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COMPENDIUM OF INFORMATION ON IDENTIFICATION AND TESTING OF MATERIALS FOR PLASTIC SOLAR THERMAL COLLECTORS

MASTER

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July 31, 1980

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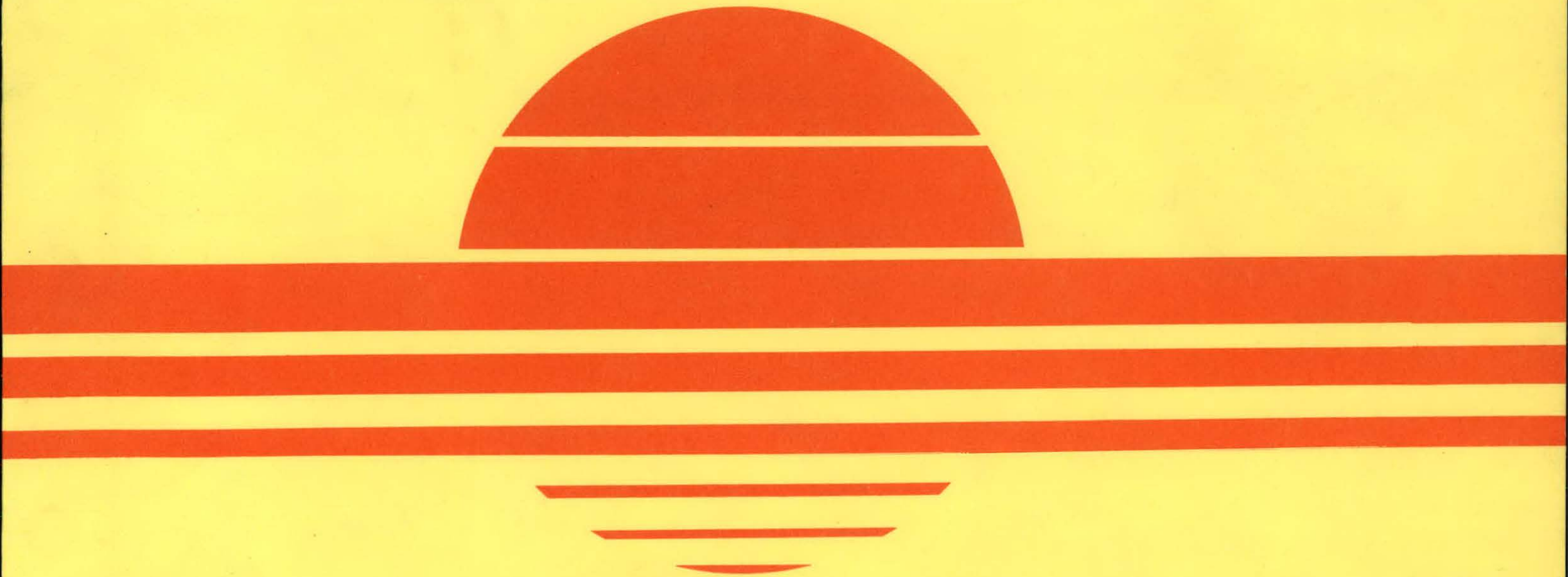

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to

U. S. DEPARTMENT OF ENERGY

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SUMMARY

This report is intended to organize and summarize prior and current literature concerning the weathering, aging, durability, degradation, and testing methodologies as applied to materials for plastic solar thermal collectors.

Topics covered include

- Rate of aging of polymeric materials
- Environmental factors affecting performance
- Evaluation and prediction of service life
- Measurement of physical and chemical properties
- Discussion of evaluation techniques and specific instrumentation
- Degradation reactions and mechanisms
- Weathering of specific polymeric materials
- Exposure testing methodology.

Major emphasis has been placed on defining the current state of the art in plastics degradation and on identifying information that can be utilized in applying appropriate and effective aging tests for use in projecting service life of plastic solar thermal collectors. This information will also be of value where polymeric components are utilized in the construction of conventional solar collectors or any application where plastic degradation and weathering are prime factors in material selection.

One important result from this study is the realization that predicting service life for materials under multiple stress conditions is a difficult task. Most published weathering studies use single stress environments and types and sizes of samples have varied to the extent that extrapolation from one set of test conditions to another has very little quantitative meaning.

The term "weatherability", although commonly used in plastics technology, is a poorly defined concept, since it refers to long-term service under complex and variable conditions and because different properties of a plastic material are affected to different degrees by a given environment. Improvement in the ability to predict weatherability therefore requires careful redefinition of the problem as well as a refinement of experimental

techniques. This review shows that no simple correlation exists between outdoor and artificial test exposure of plastics. Further, because the rates and mechanism of deterioration are different when produced by visible light, ultraviolet, heat, or moisture, an arbitrary "accelerated" weathering test will distort the balance of response observed in the slower outdoor exposure of plastics.

There is only limited information available about long-term materials problems certain to be associated with plastic materials in solar applications. Polymeric materials can exhibit long life under solar exposure alone and there is evidence to support the premise that plastic solar collectors can be designed and built to survive multiple stress environments for periods long enough to allow effective and economic use. Conditions and environments existing when applied to plastic solar collectors are generally not directly applicable to current plastic test data and the collectors and plastic components must be investigated under actual collector operating conditions to be realistic.

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INTRODUCTION

Plastics, coatings, adhesives, and other materials are all attacked by the elements of weather(1,2). Evaluating and solving these material weathering problems is not a simple matter. There are at least three main reasons for this. First, there are a large number of organic-inorganic materials and formulations, all of which are affected in different ways by the elements of weather. Second, there is a lack of standardized methods for determining how weather affects the mechanical, physical, and chemical properties of various organic-inorganic materials. Third, the combination of stresses associated with the elements of weather and their effects on materials are not well understood.

The following discussion will attempt to organize prior and pertinent literature studies concerning the weathering or durability of materials in order to select candidate materials and develop an acceptable testing methodology for analyzing plastics, coatings, and adhesives for use in plastic thermal solar collector systems.

The term "aging" as applied to a plastic solar thermal collector system refers to a change with time in some chemical or physical property of all the materials or components utilized in the solar collector which results in a decreased performance capability or decrease in expected service life of the total system. Factors most associated with the rate and extent of aging of all materials include climatic exposure and applied stresses (internal, external, sustained or cyclic).

The prediction of the rates and effects of aging is necessary to determine the proper types of usage of materials, components and systems in specific exposure environments.

Laboratory testing techniques are utilized as quality control procedures or are used to accelerate the natural aging processes of materials in order to estimae their durability or service life performance. The goal of accelerated aging is to produce, in a much shorter time, the changes in a component or material that would occur in real time of extended duration. Thus, an accelerated aging procedure which fulfills its goal would permit short-term test results to be extrapolated to long-term performance.

In designing any aging experiment under overstressed conditions (accelerated testing), a major consideration is choosing a set of environmental parameters that will accelerate those degradation rates of materials or interfaces that control failure under normal stresses. Most often, it is impractical to set up and control all environmental parameters in a test. Ideally, one would like to know a priori the dominant failure mode in the device or material. Under such circumstances, the more that is known about the behavior of the material or device, the better one can pick those environmental parameters and their magnitudes that will bring about the normal-stress failure. In an absolute sense, an acceptable accelerated test design does not require a knowledge of cause-and-effect directly; only an acceptable correlation between service life or rate of aging and some measurable quantity(ies) is required.

I. Rate of Aging of Polymeric Materials

The rate of aging of polymeric materials must be established in order to estimate when a critical property or characteristic of the material falls below an acceptable performance level under specific, general, and actual exposure conditions.

In order to establish rates of aging for materials, various testing techniques are utilized and changes in physical or chemical properties are quantitatively measured over a given time period. Some of the important parameters to be considered are the nature of the polymeric material under test and the applied stress factors such as exposure, climate, and load conditions. Another important consideration in establishing the rate of aging for materials is the complete characterization of the initial properties of the materials to be tested before application of the stress factors.

The service life of a material may be classified by one of four modes of performance for a given time period, as illustrated in Figure 1. In the figure a measure value of a property of a material relating to its intrinsic performance characteristics, e.g., hardness, extension, transparency, etc, is measured over a certain time period for a given set of applied stress conditions. In using this technique as a measure of service life, a predetermined level of performance ("acceptable performance level") for a particular property is usually identified above which the material conforms to a certain specified criteria.

A material which is very stable with respect to a given property shows essentially no change occurring with time and always exhibits an acceptable performance level (Curve A of Figure 1).

Materials which undergo a rapid initial increase in the value of a desirable property, but then adjusts to its environment and gradually decreases in performance level, is depicted in Curve B of Figure 1.

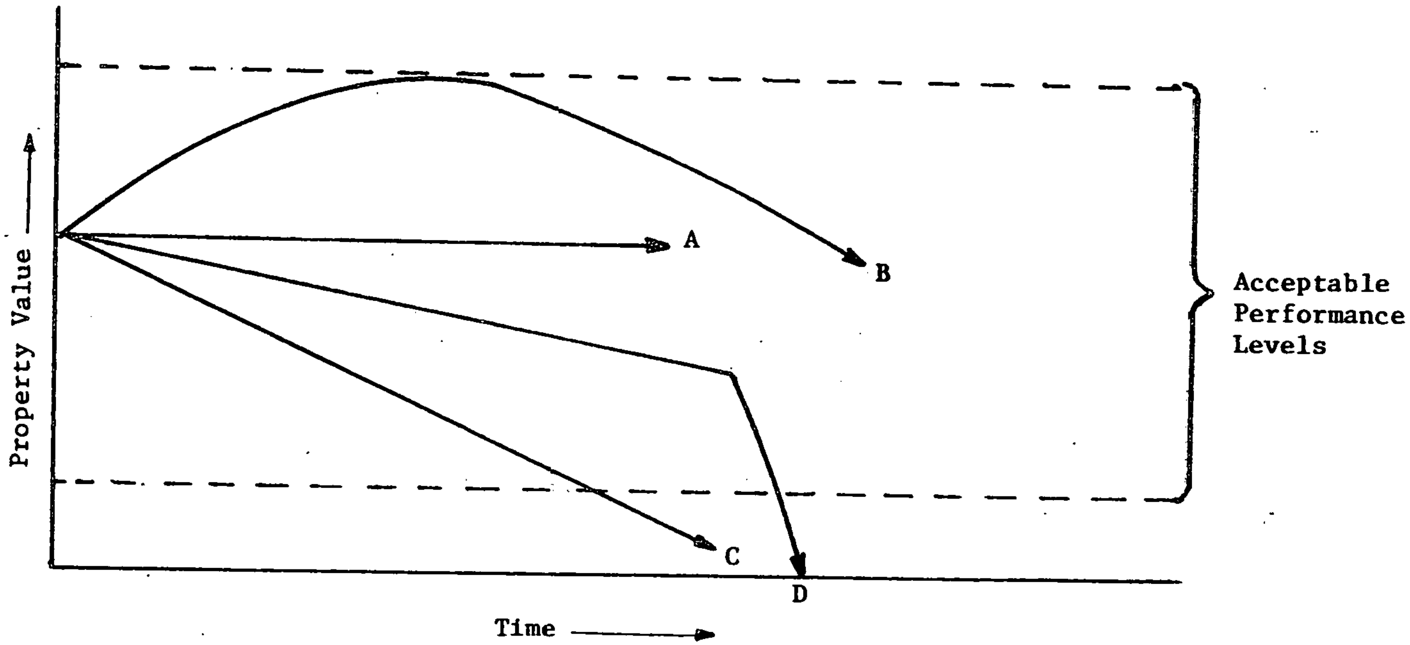

FIGURE 1. HYPOTHETICAL PERFORMANCE CURVES

Curve C illustrates a material the performance properties of which degrade linearly with time. If the slope of the line is known, durability can be predicted. Gray and Cadoff(3) found that a plot of color change versus outdoor exposure time in months was approximately linear for rigid polyvinyl chloride (PVC) polymers.

Curve D illustrates a common type of durability curve. After an initial decrease in property value, it tends to level off until some point in time where a sudden break occurs and the property drops below the acceptable performance level.

II. Environmental Factors Affecting Performance

Three key factors that may degrade the performance capabilities of a plastic solar collector component or material are (1) physical factors, (2) chemical factors, and (3) microbiological attack.

Materials such as plastics, adhesives, and coatings are composed of organic compounds. In general, organic compounds are susceptible to chemical reaction with elements and compounds such as oxygen and water, and these reactions can lead to aging. The warping in plastics, brought about by mechanical stress, is a good example of physical factors that lead to changes and aging. Fungi is an example of the third possible mechanism for degradation and microbiological deterioration. Fungi can cause damage to solar collector materials composed of organic compounds.

Accelerated aging tests are designed to yield results which can be extrapolated to some in-use time. Environmental (primarily climatic) factors that are considered to be important in the degradation process are normally incorporated in these aging tests. Some examples of environmental factors which may be causes of degradation are temperature, moisture, oxygen, ultraviolet radiation, wind, dust, sand, pollutants, ozone, salt, acids, alkalies, rot organisms, mildew, bacteria, etc. Moisture, temperature, and ultraviolet radiation are probably the principal factors, with the other factors having somewhat lesser effects. In specific climatic or geographical areas, individual factors may assume greater importance.

For example, salt spray is an important factor near an ocean as are wind and sand in desert areas and chemical pollutants in industrial areas.

(a) Effects of Moisture

Moisture, in its role in reactions based upon hydrolysis, may lead to the degradation of certain materials. Often certain hydrolyzable groups such as amides, nitriles, esters and acetals are contained in organic polymers. However, in many instances, the physical nature of absorbed moisture is more important as a degradative influence. For example, moisture absorbed in a glass-fiber reinforced polyester (GRP) material may not be detrimental in chemical deterioration, but when freezing and thawing occurs, the expansion and contraction cycling may result in a mechanical separation of the GRP fibers.

Moisture may also result in chemical corrosion of the skin material or deterioration of the core material. Vapor penetration in sandwich construction can cause delamination of the skin material by weakening the adhesive joint.

Many test methods include exposure to moisture, since moisture is an important factor in aging. Some examples are humidity chambers and light and water exposure apparatus.

(b) Effects of Temperature

The temperature changes to which solar collector components are subjected through normal seasonal changes result in substantial movements of these components. The expansion and contraction observed with temperature changes are material dependent, and restraint of component movement can result in increased stresses in a collector system.

Data published by the National Oceanic and Atmospheric Administration (NOAA) in Climatological Data - National Summary, show that normal daily temperature variations of 25 to 35 F are not uncommon in most parts of the United States. Also, recorded temperature extremes are often very large. For example, Havre, Montana, has experienced temperatures varying from a high of 111 F to a low of -57 F -- a difference of 168 F! The average temperature variation in Havre due to seasonal changes is 56 F from a high of 70 F to a low of 14 F.

Cullen(4) notes that a roof exposed to solar radiation absorbs heat, resulting in a surface temperature higher than that of the ambient air, and states that surface temperatures on outer housing components as high as 165 F are not unusual. Cullen also states that, by radiative cooling, the temperature of a roof surface at night can drop below the ambient air temperature.

The temperature changes observed in solar collector components and materials may therefore be quite large on a daily basis and even more so on an annual basis.

(c) Effects of Ultraviolet Radiation

Sunlight considerably affects materials such as coatings and adhesives, as well as those composed of plastics, and a deterioration in appearance and very often in function takes place. Many coatings fade, crack or chalk when exposed to ultraviolet radiation. Such failures are not only unsightly but may also result in the loss of serviceability, due to the fact that the protective function of nonstructural components such as coatings is largely to protect other components and this function can be severely hindered by degradation.

By definition, the solar constant is approximately 1.92 cal/min cm^2 and is the energy falling on 1 sq cm area at normal incidence, outside the earth's atmosphere, at the mean distance of the earth from the sun. Energy distribution is irregular in various parts of the spectrum, with 53% in the infrared (0.75 to 25 micrometers), 43% in the visible (0.38 to 0.75 micrometers), and 4% of the total energy falling in the ultraviolet (0.20 to 0.38 micrometers). The energy of the solar radiation decreases as the wavelength increases so that the ultraviolet range is the primary zone of photochemical action. Radiation energy is considered to be the primary portion of the sun's energy spectrum which causes materials degradation and in the ranges listed above, its energy is sufficient to break the chemical bonds(5) in the polymers of paints, plastics, and associated materials.

(d) Mechanical or Stress Factors

An important factor in the aging of structural materials and components is the presence of various types of loads in-service. Generally, the structural component must support a given load due to the dead weight of the structure. As the live loads applied to the structure change, the actual load will change, with large increases occurring when wind and snow loads are applied.

Quite often the change in the load-carrying capacity of the material is used as a measure of durability. However, most durability tests for structural materials do not require an applied load during the test. For example, in adhesives, the effect of load is included in the durability test by requiring that a dead load be applied during the test. This, however, does not account for the fluctuating or cyclic nature of the load in the actual structure.

(e) Classification of Climates

There are many different generic types of climate in the United States; however, existing accelerated aging procedures have not been designed to account for these climate variations. It is necessary to distinguish and identify types of climates to better understand the importance of specific climatic conditions on the deterioration of polymeric materials. Six major divisions of climate are defined in a survey done by Bryson and Hare.(6) In this survey, the climates vary from tropical and desert to polar climates; all divisions are found in the United States.

The correlation developed between the accelerated test results and natural aging in specific environment of ultimate exposure is the key value of any accelerated aging procedure. As new procedures for accelerated aging are developed, the varying weathering factors which are important to the aging of plastic solar thermal collector components and materials in specific climates must be defined and incorporated. Therefore, studies are needed to define these factors and to determine how they may be feasibly considered in any techniques used in accelerated aging.

A Battelle study(7) determined the frequency and duration of climatic or other environmental conditions that a related solar energy application (photovoltaic cell solar array technologies) would be expected to experience over a 20-year lifetime at various geographic locations. This extensive report identified certain environmental factors and levels that could be used in testing polymeric materials and systems for such terrestrial exposures.

The methodology developed in this report was applied to historical data for the 10-year period 1965-1974 for nine geographic locations representing widely different environmental conditions in the United States:

- Albuquerque, New Mexico
- Bismarck, North Dakota
- Boston, Massachusetts
- Brownsville, Texas
- Cleveland, Ohio
- Fairbanks, Alaska
- Los Angeles, California
- Miami Florida
- Phoenix, Arizona.

Climatic data (air temperature, relative humidity, wind speed, precipitation, etc), insolation data (total horizontal), and air pollution data (SO_2, CO_2, total oxidants, etc) were merged, whenever possible, to give sets of measured values at actual corresponding times at 3-hour intervals. Over 7×10^6 measurements were processed in making the analyses in this study.

The merged data for the geographic locations were used to generate 10-year descriptive statistics, diurnal statistics at 3-hour intervals, and statistics of weather events, in addition to the statistics related to durations, and transitions for selected combinations of environmental variables. Representative results on the following combinations of variables are included in this report:

- Air temperature, relative humidity, wind speed, and total insolation
- Air temperature and weather event (fog, rain, snow, etc)
- Air pollutant (SO_2, NO_2, total oxidants) and weather event (fog, drizzle, rain, etc).

To demonstrate feasibility, the methodology was applied to the following additional combinations of variables:

- Wind speed, wind direction, and weather event (fog, rain, snow, etc)
- Air temperature, relative humidity, wind speed, and computed values of direct insolation
- Air temperature, total insolation, and weather event (fog, rain, snow, etc)
- Air temperature, relative humidity, and air pollution (CO, SO_2, NO_2, total oxidants, and soiling index).

This methodology was also applied to identify test conditions that can be used to evaluate polymeric materials and systems, and provided procedures by which test conditions can be selected and evaluated using historical environmental data for each geographic location of interest. These procedures result in weighting factors that can be applied to experimentally determined degradation rates to obtain values for the expected total degradation for each location. In general, different weights, and hence different values, are obtained for the expected total degradation at each location.

It was noted that environmental characterizations were limited by the historical data available. For most geographic locations, for example, it was found that (1) virtually no solar spectral data in the ultraviolet range are available; (2) total horizontal insolation data, if available, are frequently of questionable validity; and (3) air pollution data are usually intermittent and since they are taken at sites where high concentrations are expected, they are not representative of suburban and rural areas. However, it was found that available historical data can serve as an effective basis for planning and analyzing laboratory tests and field tests of polymeric materials for solar collector systems.

The Battelle report also contains detailed information relating to terrestrial service environments and test conditions. Detailed climatological descriptions of nine geographic locations are given as well as the complete diurnal statistics obtained for each of the nine geographic locations. Other additional information contained in this reference is as follows:

Ultraviolet Radiation
Data Sources
Forecasting
Data Management
Meteorological Definitions
Derivation of Test Methodology Theory.

III. Estimation of Service Life on Durability of Plastic Thermal Solar Collector Components through Measurement of Physical and Chemical Properties

The starting point for service life prediction of polymeric materials begins with subjection of the material to an exposure condition (outside or in the laboratory) followed by detailed examination to determine what change may have taken place as a result of the exposure test condition. The observed changes are quantified with respect to time and are used to estimate the service life of the material. In these estimations it is important that the tests or evaluation techniques measure the extent of change in selected properties (physical or chemical) related only to the intrinsic serviceability of the material and not superficial properties or appearances (8,9).

(a) Physical Properties

Usually the first sign of deterioration or change in appearance of a plastic material is noticed through visual inspection. This is especially true in cases where climatic conditions result in changes in surface appearance such as fading, discoloration, chalking (intense ultraviolet exposure), cracking (extreme temperature cycling or hail impingement) and dirt retention (pollution or dust environments).

Properties other than appearance which may be important parameters in evaluating the durability of plastic components include hardness, softening point, gloss retention, permeability, water solubility, and others. Impact resistance is important as are stiffness, compressive, shear and tensile strengths when evaluating the durability of these components (10,11).

(b) Chemical Properties

In addition to measuring physical properties, it is also possible to measure chemical properties and attempt to relate them to service life. Aging in plastic materials is normally attributed to a chemical degradation mechanism, so that by measuring the rate of the degradation reaction one can attempt to extrapolate the results to predict long-term degradation.

Gray and Wright(12) developed a colorimetric method for measuring polyester degradation due to accelerated and natural weathering. Wallder(13) described a method using multiple internal reflection spectroscopy in the infrared region to detect and measure the carbonyl molecules which are products of polyethylene oxidation. He found the aging period necessary to detect meaningful changes to be short -- on the order of 40 hours outdoor exposure. A method(14) was developed for detecting hydroperoxide groups, which served to indicate chemical degradation, during the early stages of polyethylene oxidation.

IV. Evaluation Techniques and Instruments for Detection and Measurement of Degradation Processes for Polymeric Materials with regard to Prediction of Service Life

Molecular-level degradative changes in polymerics are, at some total integrated level, generally accompanied or followed by changes in morphological properties (e.g., such properties as crystallinity and orientation). The latter may be thought of as property changes at the microscopic level. At still higher levels of degradation, these molecular- and microscopic-level changes manifest themselves as macroscopic changes, e.g., changes in tensile strength, elongation, hardness, and heat distortion temperature.

As has been indicated earlier, measures of molecular- and microscopic-level changes, both of which occur earlier in the degradative process than macroscopic changes, are generally of more importance as early predictors of degradation. Consequently, diagnostic tools that manifest such changes must, at the same level of instrument sensitivity/precision, be of prime importance in studies directed toward prediction of service life. Thus, in such studies it is important that diagnostic tools be identified that can be used to quantify materials changes which are clearly precursors to failure <u>extremely early</u> in the degradation process, i.e., primarily at the molecular and morphological (microscopic) levels.

Some of the techniques and instruments used to detect degradation or changes in the physical properties of polymers are given in Table 1.

(a) Chemical Measurement Techniques

In the chemical techniques category, analytical instruments capable of detecting chemical changes related to degradation of components were investigated. These include such things as carbonization of polymeric surfaces due to oxidation and chain scission of polymers.

The following analytical instruments are considered most suitable for detection of degradation of solar collector components:

- Fourier Transform Infrared (FTIR) Spectroscopy including Attenuated Total Reflectance (ATR)
- Electron Spectroscopy for Chemical Analysis (ESCA), X-Ray Photoelectron Spectroscopy (XPS)
- Auger Electron Spectroscopy (AES)
- ESCA, or XPS, and AES in conjunction with ion sputtering for depth profiling
- Chemiluminescence (CI)
- Gel Permeation Chromatography (GPC)
- Combination of Mass Spectrometry/Gas Chromatography (MS/GC)
- Surface Energetics.

(1) <u>Fourier Transform Infrared (FTIR) Spectroscopy</u>. Infrared spectroscopy is based upon the interaction of infrared electromagnetic radiation with matter. This interaction results in absorption of certain wavelengths of radiation, the energy of which corresponds to the energy of specific transitions between various rotational or vibrational states of

TABLE I. TECHNIQUES AND INSTRUMENTS USED TO DETECT PHYSICAL AND CHEMICAL CHANGES IN POLYMERIC MATERIALS

Chemical

Chromatography
Wet Chemistry
Mass Spectrometer
Emission Spectrometer
EPR
NMR
Mossbauer Effect
Thermal/Vacuum Analytical Techniques
SIMS
Neutron Diffraction
X-Ray Diffraction

Electron Microprobe
IR Techniques (FTIR, etc)
Auger
Chemiluminescence
XPS/ESCA
Fluorescent Probe
Electron Stimulated Desorption Spectroscopy
ISS
LEED
Raman Spectroscopy

Electrical

Dielectrometry

Resistivity

Mechanical

Rheology
Bond Strength
Elasticity
Tensile Tests

Impact Tests
Acoustical Effects
Flexural Strength

Optical

Spectroscopy
Microscopy
Haze/Gloss Measurements
Optical Wave Guide Effects
Ellipsometry
Reflectance
Absorptance
Scattering Phenomena

Photoelasticity
Magneto-Optical Effects
Piezo-Optical Effects
Laser Diffraction
SEM
Holography
Optical Multichannel Analysis

Thermal

Thermal Conductivity
Thermal Expansion Coefficient
Heat Capacity

Thermal-Analytical Techniques (DSC, DTA, etc)

Other Physical Techniques

Water Vapor and Gas Permeability
Profilometry

Photoacoustic Spectroscopy

the molecules or groups of atoms wthin the molecule. The IR-absorption spectrum of a material is a measure of the fraction of the incident radiation absorbed as a function of wavelength. The magnitude of the absorption can be related to the concentration of specific absorbing groups and, therefore, IR spectroscopy is a quantitative tool. In polymeric systems, infrared spectroscopy can be used to measure the degree of regularity of the arrangement of macromolecules (i.e., degree of crystallinity or orientation of stretched film or fibers). Number average molecular weight and degree of branching can also be determined(15).

Conventional IR spectroscopy can be used as a quantitative tool for studying degradative changes; however, Fourier transform infrared spectroscopy offers the potential for significantly higher precision in characterizing the small changes that are encountered during the service life of a plastic material.

The photodegradation of most organic polymers results in the formation of carbonyl groups. Since the oxidation is diffusion controlled, the carbonyl concentration is higher at and near the surface than in the bulk of the material. Carbonyl can be easily detected by FTIR and ATR-FTIR can be used to measure carbonyl close to the surface. Carbonyl concentrations as low as approximately 0.01 to 0.1 percent can be detected by FTIR but calibration is required for absolute quantification. The minimum amount of carbonyl change detectable has not been determined. Increases in carbonyl can be detected after a relatively short time of outdoor exposure for many organic polymers. Since the formation of carbonyl groups is felt to be a precursor to failure caused by higher levels of oxidation, FTIR should prove quite useful in service life prediction. It is applicable to any organic polymeric material used in a plastic solar thermal collector system, but appears particularly applicable to polymeric cover materials such as polycarbonate, polyvinyl fluoride, and polymethyl methacrylate. Aside from its ability to detect carbonyl groups, FTIR can also be used to follow changes in crystallinity and by using polarized light is also capable of measuring orientation changes. These capabilities are important because both types of change can occur as the result of outdoor degradation of polymeric materials and both could conceivably lead to failure.

(2) Electron Spectroscopy for Chemical Analysis (ESCA). With ESCA, also known as X-ray photoelectron spectroscopy (XPS), the sample of interest is exposed to a 10 kv source of monochromatic X-rays. This is followed by analysis of the energy spectrum of the resulting photoelectrons emitted from the sample. The method is capable of detecting elements present at an atomic fraction of 10^{-2} to 10^{-3}. The penetration depth is 5 to 15 Å for metals, 15 to 25 Å for inorganic compounds, and 50 to 100 Å for organic compounds. ESCA provides measures of binding energies of the electrons in both the deep-lying shells and valence bands, and these in turn provide information of a chemical nature about the bulk sample and, in particular, about its surface. The experiment is carried out in ultrahigh vacuum and samples are generally solids--small plates, powders, or frozen liquids.

With ESCA, the information content per spectrum is unsurpassed by any other spectroscopic technique. The levels of available information are, as pointed out by Clark(16), the following:

(1) Absolute binding energies, relative peak intensities, shifts in binding energies. Element mapping for solids, analytical depth profiling, identification of structural features, etc.

(2) Shake up-shake off satellites. Monopole excited states: energy separation with respect to direct photoionization peaks, relative intensities of components of "singlet" and "triplet" origin.

(3) Multiple effects. For paramagnetic systems, spin state, distribution of unpaired electrons (analogue of ESR).

(4) Valence energy levels.

(5) Angular dependent studies. For solids with fixed arrangement of analyzer and X-ray source, varying take-off angle between sample and analyzer provides means of differentiating surface from subsurface and bulk effects. Variable angle between analyzer and X-ray source yields angular dependence of cross sections, asymmetry parameter β, symmetries of levels.

However, if information from any one of these levels obtained by ESCA is compared with that available from the most competitive of the other available spectroscopic techniques, ESCA almost invariably has the greater sensitivity.

Unlike all of the other surface spectroscopic tools employing an excitation beam and an analyzer such as AES, SIMS, LEED, and SSP, ESCA can be applied to polymeric systems. Because the ESCA dose rate is several orders of magnitude smaller than that of the other surface methods, it is essentially a nondestructive test when applied to polymers.

Much of the ESCA work done to date on polymers has been done by Clark(16). He lists the following advantages of ESCA in studying polymeric materials:

(1) Technique essentially nondestructive.

(2) High sensitivity and modest sample requirement.

(3) Large number of information levels available from a single experiment.

(4) For solids, unique capability of differentiating surface from subsurface and bulk phenomenon. Analytical depth profiling possible.

(5) Information level such that ab-initio investigations are feasible.

(6) Data often complementary to that obtained by other techniques. Unique capabilities central to the development of a number of important fields.

(7) Theoretical basis well understood; results of considerable interest to theoreticians and may be quantified.

For polymers used in plastic solar thermal collector systems, ESCA is particularly useful for measuring surface carbonylation, which is frequently considered to be an early precursor to several kinds of failure. It has already been mentioned that these surface carbonyls can be measured with FTIR with the aid of attenuated total reflectance. Actually, however, ATR-FTIR measures to a depth of 100 to 200 Å whereas ESCA measures 5 to 100 Å. ESCA, therefore, might be more appropriate for failure modes concerned with surface and not bulk changes.

Since ESCA is a surface tool it cannot, of course, be used for <u>in situ</u> interfacial analyses. It should be helpful, however, in diagnosing failure modes after tear-down or "autopsy" analysis of failed units providing the exposed interface is not too thick.

ESCA is also useful in conjunction with ion sputtering for determining depth profiles, i.e., plots of concentration versus depth into the polymer surface. Such information is expected to be particularly useful for interfacial tear-down failure analyses. For an interfacial failure, both surfaces could be depth profiles as an assist in the determination of the cause of failure. For example, in a delamination involving two polymeric materials, depth profiling of the surfaces may help to differentiate between failure due to a low molecular weight species migrating to the surface and that due to thermal stressing.

(3) Auger Electron Spectroscopy (AES). In Auger electron spectroscopy, an electron beam (2 to 300 ev) is directed onto the surface of a test specimen where it excites electrons in the surface and near surface atoms of the sample. This excitation results in the removal or freeing of some inner shell electrons from the atoms, creating vacant states. These states are subsequently filled by outer shell electrons, and this relaxation process is accompanied by the ejection of electrons with energies characteristic of the specific energy levels (and, hence, atomic species) involved. These electrons are called Auger electrons. An Auger spectrum consists of a plot of the derivative of the energy distribution of the ejected electrons versus energy.

AES is capable of detecting as little as 0.1 percent of an atomic layer and has a penetration depth of only a few atomic layers.

AES has become a well-established technique for the qualitative analysis of surfaces, particularly metals. Its acceptance as a quantitative technique, however, has been less widespread owing to the difficulty of establishing suitable calibration standards and to the lack of a comprehensive quantitative theoretical analysis. Most attempts to quantify AES have been empirical owing to lack of knowledge of ionization cross section, effects of diffraction, chemistry, and surface roughness, the enhancement of Auger signals by backscattered electrons, the variation in average escape depth for electrons of different energies and inability of commonly used detectors to measure absolute Auger currents.

Many papers, nevertheless, report quantitative uses of AES. These results are based on suitable calibration of the Auger signal strength relative to some independent technique such as electron microprobe, ellipsometry, piezoelectric thickness monitors, and low-energy electron diffraction. West(17) has shown that the long-term (112 days) reproducibility of AES data in a study of alloys of known composition is in the vicinity of ±20 percent. In other work, Chang(18) reported variations in the reciprocal sensitivity factor of up to 30 percent from sample to sample and in different materials.

When used in conjunction with ion sputtering, AES is capable of producing a depth profile, i.e., a plot of concentration versus depth into the specimen. Such information should be especially useful in tear-down testing for failure modes. For example, in polymer-metal delaminations, an oxide film may be detected between a polymer layer and a metallization layer indicating poor adhesion between the polymer and the metal oxide.

(4) Chemiluminescence. The chemiluminescence technique measures light, generally of very low intensity, which is emitted in the course of a chemical reaction, particularly a reaction involving oxidation or degradation of materials, e.g., hydrocarbon oxidation. In these instances, part or all of the energy change of the reaction goes to activate an electronic state in some molecule rather than appearing as heat. The activated molecule returns to the normal state by emitting the excess energy as radiation. Chemiluminescence measurements are capable of giving kinetically and mechanically significant results for reactions too slow to measure by other means. The extreme sensitivity (down to reaction rates as low as 10^{-15} moles/year) of the chemiluminescence technique makes it unexcelled in cases where very slow reactions must be detected. This nondestructive technique should be useful in determining reaction rates associated with degradation of the polymeric materials in a plastic solar collector such as the cover, coatings, and adhesives. In general, for transparent materials, the technique measures degradation of the bulk material. If the sample is opaque, it measures surface degradation. Theoretically, the technique is applicable to polymer-polymer interfaces by first measuring chemiluminescence of the interface and then measuring the separate components(19).

(5) Gel Permeation Chromatography (GPC). Gel permeation chromatography is used to measure the molecular weight distribution of polymers. It involves selective adsorption of the polymer onto various size gel particles packed in a column. A differential refractometer detector and strip-chart recorder are used to record the resulting chromatogram. It is potentially useful for detection of molecular weight reduction in polymers due to degradative chain scission. Low molecular weight additives such as plasticizers and residual monomer are also detectable, since the test output is an entire distribution curve.

In spite of its apparent lack of ultrahigh precision, gel permeation chromatography provides much information on low molecular weight additives and provides a method of separation of components for use in infrared, mass spectrometry, etc. If one considers the principal types of changes that are likely to occur in a polymer during aging and looks at tools for the study of these changes, only GPC appears relevant for following chain scission and variation in low molecular weight nonvolatile components(20).

(6) Gas Chromatography-Mass Spectrometry. Gas chromatography, a well-known technique for separating volatile organic compounds, can be applied both qualitatively and quantitatively to a wide variety of materials. The technique involves the separation of components of a mixture by passing it through a column containing a stationary phase, either solid or liquid. Usually a gas is used to transport the mixture through the column. The separation is caused by differential adsorption of the various components. The detection of these components as they exit from the column may be accomplished in many different ways. This section deals only with mass spectrometric detection. Whatever type of detection is used, gas chromatography is a very powerful tool because throughput can be effected very rapidly and compounds with only very minor physical or chemical differences can be separated.

In mass spectrometry, molecules of the material to be analyzed are introduced into a vacuum chamber, where they are then ionized. The resultant ion mixture is then resolved on the basis of the mass to charge ratio (m/e), usually by a magnetic sector analyzer, and the so-called mass spectrum -- ion intensity versus m/e -- is recorded. Usually the ionization is produced by impact of low energy (50 to 100-volt) electrons on the vapor molecules. In some cases, the ions formed originally may degrade to fragment ions and neutral species. Occasionally the parent ion is so unstable that it can be detected only indirectly by analysis of the major fragments. So-called chemical ionization may sometimes be useful in avoiding such cases. In this technique a reagent gas is introduced into the ion source at a pressure of about 1 torr. Some of the reagent gas molecules are ionized by electron impact and these ions then cause ionization of the sample molecules.

Mass spectrometry is useful in itself and also in combination with gas chromatography, where it can serve as a very sensitive detector. In either case the use of modern data processing technology increases the power and usefulness of the technique. Two types of computer-based MS/GC analysis are used: repetitive scanning and selected ion monitoring. In the first, the mass analyzer repeatedly scans over a mass range selected by the operator. Scans take from 2 to 4 seconds. In a typical MS/GC run of 30 minutes, 400 or more spectra are stored in the computer. At the completion of the run a total ionization chromatogram is obtained by computer plot of the summation of the ion intensities for each scan versus scan number. The primary use of such plots is to determine which scans contain mass spectral data corresponding to each component of interest. However, since all of the data are stored in the computer, they can be examined and plotted in many different ways. A typical example is selected ion monitoring, in which the ion current at selected masses is plotted versus time (or spectrum number). These plots can be obtained either by having the computer extract the data acquired by repetitive scanning or by having the mass spectrometer continuously monitor selected ion masses. The latter technique has the advantage of better quantitative reliability and sensitivity at least 100 times better.

The usefulness of MS/GC in the study of molecular processes leading to possible degradation of plastic solar collector performance depends critically on the generation of volatile fragments as the polymer component of the collector degrades and on whether available apparatus is sufficiently sensitive to detect and measure the material that is exuded. In the case of the photolytic degradation of PMMA at room temperature(21), several volatile products have been detected. In addition to CH_4, H_2, CO, and CO_2, methyl formate, methanol, and methyl methacrylate monomers have been detected. Studies of vacuum photodegradation at 30 C have shown that at least one molecule of carbon monoxide is produced per chain scission. The work cited in Reference 21 provides encouragement to the concept that degradation of polymeric materials does produce detectable quantities of volatile products. In order to apply MS/GC to the detection of these volatile materials, specimen solar cells would have to be exposed to typical ambient temperatures and radiation in closed chambers. A constant flow of air or other gas would be maintained to sweep the volatile materials into the inlet of a MS/GC apparatus, either directly or after concentration by cold traps.

(7) Surface Energy Analysis. Wettability measurements combined with proper mathematical analysis of the data provide an excellent means of determining the cleanliness and bondability of surfaces which are to be adhesively bonded. A good example of the usefulness of the method is seen in some recent work by D. H. Kaelble.(22) In this investigation the degradation with time of cleaned aluminum alloy surfaces was followed under various controlled relative humidities. The specific measurements made were determination of contact angles of several liquids having various surface tensions and polar-nonpolar properties. In the analysis it is assumed that the various interfacial energies can be represented as the sum of a component due to polar forces and a component due to dispersion forces. The quantity of interest γ_{SV}, the surface free energy of a solid in contact with a vapor phase (in this case, air plus water vapor), is not directly measurable. However, it can be determined by means of a procedure described by Kaelble in the cited publication. The analysis is summarized below.

For a drop of liquid in contact with a solid, the following equations are valid:

$$W_a = (1 + \cos\theta)$$

$$W_a = 2[\alpha_L \alpha_S + \beta_L \beta_S]$$

$$\gamma_{LV} = \gamma_{LV}^d + \gamma_{LV}^p = \alpha_L^2 + \beta_L^2$$

$$\gamma_{SV} = \gamma_{SV}^d + \gamma_{SV}^p = \alpha_S^2 + \beta_S^2$$

$$W_a/2\alpha_L = \alpha_S + \beta_S(\beta_L/\alpha_L) \quad ,$$

where W_a is the work of adhesion, Θ is the contact angle, γ_{LV} and γ_{SV} are the interfacial energies for the liquid-vapor and solid-vapor interfaces, and $\alpha_{L(S)}$, $\beta_{L(S)}$ are the square roots of the dispersion and polar contributions to the respective interfacial energies of the liquid-vapor and the solid-vapor interfaces.

In practice, γ_{LV}^d and γ_{LV}^p are known, Θ is measured, and W_a is calculated from the first equation above. α_L and β_L are obtained from the square roots of γ_{LV}^d and γ_{LV}^p respectively, and a plot of (β_L/α_L) versus $W_a/2\alpha_L$ is made. This plot usually gives a straight line of slope β_S and intercept α_S.

Kaelbe's work on aluminum alloys was successful in showing that surface energy analysis is applicable to detailed studies of metal surface degradation. Decreases with time were observed for the polar components of γ_{SV} and a strong dependence on relative humidity of the characteristic degradation was found.

(b) Electrical Measurement Techniques

Electrical measurements on polymeric materials is another method of detecting changes in structure as a function of applied stress and time. Typical electrical measurements made on polymeric materials are dielectric constant determination, conductivity, and resistivity.

(1) Dielectrometry. Dielectrometry has proven to be a useful tool for in-process monitoring of the degree of cure of polymeric materials.(23,24) Dielectrometry is also of potential interest in studies of polymeric degradation and its relation to the lifetime of solar collector systems. In addition to being sensitive to several of the factors associated with degradation processes (e.g., absorbed H_2O, ions, polar polymeric breakdown products), it is nondestructive and applicable to all dielectric materials.

The major parameters of interest in dielectrometry are the dielectric constant, ε, and the dielectric loss tangent of dissipation, D, given by

$$D = \mathrm{Tan}\ \delta = \frac{\varepsilon'}{\varepsilon''} ,$$

where δ is the dielectric loss angle and ε'' is the imaginary part of the complex dielectric constant

$$\varepsilon = \varepsilon' - i\varepsilon'' .$$

The dielectric constant is defined as the ratio of the permittivity of a dielectric material to the permittivity of free space. It is, then, a measure of the polarizability and, hence, of the presence of polar species in the material. The loss tangent is a measure of the energy dissipated in the material as a consequence of the attempt of dipoles, contained therein, to follow the variations of an impressed ac electric field. It is, then, sensitive to factors which determine the mobility of polar species, including the properties of the surrounding medium. Either of these parameters (ε' and D) can, under proper conditions, provide a means for detecting and identifying specific polar groups which might be present as a result of environmentally induced degradative changes, impurity absorption/diffusion, etc. The loss tangent is generally the more sensitive of the two. The dielectric loss tangent is somewhat analogous to the loss tangent in dynamic-mechanical property measurements and exhibits similar characteristics behavior (e.g., temperature dependence of peaks, sensitivity to glass transition temperatures, etc) in many cases.

A study of the sensitivity of the loss tangent to the presence of known quantities (determined by IR analysis) of specific polar groups, including carbonyl groups, methyl groups, hydroxyl groups, and terminal vinyl groups in polyethylene (a nonpolar polymer) has been published.(25) Other studies demonstrating the basic applicability of the technique to structural investigations and degradation studies(26) have also been published.

Techniques used in measuring dielectric constants and dielectric loss generally fall into one of two categories -- null methods and resonant circuit methods. The various types of capacitance bridges use null methods whereby the unknown in one arm of the bridge is balanced out against impedance elements in other arms. The resolution of the balance arms and the sensitivity of the balance indicator are key factors in the accuracy of this technique. Null-type measurements are generally suitable for measurements in the frequency range of 0 to 10^7 Hz. At higher frequencies, resonant circuit techniques are more suitable. In this technique the unknown is incorporated into a tunable microwave resonator which is then tuned through resonance. The loss is measured by the width of the resonance curve. An interesting discussion of one approach to resonant circuit measurements of dielectric properties of polymers appears in Reference 27.

The key variables in dielectric spectroscopy studies are frequency (of the ac electric field) and specimen temperature. As the frequency is scanned from very low to higher frequencies, gradual steplike drops in the dielectric constant accompanied by peaks in the loss curve are observed at specific frequencies. These correspond to resonances of specific dipole species as they reach a point where maximum energy is absorbed from the field. At higher frequencies these species can no longer respond fast enough to follow the field and, hence, their contribution to the dielectric consant drops out. The presence of ions in a material can significantly alter the low-frequency loss characteristic. Varying the temperature while monitoring dielectric characteristics at a fixed frequency allows the identification of phase changes and crystal structure changes through their effects on molecular mobility. These also will be manifested as inflection changes in the dielectric constant and loss curves.

It should be noted, however, that in certain cases the outdoor performance of a plastic material may not be reflected by a change in some of its electrical properties.

In a study by NBS on the effects of weathering for 20 plastic materials, no significant change in dielectric constant was observed for these materials exposed at three different geographical locations. The dissipation factor did, however, indicate significant changes between thick and thin samples; hence, this test may be useful as an early indicator of deterioration for some materials(28).

(2) Electrical Conductivity/Resistivity. As with dielectrometry, dc electrical conductivity measurements have been used to monitor curing cycles in polymeric materials.(29) The volume conductivity of dielectric materials is sensitive to the presence of mobile ionic species and moisture, and monitoring of this parameter could provide a sensitive means of detecting significant changes in these factors as precursors to corrosive degradation, electrical shunting effects, etc. Detailed procedures for measurements on high-resistivity dielectric materials such as glasses and polymers are available from published test standards and procedures.(30) Since these materials can have extremely high volume resistivities, care must be exercised in electrode arrangements to allow distinguishing between surface and volume conductive effects. Four-point contacting is recommended where feasible.

The conductivity of dielectrics can be highly sensitive to temperature so this parameter must be monitored and/or controlled during such measurements. The temperature dependence of the conductivity of dielectrics can also reveal significant information about the conductive species present, their activation energies, etc.(31) Glow curve-type measurements, in which excited carriers frozen into trapping levels associated with structural defects and discontinuities are gradually released by raising the temperature, thereby giving discrete peaks in the conductivity at characteristic temperatures, can also be used to derive information about the nature and density of microscopic defects.(31,32) This type of measurement could be useful in degradation diagnostic studies, although other available laboratory techniques might provide more direct and easily interpretable identifications.

(c). Optical Measurements and Techniques

Visual observation of defects in environmentally plastic materials is the most direct method of detecting change in a solar collector system. Some of the basic optically observable degradation processes are as follows:

(a) Overall spectral transmission of a plastic material. It is of fundamental importance to know the intensity and spectral distribution of light reaching the solar thermal collector surface in order to be able to ascertain what part of a change in collector efficiency output may be attributed to optical property changes and what part to other causes.

(b) First-surface damage. Damage to the first surface may be a serious factor in degradation of output. It might result from abrasion by wind-driven particles, by chemical attack by atmospheric constitutents - SO_2 for instance - or even from repeated washing.(33) Included in this category are "remediable" occlusion of the surface by dust, dirt, or grime.

(c) Delamination. Plastic solar thermal collector systems may be of a composite design; hence, delamination of components could be a major source of failure under exposure conditions and be defined as the formation of voids at interfaces of these components. The details and fundamental causes of delamination of plastic composites are complicated, but can be studied optically.

(d) Discoloration. The principal types of discoloration that may be encountered are UV-induced yellowing of polymers. Observable yellowing of polymers upon long UV exposure appears(34) to represent the "tail" of stronger absorption changes occurring at the shorter wavelengths. It should be detectable well in advance of any obvious degradation effects simply by measuring the change in UV absorption.

(e) Oxidation. Permeation of water vapor or other gases may induce chemical changes, for instance, the formation of carbonyl bonds in polymers or additives. Haze or opacification of a plastic material is usually first detected with optical measurement techniques.

(f) Strain. Changes in strain, and thus in birefringence, are obviously likely to occur wherever interfaces occur, particularly in the presence of thermal cycling, clamping, or gas permeation. The changes in strain of greatest interest are those for plastic solar thermal collectors.

The capabilities and limitations of optical techniques for measurement of environmentally produced changes of the type of interest here may perhaps be brought out by some elementary discussion of optical instruments in general. Fundamentally, there are only one or two broad categories of these optical instruments. An input beam (or set of beams) characterized by a particular wavelength, intensity, polarization state, and direction impinges on some sample. Some or all of the same characteristics are measured on an output beam (or beams) reflected, transmitted, or scattered by the sample. If the input and output beams are reasonably coherent, their relative phase can also be determined, leading to the interferometric class of instruments. In principle, with a sufficiently large set of measurements, rather complicated sample systems can be analyzed optically; that is, from our point of view, the important environmentally induced changes in optical properties of the materials in the sample system can be detected and correctly differentiated. For example, in any interferometric experiment, anything occurring to cause a change in optical-path length anywhere in the system will cause a phase difference at the detector. Such a path-length change might be due to flow-induced change in strain birefringence of a polymer, to delamination, to a refractive-index change caused by a chemical reaction, or to thermal expansion. As another example, in a simple absorption measurement, a change in transmitted intensity might result from a darkening of one or more components induced by UV exposure, from an increase in light scattering owing to abrasion of the first surface*, or from a change in reflectivity caused by delamination or possibly a change in strain birefringence. The various contributing effects might be separated out by additional measurements. In the second example, UV-induced yellowing would have a fairly characteristic spectral signature, light scattering

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* The "first surface" is defined as that which is ordinarily exposed to the atmosphere, and which is the first system interface encountered by the incident beam.

could be monitored directly, and one could measure specularly reflected as well as transmitted light. But it is not desirable to make all of these measurements, and possibly more, in every test. Moreover, abrasion-induced scattering will affect the measured reflectivity, delamination in small spots will affect the measured scattering (so will absorption if it is large enough), and so forth. This deconvolution problem is complicated by the paucity of baseline data which would help in setting up optical experiments in the optimum way -- for example, the geometry of delamination is not known; quantitative data on yellowing are available only for very few polymers; and so on. Also, it is desired to measure these changes at the lowest possible levels as well as at levels where obvious polymer deterioration occurs in order to make the most reliable possible life predictions and to indicate areas where preventive measures may be required. On the basis of these arguments, the following conclusions have been reached:

- For plastic solar thermal collector assemblies to be exposed to a variety of influences, such as by natural weathering, only the simplest optical measurements are likely to be useful.
- There is a need for a test program aimed not only at evaluating instruments and measurement techniques, but also at providing some useful baseline data on components and interfaces that are presently deemed most likely to see service in composite plastic collectors.

(1) Optical Methods for Measuring Overall Spectral Transmission of Plastic Materials. The objectives of this measurement are (a) to determine changes in the light intensity per unit spectral range reaching the plastic solar collector front surface and (b) to determine the change in absorption coefficient with time. The first measurement, an absolute measurement, is useful in determining at any given time what part of a change in collector thermal output results from optical factors. The absorption measurement, a relative measurement, is needed to predict the useful life of a polymer and is essential in determining the true impact of the optical-property degradation on total collector performance. An effective absorption coefficient can be determined by comparing intensity transmitted through the polymeric cover layers in a given spectral range with that from a glass

plate standard. This will be an effective, rather than "true", absorption coefficient since, intentionally, no corrections are to be made for reflection or scattering. The absolute change in transmissivity to be detected (i.e., required sensitivity) will depend on the planned allowable degradation in optical quality over the life of the plastic collector, and thus on the entire system design; for this reason the design requirements for transmissivity-degradation measurement techniques are difficult to specify. In some scenarios, as little as 10 percent overall degradation in optical throughput to the collector over a life of 20 years is required for system feasibility. In such circumstances, it would be necessary, in accelerated life testing and in field testing, to detect absolute changes in transmission of 1/2 percent, and preferably smaller, occurring over periods up to 1 year in order to meet the first objective, and relative measurements of about the same accuracy to meet the second. Particularly in view of the fact that the overall transmission will naturally be large by design, there is a difficult long-term precision problem to be solved -- the transmission at 500 nm, say, is 90 percent today, while 1 year from now it may be 89.5 percent. For short and long wavelengths, the precision necessary will not be as great, but neither will detector response unless very complicated and expensive multidetector measurements are resorted to. The most natural way to make the measurement is to use a white-light input and to measure the spectral transmittance with a spectrophotometer, since these instruments are widely available in designs well suited for field use.(35)

(2) Optical Methods for Measuring First-Surface Damage. The principal mechanisms of first-surface degradation will be abrasion, chemical attack, and occlusion. The principal optical effect of any of these mechanisms is an increase in scattered light. The principal optical methods of studying first-surface damage may be categorized as follows:

- Direct optical observation -- principally microscopy
- Enhanced direct observation -- enhanced by chemical enlargement or by decoration of defects
- Scanning electron microscopy

- Specular reflectivity, which will be reduced by the increase in scattering
- Direct measurement of scattered light.

(i) Direct Optical Observation. The first of these is a hand-held anamorphic* microscope(36) with its own light source. This instrument was developed for the rapid analysis of roughening of steel surfaces prior to painting, but should also work fairly well on glass or plastic. By the optical sectioning method (imaging the oblique reflection of a slit), average roughness to a level of around 15 μm over an aperture of 7.5 mm may be estimated. Something of this sort, which can be held and operated with one hand, might be convenient for the rapid surveying or sampling of surface damage to arrays _in situ_.

Of the various types of phase and interference microscopes**, the most generally useful commercially available type is the Nomarski differential interference contrast microscope. In this instrument, the incident light is passed through a Wollaston prism, producing two perpendicularly polarized beams traveling at a small angle to one another. After passing through a condensing lens, these beams pass through or are reflected from the sample, go through the magnifying optics, and pass through another Wollaston prism bringing them to the same polarization so they may interfere. The result is an image closely approximating gradients in the optical path. For the case of reflection from a rough surface, these gradients just result from the different distances adjacent rays travel before reflection, and one obtains a sharp topographic picture of the surface with excellent depth of field. Besides being useful as a laboratory instrument for qualitative assessment of the extent of damage, this type of microscope is useful for distinguishing different types of surface damage and for determining just what the effect on the surface has been. Binocular Nomarski microscopes with magnifications up to 1000X are available from Carl Zeiss, Inc. The instrument can also be used as a conventional bright-field or dark-field microscope.

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* That is, one which magnifies predominantly in one dimension; for the instrument described here, 50X vertical to 10X horizontal.

** A description and comparison of various types of interference microscopes is given by Claussen.(37)

Among the direct optical techniques, multiple beam interferometry should be mentioned. With this technique, fairly abrupt surface topography features, such as steps, in the 30 to 50 Å range may be observed and measured. To apply this technique, the surface to be investigated generally must be silvered to obtain good reflectivity. This of course requires care, is time-consuming, and must generally be considered destructive, and thus the technique has not found wide popularity.

(ii) Enhanced Optical Observation. Since the object of optical examination of the specimen surfaces is to reveal defects at the lowest level possible, anything that will enhance the visibility of the defects will be welcome. Nondestructive methods are clearly preferable, but not absolutely necessary. The most obvious technique to try is etching; besides being destructive, this will be successful only in limited circumstances, particularly on noncrystalline materials such as a plastic. An example in which etching is not particularly effective is afforded by the careful work of Tomandl(38), who studied sandblasted and etched glass by both light scattering and SEM. Calculation of shapes of pits etched with HF from stereo-SEM pairs showed that these pits were shallow ellipsoidal depressions. Upon further etching, these depressions appear just to gradually enlarge without change of shape, as determined by light-scattering measurements.(39,40)

(iii) Scanning Electron Microscopy. Under the most favorable circumstances, features as small as 2000 to 3000 Å may barely be resolved using optical microscopy. To discern smaller features, scanning electron microscopy (SEM) has become the method of choice, despite the high cost of the instrument, because of its simplicity and versatility. Only "conventional" SEM for study of surface topography is considered here, other uses being more chemical or electrical in nature. Present-day commercial instruments, when properly maintained, have a lateral resolution in the range 50 to 100 Å. Because these microscopes have great depth of field, resolution in depth is of the same order of magnitude, with a suitable "viewing angle". By the use of more than one viewing angle, the detailed topography of pits, microcracks, and other surface features can be reconstructed to a high degree of accuracy, as mentioned above. Scanning electron microscopes which can generate stereo photographs in real time are now available. For SEM of dielectric materials, it is generally necessary to lightly metallize (with gold, usually) the surface to be studied in order to avoid changing it. Metallization of the sample cannot really be considered a nondestructive technique, since the characteristics of the surface and thus its future degradation rates are likely to be altered by the film deposition and removal processes. Aside from perhaps making it possible to observe the progress of surface damage resulting from weathering at an earlier stage than otherwise possible, the chief use of SEM is to provide the detailed information which can assist in developing an interpretation of the nature of the damage process. SEM is not suitable for investigating defects in depth because of the charging problem and the energy straggling of the electron beam inside the solid material.

(iv) Specular Reflectivity. As stated above, the principle of these measurements is simply that light which is scattered is not specularly reflected, and the drop in reflectivity is generally large enough to measure.

Abrasion of plastic surfaces of only 28 Å in roughness depth is sufficient to cause a reasonable drop in reflectance(42); hence chemical attack or physical abrasion on a surface can be characterized by simple reflectivity measurements.(41,42,43)

(v) Direct Measurement of Scattered Light. The most obvious technique for this purpose can be carried out successfully by straightforward bistatic reflectometry(38) or by a number of other techniques surveyed by Eastman(41). In reflectometry, the surface is generally metallized(38), both to increase the reflectance and to eliminate problems from scattering from other interfaces and bulk materials in the system. This and the other methods generally require very careful work and/or very specialized instruments in order to get reliable quantitative scattering data. Even given the data, relating it to the statistical surface roughness is a difficult task, as a review(44) of the theoretical approaches that have been tried reveals. Overall, a combination of the other methods described seems more generally useful than direct scattering studies. Small-angle forward scattering measurements are of special interest though, since light scattering owing to first-surface deterioration is likely to complicate the study of light scattering due to other dauses, such as delamination. It is possible that the first-surface scattering might be studied under controlled conditions by use of the optical multichannel analyzer for detection, as described in the following section; whether its effects can adequately be separated from other sources of scattering is another question.

To summarize, simple specular reflectivity measurements may be satisfactory for observing the presence of first-surface deterioration at fairly low levels. Some simple experiments to verify and quantify this should be performed. Optical and electron microscopy and light-scattering measurements on the same samples would provide more detailed information on the nature of the damage, which should improve the predictive capability of the technique.

(3) Optical Methods for Detecting Delamination. The seriousness of delamination as a failure mode in composite plastic solar thermal collector systems has been already referred to several times. It is not only of consequence in itself, but also as a precursor of other modes of degradation and failure, since delamination may open pathways for chemical attack.

From the optical standpoint, the simplest delamination model consists of replacing a plane interface, between say Materials A and B, with two interfaces separated by distance d with "vacuum" or air between them. Also, in its incipient stages the delamination may not occur uniformly over a sizable area, but rather may take place at numerous small spots.

The methods that have been examined for optical observation of delamination are

- Specular reflectivity(45,46)
- Ellipsometry(47,48)
- Holographic interferometry(49-55)
- Light scattering(56).

(4) Optical Methods for Measuring Discoloration. The question that comes to mind concerning discoloration of plastics is whether the color changes involved can be detected at low levels by some method other than direct measurement of the absorption changes involved, such as a change in reflectance.

To get a feeling for the time scales involved, the results of Oster et al(34) on absorption of 12-μm-thick Saran film irradiated with UV at 2540 Å for various times were considered. The solar UV radiation reaching the earth's surface will of course be peaked at longer wavelengths and thus will have a lower quantum efficiency in producing yellowing than the source used in the experiments. To avoid problems in extrapolating the data to long wavelengths, a measurement wavelength of 4750 Å was assumed. Neglecting a small background absorption, the absorption cross section at this wavelength is approximately 0.67×10^{-22} cm^2/photon. If all air mass 2 solar photons with wavelengths shorter than 3200 Å were as effective in darkening the plastic as the 2540 Å photons were, a 12-μm Saran sheet would become completely opaque (<0.1 percent transmission at 4750 Å) in about 16 months' outdoor exposure, assuming no other chemical changes. Since the actual quantum efficiency will be much lower, obvious yellowing over a 5 to 10-year life range may be anticipated, showing the desirability of accelerated tests. Some data on exposure of polystyrene (very photosensitive) and polymethyl methacrylate to sunlight for 5 to 31 months are given by Winslow.(57) Epoxy adhesives are particularly apt to degrade in sunlight, according to data of Mauri.(58)

In laboratory experiments it is usually possible to monitor the absorptance of a material at some wavelength in the ultraviolet (say 0.3 μm) where the optical density changes from UV exposure are much greater than those in the visible spectrum. If the data on Saran is any guide, it should be possible to detect color changes this way in a time span of 10 to 20 percent of that required to observe them directly in the visible spectrum.

(5) Optical Methods for Measuring Oxidation. The oxidation of polymers proceeds by a complicated and imperfectly understood chain of reactions(57), requiring radiation to produce free radicals as well as oxygen or another oxidative reactant. The reaction propagates, though without multiplying; so one initial radical may lead to consumption of hundreds of oxygen molecules. Fortunately, it is not necessary to address the reaction details, but only to consider the net effect, which is to replace one hydrogen attached to a carbon with a hydroperoxide (OOH) radical. At the interfaces where we expect oxidation primarily to occur, these peroxide radicals will be very unstable and will soon be replaced by more stable doubly bonded oxygen (with evolution of water) or possibly by a hydroxyl group (with reevolution of oxygen). Such characteristic groups could of course be detected by infrared absorption spectroscopy; the possibility of detecting them by their effects on the refractive index in the visible spectrum will be examined here.

Such changes are easily estimated for polymers using the concept of molar refractivity. There are various approaches to this concept; the Lorenz-Lorentz type formulation is used here.(59) According to this formulation, the refractive index of a polymer at some characteristic wavelength (usually 0.589 μm) is given by

$$n = \left(\frac{1 + 2Z}{1 - Z}\right)^{1/2} ,$$

where $Z = R\rho/M$, where R is the sum of tabulated(59) universal molar refractivities for one repeat unit of the polymer, ρ is the bulk density, and M is the molecular weight of the repeat unit. As a simple example, the refractive index of polyethylene in which various small percentages of C=O or HCOH groups were substituted for CH_2's was estimated, assuming that the density changes could be neglected. It was found that for 1 percent of the repeat units altered in one of these ways, the index change expected would be 0.005 to 0.007. In a bulk sample, this would be easily measurable, but here it is just a change in the first few molecular layers at the interface, and is well below detection by any of the methods so far discussed -- this at a rather heavy degree of oxidation. The conclusion is obvious -- measurement of optical properties in the visible spectrum is not a suitable way to study interface oxidation effects, at least not unless much larger secondary effects are associated with them.

(6) Optical Methods for Measuring Strain. As mentioned previously, sizable strains are likely to be present in the composite systems of interest here, and these strains will change with time, both as a result of environmental exposure and through slow relaxation mechanisms. Strains which might be precursors of delamination between transparent layers are of particular interest. The presence of strain is recognized optically by the presence of strain birefringence. In this section, factors involved in measuring changes in this quantity are discussed.

Polymers, as is well known, show birefringence even when they are not being stressed. This birefringence depends on the polymer production method and its whole thermomechanical history. In particular, it may result from(60)

- Preferential chain alignment from high-temperature shear
- Bond distortion from low-temperature stress, including scratching
- Presence of crystalline or semicrystalline regions
- Presence of boundaries between different types of material, as in block copolymers.

These effects may be loosely referred to as strain or intrinsic birefringence.

When a polymer sheet is bonded to another material, a stress bireringence will be added to the intrinsic birefringence. In principle, the former property could be distinguished from the latter by its characteristic dependence on the geometry of the sample and the bond, but it is not known that this has been demonstrated. Even if this composite is not subjected to any environmental stress, the birefringence may in many cases change with time as the polymer tries to relieve the mechanical stress on it. If there are environmental effects in addition -- say just moderate temperature cycling -- there will be thermally produced irreversible changes in the mechanical properties of the sample, leading to changes in the stress and strain birefringence. In addition, there will be thermal changes in the basic refractive indices and stress-optic coefficients to consider if measurements are to be made at other than room temperature. The problems involved in performing and interpreting this kind of experiment for just a single polymer, not bonded to some other material, are well set forth by Kovacs and Hobbs.(60) The field of investigation where measurements at temperature are involved is called thermooptical analysis. It is still in its rudimentary stages. The present circumstance presents a rather more complicated situation to study than those generally attacked so far, even if experiments are limited to measurements at room temperature. As has been repeatedly stressed, many of the optical measurements so far discussed will be more or less sensitive to these birefringence changes. However, some part of these birefringence changes may be characteristic of internal strain changes (which must occur) preliminary to delamination; so it might be possible to obtain useful data along these lines from any of these measurements, or perhaps from more conventional birefrigence studies, as described in References 61 and 62.

(7) Summary—Optical Measurement Techniques.

- Optical experiments to be carried out should be directed partly toward obtaining relevant optical data on materials and combinations of materials likely to be used in plastic solar thermal collector systems, as well as toward evaluating instruments and measurement techniques.
- Light-scattering measurements should be made on some materials of interest to determine whether first-surface deterioration, delamination, and some polymer aging characteristics can be detected and differentiated by this technique, alone or in combination with other measurements.
- The suitability of simple reflectance measurement for evaluation of first-surface deterioration through abrasion, etching, etc, should be determined experimentally for a variety of likely first-surface materials. Data on probable rates of surface deterioration in various locations should be gathered in order to allow useful-life predictions to be made.

- Holographic interferometry experiments should be considered with the objectives of determining trade-offs between area scanned and overall sensitivity and reliability of the technique; and determining whether strain birefringence changes, possibly precursuory to delamination, can be detected in this way.

- Optical measurements performed on complete plastic solar thermal collector systems should be limited at first to simple tests, such as spectral transmitted intensity (including UV), possibly at only a few selected wavelengths.

(d) Thermal and Mechanical Measurement Techniques

This category includes the thermal and mechanical properties of materials and related diagnostic techniques. Many of the potential solar collector failure modes are related to changes in the mechanical properties of the materials caused by environmental stresses. Mechanical properties of interest here include the elastic moduli, yield strength, ultimate strength, impact strength, fatigue, elongation, hardness, creep, and bond strength. This category also includes rheological measurements which may be useful both as an analytical tool for developing data on engineering properties, and in detecting flaws such as delaminations.

Failure modes relating to changes in thermal properties are less clearly defined. The thermal conductivity, specific heat, and thermal contact resistances will determine solar collector operating-temperature distributions. Mechanical stresses will then be determined in part by differences in coefficients of thermal expansion. The contribution of changes in these properties to plastic solar collector failure is not clearly established. In addition to the usual thermal properties, this category also includes the thermal analytical tools, such as differential thermal analysis, differential scanning calorimetry, thermogravimetric analysis, and thermomechanical analysis, and special techniques such as infrared thermography.

(1) Glass Transition-Temperature Properties in Polymers. Characteristics of major importance in polymers include the changes in properties associated with various transition temperatures, the primary ones being

the glass transition temperature (T_g) and the crystalline melting temperature (T_c).(63) Below the glass transition temperature, the materials are hard, rigid glasses, while above T_g, the individual molecules are mobile and rearrange themselves to form soft and flexible (amorphous) segments with or without attendant regularly structured hard (crystalline) segments. Thus, any material degradation which shifts the glass transition temperature through the operating-temperature range of a collector has the potential of seriously altering the performance of the collector and may lead to failure.

Many factors affect the glass transition. The most important morphological factor is the chain stiffness or flexibility. Other chemical factors include the molecular polarity and backbone symmetry. Structural factors include molecular weight, degree of crosslinking, and amount of plasticization. Nielsen(64) gives some empirical rules for estimating changes in T_g. In the range of practical molecular weights, T_g is given by

$$T_g = T_g^{\,o} - \frac{K}{\overline{M}_n} ,$$

where $\overline{M}_n$ is the number average molecular weight, $T_g^{\,o}$ is the glass transition temperature at infinite molecular weight, and K is a constant which is characteristic of a given polymer. For polystyrene, $K \approx 1.75 \times 10^5$, and using $T_g^{\,o} = 100$ C, the following numerical values may be obtained:

M_n	T_g, °C	$\frac{d\overline{M}_n}{dT_g}$
10^6	99.83	5.7×10^6
10^5	98.25	5.7×10^4
10^4	82.5	5.7×10^2

The effect of crosslinking on T_g is given by the equation

$$T_g - T_{g_0} = \frac{3.9 \times 10^4}{\overline{M}_c}$$

As may be seen from the literature, shifts in T_g of more than 80 degrees are not unusual for systems such as thermosetting resins.

Plasticizers are low molecular weight liquids (and occasionally solids) which are purposely introduced to lower the glass transition temperature. The loss of these plasticizers during environmental exposure can have a significant effect on the glass transition temperature. Mixture rules for predicting T_g for a plasticized material are

$$T_g = T_{gA}\Phi_A + T_{gB}\Phi_B$$

and

$$\frac{1}{T_g} = \frac{W_A}{T_{gA}} + \frac{W_B}{T_{gB}} \quad ,$$

where T_{gA} and T_{gB} are the glass transition temperatures of the base polymer and the plasticizers, respectively, Φ_A and Φ_B are the volume fractions and W_A and W_B are the weight fractions. Typical values of T_{gB} are between -50 and -100 C, so that the T_g of the mixture may vary anywhere from T_{gB} to T_{gA}, depending upon the amount of plasticizer.

Because of the importance of the glass transition temperature, diagnostic methods based on the measurement of this quantity should be particularly useful in degradation studies. Techniques which can be used to measure T_g include

- Differential thermal analysis
- Differential scanning calorimetry
- Thermomechanical analysis
- Dynamic mechanical analysis.

(2) Thermal-Stress Properties in Polymers. Thermal stresses usually involve quantities such as $E\alpha\Delta T$, where E is the modulus of elasticity, α is the linear thermal expansion coefficient and ΔT is the temperature change from an unstressed condition. Any changes in either E or α are thus likely to change the thermal stress patterns. Empirical rules which relate the volume coefficients of expansion above and below T_g to T_g are

$$\beta_\ell T_g = 0.16^{(63)}$$

$$(\beta_\ell - \beta_g) \simeq 0.113^{(64)}$$

where β_ℓ and β_g are the coefficients of expansion above and below T_g, respectively. These rules taken together predict that $\frac{\beta_\ell}{\beta_g} \simeq 3$ to 4. While this change in expansion coefficient may be significant, it may be overshadowed by the much larger changes that can occur in the modulus of elasticity as the T_g is shifted.

Nielsen(64) has presented graphs which show the effects of several variables on the shear modulus of polymers. These graphs showed the effect of molecular weight on the modulus temperature curve for an amorphous polymer such as normal atactic polystyrene. At the glass transition temperature, the modulus drops by about three orders of magnitude. Below T_g, molecular weight has practically no effect on the modulus. For sufficiently high molecular weights, T_g and the drop in modulus are also independent of molecular weight. Above T_g, the molecular weight has a larger influence, as the tendency for viscous flow becomes more pronounced.

The effect of crosslinking on the modulus temperature curve is also important in that first, the modulus above T_g is greatly increased as crosslinking progresses and, second, the glass transition temperature shifts to higher values with increased crosslinking, as was discussed earlier. Finally, the transition region is broadened with increased crosslinking. Below T_g, crosslinking has little effect on the modulus. The shear modulus of certain polymers is essentially unaffected by crystallinity below T_g, but increases markedly with increasing degrees of crystallinity above T_g. For the most part, polymeric solar collector materials will be noncrystalline since most semicrystalline materials are opaque (ruling out top-cover applications) and have properties which are particularly sensitive to thermal history. However, where crystalline or crystallizable polymers are used, the effect of crystallinity on modulus and other physical properties will be significant.

The response of a solid under the influence of an oscillating or otherwise time-dependent forcing function is governed by a complex modulus $G^* = G' + iG''$. The imaginary term gives rise to a damping or dissipation of energy, while for small to medium damping, the real term is the same as the modulus measured by other methods. The ratio of the loss modulus, G'', to the real modulus, G', is called the loss tangent and is defined by

$$\tan \delta = G''/G' \quad .$$

Another convenient damping term is the logarithmic decrement Δ which is related to the peak amplitudes of successive damped natural vibrations A_1 and A_2 by

$$\Delta = \ln \frac{A_1}{A_2} \quad .$$

In terms of the complex modulus, Δ is given by

$$\Delta \simeq \pi \frac{G''}{G'} \text{ for } \Delta<1 \quad .$$

For a given polymer, the complex dynamic modulus depends on both the temperature and the frequency of oscillations. For most polymers, T_g and the damping peak are increased about 7 C for an order of magnitude increase in frequency.

Change in damping resonse of a polymer is also reflected by differences in its molecular weight distributions. Above T_g, the damping depends strongly on molecular weight, and the value of damping at the minimum in the curve may be used as a measurement of the number average molecular weight.

Many polymers exhibit additional damping peaks at temperatures below T_g. These are called secondary glass transitions. Prominent secondary glass transitions are usually found for tough, ductile, glassy polymers and those with high impact strength, so that changes in these secondary transitions may be indicators of changes in ductility and impact strength.

Nielsen gives approximate equations by which dynamic mechanical data taken over a range of frequency can be used to calculate the creep compliance and stress-relaxation modulus, which are useful engineering properties.(64)

The melt viscosity of a polymer which is important in manufacturing processing conditions can be extremely sensitive to changes in molecular weight(63). Above a critical molecular weight, M_{cr}, the melt viscosity may be represented by the equation

$$\log \eta = 3.4 \log \overline{M}_w + A \quad ,$$

where A is a constant which is characteristic of a given polymer, but is independent of molecular weight. The critical molecular weight depends upon the structure of the polymers and may vary from 2000 to 60,000. For low molecular weights, two empirical expressions are given for the viscosity:

$$\log \eta = n \log \overline{M}_w + B \quad ,$$

where $n \simeq 1$ and B is a constant, and

$$\log \eta = C_1 \left(\overline{M}_w \right)^n + C_2 \quad ,$$

where $n \simeq 1/2$ and C_1 and C_2 are constants.

A similar situation exists for the viscosity of concentrated polymer solutions. Below a critical molecular weight, the viscosity is proportional to M, while above M_{cr}, the viscosity is proportional to $M^{3.4}$. Thus, for sufficiently high molecular weights, a small percentage change in M produces a substantially larger change in η, and measurements of η would be a sensitive detector of degradation by modes in which the molecular weight is changed.

The dependence of many polymer properties upon molecular weight may be described by the relation:

$$X = X_\infty - \frac{A}{\overline{M}_n} \quad ,$$

where X_∞ is the value of the property X at infinite molecular weight and A is a constant for a given polymer. Many properties, including density and specific heat, attain their asymptotic values at molecular weights well below the real polymer range. On the other hand, the tensile strength does vary significantly with $\overline{M}_n$ within the range of real macromolecules.(64, 65,66)

In addition to tensile strength, other features of the stress-strain relationship for polymers may be sensitive to degradation. The elongation at break is a frequently measured property which changes with aging.(67,68)

(3) Recommended Mechanical Property Measurements. The following selected methods and instrumentation for measurement of pertinent mechanical properties of polymeric materials and components for plastic solar thermal collectors are discussed in the succeeding sections:

- Torsion Pendulum and Torsion Braid
- Rheovibron Viscoeleastometer
- Other Dynamic Mechanical Analyzers
- Ultrasonic Techniques.

(i) Torsion Pendulum and Torsion Braid. These techniques discussed below are types of dynamic mechanical measurements, which may be separated into two categories. In the first category, the response of a sample to free or natural oscillations is measured, while in the second category, the response to forced oscillations is measured. Strains imposed upon the samples are usually small so that the sample may be assumed to be in the linear viscoelastic region. The applicability of some of the instruments to the measurement of viscosity is also discussed.

The two most important free-oscillation techniques are the torsion pendulum and the torsion braid. Among all the dynamic mechanical analysis instruments, only the torsion pendulum is recognized as an ASTM method (D-2236).(70) The torsion pendulum was pioneered in this country by Nielsen(69), among others. The specimen is clamped at each end. One end is rigidly fixed, while the other is clamped to an inertia bar and is free to move. The specimen may be either rectangular or cylindrical. Rectangular specimens are to be 0.015 to 0.10 inch in thickness, 0.10 to 0.60 inch in width, and 1 to 6 inches in length (exclusive of material in the grips). Cylindrical specimens are to be less than 0.30 inch in radius and to have the same length as above. At least 1/4 inch of specimen is to be held in each grip.

In operation, the inertia member is given an initial angular displacement of less than 2.5 deg/cm of specimen length. The resulting damped harmonic oscillation is recorded by some suitable means such as linear variable differential transformers(69), rotational variable differential transformers(71), or optical techniques(72).

The useful temperature range of this technique is 4.2 to 600 K.(73) The frequency of oscillations is not generally an independent variable, but rather is determined by the properties and geometry of the sample and the moment of inertia of the inertia bar. However, a normal frequency range of 0.1 to 100 Hz may be given. Since only about ten cycles of oscillation need to be observed, a given test will take at most 2 minutes.

Estimates of the precision of the technique may be obtained from the results of an ASTM interlaboratory round robin.(70) With 15 laboratories participating, the interlaboratory precision was: for G' below T_g, ±7 percent; for G' in the glass transition region, ±30 percent; for G" and Δ above T_g, ±20 percent; and for G" and Δ below T_g, ±10 percent. Using the measured values of G' and G", or Δ, the glass transition temperature could be determined to within ±3 C. It was found that the values for intralaboratory precision were about half those for interlaboratory precision.

Although torsion pendulums have usually been laboratory-constructed devices, a commercial model is now available from Plastic Analysis Instruments, Inc., of Princeton, New Jersey. This model is automated and with a computerized data acquisition system, the precision and accuracy is improved over that quoted above. Precisions of a few tenths of 1 percent may be possible with this instrument.(74) This instrument is described in Reference (72). In addition to the usual torsion pendulum mode, the instrument may also be used for torsional braid analysis. Here, the usual film specimen is replaced by an inert braid which is dipped into a polymer solution and then the solvent is removed.

(ii) Rheovibron Viscoelastometer. The most widely used forced vibration viscoelastometer is the Rheovibron. The specimen in the form of a film or bar is held in tension by a pair of grips. An oscillating strain of a known frequency is imposed on one end of the specimen. The resulting stress is measured at the other end. From the magnitudes and phases of the stress and strain, the complex dynamic tensile modulus and the loss tangent are computed. The loss tangent may be read directly off a meter.

The temperature range of this instrument is from -150 C to 250 C. Discrete frequencies of 3.5, 11, 35, and 110 Hz and continuously variable frequencies between 0.01 and 1 Hz may be imposed on the specimen. Two models are available. Maximum specimen sizes are 0.05 x 0.4 x 5 cm for Model DDV-II-C and 0.5 x 1 x 7 cm for Model DDV-III-C. The ranges of dynamic modulus which can be measured are 10^6 to 10^{12} dyne/cm^2 for DDV-II-C and 10^7 to 10^{12} dyne/cm^2 for DDV-III-C. The range of loss tangent measurable is about 0.001 to 1.7.

Massa(75) has analyzed a mechanical model for the DDV-II, and has derived expressions for the moduli to take into account the effects of system compliance, sample yielding within the tensile grips, and system inertia. He showed that these effects require a correction to the raw data of 20 to 30 percent for glassy polymers. The correction factor was found to vary significantly with temperature, frequency, and sample width and thickness, and among different polymers. When appropriate corrections are taken into account, Massa estimates the accuracy of dynamic modulus values to be around 5 percent.(76) Accuracy is partially limited by the inability to obtain perfectly uniform specimen cross sections and by problems in mounting the samples in order to obtain uniform strain across the sample. Precision of dynamic modulus measurements is somewhat better, possibly around 1 to 2 percent. It is difficult to assign an accuracy or precision value to the loss tangent, except to note that 10^{-3} is the lower limit of detection.

Provided no damage is done to the specimen by the gripping arrangements, the measurement has no effect on the sample, so that the same sample may be remeasured after various degrees of degradation. Specimen setup time is about 10 to 15 minutes, and with a temperature scan rate of 2 C/min, a complete scan from -150 C to 250 C at a given frequency will take about 3-1/2 hours.

This is a laboratory instrument and as such is generally not portable. The instrument is produced by the Toyo Baldwin Co., Ltd., of Japan and is distributed in the United States by IMASS of Accord, Mass.

Several descriptions of modifications to the Rheovibron have appeared in the literature. Murayama and Silverman(77) have adapted the instrument to allow measurement in a gas medium, while Lowton and Nurayama(78) have extended this modification to a liquid medium. Massa(75) has developed a new sample holder to adapt the Rheovibron from a tensile geometry to a flexural geometry. Shah and Darby(79) described a parallel-plate modification of the sample holders to obtain oscillatory data in the shear mode for several high-density polyethylene melts.

(iii) Other Dynamic Mechanical Analyzers. Other instruments in this category are the Weissenberg Rheogoniometer(80), the Du Pont Dynamic Mechanical Analysis System which is a module for their Thermal Analysis System, the Rheometrics Instrument and the Dynastat Transient and Dynamic Viscoelastic Analyzer (sold by IMASS).

(iv) Ultrasonics Techniques. Techniques based upon the propagation of ultrasonic waves are widely used in nondestructive testing. When an ultrasonic wave traveling in one medium reaches an interface with another medium having a different acoustic impedance, a fraction of the incident wave is refracted. For a wave incident normal to the interface, the power reflection coefficient is given by

$$R = \left(\frac{\rho_1 C_1 - \rho_2 C_2}{\rho_1 C_1 + \rho_2 C_2} \right)^2$$

where ρ_1 and ρ_2 are the densities of the two media and C_1 and C_2 are the acoustic velocities in the two media. This reflection is the basis for the pulse-echo flaw-detection system, in which(81) an ultrasonic transducer bonded to the surface of the material under test emits a pulse which travels through the material. At each interface that the pulse encounters, a certain fraction of energy is reflected and is picked up by the transducer. This technique requires that the transducer be acoustically coupled to the material. The coupling can be effected by various means, such as cements or thin films of water or oil.

Equipment for ultrasonic inspection usually consists of three basic items:

- One or more ultrasonic transducers, possibly combined with a mechanical scanning device
- Electronic signal processors which generate and detect the ultrasonic pulses or waves and other reference signals, and which convert the signals into usable data
- Units for displaying and/or recording the various signals.

Instrumentation would probably be assembled in modular form, with applicable equipment being available from such companies as Krautkramer-Branson, Panametrics, Automation Industries (Sperry Division), Nortec Corporation, Sonic Instruments, and Balteau Electric Corporation.

While ultrasonic techniques have been used for many years to detect the presence of disbonded regions or delaminations in adhesively bonded systems, it is only recently that these techniques have been applied to the quantitative determination of the quality of an adhesive bond prior to actual disbonding. J. L. Rose and his associates at Drexel University have been working on a 5-year program which started in January, 1973, with the objective of developing ultrasonic procedures for a quantitative determination of adhesive bond strength.(82) Most of the work has been directed at predicting the failure load of aluminum-aluminum step-lap joints, although the work is to be extended to metal-to-composite structures. Mathematical models were developed to allow a study of the basic ultrasonic wave interaction mechanisms with an adhesive bond.(83) The models studied were identified as the reference bond model, the material property gradient model, the surface preparation model, and the combined property gradient and surface preparation model. The reference bond model considers the adhesive layer as a homogeneous, isotropic layer with isotropic substrate layers on either side. The material property gradient model is based upon the expectation that chemical migration, gas entrapment, and cure variation problems will cause a variation of mechanical properties across the thickness of the adhesive. This variation would then affect the transmission and reflection of ultrasonic waves through the adhesive. To make calculations, the adhesive was artificially subdivided into up to five layers having different acoustic properties. The surface preparation model is based on the expectation that surface contaminants may result in microscopic points of nonbonding uniformly distributed over the adhesive-substrate interface. This model then assumes that the interface consists of many points of contact separated by microscopic voids. The use of computer programs was necessary to obtain quantitative results. The results showed that variations in ultrasonic reflections may be small and that a careful signal analysis is necessary to avoid overlooking these small variations.

(4) Thermal Analytical Techniques. Thermal analysis involves the use of a family of techniques by which the response of some material property is studied as a function of temperature. The most widely used techniques include differential thermal analysis (DTA), differential scanning calorimetry (DSC), thermogravimetric analysis (TGA), and thermomechanical analysis (TMA). Other techniques in this family may include dynamic mechanical analysis (DMA), torsional braid analysis (TBA), thermooptical analysis (TOA), thermoacoustical analysis (TAA), electrical thermal analysis (ETA), thermometric titrimetry, and thermal evolution analysis (TEA).

All thermal analysis techniques have four main components: (1) a transducer to measure the response of the material, (2) a programmer which controls the heating and cooling rates of the material, (3) an amplifier which conditions the transducer signal, and (4) a recorder which yields a permanent record of the transducer response as a function of temperature. Various temperature scanning rates may be used and, depending upon what effects are being looked for, total running time may vary from about 10 minutes to 2 hours or so.

(i) Differential Thermal Analysis (DTA). In differential thermal analysis, the temperature of a sample and a thermally inert reference material are measured as a function of temperature. The quantity which is recorded is the temperature difference. Here the transducers are thermocouples, which are individually placed directly within the sample and the reference material. The sample and reference are placed in identical chambers in a block, the temperature of which is automatically controlled. As the block is heated, any transition which the sample undergoes will be accompanied by either liberation or absorption of heat, with a corresponding increase or decrease in the sample temperature with respect to the reference material. A record of this temperature difference (ΔT) as a function of programmed temperature (usually the sample temperature) gives information about the temperatures at which transitions occur and whether the transition is exothermic or endothermic. The area under the ΔT curve gives a rough indication of the amount of heat transferred in or out of the sample, but is not generally used for quantitative purposes.(85)

Since the thermocouples are embedded directly within the sample, DTA provides the highest thermometric accuracy of any of the thermal analysis techniques, and thus can be used to study small changes in transition temperatures which may be associated with other physical properties. Sensitivities of approximately 0.002 C are common for ΔT. However, this precision may not be realized in practice in measuring absolute temperatures of thermal events. Thermograms resulting from DTA runs generally show a number of peaks (associated with first-order transitions such as the melting point) and baseline shifts (associated with second-order transitions, such as the glass transition). Furthermore, there is inevitable rounding of the peaks and shifts so that various extrapolation techniques must be used to obtain transition temperatures. The shape of the DTA curve is influenced by two general types of variables: instrumental factors and sample characteristics.(86) Instrumental factors include furnace atmosphere, furnace size and shape, sample-holder material, sample-holder geometry, wire and bead size of thermocouple junction, heating rate, speed and response of recording instrument, and thermocouple location in sample. Sample characteristics include particle size, thermal conductivity, heat capacity, packing density, swelling or shrinkage of sample, amount of sample, effect of diluent, and degree of crystallinity. It is thus imperative that as many as possible of these factors be held constant when searching for small changes in transition temperatures. It should also be noted that the act of scanning a sample through a certain temperature range may lead to irreversible effects, such as those due to thermal degradation. In fact, a common practice is to erase a sample's previous history by performing one scan and then another. Such a procedure must be used with caution in degradation studies in order to avoid confusion between service degradation and changes induced by the actual measurement.

The use of thermal analysis in the determination of transition temperatures of polymers is covered by ASTM Designation D3418-75. Results of an interlaboratory round-robin showed that duplicate determinations of first and second-order transition temperatures on two specimens of the same sample by the same analyst should not differ by more than 1.5 C and 2.5 C, respectively. Similarly, duplicate determinations of first and second-order transition temperatures on specimens of the same sample analyzed in different laboratories should not differ by more than 2.0 C and 4.0 C, respectively.

DTA instruments are produced by a number of companies(87), with Du Pont and Perkin-Elmer being perhaps the better known ones. Many of these instruments are modular in design, with one basic programmer and recorder being used with separate modules for DTA, DSC, TGA, etc.

Numerous applications of DTA to polymers have been reported in the literature.(88) From the viewpoint of this study, the most important application appears to be the determination of shifts in the glass transition temperature. One example of this use is the study of the curing and thermal degradation of thermosetting resins by monitoring T_g.(89) In this study, changes of over 80 C in T_g were noted. Another potential use may be for determining the degree of crystallinity through measurements of the melting point.(85)

(ii) Differential Scanning Calorimetry (DSC). Differential scanning calorimetry is somewhat similar to DTA. However, DSC is more quantitative than DTA in regard to measurement of quantities of heat, i.e., DSC has a higher calorimetric accuracy than DTA. The dependent variable on the output curve of a DSC is proportional to the difference in rates of heat transfer between the sample and the reference material. The two factors which are necessary to the generation of quantitative energy data are (1) a controlled heat path into the sample and reference materials and (2) location of the temperature sensors external to the sample.

Two approaches to these requirements appear to have been taken. In a Perkin-Elmer DSC apparatus, the sample and reference material are provided with both separate temperature sensors and heaters. As the temperature is scanned, the sample and reference temperatures are continuously maintained at the same level by adjusting the power which is supplied to them. The differential power input is then recorded as the ordinate on the output chart.(90) In a Du Pont DSC apparatus, a thermoelectric disk of closely controlled dimension and configuration acts as the major path of heat flow into the sample and reference. The disk provides one leg of a thermojunction while the other leg is made by attaching thermocouple wires to the disk. Then, under operating conditions, the differential of heat flow into the sample and reference materials is proportional to the differential temperature.(85)

Calorimetric sensitivities as low as 3 μcal/sec have been reported (for DuPont Model 910).(91)

One primary use of DSC is the measurement of heats of transition at first-order transitions. An example of this is the heat of fusion; also, the degree of crystallinity may be correlated with the heat of fusion. The DSC may also be used to determine specific heats and heat effects associated with material decomposition, polymerization, and curing of thermosetting resins.

(iii) Thermogravimetric Analysis (TGA). Thermogravimetric analysis is a technique whereby the change in weight of a sample can be measured as a function of temperature.(92) Many of the commercial instruments use Cahn electrobalances. These are null-type balances, in which beam displacements caused by weight changes are detected with a light beam-shutter-photocell arrangement.(87) Changes in sample weight cause the beam to deflect momentarily. This motion produces a change in the phototube current which is amplified and fed to the coil attached to the beam. The change in coil current while in the field of the magnet produces a torque on the beam wich restores it to its original position. The coil current thus serves as an indicator of weight change. The Cahn RG electrobalance has a capacity of 2.5 g and a sensitivity of 0.1 μg. A number of companies, including Perkin-Elmer, make TGA's based on Cahn-type thermobalances.

DuPont makes a somewhat different TGA, in that a horizontal configuration, used to avoid buoyancy and aerodynamic effects, permits axial flushing of the furnace tube with various gases. This instrument has a capacity of 1 gram and a sensitivity of about 1 μg. The temperature range is room temperature to 1200 C, and the pressure range is 1 torr to 1 atmosphere pressure. Precision and accuracy of weight measurements are 0.4 and 1.0 percent of full scale, respectively. Depending upon the sample size, as little as 0.5 percent of the sample weight may be displayed full scale.

As with DTA, many factors can influence the shape of a TGA curve.(86) Instrumental factors which are important are furnace heating rate, recording or chart speed, furnace atmosphere, geometry of sample holder and furnace, sensitivity of recording mechanism, and composition of sample container. Important sample characteristics are amount of sample, solubility of evolved gases in sample, particle size, heat of reaction, sample packing, nature of the sample, and thermal conductivity. These factors must be considered when attempting to look for small changes in weight associated with degradation. The method is necessarily destructive of the sample.

TGA should be useful in degradation studies in determining changes in amounts of additives such as plasticizers and UV stabilizers. The effluent from the gas purge may be further analyzed by gas chromatography, mass spectrometry, etc.

(iv) Thermomechanical Analysis (TMA). Thermomechanical analysis is one of the newest of the thermal analysis techniques. The transducer in the TMA is a linear variable differential transformer that detects linear movement of a probe in contact with the sample. Various types of probes may be used for detecting different thermomechanical properties as the sample's temperature is scanned.(85)

In the expansion mode, changes in sample length are measured directly. The slope of the resulting curve is the coefficient of thermal expansion. The glass transition temperature may be observed by the change in slope of the expansion curve, while the melting temperature can be observed as a sudden change in dimension. The temperature range of operation is -160 C to 1200 C. The maximum sensitivity is 50 μin. of probe deflection per inch of chart paper. Linearity is ±1/2 percent up to ±0.05-inch total displacement. Various size samples up to 0.375 inch in diameter may be used.

With other probe configurations, the TMA may be used to obtain softening temperatures, tensile modulus, compression modulus, shrink temperatures, viscosity, and stress relaxation.

With regard to degradation studies, TMA should be useful in detecting changes in glass transition temperatures, as well as in providing quantitative data on changes in coefficient of the thermal expansion.

(5) Summary—Thermal and Mechanical Measurement Techniques.

- Since the properties of polymers are in some cases very sensitive to the glass transition temperature, it is recommended that this property be measured by a suitable means. The simplest and most rapid methods for measuring T_g appear to be differential thermal analysis and differential scanning calorimetry. In the absence of specific information regarding the accuracies of these two techniques with regard to measurement of T_g, DSC measurements are recommended since the resulting thermograms can be interpreted quantitatively.

- An alternative method for obtaining T_g is dynamic mechanical measurements. In addition to providing a measure of T_g, this family of techniques yields potentially valuable information on the mechanical behavior of the materials. If these measurements are undertaken, the measurements by DTA or DSC will probably be unnecessary. Dynamic mechanical measurements would thus be recommended over DTA or DSC. Since temperature is a more important variable than frequency for dynamic mechanical measurements, any of the previously described techniques may be used, depending upon availability and ease of measurement.

(e) Other Physical Property Measurement Techniques for Polymeric Materials

(1) Infrared Thermovision. Infrared thermovision is a name applied to several commercial instruments which are used for detecting temperatures or temperature differences by measuring the infrared thermal radiation emitted by objects. Radiation emitted by an object is collected by a lens system and the object of study is scanned by a system of prisms. The radiation is then focused onto an infrared detector, the voltage output of which is sent to a television-type display unit. The display may be either black and white, in which the lighter areas represent higher temperatures, or colored in which different colors represent different temperatures.

Thermovision systems have been used to monitor and test solar photovoltaic cell systems under various stress applications.(93,94)

(2) Profilometry. The stylus profilometer has been a major engineering tool for measuring surface topography and surface roughness for many years. The instrument consists basically of a diamond stylus that moves horizontally relative to the surface of the test sample. The vertical displacements of the stylus are detected and converted to analog or digital signals that can be recorded on strip charts or displayed on appropriate meters. In some cases, the sample is moved and the sensing head is stationary, while in others, the stylus is moved across a stationary sample. The technique displays an accuracy comparable to that of the X-ray and optical interference techniques used in the measurement of film thicknesses and profiles.(95) Vertical resolution for standard stylus-type

measurements is limited to about 20 Å by the electrical and mechanical noise associated with the measuring instrument. The horizontal resolution depends on the contour of the stylus tip, although it is not limited to lateral geometries larger than the stylus-tip diameter. Reference (95) gives an excellent example of a high-resolution profile in a series of strips 3.5 μm wide and 450 Å thick made by using a 0.0005-inch (∿13 μm)-radius stylus.

The combining or direct interfacing of profilometers with digital computers over the past few years$^{(96,97)}$ has had a major impact on the accuracy and effectiveness of the technique. The use of the computer greatly simplifies the task of analyzing and evaluating the data to determine average and rms surface roughness and eliminates the element of individual interpretation present in the graphical analysis techniques previously used. In addition, computer processing of the data can include elimination of the contributions of electrical noise and instrument drift. Limit sensitivities of the order of 5 Å (signal/noise = 1) are reported for configurations of this type.

In selecting instruments for characterizing the surfaces of plastic materials, and particularly degradative changes of these surfaces associated with abrasive or chemical attack, it should be kept in mind that it will probably be desirable to look at those surfaces at several different levels of magnification. Certain types of chemical attack (e.g., certain leaching or etching reactions) may result in a high density of relatively small pits or defects that might appropriately be evaluated by SEM studies. Other types of chemical and abrasive attack could have a more gross effect on surface roughness which might, for example, ultimately result in increasing the dirt accumulation rate. Some of these types of effects might be studied, particularly where quantitative assessments over large areas are desired, by profilometry. Profilometry provides a quantitative measure of surface roughness in its range of sensitivity, and evaluation of the data, especially when coupled with a computer, is reasonably straightforward. It should also be pointed out that it is not essential that the computer interface directly with the profilometer. The data can be recorded in digital form (e.g., on tape) at the measurement site and transported to the computer for processing in cases where no on-site computer is available.

In view of the sensitivity and accuracy of present-day surface profiling instruments, and particularly considering the quantitative nature of the information it supplies, it is felt that profilometry can be a useful tool in degradation and life-testing studies, particularly in environmental situations where abrasive or other forms of relatively gross attack are potentially significant factors.

(3) Photoacoustic Spectroscopy. Photoacoustic spectroscopy (PAS) is a technique which has only recently found application in the study of solids, although its origin can be traced to early studies of the "opto-acoustic effect" in gases by Alexander Graham Bell and others in the 1880's.(98) The technique consists of illuminating a sample with a modulated, high-intensity, monochromatic light beam (UV-visible-near IR) while it is enclosed in a sealed, small-volume cell which also contains a sensitive microphone. The incident light is absorbed by the sample, resulting in the excitation of electrons to higher energy states. These electrons then decay back to the lower energy states through primarily nonradiative processes, thereby creating a periodically varying (due to the modulation) heat distribution in the vicinity of the point of absorption. This heat diffuses to the surface of the sample where it creates periodic pressure waves in the gas-boundary layer. These are propagated through the bulk of the gas to drive the microphone and produce the output signal. Both the amplitude and the phase of the signal contribute useful information. These signal properties depend upon a number of factors, including the optical absorption and thermal diffusion properties of the sample, the modulation or chopping frequency, and the cell/sample geometry. The basic instrument consists of a light source with either an electrical or a mechanical modulation capability, a monochromater, the sealed sample cell, and suitable electronics for determining and displaying the amplitude and phase of the output.(99,100) The cell is basically a sealed unit with a transparent window for light admittance, containing some means of sample support and a sensing microphone. The physical dimensions of the cell and the thermal properties of the gas are important factors in determining the sensitivity of the photoacoustic spectrometer.(101)

PAS can be used to study both the optical absorption properties and the thermal diffusivity/thermal conductivity properties of a material. A principal advantage of the technique is its ability to obtain spectra directly from various types of solids such as crystals, powders, gels, and other forms and types of materials where scattered light or opaqueness might present a significant problem for more conventional spectroscopies. The technique has been used advantageously in studies of biological materials (PA-Z), organometallic compounds(102), and nonradiative processes in luminescent solids(103), and in the separate determination of bulk- and surface-absorption coefficients in weakly absorbing materials(104).

In the study of the degradation of plastic materials for solar thermal collector applications, PAS offers several potential advantages as a laboratory tool.

- PAS provides detailed information on changes in both the optical and thermal properties of materials.
- PAS has the capability of measuring changes in bulk optical properties in cases where unrelated degradation of surface properties (e.g., abrasive attack) might make characterization by more conventional techniques extremely difficult.
- With PAS, by varying the region in which the photon energy is deposited (i.e., by varying the wavelength of the incident radiation), it may be possible to obtain information that allows discrimination between several degradation modes/mechanisms occurring simultaneously.
- With PAS, with proper choice of photon wavelength and modulation frequency, changes in the properties of bonds at material interfaces might be detected, thereby providing a means of predicting delamination.(102)

(4) Water Vapor and Gas Permeability. The permeability of materials used in plastic solar thermal collector systems to gases and water vapor is of interest in degradation studies from two points of view. On the one hand, it controls the level of exposure of those elements of the collector that are vulnerable to attack by water vapor, oxygen, and other corrosive agents; hence, increases in permeability to these agents could be

precursive of increased rates of attack by them. On the other hand, changes in the permeability of polymers are generally the result of structural, morphological, and chemical changes that could lead to failure because of non-corrosive effects such as embrittlement, weakening of bonds, etc, in which case, the permeability is a potential diagnostic tool for detecting and measuring such changes. The permeability (P) of a material to a gas or vapor is the product of the solubility (S) of the gas or vapor in the barrier material and its diffusivity (D) in and through the material: $P = S \cdot D$. This quantity (P) can be directly measured and is normally expressed as the rate of transfer of the penetrant (e.g., cm^3/sec) per unit area, per unit pressure differential, and per unit thickness of the barrier material. The diffusivity is basically related to the physical limitations of diffusion, such as the size and shape of the penetrant molecule and the size and shape of the spaces or passages in the solid material through which the molecule must pass. The solubility is influenced by interactions between the penetrant molecule and the surrounding medium due to molecular forces such as those associated with the degree of the molecule's polarity and the medium. There are a number of ways to determine S and D from permeability data when that is desired. In some cases, independent data on solubility can be obtained without great difficulty (e.g., from immersion tests in the case of water vapor), thereby permitting calculation of D. This approach can, however, be difficult for species exhibiting low solubility. Alternatively, diffusion coefficients and diffusivities can be deduced from studies of the kinetics of sorption and transmission processes in the transient (nonequilibrium) stage.(105)

Permeability measurements are generally made by impressing a pressure or partial pressure gradient of the gas or vapor across a sheet of the material and measuring the rate at which it is transmitted to the low pressure side. The means of measuring the transmission rate varies with the penetrant species and the level of sensitivity desired. In the ASTM methods (E-96-66 and C355-64) for measuring water-vapor transmission, the film materials are sealed over a dish or container in which desiccant (or water) has been placed. The change in weight due to the aborption (or escape) of water is measured to determine the transmission rate. Some of the more advanced systems use IR detection systems to measure water vapor and carbon dioxide transmission rates(106), and one manufacturer uses a "coulometric" fuel cell-type detector to measure oxygen transmission rates(107). These techniques offer advantages in speed and sensitivity.

The permeability of polymeric films to water vapor, carbon dioxide, and oxygen has been of concern to the packaging industry for many years and considerable information has been developed and published in this area. The importance of water vapor permeability in photovoltaic module construction materials was recognized early in the terrestrial photovoltaic module development program, and studies of this property for a number of candidate materials have been conducted.(108)

The potential of permeability as a diagnostic tool for studying changes in polymers has been indicated recently reported studies by Professor C. Rogers of Case Western Reserve University.(109) His investigations of the effect of UV exposure on the permeability of polymers to water vapor and nitrogen have shown clear correlations between the degree of oxidation (carbonyl concentration) of the polymer, as determined by FTIR measurements, and changes in the permeability properties. Detailed examinations of the solubility and diffusivity have permitted deductions concerning the nature of some of the changes occurring (i.e., structural densification, increases in polarity). The potential applicability of permeability measurements as a laboratory technique for studying degradation associated with structural and chemical changes in polymers is apparent from these studies. One might ultimately envision a diagnostic test in which the permeabilities of a series of liquids, gases or vapors of various molecular size and polar characteristics are used to characterize degradative change in polymers.(110)

(5) Summary—Other Physical Property Measurement Techniques for Polymeric Materials

- Profilometry, when combined with computer processing of the data, could be a powerful tool for quantitative assessment of changes in surface roughness over large areas, such as that which might occur due to abrasive attack of polymeric top surfaces of thermal solar collector systems.
- Photoacoustic spectroscopy (PAS) offers some potentially unique capabilities in studying optical, thermal, and possibly bonding characteristics of polymers.
- Permeability measurements of polymeric materials are important for establishing their barrier properties against potential corrosive agents; however, these measurements could also be useful in diagnosing chemical and structural changes associated with degradation modes that are not related to corrosion.

(f) Outdoor Exposure (Natural Weathering) Techniques

Outdoor weathering is used for service life and durability testing because of the difficulty in simulating environmental conditions, particularly sunlight, in laboratory tests. The National Bureau of Standards maintains outdoor exposure sites at seven locations: Alaska, Washington, Nevada, New Jersey, Maryland, and Puerto Rico, but there are also commercial exposure stations in southern Florida (subtropical environment) and in New Mexico and Arizona (desert environment).(111)

It is difficult to correlate outdoor weathering test results between various exposure sites because of the variation in climatic conditions and the lack of sufficient measurements of important environmental parameters. Another major problem with outdoor exposure methods is the time required to obtain test results. Several years of exposure are often required to achieve detectable changes in properties. It is not always practicable or economically feasible to wait for lengthy tests except for long-range research purposes.

In natural weathering tests used for end-use service testing, the samples are usually mounted at a 45-degree angle facing south (ASTM D1435).(112) A suitable property is then measured periodically to determine material changes. This technique has many limitations. First, it is unreasonable to test a material expected to last 20 years in this manner. Second, samples have often been mechanically unstressed during the test while they may be stressed in use. Third, the mounting angle affects the results since a 45-degree angle will not always receive the maximum solar energy. The 45-degree angle also fixes the moisture runoff condition. These conditions may or may not duplicate use conditions. However, the most serious limitation in many natural weathering tests has been that the weater parameters influencing the samples' deterioration have not always been recorded. For example, the UV-radiation intensity impinging on a test sample (which is the most important single parameter) the sample temperature, thermal fluctuations and gradients, and the duration of moisture on the sample are often not measured. Also, airborne chemical pollutants have usually been ignored in data analyses because atmospheric pollution data are often lumped into a single quantity (i.e., air-quality index) which does not delineate between specific chemical pollutants.

There are still further limitations to the natural weathering tests. In tests lasting 2 years or less, the time of the year at which the test begins strongly affects both the results and the analysis.(113) A sample tested for 18 months may encompass one summer or two. A sample enduring two summers has undergone a much more severe test than the sample tested through one summer. The physical property tested is sensitive to the type of degradation that has occurred. Surface-oxidized samples, for example, may exhibit little loss in tensile strength, yet show a much more brittle behavior and a reduced elongation prior to failure.(114,115) The appropriate property for measurement is one that is sensitive to, but also indicative of, the end use of the material. Solar collector materials that retain their tensile strength yet lose their transparency are obviously failures. Ideally, the property should be tested by a nondestructive technique to eliminate the large number of samples needed to reduce sample-to-sample variation and still permit testing at various times. Finally, outdoor weather tests are valid only in the area in which the tests are conducted. Reference (116) discusses how different failure mechanisms occur in outdoor weathering in the Panama Canal Zone, a small geographical area compared with the continental United States.

Many of the above limitations can be easily overcome. Mounting the samples equatorially so that they always receive the maximum amount of radiation eliminates the mounting-angle dilemma. Although such mounting is not always consistent with end-use exposure, it maximizes the degradation rate and enables the results to be correlated with UV-radiation intensity. UV-radiation intensity impinging on the samples must be recorded. Such a measurement provides a better exposure parameter than days, sun hours, or total solar radiation. UV radiation includes both direct solar radiation and scattered skylight. If the samples are to be mechanically stressed in use, they can be stressed in the weathering test. The level of stress applied to the test sample should be the same as that in the prototype, thus avoiding a change in the failure mechanism.

Sample temperature, thermal gradients, and thermal fluctuations should also be recorded, as should humidity and the duration of surface moisture on a sample. Duration of surface moisture is more important than total rainfall. A heavy rainfall during the night that drains off the sample by morning will have much less effect on the degradation of a polymer than dew that remains and is exposed to sunlight. Airborne pollutants will also have to be monitored.

Oxygen level, while quite constant at a specific locale, does vary with altitude. For that reason, the barometric pressure should be recorded at each testing locale.

As mentioned previously, sample testing ideally should be nondestructive. Measuring changes in IR reflectance, UV absorptance, gloss, light transmission, ESR, and chemiluminescence, as discussed later, enable chemical changes in the sample to be monitored. Attempts can then be made to relate these changes to the macroscopic property of interest.

Unfortunately, the above procedures do not remove the limitations of geographical differences, nor do they abbreviate the testing time span. Equatorial mounting of the samples with focused mirrors (designated EMMA, Equatorial Mount with Mirrors for Acceleration, by Desert Sunshine Exposure Tests, Inc.) has been utilized to accelerate degradation by concentrating the solar radiation on the sample by a factor of eight. There has not been a simultaneous eight-fold increase in degradation rate, however. Correlation is lacking because UV intensity does not have a simple multiplicative effect on degradation. Various degradation reactions occur, often simultaneously, often competitively, and sometimes alternately. With several reactions possible, increasing only one parameter will not increase the overall degradation rate proportionately. Even more serious is the fact that the prime degradation mechanism may change. Schafer shows how the degradation mechanism of PVC changes when UV intensity is increased above a certain level.(117)

A summary of outdoor aging methods in ASTM Standards appears in Tables 2 and 3:

Table 2.	Outdoor Weathering Methods in ASTM Standards (1971)
Table 3.	Summary of Exposure Conditions and Procedures in ASTM Standards for Outdoor Weathering (1971)

TABLE 2. OUTDOOR WEATHERING METHODS IN ASTM STANDARDS (1971)

[Test Number (ASTM Part Number) (Materials for Possible Use in Solar Thermal Collector Systems)]

C-488 (14) (finishes for thermal insulation)

D-518 (28) (soft rubber)

D-904 (16) (adhesives)

Adhesive-bonded joints and structures: D-1828 (16), D-2918 (16), D-2919 (16)

Nonmetallic materials: G-7 (30)

Organic coatings: C-488 (14), D-1006 (21), D-1014 (21), D-1641 (21), D-1654 (21), D-2830 (20,21), G-11 (21,30)

- on metal: D-1014 (21), D-1654 (21), G-11 (21,30)
- on pipelines: G-11 (21,30)
- on thermal insulation: C-488 (14)
- on wood: D-1006 (21), D-2830 (20,21)

Varnishes: D-1654 (21)

Plastics: D-1435 (27)

Rubber: D-518 (28)

Various materials: G-24 (24,30)

Atmospheres, Definitions

C-62

C-216 Areas in the United States defined by Weathering Index

Areas in the United States defined and indicated in a map according to Weathering Index, which is, for any locality, the product of average annual number of freezing cycle days and average annual winter rainfall in inches. Areas in the map include those with

Severe index, over 500

Moderate index, 100-500

Negligible, less than 100

TABLE 2. (Continued)

D-1435	As defined by National Oceanic and Atmospheric Administration (NOAA), and shall include
	Sunlight energy data, if available, as integrated incident expressed in langleys (g-cal/cm^2)
	Description of climate and summary of data at site from NOAA
	Rainfall
	Percentage of possible sunshine
	Temperature average and extremes
	Humidity average and extremes
D-1828	Rural-pure (ASTM site: State College, Pennsylvania)
	Industrial-sulfurous gases present (Pittsburgh, PA)
	Marine-seacoast where chlorides are deposited on specimens (Kure Beach, North Carolina)
	Tropical or southern Florida under conditions of heat and high humidity (Freeport, Texas)
	and shall include
	Average monthly relative humidity
	Average monthly temperature
	Total monthly rainfall
	Average daily solar radiation, if available
	Total solar radiation on specimens, if available
	Air pollution data, if available
	References to instrumentation which meets NOAA requirements
G-7	Tropical or subtropical, inland
	Tropical or subtropical, salt atmosphere
	Temperature, inland
	Temperature, salt atmosphere
	Desert
	Arctic or subarctic
	Industrial, low pH
	Industrial, high pH
	Industrial, NO_2, SO_2, or O_3

TABLE 2. (Continued)

G-7 (continued)

Daily maximum, minimum, mean temperatures

Daily maximum, minimum, mean percent relative humidities

Daily hours of rain and dew (wetness)

Daily total inches of rainfall

Daily irradiation: total langleys, spectral langleys (wavelength band specified), ultraviolet sun-hours (solar irradiation above an intensity of 0.823 cal/cm^2 min.)

1 langley = 1 g-cal/cm^2 = 11.62 W/m^2 = 41,840 J/m^2

G-24 Includes:

Date and location, including approximate latitude

Maximum, minimum, mean daily air temperatures and relative humidity

Humidities

Exposure type

Direct (to all prevailing atmospheric conditions): B-537, C-488, D-518, D-1006, D-1014, D-1435, D-1641, D-1828, D-2830, G-11

Window (in cabinet under glass): G-24

Sunlight Exposure Method: Specimens exposed in glass-covered cabinet between 9:00 a.m. and 3:00 p.m. (standard time) on clear, sunny days. At all other times, specimens are removed from the cabinet and stored in a dark and dry storage area at normal room temperature.

Daylight Exposure Method: Same as for Sunlight Exposure Method except that the specimens are left in the exposure cabinet continuously 24 hrs a day and are removed only for inspection.

Window (under glass, not in cabinet): D-904

Direct or Window: G-7

Positioning and mounting of specimens

C-488	45 degrees from horizontal, facing south
D-518	45 degrees facing south
D-904	45 degrees facing south
D-1006	Vertically facing both south and north on test fences. If dust collection and mildew resistance are not pertinent, north vertical exposure may be eliminated.
D-1014	45 degrees facing south
	45 degrees facing north
	Vertical facing south
	Vertical facing north

TABLE 2. (Continued)

D-1435	45 degrees facing equator
	90 degrees facing equator
	Horizontal
D-1641	45 degrees facing south
D-1828	45 degrees facing south
D-2830	90 degrees on test fences as in D-1006
G-7	Most common: 45 degrees from horizontal facing equator
	Others:
	Vertical facing south and north
	Horizontal
	Angle from horizontal equal to latitude of location in degrees, facing equator
	Normal service position
	Angle of 30 degrees from horizontal, facing equator
	Angle of 5 degrees from horizontal, facing equator
G-11	Horizontal
G-24	Angle from horizontal equal to approximate latitude of location in degrees, facing equator

<u>Rack, fence, exposure cabinet design</u>

<u>Exposure cabinet</u>: G-24

<u>Racks</u>: D-1435, D-1828, G-7, G-11

<u>Test fences, racks</u>: D-1006

TABLE 3. SUMMARY OF EXPOSURE CONDITIONS AND PROCEDURES IN ASTM STANDARDS FOR OUTDOOR WEATHERING METHODS (1971)

TEST NUMBER (ASTM PART NUMBER)	TEST TITLE AND EXPOSURE DETAILS
C-488 (14)	Standard Method for Conducting Exterior Exposure Tests of Finishes for Thermal Insulation Direct esposure. Specimens mounted 45 degrees from horizontal, facing south.
D-518 (28)	Standard Method of Test for Surface Cracking Resistance of Stretched Rubber Compounds Direct exposure. Specimens mounted at 45 degrees facing south. For accelerated weathering method, see Table VI.
D-904 (16)	Standard Recommended Practice for Determining the Effect of Artificial (Carbon-Arc Type) and Natural Light on the Permanence of Adhesives Window exposure not in cabinet. Specimens mounted at 45 degrees, facing south, protected with transparent shield which transmits solar radiation. For accelerated weathering method, see Table VI.
D-1006 (21)	Recommended Practice for Conducting Exterior Exposure Tests of Paints on Wood Direct exposure. Specimens mounted vertically facing both south and north on test fences. If dust collection and mildew resistance are not pertinent, north vertical exposure may be eliminated. Description of test fences and racks.
D-1014 (21)	Standard Method of Conducting Exterior Exposure Tests of Paints on Steel Direct exposure. Specimens may be mounted as follows: 45 degrees facing south 45 degrees facing north Vertical facing south Vertical facing north
D-1435 (27)	Standard Recommended Practice for Outdoor Weathering of Plastics Direct exposure. Specimens may be mounted as follows: 45 degrees facing equator 90 degrees facing equator Horizontal Description of racks.

TABLE 3. (Continued)

D-1641 (21)	Standard Method of Test for Exterior Durability of Varnishes Direct exposure. Specimens mounted 45 degrees facing south.
D-1828 (16)	Standard Recommended Pratice for Atmospheric Exposure of Adhesive-Bonded Joints and Structures Direct exposure. Specimens mounted 45 degrees facing south Description of racks
D-2830 (20,21)	Standard Method of Test for Durability and Compatibility of Factory-Primed Wood Products with Representative Finish Coats Direct exposure. 90 degrees from horizontal on test fences as in D-1006
G-7 (30)	Tentative Recommended Practice for Atmospheric Environmental Exposure Testing of Nonmetallic Materials Direct or window exposure. Specimens usually mounted 45 degrees to horizontal facing equator. May also be mounted as follows: Vertical facing south and north Horizontal Angle from horizontal equal to latitude of location in degrees, facing equator Normal service position Angle of 30 degrees from horizontal, facing equator Angle of 5 degrees from horizontal, facing equator Description of racks
G-11 (21,30)	Tentative Method of Test for Effects of Outdoor Weathering on Pipeline Coatings Direct exposure Specimens mounted horizontally Description of racks
G-24 (24,30)	Standard Recommended Practice for Conducting Natural Light (Sunlight and Daylight) Exposures Under Glass Window exposure Angle from horizontal equal to approximate latitude of location in degrees, facing equator Description of exposure cabinet.

(g) Laboratory Accelerated Aging Techniques

There are many problems associated with lengthy outdoor testing, and in an attempt to solve these problems, several methods have been developed to accelerate the aging processes of materials. These methods and their technical problems are as follows:

- Laboratory methods must be devised to simulate outdoor weathering conditions but at the same time naturally accelerate its effects on materials and components used in plastic solar thermal collector systems.
- Climatic factors, types and extent of exposure, which cause the aging of materials must be measured accurately.
- Both chemical and physical mechanisms of aging should be accelerated and evaluated.
- The specimen size may play an important factor in accelerated aging as well as in outdoor exposure testing. The total system as well as individual materials or components should also be tested in order to obtain valid results.

In devising a laboratory method to simulate and still accelerate the aging processes, it is important to consider the effects of acceleration on the true mechanisms of aging leading to limited service life capability. Even if it is possible to accelerate the effects of aging caused by outdoor exposure in a laboratory, the mechanism of both conditions must be the same or have the same relationship between time of exposure and measured change in physical or chemical properties relating to the service life of the materials or systems tested.

Individual effects or stresses of various environmental factors separately applied to materials do not always predict the true service life of a total system. It is much more desirable to apply combinations of stresses (ultraviolet radiation, temperature cycling, humidity, etc) in order to approximate actual weathering exposure conditions closely resembling actual environments in the product to be used. For this reason, geographical testing locations throughout the United States are utilized to measure the serviceability of materials and obtain realistic information on product performance and evaluation.(118)

A summary of accelerated aging methods in ASTM Standards is presented in Tables 4 through 6.

Table 4. Accelerated Aging and Weathering Methods in ASTM Standards (1971)

Table 5. Summary of Exposure Conditions and Procedures in ASTM Standards for Accelerated Weathering (1971)

Table 6. Criteria for Outdoor and Accelerated Weathering Methods.

TABLE 4. ACCELERATED AGING AND WEATHERING METHODS IN ASTM STANDARDS (1971)

TEST (ASTM PART NUMBER) (BUILDING MATERIALS SUITABLE FOR POSSIBLE USE IN SOLAR THERMAL COLLECTOR SYSTEMS)

B-117 (7,21,31)	(metals, coatings on metals)
B-287 (7,21,31)	(metals, coatings on metals)
C-481 (16)	(sandwich constructions)
C-510 (14)	(joint sealants)
G-669 (14)	(sealants used in back bedding and face glazing)
D-518 (28)	(soft rubber)
D-529 (11)	(bituminous materials)
D-750 (28)	(vulcanized rubber)
D-756 (27)	(plastics)
D-822 (21)	(organic coatings)
D-904 (16)	(adhesives)
D-1037 (16)	(wood-based hardboard, particleboard)
D-1101 (16)	(adhesive joints in structural laminated wood)
D-1148 (28)	(vulcanized rubber)
D-1149 (28)	(vulcanized rubber)
D-1151 (16)	(adhesive bonds)
D-1167 (11,21)	(asphalt, emulsion coatings on built-up roofs)
D-1183 (16)	(adhesives)
D-1499 (27)	(plastics)
D-1501 (27)	(plastics)
D-1565 (28)	(vinyl foam)
D-1654 (21)	(organic coatings on metal)
D-1735 (21)	(organic coatings)
D-1754 (11)	(asphalt)
D-1870 (27,28)	(polymers)
D-2126 (28)	(rigid cellular plastics)
D-2246 (21)	(coated metal)
D-2247 (21)	(coated metal)
D-2248 (21)	(organic coatings)
D-2249 (14,21)	(sealants used in glazing and bedding)

TABLE 4. (Continued)

D-2366 (21)	(exterior house paints on wood)
D-2445 (26)	(propylene plastics)
D-2559 (16)	(adhesives)
D-2565 (27)	(plastics)
D-2803 (21)	(organic coatings on metal)
D-2831 (20,21)	(latex paints on masonry and asbestos-cement shingles)
D-2898 (16)	(fire-resistant treatment of wood)
D-2918 (16)	(adhesive joints)
D-2919 (16)	(adhesive joints)
G-23 (24,27,30)	(nonmetallic materials)
G-25 (24,30)	(nonmetallic materials)
G-26 (30)	(nonmetallic materials)
G-27 (30)	(nonmetallic materials)

Type of Exposure

Light exposure apparatus

Carbon-arc type: G-25, D-904

Xenon-arc type: G-27

Light and water exposure apparatus

Carbon-arc type: C-510, C-669, D-529, D-750, D-822, D-1499, D-2249, G-23

Xenon-arc type: D-2565, G-26

Fog chamber: D-1735

Humidity chamber: D-2247

Humidity chamber and cold box - cycling: D-2246

Salt-spray cabinets: B-117, B-287, B-368, D-2803

Various exposure conditions: C-62, C-67, C-88, C-216, C-217, C-481, C-510, C-666, C-669, C-671, C-682, D-518, D-545, D-756, D-1037, D-1101, D-1148, D-1149, D-1151, D-1167, D-1183, D-1501, D-1565, D-1654, D-1754, D-1870, D-2126, D-2248, D-2366, D-2445, D-2559, D-2803, D-2831, D-2898, D-2918, D-2919

TABLE 5. SUMMARY OF EXPOSURE CONDITIONS AND PROCEDURES IN ASTM STANDARDS FOR ACCELERATED WEATHERING METHODS

TEST NUMBER (ASTM PART NUMBER) TEST TITLE AND EXPOSURE DETAILS

B-117 (7,21,31) Standard Method of Salt Spray (Fog) Testing

(for organic coatings on metal)

Specimens mounted between 15 and 30 degrees from vertical, such as to permit free settling of fog on surface; preferably parallel to horizontal flow of fog.

Salt solution: 5 ± 1 parts by weight NaCl in 95 parts distilled water; pH of salt solution shall be 6.5 to 7.2 when atomized at 35 C (95 F).

Exposure zone of salt chamber at 35 (+1.1 or -1.7) C.

Period as agreed on. Recommended periods 16, 24, 48, 96, 200, 240, 500, or 720 hours.

B-287 (7,21,31) Standard Method of Acetic Acid - Salt Spray (Fog) Testing

(for organic coatings on metal)

Specimens as in B-117

Salt solution: 5±1 parts by weight NaCl in 95 parts distilled water; pH of solution shall be adjusted with acetic acid to 3.1-3.3 as measured on a sample of the collected spray and measured at 25 C (77 F).

Temperature of exposure zone and periods of exposure as in B-117.

C-481 (16) Standard Method of Test for Laboratory Aging of Sandwich Constructions

Description of two aging cycles:

Cycle A:

1. Totally immerse specimen horizontally in water at 49±2 C (120±3 F) for 1 hour.
2. Spray with steam and water vapor at 93±3 C (200±5 F) for 3 hours.
3. Store at -12±3 C (10±5 F) for 20 hours.
4. Heat at 99±2 C (210±3 F) in dry air for 3 hours.
5. Repeat Step 2.
6. Heat in dry air at 99±2 C (210±30 F) for 18 hours.

Cycle B:

1. Totally immerse specimen horizontally in water at 49±3 C (120±5 F) for 1 hour.
2. Spray with hot water at 71±3 C (160±5 F) for 3 hours.
3. Store at -40±3 C (-40±5 F) for 20 hours.
4. Heat in dry air at 71±3 C (160±5 F) for 3 hours.
5. Repeat Step 2.
6. Heat in dry air at 71±3 C (160±5 F) for 18 hours.

TABLE 5. (Continued)

C-510 (14) Standard Method of Test for Staining and Color Change of One- or Two-Part Joint Sealants

a) Expose for 100 hours in light and water exposure apparatus, Type A, conforming to ASTM G-23. Specimen temperature 140±5 F (60±3 C). Water temperature 75±4 F (24±2.4 C).

b) Expose other samples at 73.4±20 F (23±1.1 C) and 50±10 percent relative humidity for 14 days. Immerse in distilled water for 1 minute once a day (5 days per week).

C-669 (14) Standard Specification for Glazing Compounds for Back Bedding and Face Glazing of Metal Sash

Specimens prepared and exposed as in D-2249.

D-518 (28) Standard Method of Test for Surface Cracking Resistance of Stretched Rubber Compounds

(for soft rubber)

Continuous exposure of rubber test specimens, held under stain, to weather and sunlight at 45 degrees facing south or to air-ozone mixtures as in D-1149.

D-529 (11) Recommended Practice for Accelerated Weathering Test of Bituminous Materials

(modification of G-23 with optional cold air exposure in Daily Cycle B)

Daily Cycle A:

1.	Light only (140±5 F) (60±3 C) black panel temperature for	51 min
2.	Light with spray (spray water 45±5 F or 7±3 C at nozzle) for	9 min
	Total, 22 periods	60 min

Daily Cycle B:

1.	Water spray only (70±5 F) (21±3 C) for	1 hr
2.	Light exposure only (140±5 F) (60±3 C) for	1-1/2 hr
3.	Water spray only (70±5 F) (21±3 C) for	2 hr
4.	Light only (140±5 F) (60±3 C) for	16-1/2 hr
5.	Cold (refrigerator) (0±10 F) (-18±6 C) for	1-3/4 hr
	TOTAL	22-3/4 hr

D-750 (23) Recommended Practice for Operating Light- and Water-Exposure Apparatus (Carbon-Arc Type) for Artificial Weathering

Testing of Rubber Compounds.

(for vulcanized rubber) (modification of G-23)

Rubber specimens exposed either with or without elongation.

TABLE 5. (Continued)

D-756 (27) Standard Methods of Test for Resistance of Plastics to Accelerated Service Conditions

Seven test procedures which prescribe conditions for different types of exposure: six cover exposure at graduated levels of temperature and extremes of humidity; the seventh covers alternate exposure to high and low temperatures.

Procedure	Cycle or Exposure
A	24 hrs at 60 C (140 F), 88 percent relative humidity 24 hrs at 60 C (140 F) in oven
B	72 hrs at 60 C (140 F) in oven
C	24 hrs at 70 C (158 F), 70-75 percent relative humidity 24 hrs at 70 C (158 F) in oven
D	24 hrs at 80 C (176 F) over water 24 hrs at 80 C (176 F) in oven
E	24 hrs at 80 C (176 F), 70-75 percent relative humidity 24 hrs at -40 C (-40 F) or -57 C (-20.6 F) as specified 24 hrs at 80 C (176 F) in oven 24 hrs at -40 C (-40 F) or -57 C (-70.6 F) as specified
F	24 hrs at 38 C (100.4 F), 100 percent relative humidity 24 hrs at 60 C (140 F) in oven
G	24 hrs at 49 C (120.2 F), 100 percent relative humidity 24 hrs at 49 C (120.2 F) in oven

D-822 (21) Recommended Practice for Operating Light- and Water-Exposure Apparatus (Carbon-Arc Type) for Testing Paint, Varnish, Lacquer, and Related Products

(for organic coatings) (modification of G-23)

Exposure apparatus, as in G-23, specified in Table 1 for various panel sizes.

Duration of exposure may be mutually agreed on or may be the number of hours required to produce a mutually agreed on change in the test specimens or in a standard sample.

D-904 (16) Standard Recommended Practice for Determining the Effect of Artificial (Carbon-Arc-Type and Natural Light on the Permance of Adhesives (see Table II for outdoor exposure).

Specimens exposed to carbon-arc, air temperature 35-50 C, for 10 hours or until significant change is observed. Specimens mounted vertically or at angle not over 30 degrees such that the light from the arc has a normal incidence on the specimens.

D-1037 (16) Standard Methods of Evaluating the Properties of Wood-Based Fiber and Particle Panel Materials

(for wood-based hardboard, particleboard)

TABLE 5. (Continued)

D-1037 (continued)

Accelerated aging cycle, Sections 118-123:

1.	Immerse in water at 120±3 F (49±2 C) for	1 hr
2.	Spray with steam and water vapor at 200±5 F (93±3 C) for	3 hr
3.	Store at 10±5 F (-12±3 C) for	20 hr
4.	Heat at 210±3 F (99±2 C) in dry air for	3 hr
5.	Repeat Step 2	3 hr
6.	Heat in dry air at 210±3 F (99±2 C) for	18 hr
	TOTAL	48 hr

D-1101 (16) Standard Method of Test for Integrity of Glue Joints in Structural Laminated Wood Products for Exterior Use

1. Impregnate specimens with water in an autoclave by means of two vacuum-pressure cycles (specimen immersed in water):
   - 1.1 Vacuum applied 20-25 in. (508-635 mm) Hg for 15 minutes
   - 1.2 Pressure applied 150±5 psi (1034±34 kN/m^2) for 2 hours

   Total time, 2 cycles, approximately ....4-1/2 hr

2. Dry in circulating air oven at 80-85 F (27-29 C), 25-30 percent relative humidity, circulation at a rate of

   500±50 ft (150±15 m)/min .... 91-1/2 hours

   Repeat Steps 1 and 2 for total of .... 8 days

D-1148 (28) Standard Method of Test for Discoloration of Vulcanized Rubber: Organic Finish Coated or Light Colored

Exposure to ultraviolet light source: calibrated sunlamp in test chamber.

D-1149 (28) Standard Method of Test for Accelerated Ozone Cracking of Vulcanized Rubber

Specimens under tensile strain exposed to various air-ozone mixtures in a chamber at various temperatures from ambient to 70 C (158 F)

D-1151 (16) Standard Method of Test for Effect of Moisture and Temperature on Adhesive Bonds

Test specimens preconditioned for 70 days at 50±2 percent relative humidity and 23±1 C (73.4±1.8 F), then subjected to designated exposure conditions as in Table II for prescribed time.

TABLE 5. (Continued)

Standard Test Exposures (D-1151)

Test Exposure Number	Temperature[(a)] deg. C.	deg. F.	Moisture Conditions
1	-57	-70	as conditioned
2	-34	-30	as conditioned
3	-34	-30	presoaked[(b)]
4	0	32	as conditioned
5	23	73.4	50 percent relative humidity
6	23	73.4	immersed in water
7	38	100	88 percent relative humidity
8	63	145	oven, uncontrolled humidity
9	63	145	over water[(c)]
10	63	145	immersed in water
11	70	158	oven, uncontrolled humidity
12	70	158	over water[(c)]
13	82	180	oven, uncontrolled humidity
14	82	180	over water[(c)]
15	100	212	oven, uncontrolled humidity
16	100	212	immersed in water
17	105	221	oven, uncontrolled humidity
18	149	300	oven, uncontrolled humidity
19	204	400	oven, uncontrolled humidity
20	260	500	oven, uncontrolled humidity
21	316	600	oven, uncontrolled humidity

(a) The tolerance for test temperature shall be ±1 C or ±1.8 F up to 82 C or 180 F and ±1 percent for temperatures above 82 C or 180 F.

(b) Presoaking shall consist of submerging specimens in water and applying vacuum at 50 cm (20 in.) of mercury until weight equilibrium is reached.

(c) The relative humidity will ordinarily be 95 to 100 percent.

specimens are tested

(a) under conditions at which they are exposed;

(b) immediately after conditioning for 4 hrs at 50±2 percent relative humidity and 23±1 C (73.4±1.8 F);

(c) immediately after conditioning for 7 days at 50±2 percent relative humidity and 23±1 C (73.4±1.8 F).

TABLE 5. (Continued)

D-1183 Standard Methods of Test for Resistance of Adhesives to Cyclic Laboratory Aging Conditions.

Cyclic exposure to high and low temperatures, high and low relative humidities as defined in table below.

Cycles: Test Procedures

Procedure	Name	Period hrs	Temperature deg. C.	Temperature deg. F.	Relative Humidity percent
A	Interior	24	23±1.1	73.4±2	85 to 90
		24	48.5±3	120±5	less than 25
		72	23±1.1	73.4±2	85 to 90
		48	48.5±3	120±5	less than 25
B	Interior	48	60±3	140±5	less than 25
		48	38.5±2	100±3.5	85 to 90
		8	-18±2	0±3.5	about 100
		64	38.5±2	100±3.5	85 to 90
C	Exterior, land and air	48	71±3	160±5	less than 10
		48	23±1.1	73.4±2	immersed in water
		8	-57±3	-70±5	about 100
		64	38.5±2	100±3.5	about 100
D	Exterior, Marine	48	71±3	160±5	less than 10
		48	23±1.1	73.4±2	immersed in substitute ocean water
		8	-57±3	-70±5	about 100
		64	23±1.1	73.4±2	immersed in substitute ocean water

D-1499 (27) Standard Recommended Practice for Operating Light- and Water-Exposure Apparatus (Carbon-Arc Type) for Exposure of Plastics (modification of G-23)

Black panel temperature (spray off) 63±5 C (145±9 F) but lower temperature may be used if specimens are temperature sensitive. If water spray is not required, proceed as in G-25.

D-1501 (27) Standard Recommended Practice for Exposure of Plastics to Fluorescent Sunlamp

Definition of three exposure conditions for plastics:

Procedure A.	Exposure to fluorescent sunlamp at ambient temperature
Procedure B.	Exposure to fluorescent sunlamp in heated air at 50-60 C (131-140 F)

TABLE 5. (Continued)

D-1501 (27) (continued)

Procedure C. Exposure to fluorescent sunlamp at 55-60 C (131-140 F) with interruptions for exposure in fog chamber. Cycle as follows:

1. Fog chamber........................ 2 hrs
2. Heat and light as in Procedure B... 2 hrs
3. Fog chanber......................... 2 hrs
4. Heat and light as in Procedure B...18 hrs

Total..............................24 hrs

for total of 240 hrs

Turntable and fog chamber recommended: Figures 3, 4, ASTM Recommended Practice D-795 (discontinued)

D-1565 (28) Standard Specification for Flexible Foams Made from Polymers or Copolymers of Vinyl Chloride

Air oven aging test, sections 22-27: Exposure of specimens in forced-ventilation oven for 22 hours at 110 C (212 F)

D-1654 (21) Standard Method of Evaluation of Painted or Coated Specimens Subjected to Corrosive Environments

Scribe the specimen with a special tool vertically, near the center, penetrating the coating. For flat specimens, begin the scribe at approximately 1/2 inch from one edge and continue to 1/2 inch from the opposite edge.

Expose the specimen as in B-117, B-287, D-1014, or by any other method as mutually agreed.

Treat as in Method A or B:

Method A: Rinse with a gentle stream of water at 100±10 F (38±5 C) and clean with an air blast as prescribed.

Method B: Rinse with a gentle stream of water at 100±10 F (38±5 C) and scrape with a tool as prescribed.

D-1735 (21) Standard Method for Water Fog Testing of Organic Coatings

Specimens 15 degrees from vertical and parallel to horizontal flow of fog. Exposure zone of fog chamber 100±2 F unless otherwise specified. Recommended exposure period intervals of 24 hours to the total of 96, 192, 504, or 1008 hours.

D-1754 (11) Standard Method of Test for Effect of Heat and Air on Asphaltic Materials (Thin-Film Oven Test)

Heating 1/8 inch film of asphaltic specimen in oven at 163 C (325 F) for 5 hours.

D-1870 (27,28) Standard Method of Test for Elevated Temperature Aging Using a Tubular Oven

(for polymers)

Specimens heated at specified temperature in tubular oven and changes in physical properties measured.

TABLE 5. (Continued)

D-2126 (26) Standard Method of Test for Resistance of Rigid Cellular Plastics to Simulated Service Conditions

Specimens conditioned and exposed to one of seven conditions of temperature and relative humidity:

Procedure	Temperature deg. C.	Temperature deg. F.	Relative Humidity percent
A	23±1	73.4±1.8	50 2
B	-29±3	-20.2±5.4	—
C	38±1	100.4±1.8	90 - 100
D	60±1	140±1.8	90 - 100
E	70±1	158±1.8	--
F	70±1	158±1.8	90 - 100
G	100±1	212±1.8	--

D-2246 (21) Standard Method for Testing Finishes on Primed Metallic Substrates for Resistance to Humidity - Thermal Cycle Cracking

Specimens in rack 0 to 30 degrees from vertical

Cycle as follows:

1. Humidity cabinet, 100 F (38 C)
   100 percent relative humidity..............24±1/2 hrs
2. Cold box, -10±3 F (-23±2 C)..............20±1/2 hrs
3. Room temperature.........................4±1/2 hrs

TOTAL 48 hrs

Run for 15 cycles or as mutually agreed on.

D-2247 (21) Standard Method for Testing Coated Metal Specimens at 100 Percent Relative Humidity

Specimens placed in humidity chamber in such a way as to permit condensation on surfaces. Temperature of saturated air 38±1 C (100±2 F). Test may be used as a continuous humidity test or as part of a cycle.

D-2248 (21) Standard Method of Test for Detergent Resistance of Organic Finishes

(for organic coatings)

Test for resistance to detergent of organic coatings on metal panels by suspending the specimens vertically and immersing at least half of the surface area in a standardized detergent solution at 74±1 C (165±2 F), the solution being replaced every 168 hours. Period of test not prescribed.

TABLE 5. (Continued)

D-2249 (14,21) Standard Method of Predicting the Effect of Weathering on Face Glazing and Bedding Compounds on Metal Sash

(for sealants used in glazing and bedding) (modification of G-23)

Specimens knifed and pressed into a special aluminum and glass assembly to simulate face glazing and bedding.

One of the Units of Type D through G of G-23 used for exposure, with addition of 102-18 cycling cam and black panel thermometer accessories.

Specimens exposed vertically with surface of sealant facing light source, at black panel temperature of 60±3 C (140±5 F), with 102-18 cycling cam.

Specimens examined after each 100-hour exposure to a maximum of 300 hours.

D-2366 (21) Standard Method for Accelerated Testing of Moisture Blister Resistance of Exterior House Paints on Wood

Expose specimens in a blister box or cabinet containing water at a specified temperature; air temperature 100±2 F (38±1 C), 95-100 percent relative humidity. Exterior air temperature 77±2 F (25±1 C); relative humidity 50±5 percent.

D-2445 (26) Standard Method of Test for Thermal Oxidation Stability of Propylene Plastics

Exposure time determined for pellets of plastic to become embrittled in an oxygen atmosphere at 150 C (302 F)

D-2559 (16) Standard Specification for Adhesives for Structural Laminated Wood Products for Use Under Exterior (Wet Use)

Exposure Conditions

Section 13:

Test for resistance to delamination during accelerated exposure:

1. Impregnate specimens with water in an autoclave by means of two vacuum-pressure cycles (specimen immersed in water)
    1.1 Vacuum of at least 635 mm (25 in) Hg applied for 5 min.
    1.2 Pressure of 5.27±0.141 kg/cm^2 (75±2 psi) applied for 1 hour.

    Total time, 2 cycles, approximately.........2-1/6 hrs
2. Dry in an oven at 65±2 C (150±3.6 F),

    relative humidity 15 percent or less, for........21-22 hrs
3. Subject specimens to steam at 100 C (212 F) for..1-1/2 hrs
4. Add water, apply a pressure of 5.27±0.141 kg/cm^2 (75±2 psi) for 40 min.

    Repeat Steps 1 and 2; total test period.......... 3 days

TABLE 5. (Continued)

D-2565 (27) Standard Recommended Practice for Operating Xenon-Arc Type (Water-Cooled) Light and Water-Exposure Apparatus for Exposure of Plastics

(modification of G-26)

Water-cooled xenon-arc, Types A, AH, B, BH in G-26

Details on lamps, filters, operating procedures for equipment but otherwise not on exposure conditions.

D-2803 (21) Standard Method of Test for Filiform Corrosion Resistance of Organic Coatings on Metal

(Filiform corrosion is a special type which occurs under coatings on metal and is characterized by a threadlike structure and direction growth. This usually occurs between 21 and 35 C (70-95 F) and 60 to 95 percent relative humidity.)

<u>Test procedure</u>:

1. Scribe specimens as mutually agreed on. Recommended procedure: D-1654.
2. Expose specimens in a salt-spray cabinet as in B-117 for at least 4 hours but not more than 24 hours.
3. Remove specimens from the cabinet and rinse thoroughly with distilled or demineralized water.
4. Place the wet specimen in a humidity cabinet at 25±1.7 C (77±3 F) and 85±2 percent relative humidity unless otherwise specified.
5. Inspect the specimens at intervals of approximately 168 hrs.

The normal test period is 6 weeks (1008 hours) but other periods may be used as mutually agreed.

D-2831 (20,21) Standard Method of Test for Evaluating the Ability of a Latex Paint to Resist Efflorescence from the Substrate, Parts 20, 21.

Prepare test specimens from asbestos cement shingle blanks and latex paint as prescribed.

<u>Test procedure</u>:

1. Fill container with tap water so that the water level is 1 to 1-1/2 inch (2.5-3.5 cm) below the surface of the panels when in place.
2. Regulate temperature of water to 40±3 C (104±5 F).
3. Place panels on pan over the water, painted side down, and expose for 16-17 hours.
4. Turn panels over; dry 1-2 hours or more, flat, with painted side up.

TABLE 5. (Continued)

D-2898 (16) Tentative Methods of Test for Durability of Fire-Retardant Treatment of Wood

Description of apparatus, test specimens, exposure cycles

(Methods A and B)

Method A (cycle):

1. Water spray at 35-60 F (2-16 C)..................96 hrs
2. Drying in moving air stream at 135-140 F (57-60 C)..............................72 hrs

TOTAL 1 week

Method B (cycle): Ultraviolet light from sunlamps is evenly distributed over the specimen surface.

1. Water spray at not over 90 F (32 C).............. 4 hrs
2. Drying at 150±5 F (63±3 C)........................ 4 hrs
3. Repeat water spray as in Step 1.................. 4 hrs
4. Repeat drying as in Step 2........................ 4 hrs
5. Rest... 8 hrs

TOTAL 24 hrs

Repeat for total of 1,000 hrs.

D-2918 (16) Standard Recommended Practice for Determining Durability of Adhesive Joints Stressed in Peel

Clamp upper end of test panel to frame and attach desired weight to lower end. Force on adhesive should be a multiple of 450 g (1.0 lb). Suggested stress 25, 50, 75 percent of normal peel strength as measured by D-1876.

Expose specimen to one of Standard Test Environments in Table II. Test at least 3 specimens under each set of conditions (test environment and stress)

Standard Test Environments

Test Environment Number	Temperature (a)	Moisture Conditions (b)
1	23 C (73.4 F)	immersed in distilled or deionized water
2	23 C (73.4 F)	50 percent relative humidity
3	23 C (73.4 F)	15 percent relative humidity
4	35 C (75 F)	90 percent relative humidity
5	35 C (95 F)	100 percent relative humidity

(continued)

TABLE 5. (Continued)

Standard Test Environments (continued)

Test Environment Number	Temperature[(a)]	Moisture Conditions[(b)]
6	50 C (122 F)	90 percent relative humidity
7	50 C (122 F)	100 percent relative humidity
8	35 C (95 F)	5 percent salt fog
9	Ambient (outdoors)	Ambient (outdoors)
10	Other (Specify)	Other (including aqueous solutions or nonaqueous liquid (specify)

(a) The tolerance for the test temperature shall be ±1 C or ±1.8 F for environments 1 to 8.

(b) The moisture conditions may be provided by controlling the relative humidity of a box, room, or other chamber by any convenient means.

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G-25 (24,30) Standard Recommended Practice for Operating Enclosed Carbon-Arc Type Apparatus for Light Exposure of Nonmetallic Materials

Description of apparatus and operating procedure for carbon-arc light exposure apparatus without specifications for exposure conditions except as noted.

Exposure intended to simulate G-24 Sunlight Exposure (Method A) or Daylight Exposure (Method B).

Specimens mounted vertically unless vertical mount is impracticable. Temperature measurement and control based on black panel thermometer unit.

Two Types of exposure are described:

Method A. Continuous exposure to light and

Method B. Alternate exposure to light and darkness

In Method A, the relative humidity is adjusted to 30±5 percent at the exit of air from the chamber, unless otherwise specified. Black panel temperature is adjusted to 63±3 C (145±5 F). Controls are adjusted so that 20 hours continuous operation produces 18±2 standard fading hours as determined by color standards.

In Method B, the relative humidity is adjusted to 35±5 percent during the light-on period and to 90±5 percent during the light-off period. Black panel temperature at equilibrium during the light-on period shall be 63±3 C (145±5 F).

Unless otherwise specified, controls shall be adjusted for a cycle of 1 hour darkness and 3 hours light.

Apparatus is calibrated in terms of standard fading hours by means of color standards.

TABLE 5. (Continued)

G-26 (30) Standard Recommended Practice for Operating Light- and Water-Exposure Apparatus (Xenon-Arc Type) for Exposure of Nonmetallic Materials

(formerly E-239)

Description of apparatus and operating procedures for xenon-arc light and water exposure apparatus without specifications for exposure conditions except as noted.

Specimens mounted vertically.

Temperature measurement and control based on black panel thermometer unit.

Water strikes specimens as a fine spray, evenly distributed, at a temperature of 60.8±9 F (16±5 C); pH of water 6.0-8.0.

Types of light and water exposure apparatus described (6):

A, AH, B, BH, C, D

Types A, AH, B, BH are water-cooled.

Types C and D are air-cooled.

Types A, AH, C, D have automatic humidity control.

Types A, AH, B, BH, D have automatic temperature control.

All types have automatic cycle control.

G-27 (30) Standard Recommended Practice for Operating Xenon-Arc Type Apparatus for Light Exposure of Nonmetallic Materials

Description of apparatus and operating procedure for xenon-arc exposure apparatus without specification for exposure conditions except as noted.

Exposure intended to simulate G-24 Sunlight Exposure (Method A) or Daylight Exposure (Method B).

Specimens mounted vertically.

Temperature measurement and control based on black panel thermometer unit.

Apparatus may use water-cooled or air-cooled xenon-arc.

Types of exposures (Methods A and B) are described as in G-25 with the same conditions. However, the apparatus is not calibrated in terms of standard fading hours but color standards are used as references with test specimens.

TABLE 6. CRITERIA FOR OUTDOOR AND ACCELERATED WEATHERING METHODS AND EVALUATIVE TECHNIQUES IN ASTM STANDARDS (1971)

B-117 (7,21,31) (for organic coatings on metal)

Extent of corrosion(a)

Visual examination(b)

B-287 (7,21,31) (for organic coatings on metal)

Extent of corrosion(a)

Visual examination(b)

C-481 (16) (for sandwich constructions)

Measurement of properties before and after aging:

Shear test, C-273
Compressive strength, C-364
Delamination strength, C-363
Tension test, C-297
Flatwise flexure test, C-393
Climbing Drum Peel Test, D-1781

C-488 (14) (for finishes for thermal insulation)

Blistering, chalking, crazing, cracking, discoloration, flaking, and shrinkage(a)
Visual observation(b)

C-510 (14) (for joint sealants)

Change in color of sealant as compared with control specimen(a,b)

C-669 (14) (for sealants used in back bedding and face glazing)

Changes not greater than illustrated by photographs in D-2249(a,b)

Surface cracking and peeling, No. 5, Plate No. 1; Deep-bead cracking, No. 5, Plate No. 2; Loss of adhesion, Nos. 4,5, Plate No. 3; Wrinkling, Nos. 4,5, Plate No. 4; Oil exudation, No. 5, Plate No. 5

D-518 (28) (for soft rubber)

Appearance of first minute surface cracks(a) by visual observation, using 7X magnification(b)
Percent elongation of tapered specimens(a,b)

D-750 (28) (for vulcanized rubber)

Visual examination(b) for the degree and number of cracks(a) and for evidence of crazing(a)
Tensile strength and ultimate elongation, D-412(a,b)
(Specimen preparation for tests, D-15)

See footnotes on page 96.

TABLE 6. (Continued)

D-756 (27) (for plastics)

Noticeable changes in surfaces, outline, general appearance including changes in color, surface irregularities, odor, splits, as in D-883[a,b]

Changes in weight and dimensions[a,b]

D-822 (21) (for organic coatings)

Visual examination[b] for:
- Blistering, D-714[a]
- Chalking, D-659[a]
- Checking, D-660[a]
- Cracking, D-661[a]
- Erosion, D-662[a]
- Flaking (Scaling), D-772[a]
- Rusting, D-610[a]
- Change in color, E-308, D-2244[a,b]
- Change in reflectance, E-97[a,b]
- Change in specular gloss, D-523[a,b]

D-904 (16) (for adhesives)

Visual change in appearance[a,b]

Results of physical and chemical tests[a,b]

D-1006 (21) (for organic coatings on wood)

Recording on standard form as in D-1150[a,b]

D-1014 (21) (for organic coatings on steel)

Visual examination[b] for:
- Blistering, D-714[a]
- Chalking, D-659[a]
- Rusting, D-610[a]

D-1037 (16) (for wood-based hardboard, particleboard)

Visual inspection[b] for delamination or other disintegration[a]

Measurement of cupping and twisting[a,b]

Changes[a,b] in
- Static bending
- Lateral nail resistance
- Nail withdrawal
- Water absorption
- Nail Head Pull-Through

TABLE 6. (Continued)

D-1101 (16) (for adhesive joints in structural laminated wood)

Percentage delamination(a,b) $= \dfrac{\text{Length of open glue joint area on end-grain surface}}{\text{Total length of end-grain joints}} \times 100$

D-1148 (28) (for vulcanized rubber)

Change in color or test specimen in relation to original sample(a) as expressed by numerical rating of degree of discoloration(b)

D-1149 (28) (for vulcanized rubber)

Cracking(a)

Time to first observed cracking, using 7X magnification, except for triangular specimens of Method D-1171, where the magnification shall be 2X(b)

D-1151 (16) (for adhesive bonds)

Various strength properties measured before and after exposure(a,b)

Performance A = (A/C) x 100(a)

Performance B = (B/C) x 100(a), where

A = average strength after exposure

B = average strength after exposure

C = original strength after conditioning

D-1167 (11,21) (for asphalt emulsion coatings on built-up roofs)

Heat Test(b), Section 12:

Examine coating for sagging, slipping, blistering(a).

Record extent of sagging or slipping past reference line in decimals of inch, and the presence or absence of blisters.

Water Resistance(b), Section 14:

Examine for blistering and re-emulsification as evidenced by asphalt particles in the water(a).

D-1183 (16) (for adhesives)

Change in appearance(a,b)

Change in strength properties as(a,b):

- Cleavage strength of bond, D-1062
- Impact strength of bond, D-950
- Peel or stripping strength of bond, D-903
- Flexural strength of assemblies, D-1184
- Shear by tension loading, D-906, D-1002
- Tensile properties of bond, D-897
- Tensile properties of cross-lap specimens, D-1344

TABLE 6. (Continued)

D-1435 (27) (for plastics)

- Appearance Properties
  - COLOR(a)
    - Color difference(b), D-1535, D-1729, D-2244, E-308
    - Yellowness(b), D-1925, E-313
  - GEOMETRIC DISTRIBUTION(a)
    - Gloss(b), D-523, D-2457
    - Haze(b), D-1003
    - Transparency(b), D-1746
- Electrical Properties(a)
  - High-voltage, low-current arc resistance, D-495(b)
  - Dielectric breakdown, dielectric strength, D-149(b)
- Mechanical Properties(a)
  - Dimensional change, D-1042, D-1204(b)
  - Impact, D-256(b)
  - Indentation hardness, D-785, D-2240(b)
  - Stiffness, D-747(b)
  - Tear resistance, D-1004, D-1938(b)
  - Tensile and elongation properties, D-638, D-882, D-1708, D-1923, D-2289(b).

D-1499 (27) (for plastics)

Evaluation covered in ASTM methods or specifications for specific materials.

D-1501 (27) (for plastics)

Color change (none, slight, appreciable, extreme)(a,b)

Surface changes (as chalking(b), dulling) by visual observation(a)

Deep-seated changes (as checking, crazing, warping, discoloration)(a,b)

Physical and chemical tests to determine extent of degradation(a,b)

D-1565 (28) (for vinyl foam)

Percentage change in indentation-load deflection or compression-load deflection(a,b)

D-1641 (21) (for varnishes)

General appearance and visual observation(b) or checking(a), cracking(a), discoloration(a), dulling(a), recoatability(a)

TABLE 6. (Continued)

D-1654 (21) (for organic coatings on metal)

Loss of adhesion(a), extent of blistering(a), corrosion(a), and rust(a) are evaluated by measuring the distance between the scribed mark and the edge of the unaffected area of the finish(b). Numerical ratings are based on these measurements.
Rating Schedule No. 1: Report maximum and minimum creepage from the scribe and use numerical rating(b).
Rating Schedule No. 2: Rate by percentage failure as corrosion spots, blisters. Recommend use of counting grid. Photographic reference standards may be used as in D-610, D-714(b).

D-1735 (21) (for organic coatings)

Visual observation(a)
Blistering, D-714(b)
Rusting, D-610(b)

D-1754 (11) (for asphalt)

Reduction of penetration as in D-5(a,b)
Loss of weight(a,b)

D-1828 (16) (for adhesive-bonded joints and structures)

General appearance dimensions (including warpage)(a,b)
Change in properties(a,b)

D-1870 (27,28) (for polymers)

Change in physical properties in percent(a,b)

D-2126 (26) (for rigid cellular plastics)

Change in appearance(a,b)
Percentage change in weight, dimensions, and other properties(a,b)

D-2246 (21) (for coated metal)

Cracking(a) by visual observation(b), using a grid, and counting number of grid squares within which one or more cracks is visible.

D-2247 (21) (for coated metal)

Degradation of finish as observed visually(b):
- Blistering, D-714(a)
- Corrosion, D-1654(a)
- Rusting, D-610(a)
- Loss of adhesion, D-2197(a,b)
- Change in Indentation Hardness, D-1474(a,b)
- Change in Specular Gloss, D-523(a,b)

TABLE 6. (Continued)

D-2248 (21) Not prescribed but the usual coatings tests are applicable

(D-523, D-610, D-714, D-1474, D-1654, D-2197, D-2248)

D-2249 (14,21) (for sealants used in glazing and bedding)

Visual estimation(a), using numerical scale(b), of loss of adhesion(a), surface cracking and peeling(a), deep bead cracking(a), wrinkling(a), and oil exudation(a).

D-2366 (21) (for exterior house paints on wood)

Evaluate degree of blistering and percentage of test area on which blisters occur after 1, 2, 4, 7, and 14 days(a) by D-714(b). Percentage of panel area on which blisters have formed may be determined by means of a counting grid(b):

$$\text{Area blistered, percent} = \frac{\text{Number of squares covering blisters}}{\text{Total number of squares over test area}} \times 100$$

D-2445 (26) (for propylene plastics)

Pellets tested for brittleness(a) by lightly applying a light compressive force manually and observing whether they crush easily to powder form(b). The specimen has failed if at least 8 of the first 10 pellets tested are completely embrittled.

D-2559 (16) (for adhesives)

Section 13: Delamination on end-grain surfaces(a,b):

$$\text{Percentage delamination} = \frac{\text{Length of open glue joint area}}{\text{Total length of bond line}} \times 100$$

D-2565 (27) (for plastics)

Appearance(a,b)

D-2803 (21) (for organic coatings on metal)

Note filiform corrosion as threads initiating at the scribe(a). Description of photograph may be used for reporting(b).

D-2830 (20,21) (for organic coatings on wood)

Visual examination(b) for appearance(a), mildew(a), and film failure(a).

Test for adhesion between the primer and top coat(a,b):

Cut a small X to penetrate the substrate slightly.

Apply cellophane tape to cross the X; press; remove quickly.

D-2831 (20,21) (for latex paints on asbestos cement shingles)

Visual observation(b) of efflorescence(a), rated as none, slight, moderate, or severe.

D-2898 (16) (for fire-retardant treatment of wood)

Fire tests as in E-84, E-286, D-108(a,b)

TABLE 6. (Continued)

D-2918 (16) (for adhesive joints)

Rate of peel of adhesive from joint(a,b)
Plot distance peeled versus time and divide the curve into six equal parts. If the peel rate varies less than a factor of 2 over all six parts of the curve, obtain the average peel rate (slope of the curve) for the specimen. To assess durability, compare peel rates under difference levels of stress and environment and plot log peel versus stress.

D-2919 (16) (for adhesive joints)

Maximum, minimum, average length of time to failure of stressed specimens exposed to environment(a,b)

G-7 (30) (for nonmetallic materials)

One or more of the following may be pertinent:
Appearance, electrical and mechanical properties, D-1435(a,b)
Appearance, visual observation of checking, cracking, discoloration, dulling, and recoatability, D-1641(a,b)
Visual observation(b) of
  Blistering, D-714(a)
  Chalking, D-659(a)
  Checking, D-660(a)
  Color Change, G-24(a)
  Cracking, D-661(a)
  Erosion, D-662(a)
  Flaking (scaling), D-772(a)
  Rusting, D-610(a)
  Number of cracks in film as determined by high-voltage spark and counting grid, D-1670(b)

G-11 (21,30) (for pipeline coatings)

Visual observation(b) of blistering(a), checking(a), cracking(a), corrosion(a), undercutting from intentional scribe(a,b).

G-23 (24,27,30)
Evaluation covered in ASTM methods or specifications for specific materials.

G-24 (24,36)
Evaluation covered in ASTM methods or specifications for specific materials.

G-25 (24,30) (for nonmetallic materials)
Evaluation covered in ASTM methods or specification for specific materials.

G-26 (30) -- Evaluation covered in ASTM methods for specifications for specific materials.

G-27 (30) -- Evaluation covered in ASTM methods or specifications for specific materials.

(a) Criterion.
(b) Evaluative technique or test.

Accelerated Aging Procedures Without Applied Stress. Most accelerated aging methods are used without the application of stress to the test specimen. While these methods are appropriate for certain non-structural components, they are commonly used with structural components as well. They may involve continuous exposure to a given set of conditions, thermal cycling, humidity manipulation, water spray, etc. Tests are designed to determine the resistance of the material or component to such environmental conditions as temperature, moisture, oxidation, pollutants, solvents, acid, alkali, dust, sand, wind, salt, rot organisms, and bacteria.

(1) *Techniques to Intensify Sunlight, Simulate Ultraviolet Radiation, and Combine Light and Water Exposure Conditions.* Since it is seldom practical to test a material for 20 years, efforts have been made to develop accelerated testing methods which can be used to predict actual service life. In general, these efforts have been disappointing. The major difficulty is that "weather" cannot be accelerated. In accelerated aging tests, one of the degrading forces (usually UV radiation) is increased above normal levels in an attempt to accelerate the rate of degradation of the material. EMMA and EMMAQUA* are devices designed to accomplish this.(119,120) The intensity of UV radiation is increased by a factor of eight in these devices. Even though these techniques intensify solar radiation, one report(121) showed that there was little correlation between data obtained from outdoor exposure of polypropylene and polyethylene using EMMA and data obtained from accelerated aging tests involving a light and water exposure apparatus.

In laboratory units, artificial sources are used to generate UV radiation. Figure 2 shows the UV energy distribution of several common UV sources in comparison to solar radiation. The xenon arc most closely matches solar radiation, although differences are still evident. Many of the reported accelerated tests used a carbon arc as the radiation source.

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* EMMAQUA is an EMMA device which periodically sprays water on the samples. (Desert Sunshine Exposure Tests, Inc., Phoenix, Arizona).

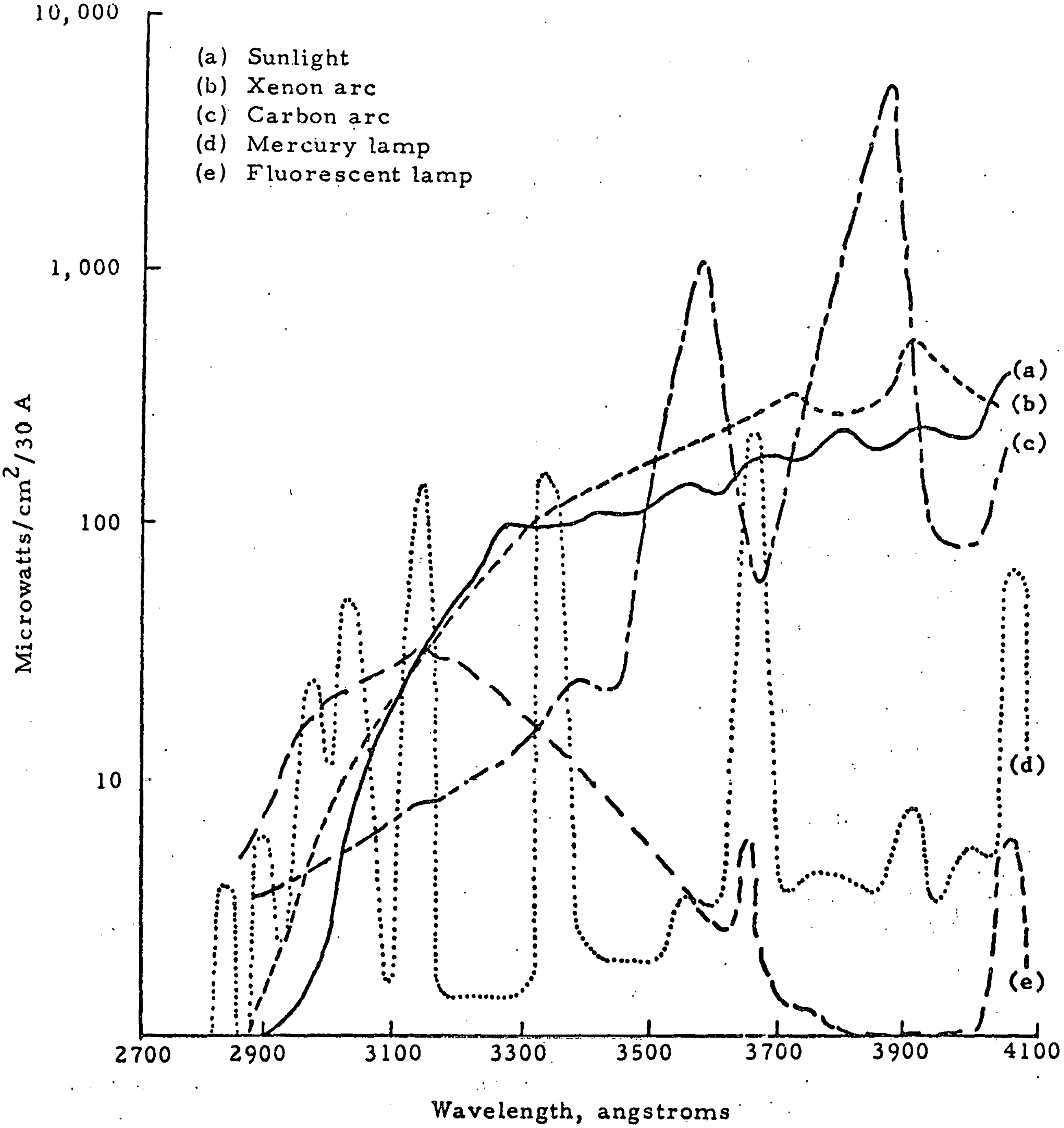


FIGURE 2. UV DISTRIBUTION OF VARIOUS UV SOURCES

The carbon arc is highly deficient in UV radiation between 0.30 and 0.34 μm, a range particularly damaging to polymers. The intensity of radiation from the various sources also fluctuates. Radiation from the carbon arc fluctuates in a highly erratic manner.(122) The sunshine carbon arc, which more closely approximates solar radiation (0.35 to 0.40 μm) than the enclosed carbon arc, consists of three filaments which burn sequentially. They were found to radiate at different intensities. Commercial xenon-arc lamps produce steady radiation (0.30 to 0.72 μm) which gradually decreases with time.(122)

Because of the variability in radiation frequency between the artificial UV sources and the sun, it is difficult to equate impinging artificial UV radiation with solar radiation. Usually, the test samples are located so that the incident radiation most closely approximates the integrated UV energy of the sun at some average condition (e.g., noon in the month of June). Attempts at increasing the radiation intensity to achieve acceleration will further distort the differences between the solar radiation and the artificial radiation. Therefore, tests are usually run continuously to accelerate the exposure time to UV radiation. Even this tactic is not ideal in that the cyclic nature of UV solar radiation is not simulated. Some accelerated-weathering devices have a night cycle, but this does not simulate the natural cyclic daytime intensity of solar UV radiation. The "night" cycles last only several minutes out of every hour.

In most accelerated testing devices, deionized, distilled water is sprayed on the samples periodically. Some devices even maintain a constant humidity. All maintain a constant temperature, and very few introduce any of the other important parameters. Clearly, artificial weathering does not simulate natural weathering. The only major accelerating parameter is UV exposure. Because natural degradation is a complex combination of many factors, it is not surprising that artificial weathering tests do not correlate with natural weathering tests. When one considers that outdoor exposure tests in Cleveland, Ohio, cannot be correlated with similar tests in Miami, Florida, because of the difference in climate, it is not surprising that laboratory tests, which simulate neither weather situation, cannot be correlated to either one.

The major use of laboratory weathering tests has been to rank materials in order of degradation stability and to evaluate the effectiveness of stabilizers and pigments.(122)

The "dew cycle"(124) is frequently used in light and water exposure apparatus. This consists of irradiating the sample with unfiltered light from a carbon arc while spraying the backs of the specimens with cold water to induce the deposition of dew on the exposed face. Other cycles used include boiling water, immersion in vapor, and water soaking alternating with exposure in the light and water apparatus.

A fluorescent sunlamp-blacklamp(125) has also been reported to yield accelerated aging results which correlate well with Florida and Arizona weathering of rigid polyvinyl chloride films(126) and the xenon arc light source has been used to accurately predict outdoor weathering of plastics (127). Another study(128) showed xenon arc weathering to compare favorably with outdoor weathering of plastics, especially when the samples were sprayed periodically with water. Studies to compare the light sources for artificial weathering have been reported.(129,130,131)

(2) Environmental Test Chambers and Specimen Size Criteria. Most laboratory accelerated testing is done in relatively small exposure cabinet facilities; hence the material sample size is small or only a model of a full-size system.

In plastic solar thermal collector systems where several plastic components having different physical properties (different extrusion processing histories, different thermal shrinkage values, dissimilar coefficients of thermal expansion, etc) are joined together, a small model of this system may not reflect the true aging deformation experienced in a life-size sample. For certain plastic solar thermal collector constructions, it would be advantageous to run accelerated tests on full-size systems.

(3) Continuous and Cyclic Testing. Exposure conditions may be held constant or cycled.

The use of various environmental conditions in a cyclic mode is basically sound, since the component is subjected to this type of environmental change in service. The problem is to accelerate the aging process by accentuating cyclic conditions. It should be noted, however, that increasing the severity of the conditions may not necessarily accelerate aging.

Cyclic tests are used to simulate alternating environmental exposures. Testing methods for laboratory aging of sandwich constructions and wood-base fiber or particle panel materials include ASTM Methods C-481-62 and D-1037-64(132), respectively. Both incorporate cycles of freezing, oven drying, steam or hot water spray, and water soaking.

An ASTM cyclic aging test for adhesives, D-1183-70(132) provides different test cycles of temperature and moisture exposures for interior and exterior services. Seven different testing methods are available for the cyclic aging of various plastic specimens as described by ASTM D756-56.(112) Each procedure in this test contains variations in the severity of temperature and moisture conditions.

(4) Accelerated Aging Tests with Applied Stress. In the development of weathering methods having an applied stress factor, the prime objective is to provide a means of evaluating structural components. A good measure of component strength will utilize short-term testing under a limited set of conditions. The stress level the component will undergo as a function of time will be the parameter of interest.

For the rheological response of a structural adhesive material to a static stress, Lewis(133) has proposed a model. The model identifies the types of damage at various levels of relative stress.

The relative stress is defined as the ratio of applied static stress to short-term bond strength. This particular type of model signifies that the strength of the short-term tests must be much greater than the design strength. The ultimate design strength of the short-term strength, based upon this model for adhesives, should be less than 25 percent, according to engineering standards. This model was developed for a specific material with the concept being (that the short-term test must be greater than the design strength) and will apply to most structural components. The distinction between short-term test results and strength retention with time is a good example of how such models can be useful in the development of relevant criteria.

In this section, testing methods will be described. They are crack propagation, dead load (sustained load) testing, cyclic load (fatigue) testing, and progressive load testing. Cyclic load testing and crack propagation are less advanced in their application to durability testing relative to progressive loading and sustained loading. In regard to these testing methods, many articles exist in the scientific literature describing their development, illustration, and applicability to durability.

(i) Crack Propagation. Fracture mechanics makes it possible to quantitatively measure a material's ability to resist fast crack extension in the presence of a flaw. Fast crack extension is associated with low toughness while slow crack extension will indicate high toughness.

Fracture mechanics have been used to evaluate the velocity of crack propagation in epoxy bond systems as reported by Mostovoy and Ripling,(134,135) The experiment utilized a contoured double cantilever beam adhesive specimen, a uniform double cantilever beam specimen, and a tensile specimen. The adherend was aluminum.

Recently, Ripling, Mostovoy, and Corten(136) concluded that fracture mechanics are applicable to the evaluation of structural adhesives. The test is designed so that energy lost during the course of crack extension is essentially zero. The major advantage is that all adherend parameters are extraneous to the value of fracture toughness. This allows (which is hopeless with peel and shear tests) a comparison of a chosen adhesive with various adherends.

A theory on crack propagation reported by Gurney and Amling(137) refers to the normal separation of elastic strips bonded to one another. If the adherend strips are relatively strong to resist bending, then the work of tearing can be extrapolated from a plot of load and deflection.

(ii) Dead Load Testing. The concept of dead load testing is defined as a sustained load to a material and the measurement of the time to failure or changes of defined parameters with time. This approach simulates the effects of long-term loading. While under a sustained stress, a material can be exposed to chosen environmental conditions closely simulating natural weather conditions.

Small specimens are evaluated by dead load testing. The smaller approach is common for 1-in. x 6-in. dogbone plastic specimens in order to evaluate adhesive-bonded lapjoints. Stress-rupture curves can be obtained from various sustained loads applied to individual specimens. This curve is a plot of a given strength factor versus time plotted on a semi-log or log-log scale.(138-145) Studies such as those by Cass and Fenner(138-140) show a subsequent flattening of the curve so that for many materials the curve can be extrapolated to obtain long-term predictions of performance from short-term data. The extrapolated value is referred to as the endurance limit and is defined as the stress level a material can withstand indefinitely. A finite time 10,000 hours has been arbitarily taken as a practical limit. Acrylic and methacrylate based plastics have been evaluated by dead load testing using a cantilever flexure test(140,141) with outdoor exposure. ASTM methods for measuring time-to-failure of plastics(112) and creep properties of adhesives(132) have been developed.

A dead load test consists of hanging weights attached to a specimen. As many as 90 specimens 2.5 cm in width and 14 cm long can be evaluated. Carter(146) has proposed an outdoor weathering rack in which lap-shear joints can be stressed up to 3000 psi. Wegman(147) has used both the Sharpe(148) jig with artificial environments and a stressed outdoor weathering frame in evaluating the durability of adhesive-bonded joints, and both were found promising for durability testing.

The basic principle behind dead load fixtures can be applied to many different materials for which sustained stress might be a factor. A major drawback exists in most cases with the dead load approach. This is due to the long time needed to obtain useful curves which can be extrapolated to give an endurance limit.

(iii) Cyclic Load Testing. The sustained load that a loadbearing material withstands is not constant load. The load varies constantly with environmental changes. A sandwich type construction with a paper honeycomb core and stressed steel facings is a good example. Surface temperatures as high as 160 F can exist on the steel facing when exposed to sunlight and would result in steel expansion. Contraction of the steel would occur in cold weather. A stress in the bond will result if the thermal expansion coefficients between core and skin materials is different. The result is unwanted differential movement between the two. This movement would place additional stresses on the composition of the sandwich panel, an effect called fatigue.

Hearle(149) published a report on fatigue in fibers and plastics in which he described a fatigue test apparatus used in his studies. This apparatus was not adapted for environmental aging, however.

Little(150) defined three kinds of fatigue tests: (1) S-N (preliminary tests), (2) P-N (life tests), and (3) P-S (strength tests). His discussion was based on the P-S-N model and described the three fatigue variables as:

S = fully reversed stress amplitude, or fully reversed strain amplitude

N = number of stress or strain cycles imposed

P = proportion failed prior to N stress or strain cycles.

(iv) Progressive Load Testing. Progressive loading is a means by which one can in a significantly shorter time determine the endurance limit of a stressed material than that required by the stress-rupture curve.

In 1948, E. Marcel Prot(151) presented a method of accelerating the cyclic fatigue testing of materials. Prot showed that by progressively increasing the load on a specimen by increments until failure occurs, one should be able to predict the stress level a specimen is capable of withstanding without rupture for an infinite time. As extrapolated from the stress-rupture curve, the stress level should be approximately the same as the endurance limit. This method consists of subjecting the specimen to progressively increasing loading rates resulting in failure. Mathematical equations are applied to the failing loads so that one can obtain a graph which can be related to the endurance limit.

Boller(152) modified the Prot equation to determine the endurance limit of glass-reinforced plastic laminates. His conclusions were that (1) the endurance limits from the Prot and sustained load methods were in good agreement at 10,000 hours duration and (2) the Prot method can be conducted on one material at one condition within 1 week, compared with 4 years by the Wohler (sustained load) method.

Prot's work was the catalyst of Loveless, Deeley, and Swanson(153) in their development of a hyperbolic equation relating rupture stress and time to break. The equation was:

$$(S_R - E)\, t_B = k \quad ,$$

where

S_R = stress at rupture

E = endurance limit

t_B = time to break

k = material constant.

They discovered it more useful to relate the endurance limit to the slope of the curve because of the substantial extrapolation involved and their equation was reliable in measuring long-term strength. Boller had related the endurance limit to the intercept after extrapolation.

Lewis, Kinmonth, and Kreahling(154) proved that the Prot method, modified by Loveless, Deeley, and Swanson gave a reasonable estimate of the long-term endurance strength of an adhesive joint.

V. General Considerations of Environmental Degradation Reactions and Mechanisms of Polymeric Materials

(a) Abrasion Resistance

Polymeric materials having superior optical clarity, abrasion resistance, and ability to withstand severe environmental exposure conditions have been the subject of several investigations. Polymeric materials having these properties can be used in periscope lenses, aircraft windshields, and covers for photovoltaic cell covers(155,156,157).

One of the earliest studies examines various commercial plastic materials for vision devices with the specific requirement of their ability to withstand severe abrasion testing. The overall object of this investigation was to find a polymer with abrasion resistance superior to that of methyl polymethacrylate and approaching that of glass. Abrasion data obtained with falling carborundum and measured by variations in surface gloss showed that such commercial plastics as methyl alpha-chloropolyacrylate, allyl diglycol carbonate, methyl polyacrylate, and crosslinked polyesters satisfied these requirements. A relative listing of commercial plastics and their abrasion resistance is given in Table 7(155).

Although this study was limited to abrasion testing of materials, many of the plastic substrates might find commercial application in present-day solar thermal collector systems.

Plastics having excellent abrasion resistance while maintaining good optical clarity also become an important factor with regard to their dirt retention capability. Easily abraded surfaces tend to collect more dirt than those having high abrasion resistance which in turn affects actual service life optical transmission qualities of plastic materials(158).

TABLE 7. ABRASION RESISTANCE OF COMMERCIAL PLASTICS

Materials Tested	Gloss After 800 Grams of Carborundum Impingement (ASTM Specification D673-44)
Plate Glass	99
Melamine Formaldehyde Coating on Cellulose Acetate	98-99
Allyl Diglycol Carbonate (CR-39 - PPG Industries)	97-98
Methyl Polyacrylate	97-98
Allyl Diglycol Carbonate Copolymer with Triallylcyanurate	97
Methyl Methacrylate-Methyl Acrylate Copolymer (1:10 ratio)	97
Methyl Alpha-Chloropolyacrylate	95
Methyl Methacrylate-Methyl Acrylate Copolymer (1:5 ratio)	94
Allyl Diglycol Carbonate Copolymer with Methyl Methacrylate	94
Methyl Polymethacrylate (Lucite - Du Pont and Plexiglas - Rohm and Haas)	92
Ethyl Polymethacrylate	92
Methyl Polymethacrylate	91
Butyl Polymethacrylate	89
Polyacrylate Coated with Methyl Polyacrylate (63-mil thickness)	89
Polyacrylate Coated with Ethyl Polyacrylate (10-mil thickness)	85
Methyl Methacrylate-Methyl Acrylate Copolymer (1:2 ratio)	85
Polyacrylate Coated with Methyl Polyacrylate (10-mil thickness)	85
Heat-treated Polystyrene	85
Polyacrylate Coated with Methyl Polyacrylate (20-mil thickness)	82
Polyacrylate Coated with Ethyl Polyacrylate (20-mil thickness)	77
Polystyrene	56

TABLE 7 (Continued)

Materials Tested	Gloss After 800 Grams of Carborundum Impingement (ASTM Specification D673-44)
Styrene Crosslinked Polytriethylene Glycol Maleates	
Styrene to Polyester Ratio: 2:10	96-98
3:10	96-98
5:10	96-98
7:10	96-98
9:10	95
Methyl Polymethacrylate Crosslinked Polytriethylene Glycol Maleates	
Polymethacrylate to Polyester Ratio:	
3:10	97-98
5:10	96
7:10	95
15:10	94
9:10	93
Styrene Crosslinked poly-(Polyethylene Glycol 200) Maleates	
Styrene to Polyester Ratio: 2:10	97-98
3:10	97-98
5:10	97-98
7:10	97-98
9:10	96
Methyl Methacrylate Crosslinked poly-(Polyethylene Glycol 200) Maleates	
Methyl Methacrylate to Polyester Ratio:	
3:10	97-98
5:10	97-98
7:10	97-98
9:10	97-98
15:10	96
Styrene Crosslinked poly-(Polyethylene Glycol 200) Itaconates	
Styrene to Polyester Ratio: 2:10	97-98
3:10	97-98
5:10	97-98
7:10	97-98
9:10	96

(b) Photochemical and Thermal Effects

The environmental aging of plastics is primarily a photooxidation-photothermal process and ultimately results in changes in mechanical (embrittlement), optical (discoloration), and electrical (increased dielectric loss) properties. Embrittlement results from molecular and/or morphological changes due to chain scissions and crosslinking reactions. Discoloration is associated with the formation of polyene chromophores. Dielectric loss increases with increases in polar group concentration produced by reactions with oxygen and other reactive species.

The principal steps in the degradation process may be described as

(1) Absorption of energy

(2) Chain scission (dissociation) and free radical formation

(3) Free radical chain reactions (with and without oxygen

(4) Radical combination to form new products (polymeric and nonpolymeric, with and without oxygen-containing groups).

Since most polymerics contain additives (stabilizers, plasticizers, processing aids, etc), changes in these low molecular weight materials also contribute to the degradation process. An example is the stabilizer system. It may contain one or several materials of relatively small molecular size and functions, through one of several mechanisms, to prevent available energy from reacting with the polymeric. However, with time, it is consumed and the degradadtion rate of the polymer accelerates. Moreover, as it is consumed, like most other additives, by-products can be formed that adversely affect materials properties. Molecular defects or impurities in the polymer also may be regarded as additives in the sense that they also cause a typical polymer performance and accelerate degradation.

Degradation in polymers can be defined in terms of the occurrence of one or more of a number of primary events within the material that produce changes <u>at the molecular level</u>. Principal among these primary events are chain scissions, crosslinking reactions, development of unsaturation, depolymerization, molecular rearrangement, volatile product formation, and attendant reactions with foreign moieties (oxygen, water vapor, etc). Certain events also can be anticipated in the low molecular weight additives present in the formulation. These principally are of the latter three types mentioned here.

Within the polymer, the occurrence of the events may be random, i.e., scattered throughout the bulk of the material or, more commonly, be concentrated at exposed surfaces of the polymer. The location at which primary events occur will be dependent on the nature of the degrading elements. UV degradation, for example, will be nonrandom. Degradation due to ionizing radiation, on the other hand, might be expected to be random.

In addition to primary events within and/or at the surface of materials, molecular level changes at materials interfaces must be considered. Here primary events of the types identified above will occur during the degradative process. However, because of multimaterial involvement, these are somewhat more complex and, therefore, less understood than those associated with individual polymerics. Further, mechanically induced stresses are more relevant precursors to failure at such interfaces.

Molecular level degradative changes in polymerics are generally, at some total level of change, accompanied or followed by changes in morphological properties, e.g., such properties as crystallinity, orientation, etc. The latter may be thought of as property changes at the microscopic level. At still greater levels of degradation, these molecular and microscopic level changes manifest themselves as macroscopic changes, e.g., changes in tensile strength, elongation, hardness, heat distortion temperature, etc.

As might be expected, measures of molecular and microscopic level changes, both of which occur earlier in the degradative process than macroscopic types, generally are of more importance as early predictors of degradation. Consequently, diagnostic tools that manifest such changes must, at the same level of instrumental sensitivity/precision, be of prime importance in studies directed toward prediction of service life. Thus, in such studies it is important that diagnostic tools be identified that can be used to quantize materials changes extremely early in the degradation process, i.e., primarily at the molecular and morphological (microscopic) levels. Moreover, a number of such tools must be available so that each of the principal modes of failure of the key materials and materials subsystems within the plastic solar collector can be followed(159,160).

Table 8 lists important weather and other environmental parameters that can affect the service life of polymeric materials. Perhaps the most important of these is ultraviolet (UV) radiation. Since radiation below a wavelength of 0.29 μm is filtered out by the earth's ozone layer, UV radiation between 0.29 and 0.40 μm is of the greatest concern.(161) This radiation is in the range (70 to 100 kcal) of the dissociation energy of the chemical bonds found in the polymers.(162) In addition to bond cleavage, UV radiation can radicalize double bonds, excite electrons, and activate other chemicals which will promote degradative chemical reactions in the polymer molecule.(163) The principal reaction results are chain scission, crosslinking, and/or depolymerization (unzipping). The specific reaction varies from polymer to polymer.(164) Chain scission reduces the polymer's molecular weight, makes it more susceptible to vapor diffusion, and weakens the macrostructure. Crosslinking entails an increase in localized density, which creates internal stresses and the development of microcracks that embrittle the material, reduce light transmittance, and increase the surface area open to chemical attack.

While UV radiation is always less than 10 percent of the total spectral radiation, its intensity varies markedly from season to season and from hour to hour. Cloud cover sharply reduces UV intensity; intensity at certain wavelengths varies more than at others.(165) Commercial polymers exhibit absorbance peaks in the UV range at which they are particularly sensitive to degradation reactions.(166,167) Some peaks are listed in Table 9. As a result of these observations, the total integrated intensity of sunlight, which is a commonly measured index of solar-radiation intensity, does not adequately represent actinic UV radiation.

TABLE 8. WEATHER AND OTHER ENVIRONMENTAL PARAMETERS AFFECTING POLYMER DETERIORATION

Parameter	Description
UV Radiation	0.29 to 0.40-μm wavelength range
Oxygen	O_2 or O_3
Water	Rain, snow, dew, frost, fog, direct heat transfer fluid contact, and humidity
Temperature	Absolute value, thermal gradient, and thermal fluctuations
Chemical Pollutants	SO_2, NO_2, CO, and NaCl
Particle Bombardment	Dust, sand, hail, bird droppings, and insects

TABLE 9. ACTIVATION-SPECTRA MAXIMA IN ULTRAVIOLET REGION FOR SEVERAL POLYMERS

Polymer	Activation-Spectrum Peak, Å
Polyesters (various formulations)	3250
Polystyrene	3185
Polyethylene	3000
Polypropylene	3700
Polyvinyl chloride	3200
Polyvinyl acetate	<2800
Polycarbonate	2850-3050 and 3300-3600
Cellulose acetate butyrate	2950-2980
Styrene acrylonitrile	2900-3250

Oxidation is one of the major reactions promoted by UV radiation. Oxygen concentration at the surface often is essentially constant, and the diffusion rate through polymers is low. And, except in thin films, the UV light does not penetrate very far into the material. Therefore, oxidation occurs at the surface of the polymer.(168,169) The specific effects of oxidation reactions vary, although they often involve discoloration and embrittlement.

Water has two effects on a polymer: it can react with the polymer to weaken it, or it can leach out stabilizing chemicals or clean a degraded surface, thereby exposing a new surface for attack.(170) The various forms in which water can contact a surface greatly complicate the analysis of its weathering effect. While oxygen concentration is constant, the amount of water vapor in the air changes continually. Water in the form of liquid moisture also varies in occurrence, thickness of coverage, and duration of coverage. The effects of rain are less well known than the effects of dew or humidity, since rain often contains atmospheric contaminants. Moreover, reactions between water and the polymer are often activated by UV radiation.(164)

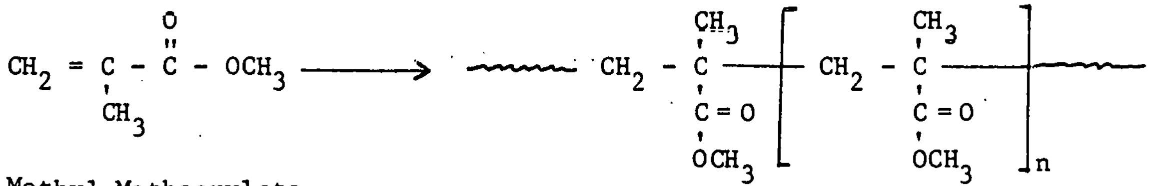
The maximum temperature that an exposed polymer can be expected to reach in a photovoltaic module has been estimated in one analysis to be 77 C(171,172), while a solar thermal collector may experience temperatures on the order of 90 C or even higher. Low temperatures are not sufficient to cause thermal breakdown of the more stable polymers.(164) High temperatures do increase degradation reaction rates, however, and this fact is particularly pertinent since the highest UV-radiation intensity occurs in the summer months when the temperature is also highest.(167) Thermal gradients and thermal fluctuations also affect material degradation. These factors result in stresses being set up in the material. Stressed polymers not only chemically react more rapidly(173), but also increase the tendency of the polymer to delaminate if it is laminated to a substrate with a different thermal expansion coefficient(174).

The effects of chemical pollutants are less well known. Studies only recently have been initiated to evaluate these effects.(175) Because of the variable nature of weather and polymers, it has been difficult to separate the effects of atmospheric pollutants from the major factors in polymer deterioration discussed previously.

(c) Selected Examples of Degradation Reactions and Mechanisms for Certain Polymeric Materials

In 1961, R.W.H. Lee compiled an extensive bibliography on "Properties and Degradation of Polyethylene Terephthalate and Methyl Methacrylate Polymers". This bibliography covered literature citations from 1947 through 1961 and is especially valuable for its documentation on weathering (degradation reactions and mechanisms) of these two polymeric materials.(176)

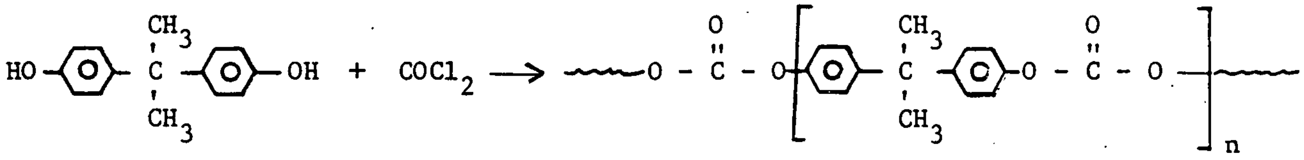
In plastic solar thermal collectors and flat-plate solar collector absorber coatings containing polymeric binders, the long-term temperature effects alone contribute greatly to the ultimate degradation of polymeric materials.(177) It should also be noted that many commercial plastics or coatings suitable for use in solar collector applications are homopolymers such as polymethylmethacrylate and polycarbonate.



$$CH_2 = \underset{CH_3}{C} - \overset{O}{\overset{\|}{C}} - OCH_3 \longrightarrow \sim\sim CH_2 - \overset{CH_3}{\underset{\underset{OCH_3}{C=O}}{C}} - \left[CH_2 - \overset{CH_3}{\underset{\underset{OCH_3}{C=O}}{C}} - \right]_n \sim\sim$$

Methyl Methacrylate
Monomer

Polymethylmethacrylate
Homopolymer

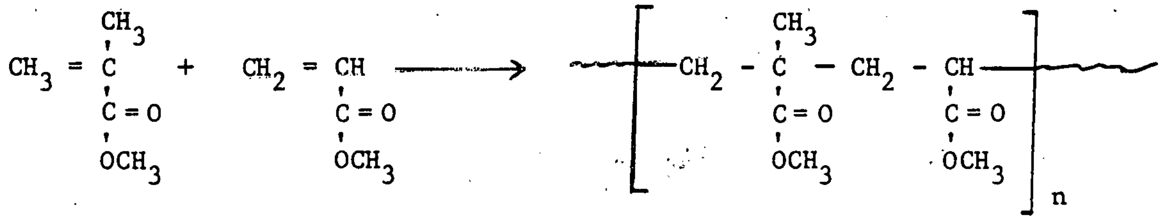


Bisphenol A

Phosgene

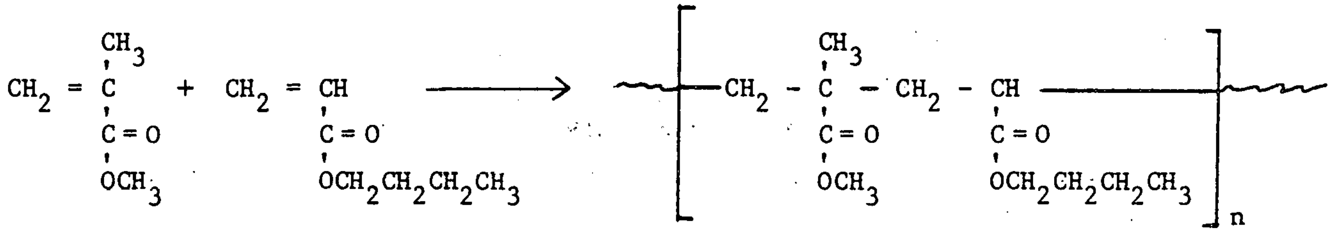
Polycarbonate Homopolymer

However, the majority of acrylic polymers on the market today are designated as copolymeric materials. These copolymers are made from several monomers which are polymerized together to form a single structure.



$$CH_3 = \underset{\underset{OCH_3}{C=O}}{\overset{CH_3}{C}} + CH_2 = \underset{\underset{OCH_3}{C=O}}{CH} \longrightarrow \sim\!\left[CH_2 - \underset{\underset{OCH_3}{C=O}}{\overset{CH_3}{C}} - CH_2 - \underset{\underset{OCH_3}{C=O}}{CH} \right]_n\!\sim$$

Methyl Methacrylate Methyl Acrylate Copolymer



$$CH_2 = \underset{\underset{OCH_3}{C=O}}{\overset{CH_3}{C}} + CH_2 = \underset{\underset{OCH_2CH_2CH_2CH_3}{C=O}}{CH} \longrightarrow \sim\!\left[CH_2 - \underset{\underset{OCH_3}{C=O}}{\overset{CH_3}{C}} - CH_2 - \underset{\underset{OCH_2CH_2CH_2CH_3}{C=O}}{CH} \right]_n\!\sim$$

Methyl Methacrylate Butyl Acrylate Copolymer

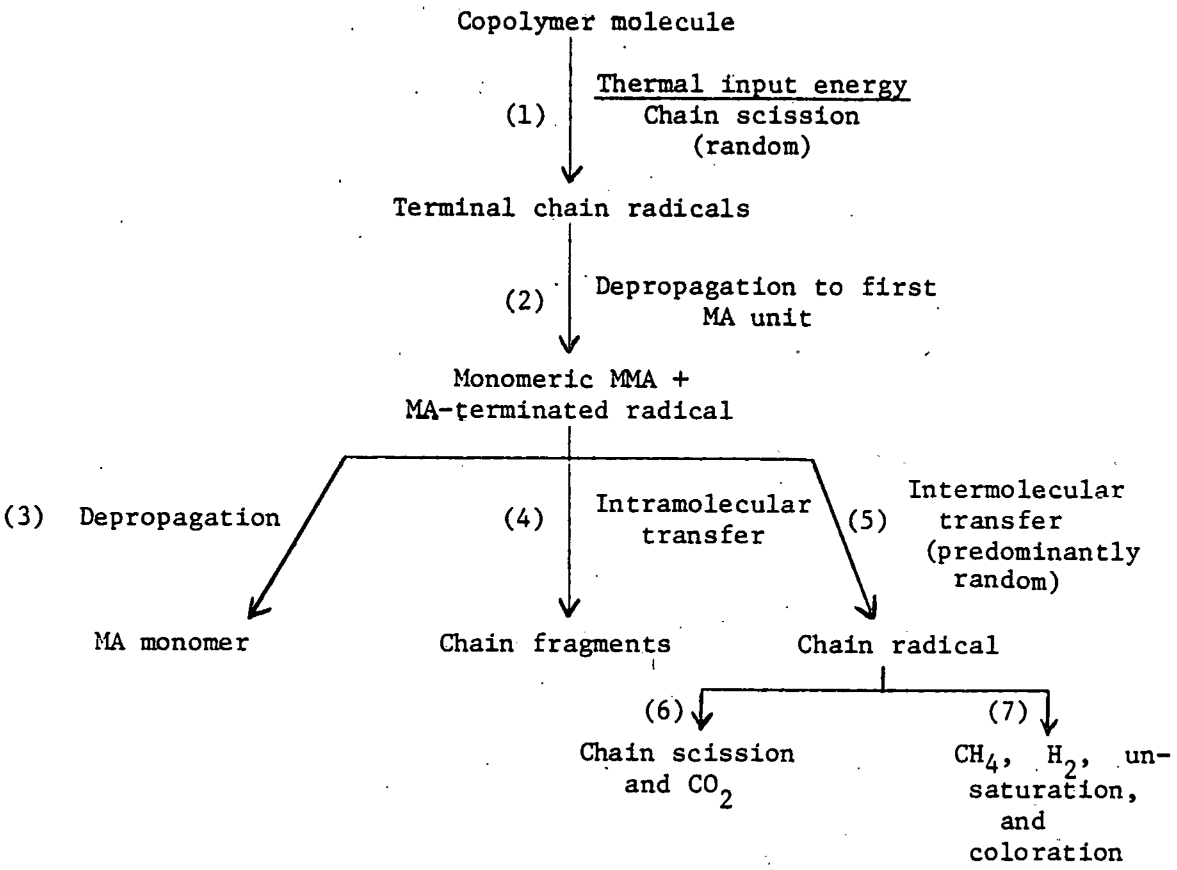
The presence of a small amount of a second or third monomer copolymerized into a single structure can have a profound effect upon the stability or other physical properties for this hybrid material which would be significantly different from a homopolymer containing only one monomer component$^{(178)}$.

Thermal degradation studies for methyl methacrylate (MMA)/methyl acrylate (MA) and methyl methacrylate (MMA)/butyl acrylate (BA) copolymers covering a wide compositional range

[112 (MMA)/1 (MA or BA) - 2 (MMA)/1 (MA or BA)]

were examined utilizing thermogravimetric analysis (TGA) and thermal volatilization analysis (TVA). The results of these studies showed that the copolymers became more stable to thermal breakdown as the acrylate content was increased. The gaseous degradation products, liquid products, chain fragments and residue were each examined separately using infrared spectroscopy, gas-liquid chromatography, and mass spectrometry. The main gaseous products were found to be carbon dioxide and smaller amounts of hydrogen from the methyl acrylate copolymer and carbon dioxide and but-1-ene from the butyl acrylate copolymer. The most important liquid products found were methyl methacrylate monomer from both systems and n-butanol from the butyl acrylate copolymers with high butyl acrylate contents.

Molecular weight measurements on the residue indicated that breakdown by random scission processes on the polymeric backbone becomes more important relative to breakdown by depolymerization processes as the acrylic content of the copolymer is increased. As degradation proceeds the color of the residual polymer goes through yellow to brown and for similar extents of decomposition the copolymers of greater acrylate content exhibit more pronounced coloration(179). A general outline of the thermal degradation reactions for MMA/MA copolymers is given below.

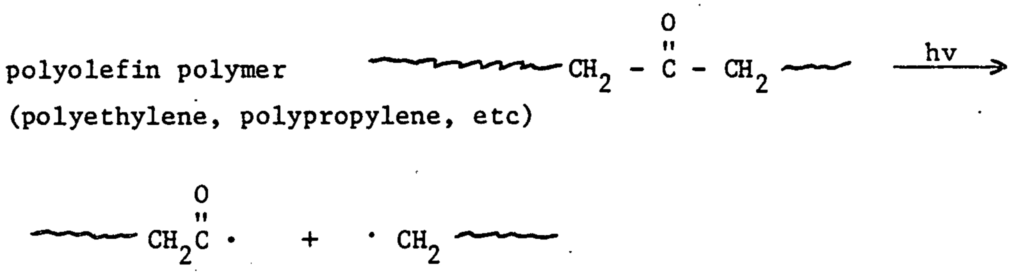


As was mentioned earlier, most outdoor weathering degradation reactions for polymers are initiated through some sort of photochemical mechanism. The first step in any photochemical sequence of events is the absorption of light energy by the polymer followed by some sort of photophysical process to an excited state intermediate. This polymeric excited state intermediate then undergoes a chemical reaction or rearrangement (chain scission, etc) which ultimately leads to a loss in physical properties of the material.

$$\text{polymer (p)} \xrightarrow{hv} p^* \text{ (excited state intermediate)}$$

$$p^* \longrightarrow \text{products}$$

Many polymers, theoretically, should not have the capability of absorbing UV energy such as polyolefinic materials since pure saturated hydrocarbons do not contain major absorption bands above 280 nm which is about the cutoff point for atmospheric solar radiation. Unfortunately, many commercial polyolefins contain trace amounts of metallic impurities or carbonyl groups which are formed during the polymerization process and hence exhibit some capability of directly absorbing UV-radiation from the environment. In these situations the polymer backbone can be easily photodegraded into an unusable product(180).



$$\text{polyolefin polymer (polyethylene, polypropylene, etc)} \quad \sim\!\sim CH_2 - \overset{O}{\overset{\|}{C}} - CH_2 \sim\!\sim \xrightarrow{hv} \sim\!\sim CH_2\overset{O}{\overset{\|}{C}}\cdot \; + \; \cdot CH_2 \sim\!\sim$$

(chain scission)

Combined stress (photochemical at low UV-radiation wavelength of 254 nm and thermal or photothermal reactions) studies on copolymers of MMA/BA showed that both degradation mechanisms are similar (photothermal or thermal alone) in that chain terminal radicals are formed from these stresses under similar conditions. The difference between photothermal and thermal-only degradation reactions is in the type and distribution of observed products. For this copolymer (MMA/BA), butyraldehyde, which is completely absent from the products of thermal degradation, is produced in the photothermal reaction while but-1-ene and carbon dioxide, which are significant products of thermal degradation, are not produced photothermally(181).

Thermal and photoinduced oxidation reactions are also common for environmentally stressed polymeric materials. In these reactions the process is initiated on the polymer chain by photochemical or photothermal mechanisms followed by direct combinations of the polymeric chain radical intermediates with atmospheric oxygen to form reactive hydroperoxides. Further thermal or photochemical stress degrades the polymeric hydroperoxide and causes further degradation of the polymer chain(182).

$$\underset{\text{(polymer chain)}}{\mathrm{HP}\text{———}\mathrm{PH}} \xrightarrow{\text{hv or thermal}} \mathrm{HP}\cdot \quad \text{(polymer radical)}$$

$$\mathrm{HP}\cdot + \mathrm{O_2} \longrightarrow \mathrm{HPOO}\cdot$$

$$\mathrm{HP}\text{——}\mathrm{PH} + \mathrm{HPOO}\cdot \longrightarrow \underset{\text{(hydroperoxide)}}{\mathrm{HPOOH}} + \mathrm{HP}\text{——}\mathrm{P}\cdot$$

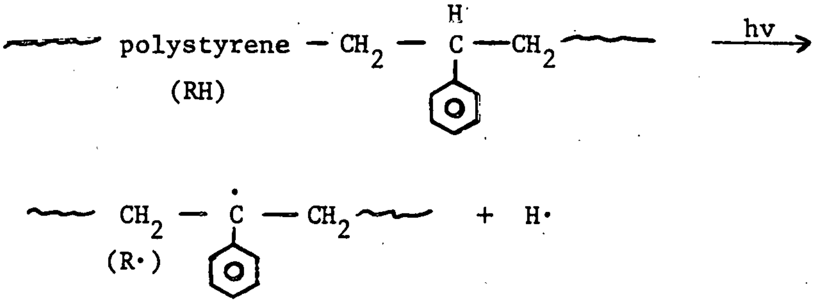
$$\mathrm{HPOOH} \xrightarrow[\text{thermal}]{\text{hv or}} \mathrm{HPO}\cdot + \cdot\mathrm{OH}$$

$$\mathrm{HPO}\cdot \longrightarrow \text{polymeric carbonyl formation.}$$

These types of stress environment (combinations of photochemical, photothermal, and photooxidation) are probably the most important considerations in accelerated testing of plastic materials.

Polystyrene materials, although not generally considered for use in plastic solar thermal collector systems, have been well characterized with regard to poor weathering performance and can be used as a control to correlate effects of outdoor degradation reactions for other polymeric materials(183,184).

Thermal degradation reactions for polystyrene results in mainly depolymerization which produces monomeric styrene starting materials; however, photochemical reactions (2537 Å UV-radiation wavelengths) for polystyrene leads to the following sequence of events:



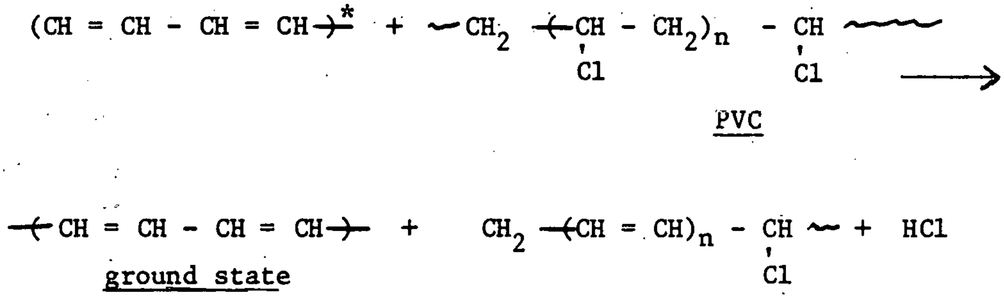
$$
\begin{aligned}
&R\cdot + R\cdot \longrightarrow \text{crosslinked polymer}\\
&H\cdot + RH \longrightarrow H_2 + R\cdot\\
&H\cdot + R\cdot \longrightarrow \text{unsaturation} + H_2\cdot\\
&\qquad\qquad\qquad\qquad\quad \searrow\\
&R\cdot + O_2 \longrightarrow ROO\cdot \longrightarrow \text{colored products.}
\end{aligned}
$$

Crosslinking reactions associated with photochemical degradation of polystyrene also have a pronounced effect on its diffusion and permability coefficients for CO_2 and can be used as a method for determining extent of degradation(185).

Polyvinyl chloride (PVC) plastics are important building materials and can be used in a wide variety of applications. The principal method of degradation for PVC is through photochemical-induced dehydrochlorination to produce conjugated unsaturated functionality along the polymer backbone. This conjugated unsaturated functionality is a strong UV absorber and can cause further photoinduced degradation reactions which lead to the ultimate failure of the polymer(186).

$$\text{PVC} \xrightarrow{hv} \sim\!\sim\!(\,\text{CH}=\text{CH}-\text{CH}=\text{CH}\,)\!-$$

$$-(\,\text{CH}=\text{CH}-\text{CH}=\text{CH}\,)- \xrightarrow{hv} \underset{\underline{\text{excited state}}}{-(\,\text{CH}=\text{CH}-\text{CH}=\text{CH}\,)^{*}}$$



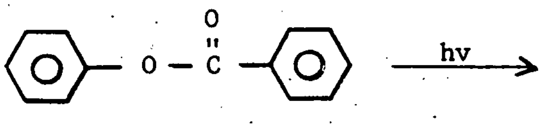
$$(\text{CH}=\text{CH}-\text{CH}=\text{CH})^{*} + \underset{\underline{\text{PVC}}}{\sim\text{CH}_2\,(\underset{\text{Cl}}{\text{CH}}-\text{CH}_2)_n-\underset{\text{Cl}}{\text{CH}}\sim\sim} \longrightarrow$$

$$\underset{\underline{\text{ground state}}}{-(\,\text{CH}=\text{CH}-\text{CH}=\text{CH}\,)-} + \text{CH}_2\,(\text{CH}=\text{CH})_n-\underset{\text{Cl}}{\text{CH}}\sim + \text{HCl}$$

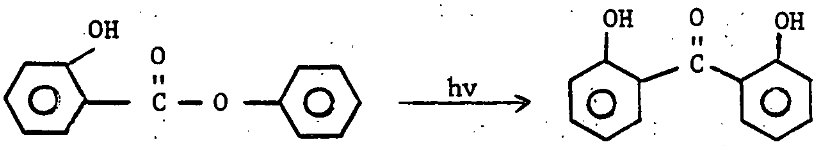
where n can vary from 2 up to 9.

Polycarbonate materials, especially UV-stabilized compositions, have the potential of being used as a major component in plastic solar thermal collector systems. These polymers are much more thermally stable than thermoplastic acrylic polymers, but they do undergo rapid photodecomposition reactions unless properly stabilized for outdoor environments.

There are two basic photochemical initiated degradation mechanisms associated with polycarbonate materials. The first such mechanism is a Fries-type rearrangement to produce yellow products attributed to dihydroxylbenzophenone molecules:



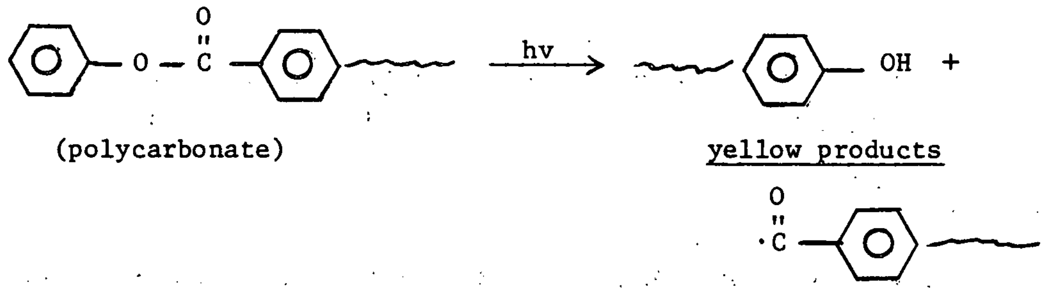
(diphenyl carbonate model structure)



(phenyl salicylate)

yellow products
(dihydroxybenzophenones)

The second mechanism, and probably the most important, is a direct chain cleavage reaction and subsequent formation of yellow phenolic products(187).



The previous discussions treat some of the general mechanisms or reactions associated with weathering of certain polymers while the following section will review more detailed weathering studies for specific polymer systems.

VI. Weathering of Specific Polymeric Materials

(a) Polyolefins

An early investigation by Titus on the "Weatherability of Polyolefins(188) covered the effects of various natural and artificial weathering conditions for a series of clear and pigmented nonreinforced plastic materials (polyethylene, polyethylene copolymers, polypropylene, polyvinyl chloride-polypropylene and an ethylene-butene copolymer). This report included exposure testing methods, weathering exposure testing at different geographical locations, as well as various means of stabilization and the effects of different pigments in the polyolefinic materials.

Correlations were also made for environmentally and unnaturally influenced stress cracking of these polymeric materials, losses in elongation and the effects of weathering conditions on stressed and unstressed polyolefin samples(188).

Some of the important results from the study showed that, in general, unmodified polyolefins have poor inherent weatherability but can be upgraded through the use of appropriate additives such as antioxidants, UV stabilizers, certain pigments and especially carbon blacks.

Oxidation, a primary cause of failure in polyolefins at high temperatures(189), was shown to be negligible under low-temperature conditions except in combination with UV light radiation. The degradation of polyethylene by UV radiation was attributed to the presence of carbonyl groups in the polymer backbone caused by the initial polymerization reaction. These carbonyl groups absorb UV energy and cause polymeric chain scission or degradation, especially in the presence of oxygen, and can be controlled through the use of UV absorbers, antioxidants, and certain pigment structures(190).

Polypropylene materials have high degrees of crystallinity which reduces the access of oxygen to the active reaction sites (substituted carbon atoms) in the main chain. In polypropylene material the antioxidants and UV absorbers concentrate in the amorphous regions of the polymer which tends to reduce its photooxidation rate and increase its outdoor exposure lifetime.

Pigment additives used to enhance polyolefin outdoor weathering stability were carbon blacks (2 to 3% concentration levels) and certain other pigments used as mercadmium red, cadmium yellow, and iron oxide (0.5 to 2% concentration levels). The stabilization of these polyolefins was also improved through the use of ultraviolet absorber compounds such as substituted benzophenone or salicylic acid derivatives.

In general, the best weathering results were obtained from a high-density, high molecular weight, carbon black-filled polyolefin material. It was also reported in this study that certain polyethylene copolymers have enhanced ultraviolet stability over the homopolymer systems.

In a similar study by Dunn and Hill(191), a series of five types of polyolefins (polypropylene, high- and low-density polyethylenes, polyethylene blends, and polyethylene-ethyl acrylate copolymer) were exposed for 4 years in Australia at four different locations having widely differing climatic conditions. The samples tested (both pigmented and clear formulations) were injection molded plaques, and mechanical or electrical properties were determined before and after exposure testing.(191)

The conclusions drawn from these studies indicated that only the polyolefins pigmented with carbon black showed excellent resistance to degradation after outdoor exposure durations of 4 years. All of the non-black pigmented and clear polyolefins were rapidly degraded under these exposure conditions (Australian climates) and their service life was considerably less than 4 years. In addition, the clear polyolefin samples all showed excessive chalking, pitting, and crazing after exposure and the crazing patterns on the sample surface were similar to stress patterns observed in unexposed specimens. If these stress patterns were eliminated during the molding process, then the resistance to weathering of polyolefins would be greatly improved.

The major cause of degradation in polyolefins, as was mentioned previously, is due in part to ultraviolet light radiation which initiates chain-scission reactions and results in cracking, embrittlement, loss in tensile strength, and reduction in elongation at break.

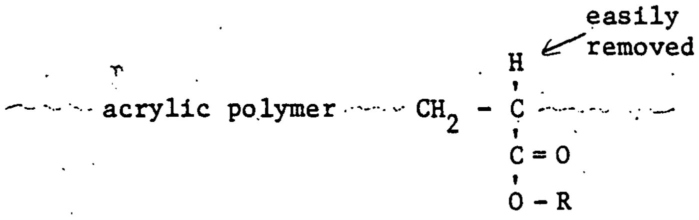
Electrical properties such as the measurement of the power factor of exposed and unexposed samples and mechanical testing were used as a method of determining the service life or weathering changes in these polyolefinic materials(191).

A short review by Baum on the "Weathering Degradation of Polyolefins" confirms the results of previous studies in that photodegradation reactions of these materials are an important factor for determining service life. This review also considered variations in spectral radiation, temperature, density of the polymer and polymer absorption spectra, orientation of the sample and sample thickness as related to property deterioration. Stabilization of polyolefins through the use of ultraviolet absorbers and special pigments were also briefly mentioned in this article(192).

(1) Applications of Polyolefins in Plastic Solar Thermal Collector Systems. One unique application of polyolefin materials in thermal solar collector systems is the use of ethylene propylene diene monomer (EPDM) rubber (carbon black loaded for maximum UV degradation protection) extruded tubular mats. These solar radiation absorbing tubular mats allow water to flow through the entire system providing thermal radiation transfer for an efficient heat collection system. Since the EPDM tubular mats are filled with carbon black, their expected service lifetime is projected for at least 30 years. Similar EPDM formulations for electrical applications have been in service for at least 20 years (193).

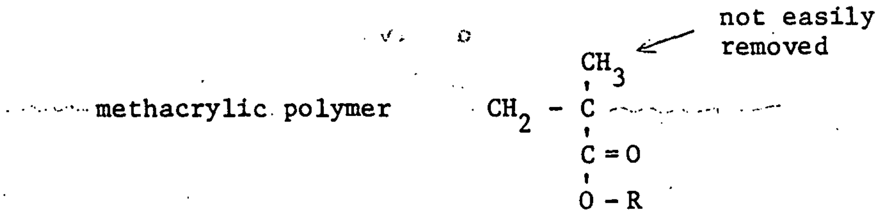
(b) Acrylic Polymers

Acrylic polymers and copolymers cover a broad class of materials synthesized from alkyl esters of acrylic acid or methacrylic acid components. One of the advantages of using these polymers is their high purity and usual absence of deleterious ultraviolet light absorbing chromophores; hence, they have an inherent capability for improved weathering resistance over other plastic materials. Acrylic and methacrylic polymers also have a unique chemical composition that tends to resist environmental attack along their backbone structures. Acrylic acid-ester polymers are more susceptible to degradation reactions than similar methacrylic acid ester polymeric structures in that hydrogen atoms are more easily removed in the former case as opposed to methyl group removal in methacrylic acid ester components.



$$\cdots\text{acrylic polymer}\cdots CH_2-\begin{array}{c}H\\|\\C\\|\\C=O\\|\\O-R\end{array}\cdots$$

acrylic acid ester polymers
where R is an alkyl group



$$\cdots\text{methacrylic polymer}\quad CH_2-\begin{array}{c}CH_3\\|\\C\\|\\C=O\\|\\O-R\end{array}\cdots$$

methacrylic acid ester polymers
where R is an alkyl group

One of the most promising materials having proven test requirements for 17-1/2 years serviceability under desert weathering conditions is a crosslinked polymethylmethacrylate premium grade sheet material containing UV absorbers and manufactured by Rohm and Haas (Plexiglas 55). This plastic was obtained from Rohm and Haas in 1956 and was mounted in an outdoor test exposure facility at Sandia Laboratories, Albuquerque, New Mexico, for 17 years and 8 months. Afer this exposure time period the sample was evaluated for extent of degradation by optical, chemical, and mechanical methods of analysis.

The optical transmittance of a material before and after weathering is an important factor in the design of plastic solar thermal collector systems. In the case of the 17-1/2-year weathered crosslinked polymethylmethacrylate sample, its integrated or total transmission was only about 10 percent less than an unexposed similar control material. These losses could also be improved upon had the sample been coated with a more abrasion-resistant material such as a crosslinked polymethylsiloxane polymer.

Chemical analysis of the aged versus a similar control polymer sample showed that there was very little change in the carbon/hydrogen ratio for both systems.

Mechanical property evaluation with thermal mechanical, torsion pendulum, and flexural analysis techniques indicated that the aged polymer had a significant reduction in the glass transition temperature, increased in brittleness, and showed a 51 percent decrease in flexural strength over a similar unaged control sample. These changes in physical properties are indicative of some form of polymer degradation related to a reduction in chain length and increases in crosslink density(194).

The superior weathering characteristics of acrylic-methacrylic polymers and copolymers qualify these materials for consideration as major components in plastic solar thermal collector systems.

(1) Applications of Acrylic Polymers in Plastic Solar Thermal Collector Systems. A black-liquid solar thermal collector system was developed at BCL using extruded acrylic materials, Acrylite SDP double-skinned sheet, available from CY/RO Industries. The black-liquid solution was circulated upward through 72 small passages and the basic flow pattern, manifolds, and hole sizes were designed to produce very uniform flow velocities in each of the 72 channels. This work is continuing and is the subject of a report on "Development of a Low-Cost, Black-Liquid Solar Collector System(195).

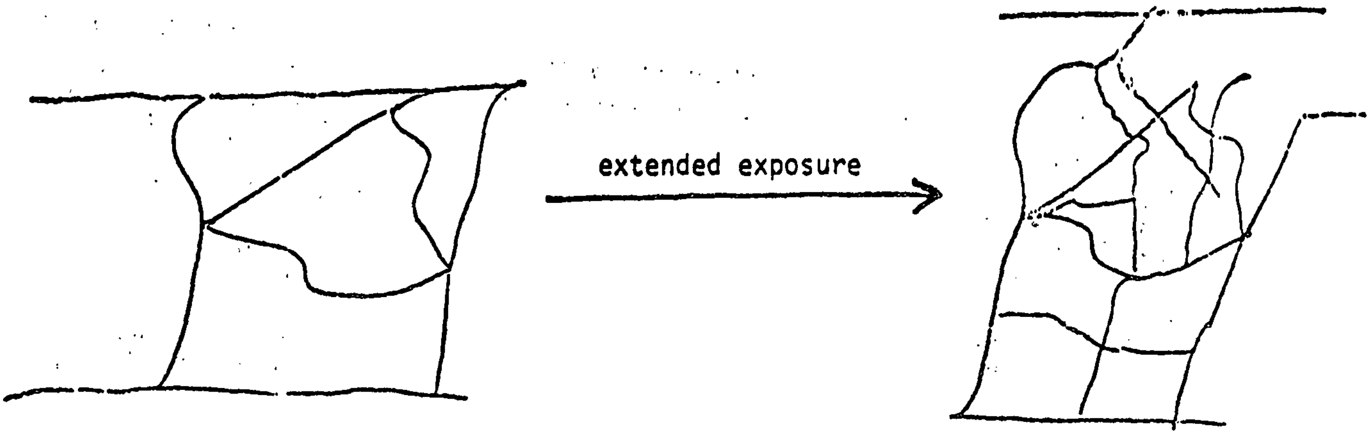
Acrylic flat sheet materials can also be used as glass glazing substitutes for more conventional metal flat plate solar thermal collector systems(196).

(c) Polycarbonates

It has been found that certain polycarbonate sheet materials (UV-stabilized and nonstabilized having a 12-mil thickness dimension) subjected to outdoor weathering in a temperate northern climate for relatively short periods (20 to 82 months) develop a network of surface microcracks on the side exposed to solar radiation. The back side or non-exposed side does not exhibit these microcrack structures. Artificial weathering produces microcrack formation under the influence of light radiation in conjunction with cycling of either temperature and moisture or temperature alone. The use of radiation by itself (4200 hours), or even relatively severe cycling of temperature and humidity without radiation (2100 cycles), does not induce microcracking(197).

(1) Surface Microcracking During Outdoor Weathering. Polycarbonate sheets (UV-stabilized and unstabilized) weathered outdoors at Ottawa, Canada, developed surface microcracks on the side exposed to solar radiation. Initial crack formation occurred after 20 to 23 months of exposure. The very narrow (0.1 to 0.2 μm in width), randomly oriented initial cracks propagate slowly with aging and interact to form a network. The network of relatively shallow cracks divides the surface of the sheet into predominantly triangular and four-sided polygons. Areas confined between the intersecting cracks decrease with weathering because the surface resin layers continue to fracture, forming new cracks.

The surface microcracks of the unstabilized sheet are deeper and wider than those of the sheet made from stabilized materials; their number per unit area of surface (density of cracks) is also greater, as evidenced by the smaller area of the polygons formed by the cracks.



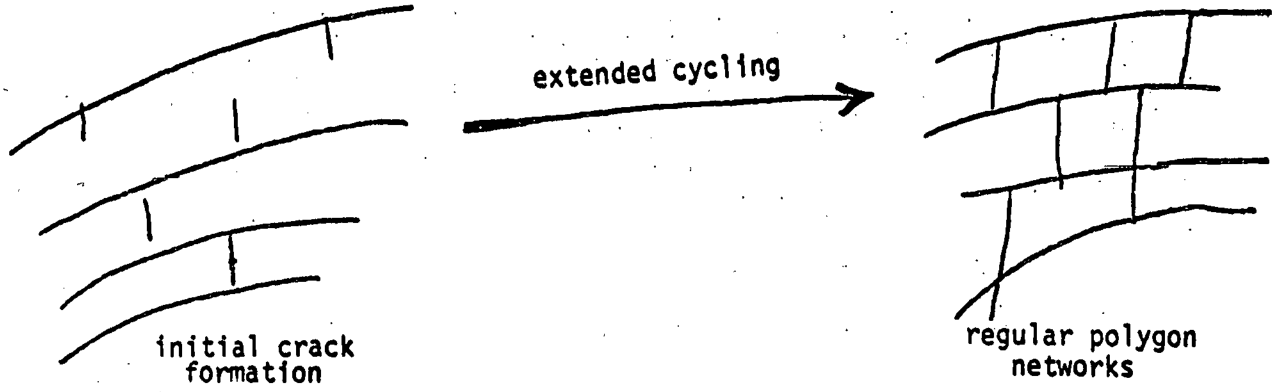
low crack line intersection

high crack line intersection and irregular polygon formation

The back side of the polycarbonate sheet which was exposed to essentially the same environmental conditions except for the action of radiation did not show any evidence of surface microcracking. This indicates that a combination of stresses (radiation + environment) is necessary for crack formation.

(2) Accelerated Weathering (Radiation Cycling and Humidity-Temperature Cycling). Polycarbonate sheets exposed to alternate cycles of radiation, water spray, and temperature in Xenon Arc Weather-Ometer resulted in the formation of surface microcracks in the exposed (front) side of the samples. Initial cracks (cracks formed after 370 to 375 cycles) were mostly parallel with a few intersecting cracks to produce a more regular polygon network.

The polygons formed by Weather-Ometer aging were regular in shape (rectangular) due to the reproducibility of cycles and relatively uniform change in the environmental conditions at the sample during any given cycle. This is in direct contrast to the irregular polygons formed during outdoor weathering, where the environmental conditions are quite variable during a cycle and from one cycle to another.



The back side (unexposed) of the polycarbonate sheets did not exhibit any signs of surface cracking.

(3) Accelerated Weathering With Continuous Radiation and Constant Temperature and Humidity. Surface microcracking in polycarbonate sheet was not observed after 4200 hours constant irradiation at 55 C sample temperature and 50 percent relative humidity.

(4) Thermal Cycling of Preirradiated Polycarbonate Sheet. Preirradiated polycarbonate sheet (2100 hours) was subjected to temperature cycling between -18 and 25 C (0 to 77 F) and developed very fine surface microcracks after 1200 temperature cycles. Network formation occurred after further cycling (1800 cycles) but only on the preirradiated side. Thermal cycling did not induce microcracking in the nonirradiated (control) sheet. Extended exposure times (4200 hours) reduced the cycle times (845) to produce single microcrack formation as well as network structures (1450 cycles).

(5) Exposure to Variable Temperatures and Humidity Without Radiation. Polycarbonate sheet subjected to cycles of humidity and temperature did not develop surface microcracks even after 2100 cycles.

Cycling of temperature and humidity induced localized, alternating microstresses between the surface resin layer and the bulk of the material and at flaws and in homogeneous points in the surface, causing a type of stress fatigue.

These observations demonstrate that surface microcracks in a sheet of polycarbonate are not caused by environmentally induced stresses alone even if they are relatively severe; the degradative action of combination of radiation stress is also required.

(6) Mechanism of Surface Microcracking. In outdoor weathering and in most of the aging treatments described, the polymeric material in the surface region of the sheet undergoes dimensional changes as a result of cyclic variations in temperature and humidity. Cyclic changes of temperature result in alternating volume expansions and contractions of the material; cyclic variations of humidity cause absorption and desorption of moisture, and this in turn results in alternating swelling and shrinking. Dimensional changes induced by both temperature and moisture may be in the same or in opposite directions, depending upon the exposure conditions to which the material has been subjected.

For example, during very hot, humid summer days, plastic sheets exposed outdoors undergo thermal expansion at the same time as swelling caused by water absorption. During hot, dry periods they undergo thermal expansion, but moisture-induced volume change may be in the opposite direction because of water desorption. Similar volume change relations can occur during artificial exposure.

Part of the mechanism of crack formation in polycarbonates is due to the action of the ultraviolet portion of solar radiation in which the material of the exposed surface layers undergoes a gradual decrease in molecular weight(198) by a process of photooxidative chain scission(199). This decrease which does not occur in the bulk material nor in the surface layers of the side not exposed to solar radiation, is in proportion to the amount of irradiation by ultraviolet light.

A decrease in molecular weight causes a lowering of the total attractive forces between adjacent polymer chains, and this, in turn, lowers the resistance of the resin to fracture. When the fatigue limit at a given site becomes lower than the environmentally induced differential stresses, fracture of the surface resin occurs, producing microcracks. Because of gradients in the stresses involved, the cracks grow from the surface inwards and are V-shaped, with the median plane approximately perpendicular to the sheet; the cracks are limited to the exposed surface region.

Studies on relatively thin polycarbonate films (1 mil thick but the researchers did not mention if the films contained UV stabilizers or not) showed that, after 1,000 hours UV exposure alone under ambient conditions, there was considerable change in the glass transition temperature (T_g) and molecular weight distribution ($\overline{M}_w$).(198)

TABLE 10. GLASS TRANSITION TEMPERATURE AND MOLECULAR WEIGHT DISTRIBUTION FOR THIN POLYCARBONATE FILMS(198)

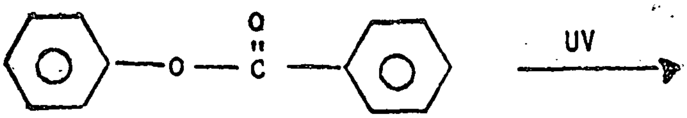
	T_g, C	$M_w \times 10^{-4}$
Unaged film	152 ± 2	2.9
1,000-hr exposure (aged with a RS sunlamp)	132 ± 1	1.0
1,000-hr UV exposure with repeated thermal analysis	123 ± 2	1.0

The drop in T_g for the UV-aged sample corresponds to a decrease in weight average molecular weight (M_w) by a factor of 2.9. Repeated thermal analysis decreases 10 C from the original value, wheras no such change was observed for the T_g of the unaged samples. These results are consistent with mixing in of low molecular weight degradation products on the surface into the bulk sample material during thermal cycling, effecting a lowering of T_g (plasticized during subsequent runs).

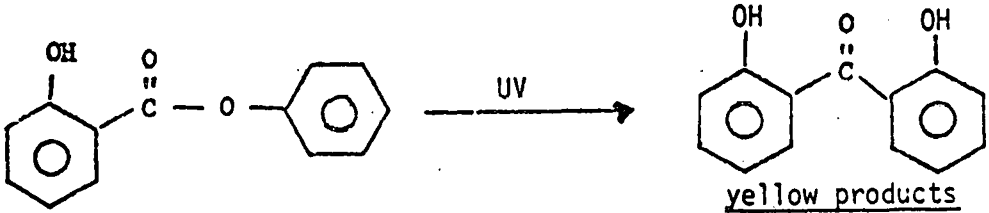
The UV-aged films showed a distinct yellow color but remained relatively transparent. In addition, the aged film is very brittle, and shows a 6 percent weight loss upon extraction with methanol.

Surface attenuated infrared reflectance measurements and UV spectral analysis detected phenolic intermediates (bisphenol-A type products) as well as aromatic esters and ketones in the degraded sample.

Photodecomposition of model compounds shown below



(diphenyl carbonate)



(phenyl salicylate)

yellow products

(dihydroxybenzophenones)

results in a Fries-type rearrangement to produce yellow products attributed to dihydroxybenzophenone molecules(200). These types of rearrangements would be similar to those expected for polycarbonate plastic materials.

In other artificial and natural environmental conditions, samples of 1/8-inch-thick polycarbonate sheet (UV-stabilized and unstabilized) were exposed to severe rain erosion (simulated rain impact testing at 400 and 600 mph) and outdoor weathering (30 weeks) in central Florida.(201)

Rain impact causes the surface of the polycarbonate to flow and lowered the optical transmission without significant removal of material.

Limited weathering studies indicated that UV-stabilized polycarbonate retained its original tensile strength (elongation was not measured) only slightly better than the unstabilized materials.(201)

A recent study on the synthesis of new, more weather-resistant polycarbonate materials and novel stabilization concepts has been initiated by Hughes Aircraft Company, but these materials are very expensive and probably would only find use in highly specialized applications(202).

(7) Applications of Polycarbonates in Plastic Solar Thermal Collector Systems. Commercial UV-stabilized polycarbonate plastics (General Electric's Lexan; Mobay's Merlon; Rohm and Haas) are easily extruded into about any configuration which readily suggests their use in low-cost solar thermal collector systems. These materials also offer unique physical properties such as high impact strength and higher heat resistance over other thermoplastic polymers and can be readily substituted for glass glazing in conventional flat plate metal solar collector systems(203,204).

Ramada Energy Systems, Inc., a Ramada Inns, Inc., company, located in Phoenix, Arizona, has developed an extruded polycarbonate solar collector having a unique triple-layer construction. The top two layers are normally used as insulating layers while the bottom channel is the fluid-carrying channel. These panels are approximately 1 foot wide and 20 feet long, although they can be produced in any length while the width is determined by the size of the extruder(205).

FAFCO, Inc., located in Menlo Park, California, is also producing an experimental coaxial extrusion plastic solar collector system utilizing polycarbonate materials(206).

Franklin Research Center has obtained a patent on a coextruded solar collector for air systems using polycarbonate.(207)

(d) Fluorocarbon Polymers

Fluorocarbon polymers are one of the best materials for resisting environmental effects of outdoor exposure. These polymers have high chemical and thermal stability, low water absorption, excellent resistance to hydrolysis reactions, and overall excellent weatherability characteristics. There are four major commercial fluorine plastics: polytetrafluoroethylene (Teflon); fluorinated ethylene-propylene (FEP) copolymer; polyvinyl fluoride (PVF); and polyvinylidene fluoride (PVF), but only thin films of FEP and PVF have high optical clarity and excellent solar transmittance values suitable for use in plastic solar collector systems(208). For example, FEP and PVF films are extremely weather resistant and after a 5-year exposure study in Florida, they retained about 95 percent of their original solar transmission(209,210).

Other weathering studies on fluorinated polymers also demonstrated their unique ability to maintain their physical properties under a wide variety of exposure test conditions(211-214).

(1) Application of Fluorinated Polymers in Plastic Solar Thermal Collector Systems. Fluorinated polymers have been used in transparent film or honeycomb structures between the absorber plate and the transparent cover in conventional flat-plate solar collectors. These composite structures increase the efficiency of the collector by suppressing heat losses and can act as an insulating barrier for the total systems(215,216).

In related solar energy technologies, fluorinated polymers have been used in plastic solar stills, lenses for concentrating collectors, and encapsulant materials for solar photovoltaic cell modules(217,218).

(e) Other Polymeric Materials

Detailed weathering studies on polystyrenes and related copolymers and terpolymers(183,220); fiber reinforced plastics and polyester gel coats(221,222); polyvinyl chloride(223-225); urethane foams(226); silicone polymers and sealants(227-233); textile polymers(234); acrylonitrile-butadiene styrene terpolymers(235,236); polyamide films(237); and elastomeric vulcanizates(238,239) have been reported but will not be discussed in this report since these polymers probably do not have major or direct applicability in plastic solar thermal collector systems. The exposure testing methodologies in these reports, however, are similar to those previously mentioned throughout this text and do not warrant further elaboration.

VII. Exposure Testing Methodology

(a) Prediction of Service Life for Materials

Prediction of service life for materials under multiple stress conditions is difficult in that many published weathering studies have not been done under carefully controlled conditions. The types and sizes of samples varied greatly between different experiments so that extrapolation from one set of test conditions to another has very little quantitative accuracy.

The term "weatherability", although commonly used in plastics technology, is a poorly defined concept since it refers to long-term service under complex and variable conditions and because different properties of a plastic are affected to different degrees by a given environment. Improvement in the ability to predict weatherability therefore requires careful redefinition of the problem as well as a refinement of experimental technique. A review of the extensive literature on outdoor and artificial exposure of plastics shows that no simple correlation exists between these two modes of testing. Further, because the rates and mechanisms of deterioration are different when produced by visible light, ultraviolet, heat, or moisture, an arbitrary "accelerated" weathering test will distort the balance of responses observed in the slower outdoor exposure of plastics.

In an ideal procedure, the effects of specific weathering parameters are established for specific properties of a given material using controlled artificial environments; the makeup of the weather at a given outdoor location is analyzed in terms of these parameters; and finally, by suitable mathematical models, the results to be expected on exposure of the material to this given composition of weather can be computed. While this approach is admittedly complex, its feasibility has been demonstrated. For routine studies where the mathematical approach may be impractical, it appears necessary to abandon the goal of a "universal" artificial weathering test unless only qualitative or screening comparisons are desired. Quantitative predictions, using present testing technology, will be most reliable if restricted to a comparison of materials which are fairly similar in composition.

The first consideration in accelerated testing is the choice of light source used to irradiate the sample. This light source should approximate the solar spectrum at a given latitude or location but with long-term stable output energies so that uniform steady state illumination can be continued over extended time periods.

The Weather-Ometer (artificial xenon arc light source) most often used in accelerated testing is capable of equating 1 hour of supposed noonday sun at Chicago comparable with actual solar illumination which gives about 12 hours of daylight with maximum intensity centered at 12:00 noon plus or minus 2 hours (Figure 2).(122)

Correlation between actual outdoor exposure testing and artificial Weather-Ometer exposure (UV wavelengths--280 to 383 nm) at constant relative humidity and ignoring other climatic conditions (rain, temperature cycling, etc) for 22 hours was found to be equivalent to 1 week of actual outdoor exposure in Stamford, Connecticut, as measured by loss in original tensile strengths of the polystyrene sheet materials (Figures 3 and 4).(240)

In order to equate UV intensity alone as a single outdoor exposure variable and to attempt to standardize a Weather-Ometer for accelerated testing, BCL analyzed available data on average hourly, daily, and monthly total daylight radiation intensities (UV range--280 to 383 nm) for the climatic region of Philadelphia, Pennsylvania. This analysis was based on studies prepared by the National Cancer Institute on the effectiveness of UV radiation in producing skin erythema at different locations throughout the U.S. through use of a "Sunburning UV-Meter". This device records a measure of the cumulative amount of ultraviolet radiation as produced in the solar spectrum which passes through its filters and photosensors. Half-hourly recordings may range from zero to slightly over 1,000 counts, depending on the geographical location and the meterological conditions prevailing at the UV-meter site. A count of approximately 400 is estimated to produce skin erythema ("sunburn") on the typical Caucasian skin(241).

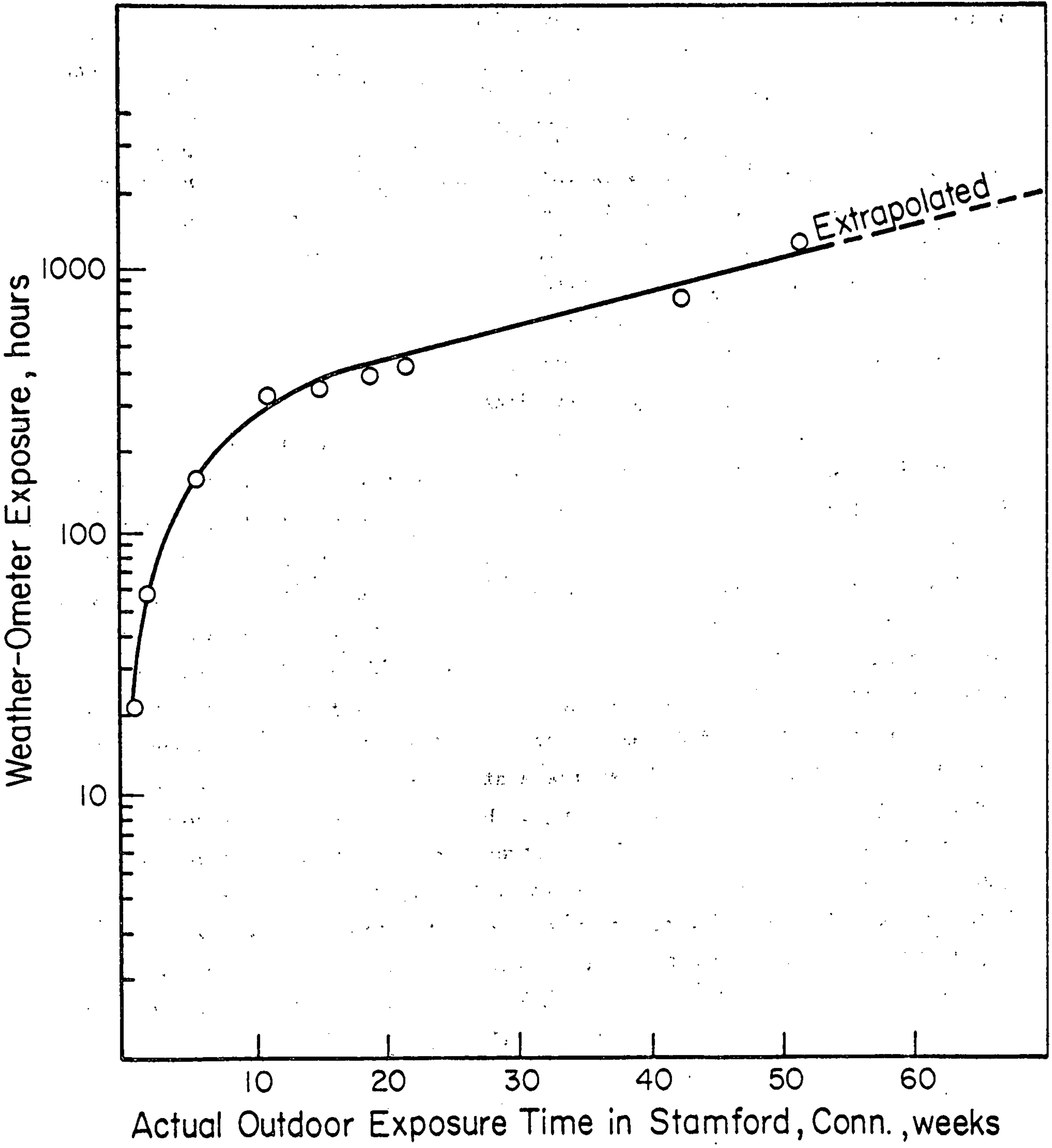


FIGURE 3. CORRELATION OF INDOOR AND OUTDOOR EXPOSURE DATA FOR POLYSTYRENE SHEET

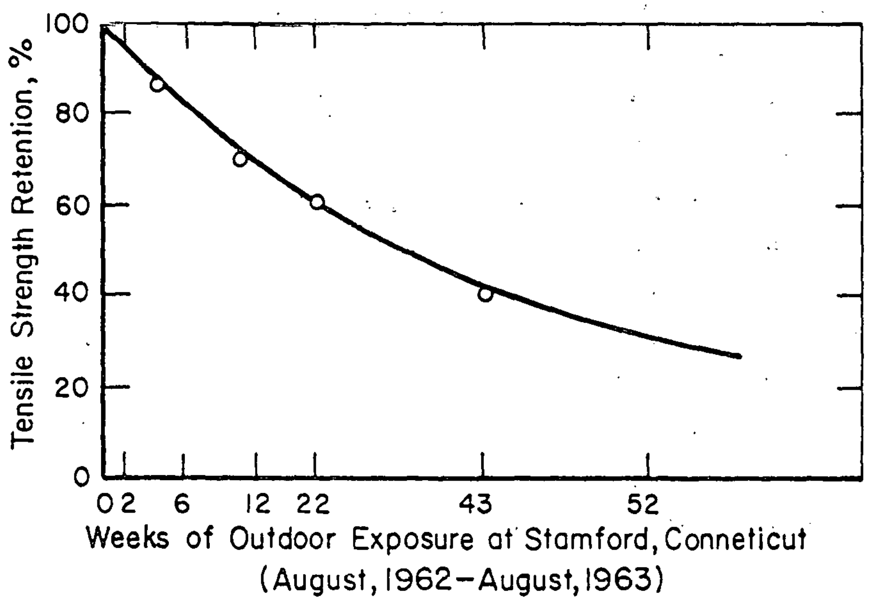


FIGURE 4. CORRELATION OF WEATHER-OMETER AND OUTDOOR EXPOSURE DATA ON POLYSTYRENE

(——) Outdoor results predicted from Weather-Ometer tests

(o) Observed outdoor results

In Figure 5 is a plot of the average number of UV counts recorded normalized to the noon hour (501 maximum UV counts) at Philadelphia, Pennsylvania, versus average number of daylight hours for each month of the year (1974). It can be readily seen from Figure 5 that the lowest UV counts recorded are in the winter months (December, January, etc) with the highest UV counts recorded being in the summer months (July = maximum UV counts). Major UV intensity effects (UV count range, 200-500) occur only at about half the total number of available daylight hours.

There appears to be a strong correlation between annual UV-count versus degrees north latitude (Figure 6). From this relationship (Figure 6), then Des Moines, Iowa; 41.5 latitude (degrees northern) should have approximately the same total annual UV-count as Chicago, Illinois; 41.5 latitude (degrees northern). The maximum average UV-count recorded at the noon hour in July for Des Moines, Iowa, was 572 counts and the total average annual UV-count for all available daylight hours was 1,251,567 counts/12 months or 1 year in 1974.

If we assume that 1 hour of noon summer sun at Chicago or Des Moines is equivalent to the average maximum UV intensity output of the Atlas Weather-Ometer per hour, then the following relationships are proposed:

$$\begin{array}{l}\text{1 hour of noon sun in}\\ \text{July, Des Moines} \simeq \text{Chicago}\end{array} \simeq \begin{array}{l}\text{572 average}\\ \text{UV counts}\end{array} \simeq \begin{array}{l}\text{1 hour in Atlas}\\ \text{Weather-Ometer}\end{array}$$

$$\frac{\begin{array}{l}\text{1,251,567 total}\\ \text{UV average counts}\\ \text{Des Moines} \simeq \text{Chicago}\end{array}}{\dfrac{\text{52 weeks}}{\text{year (1974)}}} \div \frac{\begin{array}{l}\text{572 average}\\ \text{UV counts}\end{array}}{\begin{array}{l}\text{noon hour}\\ \text{July}\end{array}} = \frac{\begin{array}{l}\text{available}\\ \text{daylight}\\ \text{42 hours}\end{array}}{\text{week}}$$

This calculation implies that 42 hours of artificial Atlas Weather-Ometer exposure time should be equivalent to 1 week of actual outdoor exposure in Des Moines or Chicago geographical locations. This, however, is not the case for Stamford, Connecticut (41.03 degrees north latitude) in which only 22 hours of UV exposure in the Atlas Weather-Ometer was experimentally determined equivalent to 1 week of outdoor exposure (240). The difference

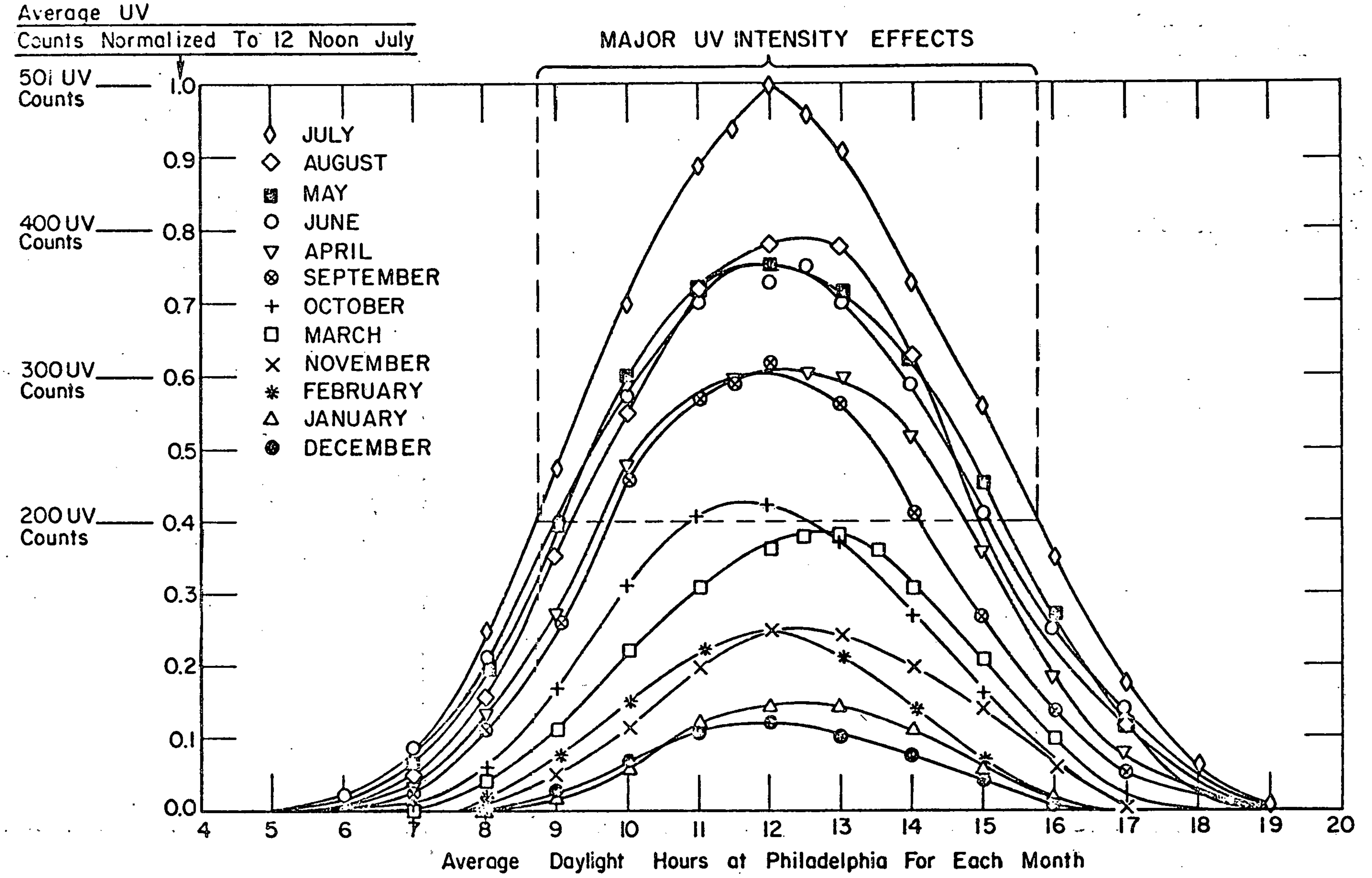


FIGURE 5. MONTHLY DISTRIBUTION OF UV RADIATION IN PHILADELPHIA

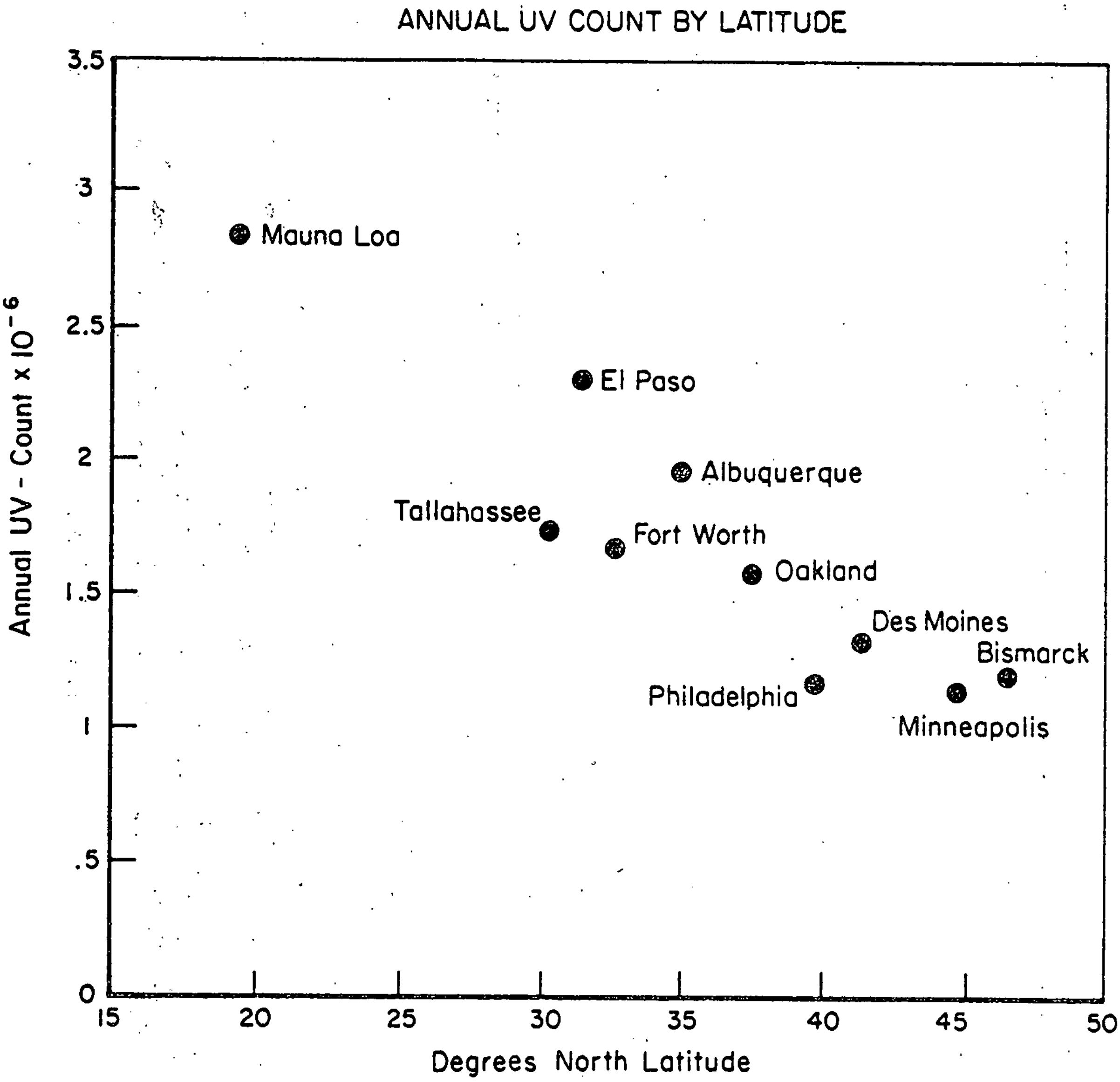


FIGURE 6. ANNUAL UV COUNT BY LATITUDE

actual and calculated exposure times could be caused by other experimental or climatic factors (altitude, cloud cover, latitude difference, etc), but is probably due to the fact that calculated 42 hours per week represents all average UV counts over a total number of available daylight hours. In reality, about 1/2 of the total number of available daylight hours represents UV counts in the range of 200 or 500 or higher intensity UV radiation.

An attempt to correlate different experimental test results on aging of polystyrene sheet materials is given in Figure 7. The variables are exposure times, conditions and time required to reach 50 to 60 percent loss in tensile strength (weeks) versus polystyrene sheet thickness.

Three-mil-thick polystyrene sheets failed in relatively short time periods (43 weeks outdoor exposure at Stamford, Connecticut, or 800 hours exposure in a Weather-Ometer). Another different set of polystyrene samples (125 mils thick) and different exposure testing conditions (3 years outdoors at Dover, New Jersey) also lost 50 to 60 percent of their tensile strength during this time period(242).

In work carried out at BCL, we have exposed some 10-mil-thick polystyrene sheets to 1600 hours of UV exposure in a Weather-Ometer and recorded a 50 to 60 percent loss in original tensile strength of the samples.

Equating over 1600-hour artificial exposure conditions to outside real time, assuming environmental conditions in Stamford, Connecticut (latitude 41.03 degrees north) are not much different than Dover, New Jersey (latitude 40.53 degrees north), requires the following considerations:

(1) 22 hours in Weather-Ometer is equivalent to 1 week outside exposure in Connecticut. Therefore, 1600 hours Weather-Ometer time approximates 73 weeks real time exposure.

(2) 1600 Weather-Ometer hours, extrapolated time, equates to 65 weeks actual outdoor exposure.

(3 Assuming Des Moines = Chicago noon summer sun or 42 hours in Weather-Ometer is equivalent to 1 week; hence, 1600 hours relates to 38 weeks outdoor exposure.

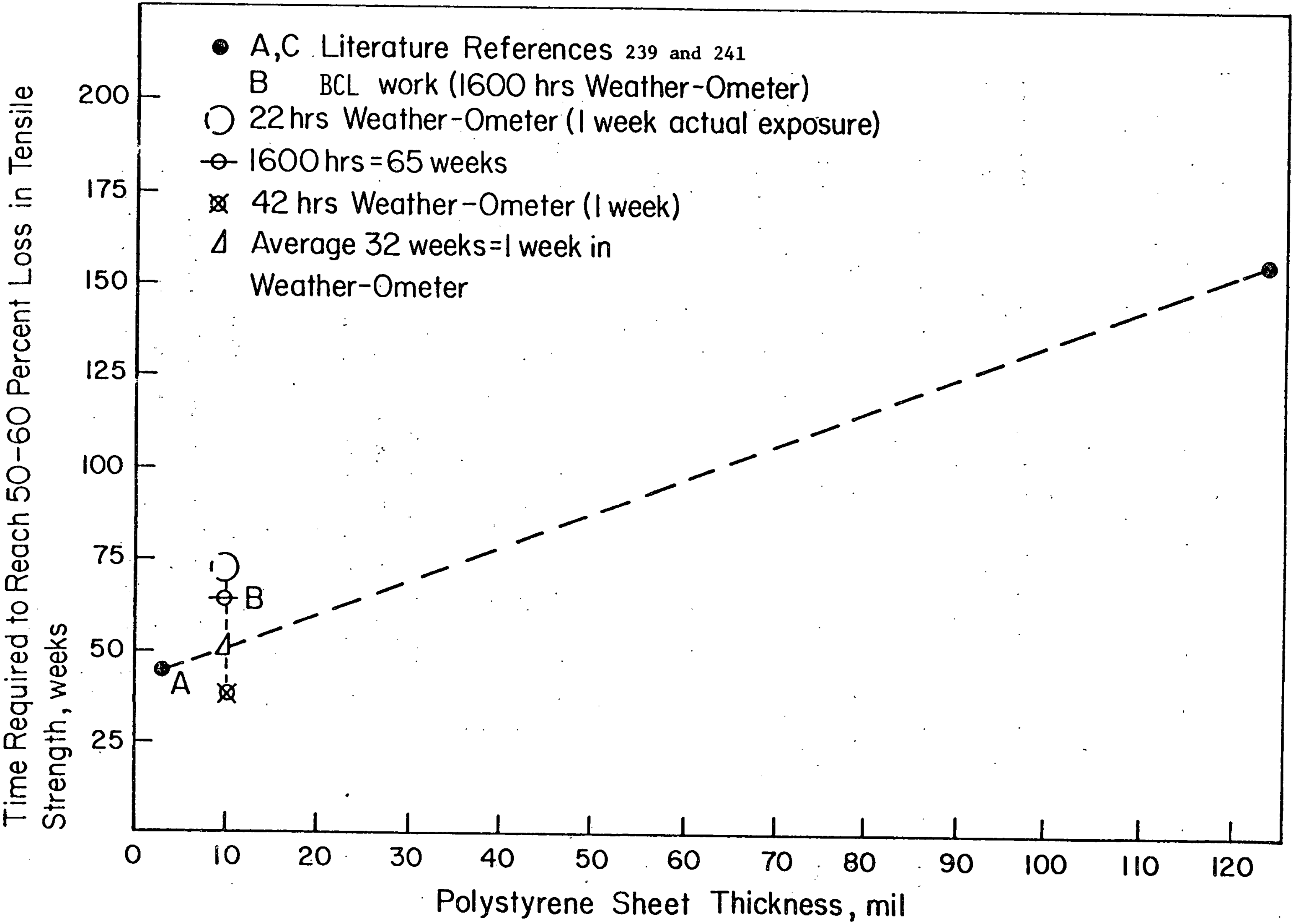


FIGURE 7. POLYSTYRENE SHEET STRENGTH LOSS CHARACTERISTICS FOR VARIOUS THICKNESSES OF POLYSTYRENE

(4) Averages of 22 and 42 Weather-Ometer hours per week of outdoor exposure; hence, 1600 Weather-Ometer hours relates to 50 weeks outside exposure.

The best fit of the data was obtained with an average Weather-Ometer exposure time of 32 hours = 1 week outdoor exposure time

(b) Major Experimental Aging Tests and Models

Literature data on aging of polymeric materials are quite extensive. The types of aging studies found can be divided into two almost mutually exclusive classes according to the goals of the study. The great bulk of literature on aging of polymeric materials provides purely descriptive data, for example, material properties before and after aging for a given time under a given set of conditions. The other class includes studies that attempt to describe aging behavior by mathematical modeling. The latter type of information is discussed in this section of the report. The most pertinent findings, both with respect to the data illustrated and to the methodology brought to bear on their mathematical representation, are summarized in this section.

(1) NBS-MCA Study. One of the most ambitious studies of natural and accelerated weathering to date was the joint industry-government program undertaken by the National Bureau of Standards and Manufacturing Chemists Association. In this program, 20 plastic materials, including six generic plastic species, were exposed beginning in 1966 in Miami, Florida; Phoenix, Arizona; and Washington, D.C. Both clear films and white films of various thicknesses were exposed. Table 11 describes the materials exposed and lists the properties measured. The program was designed to last for 10 years, but was almost aborted for lack of funds after only 6 years. Property measurements were made initially at 3-month intervals and later in the program at 1-year intervals. Since the stated goal of the study was to correlate accelerated and natural aging data, Weather-Ometer testing was also done on the same materials(243).

TABLE 11. MATERIALS AND PROPERTIES EVALUATED IN NBS-MCA STUDY ON WEATHERING OF PLASTICS

Number of Compositions	Materials Evaluated		Thickness, mils
	Generic Class	Color	
Two	Polyethylene	Translucent	1 and 60
One	Polymethylmethacrylate	Clear	60
One	Polyvinylfluoride	Clear	1
One	Polyethylene terephthalate	Clear	5
One	Crosslinked polyester	Clear	60
Fourteen	Various PVC materials	Clear and white	9, 10, and 60

Properties Evaluated	
Color	Tensile properties
Haze	Flexural properties
Glass	Electrical properties
Surface roughness	UV spectra

In the NBS data-analysis approach, the property-versus-time data were fitted to the following equation derived from the Weibull probability density function:

$$P = b_1 \exp\left\{-\left[\left(\frac{t + b_2}{b_3}\right)^{b_4}\right]\right\} + b_5 \quad ,$$

where P is the property level at time, t, and b_1 through b_5 are fitted parameters. The five fitted parameters were claimed to have physical significance as follows:

b_1 - is related to maximum property level
b_2 - is related to pre- or post-aging
b_3 - is related to characteristic life defined as the time required for 63 percent property degradation
b_4 - is related to the presence or absence of an initial induction period
b_5 - is the asymptotic property level at infinite time.

Because of these relationships, the b_1's were called the exposure parameters.

The next step in the data analysis consisted of fitting the important exposure parameters, or meaningful functions of them (such as characteristic life) to a linear equation in meterorological variables as follows:

$$b_i = C_i + C_{IL}(L) + C_{iU}(U) + C_{iH}(H) + C_{iR}(R) + C_{iT}(T)$$

where L is total radiation in langleys, U is UV radiation (Coblentz langleys), H is relative humidity, R is inches of rainfall, T is air temperature, and the C_i's were parameters fitted by stepwise regression. Thus, from the basic meterorological variables L, U, H, R, and T, it should be possible to predict the values of b_i, which in turn could be used to predict property levels or rate of property change at time, t.

The complete data analysis was made only for elongation at break and only for 3 years of data. Therefore, a judgment as to the success or failure of this approach is rather difficult. From the limited data analysis, however, successful results appear to have been obtained for some of the material-site combinations, whereas gross deficiencies were apparent for other combinations.

(2) Leikina and Tatevos'yan Study. In this accelerated aging study, a central-composite, response-surface experimental design was employed to study the effects of temperature, X_1 (30 to 65 C), and radiation intensity, X_2 (150 to 300 W/m^2), on tensile strength, elongation to break, and IR absorption (1720 to 1780 cm^{-1}) of 0.1-mm polyethylene film. This type of design allowed each response to be fitted to a second-degree polynomial equation in X_1 and X_2 as follows:

$$P = b_0 + b_1X_1 + b_2X_2 + b_{11}X_1^2 + b_{22}X_2^2 + b_{12}X_1X_2 \quad ,$$

where P is the fitted property and the b_i's are parameters fitted via multiple-linear-regression analysis. Statistical-significance testing indicated that the equations fitted within experimental error. From the fitted equations, interpolations, possible within the limits of the design, enabled property predictions for conditions not actually evaluated. Since such fitted equations are purely empirical, however, extrapolation outside of the design limits results in predictions with a high level of uncertainty.(244)

(3) Leikina, Tatevos'yan, Kuznetsova, and Melkumov Study. In this study, the effects on polyethylene and polyvinyl chloride of four variables -- irradiation intensity (350-700 w/m^2), specimen surface temperature (20 to 75 C), fraction of time exposed to water during test cycle (1/6 to 1/2), and total test time (170 to 340 hr) -- were investigated. The investigators employed a Soviet DKSTV-6000 weatherometer with a xenon lamp. The experimental design employed was a full 2^4 factorial in which all combinations of the two levels of the four variables were examined. This design permitted the fitting of the following empirical equation to the same properties as in the previous study:

$$P = b_0 + \sum_{i=1}^{4} b_i X_i + \sum_{j=1}^{4} \sum_{i=1}^{4} b_{ij} X_i X_j$$

where, again, P is the property level and the b_i's are fitted parameters. Again, the fitted equations were useful for interpolation, but not for extrapolation.

This study and the previous one are significant because the experimental designs enabled interactions between different independent variables to be elucidated. Synergistic effects go undetected in classical one-variable-at-a-time experimentation, but may be the most important information obtained from an experimental investigation. For example, for polyethylene tensile strength, the interaction between exposure time and temperature turned out to be the most significant effect(245).

(4) Kamal Study. Kamal's study is similar to the two Russian studies just cited, but is somewhat more rigorous even though it predates them. Using a xenon-arc weatherometer, he set up a program consisting of 16 sets of fixed weatherometer conditions (e.g., fixed temperature, fixed length of wet time, and fixed fraction of total time sample was wet). For each set of conditions, polystyrene, polyvinyl chloride, and crosslinked-polyester samples were exposed for 400 hours, and properties of interest, including tensile strength, color change, and UV absorption, were measured at 100-hour intervals. Property-versus-time data were fitted to the equation:

$$\log P = b_o + b_1(t-250) \quad ,$$

where P is the property level at time, t, and b_o and b_1 are fitted parameters. Clearly, b_o is the log of the property level after 250 hours, and b_1 is the logarithmic rate of change of the property with time. Kamal referred to b_o and b_1 as exposure parameters and fitted each of them to a quadratic equation in the weatherometer variables as follows:

$$b_o \text{ or } b_1 = C_o + C_1X_1 + C_2X_2 + C_3X_3 + C_{11}X_1^2 + C_{22}X_2^2 + C_{33}X_3^2 + C_{12}X_1X_2 + C_{13}X_1X_3 + C_{23}X_2X_3 \quad ,$$

where X_1 = temperature, X_2 = length of wet cycle, and X_3 = percent of time in each cycle during which the sample was wet.

As part of this study, samples of the same materials were exposed outdoors with concurrent estimates of temperature, wetness, and UV energy conditions. From information in the above equation, outdoor property values were predicted as a function of time and compared with the actual exposure data.(240)

(5) Natural Rubber Producers Research Association (NRPRA) Study. In this study, K. D. Thomas and R. Sinnott predicted room-temperature modulus changes in both polyarcylonitrile and polychloroprene elastomer systems for up to 5 years from accelerated heat-aging data at 100 to 150 C. Assuming first-order degradation kinetics, they fitted the equation:

$$\log \frac{P_t}{P_o} = kt \quad ,$$

where P_t and P_o are property levels at time, t, and time zero, respectively, and k is the first-order rate constant. Then then related k to temperature, T, using the Arrhenius equation:

$$k = A \exp (-B/RT) \quad ,$$

where A and B are fitted constants (B is the activation energy for the process causing failure). These equations were than used to predict P_t at room temperature for several properties for up to 5 years. The results are shown in Table 12 for 100 percent modulus along with actual property levels from concurrent outdoor testing. As can be seen, the results are quite good, especially in view of the fact that extrapolations were made over wide temperature and time intervals.(245)

TABLE 12. RUBBER AND PLASTICS RESEARCH ASSOCIATION (RAPRA) 5-YEAR PREDICTION RESULTS FOR MODULUS

Time at Room Temperature, years	Change in Modulus, percent			
	Nitrile Rubber		Neoprene Rubber	
	Predicted	Observed (RAPRA)	Predicted	Observed (RAPRA)
1	6	5	4	16
2	12	11	8	19
3	19	17	12	20
4	26	22	16.3	22
5	34	27	21	23

(6) Lockheed Study. The purpose of this study was to predict service life of a propellant. In the accelerated testing plan, cartons of the propellant were aged under nitrogen at 30, 70, 86, 115, and 145 F and at 0 and 5 percent strain for periods up to 120 weeks. Gel content, degree of swell, creep compliance, dilation, and crack propagation were measured at intervals during the testing period. The property-versus-time data were fitted to one of the following equations:

$$P = P_o + k \log t \quad , \text{ or}$$

$$P = P_o + k_1 \log t + k_2 \log^2 t \quad ,$$

where P and P_o are property levels at time t and 1 week, respectively, and k, k_1, and k^2 are fitted rate constants. The linear equation usually was adequate. Where it was not, the quadratic equation was employed. The rate constants k, k_1, and k_2 were related to temperature by the Arrhenius equation ($k = A \exp(-B/RT)$). The compliance equations were extrapolated over time and temperature to arrive at a predicted 1 sigma (σ) service life interval of 4.6 to 11 years. Good agreement with 7- to 8-year modulus data was claimed.(247)

(7) Hill Air Force Base Study. In this study, several properties of various components are being monitored at storage conditions to "detect changes which could reduce service life estimates". The components include various potting compounds, adhesives, spiralloy, and pressure seals. Properties include tensile strength, elongation, hardness, and lap-shear strength. Breakway torque and leak rate are measured on the seals. Data are presently available for up to 8 years for the adhesives and potting compounds and 13 years for the seals. Measurement intervals for all but the seals are approximately 1 year except for the first year where 1- and 6-month measurements were made for some properties. The number of replications at each data point vary from three to ten. Pressure seals are tested every other month with no replication.

Reported data treatment consists simply of fitting a linear relationship to the data as follows:

$$P = P_o + b_1 t \quad ,$$

where P is property level at time t, P_o is initial property level, and b_1 is the fitted slope. The slope is then tested for statistical significance. No attempts are made to predict service life or to evaluate alternative models.(248)

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APPENDIX A

BCL EXPERIENCE WITH MATERIALS AND TESTING METHODOLOGIES FOR PHOTOVOLTAIC SOLAR CELL COMPONENTS

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APPENDIX A

BCL EXPERIENCE WITH MATERIALS AND TESTING METHODOLOGIES FOR PHOTOVOLTAIC SOLAR CELL COMPONENTS

This study, although not directly aimed at plastic solar thermal collectors, does examine many related state-of-the-art materials and testing methodologies suitable for use or application in plastic thermal collector systems.

INTRODUCTION

Today, a major emerging technology is that of photovoltaic conversion, i.e., the conversion of solar energy (sunlight) into electricity. This process utilizes solar cells to generate electricity through a mechanism that involves electron displacement within the cell by photons of sunlight.

The amount of electricity generated by individual cells is small, of the order of hundredths of a watt, but larger amounts can be obtained by utilizing a number of such cells combined electrically. An initial combination of some 10 to 100 cells is called a photovoltaic array. A further combination of a number of arrays is termed a module. Ultimately, large numbers of modules are combined that occupy acres of area and are the source of large quantities of electrical power.

Before solar energy can provide electricity competitively on a large scale, however, solutions must be found to a number of technical problems. The first of these is <u>process efficiency</u>. Current solar cells, of silicon, operate at an efficiency of only about 10 percent. Maximum possible efficiency of such cells at the earth's surface is about 21 percent and is further reduced by (1) surface reflections, (2) high electrical resistivity of the transparent cell cover, and (3) imperfections in silicon due to chemical impurities and structural irregularaties.(A-1)

Next, there is the problem of long-term (20-year) protection of the cells in weathering environments. The solar cells cannot maintain optimum electrical performance unless the adverse effects of weather on both the amount of sunlight reaching the cells and the photovoltaic conversion process are minimized.

To maximize the amount of sunlight received requires that the encapsulant components on the sun-side of the cell be transparent to the solar spectrum. In addition, the optical coupling of the encapsulant to the silicon surface must be such that reflection losses in the solar spectrum are low. To maximize system performance with time in the weathering environments requires that the encapsulant provide protection to the electrical componentry. On the basis of experience with silicon devices, primarily in the microelectronics field, silicon solar cells might be expected, with substantial confidence, to have a service life of 20 years in terrestrial use, provided they are protected from the environment. This need for protection also includes protection for the cell interconnects and the metallizations on the cell which aid in the current-collection process.

Finally, it will be necessary to maximize cell efficiency and system performance at a total cost that is competitive with those of other sources of energy.

It is the overall objective of current Low Cost Solar Array (LSA) Projects to develop solutions to each of the major technological problems identified above and to provide a total high-performance system at a cost of not more than $500/kilowatt on the basis of 1974 dollars. It is the goal of this present BCL study on encapsulation materials to provide candidate encapsulant system designs with identified materials/processing methods that will insure long-term array performance at a cost that is consistent with that of the overall LSA Project goal.

OBJECTIVES OF THE BATTELLE PROGRAM

To date, the encapsulation systems (materials/processing methods/design) that have been used in the commercial manufacture of photovoltaic arrays have not met the requirements of the LSA Project. None has provided the appropriate mix of properties, i.e., high conversion efficiency, long-term performance, and low cost, that is necessary to assure a competitive position for photovoltaics in the marketplace. As a result, a considerable need exists for information on the properties, processing, and aging characteristics of polymeric and glass materials that are encapsulant candidates for low-cost array designs.

Consistent with this need, it has been the overall objective of this program at Battelle to evaluate selected materials and material combinations for use in encapsulation systems on the basis of (1) critical initial properties and characteristics and (2) their behavior over time in selected environments. The environments included high relative humidity, temperature cycling, and ultraviolet radiation. The emphasis was placed on screening evaluations, within the scope of the effort, to make possible initial comparisons for the many materials and material combinations in a relatively short time period, and to permit recommendations of materials and systems for subsequent development.

The program differs from other LSA Project studies in that system and subsystem performance, i.e., performance of combinations of materials, relevant to potentially very low-cost ensapsulant system designs has been emphasized. Materials have not been excluded from consideration solely on the basis of materials and processing costs. Rather, it has been Battelle's approach to emphasize system designs that require minimal amounts of materials and, in this way, provide an alternate approach to the attainment of program cost goals.

BACKGROUND

Materials-Performance Considerations

In any hostile environment, there appear to be two principal reasons for the deterioration in photovoltaic cell output. These are (1) reduction in the amount of light (300-1200 nm) transmitted to the cells and (2) failure of the electrical system (cells, interconnects, leads, etc) to utilize available light. A number of causes of each of these types of system deterioration have been observed and are identified below.

1. Causes of Light Transmission Losses
    - a. Degradation of encapsulant material(s) in the light path to the cells (discoloration, hazing, yellowing, etc, from UV, thermal oxidation, etc)
    - b. Delaminations at the encapsulant cover/adhesive (or pottant) or adhesive (or pottant)/cell interfaces (formation of air or water gap)
    - c. Surface abrasion of the encapsulant cover
    - d. Surface reactions of the encapsulant cover with water and/or other chemicals
    - e. Accumulation of dirt, salt, ice, snow, etc, on the encapsulant cover
2. Causes of Failure of Electrical System to Utilize Available Light
    - a. Corrosion of metallic members associated with water (or other) vapor permeation through the adhesive (or pottant)/cell interface or with outgassing (establishment of corrosive condensed phase in contact with metallic elements)

b. Corrosion of metallic members resulting from delamination and/or environmental stress cracking to provide paths for "vapors" to the corrodable components

c. Corrosion of metallic members brought about by absorption and/or reaction of the encapsulant materials with water vapor and/or other chemicals followed by swelling, degradation of barrier properties, etc.

Materials/Processing Cost Considerations

In addition to long-term performance during exposures to weathering environments, a requirement that imposes a high degree of constraint on materials selection, raw materials, and processing costs provide still a second such requirement. Currently, it would appear that short-term (1980-1985) costs for encapsulant materials, including the use of certain preformed components, e.g., polymeric sheet, reinforced substrate, etc, will be in the \$10-\$15/m^2 ($\sim$ \$1.00-\$1.50/ft^2) range. Such costs are predicated on the use of a simple sheet-film or sheet-conformal coating encapsulant design in which the sheet member is structural and may serve either as the transparent cover or as the substrate of the array.

In estimating the \$10-\$15/m^2 cost, a system was assumed composed, from top to bottom, of a transparent film cover, a 50-125 μm (2-5 mil) layer of adhesive (or pottant), a 300-μm (12-mil) layer comtaining cells at a geometric fill factor of 67 percent and adhesive, a second 50-125 μm layer of adhesive, and a structural substrate. It was further assumed that a minimal amount of sealant was applied to the edge areas of the laminate. Based on projected long-term LSA goals, this cost figure probably is high by a factor of at least 3 to 5. Consequently, cost considerations are a very major factor and further sizeable reductions must await the development of new and/or improved materials and processing methods.

EXPERIMENTAL

Encapsulant Materials Selection

Property guidelines used in the selection of encapsulant materials candidates are identified in Table A-1. In terms of specific properties, transparency in the solar spectrum was a primary consideration in the selection of all materials exclusive of the substrates discussed below. Other properties/characteristics that were weighed particularly heavily were weatherability, useful temperature range, and processability. The latter includes handleability, repairability, and ease of automation.

Materials costs and availability also were important in the selection process. However, because both processing considerations and the finalized system design(s) are basic to the establishment of total system costs, certain materials having relatively high unit costs (e.g., certain silicones, epoxies, and fluorocarbons) were selected for consideration. These materials appear to fulfill functions in certain conceptualized designs for the encapsulation system that cannot be obtained with lower cost materials. It is anticipated that design modification could be made, if required, to minimize the amounts required of certain of these high-cost materials while maintaining their unique system functions. Ultimately, it is anticipated that materials development programs are likely to provide less expensive replacements for, or modifications of, these materials.

It should be emphasized that the materials candidates that have been examined in this study are, in many cases, only representative of a number of viable ones available from a variety of manufacturers/suppliers. The identification and characterization of all materials of potential interest for each end-use application (adhesives, coatings, etc), of course, is not feasible. It is believed, however, that the materials selected are consistent with the general property requirements of interest for terrestrial solar-cell encapsulation. A discussion of the selected materials according to the several end-use applications follows.

TABLE A-1. PROPERTY GUIDELINES USED IN MATERIALS SELECTIONS

Cover

Low cost

Transmittance

UV stability/weatherability

Abrasion resistance

Barrier properties (e.g., to moisture)

Low adhesion of dirt and other pollutants

Appropriate structural properties[a]

Adhesive

Good adhesion to cover and substrate

Low cost (including primer, if necessary)

Transmittance (depending on location)

UV stability/weatherability

Low modulus (probably)

Forms moisture barrier with cell/cover

Substrate

Low cost

Moisture barrier

Weatherability

Appropriate structural properties[a]

(a) Structural properties required depend upon design of encapsulant system.

Adhesives

Adhesives can be classified either as structural or nonstructural. Generally, the former are load-bearing materials that will strengthen structures, often to the extent that the bond becomes as strong or stronger than the materials joined. These high-strength materials are resin based and are used to join various types of materials: metals, glass, plastics, etc. For the most part, they are thermosetting types and available both as dry film (prepreg) and as liquid resins. Generally, curing is effected with heat. Most epoxy adhesives are examples of this class of materials.

The nonstructural adhesives are nonload-bearing, and are used with a variety of materials where there is no need for high stress resistance. They are thermoplastic, or noncuring, and can have either a rubber or resin base. Certain silicones and urethanes are examples of the rubber-based adhesives; certain acrylics are representative of the resin-based nonstructural systems.

The high strength attributable to the structural adhesives generally is accompanied by a high degree of rigidity. The rubber-based nonstructurals, on the other hand, have excellent flexibility. Between these two extremes are the resin-based systems.

From the standpoint of the utilization of adhesives in solar-cell encapsulation, considering various potential designs for the ultimate encapsulant system, both the structural and nonstructural materials classes must be considered. However, in the current work, emphasis has been placed on the use of low-modulus materials and, consequently, nonstructurals predominate. The adhesives selected for evaluation are listed in Table A-2. A detailed property information compendium is presented in Appendix B. It is recognized here, as in the selection of materials examples for other types of end-use applications, that a number of other materials representing the same classes may provide equally good, or possibly superior, properties for the encapsulation task.

Adhesives candidates for bonding silicon cells to the plastic cover must be clear and UV stable. Of the structural types, the epoxy (Scotch-Weld 2216 B/A, clear) or an aliphatic-isocyanate-cured urethane

TABLE A-2. SELECTED ADHESIVES CANDIDATES

Trade Name	Manufacturer/Supplier	Class
	STRUCTURAL	
Cavalon 3100S (TS)[a]	Du Pont	Room temp. curing acrylic
Scotch-Weld 2216 B/A	3M	Room temp. curing epoxy
Scotch-Weld 3520 B/A	3M	Room temp. curing epoxy
Structural 3532 B/A	3M	Room temp. curing urethane
	NONSTRUCTURAL	
Acryloid B-7 (TP)[a]	Rohm & Haas	Thermoplastic acrylic
B-2397-10	Hughson	Laminating urethane
Chemlok 7000/7203	Hughson	Laminating urethane
Chemlok 7002/7203	Hughson	Laminating urethane
DC 282	Dow Corning	Pressure sensitive silicone
RTV 118	G.E.	Room temp. curing silicone
Scotch Grip 4475	3M	Resin contact (plastic)
Scotch Grip 4693	3M	Elastomeric contact
Silastic 732 RTV	Dow-Corning	Room temp. curing silicone
Silastic 734 RTV	Dow-Corning	Room temp. curing silicone
Silgrip SR-573	G.E.	Heat sealable silicone
Silgrip SR-574	G.E.	Pressure sensitive silicone

(a) The abbreviations TS and TP designate thermosetting and thermoplastic materials, respectively.

have the best potential if flexibility and moderate strength are desirable. Also acceptable are such nonstructurals as Acryloid B-7 (acrylic), Silastic 732 RTV or RTV 118 (elastomeric silicones), and Silgrip SR-573 (silicone resin).

For certain bonding applications associated with cell encapsulation (e.g., substrate-to-cell bonding), optical clarity and UV stability are not necessary requirements. Scotch-Grip 4475 and 4693 are adhesives of this type. They have been studied primarily because of their excellent bond strengths to certain plastics. Adhesives of this type also are important from the standpoint of applicability to high-speed film laminations with good bond strengths.

Cover Films and Sheets

In discussing the selection of materials for use as covers in encapsulation systems, the distinction between films and coatings (discussed later) is made on the basis that films are separate structural units formed prior to application. As such, they are generally distinguished from sheet materials only arbitrarily, on the basis of thickness. The dividing line is not well defined, but certainly materials less than 500 μm ($\approx$1/16 inch) or more can be classified as sheet.

Film and sheet selections evaluated in this study are listed in Table A-3 and have been drawn primarily from the acrylic, halocarbon, polycarbonate, and thermoplastic polyester materials classes. These classes have provided a broad range of moduli from very flexible to quite rigid. Also identified in Table A-3 is a specialty film material, 3M Company's Flexigard. It is a film laminate proposed by the manufacturer for use as outer windows of solar collectors. Film/sheet materials can be used as protective materials (moisture barriers) for the underside of the encapsulant design as well as in cover applications. Aclar-type film, for instance, is especially good for moisture-barrier properties.

TABLE A-3. SELECTED COVER CANDIDATES

Trade Name	Manufacturer/Supplier	Class	Thickness, μm (mils)
	Film		
Aclar 33C	Allied	Halocarbon	51 (2)
Dyed Mylar (weatherable)	Martin Processing Co.	Polyester (TP)(a)	178 (7)
Halar	Allied	Halocarbon	127 (5)
Korad A	Xcel	Acrylic	153 (6)
Flexigard	3M	Acrylic-Polyester	127 (5)
Tedlar 400 BG20TR	Du Pont	Halocarbon	102 (4)
"Teflon" FEP (Type C)(b)	Du Pont	Halocarbon	127 (5)
Tuffak No. 90230-D	Rohm and Haas	Polycarbonate	508 (20)
	Sheet		
Acrylite	American Cyanamid	Acrylic	3175 (125)
Lexan 9030 (UV - stab.)	G.E.	Polycarbonate	3175 (125)
Sun-Lite (Premium)	Kalwall	GR Polyester	635 (25)

(a) TP designates thermoplastic-type material.
(b) Type C is treated on one side to promote bonding.

Coatings and Pottants

In considering various designs for the encapsulation of solar cells, polymeric coatings may find utility in any of a number of different protective applications. They may be of value (1) in improving the resistance to ultraviolet (UV) radiation, abrasion, and other environmental effects on a protective cover, (2) in protecting soft potting compounds from dirt and other environmental hazards and in facilitating the removal of ice and snow from such materials, (3) in relieving thermal stresses between other components of the assembly, and (4) providing high electrical insulation between system components and a barrier against moisture and other environmental effects through direct application to the silicon cells and interconnects.

The selected list of coatings and pottants for evaluation is shown in Table A-4 and includes the acrylic, epoxy, urethane, poly(vinyl)-butyral), and silicone classes of materials. The list includes materials that may be of interest directly as solar-cell coatings as well as general-purpose coatings for various components of the encapsulation system.

Coatings with clarity, UV stability, moisture resistance, and moisture-barrier properties would be ideally suited for conformally top coating the solar cell. Most coatings in the table above were selected for such an evaluation.

Pottants (or potting compounds), generally, are one- and two-component liquid systems. Potting is an embedding process in which the material or mixture of materials is "poured" into a "container" and bonds directly to it. Usually no mold is used, and the container becomes an integral part of the assembly.

The problem areas associated with the potting of electronic components include high curing exotherms, resin shrinkage and subsequent stress development, inadequate thermal-shock resistance of materials and components, process control problems associated both with raw materials and processing, and outgassing and corrosivity problems. For the LSA Project, these problems are compounded by the added requirements for optical clarity and small coefficient-of-expansion differences with silicon, at least in the majority of envisioned encapsulation designs.

TABLE A-4. COATINGS AND POTTANT CANDIDATES

Trade Name	Manufacturer/Supplier	Class
	Coatings	
Butvar B-79	Monsanto	Poly(vinyl butyral)
Butvar B-98	Monsanto	Poly(vinyl butyral)
Butvar BR	Monsanto	Poly(vinyl butyral)
Butvar Dispersion FP	Monsanto	Poly(vinyl butyral)
DC-3140 (1204 primer)	Dow Corning	Silicone
Eccocoat AC-8	Emerson and Cuming	Acrylic
Eccocoat RTU	Emerson and Cuming	Urethane
Eccocoat VE	Emerson and Cuming	Epoxy
Glass Resin 650 (50%)	Owens-Illinois	"Silicone"
Glass Resin 650 (25%) (GR-70105-2)	Owens-Illinois	"Silicone"
	Pottants	
Q3-6527 A/B (gel)	Dow Corning	Silicone
RTV-615	General Electric	Silicone
RTV-655	General Electric	Silicone
Sylgard 184	Dow Corning	Silicone

In Table A-4, Q3-6527 A and B requires heat to accelerate cure, although it will gel at room temperature in 24 hours. Heat also will speed the cure of Sylgard 184.

The option of using a coating on the underside of the solar cell requires that the coating be selected on the basis of three principal properties. These are adhesion, weathering resistance, and low moisture permeability. In such an encapsulant design, it is likely that the top of the cell would be bonded to a clear cover (glass of polymer). Coatings for this application need not be clear, and could include materials based on butyl rubber (Butyl LM430-Exxon), polyisobutylene, polyvinylidine chloride-polyvinyl chloride copolymer, and halocarbons resins. Silane or titanate coupling agents can be used to achieve good bonding where marginal adhesion of the coating to the solar cell is experienced. (Alternatively, metal foil or sheet can be used if insulated from the cells and interconnects.)

Substrates

A number of materials types are candidate substrates, including metals, glasses, and polymerics. In considering the polymer materials, reinforced materials or laminated structures are viable forms for the thermal and mechanical requirements (e.g., epoxies and polyesters).

In considering reinforced sheet materials it was anticipated that the sheet would likely be bonded, either adhesively or mechanically, to a suitable retaining structure to form the final assembly. It has been recognized, of course, that certain system designs may not require a structural substrate as defined here. For example, the roofing-shingle or roll-out blanket-type designs would be attached directly to a retaining system without a substrate as defined.

Other Substrate Materials. Two other materials, a 102-μm (4-mil) aluminum foil and an 89-μm (3.5-mil) composite (Alure CX) were used as substrates in several film-laminate encapsulant constructions. The latter material (St. Regis) is composed of a 13-μm (0.5-mil) metallized polyester and a 76-μm (3.0-mil) coextruded high-density polyethylene. It is reported to have excellent water vapor barrier properties. Wood products coated steel sheet and styrene foams are also candidates but were not included in the scope of this evaluation.

Measurement of Materials Properties

In this section, the property measurements made on individual materials and combinations of materials are described, and the test cabinets used to expose these materials as well as the encapsulated cells are identified. Included were normal light transmittance measurements, moisture barrier property evaluations, and bond tests. These were made on "as-received" or "as-prepared" samples and following exposures to ultraviolet (UV) irradiation, thermal cycling, high humidity, and high temperature. The test methods and environmental exposures employed in this study are described, together with pertinent equipment and instruments identification.

Light Transmittance Measurements

A Cary Model 14M spectrophotometer equipped with a Model D lamp power supply was used to measure normal optical transmittances of selected cover film (sheet) materials, of free adhesive films, and of materials subsystems. Optical characterizations were performed before and after exposures to specific weathering environments. Where adhesives were used in combination with the film (sheet) materials, the latter were precleaned with methyl ethyl ketone (MEK) or hexane. Adhesive application was made by brushing where solvent attack on the film (sheet) was not a problem. Where such a problem existed, preformed free films of the adhesives were used. The free films were prepared by wet casting onto a release paper followed by a pre-application solvent-evaporation step.

Film (sheet) specimens with adhesive applied were cured at room temperature, where required, prior to environmental exposures. For exposures, samples were mounted on 7.6 x 22.9-cm (3 x 9-in.) aluminum panels using 3M Magic Mending Tape (No. 810). Following exposures, samples 2.5 x 5 cm) (1 x 2 in.) were prepared for use in the Cary. Normal transmittance (optical densities) were determined over the frequency range from 300 to 1200 nm.

Water-Vapor-Barrier Property Measurements

Water-vapor-transmission-rate (WVTR) measurements were made on film (sheet) materials according to ASTM D96-66, using a Vapometer (Thwing-Albert Instrument Company). The Vapometer is a cup 6.4 cm (2.5 in.) in diameter and 5.1 cm (2.0 in.) deep. A circular disc of the film or sheet material, measuring 7.6 cm (3.0 in.) in diameter, was held in place on the cup by a retaining ring and setscrews. Fifty grams of Drierite were used inside each cup. Total exposed film (sheet) area was 0.0032 m^2. Test temperature was 23 C, and outside the cup the relative humidity was maintained at 95 percent.

The measurement of the rate of water-vapor transmission through film/adhesives was accomplished through the use of a film pouch (or "pillow pack"). Figure A-1 shows a schematic of one-half of the pouch. A indicated, two square films (8.9 cm (3.5 in.) on a side, were bonded together along the outside edges of the films. The bond area was 1.27 cm (0.50 in.) wide; the total bonded area was 38.7 cm^2 (6.0 $in.^2$). WVTR calculations are based on the film area encompassing the desiccant, 81 cm^2 (12.5 $in.^2$). Pouches were prepared by bonding three sides, inserting the desiccant (10 grams of Drierite), and finally sealing the fourth side. Exposure to UV and temperature cycling was conducted prior to inserting the desiccant and sealing off the fourth side.

A box-type container was employed to determine the rate of water-vapor transmission through the rigid sheet/adhesives. A schematic drawing of this arrangement is shown in Figure A-2. The top and bottom of the container were pieces of sheet material 6.4 cm (2.5 in.) square. Two "picture frame" center sections were made with the same overall size as the top and bottom pieces. Each picture frame was 1.27 cm (0.50 in.) wide. When assembled, a cavity 3.8 cm (1.5 in.) square by 0.64 cm (0.25 in.) deep was formed that would hold 10 grams of desiccant. Water-vapor-transmission rate calculations are based on the total sheet area encompassing the desiccant, 29 cm^2 (4.5 $in.^2$). Exposure to UV and temperature cycling was performed before adding the desiccant and bonding the top sheet to the assembly.

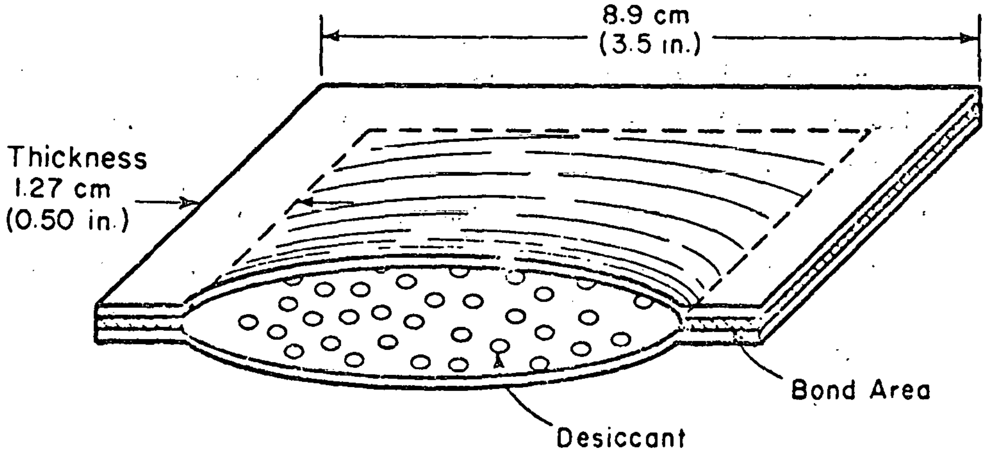

FIGURE A-1. SCHEMATIC OF FILM POUCH (PILLOW PACK) USED IN DETERMINING WATER-VAPOR TRANSMISSION THROUGH FILM/ADHESIVE SUBSYSTEMS

(Drawing shows one half of square pouch.)

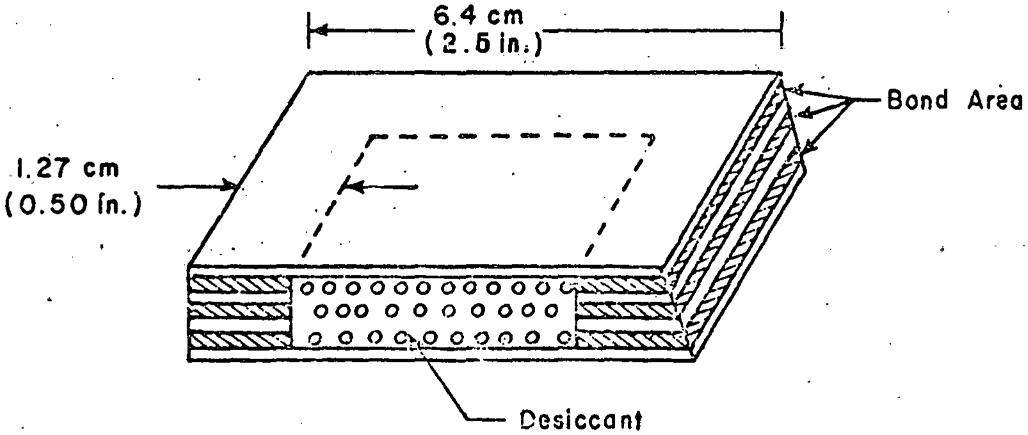

FIGURE A-2. SCHEMATIC OF BOX-TYPE CONTAINER USED IN DETERMINING WATER-VAPOR TRANSMISSION THROUGH SHEET/ADHESIVE SUBSYSTEMS

(Drawing shows one half of square container.)

Following preparation, the film pouches and sheet containers were placed in a closed 23 C environment in which the relative humidity, exterior to the samples, was maintained at 95 percent. Periodically, test samples were removed and weighed. Data from successive weighings against elapsed time were plotted. The slope of the resulting straight-line plot defined the WVTR.

Bond Strength Measurements

Tests of the adhesive bonds were performed using a modification of ASTM D1002-72. Although numerous bond test methods have been investigated, none is universally applicable nor accepted. For the screening evaluations of this study, this test appeared useful and cost effective. However, it should be noted that data suitable for design purposes are not obtained. Specimens were prepared by cutting two 2.5 x 12.7-cm (1 x 5-in.) strips of film on a TMI Precision Paper Cutter. An area of approximately 6 cm^2 on each was marked off for adhesive application. The film areas to be bonded were cleaned with either hexane or MEK. Adhesive was applied to each by brushing on a uniform layer; assembly was carried out according to adhesive manufacturers' suggestion. Most specimens were assembled within a 2 to 3-minute time period after adhesive application. Some, however, required longer drying periods for removal of solvent from the adhesive or curing agent.

For bonding with nonstructural adhesives, a 2.0-kg (4.5-lb) rubber-covered roller (Pressure Sensitive Tape Council, Glenview, Illinois) was used to apply pressure to the bonded area. For structural adhesive specimens, assembly was made by contacting the two specimens and exerting only sufficient pressure (with the fingers) to insure good wetting of the film substrates. Adhesives were cured or conditioned for 3 to 5 days, depending on adhesive type, followed by a 4-hour heat treatment at 50 C. For environmental exposures, specimens were mounted on aluminum panels as described earlier.

Four types of controls were used in this study. Included were unexposed specimens and specimens exposed to 45 C, to 58 C, and to 90 C. Humidity was not controlled in the forced-air ovens used for the thermal controls.

After exposures, bond-strength measurements were performed using a table model Instron Tester at a jaw separation speed of 0.5 cm/min (0.2 in./min). Three specimens were measured for each specimen type evaluated. For thermal controls, only one or two samples were used for each temperature. The reported values are those representative of the highest load that was applied before adhesive or cohesive failure or film breakage or elongation took place.

Measurement of Solar Cell Parameters

A substantial portion of the evaluation of encapsulant materials was augmented by measuring the current-voltage (I-V) characteristics of commercial solar cells which were encapsulated by the selected materials or material combinations. For all of the measurements reported, a single type of cell was utilized. The identifying features are listed below.

- Supplier: Spectrolab, Sylmar, California
- Material: Silicon (n/p)
- Dimensions: Round disc, 5.1 cm (2 in.) in diameter
  0.25 to 0.30 mm (10 to 12 mils) thick
- Metallization: Silver, silk screened
- Antireflecting coating: SiO_x.

According to the supplier, the metallization is applied by thick-film techniques. Different silver "pastes" are used for the collector grid and back (solid) contacts. Annealed copper leads were soldered to the metallization grids and back collector.

I-V characteristics were measured in the as-received (unencapsulated) condition for each cell for which encapsulated data are reported. It is to be noted that considerable nonuniformity in characteristics was measured among the cells used. Efficiencies ranged from approximately 8 percent to 13 percent. In most of the results reported, changes in cell parameters rather than absolute values are given so as to obviate these nonuniformities to some degree.

Specific cell parameters were measured in the as-received condition, after cleaning, after initial encapsulation, and after exposures to a particular environment for a measured length of time. The environments are described in subsequent paragraphs of this section. The parameters determined were

- Open-circuit voltage, V_{oc}
- Short-circuit current, I_{sc}
- Maximum power, P_{max}
- Current at maximum power, I_{max}
- Voltage at maximum power, V_{max}
- Fill-factor (electrical), F.F.
- Series resistance, R_s
- Shunt resistance, R_{sh}
- Efficiency, in percent, η.

These parameters assume the conventional equivalent circuit wherein a current generator is in parallel with an ideal diode and a shunt resistor. A resistance is in series with the load.

The light source for the cell measurements consisted of 4 ELH lamps (General Electric or Sylvania) mounted in a lighttight enclosure and radiating onto a water-cooled copper plate 50 cm from the lamps. A regulated supply powered the 300-W lamps. A light intensity of 100 mW/cm^2 was employed for the "high-intensity", or standard, measurement. This quantity was determined with a standard cell fabricated by NASA LeRC (Standard Cell Z-34).

Environmental Exposures

Four types of environmental exposures were used in this program. These were ultraviolet radiation, thermal cycling, high humidity, and isothermal aging at elevated temperature. The UV exposures were carried out in either a xenon lamp Weather-O-Meter, Atlas Model 60W, or a carbon arc Weather-O-Meter, Atlas Model XW-R. Thermal cycling from -40 to +90 C at a 6 cycles/day rate was performed in a Webber cabinet (Model No. WF-6-125-300). High-humidity exposures (100 percent R.H. and 38 C) were carried out in a Precision Scientific Company (Army-Navy Aeronautical-Spec AN-H-31) cabinet. High-temperature aging treatments were performed in a conventional air-circulating oven. Cell-parameter and materials-property measurements were made before and after the different environmental exposures.

Materials Evaluations

Summarized in this section are the results of the initial screening evaluations of presently available, candidate encapsulation materials, combinations of materials and their interactions/interfaces, and encapsulated cells representing the selected encapsulation system designs and materials described earlier. Comments are made about the results and the behavior of the various materials in the aging environments described previously. The first part of this section gives the results for "total polymeric systems" (all of the encapsulation components are polymers). The results for systems employing glasses as the top cover, and in some cases also as the substrate, are discussed next.

Light Transmittance Studies

Values of normal light transmittance were obtained in the Cary instrument at 100-nm increments over the range 300 to 1200 nm for selected film, sheet, adhesive, and adhesive/film or sheet combinations, before and after exposure to specific weather environments. The performance of materials has been characterized by comparing transmittances before and after exposure and computing values of R as defined by the equation:

$$R = \frac{T_{\text{exposed}}}{T_{\text{unexposed}}} ,$$

where T is the transmittance. Generally, R values are reported for the wavelength range 300 to 700 nm, the range 700 to 1200 nm, and the total range 300 to 1200 nm. These values are designated R_{VIS}, R_{IR}, and R_T, respectively. For each range, the transmittance was incrementally integrated at 100-nm intervals.

Optical Performance of Film and Sheet Cover Materials. For selected film and sheet cover candidates, the effects of individual exposures to UV, thermal cycling, and high humidity on normal light transmittance were determined. The data (summarized in Table A-4) indicate that the materials generally are not significantly affected by either UV or thermal cycling. The transmittance of Sun-Lite sheet appears to decrease somewhat after thermal cycling (R_T = 0.88).

High-humidity exposure appears to affect the optical properties of Korad A films, particularly in the IR range. The films turn milky after several hours of exposure, with a resultant R_T value of 0.91. All other film and sheet candidates were not markedly affected by the high-humidity exposure.

Optical Performance of Film- and Sheet-to-Adhesives Combinations. Film and sheet total encapsulant designs will contain a combination of a film or a sheet and an adhesive above the silicon cell. This combination will constitute the material path that must be traversed by light incident on the array. To examine the effect of weathering environments on such subsystems, combinations of film or sheet and adhesive, or three plies of these materials in which the adhesive us the inner layer of the sandwich, were subjected to ligh-transmittance measurements with R-value computations as above.

Transmittance data for a number of selected adhesives with the major cover film and sheet materials are listed in Tables A-5 and A-6. In several cases, free films of the adhesives were cast and subjected to the same environmental aging.

Scotch-Weld 2216 B/A. Data obtained for various cover candidates with Scotch-Weld B/A, a moderately flexible, structural epoxy, are presented in Table A-6. Note that three types of specimens are included: a "free" film of the adhesive, a film (or sheet)/adhesive combination, and a film/adhesive/film (or sheet) sandwich. The adhesive alone and the film/adhesive specimens were exposed to the various environments for

TABLE A-5. TRANSMITTANCE RATIOS(a) OF ENVIRONMENTALLY EXPOSED FILM AND SHEET COVER CANDIDATES

Film or Sheet Material (Thickness)	Exposure Type(b)	Exposure Time, hr	R_T	R_{IR}	R_{VIS}
Tedlar (102 μm)	none	--	--	--	--
	UV	1002	0.98	0.97	1.00
	TC	1009	0.94	0.94	0.95
	HH	500	0.96	0.96	0.97
Korad A (152 μm)	none	--	--	--	--
	UV	1002	0.99	0.99	1.00
	TC	1012	0.99	0.97	1.02
	HH	500	0.91	0.94	0.87
Tuffak (508 μm)	none	--	--	--	--
	UV	1001	0.99	1.00	0.98
	TC	1176	0.97	0.94	0.99
	HH	500	0.98	0.97	0.99
Mylar (178 μm)	none	--	--	--	--
	UV	1034	0.99	0.97	1.02
	TC	1032	0.97	0.98	0.96
	HH	--	--	--	--
"Teflon" FEP (127 μm)	none	--	--	--	--
	UV	1034	1.00	0.99	1.00
	TC	1133	0.98	0.98	0.98
	HH	500	0.97	0.98	0.95
Halar (127 μm)	none	--	--	--	--
	UV	490	0.98	0.98	0.97
	TC	500	0.96	0.96	0.97
	HH	500	1.00	0.99	1.02
Sun-Lite (635 μm)	none	--	--	--	--
	UV	490	1.23	1.37	1.09
	TC	500	0.88	0.93	0.83
	HH	500	1.11	1.10	1.11

(a) Transmittance ratios, designated R_T, R_{IR}, and R_{VIS}, are the ratios of normal light transmittance of exposed to that of unexposed samples over the ranges 300 to 1200 nm, 700 to 1200 nm, and 300 to 700 nm, respectively. Ratios were computed from measurements on single samples.

(b) UV, TC, and HH designate ultraviolet radiation (xenon lamp), thermal-cycling, and high-humidity exposures, respectively.

TABLE A-6. TRANSMITTANCE RATIOS(a) OF ENVIRONMENTALLY EXPOSED MATERIALS COMBINATIONS (SUBSYSTEMS) BASED ON SCOTCH-WELD 2216 B/A

Film/Adhesive Materials	Exposure Type(b)	Exposure Time, hr	R_T	R_{IR}	R_{VIS}
Scotch-Weld 2216 B/A Clear(c)	none	--			
ditto	U.V.	490	1.08	1.05	1.14
"	T.C.	500	1.00	0.99	1.02
"	H.H.	500	0.72	0.74	0.69
Sun-Lite/2216 (d)	none	--			
ditto	U.V.	490	1.45	1.50	1.38
"	T.C.	500	0.70	0.69	0.73
"	H.H.	500	0.54	0.65	0.41
Tuffak/2216	none	--			
ditto	U.V.	490	1.01	1.00	1.02
"	T.C.	500	0.98	0.99	0.97
"	H.H.	500	1.05	1.05	1.05
Tuffak/2216/Tuffak	none	--			
ditto	U.V.	1001	1.13	1.10	1.18
"	T.C.	1176	1.10	1.07	1.13
"	58C	1001	1.05	0.97	1.17
"	90C	1001	1.01	0.98	1.05
"Teflon" FEP/2216	none	--			
ditto	U.V.	490	0.85	0.85	0.86
"	T.C.	500	0.91	0.95	0.86
"	H.H.	500	1.01	1.04	0.96
"Teflon" FEP/2216/ "Teflon" FEP	none	--			
ditto	U.V.	1001	1.03	1.05	1.01
"	T.C.	1034	1.06	1.05	1.08
"	58C	1001	0.99	1.01	0.97
"	90C	1001	1.01	1.06	0.95
Korad A/2216	none	--			
ditto	U.V.	490	1.01	1.00	1.01
"	T.C.	500	0.87	0.88	0.85
"	H.H.	500	0.48	0.62	0.26
Korad A/2216/Korad A	none	--			
ditto	U.V.	1002	1.11	1.13	1.08
"	T.C.	1012	1.09	1.09	1.10
"	58C	1001			
"	90C	1001			

TABLE A-6. (Continued)

Film/Adhesive Materials	Exposure Type(b)	Exposure Time, hr	R_T	R_{IR}	R_{VIS}
Tedlar/2216	none	--			
ditto	U.V.	490	1.04	1.00	1.08
"	T.C.	500	0.94	0.96	0.90
"	H.H.	500	0.85	0.90	0.77
Tedlar/2216/Tedlar	none	--			
ditto	U.V.	1002	1.22	1.20	0.24
"	T.C.	1009	1.01	1.05	0.93
Halar/2216	none	--			
ditto	U.V.	490	1.02	1.04	0.98
"	T.C.	500	0.88	0.94	0.79
"	H.H.	500	0.96	0.98	0.94

(a) Transmittance ratios, designated R_T, R_{IR}, and R_{VIS}, are the ratios of normal light transmittance of exposed to that of unexposed samples over the ranges 300 to 1200 nm, 700 to 1200 nm, and 300 to 700 nm, respectively. Ratios were computed from measurements on single samples.

(b) UV, TC, and HH designate ultraviolet radiation (xenon lamp), thermal-cycling, and high-humidity exposures, respectively.

(c) Adhesive alone; no film or sheet material.

(d) Abbreviated form designates the Scotch-Weld 2216 B/A clear.

approximately 500 hours; the sandwiches were exposed for approximately 1000 hours. With the free film, only the high-humidity exposure seriously affected the transmittance.

Exposure of the film/adhesive combinations to high humidity gave rather curious results. The transmittance is of course affected by the presence of interfaces and absorption in the elements, and thus some variance in the results with the various films could be expected. However, the size of the variance is rather unexpected. Note that with Sun-Lite and Korad A the reduction in transmittance was large, while the reduction was comparatively much lower with Tuffak, "Teflon" FEP, Tedlar, and Halar. The explanation for such results is not clear; they may be due to the type of interaction between the adhesive and film.

Exposure to UV did not seriously affect the transmittance of any of the specimens, but it must be remembered that the UV exposure time is extremely short compared with 20 years. The thermal-cycle exposure likewise did not produce large effects. The large ratios for Sun-Lite after UV exposure are as yet unexplainable.

Acryloid B-7. Exposures to high humidity for 500 hours reduced the optical transmittance of free films of Acryloid B-7 significantly (Table A-7). When the adhesive was evaluated in combination with a number of film (sheet) materials, only combinations with Tedlar and Halar maintained good light transmittances after the same level of high-humidity exposure.

Generally speaking, all subsystems except those involving Sun-Lite sheet maintained stable transmittance after 500 hours of UV exposure. The Sun-Lite subsystem and, to a lesser extent, the Tuffak and "Teflon" FEP subsystems were adversely affected by thermal cyclings of 500 hours.

Silgrip SR-573. This silicon resin adhesive can be used directly as a thermoplastic laminating adhesive or can be cured with an amine or peroxide catalyst to provide increased thermal stability. Exposure to high humidity for 500 hours reduced the optical transmittance levels of Korad A, Halar, and Sun-Lite in combination with the uncatalyzed adhesive. The R_T values obtained for these subsystems were 0.54, 0.69, and 0.51, respectively (Table A-8).

TABLE A-7. TRANSMITTANCE RATIOS(a) OF ENVIRONMENTALLY EXPOSED MATERIALS COMBINATIONS (SUBSYSTEMS) BASED ON ACRYLOID B-7

Film/Adhesive Materials	Exposure Type(b)	Exposure Time, hr	R_T	R_{IR}	R_{VIS}
Acryloid B-7 Adhesive(c)	none	--			
ditto	U.V.	490	1.28	1.13	1.50
"	T.C.	500	1.09	1.01	1.22
"	H.H.	500	0.47	0.50	0.42
Korad A/Acryloid B-7	none	--			
ditto	U.V.	490	1.04	1.04	1.03
"	T.C.	500	1.04	1.06	1.01
"	H.H.	500	0.70	0.78	0.57
Halar/Acryloid B-7	none	--			
ditto	U.V.	490	0.95	0.97	0.94
"	T.C.	500	1.02	1.04	0.99
"	H.H.	500	1.02	1.01	1.02
Tedlar/Acryloid B-7	none	--			
ditto	U.V.	490	1.04	1.03	1.04
"	T.C.	500	0.98	0.99	0.97
"	H.H.	500	1.00	0.98	1.02
"Teflon" FEP/Acryloid B-7	none	--			
ditto	U.N.	490	0.94	0.93	0.96
"	T.C.	500	0.93	0.94	0.93
"	H.H.	500	0.69	0.76	0.61
Tuffak/Acryloid B-7	none	--			
ditto	U.N.	490	1.04	1.02	1.06
"	T.C.	500	0.86	0.79	0.93
"	H.H.	500	0.68	0.69	0.67
Sun-Lite/Acryloid B-7	none	--			
ditto	U.V.	490	0.84	0.87	0.80
"	T.C.	500	0.74	0.74	0.74
"	H.H.	500	0.31	0.31	0.31

(a) Transmittance ratios, designated R_T, R_{IR}, and R_{VIS}, are the ratios of normal light transmittance of exposed to that of unexposed samples over the ranges 300 to 1200 nm, 700 to 1200 nm, and 300 to 700 nm, respectively. Ratios were computed from measurements on single samples.

(b) UV, TC, and HH designate ultraviolet radiation (xenon lamp), thermal-cycling, and high-humidity exposures, respectively.

(c) Adhesive alone; no film or sheet material.

TABLE A-8. TRANSMITTANCE RATIOS(a) OF ENVIRONMENTALLY EXPOSED MATERIALS COMBINATIONS (SUBSYSTEMS) BASED ON SILGRIP SR-573

Film/Adhesive Materials Combinations	Exposure Type(b)	Exposure Time, hr	R_T	R_{IR}	R_{VIS}
Korad A/Silgrip SR-573	none	--			
ditto	U.V.	490	1.01	1.02	1.00
"	T.C.	500	1.18	1.14	1.27
"	H.H.	500	0.54	0.55	0.52
Korad A/Silgrip SR-573(c)/ Korad A	none	--			
ditto	U.V.	1002	0.98	0.94	1.07
"	T.C.	1012	0.83	0.75	0.99
Halar/Silgrip SR-573	none	--			
ditto	U.V.	490	1.04	1.09	0.98
"	T.C.	500	0.98	1.11	0.85
"	H.H.	500	0.69	0.78	0.58
Tedlar/Silgrip SR-573	none	--			
ditto	U.V.	490	1.10	1.10	1.11
"	T.C.	500	0.94	1.00	0.88
Tedlar/Silgrip SR-573(c)/ Tedlar	none	--			
ditto	U.V.	1002	0.95	1.06	0.75
"	T.C.	1009	1.63	1.64	1.61
"Teflon" FEP/Silgrip SR-573	none	--			
ditto	U.V.	490	0.94	1.00	0.87
"	T.C.	500	0.65	0.70	0.60
"	H.H.	500	0.87	0.88	0.86
"Teflon" FEP/Silgrip SR-573(d)/"Teflon" FEP	none	--			
ditto	U.V.	1046	1.79	1.74	1.87
"	T.C.	1133	1.49	1.52	1.45
"	58C	1001	1.38	1.45	1.30
"	90C	1001	1.04	1.00	1.09
Lexan-Silgrip SR-573	none	--			
ditto	U.V.	490	1.07	1.09	1.06
"	T.C.	500	1.14	1.15	1.13
"	H.H.	500	0.96	0.99	0.93

TABLE A-8. (Continued)

Film/Adhesive Materials Combinations	Exposure Type(b)	Exposure Time, hr	R_T	R_{IR}	R_{VIS}
Lexan-Silgrip SR-573(d)/ Lexan	none	--			
ditto	U.V.	1001	1.81	1.91	1.69
"	T.C.	1176	2.16	2.25	2.06
"	58C	1001	1.18	1.17	1.19
"	90C	1001	1.61	1.46	1.82
Mylar-Silgrip SR-573(d)/ Mylar	none	--			
ditto	U.V.	1034	1.09	1.11	1.06
"	T.C.	1032	2.55	2.19	3.46
"	58C	1001	2.80	2.49	3.57
"	90C	1001	2.61	2.27	3.46
Sun-Lite-Silgrip SR-573	none	--			
ditto	U.V.	490	1.47	1.64	1.30
"	T.C.	500	1.36	1.31	1.41
"	H.H.	500	0.51	0.57	0.45

(a) Transmittance ratios, designated R_T, R_{IR}, and R_{VIS}, are the ratios of normal light transmittance of exposed to that of unexposed samples over the ranges 300 to 1200 nm, 700 to 1200 nm, and 300 to 700 nm, respectively. Ratios were computed from measurements on single samples.

(b) UV, TC, and HH designate ultraviolet radiation (xenon lamp), thermal-cycling, and high-humidity exposures, respectively.

(c) Silgrip adhesive catalyzed with SRC-30, a peroxide catalyst.

(d) Silgrip adhesive catalyzed with 1.5 percent benzoyl peroxide.

It appears that under certain conditions Silgrip SR-573 continues to cure in the presence of catalyst when exposed to elevated temperatures or UV. This suggestion stems from the fact that R_T values much larger than one were obtained. It appears that only Korad A subsystems with SRC-30 catalyzed adhesive are adversely affected by thermal cycling. All subsystems tested held up well to UV exposures of up to 1000 hours.

Silastic 732 RTV. This elastomeric silicone is cured by atmospheric moisture. It was examined in combinations with films of "Teflon" FEP, Korad A, and Tedlar (Table A-9). Of the three, the "Teflon" FEP subsystem appeared to perform best over the different environmental exposures. However, decreases in transmittance were observed with these subsystems following thermal cycling, especially in the 300 to 700-nm range. The Tedlar subsystems generally were somewhat more affected than the "Teflon" FEP by the different environments. The Korad A subsystem generally performed more poorly, with the transmittances being reduced markedly by aging at both 58 and 90 C. Interestingly, the thermally cycled Korad A subsystem held up reasonably well.

RTV 118. This elastomeric silicon was evaluated only in subsystems with "Teflon" FEP and Mylar (Table A-10). The light transmittance of the "Teflon" FEP subsystem decreased substantially after thermal cycling (R_T = 0.76), while the Mylar subsystem decreased markedly after UV exposure (R = 0.60). Optical performance was unaffected or slightly improved for specimens of both subsystems exposed to the other environments.

Cavalon 3100S. This thermosetting acrylic, reacted with one of three activators (3300S, 3303S, or benzoyl peroxide), was evaluated in subsystems with "Teflon" FEP, Mylar, and Tedlar films. The adhesive system using activators 3300S and 3303S darkened in color on exposure to both UV and heat. No appreciable initial color was observed with benzoyl peroxide, but unreacted peroxide produced opaqueness.

TABLE A-9. TRANSMITTANCE RATIOS(a) OF ENVIRONMENTALLY EXPOSED MATERIALS COMBINATIONS (SUBSYSTEMS) BASED ON SILASTIC 732 RTV

Film/Adhesive Materials Combinations	Exposure Type(b)	Exposure Time, hr.	R_T	R_{IR}	R_{VIS}
"Teflon" FEP/Silastic 732 RTV/"Teflon" FEP	none	--			
ditto	U.V.	1046	1.13	1.12	1.14
"	T.C.	1133	0.75	0.80	0.69
"	58C	1001	1.20	1.18	1.23
"	90C	1001	1.07	1.04	1.11
Korad A/Silastic 732 RTV/ Korad A	none	--			
ditto	U.V.	1002	0.86	0.86	0.88
"	T.C.	1012	0.87	0.92	0.78
"	58C	1028	0.62	0.75	0.47
"	90C	1028	0.52	0.62	0.39
Tedlar/Silastic 732 RTV/ Tedlar	none	--			
ditto	U.V.	1002	0.88	0.93	0.83
"	T.C.	1009	0.88	0.97	0.76
"	58C	1028	0.85	0.91	0.77
"	90C	1028	0.88	0.92	0.82

(a) Transmittance ratios, designated R_T, R_{IR}, and R_{VIS}, are the ratios of normal light transmittance of exposed to that of unexposed samples over the ranges 300 to 1200 nm, 700 to 1200 nm, and 300 to 700 nm, respectively. Ratios were computed from measurements on single samples.

(b) UV, TC, and HH designate ultraviolet radiation (xenon lamp), thermal-cycling, and high-humidity exposures, respectively.

TABLE A-10. TRANSMITTANCE RATIOS[a] OF ENVIRONMENTALLY EXPOSED MATERIALS COMBINATIONS (SUBSYSTEMS) BASED ON RTV 118

Film /Adhesive Materials Combinations	Exposure Type[b]	Exposure Time, hr	R_T	R_{IR}	R_{VIS}
"Teflon" FEP/RTV 118/ "Teflon" FEP	none	--			
ditto	U.V.	1034	1.08	1.06	1.12
"	T.C.	1133	0.76	0.78	0.74
"	58C	1001	1.17	1.13	1.22
"	90C	1001	1.15	1.10	1.23
Mylar/RTV 118/Mylar	none	--			
ditto	U.V.	1034	0.60	0.63	0.54
"	T.C.	1133	1.23	1.31	1.13
"	58C	1001	1.01	1.14	0.85
"	90C	1001	1.11	1.28	0.90

(a) Transmittance ratios, designated R_T, R_{IR}, and R_{VIS}, are the ratios of normal light transmittance of exposed to that of unexposed samples over the ranges 300 to 1200 nm, 700 to 1200 nm, and 300 to 700 nm, respectively. Ratios were computed from measurements on single samples.

(b) UV, TC, and HH designate ultraviolet radiation (xenon lamp), thermal-cycling, and high-humidity exposures, respectively.

Optical transmittance data are presented in Table A-11. It should be noted that the Tedlar-Cavalon 3100S (5% benzoyl peroxide) subsystem improved in optical transmittance following exposure to UV, thermal cycling, or thermal aging. This is probably due to the fact that benzoyl peroxide cure is accelerated by either UV or heat and initial opaqueness of the adhesive film clears as the crosslinking reaction proceeds. If an appropriate combination of cure conditions could be established, it is possible that this adhesive may be practical for solar-cell-cover applications. Cure will be governed by the temperature and time required and the heat-distortion characteristics of the cover material used. As it is, this adhesive still may be of interest for encapsulant bonding applications where optical clarity is not important.

Scotch-Grip 4693, Scotch-Grip 4475, and Scotch-Weld 3520 B/A. These adhesives have been studied only in single subsystems in the current program (Table A-12). Scotch-Grip 4693 and Scotch-Grip 4475 normally are used to bond plastic materials. Scotch-Grip 4693 did not perform well in combination with Korad A. After thermal aging of 90 C for approximately 1000 hours, an R_T value of 0.47 was obtained. Scotch-Grip 4475 has a low softening point. In subsystems with Korad A, partial delamination and adhesive darkening occurred during both thermal cycling and 90 C thermal aging exposures.

Scotch-Weld 3520 B/A is a structural epoxy. Subsystems with Mylar maintained good optical transmittance after exposures to UV, thermal cycling, and thermal aging. Studies of other subsystems containing 3520 appear warranted.

TABLE A-11. TRANSMITTANCE RATIOS[(a)] OF ENVIRONMENTALLY EXPOSED MATERIALS COMBINATIONS (SUBSYSTEMS) BASED ON CAVALON 3100S

Film/Adhesive Materials Combinations	Exposure Type[(b)]	Exposure Time, hr	R_T	R_{IR}	R_{VIS}
"Teflon" FEP/Cavalon 3100S (Activator 3300S)/"Teflon" FEP	none	--			
ditto	U.V.	1001	0.95	1.01	0.82
"	T.C.	1133	0.87	1.09	0.41
"	58C	1001	1.46	1.43	1.53
"	90C	1001	0.98	1.18	0.57
Mylar/Cavalon 3100S (Activator 3300S)/Mylar	none	--			
ditto	U.V.	1034	1.03	1.13	0.87
"	T.C.	1032	1.10	0.87	1.02
"	58C	1001	1.16	1.25	1.02
"	90C	1001	0.84	0.98	0.62
Tedlar/Cavalon 3100S (Activator 3303S)/Tedlar	none				
ditto	U.V.	1002	0.61	0.67	0.51
"	T.C.	1009	1.07	1.12	0.99
"	58C	1028	0.69	0.81	0.53
"	90C	1028	0.61	0.78	0.39
Tedlar/Cavalon 3100S (5% benzoyl peroxide/ Tedlar	none	--			
ditto	U.V.	1002	1.76	1.89	1.57
"	T.C.	1009	1.47	1.54	1.37
"	58C	1028	1.67	1.93	1.29
"	90C	1028	2.19	1.93	2.58

(a) Transmittance ratios, designated R_T, R_{IR}, and R_{VIS}, are the ratios of normal light transmittance of exposed to that of unexposed samples over the ranges 300 to 1200 nm, 700 to 1200 nm, and 300 to 700 nm, respectively. Ratios were computed from measurements on single samples.

(b) UV, TC, and HH designate ultraviolet radiation (xenon lamp), thermal-cycling, and high-humidity exposures, respectively.

TABLE A-12. TRANSMITTANCE RATIOS[(a)] OF ENVIRONMENTALLY EXPOSED MATERIALS COMBINATIONS (SUBSYSTEMS) BASED ON SCOTCH-GRIP 4693, SCOTCH-GRIP 4475, AND SCOTCH-WELD 3520 B/A

Film/Adhesive Materials Combinations	Exposure Type(b)	Exposure Time, hr	R_T	R_{IR}	R_{VIS}
Korad A/Scotch-Grip 4693/ Korad A	none	--			
ditto	U.V.	1002	0.97	1.01	0.39
"	T.C.	1012	1.26	1.27	1.25
"	58C	1028	0.68	0.78	0.55
"	90C	1028	0.47	0.57	0.36
Korad A/Scotch-Grip 4475/ Korad A	none	--			
ditto	U.V.	1002	1.09	1.13	1.03
"	T.C.	1012	0.47	0.56	0.29
"	58C	1028	0.36	0.45	0.24
"	90C	1028	0.13	0.20	0.04
Mylar/Scotch-Weld 3520 B/A/Mylar	none	--			
ditto	U.V.	1034	1.14	1.17	1.11
"	T.C.	1032	1.26	1.33	1.16
"	58C	1001	1.22	1.34	1.06
"	90C	1001	1.23	1.35	1.06

(a) Transmittance ratios, designated R_T, R_{IR}, and R_{VIS}, are the ratios of normal light transmittance of exposed to that of unexposed samples over the ranges 300 to 1200 nm, 700 to 1200 nm, and 300 to 700 nm, respectively. Ratios were computed from measurements on single samples.

(b) UV, TC, and HH designate ultraviolet radiation (xenon lamp), thermal-cycling, and high-humidity exposures, respectively.

Normal Light Transmittance of Polymer Sheet/Film Materials

A critical evaluation parameter for cover materials for cells is, of course, the transmittance of the cover material itself and the combination of the material and the adhesive used to attach the cover to the cell. The normal transmittance was measured for selected sheet/films with and without adhesives in the wavelength range 300 to 1200 nm. Such data were taken before and after exposure to 2515 hours of ultraviolet radiation (approximately 1 sun). Table A-13 identifies the selected films and adhesives, and presents the measured results. See Tables A-2 and A-3 for further identification of the materials employed. Note that in Table A-13, the wavelength range of 300 to 1200 nm is covered only for the unexposed samples. Equipment troubles prevented obtaining comparable postexposure data in the 700 to 1200 nm range. Note also that the data represent only normal transmittance. Radiation scattered into the cell can also lead to light-generated current, so that the measured values represent a minimum flux that could actually be absorbed by the cell. Moreover, most of the flux not transmitted is represented by reflection at the surfaces, not by absorption. The thicknesses of the materials represent those easily obtainable from commercial sources. (Material development was not in the scope of this program.)

"Teflon" FEP

Compared with the rest of the materials evaluated, "Teflon" FEP alone gives rather high transmittance in both the long (700 to 1200 nm) and short (300 to 700 nm) wavelength ranges. Likewise, it exhibits low sensitivity to UV degradation, at least in the short wavelength range, as expected. (Results are given in Table A-13.)

In the long wavelength range, and before UV exposure, none of the adhesives reduced the transmittance substantially; the largest reduction was caused by the epoxy adhesive Scotch-Weld 2216. A substantial

TABLE A-13. NORMAL LIGHT TRANSMITTANCE OF POLYMER FILMS AND FILM/ADHESIVE LAMINATES BEFORE AND AFTER EXPOSURE TO 2515 HOURS OF ULTRAVIOLET RADIATION (1 Sun)

Film			Adhesive			Transmittance Before UV Exposure(a)				After UV Exposure, 3000 to 7000 A(b,c)		Change due to UV Exposure, 3000 to 7000 A	
	Thickness			Thickness		7000 to 12,000 A(c)		3000 to 7000 A(c)					
Type	µm	(mils)	Type	µm	(mils)	Percent	S.D.	Percent	S.D.	Percent	S.D.	Percent	S.D.
FEP Teflon	127	(5)	None	--	--	91.7	0.93	85.8	0.87	82.2	1.40	- 4.2	2.04
	127	(5)	Scotch-Weld 2216	76-102	(3-4)	82.2	1.28	62.7	1.96	59.8	0.81	- 4.6	2.57
	127	(5)	Silgrip-SR 573	25-51	(1-2)	90.4	1.98	83.8	2.12	73.1	4.31	-12.1	4.44
	127	(5)	Acryloid B-7	25-51	(1-2)	88.3	1.66	84.6	2.30	79.9	2.68	- 5.5	3.92
Tedlar	102	(4)	None	--	--	74.7	0.48	59.8	2.41	56.3	3.72	- 5.9	2.85
	102	(4)	Scotch-Weld 2216	76-102	(3-4)	76.6	1.15	50.4	1.24	54.6	3.26	+ 8.3	3.85
	102	(4)	Silgrip-SR 573	51	(2)	81.2	1.75	68.9	2.00	62.1	4.40	- 9.8	5.42
	102	(4)	Acryloid B-7	25-51	(1-2)	80.3	0.79	65.8	1.71	62.5	3.99	- 5.1	3.87
Halar	127	(5)	None	--	--	86.9	1.04	68.8	2.17	65.1	3.06	- 5.4	3.06
	127	(5)	Scotch-Weld 2216	76-102	(3-4)	82.3	0.77	56.8	0.71	56.9	1.76	+ 0.4	2.76
	127	(5)	Acryloid B-7	25-51	(1-2)	87.3	2.22	70.6	3.31	64.1	2.20	- 9.1	2.55
Sun-Lite(d)	635	(25)	None	--	--	16.7	3.70	14.1	2.48	11.8	4.00	-17.8	16.6
	635	(25)	Scotch-Weld 2216	76-127	(3-5)	18.9	5.04	13.6	3.19	12.1	5.08	-15.2	23.7
	635	(25)	Silgrip-SR 573	51-76	(2-3)	19.8	5.45	14.6	3.00	17.6	6.30	+18.1	23.4
Tuffak	508	(20)	None	--	--	89.2	0.71	81.5	1.64	66.3	0.61	-18.6	2.21
	508	(20)	Scotch-Weld 2216	76-102	(3-4)	82.2	2.65	62.5	1.94	58.5	2.41	- 6.4	5.14
	508	(20)	Acryloid B-7	25-76	(1-3)	82.9	2.64	78.0	4.38	63.1	0.86	-18.9	5.24
Acrylite	3175	(125)	None	--	--	89.3	0.67	75.9	1.39	70.0	1.04	- 7.8	1.46
	3175	(125)	Scotch-Weld 2216	102-127	(4-5)	81.6	1.53	62.1	2.28	63.1	2.09	+ 0.1	2.86
	3175	(125)	Silgrip-SR 573	51-76	(2-3)	86.5	2.07	68.9	1.10	68.4	1.79	- 0.6	3.85
	3175	(125)	Acryloid B-7	25-51	(1-2)	85.4	1.87	73.8	0.35	69.6	1.57	- 5.7	1.98
Flexigard	102	(4)	None	--	--	86.5	0.82	62.9	1.80	58.1	2.00	- 7.6	1.36
	102	(4)	Silgrip-SR 573	51-76	(2-3)	87.4	1.27	58.5	1.06	57.1	1.68	- 2.4	2.02

(a) Measurements made with Cary 14 spectrometer.

(b) Measurements made with Cary 118 spectrometer; values for Tedlar, Halar, and Sun-Lite corrected to Cary 14 data.

(c) Average of five samples.

(d) Low normal transmittance due to light scattering by glass fibers in Sun-Lite.

reduction did occur for the epoxy adhesive in the low wavelength range. This result occurred for all sheet/film candidates. That is, the epoxy exhibits considerable absorption in the 300 to 700 nm range.

The major effect of UV exposure in the short wavelength range was a 12 percent reduction in transmittance owing to the presence of the silicone adhesive Silgrip SR573.

Tedlar

The halocarbon, Tedlar, shows rather poor normal transmittance compared with that of the other sheet/films (with the exception of Sun-Lite which has lower normal transmittance), particularly in the 300 to 700 nm range. Exposure to 2500 hours of UV radiation reduced the transmittance in this range by about 6 percent.

Without exception the transmittance of Tedlar is increased with the application of an adhesive. This result clearly stems from improvement in optical coupling among the Tedlar/adhesive interfaces. Again, the application of Scotch-Weld 2216 adhesive leads to a decrease in transmittance in the short wavelength range. Exposure to UV radiation leads to a decrease in transmittance ranging from about 5 to 10 percent, except for Scotch-Weld 2216. Here an increase of 8 percent was manifest. Whether curing by UV was a cause for the increase is unknown but possible.

Halar

For Halar, another halocarbon, the transmittance in the long wavelength range is rather high, 87 percent, but rather low in the short wavelength range, 69 percent. Typical of the other halocarbons, the reduction due to UV exposure was about 5 percent.

Application of Scotch-Weld 2216 reduced the transmittance in both ranges, but, again, largely in the 300 to 700 nm range. Acryloid B-7, an acrylic, increased the transmittance slightly. As with Tedlar, UV exposure increased (slightly) the transmittance with Scotch-Weld 2216/Halar.

Sun-Lite

The normal transmittance of Sun-Lite, a glass-reinforced polyester, id very low for the total wavelength range examined, primarily because of light scattering by the glass fibers. As expected, the addition of adhesive layers does not affect the transmittance much. UV exposure of Sun-Lite alone reduces transmittance substantially; comparable results are found with the Scotch-Weld/Sun-Lite laminate. Silgrip caused an increase, but as in all of these samples, the variability is large: note (in Table A-13) the large values of the standard deviation. As discussed later, Sun-Lite does not decrease the light flux reaching a solar cell as much as indicated by the relatively low normal transmittance.

Tuffak

Tuffak, a polycarbonate, exhibits a comparatively high transmittance over the whole wavelength range. However, exposure to UV reduced the transmittance by approximately 19 percent. A comparable decrease was found with the Tuffak/Acryloid B-7 laminate under UV exposure. The reduction in the short wavelength range again shows up when Scotch-Weld 2216 is used.

Acrylite

Rather high transmittance is exhibited by Acrylite, an acrylic, in both wavelength ranges, especially the high range. About 8 percent reduction was found upon UV exposure. Changes in transmittance in the 300 to 700 nm range are small for this material with all adhesives examined. Note (Table A-13) the rather large thickness of the Acrylite employed.

Flexigard

Flexigard is a commercial laminate structure, probably an acrylic/polyester laminate. The film alone showed rather high transmittance in the 700 to 1200 nm range, and a rather low value in the 300 to 700 nm range. Direct comparison with the other polyester, Sun-Lite, is not fruitful because of the light-scattering effect in the latter material. An 8 percent reduction in transmittance was found which is attributable to UV exposure. With Silgrip SR573 adhesive, the reduction resulting from UV exposure was small.

Moisture-Barrier-Property Studies

Barrier Performance of Film and Sheet Materials. Water-vapor-transmission-rate measurements were made in duplicate for a number of film and sheet cover candidates using the technique described earlier. Measurements were made on unexposed samples and on samples subjected to UV radiation using a carbon arc and to temperature cycling. The data are summarized in Table A-14. These were the only carbon-arc UV exposures used in this program and were employed on the basis of equipment availability; all others were xenon-lamp exposures.

Use of the carbon-arc UV source, which contains sizable amounts of radiation in the 240 to 300-nm wavelength range, which is not present in xenon lamp exposures, had a detrimental effect on most of the materials that were exposed. Only the "Teflon" FEP and Halar films were unaffected, at least visually, by 500-hour exposures. The acrylic materials (Acrylite, Korad A, and Flexigard) yellowed and exhibited surface crazing. The polycarbonates (Lexan sheet and Tuffak), the Sun-Lite sheet, and Tedlar also yellowed. Tedlar also embrittled. Surface blooming of either an additive or degradation product of the Mylar, occurred and the bulk film became very cloudy.

Examination of the data of Table A-14 indicates, interestingly, that the carbon-arc exposure, although having pronounced effects on surface characteristics, generally did not markedly affect barrier characteristics. Only the Halar and, to a lesser extent, the Mylar and Korad A were adversely affected by carbon-arc exposures to 500 hours. The Sun-Lite sample appeared

TABLE A-14. WATER-VAPOR-BARRIER PROPERTIES(a) OF POLYMERIC FILM AND SHEET COVER CANDIDATES BEFORE AND AFTER ENVIRONMENTAL EXPOSURES

Material	Thickness, μm (mils)	WVTR, g/24 hr/m^2 at 23 C and 95% R.H. (at Indicated Thickness)		
		Unexposed	UV-50(b), 45-58 C	TC-500(b)
Halar	127 (5)	1.07	1.51	2.88
"Teflon" FEP	127 (5)	1.13	1.08	1.21
Lexan	3175 (125)	1.29	1.27	1.31
Acrylite	3175 (125)	2.16	1.67	1.53
Mylar	178 (7)	2.25	2.67	2.17
Sun-Lite	635 (25)	4.37	2.84	3.85
Tedlar	102 (4)	4.70	--	3.85
Tuffak	508 (20)	5.14	5.29	4.86
Flexigard	127 (5)	5.31	5.57	5.34
Korad A	152 (6)	22.81	26.10	23.51

(a) Measurements were performed in duplicate.

(b) UV-500 and TC-500 designate exposures of 500 hours to UV radiation and to thermal cycling, respectively. For these samples a carbon-arc source of UV was used. In all other studies described in this report the UV source was a xenon lamp.

to be improved by the UV exposure, but this may be the result of sample-to-sample variations in filler content. More work is needed with this material.

Thermal-cycling exposures of 500 hours appeared to have little effect on most of the cover candidates. Again, the barrier characteristics of the Halar film were most affected.

Barrier Performance of Film- and Sheet-to-Adhesive Subsystems. Results of WVTR measurements of subsystems of "Teflon" FEP and Mylar with selected adhesives are summarized in Tables A-15 and A-16, respectively. For studies of "Teflon" FEP, the adhesively bonded pouch was used as a measure of the effectiveness of the adhesive as a moisture-vapor barrier. The best overall performance with "Teflon" FEP was achieved with Scotch-Weld 2216 B/A, Silgrip SR-573, Silgrip SR-574, and Acryloid B-7. However, some bubble formation was observed after temperature cycling with the two Silgrip adhesives.

The Cavalon 3100S (3300S) darkened considerably during both UV and temperature-cycling exposures, and performed somewhat more poorly than other subsystems. However, it was markedly affected by UV. The greatest average increase in WVTR was recorded with the moisture-cured silicone adhesives (RTV-118 and Silastic 732 RTV).

Evaluation of the data obtained with the various Mylar subsystems (Table A-16) indicates that, with the exception of the Silastic 732 RTV subsystem, none performed satisfactorily through the UV and temperature-cycling exposures. The Silgrip SR-573, Scotch-Weld 2216 B/A, Acryloid B-7, and Cavalon 3100 S adhesives all failed, primarily because of loss of adhesion. Only the Silastic 732 RTV performed well. It is possible that the "pouch" technique may not be satisfactory for determining barrier properties with high-modulus (stiff) film materials. The "bowing" effect created by the desiccant inside the pouch puts a considerable stress on the adhesive bonds. The Silastic 732 RTV being an elastomeric, apparently was better able to withstand these stresses.

TABLE A-15. WATER-VAPOR-BARRIER PROPERTIES[(a)] OF "TEFLON" FEP (127-μm THICK) FILMS BONDED WITH VARIOUS ADHESIVES BEFORE AND AFTER ENVIRONMENTAL EXPOSURE

	WVTR, g/24 hr/m^2 at 23 C, 95% R.H.		
Adhesive	Unexposed	UV-1000[(b)], 45-58 C	TC-1000[(b)]
Silgrip SR-573	0.204	0.258	0.237
Silgrip SR-574	0.250	0.217	0.244
RTV-118	0.224	0.255	0.344
Silastic 732 RTV	0.282	0.401	0.237
Scotch-Weld 2216 B/A	0.201	0.221	0.189
Acryloid B-7	0.273	0.203	0.181
Cavalon 3100S	0.167	0.240	0.216
None (heat sealed)	0.154	0.151	0.162

(a) Measurements were performed in duplicate.

(b) UV-1000 and TC-1000 designate exposures of 1000 hours to UV radiation (xenon lamp) and to thermal cycling, respectively.

TABLE A-16. WATER-VAPOR-BARRIER PROPERTIES[a] OF WEATHERABLE MYLAR (178-μm THICK) FILMS BONDED WITH VARIOUS ADHESIVES BEFORE AND AFTER ENVIRONMENTAL EXPOSURE

	WVTR, g/24 hr/m^2 at 23 C, 95% R.H.		
Adhesive	Unexposed	UV-1000[b], 45-58 C	TC-1000[b]
Silgrip SR-573	1.85	2.25	1.90[e]
Silastic 732 RTV	1.77	1.80	1.81
Scotch-Weld 2216 B/A	1.80	1.90	1.85[e]
Acryloid B-7	2.21	(c)	(c)
Cavalon 3100S	1.74	(d)	(d)

(a) Measurements were performed in duplicate.

(b) UV-1000 and TC-1000 designate exposures for 1000 hours to UV radiation (xenon lamp) and to thermal cycling.

(c) Some loss of adhesion was noted.

(d) Some loss of adhesion and darkening were noted.

(e) Single determination. Loss of adhesion of one sample occurred.

The results from studies of the barrier properties of subsystems of the sheet material cover candidates (Acrylite, Lexan, and Sun-Lite) are presented in Table A-17. Bubble formation occurred during laminations involving the thermoplastic acrylic adhesive (Acryloid B-7) and the Acrylite and Lexan sheet materials. It is likely that solvent in the adhesive formulation is being absorbed on the surface of these sheet materials and is released to produce bubbles when heat and pressure are applied. A number of failures (high rate of water-vapor transmission depleting the desiccant) were observed in the unexposed and thermal-cycled samples. No bubbles were noted with the Sun-Lite/Acryloid B-7 subsystems.

The Acryloid B-7 and Scotch-Grip 4693 perform better than Silgrip SR-573 with the Sun-Lite material. With the Acrylite and Lexan materials, the Scotch-Grip 4693 and Silgrip SR-573 are superior to Acryloid B-7.

Bond Strength Studies

Bond tests of film/adhesive and sheet/adhesive subsystems combinations were carried out to identify, from among the adhesives candidates identified in earlier work, those adhesives that are most likely to provide encapsulation systems that are resistant to delamination and other forms of failure during exposures to various hostile weathering environments. In this work lap-shear measurements have been used to index bond strengths. Lap shear was selected primarily on the basis of its relative ease of measurement, since the screening task has dealt with very large numbers of materials combinations. It is recognized, of course, that other methods of evaluating adhesive bonds using various types of stress, e.g., tensile, shear, etc, could also have been used to provide information in characterizing the bond, as noted in the experimental procedures section.

Throughout the discussion that follows, summary tables of the bond strength data are provided; these have been condensed from original detailed tabulations of the data. Bonding characteristics of six candidate film covers with selected adhesives were studied before and after environmental exposures. All samples were preconditioned by storage at 23 C and 50 percent relative humidity for 12 to 24 hours prior to testing. Environmental exposures included UV radiation, thermal cycling, and isothermal aging at 45, 58, and 90 C.

TABLE A-17. WATER-VAPOR-BARRIER CHARACTERISTICS(a) OF VARIOUS SHEET/ADHESIVE COMBINATIONS

Sheet Material	Adhesive	Unexposed	WVTR, g/24 hr/m^2 at 23 C, 95% R.H. UV-500	UV-1000	TC-500	TC-1000
Acrylite	Acryloid B-7	(b)	3.31	4.95(b)	6.35(c)	9.31
Lexan	Acryloid B-7	6.01(c)	1.81	1.97	(b)	(b)
Sun-Lite	Acryloid B-7	3.90	4.63	4.44(b)	4.89	2.93
Acrylite	Scotch-Grip 4693	5.55	2.83	(d)	3.27	3.26
Lexan	Scotch-Grip 4693	1.34	2.62	1.63	3.36	3.38
Sun-Lite	Scotch-Grip 4693	3.53	3.76	4.09(b)	5.16	4.96
Acrylite	Silgrip SR 573	3.44	3.03	2.75	3.52	(b)
Lexan	Silgrip SR 573	1.41	1.82	1.66	3.64	4.41
Sun-Lite	Silgrip SR 573	3.76	5.53	3.96	6.06	5.25

(a) Measurements were performed in duplicate.

(b) These samples failed.

(c) One of two samples on test failed.

(d) Bubbles formed in the adhesive after lamination.

Bonding to "Teflon" FEP Film. The relative strengths of adhesive bonds formed between "Teflon" FEP (127-µm thick) and five adhesives candidates were determined before and after environmental exposures using the lap-shear method. These data are summarized in Table A-18. They reveal that the predominant failure mode is film elongation outside the bond area at a load of approximately 6.2 x $10^4 N/m^2$ (9 psi), independent of exposure. Only the Silastic 732 RTV system produced any failures of the cohesive or adhesive type at a load less than that required for film elongation. Thus, generally speaking, the yield strength of the "Teflon" FEP film defines the load limit that can be applied to these subsystems if tensile and shear forces of the type present in the lap-shear test are representative of those that might be experienced by the encapsulant. Cavalon 3100S with Activator 3300S provides a bond strength in excess of film yield strength, but discolors when exposed to UV or heat. The limitations of Silastic 732 RTV with "Teflon" FEP were mentioned above. Thus, the Scotch-Weld 2216 B/A appears to be the best structural adhesive for this application, while both RTV 118 and Silgrip SR-573 (cured with 1.5% benzoyl peroxide) are satisfactory nonstructural adhesives. The RTV 118 would be most applicable if an elastomeric is desirable, while the Silgrip material would be favored for applications in which a less flexible, more heat-resistant adhesive is required. An important point here is that thermal cyclings of 1000 hours (250 cycles) produced no delaminations. This appears to be an inherent advantage of the use of the film (sheet) laminate encapsulation design with low-modulus (flexible) adhesives.

Bonding to Mylar Film. The five adhesives evaluated with 178-µm-thick Mylar all failed adhesively or cohesively. This was not unexpected in the unexposed samples since the data suggest a tensile strength in excess of 69.0 x 10^4 N/m^2 (100 psi) for the Mylar. However, it also was found that bond strengths generally were not markedly affected by the different types of environmental exposures (Table A-19).

As in other studies, it was observed that the Cavalon 3100S with Activator 3303S discolors when exposed to UV or heat. With the Scotch-Grip 4693 and Silgraip SR-573 (1.5% benzoyl peroxide), bond strengths appear to

TABLE A-18. "TEFLON" FEP FILM LAMINATE BOND EVALUATIONS

Adhesive	Lap Shear[(a)], 10^4 N/m^2 Unexposed	1000-Hour UV Exposure	1000-Hour Temperature Cycling	1000-Hour Thermal Aging 58 C	1000-Hour Thermal Aging 90 C	Predominant Failure Mode
RTV 118	5.4-6.1	6.3	5.9-6.4	6.2	6.3	Film elongation[(b)]
Silastic 732 RTV	5.5-6.2	5.3-6.5	4.6-6.5	6.1	6.3	ditto
Silgrip SR-573 (1.5% benzoyl peroxide)	6.1-6.2	6.3-6.4	6.2-6.4	6.0	6.2	"
Scotch-Weld 2216B/A	6.1-6.2	6.2-6.4	6.2-6.5	6.2	6.3	"
Cavalon 3100S (Activator 3300S)	3.3-6.1	6.2-6.3	6.2-6.3	6.2	6.1	"

(a) The calculations of bond strength are based on a bond area of 6.45 cm^2 (1 in.^{2}); 1 N/m^2 = 1.45 x 10^{-4} psi.

(b) "Film elongation" indicates that the specimen elongated but not in the bond area. The bond strength indicated, in such cases, is equal to or smaller than the actual bond strength.

TABLE A-19. MYLAR FILM LAMINATE BOND EVALUATIONS

	Lap Shear, 10^4 N/m^2(a)					
Adhesive	Unexposed	1000-Hour UV Exposure	1000-Hour Temperature Cycling	1000-Hour Thermal Aging 58 C	1000-Hour Thermal Aging 90 C	Predominant Failure Mode
Scotch-Grip 4693	47-60	65-70	69-71	76	59	Cohesive failure
RTV-118	25-45	47-60	24-58	51	63	Adhesive/ cohesive failure
Scotch-Weld 3520 B/a	46-48	52-55	45-50	58	56	Adhesive failure
Cavalon 3100S (Activator 3303S)	48-68	41-67	57-68	48	54	Adhesive failure
Silgrip SR-573 (1.5% benzoyl peroxide)	37-46	48-66	70-76	68	70	Cohesive failure

(a) 1 N/m^2 = 1.45×10^{-4} psi.

improve after exposure to UV or thermal cycling. Thermal treatment improves bond strength, probably through further curing, as can be seen from the data for the thermally aged samples. An exception is the behavior of Scotch-Grip 4693 at 90 C. The three other adhesives seem to be completely unaffected by environmental exposures. Here again it should be noted that the low moduli of the adhesives used in bonding Mylar prevented any delamination during thermal cycling (-40 C to 90 C) after 1000 hours of exposure.

Bonding to Tuffak Film. Bond test data obtained with Tuffak film (508-μm-thick) are presented in Table A-20. This film has a yield strength of approximately 103 to 110 x 10^4 N/m^2 (150 to 160 psi). Two adhesives, the Scotch-Weld 2216 B/A and Structural Adhesive 3532 B/A, provided laminate bond strengths in excess of this yield strength; 3532 B/A is an unclear urethane and was the only adhesive from this class of materials that was available at the time of this work. It should provide a measure of the general effectiveness of this class of adhesives with Tuffak.

The adhesive next in order of effectiveness was the Silgrip SR-573 (1.5% benzoyl peroxide). Bond strengths with this material were somewhat lower than film yield strength, but were not adversely affected by UV or thermal cycling. To the contrary, as noted in other studies with this adhesive, some further curing occurred and bond strength increased. The improvement with curing was particularly noticeable with thermal controls, with bond strengths in the 62 to 76 x 10^4 N/m^2 (90 to 110 psi) range being obtained.

The two plastic adhesives (Scotch-Grip 4475 and Scotch-Grip 4693) were affected by one or more of the exposures. Scotch-Grip 4475 again was affected by the 90 C thermal cycling exposure and by thermal aging at 90 C. In both exposures, specimen delamination occurred. Bond strengths with Scotch-Grip 4693 were reduced markedly by UV exposure after as few as 500 hours of exposure. The bonds maintained strengths on thermal aging at 58 C. However, this adhesive subsystem is affected by higher temperatures; delamination occurred during aging at 90 C.

Both 4475 and 4693 are supplied with solvent, as are most "contact" adhesives. It is likely that the solvents in these adhesives caused film crazing; delamination may have resulted from built-in stresses produced

TABLE A-20. TUFFAK FILM LAMINATE BOND EVALUATIONS

	Lap-Shear, 10^4 N/m^2(a)					
Adhesive	Unexposed	1000-Hour UV Exposure	1000-Hour Temperature Cycling	1000-Hour Thermal Aging		Predominant Failure mode
				58 C	90 C	
Scotch-Grip 4475	105-113	105-109	delamination	112	delamination	Film elongation(b)
Scotch-Grip 4693	47-83	0-8.6	37-83	56	delamination	Cohesive failure
Structural Adhesive 3532 B/A	105-119	97-112	110-113	111	114	Film elongation
Silgrip SR-573, 1.5% benzoyl peroxide	13-37	19-39	18-51	73	62	Cohesive failure
Scotch-Weld 2216 B/A	104-113	113	110-117	112	124	Film elongation

(a) 1 N/m^2 = 1.45 x 10^{-4} psi.

(b) "Film elongation" indicates that the specimen elongated but not in the bond area. The bond strength indicated, in such cases, is equal to or smaller than the actual bond strength.

by solvent effects. Note that here again the moderately low-modulus structural adhesives (2216 B/A and 3532 B/A) and the nonstructural Silgrip SR-573 did not delaminate during the thermal cycling study.

Bonding to Korad A. Bond strength measurements were made for five adhesives with Korad A (152 μm thick). These are listed in Table A-21. With all but Silastic 732 RTV, failure occurred by film elongation, indicating that the adhesive is as strong or stronger than the yield value of the film, which is approximately 17 to 19 x 10^4 N/m^2 (25 to 27 psi).

Exposure to UV and thermal cycling had no effect on Silastic 743 RTV, Silgrip SR-573, or Scotch-Weld 2216 B/A. Bond strengths with Scotch-Grip 4693 and Scotch-Grip 4475 were not affected by thermal cycling, but some slight deterioration upon UV exposure was observed after 1000 hours. Interestingly, no delaminations occurred with either during 90 C thermal aging. It appears that except for 732 RTV all adhesives tested are satisfactory for bonding Korad A.

Bonding to Tedlar Film. The yield strength of 102-μm-thick Tedlar film appears to be approximately 15 x 10^4 N/m^2 (22 psi). In the study of the bonding of Tedlar with five selected adhesives, film elongation was the predominant failure mode, both before and after environmental exposures (Table A-22). Only unexposed specimens containing Cavalon 3100S (5% benzoyl peroxide) failed adhesively or cohesively (that is, at a strength much below 15 x 10^4 N/m^2). Even with this material, however, further curing appeared to occur during exposures and bond strengths improved. Some "wetting-out" problems were observed on the unexposed Silgrip SR-573 with SRC-30. Scotch-Weld 2216 B/A and Silastic 732 RTV appear to be the best overall performers with Tedlar.

Bonding to Flexigard Film. Four of the five adhesives selected for evaluations on Flexigard film (127-μm thick) were strong enough to cause film elongation for the majority of the specimens evaluated (Table A-23). Film yield strength appears to be 23 to 26 x 10^4 N/m^2 (33 to 37 psi). Film bond strengths with Silastic 732 RTV were somewhat affected by thermal aging

TABLE A-21. KORAD A FILM LAMINATE BOND EVALUATIONS

	Lap-Shear, 10^4 N/m^2 (a)					
Adhesive	Unexposed	1000-Hour UV Exposure	1000-Hour Temperature Cycling	1000-Hour Thermal Aging 58 C	1000-Hour Thermal Aging 90 C	Predominant Failure Mode
Silastic 732 RTV	10-13	12-14	10-11	13	11	Adhesive failure
Silgrip SR-573, SRC-30	16-18	19	18	18	18	Film elongation(b)
Scotch-Weld 2216 B/A	16-18	17-19	17-18	18	17	Film elongation
Scotch-Grip 4693	17-18	15-17	18	18	17	Film elongation
Scotch-Grip 4475	16-18	11-14	18	18	18	Film elongation

(a) 1 N/m^2 = 1.45 x 10^{-4} psi.

(b) "Film elongation" indicates that the specimen elongated but not in the bond area. The bond strength indicated, in such cases, is equal to or smaller than the actual bond strength.

TABLE A-22. TEDLAR FILM LAMINATE BOND EVALUATIONS

Adhesive	Lap-Shear, 10^4 N/m^2 (a)					Predominant Failure Mode
	Unexposed	1000-Hour UV Exposure	1000-Hour Temperature Cycling	1000-Hour Thermal Aging		
				58 C	90 C	
Silastic 732 RTV	13-14	14	14-15	14	15	Film elong.(b)
Scotch-Weld 2216 B/A	13-14	14-15	14-15	15	15	Film elongtion
Silgrip SR-573, SRC-30	11-14	14	14-15	14	15	Film elongation
Cavalon 3100S (Activator 3303S)	13-14	14	15	14	15	Film elongation
Cavalon 3100S, 5% benzoyl peroxide	5-10	10-14	14	14	14	Film elongation

(a) 1 N/m^2 = 1.45 x 10^{-4} psi.

(b) "Film elongation" indicates that the specimen elongated but not in the bond area. The bond strength indicated, in such cases, is equal to or smaller than the actual bond strength.

TABLE A-23. FLEXIGARD FILM LAMINATE BOND EVALUATIONS

	Lap Shear, 10^4 N/m^2 (a)					Predominant Failure Mode
				1000-Hour Thermal Aging		
Adhesive	Unexposed	1000-Hour UV Exposure	1000-Hour Temperature Cycling	58 C	90 C	
Scotch-Weld 2216B/A	23-27	25-28	23-25	34	25	Film elongation (b)
Cavalon 3100S (1.5% benzoyl peroxide)	10-11	24-31	24-25	24	26	Film elongation (b)
Scotch-Grip 4693	20-23	25-27	23-24	25	26	Film elongation
Silgrip SR-573, SRC-30	24	24-26	23	24	24	Film elongation
Silastic 732 RTV	18-23	15-21	14-19	16	16	Adhesive failure

(a) 1 N/m^2 = 1.45 x 10^{-4} psi.

(b) "Film elongation" indicates that the specimen elongated but not in the bond area. The bond strength indicated, in such cases, is equal to or smaller than the actual bond strength.

and thermal cycling, and adhesive-type failures predominated. Cavalon 3100S (1.5% benzoyl peroxide) again failed either cohesively or adhesively before exposure and improved with exposures, apparently as further curing took place. The other three adhesives (Scotch-Weld 2216 B/A, Scotch-Grip 4693, and Silgrip SR-573 with SRC-30) were unaffected by the several types of exposure.

A shortcoming with Flexigard appears to be the care required in applying the various adhesives to optimize performance. With the exception of Scotch-Weld 2216 B/A, an adhesive recommended by the manufacturer of Flexigard, film/adhesive composites generally are difficult to prepare with good optical clarity. Work to define application techniques will be necessary with a majority of the adhesives candidates.

DATA ANALYSIS

In earlier work(A-2, A-3), a number of polymeric materials identified as potential candidates for use as components of encapsulation systems for solar arrays. Potentially low-cost encapsulation designs of the film (sheet) laminate and conformally top-coated types were emphasized and principal uses of polymerics in such designs are as optical-quality adhesives, films (sheets), conformal coatings, and pottants.

In the current BCL experimental program, certain key properties of these materials types for specific end-use applications have been identified that can be associated with the major types of systems failure (loss of optical clarity, moisture transport and corrosion, and delamination) and, thus, used to index systems performance. These are the optical, moisture barrier, and strength properties of the individual and composite materials, and the effects of weathering environments (UV irradiation thermal aging, thermal cycling, high humidity, etc.) on these properties.

Throughout the discussion that follows, materials are rated principally by three designations: (+), (0), or (-), signifying levels of performance as positive, average, and negative. Occasionally, use is made of the symbol (++) to differentiate among levels of positive performance.

Table A-24 is a recapitulation of the study of the optical transmittance stability of certain of the film and sheet cover candidates during environmental exposures. From the earlier study(A-2), each of these materials was identified as having "as prepared" optical characteristics that are consistent with those required for the array cover. During exposures to each of the three environments, all six materials maintained good optical properties and were given positive ratings. The fact that all rated positively is not surprising since these materials were "handpicked" for this study on the basis of a detailed literature investigation.

TABLE A-24

RATINGS OF FILM AND SHEET MATERIALS ON BASIS OF OPTICAL TRANSMITTANCE STABILITY DURING ENVIRONMENTAL EXPOSURES

Material	R-Value(a) UV-500, 45-58 C	 TC-500	 HH-500, 38 C, 100% R.H.	Rating
Sun-Lite	1.23	0.88	1.11	+
Lexan	0.98	0.96	0.99	+
FEP	0.94(b)	0.88(b)	0.88(b)	+
Korad A	0.99	0.95	0.91	+
Tedlar	1.00	0.94	0.96	+
Halar	0.98	0.96	1.00	+

(a) $R = T_{exp}/T_{unexp}$.

(b) May be low - unexposed value appeared high (90.4%).

Continuing with the film (sheet) cover candidates, moisture barrier property measurements (Table A-14) suggest that all but Korad A are initially good and, for the most part, maintain good barrier properties after different types of environmental exposures. Perhaps even more meaningful, however, are the abilities of the materials in combination with other polymerics, principally adhesives, to provide protection from water vapor (and other contaminants) to the encapsulated photovoltalics. This latter property may not be directly relatable to barrier properties since the interfaces associated with cover/adhesive (or pottant) and adhesive (or pottant)/cell also influence cell protection. These interfacial effects have been considered in the high humidity exposure studies of encapsulated cells. These data are analyzed later in the discussion.

Also of major importance to the identification of encapsulant materials candidates are the strength of bonds formed between polymerics, e.g., between film (sheet) and adhesive in a laminate design, and between polymer and cell. Unfortunately, time has not permitted experimental studies of the latter type of bonding. Only film-to-adhesive bonding has been considered here and, as described in the text of the "Experimental" section, several adhesives, namely, Scotch-Weld 2216 B/A, Silgrip SR-573, and Acryloid B-7, have provided the best overall performances. These, consequently, have been utilized most frequently in other evaluations of materials subsystems.

In terms of the adhesive-to-cell or conformal coating-to-cell bonding, one comment again seems very pertinent. That is, with but a few exceptions, delaminations did not occur and that is very likely attributable to the fact that low-modulus materials in contact with the cell have been advocated and emphasize in this program as appropriate to the film (sheet)-laminate and conformally top-coated encapsulation designs.

In Table A-25, cover laminates, i.e., combinations of film (sheet) and adhesive, are rated on the basis of optical performance during exposures to the several environment types. The study was limited to the three adhesives, Scotch-Weld 2216 B/A, Silgrip SR-573, and Acryloid B-7, that were judged to perform best in the bond strength studies. This does not, however, rule out certain of the other adhesives used in this investigation. Limitations in terms of required work volume and time necessitated a preliminary selection from among the adhesives candidates.

As can be seen from an examination of the data of Table A-25, the performance differences among the various materials combinations are discerned primarily from their performances during 500 hours of high humidity exposure at 38 C. With a few exceptions, all systems performed well during UV irradiation and thermal cycling. Again, it is noteworthy that only rarely did delaminations occur with the selected materials combinations during the rather severe thermal cycling exposure, pointing out the very real design advantage of the use of a low-modulus adhesive in the dual role of adhesive and cell pottant.

In rating the materials combination of Table A-24, there does not appear to be any real pattern to the performance. As expected, the best performing adhesive is specific to a particular film (sheet) material. The materials ratings may be too severe in that, as pointed out above, most of the poor performances were associated with a very severe high humidity exposure. Nevertheless, neutral and negative ratings were given to those materials that had

R_T-values < 0.85 and < 0.60, respectively.

The performances of a number of adhesives and conformal coatings as protective encapsulants for photovoltaics during high humidity exposure are rated in Table A-26. The rating was made on the basis of a composite of changes in the four identified electrical characteristics of the encapsulated cells. Interestingly, in judging adhesives performance (Table A-26), two of the materials rated best in bond strength testing (Scotch-Weld 2216 B/A and Acryloid B-7) were among the best performers in this

TABLE A-25

RATINGS OF COVER LAMINATES ON BASIS OF OPTICAL TRANSMITTANCE STABILITY DURING ENVIRONMENTAL EXPOSURES

Material	R-Value			Rating
	UV-500, 45-58 C	TC-500	HH-500, 38 C, 100% R.H.	
Lexan - 2216	1.01	0.98	1.05	+
Lexan - 573	1.07	1.14	0.96	+
FEP - 2216	0.86	0.91	1.01	+
Tedlar - B-7	1.04	0.98	1.00	+
Halar - 2216	1.02	0.88	0.96	+
Halar - B-7	0.95	1.02	1.02	+
Tedlar - 2216	1.04	0.94	0.85	+
Tedlar - 573	1.15	0.94	--	+/0
Sun-Lite - 573	1.47	1.37	0.51	0
Lexan - B-7	1.04	0.86	0.68	0
FEP - B-7	0.95	0.93	0.69	0
FEP - 573	0.94	0.65	0.87	0
Korad - B-7	1.04	0.98	0.63	0
Halar - 573	1.03	0.99	0.69	0
Sun-Lite - 2216	1.45	0.71	0.54	-
Sun-Lite - B-7	0.84	0.74	0.31	-
Korad - 2216	1.01	0.87	0.48	-
Korad - 573	1.01	--	0.54	-

TABLE A-26
RATINGS OF CONFORMAL COATINGS AND ADHESIVES ON BASIS OF ELECTRICAL RESPONSE STABILITY OF COATED CELLS DURING 38 C, 100% R.H. EXPOSURE (450-550 HOURS)

	ΔP_m,(a) mW	Δn,(a) %	ΔFF(a)	ΔR_s(a) mΩ	Total	Ranking
Scotch-Weld 2216 B/A	69.6 (1)	3.78 (1)	0.181 (2)	168 (1)	(5)	+
Scotch-Grip 4475	46.5 (2)	3.11 (1)	0.182 (1)	157 (1)	(5)	+
Eccocoat AC-8	64.8 (1)	3.60 (1)	0.018 (3)	200 (1)	(6)	+
Silgrip SR-573	60.5 (1)	3.36 (1)	0.150 (2)	76 (3)	(7)	+
Scotch-Grip 4693	53.3 (2)	2.91 (2)	0.139 (2)	160 (1)	(7)	+
Acryloid B-7	47.4 (2)	2.62 (2)	0.153 (2)	165 (1)	(7)	+
DC 3140	60.0 (1)	3.3 (1)	0.088 (2-1/2)	83 (3)	(7-1/2)	0
Q3-6527 A/B	50.8 (2)	2.76 (2)	0.136 (2)	136 (2)	(8)	0
Butvar B-79	34.0 (2-1/2)	2.00 (2-1/2)	0.125 (2)	101 (2-1/2)	(9-1/2)	-
Eccocoat RTU	42.6 (2)	2.33 (2)	0.121 (2)	14 (3-1/2)	(9-1/2)	-
Eccocoat VE	14.6 (3)	2.73 (2)	0.022 (2)	61 (3)	(11)	-
RTV 118	16.3 (3)	0.87 (3)	0.028 (3)	56 (3)	(12)	-

(a) Δ Property = $(\text{Prop}_{\text{unexp}} - \text{Prop}_{\text{exp}})_{\text{uncoated cell}} - (\text{Prop}_{\text{unexp}} - \text{Prop}_{\text{exp}})_{\text{coated cell}}$.

category. Among the conformal coatings, the Eccocoat AC-8 and the DC 3140 are outstanding. These materials certainly are worthy of serious consideration in looking at candidates for use in the conformally top-coated encapsulant design. Again, the comment should be made that these conclusions are based on data from individual samples and replicate samples and longer term exposures, particularly to UV irradiation, are needed before any final comparisons of materials performance are made.

Table A-27 provides data on which a similar rating of eight total film and sheet laminate encapsulants was made. Only the Scotch-Weld 2216 B/A and Silgrip SR-573 adhesives were used in these constructions. As mentioned in the "Experimental" section, the Acrylite, Tedlar, and "Teflon" FEP-1 systems were superior to the others tested. The poor performance of the "Teflon" FEP laminate containing the Alure CX backing may be due to the use of a lower lamination temperature during its construction.

A preliminary overall assessment on the basis of performance only of the film (sheet) cover candidates has been made based on a combination of data obtained in this study and certain other key property considerations. This assessment is detailed in Table A-28. (+), (O), and (-) symbols have the same significance as indicated above. The (x) symbol refers to an absence of data associated with the particular property of interest. It should be noted that those materials known to contain UV stabilizers or plasticizers were ranked negative on this point and those materials having structural elements or other additionally desirable properties were given additional positive ratings.

Overall, the assessment indicates that "Teflon" FEP, Acrylite, and Sun-Lite are the leading cover candidats on the basis of individual properties only and that only Korad A and Mylar are rated lower than a (+) ranking. At this time, all of the materials identified here are still regarded as candidates for the encapsulant cover application.

TABLE A-27

RATINGS OF FILM AND SHEET LAMINATE ENCAPSULANTS ON BASIS OF ELECTRICAL RESPONSE STABILITY OF LAMINATED CELLS DURING 38 C, 100% R.H. EXPOSURE

Laminated Structure (Top to Bottom)	Exposure Time, hrs	ΔP_{max}[a] mw/d	Δn[a] %/d	$\Delta FF \times 10^3$[a] /d	ΔR_s[a] /d	Total	Ranking
Acrylite/2216/Alum foil	312	-0.39 (1)	-0.23 (1)	+0.15 (1)	+3.00 (1)	4	+
Sun-Lite/2216/Alum foil	312	-2.50 (3)	-0.54 (1)	-0.92 (2)	-3.30 (3)	9	+
Sun-Lite/573/Alum foil	240	-0.50 (1)	-1.50 (3)	-6.10 (4)	+5.90 (1)	9	+
FEP/573/FEP	240	-1.50 (1-1/2)	-0.70 (2)	-3.10 (3)	-2.00 (2)	8-1/2	+
FEP/573/Alure CX	144	-3.33 (3)	-1.67 (3)	-6.14 (4)	+8.33 (1)	11	0
Flexigard/573/Flexigard	144	+2.50 (1)	-0.83 (2)	-5.00 (4)	-2.33 (2)	9	+
Tedlar/573/Tedlar	144	-0.83 (1-1/2)	-1.33 (3)	-7.00 (5)	-1.17 (2)	11-1/2	0
Mylar/573/Mylar	144	-5.00 (4)	-2.67 (4)	-9.00 (5)	-7.00 (4)	17	-

(a) $$\Delta \text{ Property} = \frac{\text{Property Value}_{x \text{ hrs}} - \text{Property Value}_{0 \text{ hrs}}}{\text{Exposure Days}}$$

TABLE A-28

PRELIMINARY OVERALL PERFORMANCE RATING OF FILM/SHEET MATERIALS CANDIDATES (AT INDICATED THICKNESS)

	Optical Transmittance	Barrier Properties	Bonding Characteristics	Stability to[a]			Misc.	Overall Rating
				UV	TC	HH		
"Teflon" FEP (5 mil)	+	+	+	+++	-++	+xx	+ Low R.I.	++
Lexan (20 mil)	+	0	0	+++	+++	+xx	- UV Stab.	+
Korad A (6 mil)	+	-	+	+0+	+0+	+xx	- Plasticizer	0
Halar (5 mil)	+	+	x	+0x	+-x	+xx	--	+
Tedlar (4 mil)	+	0	+	+-+	+++	+xx	--	+
Weatherable Mylar (7 mil)	+	0/+	+	x0+	x++	xxx	- UV Stab.	0
Flexigard (5 mil)	+	0	+	x++	x++	xxx	--	++
Acrylite (125 mil)	+	0/+	x	x+x	x+x	xxx	+ Structural	++
Sun-Lite (25 mil)	+	0	x	++x	++x	+xx	+ Coeff. + Structural	++
Lexan (125 mil)	+	+	0	x+x	x+x	xxx	+ Structural - UV Stab.	+

(a) In order - optical, barrier, bonding characteristics.

TABLE A-29

PRELIMINARY OVERALL PERFORMANCE RATING OF FILM/SHEET - ADHESIVE ENCAPSULANT COVER CANDIDATES

	Optical Transmittance	Bonding Characteristics	Optical Stability to Environments (UV, TC, HH)	Rating
Tedlar - B-7	+	x	+++	+
Halar - B-7	+	x	+++	+
FEP - 2216	0/+	+	+++	+
Sun-Lite - 573	+	x	++0	0/+
Tedlar - 573	0/+	+	++x	+
Tedlar - 2216	+	+	+++	+
Halar - 2216	0/+	x	+++	+
Lexan (20 mil) - 2216	0	+	+++	0/+
Lexan (20 mil) - 573	0	0	+++	0
Lexan (20 mil) - B-7	+	x	++0	0/+
Sun-Lite - B-7	+	x	00-	0
Sun-Lite - 2216	+	x	+00	0
FEP - 573	0/+	+	+0+	0/+
FEP - B-7	0/+	x	++0	0/+
Korad - B-7	+	+	++0	0/+
Korad - 2216	+	+	++-	0
Korad - 573	0	+	+x0	0
Halar - 573	0	x	++0	0/+

GENERAL CONCLUSIONS

On the basis of the materials studies described above, three important general conclusions appear warranted. These are

(1) Utilization of film (sheet) laminate and conformally top-coated photovoltaic encapsulant designs appear attractive both from the standpoint of long-term performance and of cost. Of particular importance to the former array design has been the finding that the use of low-modulus adhesives to perform the dual function of film (sheet) bonding agent and photovoltaic encapsulant has minimized the delamination problem associated with temperature cycling, at least through some 250, 4-hour cycles covering the -40 to +90 C temperature range.

(2) A number of materials candidates for use as components (cover, adhesive, etc) of the film (sheet) laminate and conformally top-coated encapsulant designs have been identified that perform well in terms of maintenance of key properties during relevant environmental exposures. However, it appears that materials selection for any array design will have to be tailored for use in different geographic areas. A number of candidate polymerics appear to have properties that dictate against generalized usage, i.e., they have one or more property limitation, e.g., poor elevated temperature properties, poor resistance to abrasion, poor response at high humidity, etc.

(3) Before further judgments can be made relative to materials selection, evaluations of leading candidates must be extended to longer exposure times and to combinations of weathering environments with sample replication at a level in which the statistical significance of the results can be established.

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A-3. Carmichael, D. C., "Evaluation of Available Encapulation Materials for Low-Cost Long-Lived Silicone Photovoltaic Arrays", Battelle Columbus Laboratories Report to JPL, DOE/JPL-954328-78/8, June 30, 1978.

APPENDIX B

PROPERTY INFORMATION FOR SELECTED POLYMERIC MATERIALS

(From Reference 249)

PROPERTY INFORMATION FOR SELECTED POLYMERIC MATERIALS

Appendix B is set up as a series of Fact Sheets for the various materials of interest. They are further separated by applications as follow:

Page

ADHESIVES

Acryloid B-7 (Rohm and Haas Company) (a)

Acryloid B-7 is one of the family of acryloid resins which are polymeric derivatives of acrylic and methacrylic acids. The resin will "set" merely by solvent evaporation. The "set" film will remain permanently soluble. Acryloid B-7 is available as a 20 percent solids solution in ethylene chloride. It is useful in adhesive applications requiring a colorless, transparent, and flexible joint of good water, electrical, and chemical resistance. The acryloids provide tough thermoplastic bonds with such materials as metal, glass, plastics, natural or synthetic rubber, and fabrics.

Acryloid B-7 can be applied by brushing, roller coating, or spraying. Frequently it is necessary to reduce the viscosity, particularly for spray application. Such solvents as coal tar hydrocarbons, chlorinated hydrocarbons, ketones, esters, ether alcohols, and ether esters in general are suitable for thinning.

The following properties apply to Acryloid B-7:

1. Water-white color, perfect transparency, and resistance to discoloration
2. Good electrical resistance
3. Resistance to water, alcohol, alkali and acids
4. Resistance to mineral oils, vegetable oils, and greases
5. Resistance to chemicals
6. Good adhesion and flexibility.

Acryloid B-7 is available in 0.0037-0.0189 m^3 (1-5 gal) pails at \$3.79/kg (\$1.72/lb), also in 240 kg (530 lb) drums at \$1.59 kg (\$0.72/lb).

(a) Lists of trade names, company names, and cities are contained in Appendix B.

Cavalon 3100 S (E. I. du Pont de Nemours, Inc.)

Cavalon 3100 S is a 100 percent-reactive modified acrylic structural adhesive designed for high-strength bonding of steel, aluminum, fiberglass-reinforced polyester, and other high-strength structural materials. It also is a high-quality adhesive for general purpose bonding of many decorative or functional nonstructural assemblies.

Cavalon 3100 S provides an excellent balance of shear and peel strength across a wide temperature range up to 121 C (250 F). It is formulated for quick-setting, rapid-cure bonding. Fixture time on most substrates is 3 to 5 min and full cure takes place within 24 hours at room temperature. The cure takes place by contact with a surface activator, Cavalon 3300 S, placed on one of the bonding surfaces. A peroxide accelerator, 3340 S, also is available for incorporation into the adhesive where extremely rapid cure and minimum fixture times are desired. Cost is approximately $6.61/kg ($3.00/lb) for the adhesive.

Physical property data and other pertinent information for Cavalon 3100 S are listed below.

Physical Properties

Property	Property Value
Solids, percent	100
Viscosity, N s/m^2	10-14
Density, 1000 kg/m^3	1.0
Flash Point-Seta Closed Cup	17 C (62 F)
DOT Classification	Flammable
Shelf Life at 24 C (75 F)	12 months minimum
Working Life Following 3340 S Addition at 24 C (75 F)	14 days
Color	Translucent
Open Time, min	1-2
Fixture Time (With 3340 S), min	2-5
Fixture Time (Without 3340 S), min	4-9
Bondline, Min/Max, μm	76.2/762
Maximum Thermal Exposure	121 C (250 F)

Cavalon 3100 S – Continued

Typical Initial Bond Strengths

Bonded to	Tensile Shear (10^6 N/m^2)	T-Peel (N/0.0254 m)
Cold-Rolled Steel		
Oily	17	89
Abraded	22	156
Coarse grind	28	267
Cor-Ten Steel		
Oily	17	156
Coarse grind	30	222
Aluminum 2024-T3		
Oily	17	44
Abraded	27	62
FPL acid etch	27	133
Stainless Steel		
Alkaline cleaned	27	111
Abraded	19	156
Electrogalvanized Steel		
Oily	10	67
Treated	11	89
Fiberglass-Reinforced Polyester	5.5*	–
ABS	4.1*	–
PVC	8 *	–
Polycarbonate	6.3*	–
Nylon	6.2*	–
Acrylic	4.8*	–
High Impact Polystyrene	3.4*	–

*Indicates substrate failure.

Cavalon 3100 S – Continued

Bond Performance With Various Environmental Exposures

Exposure	Tensile Shear (10^6 N/m^2)	
5% Salt Spray, 38 C (100 F)	Initial	4 weeks
Cold-rolled steel	17	7
Cor-Ten steel	17	15
Aluminum 2024-T3; etched	28	26
Electrogalvanized steel	10	10
100% RH 38 C (100 F)	Initial	8 weeks
Cold-rolled steel	17	11
Cor-Ten steel	17	11
Aluminum 2024-T3 abraded	28	13
Aluminum 2024-T3 etched	28	25
Water Immersion 38 C (100 F)	Initial	4 weeks
Cold-rolled steel	17	14
Aluminum 2024-T3	28	26
Electrogalvanized steel	10	10
100-Octane Fuel Immersion	Initial	4 weeks
Aluminum 2024-T3 etched	28	28
70 C (158 F) Aging	Initial	8 weeks
Cold-rolled steel	17	17
Aluminum 2024-T3	28	28
Fiberglass reinforced	–	–
Polyester	6	6
Ambient Aging	Initial	2 years
Cold-rolled steel, oily	14	15
Florida Exposure 45 Degrees South, unprotected	Initial	6 months
Cold-rolled steel, oily	15	16
Cold-rolled steel, grit blasted	23	20
Atlas Weatherometer X-41 Cycle	Initial	1000 hr
Cold-rolled steel, oily	15	17

Eccobond 45LV (Emerson-Cuming, Inc.)

Eccobond 45LV is a clear, controlled flexibility epoxy adhesive having a viscosity considerably less than that of Eccobond 45. It is designed for use where shock and peel resistance are desired. It can be cured at room temperature or rapidly at elevated temperature. Adhesion to metals, glass, ceramic and plastic is excellent. The flexibility of Eccobond 45LV is determined by the amount of Catalyst 15LV which is used. Application is by brush, knife, or roller.

Typical average properties are presented below.

Property	Property Value
Uncatalyzed Viscosity, $N \cdot s/m^2$	35
Temperature Range for Use	-56 to 147 C (-70 to +300 F)
Hardness (Shore Durometer)	40
Bond Strength in Shear at Room Temp, N/m^2	22×10^6
After 30-Day Soak in Water, N/m^2	20×10^6
Flexural Strength, N/m^2	38×10^6
Izod Impact, J/0.0254 m of Notch	5.4
Dielectric Strength, kV/mm	16.1
Volume Resistivity, ohm-cm	3×10^{13}
Dielectric Constant, 10^2 to 10^9 Hz	3.2 to 2.9
Dissipation Factor, 10^2 to 10^9 Hz	.03 to .04

Of the several formulations given below, the semirigid one is used most frequently.

Rigid Formulation	100 parts Eccobond 45LV 25 parts Catalyst 15LV
Semirigid Formulation	100 parts Eccobond 45LV 50 parts Catalyst 15LV
Flexible Formulation	100 parts Eccobond 45LV 100 parts Catalyst 15LV

Eccobond 45LV is available at \$4.96/kg in 27.2 kg pails. The catalyst 15LV costs \$6.06/kg in 18.1 kg pails.

Epo-Tek 310 (Epoxy Technology, Inc.)

Epo-Tek 310 is a two-component, 100 percent solids, flexible, optically clear epoxy adhesive designed for bonding glass to glass, glass to metal, and metal to metal. It has low viscosity and a long pot life. It can be cured at room temperature or with heat. In addition to its excellent adhesion and optical properties, Epo-Tek 310 will withstand thermal cycling. It can be applied by spatula, brush, hypodermic needle, and commercial dispensing equipment. Other properties are listed below.

Mixing Ratio	10 parts "A" and 5.5 parts "B" (hardener)
Curing Schedule	Overnight at room temperature 2 hours at 65 C (149 F)
Pot Life	8 hours
Lap Shear Strength (Aluminum to Aluminum)	4 x 10^6 N/m^2
Temperature Cycling (3 Cycles -50 to 85 C)	Pass
Shelf Life	One year at room temperature

Epo-Tek 310 currently is available at from $40.00 kg ($18.15/lb) in lots of 0.45-4.1 kg (1-9 lb) to $26.68/kg ($12.10/lb) in 22.7 kg (50 lb and over).

Scotch-Weld 2216 B/A (3M Company)

Scotch-Weld 2216 B/A is a transparent, two-part, room-temperature-curing structural adhesive. It is of the modified-epoxy class. Normally, the epoxy base and modified amine accelerator are mixed in equal parts by weight or volume and applied with a spatula, trowel, or by pressure gravity flow. It has a work life of approximately 90 minutes at 24 C (75 F). It is used to bond rubber, metal, wood, most plastics, and masonry products. Curing requires 7 days at 4 C (40 F), 2 hours at 66 C (150 F), 5 minutes at 121 C (250 F), or 2 minutes at 177 C (350 F).

Cost is \$251.99/kg (\$114.28/gal) for the equal-parts combination of base resin and accelerator.

Performance characteristics of Scotch-Weld 2216 B/A are listed below.

Property Performance Data

Property	Property Value
Overlap Shear Strength at -55 C (-67 C)	14×10^6 N/m^2 (aluminum FPL etch)
Overlap Shear Strength at 24 C (75 F)	17×10^6 N/m^2 (aluminum FPL etch)
Overlap Shear Strength at 82 C (180 F)	2.8×10^6 N/m^2 (aluminum FPL etch)
T-Peel Strength at 24 C (75 F)	111 N/0.0254 m of width (aluminum FPL etch)
Thermal Conductivity	0.391 W/mC
Coefficient of Thermal Expansion	
0-40 C (32-104 F)	102×10^6 C^{-1}
40-80 C (104-176 F)	134×10^6 C^{-1}
Arc Resistance	130 seconds
Dielectric Strength	16.1 kV/mm
Dielectric Constant at 23 C (73 F)	5.51 (measured at 1.00 kHz)
Dielectric Constant at 60 C (140 F)	14.17 (measured at 1.00 kHz)
Dissipation Factor at 23 C (73 F)	0.112 (measured at 1.00 kHz)
Surface Resistivity at 23 C (73 F)	5.5×10^{16} ohms (measured at 500 volts dc)
Volume Resistivity at 23 C (73 F)	1.9×10^{12} ohms-cm (measured at 500 volts dc)

*The above data were developed using 7-day cure at a temperature of 24 C (77 F) and a pressure of 1.38×10^4 N/m^2.

Scotch-Weld 2216 B/A – Continued

Overlap Shear Strength After Environmental Aging

Environment	Time, days	Test Results (24 C), 10^6 N/m^2
100% RH at 49 C (120 F)	14	20
	30	14
	90	10
Salt Spray at 35 C (95 F)	14	16
	30	3.5
	60	2.1
Tap Water at 24 C (75 F)	14	22
	30	20
	90	14
Air at 71 C (160 F)	35	32
Air at 149 C (300 F)	40	34
Anti-Icing Fluid at 24 C (75 F)	7	23
Hydraulic Oil at 24 C (75 F)	7	26
JP-4 Fuel	7	22
Hydrocarbon Fluid	7	23

"Teflon" FEP (E. I. du Pont de Nemours, Inc.)

"Teflon" FEP has a balance of properties including chemical inertness, excellent dielectric properties, nonaging characteristics, performance in temperature extremes, and, in thin sections, excellent optical properties. Unlike "Teflon" TFE, which does not melt in the usual sense and must be processed by methods reminiscent of powder metallurgy, "Teflon" FEP can be melt processed by extrusion, compression-, injection-, and blow molding. The distinct melting point also permits the use of "Teflon" FEP as a hot-melt adhesive.

Few adhesives can match the broad capabilities of "Teflon" FEP film. It is excellent for bonding many materials – metals and nonmetallics as well. It produces strong bonds between two surfaces of TFE or between TFE and other substrates. It also can be used as an adhesive for bonding like FEP films to one another or to other materials.

Because it is available in film form, on a roll, in a range of thicknesses from 12.7-2286 μm (0.5-90 mils) "Teflon" FEP is more convenient to handle and store than many other adhesives. At a thickness of 127 μm (5 mils) current costs are \$28.67/kg (\$13.00/lb) or \$7.77/m^2 (\$0.72/ft^2). Property information for "Teflon" FEP is given below.

Typical Property Values – "Teflon" FEP

Property	ASTM Method	Property Value
Tensile Strength, 23 C (73 F), N/m^2	D638	21-28 x 10^6
Elongation, 23 C (73 F), percent	D638	300
Flexural Modulus, 23 C (73 F), N/m^2	D790	655-724 x 10^6
Impact Strength, -54 C (-65 F), J/0.0254 m	D256	3.9
Impact Strength, 23 C (73 F), J/0.0254 m	D256	No break
Hardness, Durometer	D2240	D55
Deformation Under Load, 23 C, 6.9 x 10^6 N/m^2, 24 hours, percent	D621	1.8
Melting Point, C	–	250-279 C (482-534 F)
Coefficient of Linear Thermal Expansion per C^{-1} (-73 to 70 C)	D696	14.9-18.7 x 10^{-5}
Thermal Conductivity, W/m·C	C177	0.251
Specific Heat, J/kg·C	–	1173
Continuous Service Temperature, C	–	204 C (400 F)
Flammability (Vertical Flame Test)	(UL 83)	Does not support combustion
Dielectric Strength Short Time, 0.254 mm, kV/mm	D149	82.7
Dielectric Constant, 60 to 10^9 Hz	D150	2.1
Dissipation Factor, 60 to 10^9 Hz	D150	.0001-.001
Volume Resistivity, ohm-cm	D257	>10^{18}
Water Absorption, percent	D570	<0.01
Weather and Chemical Resistance	–	Excellent
Specific Gravity	D792	2.12-2.17

RTV 108 (General Electric Company)

RTV 108 is a one-component dimethyl silicone adhesive/sealant with outstanding dielectric properties. It is useful throughout a temperature range from -90 to 205 C (-130 to +400 F) for bonding and sealing. Properties of the uncured and cured resin are presented below.

Property	Property Value
Typical Uncured Properties	
Color	Transparent
Consistency	Paste
Shelf Life, months	12
Typical Cured Properties	
Specific Gravity	1.07
Hardness, Shore A Durometer	30
Tensile Strength, N/m^2	2.4×10^6
Elongation, percent	400
Tear Resistance, Die B, N/0.0254 m	200
Brittle Point, C	<-68
Linear Shrinkage, percent	0.12
Maximum Continuous Service Temperature, C	204
Thermal Conductivity W/m·C at 93 C (200 F)	0.206
Coefficient of Thermal Expansion, -18 to 177 C (0-350 F), C^{-1}	$<27 \times 10^{-5}$
Dielectric Strength, kV/mm	19.7
Dielectric Constant at 60 Hz	2.8
Dissipation Factor at 60 Hz	.0026
Volume Resistivity, ohm-cm	3×10^{15}

RTV is currently available at $5.69/kg ($2.58/lb) in a 204 kg (450 lb) drum, greater than 10 drum quantities at $5.03 kg ($2.28/lb).

RTV 118 (General Electric Company)

RTV 118 is a one-component dimethyl silicone adhesive/sealant similar to RTV 108. Uncured and cured properties are given below.

Property	Property Value
Typical Uncured Properties	
Color	Transparent
Consistency	Self-leveling
Viscosity, $N \cdot s/m^2$	35
Shelf Life, months	12
Typical Cured Properties	
Specific Gravity	1.07
Hardness, Shore A Durometer	22
Tensile Strength, N/m^2	3.1×10^6
Elongation, percent	430
Tear Resistance, Die B, N/0.0254 m	147
Brittle Point, C	<-59
Linear Shrinkage, percent	0.3
Maximum Continuous Service Temperature, C	204
Thermal Conductivity, W/m·C at 93 C (200 F)	0.206
Coefficient of Thermal Expansion, -18 to 177 C (0-350 F), C^{-1}	$<27 \times 10^{-5}$
Dielectric Strength, kV/mm	19.7
Dielectric Constant at 60 Hz	2.7
Dissipation Factor at 60 Hz	.0004
Volume Resistivity, ohm-cm	2×10^{15}

RTV 118 is available at $14.33 kg ($6.50/lb) in a 204 kg (450 lb) drum, greater than 10 drum quantities at $13.67 kg ($6.20/lb).

COATINGS

Eccocoat AC-8 (Emerson and Cuming, Inc.)

Eccocoat AC-8 is a one-part water-white acrylic based coating material. It can be applied by brush, dip, or spray methods. As a clear coating for metal surfaces such as brass, aluminum, and steel, it exhibits excellent adhesion, clarity, and durability. Eccocoat AC-8 is applied at ambient conditions, and dries to a tack-free state in 15-30 min. Adhesion and film hardness are improved by a 30-min bake at temperatures up to 149 C (300 F). Property data are tabulated below.

Property	Property Value
Viscosity (No. 3 spindle)	1.5 Ns/m^2
Flexibility	Unaffected by bend over 0.635-cm mandrel
Color	Water white
Service Temperature	-54 to 177 C (-65 to 350 F) [slight yellowing at 177 C (350 F)]
Weatherability	Unaffected by 6 months' exposure in Canton, Mass.
Dielectric Constant, 60-10^6 Hz	3.0-2.6
Dissipation Factor, 60-10^6 Hz	0.04-0.01

Eccocoat AC-8 costs $4.19/kg in 18.1 kg containers.

Kynar 202 (Pennwalt Corporation)

Kynar is a polyvinylidene fluoride resin that is characterized by toughness, corrosion resistance, and application versatility. It is a crystalline, high-molecular-weight polymer and has a good balance of properties – chemical, mechanical, electrical, and thermal. Kynar 202 is a dispersion of the material in selected latent solvents. It is an excellent weather resistant barrier coating.

Cured Kynar dispersions usually measure approximately one-third of the wet film thickness. Curing normally is carried out in an air-circulating oven preheated to 160-250 C (320-482 F). The temperature is then raised to about 297 C (567 F) for about 30 min. Properties of the homopolymer resin are given below.

Property	ASTM Method	Property Value
Specific Gravity	D792	1.75-1.78
Refractive Index, n_D^{25}	D542	1.42
Clarity	–	Transparent to translucent
Melting Point, Crystalline, C	Fischer-Johns	171
Water Absorption, percent	D570	0.04
Water Vapor Permeability, g/25.4 μm/ 24 hr-/m^2/atm	–	1.0
Resistance to Weathering	–	Excellent
Tensile Strength		
25 C (77 F), N/m^2	D638	36-52 x 10^6
100 C (212 F), N/m^2	D638	19-23 x 10^6
Elongation		
25 C (77 F), percent	D638	25-500
100 C (212 F), percent	D638	400-600
Yield Point		
25 C (77 F), N/m^2	D638	36-51 x 10^6
100 C (212 F), N/m^2	D638	19-23 x 10^6
Flexural Modulus, Tangent, N/m^2	D790	1393 x 10^6
Compression Strength, 25 C (77 F), N/m^2	D695	55-69 x 10^6
Izod Impact, Notched 25 C (77 F), J/0.0254 m	D256	4.6-5.2
Hardness, Durometer, Shore, D	D676	70-80
Thermal Conductivity, 25-163 C (77-325 F), W/m·C	–	0.243-0.186
Specific Heat, J/kg·C	–	1371
Thermal Expansion, 25-60 C (77-140 F)	D696	14.4-15.3 x 10^{-5}
Thermal Stability, 1 yr 149 C (300 F)		
Weight Loss	–	None
Change in Color	–	Slightly darkened
Thermal Degradation Temperature, C	TGA (Du Pont)	410-432
Deflection Temperature, 46 x 10^4 N/m^2, C	D648	149
Deflection Temperature, 182 x 10^4 N/m^2, C	D648	91
Low-Temperature Embrittlement, C	D568	<-62

Kynar 202 – Continued

Property	ASTM Method	Property Value
Burning Rate	D635	Self-extinguishing Non-dripping
Volume Resistivity, ohm-cm	D257	2×10^{14}
Surface Arc Resistance, sec	D495	50-60
Dielectric Strength, Short Time (500 V/sec), kV/mm		
3175-μm thickness	D149	10.2
203-μm thickness	D149	50.4
Dielectric Strength, Step by Step (1 kV Steps), kV/mm	D149	37.4
Dielectric Constant		
60 Hz	D150	8.40
10^3 Hz	D150	7.72
10^6 Hz	D150	6.43
10^9 Hz	D150	2.98
Dissipation Factor		
60 Hz	D150	0.049
10^3 Hz	D150	0.019
10^6 Hz	D150	0.159
10^9 Hz	D150	0.110

Cost is $24.26/kg ($11.00/lb) in 340 kg (750 lb) quantities.

"Teflon" FEP (E. I. du Pont de Nemours, Inc.)

(The general properties of "Teflon" FEP were described earlier.)

The material is available in aqueous dispersions for coating applications. Dispersion data are listed below.

Grade	Container	Weight of Resin	Cost
Type 30	0.11 m^3 (30 gal)	93 kg (205 lb)	\$9.04 kg (4.10/lb)
Type 30B	0.11 m^3 (30 gal)	93 kg (205 lb)	\$9.15 kg (4.15/lb)
Type 42	0.11 m^3 (30 gal)	46 kg (102 lb)	\$9.04 kg (4.10/lb)

"Pyre-M.L." (E. I. du Pont de Nemours, Inc.)

"Pyre-M.L." wire enamels are solutions of polyamic acids formed by the reaction of aromatic diamines with aromatic dianhydrides. When the enamel is baked, it is converted to an inert polyimide. The polyimide is known for its excellent thermal stability, radiation resistance, solvent resistance, cryogenic resistance, and electrical properties.

Five "Pyre-M.L." enamels seem to be the most useful of several solvent variations marketed. These are listed below.

Properties of Five Pyre-M.L. Wire Enamels

	RC-5069	RC-5057	RC-5019	RC-5044	RC-5063
Polymer Solids Percent* ± 0.5%	14.0	16.5	17.0	17.0	17.5
Converted Polymer Solids Percent ± 0.5%	12.8	15.2	15.6	15.6	16.0
Gallon Wt/(kg/m^3) ± 0.05	1045	1055	1092	1092	1093
Sol. Density (kg/m^3 at 25 C) (77 F) ± 0.07	1049	1059	1096	1096	1097
Visc. (N·s/m^2 at 25 C) (77 F)**	0.5-1.2	5-7	5-7	5-7	8-11.5
Flash Point (C) (Open Cup)	75	75	96	96	96
Solvents	NMP (Aromatic Hydrocarbon)	NMP (Aromatic Hydrocarbon)	NMP	NMP	NMP

NMP = N-Methyl-2-pyrrolidone

*Solids Test Method – 1 g polymer with 1 ml NMP solvent added and baked 1 hr x 160 C (320 F).

**Viscosity at time of manufacture measured with Brookfield LVF Viscometer No. 3 Spindle at 12 rpm except for RC-5069 which uses No. 2 Spindle at 30 rpm.

The mechanical properties of "Pyre-M.L." polymer films are excellent and are retained over a wide temperature range. The zero strength temperature is 800 C (1472 F). The flex modulus, approximately 2.758 x 10^6 N/m^2 at ambient temperature, increases by only 25 percent when measured at -190 C (-310 F) and decreases by about 50 percent when measured at 250 C (482 F).

Many of the properties of wire coated with "Pyre-M.L." depend on the degree to which it is cured. This dependence is demonstrated as follows.

"Pyre-M.L." – Continued

Properties of Heavy Build No. 18 (1.024 mm) Wire Coated With "Pyre-M.L."

Degree of Cure	Incomplete	Moderate	Thorough	Very Thorough
Dissipation Factor(1), percent	2.0	0.4	0.25	0.15
Dielectric Constant(1)	4.5	3.9	3.7	3.2
Weight Loss(2), percent	16	3	1.6	1.3
Crazing(3)	Very severe	Moderate	Slight	Very slight
Crazing After 1/2 Hr x 150 C (302 F)(4)	Severe	None	None	None
Crazing After 1 Hr x 200 C (392 F)(5)	None	None	None	None
Flexibility(6)	Borderline 2X	Passes 2X	Borderline 1X	Passes 1X
Intercoat Adhesion	Good	Good	Fair	Fair
Undirectional Scrape Resistance (kg) (0.23 mm)	–	1.02-1.17	1.06-1.27	1.19-1.50
G.E. Scrape	40-70	15-35	20-40	25-45
Dielectric Strength	8.5 kV	12 kV	11 kV	11 kV
Oil and Water Resistance(7)				
Flexibility	Poor	Good	Good	Good
Dielectric Strength	2 kV	7 kV	8.5 kV	5.5 kV

(1) Clean wires with soft cloth and bend into U shape. Dip wire in mercury. Connect one end of wire to bridge; place the other lead from the bridge in mercury. Make measurement at 25 C (77 F) at 1000 Hz.

(2) Weigh 70 g of wire degreased with acetone wet cloth. Bake 5 min at 300 C (572 F) or 2 hr at 200 C (392 F). Cool 2 min and weigh.

(3) Wrap wires on 1X through 6X mandrels and dip in 2:1 mixture of N-methyl pyrrolidone and dimethylacetamide. Examine under 10X microscope. Many other solvents will give similar results.

(4) As above but bake coils for 30 min at 150 C (302 F) before dipping in solvent.

(5) As above but bake 1 hr at 200 C (392 F).

(6) Quick snap by NEMA snap test and wrap on 1X and 2X mandrels.

(7) Place NEMA twists in 7-in. (177.8 mm) length of 3/4-in. (19.05 mm) iron pipe. Add 50 cc of transformer oil and three drops of water. Seal pipe ends and age 88 hr at 200 C (392 F). Cool. Remove wires and measure dielectric strength and note flexibility.

Cost information for "Pyre-M.L." varnishes are listed below.

RC-5063	\$8748/m^3
RC-5057 (10 or more gallons)	\$7680/m^3
RC-5019/RC-5044 (10 or more gallons)	\$8584/m^3
RC-5069	\$6975/m^3

Thinner for use with "Pyre-M.L." varnish, designated T-8585 is priced at \$6605/m^3.

Parylene C (Union Carbide Corporation)

Parylene is a specialty coating produced by vapor-phase deposition and polymerization of para-xylene (or its chlorinated derivatives). The polymers are highly crystalline, straight-chain compounds that have been known for over 15 years as tough materials with excellent dielectric characteristics. Molecular weight is approximately 500,000.

Parylene is extremely resistant to chemical attack, exceptionally low in trace-metal contamination, and compatible with all organic solvents used in the cleaning and processing of electronic circuits and systems. Although parylene is insoluble in most solvents, it will soften in certain solvents at temperatures in excess of 150 C (302 F).

In current applications, parylene is deposited in thicknesses ranging from 6.4-38 μm (0.25 to 1.5 mils) in a single coating operation. Because it requires no catalysts or solvents, parylene offers the advantage that no foreign substances are present that might adversely affect the performance of the coating. Further, it is applied without increasing the temperature of the object to be coated much above room temperature, eliminating all risk of thermal damage.

Parylene C, poly-monochloro-para-xylylene, is the most widely used member of the group of parylenes because of its excellent barrier properties. Property data for Parylene C are provided in the following tabulation.

Property	Property Value
Tensile Strength, N/m^2	69×10^6
Yield Strength, N/m^2	55×10^6
Elongation to Break, percent	200
Yield Elongation, percent	2.9
Density, kg/m^3	1289
Coefficient of Friction	
Static	0.29
Dynamic	0.29
Water Absorption, percent (24 hours)	0.01 (thickness-483 μm)
Index of Refraction, N_D (23 C)	1.639
Melting or Heat Distortion Temperature, C	280
Linear Coefficient of Expansion, C^{-1}	6.9×10^{-5}
Dielectric Strength, Short Time, kV/mm at 0.0254 mm	220
Volume Resistivity, 50% RH, ohm-cm 25 C (77 F)	6×10^{16}
Surface Resistivity, 50% RH, ohms 25 C (77 F)	10^{14}
Dielectric Constant	
60 Hz	3.15
10^3 Hz	3.10
10^6 Hz	2.95
Dissipation Factor	
60 Hz	0.020
10^3 Hz	0.019
10^6 Hz	0.013

In addition to offering a license for in-house use by customers, Union Carbide provides a Development Custom Coating Service at its Bound Brook facility. Parts coating costs are as follows:

Usable coating volume 9.5 cm x 9.5 cm x 55.9 cm
Cost per run $360.00, coating thickness up to 0.038 mm
Usable coating volume 20.3 cm x 20.3 cm x 66.0 cm
Cost per run $890.00, coating thickness up to 38 μm.

DC-3140 (Dow Corning)

DC-3140 is a clear, flexible, one-component silicone coating that cures at room temperature (72 hours) without emitting acetic acid or other corrosives. It can be applied by dipping, brushing, or spraying.

The material's good tear strength and toughness allows its use in applications requiring ruggedness and high durability. It has very low water absorption and low dielectric losses that make it ideal for protecting electronic componentry. Other important features include (1) ease of processing, (2) noncorrosivity, (3) wide useful temperature range, (4) environmental resistance, and (5) ease of repair.

DC-3140 is available for $27.00/kg ($12.25/lb) in large quantities. DC-1204 primer is recommended where maximum adhesion is desired. The latter costs $9.26/kg ($4.20/lb).

Typical properties of DC-3140 are presented below.

Property	Property Value
Physical Properties	
Color	Clear
Specific Gravity	1.05
Durometer, Hardness Shore A	22
Tensile Strength, N/m^2	2.1 x 10^6
Elongation, percent	350
Tear Strength, Die B, N/0.0254 m	89
Peel Strength From Primed Aluminum, N/0.0254 m	107
Thermal Conductivity (25 to 100 C), W/m·C	0.122
Volume Expansion (25 to 100 C), C^{-1}	8.8 x 10^{-4}
ASTM D-149 Electric Strength, kV/mm	19.7
ASTM D-257 Volume Resistivity, ohm-cm	5 x 10^{14}
ASTM D-150 Dielectric Constant at 25 C (77 F)	
100 Hz	2.64
100 kHz	2.63
ASTM D-150 Dissipation Factor at 25 C (77 F)	
100 Hz	0.0016
100 kHz	0.0004
Application Properties	
Consistency	Flowable
Viscosity, N·s/m^2	35
Coating Thickness per Dip, mm	0.38
Skin-Over Time, minutes	25
Tack-Free Time, hours	1-1/2
Cure Time, 635 mm, hours	24
Cure Time, 3175-μm Thickness, hours	72
Full Cure, 3175-μm Thickness, days	7
Thin-Section Cure	Excellent
Nonvolatile Content, percent	97
Shelf Life, months	6

Glass Resin Type 650 (Owens-Illinois)

Glass resins are semi-inorganic polymers with a "back-bone" of silicon and oxygen. The manufacturer claims the materials contain more than 80 percent silicon and oxygen and are superior to commercial silicones.

The resins are presently supplied in the form of prepolymers in ethyl alcohol solution. The resin concentration is 65 percent. For use in coatings, the alcohol is evaporated and the prepolymers are redissolved in acetone. When cured, they are thermoset silicones which will not soften when heated and are insoluble in all common solvents. Heat must be applied to cure the coating.

Glass Resin Type 650 is the more reactive of the two available types and has exceptional light transparency. Coatings thicker than 25 μm (1 mil) are somewhat brittle: coating thicknesses of 12.7 μm (0.5 mil) are flexible and adhere well to a variety of substrates. General resin properties are listed below.

Property	Property Value
Specific Gravity	1.3
Flammability	Nonflammable
Tensile Strength, N/m^2	24 x 10^6
Elongation, percent	Approx. zero
Compressive Strength, N/m^2	207 x 10^6
Flexural Strength, N/m^2	34 x 10^6
Modulus of Elasticity Flexural, N/m^2	1379 x 10^6
Impact Strength, Izod (J/0.0254 m notch)	0.04
Hardness, Rockwell R Scale (6350-μm-thick sample)	120-140
Dielectric Strength: (1270-μm sample) Short Time (kV/mm)	35.4
Dielectric Constant	
60 Hz	4.1
10^6 Hz	3.2
Dissipation Factor	
60 Hz	30 x 10^{-4}
10^6 Hz	70 x 10^{-4}
Arc Resistance, seconds	130-195
Volume Resistivity, ohm-cm	
25 C (77 F)	1 x 10^{14}
75 C (167 F)	1 x 10^{16}
Coefficient of Linear Expansion: C^{-1} 0-300 C (32-572 F)	130 x 10^{-6}
Thermal Conductivity, W/m·C	0.142

Light transmission data specific to Glass Resin Type 650 follow:

Wave Length, nm	Light Transmission, percent
360-700	85-95
300	70
230	60
195	0.1

The price schedule for Glass Resin Type 650 is:

Flake Form, B Stage	
Less than 45 kg	$33.07/kg 0.45 kg in bag, 4.5 kg in drum
45 kg up to 450 kg	$30.86/kg 45 kg drum
450 kg up to 900 kg	$27.56/kg 45 kg drum

FILMS

Korad A (Rohm and Haas Company)

Korad A film is a durable all-acrylic polymer supplied in film form, and actually may be regarded as a 100 percent solid, prefabricated, quality controlled acrylic coating. As such, it provides complete freedom from the problems of solvents, pigment settling, viscosity-solids limitations, film formation, and thickness variations. It also offers a balance of fabrication, hardness, and toughness not yet attained with liquid systems.

Although adhesives are required for lamination to metal and most cellulosic surfaces, excellent adhesion to ABS and PVC plastic substrates can be achieved with heat and pressure only. Such film-to-plastic laminates can be readily embossed and vacuum formed.

Korad A is available in the clear form in gauges from 50.8 to 152.4 μm. Widths up to 1.57 meters are available routinely while greater widths are manufactured to order. Cost information for Korad A clear film in various gauges follows:

Thickness	\$/kg	Approx. Yield, m^2/kg	Approx. Cost, \$/m^2
50.8 μm	4.30	17.34	.2487
76.2 μm	4.30	11.56	.3724
152.4 μm	4.30	5.78	.7449

The following tabulation lists pertinent property information for the free film.

Property	Test Method	Typical Values*
Thickness, μm		50.8, 76.2, 152.4**
Specific Gravity, degree	ASTM D-792-60T	1.26
Area Factor, m^2 kg		10.2
Dimensional Stability, percent	10.2-cm-disk in air oven	
	10 min at 80 C (176 F)	0
	10 min at 130 C (266 F)	20
	10 min at 200 C (392 F)	59
Blocking	4.1 $(10)^4$N/m^2 16 hr C	66
Gloss (60 deg), percent reflectance	ASTM D523-62T	35
Contrast Ratio deg	ASTM D-589-66	>.98
WVTR, g/24 hr/.064 m^2/atm	ASTM E-96-63T	6 to 9
Water Absorption, percent	ASTM D-570-63 24 hr at 23 C (73 F)	1.4 to 1.6
Tear Resistance, N/mm	ASTM D-1004-61 (0.08 cm/sec)	175
Tensile Strength, N/mm^2	ASTM D-882-61T 0.08 cm/sec; 10.2-cm GL	
Yield		35×10^6
Rupture		33×10^6

Korad A (Rohm and Haas Company) – Continued

Property	Test Method	Typical Values
Tensile Elongation, percent	ASTM D-882-61T 0.08 cm/sec; 10.2-cm GL	
Yield		4.2
Rupture		75
Dielectric Strength, kV/mm	ASTM D-149-64T	86.6
Dielectric Constant, at 60 Hz	ASTM D-150-65T	4.8
Dielectric Constant, at 10^3 Hz	ASTM D-150-65T	4.4
Volume Resistivity, ohm/cm^3	ASTM D-257-61	1×10^{16}
Surface Resistivity, ohm/cm^2	ASTM D-237-61	2×10^{14}

CODE: GL – gauge length

*Based on 76.2-μm film.

**Other thicknesses can be manufactured should sufficient volume be determined.

Solvent resistance results with Korad A film, based on immersion tests [run in accordance with ASTM D-543-60 T, 7 days immersion at 24 C (75 F)] are listed below.

Solvent	Results
Ethyl Alcohol (100 percent)	Swollen
Iso-octane (100 percent)	No Change
Gasoline	No Change
JP-4 Jet Fuel	No Change
Motor Oil (SAE-30, ASTM No. 3)	No Change
Ethyl Acetate	Dissolved
Toluene	Dissolved
30 percent H_2SO_4	No Change
10 percent HNO_3	No Change
10 percent NaOH	No Change

Kynar (Pennwalt Corporation)

(The general properties of Kynar Homopolymer resin were described earlier.)

Kynar film is available in thicknesses greater than 127 μm (5 mils). It can be vacuum formed readily. The film resists aging, abrasion and biological attack. It is fungus resistant and has low moisture vapor transmission.

Typical properties of 127 μm (5 mil) Kynar film are listed below:

Property	Property Value
Transparency	Clear
Flammability	Self-Extinguishing Nondripping
Stability to Ultraviolet	Excellent
Thermal Stability (1 year, 150 C)	No Weight Loss Slight Color Change
Tensile Strength, N/m^2	41-55 x 10^6
Tensile Elongation, percent	150

Tedlar (E. I. du Pont de Nemours, Inc.)

The unique properties of Tedlar (polyvinyl fluoride) film include excellent resistance to weathering, outstanding mechanical properties, and inertness toward a wide variety of chemicals and solvents.

For glazing and solar-energy applications, a 101.6-μm (4-mil) transparent Tedlar has been developed, designed to obtain maximum strength and toughness. Transmissivities of 92 to 94 percent of total incident solar energy have been measured by pyranometer. The main losses are caused by surface reflection, with negligible absorption. After 5 years' Florida exposure, the film has retained about 95 percent of its original transmissivity and about half of its original strength and toughness.

Tedlar is available in grades designated 400BG20TR, for use with adhesives, and 400SG20TR. The former can be used to bond to a variety of substrates. Tedlar film costs \$0.43/m^2/25.4 mm (\$0.04/ft^2/mil).

Property data for Tedlar film are contained in the following tabulation.

Property	Typical Property Value	Test Method*
Density	1.38-1.57 x 10^3 kg/m^3	Weighed samples
Impact Strength	1.0-2.2 $\frac{N\cdot cm}{\mu m}$	Du Pont Pneumatic Tester
Moisture Absorption	<0.5% for all types	Water immersion
Moisture Vapor Transmission	157-205 g/(100 m^2) (hr) (25.4 μm) (53 mm Hg)	ASTM E-96-58T; 39.5 C (103 F), 80 percent RH
Refractive Index	1.46	ASTM D-542 Abbe Refractometer; 30 C (86 F)
Ultimate Tensile Strength	48-124 x 10^6 N/m^2	ASTM D-882, Method A 100% elong./min-Instron
Ultimate Elongation	115-250%	ASTM D-882, Method A 100% elong./min-Instron
Gas Permeability		
Carbon Dioxide	11.1 cc/(0.06 m^2) (24 hr) (atm) (25 μm)	ASTM D-1434
Helium	150 cc/(0.06 m^2) (24 hr) (atm) (25 μm)	ASTM D-1434
Hydrogen	58.1 cc/(0.06 m^2) (24 hr) (atm) (25 μm)	ASTM D-1434
Nitrogen	0.25 cc/(0.06 m^2) (24 hr) (atm) (25 μm)	ASTM D-1434
Oxygen	3.2 cc/(0.06 m^2) (24 hr) (atm) (25 μm)	ASTM D-1434
Weatherability	Excellent	Florida exposure; facing south at 45 degrees to horizontal
Aging	3000 hours	Circulating air oven; 150 C (302 F)
Heat Sealability	Some varieties - see Bulletin TD-14	
Linear Coefficient of Expansion	5.0 x 10^{-5} C^{-1}	

Tedlar (E. I. du Pont de Nemours, Inc.) – Continued

Property	Typical Property Value	Test Method*
Shrinkage (Type 20) MD & TD	4% at 130 C (266 F)	Air oven, 30 min
(Type 30) TD only	4% at 170 C (338 F)	Air oven, 30 min
(Type 40) TD only	2.5% at 170 C (338 F)	Air oven, 30 min
Temperature range		
Continuous use	-72 C to 107 C (-100 F to 225 F)	
Short cycles or release, 1-2 hr	Up to 175 C (350 F)	
Zero Strength	260 C to 300 C (500-572 F)	Hot bar
Dielectric Constant	9.9	ASTM D-150; 1kHz
Dielectric Strength, kV/mm	138	ASTM D-150; 60 Hz
Dissipation Factor, percent	1.4	ASTM D-150; 1000 Hz
	1.7	ASTM D-150; 1000 Hz, 70 C (158 F)
	3.4	ASTM D-150; 10 Hz
	1.6	ASTM D-150; 10 Hz, 70 C (158 F)
Volume Resistivity, ohm-cm	7×10^{14}	ASTM D-257
	1.5×10^{11}	ASTM D-257; 100 C (212 F)

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*All tests were performed at 23 C (72 F) unless otherwise noted.

"Teflon" FEP (E. I. du Pont de Nemours, Inc.)

(The general properties of "Teflon" FEP were described earlier.)

The material is available as film in continuous sheeting up to a thickness of 2413 μm (95 mils). "Teflon" FEP films can be heat bonded and sealed, vacuum formed, and laminated to various substrates.

Type 9500L lining film 2413 μm (95 mils) thick is available at \$34.18/kg (\$15.50/lb) or \$185.38/m^2 in 90.7-271.6 kg (200-599 lb) quantities and \$32.85/kg (\$14.90/lb) or \$178.21/m^2 for quantities greater than 271.6 kg (600 lb).

Lexan (UV Stabilized) (General Electric Company)

Lexan polycarbonate film offers an unusual combination of properties including clarity, dimensional stability, toughness, flexibility, heat resistance, and excellent dielectric performance. It is heat sealable and usable over a range of temperature from -101 to +135 C (-150 to +275 F). It is available in thicknesses from 25-508 μm (1-20 mils).

Lexan film 127 μm (5 mil) costs \$0.936 m^2 (\$0.087 ft^2) or \$6.17/kg (\$2.80/lb).

Property data for Lexan film are summarized in the following table.

Property	Test	Property Value
Area Factor, m^2/kg		32.8 (25 μm film)
Specific Gravity		1.20
Tensile Strength, N/m^2	ASTM D-882-56T	58-62 x 10^6
Elongation, percent	ASTM D-882-56T	85-105
Bursting Strength, Mullen points	ASTM D-774	25-35 (101.6 μm film)
Tearing Strength, N/μm	ASTM D-1004	0.20-0.27
Heat Distortion Temperature, C		153
Folding Endurance	ASTM D-643-43 (B)	250-400
Water Absorption (24 hr), percent	ASTM D-570	0.35
Dielectric Constant at 25 C	60 Hz	2.99
	1 kHz	2.99
	1 MHz	2.93
Power Factor at 25 C, percent	60 Hz	0.13-0.23
	1 kHz	0.13
	1 MHz	1.10
Dielectric Strength, kv/mm		59.1
Volume Resistivity at 25 C, ohm-cm		10^{16}

Mylar (E. I. du Pont de Nemours, Inc.)

Mylar is a polyester film made from polyethylene terephthalate. Mylar has excellent dielectric properties, good tensile, excellent resistance to most chemicals and moisture and can withstand temperature extremes from -70 to 150 C (-94 to +302 F). It is available in roll or sheet form; thicknesses range from 3.6 μm (1/7 mil) to 355.6 μm (14 mils), and widths from 6.35 mm (1/4 in.) to 3.05 m (120 in.). It can be laminated, metalized, or coated. Adhesives are available for laminating Mylar to itself and practically any other material. The film can also be coated with heat-sealable materials. Typical property data for Mylar film are presented below.

	Typical Property Value		
Property	25 μm Type A	25 μm Type T	Test Method
Ultimate Tensile Strength (MD)	172×10^6 N/m^2	310×10^6 N/m^2	ASTM D882-64T Method A-100% min
Ultimate Elongation (MD)	120%	40%	ASTM D882-64T Method A-100% min
Tensile Modulus (MD)	3792×10^6N/m^2	5515×10^6N/m^2	ASTM D882-64T Method A-100% min
Impact Strength	$2\cdot3 \ \frac{N\cdot cm}{\mu m}$	$2\cdot3 \ \frac{N\cdot cm}{\mu m}$	Du Pont Pneumatic impact
Bursting Strength (Mullen)	45.5×10^4N/m^2	37.9×10^4N/m^2	ASTM D774-63T
Density	1395 kg/m^3	1377 kg/m^3	ASTM D1505-63T
Refractive Index (Abbe)	1.64nD25	–	ASTM D542-50
Area Factor (sq m/kg/25 μm)	28.45	29.16	Calculation
Melting Point	250 C (480 F)		Fisher-Johns
Service Temperature	-70 C to 150 C (-100 F to 300 F)		
Coefficient of Thermal Expansion	17×10^{-6} C^{-1}		Modified ASTM D696-44 30 to 50 C (86 to 122 F)
Coefficient of Thermal Conductivity (25 μm Type A)	0.150 $\frac{W}{m\cdot C}$		24 to 77 C (75 to 170 F)
Heat Sealability	None unless coated or treated		
Specific Heat	1173 J/kg·C		
Dielectric Strength-Short Term for 25 μm Film	551 kV/mm		500 volts/sec, dc
	295 kV/mm		ASTM D149-64 and D2305-68; 60 Hz
	197 kV/mm		150 C – 60 Hz
Dielectric Constant	3.30		ASTM D150-65T; 60 Hz
	3.25		1 kHz
	3.0		1 MHz
	2.8		1 GHz
	3.7		150 C – 60 Hz
Dissipation Factor	0.0025		ASTM D150-65T; 60 Hz
	0.0050		1 kHz
	0.016		1 MHz
	0.003		1 GHz
	0.0040		150 C – 60 Hz
Volume Resistivity	10^{18} ohm-cm		ASTM D257-66 and D2305-68
	10^{13} ohm-cm		150 C

Mylar (E. I. du Pont de Nemours, Inc.) – Continued

Property	Typical Property Value	Test Method
Surface Resistivity	10^{16} ohms	ASTM D257-68; 23 C-30% R.H.
	10^{12} ohms	23 C-80% R.H.
Insulation Resistance	10^{12} ohms	ASTM D257-66 and D2305-68; 35 C-90% R.H.
Corona Resistance 76 μm (3 mil)	30 hours (single sheet)	Modified ASTM D2275-64T; 3000 VAC. 50 Hz

*All tests were performed at 25 C (77 F) unless otherwise noted.

Flexigard (3M Company)

Flexigard is a durable, flexible, transparent, weather-resistant composite film that is specially designed for solar applications where temperatures do not exceed 77 C (170 F). Flexigard has not had extensive usage to date. Therefore, property data are limited. However, it has been exposed in Florida at 45 degrees south for 10 years with no signs of degradation. It has also been evaluated at the 3M Solar Energy Test Site since November, 1974, with like results. Tensile strength is 145 x 10^6 N/m^2.

The product is available in rolls 1.2 m x 45.7 m x 127 μm at the following prices:

Rolls	Meters^2	Cost/Meter^2
1	56	$3.77
2-4	111	$3.34
5-7	179-390	$3.01
8	446 and over	$2.69

POTTANTS

Epocast 212/9617 (Furane Plastics, Inc.)

Epocast 212/9617 is a two-part general purpose epoxy pottant that cures at room temperature to a clear material. The flexibility of the cured resin can be modified by changing the ratio of resin to hardener used in the cure. Typical properties are presented in the following tabulation.

Property	Property Value
Uncured Resin Properties	
Resin Number	212
Hardener Number	9617
Resin in Mix, parts by weight	100
Hardener in Mix, parts by weight	60 (variable)
Typical Cure, hours/temperature, C	48/27 3/66
Viscosity, $N{\cdot}s/m^2$ at 24 C (75 F)	3.2
Pot Life, min (100 g, 25 C)	85
Cured Resin Properties	
Hardness, Shore D	87/84
Specific Gravity	1.13
Flexural Strength, N/m^2	69×10^6
Weight Loss after 48 hr at 149 C (300 F), percent	3.33
Weight gain after 24-Hr Water Immersion at 25 C (77 F), percent	0.36
Volume Resistivity at 24 C (75 F), ohm-cm	9.9×10^{14}
93 C (200 F)	1.6×10^8
149 C (300 F)	8×10^7
Dielectric Constant at 60 Hz	3.7
10^4 Hz	3.6
10^6 Hz	3.4
Dissipation Factor at 60 Hz	.009
10^4 Hz	.010
10^6 Hz	.021

Epocast 212/9617 is available in 18.1 kg (40 lb) lots for $151.60, 0.21 m^3 (55 gal) drums at $800/drum.

Stycast-1269A (Emerson and Cuming, Inc.)

Stycast 1269A is a two-part, crystal-clear epoxy casting resin well suited to optical applications. When cured, it has outstanding toughness. Where optical clarity is a prime objective, cure should be carried out at a temperature no higher than 88 C (190 F). If a fast cure is required, higher cure temperatures may be used, but the cured material will normally become light amber. When fully cured, Stycast 1269A has good high-temperature properties; some discoloration can be expected when the material is exposed to temperatures above 120 C (250 F). Except for the color change, other properties are unaffected. Stycast 1269A exhibits good adhesion to most materials, and negligible exotherm on curing. Excellent electrical properties coupled with exceptional clarity are the outstanding features of this material.

Stycast 1269A is available at a cost of $8.16 kg. Pertinent property data are tabulated below.

General Properties

Specific Gravity	1.2
Flexural Strength, (N/m^2)	228 x 10^6
Thermal Conductivity, W/m•C	0.272
Coefficient of Thermal Expansion, C^{-1}	75 x 10^{-6}
Dielectric Constant, 10^6 Hz	3.8
Loss Tangent, 10^6 Hz	Below 0.005
Volume Resistivity, ohm-cm	7 x 10^{14}
Dielectric Strength, (kV/mm)	16.9
Hardness, Shore D	
at 25 C (77 F)	85
at 93 C (200 F)	40
Index of Refraction	1.5401

Optical Transmission

Wave-Length Meters, x 10^{-6}	Percent Transmission		
	Stycast 1269A		Pyrex Glass
	4.4 mm	7.2 mm	2.0 mm
0.70	88.5	89.5	91.9
0.65	85.2	87.0	91.9
0.60	83.2	85.0	91.9
0.55	83.0	85.0	91.8
0.50	82.5	85.0	91.8
0.45	81.0	82.8	91.5
0.40	75.9	75.9	91.3
0.35	47.8	37.1	86.2
0.30	0	0	0
0.28	0	0	0

The shelf life is 6 months when stored in unopened containers at temperatures no higher than 25 C (77 F).

RTV 615 (General Electric Company)

RTV 615 is a two-part dimethyl silicone rubber pottant that cures at room temperature. It is "easily pourable" in the uncured state and has ideal dielectric properties for potting applications over a wide temperature range. RTV 615 is available in 200 kg (440 lb) kits at $18.63/kg ($8.45/lb) for 1-3 kits. Greater than 4 kits at $18.04 kg ($8.18/lb). Typical uncured and cured properties are listed below.

Property	Property Value
Typical Uncured Properties	
Color	Clear
Consistency	Easily pourable
Viscosity, $N \cdot s/m^2$	3.0
Shelf Life, months	6
Typical Cured Properties	
Specific Gravity	1.02
Hardness, Shore A Durometer	35
Tensile Strength, N/m^2	6.4×10^6
Elongation, percent	150
Tear Resistance, Die B, N/0.0254 m	111
Brittle Point, C	<-68
Linear Shrinkage, percent	<2
Thermal Conductivity, W/mC at 93 C	0.206
Coefficient of Thermal Expansion, C^{-1} (-18 to +177 C)	270×10^{-6}
Dielectric Strength, kV/mm	19.7
Dielectric Constant at 60 Hz	3.0
Dissipation Factor at 60 Hz	.001
Volume Resistivity, ohm-cm	1×10^{15}

RTV 619 (General Electric Company)

RTV 619 is a low-viscosity liquid which, with the addition of a curing agent, cures to a clear, energy-absorbant gel. It is useful as a dielectric potting material over a wide temperature range. In the uncured state, it pours easily and can be cured at room temperature without exotherm.

The following cure schedule generally is used with RTV 619:

Cure Temperature	Approximate Time
25 C (77 F)	24 hr
65 C (149 F)	1½ hr
100 C (212 F)	30 min
150 C (302 F)	15 min

Typical properties of the uncured and cured resin are presented below.

Typical Uncured Properties

	RTV-619A Base Compound	RTV-619B Curing Agent
Color	Clear	Light blue
Consistency	Easily pourable	Easily pourable
Viscosity, N·s/m^2	0.750	0.050
Specific Gravity	0.97	0.97
Solids, percent	100	100
Shelf Life at 25 C (77 F), months	6	6

Typical Uncured Properties (curing agent added)

Viscosity, N·s/m^2	0.500
Working time, hr	4

Typical Cured Properties

Color	Clear
Specific Gravity	0.97
Penetration	5 mm (Universal Penetrometer, 19.5-gram shaft, 6.35-mm diameter)
Freezing Point, C	<-65
Temperature Effect on Volume	Approximately 1% volume increase by 10 degrees C
Thermal Conductivity, W/mC	0.172
Dielectric Strength - ASTM D-149, kV/mm	19.7
Dielectric Constant - ASTM D-150, 10^3 Hz	3.0
Dissipation Factor - ASTM D-150, 10^3 Hz	0.001
Volume Resistivity - ASTM D-257, ohm-cm	1 x 10^{15}

RTV-655 (General Electric Company)

RTV-655 is a transparent, low-viscosity silicone liquid that cures with the addition of a curing agent to form a rubber-like, tough, transparent solid. It provides environmental protection and mechanical support to encapsulated or fabricated components and assemblies. RTV-655 protects against thermal shock, vibration, moisture, ozone, corona, dust, chemicals, and many other contaminants and, because of its transparency, will permit easy component identification and repair.

RTV-655 is available at \$28.53 kg (\$12.94/lb) in 1-3 kit lots (200 kg/kit). Greater than 4 kit lots are \$27.94/kg (\$12.67/lb). Typical property data for the uncured and cured material are given in the following tabulation.

Property	Property Value
Typical Uncured Properties	
Color	Clear – light straw
Viscosity at 25 C (77 F)	5 Ns/m^2
Consistency	Easily pourable
Solids Content (nominal), percent	100
Specific Gravity at 25 C (77 F)	1.07
Shelf Life at 25 C (77 F)	6 months minimum
Pot Life at 25 C (77 F) (Curing agent added)	4 hr
Refractive Index	1.435
Typical Cured Properties	
Color	4 hr/66 C (150 F) Transparent, clear light straw
Specific Gravity	1.07
ASTM-D676 Durometer, Shore A	35
Thermal Conductivity, W/m·C	>0.172
Temperature Effect on Volume	<1% volume increase per 10 C
Tensile Strength, N/m^2	$5.9\text{-}6.9 \times 10^6$
Elongation	150%
Weight Loss, percent after 1000 hr/200 C (392 F)	3.0
Dielectric Strength-ASTM D-149	19.7 kV/mm
Dielectric Constant-ASTM D-150, 10^3 Hz	3.00
Dissipation Factor-ASTM D-150, 10^3 Hz	.001
Volume Resistivity-ASTM D-257	1×10^{15}

Sylgard 184 (Dow Corning)

Sylgard 184 is a clear, low-viscosity, room-temperature curing dimethyl silicone rubber for use in potting. It is a two-part system and exhibits excellent dielectric properties from -65 to 250 C (-85 to 482 F).

The price of Sylgard 184 resin and catalyst is $18.41/kg. DC 1201 adhesion-promoting primer is $9.26/kg. Pertinent properties of the uncured and cured elastomer are presented below.

Property	Property Value
ASTM D-1298 Specific Gravity at 25 C (77 F)	1.05
ASTM D-1084B Viscosity at 25 C (77 F), N·s/m^2	5.5
Pot Life at 25 C (77 F) with Curing Agent Added, hours	2
Cured Properties	
Color	Clear
ASTM D-2240 Durometer Hardness, Shore A	35
ASTM D-412 Elongation, percent	100
ASTM D-792A Specific Gravity at 25 C (77 F)	1.05
ASTM D-412 Tensile Strength, N/m^2	6.2×10^6
Thermal Conductivity W/m·C	0.146
Linear Coefficient of Thermal Expansion, C^{-1} from -55 to 150 C	3.0×10^{-4}
Volume Expansion, C^{-1} from -55 to 150 C	9.6×10^{-4}
Weight Loss, percent after 1000 hr at 150 C (302 F)	1.6
after 1000 hr at 200 C (392 F)	4.0
ASTM D-570 Water Absorption After 7 Days Immersion at 25 C (77 F), percent	0.10
ASTM D-746 Brittle Point, degrees	-65 C (-149 F)
ASTM D-1218 Refractive Index	1.430
ASTM D-150 Dielectric Constant, at 60 Hz	2.75*
100 kHz	2.75*
ASTM D-150 Dissipation Factor, at 60 Hz	0.001*
100 kHz	0.001*
ASTM D-275 Volume Resistivity, ohm-cm	1×10^{14}
ASTM D-149 Dielectric Strength, kV/mm	21.7*
ASTM D-150 Dielectric Constant, at 60 Hz	2.65**
100 kHz	2.65**
ASTM D-150 Dissipation Factor, at 60 Hz	0.001**
100 kHz	0.001**
ASTM D-257 Volume Resistivity, ohm-cm	2×10^{14}
ASTM D-149 Dielectric Strength, kV/mm	23.6**

*1575 μm specimens cured 4 hr at 65 C (149 F).
**Aged 1000 hr at 200 C (392 F).

SEALANTS

MONO (The Tremco Manufacturing Co.)

MONO is an acrylic terpolymer sealant. It does not need modifiers for adhesion, workability, or long life. The desired sealant characteristics of exceptional adhesion and elasticity are an inherent and permanent part of the basic polymer. They will not migrate or disappear with time as is the case with many other high performing sealants. The sealant is highly weather resistant: resisting ultraviolet, oxygen, moisture, heat, and cold.

MONO is available in 0.3 kg (11 oz) tubes at $2.40/tube in case lots.

Performance characteristics of MONO are detailed in the following tabulation.

Property	Property Value	Test Method
Adhesion-In-Peel	44 to 89 N	TT-S-230a; 19-GP-5b
Staining	None	TT-S-230a; 19-GP-5b
Ultraviolet	Excellent resistance	TT-S-230a; 19-GP-5b
Through Glass	No adhesive failure	
Accelerated Aging	No adhesive, cohesive failures or oil exudation after 5000 hr	ASTM E-42, Method E
Sagging	Passes	TT-S-230a; 19-GP-5b
Curing Time	21 days at 24 C to 51 C (75 F to 120 F)	TT-S-230a
Resistance to Salt Spray	No adhesive or cohesive failure after 200 hr at 40 C (105 F) in 5% salt solution	ASTM E-117-57T
Weight Loss After Heat Aging, percent	12 to 14	TT-S-230a; 19-GP-5b
Durability (bond and cohesion after 4 days water immersion; heat and cold conditioning)	50% extension at -18 C (0 F) Passes requirements on mortar, glass, and aluminum	TT-S-230a
Gunnability	Flow rate more than 20 g in 2 min through 2.54-mm orifice 41 x 10^4 N/m^2, 25 C (77 F)	

Tremco 440 Preshimmed Tape (Tremco Manufacturing Co.) – Sealant Type

Tremco 440 tape is formulated from 100 percent solids, combining butyl and polyisobutylene. It contains no solvent or other volatiles. It is not recommended for use in joints subjected to prolonged periods of water submersion or temperatures exceeding 93 C (200 F).

440 Tape is supplied in different length rolls in the following sizes and shapes: standard sizes (thickness and width): 3.175 mm by 9.525 mm; 3.175 mm by 12.7 mm; 4.7625 mm by 12.7 mm. Other sizes such as 6.35 mm by 12.7 mm available on special order. Comes in aluminum and black/bronze colors.

The cost of Tremco 440 Tape is $0.535/roll for a 4.7625 mm x 12.7 mm x 7.6 m tape (3/16 x 1/2 x 25 ft) roll in case lots of 20 rolls per case.

Performance characteristics of 440 Tape are given below.

	Test Method	Result
Dynamic Movement and Vibration	Voss Tester (as described in Canadian Spec. 19-GP-5) 6.35-mm to 12.7-mm preshimmed Tremco 440 Tape cycled 100,000 times at laboratory conditions and 100,000 times at 71 C (160 F) using infrared lamp	No pumping, no sagging, no significant effect on adhesion
Dynamic Movement	Specimen forming a joint 4.7625-mm wide, 12.7-mm deep, and 152.4-mm long is compressed and extended 85 cycles each at 25%, 50%, and 100% extension and compression	No adhesive or cohesive failure
Adhesion	Tested on steel, aluminum, glass, and concrete after 14 days of water immersion, 14 days of conditioning at 82 C (180 F) and 14 days ultraviolet exposure	Excellent
Heat Resistance	Specimen conditioned at 135 C (275 F)	No oil exudation, blistering, flow or loss of adhesion
Squeeze-out	Specimen subjected to dynamic extension and compression, also to static and dynamic test at University of Miami	Very nominal under severe conditions
Accelerated Aging	1000 hr exposure in accelerated test unit (equivalent to 6-10 years exposure)	Adhesion still excellent: 20 to 30 Shore A hardness

Tremco 440 Preshimmed Tape – Tremco Manufacturing Co. – Sealant Tape – Continued

	Test Method	Result
Low-temperature Flexibility	152.4-mm length of tape conditioned at 88 C (190 F) for 14 days, then to -29 C (-20 F) and bent 180 degrees around a mandrel.	No loss of adhesion; no cracking
Compatibility		Compatible with all Tremco Sealants recommended for glazing, setting panels or sealing mullions

Vistalon 404 (Exxon Chemical Company)

Vistalon 404 is an ethylene-propylene copolymer for use in sealant applications. Because it is a completely saturated hydrocarbon elastomer, it can be formulated for use in a wide variety of applications in which service aging is critical. The material has exceptional ozone and heat resistance, is resistant to weather, sunlight, and chemicals, has low compression set, good resilience and good low-temperature flexibility.

The performance of peroxide-cured, filled Vistalon 404 is presented below.

Formulation

Vistalon 404	100
AgeRite Resin D	2
FEF Black	60
Zinc Oxide	5
TAC-75	2
DiCup 40 C	10
Specific Gravity	1.12

Processability Properties

Mooney Scorch at 121 C (250 F) (MS) +10, min	30
132 C (270 F) (MS) +3, min	9
Mooney Viscosity, ML 1 + 8 100 C (212 F)	90

Physical Properties

Hardness, points	68
100% Modulus, N/m^2	4.3 x 10^6
200% Modulus, N/m^2	11.4 x 10^6
Tensile Strength, N/m^2	12.4 x 10^6
Ultimate Elongation, percent	230
Compression Set, Method B, plied	
70 hr at 100 C (212 F), percent	11
22 hr at 120 C (302 F), percent	14

Physical Properties, Aged

Air Oven, 70 hr at 150 C (302 F), ASTM D573	
Hardness, points change	+5
Tensile Strength, percent change	-1
Ultimate Elongation, percent change	+4
Air Oven, 70 hr at 175 C (347 F), ASTM D573	
Hardness, points of change	+5
Tensile Strength, percent change	-28
Ultimate Elongation, percent change	-26

Lasto-Meric Liquid Polymer Sealant
(Tremco Manufacturing Co.)

Lasto-Meric is a specially-formulated 100 percent polysulfide liquid polymer base sealant.

Lasto-Meric is a nonshrinking, nonoxidizing elastic sealant for glazing and resealing all types of panels and curtain wall construction. It resists long-term exposure to sunlight and will not stain most masonry surfaces.

This long-lasting sealant bonds well to all types of masonry, metal, glass and wood. Lasto-Meric resists cyclical movement without loss of adhesion or cohesion – it remains flexible in a temperature range from -51 to 93 C (-60 to 200 F). The material returns to its original shape and dimensions after periods of deformation.

Gun-grade consistency, Lasto-Meric is designed for general use in caulking, glazing, and sealing, and for filling expansion joints. A two-part compound, Lasto-Meric is blended together before application. After proper mixing, the compound begins to cure and eventually becomes a firm, resilient rubber.

Joints sealed with Lasto-Meric are weatherproof, watertight and permanently flexible regardless of temperature, moisture, or exposure to solar radiation. Porous masonry surfaces should be primed with Tremco No. 1 Primer prior to applying Lasto-Meric.

SHEET/TUBING

Plexiglas (Rohm and Haas Company)

Plexiglas is the Rohm and Haas Company trademark for a family of thermoplastic acrylic sheets and molding powders for injection molding and extrusion. Among the many desirable properties of Plexiglas, two are preeminent:

- Unexcelled durability indoors and outdoors
- Superlative optical properties and clarity.

Resin properties are typified by those of Plexiglas V(811) which is available with varying degrees of ultraviolet transmission.

Average Physical Properties of Plexiglas Molding Pellets

Property	Test Conditions	Property Value
Refractive Index	ASTM D-542-50	1.49
Specific Gravity	ASTM D-792-64T	1.19
Tensile Strength	ASTM D-638-64T 6.35 mm specimen (0.8 x 10^{-4} m/s) maximum, N/m^2	72 x 10^6
Flexural Strength	ASTM D-790-66 Span-depth ratio 16 (0.4 x 10^{-4} m/s) maximum, N/m^2	110 x 10^6
Compressive Strength	ASTM D-695-68T (0.8 x 10^{-4} m/s) maximum, N/m^2	117 x 10^6
Impact Strength	ASTM D-256-56 (1961) Izod molded notch (per 0.0254 m of notch) J	0.5
Rockwell Hardness	ASTM D-785-62	M-97
Light Transmission "As Received"	ASTM D-1003-61 Total white, percent	92
Effect of Accelerated Weathering on Appearance of Clear Material	LY 406a-6024 (240 hr)	
	Crazing	None
	Discoloration	None
	Warping	None
	Unmolding	None
Deflection Temperature Under Load, unannealed	ASTM D-648-56 (1961) 2 C (3.6 F)/min, 182 x 10^4 N/m^2	92 C (198 F)
Melt Flow Rate by Extrusion Plastometer	ASTM D-1238-63T g/10 min	
–Condition H		1.2
–Condition I		5.0

Plexiglas (Rohm and Haas Company) – Continued

Property	Test Conditions	Property Value
Flow Temperature	ASTM D-569-59 (1961)	160 C (320 F)
Vicat Softening Point, 2 C (3.6 F)/min, 254-μm penetration, 1000-g load	ASTM D-1525-58T, C	110 C (230 F)
Shrinkage From Mold Dimension	ASTM D-955-51 (1961) (Cold mold to cold piece) mm/m 48 hr	2-6
Dielectric Strength	ASTM D-149-64 kV/mm	19.7
Dielectric Constant	ASTM D-150-68, 60 Hz	3.7
Power Factor	ASTM D-150-68, 60 Hz	0.05
Loss Factor	ASTM D-150-68, 60 Hz	0.19
Arc Resistance	ASTM D-495-61, sec	No tracking
Flammability	ASTM D–635–68 burning rate, m/s	3.0×10^{-4}
Water Absorption	ASTM D-570-63 Wt gain on 24-hr Water immersion	0.3
	Dimensional change on immersion, percent	None

Plexiglas G is the standard type of cast plexiglas sheet. Plexiglas G sheet 1.2 x 2.4 meters is available at \$11.19/m^2 in 3.175-mm thickness with masking on both sides or at \$10.11/m^2 with interlayers.

Plexiglas II UVA (ultraviolet absorbing) sheets have the same general properties as Plexiglas G but are manufactured to more exacting standards of optical quality. The cost of 3.175-mm (1/8 inch) sheet is \$29.81/m^2 (\$2.77/ft^2); 6.35 mm (1/4 inch) sheet costs \$41.64/m^2 (\$3.87/ft^2).

Average physical properties, applicable to both Plexiglas G and II UVA sheet materials, are given below.

Average Physical Properties of Plexiglas Sheet

Property	ASTM Method(2)	Property Value
Thickness, mm		6.35
Specific Gravity	D792	1.19
Refractive Index	D542	1.49
Light Transmittance and Haze, percent	D1003	
"As Received" – parallel		91
– total		92
– haze		1

Plexiglas (Rohm and Haas Company) – Continued

Property	ASTM Method(2)	Property Value
After 5-Yr Outdoor Exposure, Bristol, Pa., 45 deg. angle facing South, percent		
– parallel		90
– total		92
– haze		2
After 240-Hr Artificial Exposure, Carbon Arc Type, per ASTM G-23, percent		
– parallel		90
– total		92
– haze		2
Artificial Weathering, Fluorescent Sunlamp With Dew, 10 cycles, 240-hr Exposure – crazing	D1501 or Fed. Test Std. 406, Method 6024	None
– warping		None
Instrumental Measurement, Change In Yellowness Index After Artificial Weathering	D1925	1.0
Ultraviolet Transmission, 320 nanometers, percent	Beckman DU-792	0
Tensile Strength (6.35 mm Specimen-0.8×10^{-4} m/s)	D638	
Maximum, N/m^2		72×10^6
Rupture, N/m^2		72×10^6
Elongation Maximum, percent		4.9
Elongation Rupture, percent		4.9
Modulus of Elasticity, N/m^2		3100×10^6
Poisson's Ratio		0.35
Flexural Strength (Span Depth Ratio 16, 0.4×10^{-4} m/s)	D790	
Maximum, N/m^2		110×10^6
Rupture, N/m^2		110×10^6
Modulus of Elasticity, N/m^2		3100×10^6
Impact Strength		
Izod Milled Notch, J/0.0254 m of notch		0.5
Rockwell Hardness	D785	M-104
Thermal		
Hot Forming Temperature, C		143-182 (290-360 F)
Maximum Recommended Continuous Service Temperature, C		82-93 (180-200 F)

Plexiglas (Rohm and Haas Company) – Continued

Property	ASTM Method(2)	Property Value
Coefficient of Thermal Expansion, $C^{-1} \times 10^{-5}$	R&H P4A	
-40 C (-40 F)		5.0
-29 C (-20 F)		5.2
-18 C (0 F)		5.6
-7 C (20 F)		5.9
4 C (40 F)		6.5
16 C (60 F)		7.0
27 C (80 F)		7.6
38 C (100 F)		8.3
Coefficient of Thermal Conductivity, $\frac{W}{m \cdot C}$	Cenco-Fitch	0.186
Specific Heat at 25 C (77 F), $\frac{J}{kg \cdot C}$		1454
Electrical		
Dielectric Strength, Short Time Test, kV/mm	D149	19.7
Dielectric Constant	D150	
60 Hz		3.7
1,000 Hz		3.3
1,000,000 Hz		2.5
Power Factor	D150	
60 Hz		0.05
1,000 Hz		0.04
1,000,000 Hz		0.03
Loss Factor	D150	
60 Hz		0.19
1,000 Hz		0.13
1,000,000 Hz		0.08
Arc Resistance	D495	No tracking
Volume Resistivity, ohm/cm	D257	6×10^{17}
Surface Resistivity, ohm/square	D257	6×10^{18}
Water Absorption (weight gain) After Immersion, percent for:	D229 and D570	
1 day		0.2
2 days		0.3
7 days		0.4
28 days		0.8
56 days		1.1
84 days		1.3

Lucite (E. I. du Pont de Nemours, Inc.)

Lucite acrylic resin is a thermoplastic noted for many desirable qualities: clarity, outdoor durability, unique light-transmission characterisitics, light weight, and shatter resistance. It is available in injection molding and extrusion grades and as preformed sheet. Property information for Lucite 140 resin, a medium-molecular-weight composition priced at \$1.32 kg (\$0.60/lb) and well-suited for injection molding and extrusion processing with good heat resistance, is given below.

Properties of Molded "Lucite" 140 Acrylic Resin

Property	ASTM Method	Property Value
Tensile Strength, N/m^2 3.175 mm thick		
-57 C (-70 F)	D638	100 x 10^6
23 C (73 F)	D638	72 x 10^6
70 C (158 F)	D638	>34 x 10^6
Tensile Elongation, percent		
-57 C (-70 F)	D638	2
23 C (73 F)	D638	3-5
70 C (158 F)	D638	80
Tensile and Flexural Modulus of Elasticity 23 C (73 F), N/m^2	D638	2758-3447 x 10^6
Shear Strength, N/m^2	D732	65 x 10^6
Impact Strength, Izod, Milled Notch, 6.35-mm Bars 23 C (73 F) J/0.0254 m	D256	0.4
Stiffness 23 C (73 F) N/m^2	D747	2965 x 10^6
Flexural Strength 23 C (73 F) N/m^2	D790	110 x 10^6
Hardness, Rockwell	D785	M95
Coefficient of Linear Thermal Expansion -18 to 38 C (0-100 F, average) C^{-1}	D696	3.4 x 10^{-5}
Thermal Conductivity, W/m·C	Cenco-Fitch	0.200
Specific Heat, J/kg·C		1454
Deformation Under Load 14 x 10^6 N/m^2 24 hr, 50 C (122 F), percent	D621	0.3-0.4
Deflection Temperature		
182 x 10^4 N/m^2, C	D648	92 (198 F)
45.5 x 10^6 N/m^2, C	D648	99 (210 F)
Dielectric Strength, Short Time, 3.175 mm, kV/mm	D149	15.7
Arc Resistance	D495	No tracking
Volume Resistivity, ohm-cm	D257	>10^{15}
Dielectric Constant		
60 Hz	D150	3.5
10^3 Hz	D150	3.2
10^6 Hz	D150	2.7

Property	ASTM Method	Property Value
Dissipation Factor		
60 Hz	D150	0.06
10^3 Hz	D150	0.04
10^6 Hz	D150	0.02
Index of Refraction, n_o	D542	1.49
Luminous Transmittance, 3.175 mm, percent	E308	92
Haze, percent	D1003	<3
Water Absorption, 24 hr, percent	D570	
Weight Gain Plus Soluble Matter Loss		0.3
Soluble Matter Loss		<0.1
Flammability, 3.175 mm, mm/s	D635	0.38-0.51
Specific Gravity	D792	1.19
Mold Shrinkage	D551	0.003-0.007

"Lucite" cast acrylic sheet is available as a linear (L) and cross-linked (XL) composition. Both have excellent optics, high impact resistance, excellent weatherability, and close thickness tolerances. Either is suitable for most glazing applications; however, "Lucite" XL sheet offers advantages in solvent resistance. The high-temperature elastic strength of "Lucite" XL minimizes the probability of tear during thermoforming operations. "Lucite" L is easily cemented, using appropriate solvents.

It is available in clear sheet sizes of 91 cm x 91 cm up to 284 cm x 381 cm, cut to size within these limits; it has a nominal thickness range of 3.175 to 6.35 mm. Costs in 13,605 kg minimum quantities are \$1.72/kg (3.175 mm), \$2.25/kg (4.75 mm), and \$2.74/kg (6.35 mm). Average physical properties of "Lucite" L and XL are given below.

Property Data – "Lucite" L and XL

Property	ASTM	"Lucite" L and XL
Specific Gravity	D792	1.19
Refractive Index	D542	1.49
Light Transmittance	D1003	
Parallel		91%
Total		92%
Haze		1%
Spectral Transmission 290 to 330 nm, 6.35 mm Sheet, max percent	Beckman DU-792	5%
Tensile Strength	D638	
Rupture		74 x 10^6 N/m^2
Modulus of Elasticity		2944 x 10^6 N/m^2
Elongation at Rupture		4.5%

Lucite (E. I. du Pont de Nemours, Inc.) – Continued

Property	ASTM	"Lucite" L and XL
Flexural Strength	D790	
Rupture		103×10^6 N/m^2
Modulus of Elasticity		2944×10^6 N/m^2
Rockwell Hardness	D785	M-100
Hot Forming Temperature		135-175 C (275-350 F)
Heat Distortion Temperature, C (182×10^4 N/m^2)	D648	95 (203 F)
Coefficient of Thermal Expansion, C^{-1} (average value)	D696	7.0×10^{-5}
Maximum Recommended Continuous Service Temperature, C		80 (176 F)
Coefficient of Thermal Conductivity, W/m·C		0.207
Shrinkage, max percent		2.5
Specific Heat, J/kg·C		1454
Surface Resistivity, ohm, 75% RH	D257	$>10^{16}$
Volume Resistivity, ohm-cm	D257	10^{15}
Dielectric Strength, kV/mm Short-Time Test	D149	20
Dielectric Constant	D150	
60 Hz		4
10^3 Hz		4
10^6 Hz		3
Power Factor	D150	
60 Hz		0.06
10^3 Hz		0.04
10^6 Hz		0.02
Arc Resistance	D495	No tracking
Water Absorption (Wt Gain on Immersion For 24 Hr), percent	D570	0.3
Odor		None
Taste		None

XT-365 (American Cyanamid Company)

XT-365 is a clear, tough, rigid, modified acrylic molding compound. It is referred to as an acrylic-based multipolymer. American Cyanamid's cost for this resin is \$1.12/kg (\$0.51/lb) in truck-load quantities. Typical property data for the bulk resin are given in the following tabulation.

Property	ASTM Method	Property Value
Color		Transparent
Light Transmission, percent	D-307-44	87
Haze, percent	D-1003-52	9
Izod Impact Strength,	D-256-56	
Notched		
J/0.0254 m of notch		
(6.35 mm bar)		
23 C (73 F)		2.0
0 C (32 F)		1.6
-40 C (-40 F)		1.2
Tensile Strength, N/m^2	D-638-60T	48×10^6
Tensile Modulus, N/m^2	D-638-60T	2600×10^6
Tensile Elongation, percent	D-638-60T	28
Flexural Strength, N/m^2	D-790-59T	76×10^6
Flexural Modulus, N/m^2	D-790-59T	2400×10^6
Compressive Strength, N/m^2	D-695-54	65×10^6
Rockwell Hardness	D-785-60T	R114
		M 45
Deflection Temperature, C (182×10^4 N/m^2)	D-648-56	86 (186 F)
Coefficient of Thermal Expansion, C^{-1}	D-696-44	9.0×10^{-5}
Specific Gravity	D-792	1.1
Water Absorption, percent	D-570-59T	
24 Hr at 23 C (73 F)		0.3
Weight Change, percent		
One Week Immersion at 23 C (73 F)		
Water		0.6
Dielectric Constant at:	D-150	
100 Hz		3.25
1,000 Hz		3.21
1,000,000 Hz		2.82
Dissipation Factor at:		
100 Hz		0.028
1,000 Hz		0.30
1,000,000 Hz		0.023

Lexan (General Electric Company)

Lexan polycarbonate molding resin offers the toughness and performance of many metals, yet provides the processing and design advantages of an engineering thermoplastic. It provides impact strength and support unmatched by any other thermoplastic. It is available in a number of grades as sheet material in sizes ranging from 61 x 122 cm (24 x 48 in.) to 183 x 244 cm (72 x 96 in.), and in thicknesses from 3.175 to 12.7 mm (1/8 to 1/2 in.). Lexan 9030 sheet is the standard outdoor glazing grade. It has outstanding resistance to the detrimental effects of sunlight, rain, erosion, atmospheric chemicals, and temperature change.

Typical resin properties are listed below.

Property Data – Lexan Resin

Property	Property Value
Specific Gravity	1.20
Tensile Strength, N/m^2	62×10^6
Izod Impact Strength J per 0.0254-m Notch	21.7
Coefficient Thermal Expansion $C^{-1} \times 10^{-5}$	6.75
Heat Deflection Temperature, C	
(at 45.5×10^4 N/m^2)	138 (280 F)
(at 182×10^4 N/m^2)	132 (270 F)
UL Continuous Use Temperature, C	115 (239 F)
Flexural Modulus, N/m^2	2344×10^6
Percent Loss Apparent Modulus 14×10^6 N/m^2 1000 hr 23 C (73 F)	16
Percent Loss Apparent Modulus 14×10^6 N/m^2 1000 hr 93 C (200 F)	18
Dielectric Strength, kV/mm	15

Lexan (General Electric Company) – Continued

Property Data – Lexan 9030 Sheet

Property	Property Value
Weight, 3.175-mm Sheet, kg/m^2	3.81
4.763-mm Sheet, kg/m^2	5.71
6.350-mm Sheet, kg/m^2	7.62
9.525-mm Sheet, kg/m^2	11.42
12.70-mm Sheet, kg/m^2	15.23
UV Transmission at 0.385 μm, percent	<0.1
UV Transmission at 0.400 μm, percent	50
Tensile Strength, N/m^2	65 x 10^6
Elongation, percent	110
Compressive Strength, N/m^2	86 x 10^6
Flexural Strength, N/m^2	93 x 10^6
Modulus of Elasticity, N/m^2	2378 x 10^6

Lexan 9030 is available in 1.22 x 2.44 m (48 x 96 in.) sheet. At 3.175 mm (1/8 in.), cost is \$25.72/m^2 (\$2.39/ft^2).

Tuffak (Rohm and Haas Company)

Tuffak polycarbonate is a lightweight, high-impact sheet that transmits 82-89 percent of incident light depending on sheet thickness. It is available in 1.8 x 2.7 m (6 x 9 ft) and 2.7 x 3.7 m (9 x 12 ft) sheets and sheet thicknesses from 1.5875 mm (1/16 in.) to 12.7 mm (1/2 in.).

Average typical properties of Tuffak are given in the following tabulation.

Property	ASTM Method	Property Value
Specific Gravity	D-792	1.2
Optical		
Refractive Index	D-542	1.586
Light Transmittance, percent	D-1003	85-91
Haze, percent	D-1003	0.5-2.0
Tensile Strength, N/m^2		
Ultimate	D-638	66×10^6
Yield	D-638	58×10^6
Elongation, percent	D-638	100
Flexural Strength, N/m^2	D-790	93×10^6
Flexural Modulus, N/m^2	D-790	2300×10^6
Compressive Strength, 0.2×10^{-4} m/s, N/m^2	D-695	86×10^6
Impact Strength		
Izod (3.175 mm notched), J/0.0254 m of notch	D-256	21.7
Rockwell Hardness	D-785	R 118 M 70-78
Thermal		
Heat Deflection Temperature, C		
Under Load, 182×10^4 N/m^2	D-648	135 (275 F)
45.5×10^4 N/m^2		141 (285 F)
Coefficient of Thermal Expansion, C^{-1}	D-696	6.8×10^{-5}
Coefficient of Thermal Conductivity, $\frac{W}{m \cdot C}$	C-177	0.193
Specific Heat, J/kg•C		1246
Electrical		
Dielectric Strength, Short-Time, Test (at 3.175-mm thick), kV/mm	D-149	15.0 at 25 C 17.7 at 100 C
Dielectric Constant	D-150	
60 Hz		2.9
1,000,000 Hz		2.9
Volume Resistivity 23 C (73 F), ohm-cm	D-257	10^{14}
Miscellaneous		
Water Absorption, percent	D-570	
Equilibrium at 23 C (73 F)		0.35
Equilibrium at 100 C (212 F)		0.58

Sun-Lite (Premium Grade) (Kalwall Corporation)

Sun-Lite is a specially designed cover material for solar collectors. It is a fiberglass-reinforced polyester available in regular and premium grades. It features solar properties equivalent to or better than those of glass, low cost, low thermal expansion, light weight, and good strength. It is available in 1.2 m (4 ft) and 1.5 m (5 ft) widths up to 366 m (1200 ft) long in thicknesses of 635-1016 μm (0.025-0.040 in.). Costs are \$3.01 and \$4.95/m^2 for the thicknesses. Property data for the premium grade are presented below.

Average Property	Method	Property Value
Solar Energy Transmittance, percent	E 424 Method B	85-90
Estimated Solar Lifetime, yr		20
Thermal Sensitivity at 93 C (200 F)		Excellent
at 149 C (300 F)		Good
Heat Transmittance, percent	5-20 microns	10
Index of Refraction	D 542	1.52
Tensile Strength, N/m^2	D 638	76×10^6
Flexural Strength, N/m^2	D 790	152×10^6
Flexural Modulus, N/m^2	D 790	4100×10^6
Shear Strength, N/m^2	D 732	83×10^6
Izod Impact, J/0.025 m	D 256	13.6
Water Absorption, percent	D 570	0.50-0.60
Thermal Expansion, C^{-1} x 10^{-5}	D 696	2.5
Thermal Conductivity, W/m·C	C 177	0.124
Specific Heat, J/kg·C	D 2766	1454
Specific Gravity	D 792	1.4
Weight, kg/m^2	NBS PS53	0.85-1.43

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