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Comprehensive Testing to Measure the Response of Fluorocarbon Rubber (FKM) to Hanford Tank Waste Simulant

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**COMPREHENSIVE TESTING TO MEASURE THE RESPONSE OF
FLUOROCARBON RUBBER (FKM) TO HANFORD TANK WASTE SIMULANT**

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Abstract

This report presents the findings of the Chemical Compatibility Program developed to evaluate plastic packaging components that may be incorporated in packaging mixed-waste forms for transportation. Consistent with the methodology outlined in this report, we performed the second phase of this experimental program to determine the effects of simulant Hanford tank mixed wastes on packaging seal materials. That effort involved the comprehensive testing of five plastic liner materials in an aqueous mixed-waste simulant. The testing protocol involved exposing the materials to ~143, 286, 571, and 3,670 Krad of gamma radiation and was followed by 7-, 14-, 28-, 180-day exposures to the waste simulant at 18, 50, and 60°C. Fluorocarbon (FKM) rubber samples subjected to the same protocol were then evaluated by measuring seven material properties: specific gravity, dimensional changes, mass changes, hardness, compression set, vapor transport rates, and tensile properties. From the analyses, we determined that FKM rubber is not a good seal material to withstand aqueous mixed wastes having similar composition to the one used in this study. We have determined that FKM rubber has limited chemical durability after exposure to gamma radiation followed by exposure to the Hanford tank simulant mixed waste at elevated temperatures above 18°C.

ACKNOWLEDGMENTS

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INTRODUCTION

Hazardous and radioactive materials packaging is designed to facilitate the transport and storing of materials without posing a threat to the health or property of the general public. U.S. regulations establish general design requirements for such packagings. While no regulations have been written specifically for mixed waste packaging, regulations for the constituents of mixed wastes, that is, hazardous and radioactive substances, have been codified by the U.S. Department of Transportation (U.S. DOT, 49 CFR 173) and the U.S. Nuclear Regulatory Commission (NRC; 10 CFR 71). The materials of the packaging and any contents must be chemically compatible. Furthermore, Type A [49 CFR 173.412 (g)] and Type B (10 CFR 71.43) packaging design requirements stipulate that there be no significant chemical, galvanic, or other reaction between the materials and contents of the package.

Based on the federal requirements, a Chemical Compatibility Testing Program was developed in the Transportation Technology Department at Sandia National Laboratories/New Mexico (SNL/NM). The program attempts to assure any regulatory body that the issue of certain packaging material compatibility towards hazardous and radioactive materials has been addressed. This program was detailed in a 1993 milestone report¹ submitted to the Department of Energy (DOE). The results of this program were reported to the DOE in various unpublished milestone documents and in a number of externally published papers.²⁻⁶

The milestone report *Chemical Compatibility Test Plan and Procedure Report* (CCTP&PR) describes a program to evaluate plastic transportation packaging components that may be used in transporting mixed waste forms. Consistent with the methodology in the CCTP&PR, the first phase of this experimental program has been completed. This effort involved screening 10 plastic materials in four simulant mixed waste types.⁷ All materials that include "rubber" in their names are used as seals; the others are used as liners. These plastics were as follows:

Seals

- butadiene-acrylonitrile copolymer rubber (nitrile),
- epichlorohydrin rubber (EPI)
- isobutylene-isoprene copolymer rubber (Butyl),
- ethylene-propylene rubber (EPDM),
- fluorocarbon (FKM) rubber, and
- styrene-butadiene (SBR) rubber

Liners

- cross-linked polyethylene (XLPE),
- high-density polyethylene (HDPE),
- fluorocarbon (Kel-F™)
- polytetrafluoroethylene (Generically PTFE or Teflon®),
- polypropylene (PP).

The selected simulant mixed wastes were

- aqueous alkaline mixture of sodium nitrate and sodium nitrite,
- chlorinated hydrocarbon mixture,
- simulant liquid scintillation fluid, and
- mixture of ketones.

The first phase of the testing protocol involved exposing the materials to 286,000 rad (286 Krad) of gamma radiation followed by 14-day exposures to the waste types at 60°C. After radiation and chemical exposure, the seal or rubber materials were tested using Vapor Transport Rate (VTR) measurements, while the liner materials were tested using specific gravity as a metric. For these tests, screening criteria of ~1 g/hr/m² for VTR and a specific gravity change of 10% were used. Materials that failed to meet these criteria for all four types of waste were judged to have failed the screening tests and were excluded from the next phase of this experimental program. Based on this work, it was concluded that while all seal materials passed exposure to the aqueous simulant mixed waste, EPDM and butyl rubber had the lowest VTRs. In the chlorinated hydrocarbon simulant mixed waste, only FKM rubber passed the screening tests. This means that only FKM rubber would be selected for further testing in the chlorinated hydrocarbon simulant. In both the simulant scintillation fluid mixed waste and the ketone mixture simulant mixed waste, none of the seal materials met the screening criteria. For specific gravity testing of liner materials, the data showed that while all materials passed the screening criteria in the aqueous simulant, Kel-F™, HDPE, and XLPE offered the greatest resistance to the combination of radiation and chemicals.

The next phase of this program was the comprehensive testing of liner materials in the aqueous simulant mixed waste. Since screening tests showed that all liner materials met the screening criteria when exposed to the aqueous simulant mixed waste, the five liner materials (HDPE, XLPE, PP, Kel-F™, and PTFE) were subjected to comprehensive testing. The testing protocol

involved exposing the materials to ~143, 286, 571, and 3,670 Krad of gamma radiation followed by 7-, 14-, 28-, and 180-day exposures to the waste simulant at 18, 50, and 60°C. The radiation exposure values were calculated based on γ -ray dose rate data available to us for the components of a pump submerged in a specific storage tank at Westinghouse Hanford Co. These data indicate a maximum γ -ray dose rate in the range of 750 to 850 R/hr. The maximum dose rate of 850 rad/hr was used in calculating the dose that container materials will receive from a ^{60}Co source at Sandia National Laboratories. Using this dose rate, the four doses described above were calculated for 7-, 14-, 28-, and 180-day exposures, respectively. From the data analyses, the fluorocarbon Kel-F™ was identified as having the greatest chemical durability after having been exposed to gamma radiation followed by exposure to the Hanford Tank simulant mixed waste. The most striking observation from this study was the extremely poor performance of PTFE when exposed to the higher radiation doses. Even at lower radiation exposures, PTFE exhibited significant losses in performance. These results were reported as a Sandia Report.⁸ We also published a synopsis of these test results in the proceedings of MIXED WASTE '97.⁹

In this report, we present the results of the second phase of testing in this program. It should be recalled that since all seal materials passed the screening tests in the aqueous simulant mixed waste, all seal materials would be subjected to comprehensive testing. While earlier studies investigated the response of EPDM and butyl rubber, this study involved the comprehensive testing of FKM rubber. The results of comprehensive testing of EPDM and butyl rubber have been reported to the DOE. A synopsis of the comprehensive test results for EPDM and butyl rubber was presented at the Fourth Biennial Mixed Waste Symposium⁹ and at PATRAM '98.¹⁰ The comprehensive testing protocol involved exposing FKM to a matrix of four gamma radiation doses (~ 143, 286, 571, and 3,670 Krad), three temperatures (18, 50, and 60°C), four exposure times (7, 14, 28, and 180 days) in the aqueous simulant. The temperature and exposure times were based on values found in 49 CFR 173, Appendix B. It should be mentioned that while some FKM rubber samples were exposed to only the aqueous simulant, other samples were only irradiated, and still others were irradiated and then exposed to the simulant to mimic the action of mixed wastes. Following exposure to these conditions, the FKM rubber samples were evaluated by measuring seven material properties: specific gravity, dimensional changes, mass changes, hardness, compression set, VTR, and tensile properties.

EXPERIMENTAL

In this section, we describe the experimental aspects of the comprehensive phase of the chemical compatibility testing program for elastomeric materials.

Materials

The selected material, FKM rubber, is an elastomer having known chemical resistance to brake fluids, alcohols, and water. FKM rubber is also known by various tradenames. The most widely recognized product is VITON® manufactured by DuPont. Appendix A provides additional information on FKM, including its properties.

Simulant Preparation

The simulant mixed waste form used in this testing phase was an aqueous alkaline simulant Hanford Tank waste developed locally based on more complex formulations used by researchers at the Hanford site. It was prepared by dissolving 536 g (6.3 moles) of sodium nitrate and 150 g (2.2 moles) sodium nitrite in deionized water (1800 mL) using a 4-L beaker. After these salts had completely dissolved, 246 g (6.2 moles) sodium hydroxide was added under stirring and slight heating using a magnetic hotplate (Corning, Model PC-320). To this hot (~70°C) stirred solution, 51 g (0.30 moles) cesium chloride and 48 g (0.29 moles) strontium chloride were added. Finally, 96 g (0.9 moles) of sodium carbonate dissolved in 800 mL of deionized water was added to the solution. This latter addition resulted in the formation of a copious amount of white precipitate. Based on its insolubility, it is believed that this precipitate is strontium carbonate. To the resulting mixture was added another 400 mL of deionized water to bring the total volume of water used to 3 L. After cooling to near ambient temperature, the stirred mixture was stored in amber glass bottles (Fisher Scientific, 03-327-6). All chemicals used in the preparation of the waste simulant were American Chemical Society reagent grade chemicals. The above composition produced a mixture with the following chemical concentrations:

- 2.1 Molar (M) sodium nitrate
- 0.7 M sodium nitrite
- 2.1 M sodium hydroxide

0.3 M sodium carbonate
0.1 M cesium chloride
0.1 M strontium chloride

Sample Preparation

Standardized test methods were used to cut, condition, and test the materials. The geometry of the material samples was specified by the test method. The use of a press and dies permitted the cutting of multiple samples having uniform dimensions. The samples were cut using an expulsion press (Part 22-16-00) and dies manufactured by Testing Machines, Inc., Amityville, NY. For example, the rectangular (1" \times 2" \times 0.125", 2.5 cm \times 5.0 cm \times 0.318 cm) samples required for specific gravity and hardness measurements were cut in the expulsion press fitted with an expulsion straight edge die (Part 23-10-06). Rectangular (1" \times 3" \times 0.125", 2.5 cm \times 7.6 cm \times 0.318 cm) samples required for dimensional and mass measurements were cut in the expulsion press fitted with an expulsion straight edge die (Part 23-10-07). Circular (0.5" diameter \times 0.125" thick, 1.3 cm diameter \times 0.318 cm thick) discs required for compression set measurements were cut in the expulsion press fitted with a custom circular cutter from CCS Instruments, Akron, OH. Larger circular (2.69" diameter \times 0.125" thick, 6.83 cm diameter \times 0.318 cm thick) discs required for VTR measurements were cut in the expulsion press fitted with an expulsion die (Part 23-00-00) specifically designed for American Society for Testing and Materials (ASTM) Standard Test Method D 814¹¹ testing. Similarly, the Type C tensile samples required for tensile testing were cut in the expulsion press fitted with an expulsion die (Part 23-14-08) specifically designed for the ASTM D 412-Method A.¹²

An identification code for samples was developed to uniquely indicate the test type, sample number, and testing conditions. The brown FKM rubber samples were individually labeled using indelible ink marking pens. As recommended by ASTM D 1349,¹³ the plastics were conditioned at a standard temperature of 73.4°F (23°C) and relative humidity of 50% for at least 24 hours prior to the testing. This was done by storing the cut samples in a desiccator filled with magnesium nitrate hexahydrate (500 g) saturated with water. A humidity/temperature sensor was used to monitor the conditions in the desiccator. Procedures for generating this constant relative humidity environment are described in ASTM E 104.¹⁴ During conditioning,

the samples were stacked atop each other and separated from each other using ~1/16" (~0.16 cm) thick metal pins. The required number of samples for each test was bundled together using plastic cable ties by procedures described below.

Sample Quantities

Some FKM rubber samples were exposed to gamma radiation alone, some to the simulant (chemicals) alone, or to radiation followed by exposure to the simulant. Since radiation was expected to have the greatest effect on the compression set and tensile properties of FKM rubber, we prepared specific samples for radiation exposure alone. These samples were referred to as "Rad Only" samples. Exposing some samples to only gamma radiation, while other samples received exposure to both radiation and chemicals was done to differentiate the effects of radiation alone from the effects when two environmental conditions (radiation and simulant) were applied.

For "Rad Only" compression set measurements, 48 samples (six specimens per test) were cut for the matrix of four radiation doses, four exposure times, and three exposure temperatures for a total of 288 samples. The exposure times for "Rad Only" samples represent the time periods (7-, 14-, 28-, and 180-days) that the samples were held at the respective temperatures. Tensile property measurements (five specimens per test) for "Rad Only" samples required 240 samples. A total of 528 samples was needed for these two measurements. In view of the perceived effect of radiation on compression set and tensile property measurements, the material properties of FKM rubber were measured prior to exposure to either radiation and temperature. The values obtained from these measurements represented the initial material properties and required an additional 11 samples.

"Simulant Only" samples, referred to as "0" radiation dose samples in subsequent discussions, were required for each of the seven measurements. For specific gravity and hardness measurements, 12 samples were required. Dimensional and mass measurements (three per test) required the preparation of 9 samples. For VTR measurements (three per test), 9 samples were needed. Compression set measurements required 72 samples. Finally, tensile property measurements required 60 samples. Thus, a total of 162 samples was required for all seven

“Simulant Only” tests. These samples were exposed to the aqueous simulant for the four time periods at the three temperatures.

We now turn to the samples required for exposure to the combination of radiation and chemicals. For specific gravity and hardness measurements, 48 samples were cut out for the combination of three temperatures, four radiation doses, and four time periods. For dimensional and mass measurements, 36 samples were prepared. Compression set measurements involved 288 samples. VTR measurements involved 36 samples and tensile testing involved 240 samples. Thus, for all seven measurements, 648 samples were prepared for exposure to the three temperatures and four radiation doses. For nondestructive tests such as specific gravity, dimensional, mass, hardness, and VTR measurements, the same samples were re-used for the other exposure times, that is, one sample set was used for 7-, 14-, 28-, and 180-day exposures at each temperature and for each radiation dose.

A total of 1,349 FKM rubber samples was used to perform the various measurements.

Sample Irradiation

The elastomer samples were irradiated by an underwater ^{60}Co gamma source at SNL/NM. These samples were loaded into a metal basket in the same configuration as was used to condition the samples, that is, the samples were stacked atop each other and separated by a metal spiral or by metal pins. The basket was then inserted into a water-tight stainless steel canister (volume ~4 L). The canister was sealed and lowered into the pool to a depth of 6 feet, purged with slow steady flow (~30 mL/min) of dry air, and allowed to come to thermal equilibrium at ambient (~32), 50, or 60°C.¹⁵ Once thermal equilibrium was attained within the canister immersed in the pool of water, the canister was lowered into its irradiation location in the pool and exposure was begun to obtain the desired radiation dosage. The highest dose rate available at the Low Intensity Cobalt Array (LICA) Facility is ~540 Krad/hr. The array, which was used for irradiating these samples, typically had dose rates of ~81 Krad/hr. Thus, for an irradiation experiment in which a gamma-ray dose of 143 Krad was required, the samples were exposed for approximately 1.8 hr. For doses of 286, 571, and 3,670 Krad (3.67 Mrad), longer exposure times were needed. After the samples received the calculated radiation dosage, the

canister was removed from the pool and the samples were again placed in the conditioning chambers. No more than 24 hours typically elapsed between the time that the samples had been exposed to radiation and when they were exposed to the simulant waste or the test temperatures.

Sample Exposure to Simulant

The general exposure protocol for specific gravity samples involved placing four specimens of the FKM rubber into a container and exposing them to the aqueous simulant at three temperatures and four time periods. The four specimens were bundled together using 7.5" (19 cm) nylon cable ties. Within each bundle, the specimens were separated by $\sim \frac{1}{16}$ " (0.16 cm) metal pins used as spacers. This exposed the waste simulant to all surfaces of each specimen. A 2-L plastic container was loaded with the four bundled test specimens and then filled with 1,600 mL of the test solution. Care was taken to ensure that sufficient simulant waste was present to expose the entire surface area of all the samples. After the liquid simulant waste was added, the plastic lid was attached to the jar and tightened. The jars were placed in respective environmental chambers maintained at 18, 50, and 60°C. The jars were kept in these environmental chambers for 7, 14, 28, and 180 days. Similar procedures were followed for each of the other four testing procedures (dimensional testing, hardness testing, compression set tests, and tensile tests). For VTR measurements, each of three 1/2 pint (~236 mL) Mason Jars (Kerr Group, Inc., Los Angeles, CA, Part 70610-3) was filled with approximately 200 mL of the test solution. The FKM rubber discs were loosely attached to the jars with metal bands. The jars were placed in an upright configuration (FKM rubber and band facing up) into the environmental chambers. The jars were held at the respective test temperatures for one hour to equilibrate. After being sealed and weighed, the jars were placed in the chambers again in an inverted position and held at the specific test temperature for the required time period.

Evaluation Approach

The material properties that should be evaluated to assess the suitability of potential elastomeric materials in mixed waste packaging designs are specific gravity changes, dimensional changes (involving mass and dimensional measurements), hardness, compression set, VTR, and tensile property changes (tensile strength, tensile stress, and ultimate elongation). Since the measurement of *all* these material properties was expected to be costly and time consuming, screening tests with relatively severe exposure conditions such as high temperatures and high radiation levels were implemented to quickly reduce the number of possible materials for full evaluation. The results of these screening studies have been previously reported in a milestone document,¹⁶ at several technical conferences,²⁶ and in a SAND Report.⁷ From this screening study, it was found that all of the selected seal materials had passed the screening criteria in the aqueous simulant mixed waste. This then necessitated the testing of these six materials by exposure to a matrix of four radiation doses, three temperatures, and four exposure times in the simulant waste. In view of the extensive number of materials and exposure conditions, this second phase of the program was referred to as the Comprehensive Testing Phase.

Because of budget constraints, the testing was further subdivided into comprehensive testing on liner materials and seal materials. Additional funding constraints required an additional subdivision of the testing activity by evaluating individual elastomers. The order of testing for these individual elastomers was established by the degree of response in the aqueous simulant. In other words, the best elastomer was evaluated first while the worst would be evaluated last. From the data given in a previously submitted milestone document,¹⁶ the best-to-worst seal materials in the aqueous simulant were determined to be EPDM, butyl, SBR, nitrile, Viton®, and EPI rubber. Accordingly, the first three seal materials evaluated were EPDM, butyl, and SBR rubber.¹⁷⁻¹⁹ The next elastomer evaluated was FKM rubber.

The evaluation parameters used in this comprehensive testing phase consisted of measuring the specific gravity, dimensions, mass, hardness, compression set, VTR, and tensile properties of the seal materials. Tensile properties for elastomers included tensile strength, ultimate elongation, and tensile stress. These parameters were evaluated using standardized test methods such as those developed by the American Society for Testing and Materials (ASTM). For specific gravity measurements, ASTM D 792²⁰ was used. In measuring dimensions and mass,

ASTM D 543²¹ was used. For hardness measurements, ASTM D 2240²² was used. In evaluating compression set, ASTM D 395 - Method B²³ was used. However, in using ASTM D 395, six samples (3 sets of 2 samples) of 0.125" (3.17 mm) thickness each were held at the three temperatures with a spacer bar thickness of 4.5 mm. For VTR measurements, ASTM D 814 was used. Finally, for evaluating tensile properties, ASTM D412 - Method A was used.

Before describing the results of this study, we will discuss the comprehensive testing strategy for FKM rubber, shown in a flow diagram in Figure 1. The rubber was subjected to four different protocols (Paths A-D). To determine the intrinsic properties of the materials, Baseline property measurements (Path A) were made in each of the seven tests. These properties were measured at ambient laboratory conditions. To differentiate the effects on the materials by radiation and chemicals, one series of samples was exposed to the simulant alone (Path B) while another series of samples was exposed to both radiation and the simulant (Path C). The first series of these samples are referred to as "Simulant Only" in the flow diagram. It should be noted that both series of samples were exposed for the four time periods (7, 14, 28, and 180 days) at three different temperatures (18, 50, and 60°C). For two testing protocols, tensile testing (Tensile) and compression set (Set), where the effects of radiation and temperature alone could have significant impact on these properties, a series of samples described as "Rad Only" is shown in the flow diagram (Path D). These samples were irradiated at three temperatures and then exposed for the four time periods at the three temperatures. What may not be obvious from the flow diagram is the large number of samples tested in this comprehensive testing phase of the program. The total testing data analyzed as given in Appendices B–J involved 2,126 measurements on 1,349 samples.

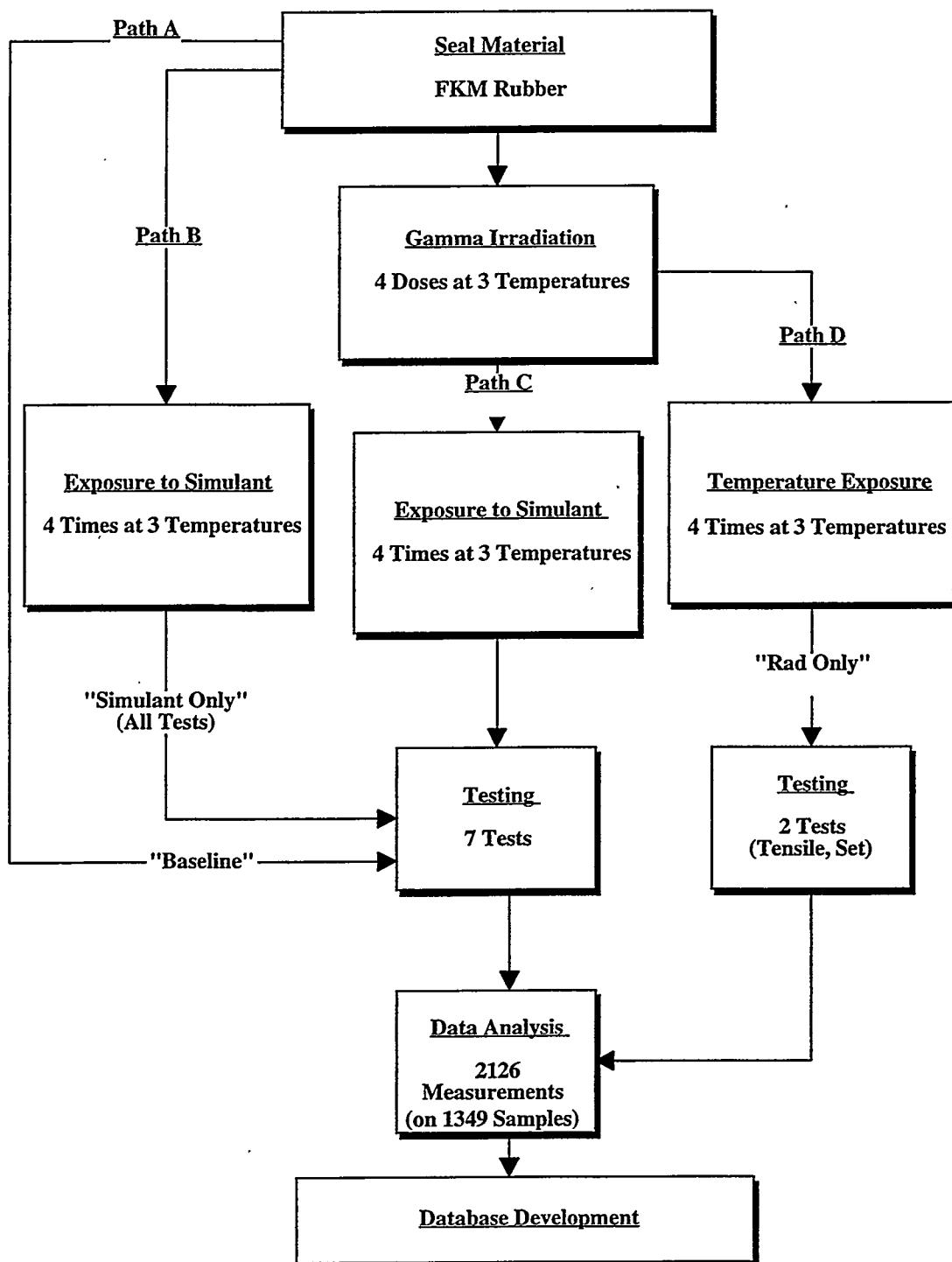


Figure 1. Comprehensive Seal Testing Strategy

RESULTS

Specific Gravity

Specific gravity measurements, also known as relative density measurements, measure the densities of materials exposed to different conditions. A decrease in density of the material can indicate leaching or swelling. Swelling can lead to increases in permeability that can be confirmed by VTR measurements. Increases in density are caused by absorption of the test liquid. This absorption leads to increases in mass of the material and can also lead to higher permeability of the test liquid.

Figure 2 shows the effects of exposure time, gamma radiation dose, and exposure temperature of the aqueous simulant on FKM rubber.

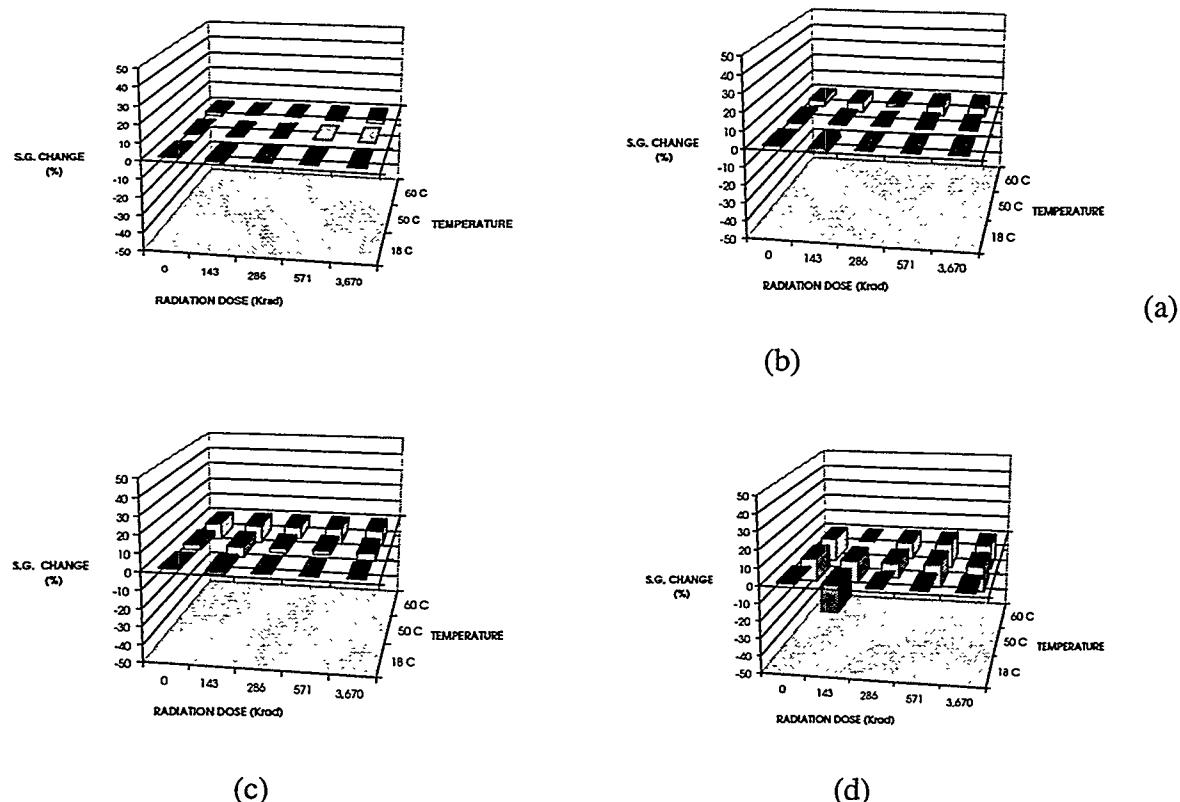


Figure 2. Specific gravity (S.G.) changes in FKM rubber after exposure to ~ 0, 143, 286, 571, and 3,670 Krad of gamma radiation followed by exposure for (a) 7 days, (b) 14 days, (c) 28 days, and (d) 180 days to the aqueous simulant waste at 18, 50, and 60°C.

These three-dimensional bar graphs plots radiation dose, exposure temperature, and the average percent specific gravity change in the x, y, and z directions, respectively. When the radiation dose is indicated as 0, the samples received no gamma radiation. These samples were only exposed to the simulant, that is, these samples represent the “Simulant Only” samples discussed earlier. It should be noted that the scale for these specific changes is rather large, for example, from 0% to 50%, and either positive or negative. In Figure 2 and all subsequent figures, negative changes can be recognized by the dark bar tops in the x-y plane. These bars project into the negative portion of the graph. The sign of the specific gravity indicates whether specific gravity has increased or decreased when compared to the pristine material, that is, the specific gravity of FKM rubber at ambient conditions. Therefore, changes in the magnitude and the sign of specific gravity values indicate changes in this property. The greater the absolute values of the changes, the more the materials are affected by the specific set of environmental conditions. Since properly engineered packaging components are not expected to be affected by contents of the package, such as aqueous mixed wastes, elastomers exhibiting the smallest changes in specific gravity should be selected as packaging components.

From an overall perspective, the data in Figure 2 show that the temperature of the simulant and the exposure time have the greatest effect on the specific gravity of FKM rubber. Increasing levels of radiation dose do not lead to dramatic changes in specific gravity. With increased temperatures and longer exposure time, increased specific gravity changes were observed. While no quantitative correlation can be made concerning temperature and exposure time effects, a doubling of specific gravity changes can be seen on samples exposed to 50 and 60°C for exposures of 28 days and below. Radiation exposure alone appears not to exhibit a significant effect on the specific gravity of FKM rubber. These results are consistent with the known sensitivity of FKM rubber to caustic chemicals at elevated temperatures.²⁵ This demonstrates that FKM rubber is *not* a suitable elastomer for use under these conditions if specific gravity is the determining package design criterion. While the exact specific gravity values are not obvious from the data in Figure 2, their actual values can be found in Appendix B.

In the following section, we present the results of the effects of only the simulant waste and a combination of radiation and the simulant on the dimensional properties of FKM rubber.

Dimensional Properties

Similar to the measurement of specific gravity, or density changes, the measurement of changes in dimensional properties can provide important information about the effects of different environments on materials. Specifically, the swelling of the material or leaching of components of the material may be manifested by increases or decreases in the dimensions of the material. The dimensional properties measured and reported in this section are changes in length, width, and thickness of the material. Since the standard test method ASTM D 543 used to measure dimensional properties includes the determination of mass as part of the test, this property was also measured. We will describe dimensional changes by evaluating the product of these changes—volume (length \times width \times thickness). The technical justification for this approach is that, while length and width changes have generally been much smaller than thickness

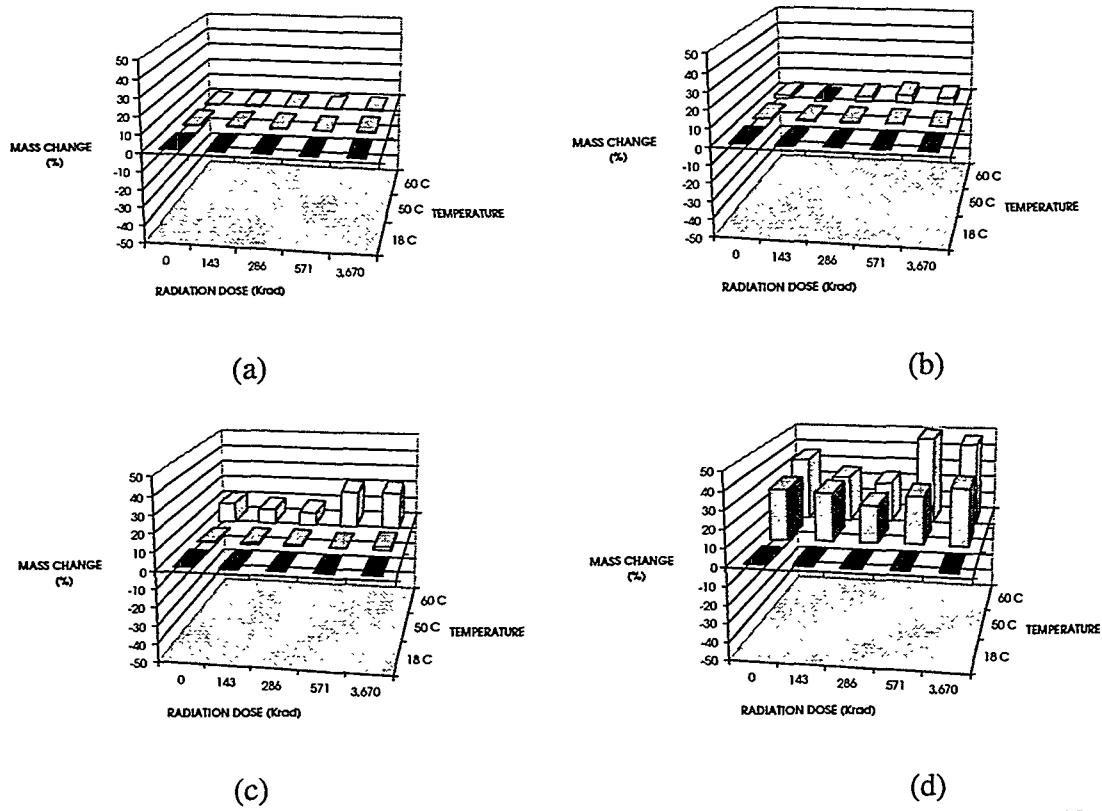


Figure 3. Mass changes in FKM rubber after exposure to approximately 0, 143, 286, 571, and 3,670 Krad of gamma radiation followed by exposure for (a) 7 days, (b) 14 days, (c) 28 days, and (d) 180 days to the aqueous simulant waste at 18, 50, and 60°C.

changes, the product of these changes encompasses individual components into one general dimensional property, the volume of the materials. The effects of the different environments on the mass changes will be presented first.

To measure the effect of exposure time and exposure temperature of the aqueous simulant on FKM rubber, the mass of the samples was measured before and after exposure to only the surrogate waste. The results are given in Figure 3 above in the 0 radiation dose data field. Similar to data shown in the previous section, the scale for average % mass change is from -50% to 50%. The sign of the mass changes indicates whether the mass of the material has increased or decreased when compared to the pristine material, that is, material mass at ambient conditions. Therefore, changes in the magnitude and the sign of % mass change values can vary in these measurements. The greater the absolute value of the changes, the more the material is affected by a particular set of environmental conditions. Since properly engineered packaging components are also not expected to be affected by contents of the package, that is, the mixed wastes, materials exhibiting the smallest changes in mass should be selected as packaging components. From an overall perspective, the data in Figure 3 show that temperature of the simulant has a slight effect on the changes in mass of the material below 14-day exposures. After 14 days exposure, substantial weight gains were observed. This is especially true at 60°C. After 180-day exposures, mass gains in excess of 20% - 50% were observed at 50°C and 60°C. While the exact mass values are not obvious from the data in Figure 3, their actual values can be found in Appendix C.

In Figure 4, the average % volume changes of FKM rubber exposed to the four gamma radiation doses followed by exposure to the aqueous simulant waste at 18, 50, and 60°C for 7, 14, 28, and 180 days are given. FKM rubber had volume changes of less than ~ 5% below 14 days and 50°C. In most cases, these volume changes were much smaller, in the ~1.5% range. At the highest temperature and longest exposure times, very large increases in volume were observed. The largest (>85%) of these changes was found at 60°C. Thus, FKM rubber swells considerably when exposed to these environmental conditions. A general trend suggests that with increasing exposure time, all the FKM rubber samples swelled. The notable exception is for samples

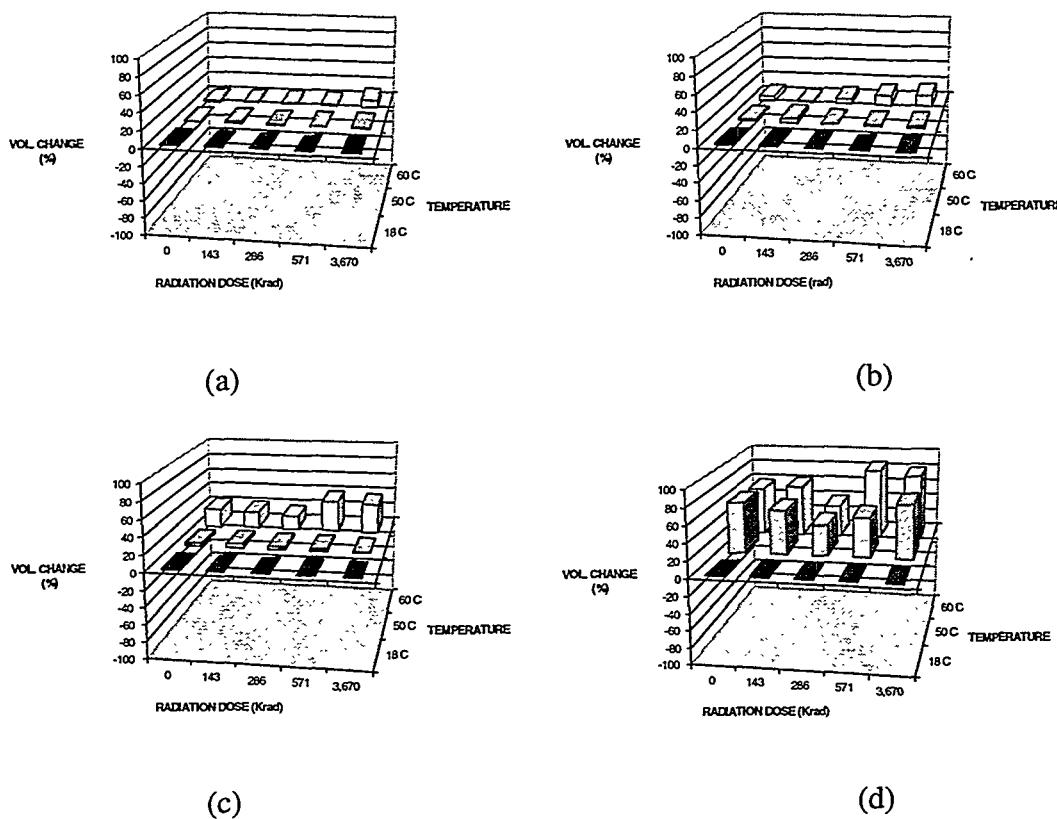


Figure 4. Volume (Vol.) changes in FKM rubber after exposure to ~ 0, 143, 286, 571, and 3,670 Krad of gamma radiation followed by exposure for (a) 7 days, (b) 14 days, (c) 28 days, and (d) 180 days to the aqueous simulant waste at 18, 50, and 60°C.

exposed at 18°C. Most of these samples exhibited slight swelling (increases in volume) with increasing exposure time. The greatest volume changes can be seen in Figure 4d, where FKM rubber nearly doubled its volume at 50°C and 60°C. It should be noted that the greatest contributor to the volume increase was an increase in the sample thickness. While the exact volume values are not obvious from the data in Figure 4, their actual values can be found in Appendix D.

Hardness Properties

The measurement of changes in the hardness of materials can provide important clues as to the effects of environmental conditions on the material. If the hardness of the material is decreasing, swelling of the material may have occurred. Alternatively, the polymeric constituents of the elastomer may have substantially degraded. Conversely, if the hardness of

the material is increasing, additional cross-linking of the polymer may have resulted. The results of these measurements, in addition to providing important data by itself, may complement other measurements such as specific gravity and dimensional and tensile properties.

The measurement of hardness in plastics involves the use of a standard instrument manufactured by Shore Instrument Company known as a Shore Durometer. The degree of hardness that the plastic exhibits will dictate the type of durometer to be used. For elastomers, which in relative terms tend to be rather soft, a Type A Durometer is used. Similar to the approach used for the previously described property measurements, the initial hardness values (~73 Shore A hardness points) were determined for pristine samples, that is, samples not exposed to anything. Using these initial hardness values, % hardness changes were measured for samples exposed to the simulant alone (see 0 radiation dose data points in Figure 5 at the three temperatures and four exposure times) and to a combination of radiation and simulant at these temperatures and exposure times. We will now present the results of these measurements.

To measure the effect of exposure time and exposure temperature of the aqueous simulant on FKM rubber, hardness testing was performed on the materials exposed to the surrogate waste alone at the three temperatures and four time periods. The results of these measurements are given in Figure 5 under 0 radiation dose. The sign of the hardness changes indicates whether the hardness of the material increased or decreased when compared to that of the pristine material. Decreasing hardness indicates that the material has become softer as a consequence of the exposure conditions. As previously mentioned, properly engineered plastic packaging components are not expected to be affected by the packaging contents. An elastomer exhibiting the smallest changes in hardness should be considered a good candidate as a packaging component. An inspection of the results in Figure 5, reveals that generally the hardness of FKM rubber essentially remained unchanged for short exposures at low temperatures. With longer exposure times and higher temperatures, most of the samples became softer. Beginning at 50°C and getting even more pronounced at 60°C, hardness changes exceeded 2% in samples

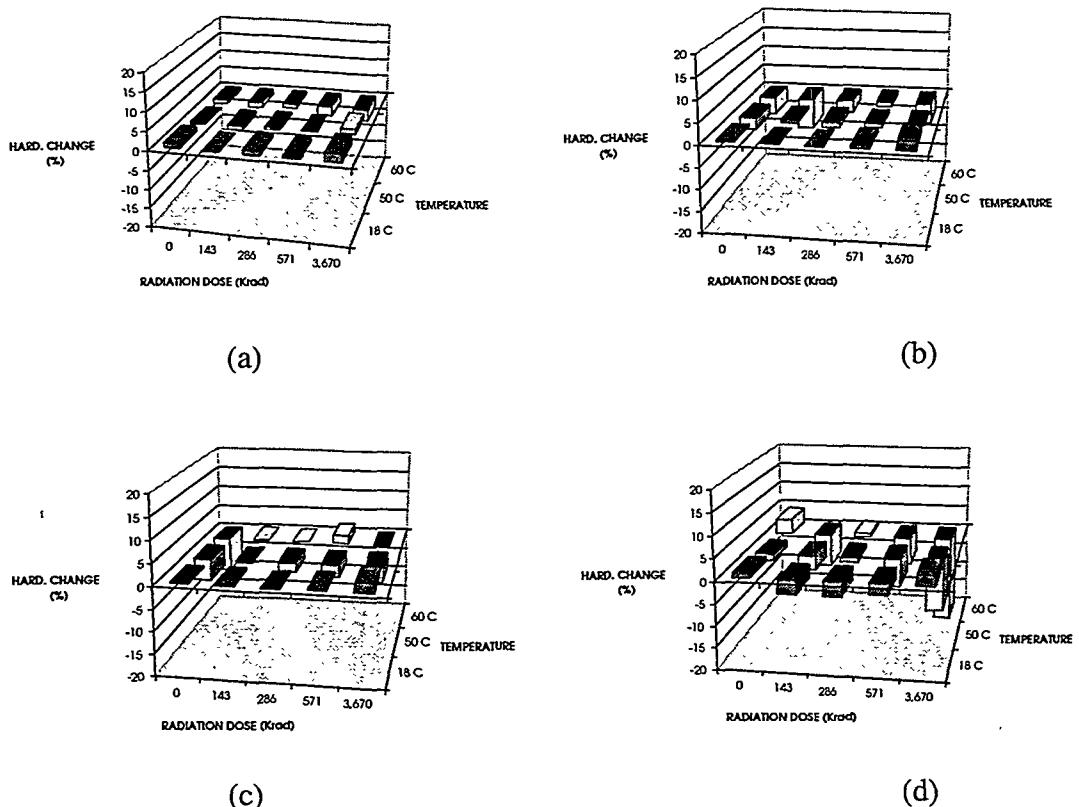


Figure 5. Hardness changes in FKM rubber after exposure to ~ 0 , 143, 286, 571, and 3,670 Krad of gamma radiation followed by exposure for (a) 7 days, (b) 14 days, (c) 28 days, and (d) 180 days to the aqueous simulant waste at 18, 50, and 60°C.

exposed only to the simulant. The effects of radiation helped restore some of the hardness in FKM by causing the material to increase in hardness. When FKM samples were exposed for 180 days (Figure 5d), all the samples that received a combination of radiation and chemical exposure exhibited a 2%-8% decrease in hardness. The hardness changes were most strongly affected by high radiation dose and high temperature. The largest hardness change (~80%) was observed in samples that received radiation doses of 3,670 Krad and 180-day exposures to the 60°C simulant. These results suggest that prolonged exposure to this caustic and oxidizing simulant leads to softening of FKM rubber. Since increases in volume under these conditions (see Figure 4) were observed, this softening appears to be because of swelling of the material. These results are consistent with the increases in mass observed under these conditions (see Figure 3). The decreasing hardness could also be explained by losses in elasticity of the material. Such losses of elasticity can be confirmed by compression set measurements discussed in the next section. These losses in elasticity could be caused by the chemical

degradation of the rubber. While the actual hardness values are not obvious from the data in Figure 5, their actual values can be found in Appendix E.

Compression Set

Compression set (Set) tests measure the ability of elastomers to retain elastic properties after prolonged action of compressive stresses. Set is usually determined in air and reported as the percent of deflection by which the elastomer fails to recover after a fixed time under a specified compression and temperature. In these experiments, the time period of 25% compression was 22 hr at 18, 50, and 60°C. A complete return by the elastomer to its original thickness after the compressive stresses are removed results in a calculated set of 0%. When the elastomer does not return to its original thickness but remains at the thickness under compression (in this case a 4.5-mm deflection), a set of 100% is calculated. These values, therefore, represent a percentage of the original deflection. The practical aspect of such a situation is that the elastomer just contacts the matting surface of the device that contains the elastomer. In the case of O-ring seals, this could lead to seal failure because the elastomeric seal makes minimum contact with the sealing surface. It should be obvious from the previous discussions that materials having a low set value are desirable. To measure set, we used the standardized test method, ASTM D 395. The FKM rubber samples were held in the compression set device at three temperatures. Figure 6 shows an example of the experimental configuration used for the compression set tests.

Since we wanted to understand effects of radiation alone on set values, a portion of the FKM rubber samples was exposed to only the four radiation doses (that is, no chemical exposure), the three temperatures, and the four exposure times. Similarly, the effects of the simulant alone were studied under these conditions. Finally, samples exposed to a combination of radiation and simulant were studied. It is important to mention that Figures 7 and 8 plot changes in compression set versus radiation dose and temperature. These changes represent the difference in the set observed under the specific environmental conditions and the set of pristine FKM

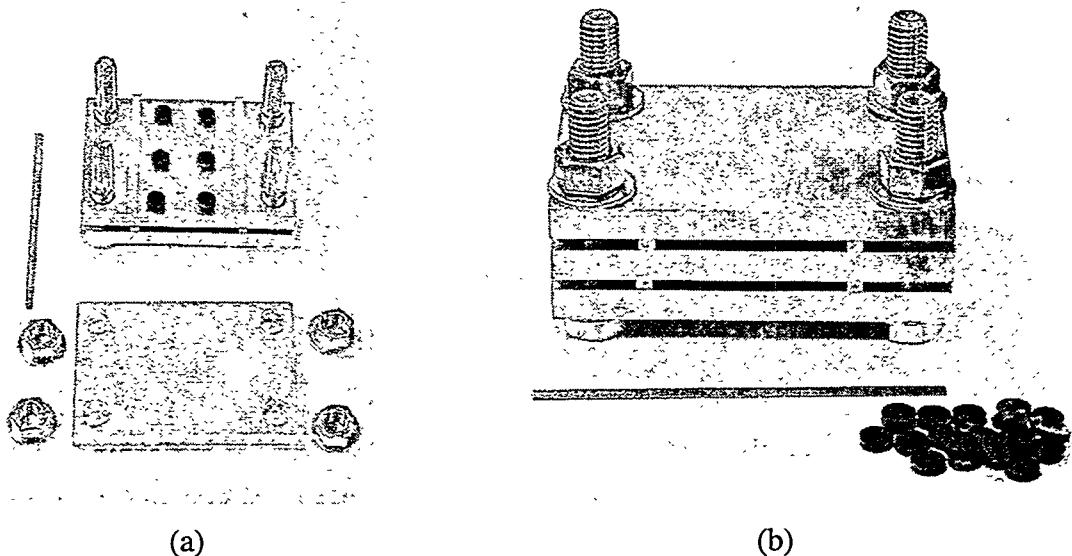


Figure 6. Compression set fixture: (a) a partly assembled fixture with the 4.5-mm spacer bars and rubber samples and (b) an assembled fixture with rubber samples.

rubber samples. Because set values are expressed in percent, the change in set is the difference of these values in percent.

In Figure 7, the set changes for FKM rubber samples exposed to four gamma ray doses followed by 7-, 14-, 28-, and 180-day exposures at the three temperatures are given. The data in Figure 7 represent when FKM rubber has only been exposed to gamma radiation. It should be pointed out that the scale is from 0 to $\pm 100\%$, that is, a 100% decrease or increase in set. Keep in mind that for these measurements, the set was measured after being held for 22 hours at the three temperatures. Another way to express this important experimental detail is that only the samples themselves saw the different radiation doses, exposure times, and exposure temperature. The compression set measurements were performed at one time and three temperatures. These compression set measurements on FKM rubber were thus different from the measurements on EPDM and butyl rubber where measurements were performed at one time and one temperature (ambient).

These results show that virtually all samples displayed a decrease in set in the range of approximately 1% to 10%, and most samples had decreases of the set in the range of approximately 7% - 8%. Decreases for changes in the set indicate that the samples, after exposure to these environmental conditions, had a smaller set than the unexposed samples; that is, these samples were more elastic than the unexposed samples. Most of these larger decreases

in set were observed in samples with larger gamma radiation dose exposures and/or longer exposure times. However, systematic trends in samples exposed to increasing radiation doses, exposure time, and exposure temperatures are not very pronounced with regard to time. These results suggest that the set changes are more dependent on temperature and radiation dose. For these environmental factors, the greatest decreases in set were noted at 3,670 Krad where a set of about -10% was observed. Since a set of comparable magnitude was observed at the lowest radiation dose of 143 Krad, the test results imply that once a threshold of radiation damage is achieved in the material, then set does not change significantly.

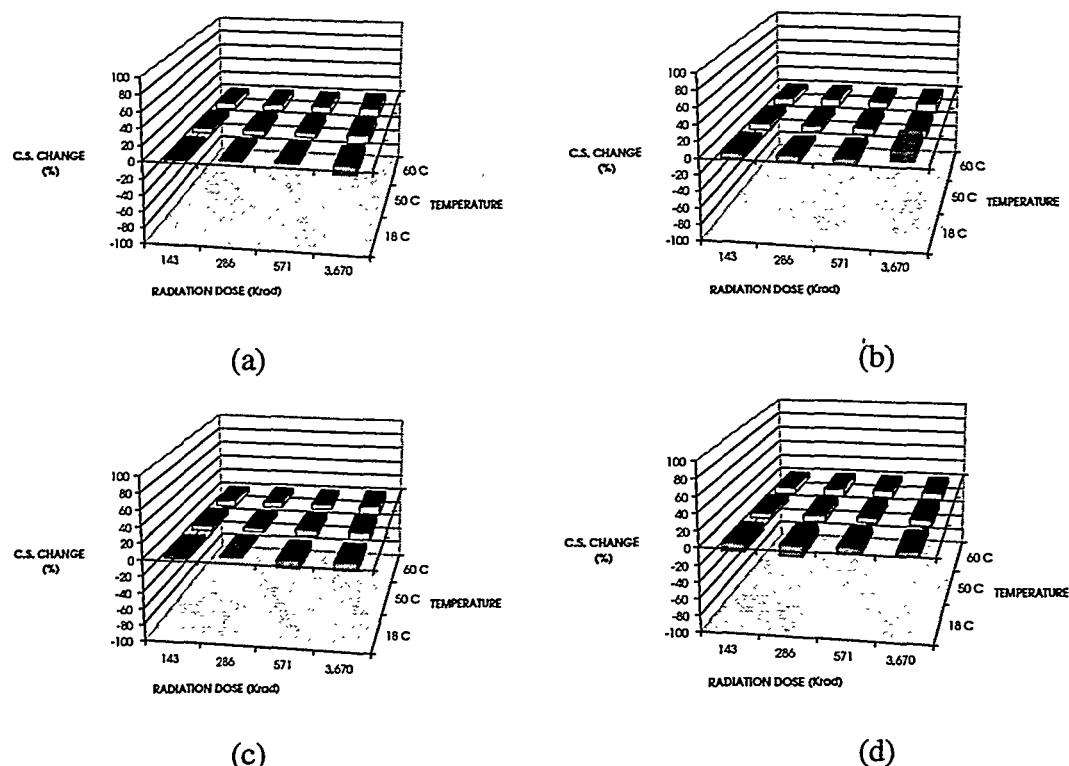


Figure 7. Compression set (C.S.) changes in FKM rubber after exposure to \sim 143, 286, 571, and 3,670 Krad of gamma radiation followed by exposure for (a) 7 days, (b) 14 days, (c) 28 days, and (d) 180 days at 18, 50, and 60°C.

In Figure 8, the set changes for FKM rubber samples exposed to a combination of the four gamma ray doses followed by 7-, 14-, 28-, and 180-day exposures to the aqueous simulant at the three temperatures are shown. In comparison to the samples exposed to gamma radiation alone, the set changes exhibited by samples exposed to a combination of radiation and the aqueous simulant were substantial, in the range of -11% to 80%. It can also be seen that the set in FKM rubber samples exposed to only the simulant (0 radiation dose data) generally also increased

with increased temperature and increased exposure time. The combination of radiation followed by exposure to the simulant has a "beneficial" effect by resulting in similar compression sets; that is, the samples were as elastic as the samples exposed only to the simulant. With the increased temperatures and increased exposure times, increases in set were noted. The greatest retention of deformation was noted in samples exposed to radiation and the simulant for 180 days at 50°C and 60°C.

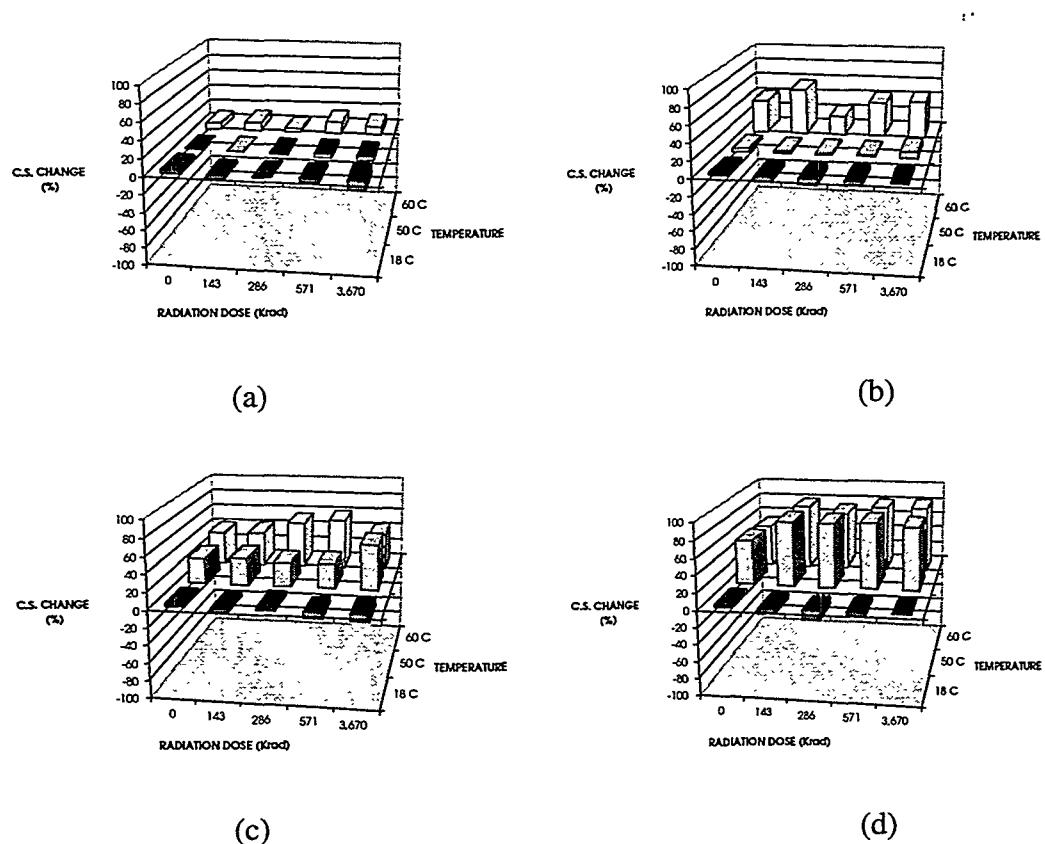


Figure 8. Compression set (C.S.) changes in FKM rubber after exposure to ~ 0, 143, 286, 571, and 3,670 Krad of gamma radiation followed by exposure for (a) 7 days, (b) 14 days, (c) 28 days, and (d) 180 days to the aqueous simulant waste at 18, 50, and 60°C, respectively.

To summarize, from the compression set results described previously, FKM rubber is affected somewhat by radiation, and greatly by the simulant or a combination of these two environmental conditions. While the actual set values are not obvious from the data in Figure 8, these values can be found in Appendix F.

Vapor Transport Rates

Vapor Transport Rate (VTR) measurements provide a measure of the permeability of various chemical agents into elastomers. The rate of transmission of a liquid through an elastomer that acts as a barrier is important in elastomer seal performance. This transmission is referred to as vapor transmission since the liquid diffuses through the elastomer in a molecular sense and escapes into the surrounding atmosphere in vapor form. This type of testing provides a steady-state measure of the rate of vapor and liquid transmission through relatively thin elastomers. While the calculated values of VTR cannot be directly converted to traditional permeability values, the VTR values can be used to give a figure of merit for permeability. These measurements were performed at three temperatures and four exposure times. In these experiments, one set of FKM rubber samples was exposed to only the simulant aqueous waste and the remaining samples were exposed to a combination of radiation and the simulant. To measure VTR, we used the standardized test method, ASTM D 814. Using this method, the FKM rubber samples were sealed to a ground-glass surface using a metal screw band. It should be noted that VTR experiments by this method with FKM rubber samples exposed to *only* gamma radiation are not possible because the testing method requires the presence of a chemical agent. Figure 9 shows a typical example of the experimental configuration used for VTR measurements.



Figure 9. Vapor Transmission Rate Cells

Because it is not possible to determine VTR on “pristine” FKM rubber, it is not possible to describe these data in terms of VTR changes. As will be recalled in prior testing methods, the pristine property value of the material was used to calculate changes in that property. For example, in the previous section the compression set value of FKM rubber that had not been exposed to radiation, the simulant, or the combination of radiation and simulant was used to define the compression set of “pristine” FKM rubber, or its initial value. In VTR measurements, however, a similar VTR value for pristine FKM rubber is not possible because its determination would require exposure to a simulant. However, one could show VTR changes between FKM rubber samples exposed to only the simulant and samples exposed to the combination of radiation and simulant. However, since in all the previously described measurements, the presentation of data for FKM rubber samples exposed to the simulant alone was included, we similarly want to retain this data format here. Thus, we will provide the actual VTR values, in $\text{g}/\text{hr}/\text{m}^2$, rather than changes in VTR. In Figure 10, we show VTR for FKM

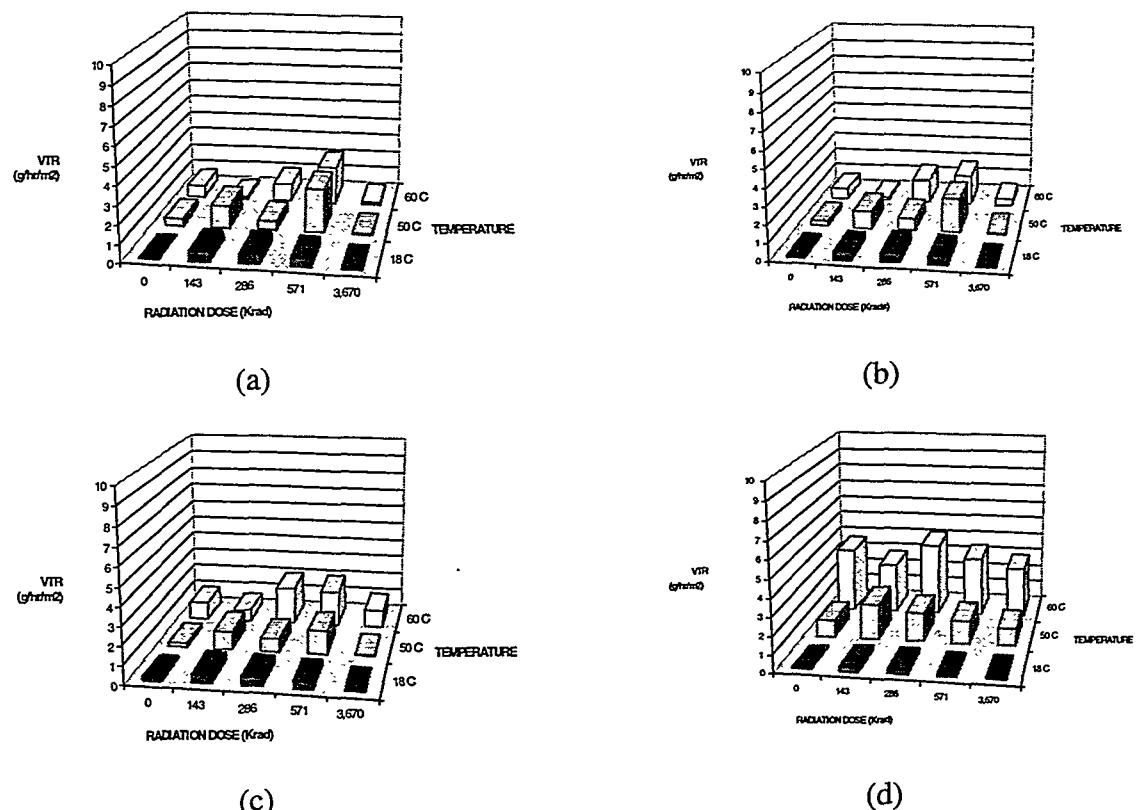


Figure 10. VTR for FKM rubber after exposure to ~ 0 , 143, 286, 571, and 3,670 Krad of gamma radiation followed by exposure for (a) 7 days, (b) 14 days, (c) 28 days, and (d) 180 days to the aqueous simulant waste at 18, 50, and 60°C, respectively.

rubber samples exposed to a combination of the four gamma ray doses followed by 7-, 14-, 28-, and 180-day exposures to the aqueous simulant at the three temperatures. The data shown for the 0 radiation dose represent samples exposed only to the simulant for the four exposure times and temperatures. As can be seen from the data, all the previously mentioned samples, except 180-day exposure at 60°C, exhibited VTR values below 1 g/hr/m². This indicated that unirradiated FKM is relatively resistant to permeation by this simulant waste if exposure times are 28 days or less and temperatures are below 50°C. With longer exposure times and temperatures at 50°C and 60°C, all VTRs for the samples were in the range of 1 to 4 g/hr/m². In general, the FKM rubber samples that had received a combination of radiation and chemical exposure exhibited more similar VTRs at all temperatures than did samples exposed to only the chemical. This observation is clearly seen for the 50°C and 60°C data points in Figure 10d. These results suggest that chemical effects dominate the VTR in FKM rubber at elevated temperatures. The actual VTR values can be found in Appendix G.

Tensile Properties

Tensile properties, also known as mechanical properties, are those associated with a material's response to mechanical forces. A quantity more useful than force is the *engineering stress* (σ), which is the ratio of the magnitude of a force to the magnitude of the originally undeformed area of the body upon which it is acting. True stress is therefore defined as $\sigma = F/A$ where A is the cross-sectional area at the time that the Force (F) is applied. The most common engineering units of stress are pounds of force per square inch (lb/in² or psi). These units may be converted to the corresponding SI unit, the Pascal (Newton/meter), by multiplying the psi value by 6895. Because we are calculating the percent of changes in properties, the units are irrelevant. However, if the actual values in Mega Pascal (MPa) are of interest, Appendix H should be consulted.

Another important tensile property to be considered is strain. A stressed material undergoes deformation or *strain* (ϵ), defined quantitatively as either the incremental deformation divided by the initial dimension or as a percent of the original dimension. Since strain is a dimensionless quantity, the precise choice of units is not important. In this study, a 1-inch gage length was used, and the units of strain are therefore inch/inch. Two fundamentally different

types of strain are observed. The first type is *elastic strain* or elastic deformation where strain is recoverable upon the release of stress. In other words, when a causal stress is removed, the resultant strain vanishes, and the original dimensions of the object are recovered. A practical example of this type of strain is the stretching of a rubber band. Since FKM rubber is a specific type of rubber, this material exhibits this type of strain. The second type of strain is *plastic strain*, which occurs when stress is increased and a value is eventually reached where permanent deformation of the object has occurred. An example of this property is the bending of wire with the fingers. Note that the term *plastic strain* does not mean necessarily that the deformed material is a plastic.

For many plastic materials that might be suitable as packaging components such as seals and liners, high strengths and high strains are expected from the material. The strains exhibited should also be elastic in nature. In certain instances, however, other specific tensile properties are desirable, that is, high strength and low strain. This study was to determine the tensile properties of the pristine material and then determine the effects of radiation alone, the simulant alone, and a combination of these environmental conditions on the tensile properties of FKM rubber. The results of these studies are described in the following sections.

Tensile Strength

The *tensile or ultimate strength* of a material is calculated by dividing the observed maximum load value placed on the material during the tensile test by the original cross-sectional area of the material. While many polymeric materials exhibit stress-strain curves having an initial maximum followed by lower stresses, this is not the case for elastomers. The maximum load value in elastomers is typically observed at the break point of the material.

The measurement of tensile properties involves using tensile testing equipment to apply controlled tensile loads to test specimens. The equipment is capable of varying the speed of load (stress) and accurately measuring the forces (strains) and elongation applied to the specimens. In this study, an Applied Test System, Inc., Universal Testing Machine (Series 1400) was used. This computer-controlled testing equipment performed the required tests using user-developed testing methods. These methods prescribe the strain rates and breaking

points along with many other experimentally important variables. The selection of these experimental variables was based on the standard test method ASTM D 412. For determining the tensile strength of elastomers, a high elongation extensometer and high rates of grip separation (50 mm/min, 20"/min) were used. The acquired data were analyzed with software developed by this manufacturer. The software calculates numerous tensile properties. The data discussed in this subsection require a determination of tensile strength calculated as described previously, using peak loads and cross-sectional area. In addition, the software also calculates ultimate elongation and tensile stress values. In this subsection, we are only interested in tensile strength values.

Because an understanding of the effect of mixed waste environments is not possible without understanding the effects of radiation and simulant alone, the latter experimental conditions were also investigated. The results of tensile strength changes in the materials exposed to gamma radiation alone at the three temperatures and four exposure times are given in Figure 11. In Figure 11, the average % tensile strength changes of FKM rubber exposed to gamma radiation alone at 18, 50, and 60°C for 7, 14, 28, and 180 days is shown. Similar to previous property measurements, these % changes were determined by measuring the change in tensile strength from that of the pristine material (11.3 MPa). Positive values of % tensile strength changes indicate that the material's tensile strength increased under the specific exposure conditions. Negative values indicate decreases in tensile strength. It should be noted that the scale for tensile strength is $\pm 100\%$.

From a general perspective, the data in Figure 11 show that for most radiation doses, exposure times, and exposure temperatures, no significant effect on the tensile strength of the material was noted. Under these conditions, the changes in tensile strength for these samples were in the range of $\sim 1\%$ to 20%. However, for most of the exposure conditions, many of these changes appear to be in the range of $\pm 1\% - 10\%$. At the longest exposure time of 180 days, 60°C, and the highest radiation dose, the tensile strength of FKM rubber appears to have decreased somewhat ($\sim 7\%$). Thus, gamma radiation does not appear to induce large changes in the tensile strength of FKM.

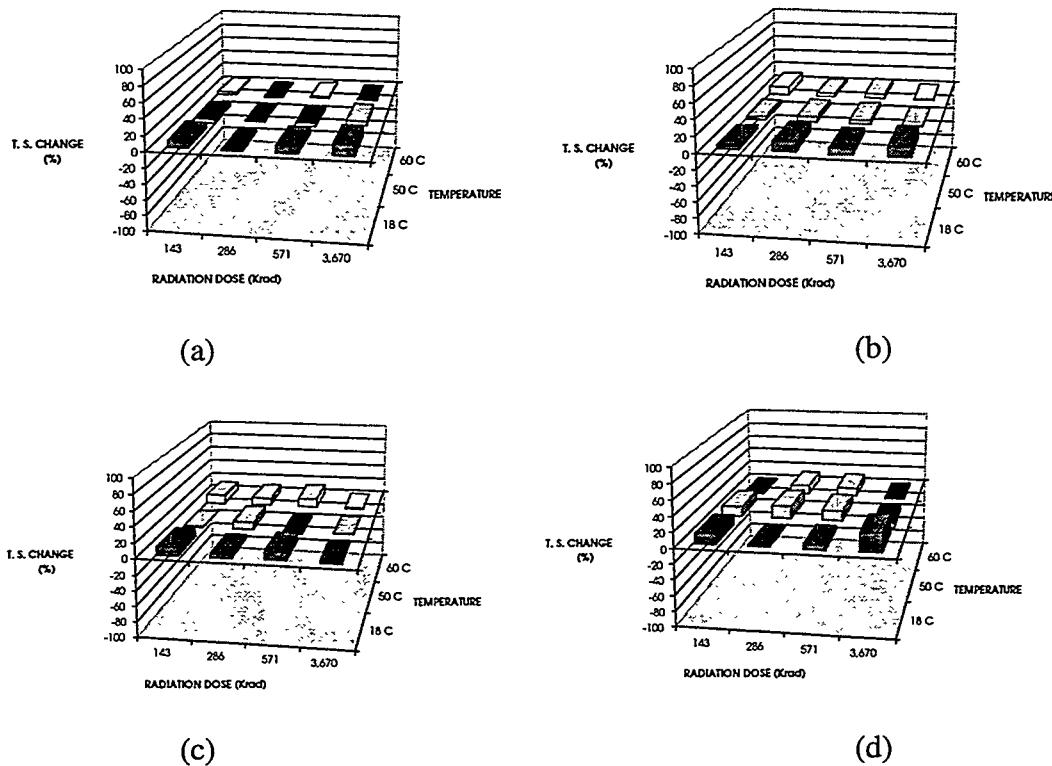


Figure 11. Tensile strength (T. S.) changes in FKM rubber after exposure to $\sim 143, 286, 571$, and $3,670$ Krad of gamma radiation followed by exposure for (a) 7 days, (b) 14 days, (c) 28 days, and (d) 180 days at $18, 50$, and 60°C .

Figure 12 shows the average % tensile strength changes of FKM rubber exposed to the four gamma radiation doses followed by exposure to the aqueous simulant waste at $18, 50$, and 60°C for 7, 14, 28, and 180 days. FKM rubber samples that were only exposed to the simulant waste (0 radiation dose) showed decreases in tensile strength at the higher temperatures. The magnitude of these changes increased with longer exposure times. At the longest exposure time (180 days), decreases in tensile strength of nearly 80% were observed at 60°C .

For FKM samples exposed to both radiation and the simulant, the general trend is that the radiation dose does not appear to have a significant effect on the tensile strength of the material. While one may notice slight decreases ($\sim 10\%$) in tensile strength in samples that received 3,670 Krad gamma radiation exposure followed by 7-day exposure to the simulant, this effect is surpassed by the effect of elevated temperatures. Even after only 7 days of exposure at 60°C , the tensile strength decreased in the range of $\sim 40\%$ irrespective of the radiation dose. At the

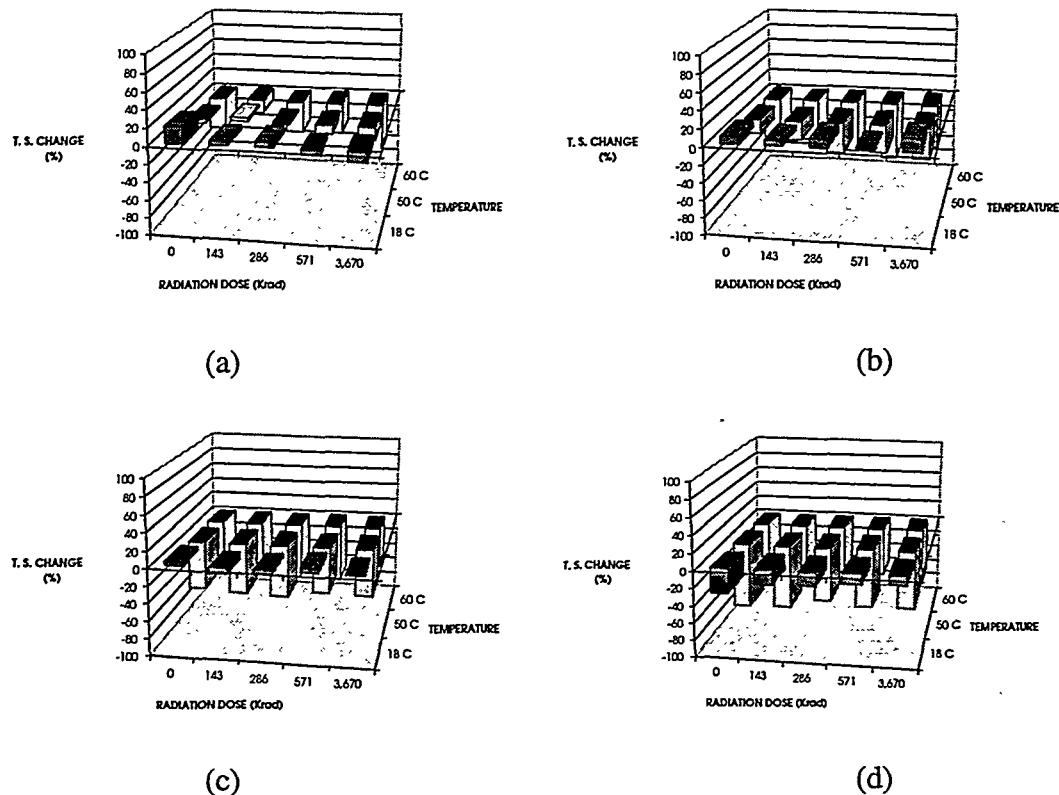


Figure 12. Tensile strength (T. S.) changes in FKM rubber after exposure to ~ 0, 143, 286, 571, and 3,670 Krad of gamma radiation followed by exposure for (a) 7 days, (b) 14 days, (c) 28 days, and (d) 180 days to the aqueous simulant waste at 18, 50, and 60°C.

slightly lower temperature of 50°C, the same levels of decreases in tensile strength were noticed after 14 days of exposure. After 180 days of exposure, all the FKM rubber samples had decreases in tensile strength in the range of 60% - 70%. These results indicate that the tensile strength of FKM rubber is influenced more by the temperature of the simulant than by the radiation dose received. This can be seen clearly in Figure 12c and d where nearly similar decreases in tensile strength (60%) were found for FKM samples that received either no radiation exposure or the maximum dose of 3,670 Krad. These observations suggest that FKM is dominated by chemical effects. While the exact tensile strength values are not obvious from the data in the figure, their actual values can be found in Appendix H.

Elongation at Break or Ultimate Elongation

As was discussed previously, the stress-strain diagrams of linear polymers exhibit an initial maximum stress value. This maximum stress value occurs at the yield point of the material. At this point, deformation starts to localize in the material, forming a “neck,” and the material is said to undergo necking. However, because elastomers, as reflected by their name, are extremely elastic, necking is not observed in these materials. The determination of elongation at yield in elastomeric materials, as opposed to thermoplastic materials, is not possible. In elastomers, the maximum stress value occurs at the break point of the material. The amount of elongation that the material exhibits at this point is known as the elongation at break. For elastomers, the term *ultimate elongation* is used rather than elongation at break. The ultimate elongation of an elastomer is defined by Eq. 1 as

$$\text{Ultimate Elongation} = [(L_t - L_o)/L_o] \times 100. \quad \text{Eq. 1}$$

where L_o is the initial gage length (1" in this study) and L_t is the gage length at the break point. These ultimate elongation values are expressed in percentages. It should be clear that increasing values for ultimate elongation mean increasing elasticity in the material, and decreasing values represent decreasing elasticity. The data presented in the following sections describe the change in elongation. These values were obtained by subtracting the ultimate elongation of the pristine material (140%) from the ultimate elongation observed in the material at the specific environmental conditions. As in previous measurements, positive and negative values for changes in ultimate elongation are possible. If the ultimate elongation values are of interest, Appendix I should be consulted.

In Figure 13, the average changes in ultimate elongation of FKM rubber exposed to the four gamma radiation doses followed by exposure at 18, 50, and 60°C for 7, 14, 28, and 180 days are given. In this figure, the scale ranges from -100% to 100%. Since the pristine elongation of SBR rubber is 140%, this range corresponds to an ultimate elongation range of ~40% to 240%. While the scale is larger than in previous property measurements, it is still less than observed⁸ for thermoplastics. For thermoplastic materials, the scales ranged from -600% to 1000%. These

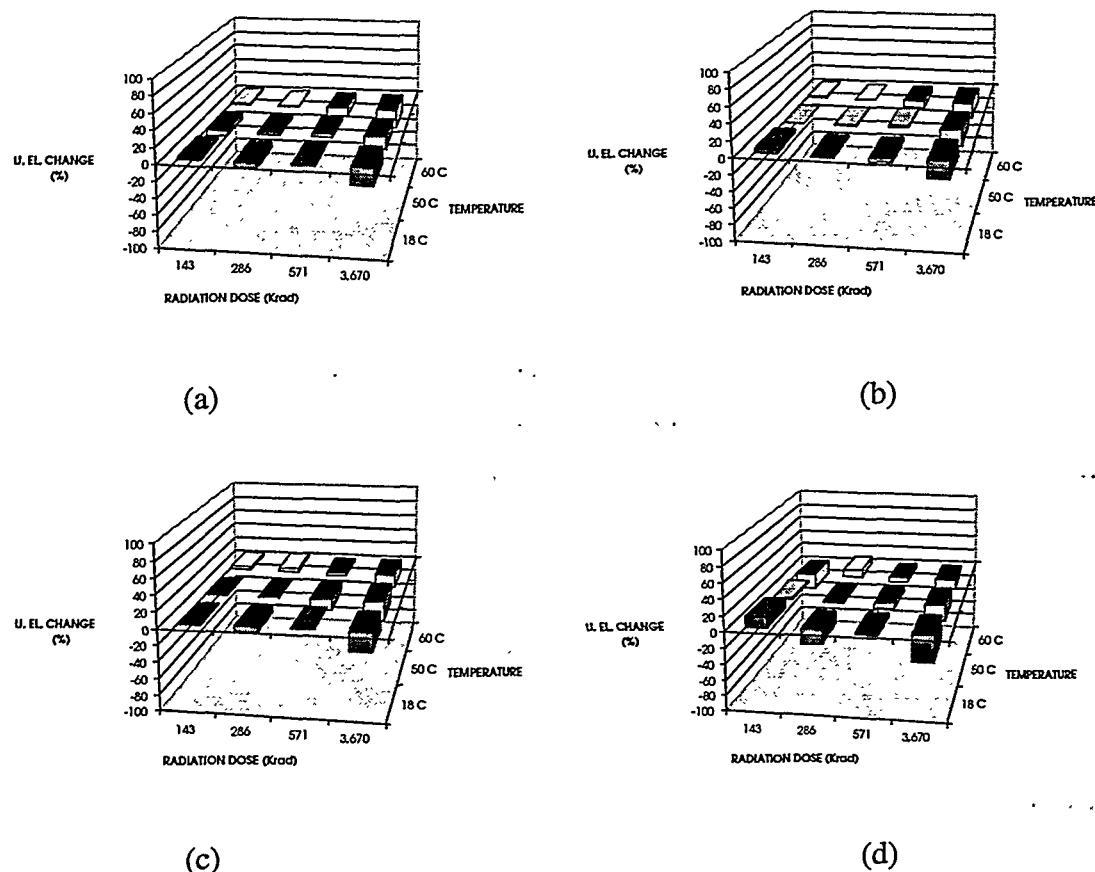


Figure 13. Ultimate Elongation (U. El.) changes in FKM rubber after exposure to $\sim 143, 286, 571$, and $3,670$ Krad of gamma radiation followed by exposure for (a) 7 days, (b) 14 days, (c) 28 days, and (d) 180 days at $18, 50$, and 60°C .

results suggest that the “necking” observed in thermoplastics plays an important role for accommodating deformation.

The data generally show a 6% decrease in ultimate elongation. With increasing exposure time, there is a net decrease in ultimate elongation, but as a whole, the ultimate elongation did not change substantially. At the longest exposure time of 180 days, the largest changes in ultimate elongation were observed. For this exposure time, decreases in ultimate elongation of 13% to 33% were observed. With increasing temperatures, relative increases in ultimate elongation in the 5% to 10% range were generally observed. With increasing radiation dose from 143 to 571 Krad, no changes greater than 10% were typically observed in FKM rubber. The notable exception was samples exposed to 3,670 Krad gamma radiation. For these samples, the

ultimate elongation decreased up to 30%. These results indicate that the elasticity of FKM rubber is strongly affected only by high doses of gamma radiation.

Figure 14 shows the average changes in ultimate elongation for FKM rubber exposed to the four gamma radiation doses followed by exposure to the aqueous simulant waste at 18, 50, and 60°C for 7, 14, 28, and 180 days. Similar to the FKM rubber exposed only to gamma radiation, there is a general decrease in ultimate elongation with increased exposure time. These decreases in ultimate elongation are in the 1% to 46% range. The FKM rubber samples exposed to only the aqueous simulant (0 radiation dose data) also follow this trend but to a lesser degree. These results suggest that the simulant alone does not result in extremely large decreases in ultimate elongation in FKM rubber; that is, the relative elasticity of FKM rubber is retained. With increasing exposure times, FKM rubber exhibited large decreases in ultimate elongation values.

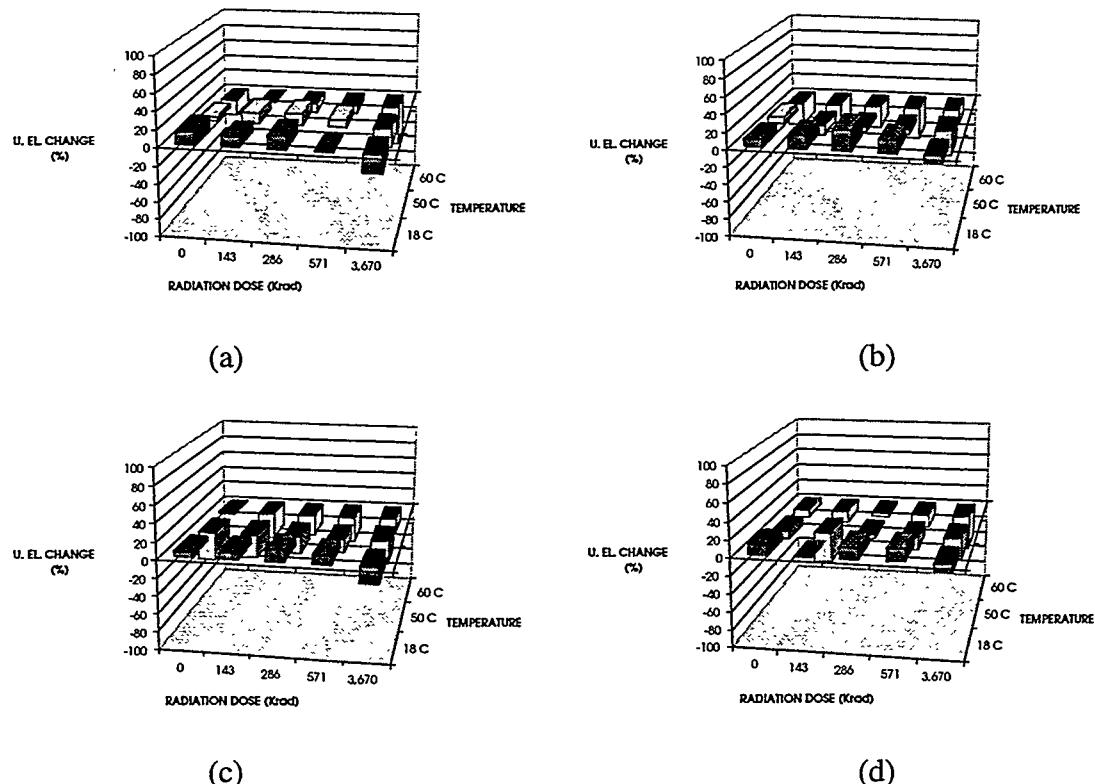


Figure 14. Ultimate elongation (U. El.) changes in FKM rubber after exposure to ~ 0, 143, 286, 571, and 3,670 Krad of gamma radiation followed by exposure for (a) 7 days, (b) 14 days, (c) 28 days, and (d) 180 days to the aqueous simulant waste at 18, 50, and 60°C.

These decreases are in the range of 20%. Upon exposure to the highest radiation dose and the highest temperature, most FKM samples showed a net loss in ultimate elongation of nearly 50%. These results indicate that when FKM rubber receives a longer exposure to the simulant, its elasticity decreases significantly. While the actual ultimate elongation values are not obvious from the data in the previous figures, their actual values can be found in Appendix I.

Tensile Stress or 100% Modulus

For most materials, the initial portion of a stress-strain diagram is linear. This implies that strain is proportional to stress. The proportionality constant (slope of this linear region) is called the *modulus of elasticity*. The modulus of elasticity or *Young's modulus* is a property of the stressed material. In fact, the magnitude of the modulus can be related to the nature of the chemical bonds in the material. Therefore, the modulus provides a measure of the strength of the bonding in the material being investigated. High values of modulus indicate that strong bonding is present in the material. As one might surmise from the previous discussion, materials having strong covalent bonding have the highest modulus values. Thus, the larger the value for modulus, the stronger the bonding is expected to be in the material. Modulus has the same units as stress (psi or MPa). The rubber industry also refers to the modulus of a compound. They give it a specific designation such as 100% modulus or 300% modulus. This is because the value generated is not an engineering modulus as mentioned previously, but rather is the stress required to obtain a given strain. Therefore, the "100% modulus," is simply the stress (σ) required to elongate the elastomer to twice its reference gage length. Rather than representing the slope of a region in a stress-strain curve, the 100% modulus represents a single data point on the curve. The 100% modulus or tensile stress of an elastomer has identical units as the engineering modulus.

Since we are interested in measuring changes in the tensile stress of the exposed material from that of unexposed or pristine material (7.57 MPa), we will discuss the % change in tensile stress of the materials. This is calculated from the relationship given in Eq. 2

$$\% \text{ Change in Tensile Stress} = (\sigma_f - \sigma_o) / \sigma_o \times 100, \quad \text{Eq. 2}$$

where σ_f is the measured tensile stress under the specific environmental conditions at 100% elongation and σ_0 is the tensile stress of the pristine material at 100% elongation. The 100% modulus changes can be positive or negative in value depending on the magnitude of either σ_f or σ_0 . Positive changes in % tensile stress indicate that the material of interest required greater application of stress to elongate the elastomer 100% than did pristine material. Negative values indicate that the material of interest required less application of stress than did the pristine material. Appendix J provides the actual tensile stress values of FKM rubber under the different environmental conditions along with the % change.

In Figure 15, the average % change in tensile stress of FKM rubber exposed to the four gamma radiation doses followed by exposure at 18, 50, and 60°C for 7, 14, 28, and 180 days is given. It should again be mentioned that the scale is $\pm 100\%$. Therefore even small bar heights could represent changes of up to 10% in tensile stress. As can be seen from these data, exposure to

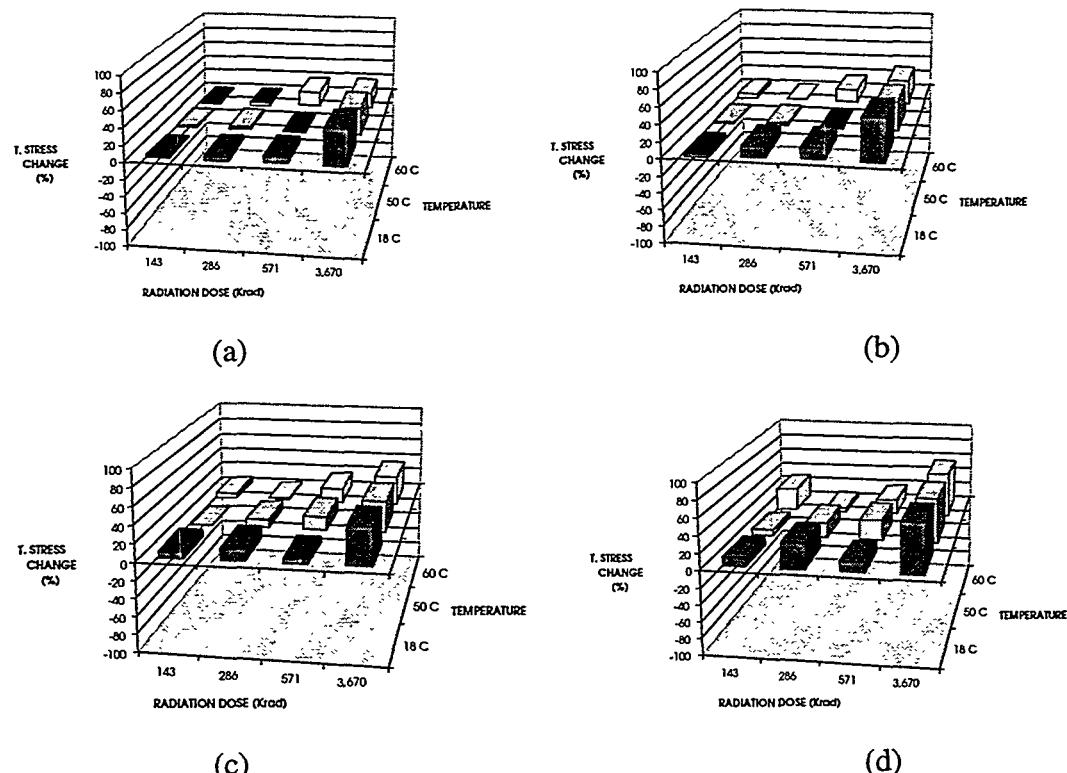


Figure 15. Tensile stress (T. Stress) changes in FKM rubber after exposure to ~ 143 , 286 , 571 , and $3,670$ Krad of gamma radiation followed by exposure for (a) 7 days, (b) 14 days, (c) 28 days, and (d) 180 days at 18 , 50 , and 60°C .

increasing gamma radiation doses below 3,670 Krad does not result in very large (>20%) increases in tensile stress. At the highest radiation dose of 3,670 Krad, extremely large (20% - 60%) changes in tensile stress were observed. Increasing the exposure temperatures has no dramatic effect on the tensile stress of FKM rubber, especially at the shorter exposure times. The changes ranged from -3% to just over 18%. Even though some of the samples exhibited a slight decrease in tensile stress, most samples exhibited increases in tensile stress. At the highest radiation dose, there is a general trend in decreasing tensile stress values with increasing temperatures. Over the four exposure times, the decreases in tensile stress ranged from 58% to 18%. These trends are generally consistent with hardening in the material, that is, decreased elasticity of the elastomer. The latter observation is in agreement with a decrease in the rubbery qualities of the material confirmed by decreases in compression set (Figure 7) and decreases in ultimate elongation (Figure 13).

Figure 16 shows the average % change in tensile stress of FKM rubber exposed to the four gamma radiation doses followed by exposure at 18, 50, and 60°C to the aqueous simulant waste for 7, 14, 28, and 180 days. In contrast to the data in Figure 15, the larger decreases in tensile stress were observed in FKM samples where exposure to only the simulant occurred. The other notable difference is the effect of elevated temperatures. For samples exposed to only the simulant, the largest decrease in tensile stress was on the order of ~73%. With exposure to radiation, the tensile stress values actually increased slightly; that is, the changes became less negative in value. For example, in Figure 16c the tensile stress change values for samples exposed at 50°C increased from -48% to -37%. At longest exposure time of 180 days (Figure 16d), the values at 50°C ranged from approximately -73% to -57% of the pristine FKM rubber's tensile stress values. However, when the case of 180-day exposures at 60°C is examined, a calculation of change in tensile stress was not possible for samples exposed to 3,670 Krad. The inability to calculate tensile stress change requires further explanation. For these samples, it should be mentioned that the average ultimate elongation was 84%. It should be recalled that tensile stress, as discussed in this section, is also known as the 100% modulus. Since these samples only elongated to 84%, it should be clear that a 100% modulus is not applicable here. For this reason, the bar in Figure 16d for this data point has a notation of N/A applied to it. Comparing the results of Figures 15 and 16, we find that FKM rubber exposed to both radiation and the simulant waste had lower tensile stress values at the higher radiation doses. Since

tensile stress is a measurement of the stiffness²⁴ of the elastomer, the combination of radiation and chemical exposure resulted in a decrease in the stiffness of the material. These interesting results suggest that simulant counteracted the embrittling action of radiation. Further studies are required to more fully understand this interesting phenomenon. However, since the simulant causes such dramatic degradation in FKM rubber at elevated temperatures, the effect of radiation is overcome by the much greater chemical effect. It appears that FKM rubber is remarkably susceptible to the effects of the combination of radiation and simulant. At the higher radiation doses and higher temperature, the loss of tensile stress in FKM rubber is quite evident.

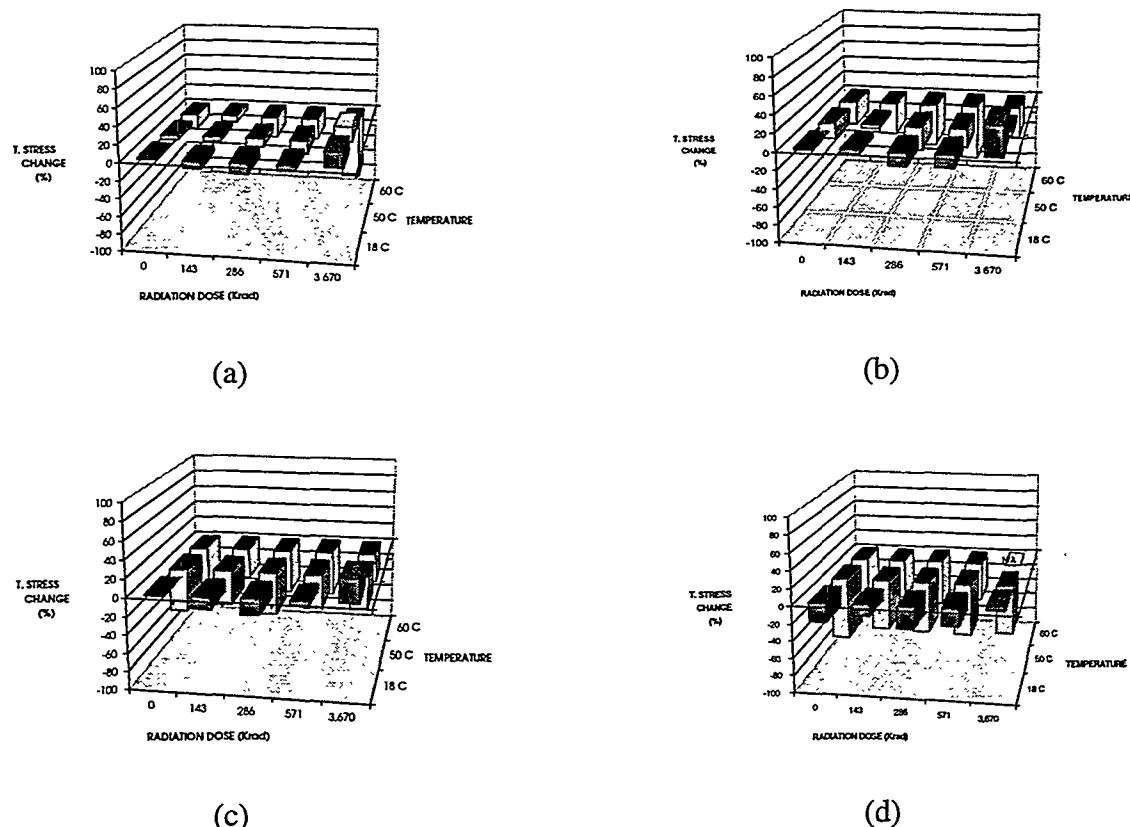


Figure 16. Tensile stress (T. Stress) changes in FKM rubber after exposure to ~ 0, 143, 286, 571, and 3,670 Krad of gamma radiation followed by exposure for (a) 7 days, (b) 14 days, (c) 28 days, and (d) 180 days to the aqueous simulant waste at 18, 50, and 60°C.

DISCUSSION

The Chemical Interaction Program, previously referred to as the Chemical Compatibility Program,¹ provides a scientifically defensible methodology for measuring the chemical interactions of polymeric liner and seal materials with hazardous wastes. These polymeric materials are those that may be used in current and future container designs for the transportation of hazardous and mixed wastes throughout the DOE complex.

Since the completion of the screening phase of the program several years ago, the comprehensive phase of this program has been in progress. Because all seal and liner materials passed the screening tests *when exposed to the aqueous simulant Hanford tank waste*, 10 materials needed to be subjected to the test matrix. This resulted in an extremely large sample and test set. In view of manpower and budget constraints, the comprehensive testing phase of the program was further subdivided into the testing of liner materials and seal materials. The results of liner testing were the subject of a SAND report.⁸ Because of further funding constraints, the comprehensive testing of seal materials was subdivided into the testing of individual elastomers. In previous reports,¹⁷⁻¹⁹ we discussed the results from testing of the first three elastomeric seal materials, EPDM, butyl, and SBR rubber. This report describes the testing results from another elastomeric seal material, FKM rubber. We now discuss the results for each of the properties measured starting with specific gravity and ending with tensile stress of FKM rubber.

Overall, the specific gravity data show that simulant temperatures near 18°C and 7-day exposure times have slight effects on the specific gravity of FKM rubber. With higher temperatures and longer exposure times, substantial changes in specific gravity are observed. These results are consistent with the known sensitivity of this elastomer towards caustic aqueous solutions at elevated temperatures.²⁰ It also demonstrates that FKM rubber is not a suitable elastomer for use under these conditions if specific gravity is the determining design criterion for selection of packaging seal components. Increasing exposure times, radiation doses, and exposure temperatures generally caused decreases in specific gravity of nearly than 4%. The greatest changes were noted for the samples exposed to the most extreme exposure conditions, that is, at 3,670 Krad and 60°C. The FKM rubber samples, which were not irradiated in most instances, displayed very similar decreases in specific gravity compared to samples exposed to both

radiation and the simulant. However, with longer exposure times, specific gravity values similar to combination conditions were observed. These results indicate that FKM rubber is significantly affected by the chemical exposure, which is then further affected by radiation exposure. This suggests that FKM rubber is less resistant to chemical exposure, and radiation may actually help reduce the negative effect of such exposure.

The mass of FKM rubber increased slightly after exposure to the simulant or the combination of radiation and simulant at increasing exposure times and low exposure temperatures. These increases in mass were most pronounced at the elevated temperatures of 50°C and 60°C. At 18°C, FKM rubber exhibited slight (~0.3%) weight gains. The largest increases in mass of ~52% were observed after 180-day exposures. Since the mass changes are large, the slight decreases in specific gravity noted earlier could be from a combination of mass and dimensional changes. Specifically, the volume of the FKM rubber could increase to a greater degree than the increases in mass, resulting in a net decrease in specific gravity. In fact, this is exactly what was observed. For most sample volumes, increases in volume were observed. As will be recalled, the volume of the samples was calculated through multiplication of their dimensions. A further inspection of the changes in dimensions revealed that while the length and width of most samples increased substantially, the sample thickness increased to a much greater extent. Since thickness measurements are more accurate than measurements of sample length or width, increasing thickness indicates swelling of the material. It should be kept in mind that this swelling is relatively large (~0% to 52%). The large amount of swelling was observed at the 180-day exposures and elevated temperatures. The interesting aspect is that swelling appears not to be significantly dependent on radiation dose. More specifically, the thickness changes in samples that received no radiation exposure were similar to those in samples that received the highest radiation dose. Since dimensional changes are not isotropic in the rectangular geometry of the samples, large changes in one of the sample dimensions dominate changes in volume. In actual packaging, seals are in the form of O-rings. O-rings with their circular geometry may exhibit more isotropic behavior. Thus, the relatively small anisotropic changes in dimension may be even larger in O-rings where isotropic behavior is expected. The practical implication of these results are that FKM rubber O-rings, when directly exposed to a Hanford tank waste under similar conditions as used in this study, are expected to change dimensionally very much.

Analogous to the previously discussed dimensional property changes, the hardness of FKM rubber decreases with increasing exposure time under both radiation and simulant exposure. For samples exposed to only the simulant, the hardness does not change significantly at 18°C. With increasing temperature, larger decreases in hardness were noted. Additionally, increasing radiation levels appear to exert a beneficial effect on the retention of hardness by FKM rubber. This was found to be especially the case for FKM rubber exposed for 28 days. Under these conditions, many of the FKM rubber samples that received radiation doses below 3,670 Krad became harder after previously being softer. As the exposure time approached 180 days, radiation exposure caused large softening in FKM. Since FKM rubber expanded greatly under these conditions, the observed softening of the material at these severe conditions could be ascribed purely to a swelling phenomenon. Softening because of swelling could be caused by chain scission in the polymer or the chemical degradation of the compounded rubber itself. Confirmation of this process will require additional tests. The observation that radiation in combination with chemical exposure may have a beneficial effect on hardness suggests that the cross-linking and chain scission processes occur simultaneously. Possibly the combination of processes leads to a polymeric structure having generally shorter polymer segments, which are crosslinked because of radiation and chemical reactions.

Compression set measures the retention of elastic properties of material after exposure to compressive stresses. When FKM rubber samples were exposed to only gamma radiation, set changes in the range of -12% to 1% were found. For samples exposed to both radiation and the simulant, the compression set change values increased slightly from -11% to 80%. The combined effects of radiation and chemical exposure resulted in increased compression sets; that is, the material became more deformed or less rubbery. These deformations are most pronounced in samples to the simulant at 60°C. An example of the degree to which FKM samples were deformed on compression is shown in Figure 17.

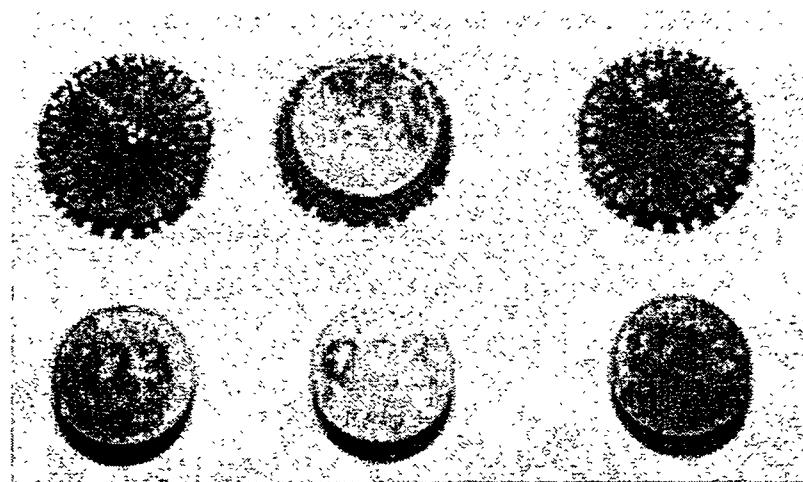


Figure 17. Compression set samples exposed for 180 days at 60°C – 286 Krad gamma radiation alone (R23-Bottom) and a combination of 286 Krad gamma radiation and simulant (23-Top).

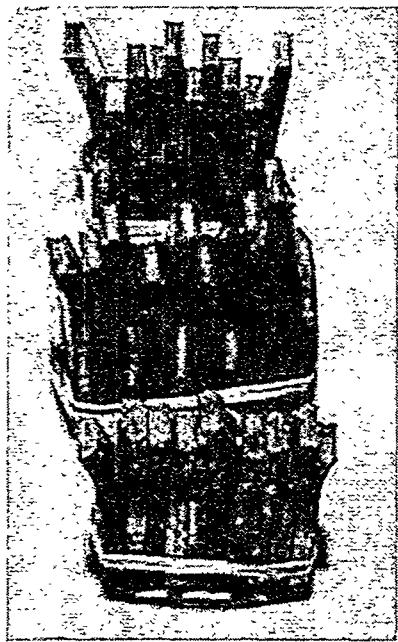
While the bottom samples (R23), which had only been exposed to the gamma radiation at elevated temperature, appear undeformed, the top samples (23), which received both radiation and simulant exposure, were cracked, that is, no longer rubbery. Compared with the hardness results, while the FKM rubber became softer at the longest exposure time, its elastic properties appear to have suffered dramatically under these conditions. As was discussed previously, perhaps the competition between cross-linking, chain scission, and chemical degradation reactions could offer an explanation. However, other factors such as the interaction of the other constituents of elastomers (oils, vulcanizing aids, fillers, etc.) at these environmental conditions would need to be considered.

The VTR of FKM rubber changed very little when exposed to radiation and the aqueous simulant at 18°C. Most of the Vapor Transmission Rates (VTRs) for FKM rubber samples were less than 0.5 g/hr/m² under these conditions. These results are not unexpected because the permeation of water molecules through this polymeric network is expected to be slow. That this process is temperature dependent is confirmed by our results. As temperature is increased, the VTRs also increased. The interesting aspect of our results is that the VTRs appear to be lower in samples receiving the highest radiation dose when compared to other radiation doses. A possible explanation is that the radiation has resulted in a layer that reduces the transport of water vapor. It should however be noted that large VTRs were found after 180-day exposures at elevated

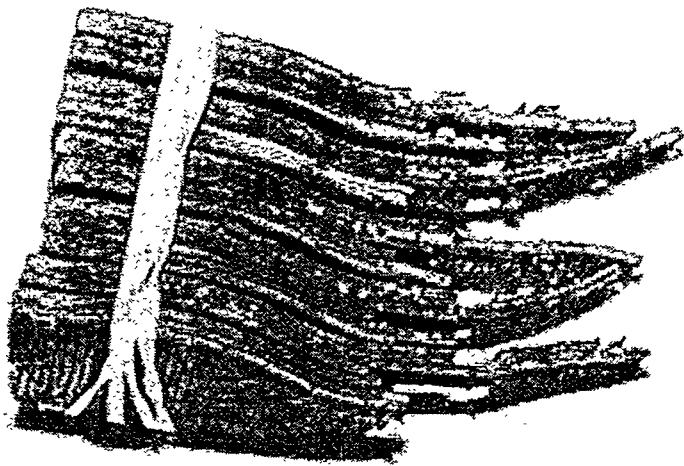
temperatures. These observations suggest that the postulated layers break down after some time period between 28 and 180 days. This leads to VTRs in the range of 2 – 4 g/hr/m².

The tensile strength of FKM rubber exhibits small changes when exposed to radiation and large changes when exposed to the simulant, and both radiation and simulant. For samples exposed to only radiation, increases in tensile strength of less than 20% were noted. Many of these samples increased less than 12% in tensile strength. The combination of high radiation doses and chemical exposure resulted in 50-80% decreases in tensile strength. At the highest radiation dose, the scission of polymeric chains may predominate as the determining process that leads to lower strength. While the material's strength may be retained at the lower temperature, the material's elasticity decreased dramatically at the highest temperature even for only 7-day exposures.

The elastic property of materials can be measured by evaluating their degree of ultimate elongation. For FKM rubber samples exposed to only gamma radiation, a general decrease in elongation was observed with increasing exposure time. These results indicate that the material is still elastic after shorter exposure times but less elastic at longer exposure time. However, because most FKM rubber samples were still rather elastic, that is, stretching about ~130%, a change of ~10% in ultimate elongation still results in fairly elastic material. For FKM rubber exposed to both radiation and simulant, there is a general decrease in elongation. This effect is most pronounced at the highest radiation dose, longest exposure time, and highest temperature. At intermediate radiation doses, the changes in ultimate elongation do not vary greatly. This particular material property appears not be affected greatly by various environmental parameters. A possible explanation is that the interior of the FKM sample may still be relatively shielded from the penetration of the simulant, that is, the simulant has not completely diffused into the interior of the sample. Thus, the interior of the material is still rather elastic, thereby maintaining the elongation of the material. This lack of total penetration by the simulant can be seen in the cross section of the fracture tensile specimens in Figure 18.



(a)



(b)

Figure 18. Tensile specimens after completion of several tests showing (a) cross section of fracture and (b) length-wise view.

Figure 18a clearly shows the cross section of three samples sets. From top to bottom, these samples represent tensile test specimens, which received 143 Krad gamma radiation followed by 180-day exposure at 18, 50, and 60°C, respectively. The presence of a relatively light area in the center of the specimens exposed to the higher temperature indicates relatively intact FKM. In Figure 18b, a set of tensile test specimens exposed for 180 days to the simulant at 60°C after the tensile test is shown. These specimens show that the outside layer had cracked and flaked off during elongation. The interior portion remained intact until the point of breaking. It is suspected that the inner layer retains some of the mechanical properties of FKM during the tensile test.

Finally, the tensile stress properties of FKM rubber are discussed. Tensile stress or 100% modulus measurements provide a measure of the stiffness of the elastomer. A larger tensile stress value indicates that the elastomer is more likely to recover from localized forces and thereby resist extrusion. The effect of radiation on the tensile stress values of FKM rubber did

not increase by more than 54%, nor did they decrease by more than 4%. The effects of radiation alone on FKM rubber resulted in much stiffer material at the highest radiation doses. The stiffness also decreased with increasing temperature at each of the four exposure times. This effect may be related to the flaking off of the outer layers during the test. Since the layer is least pronounced in the samples exposed to the lowest temperature, the largest

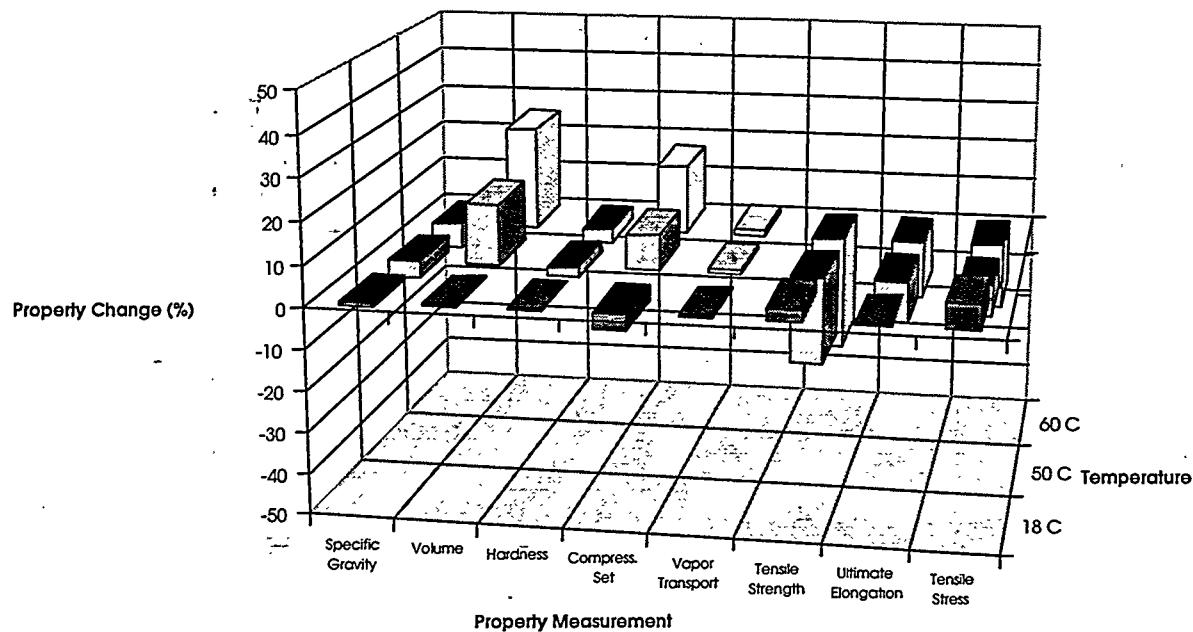


Figure 19. Summary graph for FKM rubber samples exposed to radiation, simulant, and to a combination of both radiation and simulant at 18, 50, and 60°C.

tensile stress is expected in these samples. For samples exposed to either only the simulant or to a combination of both radiation and simulant, decreases in tensile stress of as much as 73% and 62%, respectively, were observed. These results suggest that the simulant has a greater effect on the material, that is, the chemical makes FKM less stiff. The effect of radiation counteracts the effects of the simulant to some degree by slightly increasing the stiffness. As in previous observations, the simulant appears to cause significant degradation in the material. The observation that decreases in hardness, tensile strength, ultimate elongation, and tensile stress were observed under radiation and chemical exposures, while compression set increased, suggest that FKM rubber indeed has lost much of its elastic properties.

In summary, the measurement of changes in specific gravity, mass, volume, hardness, compression set, and VTRs indicated that FKM rubber is remarkably sensitive to the effects of radiation and the aqueous simulant at elevated temperatures and exposure times. A summary of the measurements is shown in Figure 19. The data shown in graphical form is an average of all the property change values obtained for samples exposed to radiation alone, the simulant alone, and the combination of both radiation and chemical exposure and all exposure times. While not differentiating the effects of each environmental condition, the summary graph does show a trend in the effect of increasing temperature on property changes. The material properties of specific gravity and hardness decrease with increasing temperature while volume, VTRs, and compression set increase under these conditions. From the tensile testing results, consisting of tensile strength, ultimate elongation, and tensile stress measurements, FKM rubber appears to be more susceptible to the effects the aqueous simulant at elevated temperatures. When compared to the previously obtained tensile testing results for SBR, EPDM rubber and butyl rubber, FKM appears to be the least resistant under these environmental conditions. The effect of radiation in counteracting the effects of the simulant in helping to reduce the changes in some material properties was noted. These results suggest that this particular aqueous mixed waste at elevated temperatures has large detrimental effects on FKM rubber.

CONCLUSIONS

We have developed a program for studying the chemical interactions of plastic packaging components that may be used in packaging for transporting mixed waste forms. Consistent with the methodology outlined in this report, we have performed the second phase of this experimental program to determine the effects of simulant Hanford tank mixed wastes on a packaging seal material, FKM rubber. This involved the comprehensive testing of FKM rubber with an aqueous mixed waste simulant. The testing protocol exposed the respective materials to ~143, 286, 571, and 3,670 Krad of gamma radiation followed by 7-, 14-, 28-, 180-day exposures to the waste simulant at 18, 50, and 60°C. From the analyses, we determined that FKM rubber has reasonably good resistance to radiation, but has poor resistance to the aqueous simulant, and a combination of both environmental factors. These results suggest that FKM rubber is a poor choice as a seal material to withstand aqueous mixed wastes having similar composition to the one used in this study at high temperatures. FKM rubber appears to respond less favorably compared to EPDM and SBR rubber when exposed to the simulant in this study at low temperatures (18°C). Both of these elastomers appear to be superior in performance when compared to butyl rubber.

REFERENCES

1. P. J. Nigrey. Chemical Compatibility Test Plan & Procedure Report, September 29, 1993, Sandia National Laboratories, Albuquerque, NM, unpublished (1993).
2. P. J. Nigrey, M. Conroy, and L. B. Blalock. "Mixed Waste Chemical Compatibility with Packaging Components," in *Proc. of SPECTRUM '94, Nuclear and Hazardous Waste Management International Topical Meeting*, Vol.1, pp. 386 - 391, American Nuclear Society, La Grange Park, Illinois (1994).
3. P. J. Nigrey and T. G. Dickens. "Effects of Mixed Waste Simulants on Transportation Packaging Plastic Components," Paper 95-TP-68.02, Presented at the 88th Annual Meeting & Exhibition of the Air & Waste Management Association San Antonio, TX (1995).
4. P. J. Nigrey. "Mixed Waste Chemical Compatibility: A Testing Program for Plastic Packaging Components," in *Proc. of PATRAM '95, The 11th International Symposium on the Packaging and Transportation of Radioactive Materials*, Vol. III, pp. 1372 - 1379 (1996).
5. P. J. Nigrey and T. G. Dickens. "Effects of Hanford Tank Simulant Waste on Plastic Packaging Components," in *Proc. of PATRAM '95, The 11th International Symposium on the Packaging and Transportation of Radioactive Materials*, Vol. III, pp. 1210 - 1216 (1996).
6. P. J. Nigrey and T. G. Dickens. "Chemical Compatibility Screening Results of Plastic Packaging Components to Mixed Waste Simulants," in *Proc. of PATRAM '95, The 11th International Symposium on the Packaging and Transportation of Radioactive Materials*, Vol. III, pp. 1202 - 1209 (1996).
7. P. J. Nigrey and T. G. Dickens. Chemical Compatibility Screening Test Results, SAND97-3104, Sandia National Laboratories, Albuquerque, NM (1997).
8. P. J. Nigrey and T. G. Dickens. Comprehensive Testing to Measure the Response of Liner Materials to Hanford Tank Waste Simulant, SAND99-0419, Sandia National Laboratories, Albuquerque, NM (1999).
9. P. J. Nigrey and T. G. Dickens. "A Testing Program to Evaluate the Effects of Simulant Mixed Wastes on Plastic Transportation Packaging Components," *Technology: Journal of the Franklin Institute*, Vol. 334A, pp. 337-349 (1997).
10. P. J. Nigrey and T. G. Dickens. "Effects of Simulant Mixed Waste on EPDM and Butyl Rubber," in *Proc. of PATRAM '98, The 12th International Symposium on the Packaging and Transportation of Radioactive Materials*, Vol. 1, pp. 423-428 (1998).
11. D 814-86, *Standard Test Method for Rubber Property - Vapor Transmission of Volatile Liquids*, American Society for Testing and Materials, Philadelphia, PA (1991).

12. D 412-87, *Standard Test Method for Rubber Properties in Tension*, American Society for Testing and Materials, Philadelphia, PA (1987).
13. D 1349-87, *Standard Practice for Rubber - Standard Temperatures for Testing*, American Society for Testing and Materials, Philadelphia, PA (1987).
14. E-104- 85, *Standard Practice for Maintaining Constant Relative Humidity by Means of Aqueous Solutions*, American Society for Testing and Materials, Philadelphia, PA (1991).
15. K. T. Gillen, R. L. Clough, and L. H. Jones. *Investigation of Cable Deterioration in the Containment Building of Savannah River Nuclear Reactor*, Sandia National Laboratories Report, SAND 81-2613 (1982).
16. P. J. Nigrey and T. G. Dickens. *Chemical Compatibility Screening Test Results*, DOE/HQ Milestone AL239004 (MS#106), Sandia National Laboratories, unpublished (1994).
17. P. J. Nigrey and T. G. Dickens. *Response of Ethylene Propylene Rubber (EPDM) to Simulant Hanford Tank Waste*, March 11, 1997, DOE/HQ Milestone 8986.41(#20), Sandia National Laboratories, unpublished (1997).
18. P. J. Nigrey and T. G. Dickens. *Comprehensive Testing of Butyl Rubber with Simulant Hanford Waste*, August 30, 1997, DOE/HQ Milestone # 8986.41 (MS#22), Sandia National Laboratories, unpublished (1997).
19. P. J. Nigrey and T. G. Dickens. *Response of Styrene Butadiene (SBR) Rubber to Simulant Hanford Tank Waste*, August 10, 1998, DOE/HQ Milestone 8986.41(FY98 MS#18), Sandia National Laboratories, unpublished (1998).
20. D 792-91, *Standard Test Method for Density and Specific Gravity (Relative Density) of Plastics by Displacement*, American Society for Testing and Materials, Philadelphia, PA (1991).
21. D 543-87, *Standard Test Method for Resistance of Plastics to Chemical Reagents*, American Society for Testing and Materials, Philadelphia, PA (1987).
22. D 2240-91, *Standard Test Method for Rubber Property - Durometer Hardness*, American Society for Testing and Materials, Philadelphia, PA (1991).
23. D 395-89, *Standard Test Method for Rubber Property - Compression Set*, American Society for Testing and Materials, Philadelphia, PA (1989).
24. R. J. Del Vecchio. "Tensile Testing of Elastomers," in *Tensile Testing* (P. Han, ed.), ASM International, The Materials Information Society, Materials Park, Ohio (1992), pp. 136- 146.

25. *Seals and Sealing Handbook*, M. Brown (Ed.), 4th Edition, Elsevier Advanced Technology, Kidlington, Oxford, UK, p. 554 (1995).

APPENDIX A
FKM Rubber Material Information

Material Supplier: Parker Seal Group*
 O-Ring Division
 2360 Palumbo Drive
 P.O. Box 11751
 Lexington, KY 40512
 (606) 269-2351

<u>Measurement</u>	<u>Initial Value</u>	<u>Cure Date</u>	<u>Batch Number</u>
Specific Gravity	2.1430	CD 1Q98	B324830
Dimensional	NA	CD 1Q98	B324830
Mass	NA	CD 1Q98	B324830
Hardness (Shore A)	72.6 points	CD 1Q98	B324830
Compression Set	16.4	CD 1Q98	B324830
Vapor Transport Rates	NA	CD 1Q98	B324830
Tensile Property		CD 1Q98	B324830
Tensile Strength	11.3 Mpa		
Ultimate Elongation	140%		
Tensile Stress	7.57 MPa		

* Procured from Parker Seal Group (Compound No. V0884-75) through Southwest Seal and Supply, 1413 1st Street NW, Albuquerque, NM 87102-1533, (505) 247-0265. This material was obtained in the form of molded sheets. The 12" (30.5 cm) square sheets were ~0.125" (0.317 cm) thick. **Cost: ~\$147/ft² (~ \$0.15/cm²)**

APPENDIX B
FKM Rubber Specific Gravity Data

AVERAGE SPECIFIC GRAVITY (SP.GR.) AND % CHANGE: FKM									
18 C	INITIAL	7 DAYS		14 DAYS		28 DAYS		180 DAYS	
RAD DOSE	SP. GR.	SP. GR.	% CHANGE	SP. GR.	% CHANGE	SP. GR.	% CHANGE	SP. GR.	% CHANGE
0K	2.1430	2.1466	0.17	2.1460	0.14	2.1448	0.08	2.1311	-0.56
143K	2.1467	2.1263	-0.95	2.0829	-2.97	2.1042	-1.98	1.8174	-15.34
286K	2.1449	2.1468	0.09	2.1464	0.07	2.1460	0.05	2.1284	-0.77
571K	2.1401	2.1434	0.15	2.1442	0.19	2.1404	0.01	2.1240	-0.75
3670K	2.1419	2.1447	0.13	2.1431	0.06	2.1416	-0.01	2.1302	-0.55
50 C	INITIAL	7 DAYS		14 DAYS		28 DAYS		180 DAYS	
RAD DOSE	SP. GR.	SP. GR.	% CHANGE	SP. GR.	% CHANGE	SP. GR.	% CHANGE	SP. GR.	% CHANGE
0K	2.1437	2.1344	-0.43	2.1222	-1.00	2.0759	-3.16	1.8641	-13.04
143K	2.1434	2.1332	-0.48	2.1302	-0.62	2.0190	-5.80	1.8652	-12.98
286K	2.1421	2.1369	-0.24	2.1305	-0.54	2.0909	-2.39	1.9570	-8.64
571K	2.1433	2.1405	-0.13	2.1304	-0.60	2.0937	-2.31	1.8643	-13.02
3670K	2.1334	2.1367	0.15	2.1219	-0.54	2.0403	-4.36	1.8295	-14.24
60 C	INITIAL	7 DAYS		14 DAYS		28 DAYS		180 DAYS	
RAD DOSE	SP. GR.	SP. GR.	% CHANGE	SP. GR.	% CHANGE	SP. GR.	% CHANGE	SP. GR.	% CHANGE
0K	2.1439	2.1128	-0.15	2.0702	-3.44	1.9377	-9.62	1.8457	-13.91
143K	2.1364	2.1252	-0.52	2.0169	-5.59	1.9120	-10.50	2.1257	-0.50
286K	2.1364	2.1311	-0.25	2.1114	-1.17	1.9874	-6.97	1.9231	-9.98
571K	2.1399	2.1151	-1.16	2.0135	-5.91	1.9526	-8.75	1.8276	-14.59
3670K	2.1410	2.1114	-1.38	2.0200	-5.65	1.9575	-8.57	1.8257	-14.73

APPENDIX C
FKM Rubber Mass Data

MASS (g) AND % CHANGE: FKM									
18 C	INITIAL	7 DAYS		14 DAYS		28 DAYS		180 DAYS	
RAD DOSE	WEIGHT	WEIGHT	% CHANGE	WEIGHT	% CHANGE	WEIGHT	% CHANGE	WEIGHT	% CHANGE
0K	12.9267	12.9337	0.05	12.9378	0.09	12.9422	0.12	12.9678	0.32
143K	12.6461	12.6574	0.09	12.6602	0.11	12.6694	0.18	12.7531	0.85
286K	12.8850	12.8962	0.09	12.9002	0.12	12.9062	0.16	13.0030	0.92
571K	12.8553	12.8671	0.09	12.8703	0.12	12.8760	0.16	12.9330	0.60
3670K	12.6684	12.6877	0.15	12.6888	0.16	12.6986	0.24	12.7529	0.67
50 C	INITIAL	7 DAYS		14 DAYS		28 DAYS		180 DAYS	
RAD DOSE	WEIGHT	WEIGHT	% CHANGE	WEIGHT	% CHANGE	WEIGHT	% CHANGE	WEIGHT	% CHANGE
0K	12.7559	12.8164	0.47	12.8363	0.63	12.8131	0.45	16.4337	28.83
143K	12.4404	12.5055	0.52	12.4898	0.40	12.5505	0.89	15.8605	27.49
286K	12.8501	12.9042	0.42	12.9165	0.52	13.0170	1.30	15.5749	21.20
571K	13.0244	13.0708	0.36	13.0642	0.31	13.0307	0.05	16.5179	26.82
3670K	12.8471	12.9184	0.55	12.8510	0.03	13.0642	1.69	17.0231	32.51
60 C	INITIAL	7 DAYS		14 DAYS		28 DAYS		180 DAYS	
RAD DOSE	WEIGHT	WEIGHT	% CHANGE	WEIGHT	% CHANGE	WEIGHT	% CHANGE	WEIGHT	% CHANGE
0K	13.0748	13.1262	0.39	13.3367	2.00	14.5408	11.21	17.6982	35.36
143K	13.1488	13.2131	0.49	13.0139	-1.03	14.2631	8.47	16.5243	25.67
286K	12.9423	13.0235	0.63	13.3426	3.09	13.9371	7.69	15.8942	22.81
571K	12.8620	12.9417	0.62	13.4611	4.66	15.5866	21.18	19.5523	52.02
3670K	12.9625	13.0457	0.64	13.4616	3.85	15.6892	21.04	19.0439	46.92

APPENDIX D
FKM Rubber Dimensional Data

VOLUME (mm ³) AND % CHANGE: FKM										
18 C	INITIAL	7 DAYS		14 DAYS		28 DAYS		180 DAYS		
		RAD DOSE	VOLUME	VOLUME	% CHANGE	VOLUME	% CHANGE	VOLUME	% CHANGE	
0K	5957	5956	5956	-0.02	5983	0.44	5961	0.07	5976	0.32
143K	5834	5824	5824	-0.17	5912	1.34	5881	0.81	5920	1.47
286K	5954	5959	5959	0.08	6000	0.77	5961	0.12	6030	1.28
571K	5941	5917	5917	-0.40	5932	-0.15	5964	0.39	6001	1.01
3670K	5848	5845	5845	-0.05	5875	0.46	5875	0.46	5911	1.08
50 C	INITIAL	7 DAYS		14 DAYS		28 DAYS		180 DAYS		
		RAD DOSE	VOLUME	VOLUME	% CHANGE	VOLUME	% CHANGE	VOLUME	% CHANGE	
0K	5882	5924	5924	0.71	5969	1.48	6128	4.18	9474	61.07
143K	5732	5831	5831	1.73	6019	5.01	6017	4.97	8793	53.40
286K	5930	5962	5962	0.54	6010	1.35	6134	3.44	8139	37.25
571K	6014	6026	6026	0.20	6092	1.30	6179	2.74	8925	48.40
3670K	5953	6001	6001	0.81	6056	1.73	6035	1.38	9838	65.26
60 C	INITIAL	7 DAYS		14 DAYS		28 DAYS		180 DAYS		
		RAD DOSE	VOLUME	VOLUME	% CHANGE	VOLUME	% CHANGE	VOLUME	% CHANGE	
0K	6053	6132	6132	1.31	6400	5.73	7394	22.15	9531	57.46
143K	6106	6167	6167	1.00	6215	1.79	7350	20.37	9861	61.50
286K	5979	6071	6071	1.54	6375	6.62	6965	16.49	8233	37.70
571K	5940	6045	6045	1.77	6653	12.00	8162	37.41	11001	85.20
3670K	5988	6504	6504	8.62	6825	13.98	8099	35.25	10730	79.19

LENGTH (mm) AND % CHANGE: FKM										
18 C	INITIAL	7 DAYS		14 DAYS		28 DAYS		180 DAYS		
		RAD DOSE	LENGTH	LENGTH	% CHANGE	LENGTH	% CHANGE	LENGTH	% CHANGE	
0K	75.57	75.59	75.59	0.03	75.60	0.04	75.59	0.03	75.60	0.04
143K	75.70	75.52	75.52	-0.24	75.88	0.24	75.88	0.24	75.87	0.22
286K	75.65	75.72	75.72	0.09	75.98	0.44	75.75	0.13	75.78	0.17
571K	75.69	75.90	75.90	0.28	75.68	-0.01	75.75	0.08	75.80	0.15
3670K	75.66	75.66	75.66	0.00	75.86	0.26	75.86	0.26	75.82	0.21
50 C	INITIAL	7 DAYS		14 DAYS		28 DAYS		180 DAYS		
		RAD DOSE	LENGTH	LENGTH	% CHANGE	LENGTH	% CHANGE	LENGTH	% CHANGE	
0K	75.60	75.69	75.69	0.12	75.67	0.09	75.53	-0.09	81.06	7.22
143K	75.67	75.66	75.66	-0.01	75.88	0.28	75.77	0.13	79.77	5.42
286K	75.69	75.61	75.61	-0.11	75.61	-0.11	75.60	-0.12	80.14	5.88
571K	75.66	75.74	75.74	0.11	75.66	0.00	75.29	-0.49	80.11	5.88
3670K	75.62	75.61	75.61	-0.01	75.50	-0.16	74.58	-1.38	80.36	6.27
60 C	INITIAL	7 DAYS		14 DAYS		28 DAYS		180 DAYS		
		RAD DOSE	LENGTH	LENGTH	% CHANGE	LENGTH	% CHANGE	LENGTH	% CHANGE	
0K	75.67	75.67	75.67	0.00	75.69	0.03	78.07	3.17	82.36	8.84
143K	75.64	75.49	75.49	-0.20	74.53	-1.47	77.91	3.00	80.64	6.61
286K	75.74	75.67	75.67	-0.09	75.98	0.32	78.32	3.41	80.58	6.39
571K	75.65	75.57	75.57	-0.11	76.37	0.95	79.99	5.74	83.02	9.74
3670K	75.65	76.18	76.18	0.70	76.56	1.20	79.51	5.10	82.01	8.41

APPENDIX D (cont.)
FKM Rubber Dimensional Data

WIDTH (mm) AND % CHANGE: FKM										
18 C	INITIAL	7 DAYS			14 DAYS		28 DAYS		180 DAYS	
RAD DOSE	WIDTH	WIDTH	% CHANGE	WIDTH	% CHANGE	WIDTH	% CHANGE	WIDTH	% CHANGE	
0K	25.19	25.20	0.04	25.23	0.16	25.21	0.08	25.24	0.20	
143K	25.21	25.18	-0.12	25.26	0.20	25.26	0.20	25.23	0.08	
286K	25.25	25.25	0.00	25.28	0.12	25.25	0.00	25.25	0.00	
571K	25.22	25.23	0.04	25.18	-0.16	25.24	0.08	25.27	0.20	
3670K	25.22	25.15	-0.28	25.19	-0.12	25.18	-0.16	25.23	0.04	
50 C	INITIAL	7 DAYS			14 DAYS		28 DAYS		180 DAYS	
RAD DOSE	WIDTH	WIDTH	% CHANGE	WIDTH	% CHANGE	WIDTH	% CHANGE	WIDTH	% CHANGE	
0K	25.21	25.25	0.16	25.26	0.20	25.32	0.44	27.54	9.24	
143K	25.18	25.22	0.16	25.24	0.24	25.48	1.19	26.93	6.95	
286K	25.18	25.23	0.20	25.24	0.24	25.30	0.48	27.31	8.46	
571K	25.18	25.27	0.36	25.22	0.16	25.28	0.40	27.29	8.38	
3670K	25.29	25.24	-0.20	25.19	-0.40	24.54	-2.97	27.14	7.32	
60 C	INITIAL	7 DAYS			14 DAYS		28 DAYS		180 DAYS	
RAD DOSE	WIDTH	WIDTH	% CHANGE	WIDTH	% CHANGE	WIDTH	% CHANGE	WIDTH	% CHANGE	
0K	25.23	25.26	0.12	25.48	0.99	26.49	4.99	27.76	10.03	
143K	25.34	25.25	-0.36	24.94	-1.58	26.29	3.75	27.58	8.84	
286K	25.22	25.30	0.32	25.55	1.31	26.19	3.85	27.26	8.09	
571K	25.22	25.30	0.32	25.72	1.98	27.06	7.30	27.95	10.82	
3670K	25.21	26.32	4.40	24.77	-1.75	26.84	6.47	27.71	9.92	

THICKNESS (mm) AND % CHANGE: FKM										
18 C	INITIAL	7 DAYS			14 DAYS		28 DAYS		180 DAYS	
RAD DOSE	THICKNESS	THICKNESS	% CHANGE							
0K	3.13	3.13	0.00	3.14	0.32	3.13	0.00	3.13	0.00	
143K	3.06	3.06	0.00	3.08	0.65	3.07	0.33	3.09	0.98	
286K	3.12	3.12	0.00	3.12	0.00	3.12	0.00	3.15	0.96	
571K	3.11	3.09	-0.64	3.11	0.00	3.12	0.32	3.13	0.64	
3670K	3.06	3.07	0.33	3.07	0.33	3.08	0.65	3.09	0.98	
50 C	INITIAL	7 DAYS			14 DAYS		28 DAYS		180 DAYS	
RAD DOSE	THICKNESS	THICKNESS	% CHANGE							
0K	3.09	3.10	0.32	3.12	0.97	3.20	3.56	4.24	37.22	
143K	3.01	3.06	1.66	3.14	4.32	3.12	3.65	4.09	35.88	
286K	3.11	3.12	0.32	3.15	1.29	3.21	3.22	3.72	19.61	
571K	3.16	3.15	-0.32	3.19	0.95	3.25	2.85	4.08	29.11	
3670K	3.11	3.14	0.96	3.18	2.25	3.29	5.79	4.51	45.02	
60 C	INITIAL	7 DAYS			14 DAYS		28 DAYS		180 DAYS	
RAD DOSE	THICKNESS	THICKNESS	% CHANGE							
0K	3.17	3.21	1.26	3.32	4.73	3.58	12.93	4.17	31.55	
143K	3.19	3.24	1.57	3.34	4.70	3.59	12.54	4.44	39.18	
286K	3.13	3.17	1.28	3.28	4.79	3.40	8.63	3.75	19.81	
571K	3.11	3.16	1.61	3.39	9.00	3.77	21.22	4.74	52.41	
3670K	3.14	3.24	3.18	3.46	10.19	3.80	21.02	4.72	50.32	

APPENDIX E
FKM Rubber Hardness Data

AVERAGE HARDNESS (TYPE A) AND % CHANGE: FKM									
18 C	INITIAL	7 DAYS		14 DAYS		28 DAYS		180 DAYS	
RAD DOSE	HARDNESS	HARDNESS	% CHANGE						
0K	71.5	72.5	1.4	71.8	0.4	71.7	0.3	72.3	1.1
143K	72.3	72.5	0.3	72.2	-0.1	72.6	0.4	69.9	-3.3
286K	72.3	73.1	1.1	72.3	0.0	72.1	-0.3	70.0	-3.2
571K	72.3	72.8	0.7	72.7	0.6	72.5	0.3	70.6	-2.4
3670K	71.9	73.5	2.2	73.4	2.1	73.4	2.1	73.7	2.5
50 C	INITIAL	7 DAYS		14 DAYS		28 DAYS		180 DAYS	
RAD DOSE	HARDNESS	HARDNESS	% CHANGE						
0K	72.0	71.7	-0.4	70.1	-2.6	68.5	-4.9	70.9	-1.5
143K	72.4	71.5	-1.2	71.9	-0.7	72.2	-0.3	69.9	-3.5
286K	72.7	72.3	-0.6	71.8	-1.2	71.2	-2.1	72.0	-1.0
571K	72.9	72.8	-0.1	72.3	-0.8	70.9	-2.7	68.2	-6.4
3670K	73.1	74.1	1.4	73.0	-0.1	67.7	-7.4	64.5	-11.8
60 C	INITIAL	7 DAYS		14 DAYS		28 DAYS		180 DAYS	
RAD DOSE	HARDNESS	HARDNESS	% CHANGE						
0K	72.5	71.8	-1.0	69.5	-4.1	66.9	-7.7	74.5	2.8
143K	73.4	72.3	-1.5	70.0	-4.6	73.5	0.1	67.8	-7.6
286K	73.4	72.8	-0.8	71.3	-2.9	73.5	0.1	74.0	0.8
571K	73.1	71.3	-2.5	72.4	-1.0	74.5	1.9	68.4	-6.4
3670K	72.6	70.5	-2.9	69.2	-4.7	72.3	-0.4	54.2	-25.3

APPENDIX F
FKM Rubber Compression Set Data

COMPRESSION SET (SET, %) AND CHANGE: FKM										
18 C	INITIAL	7 DAYS			14 DAYS		28 DAYS		180 DAYS	
		SET	SET	CHANGE	SET	CHANGE	SET	CHANGE	SET	CHANGE
RAD DOSE	SET	SET	SET	CHANGE	SET	SET	SET	SET	SET	CHANGE
0K	16.39	20.09	3.70		18.93	2.54	19.89	3.50	19.39	3.00
143K	16.39	12.47	-3.92		10.91	-5.48	12.63	-3.76	10.92	-5.47
286K	16.39	16.49	0.10		9.63	-6.76	20.17	3.78	5.46	-10.93
571K	16.39	13.28	-3.11		11.21	-5.18	10.13	-6.26	11.93	-4.46
3670K	16.39	5.50	-10.89		14.66	-1.73	8.39	-8.00	15.98	-0.41
50 C	INITIAL	7 DAYS			14 DAYS		28 DAYS		180 DAYS	
		SET	SET	CHANGE	SET	SET	SET	SET	SET	CHANGE
RAD DOSE	SET	SET	SET	CHANGE	SET	SET	SET	SET	SET	CHANGE
0K	16.39	16.22	-0.17		20.50	4.11	46.46	30.07	70.64	54.25
143K	16.39	16.60	0.21		18.86	2.47	48.52	32.13	95.34	78.95
286K	16.39	14.26	-2.13		18.79	2.40	44.02	27.63	94.89	78.50
571K	16.39	12.02	-4.37		18.65	2.26	44.77	28.38	96.85	80.46
3670K	16.39	10.85	-5.54		24.11	7.72	69.11	52.72	93.21	76.82
60 C	INITIAL	7 DAYS			14 DAYS		28 DAYS		180 DAYS	
		SET	SET	CHANGE	SET	SET	SET	SET	SET	CHANGE
RAD DOSE	SET	SET	SET	CHANGE	SET	SET	SET	SET	SET	CHANGE
0K	16.39	24.72	8.33		56.61	40.22	56.12	39.73	67.27	50.88
143K	16.39	26.62	10.23		72.33	55.94	57.16	40.77	95.14	78.75
286K	16.39	19.86	3.47		40.05	23.66	70.40	54.01	87.29	70.90
571K	16.39	29.81	13.42		58.36	41.97	75.78	59.39	95.91	79.52
3670K	16.39	25.75	9.36		61.34	44.95	59.73	43.34	96.49	80.10
18 C, RAD ONLY	INITIAL	7 DAYS			14 DAYS		28 DAYS		180 DAYS	
		SET	SET	CHANGE	SET	SET	SET	SET	SET	CHANGE
RAD DOSE	SET	SET	SET	CHANGE	SET	SET	SET	SET	SET	CHANGE
143K	16.39	12.31	-4.08		10.66	-5.73	11.82	-4.57	7.34	-9.05
286K	16.39	14.44	-1.95		10.07	-6.32	17.10	0.71	3.98	-12.41
571K	16.39	15.81	-0.58		10.21	-6.18	7.12	-9.27	9.15	-7.24
3670K	16.39	5.42	-10.97		29.16	12.77	8.02	-8.37	10.24	-6.15
50 C, RAD ONLY	INITIAL	7 DAYS			14 DAYS		28 DAYS		180 DAYS	
		SET	SET	CHANGE	SET	SET	SET	SET	SET	CHANGE
RAD DOSE	SET	SET	SET	CHANGE	SET	SET	SET	SET	SET	CHANGE
143K	16.39	9.73	-6.66		8.57	-7.82	9.17	-7.22	9.11	-7.28
286K	16.39	9.44	-6.95		8.84	-7.55	10.99	-5.40	7.65	-8.74
571K	16.39	8.81	-7.58		8.45	-7.94	8.78	-7.61	9.01	-7.38
3670K	16.39	5.5	-10.89		7.15	-9.24	6.83	-9.56	7.76	-8.63
60 C, RAD ONLY	INITIAL	7 DAYS			14 DAYS		28 DAYS		180 DAYS	
		SET	SET	CHANGE	SET	SET	SET	SET	SET	CHANGE
RAD DOSE	SET	SET	SET	CHANGE	SET	SET	SET	SET	SET	CHANGE
143K	16.39	8.23	-8.16		7.99	-8.40	8.25	-8.14	8.05	-8.34
286K	16.39	7.76	-8.63		7.33	-9.06	9.62	-6.77	6.15	-10.24
571K	16.39	7.47	-8.92		8.33	-8.06	9.98	-6.41	7.95	-8.44
3670K	16.39	5.26	-11.13		5.54	-10.85	5.82	-10.57	7.22	-9.17

APPENDIX G
FKM Rubber Vapor Transport Rate Data

VAPOR TRANSMISSION RATE (g/hr/m²): FKM				
18 C	7 DAYS	14 DAYS	28 DAYS	180 DAYS
RADIATION DOSE	VAPOR TRANSMISSION	VAPOR TRANSMISSION	VAPOR TRANSMISSION	VAPOR TRANSMISSION
0K	0.0975	0.1245	0.1597	0.0726
143K	0.5323	0.4562	0.4805	0.2654
286K	0.5361	0.5036	0.4752	0.2142
571K	0.4224	0.4955	0.3317	0.1384
3670K	0.0921	0.0758	0.0690	0.0491
50 C	7 DAYS	14 DAYS	28 DAYS	180 DAYS
RADIATION DOSE	VAPOR TRANSMISSION	VAPOR TRANSMISSION	VAPOR TRANSMISSION	VAPOR TRANSMISSION
0K	0.4061	0.2708	0.2396	0.9771
143K	1.2458	1.0060	1.0047	1.9879
286K	0.4603	0.6931	0.7026	1.6080
571K	2.3853	1.9282	1.2711	1.2814
3670K	0.0668	0.0623	0.0934	0.9253
60 C	7 DAYS	14 DAYS	28 DAYS	180 DAYS
RADIATION DOSE	VAPOR TRANSMISSION	VAPOR TRANSMISSION	VAPOR TRANSMISSION	VAPOR TRANSMISSION
0K	0.6498	0.6038	0.9314	3.6571
143K	0.1082	0.1813	0.6101	2.7959
286K	0.9801	1.3565	1.9684	4.1079
571K	2.0902	1.7247	1.8899	3.3637
3670K	0.1625	0.3736	0.8948	2.8623

APPENDIX H
FKM Rubber Tensile Strength Data

TENSILE STRENGTH (TENS. STR., MPa) AND % CHANGE: FKM										
18 C		INITIAL	7 DAYS		14 DAYS		28 DAYS		180 DAYS	
RAD DOSE		TENS. STR.	TENS. STR.	% CHANGE						
0K		11.3	13.4	18.6	12.1	7.4	11.8	4.8	8.0	-29.2
143K		11.3	11.8	4.9	11.9	5.7	10.5	-7.3	9.3	-17.5
286K		11.3	11.9	5.5	12.4	9.8	10.8	-4.2	9.6	-15.4
571K		11.3	10.7	-5.0	11.6	2.3	11.7	3.8	10.0	-11.2
3670K		11.3	10.1	-10.3	13.0	14.8	11.1	-1.7	10.1	-10.3
50 C		INITIAL	7 DAYS		14 DAYS		28 DAYS		180 DAYS	
RAD DOSE		TENS. STR.	TENS. STR.	% CHANGE						
0K		11.3	10.9	-3.9	9.4	-16.5	4.9	-56.6	2.6	-77.1
143K		11.3	11.7	3.9	8.8	-22.1	4.5	-60.3	2.7	-76.1
286K		11.3	11.1	-1.8	7.3	-35.7	4.3	-62.2	3.9	-65.4
571K		11.3	10.2	-10.0	7.6	-32.8	5.2	-54.2	3.3	-70.5
3670K		11.3	9.5	-15.5	7.1	-37.1	4.9	-56.6	3.4	-69.9
60 C		INITIAL	7 DAYS		14 DAYS		28 DAYS		180 DAYS	
RAD DOSE		TENS. STR.	TENS. STR.	% CHANGE						
0K		11.3	7.1	-37.5	5.7	-49.9	4.6	-59.1	3.1	-72.6
143K		11.3	9.6	-14.8	5.7	-49.8	4.8	-57.3	4.6	-59.6
286K		11.3	7.1	-37.4	4.3	-62.1	3.8	-66.7	4.7	-58.8
571K		11.3	7.1	-36.9	3.6	-68.6	3.6	-68.6	4.7	-58.7
3670K		11.3	4.6	-59.0	5.4	-52.2	5.9	-48.0	3.6	-68.1
18 C, RAD ONLY		INITIAL	7 DAYS		14 DAYS		28 DAYS		180 DAYS	
RAD DOSE		TENS. STR.	TENS. STR.	% CHANGE						
143K		11.3	12.2	7.9	11.8	4.6	12.4	10.1	12.7	12.8
286K		11.3	11.2	-0.7	12.6	11.7	11.8	4.8	11.6	2.4
571K		11.3	12.3	8.5	12.1	7.0	12.5	10.9	12.0	5.9
3670K		11.3	12.8	13.1	12.6	11.6	11.6	3.1	13.9	23.3
50 C, RAD ONLY		INITIAL	7 DAYS		14 DAYS		28 DAYS		180 DAYS	
RAD DOSE		TENS. STR.	TENS. STR.	% CHANGE						
143K		11.3	10.6	-5.7	11.7	3.8	11.4	0.7	12.8	13.1
286K		11.3	11.2	-0.6	12.0	6.6	12.3	8.9	13.1	16.0
571K		11.3	10.9	-3.5	11.9	5.0	10.9	-3.2	12.7	12.2
3670K		11.3	12.0	5.9	11.3	0.1	11.3	0.4	10.5	-7.0
60 C, RAD ONLY		INITIAL	7 DAYS		14 DAYS		28 DAYS		180 DAYS	
RAD DOSE		TENS. STR.	TENS. STR.	% CHANGE						
143K		11.3	11.7	3.5	12.4	9.9	12.6	11.7	11.0	-2.9
286K		11.3	11.1	-1.3	11.7	3.9	12.3	9.2	12.6	11.9
571K		11.3	11.6	2.3	11.9	5.4	12.6	11.8	12.5	10.3
3670K		11.3	11.3	-0.1	11.3	0.5	11.4	0.9	11.0	-2.6

APPENDIX I
FKM Rubber Ultimate Elongation Data

ULTIMATE ELONGATION (ELONG., %) AND CHANGE: FKM									
18 C	INITIAL	7 DAYS		14 DAYS		28 DAYS		180 DAYS	
RAD DOSE	ELONG.	ELONG.	CHANGE	ELONG.	CHANGE	ELONG.	CHANGE	ELONG.	CHANGE
0K	140	161	15	152	9	149	6	156	11
143K	140	156	11	161	15	150	7	138	-1
286K	140	161	15	173	24	158	13	157	12
571K	140	142	1	161	15	153	9	159	14
3670K	140	111	-21	127	-9	115	-18	127	-9
50 C	INITIAL	7 DAYS		14 DAYS		28 DAYS		180 DAYS	
RAD DOSE	ELONG.	ELONG.	CHANGE	ELONG.	CHANGE	ELONG.	CHANGE	ELONG.	CHANGE
0K	140	147	5	152	9	95	-32	128	-9
143K	140	157	12	123	-12	102	-27	91	-35
286K	140	158	13	133	-5	113	-19	135	-4
571K	140	151	8	130	-7	117	-16	117	-16
3670K	140	109	-22	98	-30	104	-26	107	-24
60 C	INITIAL	7 DAYS		14 DAYS		28 DAYS		180 DAYS	
RAD DOSE	ELONG.	ELONG.	CHANGE	ELONG.	CHANGE	ELONG.	CHANGE	ELONG.	CHANGE
0K	140	115	-18	102	-27	137	-2	127	-9
143K	140	138	-1	95	-32	98	-30	122	-13
286K	140	126	-10	102	-27	106	-24	135	-4
571K	140	126	-10	86	-39	99	-29	119	-15
3670K	140	76	-46	113	-19	117	-16	84	-40
18 C, RAD ONLY	INITIAL	7 DAYS		14 DAYS		28 DAYS		180 DAYS	
RAD DOSE	ELONG.	ELONG.	CHANGE	ELONG.	CHANGE	ELONG.	CHANGE	ELONG.	CHANGE
143K	140	144	3	147	5	142	1	160	14
286K	140	132	-6	137	-2	131	-6	116	-17
571K	140	143	2	134	-4	142	1	135	-4
3670K	140	112	-20	110	-21	108	-23	94	-33
50 C, RAD ONLY	INITIAL	7 DAYS		14 DAYS		28 DAYS		180 DAYS	
RAD DOSE	ELONG.	ELONG.	CHANGE	ELONG.	CHANGE	ELONG.	CHANGE	ELONG.	CHANGE
143K	140	129	-8	140	0	138	-1	143	2
286K	140	135	-4	142	1	139	-1	137	-2
571K	140	135	-4	143	2	121	-14	129	-8
3670K	140	117	-16	110	-21	107	-24	112	-20
60 C, RAD ONLY	INITIAL	7 DAYS		14 DAYS		28 DAYS		180 DAYS	
RAD DOSE	ELONG.	ELONG.	CHANGE	ELONG.	CHANGE	ELONG.	CHANGE	ELONG.	CHANGE
143K	140	143	2	143	2	146	4	112	-20
286K	140	141	1	140	0	146	4	152	9
571K	140	124	-11	128	-9	135	-4	132	-6
3670K	140	106	-24	112	-20	118	-16	122	-13

APPENDIX J
FKM Rubber Tensile Stress Data

TENSILE STRESS (STRESS, MPa) AND % CHANGE: FKM								
18 C	INITIAL	7 DAYS		14 DAYS		28 DAYS		180 DAYS
RAD DOSE	STRESS	STRESS	% CHANGE	STRESS	% CHANGE	STRESS	% CHANGE	STRESS % CHANGE
0K	7.57	7.60	0.36	7.46	-1.5	7.45	-1.64	5.87 -22.4
143K	7.57	6.96	-8.11	7.26	-4.10	6.54	-13.57	6.58 -13.11
286K	7.57	6.67	-11.93	6.43	-15.03	6.25	-17.49	5.67 -25.14
571K	7.57	7.12	-6.0	6.47	-14.6	7.17	-5.3	6.01 -20.7
3670K	7.57	8.85	16.8	10.07	33.0	9.52	25.7	7.98 5.5
50 C	INITIAL	7 DAYS		14 DAYS		28 DAYS		180 DAYS
RAD DOSE	STRESS	STRESS	% CHANGE	STRESS	% CHANGE	STRESS	% CHANGE	STRESS % CHANGE
0K	7.57	7.16	-5.46	6.22	-17.85	3.95	-47.81	2.07 -72.68
143K	7.57	7.20	-4.92	7.28	-3.83	4.63	-38.80	3.10 -59.11
286K	7.57	6.81	-10.11	5.60	-26.05	3.99	-47.4	2.98 -60.7
571K	7.57	6.40	-15.5	5.96	-21.2	4.72	-37.7	2.88 -61.9
3670K	7.57	8.95	18.2	7.17	-5.3	4.81	-36.5	3.23 -57.4
60 C	INITIAL	7 DAYS		14 DAYS		28 DAYS		180 DAYS
RAD DOSE	STRESS	STRESS	% CHANGE	STRESS	% CHANGE	STRESS	% CHANGE	STRESS % CHANGE
0K	7.57	6.27	-17.12	5.47	-27.78	3.47	-54.2	2.52 -66.67
143K	7.57	7.04	-7.01	4.74	-37.4	4.94	-34.70	3.93 -48.09
286K	7.57	5.73	-24.32	3.87	-48.8	3.79	-49.9	3.50 -53.7
571K	7.57	5.65	-25.3	2.79	-63.1	3.75	-50.5	4.03 -46.7
3670K	7.57	2.21	-70.9	4.92	-35.1	5.11	-32.5	N/A N/A
18 C, RAD ONLY	INITIAL	7 DAYS		14 DAYS		28 DAYS		180 DAYS
RAD DOSE	STRESS	STRESS	% CHANGE	STRESS	% CHANGE	STRESS	% CHANGE	STRESS % CHANGE
143K	7.57	7.75	2.37	7.31	-3.46	8.11	7.1	8.36 10.4
286K	7.57	7.92	4.55	8.51	12.39	8.58	13.30	9.81 29.60
571K	7.57	8.12	7.29	8.49	12.20	8.00	5.65	8.45 11.66
3670K	7.57	10.78	42.4	11.43	51.0	10.78	42.3	11.98 58.3
50 C, RAD ONLY	INITIAL	7 DAYS		14 DAYS		28 DAYS		180 DAYS
RAD DOSE	STRESS	STRESS	% CHANGE	STRESS	% CHANGE	STRESS	% CHANGE	STRESS % CHANGE
143K	7.57	7.76	2.6	7.69	1.6	7.61	0.5	8.14 7.47
286K	7.57	7.81	3.10	7.86	3.83	8.27	9.29	8.99 18.76
571K	7.57	7.47	-1.28	7.49	-1.09	8.76	15.66	9.47 25.14
3670K	7.57	9.97	31.7	10.25	35.3	10.36	36.8	11.65 53.9
60 C, RAD ONLY	INITIAL	7 DAYS		14 DAYS		28 DAYS		180 DAYS
RAD DOSE	STRESS	STRESS	% CHANGE	STRESS	% CHANGE	STRESS	% CHANGE	STRESS % CHANGE
143K	7.57	7.42	-2.0	7.98	5.5	8.03	6.01	9.67 27.8
286K	7.57	7.29	-3.64	7.61	0.55	7.71	1.82	8.00 5.65
571K	7.57	8.92	17.85	8.77	15.85	8.81	16.39	8.92 17.85
3670K	7.57	8.96	18.3	9.82	29.7	10.14	33.9	11.71 54.6

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