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CHARACTERIZATION OF AN EPOXY FOAM

by

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THE Bendix CORPORATION
KANSAS CITY DIVISION

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INTRODUCTION

The premium placed on lightweight, high component density electronic packages by current aerospace design requirements dictates the investigation of foam type materials. Materials under consideration are primarily rigid urethane foam-in-place systems and to a lesser degree the syntactic foams. Recently a new candidate has been added to the list -- a foam-in-place one-component epoxy resin based foam.

This epoxy foam system has the same inherent properties as epoxy casting resins. Desirable characteristics include excellent chemical resistance, minimum moisture absorption, superior adhesive qualities, low shrinkage during cure, acceptable mechanical properties, and satisfactory electrical characteristics. In addition, the material is well suited for lightweight electronic potting because of its long pot life, excellent fluidity, low thermal conductivity, and its ability to be blown to a relatively low density.

Several mechanical, electrical, and thermal properties of this new one-component rigid epoxy foam were studied. All the properties that are necessary to fully characterize the material were not investigated. However, the data given indicates the foam system has a good potential for foamed-in-place electronic potting and encapsulation applications.

EXPERIMENTAL PROCEDURE

MATERIAL

The rigid epoxy foam system investigated is manufactured by Ablestik Adhesive Company, 833 West 182nd Street, Gardena, California. The material formulation is claimed to be proprietary, but is known to be a blend of aromatic and aliphatic epoxy resins, amine curing agents, and a solid blowing agent that decomposes when heated. Since the epoxy resins are premixed with the curing and blowing agents, the one-component system must be continuously maintained at minus 40°F or colder to prevent premature reaction. When stored at minus 40°F the shelf life is at least three months.

The premixed material is normally packaged in Semco polyethylene cartridges. In a room temperature environment the frozen premix will thaw to an extrudable temperature within one hour. The liquid premix has a pot life of approximately one hour after thawing.

FOAMING PARAMETERS

According to the manufacturer, this material was developed specifically as an encapsulating foam for electronic components. The material has been molded successfully in this application in foam densities of between 10 and 30 pounds per cubic foot (pcf). When molded at low densities or in open molds, the cell structure is undesirable. At high densities or in thick cross sections of foam the exotherm temperatures developed may be detrimental or prohibitive.

Since the blowing of foam is due to thermal decomposition of one component, the liquid premix will cure to a solid without blowing if the temperature does not exceed 120°F. For this reason, mold preheats usually are not required. If the time required for the material inside a mold to reach 120 to 130°F (blowing agent decomposition temperature) is greater than 45 to 60 minutes the molds should be preheated to insure consistent moldings. The minimum cure temperature recommended is 150°F. The maximum cure temperature evaluated was 325°F. The optimum cure time is four hours at 150°F for most applications. As with most other polymeric cellular materials, this epoxy foam has reduced strength and dimensional stability if used above its optimum cure temperature.

TEST METHODS

Whenever possible, specimens were tested by standard ASTM procedures. In some cases other accepted test methods were employed, particularly for the determination of thermal properties of the foam system. In all cases, test specimens were machined from 6 by 6 by 1 inch test blocks. Listed in Table 1 are the properties tested, testing method, and specimen size.

RESULTS AND DISCUSSION

MECHANICAL PROPERTIES

Compressive strength is a convenient test of the mechanical strength of a foam. Throughout this characterization study on

epoxy foam, the compressive strength was determined using 1 by 1 by 1 inch cubes. Since this foam system is basically an epoxy polymer using an amine curing agent, the exotherm temperature developed is significant and must be considered in any molding application. Since thick cross sections cannot be molded from the material, the compressive test specimens were not homogeneous foam samples cut from the center of a larger molded block as is the case for most data listed for rigid urethane foams. The samples were taken from molded 6 by 6 by 1 inch test blocks.

The test cubes cut from the 6 by 6 by 1 inch block had molded skin on two faces. Thus the two standard test directions (perpendicular and parallel to platen travel) have more significant effect on compressive strengths. Borrowing from the terminology of sandwich structure, edge testing is parallel to foam rise with the two skin faces carrying most of the load. In flat testing, the molded skin surfaces are on the platens and the lower density center portion is loaded perpendicular to foam rise. Figure 1 graphically describes a typical foam block.

The fact that the test specimens have high density skins on two faces significantly alters the strength depending upon the direction of testing. Figure 2 shows a typical stress-strain relationship for the epoxy foam investigated. Normally, both the edge and flat tests have about the same compressive moduli of approximately 30,000 psi at room temperature. At 200°F the modulus drops to about 12,000 psi. The flat specimens exhibit a yield point at between two and five percent strain. The edge specimens do not reach an ultimate before 10 percent strain.

As with other types of rigid polymeric foam materials, the strength is a function of the density. Figure 3 is a log-log plot of density versus strengths for foam cured four hours at 165°F. These curves allow the prediction of strength from density measurements, and are used to normalize test data to a particular chosen density. The curves for room temperature strengths have slopes of 1.7 and 1.9.

Figure 4 is a log-log plot of curves for compressive modulus versus density. The slope for the room temperature tests is about 1.65 with intercepts of 2 to 3. The 200°F test has a slope of about 1.2 with an intercept of 3. The data scatter is much greater for the 200°F tests than for the room temperature tests.

Due primarily to the exotherm temperature developed during cure, the recommended cure temperature for the epoxy foam investigated is 150°F. Since the strength is a function of cure temperature, most of this characterization study was carried out at a cure temperature of 165°F. The relationship between strength and test temperature for various densities is shown in Figure 5. All specimens were cured for four hours at 165°F. Regardless of the density, the ultimate test temperature seems to be between 250 and 300°F. This is approximately 100°F higher than the cure temperature.

Higher cure temperatures do give higher strengths. Figure 6 shows the effect of increasing cure temperature on strength of foam specimens at 14 pcf.

The tensile strength of the epoxy foam system was determined. The tests were performed at room temperature to obtain some idea of the tensile strength expected at various densities. It is evident from the data given in Table 2 that as the density increases so does the tensile strength of the foam.

Limited data has been collected on the shear strength of this epoxy foam. The shear strength was determined as a function of density for specimens cured at 165°F for four hours and tested at temperatures of 75 and 200°F. As shown by Figure 7, the shear strength varies from 650 psi at 25 pcf to about 200 psi at 10 pcf. The shear strength is, of course, reduced when the test temperature is increased to 200°F. At 200°F the shear strength is about 500 psi at 25 pcf and 150 psi at 10 pcf.

Flexural strength data as a function of density was collected on one set of test specimens. This data is shown in Table 3. Increasing the cure temperature from 165 to 200°F did tend to increase the flexural strength, but not significantly. When tested at room temperature, the flexural strength varies from about 1100 psi at 20 pcf to 300 psi at 10 pcf. At a 200°F test temperature the flexural strength of the epoxy foam varies from about 800 psi at 20 pcf to 200 psi at 10 pcf.

ELECTRICAL PROPERTIES

Electrical properties of polymeric materials can be important especially when the materials are used to package electronic equipment. However, the electrical properties are relatively difficult to measure and the test data still may not be reliable.

The dielectric strength, volume resistivity, dielectric constant, and dissipation factor have been measured as functions of density and frequency for the epoxy foam investigated.

To determine dielectric strengths, the 4 by 4 by 0.1 inch specimens were machined from the standard 6 by 6 by 1 inch test blocks. The dielectric strength is the voltage a material can withstand at electrical breakdown compared to the thickness of the test specimen. Thus, the units are volts per mil. The dielectric strength of solid epoxy in comparable thickness is 425 volts per mil. Tested values of the epoxy foam are shown in Figure 8 and are about 50 to 70 volts per mil over a density range of 10 to 15 pcf. Contrary to expected results, the dielectric strength decreased with increasing density.

The volume resistivity also was determined from specimens machined from 6 by 6 by 1 inch test blocks. Basically, the volume resistivity is a measure of the electrical resistance of a material or is a measure of the ability to resist current flow. The units are ohm-centimeters. Typical resistivity values of 10^{13} ohm-cm have been reported in the literature for epoxy foams. Figure 9 is a plot of volume resistivity versus density for the one-component epoxy foam system; values in the order of 10^{13} to 10^{10} ohm-cm over a density range of 10 to 18 pcf were measured. Volume resistivity measurements are difficult tests to run, and repeatable results are not always obtained. However, these data are consistent with data for other cellular materials.

The dielectric constant of a material is the ratio of the capacitance of the material to the capacitance of a vacuum measured with the same electrode configuration. The capacitance is that property of a system of conductors and dielectrics that allows the storage of electricity when a potential difference exists between the conductors. The units of capacitance are Farads or coulombs per volt. The dissipation factor is the tangent of the loss angle. Test values as a function of density for the dielectric strength are depicted in Figure 10 and the corresponding dissipation factors are shown in Figure 11.

THERMAL PROPERTIES

Thermal expansion of the epoxy foam was determined using a quartz tube dialtometer in a horizontal position. With this apparatus, the maximum test temperature was 165°F. The samples tended to deform at temperatures greater than 165°F and

gave erroneous results. In the range of -65°F to 165°F, the linear thermal expansion for densities of 17 to 32 pcf was 21 to 28 x 10^{-6} in/in/°F.

Thermogravimetric Analysis (TGA) tests have been made on samples of unfoamed solid epoxy cured at room temperature as well as samples cured at 165 and 325°F. Foam densities of 11 pcf and 22 pcf were investigated. The thermograms for all samples show a weight loss commencing at 220°C to 250°C and having the same general shape. From this limited data, it was shown that the percent residue increased with density and with cure temperature. A typical curve is shown in Figure 12.

Differential Thermal Analysis (DTA) data was collected on samples cured the same as for the TGA tests. The DTA traces all show an exotherm at 210 to 250°F which corresponds to the initial weight loss of the TGA. The DTA of the unfoamed epoxy cured at room temperature is interesting. This trace shows an endotherm at 50°C (120°F) followed immediately by a broad exotherm, as shown in Figure 13. The endotherm is the decomposition of the solid blowing agent followed by additional curing of the epoxy resin. The initial decomposition exotherm starts at about 260°C.

Thermomechanical Analysis (TMA) was used to determine the glass transition temperatures of samples cured at 165°F, 250°F, and 325°F for two, four, and eight hours. These results are given in Table 4. The results indicate that the T_g is increased markedly by increasing the cure temperature from 165 to 325°F. Increasing the cure time also increases the T_g.

CONCLUSIONS

A review of several of the mechanical, electrical, and thermal properties of a one-component rigid epoxy foam system indicates that the foam meets many of the requirements for an embedment material for electronic components. Because of the excellent fluidity, long pot life, and convenient packaging of the premix, many production and processing problems are reduced. The mechanical and electrical properties plus the thermal stability of the cured foam appears to make this epoxy foam a prime candidate for foamed-in-place electronic potting and encapsulation applications.

ACKNOWLEDGEMENTS

The authors wish to express their appreciation to the Test Laboratory for the various tests performed and to the Bendix Corporation, Kansas City, Missouri, for allowing the presentation of their work.

Table 1

Test Methods for Characterization of Epoxy Foam

PROPERTY	TEST METHOD	SPECIMEN SIZE
Mechanical		
Compressive Strength	ASTM D-695	1 by 1 by 1 inch
Tensile Strength	ASTM D-1623	1 by 1 by 1 inch Type B
Shear Strength	ASTM D-732	2 by 2 by 1/4 inch
Flexure Strength	ASTM D-790	5 by 1/2 by 1/2 inch
Thermal		
Thermal Expansion	ASTM D-696	0.45 inch diameter by 4 inches
Thermogravimetric Analysis (TGA)	15°C/minimum temperature rise/nitrogen atmosphere	
Thermomechanical Analyzer (TMA)	10°C/minimum temperature rise/5 gram weight penetration	
Differential Thermal Analysis (DTA)	15°C/minimum temperature rise/nitrogen atmosphere	
Electrical		
Dielectric Strength	ASTM D-149	4 by 4 by 0.1 inch
Volume Resistivity	ASTM D-257	4 by 4 by 0.1 inch
Dielectric Constant	ASTM D-150	2 inch diameter by 0.1 inch
Dissipation Factor	ASTM D-150	2 inch diameter by 0.1 inch

Table 2

Tensile Strength for Epoxy Foam

Foam Density, pcf	Test Orientation	Tensile Strength, psi
10	Parallel to rise	186
10	Perpendicular to rise	145
20	Parallel to rise	373
20	Perpendicular to rise	400
30	Parallel to rise	326
30	Perpendicular to rise	392

Table 3

Flexure Strength of Epoxy Foam

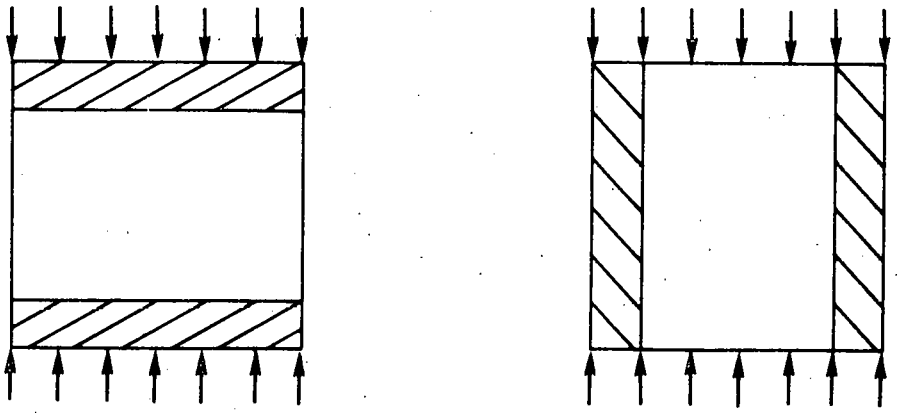
Density, pcf	Cure Temperature ^{o}F	Test Temperature ^{o}F	Flexural Strength, psi
10.0	165	75	381
16.5	165	75	657
17.3	165	75	907
9.9	165	200	216
14.6	165	200	445
16.8	165	200	575
12.2	200	75	475
18.4	200	75	955
20.7	200	75	1156
12.4	200	200	362
16.8	200	200	645
19.7	200	200	864

(Tested perpendicular to foam rise)

Table 4

Glass Transition Temperatures (T_g) of Epoxy Foam

Cure Condition		Glass Transition Temperature	
Temperature, °F	Time/Hrs.	°C	°F
165	4	101	214
165	4	113	235
165	4	110	230
165	4	103	217
250	2	124	255
250	4	138	280
250	4	137	279
250	8	140	284
325	2	133	271
325	4	140	284
325	8	160	320



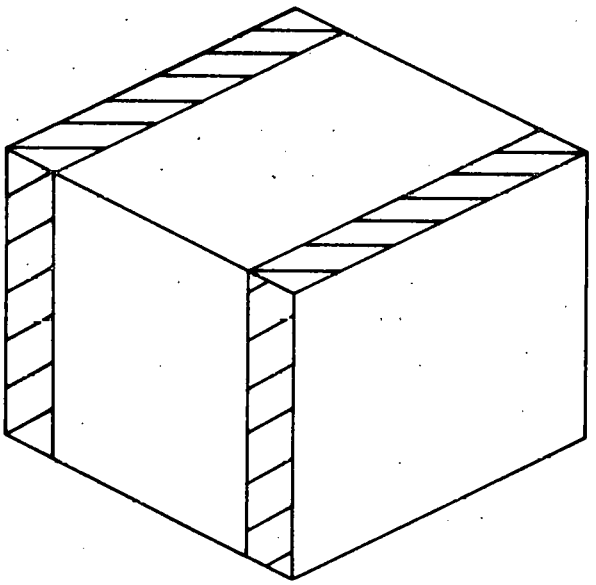


Figure 1. TYPICAL FOAM TEST SPECIMEN

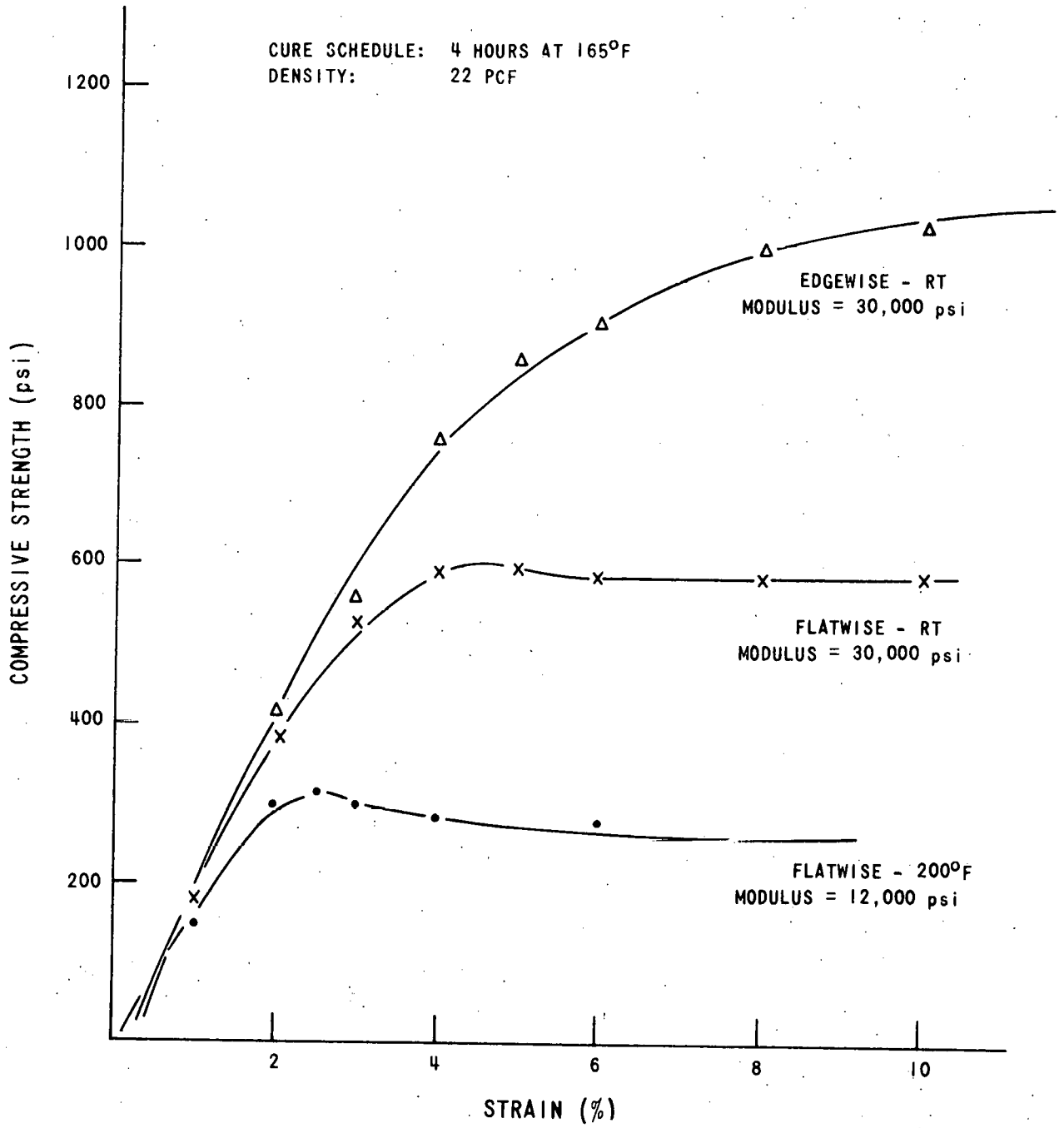


Figure 2. COMPRESSIVE STRESS-STRAIN CURVES FOR EPOXY FOAM

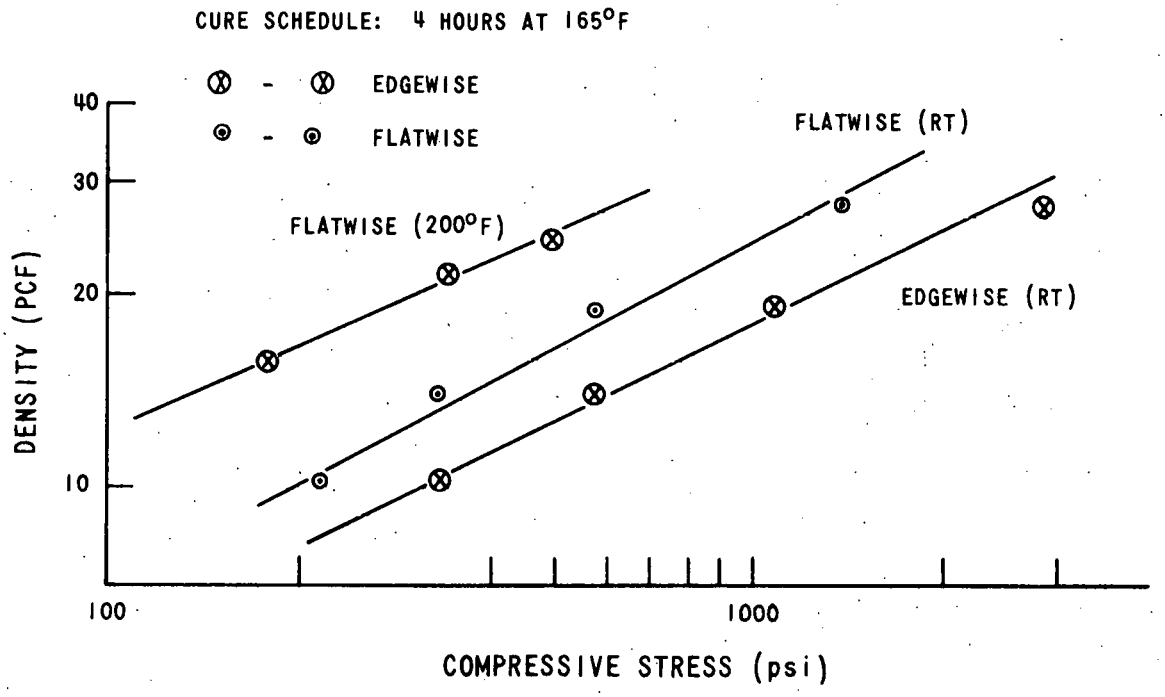


Figure 3. DENSITY VERSUS STRENGTH FOR EPOXY FOAM

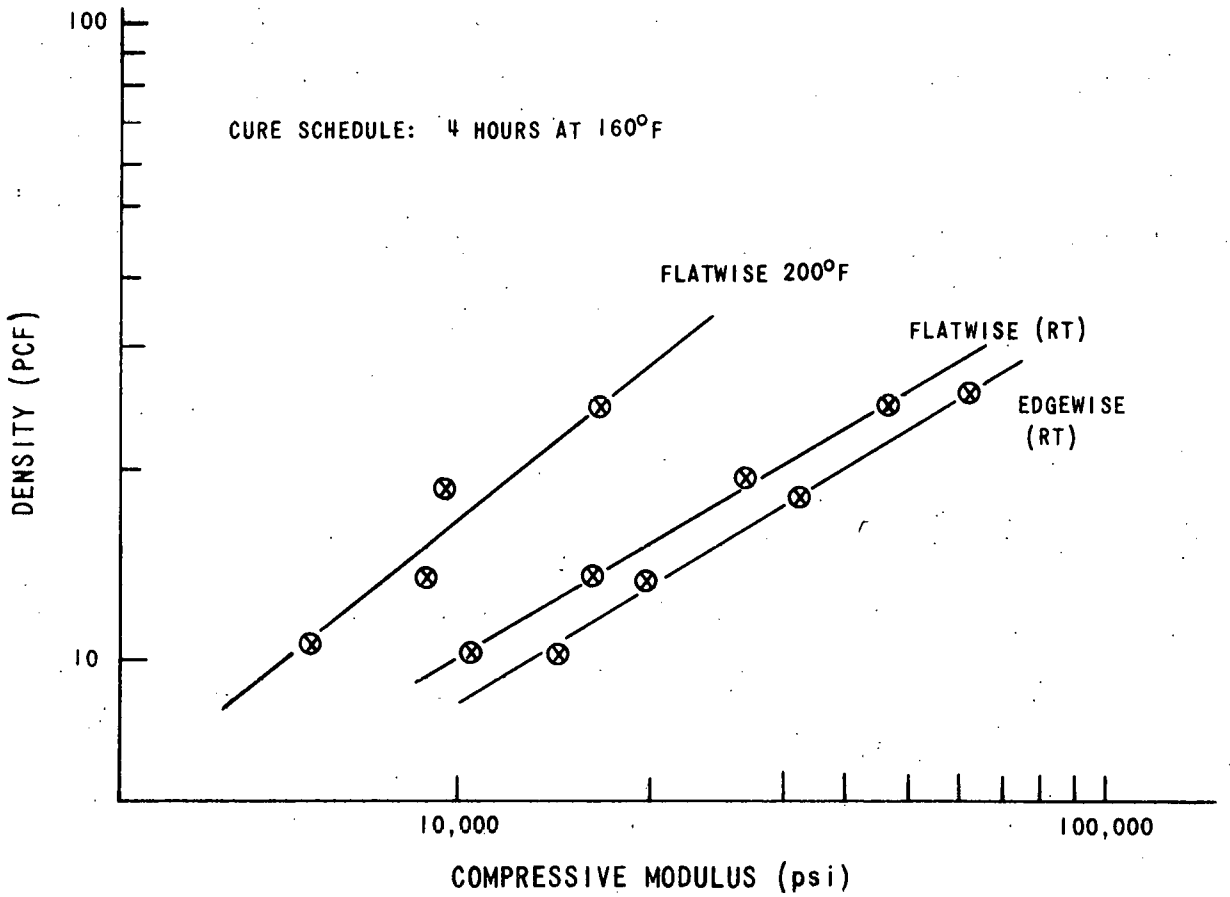


Figure 4. DENSITY VERSUS MODULUS FOR EPOXY FOAM

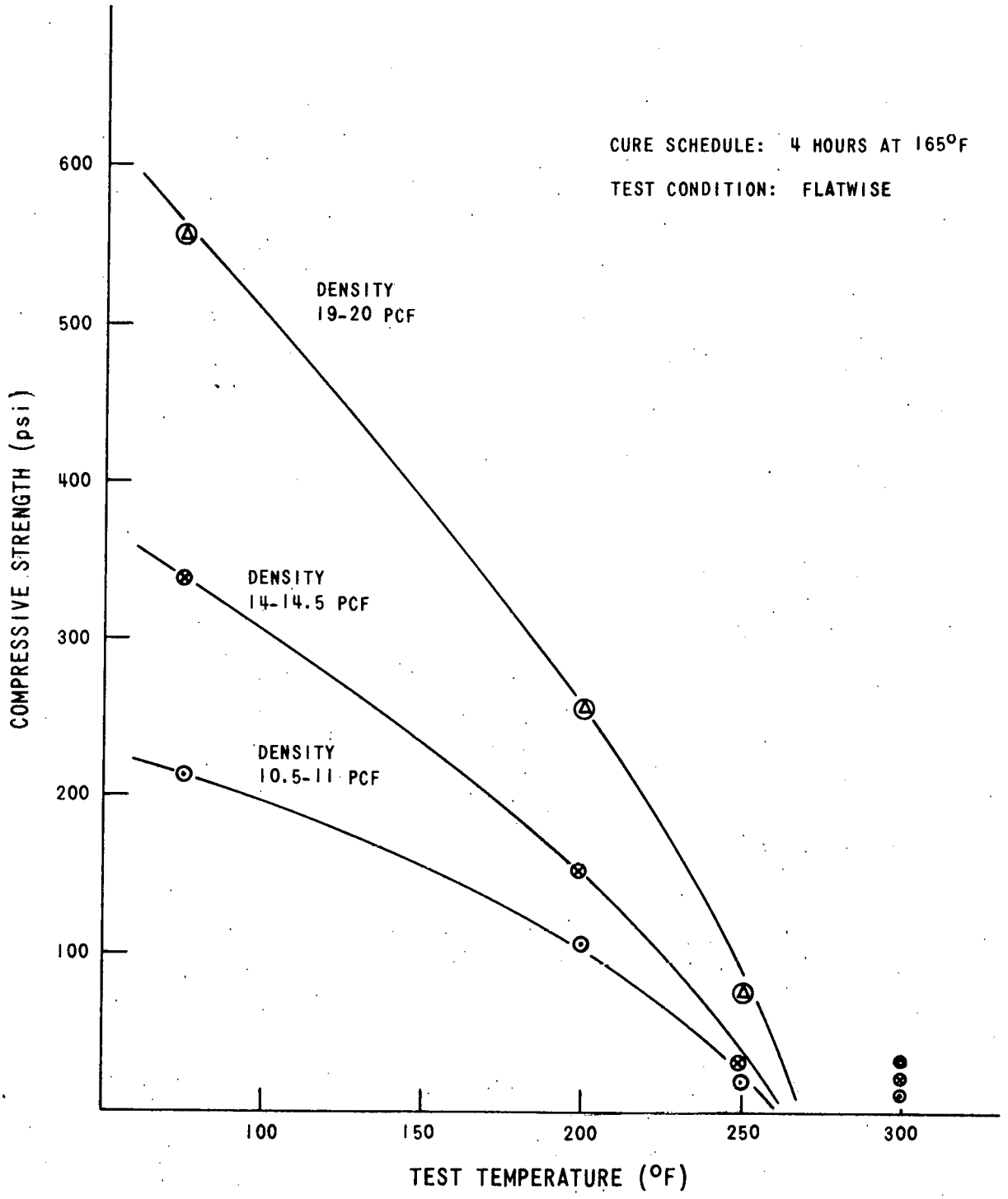


Figure 5. COMPRESSIVE STRENGTH VERSUS TEST TEMPERATURE FOR EPOXY FOAM

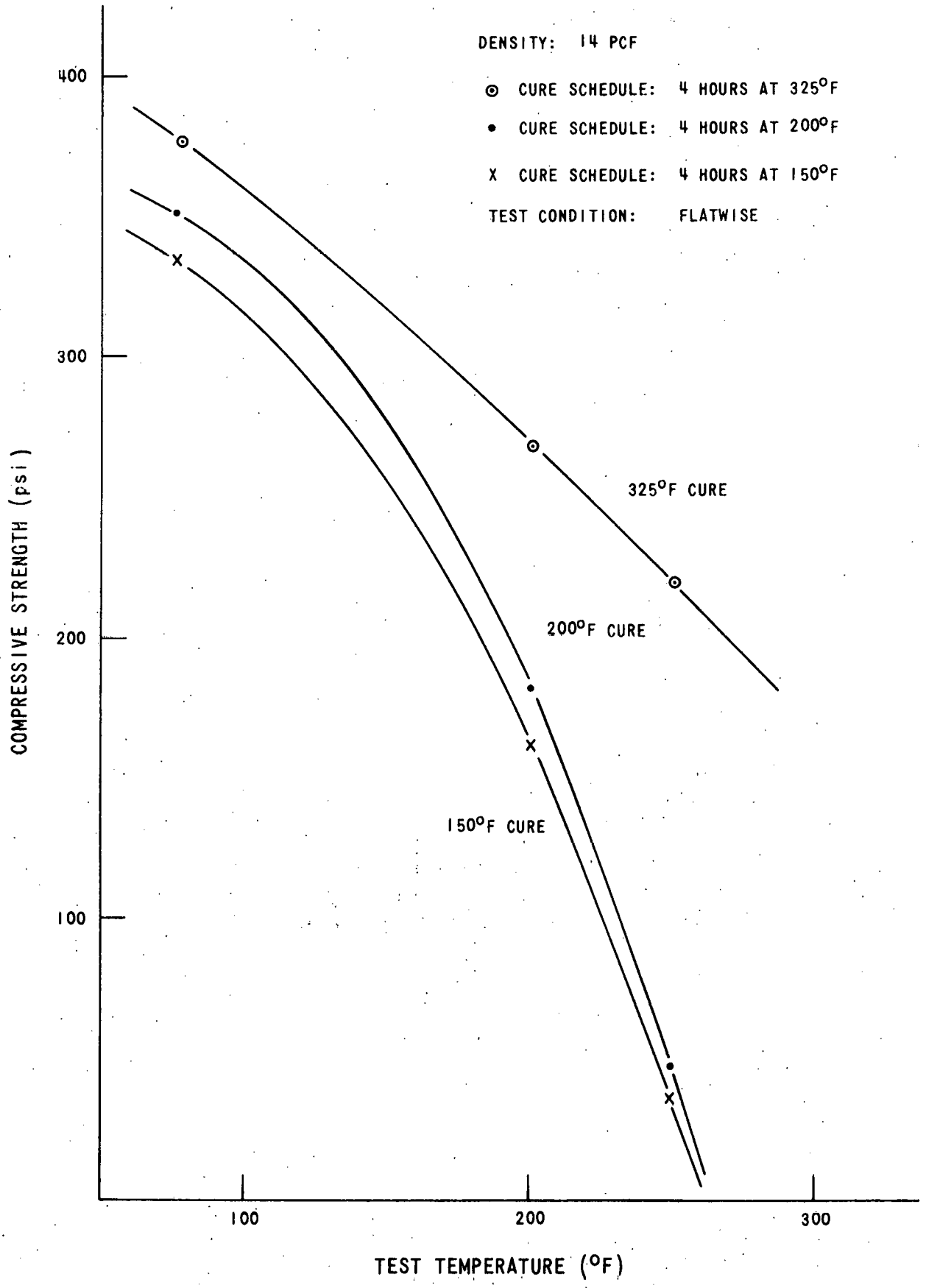


Figure 6. COMPRESSIVE STRENGTH VERSUS TEST TEMPERATURE FOR EPOXY FOAM

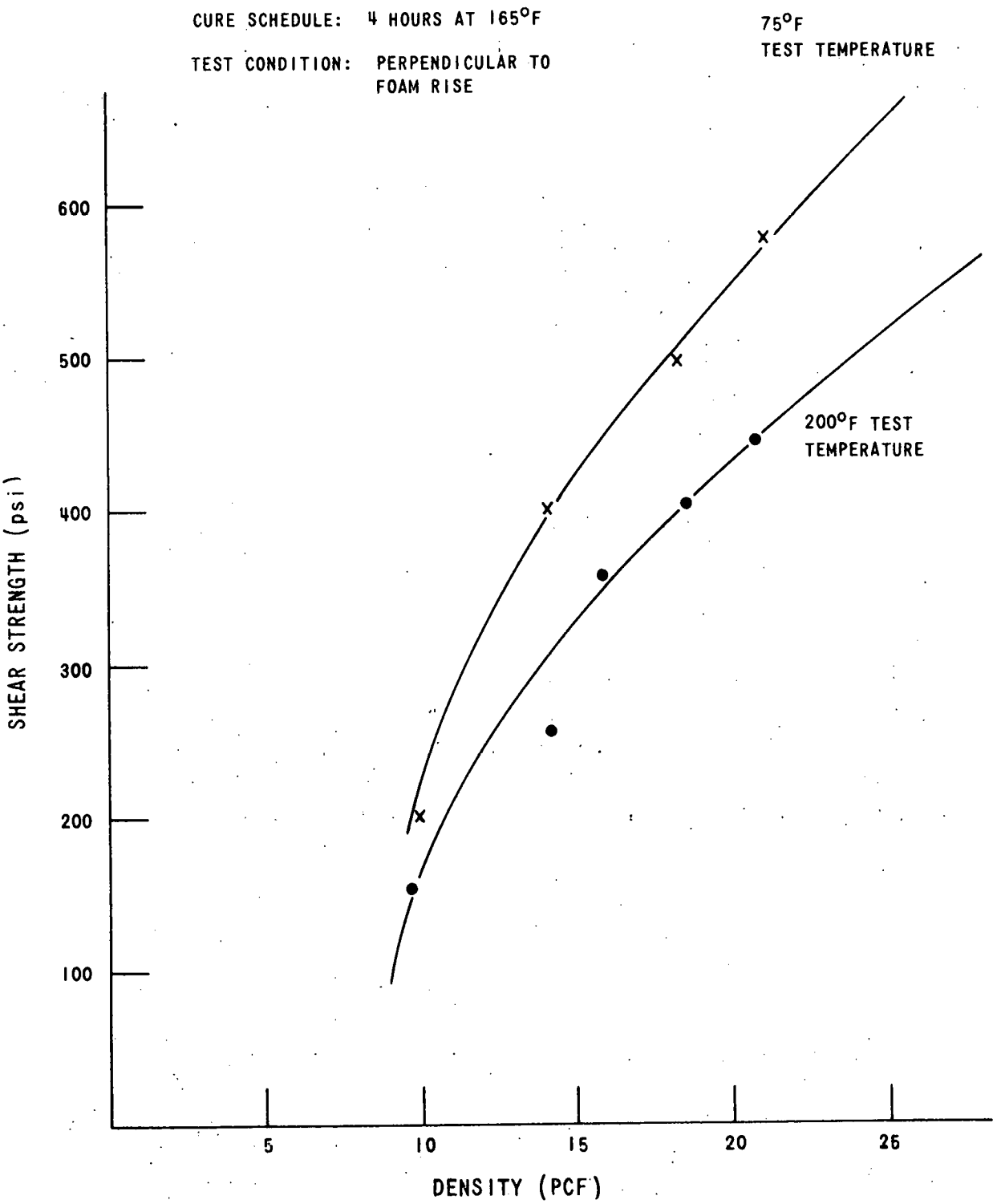


Figure 7. SHEAR STRENGTH VERSUS DENSITY FOR EPOXY FOAM SYSTEM

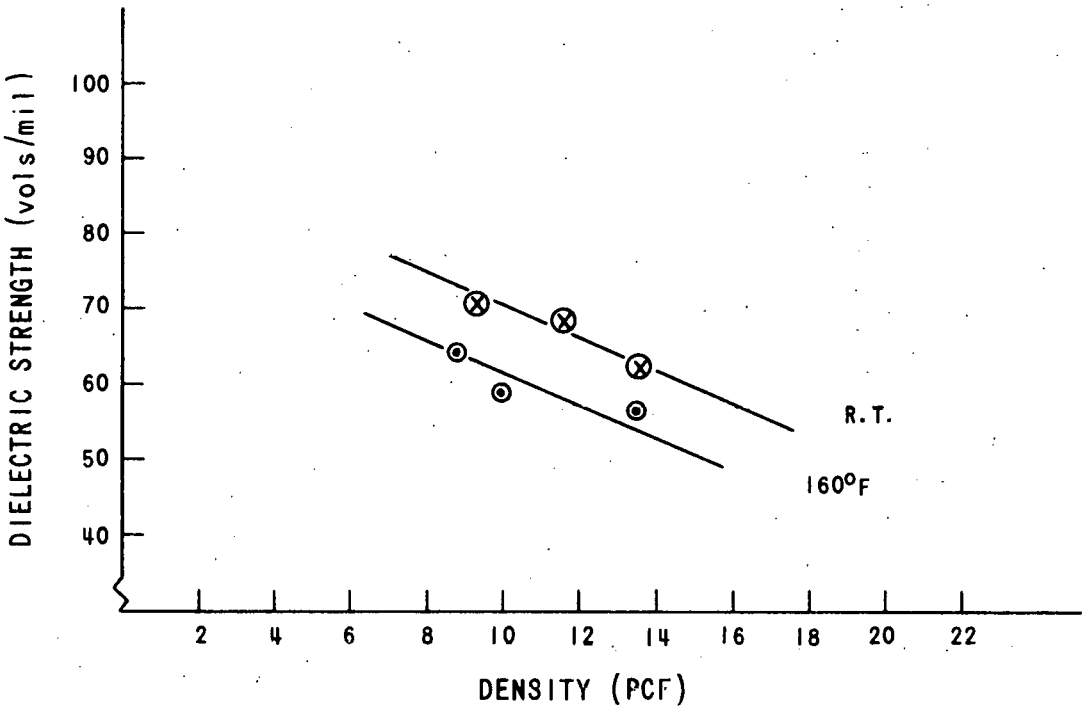


Figure 8. DIELECTRIC STRENGTH VERSUS DENSITY FOR EPOXY FOAM

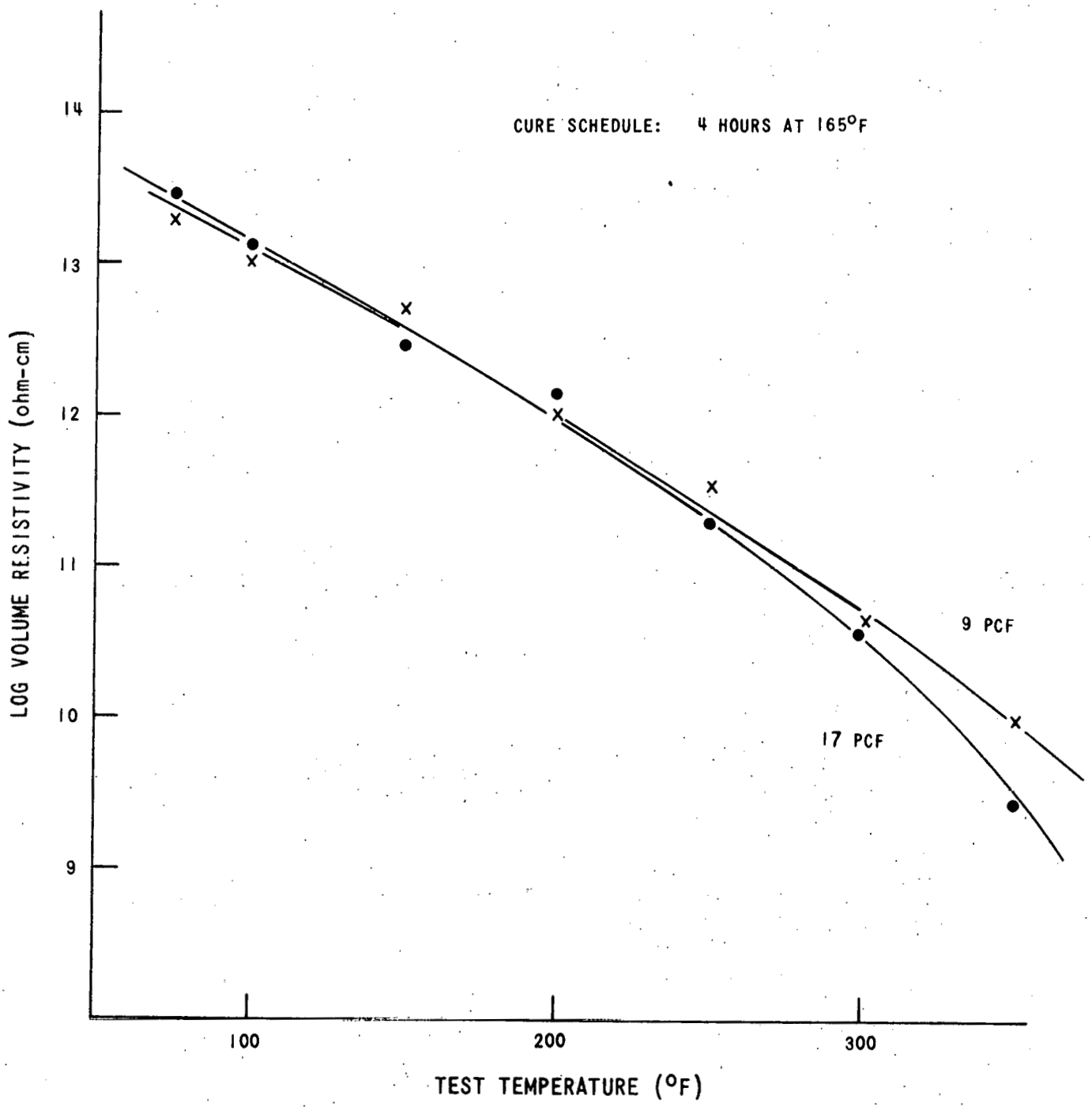


Figure 9. VOLUME RESISTIVITY VERSUS TEST TEMPERATURE FOR EPOXY FOAM

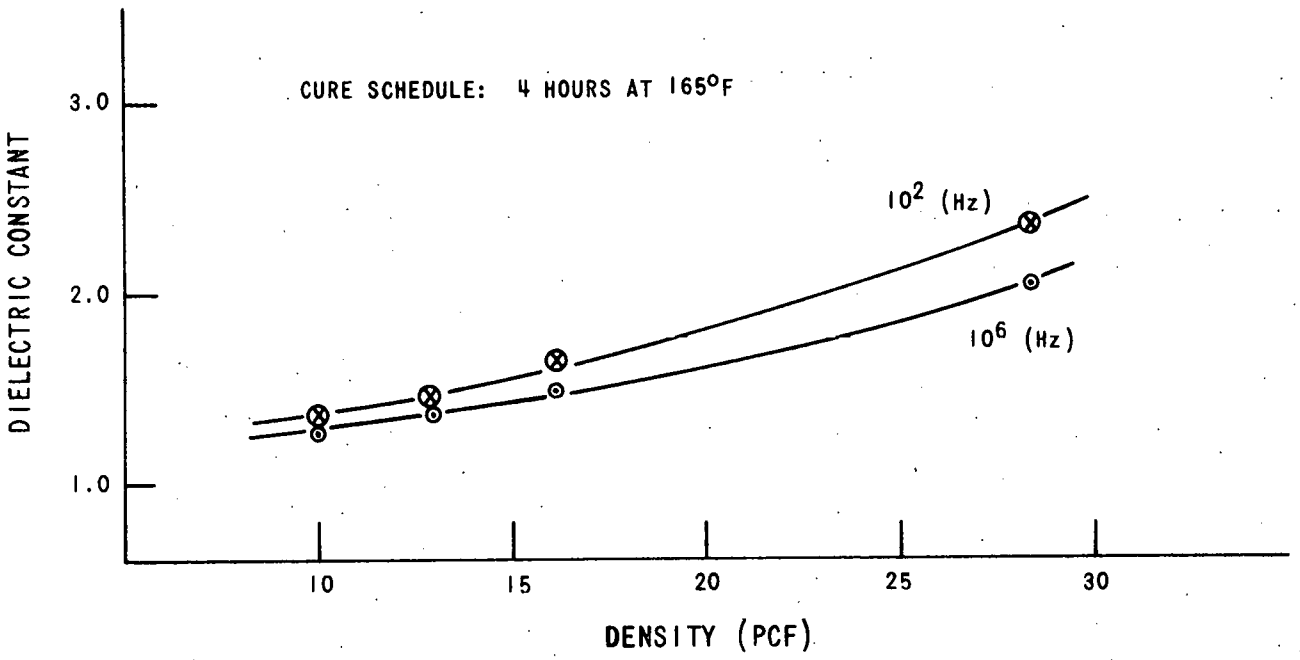


Figure 10. DIELECTRIC CONSTANT VERSUS DENSITY FOR EPOXY FOAM

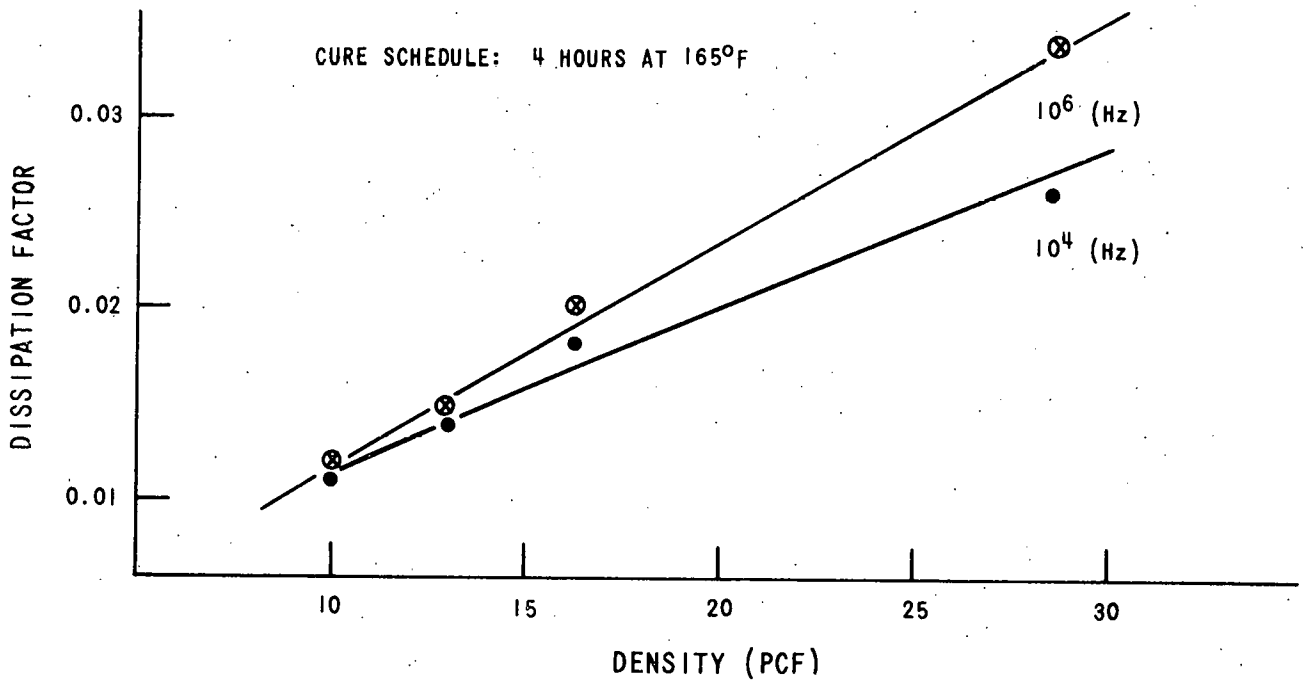


Figure 11. DISSIPATION FACTOR VERSUS DENSITY FOR EPOXY FOAM

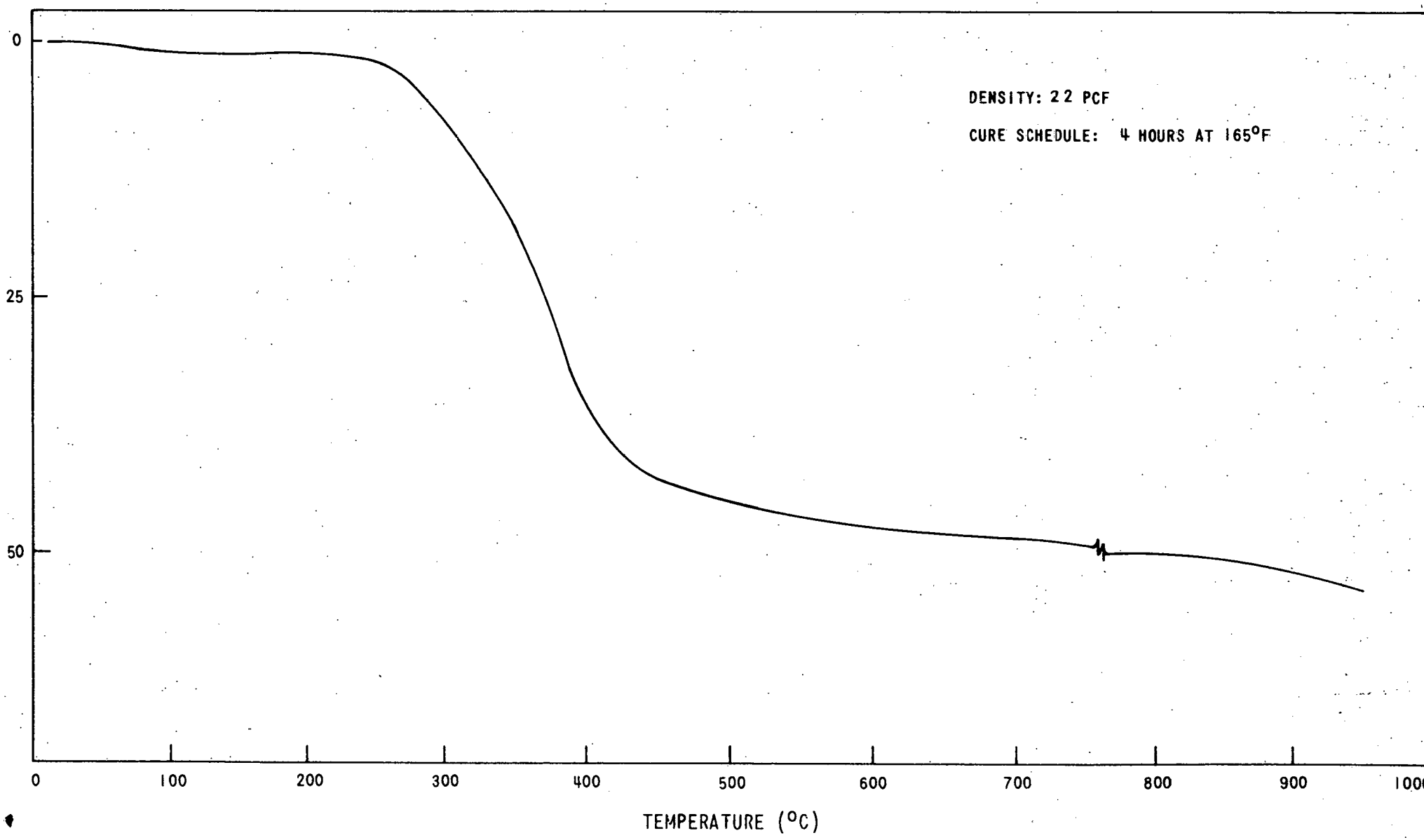


Figure 12. TYPIC TGA CURVE OF EPOXY FOAM

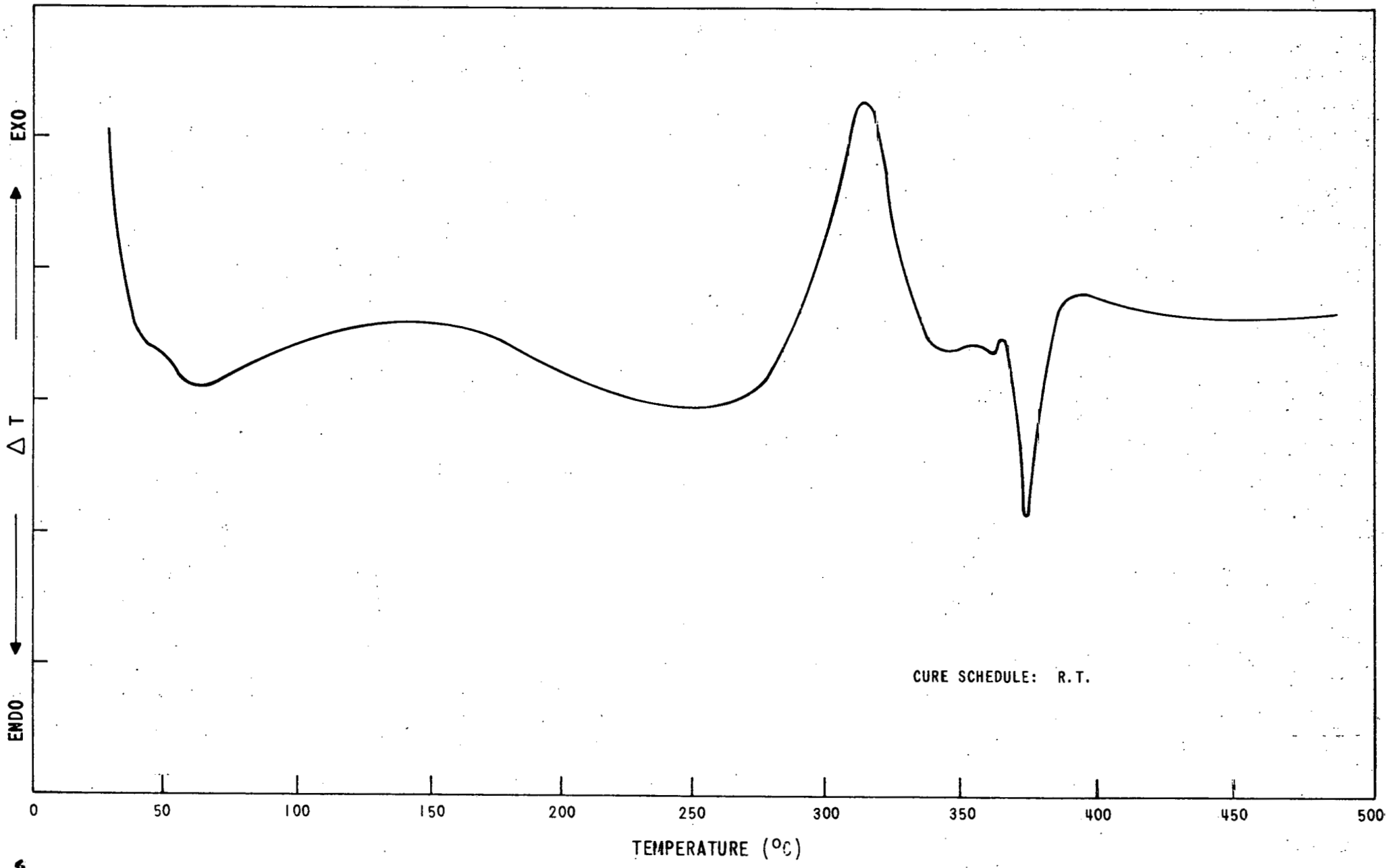


Figure 13. TYPICAL DTA CURVE OF UNFOAMED EPOXY