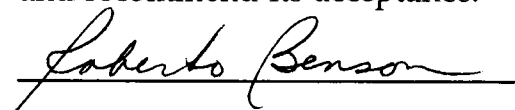


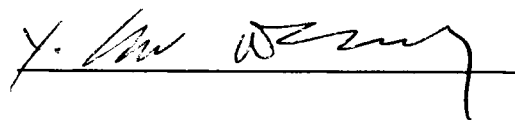
To the Graduate Council:

I am submitting herewith a thesis written by Johnny Hampton Spence, II entitled "The Effects of Isothermal Aging on the Interfacial and Shear Properties of Composites." I have examined the final copy of this thesis for form and content and recommend that it be accepted in partial fulfillment of the requirements for the degree of Master of Science, with a major in Engineering Science.


Madhu S. Madhukar, Major Professor

We have read this thesis
and recommend its acceptance:





Accepted for the Council


Associate Vice Chancellor and
Dean of the Graduate School

THE EFFECTS OF ISOTHERMAL AGING ON THE INTERFACIAL AND SHEAR PROPERTIES OF COMPOSITES

A Thesis
Presented for the
Master of Science
Degree
The University of Tennessee, Knoxville

Johnny Hampton Spence, II
May 1996

DEDICATION

This thesis is dedicated to my parents

John and Louise Spence

and to my sister

Betty Spence Whitehouse

and her family

who have contributed so much to

my growth, both personally and

professionally, over the years.

ACKNOWLEDGMENTS

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Finally, I am grateful to my parents John and Louise Spence, my sister Betty Spence Whitehouse, and to my brother-in-law Ed Whitehouse for assistance and encouragement all throughout my academic career.

ABSTRACT

The research for this thesis was divided into two segments. The first segment examines the effects of isothermal aging on the shear properties of a G40/BMI composite. The composite was aged at 150°C for periods ranging from 0 to 10,000 hours. Tests were conducted after the composite was aged in order to determine weight loss, change in glass transition temperature, inplane shear strength and shear modulus. After 10,000 hours of aging, the composite specimens had a weight loss of 0.3 percent and a shift in glass transition temperature from 215°C to 265°C. Using the Iosipescu test method, the shear properties were measured, and it was determined that no change occurred in either the inplane shear strength or shear modulus after 10,000 hours of aging. Thus, long-term exposure at 150°C for 10,000 hours appears to have no effect on the shear properties of this composite.

The second segment examines the effects of isothermal aging on the interfacial properties of a IM7/epoxy composite. Single fiber coupons were manufactured and aged at 100°C for periods ranging from 0 to 2000 hours. Tests were conducted after the coupons were aged in order to determine changes in glass transition temperature and changes in the fiber-matrix interface. After 2000 hours of aging, no change in glass transition temperature was detected. Using the single fiber fragmentation method, the fiber-matrix interfaces of the composite coupons were examined. After 2000 hours of aging, interfacial shear strength decreased, fiber-matrix debonding increased, and fiber slippage increased. Also, SEM examination revealed aging-induced cracking between the fiber and the matrix along the interface. Thus, exposure to elevated temperatures can severely degrade a composite's interfacial properties.

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**PART I: GENERAL INTRODUCTION
AND OVERVIEW**

CHAPTER 1

INTRODUCTION, BACKGROUND, AND MOTIVATION

For thousands of years, the materials of choice have been wood, metal, and ceramics. During this past century, engineers and scientists have developed many new materials from which to choose. These materials range from new super alloys and plastics to engineered ceramics and smart materials. One of the most important classes of materials to emerge have been composites.

Combinations of two or more materials, composites offer tremendous property improvements over conventional materials. Also, composites usually weigh less than conventional materials, which is a plus in situations where total weight is important.

The aerospace field was one of the first areas in which advanced composite materials were widely used. Early in their development, the high manufacturing cost of composites kept them limited to areas where performance was more important than cost. Now, however, new ways to produce composites have opened up other areas such as the automotive, construction, recreational, and health industries.

Presently, the most widely used composites are composed of a polymer matrix with carbon or glass fibers used as the reinforcement. Metal matrix and ceramic matrix composites also exist, but so far these have not been as popular. Ceramic matrix composites are strong and can be used at high temperatures. However, they are extremely brittle. Metal matrix composites are not brittle and have a lot of advantages over polymer matrix composites

such as higher temperature use, better transverse strength, and no moisture absorption. However, they have high costs, complex fabrication methods, and limited service experience. In comparison, polymer matrix composites are more advanced in fabrication technology and have lower raw material and fabrication costs than metal matrix composites. Therefore, polymer matrix composites are more popular for the time being.

Significant research has been conducted in order to better understand and improve the properties of these polymer matrix composites (PMC's). One of the most important research areas has been the thermal oxidative stability (TOS) of PMC's. Many applications of composites involve exposure to temperatures that can rise as high as 300-400°C. These are high temperatures for polymeric materials, and it is extremely important that they resist degradation and retain their properties.

Long term exposure to high temperatures can affect polymers severely. When temperatures approach a polymer's glass transition temperature or melting point, the mechanical properties can change dramatically. Even if the temperature is well below the glass transition temperature, long term exposure can still cause property degradations due to thermal oxidation and chemical attack. In a composite, the fibers are the main load bearers, but the matrix protects the fibers from adverse environments and transfers the load between the fibers. The matrix also has a big influence on the in-plane shear and interlaminar shear properties of the composite. Therefore, when the matrix is affected, the entire composite is affected [1].

If a polymer composite has a poor TOS, it can experience weight loss and a deterioration of properties such as creep, tensile strength, compressive strength, fatigue behavior, and fracture toughness. The matrix can become

embrittled, the fibers can oxidize or undergo some other chemical attack, and the interphase can be weakened. Therefore, it is important that a thorough understanding of the high temperature behavior of PMC's be attained.

The purpose of this project was to further study the TOS of polymer matrix composites. The National Aeronautics and Space Administration (NASA) sponsored this study as part of their High Speed Research Program. One of the goals for this program was the research and development of materials for possible use in the next generation of civilian supersonic aircraft. Strong and lightweight, PMC's would be ideal for the airframe of the aircraft as well as parts of the engine structure. At supersonic speeds, air friction heats the airframe. At speeds of Mach 2 and Mach 2.4, the temperatures of the airframe would be respectively 125°C and 175°C. Therefore, TOS is an extremely important consideration.

This research project consists of two segments. The first segment examines the effects of isothermal aging on the shear properties of a G40/BMI composite. It was motivated by a previous study conducted by researchers at NASA's Langley Research Center [2]. In that study, composites were aged at temperatures of 125°C and 175°C and then tested. The researchers monitored the weight loss and property degradation of the composites. One composite, an IM7/BMI system, experienced significant weight loss and property degradation as well as micro-cracking when aged at 175°C. For this project, a similar composite (G40/BMI) was aged at 150°C to study the effects of aging on shear properties. The goal was to increase the understanding of isothermal aging effects on a composite system with a low TOS.

The second segment of the project is a study of isothermal aging effects on interfacial properties of an IM7/epoxy system. A different material was

used for this part because the BMI system is not transparent, which is necessary for the interfacial characterization technique used in this project. Although IM7/epoxy is not suitable for high temperatures, it is comparable in several ways to aerospace matrices such as BMI and is transparent which makes it acceptable for this study.

The motivation for the second part stemmed from a report by researchers at NASA's Lewis Research Center [3]. The report was a study of specimen geometry effects on the TOS of composites. In particular, they examined the three different surfaces of a unidirectional composite in order to determine which surface had the greatest weight loss flux when aged. The surface perpendicular to the fiber direction was found to have the greatest flux. In addition, cracks were found on this surface around the fiber/matrix interface. This suggests that aging can cause significant damage to the interface. The goal of this part of the project was to provide a better understanding of what exactly is happening at the interface when the composite is aged.

This second part of the research was done parallel with a similar project at NASA's Langley Research Center. Engineers at Langley developed techniques to conduct the interfacial studies using an acoustic microscope. With such an instrument, opaqueness was no longer a problem. The results from the UT study were used to verify the results obtained from the acoustic microscope.

It was expected that the combined results from both parts of the research project would provide a better understanding of polymer composites. Specifically, the response of polymers with low thermal oxidative stability to long term aging at high temperatures.

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REFERENCES

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**PART II: THE EFFECTS OF
ISOTHERMAL AGING
ON SHEAR PROPERTIES
OF G40/BMI COMPOSITES**

CHAPTER 1

INTRODUCTION

When polymer matrix composites are exposed to elevated temperatures, they can undergo property degradation, even though the temperature is well below the glass transition temperature. This degradation is usually due to oxidation, so the thermal oxidative stability (TOS) of the polymer composite is very important.

Researchers have published many papers dealing with the TOS of polymer matrix composites (1-12). They used a variety of different methods to gauge a composite's TOS. In addition to weight loss, they have tested tensile, compressive, shear, and flexure properties; studied the effects on fracture toughness and viscoelastic properties such as creep resistance; and monitored changes in the glass transition temperature. Finally, they have attempted to devise fiber/matrix combinations that yield higher and higher degrees of TOS.

For this project, the TOS of a G40/BMI composite was studied because a previous project had indicated that an IM7/BMI composite, a very similar material, had shown signs of a low TOS when aged at 175°C for 5000 hours (13). The material in that study experienced microcracking and property degradation in addition to weight loss.

For this study, the inplane shear properties of the composite were monitored for changes due to aging. Unlike tensile and compressive testing which each have proven standard techniques, shear testing can be done by several ways (14). This includes short beam shear, off-axis tension, ± 45 -degree laminate, and rail-shear tests. The only problem with shear testing is that it is

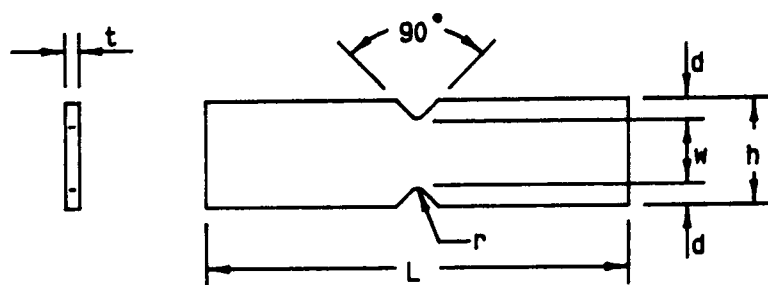
hard to get a state of pure, uniform shear in a specimen. Tensile or compressive components that taint the results are always present. Because of this, values for the shear strength and shear modulus differ from test to test.

A test developed by Iosipescu, which bears his name, tried to overcome these drawbacks (15). This test was refined by Walrath and Adams by using a modified fixture and slightly different specimen dimensions (16). The specimen configuration is shown in Figure 1a and is loaded as depicted in 1b. The modified fixture is shown in Figure 2. The test produces a region of almost pure shear between the notches. Some tensile stresses are present at the root of the notches. However, the magnitude of these stresses is small, so they have negligible effect on the shear values.

A number of research studies have used the Iosipescu shear test (17-21). Almost all the papers agreed that the test was one of the best for shear testing. This test can perform either inplane or interlaminar shear testing, depending on the specimen configuration. The only major drawback is that the shear is not uniform across the entire specimen (Figure 3). This characteristic taints the results slightly. However, a variety of techniques, including finite element analysis, can determine correlation factors which will incorporate the non-uniformity into the test results.

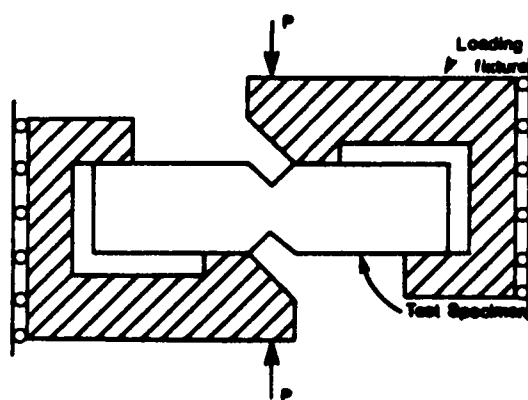
Correlation factors were not used for this study. The main concern here was not the absolute correct values of inplane shear strength and modulus, but the changes in them caused by aging. Therefore, correlation factors were beyond the scope of this study and were not calculated.

Overall, the purpose of this part of the project was to examine the TOS of this material. The goal was to determine if aging the G40/BMI composite at 150°C would cause damage and property degradation, specifically inplane shear properties.



$t = 1.7 \text{ mm (0.0660 in)}$
 $h = 19.1 \text{ mm (0.75 in)}$
 $d = 4.3 \text{ mm (0.17 in)}$
 $L = 76 \text{ mm (3 in)}$
 $r = 1.3 \text{ mm (0.05 in)}$

a. Iosipescu shear test specimen.
(From Reference 16)



b. Loading of the Iosipescu specimen.
(From Reference 18)

Figure 1. Iosipescu specimen.

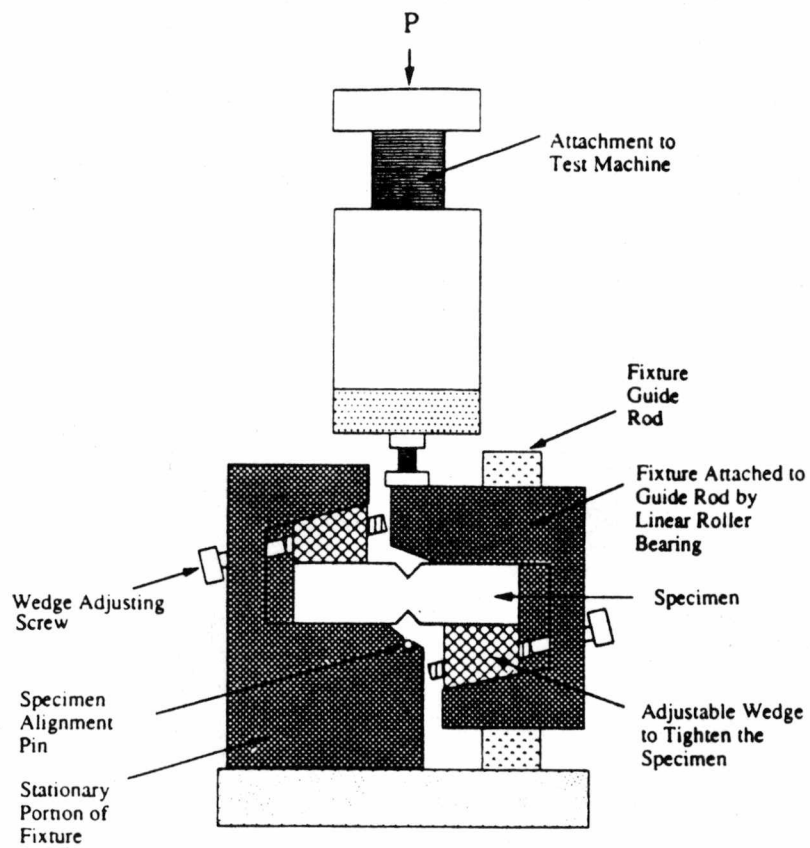


Figure 2. Modified Iosipescu test fixture.
(From Reference 21)

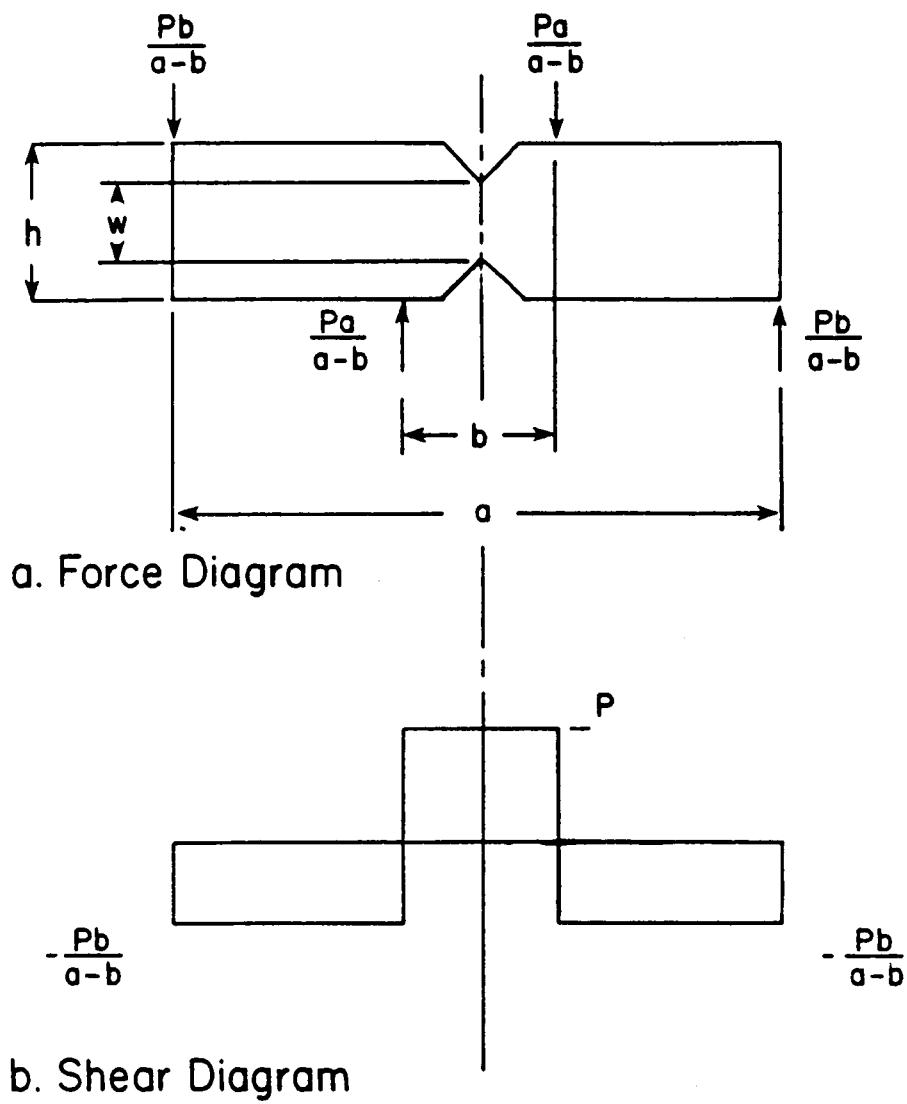


Figure 3. Force and shear diagrams for the Iosipescu specimen.
(From Reference 16)

CHAPTER 2

EXPERIMENTAL PROCEDURE

The specimens were made from a G40/5260 BMI prepreg manufactured by CYTEC Engineered Materials, Inc. BMI is short for bismaleimide, which is a thermoset polymer that has excellent toughness and high temperature resistance. It has a continuous use temperature of up to 177°C and a short term use temperature of up to 232°C. The G40 fiber is a carbon fiber that is very similar to the better known IM7 fiber. It has a tensile strength of 5000 MPa (700 ksi) and a modulus of 276 GPa (40,000 ksi). Combined together in the prepreg, the material has a typical tensile strength of 2932 MPa (425 ksi) and a modulus of 168 GPa (24,400 ksi).

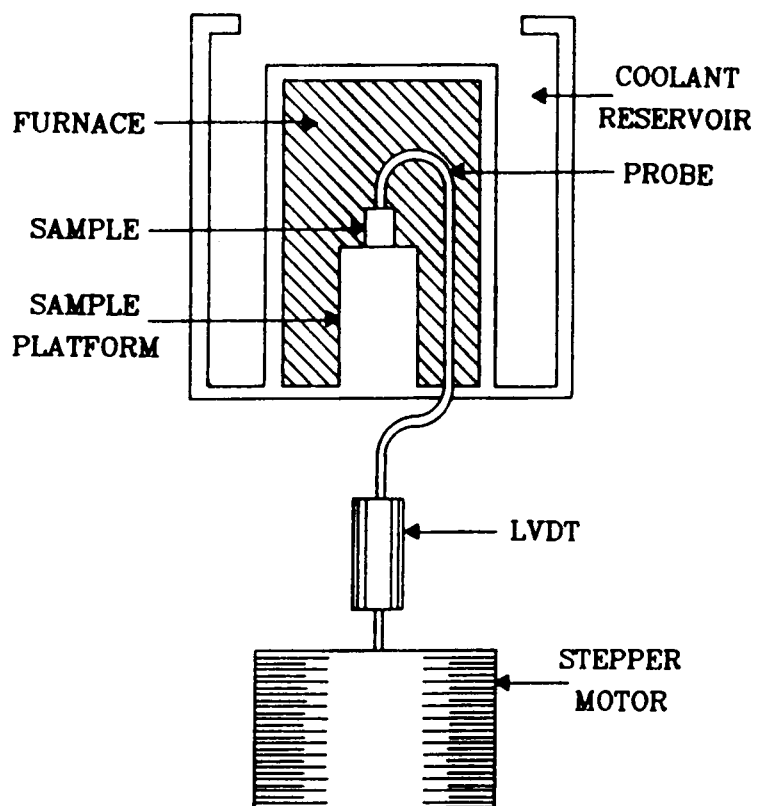
The prepreg was cut into 12x12 inch squares and stacked into 12-ply laminates. The laminates were then cured in a vacuum press by a two-part curing process. The first part consisted of raising the temperature from 23°C to 150°C at 1.7°C/minute under a vacuum, holding for 30 minutes at 150°C, raising the pressure to 0.59 MPa, raising the temperature to 192°C at 1.7°C/minute, and holding at 192°C for four hours. The second step is a post-cure which consisted of raising the temperature from 23°C to 220°C at 2.8°C/minute, and holding for 6 hours.

After curing, the edges of the plates were trimmed. Then the plates were cut into smaller 5x5 inch squares. The squares were then aged in a Blue M® air circulating oven at 150°C for periods of 0, 100, 200, 500, 1000, 2000, 5000, 7500, and 10,000 hours. The specimens were weighed before and after aging to determine if any weight loss occurred.

The glass transition temperature was measured at each aging time using thermo-mechanical analysis (TMA). This method employed a glass probe which was pressed on the specimen with a specified force (see Figure 4a). The specimen was heated over a selected temperature range which caused it to undergo volume changes. The displacement of the probe due to these expansions and contractions was measured and plotted against the temperature. When a material reaches its glass transition temperature, a major volume change occurs. Thus, any sudden change in the slope of the line on the temperature/displacement plot indicates the glass transition temperature (22). In this project, the force of the probe on the specimen was 0.1 Newtons, and the temperature was increased from 50°C to 350°C at a rate of 10°C/minute. Nitrogen gas was the only gas used in the chamber containing the specimen and probe to prevent any reaction with oxygen at elevated temperatures. After a temperature/displacement plot was obtained, the glass transition temperature was determined by the location of the slope change of the line (see Figure 4b).

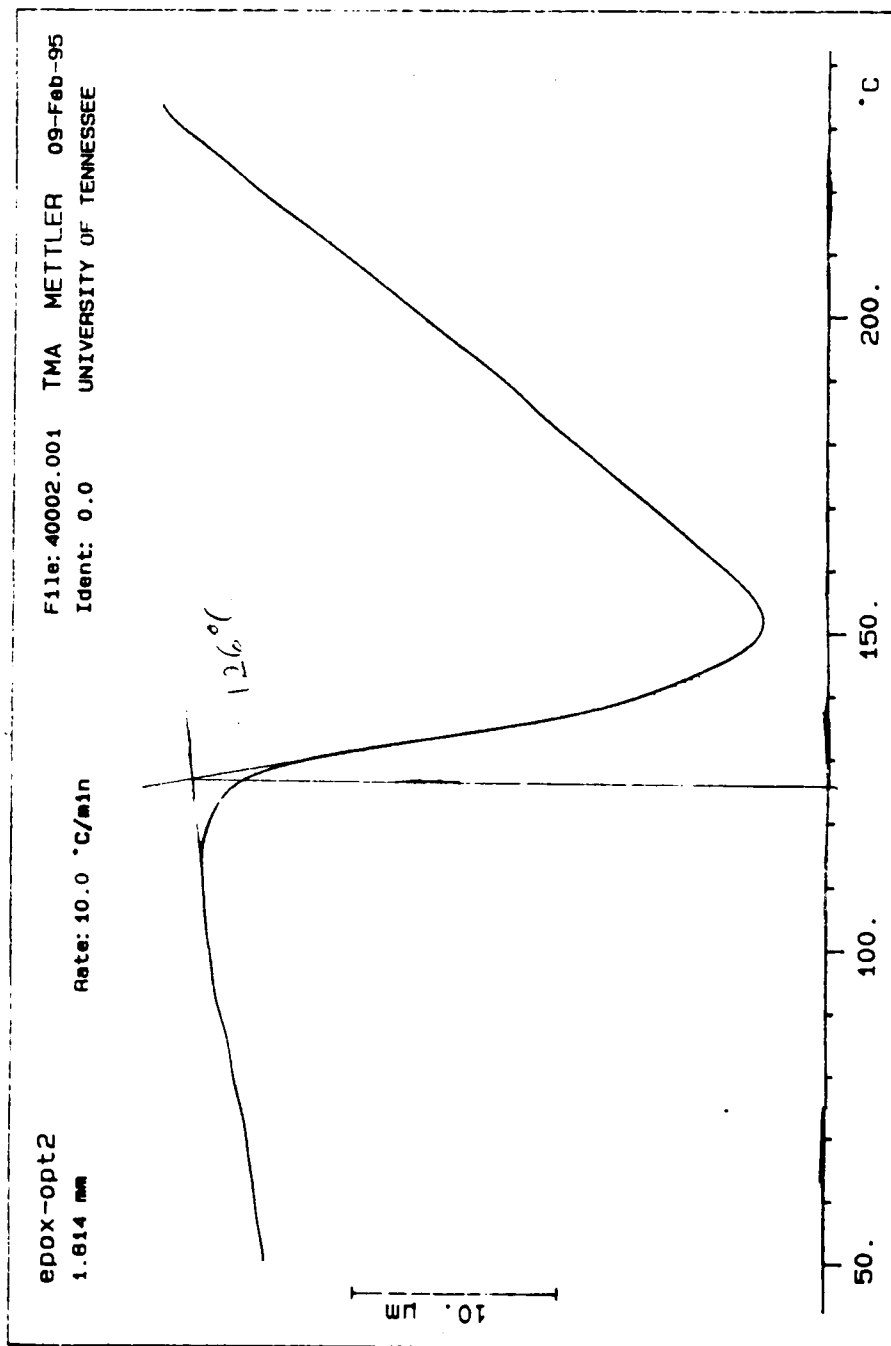
For the Iosipescu shear test, the 5x5 inch plates were cut into the specimen configuration shown in Figure 1. The specimens were then fitted with fiberglass tabs to stabilize them during the test. The tabs were attached with an epoxy adhesive, and the edges of the tabs were sanded to make them flush with the specimen edges.

The specimens were fitted with 350 ohm strain gauges of the EA-13-062TV-350 type manufactured by Measurements Group, Inc. The surfaces to which the gauges were bonded were degreased, sanded, and cleaned. After this preparation, the gauges were glued to the specimens using an M-bond 200 adhesive.



a. Schematic of TMA experimental set-up.
(From Reference 22)

Figure 4. Glass transistion temperature procedure.



b. T_g determination by change in slope.

Figure 4 (continued). Glass transition temperature procedure.

The apparatus used to conduct the Iosipescu shear test is shown in Figure 2. It was placed into an Interlaken Series 3300 testing machine operating in compression at room temperature. Four specimens were tested at each aging time. With the strain gauges wired to an amplifier, the data was collected by a Quickbasic data acquisition program on an Apple computer.

This set-up enabled the measuring of the inplane shear strength and the calculation of the shear modulus. The load was divided by the specimen area between the notches to calculate shear stress. The highest value, of course, was the inplane shear strength. The shear stress was plotted against the shear strain. From this graph, the shear modulus was calculated from the slope of the line in the elastic region. Using a linear regression program on the computer, a line was plotted through the data points in the elastic portion of the stress-strain plot. The slope of this line was the shear modulus. The results from all the specimens in each aging period were combined to obtain average values of inplane shear strength and shear modulus.

Another task performed in this study was the investigation of surface flux, or the amount of weight loss from each surface on the composite. There are three different types of surfaces on a uni-directional composite plate (see Figure 5). Previous research found that the surface perpendicular to the fiber direction had the greatest weight loss flux. It was decided to verify that result in this research, so 8-ply, 12-ply, and 24-ply specimens cut into a variety of different shapes were aged. The specimens were cut so that each of the three main surfaces varied in area from specimen to specimen. By comparing the weight loss percentage of each specimen, the surface area prone to the most weight loss could be determined.

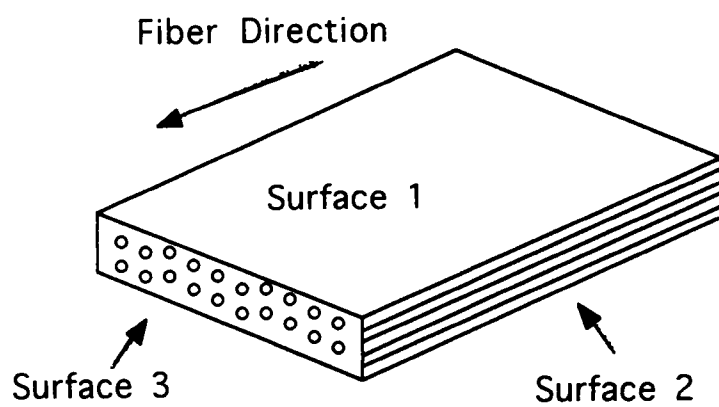


Figure 5. Surfaces of a composite plate.

CHAPTER 3

RESULTS AND DISCUSSION

The weight loss data for the 5x5 inch 12-ply plates is plotted in Figure 6. Any weight loss above 0.1% was considered significant. As the graph shows, significant weight loss began to occur at 5000 hours. There was a small spike at 1000 hours, but it was within the error of the balance used to measure the weight of the plates. At the maximum aging time of 10,000 hours, the weight loss was just under 0.3%.

Although 0.3% cannot be deemed insignificant, it is not really a large amount of weight loss. The paper which was the basis for this study monitored the weight loss at two temperatures, 125°C and 175°C (13). No significant weight loss occurred at 125°C while 2% weight loss occurred after 5000 hours of aging at 175°C. Compared with the result in this project, it can be concluded that 150°C is probably where the TOS of the BMI starts to weaken. A 0.3% weight loss is not significant by itself, but if the material were to be used in an actual application where the exposure time at 150°C would be substantially longer, the weight loss and degradation will be a major concern.

The TMA temperature/displacement plots for the different aging times are shown in Figures 7-15. The T_g is plotted against the aging time in Figure 16. The T_g shifted 50 degrees over the 10,000 hour aging period. Originally, it was 215°C and did not change until after 1000 hours of aging where it increased to 225°C. By 2000 hours, the T_g had shifted 35 degrees total to 250°C. From that point until 10,000 hours it shifted another 15 degrees to 265°C.

This shifting of the T_g was due to continued curing and cross-linking. Most of the time, composite resins are not fully cross-linked during the cure

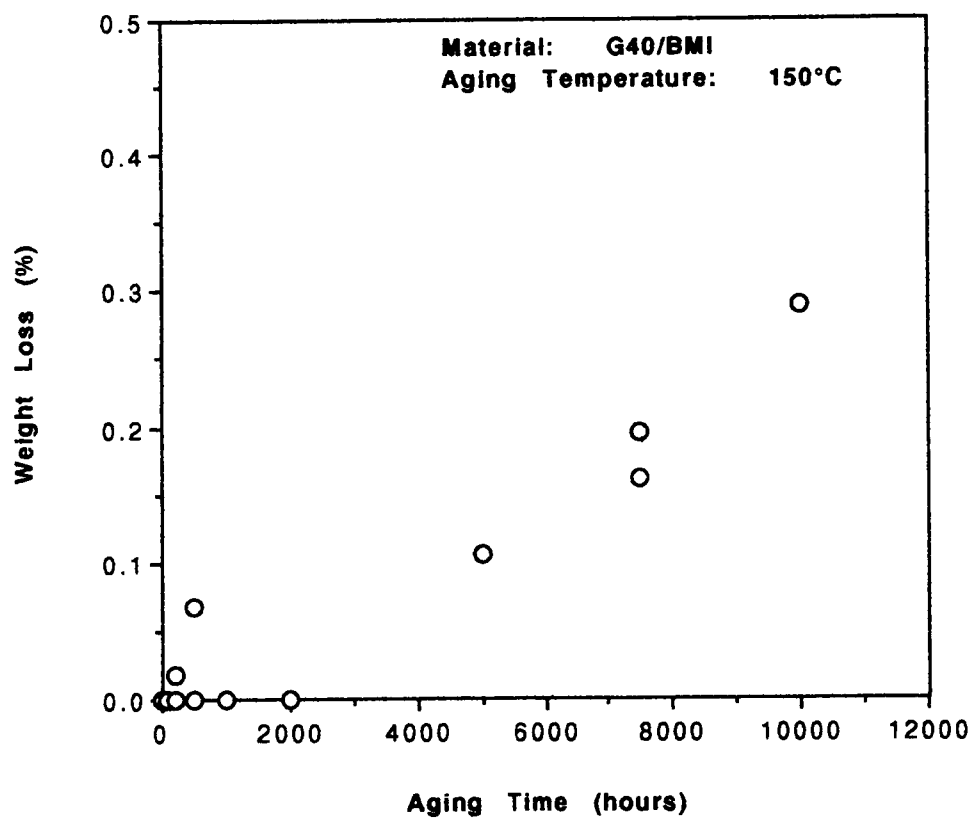


Figure 6. Plot of weight loss versus aging time.

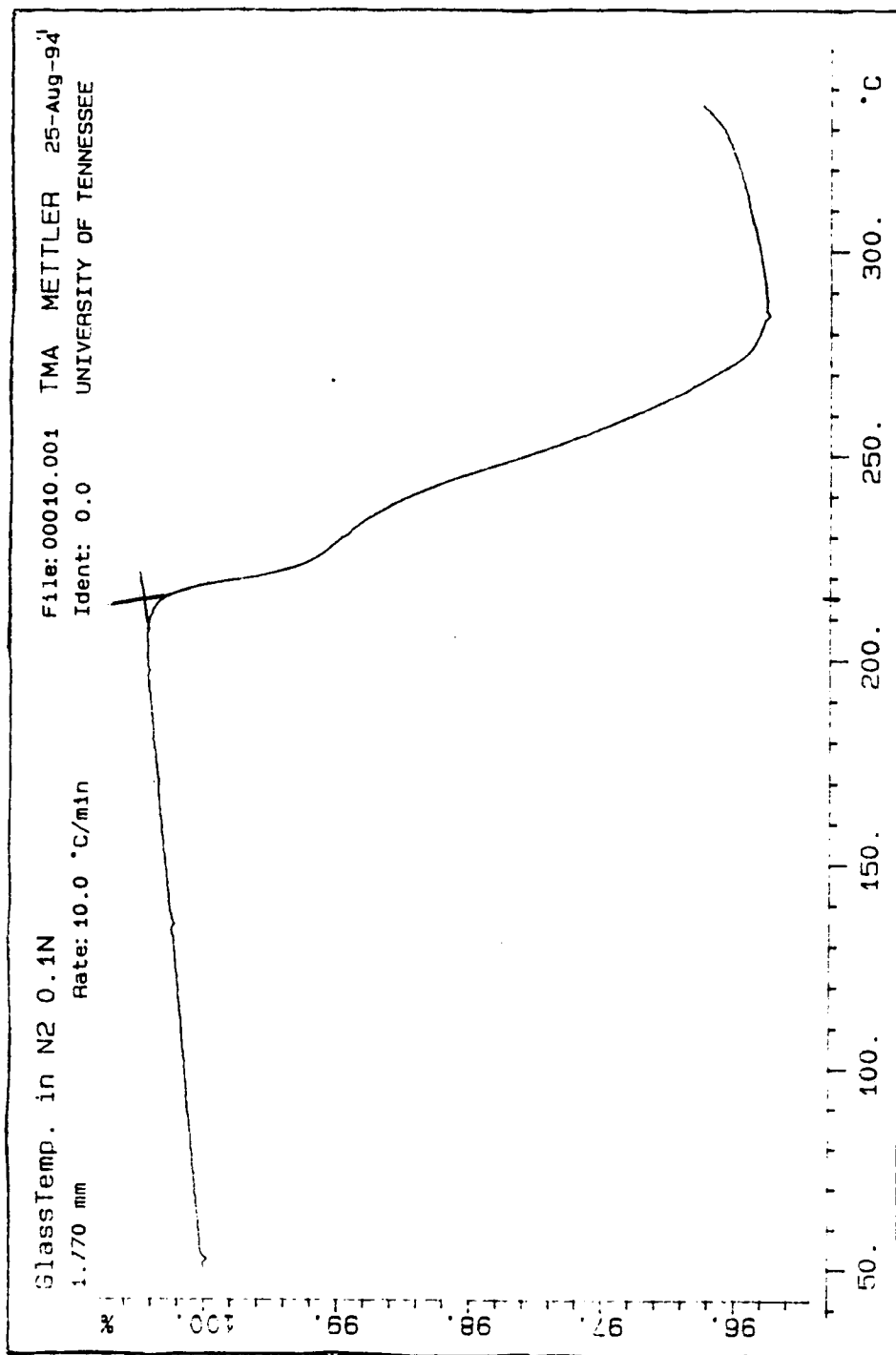


Figure 7. Temperature/displacement plot for specimen aged for 0 hours.

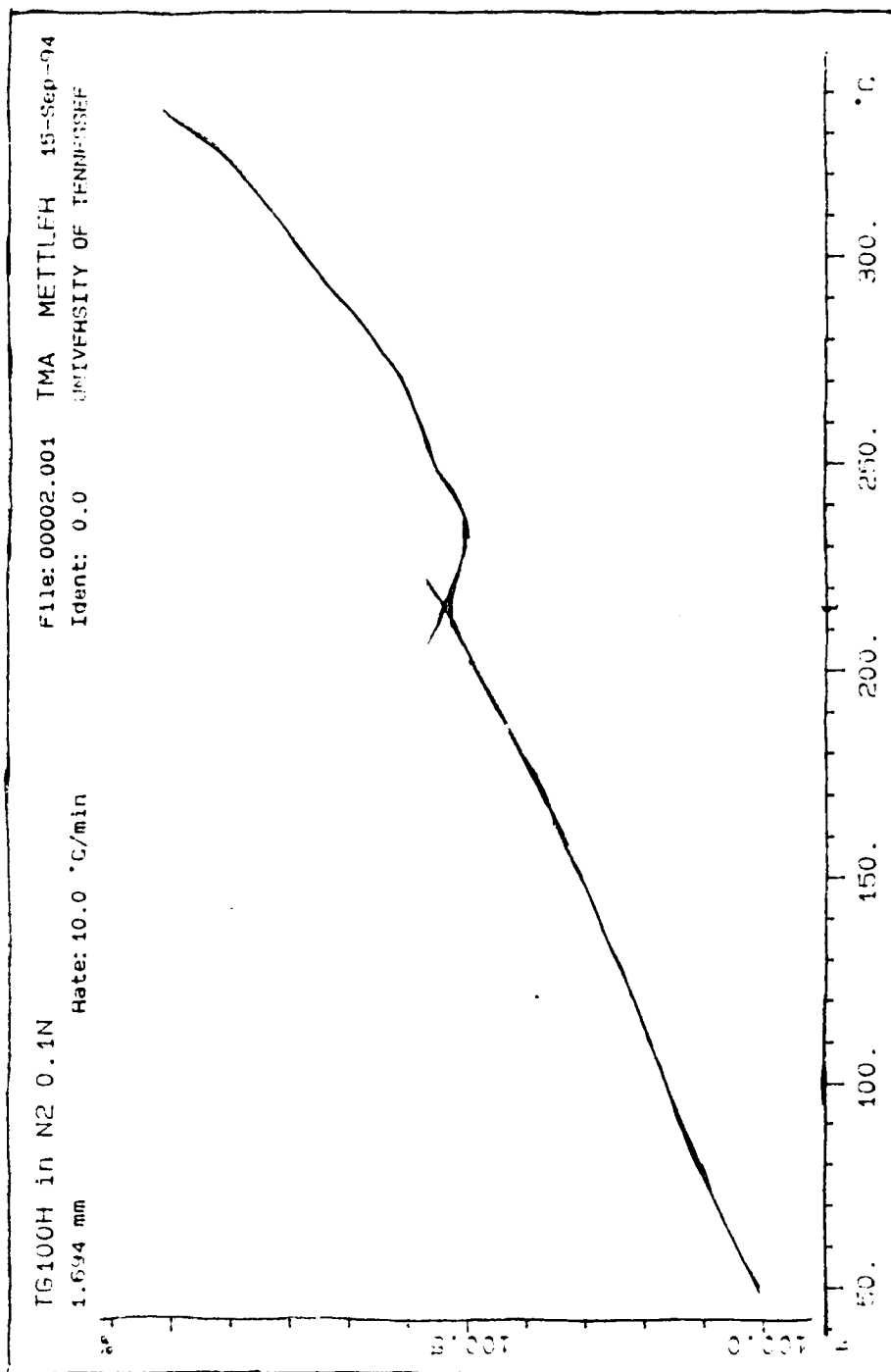


Figure 8. Temperature/displacement plot for specimen aged for 100 hours.

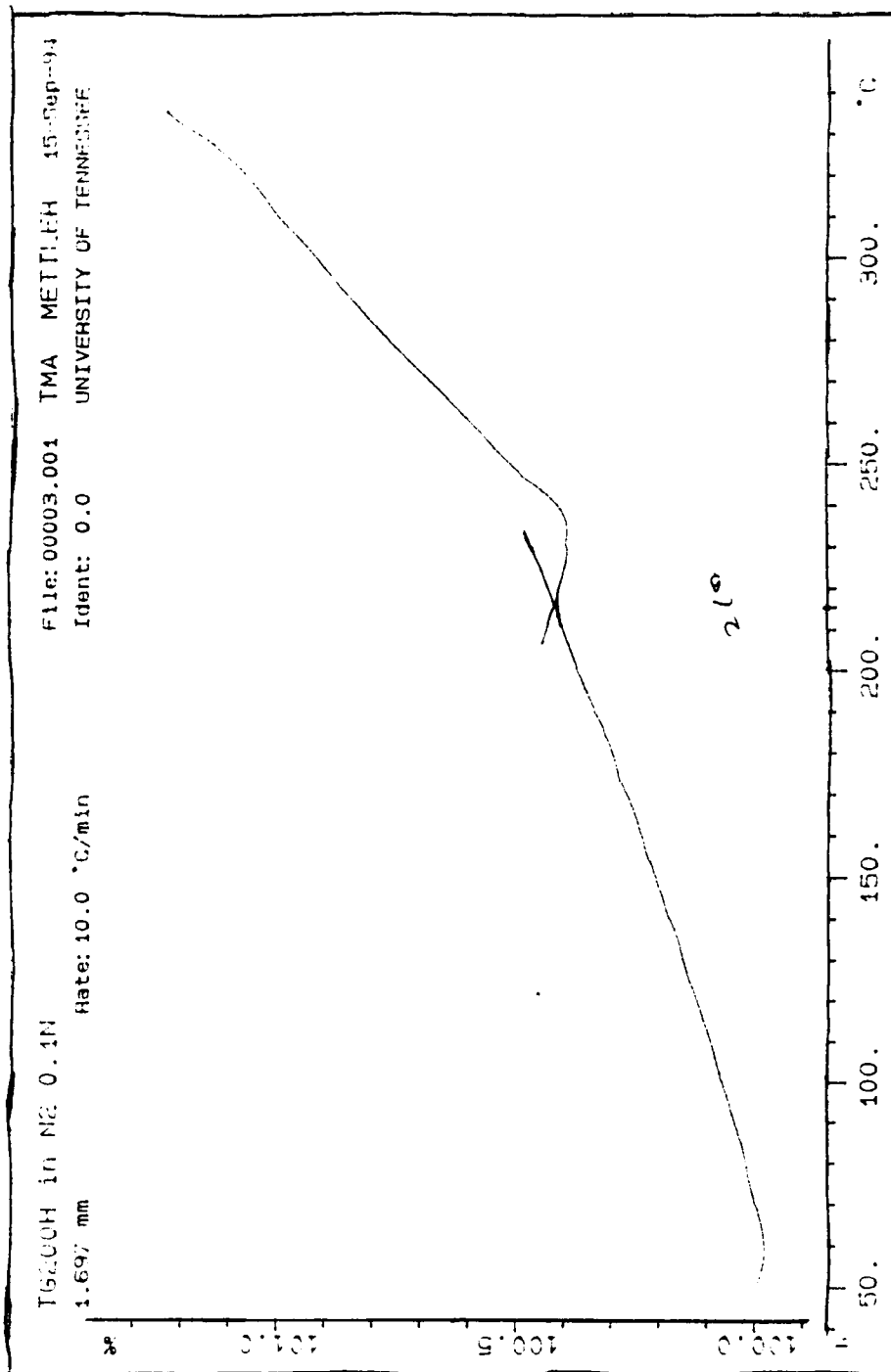


Figure 9. Temperature/displacement plot for specimen aged for 200 hours.

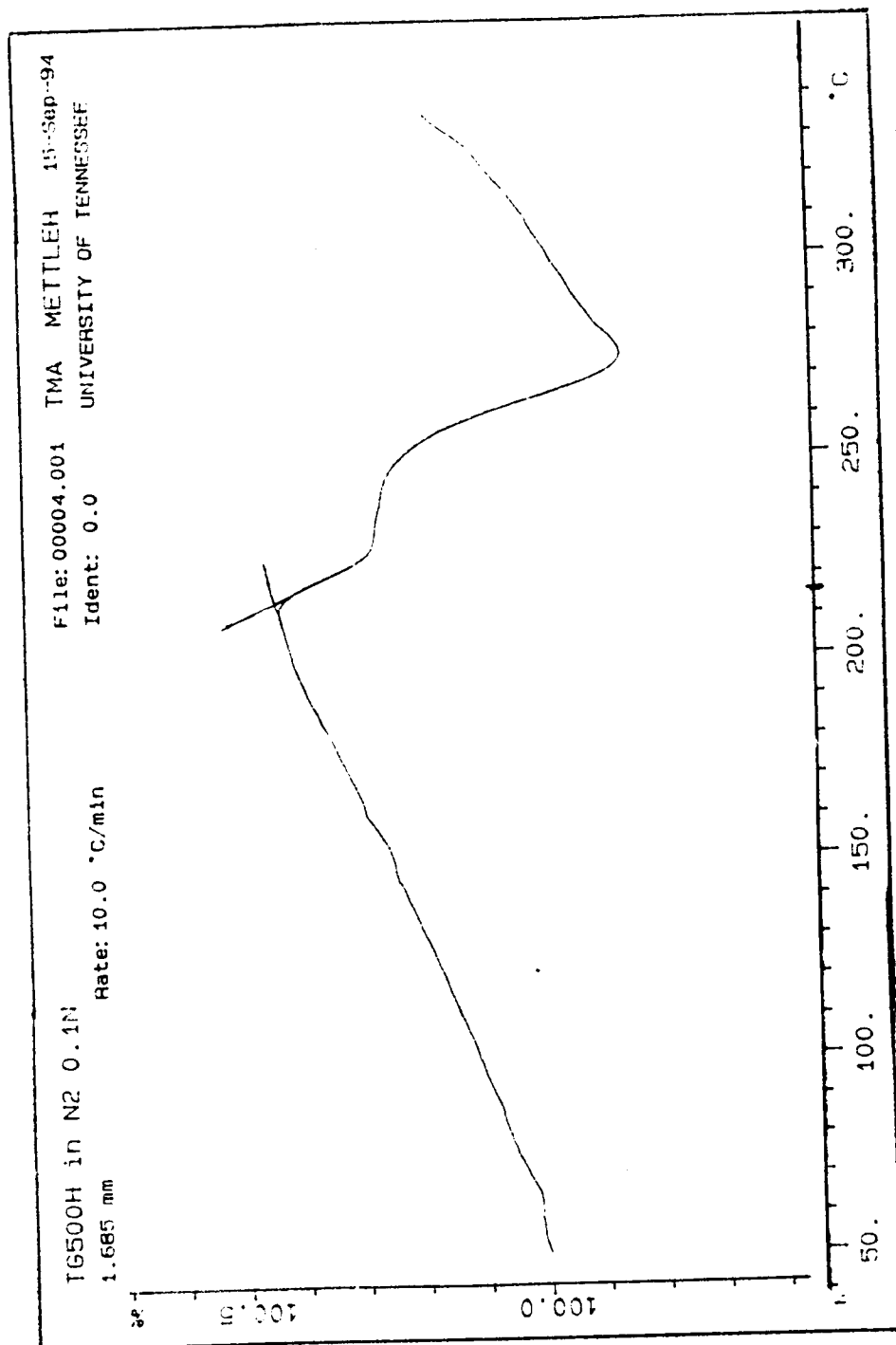


Figure 10. Temperature/displacement plot for specimen aged for 500 hours.

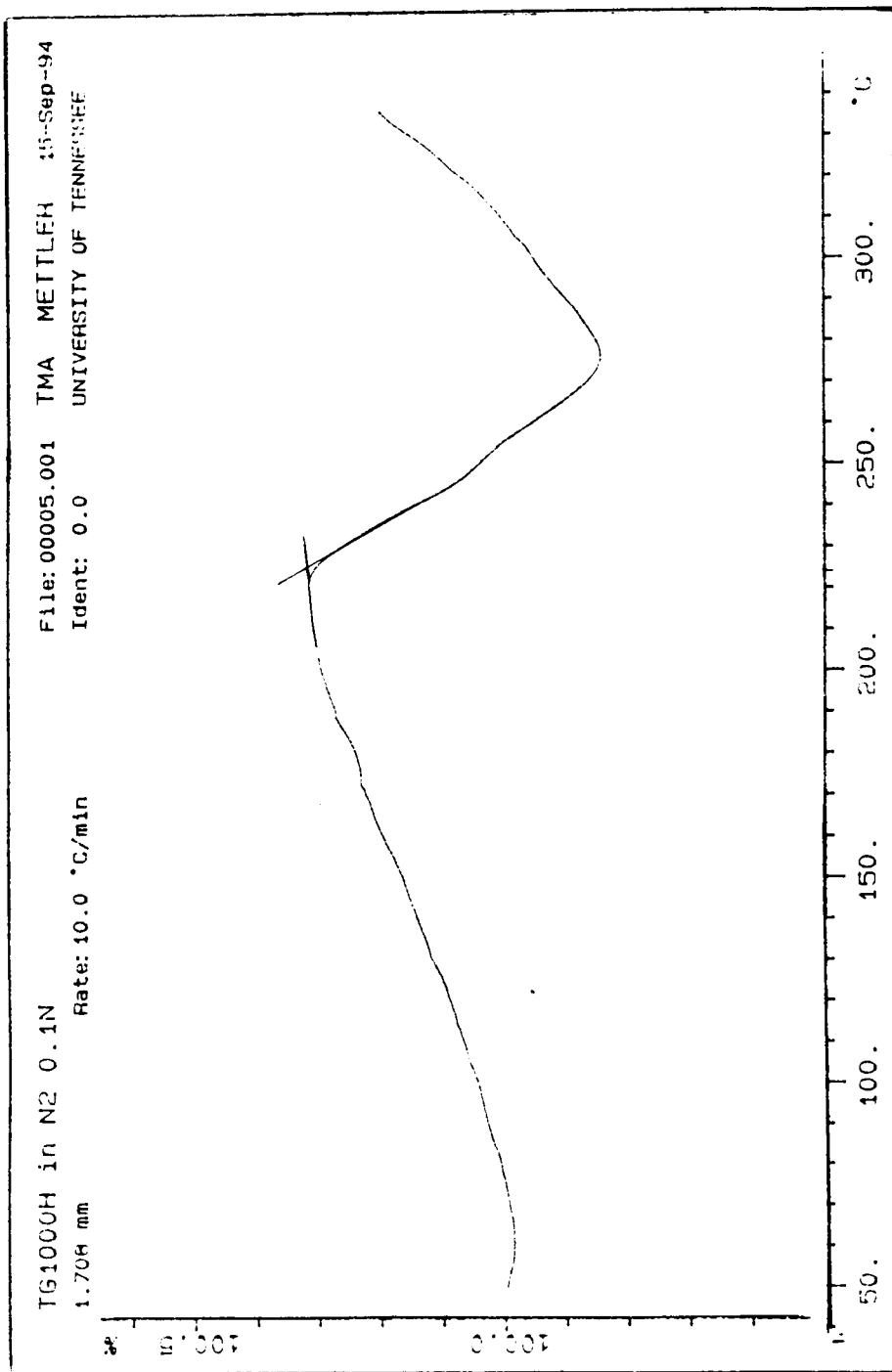


Figure 11. Temperature/displacement plot for specimen aged for 1000 hours.

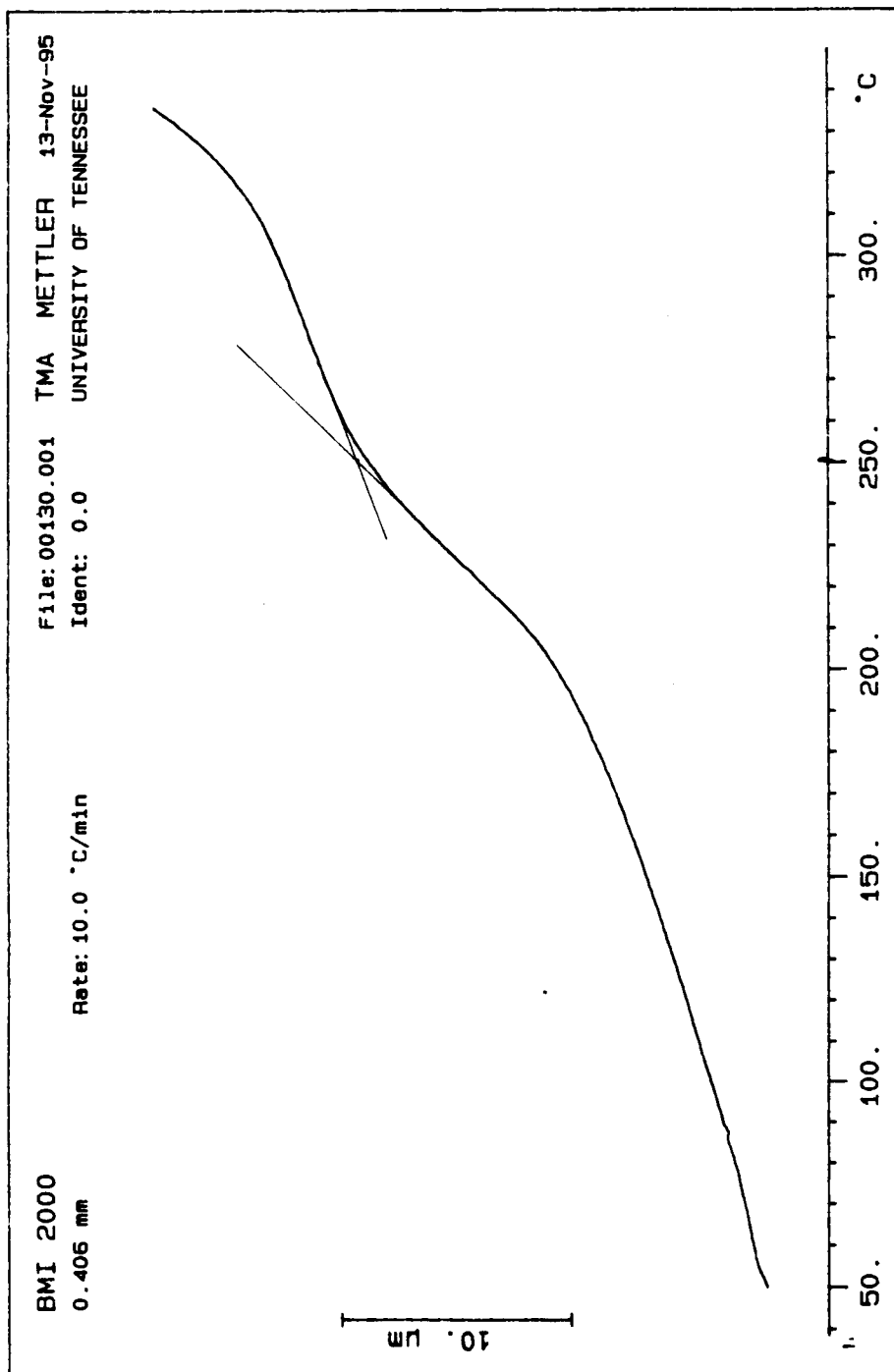


Figure 12. Temperature/displacement plot for specimen aged for 2000 hours.

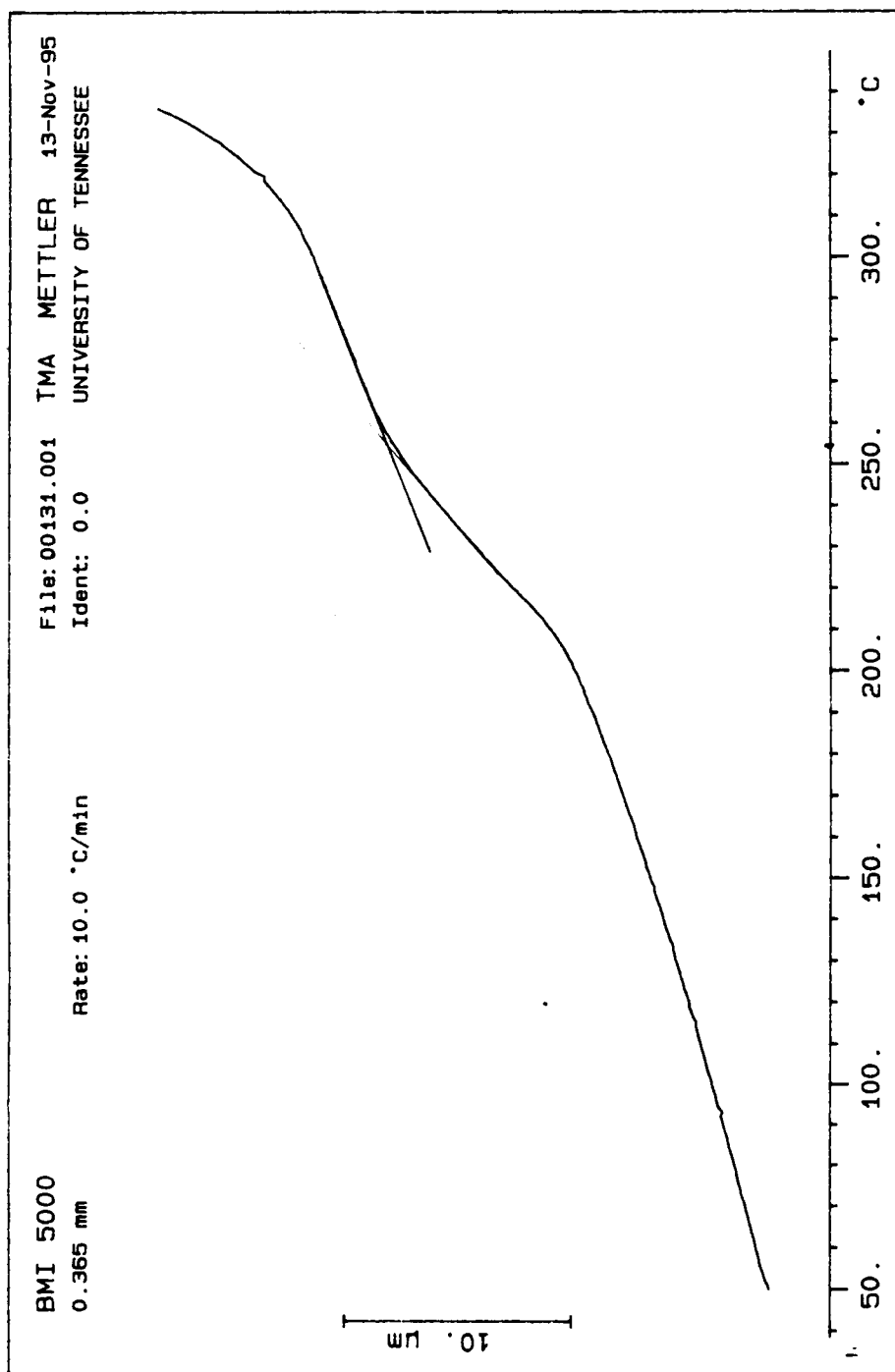


Figure 13. Temperature/displacement plot for specimen aged for 5000 hours.

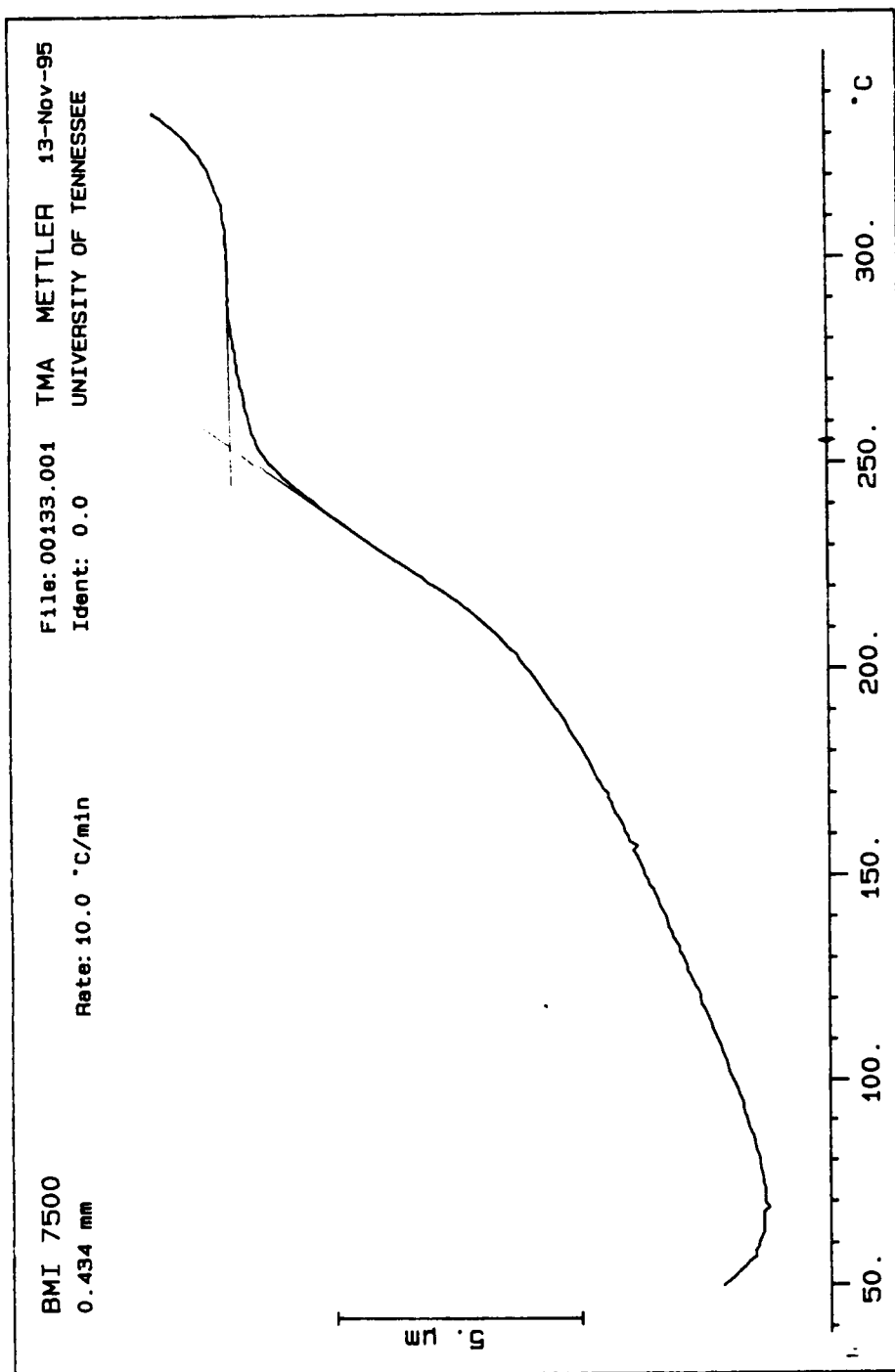


Figure 14. Temperature/displacement plot for specimen aged for 7500 hours.

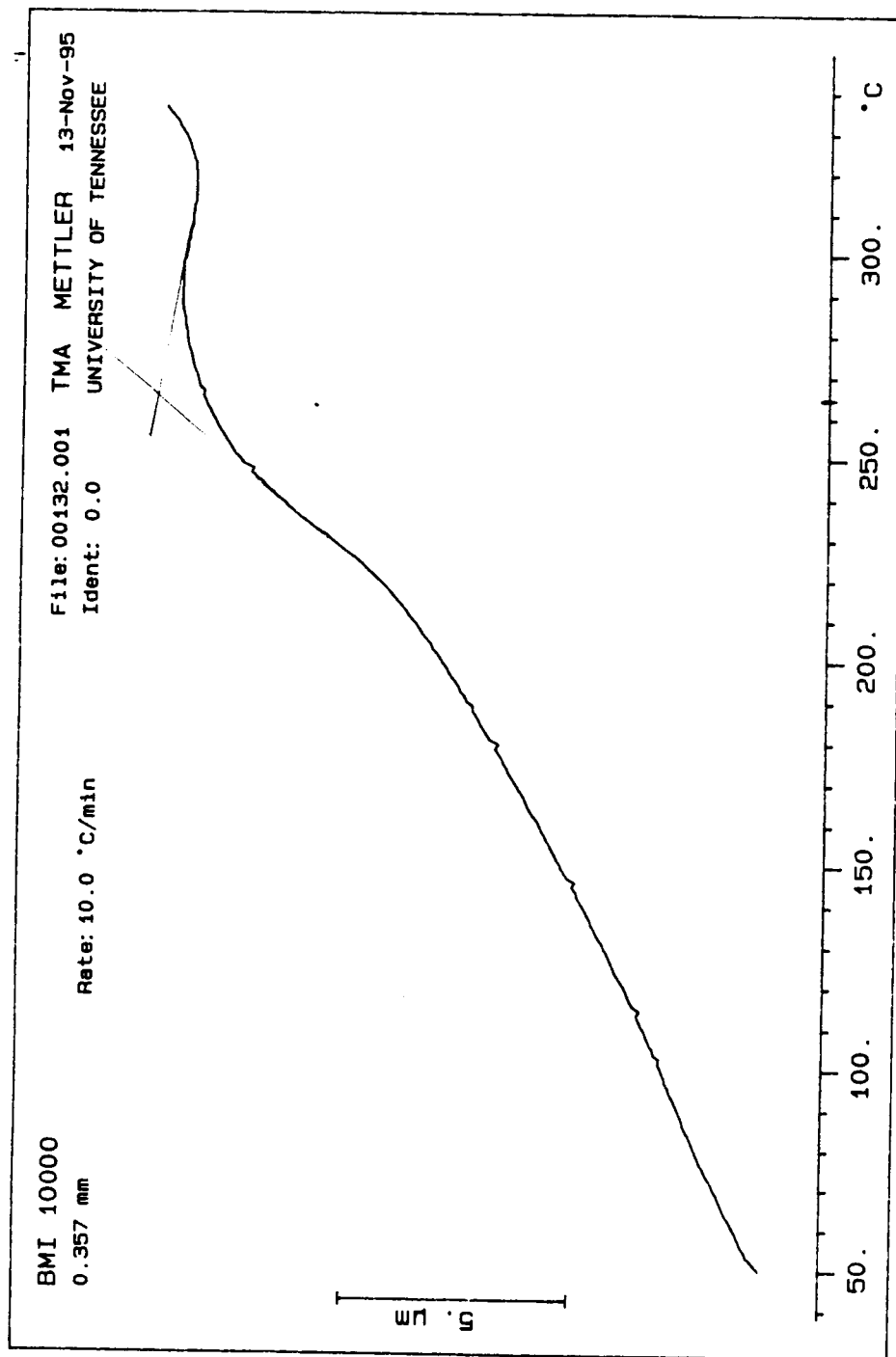


Figure 15. Temperature/displacement plot for specimen aged for 10000 hours.

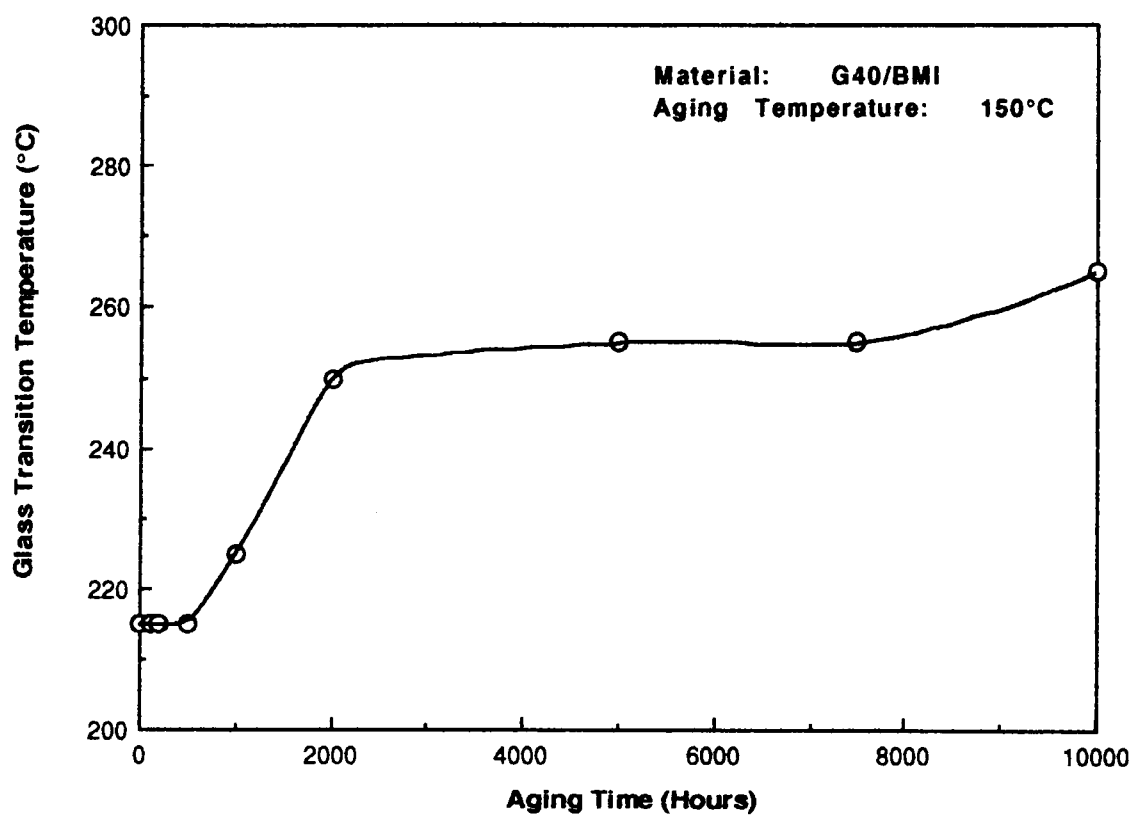


Figure 16. Glass transition versus aging time.

cycles. Therefore, when they are exposed to elevated temperatures for long periods of time, the resins continue to cure and form cross-links between their polymer chains. As can be seen, the shifts in T_g do not appear uniform. For example, a large shift is seen between 1000 and 2000 hours. The reason for this is unknown. Perhaps if more specimens had been tested the increase would not have been as sharp. The statistical average of several test specimens may have produced a smoother curve.

Another change on the TMA plots other than the T_g shift was the change in the shape of the curves. After 2000 hours of aging, the change in slope of the curve was not as severe after the T_g was reached. This effect was due to the continued cross-linking as well. Because more cross-linking has occurred, the polymer chains can't shift as much when the T_g is reached. Therefore, the change in the curve is not as pronounced as specimens with less cross-linking.

The inplane shear strength and shear modulus data is displayed in Table 1 along with the T_g and weight loss data for each aging period. Individual stress/strain curves are included in the appendix. Due to insignificant weight loss, it was not necessary to test the 100, 200, 500, and 1000 hours data because the results would have been similar to the unaged specimen. Therefore, only the 0, 2000, 5000, 7500, and 10,000 hours specimens were shear tested.

A plot of the inplane shear strength versus aging time is shown in Figure 17. There is not much difference between the shear strength of each aging period, so aging at this temperature and duration does not appear to have much effect on the shear strength.

A plot of the shear modulus versus aging time is shown in Figure 18. A small spike occurred at 2000 hours of aging, but the modulus is similar for

Table 1. Shear properties, weight loss, and glass transition temperature data.

Aging Time (hours)	Weight Loss (%)	T _g (°C)	Shear Strength (MPa)	Shear Modulus (GPa)
0	0	215	127.4	5.73
0	0	215	113.9	5.20
100	0	215	--	--
200	0.017	215	--	--
500	0.067	215	--	--
1000	0	225	--	--
2000	0	250	139.1	7.15
2000	0	250	137.6	11.1
2000	0	250	133.7	7.46
2000	0	250	141.0	7.22
5000	0.106	255	123.3	6.86
5000	0.106	255	124.9	6.77
7500	0.196	255	127.8	7.13
7500	0.196	255	136.4	5.51
7500	0.196	255	127.2	5.98
7500	0.196	255	130.9	5.65
10,000	0.289	265	118.8	6.58
10,000	0.289	265	135.8	6.07
10,000	0.289	265	130.4	6.96
10,000	0.289	265	125.7	4.05

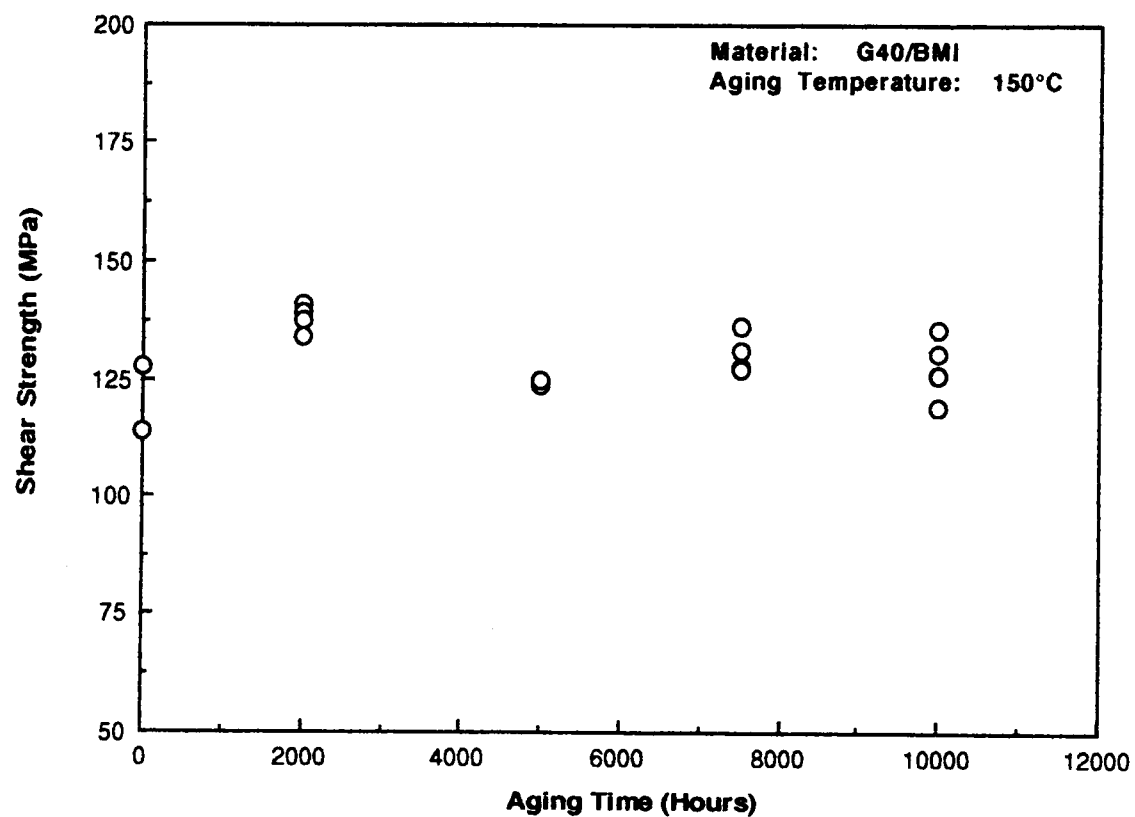


Figure 17. In-plane shear strength versus aging time.

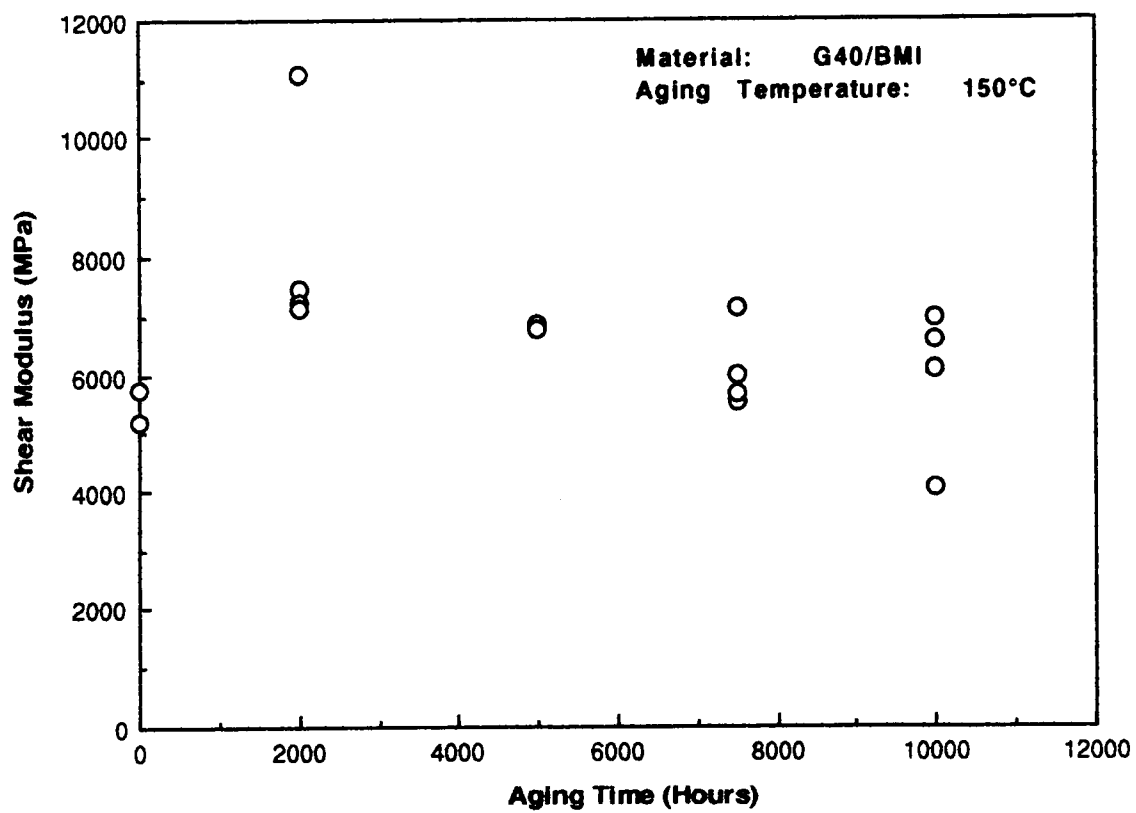


Figure 18. Shear modulus versus aging time.

the other aging periods. The increase in the modulus at 2000 hours of aging can be explained by the increase in T_g which would make the composite stiffer, but the reason for the decrease in modulus after 2000 hours is unknown. The decrease might have been caused by property degradation due to aging, or the entire spike might have been due to a defective specimen. As can be seen on the plot, one specimen aged for 2000 hours had a much higher value than the others. The shear modulus was not expected to change a great deal, a fact substantiated by a previous report (13). The decrease stabilized at the 7500 and 10,000 hours specimens with values similar to the unaged specimen. Therefore, over the entire 10,000 hours the shear modulus did not change very much.

One needs to use caution when using shear data as a marker of aging induced damage. The weight loss was low in this case, so there was no reason to expect major changes in the inplane shear strength or shear modulus. However, these specimens were cut from 5x5 inch plates after being aged. The most severe aging damage would be around the edge. The region being tested in the Iosipescu test is between the notches, which were machined after aging. Therefore, the area that would undergo the most damage is not being tested. In the future, it would be better to machine the specimens before the aging.

Finally, the experiments with the different size and shape of specimens proved to be inconclusive. The weight loss percentages ranged between 0.2% and 0.3%. Any differences between the specimens would be within the error of the balance, so no conclusions about which side suffered the most aging damage could be drawn.

CHAPTER 4

CONCLUSIONS

The specimens started suffering significant weight loss after 5000 hours of aging. The maximum weight loss was 0.3%. The glass transition temperature shifted a total of 50 degrees from 215°C to 265°C over the 10,000 hour aging range. The biggest increase occurred between 1000 and 2000 hours.

There was no major change in the inplane shear strength after aging. Over the entire aging range, the shear modulus did not change much. However, there was a spike at 2000 hours where the modulus increased and then decreased. The increase could be explained by the change in T_g , but the reason for the decrease is not known. Further testing needs to be done to determine if the decrease is indeed due to aging damage, or if the entire spike was the result of a defective specimen.

The aging of the different size specimens did not reveal anything conclusive about which side of the plate suffered the most damage. The weight loss was not large enough.

Even though the weight loss was not very large, it was enough to take into consideration if the material were to be used in an application where it would be exposed for long periods of time at this temperature. Overall, the TOS of this material is good below 150°C. Above that temperature, the TOS starts to decline.

For future aging studies, more mechanical properties need to be tested. Not only inplane shear, but tensile, compressive, and flexure properties would give a more thorough understanding of the TOS. Also, for this material, a higher temperature would be best in order to get more aging

damage. With this, a better understanding of how a material with low TOS responds to elevated temperature could be obtained.

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APPENDIX

APPENDIX

This appendix contains the shear stress-strain plots for each of the specimens tested by the Iosipescu shear method. The specimens were aged at 150°C for 0, 2000, 5000, 7500, and 10,000 hours. Four specimens were tested from each aging period. Due to instrument mishap, two specimens were accidentally destroyed from the 0 hours set and two were destroyed from the 5000 hours set.

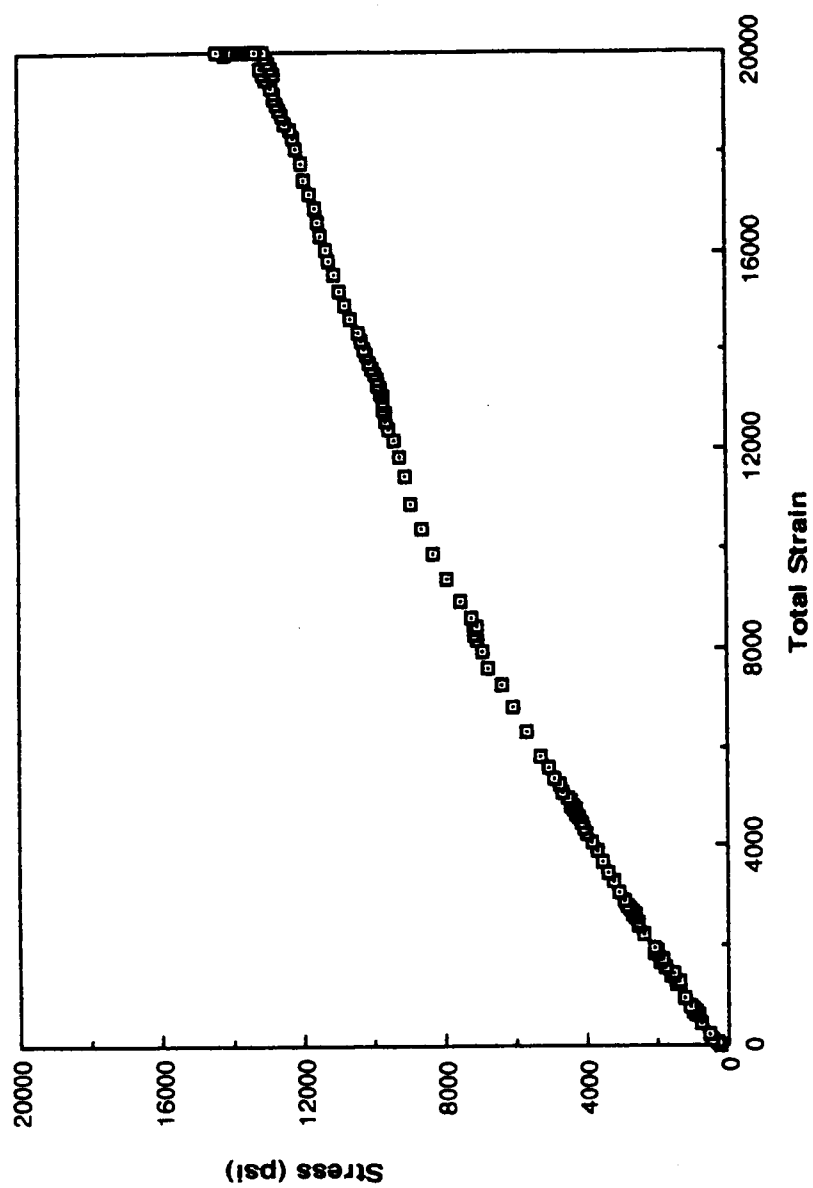


Figure A1. Shear stress/strain plot of unaged specimen no. 1.

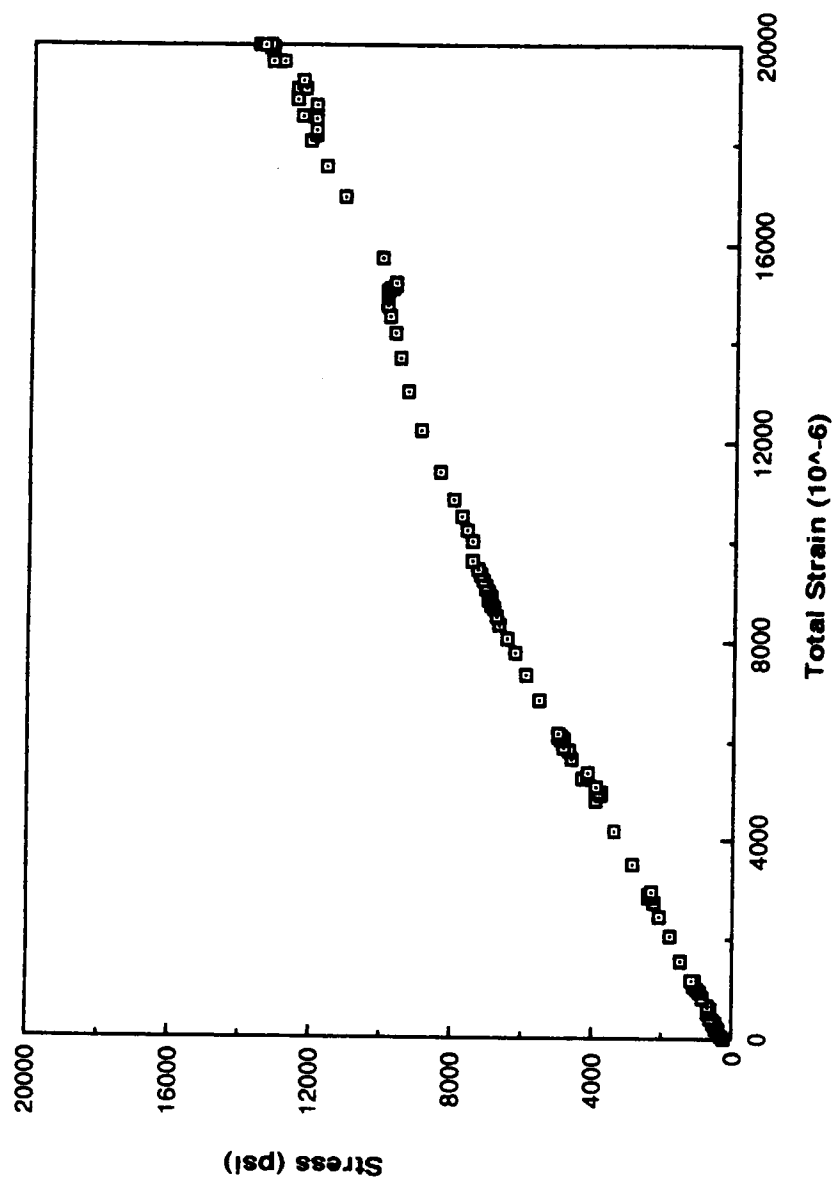


Figure A2. Shear stress/strain plot of unaged specimen no. 2.

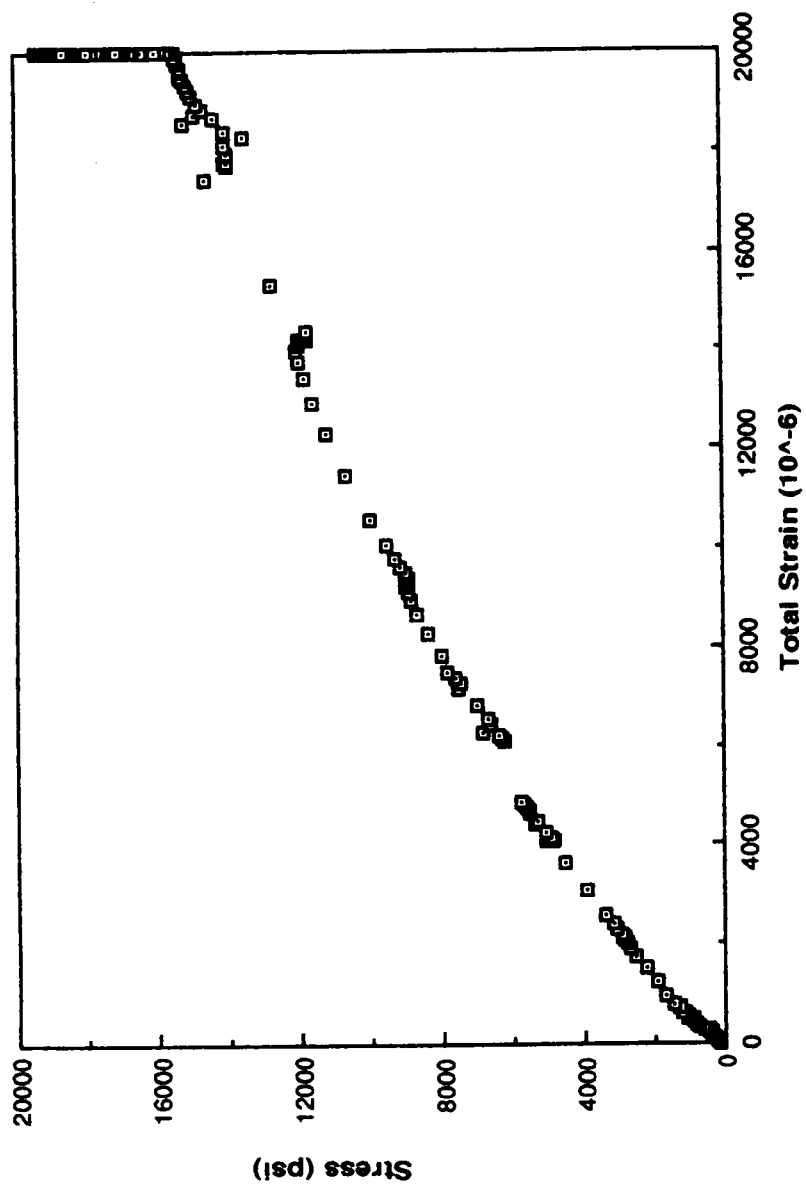


Figure A3. Shear stress/strain plot of specimen aged 2000 hours, no. 1.

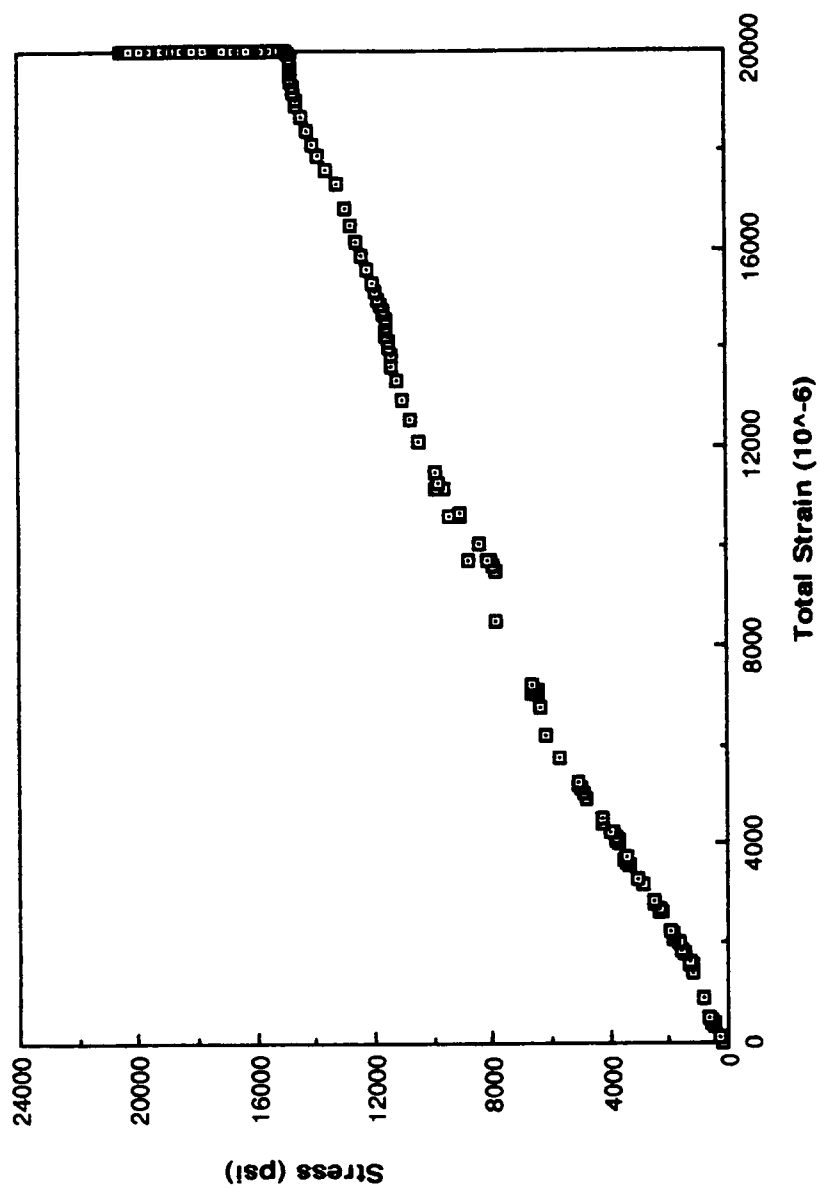


Figure A4. Shear stress/strain plot of specimen aged 2000 hours, no. 2.

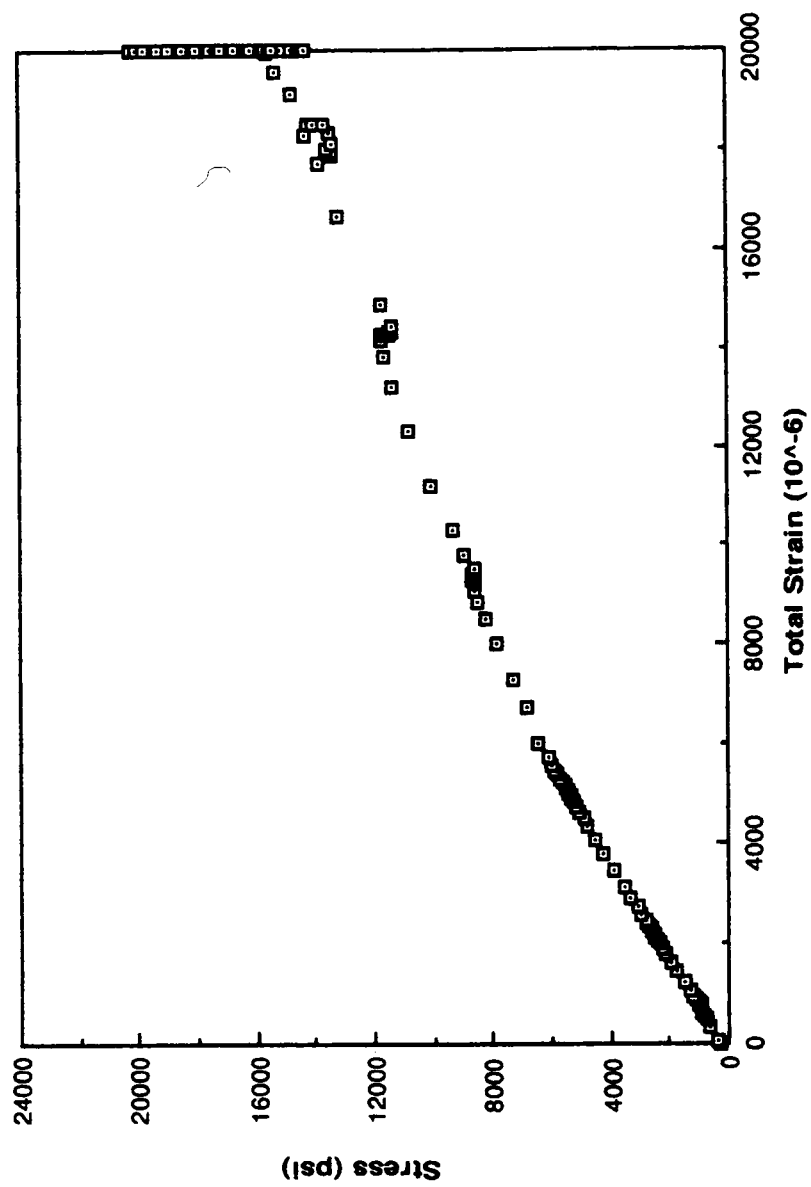


Figure A5. Shear stress/strain plot of specimen aged 2000 hours, no. 3.

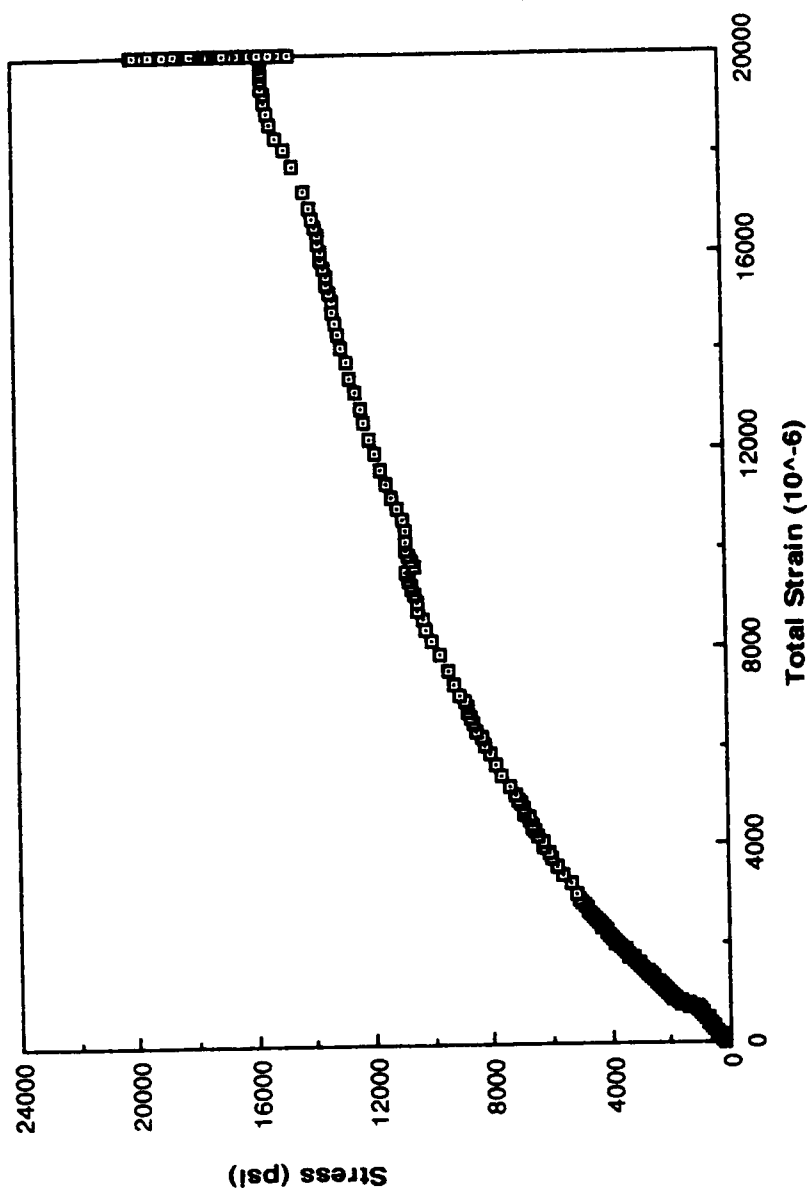


Figure A6. Shear stress/strain plot of specimen aged 2000 hours, no. 4.

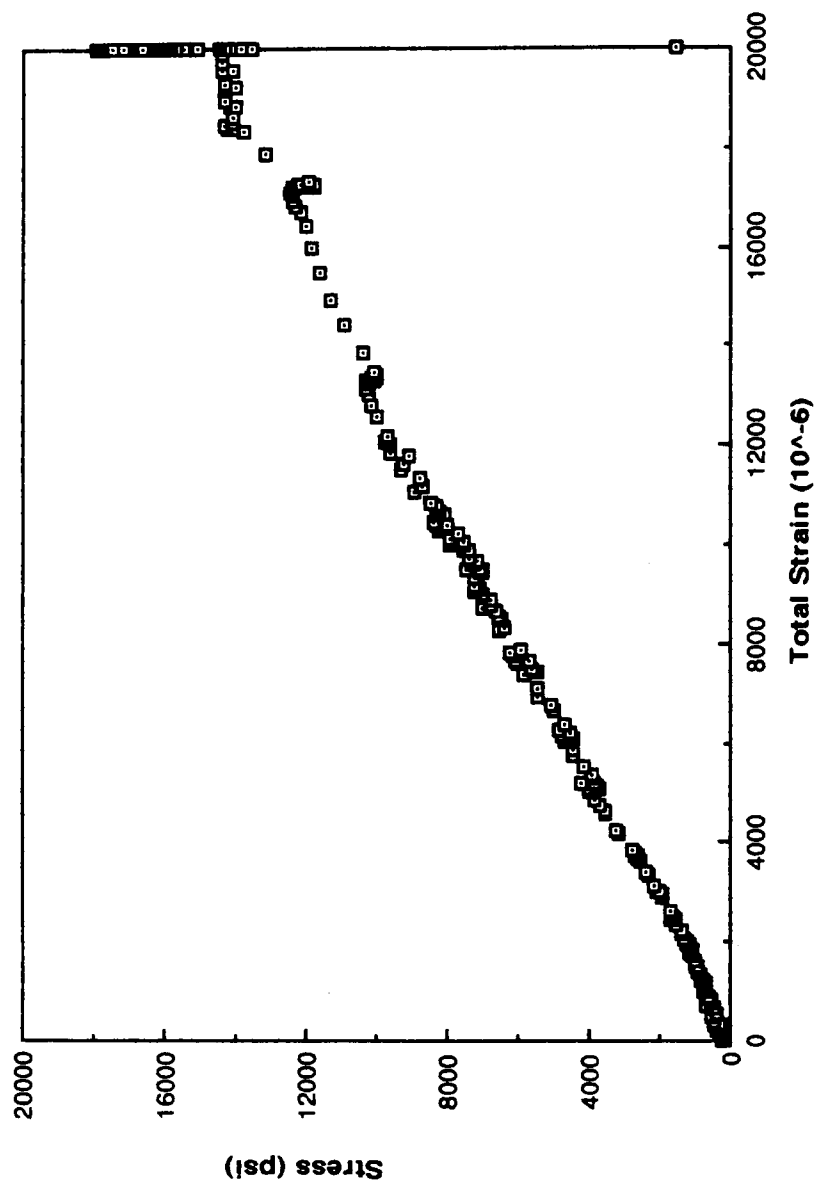


Figure A7. Shear stress/strain plot of specimen aged 5000 hours, no. 1.

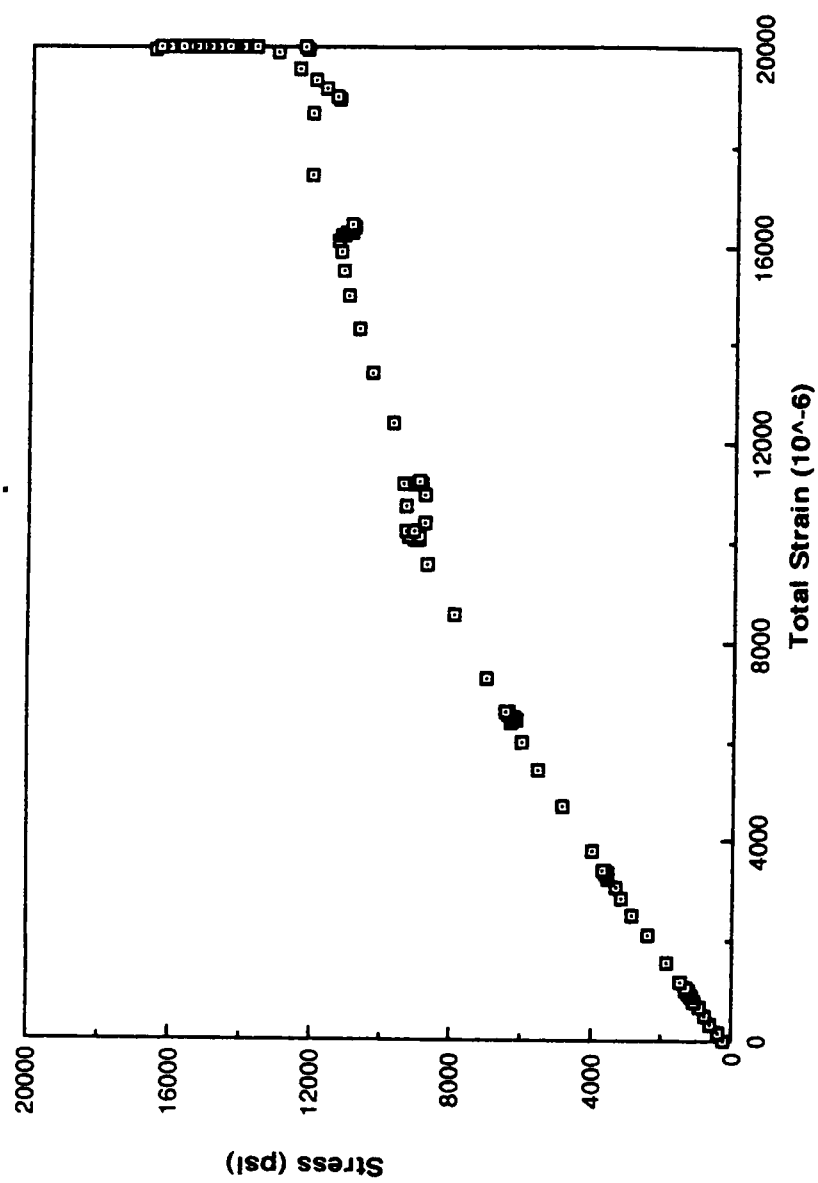


Figure A8. Shear stress/strain plot of specimen aged 5000 hours, no. 2.

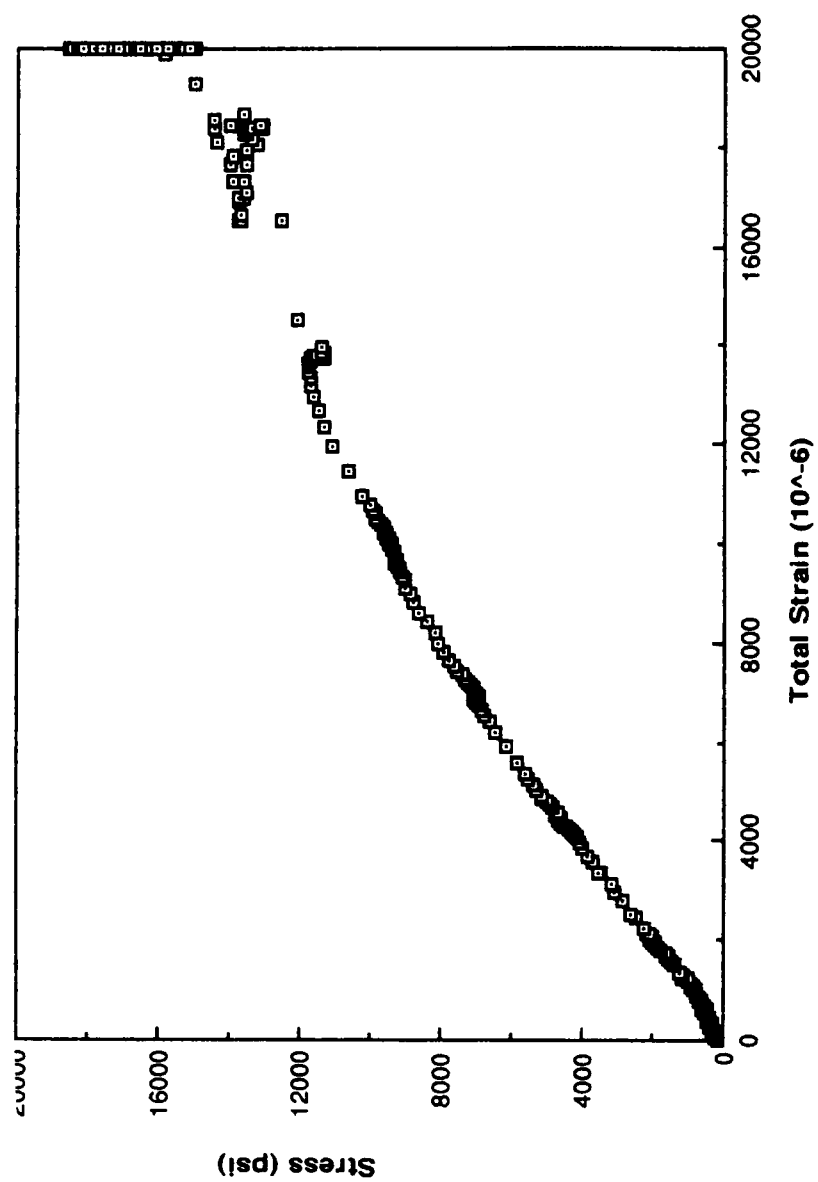


Figure A9. Shear stress/strain plot of specimen aged 7500 hours, no. 1.

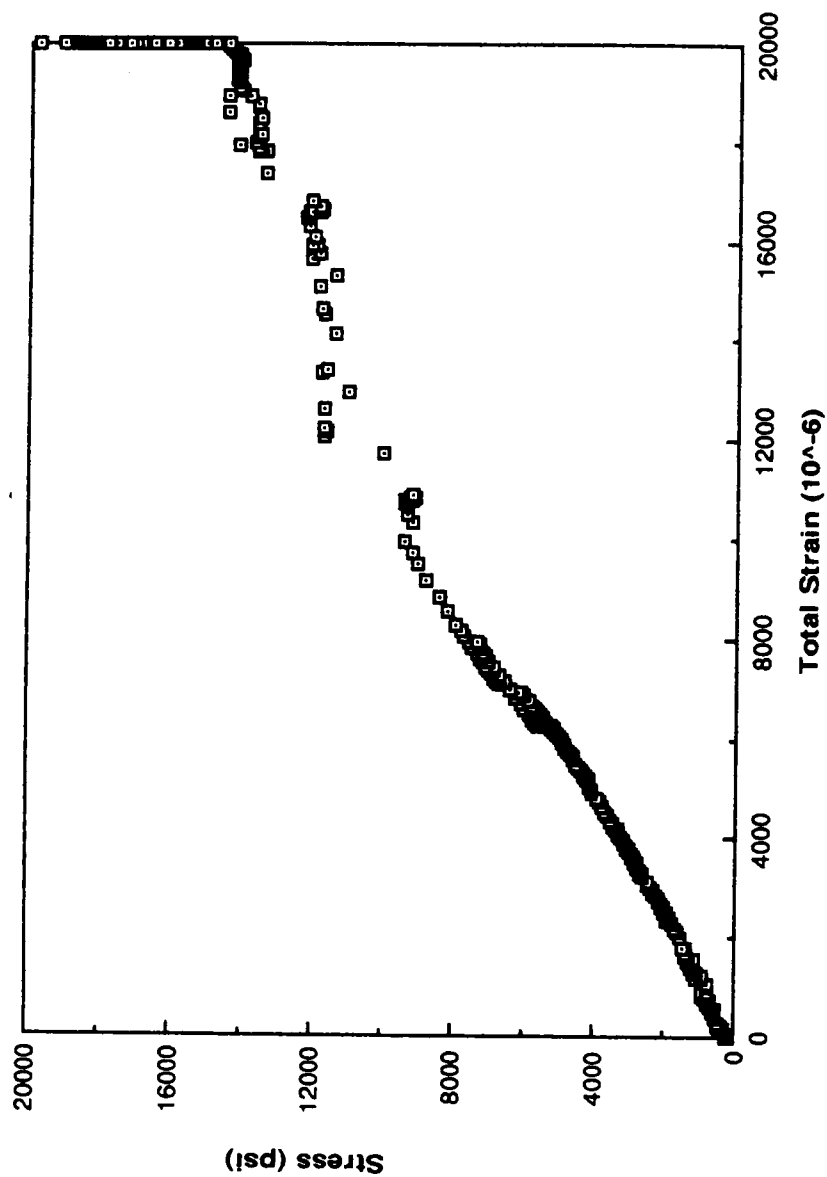


Figure A10. Shear stress/strain plot of specimen aged 7500 hours, no. 2.

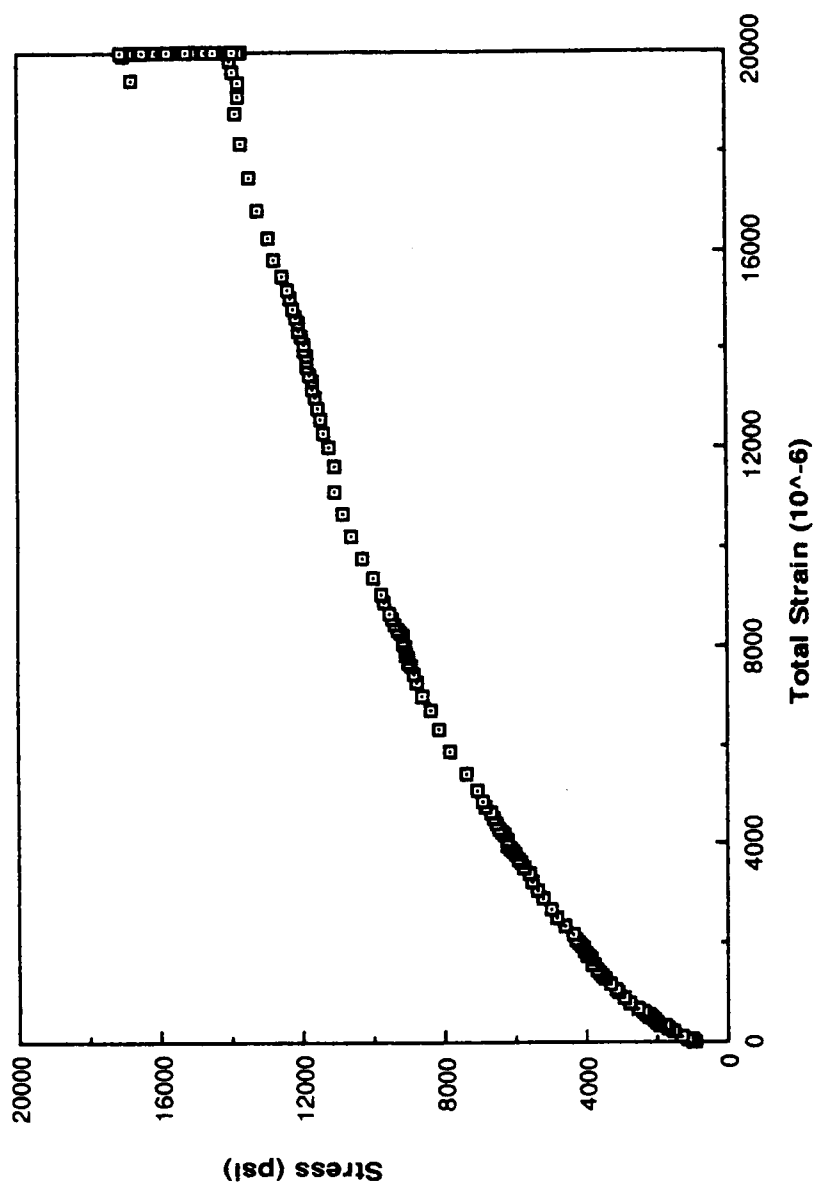


Figure A11. Shear stress/strain plot of specimen aged 7500 hours, no. 3.

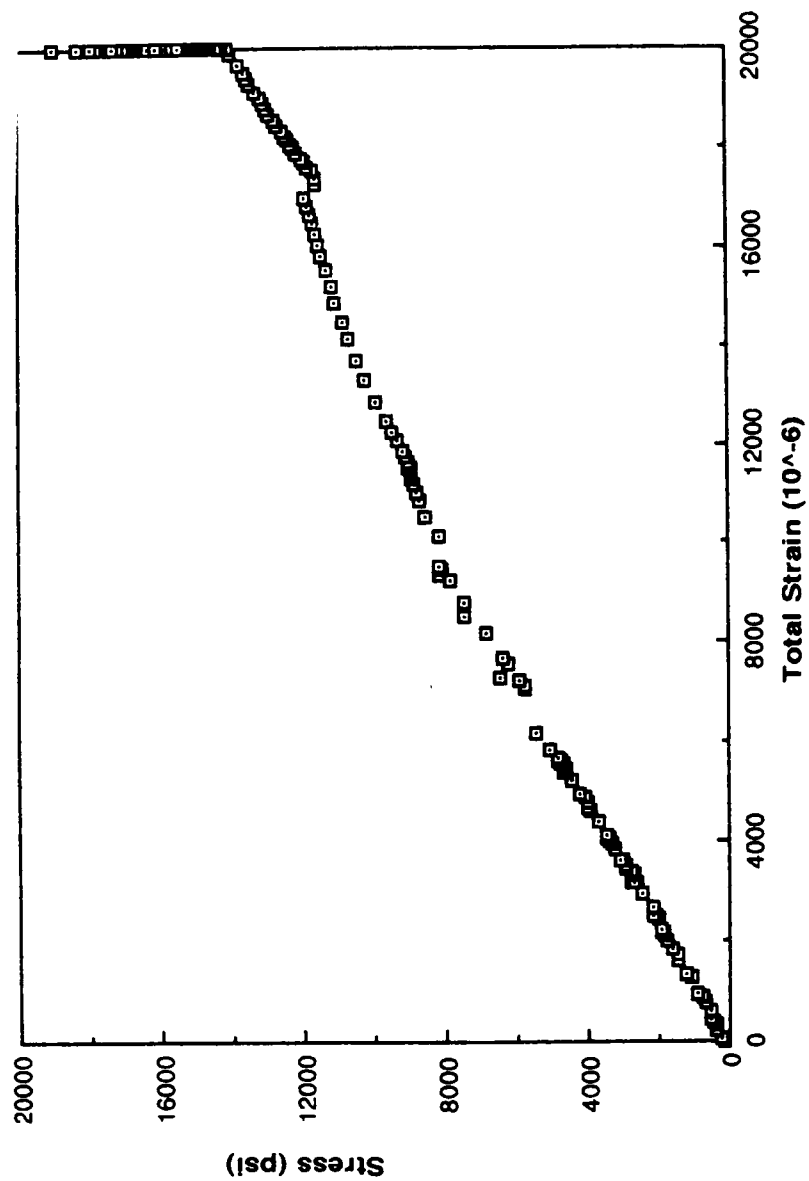


Figure A12. Shear stress/strain plot of specimen aged 7500 hours, no. 4.

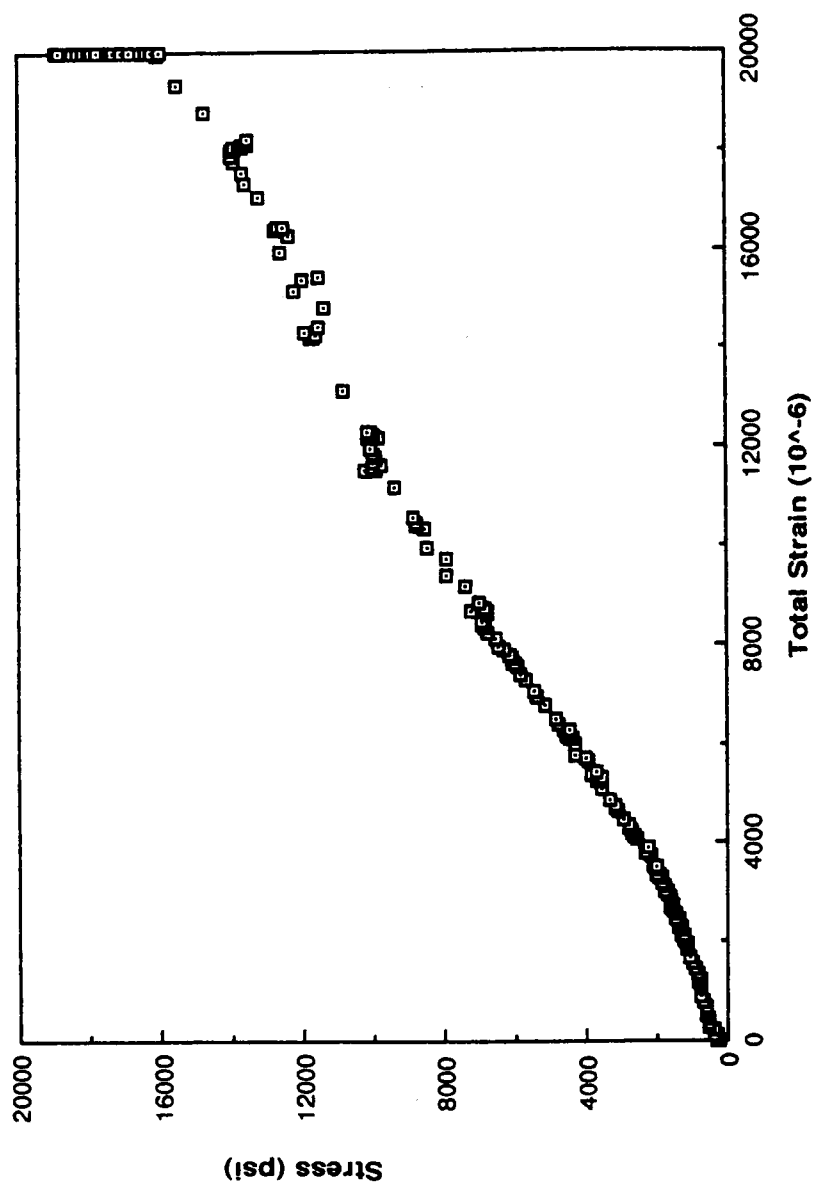


Figure A13. Shear stress/strain plot of specimen aged 10,000 hours, no. 1.

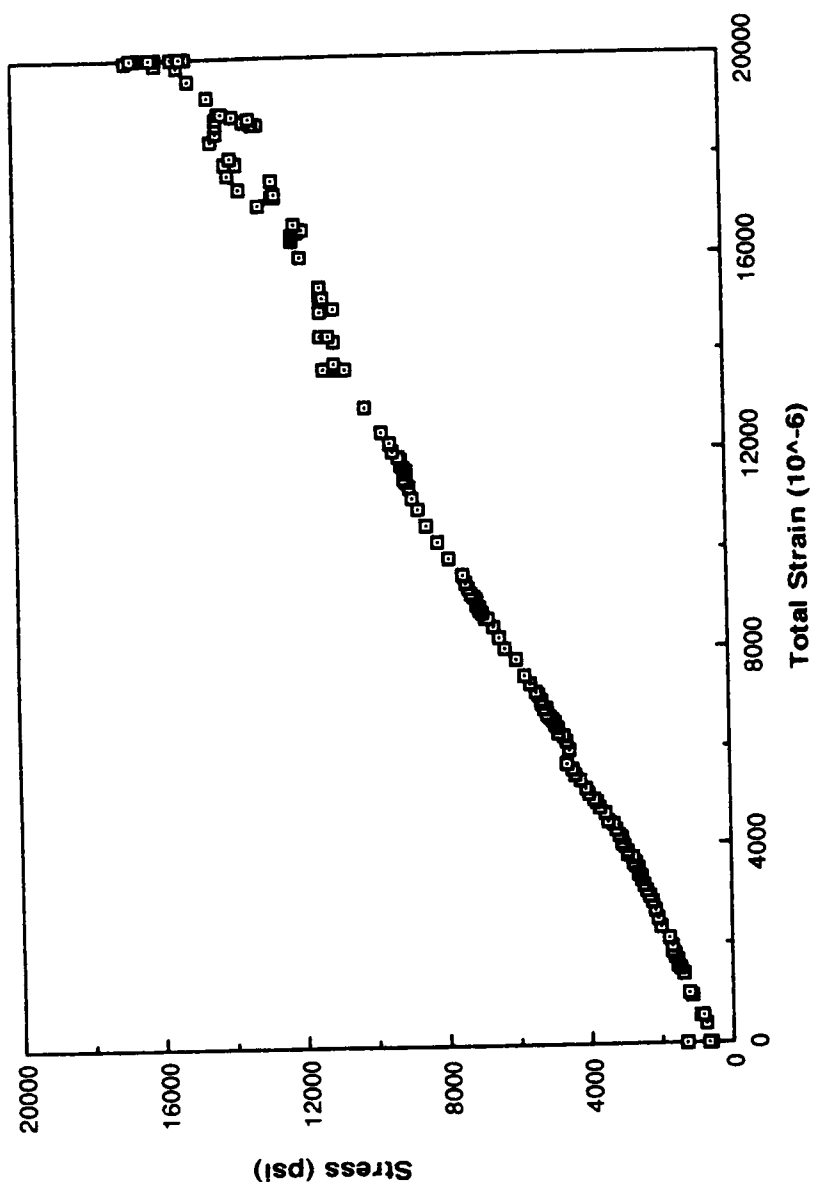


Figure A14. Shear stress/strain plot of specimen aged 10,000 hours, no. 2.

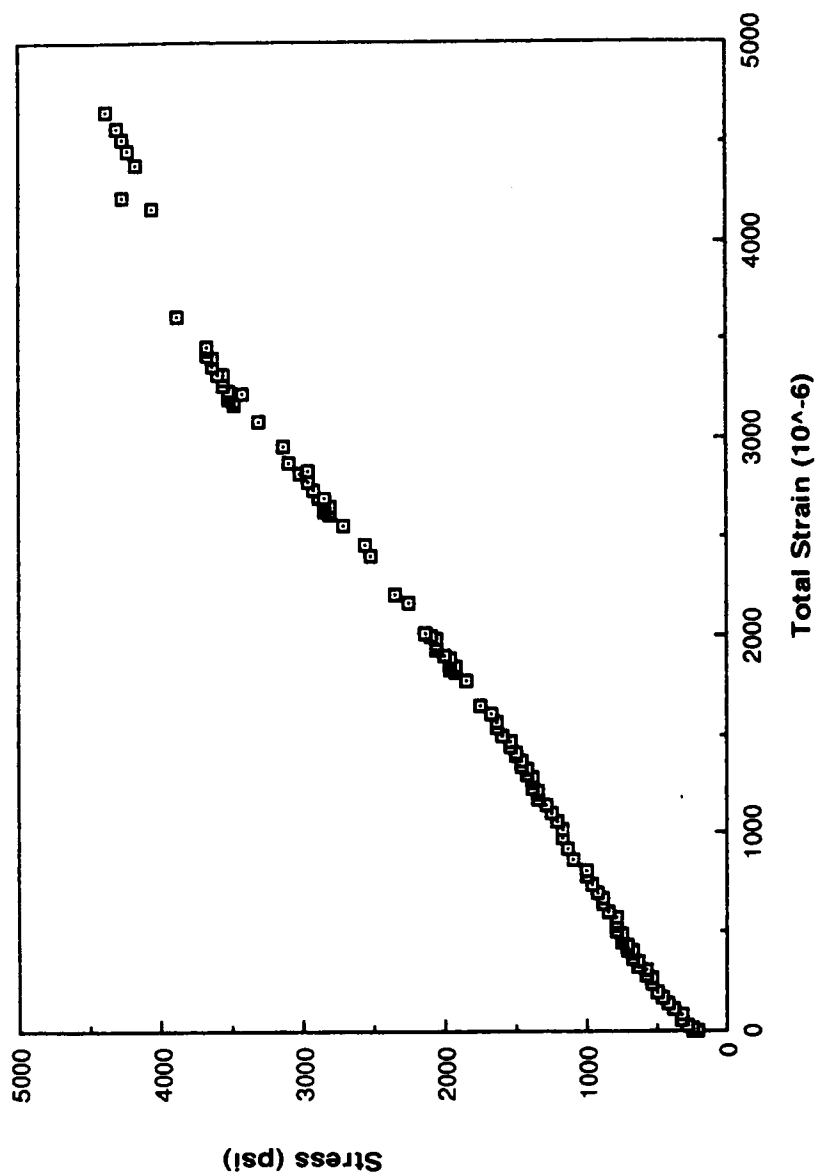


Figure A15. Shear stress/strain plot of specimen aged 10,000 hours, no. 3.

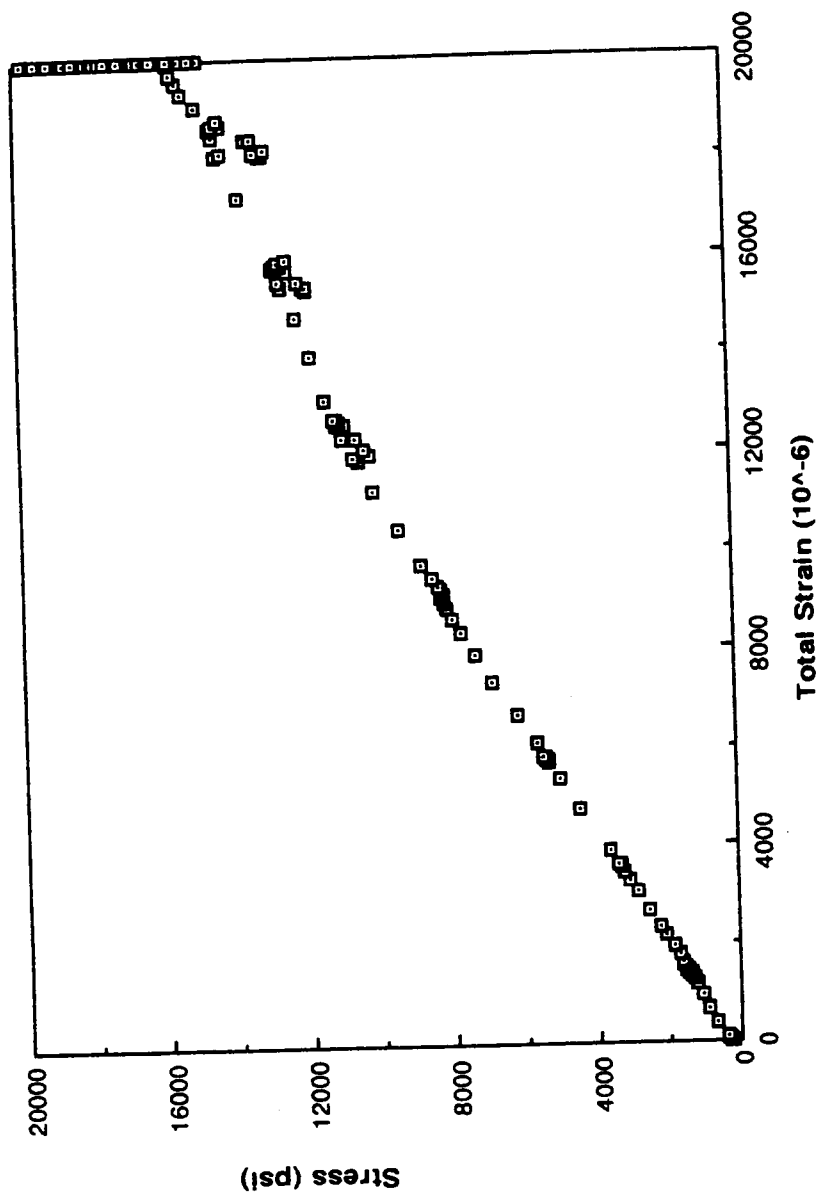


Figure A16. Shear stress/strain plot of specimen aged 10,000 hours, no. 4.

**PART III: EFFECTS OF ISOTHERMAL
AGING ON INTERFACIAL PROPERTIES
OF IM7/EPOXY COMPOSITES**

CHAPTER 1

INTRODUCTION

During the past few years, interest in interfacial research has increased. Scientists and engineers are realizing that the interface between the fiber and the matrix is one of the most important considerations when working with composites. Several papers have been written which show the effect an interface has on the mechanical properties of a composite (1-8).

Usually a good interface means good adhesion between the fiber and the matrix. A weak interface may cause early debonding, but a very strong interface may cause the composite to behave in a brittle matter. Therefore, there is an optimum value of adhesion which will give the best interface. Several papers have been written which focus on the fiber/matrix adhesion (9-11).

Originally, the interface was viewed as a two dimensional zone. It was the boundary between the fiber and the matrix. Now, however, the interface is viewed as a three dimensional region and is termed the interphase. Figure 1 shows a schematic of this interphase. At some point in the matrix, the bulk properties change. This is the starting of the interphase. It continues through the actual fiber/matrix interface and into the fiber where the fiber properties along the interface are a little different from the properties of the bulk fiber. When these properties change back to those of the bulk fiber, the interphase ends. Any accurate understanding of the interface needs to take this three dimensionality into account (12).

Several test methods are available for characterizing the interfacial strength (13-15). The method used for this study is the single fiber

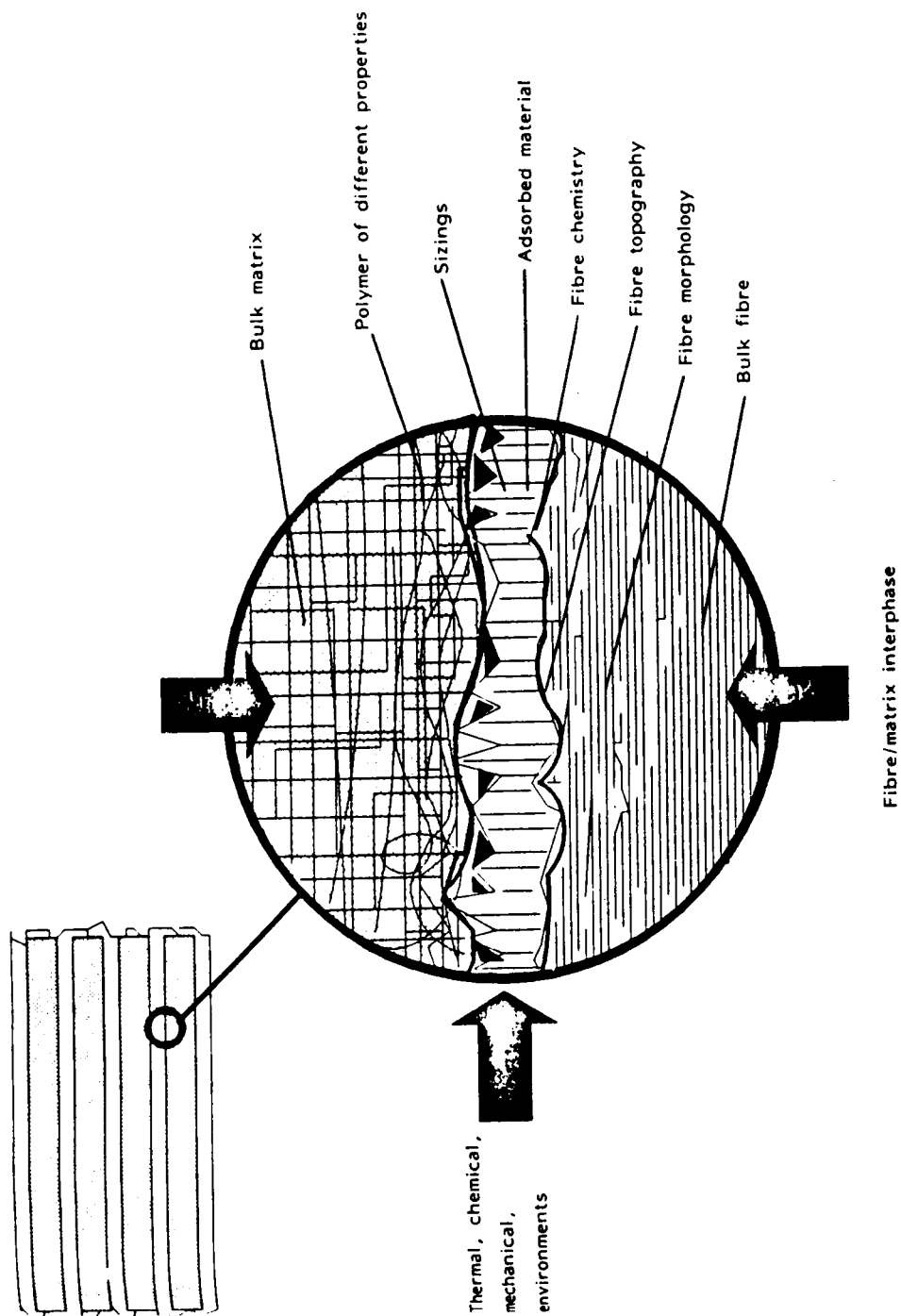


Figure 1. Schematic of the fiber/matrix interphase.
(From Reference 15)

fragmentation test. In this test, a single-fiber dog-bone-shaped coupon is put into a loading apparatus and subjected to a monotonically increasing load. The load is transferred from the matrix to the fiber through shear forces at the interface. When the stresses in the fiber build to a high enough level, the fiber will fracture. The fiber will continue to fracture as the load is increased. At the saturation point, the fiber segments become too short to receive sufficient stress for further fragmentation. The fiber will not fracture anymore, and this length is called the critical length. This process is shown schematically in Figure 2. Several studies concerning the theoretical aspects of this test have been published (16-18).

The equation shown in Figure 2 is obtained from a simple force balance on a fiber element and relates the interfacial shear strength to the fiber critical length l_c . As the critical length increases in the equation, the interfacial shear strength decreases, and a lower interfacial shear strength means a weaker interface. There is some controversy over this equation. It is derived from a force balance along the fiber and assumes constant shear stresses along the interface. This assumption is invalid. However, this assumption does not affect this study because the equation was used in a qualitative manner to understand the effect of aging on interface degradation.

When working with composites, an engineer or scientist needs to be aware of any changes during the lifetime of the composite part. In particular, changes in the interface need to be closely monitored because these changes could cause a degradation of the composite's mechanical properties. Exposure to high temperatures could affect the interface severely (19). Therefore, it is important to know how the interface reacts when exposed to elevated temperatures.

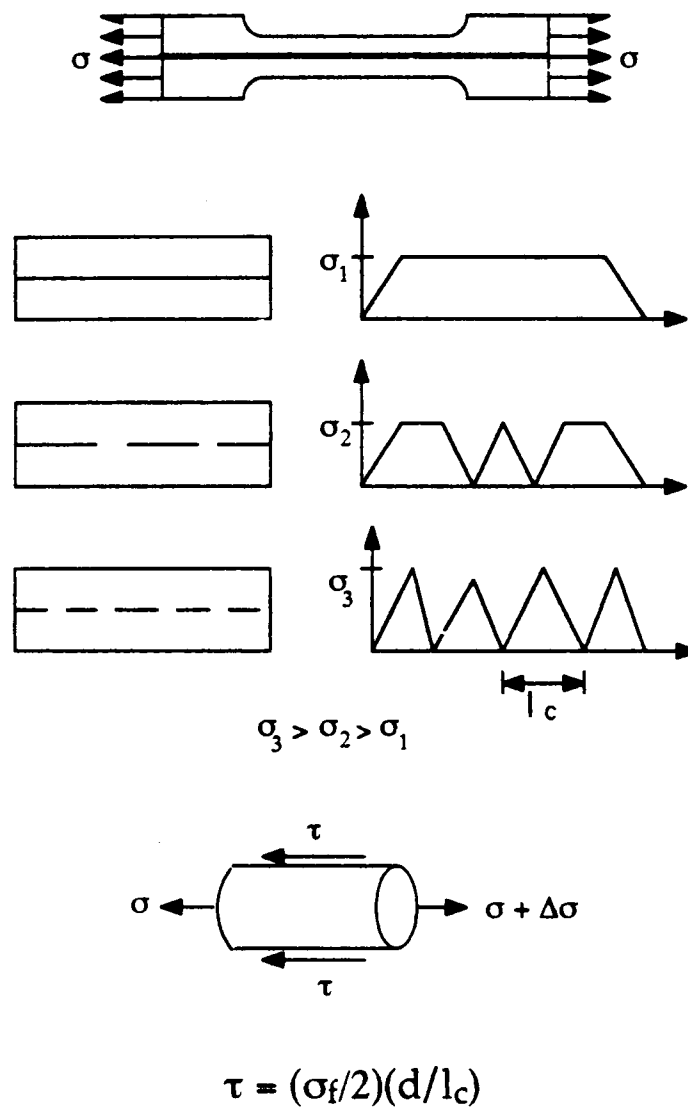


Figure 2. Schematic of the single fiber fragmentation test process.

Several studies have looked at the temperature dependence of the interface (20-24). However, these studies have not included the effects of isothermal aging on the critical length or interfacial failure mechanism. This research project uses the fragmentation test along with some other techniques to study these aging effects. The purpose of this research is to determine how isothermal aging affects the interface and to what extent.

CHAPTER 2

EXPERIMENTAL PROCEDURE

Dog-bone-shaped single-fiber specimens were made using an epoxy/carbon fiber material system. The fiber was an IM7 type carbon fiber, which is a PAN-based fiber manufactured by Hercules, Inc. It has a tensile strength of 5308 MPa (770 ksi) and a tensile modulus of 275,760 MPa (40,000 ksi). The resin was a diglycidyl ether of bisphenol-A (commercial name Epon 828) manufactured by Shell Chemical Company and was cured with an aromatic diamine named meta-phenylene diamine (m-PDA). The resin typically has a tensile strength of 89.6 MPa (13 ksi) and a tensile modulus of 3619 MPa (525 ksi). Simple to prepare, this material system is commonly used in interfacial studies. It is well characterized, has a low temperature cure cycle, and is similar to actual aerospace matrices.

The resin mixture was a ratio of 14.5 parts by weight of m-PDA to 100 parts by weight of Epon 828. After mixing, the resin was placed into an oven chamber under vacuum at a temperature of 75°C in order to degas the mixture and remove any potential gas bubbles. After 10 minutes of degassing, the resin was poured into a mold in which the IM7 fiber had already been placed. The entire specimen was then cured.

The total cure cycle was five hours with one hour at 75°C, two hours increasing from 75°C to 125°C, one hour at 125°C, and finally a one hour cool-down to room temperature. This is an optimized cure cycle determined to give the coupon the least amount of residual stresses. A schematic of the curing set-up is shown in Figure 3.

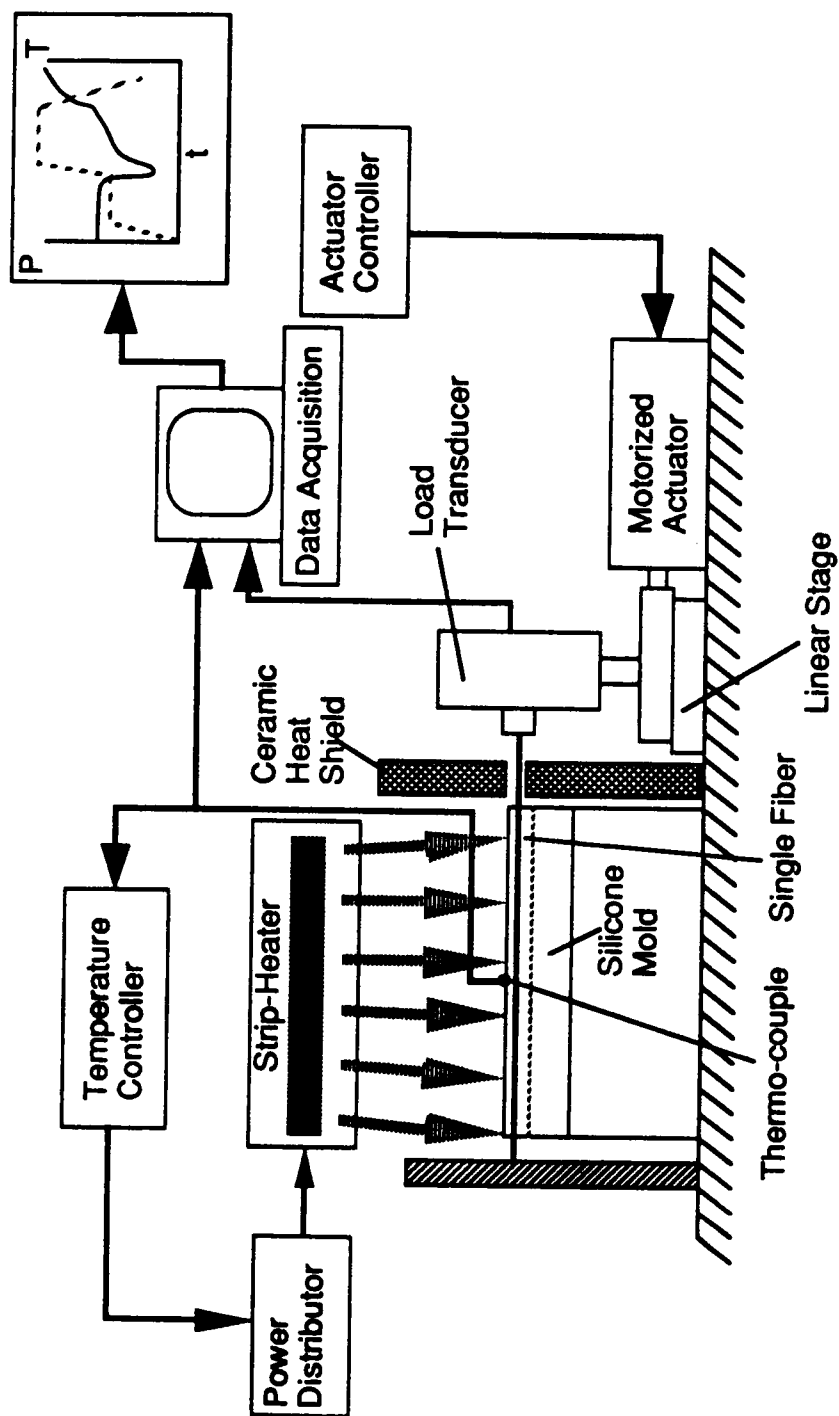


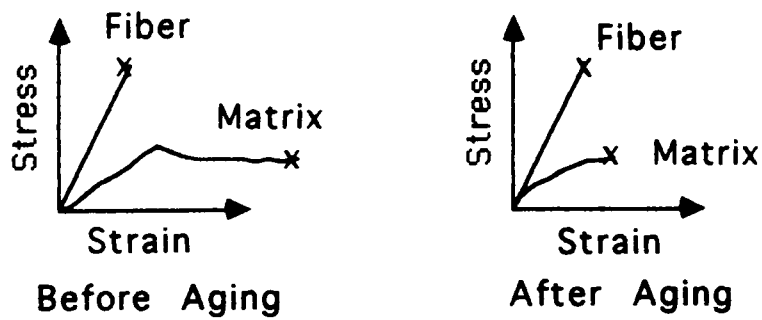
Figure 3. Schematic of the experimental set-up for curing specimens.

During the cure cycle, one end of the fiber was attached to a fixed mount while the other end was attached to a load cell mounted on a motorized track. The load cell monitored the tension in the fiber while the track allowed the fiber to be loaded to the desired tension level during curing. This pre-tensioning of the fiber was a solution to two problems encountered in this study.

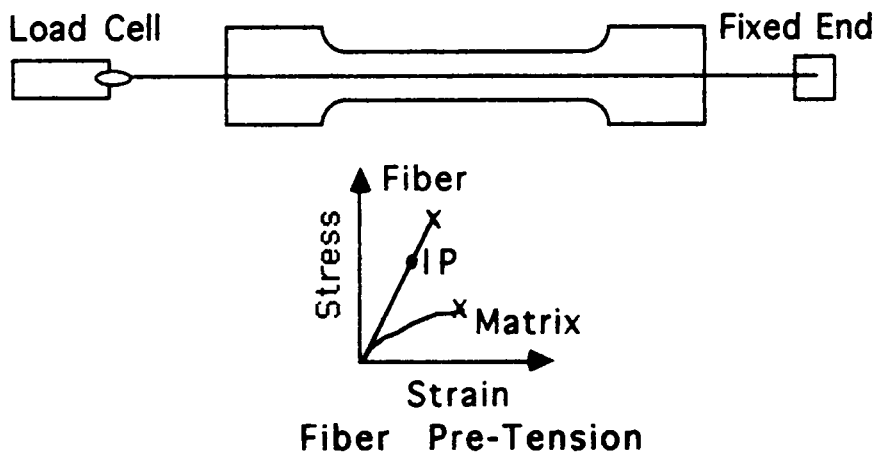
The first problem was the variability of fiber tension from specimen to specimen. When a fiber is placed in the mold and attached to two fixed ends, a tension is introduced into the fiber. There is no way of knowing what this tension is, and it will vary from specimen to specimen. Thus, another variability has to be taken into account during the fragmentation test. The load cell allowed a constant pre-tension to be set for each fiber in this study, and thus, eliminated the variability problem.

The second problem was matrix embrittlement. As specimens age, the failure strain of the matrix decreases due to embrittlement. Embrittlement reduces the matrix failure strain and causes the specimen to fail before the fiber fracturing reaches its saturation point (See Figure 4a). The fragmentation test requires a matrix with a failure strain three times as great as that of the fiber. A pre-tension on the fiber causes the fiber loading to begin at an initial point, IP, during the fragmentation test (See Figure 4b). As a result, the fiber will fracture earlier and reach its saturation point before the matrix reaches its failure strain and the specimen fails. Therefore, the problems resulting from the embrittlement of the matrix are overcome when a fiber pre-tension is used. By trial and error, a pre-tension of 2996 MPa (435 ksi) was found to work best for this study.

As seen in Figure 3, a quartz strip heater applied the temperature cycle to the resin while the load cell monitored the changes in fiber tension due to



a) Reduction of the matrix failure strain due to embrittlement.



b) Application of pre-tension which starts fiber tension from an initial point, IP.

Figure 4. Fiber pre-tension solution.

thermal expansion and contraction. A data acquisition program recorded the temperature and tension data on a computer. From this data, a plot of temperature and fiber tension versus curing time was obtained (See Figure 5). Although not a part of this research project, these plots can be used to find optimum cure cycles that give composite resins the least amount of residual stresses.

After curing, the specimens were aged in a Blue M® circulating air oven at a temperature of 100°C for periods of 0, 500, 1000, and 2000 hours. Upon removal, the specimens were visually inspected and then stored in a dessicator until they were tested. Specimens were weighed before and after being placed in the furnace in order to determine any weight loss by oxidation or other chemical attack due to aging.

The glass transition temperature was measured at each aging time using thermo-mechanical analysis (TMA). This method employed a glass probe which is pressed on the specimen with a specified force (see Figure 6a). The specimen was heated over a selected temperature range which caused it to undergo volume changes. The displacement of the probe due to these expansions and contractions was measured and plotted against the temperature. When a material reaches its glass transition temperature, a major volume change occurs. Thus, any sudden change in the slope of the line on the temperature/displacement plot indicates the glass transition temperature (25). In this project, the force of the probe on the specimen was 0.1 Newtons, and the temperature was increased from 50°C to 250°C at a rate of 10°C/minute. Nitrogen gas was the only gas used in the chamber containing the specimen and probe to prevent any reaction with oxygen at elevated temperatures. After a temperature/displacement plot was obtained,

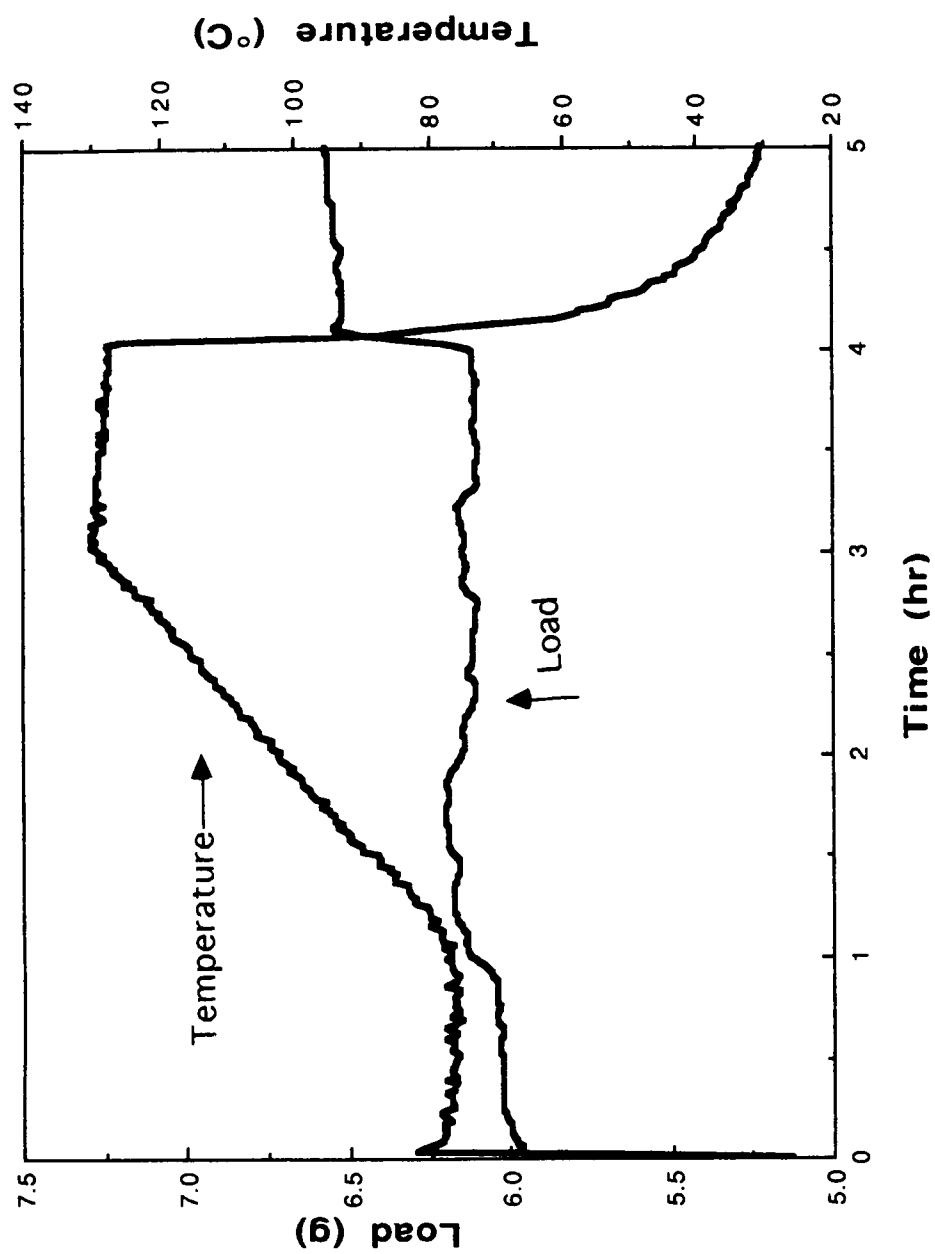
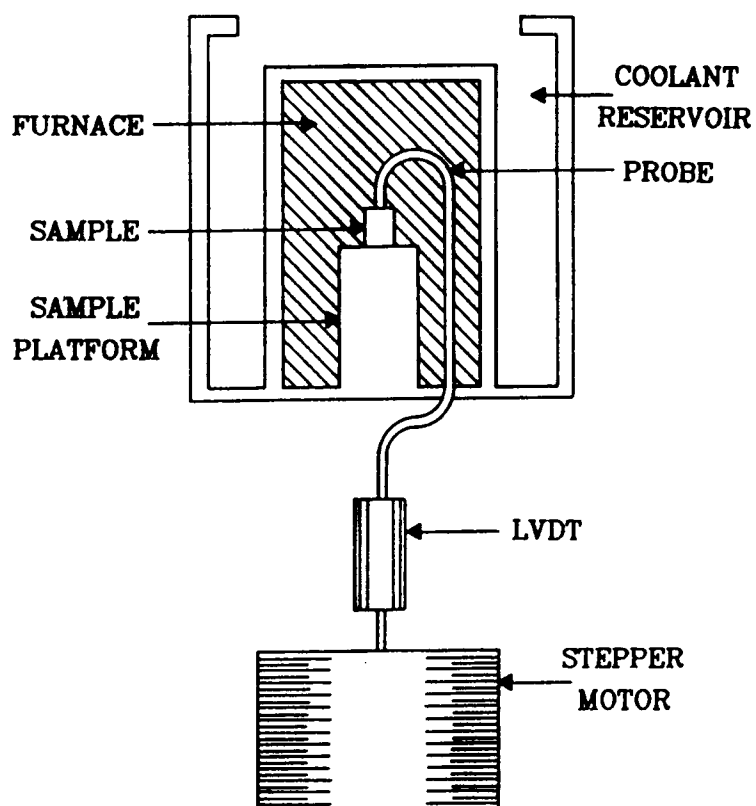
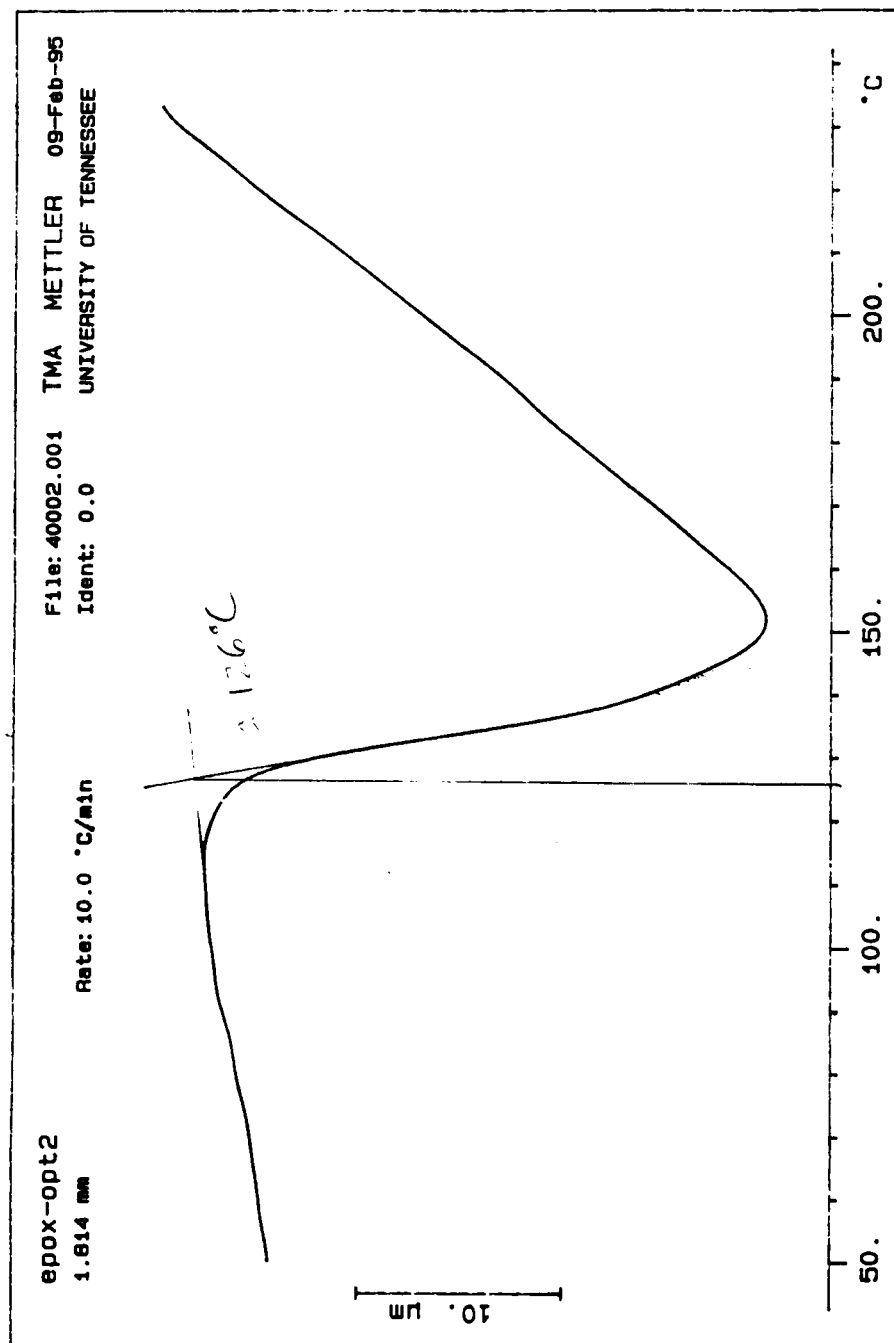


Figure 5. Cure cycle plot of temperature and fiber tension vs. curing time.



a) Schematic of TMA experimental set-up.
(From Reference 25)

Figure 6. Glass transition temperature procedure.



b) T_g determination by change in slope

Figure 6 (continued). Glass transition temperature procedure.

the glass transition temperature was determined by the location of the slope change of the line (see Figure 6b).

The interfacial properties of the specimens were tested using the single fiber fragmentation test described in the introduction. The experimental set-up is shown in Figure 7. Three specimens were tested from each aging time, except for the unaged case where only two specimens were tested. All specimens were loaded until they reached their saturation point. Then, the fiber lengths were measured and the average value of these lengths was taken to be the critical length.

No statistical analysis of the fiber length data was done. Many researchers use Weibull analysis or other methods to account for variations the fiber critical length. However, the method used, whether Weibull statistics or the normal average, would not have affected the research results. Therefore, in-depth statistical analysis seemed redundant and was not performed.

As previously mentioned, the equation in Figure 2 relating critical length to interfacial shear stress was used on a qualitative basis only. Basically, as the critical length increases, the interfacial shear strength decreases. Thus, increases in the critical length indicate a degradation of the fiber/matrix interface while decreases in the length indicate an improvement.

When the fiber fractures, it tries to "recoil" to its unstressed position which the bond between the matrix and the fiber prevents it from doing. This introduces shear stresses around the fracture area at the interface. Transmitted polarized light from a microscope forms a birefringence pattern in areas with high shear stresses. Thus, the stress state around the fiber fracture was observed and studied by making use of this phenomenon. For

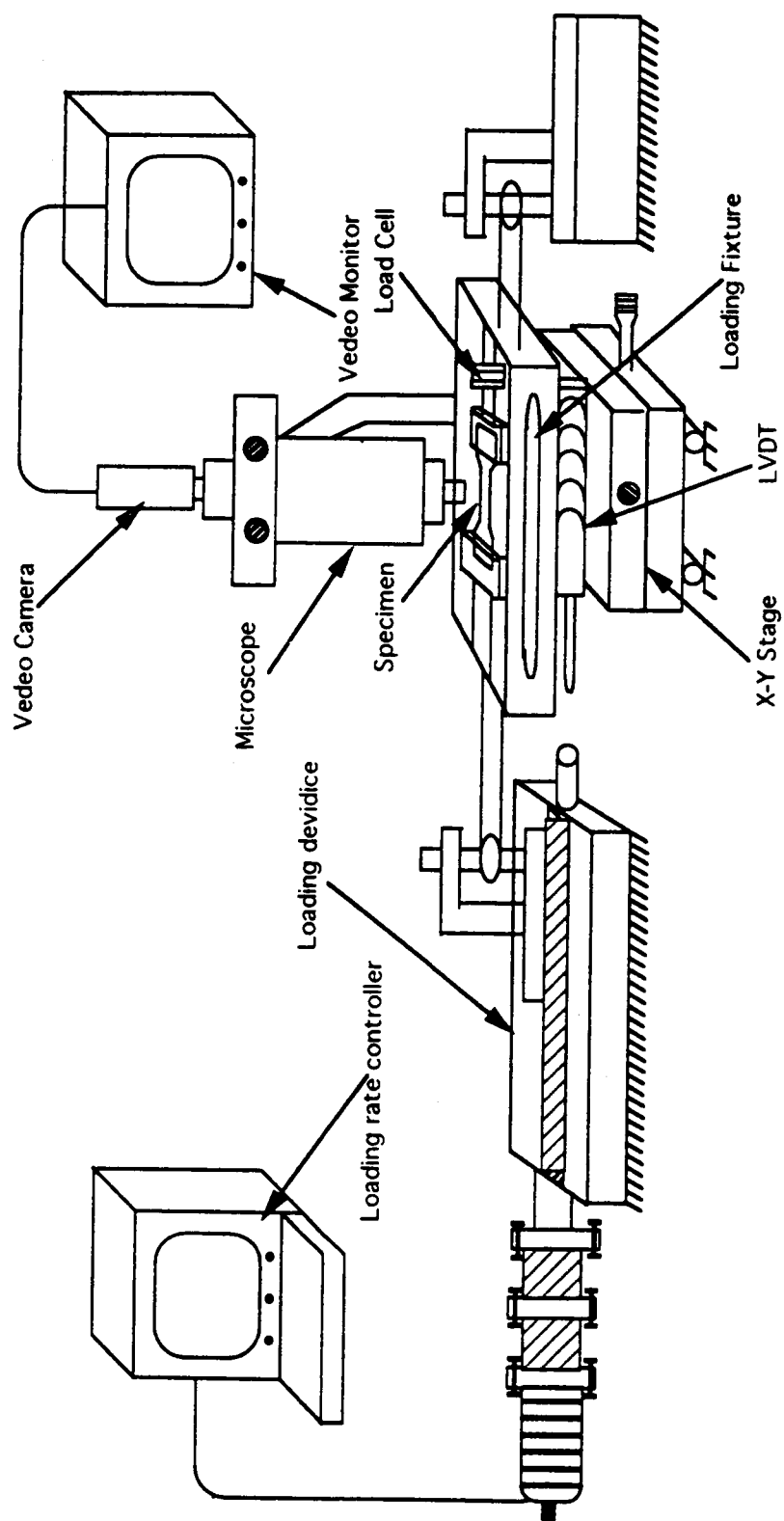
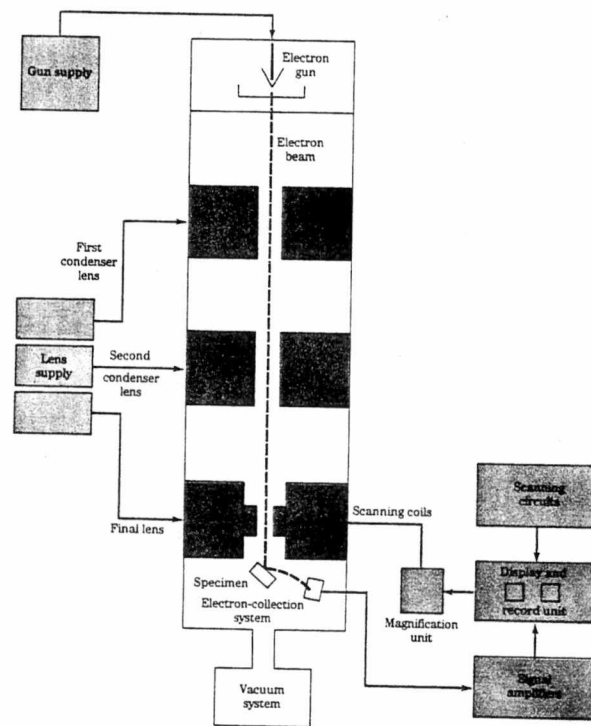


Figure 7. Schematic of fragmentation test experimental set-up.

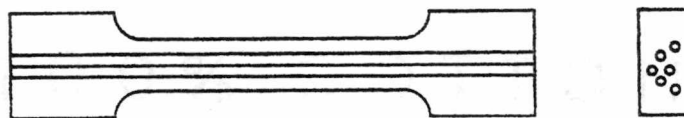
each specimen, video and still pictures of the fringe patterns around various fractures were taken and analyzed.

Finally, an analysis using the scanning electron microscope (SEM) was conducted in this study (see Figure 8a). Multi-fiber specimens were used for this part. These specimens were similar to those used in the fragmentation test except they contain more than one carbon fiber. Multiple fibers made it easier to locate a fiber under the microscope (see Figure 8b).

After curing, using the same process described earlier, the surface perpendicular to the fiber direction was polished. The polishing procedure included grinding on silicon carbide paper and emery paper, coarse polishing using diamond paste on a nylon cloth, and fine polishing using a 0.01 micron aluminum oxide slurry on a Microcloth polishing paper. These specimens were then aged at 100°C for periods of 0, 200, 500, 1000, 1500, and 2000 hours in the Blue M circulating air oven. The polished surfaces were examined in the SEM with particular attention paid to the fiber/matrix interface area. Weight loss was also monitored for these specimens.



a) Schematic of the basic parts of an SEM.
(From Reference 26)



b) Multi-fiber coupon used for SEM examination.

Figure 8. SEM Examination

CHAPTER 3

RESULTS AND DISCUSSION

As the specimens were aged, they darkened in color and became slightly embrittled. However, they were still transparent enough for the fragmentation test. Due to the fiber pretension, the decreased ductility did not prevent the fiber fragmentation process from reaching saturation. Weighing the specimens revealed no significant weight loss during the aging.

The TMA temperature/displacement plots for specimens at each aging period are shown in Figures 9-12. The glass transition temperature (T_g) for the unaged specimen was determined to be 135°C. As shown by the plots, the isothermal aging did not change this temperature, which remained at 135°C in all cases. Increased brittleness is usually associated with increased cross-linking and curing which means an increase in T_g . However, the constant T_g showed that the specimens were fully cured. Therefore, the increased brittleness was not due to increased curing but was due to aging-induced degradation of the matrix.

Table 1 shows the critical length data for the tested specimens. This data is plotted in Figure 13 where the solid line is the average of all the specimens. Notice that the critical length increased significantly between 0 and 500 hours. This increase signifies that the interfacial shear strength decreased and therefore, indicates the degradation of the interface as a whole. This was an important result because it demonstrated that isothermal aging has a significant effect on the interface.

Now notice that the critical length peaked at 500 hours and then decreased at 1000 hours and 2000 hours. This would seem to indicate that the

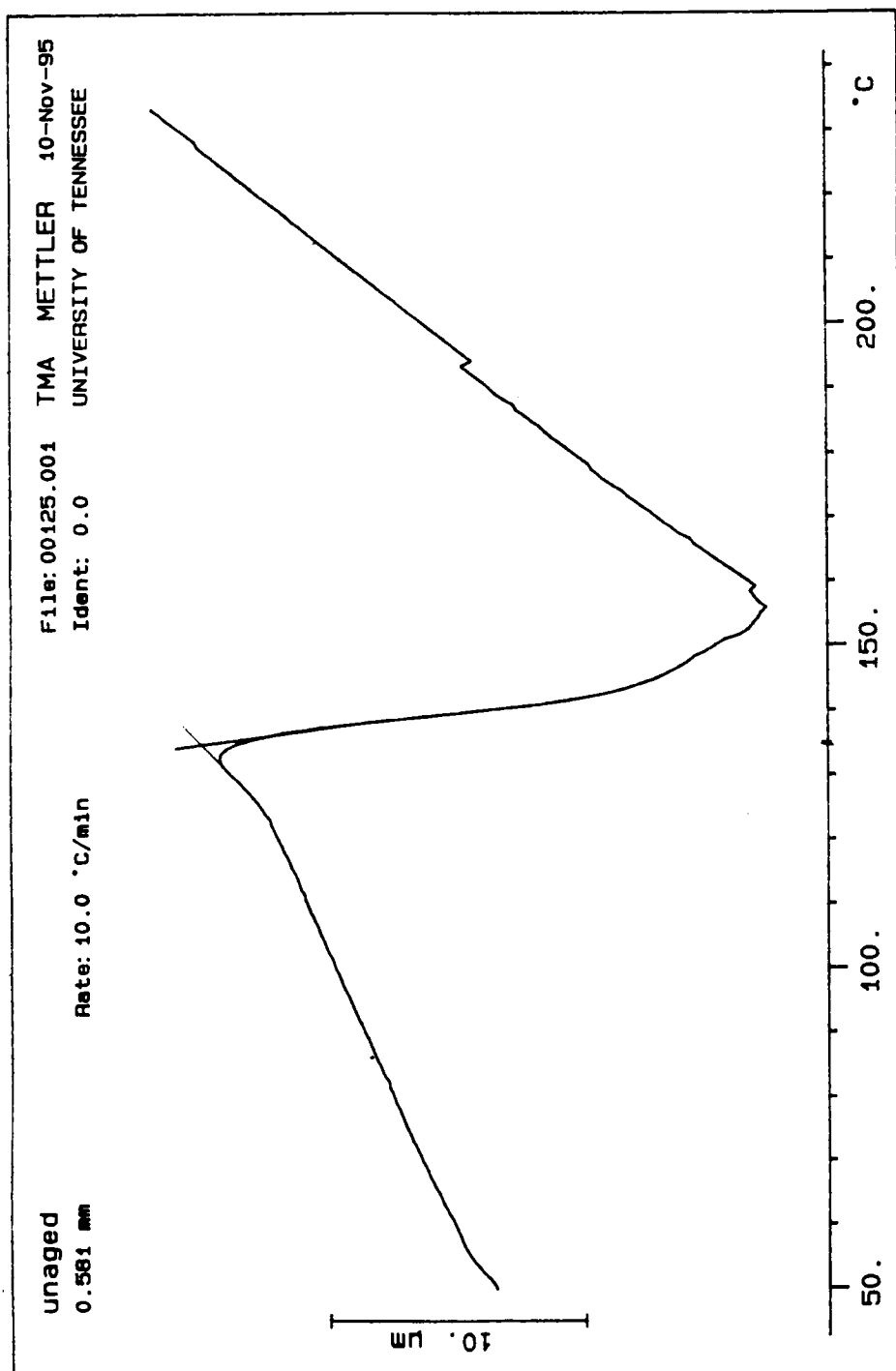


Figure 9. Temperature/displacement plot for unaged specimen.

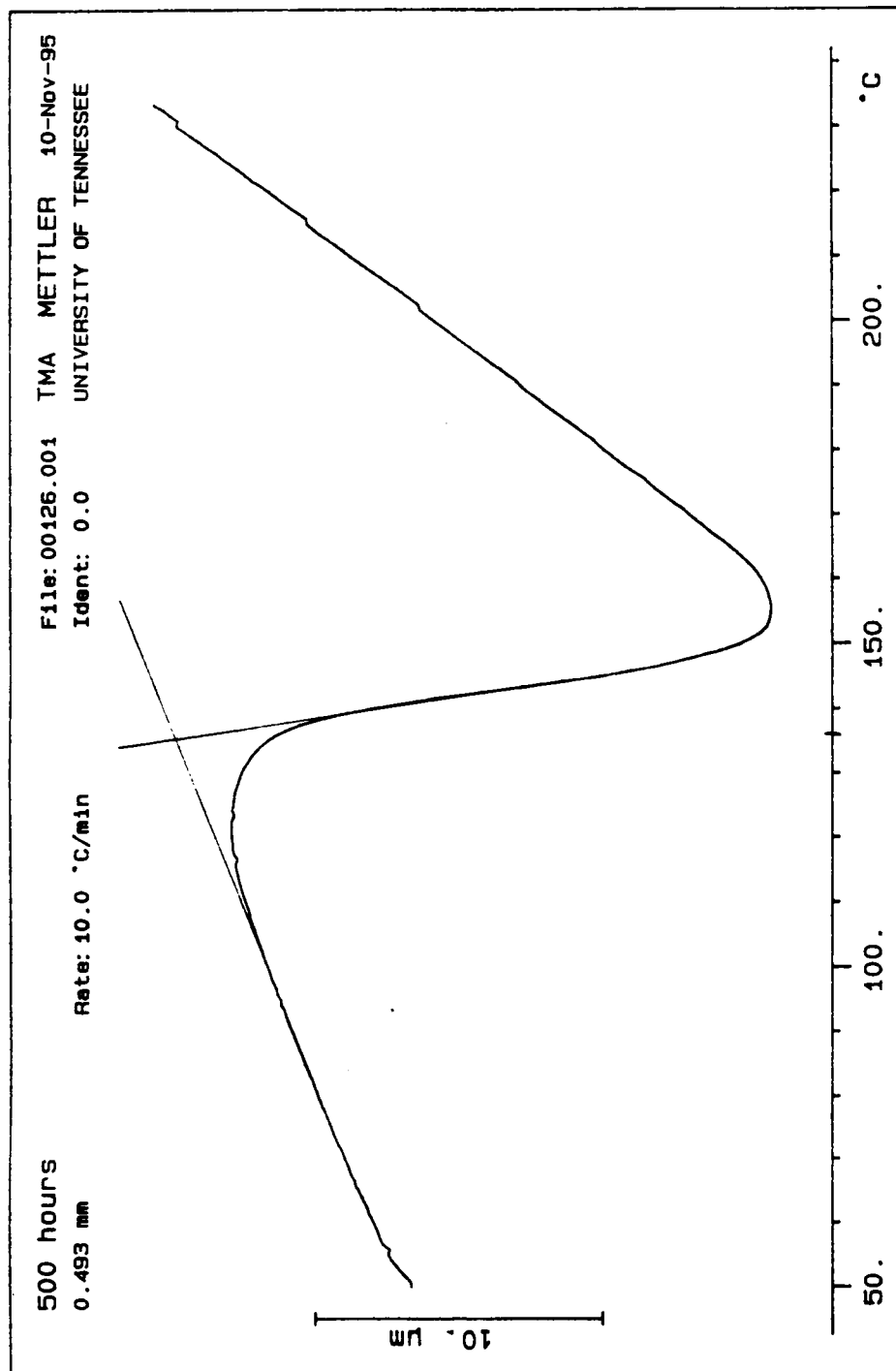


Figure 10. Temperature/displacement plot for specimen aged for 500 hours.

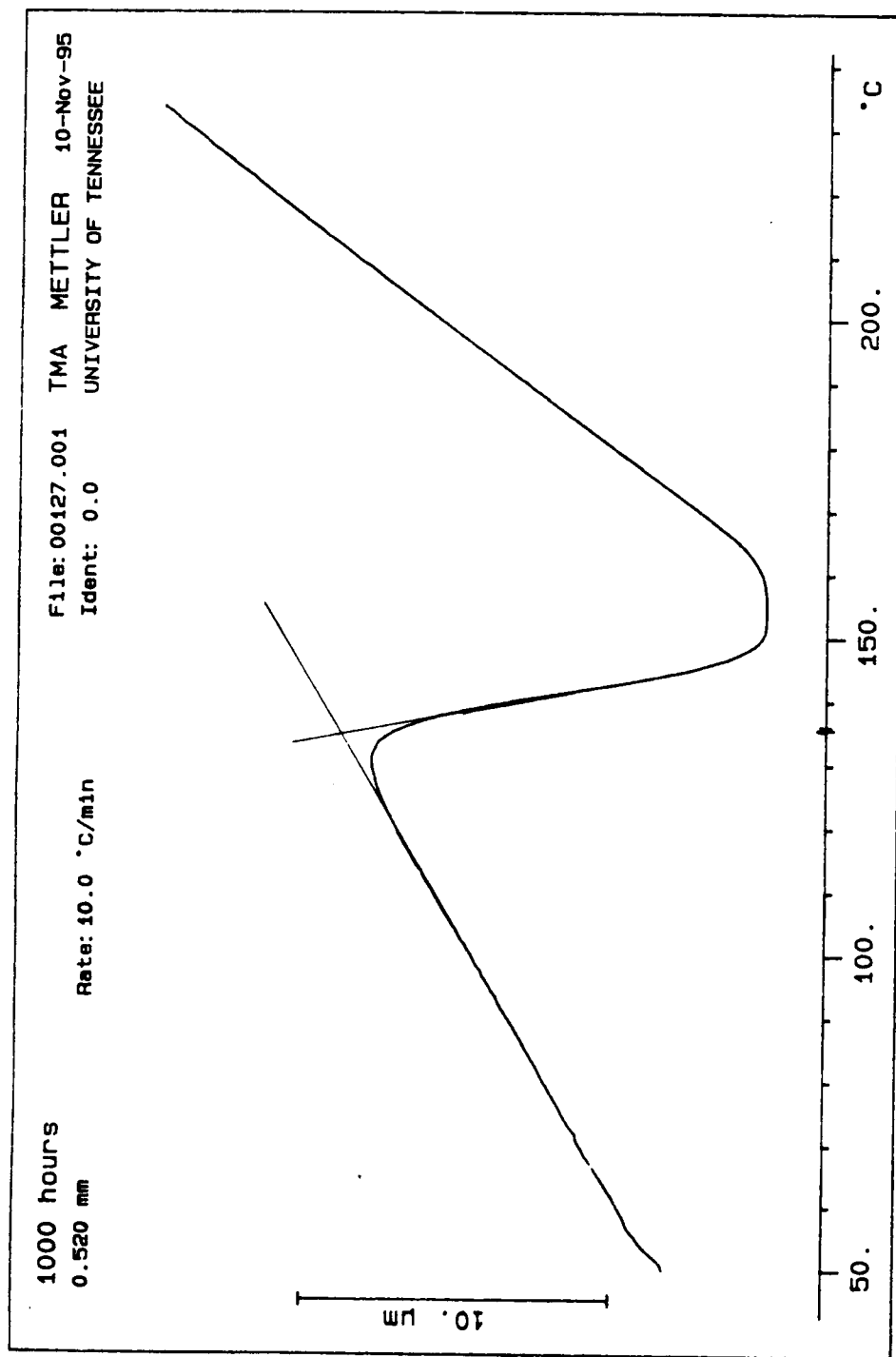


Figure 11. Temperature/displacement plot for specimen aged for 1000 hours.

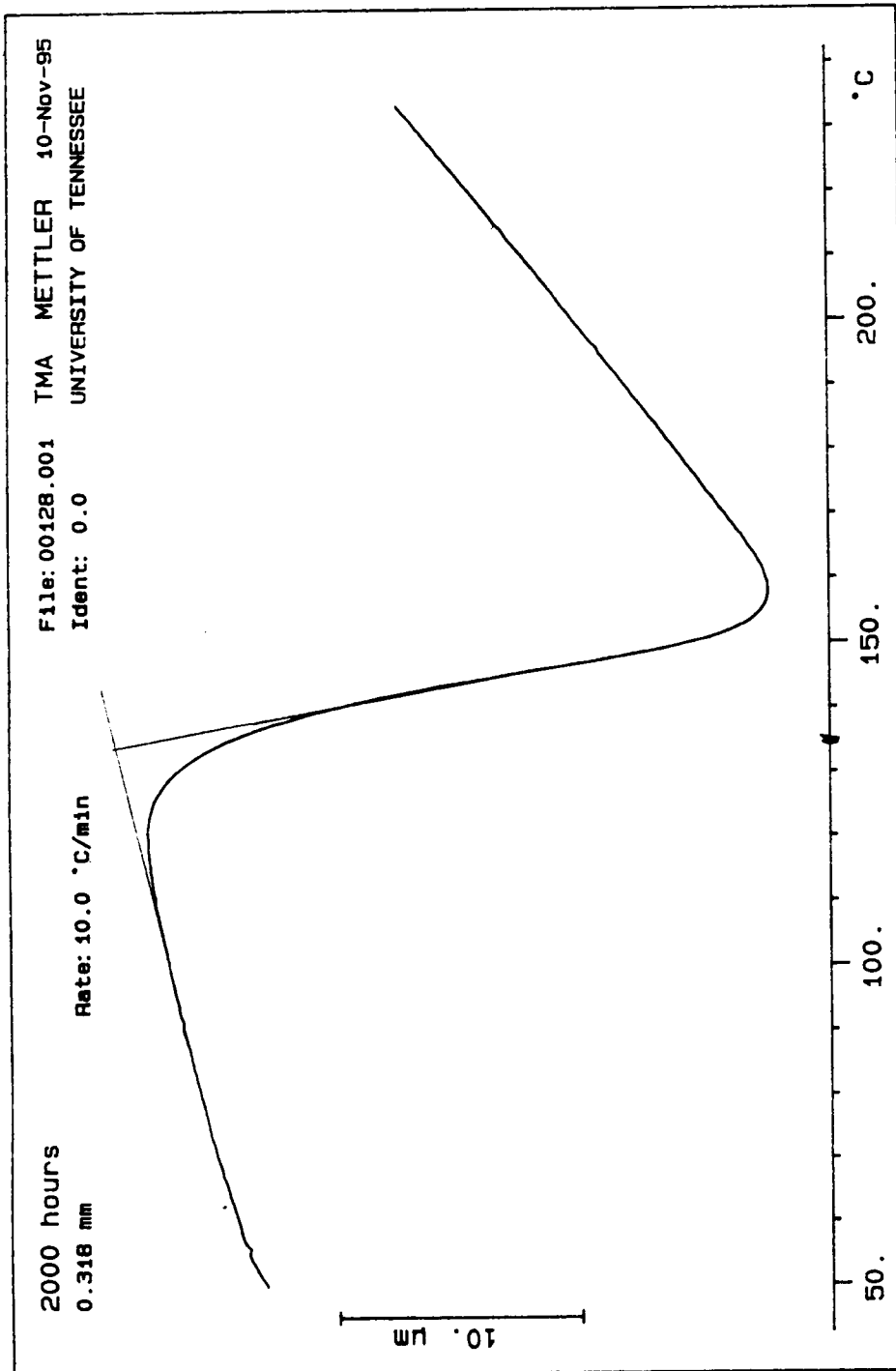


Figure 12. Temperature/displacement plot for specimen aged for 2000 hours.

Table 1. Critical Length Data

Aging Time (hr)	Critical Length (micron)	Minimum Length (micron)	Maximum Length (micron)
0	341.12	113.68	686.86
0	347.58	176.69	627.54
500	466.69	194.92	866.53
500	456.38	183.9	677.97
500	473.4	278.39	763.98
1000	442.24	229.24	866.53
1000	408.38	157.2	788.98
1000	461.31	194.92	777.97
2000	387.02	177.54	700.85
2000	429.39	191.53	758.9
2000	423.0	191.53	822.46

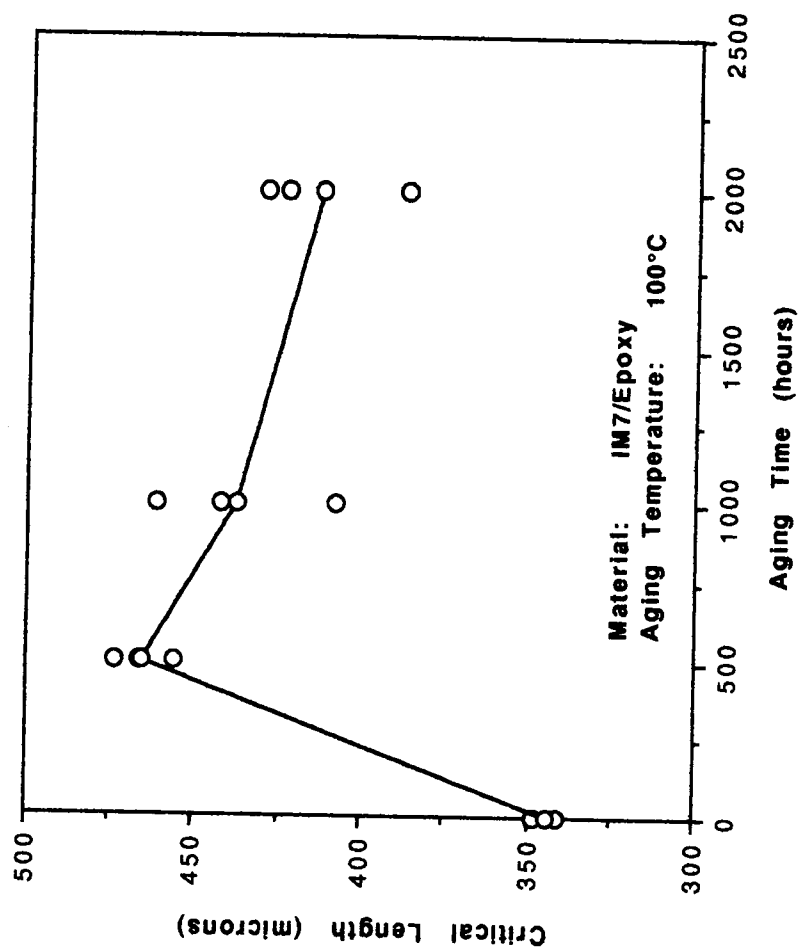


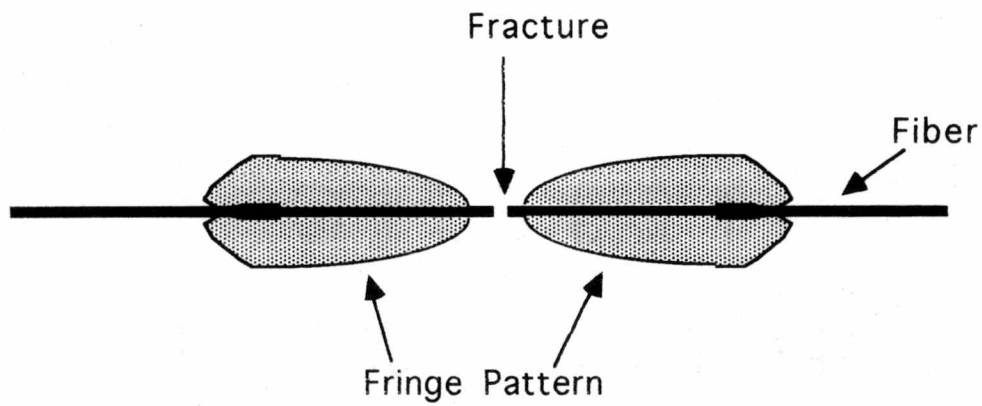
Figure 13. Plot of critical length vs. aging time.

interfacial shear strength increased and that the interface had improved. However, that is not a very logical assumption. Why would the interface suddenly start improving after it had initially undergone degradation? There was really no reason why the interface should start improving. Instead, the possible explanation for this critical length increase might be due to carbon fiber degradation. Carbon fibers can undergo property degradation when subjected to elevated temperatures (27). The degradation of the interphase during the first 500 hours may have provided a convenient path for the hot air to reach the embedded fiber and degrade the fiber's properties. Thus, the fiber would fracture more easily into smaller pieces. This would interfere with the test because the critical length is affected not only by the interface properties but also by the variability of the fiber condition. In order to verify this hypothesis, samples of carbon fibers are currently being examined for degradation after being aged for different lengths of time. Complete data on the critical lengths of each specimen is included in Appendix A.

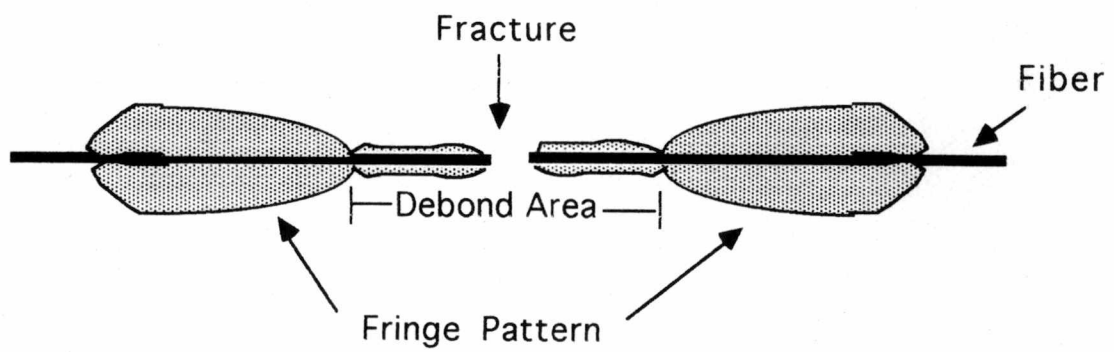
As a specimen was loaded, the fiber was continuously scanned until the first fracture occurred. This first fracture and its ensuing fringe pattern were photographed several times as the load was increased. This was done in order to document any changes in the fringe pattern from the time the fracture occurred until saturation.

A fringe pattern consists of a "bulb" on either side of the fracture (See Figure 14). As mentioned earlier, this bulb is due to the shearing stresses caused by the fiber as it tries to rebound to its unstressed position. When the interface is strong, the fiber is prevented from retracting.

For any given specimen, the shearing stresses around a fracture will increase as the load is increased. If the shearing stresses become greater than the interfacial strength, the fiber and the matrix will debond locally and the



a) Little or no debonding and slippage.



b) Significant debonding and slippage.

Figure 14. Schematic of the birefringence pattern around a fiber fracture.

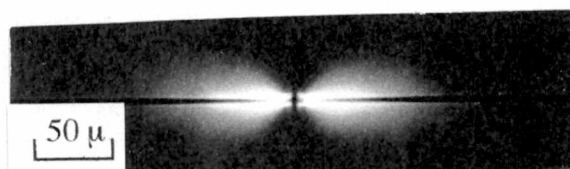
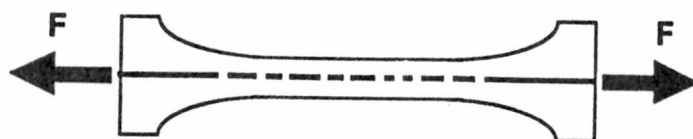
fiber will slip. However, the fiber does not debond along the entire length of the fragment. At some point, there will still be an interfacial bond between the fiber and the matrix. At this point, the bulb part of the fringe pattern will still be present indicating shear stresses. Therefore, the fringe pattern has shifted.

Along the debond area, frictional forces between the fiber and the matrix will show up as a bright line or sheath along the interface. By looking at the location of the bulb part of the fringe pattern, it is possible to tell how much debonding and slippage, if any, occurs during the fragmentation test. The weaker the interface becomes, the more slippage and debonding occurs.

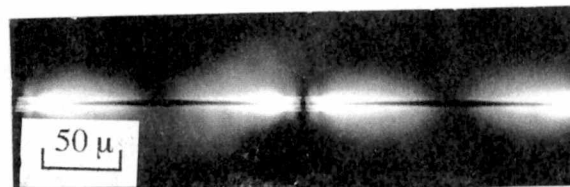
Figure 15 shows the first crack that formed in an unaged specimen. The magnification is 200X. The fringe pattern formed by shearing stresses along the interface can be plainly seen. The pattern may have shifted a little at the higher load indicating a small amount of slippage, but the amount was considered insignificant.

Figure 16 shows the first crack in a specimen aged for 500 hours. The picture on the left side is magnified at 200X while the right side is an increased magnification of 500X. The fringe pattern when the fracture first occurred was the same as the unaged specimen's pattern. At the maximum load, however, the shift in the fringe pattern was a little larger. Debonding and fiber slippage had increased, suggesting the weakening of the interface during the aging.

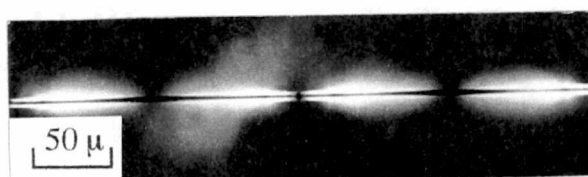
The specimen aged for 1000 hours is shown in Figure 17. The first crack appeared at a higher load this time due to matrix embrittlement. It is similar to the first crack of the unaged specimen except for the fact that the space between the fiber fragments now appeared larger. As the load was increased to the maximum, the fringe pattern shifted much more than the



a) 57 lbs

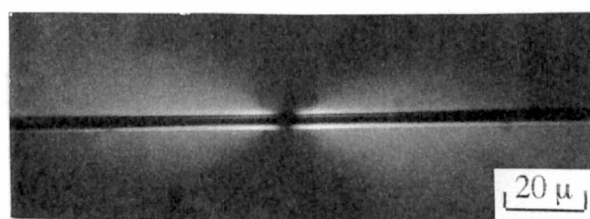
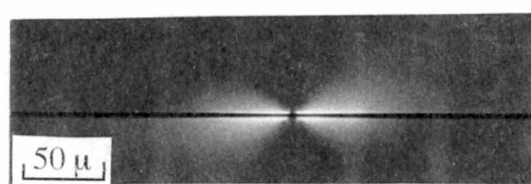
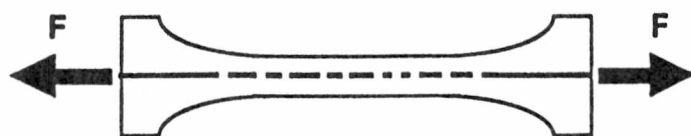


b) 76 lbs

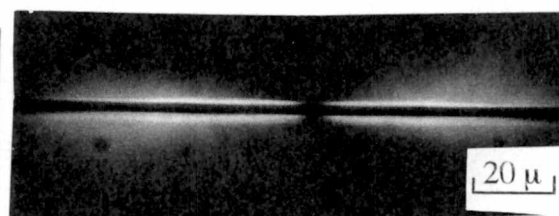
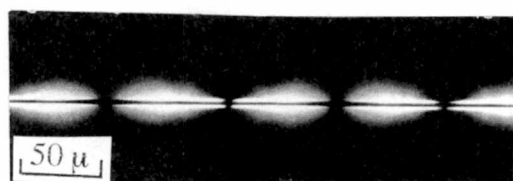


c) 100 lbs

Figure 15. Birefringence pattern of first fiber fracture formed in unaged specimen.

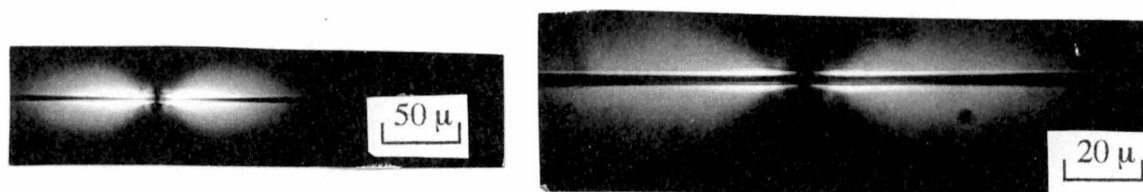
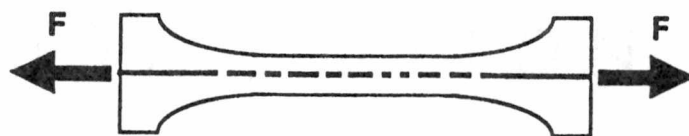


a) 57 lbs

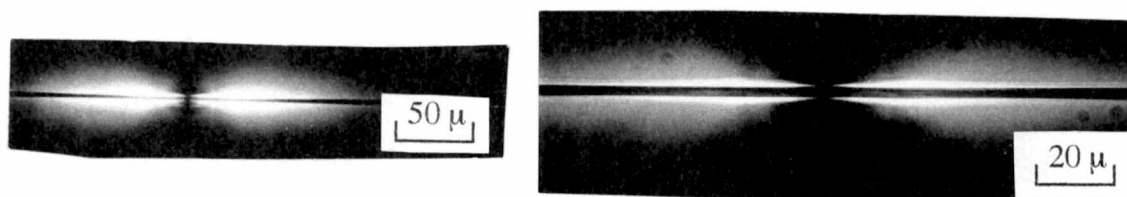


b) 110 lbs

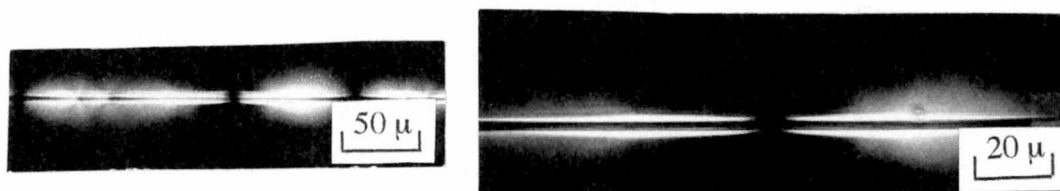
Figure 16. Birefringence pattern of first fiber fracture formed in specimen aged for 500 hours .



a) 75 lbs



b) 100 lbs



c) 120 lbs

Figure 17. Birefringence pattern of first fiber fracture formed in specimen aged for 1000 hours.

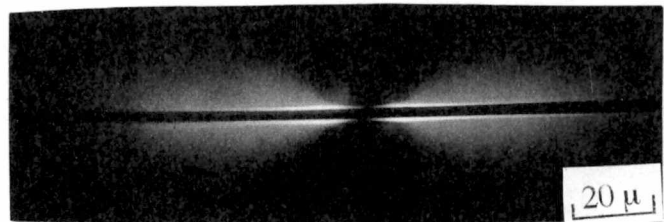
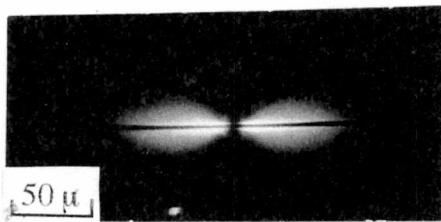
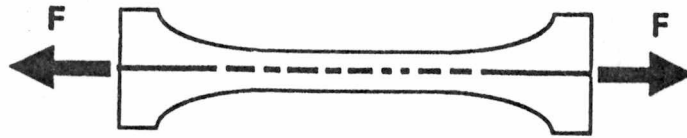
shifts for shorter aging times. Debonding and fiber slippage was much greater and the interface had degraded further.

This degradation process continued in the specimen aged for 2000 hours and is shown in Figure 18. Once again, the spacing between the fiber fragments had become larger, and the shift in the fringe pattern indicated increasing debonding and slippage.

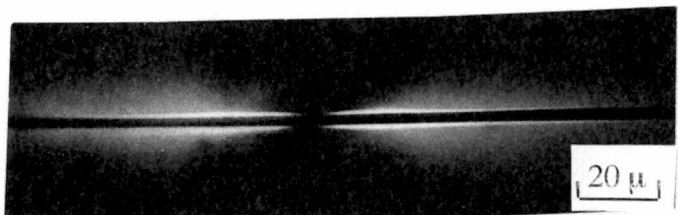
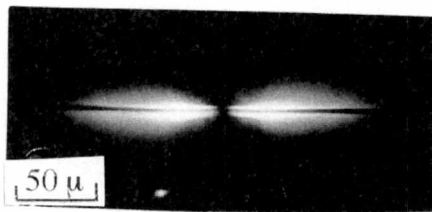
Observation of fiber fractures formed at higher load levels demonstrated this degradation effect also. When fractures occur at high loads, the initial shear stresses can be greater than the interfacial bond strength, and the fiber will debond and slip almost immediately. The amount of debonding and slippage depends on the strength of the interface. Figure 19 shows fractures pictured immediately after they were formed for each of the different aging times. All these fractures occurred at load levels close to the maximum loads. For the unaged specimen, a small amount of debonding and slippage was observed by the slight shift in the fringe pattern. The figure shows that this debonding and slippage increased as the aging times increased to 500, 1000, and 2000 hours. Therefore, once again, the increased debonding and slippage indicated the degradation of the interface by isothermal aging.

How is it known that the observed pattern shifts were due to debonding and slippage and not some other mechanism?

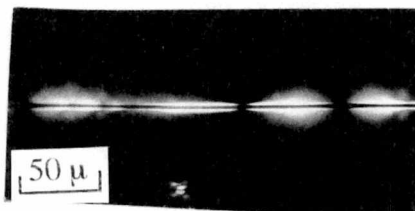
Figure 20 shows one of the many fractures observed on an unaged specimen. Because this specimen had a strong interface, this was the only fracture that had significant slippage. This fracture was photographed while the specimen was being unloaded. As the specimen was unloaded, the shear stresses began relaxing and the fringe pattern faded slightly in intensity. As unloading continued, the pattern gradually brightened around the fracture area again. This brightening was due to stresses caused by frictional forces



a) 75 lbs

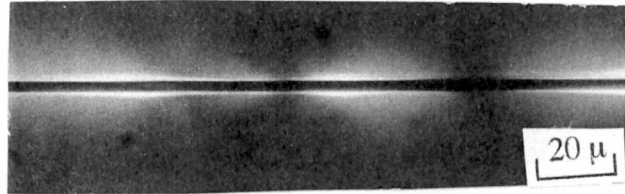
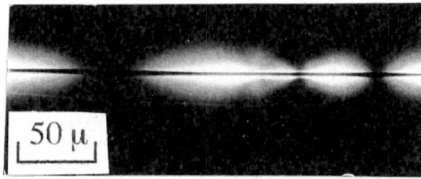
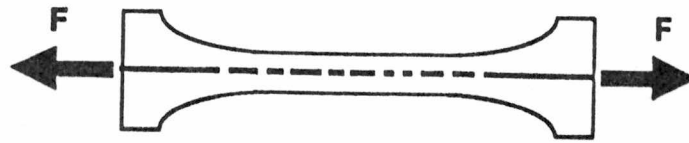


b) 95 lbs

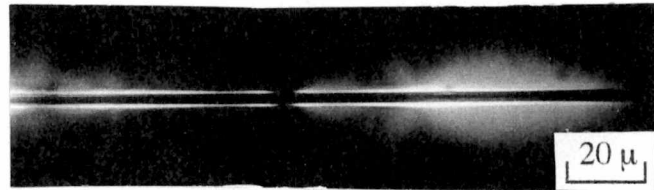
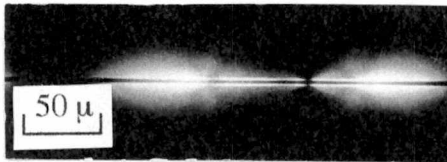


c) 120 lbs

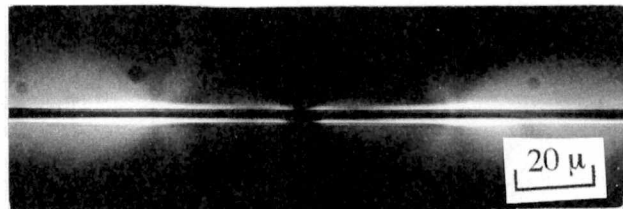
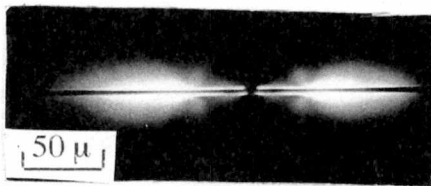
Figure 18. Birefringence pattern of first fiber fracture formed in specimen aged for 2000 hours.



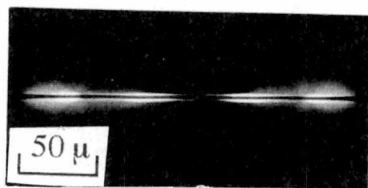
a) Unaged (Saturation Level: 100 lbs)



b) 500 Hours (Saturation Level: 110 lbs)

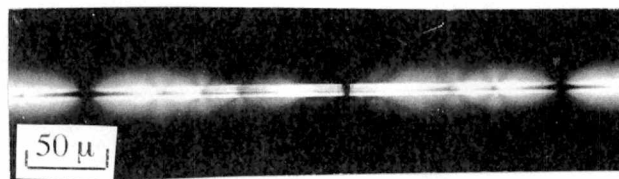


c) 1000 Hours (Saturation Level: 120 lbs)

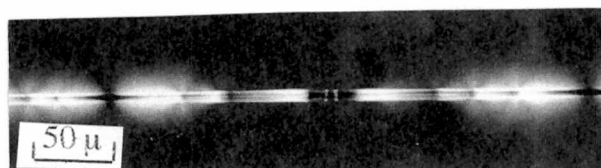


d) 2000 Hours (Saturation Level: 120 lbs)

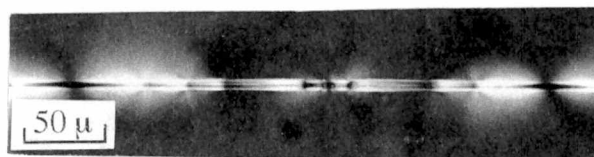
Figure 19. Birefringence patterns of fiber fractures formed at tension levels approximately ten pounds below saturation level.



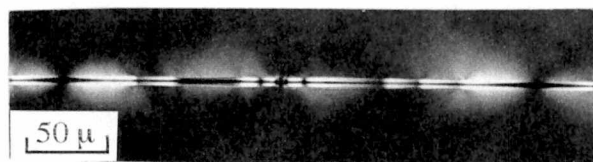
a) 100 lbs



b) 75 lbs



c) 45 lbs



d) 10 lbs

Figure 20. Birefringence pattern of fiber fracture in unaged specimen as it is unloaded.

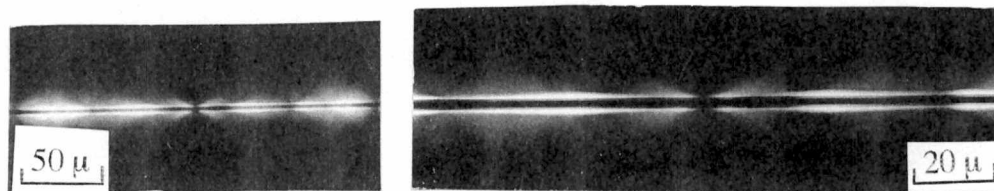
between the now debonded fiber and matrix. The fiber was moving back into its original position and rubbing against the matrix. The 500 hours specimen also showed this phenomenon in Figure 21.

Figure 22 shows the unloading of the 1000 hours specimen. Once again, the gradual fading and then brightening of the fringe pattern around the fracture area was observed. This time, however, the renewed brightness around the fracture area suddenly snapped off as if someone had thrown a switch. The fiber had overcome the frictional forces and had slipped back towards its original position. This same event was observed in the 2000 hours specimen shown in Figure 23.

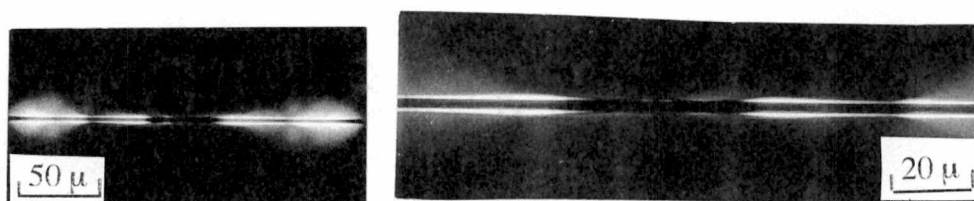
Therefore, as a specimen is unloaded, the fringe pattern gradually fades around the fracture areas as the shearing stresses relax. Then, the buildup of frictional forces along the debonded area cause the pattern to gradually brighten once more as the fiber tries to move to its original position. In specimens where the amount of debonding is great, the fiber overcomes these frictional forces and slips. This sudden slip causes the fringe pattern to dim immediately. Thus, the pattern changes that occur during unloading verify slippage and debonding in the specimen.

The multi-fiber SEM specimens were monitored for weight loss also. As with the single fiber specimens, no significant weight loss occurred (less than 0.01%). The SEM pictures of each specimen are shown in Figures 24 through 29. The aging times were 0, 200, 500, 1000, 1500, and 2000 hours respectively. The "a" part of each figure shows several fibers while the "b" part is a close-up of one or two of the fibers.

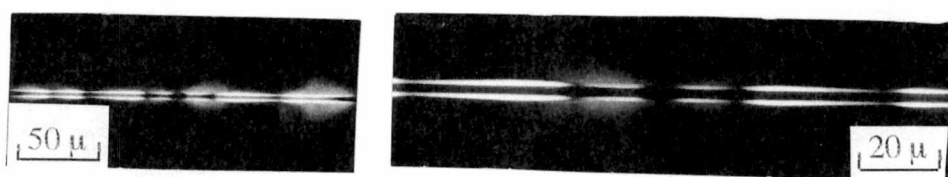
The unaged specimen obviously showed no sign of cracking or other damage. The dark line around the fiber seen in the figure is a shadow due to the fact that the fiber is slightly above the matrix surface.



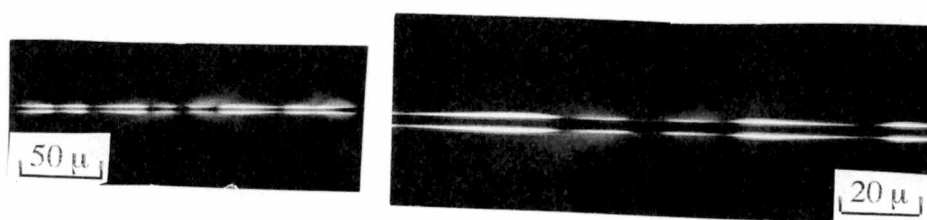
a) 110 lbs



b) 70 lbs

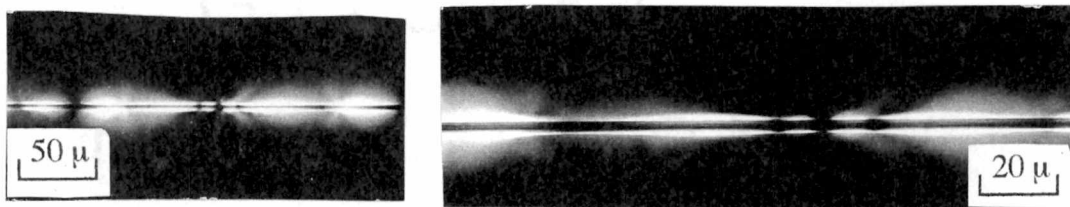


c) 22 lbs

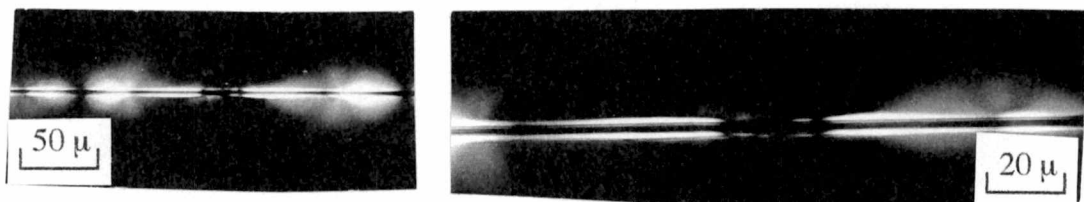


d) 10 lbs

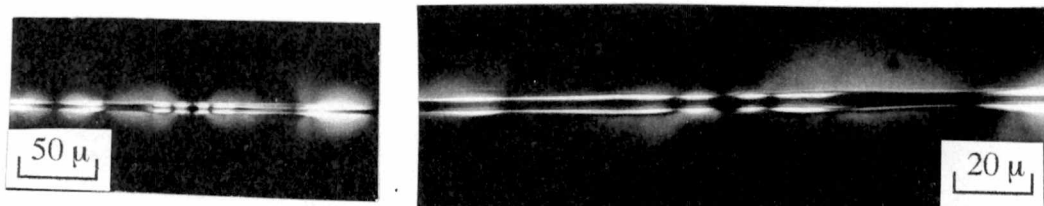
Figure 21. Birefringence pattern of fiber fracture in specimen aged for 500 hours as it is unloaded.



a) 120 lbs



b) 70 lbs

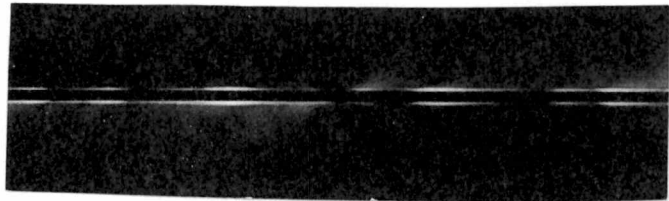
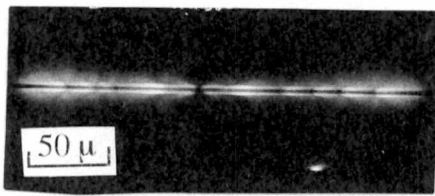


c) 25 lbs

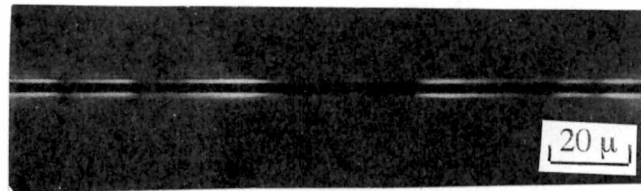
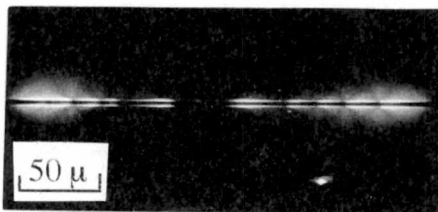


d) 20 lbs

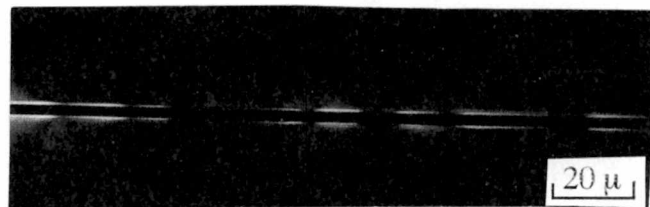
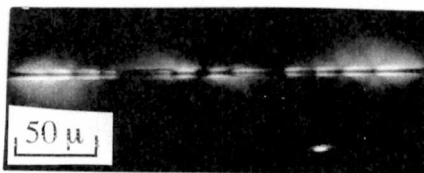
Figure 22. Birefringence pattern of fiber fracture in specimen aged for 1000 hours as it is unloaded.



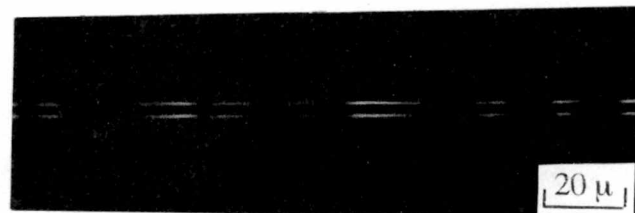
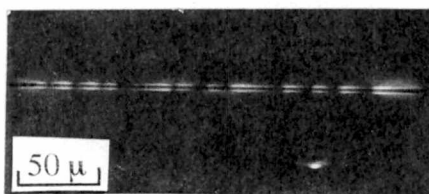
a) 120 lbs



b) 75 lbs

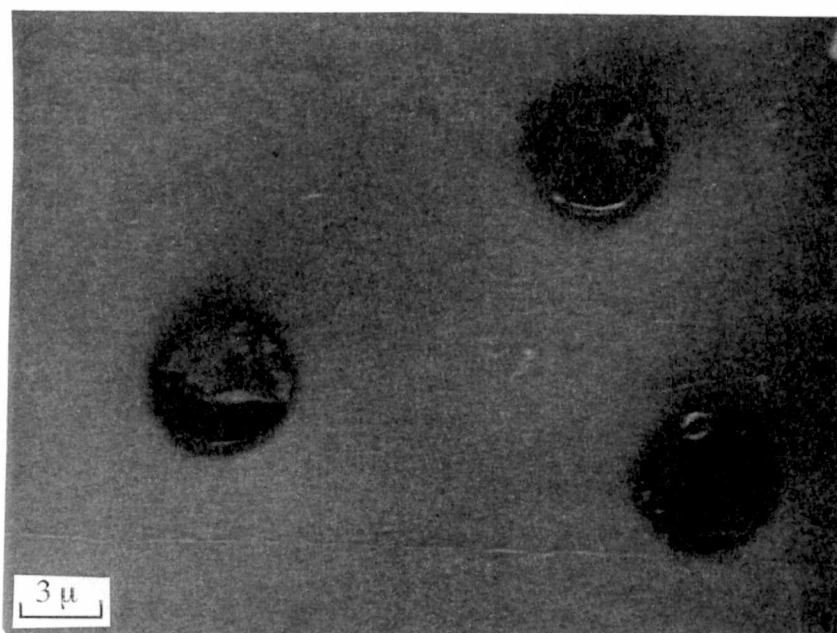


c) 50 lbs

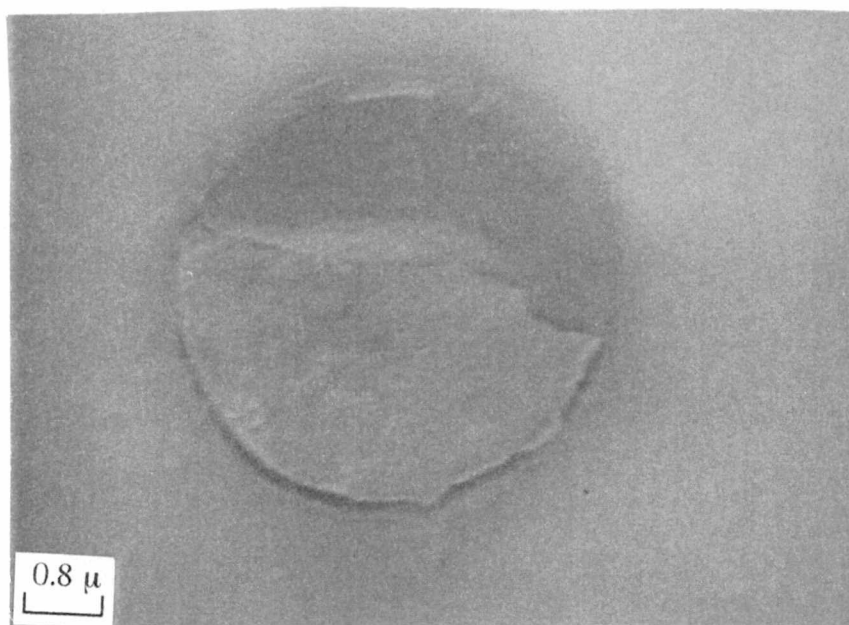


d) 20 lbs

Figure 23. Birefringence pattern of fiber fracture in specimen aged for 2000 hours as it is unloaded.

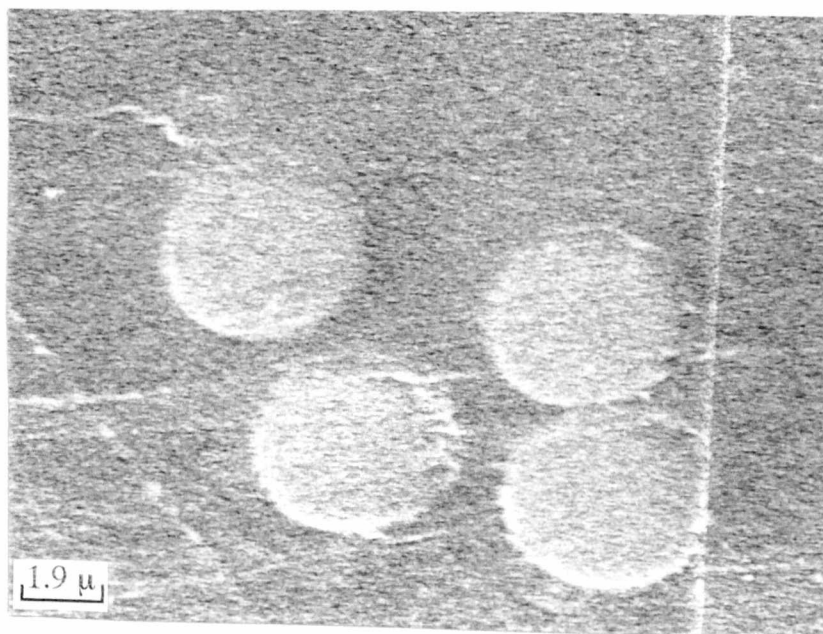


a)



b)

Figure 24. SEM photograph of unaged specimen.

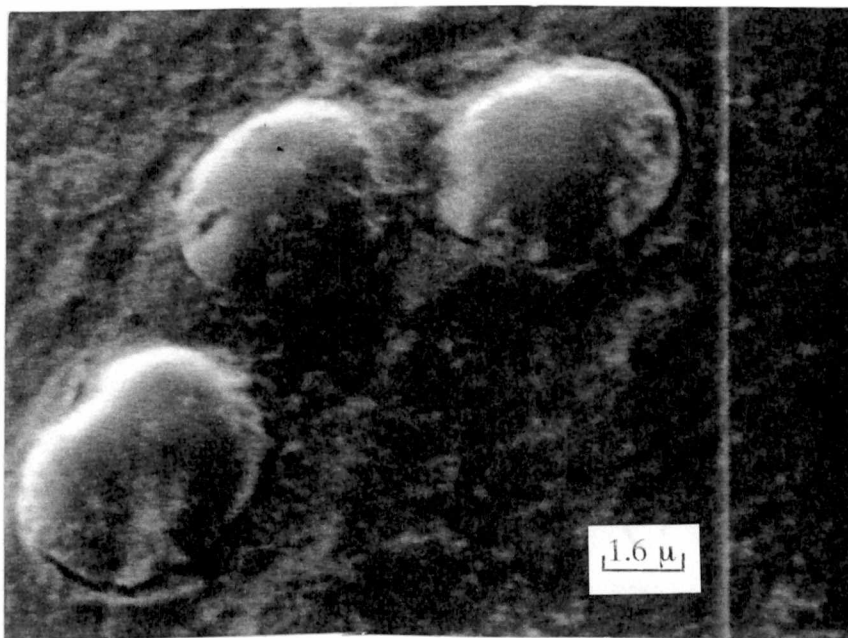


a)

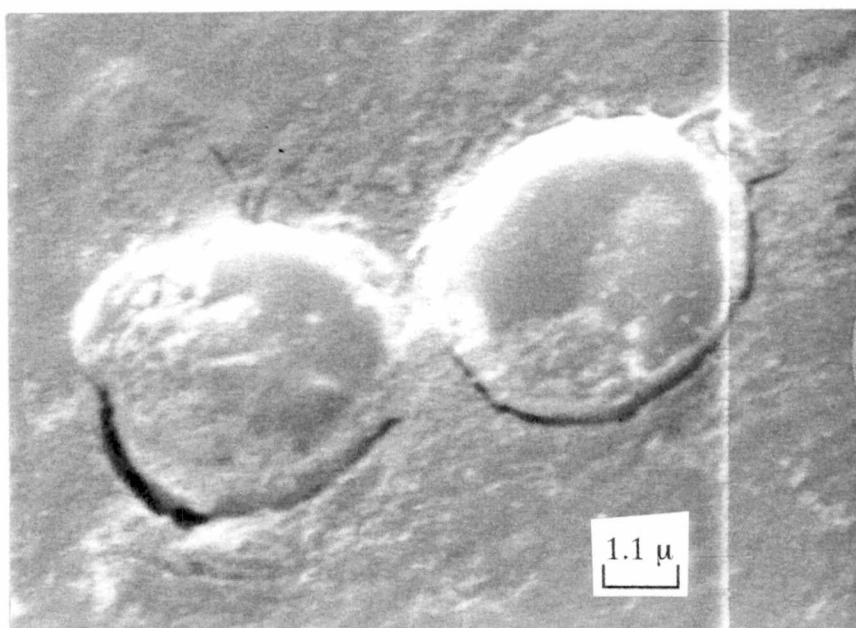


b)

Figure 25. SEM photograph of specimen aged for 200 hours.

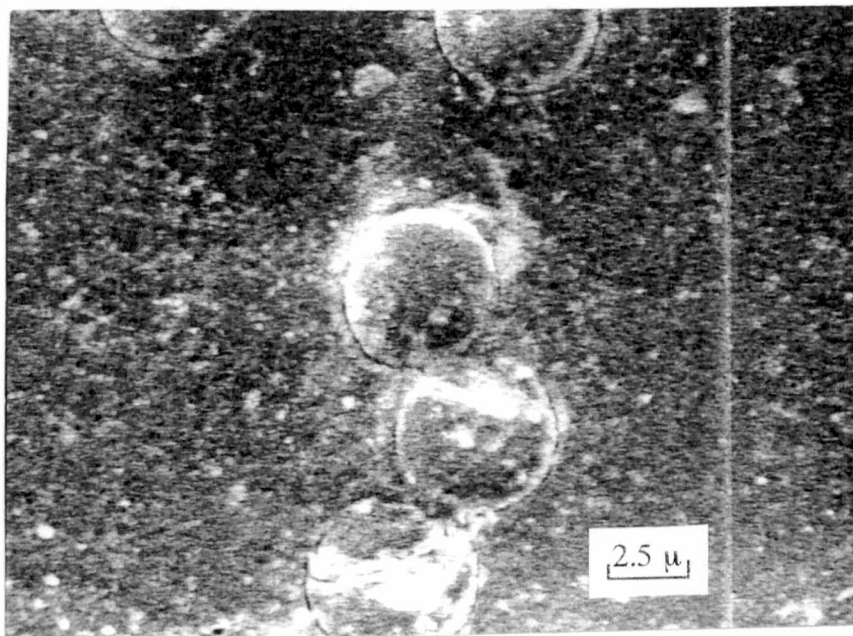


a)

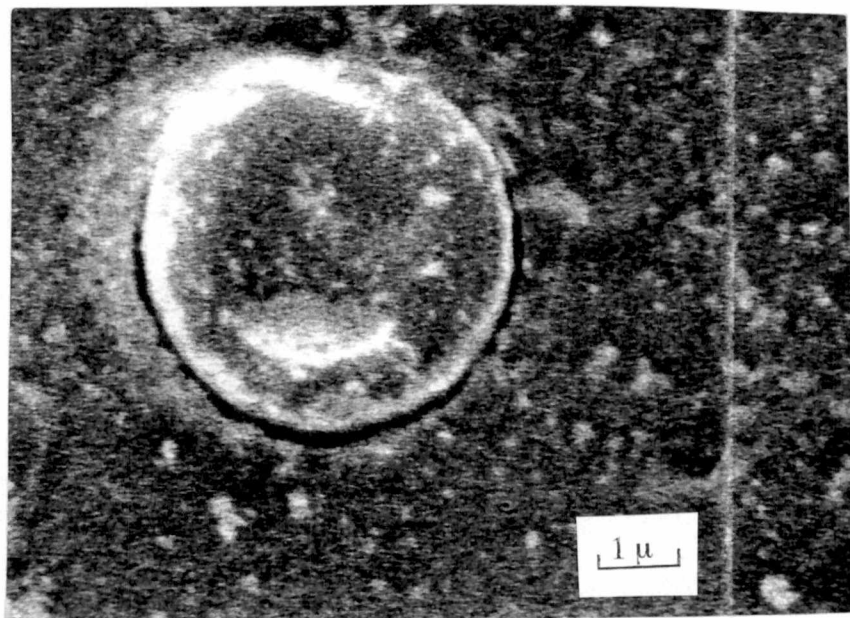


b)

Figure 26. SEM photograph of specimen aged for 500 hours.

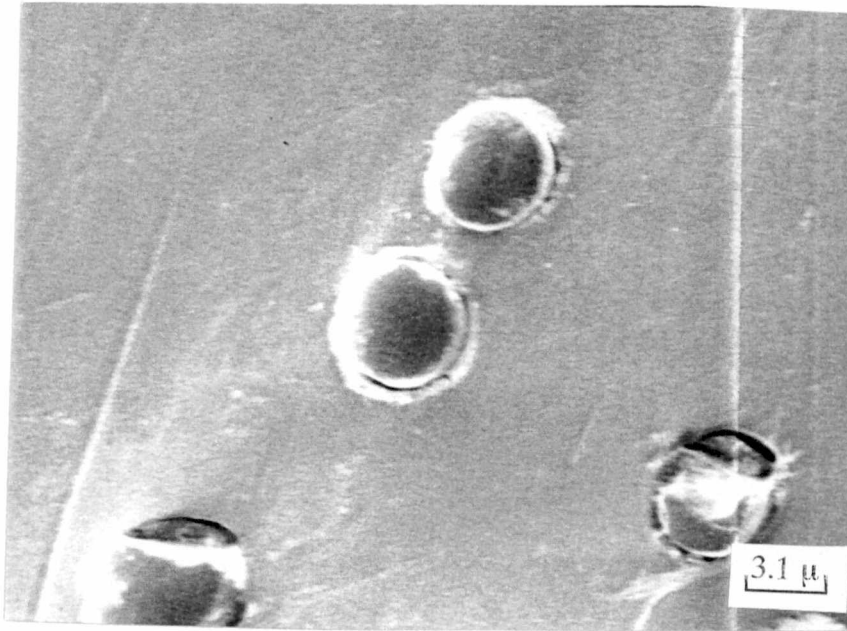


a)

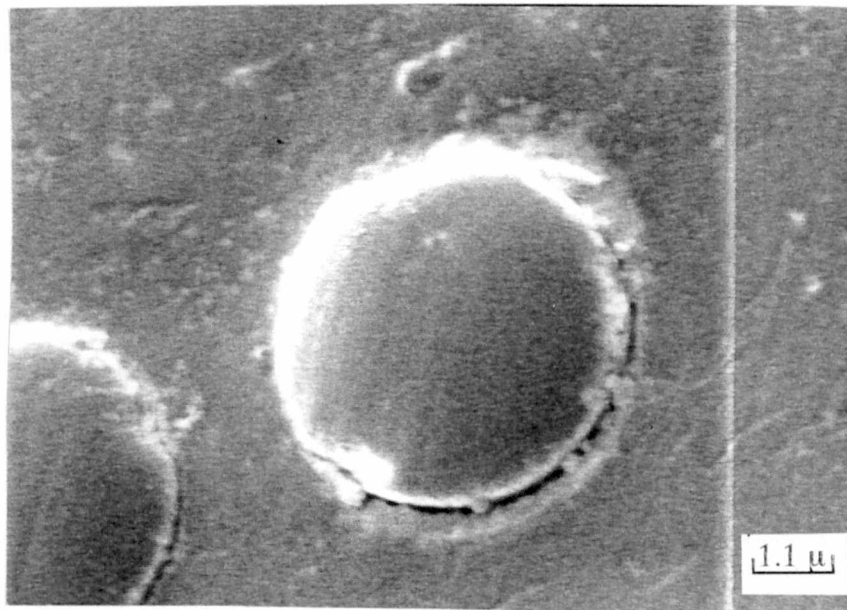


b)

Figure 27. SEM photograph of specimen aged for 1000 hours.

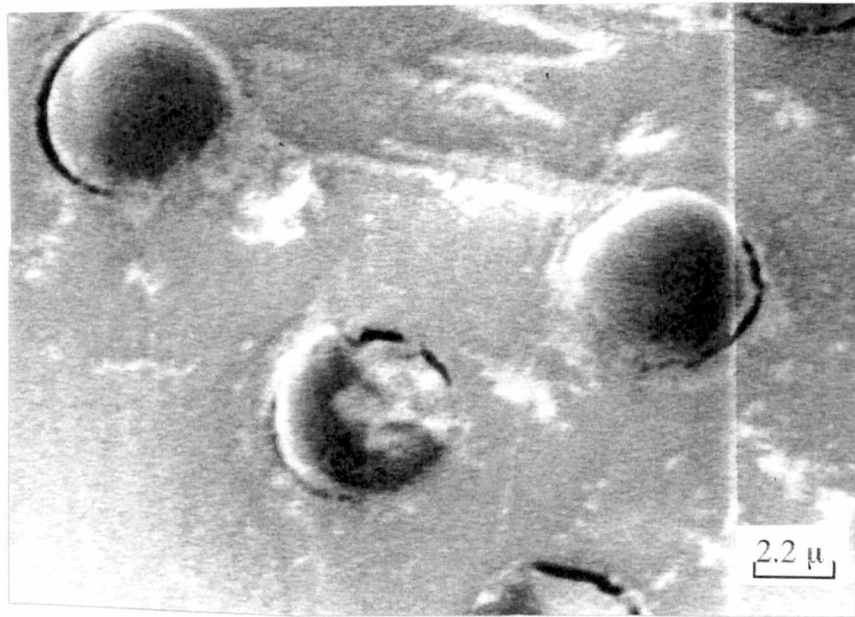


a)

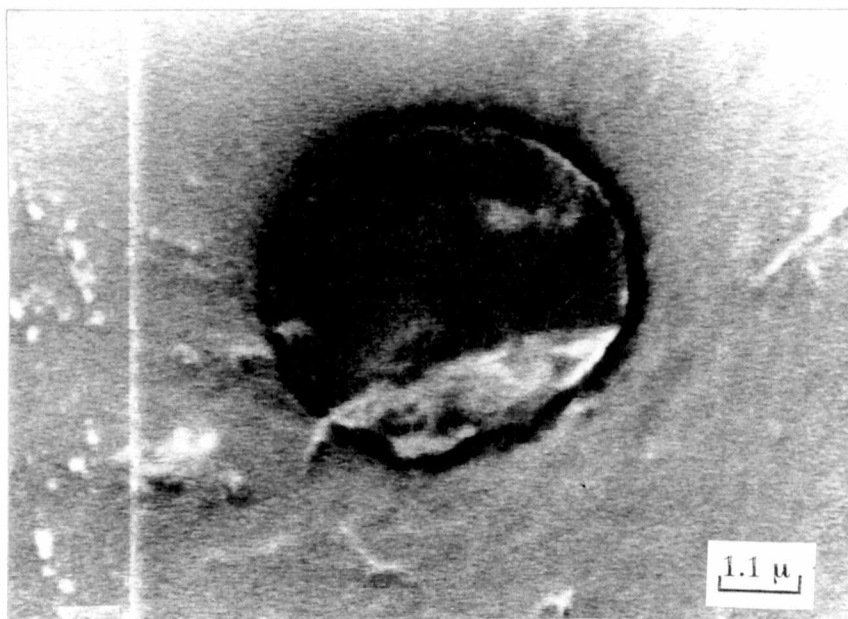


b)

Figure 28. SEM photograph of specimen aged for 1500 hours.



a)



b)

Figure 29. SEM photograph of specimen aged for 2000 hours.

For the specimen aged for 200 hours, some aging damage was observed. Crevices and pits had appeared around the fiber, but no major damage had yet occurred.

However, major damage had occurred in the 500 hours specimen. Cracks had appeared at the interface between the fiber and matrix. Thus, the interface had been subject to significant degradation. This cracking was also observed in the specimens aged for 1000, 1500, and 2000 hours.

Figure 30 shows a fiber in the specimen aged for 2000 hours. This fiber has a crack through its diameter. Not only is the interface being assaulted by isothermal aging, but the fibers are being damaged also.

These pictures correlate with the other data very well and also support the explanation given for the decrease in critical length values at 1000 and 2000 hours of aging time (See Figure 13). The cracks between the fiber and the matrix could expose the carbon fiber to the hot air, which could degrade the fiber's properties through oxidation. The pictures also confirm that the interface continued to degrade at higher aging times despite the critical length decrease observed in the 1000 and 2000 hours specimens.

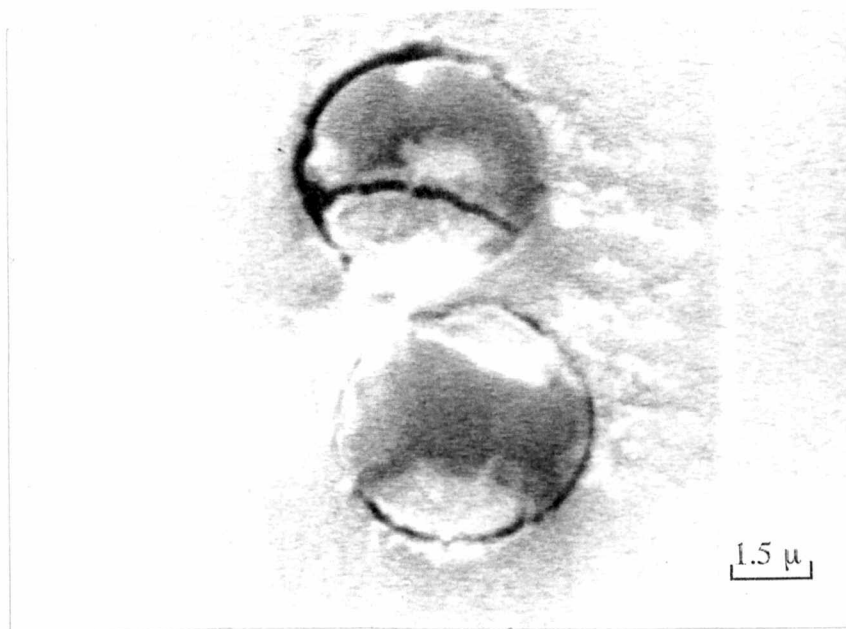


Figure 30. SEM photograph of fiber crack in specimen aged for 2000 hours.

CHAPTER 4

CONCLUSIONS

The major conclusion from this study was that isothermal aging has a significant effect on the interface. The critical length measurements, fringe pattern observation, and SEM examination all indicated interfacial degradation. The increase in matrix brittleness and the fiber crack observed in the 2000 hours specimen in Figure 30 show that the matrix and fiber themselves are also affected. However, the key area is the interface.

Critical length measurements from the single fiber fragmentation test show this degradation up to 500 hours. For longer aging times, it is believed that the degradation of the carbon fiber tainted the critical length results.

Observation of the fringe patterns show that isothermal aging caused fiber/matrix debonding and fiber slippage after 500 hours of aging. Furthermore, this debonding and slippage increased with aging time.

Finally, the SEM examination showed a little damage after 200 hours of aging, but the heavy damage occurred after 500 hours aging. Major cracking around the interface had occurred by this time. This cracking continued up until the maximum aging time of 2000 hours.

This cracking could have exposed the fiber to the hot circulating air which would have degraded the fiber by oxidation. This along with the fracture of the fiber seen in the 2000 hours specimen correlates with the fiber degradation hypothesis.

When designing composites for use in applications which involve exposure to elevated temperatures, the interface must be taken into account. Choice of matrix and fiber will enable a composite to be used in high

temperatures, but if the interface is not sufficiently protected, it will eventually degrade. This, of course, results in the degradation of composite properties as a whole.

Several ideas for future work have been formed.

- The first one is to verify the carbon fiber degradation during aging, which is currently being done.
- The second idea is to determine the depth of the interfacial cracking . In other words, the extent of the aging damage needs to be better quantified.
- Finally, it would be interesting to determine the effect of different fiber surface treatments on the extent of interfacial aging damage. It appears reasonable that the surface treatment that gives the best adhesion would have the best thermo-oxidative stability. Is this true?

As one can see, interfacial research is an important area in the composites field. A lot of work remains to be done in studying interfacial effects.

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APPENDIX

APPENDIX

This appendix contains the fiber critical length data for the specimens aged for 0, 500, 1000, and 2000 hours. Each specimen had many fiber fragments by the time the saturation point was reached. The average of these fiber fragment lengths was the critical length. Each plot shows the length for each of the fiber fragments across the span of the specimen. In the legend to the right of plot, the average, standard deviation, average standard deviation, minimum, maximum, and median of all the fragment lengths are listed.

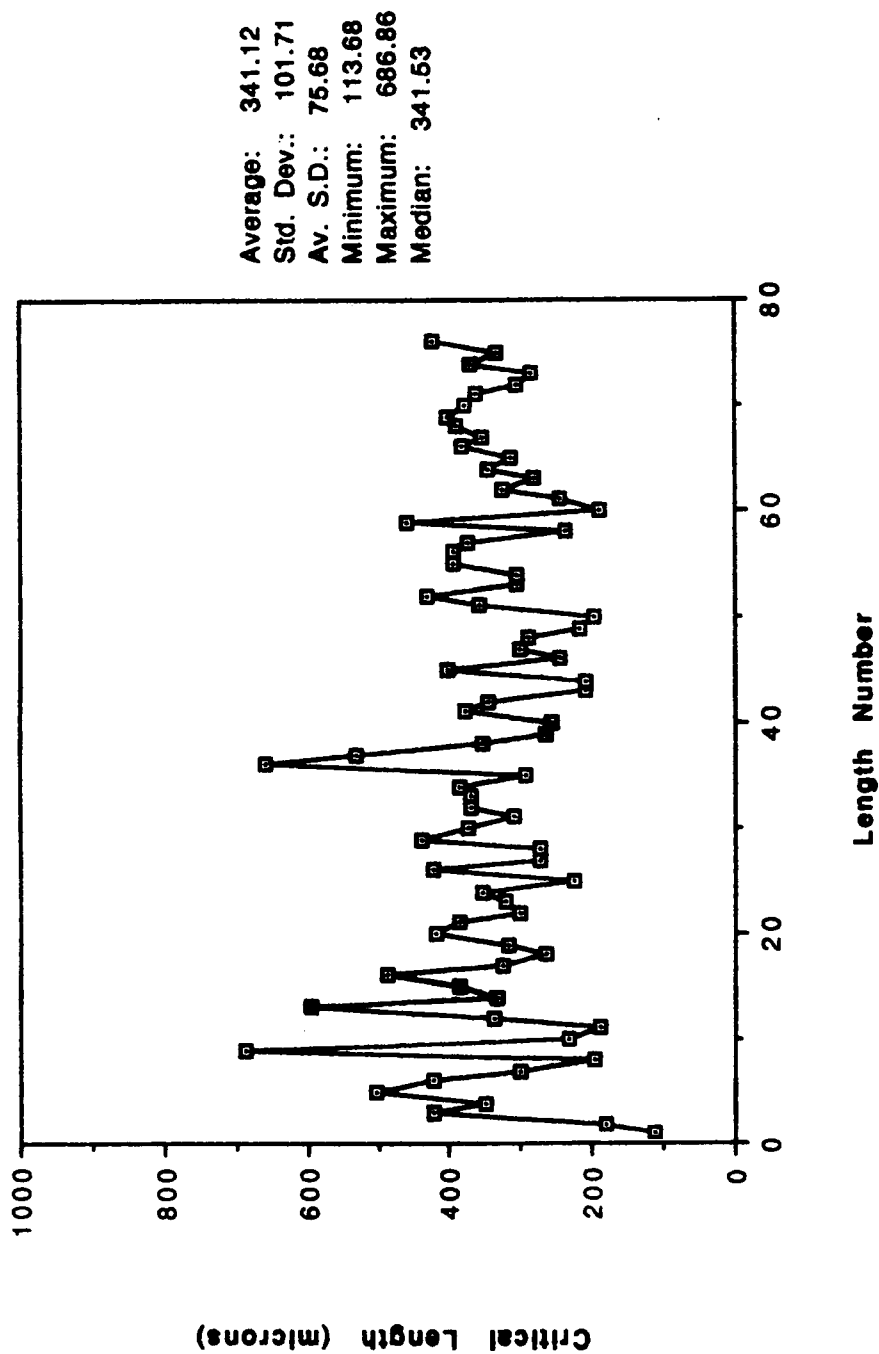


Figure A1. Critical length data for specimen number one, unaged .

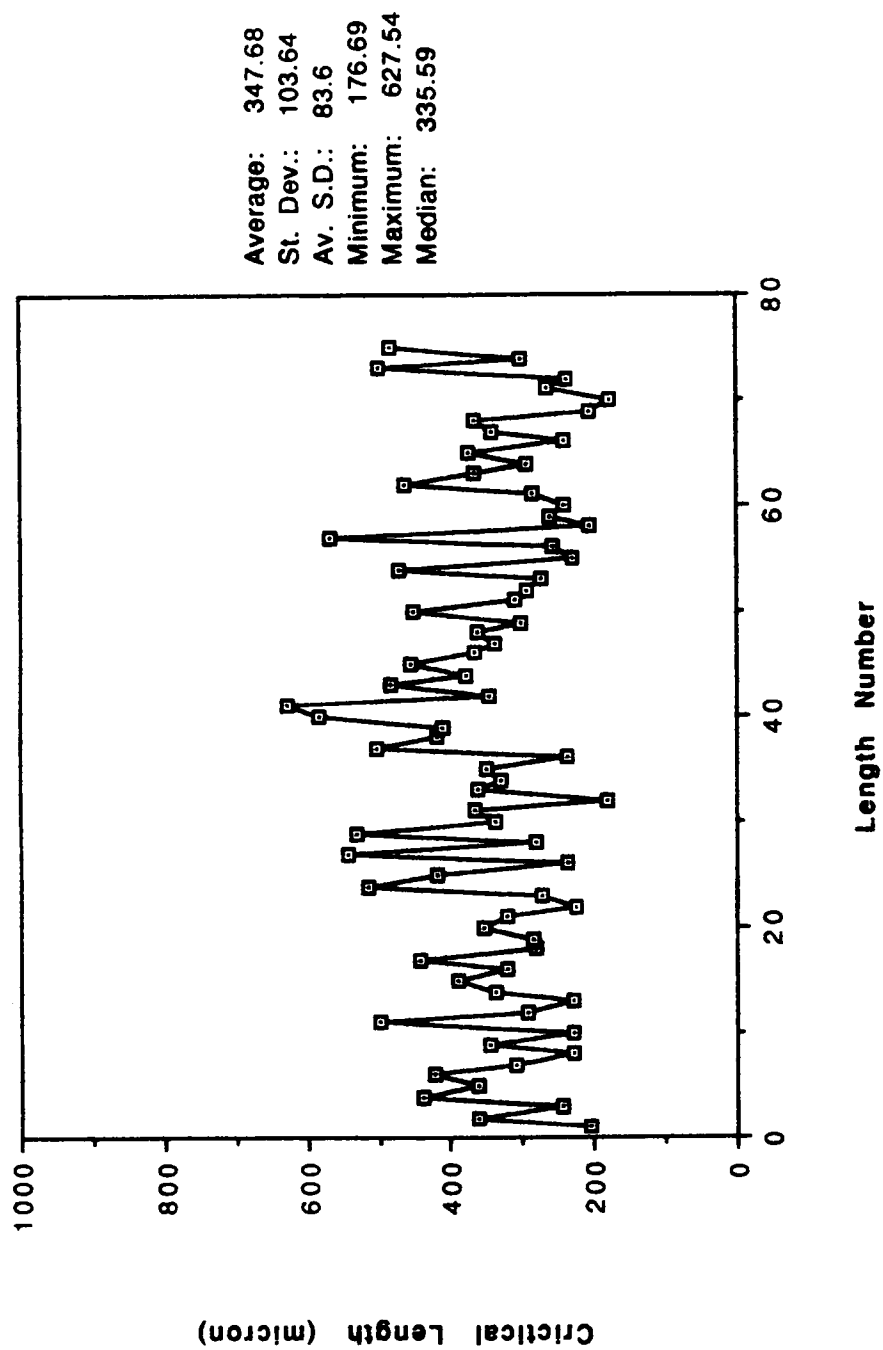


Figure A2. Critical length data for specimen number two, unaged .

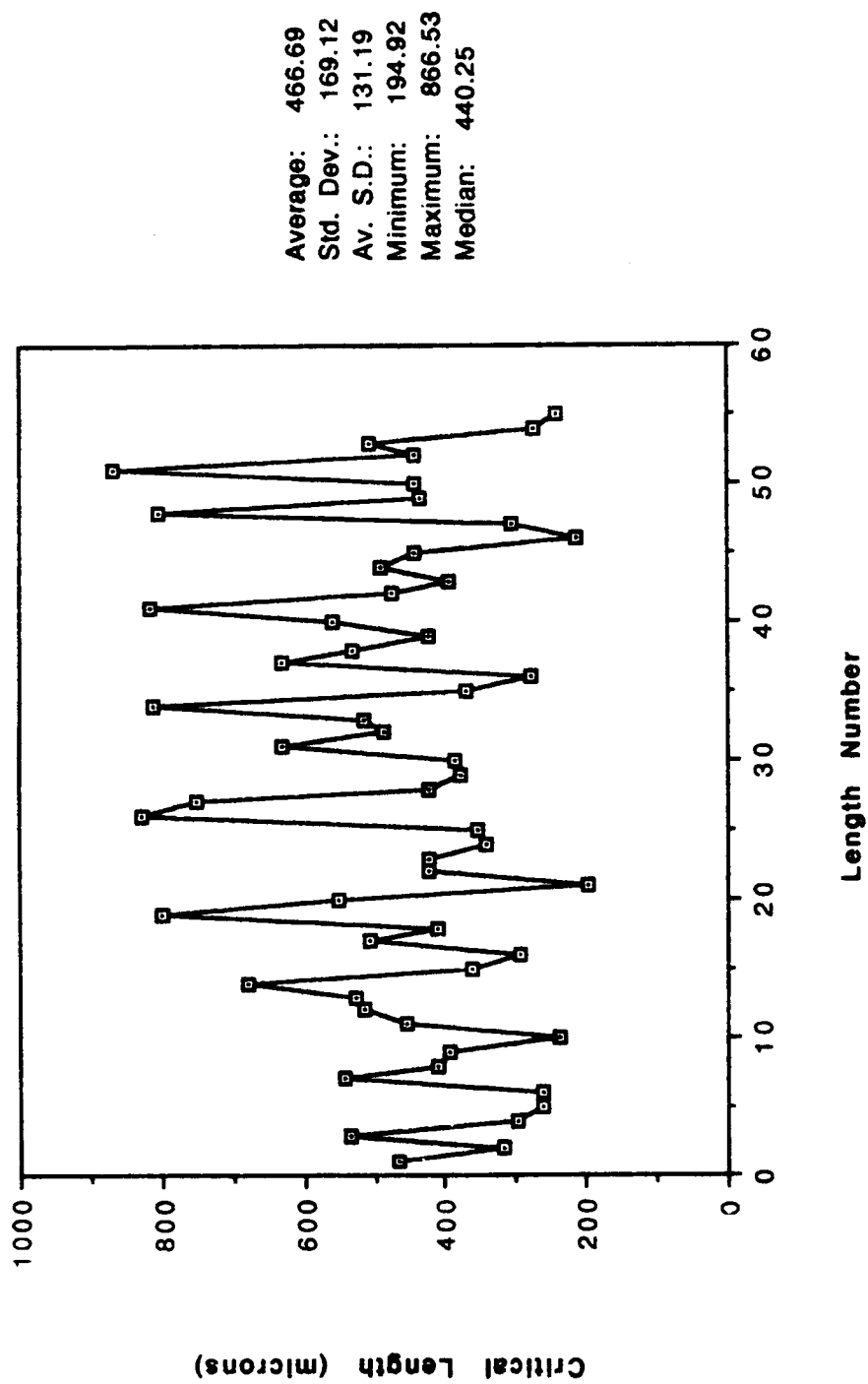


Figure A3. Critical length data for specimen number one, aged 500 hours.

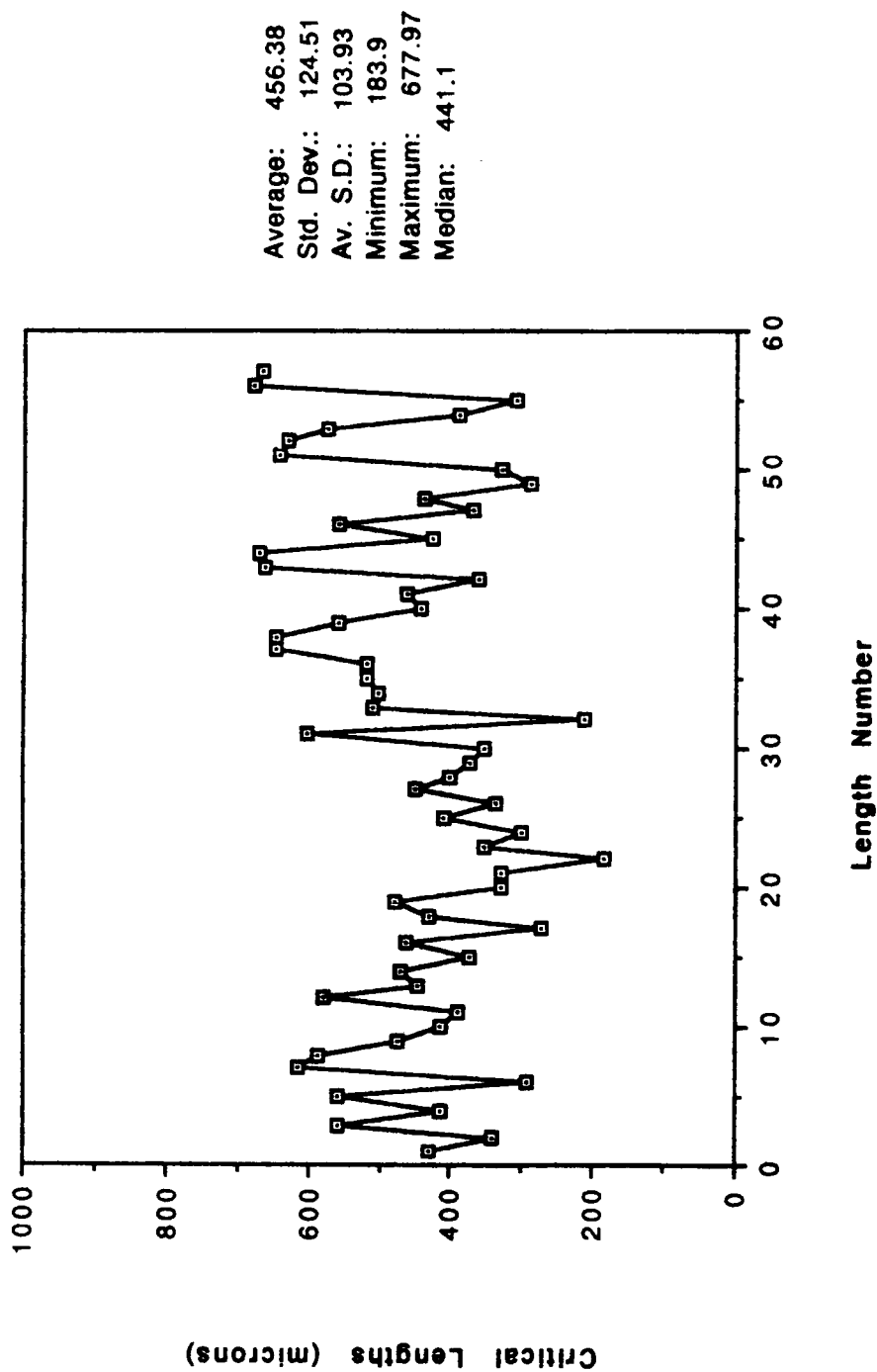


Figure A4. Critical length data for specimen number two, aged 500 hours.

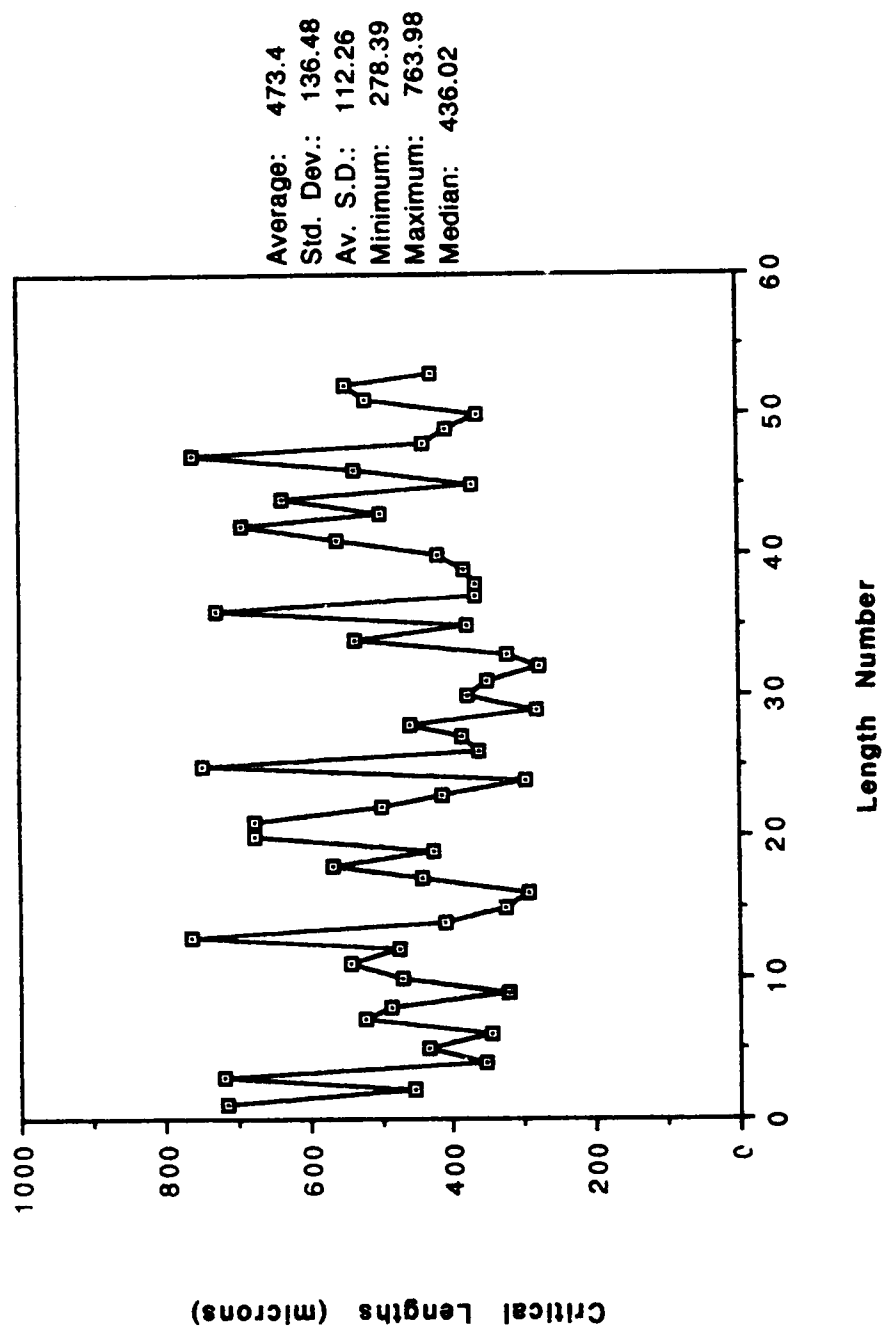


Figure A5. Critical length data for specimen number three, aged 500 hours.

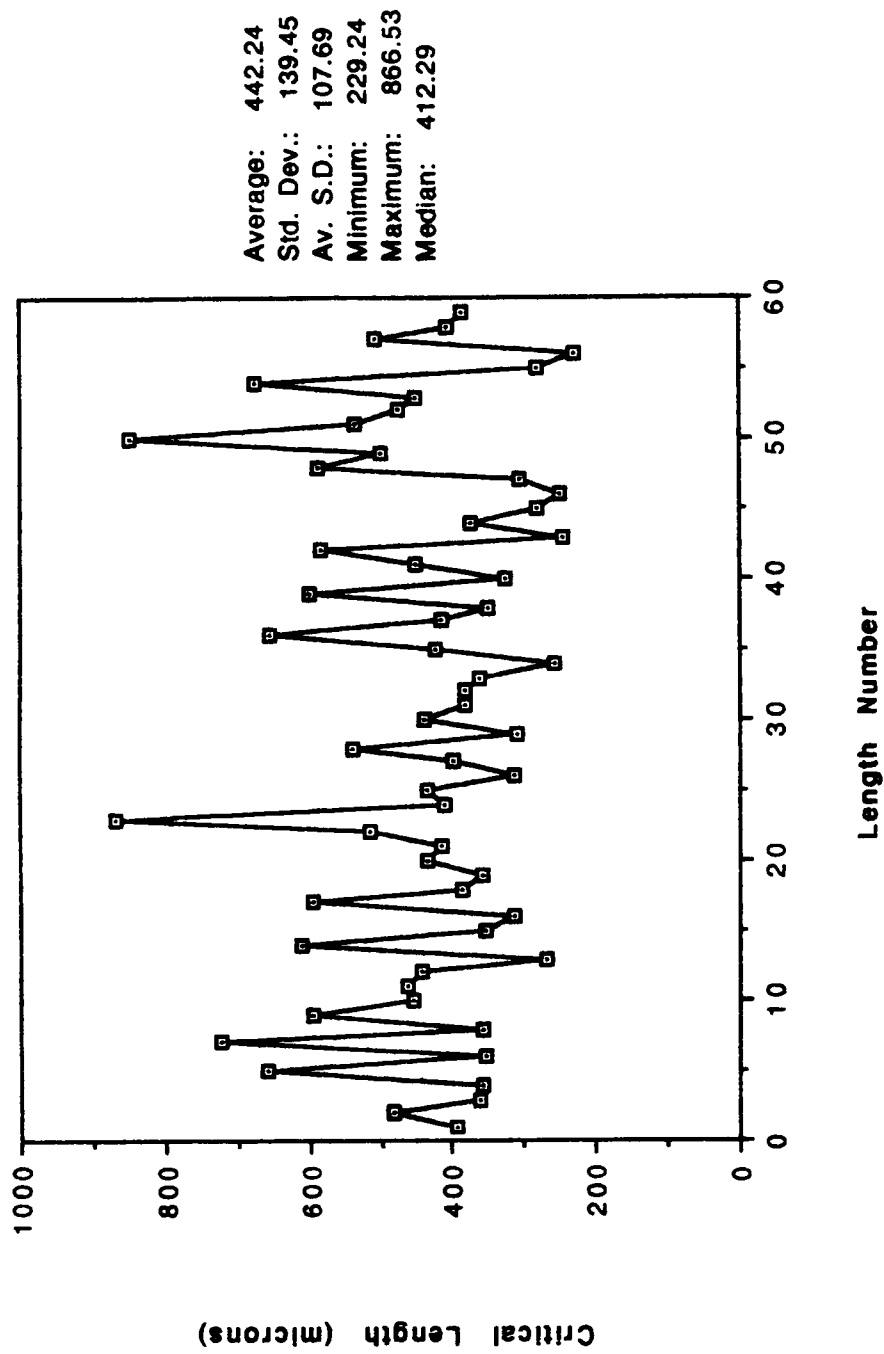


Figure A6. Critical length data for specimen number one, aged 1000 hours.

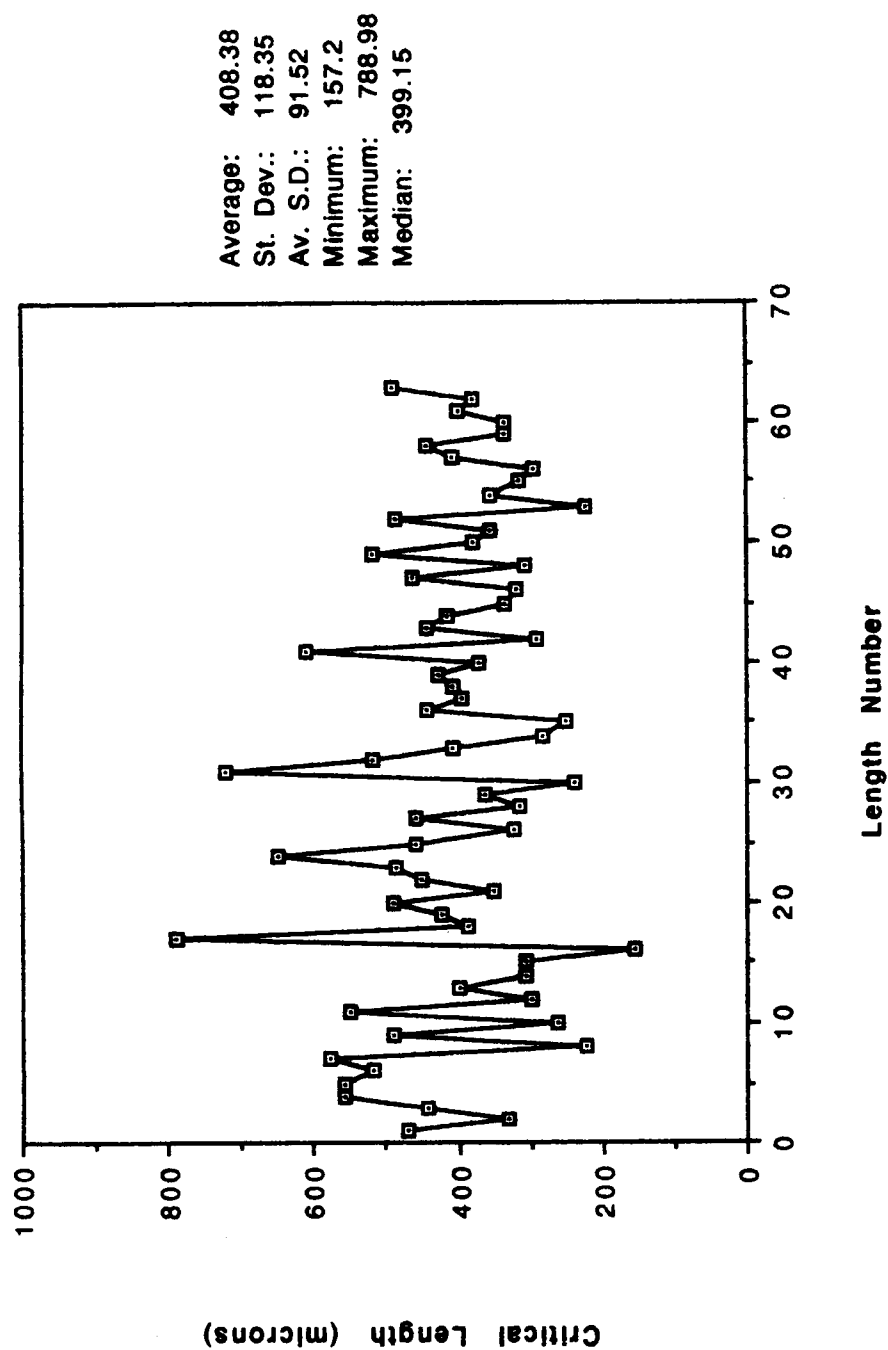


Figure A7. Critical length data for specimen number two, aged 1000 hours.

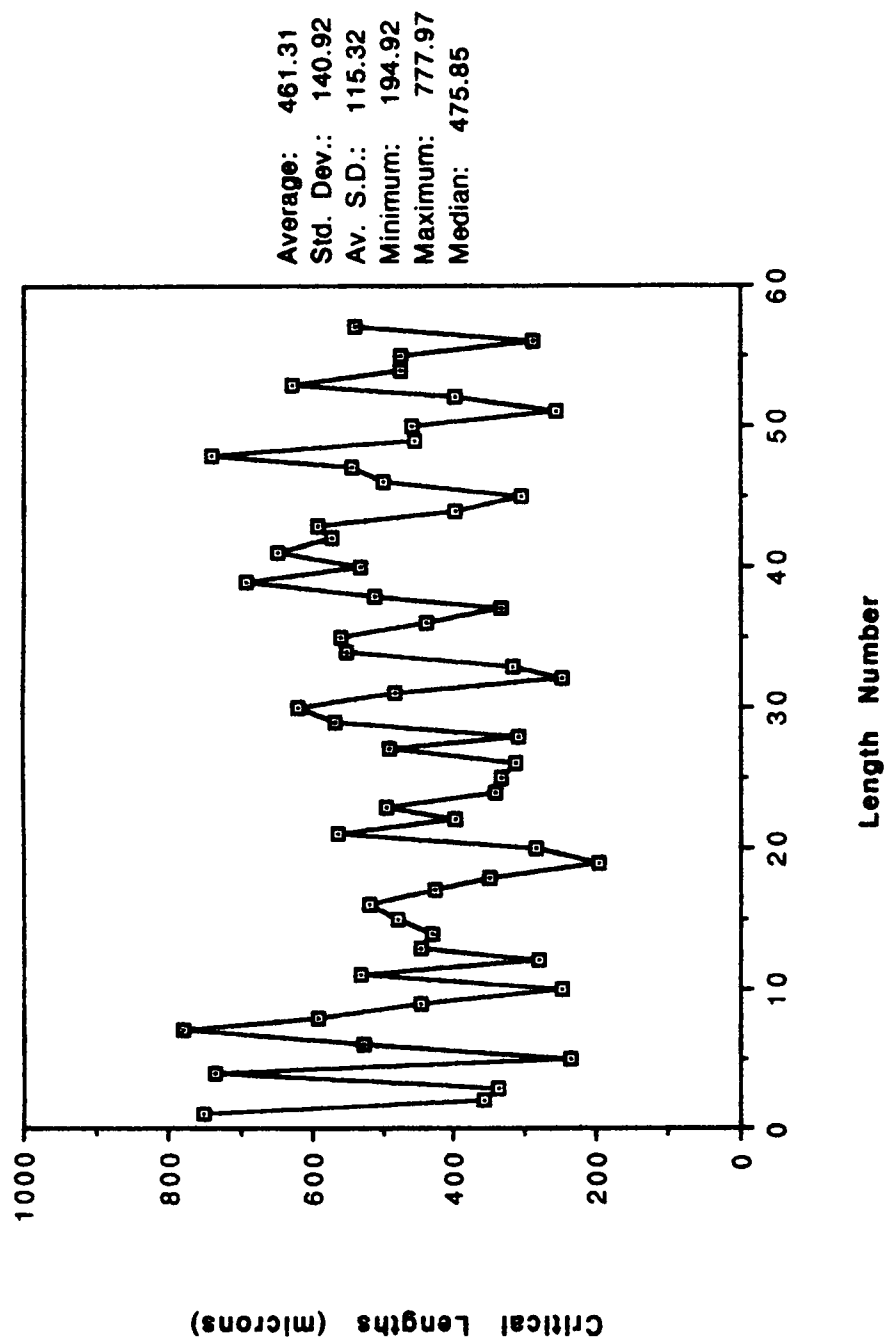


Figure A8. Critical length data for specimen number three, aged 1000 hours.

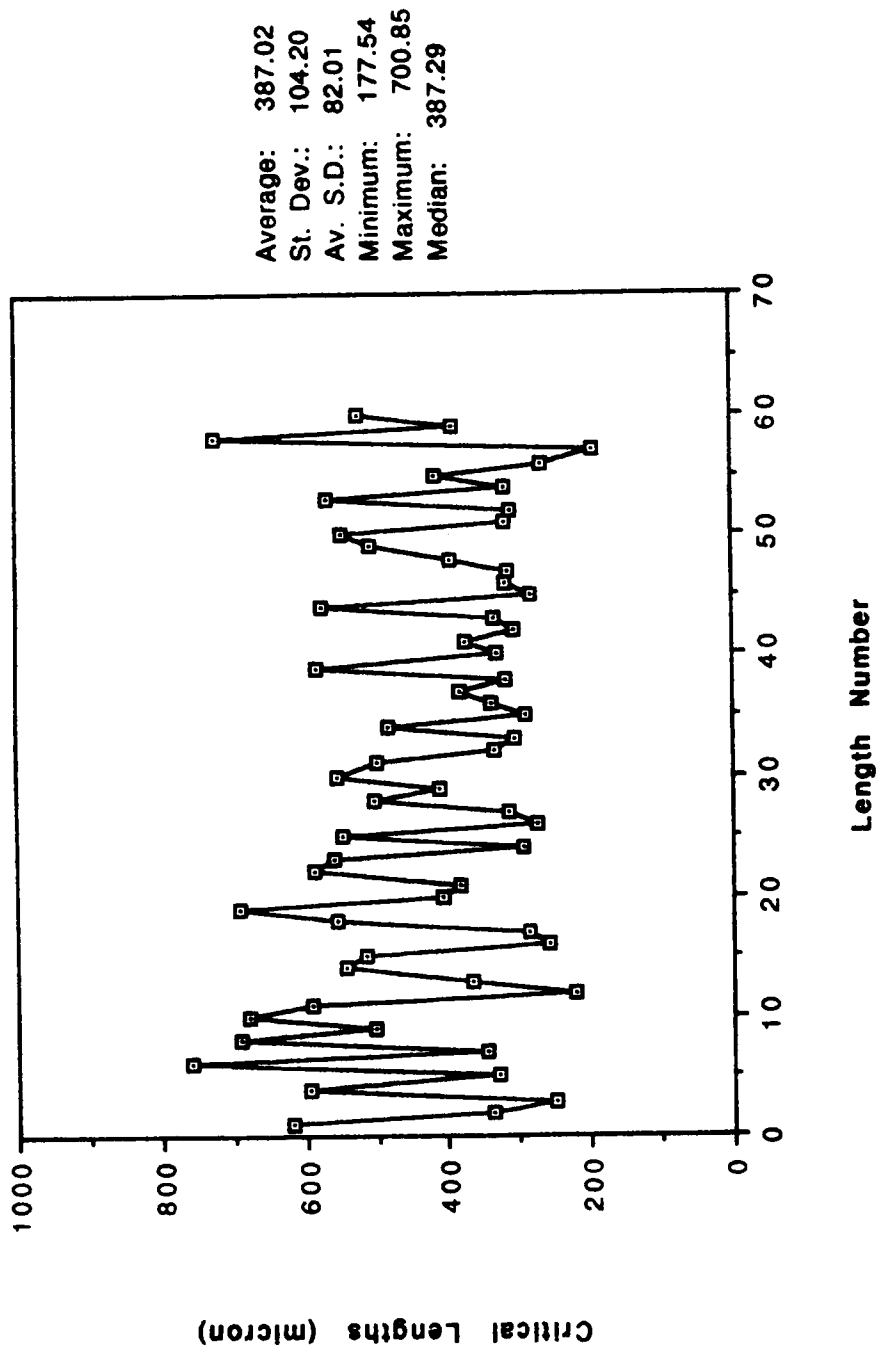


Figure A9. Critical length data for specimen number one, aged 2000 hours.

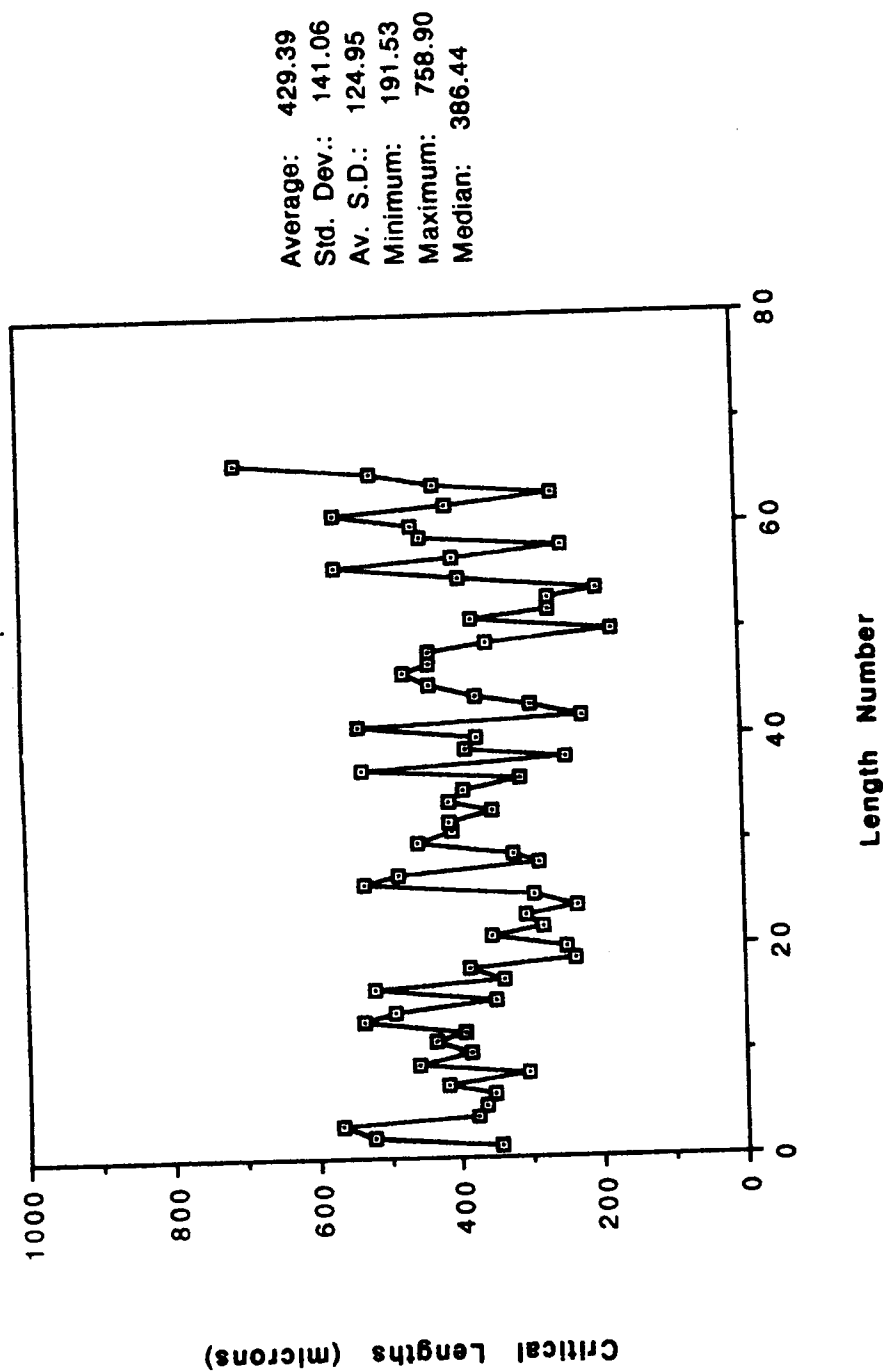


Figure A10. Critical length data for specimen number two, aged 2000 hours.

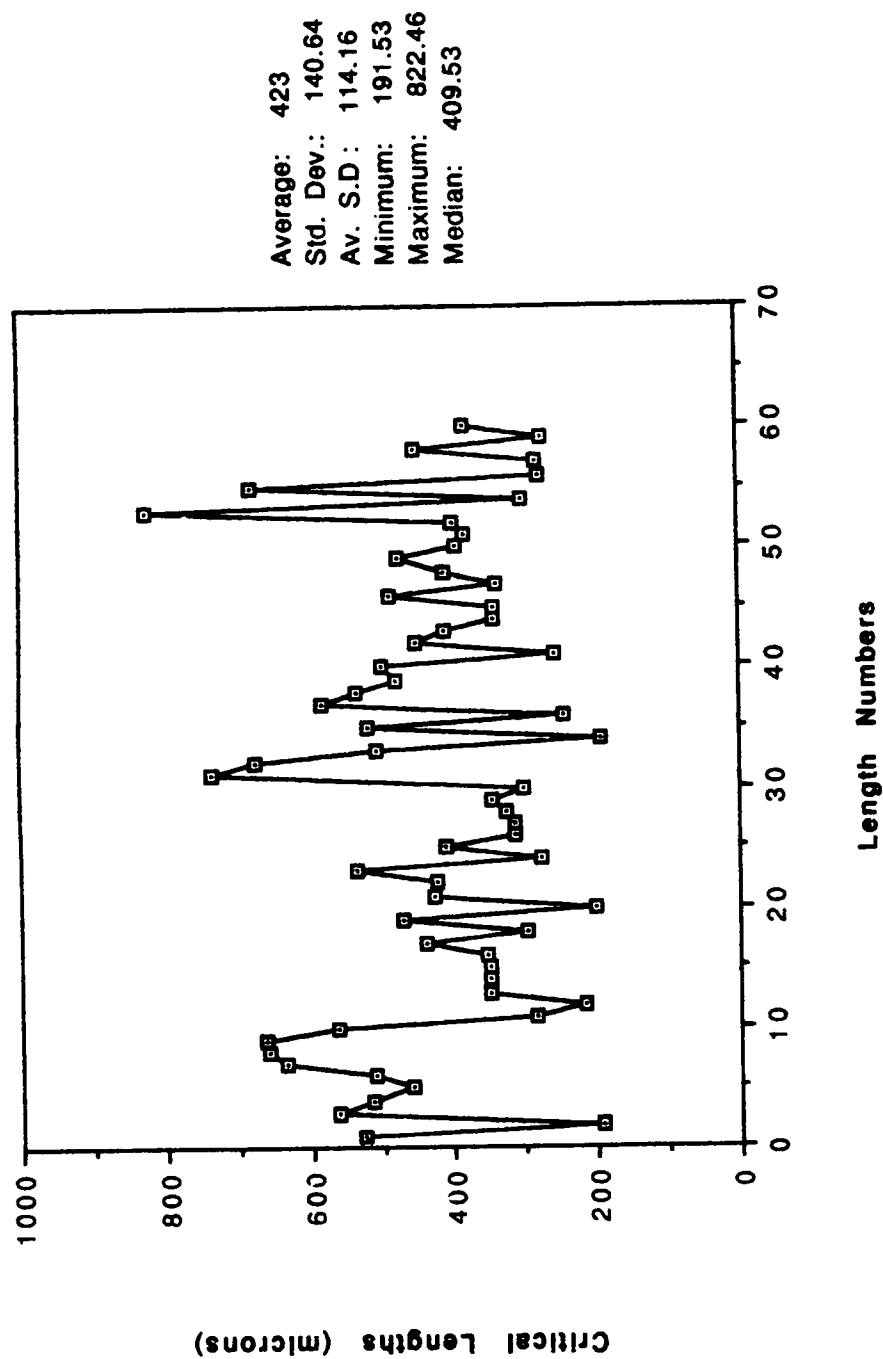


Figure A11. Critical length data for specimen number three, aged 2000 hours.

VITA

Johnny Hampton Spence, II was born in Maryville, Tennessee on August 8, 1972. He attended schools in the Blount County, Tennessee public school system where he graduated with honors from Heritage High School on May 31, 1990. In August 1990, he entered the Engineering College at the University of Tennessee, Knoxville. He graduated cum laude with a Bachelor of Science degree in Materials Science and Engineering on May 13, 1994. That same month, he started graduate school, also at the University of Tennessee, Knoxville, where he studied composite materials as a graduate research assistant. He graduated on May 10, 1996 with a Master of Science degree in Engineering Science.