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Specimen Geometry Effects on Graphite/ PMR-15 Composites During Thermo-Oxidative Aging

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SPECIMEN GEOMETRY EFFECTS ON GRAPHITE/PMR-15 COMPOSITES DURING THERMO-OXIDATIVE AGING

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SUMMARY

Studies were conducted to establish the effects of specimen geometry on the thermo-oxidative stability and the mechanical properties retention of unidirectional Celion 12000 graphite fiber reinforced PMR-15 polyimide composites. Weight loss, flexural strength and interlaminar shear strength were measured at isothermal aging times as long as 1639 hr at a temperature of 316 °C for three different specimen geometries. It was found that the three different types of specimen surfaces did exhibit different values of weight loss/unit area. The mechanical properties retention was also found to be dependent on geometry for these composites. The interlaminar shear strength decreased significantly over the complete range of aging times. The flexural strength retention started showing geometric dependency after about 1000 hr of aging at 316 °C. Weight loss fluxes, associated with the three different types of exposed surfaces, were calculated and used to develop an empirical mathematical model for predicting the weight loss behavior of unidirectional composites of arbitrary geometries. Data are presented comparing experimentally determined weight losses with weight loss values predicted using the empirical model.

E-2860

INTRODUCTION

In response to the growing need for high strength, low weight, high temperature materials for aerospace applications, polymer matrix composites, reinforced with graphite fibers are being increasingly utilized. The efforts of Serafini et al. (ref. 1) have led to the successful development and commercialization of one such material, namely graphite fiber reinforced PMR-15 composites. Currently this material is being used in aerospace components which experience temperatures up to 316 °C.

The selection of composite materials for use at the higher temperatures depends on many variables. In general, preliminary screening evaluations are based on the retention of physical and mechanical properties of the materials under investigation. These properties include thermo-oxidative stability, flexural strength retention and shear strength retention at elevated temperatures. As reported by Nelson (ref. 2), a specimen geometry effect occurs in weight loss and mechanical property data for unidirectional composites. Although this effect has also been noted previously by Burns et al. (ref. 3), Hanson et al. (ref. 4), and Scola (ref. 5), no geometry independent expression

has been developed for reporting weight loss data for these types of materials. No method exists for predicting isothermal aging weight loss values for composite materials.

This paper examines the effect of the specimen geometry on the physical and mechanical properties of graphite fiber reinforced PMR-15 composites during thermo-oxidative aging. Weight loss, flexural strength and shear strength changes are documented as a function of aging time at 316 °C.

Mathematical relationships are developed to help optimize the design of aging specimens so the resulting data can be used to successfully predict the aging behavior of identical composite material of more realistic geometries. Also the relationships that are developed can be used to compare and correlate data from different investigators.

MATERIALS

Three monomers were used in formulating the 1500 molecular weight PMR polyimide matrix. These include the monomethylester of 5-norbornene-2,3-dicarboxylic acid (NE) 4,4'-methylenedianiline (MDA) and 3,3',4,4'-benzophenonetetracarboxylic dianhydride (BTDA). These were obtained from commercial sources.

The reinforcement used in this study was the Celanese Celion 12000 PI-03 polyimide sized fiber. The properties of the fiber are listed in table I.

LAMINATE FABRICATION AND SPECIMEN PREPARATION

Esterification of BTDA to the dimethylester of 3,3',4,4'-benzophenone tetracarboxylic acid (BTDE) was accomplished by refluxing the anhydride in a 50 wt Δ methanol solution. The monomer solution was prepared at room temperature by the addition of the three monomers in a 2 NE: 3.087 MDA: 2.087 BTDE stoichiometric ratio with the proper amount of methanol to make a 50 wt Δ solution. Laminate fabrication then proceeded via the following steps:

(1) Fiber winding.
(2) Fiber impregnation by the monomer solution.
(3) Ply cutting and layup.
(4) Imidization.
(5) Compression molding.

This procedure is fully described elsewhere (ref. 6), and yielded low void, high quality laminates. A free standing postcure was given in air at 316 °C for 16 hr to complete the curing process.

A series of four Celion 12000/PMR-15 laminates were fabricated. Laminate (1-12) had a ten ply, unidirectional layup and had nominal measurements of 0.152 by 0.076 m. The fiber direction was parallel to the 0.152 m direction. This laminate was machined into specimens having the approximate geometries as follows:

(1) 0.025 by 0.076 m coupons.
(2) 0.0051 by 0.076 m flexural specimens.
(3) 0.0051 by 0.0152 m shear specimens.

All of these specimens had a thickness of about 0.0025 m. Laminates (1-41A) and (1-78B) also consisted of ten ply unidirectional layups having 0.203 by 0.076 m and 0.0916 by 0.0916 m overall dimensions respectively. Laminate (1-78A) was fabricated using a 25 ply unidirectional layup having overall dimensions of 0.203 by 0.076 m. The fiber direction again is in the direction of the larger dimension. A complete list of specimen sizes are shown in table I.

PHYSICAL AND MECHANICAL PROPERTIES MEASUREMENTS

All thermal aging was done in air convection ovens. The air change rate was 1×10^{-6} m^3/min. The specimens were allowed to cool to room temperature in a dessicator before they were weighed. Macroscopic measurements were determined using dial calipers having an accuracy of $\pm 2.54 \times 10^{-5}$ m. Specimen dimensions were measured and recorded every time the specimens were removed from the oven for weighing.

The densities of the composites were measured by a water displacement technique as described in ASTM-D792.

The mechanical properties that were measured to monitor the properties retention during the aging time were flexural and shear strengths. The apparent horizontal shear strength was determined using the short beam method as detailed in ASTM-D2433. The two support pins measured 0.0064 m in diameter instead of the recommended 0.0032 m diameter, and a span to depth ratio of 5:1 was used. The flexural properties were measured using the procedure described in ASTM-D790. A span of 0.064 m was used. This span yielded a span to depth ratio of approximately 26:1 for most of the flexural specimens that were tested.

All laminates were found to be ultrasonically sound. Using an acid digestion technique along with composite density measurement data, the void contents of the composites were determined to be less than 0.6 vol Δ.

RESULTS AND DISCUSSION

Composite weight loss

The weight loss data are presented in figure 1. In figure 1(a), one can see that, as expected, the actual weight loss, in kg, is dependent on the size of the specimen. However, if the data are plotted in terms of percent weight loss, as in figure 1(b), one can see that the weight loss is not significantly controlled by the specimen weight or volume. The ranking of specimens on a percent weight basis is inverse to the ranking on a weight basis (fig. 1(a)). The ranking of the specimen does appear to suggest the influence of a surface phenomenon when one examines figure 1(c). In this figure the weight loss is presented as weight/unit area. The calculated ratios of surface areas in the order; shear; flexural; coupon are 1: 4.1: 16.3. The ratios for weight losses on a weight per unit basis for the three types of specimens are 1: 3.6: 14.4.

The surface area ratios and the weight loss ratios are in close agreement, suggesting a significant effect of the specimen surface area on the absolute weight loss magnitude. If one examines figure 1(c) more closely, two points of interest are apparent. The first point is that the curves for the coupons and the flexural specimens lie very close together. In fact, a single curve could adequately represent the data from both types of specimens, even though the weight loss data are consistently slightly greater for the coupons than for the flexural specimens. The second point of interest is that the weight loss/unit area for the shear specimens is significantly higher than those of the other two types of specimens after isothermal aging at 316 °C for over 1600 hr. One might conclude from these two points that one type of surface might be experiencing a significantly greater weight loss than the other two types of surfaces.

In exploring the above possibility, one can describe three different types of specimen surfaces that comprise the total surface area of each of the three types of specimens. One of the surfaces is that surface which was compressed by the metal die surface during the consolidation and curing of the laminate. This surface is shown in figure 2 and is designated as surface S1. This surface can be considered as being an "all resin" surface in the as-fabricated condition. The second type of surface is the surface which is created by cutting the specimen through the thickness in a direction parallel to the unidirectional fibers. This surface is designated surface S2 in figure 2. The fraction of this surface, which is exposed matrix, is equivalent to the volume fraction of matrix in the specimen. The third type of surface, S3 in figure 2, is the surface created when one slices the specimen through the thickness in a direction normal to the fiber direction. The fraction of matrix exposed to the environment on surface S3 is also equal to the volume fraction of matrix in the specimen.

The areas of each of these three types of surfaces and the fractions of the total exposed surfaces which are contributed by each type of surface for each type of specimen are presented in table II. If one compares the data in table II with the presentation of weight loss/unit area in figure 1(c), it appears that there might be a relationship between the fraction of surface, S3, in the specimens and the ranking of the specimens from the data shown in figure 1(c).

In order to explore the possibility of such a relationship, as proposed above, one can ideate that during isothermal aging of the graphite/polyimide composites, weight was being lost at three different rates from the three different surfaces. The rates (fluxes) are expressed in kg/sq m-hr. If the relationship suggested by the comparison of the data in figure 1(c) and table II is real, then one may consider that the total weight loss from a specimen, during isothermal aging, is a function of both time and the fraction of each type of area in a particular composite geometry. The relationship between weight loss and time and surface area can be expressed as follows:

$$Delwt = [A1K1 + A2K2 + A3K3]t^{B} \qquad (1)$$

A1, A2 and A3 = Areas of surfaces S1, S2 and S3 respectively.
K1, K2 and K3 = Weight loss rates from surfaces S1, S2 and S3 respectively.
Delwt = Weight loss.
B = Constant.

This relationship indicates that composite weight loss during isothermal aging is almost totally a surface phenomenon. It does not involve the bulk material within the specimen to any significant degree during the 1600 hr time span of this test program. Figures 3 to 5 show microphotographs of sections of the graphite/polyimide composite specimens after being exposed to an environment of 316 °C for various times. It does appear that for the times investigated in this study, the degradation of the composite is visibly occurring at the surface and not in the bulk material.

It must be noted that there are differences between the depth of penetration of oxygen through the surfaces S1 and S2 with that of S3. The amount of material involved in the degradation process through S3 appears to be significantly greater than the amounts of material which have degraded along the other two types of surfaces. These figures (3 to 5) corroborate the assumptions previously made and based on figure 1(c) and table II, that the weight losses of the three types of specimens studied were significantly influenced by the amount of surface, S3, that was exposed to the environment.

To further test the validity of the above assumptions, a number of regression analyses were run to fit the weight loss data to an equation similar to equation (1). The calculations were made using an HP Statistical Library Program (HP 98820). The aging data for the three types of specimens were fitted to an equation of the form :

$$Delwt = c + Dt^{B} \tag{2}$$

The calculated equations, data points and curves are shown in figures 6 to 8 for each of the types of specimens studied. Note the spread in data increases as the time of exposure increases.

If one compares equations (1) and (2), it is evident that:

$$D = [A1K1 + A2K2 + A3K3] \tag{3}$$

K and A are as defined for equation (1). If this relationship is valid, then one should be able to solve the four equations presented in figures 6 to 8 for the three weight loss fluxes (K1, K2, K3) from the surfaces S1, S2 and S3. These equations were solved simultaneously and the calculated K values are as follows:

$$K1 = 1.817 \times 10^{-7} \text{ kg/sq m-hr}^{1.82}$$

$$K2 = 4.652 \times 10^{-8} \text{ kg/sq m-hr}^{1.82}$$

$$K3 = 1.648 \times 10^{-6} \text{ kg/sq m-hr}^{1.82}$$

These values indicate that the greatest loss of composite weight is from the ends cut perpendicular to the unidirectional fibers (S3).

The weight loss from the compressed (molded) surface is calculated to be four times that of the surface cut parallel to the fiber direction. If one considers the molded surface to initially consist totally of matrix material, this surface has 2.2 to 2.5 times more matrix exposed to the environment than does the cut surface. Laminate fiber volume fractions vary from 0.55 to 0.60.

The lower calculated weight loss flux for the cut surface may be due to a protective mechanism provided by the fibers. This could entail the retardation of oxygen diffusion into the specimen. The order of magnitude of the difference between the loss fluxes between surfaces S3 and S1 is not unexpected as others (refs. 1 and 2) have postulated accelerated weight losses from these end surfaces.

Although the numerical values presented herein were acquired empirically, they can be rationalized as being reasonable numbers.

Under the conditons of this study, the time exponent, B, is 1.82 and it appears to be insensitive to geometry. When one performs a regression analysis using the data measured by Nelson for Celion 6000/PMR-15 composites aged for times up to 15 000 hr at 232 °C, the exponent, B, is almost unity. In order to satisfactorily explain the significance of the time term in equation (2), a more extensive testing program must be developed.

Even though the calculated values of weight loss fluxes appear to be reasonable values, the credibility of these values must be confirmed by using them to predict isothermal aging weight losses for specimens with different dimensions.

Three laminates were fabricated for this purpose. Their dimensions and weights are listed in table II. The monomers were mixed separately from the monomers used to fabricate laminate (1-12).

One of the laminates fabricated (1-78A) was fabricated with a surface area equal to that of the coupons from laminate (1-12) but with a thickness twice as great. The weight loss data are shown as a function of time in figure 9. The open circles are the predicted values of weight loss from the equations calculated by regression analysis.

Similar curves are presented in figures 10 and 11 for two other composite laminate specimens which were isothermally aged at 316 °C. In figure 10, data for a 0.073 by 0.073 by 0.0022 m laminate (1-78B) are presented. The observed data, the predicted data, and the calculated curve are all presented in this figure. The results of the regression analyses predict weight loss values slightly higher than those observed. The average difference is 5.0 percent with a maximum of 6.5 percent at 1525 hr. This is a reasonable prediction.

Figure 11 shows observed and predicted weight loss values for a 0.073 by 0.173 m laminate specimen. This specimen contains about six times the surface area of the smaller coupon specimen. The predicted data and the observed data agree very closely. Again, the values of the observed data are slightly lower than those of the predicted values.

Overall, the three weight loss fluxes calculated from the graphite/polyimide weight loss data appear to be reasonable values which can be used to predict composite material response to isothermal aging for times up to about 1600 hr at 316 °C.

SURFACE AREA CHANGES

The surface areas and volumes used in computing weight loss fluxes are based on the original macroscopic dimensions of the specimens that were made prior to the initiation of aging. In order to account for the possibility of

error in the calculations due to surface area changes during aging, the changes in surface areas were monitored. The changes in surface areas were between -2.0 and -3.5 percent. This was considered to be negligible.

COMPOSITE MECHANICAL PROPERTIES

Mechanical property differences between precut, aged flexural and shear specimens and identical specimens freshly cut from aged coupons are shown in figures 12 and 13. Since it appears that the aging of the composites causes no appreciable degradation of the bulk of the composite material, the degradation of the mechanical properties of the specimens cut from the aged coupons should be due to the degradation of the two S2 surfaces. Then it can be rationalized that any differences between mechanical properties of the aged, precut specimens and the freshly cut, aged specimens is due to the cut edge deterioration in the precut test specimens.

If one compares the flexural strengths of the two types of aged specimens, as shown in figure 12, it appears that the above reasoning may be true. If one uses the effective widths (undegraded material) of the two types of specimens to calculate ultimate loads, one finds that the precut specimen should fail at a load or apparent flexural strength of 0.85 those of the freshly cut specimen. This would result in an apparent flexural strength of 0.78 GPa. This value is for a specimen that has been aged for 1639 hr at 316 °C. The observed value as shown in figure 12 is 0.75 GPa.

If one performs the same types of calculations to determine shear test loads or apparent shear strengths, one will find that the predicted apparent shear strength for the precut specimen, aged for 1639 hr is 35.5 MPa. The observed value is 26.2 MPa. The short beam shear specimens were tested using an overhang of 1.6×10^{-2} m. The measured length of oxygen penetration into the cut ends of the shear specimens that were aged for 1639 hr is 3.9×10^{-2} m. This means that the the oxidized end material was supporting the specimen during the test. When the specimen is visually examined, there are no signs of failure in the specimen where it was supported by the two end pins. The visible evidence of failure is that of compressive failure in the center of the top surface where the load pin contacted the specimen. The load-time traces recorded by during the testing also indicate a bearing failure as the mode of failure. One other item of importance in assessing the validity of short beam shear test results from precut aged composites is that due to the large amount of cracking occurring at the ends of the specimens, the shear failure may start at the end of one of the cracks. This would then alter the test to a test for measuring crack sensitivity rather than a test for measuring shear strength.

While the flexural failure strength can be reasonably well explained in a simple manner by the specimen degradation, the short beam shear strength reductions are due to more complex causes. It appears that from the results of this study, the short beam shear strength of an aged, precut specimen cannot be measured with an degree of confidence.

CONCLUSIONS

This study confirms the results of previous work (refs. 2 to 5) that the isothermal aging behavior and the mechanical properties retention of graphite fiber reinforced composites are indeed geometry dependent. In both instances, it appears that as the surface area of the specimen decreases, the degradation effect of the aging process increases. In all cases, including this study, the enhanced degradation is attributed to specimen edge surface effects of edge surfaces perpendicular to the fiber direction.

The significant conclusions from this study, basically provide detail and clarification to the general conclusions presented above. These conclusions are listed as follows:

1. Surface weight loss rate values for composites can be calculated from aging data.

2. The weight loss of graphite fiber reinforced composites is a function of time to the 1.82 power. This is not dependent on the geometry of the specimen.

3. Weight losses can be reasonably predicted for graphite fiber reinforced composites of arbitrary geometries.

4. The high temperature aging degradation of the through-the-thickness fiber end surface is almost one order of magnitude greater than the compressed surface, and almost 40 times greater than the surface cut parallel to the fibers.

5. It appears that flexural strength retention, during aging, can be related to weight loss and is geometry related.

6. Short beam shear strength degradation is geometry dependent and is susceptible to a number of mechanisms which can cause nonshear failure to occur.

BIOGRAPHIES

Kenneth Bowles

Kenneth Bowles received his Ph.D. in Materials Science from Case Western Reserve University. Since the start of his employment at the Lewis Research Center in 1958, he has conducted research in the areas of liquid metal corrosion, advanced nuclear fuel element materials and composite materials. He is currently working in the Polymer Branch in the are of composite toughness.

Anne Meyers

Anne Meyers received her B.S. in Chemical Engineering from Ohio State University in 1983. After graduation, she accepted a position in the Composites Branch at the Lewis Research Center as a Materials Engineer. Her work was directed toward environmental effects and processing science. In September of 1984, Anne entered medical school.

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TABLE I. - PROPERTIES OF
CELION 12 000
PI-03 FIBERS

Tensile strength, MPa	3889.7
Modulus, GPa	235.2
Elongation, percent	1.65
Density, Kg/m^3	17.8

TABLE II. - SPECIMEN DIMENSIONS

Laminate		No.	Thick, $mx10^3$	Wide, $mx10^3$	Long, m	Volume, m^3x10^7	Surface area, $mx10^4$	End area, $mx10^4$
Shear	(1-12)	43	2.46	5.21	0.0156	1.60	2.64	0.26
Flexure	(1-12)	22	2.44	5.21	0.0703	8.00	11.03	0.26
Coupon	(1-12)	10	2.44	25.40	0.0735	45.90	42.13	1.23
Thick coupon	(1-78A)	9	5.36	24.92	0.0652	86.90	42.13	2.64
Laminate 1	(1-41A)	1	2.39	72.64	0.1730	308.10	264.90	3.48
Laminate 2	(1-78B)	1	2.16	93.09	0.0967	195.00	188.26	4.00

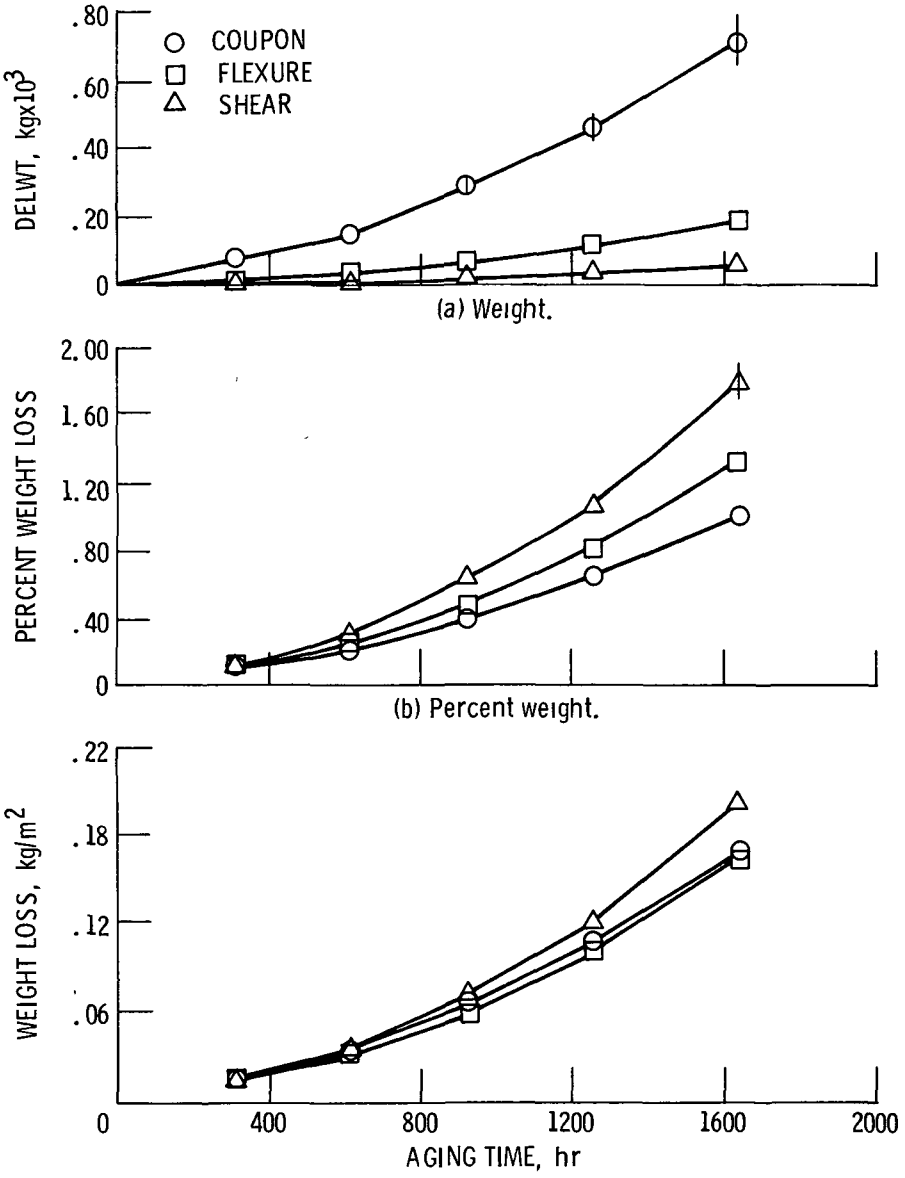

Figure 1. - Weight loss data from (1-12) laminate specimens.

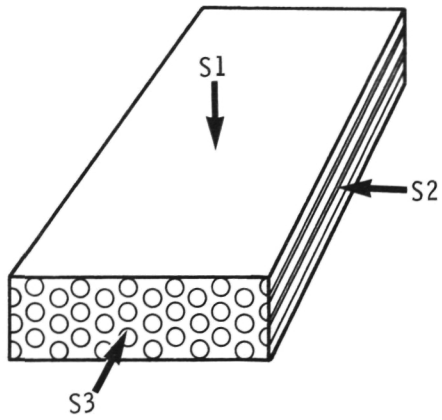

Figure 2. - Specimen surfaces.

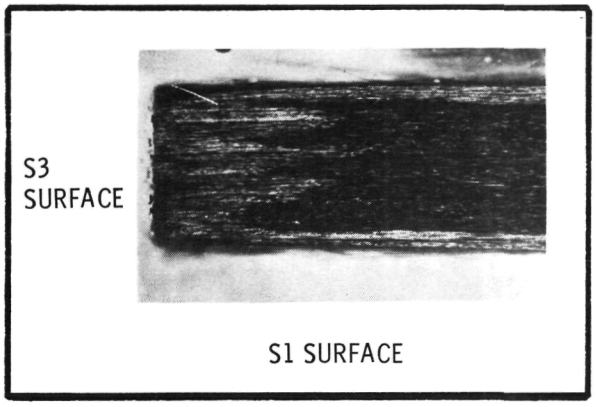

Figure 3. - Through-the-thickness surface of a composite specimen aged for 1639 hr at 316° C. Light area is degraded material. Thickness is about 0.0024 m.

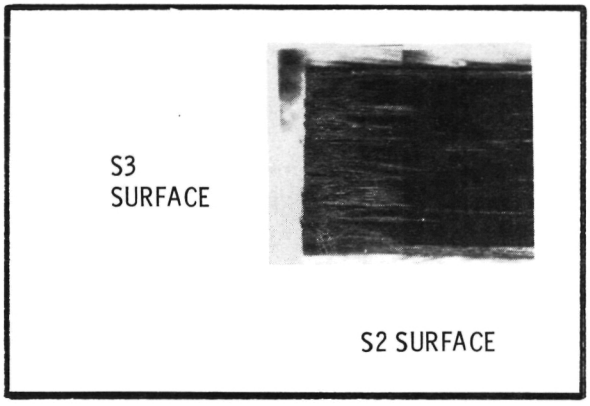

Figure 4. - Composite section parallel to S2 surface. Material was aged for 1639 hr at 316 °C. Light area is degraded material. Width is about 0.0051 m.

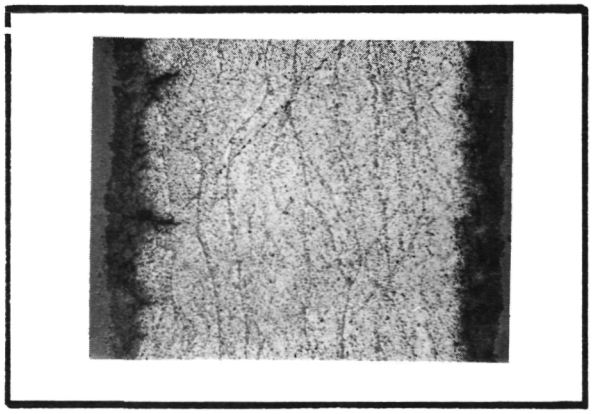

Figure 5. - Typical appearance of the interior of Celion 12000/PMR-15 composite specimen after aging at 316 °C for 1639 hr. Specimen thickness is 2.54×10^{-3} m.

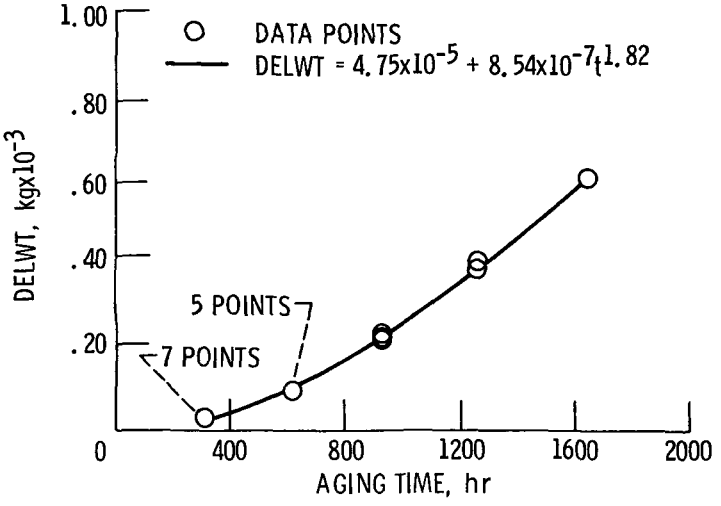

Figure 6. - Weight loss data and weight loss equation for (1-12) laminate coupons. Aging done at 316 ^{0}C.

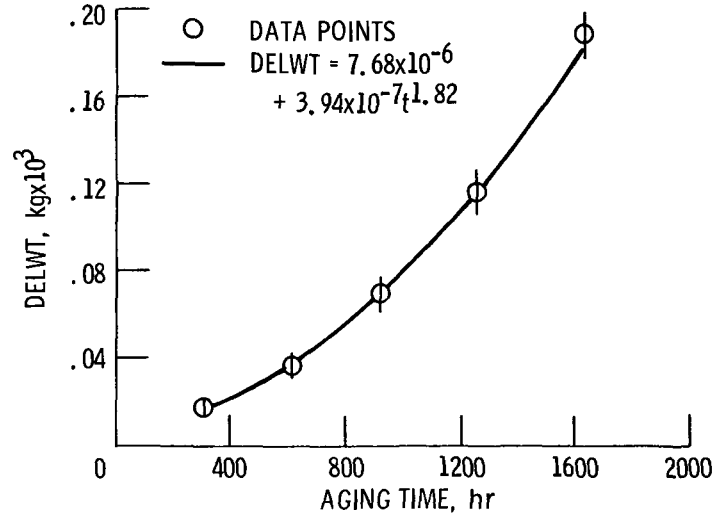

Figure 7. - Weight loss data and weight loss equation for (1-12) laminate flexure specimens. Aging done at 316 ^{0}C.

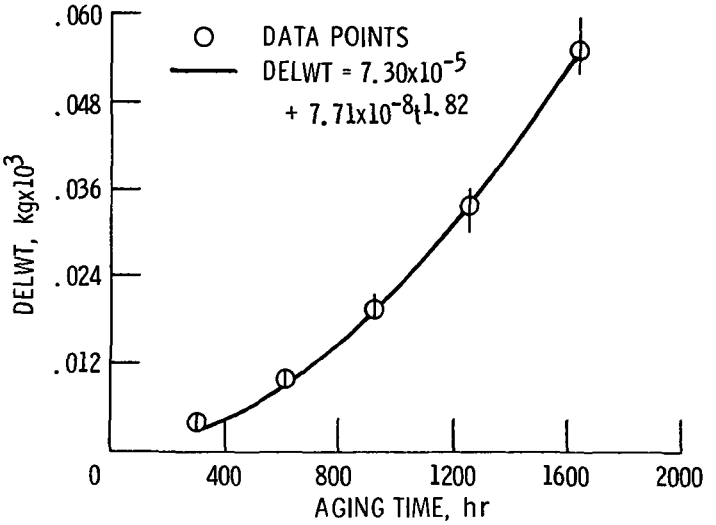

Figure 8. - Weight loss data and weight loss equation for (1-12) laminate shear specimens. Aging done at 316 ^{0}C.

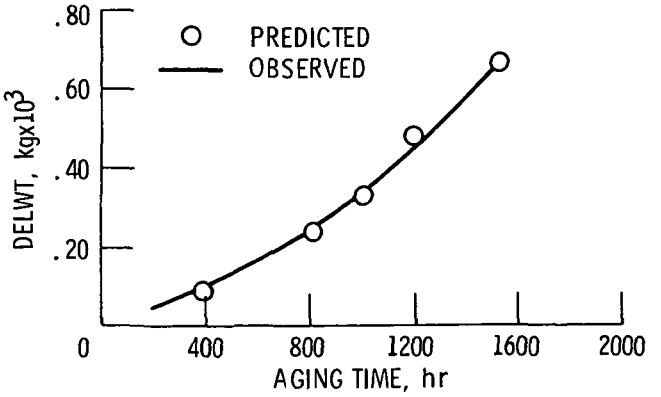

Figure 9. - Comparison of predicted and observed weight loss data for thick laminate (1-78A). Aging done at 316 ^{0}C.

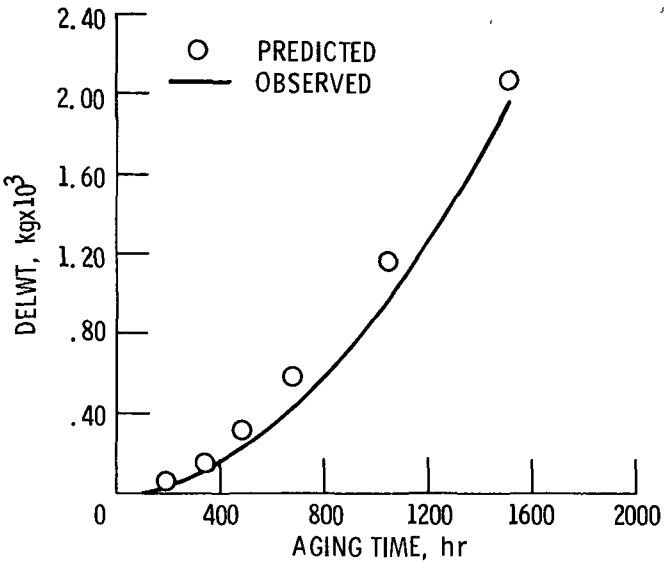

Figure 10. - Comparison of predicted and observed weight loss data for laminate (1-78B). Aging done at 316 °C.

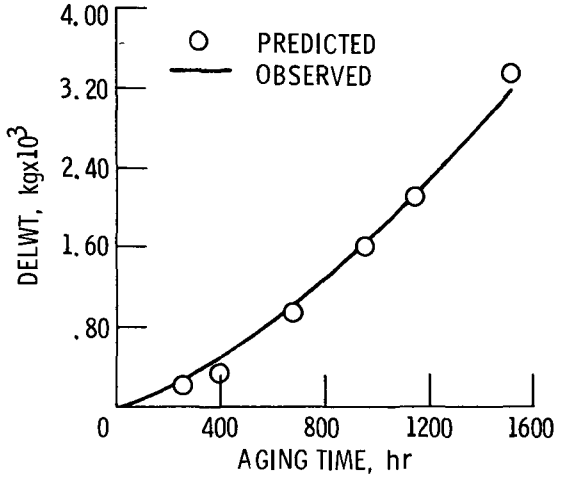

Figure 11. - Comparison of predicted and observed weight loss data for laminate (1-41A). Aging done at 316 °C.

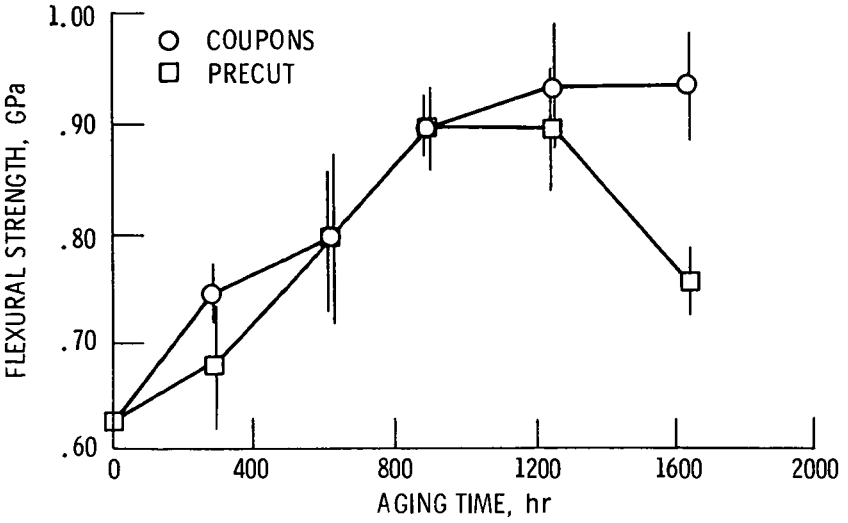

Figure 12. - Comparison of flexural strengths of precut and large composite specimens after aging at 316 °C. Tests conducted at 316 °C.

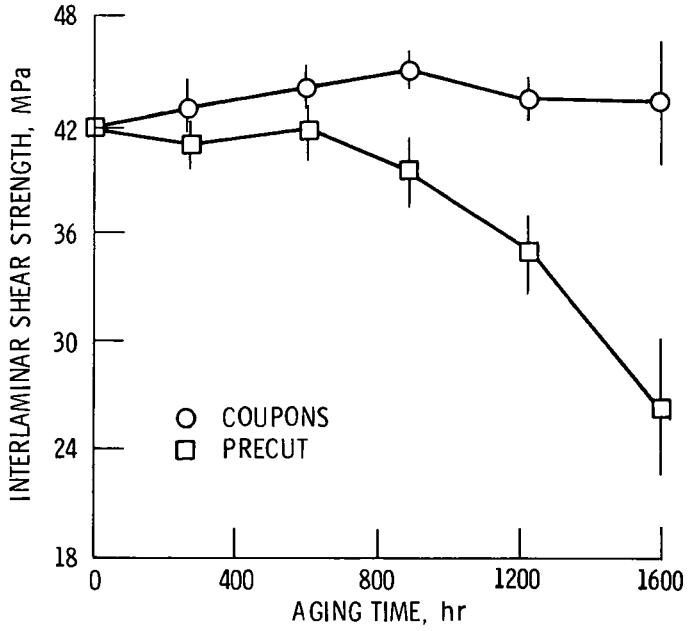

Figure 13. - Comparison of interlaminar shear strengths of precut and large composite specimens after aging at 316 °C. Tests conducted at 316 °C.

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15 Supplementary Notes

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16 Abstract

Studies were conducted to establish the effects of specimen geometry on the thermo-oxidative stability and the mechanical properties retention of unidirectional Celion 12000 graphite fiber reinforced PMR-15 polyimide composites. Weight loss, flexural strength and interlaminar shear strength were measured at isothermal aging times as long as 1639 hr at a temperature of 316 °C for three different specimen geometries. It was found that the three different types of specimen surfaces did exhibit different values of weight loss/unit area. The mechanical properties retention was also found to be dependent on geometry for these composites. The interlaminar shear strength decreased significantly over the complete range of aging times. The flexural strength retention started showing geometric dependency after about 1000 hr of aging at 316 °C. Weight loss fluxes, associated with the three different types of exposed surfaces, were calculated and used to develop an empirical mathematical model for predicting the weight loss behavior of unidirectional composites of arbitrary geometries. Data are presented comparing experimentally determined weight losses with weight loss values predicted using the empirical model.

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