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# 371 °C Mechanical Properties of Graphite/Polyimide Composites

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NASA

# 371 °C MECHANICAL PROPERTIES OF GRAPHITE/POLYIMIDE COMPOSITES

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## SUMMARY

A series of condensation polyimides based on pyromellitic dianhydride was synthesized and evaluated for potential application at 371 °C. Several three- and four-ring benzenoid diamine systems containing oxygen bridging groups were investigated in this study. Thermomechanical analysis of neat resin specimens indicated that the polyimide prepared from the dimethyl ester of pyromellitic acid (PMDE) and 2,2-bis[4-(4'-aminophenoxy)phenyl]-1,1,1,3,3,3-hexafluoropropane (BDAF) was the only resin system which had a glass transition temperature (Tg) above 371 °C. The Tg of the PMDE/BDAF polyimide was found to be 390 °C after a postcure in air at 371 °C for 24 hr. Unidirectional composites were fabricated from the PMDE/BDAF system and unsized Celion 6000 graphite fibers. Final cure temperatures in the range of 371 to 427 °C with an applied pressure of 10.34 to 13.78 MPa were investigated. The void content of the composites ranged from 4.6 to 8.6 percent. Composites cured at 399 °C under a pressure of 10.34 MPa and postcured in air at 371 °C for 24 hr exhibited the highest 371 °C interlaminar shear strength (ILSS, 40.7 MPa) and flexural strength (758 MPa). The thermo-oxidative stability of the composites was determined by subjecting specimens to isothermal exposure at 371 °C in air at atmospheric pressure, as well as a pressure of 0.52 MPa. Specimens exposed at atmospheric pressure exhibited a weight loss of 12 percent after 200 hr of exposure and 88 percent retention of its original 371 °C ILSS. In contrast, the specimens exposed at 0.52 MPa pressure exhibited a comparable weight loss after only 72 hr, and a 71 percent retention of its original 371 °C ILSS. Based on the results of this study, it appears that the PMDE/BDAF polyimide system has potential as a composite matrix resin for continuous exposure in air at 371 °C and atmospheric pressure up to 200 hr. However, the service life at 371 °C and elevated air pressures appears to be limited to 48 to 72 hr.

## INTRODUCTION

Studies conducted at NASA Lewis Research Center led to the development of a class of readily processable polyimides known as PMR polyimides (refs. 1 and 2). The commercially available version known as PMR-15 has gained a wide acceptance as a matrix resin in fiber-reinforced composites for high performance structural applications (ref. 3). The development of PMR-15 has made it possible to extend the upper use temperature limit of fiber-reinforced composite materials to 316 °C. However, it would be highly desirable to identify matrix resins with an even higher temperature capability up to 371 °C. A previous study (ref. 4) has shown that it is difficult to obtain PMR-15 resins having a glass transition temperature (Tg) in the 371 °C range, even after extensive postcure. The purpose of this study was to synthesize and identify condensation type polyimides having potential as matrix resins for applications at 371 °C and air pressures up to 0.5 MPa.

The approach taken in this study was to synthesize a series of condensation polyimides based on pyromellitic dianhydride (PMDA). Because of its rigid structure, PMDA can be expected to confer high Tg values to a polyimide structure. However, to avoid processing problems due to excessive rigidity in the polyimide molecular structure, it was felt to be necessary to use flexibilized aromatic structures for the diamine component of the polyimide.

A series of neat polyimide resins was synthesized from the dimethyl ester of pyromellitic acid (PMDE) and multi-ring aromatic diamines containing ether, as well as other connecting groups. The resins were screened for their Tg values and thermo-oxidative stability. One resin system, prepared from PMDE and 2,2-bis[4-(4'-aminophenoxy)phenyl]-1,1,1,3,3,3-hexafluoropropane (BDAF), was selected for composite evaluation. Unidirectional composites, using unsized Celion 6000 graphite fiber, were compression-molded at temperatures ranging from 371 to 427 °C and pressures ranging from 10.34 to 13.78 MPa. Short-term mechanical properties at room temperature and 371 °C were determined. Composite weight loss and mechanical property retention characteristics as a function of exposure time at 371 °C in air at both atmospheric pressure and a pressure of 0.52 MPa were also determined.

## RESULTS AND DISCUSSION

### Resin Screening Study

In theory, the polyimides prepared from pyromellitic dianhydride (PMDA) and rigid aromatic diamines without connecting groups (such as p-phenylenediamine or 2,6-diaminonaphthalene) should exhibit a combination of high glass transition temperatures (Tg) and excellent thermo-oxidative stability. However, in the fabrication of fiber-reinforced composite components, the processability of the matrix resin plays an important role. Monomer combinations such as those mentioned above exhibit an extremely limited resin flow during the fabrication cycle, making it very difficult to fabricate high-quality, low-void composites. To improve the processing characteristics, it is necessary to flexibilize the diamine portion of the polymer by introduction of connecting groups between two or more aromatic rings.

In this study, a series of ether-modified, flexibilized diamines was synthesized according to the scheme outlined in figure 1. The disodium salt of a bisphenol was reacted with p-chloronitrobenzene in refluxing N,N-dimethylacetamide to yield the dinitro compound, which was then hydrogenated to the corresponding diamine in N,N-dimethylformamide solution, using Raney Ni as the catalyst. The monomers used in this study are listed in table I. It should be noted that the diamine BDAF was first synthesized by TRW under a NASA contract (ref. 5), and is now commercially available. The three-ring diamines PBAB and MBAB contain two oxygen bridges between the phenyl rings to provide flexibility to the molecular structure. In addition to two oxygen bridges, the four-ring diamines BDAF, BDAP, BDAS, and BDAO also contain a third bridging group.

Molding powders were prepared from PMDE and each of the diamines listed in table I. Neat resin specimens were compression-molded using a final cure pressure of 13.78 MPa and a temperature of 399 °C. Well consolidated resin moldings were obtained in each case except from the PMDE/PBAB combination, which exhibited a large number of voids. When this resin was molded at a pressure of 17.23 MPa, the void content was diminished, but not entirely eliminated. It appears that the PMDE/PBAB combination provides a rigid molecular structure which prevents adequate resin flow during fabrication.

Table II shows Tg values of the resins before and after postcure in air at 371 °C, as well as weight loss data after exposure at 371 °C. As expected, postcuring for 24 hr increased the Tg values of the resins. The increase was 40 to 60 °C, except for the PMDE/BDAF resin, where the increase was 96 °C. It is interesting to note that postcure times longer than 24 hr conferred no significant increase in Tg for any of the resins. It can be seen that the resins prepared from the four ring diamines BDAP, BDAS, and BDAO exhibited the lowest Tg values, ranging from 318 to 330 °C. This is not unexpected since each diamine contains three flexibilizing groups. The three-ring diamine MBAB, which contains only two flexibilizing groups and a meta orientation on the central phenyl group, expectedly confers an increase in Tg to 334 °C, compared to 318 °C for its four-ring homolog BDAO. The more linear isomer PBAB, which is completely para, confers a further increase in Tg to 357 °C. The above trends thus follow an expected pattern. However, the resin from the diamine BDAF exhibits an anomalously high Tg value, compared to the resins from the other four-ring diamines. Molecular models indicate that the central hexafluoroisopropylidene group in BDAF is relatively rigid compared to the central linking groups in the three other four-ring diamines. This rigidity would be expected to confer a higher Tg value to the PMDE/BDAF polymer. In addition, it is possible that the presence of the hexafluoroisopropylidene group allows an efficient pathway for oxidative crosslinking during cure and postcure at high temperatures. One possibility is that cleavage of the $C-CF_3$ bond produces a very stable radical which can readily react with other crosslinking sites in the polymer molecule. It is more likely, however, that crosslink formation occurs by cleavage of the C-O bonds and subsequent coupling of the phenyl ether portions of the polymer chain. This type of crosslinking mechanism was found to occur when poly-N,N'-(4,4'-diphenylether)pyromellitimide film was exposed in air at 400 °C (ref. 6). It is speculated that the stronger electron-withdrawing character of the hexafluoroisopropylidene group in BDAF promotes a more efficient oxidative crosslinking mechanism, compared to the central bridging groups in BDAP, BDAS, and BDAO.

It can be seen in table II that based on Tg values the only polymer with potential for 371 °C applications is the polyimide prepared from PMDE/BDAF. This resin also exhibited the lowest weight loss after exposure in air at 371 °C for 72 hr. Based on these considerations, it was selected for composite evaluation studies.

## Composite Fabrication and Evaluation

Unidirectional composites were compression-molded, using the PMDE/BDAF monomer combination and unsized Celion 6000 graphite fiber. In the initial studies a final cure temperature of 371 °C was employed at two different molding pressures: 10.34 and 13.78 MPa. Some properties of the composites are shown in table III. It should be noted that the interlaminar shear strength (ILSS) values are quite low. For comparison, the room temperature ILSS values of Celion 6000/PMR-15 polyimide composites are in the 110 to 120 MPa range (ref. 7). The low ILSS values can at least partly be attributed to the high void content of the composites. The higher cure pressure of 13.78 MPa did not improve the composite properties. It can be speculated that cure temperatures higher than 371 °C may be necessary to achieve improved resin consolidation and to minimize the void content. Consequently, further experiments were performed, using final cure temperatures of 399 and 427 °C. The Tg values, void content, and short-term mechanical properties of the composites are shown in table IV. It can be seen that the void content of the two composites was lower than that of the composites cured at 371 °C (see table III). The values ranged from 4.6 to 5.0 percent, and are within the void content range expected for state-of-the-art condensation type polyimide composites. It is interesting to note that the Tg values of the composites appear to be a linear function of the final cure temperature, and are well above the potential 371 °C use temperature. The Tg values shown in table III and IV were achieved after a free-standing postcure in air at 371 °C for 24 hr. Postcure times beyond 24 hr did not further increase the Tg values. This is in agreement with the Tg data measured for neat resin specimens (see table II).

The room temperature mechanical properties are still somewhat lower than those of Celion 6000/PMR-15 composites, but exhibit significant improvement compared to those of the composite cured at 371 °C. The short-term 371 °C mechanical properties are also very respectable. Since this study represents only the first effort in composite fabrication using the PMDE-BDAF matrix resin, it is likely that further optimization of the cure cycle would result in a lower void content and improved mechanical properties.

Next the attention was focused on composite mechanical property retention after exposure at elevated temperatures. The two composites (cured at 399 and 427 °C) were exposed at 371 °C in flowing air at both atmospheric pressure and a pressure of 0.52 MPa. The weight loss characteristics of the composites are shown in figure 2. The exposure was terminated when the specimens exhibited a loss of 11 to 12 percent of their original weight. At this point there was considerable degradation of the composite surface, as exhibited by the presence of loose fibers. It can be seen that the rate of degradation for the specimens exposed at a pressure of 0.52 MPa was considerably faster than that at atmospheric pressure. The mechanical property retention characteristics of the composites after exposure in air at 371 °C at atmospheric pressure and a pressure of 0.52 MPa are shown in figures 3 to 5. Several points should be noted. At both air pressures the composite cured at 427 °C exhibited a lower level of ILSS (fig. 3) and flexural strength (fig. 4) throughout the exposure time compared to the composite cured at 399 °C. It is quite possible that some degradation of the polymer matrix occurs during curing at 427 °C, leading to chain scission and lowering of the mechanical properties of the composite. The flexural modulus values of both composites (fig. 5) were closely comparable at each exposure condition. Another point to be noted is that both composites exhibited a very good retention of their mechanical properties on exposure at 371 °C in air at atmospheric pressure. After 192 hr the composites retained 72 to 83 percent of their initial 371 °C ILSS, 70 to 71 percent of the flexural strength, and 85 to 88 percent of the initial flexural modulus values. When the composites were exposed at an air pressure of 0.52 MPa, the decrease of the mechanical properties paralleled the rapid rate of weight loss. After

72 hr of exposure at this condition, the composites exhibited a 50 to 65 percent retention of their original ILSS, a 60 to 64 percent retention of the flexural strength, and a 80 to 83 percent retention of the flexural modulus.

Based on the above composite evaluation studies, it appears that the PMDE-BDAF matrix resin is a viable candidate for composite applications requiring continuous exposure in air at 371 °C.

## EXPERIMENTAL

### Monomers

The monomers used in this study are shown in table I. The PMDE was synthesized according to the procedure outlined in reference 8. The synthetic scheme for the diamines used in this study is outlined in figure 1. A typical synthesis procedure is as follows: a three-neck flask equipped with a $N_2$ inlet, a magnetic stirrer, and a Dean-Stark trap was charged with 4,4'-dihydroxydiphenyl ether (20.2 g, 0.1 mole), N,N-dimethylacetamide (192 ml), toluene (75 ml), NaOH (9.0 g, 0.225 mole), and $H_2O$ (4 ml). The mixture was heated to reflux, and the $H_2O$ was removed by azeotropic distillation until the pot temperature reached 150 °C. Next, p-chloronitrobenzene (39.4 g, 0.25 mole) was added, and the mixture was stirred at gentle reflux for 24 hr. The mixture was cooled to room temperature and poured into crushed ice. The brown product was collected by filtration, washed with $H_2O$ and dried at 75 °C to yield 39.9 g of crude product. Crystallization from acetone gave 29.4 g (66 percent) of purified dinitro compound, mp 144 to 146 °C. A mixture of the dinitro compound (28.86 g, 0.065 mole), N,N-dimethylformamide (120 ml), and Raney Ni catalyst (approximately 5 g) were shaken in a Parr apparatus under 0.345 MPa of $H_2$ for 48 hr. The mixture was filtered, and the filtrate was warmed on a hot plate to 60 °C. Water was added to slight cloudiness, and the solution was cooled, then filtered to yield 11.60 g of diamine. The filtrate from this crop was worked up in the same manner to give a second crop of 11.40 g. Crystallization from acetone gave 20.4 g (82 percent) of purified diamine BDAO.

### Neat Polyimide Resin Disks

A stoichiometric mixture of PMDE and the diamine was dissolved in 4:1 acetone:N,N-dimethylformamide at a solids concentration of 40 wt %. The solvent was removed in a rotary evaporator, and the residue was staged for 0.5 hr at 175 °C to yield a molding powder. The molding powder (2 g) was placed in a cold circular matched metal die (diameter = 2.54 cm) and placed in a press preheated to 232 °C at contact pressure. When the die temperature reached 232 °C, a pressure of 13.78 MPa was applied, and the platen temperature increased to 399 °C. The pressure was released and reapplied when the die temperature was 260, 288, and 316 °C. When the die temperature had reached 399 °C, the mold was maintained at 399 °C and 13.78 MPa for 2 hr.

### Composite Fabrication

Prepregging solutions were prepared by dissolving a stoichiometric ratio of PMDE and BDAF in a 10:1 mixture of acetone:N,N-dimethylformamide calculated to yield a solids content of 40 wt %. Prepreg tapes were prepared by drum-winding and impregnating unsized Celion 6000 graphite fiber with the PMDE/BDAF solutions. The prepreg tapes were air-dried on the drum at ambient conditions for 18 hr. The prepreg was cut into twelve 10.16 by 10.16 cm plies and stacked unidirectionally into a matched metal die. The prepreg stack was staged for 1 hr at 120 °C. The molding cycle was identical to that used for neat resin specimens, except that the fabrication was carried out at three different final cure temperatures (371, 399, and 427 °C), and two pressures (10.34 and 13.78 MPa). The laminates were cooled to 204 °C or less prior to releasing the pressure and removal from the mold. The laminates were given a free-standing postcure in air at 371 °C by increasing the temperature from ambient to 371 °C at about 1.5 °C/min, then holding at 371 °C for 24 hr.

## Resin and Composite Testing

Glass transition temperatures were measured with a thermomechanical analysis (TMA) apparatus, using a penetration probe under a load of 5 g. Environmental exposure of the resins and laminates was conducted in a forced-draft oven at 371 °C with an air change rate of 100 $cm^3$/min. Laminate exposure was also conducted at 371 °C at a pressure of 0.52 MPa. Flexural strength tests were performed in accordance with ASTM D-790 using a three-point loading fixture and a span of 5.08 cm. The flexural property values were normalized to 60 vol % fiber. Interlaminar shear strength tests were performed in accordance with ASTM D-2344 at a constant span/depth ratio of 4. The fiber content was determined by $H_2SO_4/H_2O_2$ digestion.

## CONCLUSIONS

Based on the results of this study, the following conclusions can be drawn: it appears that the polyimide prepared from the dimethylester of pyromellitic acid (PMDE) and 2,2-bis[4-(4'-aminophenoxyl)phenyl]-1,1,1,3,3,3-hexafluoropropane (BDAF) has potential as a composite matrix resin for continuous exposure in air at 371 °C and atmospheric pressure up to 200 hr. However, its service life at 371 °C and elevated air pressures appears to be limited to 48 to 72 hr.

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TABLE I. - MONOMERS USED IN POLYIMIDE SYNTHESIS

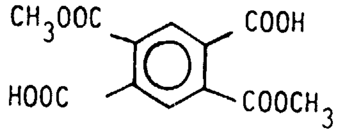
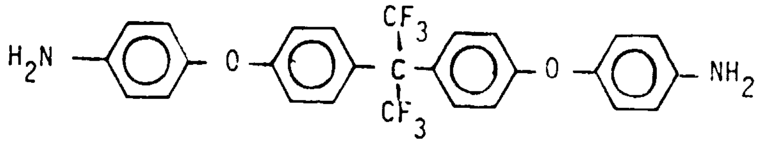
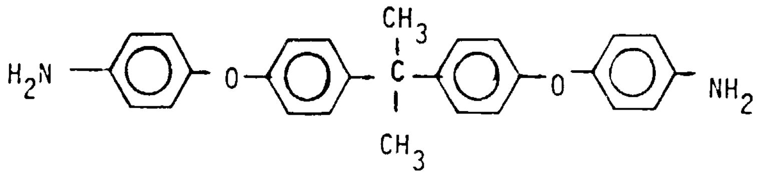
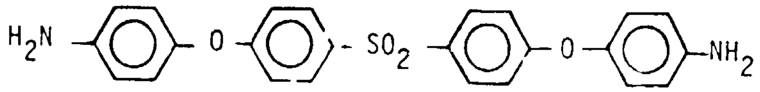
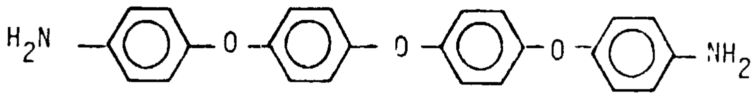
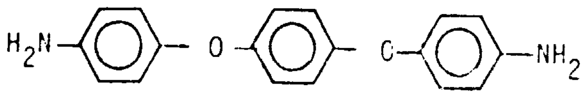
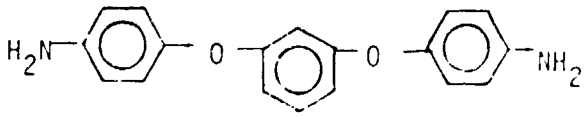
| Structure | Abbreviation |
|---|---|
|  | PMDE |
|  | BDAF |
|  | BDAP |
|  | BDAS |
|  | BDAO |
|  | PBAB |
|  | MBAE |

TABLE II. - PROPERTIES OF NEAT POLYIMIDE RESINS

| Diamine | Tg (°C) after postcure in air at 371 °C, hr | | | | Percent[a] weight loss |
|---|---|---|---|---|---|
| | 0 | 24 | 48 | 72 | |
| BDAF | 291 | 387 | 388 | 390 | 5.8 |
| BDAP | 270 | 330 | 334 | 328 | 9.0 |
| BDAS | 273 | 325 | 329 | 330 | 17.7 |
| BDAO | 279 | 318 | 317 | 319 | 9.4 |
| MBAB | 279 | 334 | 335 | 332 | 12.5 |
| PBAB | 305 | 357 | 360 | 360 | 19.9 |

[a] After 72 hr exposure in air at 371 °C.

TABLE III. - PROPERTIES OF CELION 6000/PMDE-BDAF POLYIMIDE COMPOSITES CURED AT 371 °C[a]

| Final molding pressure, MPa | Tg, °C | Void content, % | Interlaminar shear strength, MPa | |
|---|---|---|---|---|
| | | | 25 °C | 371 °C |
| 10.34 | 407 | 8.5 | 59.8 | 29.3 |
| 13.78 | 408 | 8.6 | 60.3 | 28.7 |

[a] Postcured for 24 hr in air at 371 °C.

TABLE IV. - PROPERTIES OF CELION 6000/PMDE-BDAF POLYIMIDE COMPOSITES[a]

| Final cure temperature, °C | Tg, °C | Void content, % | Interlaminar shear strength, MPa | | Flexural[b] strength, MPa | | Flexural[b] modulus, GPa | |
|---|---|---|---|---|---|---|---|---|
| | | | 25 °C | 371 °C | 25 °C | 371 °C | 25 °C | 371 °C |
| 399 | 422 | 4.6 | 83.3 | 40.7 | 1500 | 758 | 114 | 95.7 |
| 427 | 441 | 5.0 | 89.6 | 35.9 | 1420 | 620 | 117 | 100 |

[a] Postcured in air at 371 °C for 24 hr.

[b] Normalized to 60 vol % fiber.

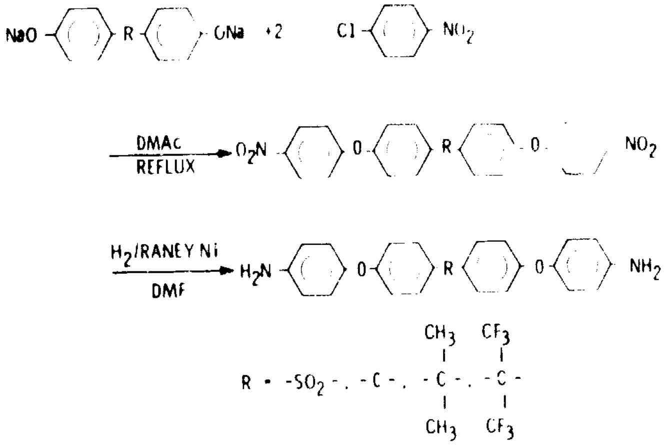


Figure 1. - Synthesis of flexibilized aromatic diamines.

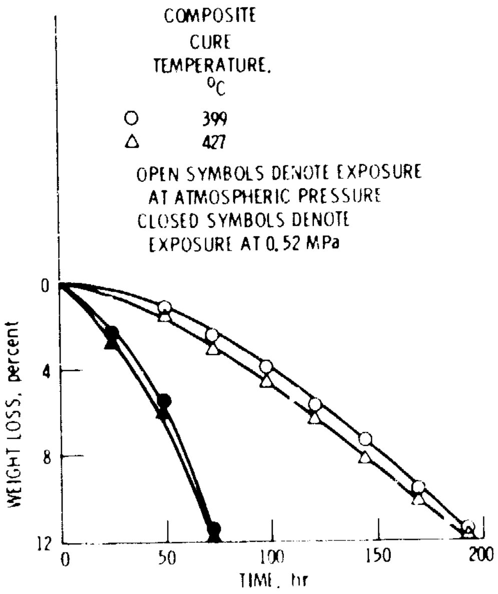


Figure 2. - Weight loss of Celion 6000/PMDE-BDAF composites exposed in air at 371 °C.

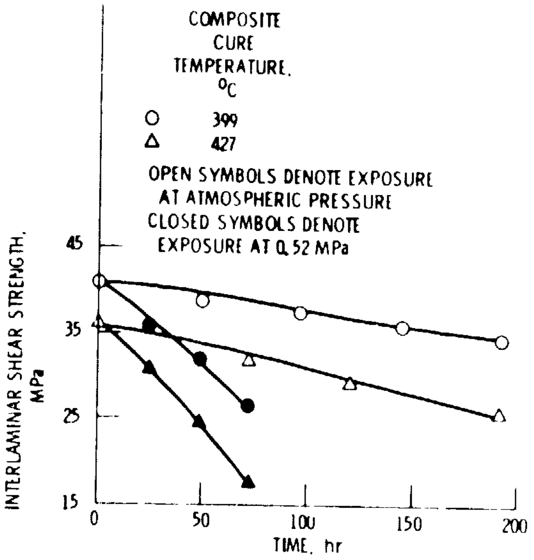


Figure 3. - Interlaminar shear strength of Celion 6000/PMDE-BDAF composites exposed and tested in air at 371 °C.

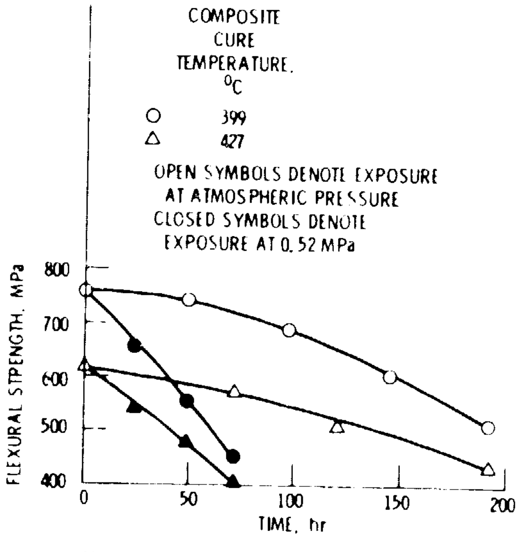


Figure 4. - Flexural strength of Celion 6000/PMDE-BDAF composites exposed and tested in air at 371 °C.

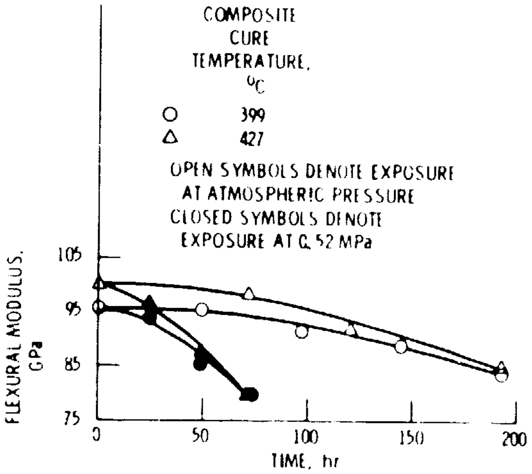


Figure 5. - Flexural modulus of Celion 6000/PMDE-BDAF composites exposed and tested in air at 371 °C.

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15. Supplementary Notes

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16. Abstract

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