CHARACTERIZATION OF RECENTLY DEVELOPED
HIGH TEMPERATURE POLYMERS: RADIATION
RESISTANCE OF POLYIMIDE AT ELEVATED
TEMPERATURES

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By

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INTRODUCTION

In recent years, several new polymers have been developed which have good mechanical properties at moderately high temperatures. If these materials also retain these properties in radiation environments, they could be applied in the design of nuclear reactors where only limited use of polymers is made at present. In this study, an analysis was made of the effect of gamma radiation on the mechanical properties at elevated temperatures of one of the new polymers, a glass-reinforced polyimide plastic.

SUMMARY AND CONCLUSIONS

No systematic effect was found from gamma radiation doses up to 10^9 roentgen on the modulus of elasticity or strength at constant test temperature. Heat treating (postcuring) of the polyimide before irradiation produced improved mechanical properties. The exclusion of air during irradiation to eliminate any oxidative degradation contributed no further improvement.

Therefore, the excellent mechanical performance of this plastic at elevated temperatures is maintained in radiation environments to doses of

at least 10^9 roentgen. This capability should permit the use of this polyimide, and perhaps other similar polymers, in nuclear reactor applications. A survey of such potential applications is recommended.

DISCUSSION

EXPERIMENTAL

Laminated panels were formed from a roll of du Pont's PI-2501 "B-staged" Pyralin polyimide glass fabric prepreg by cutting into 5-1/2 x 10 inch prepreg sheets, stacking the sheets 12 deep with the fabric warp direction of all sheets parallel, and curing the stack in a press at 57 psi and 177°C for five hours. The cured panels, with thickness of about 0.093 inch, were cut into 1 x 3 inch flexural test samples. The three-inch direction was the warp direction of the 181 style, A1100 finish, E glass fabric reinforcement.

The samples designated in Table 1 as "postcured" were heated in a circulating air oven for four hours each at 202°, 227°, 254°, 280° and 293°C, in that order. Samples then were irradiated in a ^{60}Co source at doses ranging from 1.0×10^6 to 6.4×10^9 roentgen. A few of the samples, as noted in Table 1, were irradiated in the absence of oxygen by sealing in ampoules filled with nitrogen.

Flexural tests were conducted in accordance with ASTM Standard Test D790-63 in a floor model Instron universal testing instrument. The samples were supported on two cylinders of 1/4 inch radius with a 2 inch span between cylinders. A third cylinder of 1/4 inch radius applied the test load from above the samples at the center of the 2 inch span. The loading rate was 0.05 inch per minute. Samples tested at the elevated temperatures of approximately 150° and 315°C were enclosed in an environmental chamber.

TABLE 1

FLEXURAL TEST RESULTS FOR GLASS-REINFORCED POLYIMIDE

<u>Test Temperature (°C)</u>	<u>Tensile Modulus of Elasticity (psi) x 10⁻⁶</u>			<u>Yield Strength (psi) x 10⁻⁴</u>		
	<u>25</u>	<u>150</u>	<u>315</u>	<u>25</u>	<u>125</u>	<u>315</u>
Not Postcured;						
Dose (R) = 0	3.95 ±.30	--	--	8.45 ±.73	--	--
1.0x10 ⁶	3.51 ±.22	3.22 ±.18	2.54 ±.16	7.42 ±.37	5.79 ±.47	3.32 ±.52
1.0x10 ⁷	3.51 ±.20	3.21 ±.28	2.58 ±.41	7.74 ±.27	5.93 ±.57	3.60 ±.72
1.0x10 ⁸	3.46 ±.29	3.33 ±.19	2.72 ±.30	7.49 ±.60	6.70 ±.48	3.93 ±.59
1.0x10 ⁹	3.85 ±.17	3.32 ±.40	2.21 ±.48	8.24 ±.55	6.27 ±.91	3.13 ±1.16
(a) 6.4x10 ⁹	--	3.57 ±.21	2.80 ±.24	--	7.66 ±.42	4.01 ±.59
Postcured;						
Dose (R) = 1.0x10 ⁷	--	3.74 ±.24	3.28 ±.33	--	7.27 ±1.31	4.42 ±.81
1.0x10 ⁸	--	3.80 ±.18	3.40 ±.25	--	7.52 ±.69	4.82 ±.68
1.0x10 ⁹	--	3.93 ±.23	3.14 ±.18	--	5.40 ±.87	4.49 ±.46
(a) 1.0x10 ⁹	3.70 ±.37	3.68 ±.17	3.14 ±.56	7.66 ±.45	7.21 ±.21	4.01 ±.26

(a) Irradiated in absence of oxygen.

FIGURE 1
Tensile Modulus of Elasticity of Glass-Reinforced Polyimide without Postcure after Various Gamma-Irradiation Doses

FIGURE 2
Yield Strength for Glass-Reinforced Polyimide without Postcure after Various Gamma-Irradiation Doses

FIGURE 3
Tensile Modulus of Elasticity of Glass-Reinforced Polyimide with Postcure after Various Gamma-Irradiation Doses

FIGURE 4
Yield Strength for Glass-Reinforced Polyimide with Postcure after Various Gamma-Irradiation Doses

Usually, five duplicate samples were tested at each selected combination of radiation level, test temperature, and postcuring; no sample was tested at more than one set of conditions. The yield strength and the tensile modulus of elasticity were calculated from the test results using equations 3 and 5, respectively, in the ASTM standard.

RESULTS

Sample Composition

The weight percent of glass reinforcement in the samples was determined by heating five weighed samples from three different panels to constant weight in a 600°C oven. The glass residue from each sample was weighed, giving the glass content as 83.1 ± 1.1 percent by weight.

Flexural Test Results

The tensile modulus of elasticity and yield strength calculated from the flexural test data are presented in Table 1 and Figures 1 to 4. The estimated standard deviation was also calculated and appears with the averages in the table and figures.

It may be seen from Figures 1 to 4 that a straight line of zero slope can be drawn through the modulus or strength data at each temperature such that the line falls within one standard deviation of each average data point with one exception. Thus, no systematic increase or decrease is apparent in either modulus or strength with increasing dose up to 10^9 roentgen.

In each figure, the decreasing modulus or strength with increasing test temperature is apparent. The relative decrease in strength is considerably more abrupt than the decrease in modulus with increasing temperature.

The effect of postcuring on the test results can be seen by comparing Figure 1 with Figure 3 and Figure 2 with Figure 4. Appreciable improvements in both modulus and strength at the various doses and test temperatures are obtained by postcuring.

To determine the effect of any oxidative degradation on the test results, oxygen was excluded during irradiation of some samples. The effect can be seen from Table 1 to be small. With no postcuring, modulus and strength values obtained with oxygen absent are slightly greater than values obtained with oxygen present at corresponding test temperatures. However, this is not true with postcured samples. Postcuring would be conducted in practice since it has been shown to give improved properties; thus, no additional advantage would be gained by excluding oxygen during irradiation.

Flammability Test Results

Flammability tests were conducted on two 1 x 3 inch samples in accordance with ASTM Standard Test D635-63. The samples were found to be self extinguishing by this test.

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