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IN SITU POLYMERIZATION OF MONOMERS FOR POLYPHENYLQUINOXALINE - GRAPHITE FIBER COMPOSITES

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16. Abstract

In situ polymerization of monomers was used to prepare graphite-fiber-reinforced polyphenylquinoxaline composites. Six different monomer combinations were investigated. Composite mechanical property retention characteristics were determined at 316° C (600° F) over an extended time period.

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SUMMARY

Methods currently used to prepare fiber-reinforced, high-temperature-resistant polyphenylquinoxaline (PPQ) composites employ extremely viscous, low-solid-content solutions of high-molecular-weight PPQ polymers. An improved approach, described in this report, consists of impregnating the fiber with a solution of the appropriate monomers instead of a solution of previously synthesized high-molecular-weight polymers. Polymerization of the monomers occurs in situ on the fiber during the solvent removal and curing stages. The in situ polymerization approach greatly simplifies the fabrication of PPQ - graphite fiber composites. The use of low-viscosity, monomeric-type solutions facilitates fiber wetting, permits a high solid content, and eliminates the need for prior polymer synthesis.

INTRODUCTION

Polyphenylquinoxalines (PPQ) are considered to have excellent potential for use as high-temperature-resistant matrix resins in advanced fiber-resin composites (ref. 1). However, because of processing problems, their potential as high-temperature matrix resins has not been realized. Current methods used

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to fabricate PPQ-fiber composites consist of impregnating the reinforcement with high-molecular-weight PPQ polymers dissolved in a solvent or solvent mixture containing m-cresol. Because of the inordinately high viscosity of these solutions, which results from both the high molecular weight of the polymer and the use of m-cresol, complete wetting of the fiber during impregnation is difficult to achieve. Prior approaches used to overcome the viscosity problem are (1) to limit the polymer molecular weight by unbalancing the stoichiometry of the system or (2) to use dilute solutions. Unbalancing the stoichiometry can adversely affect the polymer thermo-oxidative stability, and even dilute solutions of high-molecular-weight polymers are extremely viscous.

A new approach was developed to circumvent some of the composite processing problems associated with the use of PPQ polymers as the matrix material. The method used to prepare high-performance PPQ - graphite fiber composites consists of impregnating the fiber with a freshly made solution of the appropriate monomers instead of a solution of high-molecular-weight polymers. Impregnation of the fiber prior to appreciable polymerization completely eliminates the impregnation problems encountered with the use of high-viscosity, high-molecular-weight PPQ solutions. Note that the major part of the polymerization of the reactant mixture is conducted on the fiber during the solvent removal and final curing stages. Although at the time of impregnation the solution is not truly monomeric, we refer to this approach as the in situ polymerization of monomers because of its similarity to the monomeric reactant approach developed in our laboratories for A-type polyimides (ref. 2).

A screening study was made of six different PPQ - Hercules HMS graphite fiber composite systems fabricated by the in situ polymerization approach. Composite mechanical properties and thermo-oxidative stability characteristics were determined at 316° C (600° F) over an extended time period. Also presented are weight loss measurements of PPQ films fabricated by conventional polymerization procedures.

EXPERIMENTAL PROCEDURE

Monomers

The monomers used in this study are shown in table I. The monomers 3,3'-

diaminobenzidine (DAB), 3,3',4,4'-tetraaminobenzophenone (TABP), 1,4-bis(phenylglyoxalyl)benzene (PPGB), 1,3-bis(phenylglyoxalyl)benzene (MPGB), and 4,4'-oxydibenzil (ODB) were obtained from commercial sources and were used as received. The monomer 3,3',4,4'-tetraaminodiphenyl sulfone (TADPS) was prepared according to the method of Stille and Arnold (ref. 3), and 3,3', 4,4'-tetraaminodiphenylmethane (TADPM) was prepared essentially according to the method of Bell and Jewell (ref. 4).

Film Fabrication and Testing

The PPQ polymers were prepared according to the following representative procedure. A stoichiometric quantity of tetraketone was added during 5 minutes to a stirred slurry of the tetraamine in m-cresol. The solid content was in the range of 10 to 15 weight percent. The reaction mixture was stirred for 18 hours at room temperature to provide a viscous solution. Films were cast by doctoring the solution on a glass plate followed by drying in air at 70° C (158° F) for 4 hours, then in vacuum at 130° C (266° F) for 4 hours. The film thickness was in the range of 0.025 to 0.051 mm (1 to 2 mils).

Isothermal exposure of the PPQ films was performed at 316° C (600° F) in a circulating air oven. The air change rate was 100 cm^3/min. The film size was 5.08 cm by 5.08 cm (2 in. by 2 in.).

Composite Fabrication and Testing

Solutions of PPQ systems I to VI (table II) were prepared by separately dissolving stoichiometric quantities of each monomer in N-methylpyrrolidone (NMP) at a solid content of 25 to 35 weight percent. The solutions were then combined, stirred for 30 seconds, and applied within 1/2 hour to drum-wound Hercules HMS graphite fiber. The impregnated fibers (prepregs) were then dried with heat lamps as the drum was rotated to yield tape with a volatile content of approximately 28 weight percent. The quantities of fiber and solution were calculated to yield a composite containing 40-weight-percent resin. The volatile content was determined by placing a 7.62-cm by 7.62-cm (3-in. by 3-in.) section of dried impregnated tape into a 316° C (600° F) oven for 30 minutes and then measuring the weight loss. At a volatile content of 28 percent, the tapes were flexible but not tacky. The tapes were then removed from the drum, cut, stacked unidirectionally 8 to 12 plies thick, and wrapped in 0.051-mm (2-mil) thick stainless-steel foil. Composites were then molded by placing the wrapped stack into a preheated matched metal die in a press also heated to the desired cure temperature. After a dwell time of 45 to 130 seconds, pressures ranging from 3.5 to 6.2 MN/m^2 (500 to 900 psi) were applied for 30 to 60 minutes. In most instances, the composites were given elevated-temperature postcures without applied pressure.

Flexural strength tests were performed by using a three-point loading fixture with a fixed span of 5.1 cm (2 in.). The specimen size was 0.635 cm by 6.03 cm (1/4 in. by $2\frac{3}{8}$ in.). The thicknesses of the laminates ranged from 0.18 cm to 0.23 cm (0.07 in. to 0.09 in.). The resultant span-depth ratio ranged from 22 to 28. The rate of center loading for flexural testing was 0.127 cm/min (0.05 in./min). The interlaminar shear strength tests were conducted at a constant span-depth ratio of 5 by using the interlaminar shear test fixture described in reference 5. Elevated-temperature tests were performed in an environmental heating chamber. For the flexural and shear tests the load was applied after a 15-minute soak at 316° C (600° F). Composite weight loss was determined by using the apparatus and conditions previously described for the PPQ film. Generally, specimens were tested in triplicate.

RESULTS AND DISCUSSION

The monomer combinations used for the fabrication of PPQ - graphite fiber composites are shown in table II. The synthesis of PPQ polymers from several of these monomer systems and the properties of these polymers have been reviewed (ref. 6). In the prior studies the polymers were synthesized by using carefully controlled laboratory procedures. Polymers from monomer systems I and VI have also been evaluated as matrix resins in graphite fiber composites (ref. 1). Some difficulty has been experienced in obtaining void-free composites when solutions of high-molecular-weight PPQ polymers were used. The processing difficulties could be attributed to a number of factors, such as the inability to achieve complete fiber wetting or to remove completely the m-cresol - xylene solvent mixture. In order to eliminate these processing problems, we used the approach of conducting the polymer synthesis step on the surfaces of the reinforcing fibers. In this approach a low-viscosity solution of monomer mixtures in N-methylpyrrolidone (NMP) was used as the preimpregnation varnish. Because of the fast reaction rate of the tetraamine-tetracarbonyl monomer system, some reaction occurs immediately upon mixing the two separate monomer solutions. The pot life of the varnish is limited by the solubility of the reaction products, perhaps oligomers, in the NMP solvent. The solution, however, has sufficient pot life for use as a preimpregnation varnish.

The conventional fabrication method employs m-cresol because high-molecular-weight PPQ polymers remain soluble in this solvent. Apparently, because of the formation of polymer-solvent complexes, the m-cresol serves as an internal plasticizer, providing flow during processing. Consequently, the m-cresol is difficult to remove during precuring. It therefore volatilizes during the final cure, producing voids. The voids in composites prepared from high-viscosity polymer solutions can also be attributed to incomplete fiber wetting during impregnation, combined with little or no resin flow after the precure step.

The various monomer combinations shown in table II were selected on the basis of processability without sacrificing the thermo-oxidative stability of the PPQ resin.

Isothermal exposure of PPQ films was performed in air at 316° C (600° F) to obtain an estimate of polymer thermo-oxidative stability. The isothermogravimetric analysis (ITGA) results for the films are shown in figure 1. The data shown in the figure were obtained on PPQ films cast from m-cresol solutions of high-molecular-weight polymers. The m-cresol solutions were used because of the ease of film preparation. The curves shown in figure 1 do not take into account the initial weight loss experienced by the films during the first hour of exposure at 316° C (600° F). The investigators of reference 1 attribute the initial weight loss to volatilization of retained solvent. The film weight loss after the 1-hour exposure is given in table III. These weight losses also point out the difficulty in completely removing the m-cresol. Figure 1 shows that polymers I and II lost considerably less weight after 600 hours than polymers III to V. Films of polymer VI were reported to exhibit a thermo-oxidative stability comparable to that of polymer I (ref. 1).

As previously mentioned, ITGA of polymer films provides only a "best first guess" of polymer thermo-oxidative stability. Therefore, composites were fabricated and evaluated from Hercules HMS graphite fiber and all six monomer systems. Solutions of monomers in NMP having a solid content of 25 to 35 weight percent were used. All six monomer combinations provided low-viscosity solutions that possessed an adequate pot life, ranging from about 1/2 hour for system VI to more than 7 hours for system I. Excellent to good fiber wetting was obtained with systems I to IV. Void-free composites resulted from systems I to III. System V provided fair wetting, while system VI was rated as poor. As used in this investigation, the relative ratings of fiber wetting were determined by microscopic examination of sectioned composite specimens. It is possible that some of the observed voids attributed to poor wetting might have resulted from volatilization of entrapped solvent.

Figure 2 compares photomicrographs of cross-sectioned composites obtained from systems I and VI. The photomicrographs clearly show that a void-free composite was obtained from system I. Photomicrographs of composites from systems II and III were very similar to that of system I. Several attempts to improve the quality of composite system VI were unsuccessful. Difficulty was also encountered in the fabrication of composites from system V. Monomer systems V and VI are highly reactive; and it is possible that, upon mixing the monomer solutions, oligomers were formed that were unable to penetrate the fiber bundle. It must be emphasized that the overall processability of all the PPQ monomer-fiber systems was considerably better than that obtained by conventional methods, which employ solutions of high-molecular-weight polymers. It is entirely possible that further improvement in solvent-monomer systems, in impregnation techniques, and so forth, would result in void-free composites for all of the systems.

Table IV indicates the processing parameters that were selected after preliminary experimentation. The cure temperatures were selected to be higher than available published glass transition temperature T_g values (ref. 7) for each polymer system. The dwell time given for each system is the time interval required for complete evolution of volatile material. During the dwell time a cloud of vapor was observed. Means are currently being developed to utilize instrumental methods to establish the proper dwell time. As shown in table III, the cure pressures ranged from 3.5 to 6.2 MN/m^2 (500 to 900 psi), and cure times from 30 to 60 minutes. The composites were given an additional 60-minute cure at 371° C (700° F) at the cure pressure.

Preliminary evaluation of composite properties at 316° C (600° F) indicated that elevated-temperature postcuring was required to decrease thermoplastic behavior at elevated temperatures. Thermoplasticity was manifested as continued yielding of the composite without failing during application of load. After some experimentation, the postcuring cycle shown in table V was adopted.

The room-temperature properties and the short-time 316° C (600° F) properties of the postcured composites are shown in table VI. The data show that, even after the extensive postcuring cycle, composite systems II, III, and VI exhibited thermoplastic behavior at 316° C (600° F). In addition to the flexural strength values, the failure mode resulting from the three-point flexural test is indicated. In the room-temperature flexural test, composite system II exhibited a tension-type failure, which is responsible for its higher flexural strength. All other flexural test specimens, tested at room temperature or at 316° C (600° F), exhibited compression-type failure, indicating that the resin matrix did not completely transfer the applied loads to the fiber reinforcement. Except for composite system II, which had a fiber translation efficiency (FTE) of 96 percent based on a nominal fiber tensile strength of 2.07 GN/m^2 (300 ksi), the composites exhibited FTE values of approximately 75 percent. The 316° C (600° F) retention of flexural strength ranged from 22 percent for system III to 68 percent for system IV.

The specimens tested for interlaminar shear strength did not exhibit shear failures. Instead, they exhibited a bearing type of failure. The interlaminar shear strength, therefore, is at least equal to or greater than the interlaminar shear stress values given in table VI. Even after the thermal postcuring, composite systems II, III, and VI displayed thermoplasticity at 316° C (600° F). The thermoplastic nature of systems II and III can perhaps be attributed to relatively low glass transition temperature T_g values for the resin matrix. This argument, however, is not valid for system VI, because the T_g value for the polymer resulting from the DAB/PPGB combination is 372° C (702° F) (ref. 7). The T_g value for the TABP/PPGB polymer used in system I is appreciably lower, 327° C (619° F), yet composite system I did not exhibit thermoplasticity at 316° C (600° F).

Data are not presented for system V in table VI because of previously discussed fiber wetting problems in the fabrication of composites. Similar problems were encountered with system VI; therefore, investigation of composite systems V and VI was terminated.

Figure 3 shows the variation of composite weight loss, flexural strength, and interlaminar shear strength as a function of time in air at 316° C (600° F). Also given in the figure are the room-temperature mechanical properties. All data points are an average of three samples except the room-temperature flexural strength for system II, which is an average of two samples. Figure 3 shows that the composite weight retention ranged from good to poor. A correlation between weight loss for the composites and weight loss for the corresponding PPQ films was not apparent.

The results of flexural tests after isothermal exposure in air at 316° C (600° F) (fig. 3) showed that composite systems I and II exhibited good retention of their short-time 316° C (600° F) strength. System III exhibited an increase of flexural strength after 100 hours of exposure. This could be attributed to further postcuring during the exposure. The flexural strength of system IV decreased rapidly during exposure. Microscopic examination of sectioned specimens of system IV showed the presence of interply voids rather than the intraply voids which would have resulted from poor fiber wetting. The formation of interply voids is attributed to degradation of the polymer during the elevated-temperature cure.

The interlaminar shear data (fig. 3) show excellent retention of short-time 316° C (600° F) values on exposure at 316° C (600° F) in air. Composite system III lost its thermoplasticity somewhere between 0 and 100 hours of exposure. Composite system II lost its thermoplasticity between 100 and 200 hours. The interlaminar shear strength test was conducted in a manner which precludes taking a thermoplastic specimen to failure. Therefore, no failure points for composite systems II and III are presented before 200 and 100 hours, respectively.

The weight loss and mechanical property tests were discontinued at various times at which appreciable surface degradation of the resin matrix had occurred, as indicated by the appearance of loose fibers.

Of all the monomer combinations investigated in this study, composite system I (TABP/PPGB) displayed the best overall performance. In fact, the data obtained in this investigation using the in situ polymerization of monomers approach are significantly better than the data reported in reference 1, which used conventional methods for a PPQ copolymer - HMS fiber composite. The composition of the copolymer was TABP, DAB, and PPGB in a molar ratio of 3: 1: 4. This particular combination was selected as the PPQ polymer which had the best balance of processability and thermo-oxidative stability. Our results show that the in situ polymerization of monomers approach can be used to fabricate PPQ-fiber composites. However, considerable work still must be done to eliminate thermoplasticity of PPQ composites without using elevated-temperature postcuring cycles. In addition to introducing cross-links, the elevated-temperature postcuring undoubtedly causes degradation of the resin matrix or introduces reactive sites for subsequent degradation. Cross-links introduced by chemical reactions at milder conditions would eliminate resin degradation resulting from exorbitantly high postcuring temperatures.

CONCLUSIONS

Based on the results of this preliminary investigation, the following conclusions can be drawn:

1. The in situ polymerization approach greatly simplifies the fabrication of polyphenylquinoxaline (PPQ) - graphite fiber-reinforced composites. This approach eliminates the need for polymer synthesis, improves fiber wetting, and results in an overall improvement in processability. Our results show that the PPQ monomer combination which provided the best performance at 316° C (600° F) in air was 3,3',4,4'-tetraaminobenzophenone/1,4-bis(phenylglyoxalyl) benzene (TABP/PPGB).

2. Further work must be done to eliminate the high-temperature thermoplasticity of PPQ polymers by introducing cross-linking by chemical means rather than by thermal means during elevated-temperature postcuring.

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, June 20, 1974,
501-21.

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TABLE I. - MONOMERS USED FOR POLYPHENYLQUINOXALINE (PPQ) SYNTHESIS

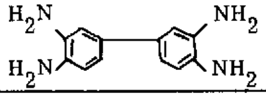
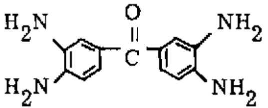
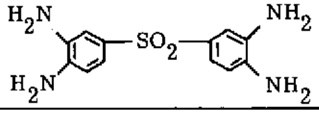
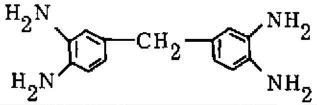
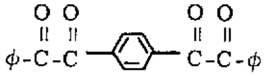
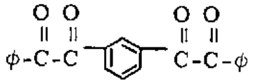
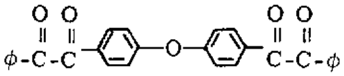
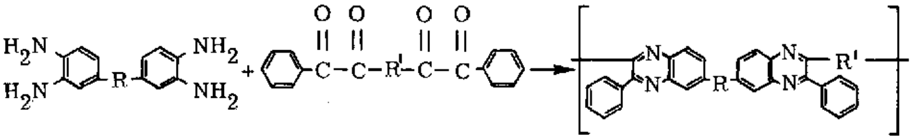
Structure	Name	Abbreviation
	3, 3'-Diaminobenzidine	DAB
	3, 3', 4, 4'-Tetraaminobenzophenone	TABP
	3, 3', 4, 4'-Tetraaminodiphenyl sulfone	TADPS
	3, 3', 4, 4'-Tetraaminodiphenylmethane	TADPM
	1, 4-Bis(phenylglyoxalyl)benzene	PPGB
	1, 3-Bis(phenylglyoxalyl)benzene	MPGB
	4, 4'-Oxydibenzil	ODB

TABLE II. - POLYPHENYLQUINOXALINE (PPQ) SYSTEMS INVESTIGATED



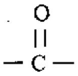
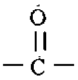
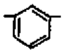
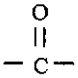
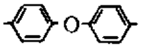
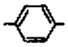
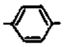
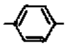
PPQ system	Monomers	R	R'
I	TABP/PPGB	 $-\overset{O}{\overset{\|}{C}}-$	
II	TABP/MPGB	 $-\overset{O}{\overset{\|}{C}}-$	
III	TABP/ODB	 $-\overset{O}{\overset{\|}{C}}-$	
IV	TADPS/PPGB	$-SO_2-$	
V	TADPM/PPGB	$-CH_2-$	
VI	DAB/PPGB	Nil	

TABLE III. - WEIGHT LOSS OF POLYPHENYLQUINOXALINE (PPQ) FILMS EXPOSED IN AIR AT 316° C (600° F) FOR 1 HOUR

PPQ system	Monomers	Film weight loss, percent
I	TABP/PPGB	5.8
II	TABP/MPGB	8.4
III	TABP/ODB	9.8
IV	TADPS/PPGB	7.7
V	TADPM/PPGB	6.2

TABLE IV. - SUMMARY OF POLYPHENYLQUINOXALINE (PPQ) - GRAPHITE FIBER COMPOSITE PROCESSING CONDITIONS

PPQ system	Monomers[a]	Volatile content,[b] wt %	Fiber content,[c] vol %	Cure parameters[d]					
				Cure temperature		Dwell time, sec	Cure pressure		Cure time, min
				°C	°F		MN/m^2	psi	
I	TABP/PPGB	28	54.0	330	625	90	6.2	900	60
II	TABP/MPGB	25	50.0	330	625	60	6.2	900	60
III	TABP/ODB	28	52.4	321	610	90	5.5	800	30
IV	TADPS/PPGB	22	55.0	343	650	45	4.1	600	30
V	TADPM/PPGB	30	----	330	625	105	3.5	500	30
VI	DAB/PPGB	36	51.0	382	720	130	6.2	900	60

[a] Solid content of impregnating solutions, 25 to 35 wt %.

[b] Volatile content of impregnated fiber before curing.

[c] Resin content determined by H_2O_2/H_2SO_4 digestion.

[d] All laminates except composite system VI were subjected to additional curing for 60 min and 371° C (700° F) at cure pressure.

TABLE V. - SCHEDULE FOR ELEVATED-TEMPERATURE POSTCURING IN AIR

Time at temperature, hr	Postcuring temperature	
	°C	°F
2	200	392
2	225	437
2	250	482
2	300	572
2	325	617
2	350	662
2	371	700

TABLE VI. - MECHANICAL PROPERTIES OF POLYPHENYLQUINOXALINE (PPQ) - GRAPHITE FIBER COMPOSITES AT ROOM TEMPERATURE AND 316° C (600° F)

Composite system	Test temperature		Flexural strength		Flexural modulus		Interlaminar shear stress at failure	
	°C	°F	MN/m^2	ksi	GN/m^2	psi	MN/m^2	ksi
I	(a)	(a)	847	123	143	[b]20.7×10^{6}	52.4	7.6
	316	600	482	70	70	[b]10.2	28.3	4.25
II	(a)	(a)	985	143	142	[c]20.6	55.1	8.0
	316	600	476	69	67.5	[b]9.8	(d)	(d)
III	(a)	(a)	840	122	137	[b]19.8	58.6	8.5
	316	600	182	26.5	(d)	(d)	(d)	(d)
IV	(a)	(a)	785	114	146	[b]21.2	32.6	4.7
	316	600	544	79	91	[b]13.2	27.0	3.9
VI	(a)	(a)	---	---	---	----	24.8	3.6
	316	600	---	---	---	----	(d)	(d)

[a]Room temperature.
[b]Failed in compression.
[c]Failed in tension.
[d]Thermoplastic.

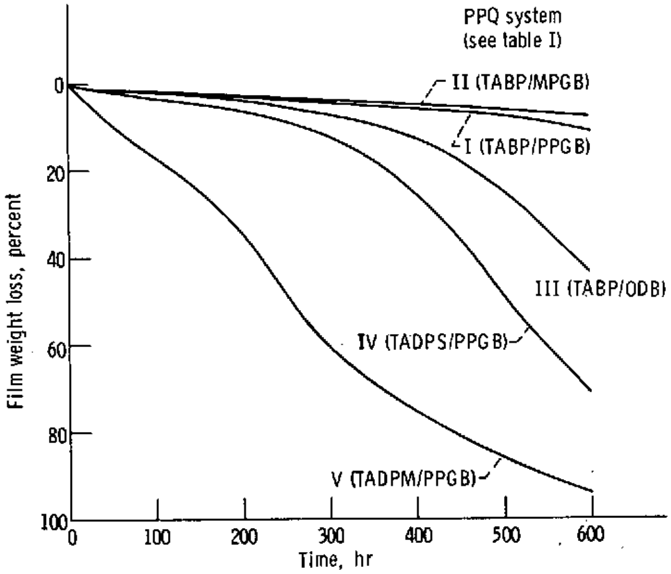
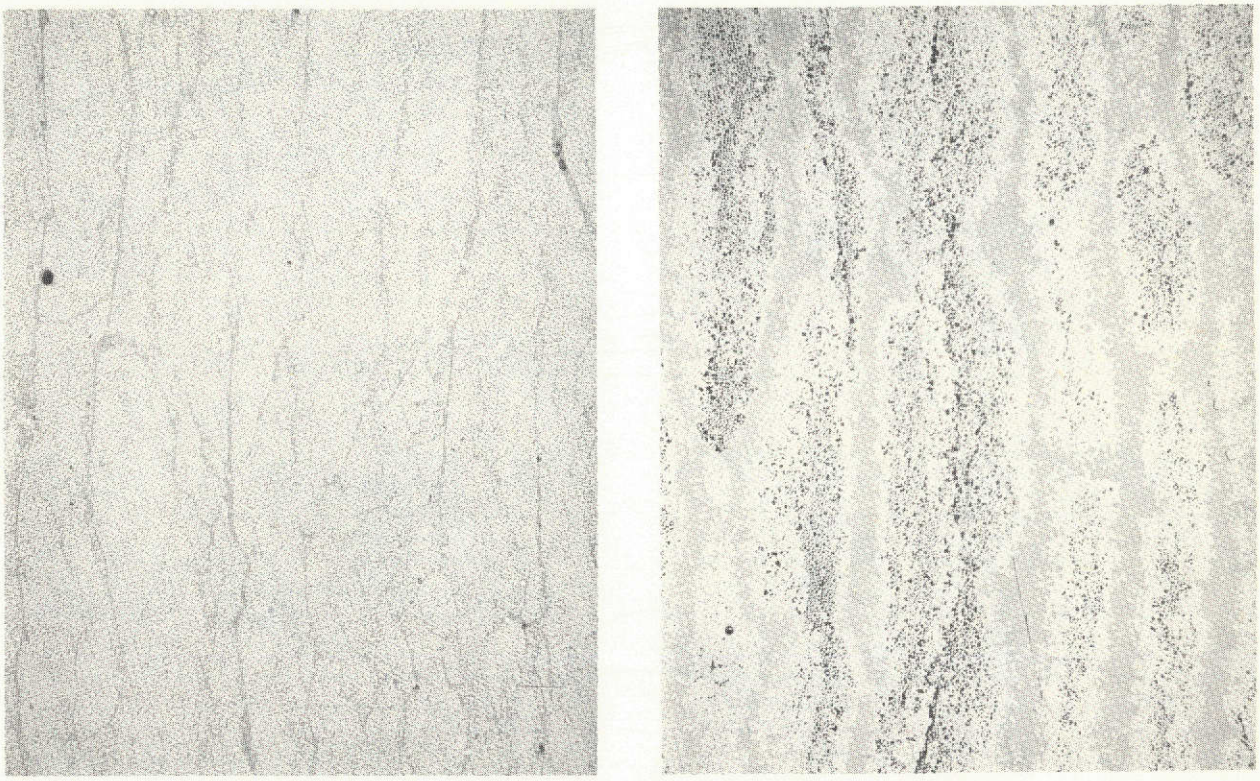


Figure 1. - Weight loss of polyphenylquinoxaline (PPQ) films exposed in air at 316° C (600° F). (PPQ films cast from high-molecular-weight polymers.)



I VI

Figure 2. - Photomicrographs of polyphenylquinoxaline(PPQ) - graphite-fiber composite systems I and VI. X50.

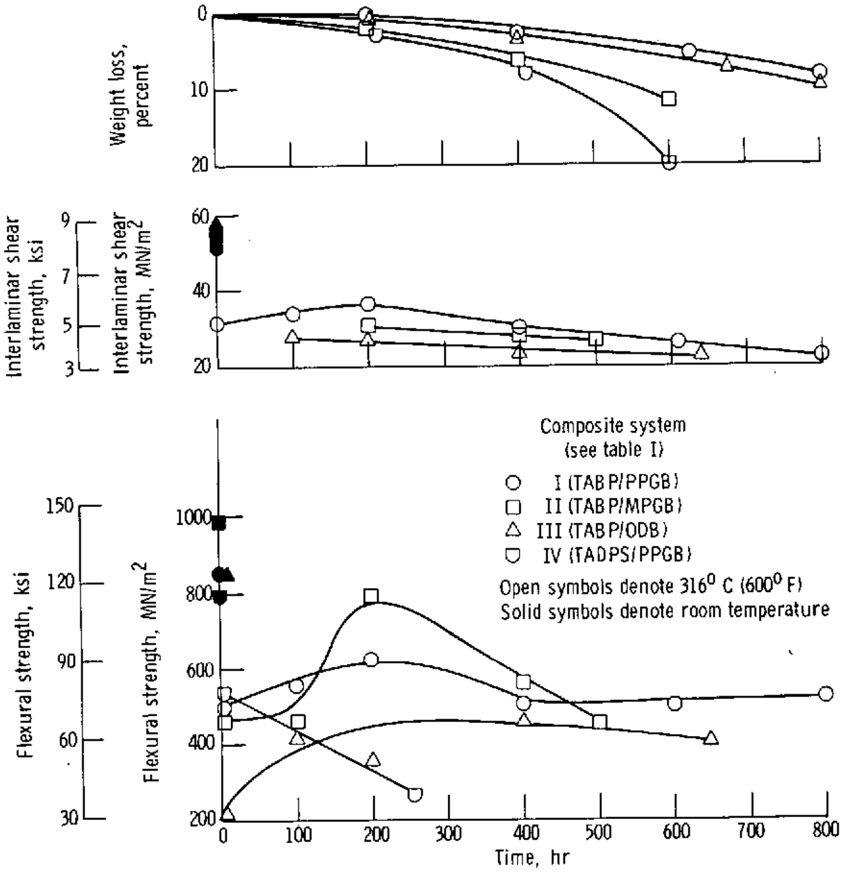


Figure 3. - Properties of polyphenylquinoxaline (PPQ) - graphite fiber composites as a function of exposure time at 316° C (600° F).

NASA-Langley, 1974