JOURNAL OF APPLIED POLYMER SCIENCE VOL. 13, PP. 2045-2056 (1969)

The Effect of Impulsive Preloadings on the Behavior of Quartz Phenolic

R. D. PERKINS, S. G. BABCOCK, F. L. SCHIERLOH, and A. H. JONES, *Manufacturing Development, Materials and Structures Laboratory, General Motors Corporation, Warren, Michigan*

Synopsis

Uniaxial strain and uniaxial stress preloadings were performed on quartz phenolic in order to assess the effect of an impulsive preload on the subsequent uniaxial stress behavior of a composite material. In these tests observable damage was limited to the phenolic matrix. As a result, postpreload experiments on the specimen under uniaxial stress correlated the dependence of the fracture strength with the extent of the damage to the phenolic matrix as well as the direction of the reload relative to the fiber layup.

INTRODUCTION

Fiber-reinforced composite materials achieve their strength by the reinforcement of a low modulus matrix with high modulus, high strength fibers. Under load these materials have complex local stress distributions which produce local failures. Two possible types of failure are cracking of the matrix from the local stresses and debonding between the matrix and the fiber bundles from the high interfacial shear stresses.

In order to predict the capability of a composite material to withstand a given loading, it is necessary to determine the extent to which a previous loading affects the load-carrying ability of the material by microcracking and debonding. The study reported here concentrated on the evaluation of the degradation of a fiber-reinforced composite material (quartz phenolic) after a uniaxial strain shock passage or a uniaxial stress loading to a known stress state. (The quartz phenolic was compression molded by Reflective Laminates, Inc. from J. P. Stevens Astroquartz 581 fibers and Monsanto SC1008 phenolic resin.) Experiments were conducted at 23°C and 315°C in order to determine the combined effects of elevated temperatures and preloads. The damage was assessed through uniaxial stress tests to failure and photomicrographic analysis.

EXPERIMENTAL TECHNIQUES

Uniaxial Strain Preshock

The experimental arrangement, schematically shown in Figure 1, consisted of two right circular cylinders 0.478 cm in diameter and 0.635 cm

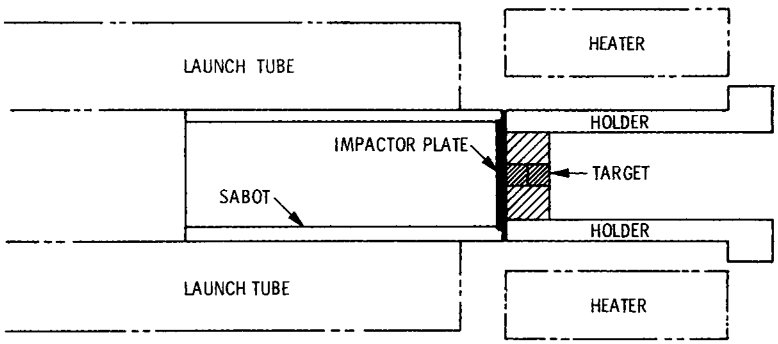

Fig. 1. Schematic diagram of preshock experimental techniques.

long. The first was the specimen to be shocked and the second was the momentum trap to keep reflected tensile waves from entering the specimen. Damage from edge rarefactions was prevented by placing the small cylinders within an annular ring of 2.54 cm outside diameter and 0.478 cm inside diameter. All mating surfaces between the three parts composing the target were joined by thin epoxy layers which formed a zero tensile strength interface. By using the same material for the specimen and the momentum traps, the production of unknown waves at the various interfaces was minimized.

The assembled target was mounted into a steel holder which not only provided for alignment of the target face with the impactor face but also provided for target recovery. The inside diameter of the holder was just slightly larger than the target diameter, thereby limiting the amount of tumbling of the target after preshocking.

Two types of impactors were used in this study, one of 4340 steel (R_c 38–42) and one of material identical to that of the target; the impactors were 2.54 cm in diameter and 0.25 cm thick. The transit time for a wave in the impactor to propagate from the front face, reflect from the rear face, and return to the front face was 0.87 microsec for a steel impactor and about 1.5 microsec for the composite impactor. The impactors were mounted in 3.8 cm diameter light-weight Plexiglas tubular sabots which were filled with very low impedance foam. The backs of the sabots were closed with a Plexiglas plate. The saboted impactor was launched toward the target from a compressed gas gun of 3.8 cm bore and 2.5 meters long. The velocities were measured by a photomultiplier velocity system during the last 15 cm of travel in the launch tube before entering the impact chamber. This chamber, which contains the specimen during impact, was evacuated to about 2 mm Hg prior to each test.

Specimens were preshocked at elevated temperature by inserting the entire specimen and holder into a radiantly heated oven. The oven used four 500-watt quartz-iodine lamps to bring the specimen to temperature. Temperature differences through the target were found to be less than 5°C; therefore, the test temperature was measured by a single thermocouple mounted on the rear of the target. The specimen was brought to temperature slowly over a period of about 15 min; after preshock the specimen was allowed to cool in the target chamber for another 15 min.

The specimen under shock conditions is in a state of uniaxial strain. In terms of stress, the specimen is loaded in a triaxial state of stress, i.e., a hydrostatic component together with deviatoric stresses whose upper limits are determined by the shear strength of the material. From the equation of state of the target[1] and the impactor materials[1,2] and the impact velocity, the magnitude of the shock pulse may be determined through the "impedance matching" method.[1,3] Unfortunately, no equation of state is available for this composite at elevated temperatures. Therefore, all shock pressures were calculated using the room temperature data.

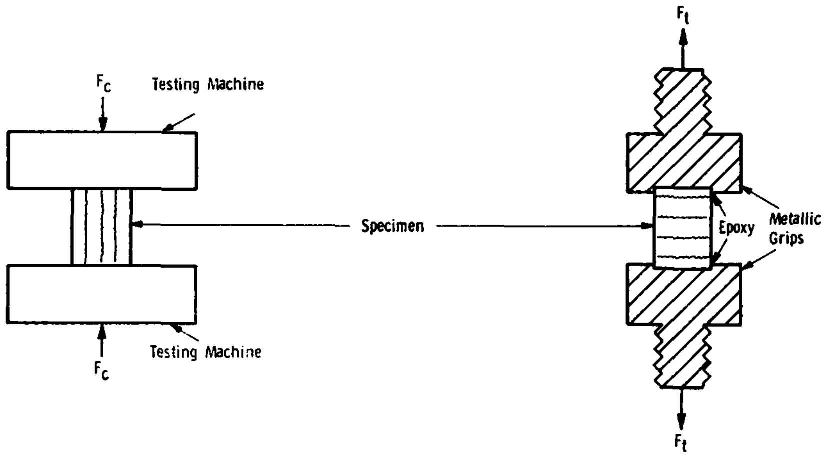

Fig. 2. Testing of preshocked specimens.

After the target had been shocked, the fracture strength of the material was obtained. Samples with fiber layup parallel to the specimen axis were tested in uniaxial compression, as shown in Figure 2. Samples with fiber layup perpendicular to the specimen axis were tested only in uniaxial tension by bonding the samples with epoxy to metallic grips, as shown in Figure 2.

Uniaxial Stress Preload

Specimens were subjected to uniaxial stress preloads in the Medium Strain-Rate Machine (Fig. 3) which was developed at General Motors to enable the testing of various materials from ductile plastics to brittle ceramics as well as high-strength metals and composites. This device is similar in principle to machines developed by Clark and Wood[4] and Campbell and Marsh.[5] However, the present device has undergone extensive modifications in order to improve the accuracy of the experimental techniques as well as to accommodate a wide variety of materials.[6] De-

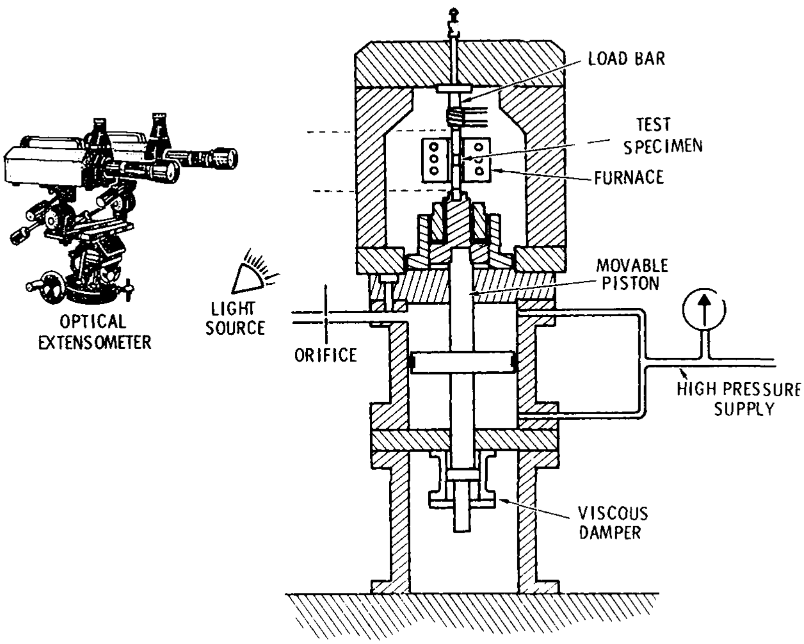

Fig. 3. Medium Strain-Rate Machine.

pending upon the size and strength of the specimen, strain rates from 10^{-3} to about 10^{2}/per sec are achievable with this device.

For elevated temperature testing, specimens were heated in air by a furnace, similar to the one used in the uniaxial strain work, attached to the Medium Strain-Rate Machine. In order to eliminate potential problems induced by thermal cycling and minimize the effects of time-at-temperature, specimens were heated to 315°C in approximately 7 min, loaded to a strain level below fracture, unloaded, and then reloaded to fracture without variation in temperature. The total length of time to perform the experiment was approximately 10 to 12 min. Other specimens under identical conditions were fractured on the first loading. Stiffness and strength were compared for the two loading conditions to assess the preload damage.

As an assist to determining preload damage, specimens subjected to initial loadings below ultimate fracture strain and then removed from the machine without reloading were sectioned and photomicrographically analyzed. Correlation was then attempted between micrographic observations and data from identical specimens loaded a second time to fracture.

DISCUSSION OF RESULTS

Uniaxial Strain Preshock

Photomicrographic studies of a composite material shocked in a previous study[7] showed that damage was limited to cracking of the matrix. There was no evidence of fiber mat damage. Furthermore, the matrix cracking was found to be independent of the shock direction with respect to the fiber layup. In the present tests, therefore, the conditions in which the fiber mats dominate the material behavior were neglected in favor of more thorough studies of the limiting cases: (1) tension with fiber layup normal to the specimen axis where the resin matrix determines the tensile strength; and (2) compression with fiber layup parallel to the specimen axis where the resin matrix helps distribute the load between the fiber mats.

The experimental results obtained from the study of the preconditioning effects by shocking on quartz phenolic are shown in Tables I and II. These tables contain data from uniaxial stress loadings for the conditions enumerated above. Throughout the collected data, variations in fracture stress (maximum of ±10%) for a given condition were found. These variations are a result of partial failure of the wave traps, damage during recovery, material scatter, and loading variations. (Note that the specimens were tested directly after loading without remachining of the end surfaces.)

TABLE I

Quartz Phenolic—Fiber Layup Parallel to Specimen Axis

Temp., °C	No. of tests	Impactor material	Av. preshock stress, kb	Average uniaxial compressive strength, kg/cm²	% Difference from room temp. norm, °C	Group
23	9	none	0	4.53×10^3	0	a
315	3	none	0	3.93×10^3	−13	b
23	3	4340	14.3	2.43×10^3	−46	c
315	2	quartz phenolic	16.5	1.53×10^3	−66	d
23	5	quartz phenolic	16.3	3.79×10^3	−16	e
23	5	4340	20.2	1.79×10^3	−60	f

TABLE II

Quartz Phenolic—Fiber Layup Normal to Specimen Axis

Test temp., °C	No. of tests	Impactor material	Av. preshock stress, kb	Av. uniaxial tensile strength, kg/cm²	% Difference from room temp. norm, °C	Group
23	2	none	0	2.49×10^2	0	a
23	4	4340	4.1	3.08×10	−87	b
23	2	quartz phenolic	3.7	1.27×10	−94	c
315	3	4340	4.4	4.63×10	−81	d

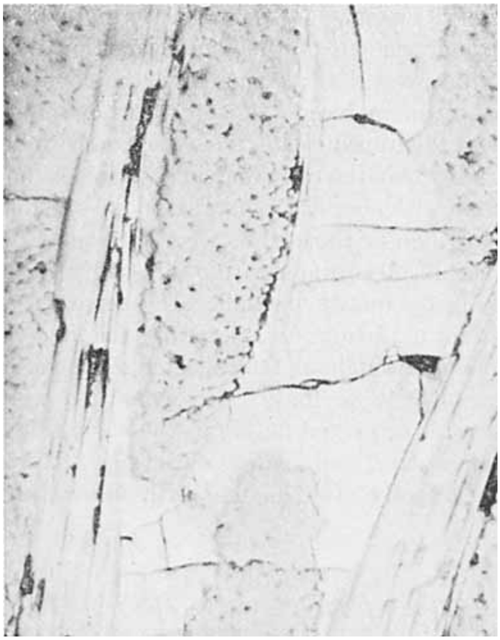

Fig. 4. Photomicrograph showing cracks in resin matrix in preshocked quartz phenolic.

Table I contains the results on material with loading parallel to the fiber layup. For this condition, two sets of control samples were tested. One set was tested as machined from bulk material. The other set was preheated to 315°C over a period of 15 min, then oven cooled in order to simulate only the temperature effects on the specimens preshocked at temperature. This simulation was necessary as micrographic studies showed that this type of temperature cycling tended to crack the phenolic, thereby degrading the material's mechanical strength much like the effects of a preshock (Fig. 4).[3]

It is seen from column 6, Table I, that two sets of conditions produce similar degradation, namely, elevated temperature control samples and the specimens shocked to about 16 kilobars at room temperature with a quartz phenolic impactor. The former group, (b), exhibited about -13% deviation from original strength and the latter group, (e), about -16%. The second set are targets shocked at 315°C to about 16 kb with a quartz phenolic impactor and those shocked at room temperature to about 20 kb with a steel impactor. The former group, (d), exhibited about -66% deviation and the latter group, (f), about -60%.

For groups (b) and (e) the strength degradation is a result of the cracked matrix not being able to distribute the load between the fiber mats. The second set, groups (d) and (f), however, show more extensive effects of preconditioning. From the above results, it appears that these groups underwent more extensive degradation of the matrix than groups (b) and (e). While the cracks from groups (b) and (e) were produced only by

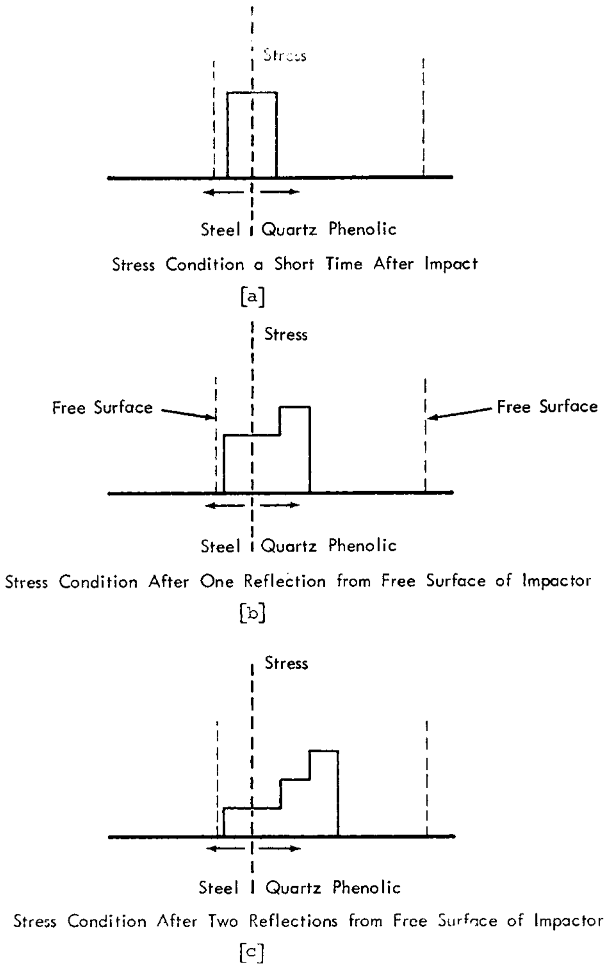

Fig. 5. Diagram of stepped release wave in target:—impactor system with mismatched impedances.

thermal cycling and compressive shock loadings, respectively, cracking from untrapped tensile waves is the probable cause for strength degradation of specimen groups (d) and (f). These tensile waves result from impedance mismatch between the target and the impactor. (Impedance here is defined as ρc, where ρ is the density and c is the wave speed. The impedance is the chord from the origin to the Hugoniot point on the pressure-particle velocity plane. This definition applies to a "linearly elastic" material such as quartz phenolic.) For group (f) the mismatch was the result of using a higher-impedance steel impactor to generate high stresses in the lower-impedance composite targets using relatively low impact velocities. For group (d), however, the mismatch resulted from the target being at elevated temperature and the impactor at room temperature:

the impedance of quartz phenolic at 315°C is estimated to be about one-half its impedance at 23°C.

When similar materials are impacted, the unloading wave from the rear surface of the impactor, which is equal in magnitude and opposite in sign to the generated wave in the target, is totally transmitted into the target and thereby unloads it. However, for mismatched systems (assuming the impactor is of greater impedance than the target) the same factors which allow higher target stresses at lower impact velocities (Fig. 5a) produce only partial transmittal of the unloading wave into the target for each reflection at the impactor–target interface (Fig. 5b, 5c).

Therefore, the point in the target at which the resultant of the loading wave and the wave reflected from the rear surface becomes tensile may be inside the specimen and not inside the momentum trap (which is thrown off when a resultant tensile wave cannot cross the specimen–momentum trap interface). Furthermore, even if the resultant becomes tensile in the momentum trap (thereby trapping the primary source of longitudinally propagating tensile waves), trapped compressive waves from the "tail" of the loading wave will reflect from the new target rear surface continuing to degrade the material.

Shock-wave-produced matrix cracking in quartz phenolic resulted in large tensile strength degradation when loaded normal to the fiber layup (Table II). For this condition the tensile strength is essentially dependent upon the matrix, and if this matrix is cracked, extreme strength degradation occurs.

Uniaxial Stress Preload

In order to investigate the response of composite materials to initial uniaxial stress high strain-rate loadings below fracture, several factors must be considered. These factors include the effects of the rate of loading, the relaxation characteristics of the material, and the effect of the temperature environments on the material, including heating rate and time at temperature.

Quartz phenolic behaves viscoelastically when loaded at various high rates parallel to the fiber layup.[7] Specimens subjected to short-time loadings (30 to 45 millisec) to above the quasistatic fracture stress (10^{-3} per sec) relax when the strain rate goes to zero and within 15 millisec fracture at the quasistatic stress level. Since the time to unload the machine is long compared to this material's relaxation time, intact specimens dynamically loaded to above the quasistatic fracture stress are not recoverable.

The effect of temperature environment on the observed behavior of quartz phenolic was minimized in this study by heating specimens at a relatively slow rate (above 40°C/min), eliminating cooldown between initial loadings and subsequent reloadings to fracture, and minimizing the total time at temperature.

The experimental results from specimens preloaded in uniaxial stress are contained in Tables III and IV. Table III contains the results of quartz phenolic loaded in compression both normal and parallel to the fiber layup direction. As shown, there is essentially no strength degradation at room temperature to about 70–80% preload. However, for loadings parallel to the fiber layup to 88% of fracture stress, subsequent reloading produced a −30% degradation.

At elevated temperatures (315°C) in compression, loadings normal to the layup to almost 60% of the elevated temperature fracture stress produced no degradation. Loading parallel to the layup to about the same percentage to fracture, however, produced as much as −66% degradation.

TABLE III
Quartz Phenolic Compression Tests

Test temp., °C	Fiber layup relative to direction of loading	Strain rate, sec^{-1}	Preload % of fracture stress	Degradation on reload to fracture
23	normal	2	70	< -5
23	parallel	4×10^{-1}	77	< -5
		6×10^{-1}	88	−30
315	normal	2	58	nil
315	parallel	4×10^{-1}	62	−66

All conditions represent the average of a minimum of four tests.

TABLE IV
Quartz Phenolic Tension Tests

Test temp., °C	Fiber layup relative to direction of loading	Strain rate, sec^{-1}	Preload % of fracture stress	% Degradation on reload to fracture
23	parallel	5×10^{-4}	75	nil
		5×10^{-1}	63	nil
315	parallel	1	86	−25

All conditions represent the average of a minimum of 4 tests.

Figure 6 contains photomicrographs of specimens which were loaded below fracture, then unloaded. These typical photographs show cracks in the phenolic matrix but do not show any broken fiber bundles.

For tensile loadings, no preload effects were observed for room temperature loadings parallel to the layup to 75% of fracture stress. At 315°C, however, preloads for this layup to 86% of elevated temperature fracture stress produced as much as −25% degradation. Tension tests on quartz phenolic with layup normal to the loading axis were not conducted due to the very weak strength and brittleness of this layup. From previous tests and tests conducted in the preshock damage study, large degradations in strength would be expected if the phenolic matrix was damaged by preloading.

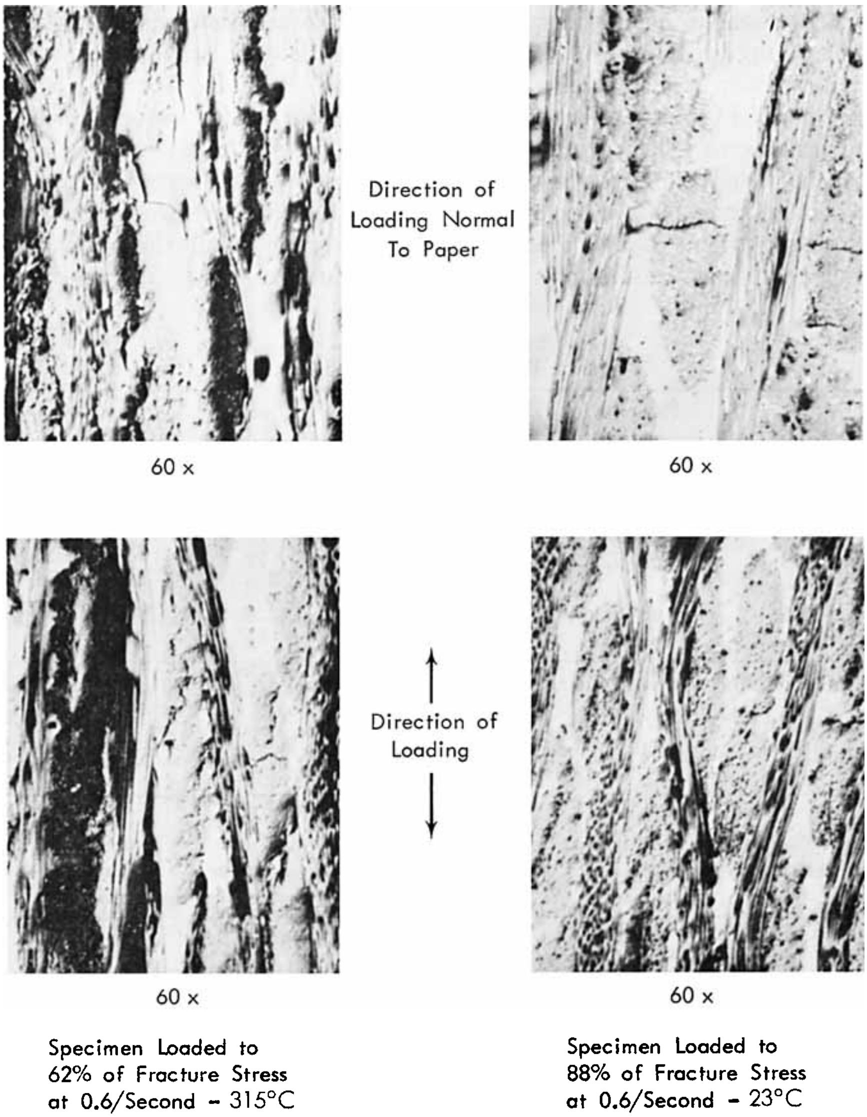

Fig. 6. Photomicrographs of quartz phenolic after loading in compression parallel to fiber layup.

SUMMARY

From this study it has been found that if a fiber-reinforced composite is exposed to a single compressive shock wave and subsequently completely unloaded, the uniaxial compressive strength when loaded parallel to the fiber layup will be decreased by about -15 to -20%. Under shock loading with gradual stepped unloading, compressive strength degradation up to -60% to -70% will result for this layup. Regardless of the shock profile, the uniaxial tensile strength when loaded normal to the fiber layup will exhibit −80% to −90% degradation. Although compressive strengths loaded normal to the fiber layup and tensile strength loaded parallel to the fiber layup were not determined, the strength degradation should be about −20% because, for these layups, the material strength is primarily dependent upon the undamaged fiber mats.

At room temperature, uniaxial stress compressive high strain-rate loadings to about 88% of fracture stress produced about −30% degradation in fracture strength when loaded parallel to the fiber layup. For compressive loadings normal to the fiber layup, only slight degradation in strength and no change in stiffness were observed. Tensile room temperature loadings parallel to the fiber layup below 75% of fracture stress produced no degradation. At 315°C, high strain-rate compressive loadings parallel to the fiber layup above 60% of fracture stress produced degradation of about −66% on reloading to fracture. Loadings normal to the fiber layup in compression at high strain rate up to static fracture stress produced no degradation at temperatures up to 315°C. High strain-rate tensile loading parallel to the fiber layup at 315°C above 85% of fracture stress produced about −25% degradation. Tension tests normal to the fiber layup could not be conducted due to the weakness of the material in this direction; however, cracking of the phenolic matrix should highly degrade the tensile strength of this layup.

Throughout this study, the strengths of composite materials were found to be most affected by previous loadings when the fracture mode was dependent upon the phenolic matrix: i.e., compression loadings parallel to the fiber layup and tension loadings normal to the fiber layup. For conditions in which the fiber mats provide the primary strength influence, i.e., compression normal to the fiber layup and tension parallel to the fiber layup, preload studies show little influence of the cracked phenolic. The cracked matrix, however, may only be a gross indicator for predamage. Recent studies[8] have shown that fiber-to-matrix bonding plays an important role in determining a composite material's behavior. The present study did not include detailed micrographic studies on debonding and therefore samples that showed little matrix cracking but still showed a decrease in strength probably did undergo debonding.

Although both parts of this impulsive loading study concentrated on quartz phenolic, the basic findings may be extended to other fiber-reinforced composites. The magnitudes of strength degradation, however, will depend on quantities such as the relative influence of the resin matrix on the strength of each composite.

The work presented in this paper was conducted as part of the Materials Response Studies (MARS I) under Air Force Contract FO 4694-67-C-0033, monitored by Major N. J. Azzarita, Space and Missile Systems Organization, Air Force Systems Command, Norton Air Force Base, California 92409. The authors would like to especially thank Mr. S. J. Green for his many excellent technical contributions during this study.

References

1. W. M. Isbell, N. Froula, and F. H. Shipman, GM AC Electronics—Defense Research Laboratories Report TR 67-07, Vol. III; also BSD-TR-67-25, Feb. 1967.

2. B. M. Butcher and J. R. Canon, *A.I.A.A. J.*, **2**, 2174 (1964).

3. G. E. Duvall and G. R. Fowles, in *High Pressure Physics and Chemistry*, Vol. 2, R. S. Bradley, Ed., Academic Press, New York, 1963, Chapter 9.

4. D. S. Clark and D. S. Wood, *Proc. ASTM*, **49** (1949), p. 717.

5. J. D. Campbell and K. J. Marsh, *Philosophical Mag.*, **7**, No. 78, June (1962).

6. C. J. Maiden and S. J. Green, "Compressive Strain Rate Tests on Six Selected Materials at Strain Rates from 10^{-3} to 10^{4} in./in./second," *J. Appl. Mechanics*, **33**, Sept. (1966).

7. S. J. Green, S. G. Babcock, and R. D. Perkins, Final Report for Air Force Contract AF 04-(694)-806, BSD Report TR-67-23 (AD-822 803L), Vol. I, Feb. 1967.

8. A. Kumar, R. D. Perkins, F. L. Schierloh, and D. L. Holt, Final Report on Materials Response Studies (MARS I), SAMSO Report TR 68-71, Jan. 1968.

Received February 21, 1969
Revised June 18, 1969