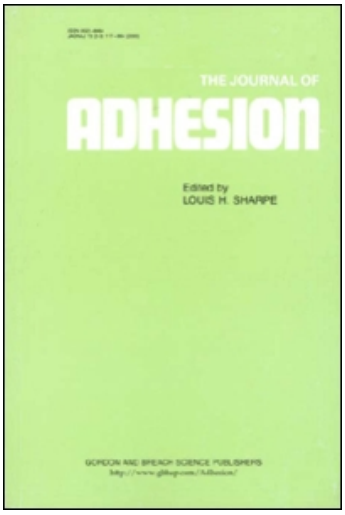

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Effects of Microwave Processing on Fiber-matrix Adhesion in Composites†

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Experiments have been done using a single mode (TE_{111}, 2.45 GHz) cylindrial microwave cavity with single fiber composite specimens. After obtaining a cure cycle with microwaves to match that achieved with a conventional thermal cure cycle as measured by tensile tests, dynamic mechanical analysis and differential scanning calorimetry, quantitative measurements of interfacial shear strength and physical properties have been carried out and compared with the results from conventional thermally-cured systems. Under the conditions studied for single fiber specimens, the fiber-matrix interfacial shear strength decreases slightly in both glass-epoxy and aramid-epoxy cases as compared with thermally-cured specimens. Graphite fiber-epoxy adhesion, on the other hand, increases significantly in these single fiber studies in microwave processed specimens as indicated by an increase in the interfacial shear strength. The failure mode changes from interfacial (thermal curing) to matrix failure.

KEY WORDS Microwave curing; interface/interphase; adhesion; fiber reinforced composite; interfacial shear strength; single fiber testing.

1. INTRODUCTION

The processing of high performance and thick composite materials requires the transfer of energy efficiently into the polymer mass. The introduction of high temperature matrix materials necessitates the use of very high temperature processing equipment for the production of these new-generation composite materials if traditional thermal processing techniques are used. Electromagnetic heating of polymer systems is an alternative to conventional thermal processing. Microwave curing of composites offers the potential for the development of an extremely fast and versatile method of composite processing. Control of energy input, duration and location provides an inherent flexibility not present in any other technique. Limitations in thermal processing due to poor heat conductivity of polymers and accelerated thermal degradation at elevated temperatures due to

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prolonged heating are of little concern in microwave processing. A key factor in achieving this capability lies in creating an "acceptable" interphase between fiber and matrix in the cured composites. Successful composite processing requires that not only the matrix but the polymer-fiber interphase be processed in an optimum manner. Composite properties are highly dependent on adhesion between fiber and matrix and the interphase region is a major factor in determining the type and level of adhesion.

Microwave processing of polymers and polymeric composites at 2.45 GHz has been studied in conventional microwave (multimode) ovens,[1–3] in TE_{01} wave guides[4–6] and in a TE_{10} wave guides.[7] Jow *et al.*[8] have reported the on-line dielectric properties of a reacting epoxy system under a microwave environment in a single-mode cavity applicator. One of the unknown parameters necessary to process effectively an uncured polymeric matrix reinforced with continuous fibers is the interaction of microwave radiation with the fiber-matrix interface/interphase. This study investigates the coupling of microwave energy with the fiber-matrix interphase and the resultant effects on fiber-matrix adhesion in composites.

Coupling of microwave energy depends on the complex dielectric property of materials. Differences in dielectric properties of fibers, polymers and other constituents of composites could cause selective absorption of microwave energy as well as other identifiable molecular phenomena in the interphase region. These phenomena could be: generation and release of volatiles from reinforcements or the polymer matrix in the interphase region; alteration of the specific reinforcement surface chemical groups and their reactions with the polymeric matrix; and the introduction of variations in cure morphology in the interphase region.

Moisture is present in both the polymeric matrix and on the surface or in the bulk of the reinforcement fiber. Although the quantities of moisture present in the bulk material may be low on an absolute scale, concentration of even one percent moisture at the fiber-matrix interface would be a disruptive force causing the nucleation and growth of voids which are a source for the reduction of composite properties. For graphite or glass reinforcements, the moisture is located on the fiber surface. For a polymeric reinforcement, bulk absorption of moisture is an additional source. Preferential heating of water molecules by microwave radiation would cause water vaporization. Interfacial free energies between fiber and matrix could dictate that the water molecules would preferentially concentrate and nucleate at the interface.

The reinforcement fiber surface contains surface chemical groups either native to the surface or placed there through surface treatments. These groups are effective in the promotion of wetting of the fiber surface by the polymer and in chemical coupling of the polymer with the fiber surface in order to promote mechanical, thermal and environmental stability. In the presence of microwave radiation, these surface groups (*e.g.* carboxylic, phenolic, carbonyl, lactone and hydroxyl) could desorb from the surface or could react with the matrix in a different manner. Either mechanism, *i.e.* the reaction between the adjacent surface groups resulting in deactivation of the surface chemical species or

enhanced reactivity of the surface groups with the matrix will affect fiber-matrix adhesion.

Absorption of microwave energy as heat is directly proportional to the loss factor of the material being processed and is given by:[9]

$$\langle P \rangle = \tfrac{1}{2} E_0^2 \omega \varepsilon'' \tag{1}$$

where $\langle P \rangle$ is the rate of power absorption, E_0 is the electric field intensity, ω is the frequency of radiation and ε'' is the loss factor. Differences in dielectric properties between fiber and matrix may give rise to energy localization effects in the interphase region. Further, the reflection of microwave radiation from the conducting carbon fiber surface in laminated composites could cause different unidentified phenomena in the interphase region. Sharp temperature gradients at the interface may cause the processing of the interface to be different from the bulk. All these could be the source for variations in the local morphology of polymers in the interphase region.

Each of the different possible phenomena occurring in the interphase region as discussed above could be a beneficial or deterimental development depending on the polymer chemistry. The creation of an excessively-crosslinked region could increase the efficiency of stress transfer from fiber to matrix while reducing the composite fracture toughness. The promotion of fracture toughness with a reduction in mechanical properties could be a result if matrix crosslink density is reduced.

2. APPROACH

Adhesion between fiber and matrix was evaluated by single fiber critical length tests.[10,11] When a matrix tensile coupon containing a single fiber is subjected to an increasing tensile load, the fiber begins to fracture until the final fiber fragments are shorter than the critical length. Determination of the fiber critical length-to-diameter ratio allows the interfacial shear strength to be determined according to:

$$\tau = \frac{\sigma_f}{2}\left(\frac{d}{l_c}\right) \tag{2}$$

where σ_f is the fiber tensile strength at the fragment length l_c for a fiber of diameter d.

Due to random defects in the fiber, fiber fragments have a range of lengths. It has been shown that the critical length data fit a two-parameter Weibull distribution.[11] A mean value of interfacial shear strength can be calculated according to:

$$\bar{\tau} = \frac{\sigma_f}{2\beta}\Gamma\left(1 - \frac{1}{\alpha}\right); \tag{3}$$

where α and β are the two parameters of the distribution and can be evaluated by maximum likelihood methods.

Valuable information regarding the stress transfer and the locus of failure between the fiber and the matrix can be obtained by examining the fiber fragmentation process under polarized light.[12]

Evaluation of the interface in microwave-cured specimens as compared with thermally-cured specimens requires that the bulk matrix properties be the same in both cases. In order to achieve this the following steps were followed:

1) Establish a database of mechanical properties and extent of cure for the matrix cured thermally under a standard cure cycle.

2) Develop a microwave cure cycle which would produce the same matrix properties as the baseline data.

3) Evaluate and compare the interfacial shear strength and failure mode of various fibers in thermally-cured and microwave-cured (under the above determined cure cycle) specimens.

3. EXPERIMENTAL

The materials used in this study were chosen to facilitate the detection of different mechanisms of microwave interactions at the interphase. A DGEBA-based epoxy resin (DER 331, Dow Chemical) cured with the stoichiometric amount of meta-phenylene diamine (*m*-PDA) is used as the matrix material with various reinforcing fibers including carbon (AS4), *E*-glass and an aramid fiber (Kevlar 49®, Du Pont).

A. Thermal curing

Single fiber specimens of epoxy (Figure 1) were prepared by casting into a silicone RTV-664 eight cavity mold. Standard ASTM 63.5 mm (2.5″) dogbone specimen cavities with a 3.175 mm (1/8″) wide × 1.59 mm (1/16″) deep × 25.4 mm (1″) long gage section were molded into a 7.62 cm (3″) × 20.32 cm (8″) × 1.27 cm

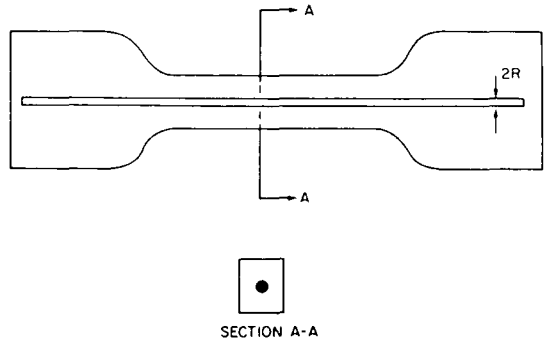


FIGURE 1 Schematic diagram of a single-fiber interfacial shear strength specimen.

(1/2″) thick silicone piece. 0.8 mm (1/32″) deep sprue slots were molded at both the ends of each dogbone cavity for aligning the fiber axially in the dogbone. Single filaments of each desired fiber were taken from a fiber bundle and placed tightly in the sprues of a dogbone cavity with rubber cement so that the fiber was centered across the cavity axially.

Epoxy resin (DER 331) with a stoichiometric amount (14.5 phr) of *m*-PDA were weighed in separate beakers. These beakers were then transferred to an oven and heated at 65°C until the *m*-PDA had melted. Then the epoxy resin and the curing agent were mixed thoroughly and degassed in a vacuum oven along with the silicone mold at 65°C and 29″ Hg vacuum. The degassed resin and curing agent mixture was then poured into all the cavities of the mold with the help of a drip rod and to a level just above the height of the mold surface. The assembly was then transferred to an electronically temperature-controlled, air-circulating oven which was preprogrammed for a curing cycle of 2 hrs @ 75°C followed by 2 hrs @ 125°C. At the end of the cure cycle, the oven temperature was brought down to room temperature at a cooling rate of 2°C/min. The cured specimens were then taken out of the silicone mold and the extra material was sanded off the surface of the specimens in order to get smooth and uniform specimens. The samples were stored in a desiccator until ready for analysis.

B. Microwave curing

Coupling of microwave energy to polymer specimens was achieved by interactions between a sustained electric field in the microwave applicator and the dipoles contained in the polymer system. The schematic of the experimental setup is shown in Figure 2. A cylindrical brass cavity,[13] of 17.8 cm (7″) inner diameter covered by two transverse brass shorting planes and constructed in the Michigan State University machine shop, was used as a microwave applicator (Figure 3). The cavity length could be adjusted by moving the top plane. The bottom plane was fixed but could easily be removed. The cavity wall and the bottom had

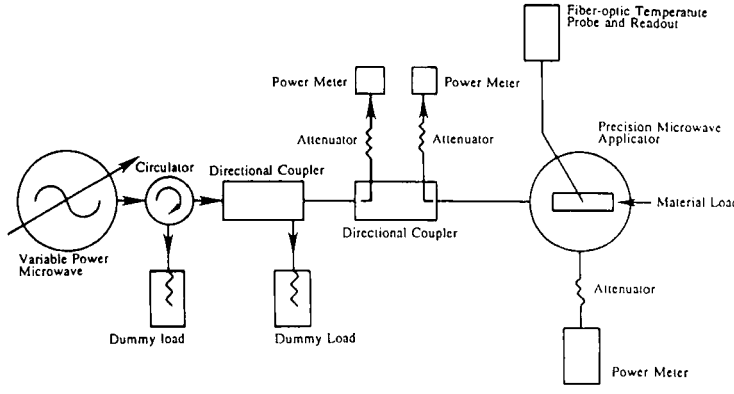


FIGURE 2 Experimental microwave system circuit diagram.

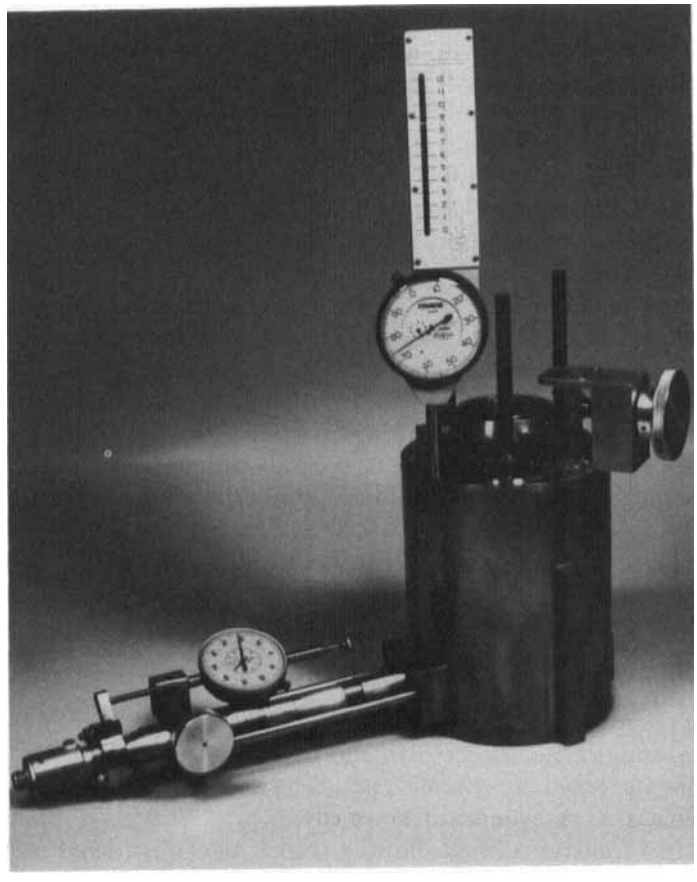


FIGURE 3 Cylindrical brass cavity for microwave processing.

several diagnostic holes. Microwave power was coupled into the cavity through an adjustable excitation probe. The coaxial probe had an outer conductor of 1.27 cm (0.5″) diameter and an inner conductor of 0.442 cm (0.174″) diameter and was located 3.81 cm (1.5″) above the bottom plate.

A magnetron-based, fixed frequency (2.45 GHz) generator (Opthos MPG-4) with adjustable duty cycles from 5% to 85%, and power output from 0 to 120 watts, was used as the microwave source. For stability considerations, the power source was operated at higher power levels and the desired power was channelled through a 10 db directional coupler. A circulator was used to protect the source from the reflected power. 50-ohm impedance coaxial cables were used to transmit the power from the source to the cavity. Two 20 db directional couplers were used to decouple the incident and reflected signals. Both incident and reflected signals were attenuated and measured directly by power meters.

Silicone dogbone cavities filled with the epoxy mixture were placed in the center of the bottom of the cavity and in alignment with the excitation probe.

Due to non-uniformity of the fields inside the cavity, only one dogbone specimen was cured at a time. A fluoroptic temperature measuring system (Luxtron Model 750) equipped with four channels for measurement was used to monitor continuously the temperature of the reacting mixture at different locations. The cavity was tuned initially to couple the microwave energy critically to the epoxy in the lowest order mode TE_{111}, by moving the top plate of the cavity and the excitation probe. The cavity was tuned continuously throughout the experiment as the dielectric properties of the reacting mixture were changing.

C. Material properties

Specimens cured thermally or in the microwave environment were tested for thermal and mechanical properties. The extent of conversion (α) and glass transition temperature (T_g) were determined using Differential Scanning Calorimetry (DSC model 9900, DuPont). DSC scans of cured and uncured matrices were made at 5°C/min under a nitrogen purge using open pans.

Mechanical properties, including tensile strength and tensile modulus were determined using a servohydraulic tensile tester (Material Testing System 880). ASTM standard dogbone specimens were loaded in the hydraulic grips with specially-designed supplementary grips to protect the specimens. The load-elongation curves were recorded on a chart recorder at a strain rate of 0.02″/min (about 2%/min). In calculating the strain from crosshead displacement, the gage length of the sample was taken as the distance between the supplementary grips (25.8 mm). Young's modulus was calculated from the initial slope of the load-elongation curve whereas the tensile strength to failure was recorded from the machine itself.

Glass transition temperatures, dynamic mechanical properties such as flexural modulus (E') and loss tangent (tan δ) of cured specimens were determined using a Dynamic Mechanical Analyser (DMA model 9900, DuPont). Fixed frequency (1 Hz) DMA scans were made at 10°C/min and 0.2 mm amplitude.

D. Single fiber critical length test

Single-fiber dogbone specimens of the desired fiber were prepared and cured thermally or with microwaves as discussed above. Once fabricated, these specimens were then tested for fiber critical lengths. Prior to testing, each specimen was examined visually and under the microscope for voids, fiber breaks, additional fibers and other foreign materials, and other fabrication defects which would affect the testing and the results. The testing procedure involved the careful straining of the specimen in a hand-operated tensile testing fixture, designed and built in the Composite Materials and Structures Center Laboratory and Michigan State University machine shop. The fixture was capable of applying enough load to the specimen to cause fracture and incorporated a dial gage to give an indication of the amount of strain. The fixture was then mounted on the stage of a microscope (Olympus BH-2). The clarity of the image was improved by

using two cover glasses along with the proper refractive index fluid, one on the top surface of the specimen and one on the bottom surface, to eliminate distortions due to specimen roughness.

The specimen was then subjected to an increasing tensile load and fiber breaks were monitored *in-situ* through the microscope. When the specimen reached a state of constant number of fiber breaks irrespective of the increasing load, the length of each fiber segment was measured using a calibrated filar eyepiece. Fiber diameter was also measured using the same filar eyepiece. The fiber fractures were then examined under transmitted polarized light and photomicrographs were taken using the attached camera assembly to the microscope.

E. Interphase temperature

The determination of the graphite–epoxy interphase temperature is essential in order to develop an understanding of the interphase. The interphase region is usually 5–5000 Å thick and so it is very difficult to evaluate its local temperature directly and accurately. An indirect method was used to determine the interphase temperature. The method consisted of directly measuring the temperatures at different locations in the vicinity of the fiber in the specimen using fluoroptic microprobes. The interphase temperature was then evaluated analytically using inverse heat conduction methods. The details of the experimental procedure are given below.

Several holes of about 1 mm diameter were made in the gage section of the silicone dogbone mold (Figure 4). The silicone mold was then placed on the bottom plate of the cavity in such a way that the plate holes were aligned with the silicone mold holes. Fluoroptic temperature probes were then inserted through the holes and fixed at the different locations and heights across the thickness of the dogbone. The distances of these probes from the mold surface were then measured using a digital caliper. A single graphite fiber was laid in the mold and its height above the mold bottom was also recorded. Heated (70°C) epoxy resin (DER 331) was then poured in the mold with the help of a drip stick. No curing

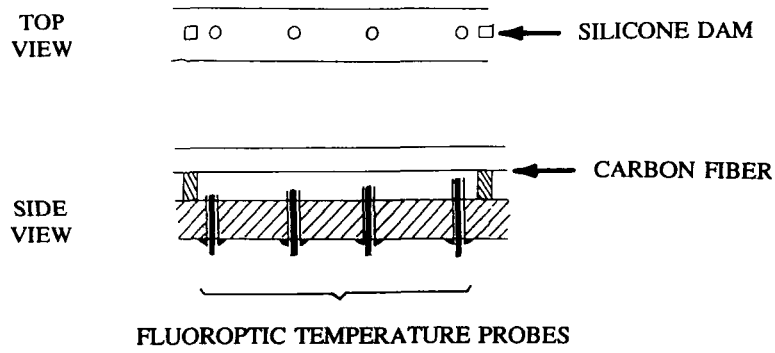


FIGURE 4 Diagram of a carbon fiber–epoxy interphase temperature measurement experiment.

agent was used with the epoxy, since with the curing agent the epoxy mixture would solidify and destroy the fluoroptic probes. The cavity was then tuned in the TE_{111} mode and the specimen was heated under 4.5 watts (the same low-power curing cycle for graphite fiber specimens) of microwave power. Temperatures of all the probes were then recorded every second with the help of an automated data acquisition system. Temperature profiles at distances of 0.09 mm (T1), 0.34 mm (T2) and 0.59 mm (T3) from the fiber are shown in Figure 5. The graph shows that the steady state temperature in the interphase region is attained in less than 350 seconds.

A steady state heat balance around the fiber allows us to back calculate the fiber surface temperature from the above-obtained, steady-state temperature data. The heat balance led to the expression:

$$T = C_1 \ln r + C_2$$

where T is the temperature at a distance r from the fiber axis. Constants C_1 and C_2 are determined from the temperature data using the Least Squares method and the values are:

$$C_1 = -13.81, \qquad C_2 = 73.98$$

This simplistic approach provides an initial approximation of the interphase temperature from the steady-state data. The fiber is assumed to be infinitesimally small as compared with the mold dimensions and thus the boundaries are ignored in the heat balance. In practice, the fluoroptic temperature probes were placed at significant distances away from the fiber and any heat absorbed by the fluoroptic probes is ignored in the heat balance.

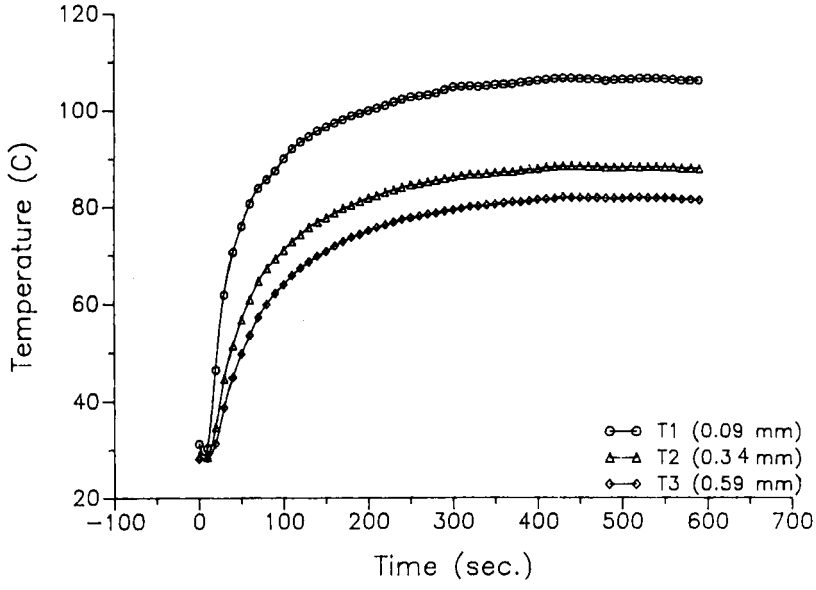


FIGURE 5 Carbon fiber–epoxy interphase temperature profiles under microwave environment.

TABLE I
Mechanical properties of epoxy cured under thermal and microwave environments

	Tensile strength (Ksi) Mean (S.D.)	Modulus (Ksi)[b] Mean (S.D.)
Thermal curing		
2 hrs @ 75°C, 2 hrs @ 125°C	12.27 (0.97)	231.63 (10.73)
Microwave curing[a]		
9.0 W for 15 min	12.77 (1.09)	238.24 (6.34)
9.5 W for 15 min	12.72 (0.83)	234.61 (9.27)
12 W for 15 min	12.68 (0.62)	219.27 (15.29)
5.0 W for 15 min	13.79 (0.65)	234.48 (6.92)
4.5 W for 25 min	13.20 (0.40)	230.00 (4.11)

[a] With 2 hrs @ 125°C post cure.
[b] Values are low as compared with the literature values due to the use of machine strain in evaluating Young's modulus. These values are used only for comparison in this study.

4. RESULTS AND DISCUSSION

A. Matrix properties

Baseline mechanical properties of the thermally-cured specimens under the curing cycle of 2 hrs @ 75°C followed by 2 hrs @ 125°C are listed in Table I. For microwave processing, experiments were performed by aligning the specimens with the E field inside the cavity. Specimens were cured at various power levels and durations and their temperature-time profiles are shown in Figure 6. In the beginning of the process, the temperature increases very rapidly as the dipoles are

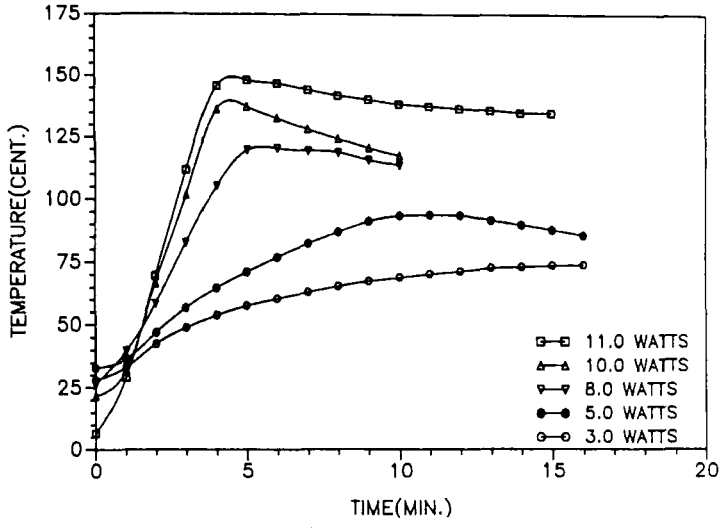


FIGURE 6 Temperature-time profiles of DER 331/*m*-PDA specimens at different microwave power levels.

free to rotate and thus the coupling of microwave energy is very efficient. As the reaction progresses, the network starts to form and as a result, dipoles are no longer free to rotate. Power absorption decreases and temperature decreases slowly. Comparison of various profiles shows that the peak temperature of a curing cycle increases with increasing power level. These specimens are then tested for mechanical properties including modulus and tensile strength. The results of mechanical properties are listed in Table I.

By comparing mechanical properties at various power levels with the baseline data, it was found that a curing cycle of 9.5 watts for 15 minutes would produce the same matrix mechanical properties as the baseline data. This curing cycle of 9.5 watts for 15 minutes was used to evaluate the interfacial shear strength.

B. E-glass fiber

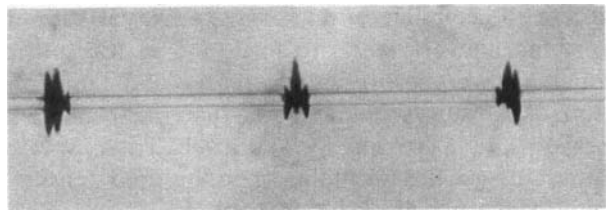
Interfacial shear strength (ISS) data for *E*-glass fiber are tabulated in Table II. ISS in the microwave cured specimen is found to be 8.29 Ksi as compared to 9.73 Ksi for thermally-cured specimens. The fracture behaviour of *E*-glass in epoxy matrix is shown in the transmitted light micrograph (Figure 7(A)). Black spots show the point of fiber failure in the matrix. At the point of fiber breakage, the matrix also fails and a crack starts to grow in the matrix perpendicular to the fiber. This type of failure, termed matrix failure, is characteristic of good adhesion between fiber and matrix. No difference is observed in the failure mode of *E*-glass fibers whether under microwave curing or thermal curing even though the ISS decreased by 15%.

C. Aramid fiber

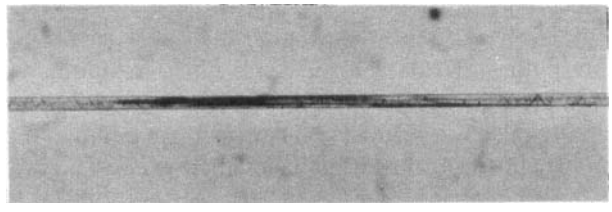
Similar experiments were done with an aramid fiber specimen and the results are tabulated in Table III. The bonding strength also decreases by 15% in the microwave-cured specimens. ISS in thermally-cured specimens is 3.23 Ksi which decreases to 2.74 Ksi in microwave-cured specimens. The ISS value of aramid fibers is lower than that of glass fiber indicating poor adhesion between aramid fibers and epoxy. The unique fracture of aramid fibers is indicated by the

TABLE II
Interfacial shear strength of *E*-glass fiber specimens cured under thermal and microwave environments

	Thermal curing	Microwave curing
Total fragments	580	681
Weibull α	4.366	3.628
Weibull β	18.429	22.749
Fiber tensile strength (Ksi) (1" gage length)	299	299
Interfacial shear strength (Ksi)	9.73	8.29



(A) TRANSMITTED LIGHT MICROGRAPH OF E-GLASS FIBER FRACTURE



(B) TRANSMITTED LIGHT MICROGRAPH OF KEVLAR-49 FIBER FRACTURE

FIGURE 7 Micrographs of (A) *E*-glass, (B) Kevlar 49 fiber fractures in thermally-cured specimens.

transmitted light micrograph (Figure 7(B)). An aramid fiber does not break at sharp points as in the case of the brittle glass fiber. Instead the fiber breaks by an axial fibrillation mechanism with the whole dark region being the fiber fracture. The failure mode in microwave-cured specimens is observed to be the same as in thermally-cured specimens.

D. Carbon fiber

Carbon fiber, being a good conductor, absorbs a majority of the power causing a very high temperature rise in the fiber and interphase region. The carbon fiber

TABLE III
Interfacial shear strength of Kevlar 49® fiber specimens cured under thermal and microwave environments

	Thermal curing	Microwave curing
Total fragments	292	186
Critical length (mm)	0.904	1.065
Fiber tensile strength (Ksi) (1" gage length)	487	487
Interfacial shear strength (Ksi)	3.23	2.74

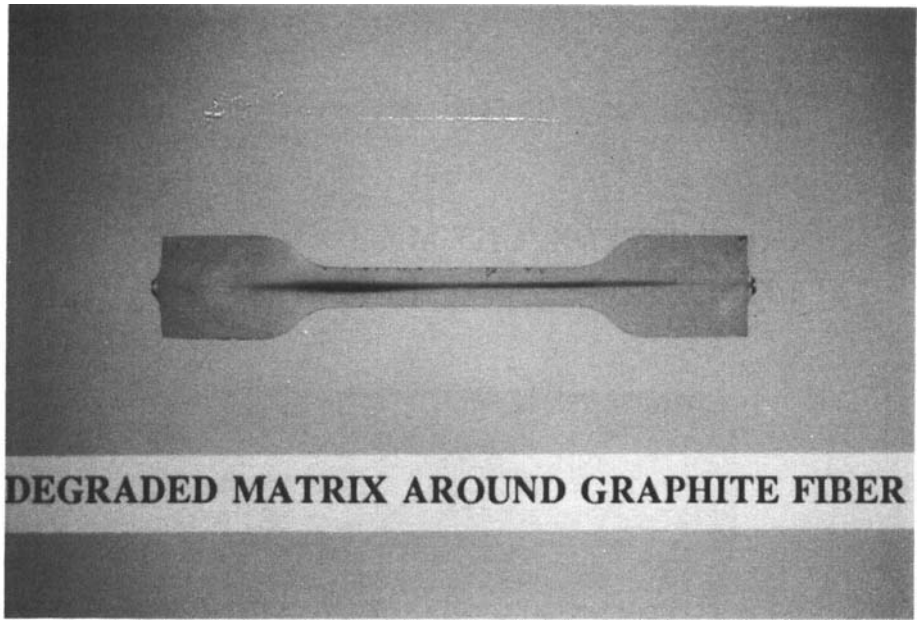
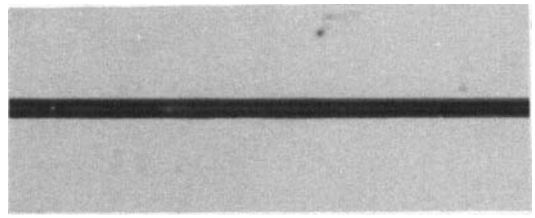


FIGURE 8 Degraded matrix around graphite fiber in a microwave-cured specimen.

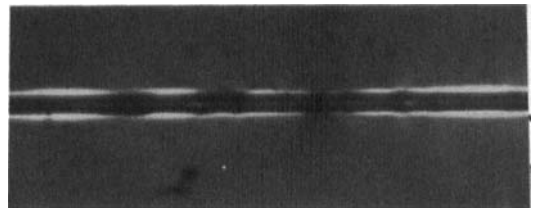
(AS4)-epoxy interphase was observed to have been thermally degraded under the microwave curing conditions of 9.5 watts for 15 minutes (Figure 8). This necessitated the development of a lower power curing cycle for carbon fiber specimens. The results of ISS are tabulated in Table IV for this reduced power cycle. The ISS in the microwave-cured specimen is found to be 14.15 Ksi as compared with 8.3 Ksi in the thermally-cured specimen. A significant increase of 70% in bonding strength between the carbon fiber and the matrix is detected. The mode of failure also changes from axial (thermal curing) to matrix (microwave curing). Micrographs of carbon fiber fracture in a thermally-cured specimen under transmitted light and polarized transmitted light are shown in Figure 9. In the transmitted light micrograph, it is difficult to notice the fiber fracture whereas, in the polarized transmitted light micrograph, the birefringent pattern clearly

TABLE IV
Interfacial shear strength of graphite fiber specimens cured under thermal and microwave environments

	Thermal curing	Microwave curing
Total fragments	515	553
Weibull α	3.568	3.789
Weibull β	60.00	34.953
Fiber tensile strength (Ksi) (1" gage length)	795	795
Interfacial shear strength (Ksi)	8.30	14.15



TRANSMITTED LIGHT MICROGRAPH OF AS4 FIBER FRACTURE (THERMAL CURING)



POLARIZED TRANSMITTED LIGHT MICROGRAPH OF AS4 FIBER FRACTURE (THERMAL CURING)

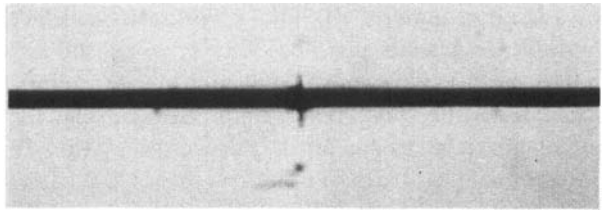
FIGURE 9 Micrographs of a graphite fiber fracture in a thermally-cured specimen.

shows an axial mode of failure.[14] Light around the fiber shows that debonding has occurred between the fiber and the matrix. Figure 10 shows carbon fiber fracture in a microwave-cured specimen under transmitted light and polarized transmitted light. A crack grows in the matrix direction at the fiber break, depicting a matrix-failure mode.

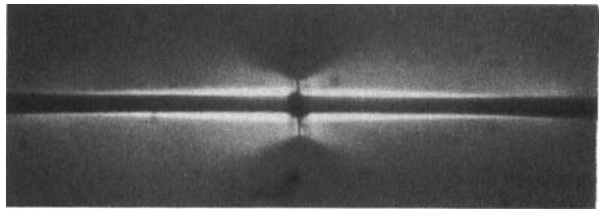
E. Interphase temperature in graphite-epoxy specimens

The question that remains unanswered is what makes the graphite–epoxy interphase so unique, under microwave processing, that it shows dramatic changes in interfacial properties as opposed to the glass-epoxy and Kevlar-epoxy interphases. The conductive nature of the carbon fiber causes the concentration of microwave energy in the form of heat in the interphase region. Carbon fiber acts as a heat source causing some unidentified phenomena in the interphase region which needs to be investigated. Various possible interphase parameters which could be affected significantly under the microwave environment, thus affecting the interfacial shear strength and the failure mode are:

1) Final cured material properties of the interphase region could possibly be different from the bulk properties when the material is cured in a microwave environment, due to the high interphase temperature.



TRANSMITTED LIGHT MICROGRAPH OF AS4 FIBER FRACTURE (MICROWAVE CURING)



POLARIZED TRANSMITTED LIGHT MICROGRAPH OF AS4 FIBER FRACTURE (MICROWAVE CURING)

FIGURE 10 Micrographs of a graphite fiber fracture in a microwave-cured specimen.

2) The graphite fiber surface chemistry could be altered in the microwave environment resulting in an improved chemical bonding between the fiber and the matrix.

3) A sharp temperature gradient in the interphase region could affect the bulk material properties of the graphite fiber.

4) In microwave curing, the graphite fiber may absorb the majority of the power as heat and the curing proceeds from the fiber surface out to the bulk matrix. This could result in a stress-free interphase with improved adhesion between the fiber and the matrix.

Although differentiation between these possible mechanisms will be the subject of further research, the estimation of the fiber surface temperature is an important factor in explaining possible causes for the observed difference in behaviour. Using the inverse method explained above, the fiber temperature was estimated to be 150.23°C (at $r = 4\mu$) at its surface while the surrounding epoxy was at a much lower temperature.

DSC studies show that it takes about 23 minutes to cure the DER 331 and *m*-PDA mixture completely at the above-determined fiber surface temperature (150°C) as compared with more than 4 hrs when cured at the bulk temperature (75°C). This result confirms our observation that in a graphite fiber specimen

under the microwave environment, the epoxy curing front moves from the fiber surface out to the bulk. In aramid and glass fiber systems, the dielectric properties of these fibers make them transparent to the microwave radiation, therefore the cure would proceed from the bulk to the fiber surface. The temperature of the fiber might also be expected to be initially lower than the surroundings. This phenomenon could result in a stress-free interphase for the carbon fiber which, in turn, might cause better adhesion between the carbon fiber and the epoxy matrix while, conversely, providing a "poorer" interface for the aramid and glass systems.

5. CONCLUSIONS

The conclusions derived as a result of this study are as follows:

- Microwave curing of epoxy is much faster and more flexible than any other conventional thermal heating technique and provides equally good interfacial and mechanical properties. With conductive fibers, the adhesion may be superior.
- Under the curing conditions used in this investigation with single-fiber specimens, the fiber-matrix interfacial shear strength decreases by 15% when the glass-epoxy and aramid-epoxy specimens are cured with microwave energy as opposed to conventional thermal curing. The fiber failure mode is not affected by the curing techniques for either system.
- Microwave curing of specimens with carbon fibers requires extra caution because the conductive nature of the carbon fiber can cause selective absorption of microwave energy resulting in localized heating of the fibers.
- Microwave curing increases the level of adhesion in single-fiber, carbon-epoxy specimens. Under the curing conditions used for carbon fiber specimens, the interfacial shear strength in microwave-cured specimens increases by 70% as compared with that in thermally-cured specimens. However, the fiber fracture mode changes from interfacial failure in thermally-cured specimens to matrix failure in microwave-cured specimens.
- The interphase temperature in the single-fiber, carbon-epoxy specimens under 4.5 watts of microwave power is found to be 75°C higher than the bulk temperature. This results in a much faster curing reaction in the interphase region than in the bulk matrix.

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