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Effect of Fiber Treatment on the In-plane Shear Properties of Composite Materials

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The effect of the fiber surface treatment on composite material shear behavior is evaluated. The Iosipescu shear test is employed to measure the in-plane shear properties of unidirectional AU4- and AS4-BMI/PES, as well as IM8-BMI/PES with 50%, 100% and 400% fiber surface treatment. In addition, a meso-indentation technique is employed to assess the effect of degrees of surface treatment on interfacial shear strengths. It was found that the in-plane shear properties of AU4- and AS4-BMI/PES laminates are essentially the same. The in-plane shear properties of IM8-BMI/PES laminates with 50%, 100% and 400% fiber surface treatment are approximately the same. Meso-indentation results confirm the Iosipescu shear strengths obtained.

KEY WORDS polymer composite; fiber surface treatment; shear modulus; shear strength; losipescu specimen; meso-indentation.

INTRODUCTION

Characterization of the in-plane shear properties is a prerequisite to the understanding of the mechanical behavior of a laminated composite structure. For unidirectional composite materials, fiber properties dominate the mechanical behavior in the tensile mode, whereas shear behavior provides a discriminator of matrix and fiber/matrix interfacial properties. Thus, shear modulus or strength can provide a parameter for assessment of the effects of processing variables, such as fiber treatment, upon the performance of a composite system.

In a fiber-reinforced composite, the fiber-matrix interface transfers the stress from the weak and often low modulus matrix to the fiber. Thus, in addition to the material properties of the fiber and matrix, composite behavior can also be governed by the chemical-physical interactions occurring at the fiber-matrix interface and encompassing interphase. The interphase includes the contact region between the fiber and the matrix (the interface) and the regions of finite thickness extending on both sides of the interface. The interphase starts from some point within the fiber where

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the local properties begin to change from the fiber bulk properties through the actual interface into the matrix where the local properties again equal the bulk properties.¹ Thus, the interphase encompasses the bulk fiber, the surface layer of the fiber, a surrounding layer of polymer matrix of different properties and the bulk adhesive matrix. The surface region of the fiber may contain pores or cracks, and the atomic and molecular composition of the fiber surface is different from the bulk of the fiber. The effect of the surface treatment is to etch away the outer fiber layer initially present on the fiber and to add surface chemical groups to increase the interaction with the matrix.¹

It was shown^{1,2} that the adhesion of surface treated AS4 carbon fibers to thermosetting polymer increased significantly as compared with the untreated AU4 carbon fibers. It is generally known that thermoplastic polymers do not form as strong a bond to carbon fibers as thermosetting polymers do. Therefore, it is of interest to investigate the adhesion of the carbon fibers to the thermoplastic-toughened thermosetting polymers. In this study, the effect of the fiber surface treatment (AU4 and AS4 fibers) on the shear properties of the thermoplastic-toughened thermosetting composites is evaluated. In addition, the effect of different degrees of fiber surface treatment (50%, 100% and 400% for IM8 fibers) on the thermoplastictoughened thermosetting composites is also investigated.

The importance of surface treatment has been evaluated using the single fiber fragmentation technique,² which provides the interfacial shear strength and the failure mechanism for a single fiber in a block of matrix material. Although this technique provides critical information about fiber-matrix bond quality, given the base constituents, it is desirable to know the interfacial quality in the context of the as-processed composite. Thus, in this study a meso-indentation test is used to interrogate the interfacial/phasal characteristics of composite laminates.³ The test is performed by pressing a hardened steel spherical penetrator into the fiber direction of a polished composite surface. This technique has been demonstrated to provide a quantitative measure of interface bond quality for unidirectional laminates with minimal cost, complexity and quantity of material required.

In structural applications, the mechanical properties of processed composite laminates are needed. Therefore, the effect of the surface treatment has to be evaluated in a global sense by measuring, for example, the in-plane shear modulus and strength of the bulk composite material. In the process of developing a new material system, large quantities of the material are not readily available for material property characterization. Among the existing shear testing methods, the Iosipescu shear test is chosen here for the material in-plane shear property characterization due to the following special features: (1) small specimen size combined with the ability to test a range of specimen thickness, (2) the potential to measure shear modulus and strength. To provide consistent and accurate shear modulus measurement, the 90° (fibers parallel to the applied load) Iosipescu shear specimen is used.⁴ The in-plane shear properties of 90° AU4- and AS4-BMI/PES and IM8-BMI/PES Iosipescu shear specimens with 50%, 100% and 400% fiber treatment are measured and compared.

EXPERIMENTAL PREPARATION

(i) AU4/AS4 Fiber Composites

AU4 fibers, which were removed from the heat treatment ovens without any further processing, and AS4 fibers, which were treated with a proprietary electrochemical oxidation step, were used. The XPS (X-Ray Photoelectron Spectroscopy) technique was employed to examine the chemical compositions on the fiber surface of AU4 and AS4 fibers.

The bismaleimide matrix material was a two-part system, Matrimid 5292 A/B, Figure 1, obtained commercially from Ciba Geigy. Part A was the common bismaleimide, 4,4' bismaleimidodiphenyl methane and part B was a reactive diluent, 0,0' diallyl bisphenol A, added as a processing aid and which also served as a solvent for the polysulfone, a thermoplastic toughness modifier. The polysulfone, Figure 2, was specifically synthesized to control its molecular weight to aid in processing, and prepared with reactive (maleimide) end groups to improve the modified BMI fracture toughness properties.^{5,6}



⁽a)



(b)

FIGURE 1 Chemical structure of (a) 4,4' Bismaleimidodiphenyl methane, (b) 0,0' Diallyl Bisphenol A.



FIGURE 2 Repeating unit of polysulfone.

The two sets of fibers, AU4 and AS4, were combined with the thermoplasticmodified thermoset using hot-melt processing techniques. The hot-melt solution was prepared by initially weighing the diallyl bisphenol A compound into a twonecked, round bottom flask. The thermoplastic modifier (dried overnight in a vacuum oven) was weighed and added to the flask. In this study, a polysulfone of molecular weight 12800 g/mole with maleimide end groups was added to the BMI resin at a 15% (by weight) loading. A mechanical stirrer with Teflon[®] paddle and vacuum adapter was fitted to the flask and placed in an oil bath heated to 130°C. A vacuum was slowly applied to the stirred mixture in order to degas the resin system and to remove volatiles such as residual solvent and entrapped air. Upon completing this first step a homogeneous yellow solution was obtained. After raising the temperature to 140°C the bismaleimide resin was added via a powder funnel. Again, vacuum was applied to the flask and, with further stirring for approximately 20 minutes, a dark, reddish-brown, homogeneous hot-melt solution was obtained. In this form, the hot-melt resin could be added to the resin pot within the hot-melt drum winder for fiber prepregging.

The unidirectional AU4 and AS4 composite panels have in-plane dimensions of 76mm by 127mm $(3'' \times 5'')$ and are 18-plies thick. Fiber volume fractions were analyzed according to ASTM test method D3171. The fiber volume fractions for the AU4 and AS4 panels were measured to be 50% and 70%, respectively. Due to the difference in fiber volume fractions, the average thicknesses of the AU4 and AS4 specimens are 2.5mm and 3.8mm, respectively. The extensional properties are $E_{11} = 131$ GPa, $E_{22} = 8.65$ GPa for AU4-BMI/PES and $E_{11} = 140$ GPa, $E_{22} = 8.15$ GPa for AS4-BMI/PES, respectively.⁶ Six 90° specimens were cut from each panel to the dimensions suggested by ASTM,7 Figure 3a. A two-dimensional drawing of the modified Wyoming fixture is shown in Figure 3b. Each specimen is numbered according to its panel position for further reference, Figure 4. Five specimens were instrumented with unstacked two-gage rosettes (1.5mm gage length) at the center of the front and back surfaces of the specimen. One specimen was tested with a Moiré grating on one face and the strain gage rosette on the other face to evaluate the uniformity of the strain fields. Masking tape was applied to the long edges of the specimen to reduce the twisting effect.⁴

(ii) IM8 Fiber Composites

The IM8 carbon fibers were acquired with varying levels of a proprietary surface treatment. Three unidirectional composite panels with 50%, 100% and 400% surface-treated IM8 fibers in BMI/PES matrix were fabricated.⁶ The surface chemistry of the IM8 fibers was then determined by XPS. The corresponding fiber volume fractions for the 50%, 100% and 400% surface-treated panels were 71%, 69% and 70%, respectively.

The composite panels were nominally 2mm (18-plies) thick with 76mm by 127mm $(3'' \times 5'')$ in-plane dimensions. Five specimens obtained from each panel were instrumented with unstacked two-gage rosettes (1.5mm gage length) on both faces of the specimens and one specimen from each panel was tested using the Moiré interferometry technique to evaluate the uniformity of the strain fields.



FIGURE 3 (a) Modified Iosipescu specimen and (b) modified Wyoming fixture (W2).

For these IM8 fiber composites, meso-indentation specimens were taken from failed Iosipescu test specimens in the central region of the laminated panel, *i.e.* one of specimens #2 through #5 of Figure 4. The region close to the notch and the panel edge were discarded and the remaining lengths were sectioned in half along



FIGURE 4 Panel size, specimen position and thickness for AU4-BMI/PES and AS4-BMI/PES composite materials.

the 76mm dimension. Specimens 20mm in length and 7mm in depth (*i.e.* fiber direction) resulted following trimming. The sections were then mounted in a metallographic epoxy casting compound and polished such that indentations were made on the freshly cut face so as not to interrogate the "loading" edges which originally made up the boundaries of the Iosipescu specimen.

The Iosipescu shear test was conducted according to the procedure proposed by Ho⁸ et al. The Moiré experiments were first performed to assess the uniformity of the displacement and strain fields and to ensure the quality of the fabricated composite specimens. From the Moiré fringe patterns of the AU4-, AS4-BMI/PES and the IM8-BMI/PES composites, it was found that the uniformities of the displacement and strain fields of these composites were essentially similar and were representative of those of the 90° specimens of high orthotropies.^{4,9} Characteristic Moiré fringe patterns and the associated strain fields of the 90° specimens are discussed in References 9 and 10.

EXPERIMENTAL RESULTS AND DISCUSSION

(i) AU4/AS4 Fiber Composites

The chemical compositions of the AU4 and AS4 fiber surfaces obtained from XPS are shown in Table I. The oxygen concentration on the AS4 surface is increased by a factor of 5 over the AU4 fiber while the nitrogen concentration is increased by a factor of 2.5.

		Atomic concentration				
	Fiber C(1s)	N(1s)	O(1s)	Other	O/C	N/C
AU4 AS4	96.0 84.0	2.0 5.0	1.8 9.9	0.8	0.02 0.12	0.02 0.06
IM8 50% ST IM8 100% ST IM8 400% ST	88.8 83.7 82.1	2.9 4.1 6.3	8.3 11.4 10.7	0.8 0.9	0.09 0.14 0.13	0.03 0.05 0.08

 TABLE I

 Atomic concentrations of various elements on the surface of carbon fibers from XPS analysis

The shear stress-strain data plotted as shear stress against average shear strain obtained from the front and back faces of the strain gaged AU4 ($V_f = 50\%$) and AS4 ($V_f = 70\%$) specimens are shown in Figure 5a. The shear responses for the AU4- and AS4-BMI/PES are mostly linear elastic up to 0.6% shear strain and show slight nonlinearity afterwards. The average failure shear strains for the AU4- and AS4-BMI/PES are about 0.92%. Thus, a direct comparison of the shear modulus between the two material systems (AU4- and AS4-BMI/PES) can be performed by normalizing the fiber volume fraction of the AU4-BMI/PES from 50% to 70% according to the following equation,¹¹

$$G_{12}^{m} \approx \frac{G_{m}}{V \left[\frac{G_{m}}{G_{f}} + (1 - V_{f}) \right]},$$
(1)

where G_{12}^{m} is the measured shear modulus of the composite material corresponding to a fiber volume fraction V_f ; and G_m (≈ 1.65 GPa) and G_f (≈ 28 GPa) are the shear moduli for matrix and fiber, respectively. Assuming that the shear strain is proportional to the reciprocal of equation (1) in the elastic response range, modified shear stress-strain curves for the AU4-BMI/PES specimens are obtained as shown in Figure 5b. The shear responses of the AU4- and AS4-BMI/PES are coincident. Before normalization of the shear properties, the in-plane shear modulus and strength for the AU4 ($V_f = 50\%$) panel are about 40% lower than those of the AS4 ($V_f = 70\%$) panel. After normalization with respect to 70% fiber volume fraction, the shear modulus and strength for the AU4 ($V_f = 70\%$) panel are about 5% and 6% lower than those of the AS4 ($V_f = 70\%$) panel, respectively, Figure 6.

To evaluate the effect of twisting on the apparent shear strength measurement,⁴ front and back shear responses of the AU4- and AS4-BMI/PES were recorded and compared. It was found that the AU4 specimens were subjected to twisting but the AS4 specimens were not. The twisting of the AU4 specimen is attributed to its large specimen thickness such that the load eccentricity on the specimen edges is more profound.⁴ Typical front and back shear responses of the AU4 and AS4 specimens are shown in Figure 7. From Figure 7a, it is shown that the shear strength of the AU4 specimen was reduced by about 7% due to the twisting effect. Thus, the apparent shear strength obtained from the AU4 and AS4 specimens would be essentially the same if the twisting effect was eliminated from the AU4 specimens.



FIGURE 5 Average of front and back shear stress-strain data of (a) AU4-BMI/PES ($V_f = 50\%$) and AS4-BMI/PES ($V_f = 70\%$), (b) AU4-BMI/PES (normalized $V_f = 70\%$) and AS4-BMI/PES ($V_f = 70\%$) specimens.



FIGURE 6 (a) G_{12}^{*} (before application of correction factor^{4,10}), (b) S_{12} of AU4-BMI/PES and AS4-BMI/PES specimens.



FIGURE 7 Typical front and back shear stress-strain data of (a) AU4-BMI/PES (specimen thickness t = 3.8mm), (b) AS4-BMI/PES (specimen thickness t = 2.5mm) specimens.

The fracture surfaces of the AU4- and AS4-BMI/PES were examined by scanning electron microscopy (SEM) and the resulting micrographs are shown in Figure 8. The AU4-BMI/PES failure surface consisted of clean fibers with little or no matrix debris, fiber pullouts or fiber breaks. Because most of the exposed fibers are devoid of the matrix, the fracture surface appears to be of extensive fiber-matrix interfacial failure. It suggested that the normalization scheme of equation (1) is a valid approxi-





а

FIGURE 8 Scanning electron microscopy (SEM) of the fracture surface of (a) AU4-BMI/PES, (b) AS4-BMI/PES specimens.

mation because there is no sign of matrix failure in the fracture surface, so that the change of fiber spacing will not change the behavior of the matrix materials. For the AS4-BMI/PES fracture surface, clean fibers with some matrix material and fiber bundle breakage were observed. In addition to fiber-matrix interfacial failure as shown by the clean fibers, the matrix debris on some of the exposed fiber surfaces indicated the presence of matrix failure.

One of the primary purposes of surface treatment is to increase the interfacial shear strength of the composite materials. It was shown earlier that the oxygen and nitrogen concentrations on the AS4 fiber are greater than those of the AU4 fiber. However, the shear modulus of the AS4-BMI/PES composite is only about 5% higher than that of the AU4-BMI/PES composite, while the shear strengths of the AU4- and AS4-BMI/PES composites are essentially the same. Thus, the surface chemistry of the carbon fibers is not relevant to the shear strength of the BMI/PES polymer composite. In summary, the effect of fiber surface treatment on the shear properties of the AU4 and AS4 fiber-reinforced BMI/PES composites is insignificant.



FIGURE 9 Average of front and back shear stress-strain data of (a) 50%, (b) 100%, (c) 400% surface-treated IM8-BMI/PES specimens.

(ii) IM8 Fiber Composites

After discarding the shear response of an apparently anomalous specimen (specimen with a very low shear strength), the shear stress-strain data obtained from averaging the front and back shear strains of the strain gaged specimens are shown in Figure 9 for 50%, 100% and 400% surface-treated IM8-BMI/PES specimens, respectively. Each specimen group showed some degree of data scatter. The degree of twisting for specimens of the 50%, 100% and 400% surface-treated panels are equally small, about 4 to 5%, Figure 10. When the shear responses of the 50%, 100% and 400% surface-treated IM8-BMI/PES specimens were presented in one plot, such as Figure 11a, it was found that under an equivalent applied shear stress, the magnitudes of the measured shear strains were in ascending order for the 50%, 100% and 400% surface-treated IM8-BMI/PES specimens, respectively, Figure 11b. The shear moduli and strengths of the three surface-treated IM8-BMI/PES panels are shown in Figure 12. It is shown that the shear moduli (0.02% and 0.2% chord modulus) obtained from the 50%, 100% and 400% surface-treated specimens are in descending order, as suggested by Figure 11. The shear strengths for the 50%



FIGURE 10 Typical front and back shear stress-strain data for (a) 50%, (b) 100%, (c) 400% surface-treated IM8-BMI/PES specimens.



FIGURE 11 (a) Shear stress-strain data (four specimens in each group), (b) typical shear stress-strain data (one specimen in each group) of the 50%, 100% and 400% surface-treated IM8-BM1/PES specimens.



FIGURE 12 (a) G_{12}^{*} (before application of correction factor^{4,10}), (b) S_{12} of surface-treated IM8-BMI/PES specimens.

and 100% surface-treated IM8-BMI/PES panels are essentially the same, while the shear strength of the 400% surface-treated panel is about 4.5% higher than the other two panels. However, it is emphasized here that the 4.5% increase in shear strength of the 400% surface-treated panel is small, and probably insignificant, because some of the measured strengths were within the data scatter of the other two surface-treated panels.

The fracture surfaces of the 50%, 100% and 400% surface treated specimens are very similar, as shown in Figure 13. It was found that the fracture surfaces consisted of clean fibers and fibers with matrix debris. The predominant failures appear to be interfacial/phasial and matrix tearing.

The meso-indentation results corroborate the laminate shear strength sensitivity observed. The characteristic indentation responses are summarized in Table II. The Mean Maximum Hardness Pressure (MMHP) is the applied indenter pressure when interfacial failure was observed. The representative strain is an equivalent contact strain recorded at the point of interfacial failure as well. These values may be regarded as the ultimate stress and strain to effect shearing of the interface. Weibull averages and standard deviations are presented for each quantity in Table II. The interfacial shear strength τ_i is related to the mean maximum hardness pressure (MMHP) by¹²

$$\tau_i = 0.153 \times MMHP \tag{2}$$

Note that the interfacial shear strengths, τ_i , obtained from meso-indentation results are not comparable with the Iosipescu S₁₂ because the two techniques measure a shear response at two distinct levels: a global or laminate level and at a meso-level. Nonetheless, the surface treatment appears not to affect the character of the interface/phase.

% Surface treatment	# Samples	MMHP and standard dev. (MPa)	Representative strain and standard dev.	
50	20	101.5 ± 8.4	0.248 ± 0.023	
100	16	93.4 ± 10.1	0.252 ± 0.023	
400	18	97.0 ± 6.9	0.246 ± 0.013	

TABLE II Meso-Indentation results for the IM8-BMI/15% PES composites given various fiber surface treatments



a

FIGURE 13 Scanning electron microscopy (SEM) of the fracture surface of (a) 50%, (b) 100%, (c) 400% surface-treated IM8-BMI/PES specimens. Index marks are $10 \ \mu m$.



FIGURE 13 (Continued)

CONCLUSIONS

The Iosipescu shear test was applied to evaluate the effect of surface treatment on the shear properties of composite materials. It was shown that the in-plane shear moduli and strengths of the AU4- and AS4-BMI/PES laminates are essentially the same after normalization of the shear properties with respect to the same volume fraction. The effect of degree of fiber surface treatment was also investigated. Meso-indentation results revealed that the interphase/face quality was essentially unchanged by the level of surface treatment. The in-plane shear strengths of the IM8-BMI/PES laminates with 50%, 100% and 400% fiber surface treatment are approximately the same, but the shear moduli (0.02% and 0.2% chord modulus) are in of descending order for 50%, 100% and 400% fiber surface treatments. Note that while the Iosipescu shear test provides accurate measurement of composite material shear modulus, the shear strength obtained is not the true shear strength of the material system due to the mixed mode failure. Nevertheless, the apparent shear strength can be used as a reference value in the comparison of processing variables in the manufacturing of composite materials.

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