MECHANICAL BEHAVIOUR OF WOVEN GRAPHITE/POLYIMIDE COMPOSITES WITH MEDIUM AND HIGH MODULUS GRAPHITE FIBERS SUBJECTED TO BIAXIAL SHEAR DOMINATED LOADS

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SUMMARY: A major limitation of woven fiber/polymer matrix composite systems is the inability of these materials to resist intralaminar and interlaminar damage initiation and propagation under shear-dominated biaxial loading conditions. There are numerous shear test methods for woven fabric composites, each with its own advantages and disadvantages. Two techniques, which show much potential, are the Iosipescu shear and ±45° tensile tests. In this paper, the application of these two tests for the room and high temperature failure analyses of woven graphite/polyimide composites is briefly evaluated. In particular, visco-elastic micro-, meso- and macro-stress distributions in a woven eight harness satin (8HS) T650/PMR-15 composite subjected to these two tests are presented and their effect on the failure process of the composite is evaluated. Subsequently, the application of the Iosipescu tests to the failure analysis of woven composites with medium (T650) and high (M40J and M60J) modulus graphite fibers and PMR-15 and PMR-II-50 polyimide resins is discussed. The composites were tested as-supplied and after thermal conditioning. The effect of temperature and thermal conditioning on the initiation of intralaminar damage and the shear strength of the composites was established. It has been shown in this work that the onset of intralaminar damage and the shear strength of the T-650/PMR-15 composite are very strongly dependent on the type of test. The biaxial tension/shear stress fields in the gage sections of the ±45° specimens resulted in much lower estimates of the critical loads for the initiation of intralaminar damage and the composite strength in comparison with the Iosipescu test data at both room temperature and at 316°C. This agreed very well with the conclusions from visco-elastic micro- and meso-stress analyses. It has also been shown in this research that the room and high temperature (at 316°C) ultimate shear strengths of the T-650/PMR-15, M40J/PMR-II-50 and M60J/PMR-II-50 composite systems tested using the Iosipescu shear test increase linearly with an increase in the strains at failure of the T-650, M40J and M60J fibers. The room and high temperature shear strengths of the M60J fiber system are the lowest followed by the strengths of the M40J and subsequently the T-650 fiber composite systems. No noticeable effect of the thermal conditioning and moisture on the shear strengths of the M40J and M60J fiber composites was found. However, the critical shear stresses for the initiation of tow cracking in the M40J and M60J fiber Iosipescu specimens increased substantially after pre-conditioning.

KEYWORDS: woven graphite/polyimides, shear testing, high modulus fibers, modeling

INTRODUCTION

Considerable efforts have been underway to develop multidisciplinary technologies for affordable propulsion engine components that will enable the system to operate at higher temperatures with reduced cooling while sustaining performance and durability. As part of these efforts, high temperature polymer matrix composites and fabrication technologies are being developed suitable for manifolds, combustion chamber supports and attachments [1]. The use of such composites should allow the replacement of heavy metal engine components to provide a high thrust to weight ratio. Graphite/polyimide woven composites are good candidates for these applications. However, one of the limitations of such composites for engine applications is their low strength under shear and biaxial shear dominated loading conditions at room and elevated temperatures [2-7]. Accurate experimental in-plane shear properties are essential for material selection and also as design data. While T650/PMR-15 composites were focused on high strength/medium stiffness applications, the combustion chamber support structure requires much stiffer fibers. The two candidates that have received a considerable amount of attention are M40J/PMR-II-50 and M60J/PMR-II-50 composite systems [1,7]. The M40J and M60J fibers are graphite fibers with significantly increased longitudinal stiffness properties in comparison with their T650 counterparts [7]. The PMR-II-50 matrix is a polyimide resin with improved high temperature performance compared to PMR-15 [7]. During manufacturing of graphite/polyimide composites, large residual thermal stresses are generated in the composites [6-12]. A greater discrepancy between the thermal expansion coefficients of the fibers and that of the resin, and a larger temperature difference between the manufacturing and in-service temperatures leads to the generation of higher residual thermal stresses. The non-zero state of stress at room temperature can significantly affect the strength of the composite materials, especially at low temperatures.

Recently, the residual thermal stresses in unidirectional and woven graphite fiber/polyimide matrix composites have been investigated by Benedikt et al. [8-10] by performing X-ray diffraction experiments on embedded metallic inclusions and by calculating residual stresses in the composites from the measured residual stresses in the particles. It was shown that the interlaminar residual thermal stresses in an eight harness satin (8HS) T650/PMR-15 polyimide matrix composite can be especially high, almost equal to the tensile strength of the polyimide resin [10]. It has also been recently shown by Rupnowski and Kumosa [12] that the effect of residual stresses cannot be ignored in the failure analysis of 8HS graphite/PMR-15 composites subjected to biaxial shear dominated loads. Both the micro- and meso-intralaminar stresses in the composite were found to be very high, affecting the strength of the composite under in-plane biaxial loads [12].

Since the manufacturing temperature of the M40J/PMR-II-50 and M60J/PMR-II-50 composites (371°C) is higher than that of the T650/PMR-15 system (316°C) and the stiffness properties of the M40J and M60J fibers are higher than those of the T650-35 fibers [7], the residual thermal stresses in these two composites could be very high and tow micro-cracking during manufacturing could be significant. If present, the micro-cracks could seriously reduce the strength of the composites, especially when tested under in-plane biaxial shear dominated loads. Therefore, the primary objective of this study was to evaluate the in-plane shear strength properties of the composites based on the high stiffness M40J and M60J graphite fibers with the PMR-II-50 resin at room and elevated temperatures (at 316°C) and compare them with the shear strength of the T650/PMR-15 composite.

MACRO-, MESO- AND MICRO-STRESS ANALYSES OF WOVEN IOSIPESCU AND ±45° SPECIMENS

The T650, M40J and M60J graphite fiber/polyimide woven composites were investigated using the Iosipescu shear and ±45° tensile [2-7] tests. To understand the effect of specimen geometries, loading conditions, composite architectures, temperature, manufacturing, etc., both tests have been numerically evaluated using finite element techniques on the macro-, meso- and micro-levels [2-7, 11-14].

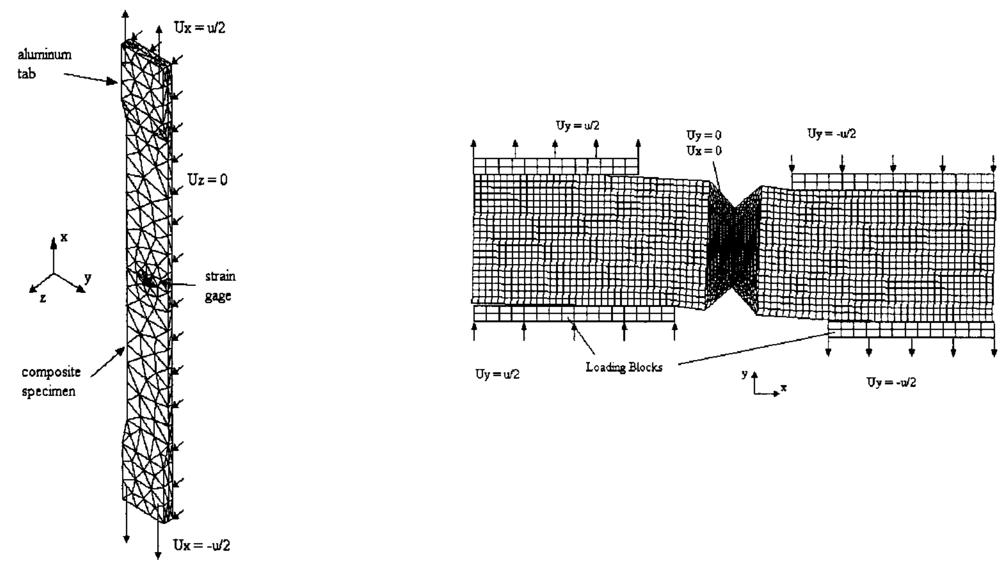

Figure 1. Finite element representations of the ±45° tensile (left) and Iosipescu (right) specimens [3-5].

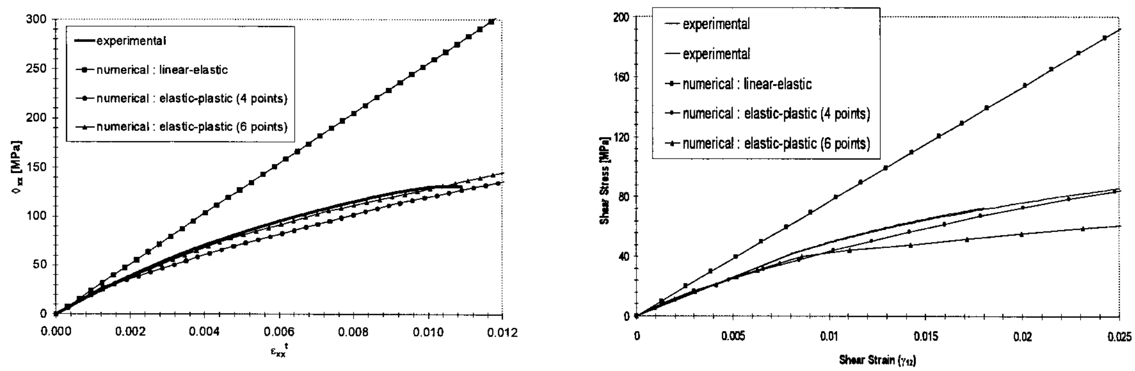

Figure 2. Shear stress/shear strain diagrams from the ±45° tensile (left) and Iosipescu shear (right) tests performed on the 8HS T650/PMR-15 composite [3-5].

An elastic-plastic, time-independent, macroscopic, homogenous model of an 8HS woven graphite/PMR-15 composite material has been developed by Odegard et al. [13] that predicts the non-linear response of the material subjected to shear-dominated biaxial loads. The model has been used to determine the response of woven composite Iosipescu shear and ±45° tensile test specimens in non-linear finite element analyses (Fig. 1) using a multi-linear averaging technique [3-5]. The finite element computations of the ±45° and Iosipescu tests were fully non-linear with the material, geometrical, and boundary contact non-linearities considered. It has been shown that the numerically calculated stress-strain diagrams of the off-axis specimens are very close to the experimentally obtained curves for small strains without inter- and intralaminar damage (Fig. 2). It was also indicated that the non-linear effects had to be considered in the modeling of the composite under in-plane shear dominated biaxial loads. Large differences were found in the mechanical response of the composite to shear when modeled under linear and non-linear conditions (Fig. 2). It was also shown [3-5] that the stress distributions in the Iosipescu and ±45° woven specimens were entirely different. In the gage sections of the woven Iosipescu specimens, the stress fields are essentially pure shear (Fig. 3, left) whereas the ±45° test generated biaxial tension/tension and shear stress fields (Fig. 3, right).

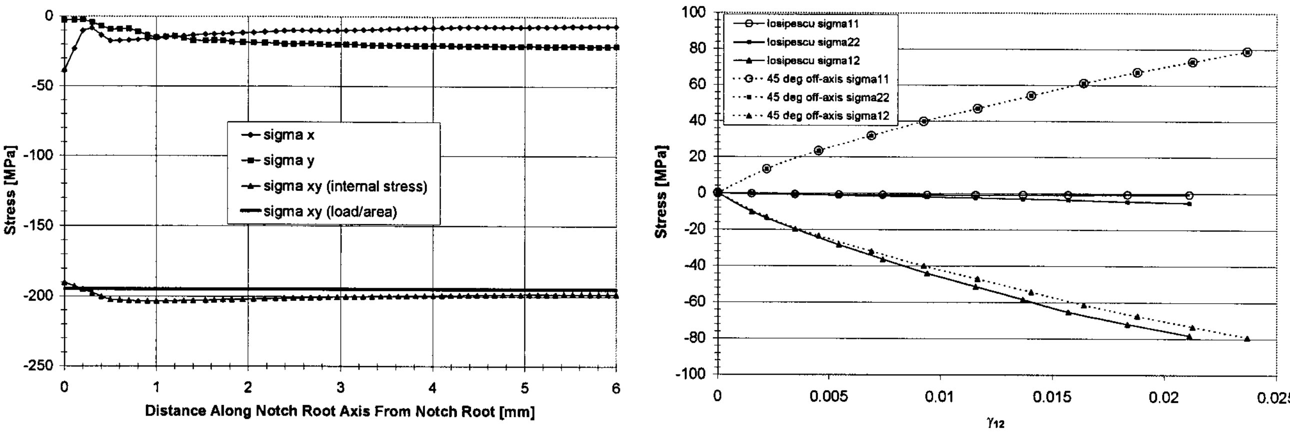

Figure 3. Stress distributions in the gage sections of the 8HS T650/PMR-15 Iosipescu shear (left) and ±45° tensile (right) specimens [3-5].

In the macro-stress analysis of the Iosipescu and ±45° specimens, the effect of composite manufacturing on the internal stress distributions was not considered. This was done however by performing visco-elastic computations of both residual and applied stresses in the 8HS T650/PMR-15 composite on the meso- and micro-scales (Fig. 4) [12]. The composite manufacturing conditions, visco-elastic properties of the polyimide resin, orthotropic properties of the graphite fibers and composite architecture (weave type, fiber distribution, etc.) were considered in the stress computations performed for a variety of in-plane biaxial loading conditions ranging from pure tension, biaxial tension along the warp and fill tows, biaxial tension and shear, and pure shear [12].

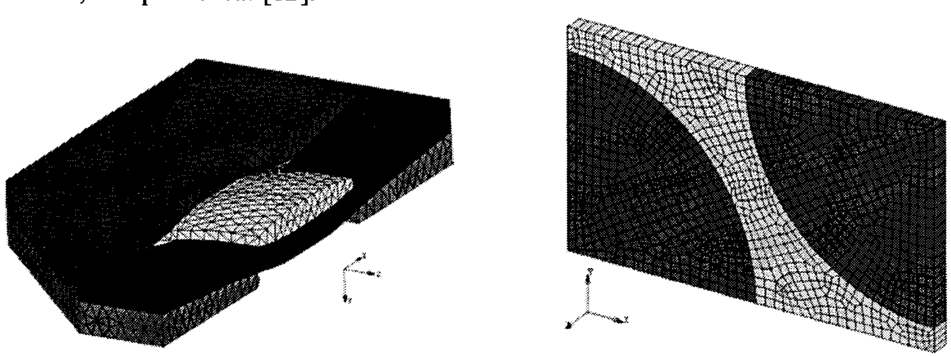

Figure 4. Finite element representations of the 8HS graphite/polyimide composite for the visco-elastic meso-stress (left) and micro-stress (right) analyses [12].

The visco-elastic computations of the internal stresses on the meso- and micro-scales in the 8HS graphite (T650)/PMR-15 composite have shown that the local stress concentrations in the composite are very strongly dependent on the loading conditions (Table 1). Under biaxial loads consisting of biaxial tension applied in the direction of the warp and fill tows in addition to in-plane shear, there exist the largest principal stresses in the polyimide matrix at the fiber matrix interface. The meso-stress analysis has also shown that this type of biaxial loading creates the most favorable combination of meso- transverse and shear stresses for the initiation of transverse tow cracks in the composite. It has also been stressed that for the accurate evaluation of stress distributions in the woven graphite/polyimide composites on both the meso- and micro-levels, the residual thermal stresses from composite manufacturing must be considered in the stress analysis [12].

Table 1. Meso- and micro-stresses in the center of the tow in the undulation region of 8HS T650/PMR-15 for various external in-plane loads at room temperature [12].

	Meso-stresses [MPa]				Micro-stresses [MPa]
Load case	σ_{XX} (transverse in-plane)	σ_{ZZ} (longitudinal in-plane)	σ_{YY} (transverse out of plane)	σ_{XZ} (in-plane shear)	Max. principal stresses in the matrix
Case 1 (ΔT only)	64	-119	18	-1	94
Case 2 (ΔT+TX)	79	-129	16	-1	107
Case 3 (ΔT+TZ)	68	145	20	-1	102
Case 4 (ΔT+S)	66	-98	19	108	208
Case 5 (ΔT+TX+TZ)	83	136	17	0	111
Case 6 (ΔT+TX+S)	81	-107	17	109	222
Case 7 (ΔT+TZ+S)	70	167	20	109	216
Case 8 (ΔT+TX+TZ+S)	85	158	18	110	230

(ΔT residual stresses only, TX(Y) applied in-plane tension along the tows, TZ transverse tension and S in-plane shear).

The stress data obtained from the micro- and meso-stress analyses agreed very well with the experimental strength results obtained by performing Iosipescu shear and ±45° tensile tests on the 8HS T650/PMR-15 composite at room and elevated temperatures. Under almost pure in-plane shear (Iosipescu test), the shear stresses for the initiation of intralaminar damage and at the maximum loads were significantly higher than those from the case of biaxial tension and shear (±45° tensile test). This effect can be clearly seen in Table 2 [4-6].

Table 2. Shear stresses at the onset of intralaminar damage and at the maximum loads from the ±45° and Iosipescu tests at room and 316°C temperatures for the 8HS T-650/PMR-15 system [4-6].

	Shear Stresses at the Onset of Intralaminar Damage [MPa]		Shear Stresses at Maximum Loads [MPa]	
Test	±45°	Iosipescu	±45°	Iosipescu
at RT [1, 2]	56.6 ± 2.0	94.8 ± 1.3	82.0 ± 0.15	105.8 ± 2.6
at 316°C	37.3 ± 5.2	59.9 ± 1.2	50.8 ± 6.0	71.8 ± 4.2

SHEAR STRENGTH OF WOVEN MEDIUM AND HIGH MODULUS GRAPHITE FIBER/POLYIMIDE COMPOSITES

The room and elevated temperature shear strength properties of the 8HS T650/PMR-15 system were subsequently compared to the properties of the 4HS M40J/PMR-II-50 and M60J/PMR-II-50 composites obtained from the Iosipescu shear tests [7]. Both the shear stresses for the initiation of intralaminar damage and the shear strengths determined at the maximum loads were compared at room temperature and at 316°C. The selected fiber and composite properties are shown in Tables 3a and 3b, respectively [7]. It can be seen in Table 3a that the high modulus M40J and M60J fibers exhibit considerably reduced tensile strengths in comparison with the T650 fibers. Especially, their strains at failure are significantly reduced.

Table 3a. Selected mechanical properties and coeeficients of thermal expansion (CTE) of T650,

M40J and M60J fibers [7].

Fibers \ Properties	E_L [GPa]	E_T [GPa]	Strength [GPa]	Strain at Failure [%]	CTE_L [10^{-6}/°C]	CTE_T [10^{-6}/°C]
T650-35	241	20	4.55	1.7	-0.5	10
M40J	377	N/A	4.41	1.2	-0.83	N/A
M60J	588	N/A	3.92	0.7	-1.1	N/A

Table 3b. Fiber, resin, void contents, T_g and T_d for the three composite systems [7].

Composite \ Property	Fiber by volume [%]	Matrix by weight [%]	Void fraction [%]	Tg [°C]	Td [°C]
T650-35/PMR-15	61.38	31.06	1.35	342	473
M40J/PMR-II-50	58.13	35.98	1.80	389	N/A
M60J/PMR-II-50	58.26	34.1	1.32	376	N/A

The 8HS T650-35/PMR-15 composite system was investigated after post-curing, as-supplied. The high modulus graphite fiber composites based on the M40J and M60J fibers were tested as supplied (after post-curing), and after thermo-cycling [7]. Thermo-cycling was carried out on dried composite panels by heating them from room temperature to 316°C in two minutes, held at 316°C for ten seconds and rapidly cooled with air to room temperature. The panels were thermo-cycled two hundred times. After thermo-cycling Iosipescu specimens were cut from the panels. After cutting, a set of specimens was hydrated in a moist environment prior to testing.

a b c

Figure 5. Optical micrographs of as supplied 8HS T650-35/PMR-15 (a), 4HS M40J/PMR-II-50 (b) and 4HS M60J/PMR-II-50 composites [7].

The residual thermal stresses in the M40J and M60J fiber composites with the PMR-II-50 resin were found to be much higher than in the case of the T-650/PMR-15 composite [7]. This resulted in the formation of multiple transverse micro-cracks in the fill and warp tows during manufacturing (Fig. 5), particularly in the case of the M60J fiber system [7]. Thermo-cycling and moisture only slightly increased the number of tow cracks in comparison with the as-supplied composites [7]. No tow micro-cracks were found in the as supplied T650/PMR-15 system. The micro-features of the tow cracking in the M40J and M60J/PMR-II-50 composites were found to be very similar (Fig. 6). In both cases, the resin is pulled away from the fiber/resin interface. A closer investigation of individual fibers leads to the conclusion that the resin is pulled away from that region of the fiber that is facing a large localized concentration of resin. When the propagating crack reaches a region of resin it is disrupted, and the resin appears to be stretched and not broken and the crack starts again after the resin rich region. The exact magnitudes of the intralaminar and interlaminar residual stresses in the M40J and M60J fiber composites cannot be determined at present due to the lack of visco-elastic properties of the PMR-II-50 resin and the whole set of the physical properties of the fibers.

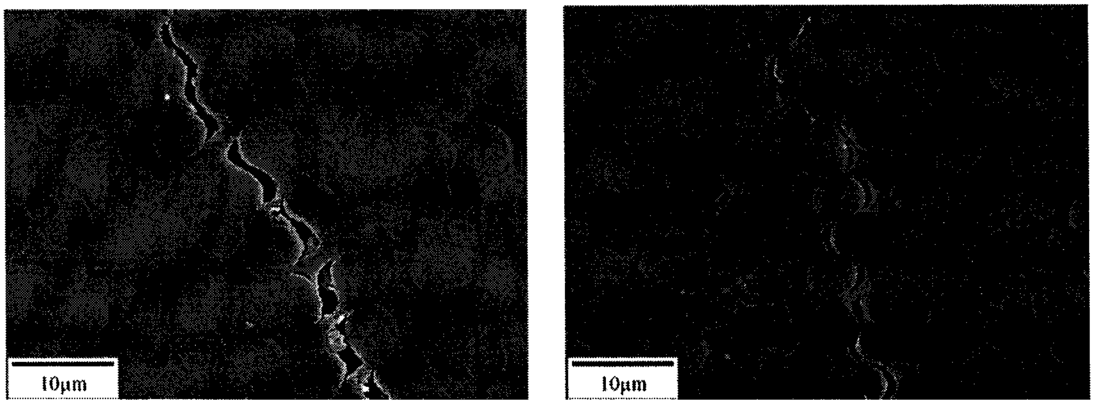

Figure 6. Micro-cracking in the as-supplied M40J/PMR-II-50 (left) and as supplied M60J/PMR-II-50 (right) composites [7].

Iosipescu test specimens were displaced at a rate of 1.27 mm/min. until failure. For modulus testing at room temperature, Micro-Measurements Division Measurements Group Inc. WK series 0°, 45°, 90° strain gage rosettes were used. The gages were mounted on the front and back surfaces and the effect of specimen twisting was evaluated. Iosipescu shear testing at 316°C was performed in a MTS 651.10E-04 environmental chamber. The Iosipescu fixture and supports were heated in the chamber at 5.7°C/min. to 316 ±1°C and held for one hour. After the hold, the chamber was opened and the test specimen was mounted in the Iosipescu fixture. Then, the chamber was closed and the temperature of the gage section on each side of the specimen was monitored. Testing proceeded ten minutes after installation of the specimen into the fixture. The shear moduli of the composites were not evaluated at 316°C. Acoustic emission (AE) was monitored during the room and high temperature Iosipescu tests. Signal location techniques were used to eliminate AE signals generated by the loading blocks of the Iosipescu fixture [4-7]. The shear stresses at the significant onset of AE were determined and used as the shear stresses for the initiation of intralaminar damage in the composites.

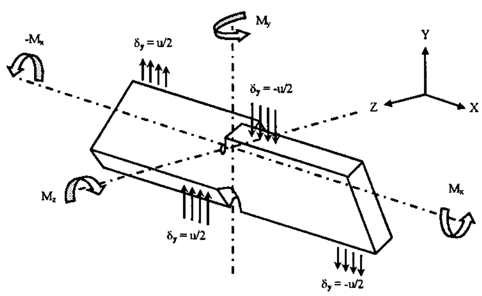

Figure 7. Iosipescu specimen twisting

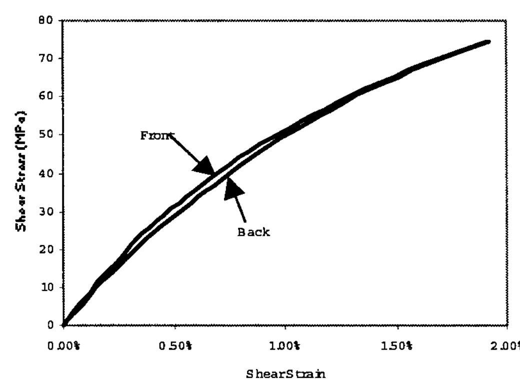

Figure 8. Room temperature shear stress/shear strain diagrams for the M40J/PMR-II-50 composite from the front and back strain gages.

The effect of possible out of plane deformations on the shear stress/shear strain response of the composites was evaluated at room temperature by placing strain gages on the front and back of the T650, M40J and M60J fiber specimens. In addition, the effect out-of-plane specimen deformations on the shear modulus determination of unidirectional and woven T650/PMR-15 composites was numerically investigated by Searles et al. [14]. Various out of plane deformations of the Iosipescu specimens (twisting (Fig. 7), bending, combined twisting and bending) were studied and the type of deformation with the strongest undesirable effect on the shear modulus determination was established [14]. Since the shear stress/strain response measured by the back and front gages was very similar (Fig. 8) for all three composites, it was concluded that the effect of undesirable out of-plane deformations could be neglected at RT [7]. This observation was important since the M40J and M60J fiber Iosipescu specimens were more than two times thinner than the T650/PMR-15 specimens.

Table 4. Shear stresses for the initiation of intralaminar damage and at the maximum loads at room temperature and at 316°C for the woven T650, M40J and M60J fiber composites [7]

Composite System	At Room Temperature		At 316°C	
	Shear Stresses at the Significant Onset of AE [MPa]	Shear Stresses at Maximum Loads [MPa]	Shear Stresses at the Significant Onset of AE [MPa]	Shear Stresses at Maximum Loads [MPa]
T-650/PMR-15	94.8 ± 1.3	105.8 ± 2.6	59.9 ± 1.2	71.8 ± 4.2
M40J/PMR-II-50	30.3 ± 3.1	86.7 ± 2.7	56.5 (one test) (**54.9 ± 4.3)	61.2 ± 4.1
M60J/PMR-II-50	9.9 ± 2.5	63.8 ± 0.4	(*** 40.0 ± 5.8)	50.2 ± 0.8

Number of * are the number of tests with onset of AE after the maximum load.

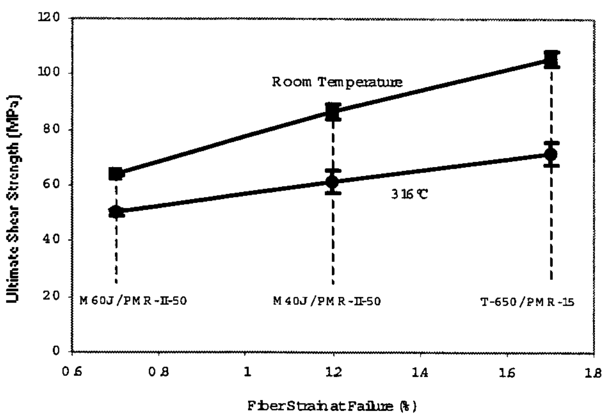

Figure 9. Shear strength of the as supplied T-650, M40J and M60J fiber composites at room temperature and 316°C as a function of the fiber strain at failure [7].

There exists a very noticeable effect of residual thermal stresses on the initiation of intralaminar damage in the composites, as determined by acoustic emission (Table 4). The large magnitudes of residual stresses in the M60J fiber composite resulted in the formation of the cracks under very low shear stresses at room temperature. At this temperature, the critical shear stresses for the initiation of tow cracking are much higher in the M40J/PMR-II-50 and the T-650/PMR-15 composites.

The room and high temperature (at 316°C) ultimate shear strengths of the T650-35/PMR-15, M40J/PMR-II-50 and M60J/PMR-II-50 composite systems tested using the Iosipescu shear test increase linearly with an increase in the strains at failure of the T650-35, M40J and M60J fibers (Fig. 9, Table 4) [illegible] room and high temperature shear strengths of the M60J fiber system are the lowest followed [illegible] strengths of the M40J and subsequently T650-35 fiber composite systems. There is no noti[illegible]ct of the thermal conditioning and moisture on the shear strengths of the M40J and M60J [illegible] composites (Fig. 10) However, the critical shear stresses for the initiation of tow micro-cracking [illegible] M40J and M60J fiber Iosipescu specimens increased substantially after pre-conditioning (Fig. 11). This indica[illegible] thermo-cycling (by the formation of additional tow micro-cracks) and moisture can significantly r[illegible] residual thermal stresses in the composites at room temperature. At 316°C the effect of the residual [illegible] the initiation of damage in the as-supplied and pre-conditioned composites [illegible] [7].

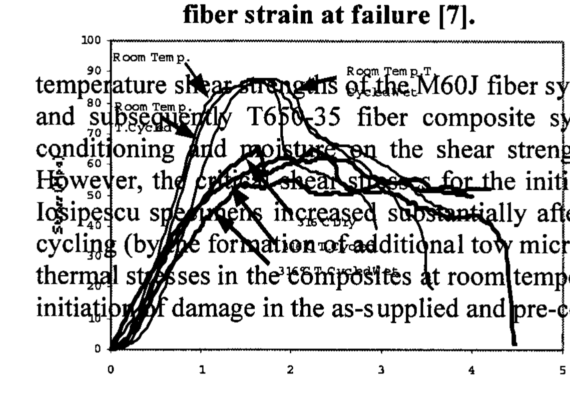

Figure 10. Typical stress vs. displacement curves for the M40J/PMR-II-50 Iosipescu specimens tested at room and at 316°C under dry, thermo-cycled and thermo-cycled with moisture condition [7].

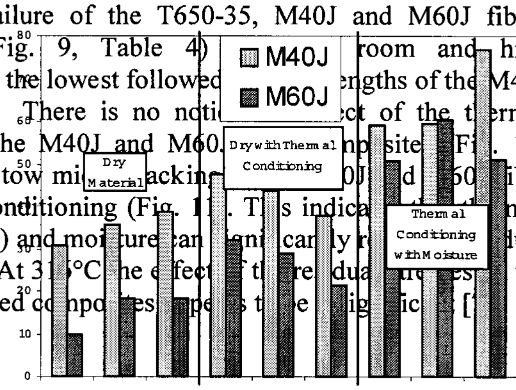

Figure 11. Intralaminar damage initiation shear stresses (% of the maximum stresses) for the M40J/PMR-II-50 and M60J/PMR-II-50 specimens tested at room temperature [7].

CONCLUSIONS

In this work, a combined numerical/experimental testing methodology has been described for the determination of mechanical response of woven graphite/polyimide composites subjected to either shear (Iosipescu test) or biaxial shear dominated (±45° test) in-plane loads. The methodology was successfully used to determine the initiation of intralaminar damage and the shear strengths of medium and high modulus graphite fiber composites with PMR-15 and PMR-II-50 polyimide resins. The room and elevated temperature testing of the composites was supported by performing comprehensive macro-, meso- and micro-stress analyses using non-linear finite element techniques. It has been shown in this research that the effect of residual thermal stresses on the prediction of intralaminar damage initiation in the composites should not be ignored. This effect can only be examined if the visco-elastic response of a polyimide matrix is considered in the meso- and micro-finite element computations. It has also been shown that the initiation of intralaminar damage in the composites is very strongly dependent on the in-plane loading conditions. It was found that the combined effect of biaxial tension along the warp and fill tows and in-plane shear created the most suitable stress conditions for the initiation of tow micro-cracking. Strong effects of fiber type and manufacturing conditions were found on the magnitudes of residual stresses, tow micro-cracking and the shear strength of the composites. It was also presented in this research that both the thermo-cycling and moisture significantly reduced the residual thermal stresses and increased the critical shear stresses for the initiation of tow micro-cracking.

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