ANALYSES OF FAILURE MECHANISMS IN WOVEN GRAPHITE/POLYIMIDE COMPOSITES WITH MEDIUM AND HIGH MODULUS GRAPHITE FIBERS SUBJECTED TO IN-PLANE SHEAR

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ABSTRACT

- The application of the Iosipescu shear test for the room and high temperature failure analyses of the woven graphite/polyimide composites with the medium (T-650) and high (M40J and M60J) modulus graphite fibers is discussed. The M40J/PMR-II-50 and M60J/PMR-II-50 composites were tested as supplied and after thermal conditioning. The effect of temperature and conditioning on the initiation of intralaminar damage and the shear strength of the composites was established.

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BACKGROUND

- **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. The usage of such composites should allow the replacement of heavy metal engine components to provide a high thrust to weight ratio.**

BACKGROUND

- 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. Accurate experimental in-plane shear properties are essential for material selection decision and also as design data.

- While the T650-35/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.

FIBER PROPERTIES

Properties / Fibers	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

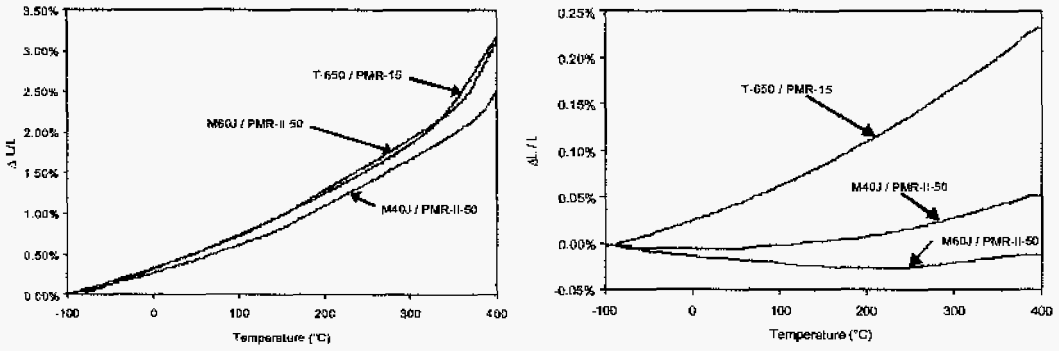
The M40J and M60J fibers are graphite fibers with significantly increased longitudinal stiffness properties in comparison with their T650-35 counterparts. The PMR-II-50 matrix is a polyimide resin with improved high temperature performance compared to PMR-15.

COMPOSITE PROPERTIES

Composite \ Properties	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

Since the manufacturing temperature of the M40J/PMR-II-50 and M60J/PMR-II-50 composites (371°C) is higher than that of the T650-35/PMR-15 system (315°C) and the stiffness properties of the M40J and M60J fibers are higher than those of the T650-35 fibers, the residual thermal stresses in these two composites could be very high and tow micro-cracking during manufacturing could be significant affecting the strength of the composites, especially when tested under in-plane biaxial shear dominated loads.

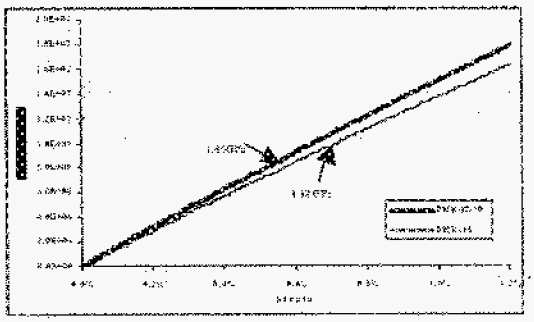
COMPOSITE PROPERTIES



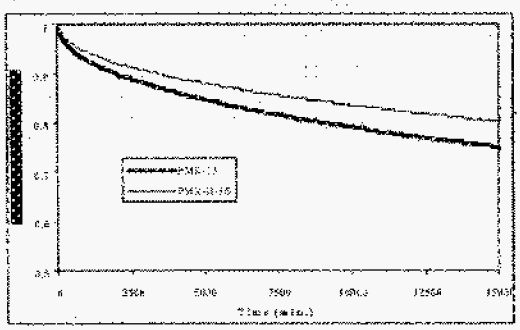
a **b**

Thermal expansions out-of-plane (a) and in-plane (b) for the T-650/PMR-15, M40J/PMR-II-50 and M60J/PMR-II-50 composites as a function of temperature.

COMPARISON BETWEEN PMR-15 AND PMR-II-50 RESINS



a



b

Room temperature shear stress/shear strain curves for PMR-15 and PMR-II-50 resins (a) and the normalized isothermal length relaxation of PMR-15 at 315°C and PMR-II-50 at 365°C (b).

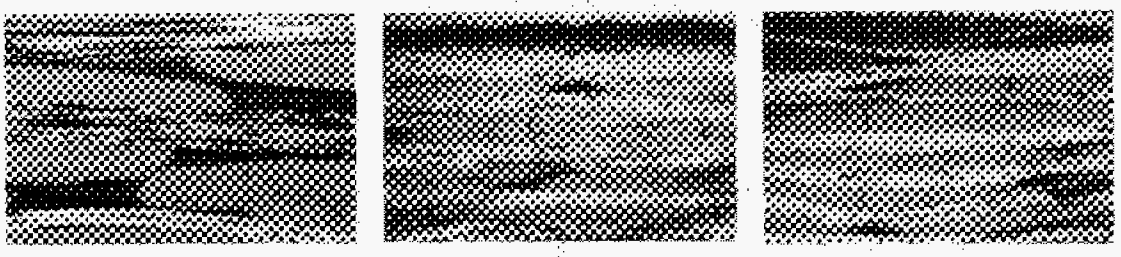
PRE-CONDITIONING OF M40J and M60J/PMR-II-50

8HS T650-35/PMR-15 tested as supplied only.

4HS M40J/PMR-II-50 and M60J/PMR-II-50 were tested

- **as-supplied,**
- **after thermo-cycling; thermo-cycling was carried out on dried panels by heating 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.**
- **thermo-cycling with water added; thermo-cycled specimens were hydrated in a moist environment prior to testing.**

TOW MICRO-CRACKING DURING MANUFACTURING

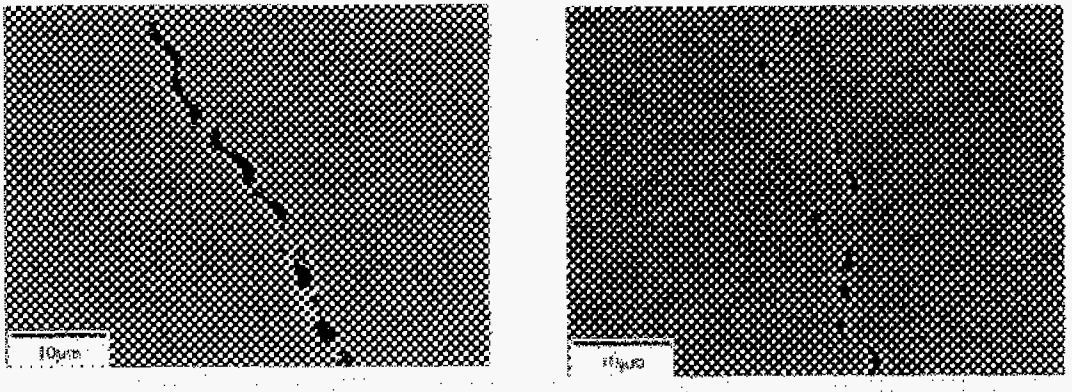


a b c

Optical micrographs of as supplied 8HS T-650/PMR-15 (a), 4HS M40J/PMR-50 (b) and 4HS M60J/PMR-II-50 composites.

- Thermo-cycling and moisture only slightly increased the number of tow cracks in comparison with the as-supplied M40J and M60J/PMR-II-50 composites.

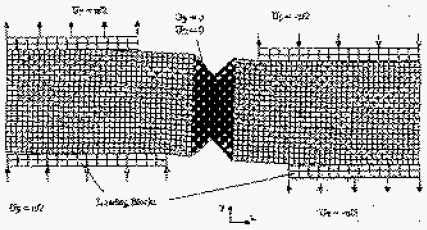
TOW MICRO-CRACKING DURING MANUFACTURING



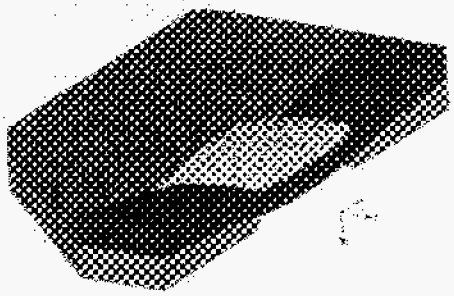
a b

Micro-cracking in the as-supplied M40J/PMR-II-50 (a) and as-supplied M60J/PMR-II-50 (b) composites.

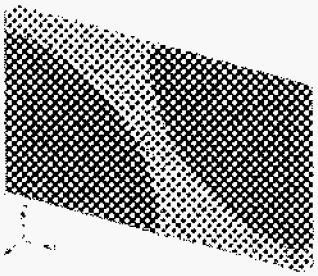
MACRO-, MESO- AND MICRO-FINITE ELEMENT REPRESENTATIONS



Iosipescu Shear Specimen



Meso-Representative Volume Element (RVE)



Micro-Representative Volume Element (RVE)

FINITE ELEMEM SIMULATIONS OF WOVEN GRAPHITE/POLYIMIDE COMPOSITES (Capabilities and Limitations)

- **Macro-stress/strain distributions (on the structural level) in woven structures can be numerically predicted as a function of temperature, multiaxial loading conditions and composite architectures. However, the effect of residual stresses on the mechanical response of the composites cannot be evaluated using the macro-approach. This can only be done using the micro- and meso- approaches.**
- **Meso-stress and strain distributions (on the tow level) can be numerically predicted in the PMR-15 type of composites at room and elevated temperature with residual stresses under any loading conditions and composite architectures. At high temperatures, the effect of plastic deformation of the PMR-15 resin cannot be consider (no elasto-visco-plastic properties of PMR-15 are available).**
- **Stress and strain distribution (on the fiber level) can be numerically predicted under any loading conditions, composite architectures and temperatures in the PMR-15 type composites without considering the plastic deformation of the resin.**

MESO- RESIDUAL STRESSSES IN 8HS T650/PMR-15, 4HS M40J/PMR-II-50 and 4HS M60J/PMR-II-50

Residual Stresses / Composite Systems	σ_x (transverse in-plane) [MPa]	σ_z (transverse through-the-thickness) [MPa]
8HS T650-35 PMR-15	62	20
4HS M40J/PMR-II-50 (PMR-15 = PMR-II-50)	78	13
4HS M60J/PMR-II-50 (PMR-15 = PMR-II-50)	81	13

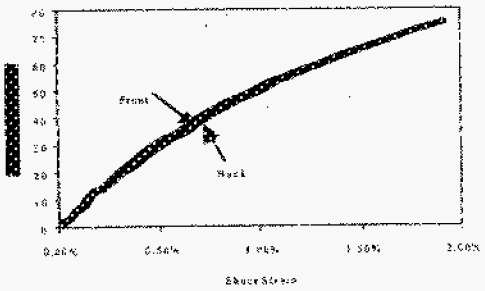
Numerically estimated residual intralaminar meso-stresses in plane and through the thickness in the three composite systems.

IOSIPESCU SHEAR TESTS -TESTING CONDITIONS

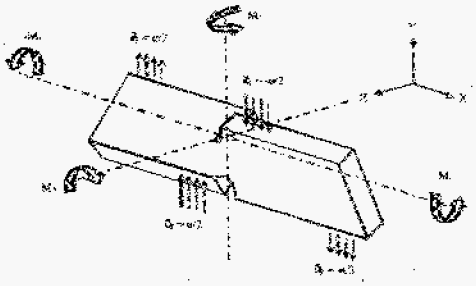
Iosipescu test specimens were displaced at a rate of 1.27 mm/min. until failure. For modulus testing at room temperature, Micro-Measurements Group Inc. WK series 0°, 45°, 90° strain gage rosettes were used. The gages were mounted on the front and back surfaces and the effect of specimen twisting was 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 modulus was not evaluated at 316°C.

IOSIPESCU SHEAR TESTING OF 8HS T650/PMR-15, 4HS M40J/PMR-II-50 and 4HS M60J/PMR-II-50 COMPOSITES

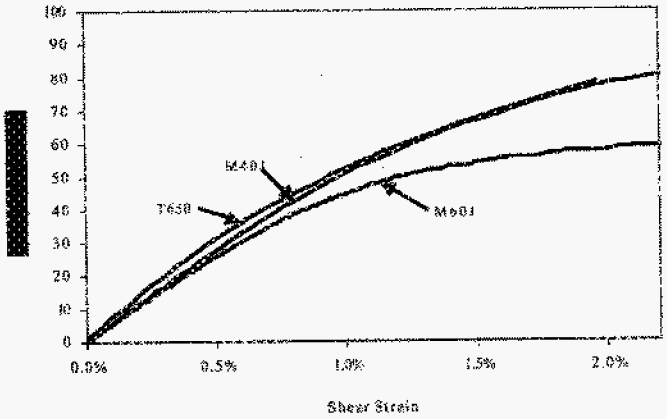


Shear stress/shear strain curves from the back and front strain gages for the M40J/PMR-II-50 composite tested at RT



An example of shear (in-plane) and out-of-plane deformations of Iosipescu specimens

IOSIPESCU SHEAR TESTING OF 8HS T650/PMR-15, 4HS M40J/PMR-II-50 and 4HS M60J/PMR-II-50 COMPOSITES



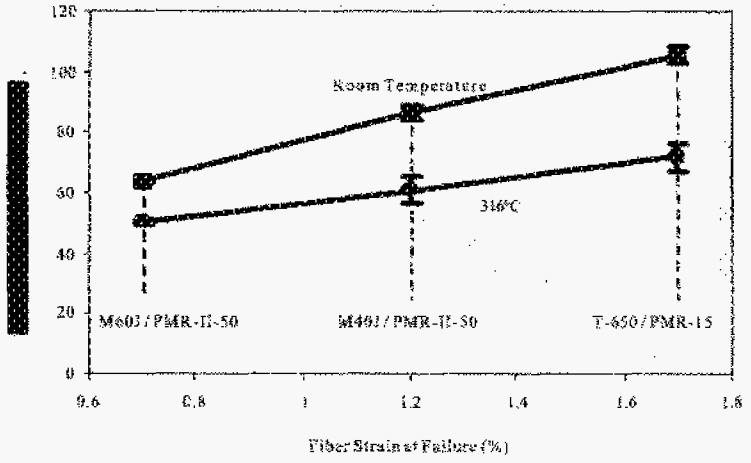
Shear stress vs. shear strain curves for the T-650/PMR-15, M40J/PMR-II-50 and M60J/PMR-II-50 composites (as supplied) tested at room temperature.

IOSIPESCU SHEAR TESTING OF 8HS T650/PMR-15, 4HS M40J/PMR-II-50 and 4HS M60J/PMR-II-50 COMPOSITES

	At Room Temperature		At 316°C	
Composite Systero	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

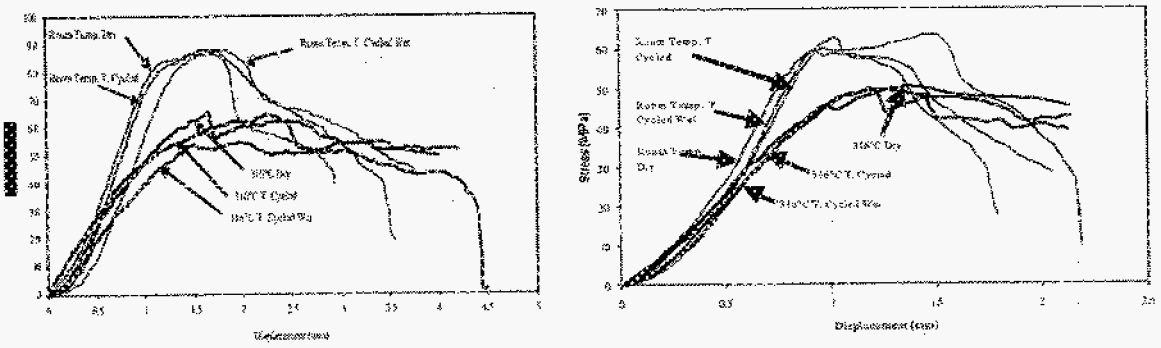
Number of * are the number of tests with onset of AE after the maximum load.

IOSIPESCU SHEAR TESTING OF 8HS T650/PMR-15, 4HS M40J/PMR-II-50 and 4HS M60J/PMR-II-50 COMPOSITES



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.

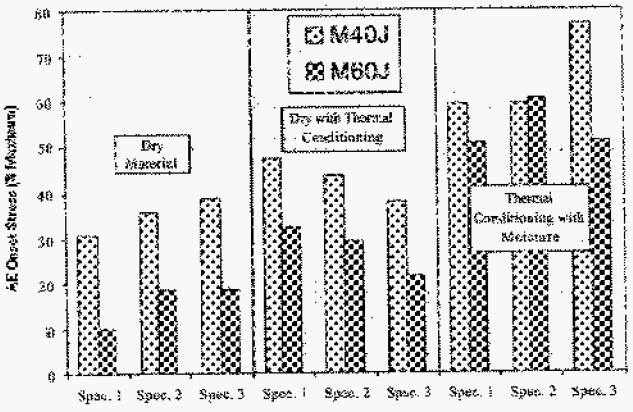
EFFECT OF PRE-CONDITIONING ON THE SHEAR STRENGTH OF M40J/PMR-II-50 AND M60J/PMR-II-50



a b

Typical shear stress vs. displacement curves for the M40J/PMR-II-50 (a) and M60J/PMR-II-50 (b) Iosipescu specimens tested at room and at 316°C under dry, thermo-cycled and thermo-cycled with moisture conditions.

EFFECT OF PRE-CONDITIONING ON THE SHEAR STRENGTH OF M40J/PMR-II-50 AND M60J/PMR-II-50



AE initiation stresses (% of the maximum stresses) for all the M40J/PMR-II-50 and M60J/PMR-II-50 specimens tested at room temperature.

- **The critical shear stresses for the initiation of new tow cracking in the M40J and M60J fiber Iosipescu specimens subjected to shear increased substantially after pre-conditioning.**
- **At 316°C the effect of the residual stresses on the initiation of damage in the as-supplied and pre-conditioned composites appears to be insignificant.**

EFFECT OF PRE-CONDITIONING ON THE SHEAR STIFFNESS OF M40J/PMR-II-50 AND M60J/PMR-II-50

Conditioning / System	Dry [GPa]	Dry and Thermo-cycled [GPa]	Thermo-cycled and moisture [GPa]
M40J/PMR-II-50	5.78	5.29	5.75
M60J/PMR-II-50	5.72	5.63	5.67
T650-35/PMR-15	6.97	N/A	N/A

Shear moduli of the T650-35/PMR-15, M40J/PMR-II-50 and M60J/PMR-II-50 at room temperature as a function of pre-conditioning

CONCLUSIONS

1. The magnitudes of the residual thermal stresses in the M40J and M60J fiber composites with the PMR-II-50 resin are much higher than in the case of the T650-35/PMR-15 composite. This resulted in the formation of multiple transverse micro-cracks in the fill and warp tows of the composites during manufacturing, particularly in the case of the M60J fiber system.

2. Thermo-cycling and moisture only slightly increased the number of tow micro-cracks in comparison with the as-supplied composites.

3. There exists a very noticeable effect of residual thermal stresses on the initiation of intralaminar shear damage (transverse tow micro-cracks) of the composite.

CONCLUSIONS (Cont.)

4. The critical shear stresses for the initiation of tow cracking at room temperature in the M40J and M60J fiber Iosipescu specimens increased substantially after pre-conditioning.

5. At 316°C the effect of the residual stresses on the initiation of damage in the as-supplied and pre-conditioned composites appears to be insignificant.

6. 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.

CONCLUSIONS (Cont.)

7. The room and high temperature shear strengths of the M60J fiber system are the lowest followed by the strengths of the M40J and subsequently T650-35 fiber composite systems.

8. There is no noticeable effect of the thermal conditioning and moisture on the shear strengths of the M40J and M60J fiber composites at room temperature and at 316°C.