ANALYSES OF FAILURE MECHANISMS AND RESIDUAL STRESSES IN GRAPHITE/POLYIMIDE COMPOSITES SUBJECTED TO SHEAR DOMINATED BIAXIAL LOADS

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JOINTLY SPONSORED BY AFOSR AND NASA GLENN RESEARCH CENTER UNDER GRANT F49620-00-1-0159

This research contributes to the understanding of macro- and micro-failure mechanisms in woven fabric polyimide matrix composites based on medium and high modulus graphite fibers tested under biaxial, shear dominated stress conditions over a temperature range of -50°C to 315°C. The goal of this research is also to provide a testing methodology for determining residual stress distributions in unidirectional, cross/ply and fabric graphite/polyimide composites using the concept of embedded metallic inclusions and X-ray diffraction (XRD) measurements.

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- Objectives

This research contributes to the understanding of macro- and micro-failure mechanisms in woven fabric polyimide matrix composites based on medium and high modulus graphite fibers tested under biaxial, shear dominated stress conditions over a temperature range of -50°C to 315°C. The goal of this research is also to provide a testing methodology for determining residual stress distributions in unidirectional, cross/ply and fabric graphite/polyimide composites using the concept of embedded metallic inclusions and X-ray diffraction (XRD) measurements.

- State-of-the-art

There is no reliable test method for the evaluation of the mechanical behavior of woven graphite/polyimide composites subjected to in-plane biaxial shear dominated loads at low and elevated temperatures. In particular, the effect of residual thermal stresses on the failure process of the composites under biaxial shear dominated loads has not yet been established.

- Approach

Shear (Iosipescu) and biaxial (±45° off axis) tests are being performed on woven graphite/polyimide composites with medium and high modulus graphite fibers with PMR-15 and PMR-II-50 resins. The mechanical response of the composites is being numerically predicted. Macro- and micro-failure mechanisms in the composites are being investigated both experimentally and numerically. Particular attention is being given to the evaluation of residual thermal stresses in the composites and their influence on the failure process as a function of temperature as well as physical and chemical aging.

- Systems

There is a strong need to understand the shear strength properties of graphite/polyimide composites based on high modulus graphite fibers for space applications. Space systems will benefit from this research (according to NASA).

Background Information (Past History)

- The effect of either in-plane shear or biaxial shear dominated loads on the failure process of woven graphite/polyimide composites based on medium and high modulus graphite fibers with PMR-15 and PMR-II-50 resins at room and elevated temperatures has not been investigated in the past. In particular, no comparative studies of the ±45° off axis and Iosipescu tests as applied to woven composites have been performed. In this project, both of the tests were numerically evaluated by performing fully non-linear finite element computations (for the first time). Subsequently, the shear strengths of an 8HS graphite/PMR-15 composite were determined at room and 315°C temperatures.
- Predecki and Barrett showed in the 70's that it was possible to use X-ray diffraction methods to measure the state of strain in various metallic inclusions embedded in the polymer matrix of a graphite/epoxy composite system. However, they could not extract the actual residual stresses in the composite from the XRD measurements. For the first time, this was accomplished in this project by performing numerous XRD tests on unidirectional and woven graphite/PMR-15 composites with either embedded aluminum or silver particles. The experimental data were numerically verified by performing visco-elastic computations using the Eshelby method for multiple inclusions and plate theory.

MOST RECENT PUBLICATIONS

1. G. Odegard and M. Kumosa, Elastic-Plastic and Failure Properties of a Unidirectional Carbon/PMR-15 Composite at Room and Elevated Temperatures, *Composites Science and Technology*, Vol. 60 (2000) pp. 2979-2988.

2. G. Odegard, D. Armentrout, K. Searles, L. Kumosa, J. K. Sutter and M. Kumosa, Failure Analysis of ±45° Off-Axis Woven fabric Composite Specimens, *Journal of Composites Technology & Research*, Vol. 23, No. 3, (2001) pp. 205-224.

3. M. Kumosa, P.K. Predecki, G. Odegard, K. Searles, B. Benedikt, D. Armentrout, L. Kumosa, M. Gentz and J.K. Sutter, Analysis of Failure Mechanisms and Residual Stresses in Unidirectional and Woven Graphite/PMR-15 Composites Subjected to Shear Dominated Biaxial Loads, HIGH TEMPLE Workshop XXI, 12-15 February 2001, Clearwater Beach, Florida.

4. B. Benedikt, M. Kumosa, P.K. Predecki, L. Kumosa, M.G. Castelli and J.K. Sutter, An Analysis of Residual Thermal Stresses in a Unidirectional Graphite/PMR-15 Composite Based on the X-ray Diffraction Measurements, *Composites Science and Technology*, Vol. 61, No. 14 (2001) pp. 1977-1994.

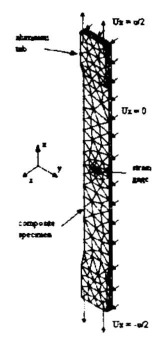
5. M. Kumosa, G. Odegard, D. Armentrout, L. Kumosa and J. K. Sutter, Comparison of the ±45° Off Axis and Iosipescu Shear Tests for Woven Fabric Composite Materials, in *Journal of Composites Technology & Research*, Vol. 24 (2001) pp. 3-16.

6. B. Benedikt, P. Predecki, L. Kumosa, D. Armentrout, J. K. Sutter and M. Kumosa, The Use of X-Ray Diffraction Measurements to Determine the Effect of Bending Loads on Internal Stresses in Aluminum Inclusions Embedded in a Unidirectional Graphite/PMR-15 Composite, *Composites Science and Technology*, Vol. 61, No. 14 (2001) pp. 1995-2006.

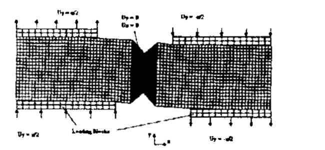
7. B. Benedikt, P. Rupnowski, L. Kumosa, J. K. Sutter, P.K. Predecki and M. Kumosa, Determination of Interlaminar Residual Thermal Stresses in a Woven 8HS Graphite/PMR-15 Composite Using X-Ray Diffraction Measurements, *Mechanics of Composite Materials and Structures*, submitted.

8. M. Gentz, D. Armentrout, P. Rupnowski, L. Kumosa, J.K. Sutter and M. Kumosa, Mechanical Behavior of a Woven Graphite/PMR-15 Composite at Room and Elevated Temperatures Determined from the ±45° Off-Axis and Iosipescu Tests, *Composites Technology & Research*, submitted.

±45° and Iosipescu Tests (FEM)



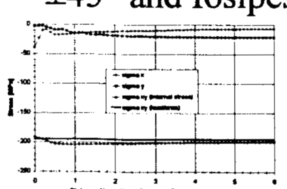
Finite element mesh of the ±45° specimen



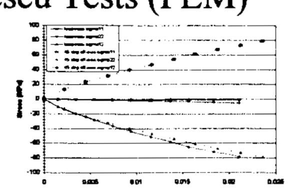
Finite element mesh of the Iosipescu specimen.

The application of the Iosipescu and ±45° off axis tests for the shear testing of woven graphite/polyimide composites at room and elevated temperatures has been evaluated both experimentally and numerically.

±45° and Iosipescu Tests (FEM)



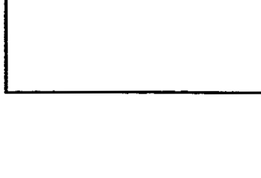
Normal and shear stress components along the notch root axis of the Iosipescu specimen



Normal and shear stress components versus shear strain at the centers of the Iosipescu and ±45° off axis specimens

The finite element computations of the ±45° off-axis and Iosipescu tests were fully non-linear with the material, geometrical, and boundary contact non-linearities considered. It was shown that the stress distributions in the two specimens were entirely different. In the gage sections of the woven Iosipescu specimens, the stress fields are esentially pure shear whereas the ±45° test generated biaxial tension/tension and shear stress fields.

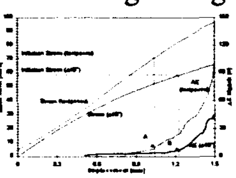
±45° and Iosipescu Tests (Experimental)



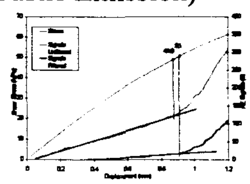
Shear stress vs. displacement curves from the Iosipescu tests performed at room and 315°C temperatures.

Shear stress vs. displacement curves from the ±45° tests performed on 25.4 mm wide specimens at 315°C and at room temperature.

±45° and Iosipescu Tests (Initiation of Damage using Acoustic Emission)



Load and AE vs. displacement curves for the 8HS graphite/PMR-15 system at RT from the Iosipescu and ±45° off axis tests

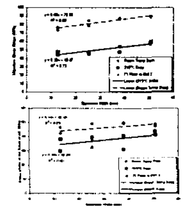


Shear stress vs. displacement and the filtered and unfiltered AE signals vs. displacement for a 12.7 mm wide ±45° tensile test.

The initiation of interlaminar damage was monitored by acoustic emission both at room and high temperatures. Unique localization techniques were employed to avoid signals generated outside the gage sections of the specimens in the tests performed at RT. At high temperatures the localization techniques could not be used.

Comparison between the Iosipescu and ±45° tests

It has been shown in this research that the critical loads for the initiation of intralaminar damage in an 8HS woven graphite/PMR-15 composite tested at room temperature and at 315°C are significantly lower from the ±45° off axis (biaxial) tests than from the Iosipescu (shear) tests. The shear strengths of the composites at room and elevated temperatures, determined from the maximum loads, are also lower in the ±45° off axis tests in comparison with the Iosipescu test data. There is a strong specimen width effect in the ±45° off axis graphite/PMR-15 specimens both at room and at 315°C. It appears that the weakest failure mode is in-plane biaxial failure under tensile stresses along the warp and fill tows in addition to shear.

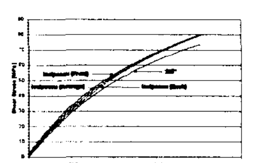


Specimen width effect in the ±45° off-axis 8HS graphite/PMR-15 specimens at room and 315°C temperatures.

	Shear Stresses at the Onset of Intralaminar Damage [MPa]		Shear Stresses at Maximum Loads [MPa]	
Test	±45°	Iosipescu	±45°	Iosipescu
at RT	56.6 ± 2.0	94.8 ± 1.3	82.0 ± 0.15	105.8 ± 2.6
at 315°C	37.3 ± 5.2	59.9 ± 1.2	50.8 ± 6.0	71.8 ± 4.2

Shear stresses at the onset of intralaminar damage (determined by AE) and at the maximum loads from the ±45° off-axis and Iosipescu tests at room and 315°C temperatures (8HS graphite/PMR-15).

Strain Measurements



Shear stress vs. shear strain diagrams from the ±45° and Iosipescu tests at room temperature

Temperature	Experimental [GPa]	Numerical [GPa]
RT	5.54	5.24*
315°C	0.61 and 0.34	3.35*

* after 30 sec which corresponds to 6 MPa at 1 mm/min

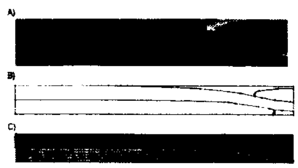
Experimental and numerical shear moduli at room and 315°C temperatures from the ±45° tests.

Strain Gage "Effect"

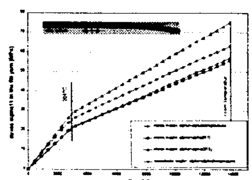
Plate #	Specimen #	# of Gages	Specimen Width (mm)	Thickness (mm)	Onset of AE (MPa)	Max. Stress (MPa)
B	4	0	25.43	5.41	39.9	45.8
A	7	0	25.40	5.38	33.0	46.9
A	6	1	25.45	5.42	23.3	36.3
A	5	1	25.65	5.42	21.2	37.4
(*)	7	2	25.44	5.40	17.1	31.3
A	4	2	25.45	5.46	11.0	26.7
B	1	2	25.37	5.32	13.3	18.7

Shear stresses at the significant onset of AE and at the maximum loads from the ±45° tests performed at 315°C with and without strain gages.

Visco-elastic Calculations of Residual Stresses

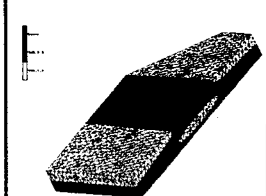


To understand the effect of biaxial shear dominated loads on the failure process, two- and three dimensional visco-elastic finite element computations of residual thermal stresses in the 8HS graphite/PMR-15 system have been initiated. Particular attention is being given to the determination of the effect of residual thermal stresses and biaxial loads on the initiation of transverse cracks in the warp and fill tows of the composite.



Residual transverse stresses in the fill tows

Three dimensional model of 8HS unit cell



To understand the effect of biaxial and shear dominated loads, a 3D model of the unit cell was built. The magnitude of residual meso-stresses in the warp and fill tows and the stresses caused by purely mechanical shear and biaxial loads were numerically determined.

External Loads	σ_{11} [MPa]	σ_{22} [MPa]	σ_{33} [MPa]	σ_{12} [MPa]
Residual Only	62	-119	18	-2
Residual and Iosipescu	64	-97	19	111
Residual and ±45°	74	49	18	66

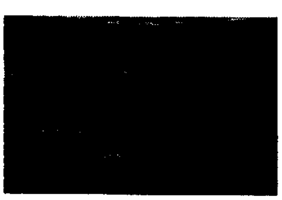
FEM model parameters:

- simulation type: thermo-visco-elastic cooling
- periodic boundary conditions
- 5427 nodes, 26248 elements
- element type: tetra4

Employing the Tsai-Hill failure criterion, the transverse and shear strengths of the tows with a high volume fraction of fibers (72%) were estimated.

Indirect Estimation of Tow Strength	
Transverse [MPa]	Shear [MPa]
60	174

Examples of Tow Cracking During Manufacturing



M40J Fibers with PMR-II-50 Resin
EES-29

M60J Fibers with PMR-II-50 Resin
EES-13

Composite systems investigated so far:

1. 8HS T650/PMR-15 (no tow cracks after manufacturing, tow cracks observed only after mechanical testing at RT and 315C)
2. 4HS M40J/PMR-II-50 (few cracks observed after manufacturing)
3. 4HS M60J/PMR-II-50 (multiple cracks observed after manufacturing)

Effects of Stiffness Properties and CTEs of Fibers on Residual Meso-Stresses

Residual thermal meso- and micro-stresses depend significantly on the stiffness properties of graphite fibers and their CTEs.

fiber properties			transverse
alpha1 [10^-6/K]	E11 [GPa]	E22 [GPa]	residual stress [MPa]
-0.5	241	20	62.1
-0.5	241	40	72.2
0.83	377	20	62.5
-1.1	588	20	69.9
-0.5	588	20	68.3
-0.5	377	20	65.8
-0.5	482	40	79.2

Residual meso-stresses as a function of fiber stiffness and CTE

X-ray diffraction tests

- **Major advances have also been made recently in this project in the development of an experimental/numerical technique for the determination of interlaminar residual stresses in woven graphite/polymer composites based on X-ray diffraction measurements of residual strains in embedded metallic inclusions. The residual thermal stresses between unidirectional and woven 8HS graphite/PMR-15 plies have been determined both experimentally and numerically as a function of the manufacturing process.**

Aluminum inclusion

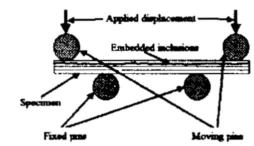
Silver inclusion

Embedded Al inclusions between woven 8HS plies in the graphite/PMR-15 system.

Aluminum (spherical), silver (irregular) and niobium (irregular) inclusions were used. The best results were obtained using the aluminum inclusions both in the case of the unidirectional and woven systems.

X-ray diffraction tests

Numerous XRD measurements were made to determine residual strains and stresses in embedded Al and Ag inclusions placed in four ply 8HS woven and 6 ply unidirectional graphite/PMR-15 composites. In the modeling part of this research, the residual thermal stresses in the composites were evaluated by performing linear and visco-elastic computations. The magnitudes of the stresses in the composites as a function of temperature, time and composite architecture have been calculated. The numerically determined residual stresses in the composites were subsequently compared to the residual stresses determined from the X-ray analysis in conjunction with the application of the linear elastic and visco-elastic Eshelby models for multiple ellipsoidal inclusions. The effect of external bending loads on the residual strains and stresses in the embedded particles was also experimentally and numerically investigated.



X-ray four point bend tests

	X-ray with Eshelby			Plate theory		
	σ11 [MPa]	σ 22 [MPa]	σ 33 [MPa]	σ 11 [MPa]	σ 22 [MPa]	σ 33 [MPa]
Linear elastic	70.7 ± 17	71.1 ± 17	36.7 ± 16	94.0	94.0	0
Visco-elastic	67.3 ± 17	67.6 ± 16	33.0 ± 16	63.1	63.1	0

Interlaminar residual stresses in the 8HS graphite/PMR-15 composite from XRD measurements and the Eshelby model for multiple inclusions as well as from laminated plate theory with linear-elastic and visco-elastic assumptions.

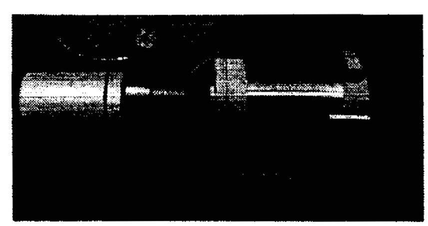
Residual stresses from the X-ray diffraction tests

Comparison between the interlaminar residual stresses in the unidirectional and woven (8HS) graphite/PMR-15 composites.

	Excluding bending			Including bending			
	σ_{11} [MPa]	σ_{22} [MPa]	σ_{33} [MPa]	σ_{11} [MPa]	σ_{22} [MPa]	σ_{33} [MPa]	Volume fraction of Al inclusions
Unidirectional	53.5± 9	39.6 ± 8	25 ± 5	55 ± 6	40.5 ± 5	26.6± 4	40 ±7%
8HS woven	67.3±17	67.6 ± 16	33 ± 16	62.3± 8	61.7 ± 8	27.6± 7	46 ±7.4%

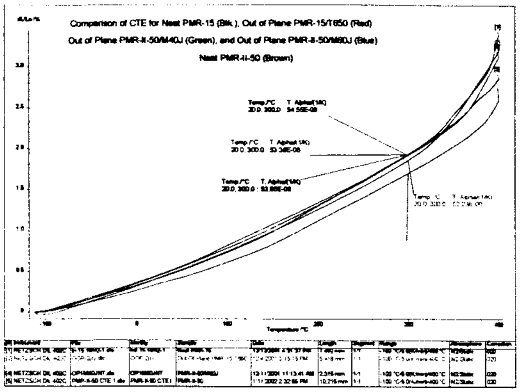
The magnitudes of the interlaminar residual stresses along the fibers (σ_{11}, unidirectional) and along the tows ($\sigma_{11} = \sigma_{22}$, 8HS) in both the graphite/PMR-15 systems are high. Especially, the large tensile stresses along the tows (62.3 ± 8 MPa) might create cracking of the polyimide layers in service since they are only slightly lower than the tensile strength of the PMR-15 resin, which is approximately 80 MPa. They might be even higher in graphite/polyimide composite structures subjected to large temperature variations in-service.

New Dilatometer

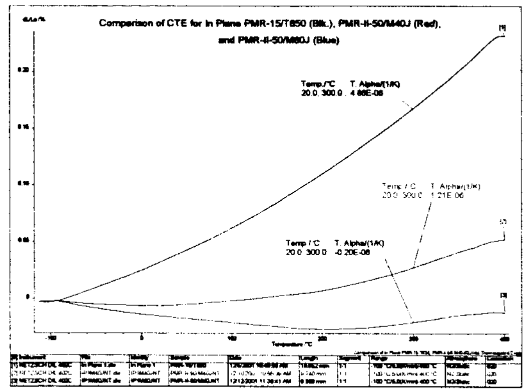


Netzsch Dil 402 C
Temperature Range –180C to 500C
Resolution 1.25nm/digit
Displacement Range 5000μm
Thermostatic Controlled Transducer
Atmospheric Control

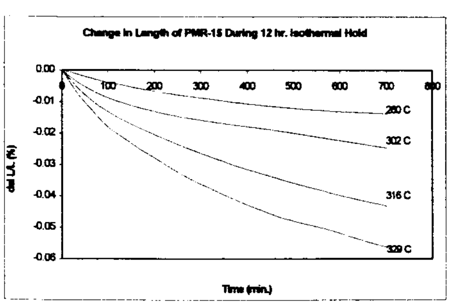
Through-thickness CTEs for 8HS T650/PMR-15, 4HS M40J/PMR-II-50, 4HS M60J/PMR-II-50 and neat PMR-15 and PMR-II-50



In-Plane CTEs for 8HS T650/PMR-15, 4HS M40J/PMR-II-50, 4HS M60J/PMR-II-50 and neat PMR-15 and PMR-II-50



Short Term Aging Effects in PMR-15



Major Conclusions/Biaxial Testing

- Three-dimensional fully non-linear finite element computations have shown that the ±45° off-axis test does not generate a pure shear stress field in the specimen gage section. Instead, a biaxial stress field is present consisting of tension along the warp and fill tows in addition to shear. In the gage section of the woven Iosipescu specimens the stress field is almost pure shear .
- The mechanical data clearly showed that the ±45° off-axis tests are significantly affected by the specimen width effect not only at room temperature but also at elevated temperatures.
- The onset of intralaminar damage and the shear strength of the composite determined at the maximum loads are very strongly dependent on the type of test. The biaxial tension/shear stress fields in the gage sections of the ±45° off-axis 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. It appears that the lowest mode of failure of the composite at room temperature and at 315°C is the combined effect of tension along the warp and fill tows in addition to shear.

Major Conclusions/Residual Stress Analysis

- It has been shown in this research that the thermal residual interlaminar stresses can be determined in unidirectional and woven 8HS graphite/PMR 15 composites using the methodology based on the XRD measurements of residual strains in embedded metallic inclusions. The stresses are three dimensional in nature with significant stresses present between the plies in the thickness direction of the specimen.
- There is a good agreement between the thermal residual stresses in the directions of the tows determined from the XRD measurements in conjunction with application of the visco-elastic Eshelby model for multiple inclusions, with the stresses from the visco-elastic plate theory. Only the stresses through the thickness are entirely different.
- The scatter in the XRD measurements is predominately caused by the non-uniform distribution of the particles and the non-uniform distribution of residual stresses in the composites.
- The accuracy of the entire analysis of the residual stresses is very strongly dependent on the physical properties of the investigated composite system. The temperature and time dependent physical properties of the composites and inclusions must be known for the accurate determination of the residual stresses either from laminate theory or from the XRD measurements of residual strains in the inclusions in conjunction with the Eshelby model.
- There is a strong effect of inclusion shape on the measured X-ray strains and stresses. In particular, the irregular silver inclusions significantly overestimated the residual stresses in the composites.

Current and Future Research Activities

- The newly developed combined numerical and experimental methodology for the determination of the shear properties of woven graphite/polyimide composites (verified so far only on graphite/PMR-15 systems) is being used for other types of woven composites such as 4HS graphite (M40J and M60J)/PMR-II-50 systems. The Iosipescu and ±45° off axis tests are being performed at room and elevated temperatures supported by finite element computations.
- Interlaminar residual stresses in the unidirectional and woven graphite/PMR-15 systems are being measured by XRD as a function of physical and chemical aging. New types of inclusions are also being evaluated (no oxidation, better inclusion distributions).
- Residual thermal stresses in the 4HS M40J and M60J fibers with PMR-II-50 will be measured and calculated. The effect of the stresses on the failure process in the composites subjected to in-plane shear and biaxial loads as a function of temperature as well as physical and chemical aging will be determined.