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

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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 composite 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, crossply 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 (isotensor) 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-H1-50 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-H1-50 resins at room and elevated temperatures has not been investigated in the past. In particular, no comparative studies of the +45° off axis and isotensor 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 RIS 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.
Normal and shear stress components along the notch root axis of the Iosipescu specimen

Normal and shear stress component 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 gauge sections of the woven Iosipescu specimen, the stress fields are essentially pure shear whereas the ±45° test generated bi axial tension/torsion and shear stress fields.

Most Recent Publications


±45° and Iosipescu Tests (FEM)

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 (Experimental)

Shear stress vs. displacement curves from the Iosipescu tests performed at room and 355°C temperatures.
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 interlaminar damage in an ESS woven graphite/PHS-15 composite tested at room temperature and at 315°C are significantly lower from the ±45° off axis (isotropic) tests than from the Iosipescu (shear) tests. The shear strengths of the composite 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/PHS-15 specimens both at room and at 315°C. It appears that the weakest failure mode is in-plane bi-axisial failure under nominal stresses along the warp and fill axes in addition to shear.

**Strain Gage “Effect”**

<table>
<thead>
<tr>
<th>Plate</th>
<th>Specimen</th>
<th># of Gages</th>
<th>Specimen</th>
<th>Thickness (mm)</th>
<th>Onset of AE (MPa)</th>
<th>Max Stress (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>B</td>
<td>4</td>
<td>0</td>
<td>25.43</td>
<td>5.41</td>
<td>39.9</td>
<td>45.9</td>
</tr>
<tr>
<td>A</td>
<td>7</td>
<td>0</td>
<td>25.40</td>
<td>5.38</td>
<td>33.0</td>
<td>46.9</td>
</tr>
<tr>
<td>A</td>
<td>6</td>
<td>1</td>
<td>25.45</td>
<td>5.42</td>
<td>23.3</td>
<td>36.3</td>
</tr>
<tr>
<td>A</td>
<td>5</td>
<td>1</td>
<td>25.65</td>
<td>5.42</td>
<td>21.2</td>
<td>37.4</td>
</tr>
<tr>
<td>(*)</td>
<td>7</td>
<td>2</td>
<td>25.44</td>
<td>5.40</td>
<td>17.1</td>
<td>31.3</td>
</tr>
<tr>
<td>A</td>
<td>4</td>
<td>2</td>
<td>25.45</td>
<td>5.46</td>
<td>13.0</td>
<td>36.7</td>
</tr>
<tr>
<td>B</td>
<td>1</td>
<td>2</td>
<td>25.57</td>
<td>5.32</td>
<td>13.3</td>
<td>18.7</td>
</tr>
</tbody>
</table>

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 bi-axial shear dominated loads on the failure process, a three dimensional visco-elastic finite element computations of residual thermal stresses in the 8HS graphite/PMR-11-50 system were initiated. Particular attention is being given to the determination of the effect of residual thermal stresses and bi-axial loads on the initiation of transverse cracks in the warp and fill tows of the composite.

Three dimensional model of 8HS unit cell

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

FEM model parameters:
- simulation type: thermo-visco-elastic cooling
- periodic boundary conditions
- 5427 nodes, 26248 elements
- element type: tetrah

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 115°C)
2. 8HS M40J/PMR-II-50 (few cracks observed after manufacturing)
3. 8HS 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.

<table>
<thead>
<tr>
<th>fiber properties</th>
<th>E11</th>
<th>E22</th>
<th>transverse residual stress</th>
</tr>
</thead>
<tbody>
<tr>
<td>alpha1</td>
<td>10^6 [GPa]</td>
<td>[GPa]</td>
<td>[MPa]</td>
</tr>
<tr>
<td>-0.5</td>
<td>241</td>
<td>20</td>
<td>62.1</td>
</tr>
<tr>
<td>-0.5</td>
<td>241</td>
<td>40</td>
<td>72.2</td>
</tr>
<tr>
<td>-0.5</td>
<td>377</td>
<td>20</td>
<td>62.5</td>
</tr>
<tr>
<td>-0.5</td>
<td>588</td>
<td>20</td>
<td>68.3</td>
</tr>
<tr>
<td>-0.5</td>
<td>377</td>
<td>40</td>
<td>65.8</td>
</tr>
<tr>
<td>-0.5</td>
<td>482</td>
<td>40</td>
<td>79.2</td>
</tr>
</tbody>
</table>

Residual meso-stresses as a function of fiber stiffness and CTE
Residual stresses from the X-ray diffraction tests

<table>
<thead>
<tr>
<th>Conditions</th>
<th>Excluding Inclusion</th>
<th>Including Inclusion</th>
<th>Maximum CTE °C</th>
</tr>
</thead>
<tbody>
<tr>
<td>Unidirectional</td>
<td>49.0 ± 0.0</td>
<td>48.0 ± 0.0</td>
<td>49.7 ± 0.0</td>
</tr>
<tr>
<td>Bidirectional</td>
<td>47.0 ± 0.0</td>
<td>46.0 ± 0.0</td>
<td>47.5 ± 0.0</td>
</tr>
</tbody>
</table>

The magnitudes of the interlaminary residual stresses along the fiber (unidirectional) and along the tow (b = c = 0°, 3°) in both the graphite/PMR-15 systems are high. Especially, the large tensile stresses along the tow (6.2 ± 8 MPa) might cause cracking of the polyimide layers in service since they are only slightly lower than the ultimate 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.

Netzsch Dil402 C
Temperature Range –180°C to 500°C
Resolution 1.25mm/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

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 losipescu 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 specimen resulted in much lower estimates of the critical loads for the initiation of intralaminar damage and the composite strength compared with the losipescu 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 4HS 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 axes 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 isotropic 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.