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POLYIMIDE COMPOSITES*

MACIEJ S. KUMOSA^{**}, Kevin H. Searles^{***} and Greg Odegard^{**}

**Center for Advanced Materials and Structures Department of Engineering University of Denver Denver, Colorado

*** Department of Materials Science and Engineering Oregon Graduate Institute of Science and Technology Portland, Oregon

Introduction

High temperature polymers and polymer matrix composites (PMC's) are finding increasing use in the aerospace and electronics industries. Graphite fiber reinforced polyimides are advanced thermosetting polymer composites which have become highly relevant in the realm of high temperature applications (ref. 1). These composites possess exceptional specific properties and are stable to temperatures as high as 360 °C (680 °F). For successful application of graphite/polyimide composites, it is essential to have a reliable database of material properties and a detailed understanding of fracture and failure behavior, especially at elevated temperatures. A major limitation of many fiber/polymer composite systems is the inability of these materials to resist intralaminar and interlaminar damage initiation and propagation under biaxial, shear dominated, monotonic and cyclic loading conditions. The purpose of this research is to investigate failure properties of fabric and unidirectional graphite/polyimide composites tested over the temperature range -50 °C to 315.6 °C (-58 °F to 600 °F) under biaxial, shear dominated loading conditions.

The Josipescu shear test, which was originally designed for determining shear properties of metals (ref. 2), was first applied to composite materials by Walrath and Adams (ref. 3). The traditional losipescu shear test essentially consists of a double edge-notched beam specimen, to which two counteracting force couples are applied such that the net bending moment at the specimen midlength is zero, and a relatively uniform shear stress field exists in the central gage section of the specimen. Based upon the traditional Iosipescu shear test, an in-plane biaxial Iosipescu test fixture has been designed and developed at the University of Cambridge (ref. 4). The fixture (Fig. 2) is capable of testing Iosipescu specimens in either pure shear or a combination of shear and transverse tension/compression under static or cyclic loads. Shear tests can be performed under externally applied compressive loads (P_0) normal to the longitudinal axis of the specimen as in the traditional test (refs. 1 and 2). For in-plane, biaxial tests, the specimen is rotated clockwise (c.w.) or counter-clockwise (c.c.w.) such that the compressive load (P_{α}) is applied at various angles (α) to the normal (Fig. 3).

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Recent studies have employed the fixture for obtaining biaxial failure and fracture properties of unidirectional GRP and CFRP composites, teak wood, Ti/SiC composites and adhesive joints (refs. 4-8). For this study, a series of shear and biaxial losipescu tests were performed at room and elevated temperatures on the following three composite materials:

- 1. Graphite/PMR-15 (fabricated at the NASA Lewis Research Center); Fiber: T650-35, 8-H Satin Cloth; Ply Layup: 16-ply (warp-aligned); Matrix: PMR-15.
- 2. Graphite/Avamid-R (fabricated by DuPont); Fiber: T650-35, 8-H Satin Cloth; Ply Layup: 10-ply (warp-aligned and 0/90); Matrix: Avamid-R.
- 3. Graphite/PMR-15 (fabricated at the NASA Lewis Research Center); Fiber: T650-35, unidirectional; Ply Layup: 34-ply; Matrix: PMR-15.

Composites 1 and 3 were fabricated using a simulated autoclave according to G.E.'s specifications. The panels were post-cured according to G.E.'s schedule. In this paper, only the results from the biaxial testing of the fabric composites are presented.

The load-displacement diagrams for the graphite/PMR-15 Iosipescu specimens tested in the biaxial fixture at room temperature are presented in Fig. 4. It can be observed that the shape of the load-displacement curves depends on the loading angle. Similar tests were performed on the graphite/Avamid-R composites (warp-aligned and 0/90). The loads at failure as a function of the loading angle (α) for the investigated composites are shown in Fig. 5. At room temperature, the biaxial strength properties of these three composite systems appear to be different. Moreover, different failure modes were identified and it was also established that the shear and biaxial strength properties of the graphite/PMR-15 system may be significantly influenced by the manufacturing process.

The micro-failure process in the PMR-15 system tested at room temperature starts from the initiation of interlaminar cracks between the layers (Fig. 6a). This usually leads to the formation of large delaminations within the gage section of the losipescu specimens. Often, one of the interlaminar cracks propagates catastrophically along the sample causing a significant drop on the load-displacement curves. Such load drops can be observed in Fig. 4. This effect, however, seems to be dependent on the manufacturing process. If the strength of the interfaces between the layers is high, the load drops do not occur. The interlaminar damage in the PMR-15 system creates large out-of-plane deformations on the specimen surface i.e., bulging (see Fig. 6b). The micro-failure process which determines the failure of the Avamid-R system is the formation of interlaminar cracks between the layers. Since the interlaminar strength properties of the Avamid-R system appear to be higher compared to the PMR-15 system, the bulging effect on the composite surface is substantially less pronounced.

The micro-damage generated in the composite Iosipescu specimens tested under biaxial loading conditions can be evaluated by capturing and performing qualitative analyses of scanning electron microscope (SEM) images from planar specimen slices. Subsequently, the slices are reassembled into 3-D space and the net volumetric effect of damage can be determined. In Fig. 7, a 3-D projection of damage within the gage section of the PMR-15 Iosipescu specimen tested in shear is presented.

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The biaxial tests at elevated temperatures were performed using a high temperature setup assembled at the University of Denver (Fig. 8). Since the high temperature, biaxial losipescu research is still in progress, only preliminary results from the high temperature investigation are presented and discussed in this paper. As an example, the load-displacement diagrams for the graphite/PMR-15 and graphite/Avamid-R (0/90) composites tested in shear at various temperatures are shown in Figs. 9 and 10, respectively. The effect of elevated temperature on the load-displacement curves for the graphite/Avamid-R composite is significantly stronger in comparison with the graphite/PMR-15 system. From the maximum loads determined using the curves in Figs. 9 and 10, the shear strength properties were subsequently estimated. The shear strengths of the composites as a function of temperature are presented in Fig. 11. At room temperature, it appears that the shear strength of the graphite/Avamid-R system is slightly higher than that of the graphite/PMR-15 system. However, at elevated temperatures above 232 °C (450 °F), the graphite/Avamid-R composite exhibits a rapid decrease in its shear strength, whereas the shear strength of the PMR-15 system gradually decreases with an increase in temperature.

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Objectives

- Determine failure and fracture properties of unidirectional and fabric graphite reinforced polyimide composites based on PMR-15 and Avamid-R resins tested under biaxial, shear dominated loading conditions over the temperature range -50 °C to 315.6 °C (-58 °F to 600 °F)
- Characterize micro-damage initiation and development in the composites as a function of:
 - Testing temperature
 - Biaxial, shear dominated in-plane stress conditions
 - Type of polyimide resin
 - Manufacturing process
- Develop a finite element model of biaxial, graphite/polyimide losipescu specimens for elevated temperature applications



Loading Diagram for Inducing an In-Plane Biaxial Stress State











Damage at the Notch Root in Graphite/PMR-15 losipescu Specimens: a) Inter- and Intralaminar Cracks



Fig. 6a

Damage at the Notch Root in Graphite/PMR-15 losipescu Specimens: b) Out-of-Plane Damage (Bulging)



Fig. 6b

3-D Projection of Damage in a Graphite/PMR-15 losipescu Specimen Tested in Shear at Room Temperature







High Temperature Experimental Setup



Load-Displacement Diagrams for Graphite/PMR-15 losipescu Specimens Tested at Elevated





Shear Strength as a Function of Temperature



Fig. 11

Summary of Results

- Biaxial strength properties and failure modes for graphite/PMR-15 and graphite/Avamid-R are different at room temperature:
 - It appears that the shear strength properties of the Avamid-R system are better than the PMR-15 system
 - Under biaxial shear/compression loading conditions, the PMR-15 composite exhibits significantly higher strength in comparison with the Avamid-R system
- The effect of elevated temperatures on the load-displacement curves in shear for the graphite/Avamid-R composite is significantly more prevalent than for the graphite/PMR-15 composite
- The shear strengths of the Avamid-R system at elevated temperatures are significantly lower than the high temperature shear strengths of the graphite/PMR-15 system

Fig. 12

Conclusion

- Application of the biaxial losipescu test fixture can be successfully extended to include graphite reinforced polyimide fabric and unidirectional composites when determining:
 - shear strength properties at room and elevated temperatures
 - shear dominated, biaxial failure mechanisms

Future Work

- Complete biaxial losipescu testing on fabric and unidirectional graphite polyimide composites over a range of temperatures
- Complete development of image analysis techniques for reproduction of damage from planar specimen slices
- Develop acoustic emission and resonant frequency methodologies for monitoring and identifying damage progression in the composites at high temperatures
- Develop numerical schemes for modeling damage in composite biaxial losipescu specimens at high temperatures