INTERFACIAL CHARACTERIZATION OF GLASS AND GLASS-CERAMIC MATRIX/NICALON SiC FIBER COMPOSITES

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ABSTRACT

The strong and tough composite systems consisting of Nicalon SiC yarn reinforced lithium aluminosilicate (LAS) glass-ceramics, with and without Nb₂O₅ additive as a means to form a NbC reaction layer around the SiC fibers during fabrication, have been characterized through the use of TEM replica and thin foil analysis and scanning Auger microprobe (SAM) analysis of composite fracture surfaces. From these studies, it has been found that the chemistry of the fibers within a few hundred angstroms of the fiber/matrix interface has changed significantly from as-received fibers in that a carbon rich zone of 100-400Å in thickness has formed at the fiber surface and that aluminum has diffused into the fibers from the glass-ceramic matrix.

From SAM analysis of Nicalon fiber surfaces from extremely weak and brittle glass and glass-ceramic matrix composites, this carbon rich layer is either nonexistent or much reduced in carbon content. It appears, therefore, that the formation of this carbon rich interfacial zone in the LAS matrix composites under study leads to quite weak bonding at the fiber/matrix interface that directly contributes to the high toughness observed for these materials.

INTRODUCTION

The ceramic composite systems based on the reinforcement of glass and glass-ceramic matrices with Nicalon* SiC fibers have been under study at United Technologies Research Center (UTRC) for the past several years.¹⁻³ The basic goal of this work was to develop a high strength, high toughness, low density ceramic matrix composite that exhibited a use temperature of at least 1000°C. This goal was essentially met with the development of the lithium aluminosilicate (LAS) glass-ceramic matrix/Nicalon fiber composite system.³ However, during the course of this investigation it was found that certain fabrication conditions and/or matrix compositions resulted in quite weak and brittle composites with fracture characteristics quite different than that usually observed for this class of composite. For example, Fig. 1 shows the

*Nippon Carbon Co. Ltd., Tokyo, Japan

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fracture surfaces of two different LAS matrix/Nicalon SiC fiber composites; one is a very weak [31 ksi (214 MPa)] and brittle material while the other is strong [119 ksi (820 MPa)] with a very tough and fibrous mode of fracture. In order to gain an understanding of the factors controlling the type of fracture behavior observed in these composite systems, a comprehensive scanning transmission electron microscope (STEM) analysis of replicas of polished composite cross-sections as well as ion beam thinned composite sections and scanning Auger microprobe (SAM) analysis of composite fracture surfaces was undertaken. These analytical methods allowed the phase assemblages and microchemical composition of the fiber/matrix interfacial regions to be determined to a fine degree of spatial resolution. The results of this study are discussed in the following sections of this paper.

MATERIALS

The SiC fiber used throughout this study is that produced by Nippon Carbon Co. in Japan and currently distributed in this country by Dow Corning Corp., Midland, Michigan. The fibers are obtained on spools of continuous length (~500 m) tows of 500 fibers/tow with an average fiber diameter of ~15 μm. The average tensile strength and elastic modulus of this fiber, as measured at UTRC, is 2400 MPa (350 ksi) and 193 GPa (28 x 10^6 psi), respectively.

The LAS matrix materials used in this study are designated LAS-I, LAS-II, and LAS-III. LAS-I is the matrix very similar in composition to Corning 9608, except that ~3 wt% ZrO₂ replaces the ~3 wt% TiO₂ used as a nucleating agent in 9608. LAS-II is identical to LAS-I except that it contains an addition of ~5 wt% Nb₂O₅ for NbC reaction barrier formation. LAS-III also contains 5 wt% Nb₂O₅ and, in addition, is formulated to be much more refractory than either LAS-I or LAS-II. The glass matrix material evaluated in composite form is Corning 1723 aluminosilicate. All of the matrix materials were received from Corning Glass Works as glassy powder of 8-12 μm average particle size.

COMPOSITE FABRICATION

The glass and glass-ceramic matrix composites were fabricated by passing the SiC yarn through an agitated slurry of glass powder, water, and an acrylic binder, onto a rotating drum. The resultant tape was then cut into the