

Ambient-Temperature Mechanical Response of Alumina-Fluoromica Laminates

Todd T. King* and Reid F. Cooper*

Department of Materials Science and Engineering, University of Wisconsin, Madison, Wisconsin 53706

Flexural delamination experiments were used to evaluate the mechanical performance of thermochemically stable alumina-fluoromica laminates. Hot-pressed, precracked laminate specimens, in which two MgAl_2O_4 -spinel-coated alumina substrates were separated by a thin layer of fluorophlogopite ($\text{KMg}_3(\text{AlSi}_3)\text{O}_{10}\text{F}_2$), were tested in four-point flexure at room temperature. Two types of mechanical response were observed: steady-state delamination and brittle failure. Microstructural analysis showed that the delamination response was associated with fine ($\leq 5 \mu\text{m}$) grains of the mica; the brittle response occurred when the mica interphase consisted of large ($>30 \mu\text{m}$) grains that bridged the interphase. The steady-state strain-energy release rate (G_{ss}) measured on the graceful, delaminating beams was $9.1 \pm 0.4 \text{ J}\cdot\text{m}^{-2}$ for randomly oriented $\sim 5\text{-}\mu\text{m}$ grains but only $2.8 \pm 0.2 \text{ J}\cdot\text{m}^{-2}$ for $\sim 1\text{-}\mu\text{m}$ grains that were aligned with easy-cleavage planes parallel to the laminate interfaces. The results suggested that debonding of the specimens occurred via cleavage of the mica grains. Observation of delamination cracks confirmed this point: propagation occurred within the fluoromica interphase rather than along the spinel/alumina or spinel/fluorophlogopite interfaces. The mechanical feasibility of laminate specimens without the protective spinel coating on the substrate containing the notch was also tested to address an issue related to the preparation of alumina fiber/mica interphase/alumina matrix composites. The delamination response again occurred for the case of a fine-grained mica interphase.

I. Introduction

FIBER-REINFORCED ceramic composite systems must fulfill three general criteria to be applicable in high-temperature, oxygen-rich environments (e.g., as experienced in engine applications). First, the ceramic composite system must display the appropriate response to high-rate deformation, specifically fiber debonding and frictional sliding along the fiber/matrix interface. Second, planar interfaces must be formed (during composite fabrication) and maintained (during application) between each of the composite component phases, particularly to avoid the introduction of Griffith flaws on the fiber surface; this criterion thus indicates that appropriate thermokinetic compatibility must exist between the fiber, matrix, and any interphase materials. Third, the fiber, matrix, and interphase materials must exhibit either stability against, or kinetic resistance to, high-temperature oxidation. It is this final criterion that

is the primary limitation in a variety of ceramic composite systems, such as those using carbide and nitride phases.

Our efforts have concentrated on using oxides and silicates as an approach to the fulfillment of these criteria. Using the structural diversity of silicates and the constraints of geochemistry, Cooper and Hall¹ have articulated a thermochemically stable approach to use synthetic mica as a mechanical and chemical protective interphase for alumina fibers in a simple oxide or silicate matrix. The specific interphase material considered is fluorophlogopite ($\text{KMg}_3(\text{AlSi}_3)\text{O}_{10}\text{F}_2$), an easily cleavable trioctahedral fluoromica mineral capable of withstanding temperatures over 1300°C .² A proposed composite of MgAl_2O_4 -spinel-protected alumina fibers/fluorophlogopite interphase/forsterite matrix has demonstrated thermodynamic stability up to 1280°C under standard-state conditions (e.g., within a composite) and up to 1230°C in an open-air environment. The primary purpose of the spinel phase is to establish pseudo-binary local equilibrium at the fluorophlogopite/spinel and spinel/alumina interfaces at temperatures exceeding $\sim 1280^\circ\text{C}$. An additional advantage of the spinel layer derives from its close-packed oxygen-anion sublattice, which allows the spinel phase to act as an effective barrier against K^+ diffusion, dramatically slowing the reaction between fluorophlogopite and alumina that occurs when the temperature exceeds that noted above. Reaction couple studies have also shown that the proposed materials retain distinctly planar interfaces between all the composite components, even if the temperature is sufficiently high that the alumina-mica reaction proceeds.

Preliminary studies also indicate tendencies toward a favorable high-rate mechanical response, specifically the occurrence of debonding at the mica interphase. Cook and Gordon³ and He and Hutchinson⁴ have articulated a debonding criterion that stipulates that, for interfacial debonding to occur at a bimaterial interface, the fracture toughness (strain-energy release rate) of the interface must be less than $\sim 25\%$ that of the phase opposite the interface from the propagating crack. In our composite system, debonding is predicted when considering the intrinsic fracture toughness of the materials involved: alumina, 35 to $40 \text{ J}\cdot\text{m}^{-2}$;⁵ spinel, 10 to $16 \text{ J}\cdot\text{m}^{-2}$;⁶ and fluorophlogopite, $0.8 \text{ J}\cdot\text{m}^{-2}$ (in the basal plane, perpendicular to the c -axis).⁷ Additionally, cracks generated by Vickers microhardness indents in spinel-protected sapphire/fluorophlogopite/spinel-protected sapphire reaction couples provide qualitative evidence supporting appropriate debonding in this composite system.¹

However, the work to date concerning the mechanical behavior of oxide composites using a fluoromica interphase has been predominately qualitative in nature. For this ceramic composite system to be truly useful in engineering applications, more quantitative information must be acquired. In particular, the energy release behavior of the interphase/interphase must be gauged to ascertain debonding potential in a fiber-reinforced composite. Interphase microstructural features, such as grain size and orientation, also influence the mechanical response of the composite. Therefore, the focus of this investigation is to evaluate the interfacial fracture strength of notched spinel-protected alumina/fluorophlogopite laminate beam specimens under four-point flexure, following the test protocol outlined by

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*Member, American Ceramic Society.