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THE INFLUENCE OF FIBER/MATRIX INTERFACE ON THE MECHANICAL BEHAVIOR OF NICALON SiC FIBER REINFORCED GLASS-CERAMIC COMPOSITES

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ABSTRACT

The mechanical properties of unidirectional Nicalon SiC fiber reinforced calcium aluminosilicate (CAS/SiC) and magnesium aluminosilicate (MAS/SiC) glass-ceramic composites have been investigated by tensile testing and a nondestructive laser-ultrasound technique. The barium-stuffed MAS was either undoped or doped with 5% borosilicate glass. The degradation of the elastic stiffness constant C_{11} in the transverse direction due to interface damage was monitored *in-situ* by measuring the laser-generated ultrasound wave velocity. The three composite materials show distinctly different characteristics of macroscopic deformation behavior, which are correlated strongly to the interface degradation. A stronger reduction trend of the elastic constant C_{11} is associated with a larger degree of inelastic deformation. Observations of the fracture surfaces also reveal the close relation between fiber pullout length and interfacial characteristics. Interfaces of these composites have been studied by TEM, and their influence on inhibiting and deflecting matrix cracks is discussed.

INTRODUCTION

It is well known that the fiber/matrix interface has a profound influence on the mechanical behavior of ceramic matrix composites (CMCs).[1,2] Since the failure strain of the matrix is usually smaller than that of the fiber, matrix cracking occurs first under tensile loading.[3] Matrix cracks run into nearby fibers and are deflected along the fiber/matrix interface if the interface is relatively weak. CMCs exhibit nonlinear deformation behavior as a result of multiple matrix cracking and interface debonding/sliding.[4]

Although the importance of the interface has long been recognized, little has been done to investigate the interface damage *in situ* and correlate it to macroscopic deformation. In this study, a laser-ultrasound technique has been applied to reveal the relation between interface degradation and axial deformation under uniaxial tension. Laser-generated ultrasound velocities in transverse plane are measured at various stress (damage) levels. The transverse elastic stiffness can then be determined from these measurements. The deterioration of transverse elastic stiffness due to interface damage is reflected by the reduction of ultrasound velocity measured in the transverse plane. Three Nicalon SiC fiber reinforced glass-ceramic matrix systems, calcium aluminosilicate (CAS), barium-stuffed magnesium aluminosilicate (MAS) undoped or doped with 5% borosilicate glass (BSG), are used in this investigation. BSG functions as a source of boron and diffuses into the interface and fiber during hot consolidation, which affects the interfacial layer formation.[5] The degree of stiffness reduction in the transverse and axial directions will be studied for the above CMCs. The important role of fiber/matrix interface on the macroscopic deformation behavior will be discussed.

EXPERIMENTAL

Nicalon SiC fiber reinforced unidirectional CAS and MAS composite panels were supplied by Corning Inc. The manufacturing procedure and material properties are described elsewhere[6,7]. The Nicalon SiC fiber volume fraction is about 0.35 ~0.4, and the average fiber diameter is 15 μm. There is a carbon-rich interface formed *in situ* during fabrication, which plays an important role in the deformation of Nicalon fiber reinforced ceramics.[8,9,10,11]

Tensile specimens were ~150 mm × 10 mm × 3 mm. Side surfaces of some samples were polished before tensile testing in order to take acetate replicas of surface matrix cracks under loading. Cross-head speed of 0.03 mm/min was used. Axial strain was measured with a one-inch gauge length extensometer. Both continuous and loading/unloading tests[12] were conducted for each CMC system.

Detailed procedures of the laser-ultrasound test have been given previously.[13] The laser pulse from a Q-switch Nd:YAG laser with an operating wavelength of 1.064 μm was delivered to the sample under loading by an optical fiber with a 600 μm core diameter.[14] The laser beam was coupled into one end of the optical fiber by a small convex lens, and the outcoming beam from the fiber was focused onto to the sample by two convex lenses. A small portion of the laser pulse was deflected onto a photodiode and used to trigger a pair of 8-bit, 1 GHz digital oscilloscopes. The signals from two ultrasonic transducers were also connected to the oscilloscopes. These signals were amplified and low pass filtered at 10 MHz. The arrival time of the ultrasound wave was measured by observing the difference in time between the photodiode and the transducer pulse. The systematic delay arising from the propagation of the laser pulse through the optical system was measured and subtracted.

Thin sections parallel to the fiber direction were cut from as-received material, mechanically polished to ~20 μm, and ion-milled. The fiber/matrix interface was then analyzed by transmission electron microscopy (TEM).

RESULTS AND DISCUSSION

Stress-strain behavior of CAS/SiC, MAS0/SiC, MAS5/SiC are shown in Fig. 1. MAS0 refers to undoped MAS and MAS5 refers to MAS doped with 5% BSG. Axial Young's modulus E_L is almost identical (~150 GPa) for MAS5/SiC and MAS0/SiC, and is ~ 130 GPa for CAS. Deformation beyond the linear elastic regime is quite different for the three CMCs. CAS/SiC has a plateau region in the stress range of 200-270 MPa, and shows a "stiffening" effect in the final deformation stage. MAS0/SiC deviates appreciably from linear elastic behavior near 300 MPa, and deforms nonlinearly up to failure without a plateau regime, while MAS5/SiC displays very little nonlinear deformation.

The above characteristics are closely correlated to their interfacial characteristics and corresponding transverse stiffness degradation. The transverse elastic stiffness C_{11} is directly related to the ultrasound velocity by $C_{11} = \rho V_L^2$, where ρ is the density of the composite and V_L is the longitudinal velocity in the transverse direction. The change of C_{11} as a function of applied stress is illustrated in Fig. 2, where symbols are the experimental data from various tests, and continuous lines are from curve fitting to show the general reduction trends. Among the three Nicalon SiC reinforced glass-ceramics, CAS/SiC exhibits the largest degree of reduction of C_{11}, while MAS5/SiC shows the least reduction. The same order applies for axial nonlinear deformation. A large amount of nonlinear deformation from 200 to 270 MPa for CAS/SiC is also associated with the rapid reduction of C_{11} in this range, where the matrix cracking density increases significantly.[15]

Since matrix cracks are nearly parallel to the transverse direction, transverse stiffness is insensitive to the presence of matrix cracks. The reduction of C_{11} is attributed to the degradation of interfacial adhesion as a result of the interaction between transverse matrix cracks and the interface. Although MAS5/SiC shows little nonlinear deformation, matrix cracking is found from surface replicas. Matrix cracking of MAS5/SiC at a stress level of 255 MPa is illustrated in Fig. 3. The absence of a reduction of C_{11} in MAS5/SiC indicates that interfacial damage in the form of debonding/sliding is inhibited substantially in a strongly bonded interface system, and has negligible effect on the overall transverse elastic stiffness. The above observations demonstrate that, to exhibit appreciable amount of nonlinear deformation, significant interfacial damage must occur in addition to multiple matrix cracking. Strong fiber/matrix interface prohibits matrix crack opening considerably and results in small inelastic strain. This observation also confirms that, as pointed out by Kim *et. al.*[16], defining the initiation of matrix cracking from stress-strain curves using the deviation point from the linear regime is inappropriate in CMCs. For CMCs where multiple matrix cracking occurs, the beginning of macroscopic nonlinear deformation is a result of *damage accumulation* due to interface debonding/sliding and consequent matrix crack opening.

As expected, the fiber pull-out length after failure in MAS5/SiC is quite short compared to that of MAS5/SiC and CAS/SiC. This is illustrated in Figs. 4 (a)-(c).

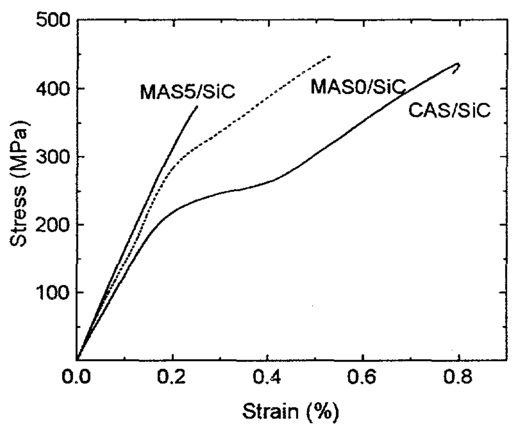


Fig. 1 Stress-strain curves for CAS/SiC, MAS0/SiC, and MAS5/SiC composites.

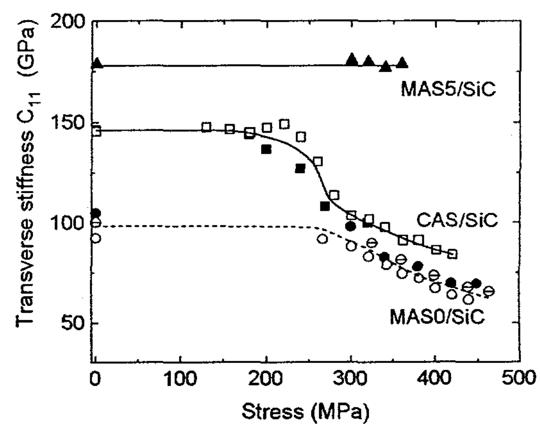
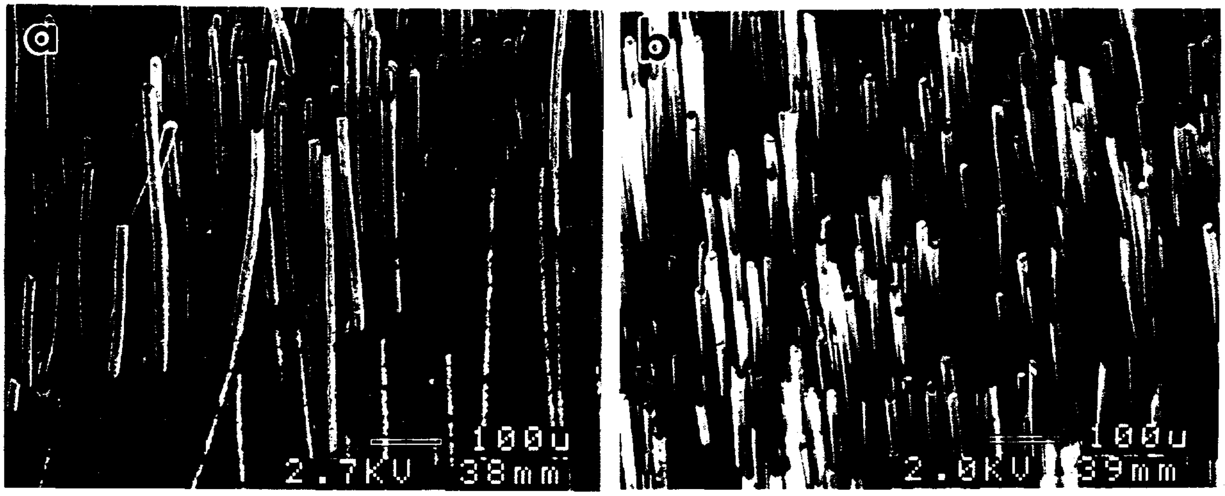


Fig. 2 Reduction of transverse elastic stiffness C_{11} with the increase of stress.

Fig. 3 Optical micrograph of surface replica of MAS5/SiC revealing matrix cracking at 255 MPa.



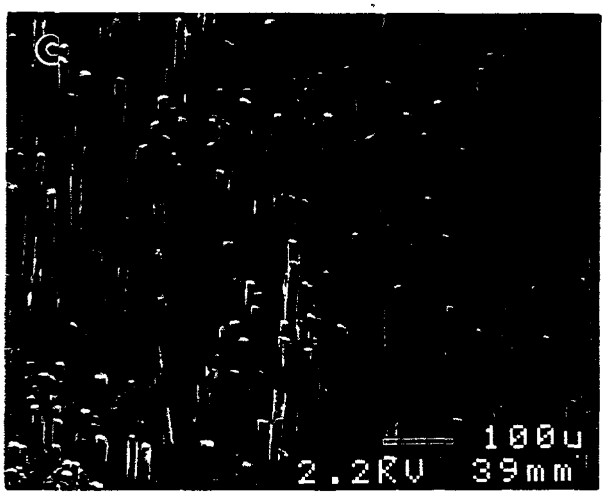


Fig. 4 Fracture surface of unidirectional composites: (a) CAS/SiC; (b) MAS0/SiC; (c) MAS5/SiC.

C_{11} is seen to be quite low for as-processed MAS0/SiC (~100 GPa) compared to MAS5/SiC (~175 GPa). Because the MAS matrix has a lower thermal expansion coefficient (~ 2.6×10^{-6} K^{-1}) than that of the fiber (4.0×10^{-6} K^{-1}),[17] the fiber/matrix interface is under residual tension after processing, which could result in local gaps at the interface. Whereas for MAS5/SiC, this gap is filled by reaction layers because of doping, and strong adhesion at the interface is achieved. This is reflected by the higher value of C_{11} in as-processed MAS5/SiC. Transverse tensile testing also confirms that the transverse Young's modulus E_T of MAS0/SiC is much lower than that of MAS5/SiC.[18] However, the interfacial bonding has little effect on the initial E_L of MAS5 and MAS0 (before matrix cracking occurs). It is the interaction between matrix cracking and the interface that determines the nonlinear deformation behavior.

TEM thin foil observations of MAS5/SiC and MAS0/SiC reveal that a uniform interaction layer formed in MAS5 compared to a slightly porous interfacial structure in MAS0. This is illustrated in Figs. 5 (a)-(b). Preliminary chemical analysis on MAS5/SiC by X-ray energy dispersive spectroscopy (EDS) reveals there is barium segregation at fiber/matrix interface.[19] XPS investigation on 7.5% doped MAS by Larsen *et al.* indicated that boron is present at interfaces in the form of B_2O_3 and BN.[20] We have attempted to detect B or N on MAS5/SiC by parallel energy loss spectroscopy (PEELS) but have so far been unsuccessful due to the low concentration of B and N. A stronger bond does form at fiber/matrix interface because of doping in the MAS system, as revealed by laser-ultrasonic measurements and transverse tensile testing. More study is underway to characterize the chemistry and microstructure of the interface in the MAS system.

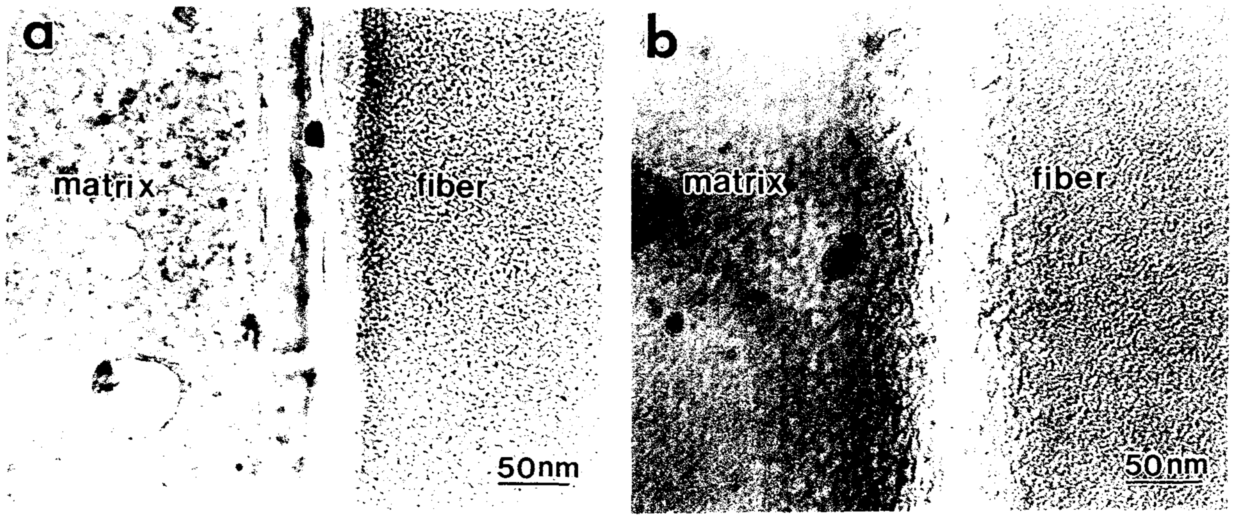


Fig. 5 TEM bright field images of fiber/matrix interfacial region:(a) MAS5/SiC; (b) MAS0/SiC

CONCLUSIONS

Using a laser-ultrasonic technique, progressive interface damage has been monitored *in situ* during uniaxial tension. Strong correlation of interface debonding/sliding with axial nonlinear deformation is found. A stronger reduction trend of the transverse elastic constant C_{11} is associated with a larger degree of inelastic deformation. Among three CMCs investigated, MAS5/SiC shows little nonlinear deformation as a result of doping, consistent with a negligible reduction on transverse stiffness and a short fiber pull-out length after failure. Results indicate that the interaction between matrix cracking and the interface has a significant role on the nonlinear deformation of CMCs. Strongly bonded fiber/matrix interface will inhibit matrix crack opening and results in little inelastic deformation in axial direction.

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