LASER-ULTRASONIC EVALUATION OF DAMAGE IN UNIDIRECTIONAL CERAMIC MATRIX COMPOSITES

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INTRODUCTION

Ceramic matrix composites (CMCs) have attracted great attention because of their potential for high temperature structural applications. Among the CMCs, calcium aluminosilicate (CAS) glass ceramic composite reinforced by Nicalon\textsuperscript{TM} SiC fiber with a carbon-rich interface has been extensively investigated because of their "notch-insensitivity": stresses near holes and notches can be redistributed by inelastic deformation in the form of multiple matrix cracking [1-3]. Thus, stress concentrations can be alleviated near these sites and the risk of catastrophic failure is consequently reduced.

Understanding damage evolution during the deformation of CMCs is very important for constitutive model development of as well as materials design. To contribute to these goals, techniques for \textit{in-situ} monitoring of damage initiation and accumulation are needed. In previous work, damage was characterized mostly by the change of Young's modulus along the loading direction (measured by loading/unloading) in conjunction with metallography [1,4,5]. However, this approach does not provide detailed insight into the anisotropic nature of damage, or about the cumulative effects of fiber-matrix debonding/sliding, radial cracking and shear deformation. In this study, we have pursued an \textit{in-situ} laser-ultrasonic technique to nondestructively measure the anisotropic stiffness degradation under loading.

When a laser pulse is directed at a sample surface, high frequency acoustic waves can be generated by thermal or ablation mechanisms depending on the incident power intensity [6,7]. The resulting propagation of elastic waves through an anisotropic media can then be characterized by the well-known Christoffel equation [8]:

\[ \Omega(V, n) = \det[C_{ijkl} n_j n_k - \rho V^2 \delta_{ik}] = 0, \]

where \( C_{ijkl} \) are the second-order elastic stiffness constants of the material, \( n \) is a unit vector along the bulk wave propagation direction, \( \rho \) is the density of the medium, \( V \) is the phase velocity and \( \delta_{ik} \) is the Kroneker delta. Based on a set of wave velocities measured along various propagation directions, the elastic constants can be deduced by fitting the experimental data to the solutions of the Christoffel equation through nonlinear optimization procedures. Unidirectional composites can be regarded as transversely isotropic materials. Using abbreviated stiffness subscripts, the five independent elastic constants are \( C_{11}, C_{33}, C_{12}, C_{13} \) and \( C_{44} \), while \( C_{22} = C_{11}, C_{23} = C_{13}, C_{55} = C_{44} \) and \( C_{66} = (C_{11} - C_{12})/2 \). As damage accumulates in such composites, the elastic moduli are reduced, resulting in a decrease of the wave propagation velocities [9].

When damage in the form of cracking occurs, elastic waves are generated, i.e., acoustic emission occurs. These dynamic elastic signals propagate through the composites and cause surface displacements which can be ultrasonically detected by a piezoelectric transducer attached to the sample surface [10,11]. This nondestructive technique also provides additional insight into the evolution of damage. In the present study, acoustic emission (AE) events were continuously recorded during loading/unloading of CAS/SiC composite. This was combined with data obtained from laser-ultrasonic and mechanical testing to shed new light on the damage evolution process in unidirectional fiber-reinforced ceramic composite materials.

**EXPERIMENTAL**

Tensile specimens of dimensions \( 150 \text{ mm} \times 10 \text{ mm} \times 3 \text{ mm} \) with continuous fibers parallel to the loading direction (the length direction of the specimen) were cut from the unidirectional CAS/SiC composite plate (provided by Corning Inc.). The specimen ends were bonded with aluminum tabs using modified epoxy (3M Company) to ensure even load transfer. Tensile testing was performed on an Instron 4200 machine at room temperature. Axial strain was measured by a 2.54 cm gauge length extensometer.

Laser pulse was generated by a Q-switch 1.064 \( \mu \text{m} \) wavelength Nd:YAG laser. Pulses with an energy of about 5 mJ were delivered to the loaded sample through an optical fiber [12,13]. A laser scan along the two principal directions of the sample was controlled by an X-Y-Z positioner. Ultrasonic wave arrivals were detected by two broadband piezoelectric transducers in contact with the sample. The detected signal was transferred to digital oscilloscopes. The chosen coordinates and the experimental setup are shown schematically in Fig. 1.
During the experiments, a laser scan was performed at a pre-set stress level, and then repeated after unloading to 10 MPa. Before each scan, the laser source was re-aligned to keep the laser scan along the same positions. Acoustic emission events were recorded continuously by the two sensors during the entire tensile test. The root-mean square average of the waveform amplitude was calculated automatically each time the waveform was recorded.

Fig. 1. Experimental setup and scan-path for laser-ultrasonic experiments.
RESULTS AND DISCUSSION

Fig. 2 shows a tensile stress-strain curve for a unidirectional CAS/SiC sample during repeated loading/unloading up to 320 MPa. The acoustic emission events corresponding to loading the sample to 200 MPa are also shown in Fig. 2. Fig. 3 displays the accumulated AE counts during the entire loading/unloading cycles.

Fig. 2. Tensile stress-strain curve and corresponding AE events over stress range 0 ~ 200 MPa.

Fig. 3. Tensile stress-stain curve and accumulated AE during the entire loading/unloading cycles.
process. From these figures, it is clear that the σ-ε curve starts to deviate from linearity over the stress range of 130-150 MPa, which is the accepted matrix cracking stress threshold [1]. However, in the linear region, AE events are detected, associated with cracks extension from initial flaws formed during fabrication. They appear to have little effect on the macroscopic deformation behavior and they have only partially spread across the sample. With the development of matrix cracking, numerous AE events are detected and an appreciable amount of inelastic deformation occurs over the stress range of 130 MPa to 320 MPa. Fig. 4 (a)-(c) show matrix cracks at different loading stages.

During unloading from 320 MPa, a number of AE events were observed whereas only one or two AE signals were recorded when unloading from 200 MPa or 270 MPa. AE events during unloading might originate from interface sliding [14] and some crack closure effects; these results imply that limited damage are occurring on unloading and they are not sufficient to generate AE signals at low stress levels, although the hysteresis loops indicate that there was energy dissipation during unloading/reloading. The unloading curves from 270 MPa and 320 MPa (Fig. 2) indicate that the unloading elastic modulus does not change much, which implies that the density of matrix cracks seems to saturate near the end of the test (320 MPa) as shown in Fig. 4(b) and (c).

It is noticed that, for most paths of reloading, AE events do not occur during reloading until near the previous peak stress level. These observations indicate that unloading/reloading does not contribute to significant additional damage. Although the hysteresis loops were formed as a results of energy dissipation during loading/unloading through frictional sliding at the fiber/matrix interface, the results suggest that interface sliding at low damage levels is not sufficient to yield detectable AE signals.

With the development of damage, the elastic stiﬀnesses are degraded. This is manifest by a reduction of the ultrasonic wave velocity as well as a reduced unloading modulus on the stress-strain curve. The elastic stiﬀness constant $C_{11}$ can be found directly from the longitudinal wave velocity at the epicenter position. In the transversely isotropic $X_1X_2$ plane, independent elastic constant measurements by the resonant ultrasound spectroscopy technique [15] indicated that $C_{44} \approx C_{66}$, thus, $C_{44}$ (or $C_{66}$) can determined by averaging the shear velocity at different scan positions in the $X_1X_2$ plane, because the velocity of the pure shear wave is given by $V = \sqrt{C_{44} / \rho}$.

The other unknown elastic constants $C_{33}$ and $C_{13}$ are determined from the wave velocity measured within the principal $X_1X_3$ plane. Again, the two shear modes are not clearly distinguished, thus identifying $C_{33}$ and $C_{13}$ are based on the longitudinal wave speeds using Eq. (1) through a nonlinear curve fitting procedure.
Fig. 4. Optical micrographs of replicas show surface matrix cracks at different stress levels: (a) 230 MPa; (b) 270 MPa; (c) 330 MPa.

Fig. 5. Measured and curve fitted longitudinal wave velocity in $X_1X_3$ plane at four stress levels.
The measured wave velocities in the $X_1X_3$ plane together with the ones determined by curve fitting at four different stress levels (0, 200, 270 and 320 MPa) are plotted in Fig. 5. Significant delay in the wave arrival time is observed when the tensile stress is increased from 200 MPa to 270 MPa, and reflects the contribution of damage to the elastic properties. This is consistent with the plateau region shown on the $\sigma$-$\varepsilon$ curve. Similar measurements and calculations were carried out for another sample at stress levels of 180 and 240 MPa.

The measurements performed at 10 MPa after unloading from each successively higher stress indicate that the wave arrival velocities increase slightly after unloading, although not significantly compared with the data before unloading. This result implies that there may be some crack closure but the damage certainly remained after unloading.

Elastic constants $C_{11}$, $C_{33}$ and $C_{44}$ together with the unloading elastic moduli $E^*$ obtained from the partial unloading test are plotted in Fig. 6 (reloading modulus was used at the stress 180 MPa). $C_{11}$, $C_{33}$ and $E^*$ show the same reduction trend, although the degree of reduction near the end of matrix crack saturation for $E^*$ seems to be less than that of $C_{11}$ and $C_{33}$.

Laser-ultrasonic measurements showed that there is an overall degradation of the elastic stiffness constants. Micrographs of the fracture surface show "crack-

![Graph showing elastic constants and stress relationship](image-url)

Fig. 6. Elastic constants determined by laser-ultrasonic method and unloading elastic modulus by tensile test.
like” discontinuities in the transverse plane normal to the loading direction [13]. The decrease of transverse elastic constants might be due to fiber/matrix interface debonding, residual matrix cracking and shear deformation involved in matrix crack propagation. This softening effect is still under investigation.

CONCLUSIONS

The laser-ultrasonic technique has been successfully applied to study damage evolution in a unidirectional fiber-reinforced CAS/SiC ceramic composite. Elastic constants were determined based on ultrasound wave velocity measurements along various propagation directions. The results show that wave propagation is sensitive to damage accumulation in the sample. In conjunction with AE recording, the nondestructive laser-ultrasonic method provides valuable knowledge of the overall anisotropic damage in fiber-reinforced ceramic composites. Damage accumulation under loading is manifest by a reduction of the elastic constants, AE signals, and the σ-ε hysteresis loops.

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REFERENCES