ANALYSIS OF LOCAL IN SITU STRAIN IN CERAMICS AND CERAMIC MATRIX COMPOSITES*

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ABSTRACT

A nonintrusive technique using scanning laser acoustic microscopy (SLAM) for measuring the localized strain of ceramics was developed for a LAS monolithic, a SiC/LAS monofilament composite, and a SiC/CAS unidirectionally reinforced composite. The localized strain analysis was used to assess residual stresses due to processing. A correlation between the relaxation stress and the fracture behavior of each material was found and shown to correspond to matrix micro-cracking and secondary cracks.

INTRODUCTION

Ceramics and ceramic matrix composites (CMCs) have desirable properties which make them viable candidates for use in applications at elevated temperatures [1-5]. High resolution, nonintrusive measurement methodologies provide an effective mechanism for accurate evaluation of these materials. These technologies uniquely provide a noncontacting analysis of the localized effect of fracture behavior and its influence on the macroscopic material behavior.

This work describes the analysis of localized strain behavior for selected ceramic systems. The matrix constituent was also analyzed to provide a baseline standard against which to monitor the behavior of the composite.

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EXPERIMENTAL PROCEDURE

Lithium alumino-silicate (LAS) glass ceramic†, unidirectionally silicon carbide reinforced calcium alumino-silicate (SiC/CAS) composite†, and monofilament silicon carbide reinforced lithium alumino-silicate (SiC/LAS) composite were evaluated in this work. The SiC/LAS composites were formed at the University of Dayton Research Institute by hot pressing individual Nicalon SiC fibers between two as-received LAS plates.

The four-point flexural strengths of half of the as-received specimens (Military Standard 1942A specimen configuration B [6]) from each material system were measured using an Instron LM1 Universal Tester operating at a crosshead rate of 0.0085 mm/second. The mean flexural strength of each system was used as an upper bound for subsequent nonintrusive testing of the remaining test specimens.

Nonintrusive testing was performed using an in situ MOR fixture in the scanning laser acoustic microscope (SLAM) [7]. Each test bar was stressed to a pre-selected maximum applied stress state and then relaxed to its initial stress state. The SLAM interference fringe shift was measured at the initial stress state, at a series of arbitrarily selected (predetermined) stress values during loading, and at the relaxed stress state. The normalized fringe shift at each stress state was used directly to calculate the strain induced within the maximum tensile zone of the specimens, as described by Kent et al. [8].

To confirm the independence of the maximum applied stress on the measured behavioral response, a small sample of LAS test specimens were stressed to three different maximum stress values and strain was measured nonintrusively.

The four-point flexural strength of the test specimens was measured following nonintrusive testing. The post-strain analysis results were correlated with the nondestructive testing parameters.

RESULTS

The mean flexural strength measured for the monolithic LAS was 117.2 MPa. This value agrees well with the median flexural strength reported in the literature for this material (113.0 MPa) [9]. The load-deflection curve recorded during strength testing was linear to failure. This deflection curve provides the macroscopic response of

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† provided by Corning

the system to specimen stressing.

The SLAM strain data represent mechanical behavior at the localized level. The typical local stress-strain behavior for LAS is shown in Figure 1. On the localized scale, significant deviations from

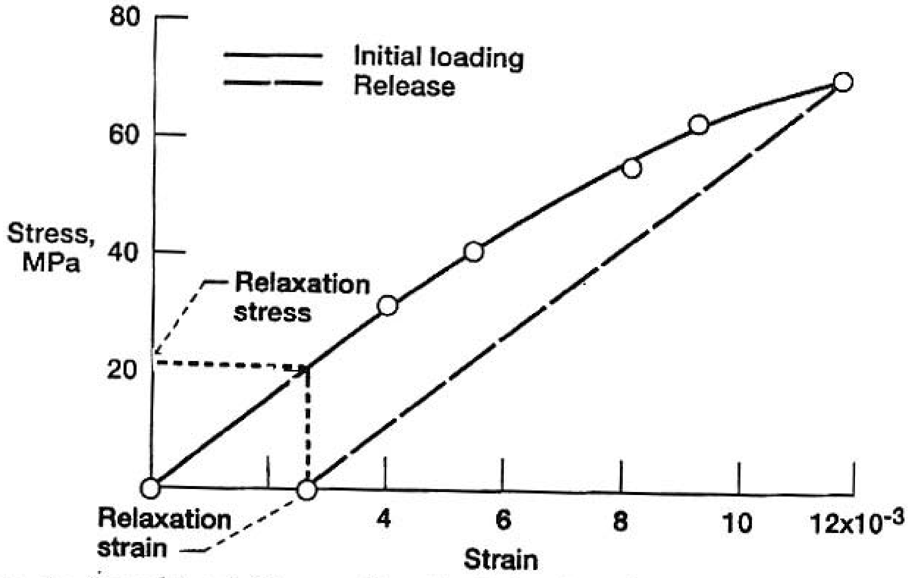

Figure 1: Localized Stress-Strain Behavior for LAS

linearity and apparent hysteresis upon unloading was observed. A permanent strain, defined as relaxation strain (ϵ_r), was indicated. The relaxation strain due to subcritical loading is attributed to relaxation of the inherent residual tensile stresses present in LAS due to processing. At the local level, microcracks initiate in the high stress regions to relieve the residual stress. However, the macroscopic applied stress is not sufficient to sustain crack propagation to failure and only the local phenomena is observed. The relaxation strain corresponds to a relaxation stress, as denoted in Figure 1.

As shown in Table 1, the standard deviations of the measured relaxation and fracture stresses are both approximately 15 percent about the means. However, the mean stress relaxation ratio (the ratio of the relaxation stress to the fracture stress) on a specimen to specimen basis is 18.5 ± 1.5 percent (a standard deviation of 8 percent about the mean). This demonstrates that there is a definitive correlation between the relaxation stress and the fracture stress.

Table 1: Empirical Localized Relaxation and Fracture Stresses (LAS)

Specimen	Rel. Stress (σ_r), MPa	Frac Stress (σ_f), MPa	Ratio (σ_r/σ_f)
1	29.6	148.3	0.200
2	20.0	115.0	0.174
3	22.3	120.6	0.185
4	23.6	139.8	0.169
5	20.2	107.0	0.189
6	14.8	92.7	0.160
7	18.5	105.7	0.175
8	22.5	106.4	0.212
9	13.0	63.0	0.206
Mean±Sigma	20.5 ± 4.9	110.9± 25.4	0.185±.018

The stress relaxation ratio appears to be a constant parameter for each material system. Table 2 shows the measured relaxation strains and corresponding stress relaxation ratios for three LAS test specimens unloaded from different subcritical applied stress values. Although this is a limited sample set, the data clearly indicate that the stress relaxation ratio is constant for the LAS material. Post-mortem analysis of the fracture surface within the tensile region of the LAS indicates the presence of multiple local micro-crack initiation sites and secondary cracking from the primary crack path. This corroborates the presence of localized nonlinear mechanical behavior. Figure 2 shows SEM micrographs from two representative LAS specimens.

The SiC/LAS monofilament composite specimen stress-strain behavior also shows the development of nonlinear behavior (Figure 3). In this case, the stress relaxation ratio exhibits a bimodal distribution with the bifurcation predicated on the dominant fracture mode of the test specimen. The mean stress relaxation ratios were 0.358 for specimens which failed due to shear and 0.220 for specimens failing due to uninhibited tensile cracking. In general, stress relaxation ratios greater than 0.30 correspond to shear failure at the fiber matrix interface and a correspondingly weaker interface.

Table 2: Relaxation Strain and Stress of LAS for Unloading from Selected Values

Spec.	Unload Stress	ϵ_r,E-3	σ_r, MPa	σ_f, MPa	Ratio
1	55	3.53	28	152	0.184
	67	3.42			
	83	3.53			
2	55	2.83	24	128	0.188
	67	2.71			
	83	2.94			
3	55	2.85	24	118	0.203
	67	2.85			
	83	2.96			

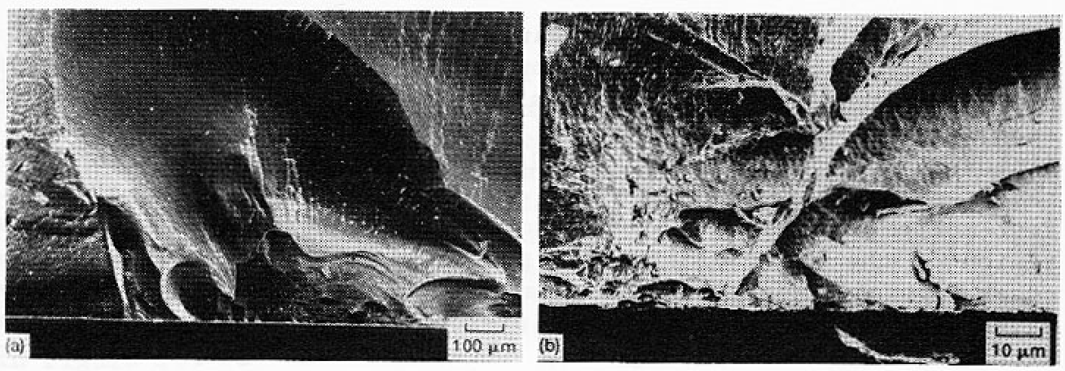

Figure 2: SEM Micrographs of Fracture Surface of LAS

SEM micrographs of the fracture surface of the interfacial bond region are shown in Figure 4. In Figure 4a, there is strong chemical bonding between the fiber and matrix. In this case, the load is not effectively transferred from the matrix to fiber and cracks propagate through the fiber without inhibition. Failure of these test specimens occurs due to tensile cracking through the specimen; the fiber has no observable effect on the fracture behavior of the material. Figure 4b shows the interfacial region of

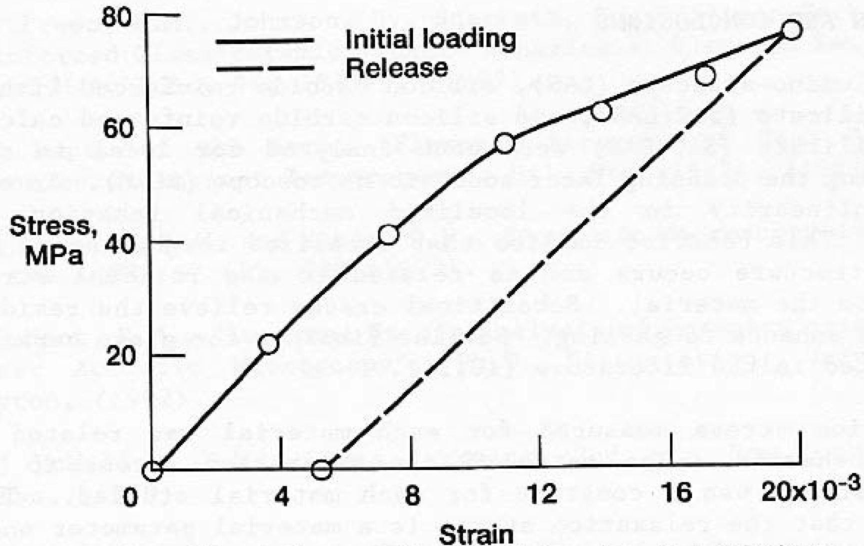

Figure 3: Localized Stress-Strain Behavior for SiC/LAS

(a) (b)

Figure 4: SEM Micrographs of Fracture Surfaces of SiC/LAS

a test specimen which exhibited a high stress relaxation ratio. The presence of excessive voids around the fiber-matrix interface suggests lower interfacial bond strength. These test specimens characteristically failed in shear at the fiber-matrix interface.

The failure process for the fully reinforced SiC/CAS composite specimens was much more complex than observed for the monofilament composites. Local strain analysis of these test specimens demonstrated nonlinearity and relaxation strains, analogous to that described for LAS and SiC/LAS. However, a lower mean stress relaxation ratio (0.11) was found and attributed to a change in the residual stress distribution in the composite; the CAS matrix is in residual tension due to the CTE mismatch with the fiber.

DISCUSSION AND CONCLUSIONS

Lithium alumino-silicate (LAS), silicon carbide reinforced lithium alumino-silicate (SiC/LAS), and silicon carbide reinforced calcium alumino-silicate (SiC/CAS) were each analyzed for local in situ strain using the scanning laser acoustic microscope (SLAM). In each case, nonlinearity in the localized mechanical behavior was observed. This behavior implies that localized toughening of the ceramic structure occurs and is related to the residual stress inherent to the material. Subcritical cracks relieve the residual stress and enhance toughening. Similar findings for glass ceramics are reported in the literature [10,11].

A relaxation stress measured for each material was related to fracture behavior. The ratio of the relaxation stress to the fracture stress was a constant for each material studied. This indicates that the relaxation stress is a material parameter and a specific threshold for microcrack initiation to relieve inherent residual stresses exists. Preliminary investigation of an analogous correlation for threshold stress intensity to the fracture toughness of LAS provided in the literature [12] gives a ratio of 0.20. This correlation, within the experimental uncertainty, is consistent with that observed in the present study.

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