MATERIALS

Reliable Ceramic Structural Composites Designed with a Threshold Strength

Technical Report # 3

Residual Stress Induced R-Curves in Laminar Ceramics that Exhibit a Threshold Strength

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Abstract

A stress intensity (K) function describing the extension of a crack through bounding compressive layers has been experimentally verified using two methods. For one method the crack length was measured using a replicating technique; in the other, the crack length was continuously measured in-situ with an optical microscope during loading. In both, cracks were observed to propagate in a stable, nearly straight path across the compressive layer with increasing applied stress. Catastrophic failure was observed when the crack fully extended across the compressive layers at an applied stress predicted and described as the threshold stress [1]. This behavior and agreement was only valid when either the compressive stress and/or thickness of the compressive layer were small.

1 Introduction

It was recently shown that a threshold strength (i.e. a strength below which there is theoretically zero probability of failure) can be obtained in laminar ceramics composed of alternating layers of one material separated by thinner layers of a second material which contains biaxial, residual, compressive stresses [1]. Large flaws within the thicker layers were observed to propagate and then arrest as they entered the compressive layers. For laminates of identical architecture, failure occurred at the same applied tensile stress despite large differences in the initial size of the crack introduced into the thicker layers. This stress was the threshold strength, i.e., the stress below which failure could not occur.

A stress intensity solution developed to describe this phenomena was expressed as

$$K = \sigma_a\sqrt{\pi a} + \sigma_c\sqrt{\pi a}\left[\left(1+\frac{t_1}{t_2}\right)\frac{2}{\pi}\sin^{-1}\left(\frac{t_2}{2a}\right)-1\right] \quad (1)$$

where σ_a is the applied tensile stress, $2a$ is the crack length, and t_1 and t_2, the thicknesses of the compressive and tensile (thicker) layers, respectively. The magnitude of the biaxial residual compression within the compressive layers, σ_c, is given by

$$\sigma_c = \varepsilon_r E_1' \left(1 + \frac{t_1 E_1'}{t_2 E_2'}\right)^{-1} \tag{2}$$

where ε_r is the residual differential thermal strain, $E_i'=E_i/(1-\nu_i)$, E the Young's modulus, and ν_i the Poisson's ratio. Inspection of Eq. 1 shows that as the crack enters the compressive layer, the second term on the right side of the equation reduces the value of the stress intensity function. Since further extension through the compressive layer continues to reduce K, propagation must occur in a stable manner with increasing applied stress.

It was further suggested that failure would occur once the crack fully extended across the compressive layer (i.e. when $2a = t_2 + 2t_1$) and $K \geq K_c$, when the applied stress reached a critical value called the threshold strength

$$\sigma_{thr} = \frac{K_c}{\sqrt{\pi \frac{t_2}{2}\left(1 + \frac{2t_1}{t_2}\right)}} + \sigma_c \left[1 - \left(1 + \frac{t_1}{t_2}\right)\frac{2}{\pi}\sin^{-1}\left(\frac{1}{1 + \frac{2t_1}{t_2}}\right)\right] \tag{3}$$

Fracture surface observations indicated, however, that the crack did not propagate straight across the compressive layers as assumed in the development of Eqs. 1 and 3. Instead, the crack was observed to bifurcate within the compressive layer. Experimental results described elsewhere [2] will show that when the compressive stress and/or compressive layer thickness is small, the crack does propagate straight across the compressive layer as assumed in Eq. 1. It is these conditions that will be explored here in order to experimentally determine the validity of Eq. 1.

2 Experimental Procedure

2.1 Specimen Preparation

Multilayered laminar composites consisting of alternating 540 ± 10 μm thick tensile layers and 55 ± 5 μm thick compressive layers were fabricated via a sequential slip casting technique described elsewhere [3]. The specimens contained tensile layers composed of 0.95 volume fraction Al_2O_3[†] and 0.05 volume fraction $Zr(3Y)O_2$[‡]; the ZrO_2 was included to control the grain size of the Al_2O_3. The compressive layers were composed of a mixture of 0.25 volume fraction mullite[§] / 0.75 volume fraction Al_2O_3. Experiments described elsewhere [2] showed that this particular choice of compressive layer composition and thickness would yield the desired laminate specimens in which cracking propagated straight across the compressive layers without bifurcation. Dispersed aqueous slurries of 0.30 volume fraction for the Al_2O_3 tensile layers and 0.15 volume fraction for the mullite / Al_2O_3 compressive layers were made at pH 11 (TMA-OH),

† AKP-15, d_{50}=0.6 μm, Sumitomo, Japan
‡ TZ-3Ys, d_{50}=0.4 μm, Tosoh, Japan
§ MULSM, d_{50}=0.7 μm, Baikowski, France

homogenized by attrition milling (Union Process Szegvari Attritor, 3 mm $Zr(3Y)O_2$ milling media), and then allowed to equilibrate for 12 hours prior to casting.

Laminate plates (approximately 70 x 60 x 4 mm before firing) were cast, dried at room temperature for several days, and fired at 1550°C / 2 hrs in air (5°C/min heating and cooling rate). Biaxial, residual compression within the compressive layers, caused by differential thermal contraction of the laminae during cooling from sintering, was calculated to be 350 MPa using Eq. 2 and the property data listed in Table 1. **The properties of the multiphase compressive layers reported in Table 1 were estimated using the non-linear analyses of Ravichandran [5] for the elastic modulus and Turner [6] for the coefficient of thermal expansion. The Poisson's ratio, however, was estimated using a simple linear rule of mixtures, as suggested by Ravichandran [5]. Linear rule of mixtures estimates were also used to determine the properties of the multiphase tensile layers, due to the relatively low second phase content. In addition, the residual, compresive stress was measured on a surface of the compressive layer, far from any edge, that was produced by a fracture. A value of 363 ± 2 MPa was obtained using the piezospectroscopic method which uses the stress-induced shift in the fluorescence spectra of trace Cr^{3+} impurities in alumina [4]. The measured and calculated values of the compressive stress are in reasonable agreement.**

Material	CTE ($x10^{-6}$ C^{-1})	E (GPa)	ν
Al_2O_3	8.30 *(7)*	401 *(7)*	0.22 *(7)*
$Zr(3Y)O_2$	11.35 *(8)*	205 *(8)*	0.32 *(8)*
Mullite	5.30 *(7)*	220 *(7)*	0.27 *(7)*

Table 1. Materials property data for laminate constituents. References are included in parenthesis next to values.

Bars, used for flexural loading experiments, were diamond cut (approximately 3.5 x 3 x 50 mm) from the plates and one of the lateral surfaces of each specimen was polished to 6 μm with diamond abrasive. An ~300 μm long pre-crack was introduced into the middle of the central Al_2O_3 layer on the polished surface of each specimen with a Vickers indenter loaded to 5 kg. This pre-cracking ensured that crack extension across the tensile layer to the bounding compressive layers would occur at low enough load to allow stable growth of the crack through the compressive layers.

2.2 Indirect Crack Path Observation Using Cellulose Acetate Replicas

Two specimens were tested in transverse, 4-pt flexure (i.e. loading direction parallel to the laminate planes) on a screw-driven testing machine operating in displacement control mode (Instron Model 8562, crosshead speed = 0.01 mm/min, spans = 13 and 30 mm), such that the indented face of the specimen was put in tension, as shown in Fig. 1. Cellulose acetate replicas of the crack on the tensile surface were made at different loads by stopping the loading machine and holding the load at the desired value while replicating the crack. The replicas were made at the initiation of the load dwell; at least three replicas were made at each load interval to ensure reproducibility. Crack lengths were then measured from the replicas with an optical microscope (Nikon Labophot) fitted with a Nomarski polarizing filter.

2.3 Direct Optical Crack Path Observation

Another specimen was tested with a different transverse, 4-pt flexure fixture (spans = 10 and 20 mm) [9] that was mounted on the stage of an optical microscope to allow direct, in-situ observation of the crack on the tensile surface of the beam. Mechanical loading was achieved with a piezoelectric actuator. Observation of stable crack propagation during loading was made using ultraviolet lighting and fluorescent penetrating dye (Met-L-Clek FP 90, Helling KG, GmbH, Hamburg, Germany). Although the loading was carried out under displacement control, no attempt was made to set the displacement rate at any given value. After a predetermined load was achieved, it was held for 10 min to allow time for the crack to reach its full extension. Crack lengths were then measured from a video recording taken during the testing.

3 Results

As reported earlier [1], the pre-crack introduced with the indenter would extend across the thick, tensile layer at a low load (≤100 MPa) and arrest at the interface between the tensile and compressive layers. Figure 1 shows a series of micrographs of cellulose acetate replicas for the arrested crack as it penetrated one of the two bounding compressive layers with the sequentially increasing applied load. The crack opening displacement under load appears to be helpful for accurate crack length measurements. Failure eventually occurred at a load slightly higher than the load used for the final replica. For this specimen, the failure stress of 208 MPa is in good agreement with the strength of 194 MPa predicted by Eq. 3, using values of K_c = 2.7 MPa•m$^{1/2}$ for the mullite/alumina material [10]. Similar agreement was obtained for a second specimen.

Similar behavior was also seen during the direct observation of the crack under loading using the dye penetrant and fluorescent lighting. However, in this experiment the load that could be achieved with the piezoelectric actuator was insufficient to fully drive the crack through the compressive layer.

The tensile stress applied to the crack on the surface of the specimen, σ_a, was calculated by substituting the applied load and specimen dimensions into the bending beam formula for 4 point flexural loading

$$\sigma_a = \frac{3P(s_o - s_i)}{2bh^2} \tag{4}$$

where P is the applied load, s_o and s_i the outer and inner spans, b the specimen width, and h the specimen height. The pertinent crack geometry was taken to be a through-thickness crack as observed from the fracture surface in the experiments performed before. Therefore, the applied stress determined with Eq. 4 and the appropriate crack length were then substituted into the stress intensity expression for a through-thickness slit crack in an infinite plate subjected to uniaxial tension

$$K_a = \sigma_a \sqrt{\pi a} \tag{5}$$

to obtain the applied stress intensity factor vs. crack length data shown in Fig. 2.

4 Discussion

The solid line in Fig. 2, representing the theoretical prediction, was determined by substituting the appropriate values for these laminates (σ_c = 350 MPa, t_1 = 55 μm, t_2 = 540 μm, and $K = K_c$ = 2.7 MPa•m$^{1/2}$) into Eq. 1 to determine the penetration depth of the crack into the compressive layers. The crack length that includes the penetration depth, along with the applied stress used to calculate it, were then substituted into Eq. 5 to determine the predicted applied stress intensity shown by the solid line in Fig. 2.

As shown, the data from the two different experiments **are in good agreement** with the fracture mechanics model for the straight through crack as expressed by the stress intensity function shown in Eq. 1. In addition, as predicted, the extension of the crack through the compressive layer occurred in a stable manner. It should also be noted that the predicted value of the threshold strength is **in good agreement** to the measured value. It can thus be concluded that current stress intensity analysis accurately predicts the stress intensity (Eq. 1) and threshold strength (Eq 3) for conditions where the crack extends straight through the compressive layers. It can also be seen, that toughening arises from the shielding of the applied stress by the residual compression as the crack enters the compressive layers.

It is also important to note that, although Eq. 1 was derived for the case of a slit crack in a semi-infinite plate under uniaxial tension, it appears to be applicable to the bending test geometry as well. One of the reasons for this good correlation could rest with the fact that when the surface crack introduced by indentation extends across the surface to the compressive layers, it also extends to the center of the bending beam as well [1]. Thus, although the specimen is not placed in pure tension but is instead subjected to a stress gradient, the crack is a slit crack that fully extends through the applied tensile stress gradient.

Other studies not detailed here [2] have further supported the validity of the model for other laminates of widely varying architectures and composition. These studies also confirm the initial observation [1] that for conditions of higher compressive stress and/or thicker compressive layers, the crack bifurcates as it enters the compressive layers. For the case of bifurcation, Eq. 1 does not physically model the crack, and Eq. 2 under estimates the threshold strength. Work is in currently in progress to explore the bifurcation phenomena more extensively, both experimentally [2] and analytically [11].

Conclusions

Observations have been made that bolster confidence in a previously developed model describing the phenomena of threshold strength. It has been shown that this model is applicable in cases where the crack proceeds in a straight path through the compressive layer. Current studies underway further validate this conclusion [2], and seek to develop a more robust model describing the threshold phenomena that also describes the additional case where the crack bifurcates within the compressive layer [11].

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FIGURES

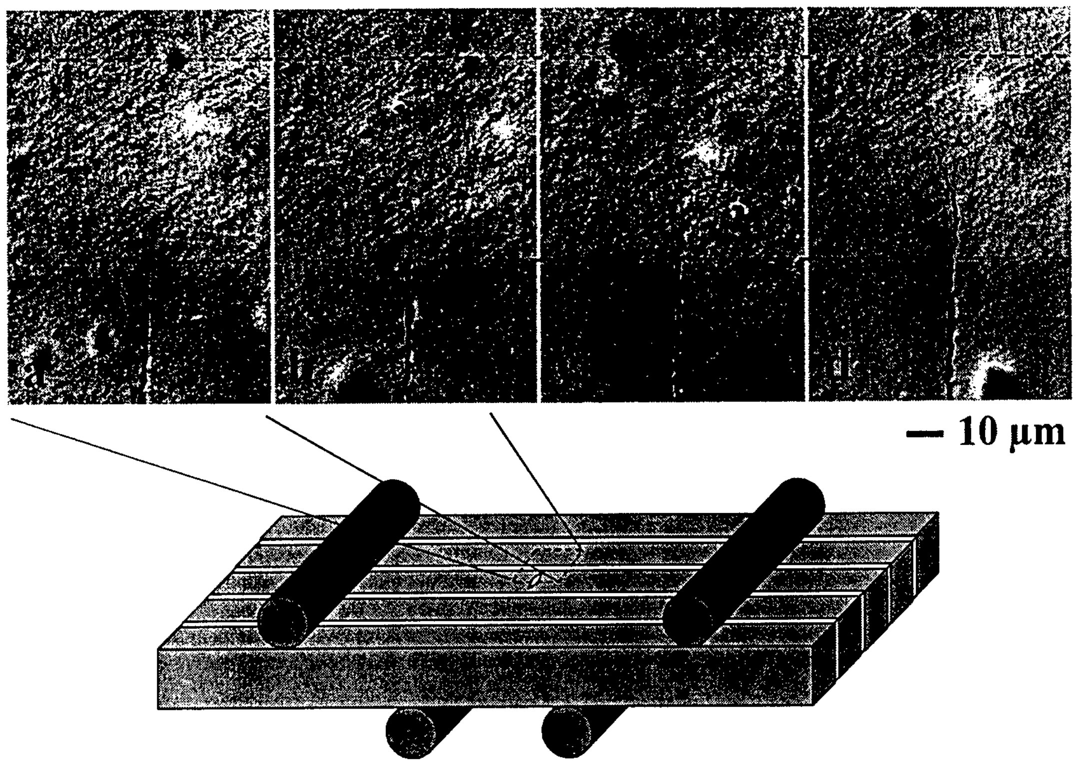

Fig. 1. Schematic of the transverse 4-point flexure configuration and optical micrographs of cellulose acetate replicas of the arrested crack within one of the compressive layers of one of the specimens taken during loading at applied stresses of: a) 112 MPa; b) 140 MPa; c) 168 MPa; and d) 195 MPa. The specimen failed soon after at an applied stress of 208 MPa. Crack penetration depths within the compressive layers of 9.1, 17.5, 28.4, and 48.2 μm, respectively, were measured from the micrographs. Note: Dashed lines were added to indicate the boundaries of the compressive layer.

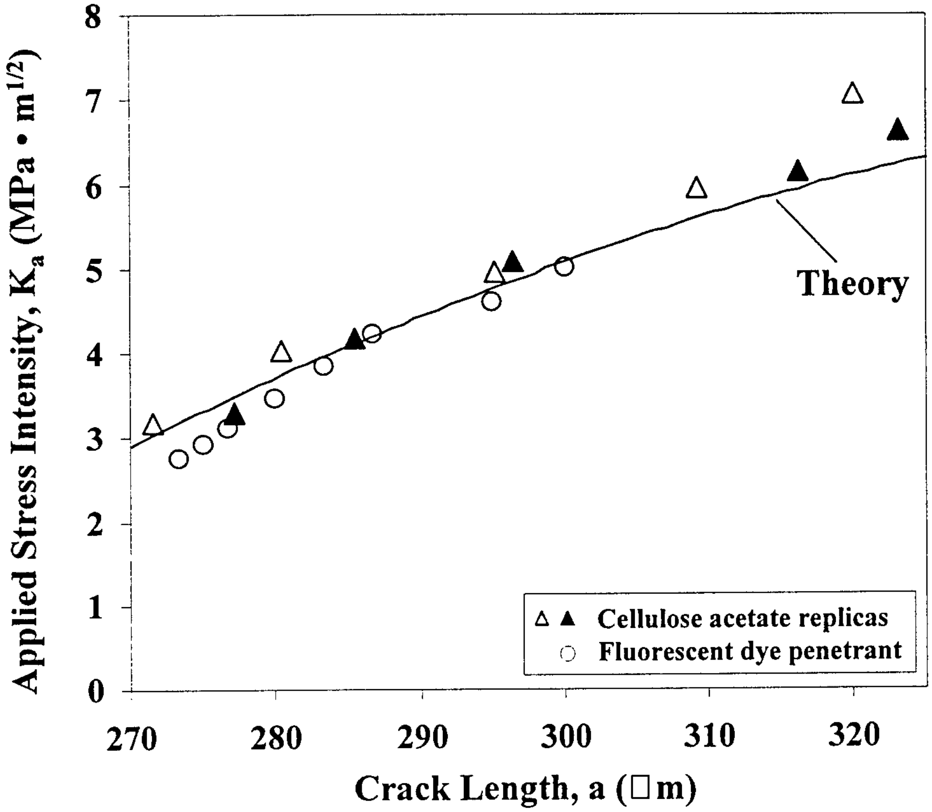

Fig. 2 Plot of applied stress intensity, K_a, for cracks propagating through the compressive layers of the test specimens. Solid line represents model prediction. Crack growth was observed by either cellulose acetate replication or fluorescent dye penetrant.

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13. ABSTRACT (Maximum 200 words)

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