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6. AUTHOR(S)
Frederick F. Lange, PI

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)
Materials Department
College of Engineering
University of California
Santa Barbara, CA 93106-5050

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13. ABSTRACT (Maximum 200 words)CERAMICS THAT EXHIBIT A THRESHOLD STRENGTH

F. F. Lange, M.P. Rao, K. Hbaieb, and R.M. McMeeking, UCSB, Materials Dept

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As reviewed below, a threshold strength can also be obtained by incorporating periodic compressive layers within a brittle material. It is shown that the compressive layers can stop cracks larger than a given size, and an increasing stress must be applied to 'push' the crack through the compressive layers to cause catastrophic failure. Factors that affect the threshold strength are reviewed.

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CERAMICS THAT EXHIBIT A THRESHOLD STRENGTH

F. F. Lange, M.P. Rao, K. Hbaieb, and R.M. McMeeking
Materials Department
University of California at Santa Barbara
Santa Barbara, CA 93106

ABSTRACT

The strength of a brittle material is not a singular value, but a distributed set of values that reflect the large variety of flaws (types and sizes) that are incorporated during processing. The distribution of strength values obtained during testing of ceramic specimens, all made at one time, are generally characterized by statistical parameters that vary with the processing method and processing period, That is, most manufactures cannot control the nature of the flaws they inadvertently incorporate during processing. Proof testing, i.e., the pre-stressing of a component to a specific value, can be used to truncate the statistical distribution, therefore defining a minimum, or threshold strength for components that do not fail the proof test. Although there is added cost to proof testing, it allows the designer to ensure reliability.

As reviewed below, a threshold strength can also be obtained by incorporating periodic compressive layers within a brittle material. It is shown that the compressive layers can stop cracks larger than a given size, and an increasing stress must be applied to 'push' the crack through the compressive layers to cause catastrophic failure. Factors that affect the threshold strength are reviewed.

INTRODUCTION

It was recently shown that a threshold strength (i.e. a strength below which the probability of failure is zero) can be obtained in laminar ceramics composed of periodic, alternating layers of one material separated by thinner layers of a second material. [1] The second layer contains biaxial, residual, compressive stresses due to either differential thermal contraction or a phase transformation during cooling from the fabrication temperature. A threshold strength has been the 'holy grail' of structural ceramics. Large flaws within the thicker layers that extend at a lower stress arrest as they entered the compressive layers. For periodic laminates, failure never occurs below a threshold stress despite large differences in the initial size of the crack present in the thicker layers.

STRESS INTENSITY FUNCTION OF A STRAIGHT CRACK

The stress intensity function that describes the stable extension of the crack through the thin, compressive layer is expressed as [1]

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

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

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

where ε_r is the residual differential thermal strain, $E_i' = E_i/(1-\nu_i)$, E the Young's modulus, and ν the Poisson's ratio. Inspection of eq. (1) shows that as the crack enters the compressive layer, the second term reduces the value of the stress intensity function. Since extension through the compressive layer continues to reduce K, the stress must be continuously increased to extend the crack through the compressive layer.

Equation (1) was confirmed by using both Finite Element Analysis (FEA) and an experimental method to directly observe the stable extension of the crack through the compressive layer.

Figure 1 reports the results of the FEA experiments where the elastic modulus of the compressive layer (layer 1) and the thicker tensile layer (layer 2) were different. [2] When $E_1/E_2 = 1$, one obtains the same K expression obtained from the superposition of stresses used to obtain eq. (1). In addition, the FEA showed that when $E_1/E_2 \leq 1$, the compressive layer has a greater effect in hindering the extension of the crack. The reason for this is that less strain energy is stored in the compressive layer when its modulus is small. Thus, compressive layers with a smaller elastic modulus are better for hindering stable crack extension.

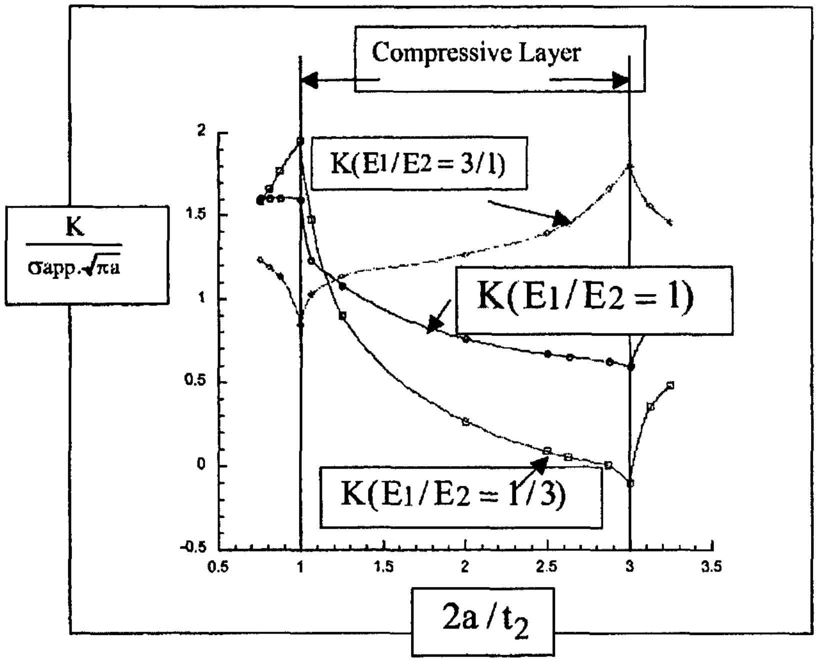


Figure 1 Stress intensity (normalized by applied stress and crack size) vs crack size (normalized by distance between compressive layers). Three functions are for three different elastic modulus ratios, showing that a low elastic modulus for the compressive layer is much better for retarding cracks. [2]

Because the crack extends in a stable manner, it can be directly observed as it sequentially propagates across the compressive layer with increasing applied stress. Figure 2 shows a crack that was replicated, in situ, with acetate tape at different stresses as it extended across the compressive layer at increasing applied stress. [3] The shadowed replica was then observed with an optical microscope using Nomarski interference as shown in Fig. 2. The stress intensity function can be experimentally determined by plotting the crack length vs applied stress. As shown in Fig. 3, these measurements confirm the theoretical K function given by Eq. (1). [3]

Equation (1) shows that an increasing stress is needed to extend the crack through the compressive layers, which implies a new toughening mechanism.

From an engineering standpoint, eq (1) can be rearranged to state a more important concept, i.e., a threshold exists, below which, failure is impossible. The applied stress needed to fully extend the crack through the compressive layer is called the threshold strength, $\sigma_{th.}$. Failure occurs once the crack fully extends through the compressive layer (i.e. when $2a = t_2 + 2t_1$) and $K \geq K_c$.

THE THRESHOLD STRENGTH

By substituting $2a = t_2 + 2t_1$ and $K = K_c$ into eq.(1) and rearranging, one obtains the threshold strength, $\sigma_{th.}$. [1]

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

As shown in eq. 3, the three independent variables that control the threshold strength are the critical stress intensity factor of the compressive layer material, K_c, the magnitude of the compressive stress, σ_c, and the separation distance between the compressive layers t_2 (the thickness of the thicker, tensile layer). Although not implicit in eq (3), the elastic modulus ratio of the two layers is a forth independent variable. [2] The thickness ratio of the two layers (t_1/t_2) represents the volume fraction of the two phases. This ratio is a dependent variable; it also controls the magnitude of the compressive stress.

Observations show that the crack does not always propagate straight across the compressive layers as implied by eq (1) and (2). [1,4] Instead, when either the compressive stress or the thickness of the compressive layer is large, the crack bifurcates as two cracks as it traverses the compressive layer. On the other hand, when the compressive stress and/or compressive layer thickness is small, the crack does not bifurcate. Instead, the crack extends straight across the compressive layer as assumed to develop both eqs. (1) and (3). [4]

Before discussing the probable cause for bifurcation, the effect of the different variables on the threshold strength will be reviewed. Several of the

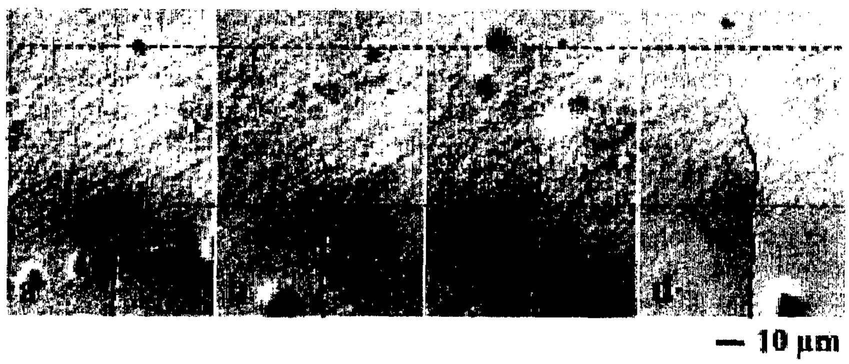


Figure 2 Micrographs of acetate replicas for a crack extending straight though a compressive layer (defined by broken lines) with increasing applied stress. As shown in Fig. 3, the length of crack as a function of stress was used to confirm the K expression given in eq(1). [3]

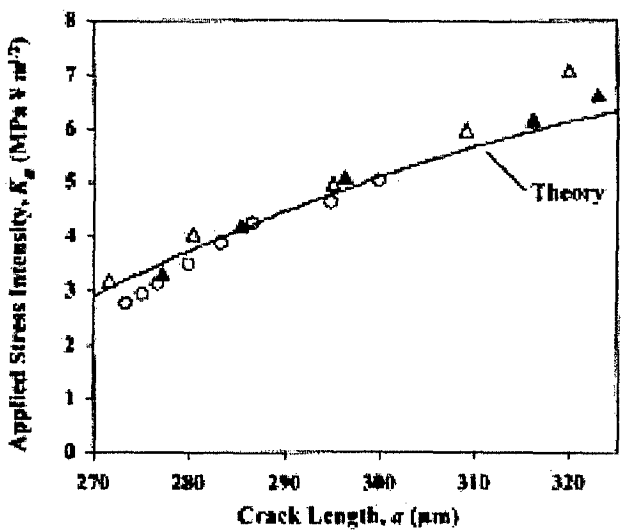


Figure 3 Results of stress intensity determination via observing stable crack extension through a compressive layer via a replica method (see Fig. 1) and an optical method. Solid line represents the theory predicted by eq (1). [3]

variables can be easily changed to determine their effect on the threshold strength. For these studies, laminar composites composed of thicker alumina layers separated by thinner alumina/mullite layers were fabricated. One of the variables, the compressive stress, was systematically changed by changing the volume fraction of the mullite, to reduce thermal expansion, in the thinner alumina/mullite compressive layers for fixed t_1/t_2 ratios. [4] A second variable, the distance between the compressive layers, t_2, was changed by fabricating different laminates with different values of t_1 at a fixed compressive stress (i.e., fixed mullite content) while also fixing the t_1/t_2 ratio. The residual compressive stresses within the thin compressive layers of some representative architectures were measured using a piezospectroscopic method which determines stress by measuring the stress-induced shift in the fluorescence spectra of trace Cr^{3+} impurities within the alumina. [5]

The results of these studies can be generalized in Fig. 4, which shows the effect of the compressive stress on the threshold strength for two different architectures, one with t_1/t_2 = 25 μm/200 μm and the other with t_1/t_2 = 55 μm/550 μm. The results could be divided into two regimes. [4] In the first regime, in which the compressive stress was < 400 MPa, the threshold strengths for the two architectures agreed well with those predicted by eq. 3. For this regime, the crack propagated straight through the compressive layer as shown in Fig. 2. On the other hand, for compressive stresses > 400 MPa, the experimental values of the threshold strength were progressively greater than those predicted by eq. 3. For this second regime, the crack was observed to bifurcate though the compressive layer as shown in Fig. 5.

Figure 5 shows that the crack bifurcates either within or as it enters the compressive layer of laminates with the 55μm/550μm architecture. [4] The figure also shows that the angle between the two cracks changes from 115° to 122° to 135° with increasing compressive stress for compressive layers containing 0.40, 0.55 and 0.70 volume fraction mullite, respectively. It should be noted that in addition to the bifurcated crack, except for the region between the bifurcated cracks a linear crack is also observed along the centerline of the compressive layer. [4]

The reason why a center crack, generally known as an edge crack, forms on the surface of a compressive layer is well known. [6] Figure 6 illustrates a compressive layer from the surface to the interior, and illustrates an edge crack.

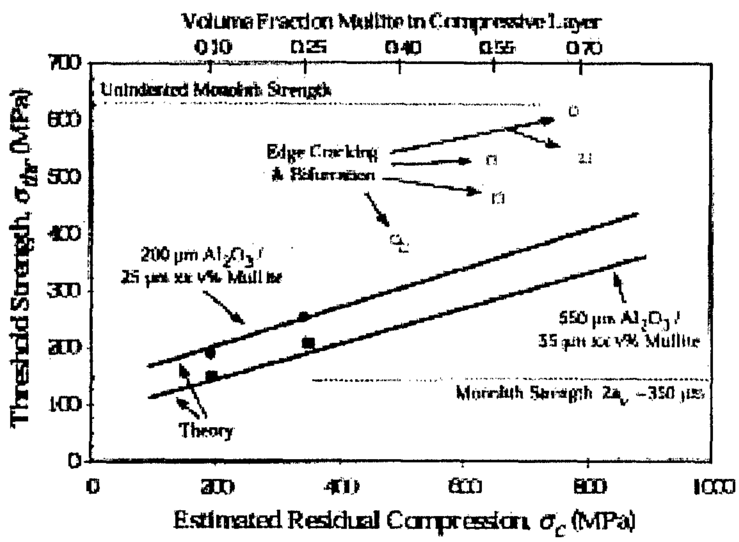


Figure 4. Effect of compressive stress on threshold strength measure for laminates with two different architectures (laminate dimensions). [4]

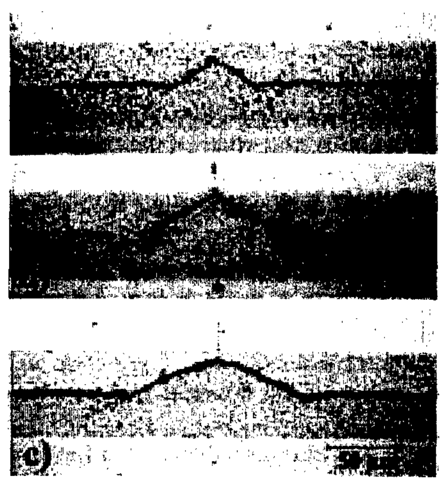
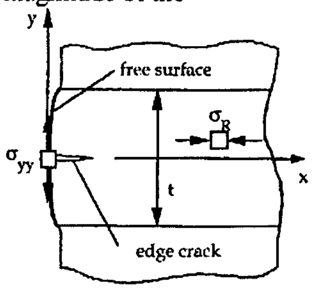


Figure 5 Crack bifurcation in compressive layers with increasing compressive stress (a to c). [4]

Because the layer is not constrained at the surface, a tensile stress, σ_{yy}, is present as shown. σ_{yy} has a maximum value on the surface at the center of the layer, and diminishes to zero at a distance from the surface equal to several layer thickness, where the stress is compressive and biaxial as expected. Because the strain energy associated with the tensile stress is highly localized at the surface, one can show that the condition for crack extension is given by the strain energy release rate function, G. As shown in eq (6), G not only depends on the magnitude of the



$$\sigma_{yy}(x)\Big|_{y=0} = \frac{2}{\pi}\left[\theta - \frac{1}{2}\sin 2\theta\right]\sigma_c \quad (4)$$

$$\tan\theta = \frac{t}{2x} \quad (5)$$

$$G = \frac{0.34\sigma_c^2\left(1-\nu^2\right)}{E}t \quad (6)$$

Figure 6 Tensile stress is present at compressive stress meets the surface. Eq (4) relates the magnitude of the tensile stress to the distance from the surface into the layer. Eq (6) shows that the strain energy release rate is related to the thickness of the compressive layer; for this reason, edge cracks will only occur when the compressive layer is greater than a critical value. The critical value depends on the magnitude of the compressive stress. [6]

compressive stress and the elastic properties of the compressive layer, but also, on the layer thickness. For a given compressive stress, a critical layer thickness exists (t_c), above which, a crack will extend along the centerline to a depth that is proportional the layer thickness. [6]

The bifurcated cracks shown in Fig. 5 were observed after removing the surface by diamond matching to a depth below the penetration depth of the original edge crack. [4] The edge cracking observed in Fig. 5 is an artifact which reformed on the new free surface exposed by grinding, as is evidenced by it's absence between the branches of the bifurcated crack where the tensile surface stresses are relieved.

The threshold strength was also determined as a function of the compressive layer thickness and the distance between the compressive layers with the other variables held constant. These data, along with those shown in Fig. 4 can be summarized with the following statement. When either the compressive stress or layer thickness was small, the crack did not bifurcate and the threshold strength could be predicted with eq. (1); when either the compressive stress or compressive layer thickness was large, the crack bifurcated and the threshold strength was larger than that predicted by eq. (1). [4]

Figure 7 plots the observed crack path relative to the compressive stress and the compressive layer thickness. Closed symbols represent specimens in which neither edge cracking nor bifurcation were observed; open symbols represent specimens in which both edge cracking and bifurcation were observed. The broken lines indicate the predicted critical layer thickness for edge cracking (eq. 6) for values of the dimensionless parameter of a) 0.34 and b) 0.17. [6] The underlying feature is that edge cracking and bifurcation are observed together.

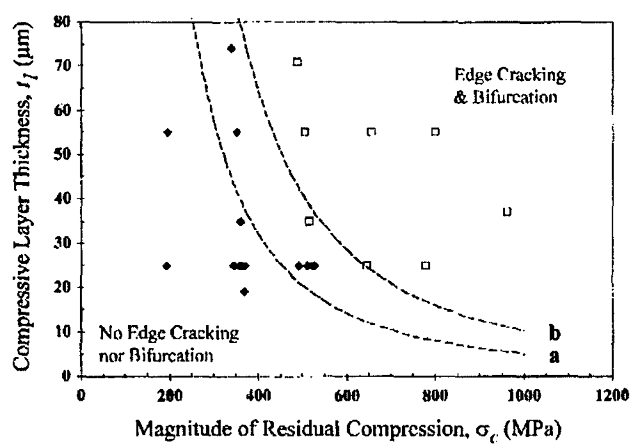


Figure 7. Observed crack path as a function of compressive stress and compressive layer thickness. Composites on right of lines exhibit edge cracking and crack bifurcation, whereas crack propagates straight through the compressive layer for conditions on the left hand side. [4, 6]

FUTURE RESEARCH

Research is currently underway to understand the physical mechanisms underlying bifurcation occurs and to develop a stress intensity function to predict the threshold strength under conditions that produce bifurcation. Methods for the fabrication of three dimensional, crack arresting architectures, as opposed to the laminar (2D) architectures reviewed above, have also been demonstrated. Although this review emphasized one material system (alumina/alumina-mullite), other systems have also been studied including one where the compressive stress is developed by a phase transformation. The embryonic research reviewed above is expected to produce brittle materials with high structural reliability.

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