Structural and Microstructural Design in Brittle Materials

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Abstract
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Keywords
Nondestructive Evaluation

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STRUCTURAL AND MICROSTRUCTURAL DESIGN IN BRITTLE MATERIALS

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ABSTRACT

Structural design with brittle materials requires that the stress level in the component correspond to a material survival probability that exceeds the minimum survival probability permitted in that application. This can be achieved by developing failure models that fully account for the probability of fracture from defects within the material (including considerations of fracture statistics, fracture mechanics and stress analysis) coupled with non-destructive techniques that determine the size of the large extreme of critical defects. Approaches for obtaining the requisite information are described in this paper. The results provide implications for the microstructural design of failure resistant brittle materials by reducing the size of deleterious defects and enhancing the fracture toughness.

INTRODUCTION

The design of structural components from brittle solids, in concept, is straightforward. It simply requires that the stress level in the component should not exceed the strength of the material, at the permissible level of survival probability. The implementation of this concept is, however, very involved. It requires the combination of information derived from the disciplines of fracture statistics, fracture mechanics and flaw detection (or non-destructive evaluation). The description of the general scientific framework for structural design, utilizing these disciplines, and of the future prospects for this class of materials, are the primary intents of the present paper.

Ultimately, design might take the form of a computer simulation of crack growth in real microstructures, coupled with microstructural characterization techniques (such as acoustic scattering). Presently, however, useful progress is being achieved using a partially decoupled approach. The evolution of failure from defects and the crack extension mechanisms are studied separately, and merge where possible. This approach has influenced the structure of the paper, which includes separate considerations of fracture initiating flaws, crack propagation and defect characteristics.

The character of the design problem is illustrated in Fig. 1a, which plots the probability of fracture of a ceramic (measured, say, in flexure) as a function of stress level. It might be construed that for design purposes, it is simply necessary to superimpose the permissible level of allure onto this figure, to obtain a maximum allowable stress in the component: and then to design the component accordingly. The limitations of this approach should be emphasized when it is appreciated that the fracture probability curve can be substantially perturbed by a wide variety of phenomena. These include: the incidence of slow crack growth (Fig. 1b), the occurrence of undetected flaw populations in the inevitable region of extrapolation (Fig. 1c), and effects of stress rate (Fig. 1d). Because of the problems associated with the direct use of statistical design procedures, alternate approaches have been sought, which attempt to effectively truncate the strength distribution at a level above the design stress (Fig. 1e). One such approach, involving the characterization of fracture initiating defects and of the evolution of failure, is emphasized in the present paper. Implications for microstructural design are included, as they emerge from the general scheme.

Fracture in brittle solids usually occurs either by direct extension of a single pre-existent flaw (from the large extreme of the flaw population) or by the coalescence of small flaws. The level of stress needed to activate these flaws relates to the size of the flaw in a manner that depends upon the interactions and the resultant strength, flaw size, probability relations are described in the first part of the paper, for each of the prevalent flaw types: inclusions, voids, surface cracks, microcracks. Immediate implications for microstructural design derive from these descriptions of strength.

The formation of flaws (especially extrinsic flaws such as surface cracks, impact damage, thermal cracks), and their extension susceptibility, depend sensitively upon the fracture toughness of the material. The toughness thereby emerges as a critical structural parameter. The fracture toughness of brittle materials and its dependence on microstructure is discussed in the second part of the paper. Implications for improved microstructural and structural design, based on toughness considerations, are then explored.

The ultimate survival of a brittle structural component at an acceptable survival probability requires the use of a flaw characterization technique in conjunction with a failure model. Such techniques involve the detection and analysis of waves scattered or absorbed in the most versatile and sophisticated mode of flaw characterization involves the use of acoustic waves: either bulk waves or surface waves. The utility of acoustic waves for providing the requisite survival information, including the combination of the measurement and fracture results to derive optimum accept/reject decision schemes, is described in the third part of the paper.
Fig. 1 A schematic illustrating some of the issues that limit the use of a direct statistical approach for structural design. The diagrams relate the fracture probability $p$ to the strength level $S$ and the sample volume $V$.

Finally, some prospects for further advancement of our comprehension of the failure process are discussed.

**FRACUTRE INITIATING DEFECTS**

The flaws that ultimately initiate fracture in brittle solids can be conveniently classified as intrinsic or extrinsic. The intrinsic flaws are introduced during the fabrication and are predominantly inclusions or voids. The extrinsic flaws are stress induced cracks, such as the surface cracks introduced during machining and the microcracks that result from large residual stresses (e.g., due to thermal contraction anisotropy). Each class of defect will be discussed separately.

The only available analyses of fracture from defects that provide a consistent description of effects of defect size, type and shape invoke the existence of preexistent microflaws, activated by the concentrated stress fields around and within the defects. However, the character of these small flaws is not well-defined. It is supposed that the flaws are the small voids (or precipitates) that typically occur at grain boundaries (e.g., at triple points). These flaws are prone to activation at relatively small levels of applied stress because of the large residual stresses that can exist at grain boundaries due to thermal contraction anisotropy. Such flaws located in high energy boundaries would be particularly susceptible to microcrack formation. Direct evidence of this mode of microcracking has not been obtained, however, and the concept must be treated as phenomenological at this juncture.

The statistical character of the microcracking process can be conveniently posed by
commencing with the premise that the microflaws exhibit an extreme value size distribution that leads to the probabilistic relation:  

$$\phi(a) = 1 - \exp\left[-\left(\frac{a}{a_0}\right)^k\right]$$  

(1)

where $\phi(a)$ is the probability of finding a microflaw larger than $a$ on a grain boundary of area $A$, $a_0$ is a scale parameter, $k$ is a shape parameter and $\lambda$ is a normalizing constant. Noting that a flaw will extend under the condition that the stress intensity factor $K$ reaches the critical value for grain boundary fracture, $K_c$, then gives the approximate result:  

$$K_c = \frac{9}{2}^{\frac{1}{2}}\frac{a_0}{\lambda}$$

(2)

where $a$ is the total stress (applied plus residual) normal to the boundary and $\tau$ is the total in-plane shear stress needed to induce crack extension. Substituting $a$ from Eq. (2) into Eq. (1) gives the probability of microcracking as a function of applied stress $(\sigma_a, \tau_m)$ as:  

$$\phi(\sigma_a, \tau_m) = 1 - \exp\left[-\left(\frac{a}{a_0}\right)^k\frac{K_{cr}}{\sigma_0}\int_A \left(\frac{(2-\nu)^2}{\sigma_a^3} + \frac{4\tau^2}{(2-\nu)^2}\right) dA\right]$$  

(3)

It should be noted that, since the residual stresses and the toughness are variables, the microcrack probability associated with a specific boundary is not uniquely related to the applied stress; rather, a distribution of probabilities generally exists. This effect allows the origination of a crack tip microcrack zone (i.e., microcracks do not necessarily initiate first at the most highly stressed boundary contiguous with the crack tip). For many other problems, the probability of crack formation averaged over many grains is more pertinent. For this situation, the average residual stress must be zero, and Eq. (3) reduces to the simple form:  

$$\phi(\sigma_A) = 1 - \exp\left[-\frac{1}{V_0} \int_V \left(\sigma_A/S_0\right)^m dV\right]$$  

(4)

where $\sigma_A$ is the applied stress, $m$ is the shape parameter $(=2k)$, $S_0$ is the scale parameter (which includes $K_c$, $a_0$ and $\nu$ as well as the coefficient that reflects an averaging of the normal and shear stresses over the grain boundaries) and $V$ is the volume of material. A relation similar to Eq. (4) is also generally assumed to describe microcrack extension, except that $S_0$ will have a different significance.

Intrinsic Defects

Voids - The probability of fracture from a void can be ascertained by combining the void stress field with the appropriate statistical relation for extension of microcracks existing in the vicinity of the void. If the microcracks are very much smaller than the void radius, a direct statistical analysis using Eq. (4) suffices.  

For example, when the microcracks predominate at the void surface (Fig. 3), the surface stress field:  

$$\sigma_0/\sigma_A = \frac{3}{2}(1-5v)[(4-5v) + 5\cos 2\theta]$$  

(5)

$$\sigma_0/\sigma_A = \frac{3}{2}(1-5v)[5v\cos 2\theta - 1]$$

(6)

can be combined with Eq. (4), and integrated over the tensile portion of the void surface, to yield:  

$$\varepsilon = -\pi [1+\frac{1}{3}r^3] = 8r^2(\sigma_A/S_0)^m \exp[0.52m-1.4]$$  

(7)

where $r$ is the void radius. A stronger dependence on $r$ emerges for volume distributed microflaws; viz., $\varepsilon = r$, as deduced by Vardar, et al.  

When the microcracks are not small, vis-a-vis the void radius, a stress gradient correction derived from fracture mechanics solutions must be applied. This correction arises because the stress intensity factor for a flaw located in a rapidly varying stress field depends sensitively on the exact flaw location and on its size relative to the gradient. The effect is especially manifest for surface located microcracks, which are subject to the following approximate peak stress intensity factor:  

$$\hat{K} = \frac{2\sigma_a\sqrt{a}}{\sqrt{\pi(1+0.3(0.2a/r))}}$$  

(7)

This relation for $\hat{K}$ can be used to obtain an effective stress $\sigma_{eff}$ that replaces the applied stress in Eq. (16), given by:
the inclusion has a larger toughness than the matrix (an unusual occurrence) fracture initiates within the matrix, usually from microcracks located within (or adjacent to) the interface. The process then resembles the void fracture problem. However, one additional distinction must be introduced. When the bulk modulus of the inclusion exceeds that of the matrix, the tensile stresses (in a direction suitable for continued extension of the crack due to the applied stress) are confined to a relatively small zone near the poles of the inclusion (Fig. 4). The fracture probability can then be anticipated to be relatively small, as exemplified by the high survival probability for WC inclusions in silicon nitride. Alternatively, when the modulus of the inclusion is smaller than that of the matrix, the maximum tensile stresses occur near the equatorial plane. The fracture condition is then comparable to that for a void, modified by a stress coefficient $\lambda$ that depends on the modulus ratio;

$$\lambda = 1 + \frac{2[(\kappa_i/\kappa_m) - 1](1 - 2\nu)}{3(1 - \nu)} \left[ \frac{4\nu + 3\kappa_m}{4\nu + 3\kappa_i} \right]$$

where $\kappa$ is the bulk modulus and $\nu$ is the shear modulus. This case is expected to be an important one in ceramics, because the inclusions are often porous\(^3\) (following high temperature mass transport driven by thermal contraction anisotropy) and hence, of low effective modulus.

Most inclusions typically encountered in brittle matrices are of low toughness, because they are usually the friable product of chemical reaction with the matrix (Fig. 6). If such an inclusion also has a relatively high modulus (approaching that of the matrix), the inclusion can fracture sub-critically to create a crack of dimensions comparable to the cross-section of the inclusion. The ultimate fracture strength is then dictated by the usual fracture mechanics relation for an internal crack\(^1\)

$$\sigma = z(a/c)K^\text{eff}_{\text{c}}F(\kappa_i/\kappa_m)$$

where $a$ and $c$ are the dimensions of the crack, $K^\text{eff}_{\text{c}}$ is the effective toughness of the matrix phase neighboring the defect and $z$ is an exponent ($=0.5$) that depends on the modulus ratio. This type of defect is the most deleterious of the high expansion defects (Fig. 4). Defects in this category are exemplified by Si inclusions in Si$_3$N$_4$ (Fig. 5). When the modulus of the defect becomes very small, because of extensive porosity (Fig. 6), the stresses do not attain a sufficient level to induce defect fracture (despite their friability); the situation is then identical to that of low modulus, high toughness inclusions. However, an intermediate condition is also possible; wherein fracture can initiate within the defect and then propagate directly into the matrix to cause complete failure. In this situation, fracture is dictated by the probability of activating microcracks within the inclusion, and the fracture probability becomes;

$$\phi = 1 - \exp \left[ -V_i \left( \frac{\lambda a + \sigma a}{f_0} \right)^\gamma \right]$$
Fig. 4 A schematic indicating the various cracking responses that can occur in the presence of inclusions.

inclusions. This is a plausible situation considering the potential for a transition, with decrease in size, from inclusion initiated fracture (volume dependent) to matrix fracture (area dependent).

Fig. 5 Strength, size relations for various fracture initiating defects in silicon nitride.

where $p_0$ is the scale parameter, $\gamma$ is the shape parameter, and $V_i$ is the inclusion volume. Fracture results obtained for iron silicide inclusions in silicon nitride satisfy a joint fracture relation involving a combination of the critical defect fracture model (Eq. 11) and the matrix fracture model pertinent to low modulus inclusions. This is a plausible situation considering the potential for a transition, with decrease in size, from inclusion initiated fracture (volume dependent) to matrix fracture (area dependent).

Fig. 6 A scanning electron micrograph of an iron silicide inclusion in silicon nitride.
Extrinsic Defects

Extrinsic defects are usually cracks produced by large transient or localized stress states. The most common sources of extrinsic defects are surface cracks produced by machining,\textsuperscript{14} impact,\textsuperscript{15} or thermal shock. The machining induced cracks are the most prevalent (and comparable in character to the cracks introduced by projectile impact).\textsuperscript{15} The evolution of the cracks, and their resultant influence on strength, is analogous to the cracking that occurs during indentation\textsuperscript{16} (Fig. 7). The ultimate dimensions of the cracks are dictated by the residual indentation field, as controlled by the hardness, $H$, toughness, $K_c$, and modulus, $E$, of the material. A specific relation recently derived for the strength controlling radial cracks is:\textsuperscript{16}

$$a^{3/2} = 2.10^{-2}(\cot^{2/3} \psi)(E/H)^{2/3}K_c^{-1}P_N$$  \hspace{1cm} (12)

where $\psi$ is the included angle of the grinding particle and $P_N$ is the normal force applied to the particle. The term $E/H$ arises because fracture is a residual stress dominated process.\textsuperscript{17} The radial cracks are usually semi-circular, because of the symmetry of the residual field.

The extension of the surface cracks introduced by grinding is explicable using standard fracture mechanics relations for mode I,\textsuperscript{13}

$$K = F(\psi) \frac{2}{\sqrt{\pi}} \sigma \sqrt{a}$$ \hspace{1cm} (13)

where $F(\psi)$ is the function, plotted in Fig. 8, that describes the variation in $K$ around the crack periphery. Extension to the mixed mode fracture of inclined cracks appears to be adequately described, over an appreciable angular range, by the simple coplanar strain energy release rate criterion.\textsuperscript{18} However, the effective stress that produces crack extension can include a significant residual component; particularly in coarse grinding situations, where the plastic zone is not removed by subsequent fine grinding. Consequently, the applied stress at fracture can exhibit both
systematic and random variations from that anticipated by direct application of Eq. (13) (with the peak stress intensity factor $K$ equated to $K_c$). Available test results suggest a probability of fracture given by the normal distribution:

$$\phi(a \mid \sigma_p) = \frac{1}{\sqrt{2\pi} \sigma_p} \int_{-\infty}^{\infty} \exp \left[ -\frac{(a - \sigma_p)^2}{2\sigma_p^2} \right] \, da$$  \hspace{1cm} (14)

where $\sigma_p$ is the predicted strength obtained from Eq. (13):

$$\sigma_p = \frac{K_c}{\hat{F}(\hat{a})} \frac{\sqrt{\pi}}{2a}$$  \hspace{1cm} (15)

$\hat{F}(\hat{a})$ corresponds to the value at $\hat{a}$, $\hat{v}$ is the variance in $\sigma_p$, $\hat{a}$ is a systematic error coefficient and $\alpha$ is a parameter related to the mean strength.

**Microstructural Design**

Several direct implications for microstructural design emerge from the fracture models. A reduction in the size of the large extreme of voids and inclusions is the most obvious. Inclusions derive from several different sources, usually in connection with the powder preparation and compaction stages of fabrication. The implementation of more stringent powder handling controls (both chemical and size distribution) provides direct benefits. Voids are more difficult to minimize. Large voids form due to coarsening phenomena (driven by surface diffusion or evaporation/condensation) that can occur concurrent with densification. Their existence can be minimized by avoiding domination of the mass transport by surface diffusion control or evaporation condensation control: as ascertained by reference to initial stage sintering maps for the material and the sintering environment.20

The formation of extrinsic cracks is determined by those microstructural parameters that influence the toughness and hardness, as indicated by Eq. (12). The relevant issues are described in the following section.

**THE FRACTURE TOUGHNESS**

Inspection of the above relations for the fracture probabilities from both intrinsic and extrinsic defects indicates that the fracture toughness of the matrix is usually a strength controlling parameter. It is essential, therefore, that the pertinent toughness be understood and, where possible, optimized. For this purpose, it is convenient to introduce the concept of microtoughness and its relation to the macrotoughness of a material. This concept is necessary because the ultimate fracture of brittle materials (especially those with coarse microstructures) can occur at crack lengths which are sufficiently small, vis-a-vis the important microstructural dimensions, that the macrotoughness of the material is not directly pertinent to the fracture problem.21,22,23

The variation of toughness with crack length is still rather ill-defined, because of the difficulties associated both with the conduct of critical experiments and the development of theoretical solutions. The only unequivocal statement currently permissible is that the effective toughness $K_{ef}$ varies from its single crystal value $K_s$ at $a/d$ (where $d$ is the grain diameter) to the macroscopic value at $a/\hat{a}$, where $\hat{a}$ is the approximate range 5-100 (depending upon the nature of the crack/microstructure interaction), as depicted in Fig. 9. The significance of this variation is illustrated by considering that the effective toughness varies as:

$$K_{ef} = K_s + \frac{K_c - K_s}{1 + \alpha d_0/\hat{a}}$$  \hspace{1cm} (16)

where $\omega$ is a constant ($\sim 5$). The crack extension stress for a circular crack then becomes:

$$\sigma = \left( \frac{\sqrt{\pi}}{2} \right) a^{-1/2} \left( \frac{K_c - K_s}{K_s + \frac{K_c - K_s}{1 + \alpha d_0/\hat{a}}} \right)$$  \hspace{1cm} (17)

which can be rearranged into dimensionless groups to give:

$$\left( \frac{2\sqrt{\omega}}{\pi} \right) \left( \frac{\sqrt{d}}{K_s} \right) = \frac{1 + \xi (1 + \epsilon)}{\xi^{1/2} (1 + \epsilon)}$$  \hspace{1cm} (18)

where $\xi = a/\hat{a}$ and $\epsilon = (K_c - K_s)/K_s$. Typical stress variations are plotted in Fig. 10a. For $\epsilon > 0$, the curve shows a maximum. This maximum will control the fracture stress $\sigma_f$ if the initial crack is in a range that yields an initial crack extension stress less than the maximum, as depicted in Fig. 10b. Differentiating Eq. (18), and setting to zero to obtain the maximum, yields a critical crack length at fracture ($\epsilon > 0$), given by:

$$\xi_c = \frac{[\epsilon - 2\xi (1 - \phi/(1 + \epsilon))^{1/2}]}{2[1 + \epsilon]}$$  \hspace{1cm} (19)

which reduces for large $\epsilon$ to $\xi_c - 1$, i.e., $a-d\hat{a}$. The fracture stress is thus given (for large $\epsilon$) by:

$$\sigma_f = \frac{K_c}{4\sqrt{\hat{a}} a}$$  \hspace{1cm} (20)

![Fig. 9 A typical variation in toughness with relative crack length for small cracks.](image-url)
The variation in the normalized crack extension stress with relation given in Fig. 9.

A schematic indicating the growth of cracks for large $K - K_c$, up to the fracture stress $\sigma_f$; A to B occurs unstably while B to C corresponds to stable growth.

The interesting result thus emerges that the strength is inversely proportional to $d^{1/2}$ (regardless of the function selected for $K_{eff}$), a behavior typical of coarse grained ceramics, for which the pre-existent cracks are in the microcrack range. Catastrophic crack extension is thus often a nebulous one for coarse grained materials; and the conventional use of toughness should be confined to materials with fine microstructures (for which strength does not relate systematically to grain size).

The development of theoretical relations between the intrinsic toughness of brittle materials, microstructure and crack length is presently at a very elementary level, primarily because crack extension is a complex three-dimensional problem. Reliance on toughness levels for correlating strength results with defect dimensions (through the fracture models) must presently be placed on measured values, obtained on single crystals and polycrystals. However, a more advanced comprehension of extrinsic toughening has recently emerged. This comprehension provides useful insights pertinent to microstructural design, and will be presented in some detail.

**Microstructural Design**

Two important mechanisms of toughening have been subject to recent analysis: toughening induced by martensitic transformations and by microcracking. Both processes involve a zone of dilation and induced around the crack tip, which results in a reduced crack tip tension and hence, an increase in toughness. Also, it is self-evident that the closer the system is to transformation (or microcracking) in the absence of stress, the larger will be the process zone size and hence, the larger the toughness. However, in detail, the processes are quite different.

**Martensitic Toughening**

A toughening can be induced in brittle materials if particles are introduced that undergo a diffusionless transformation, induced by a stress field of combined shear and hydrostatic tension. Typical examples are $\text{ZrO}_2$, $\text{HfO}_2$, $\text{BaTiO}_3$ and BN. A prerequisite for the toughening is that the particles be incorporated in a relatively stiff elastic matrix. This constrains the transformation strain and thus, permits the thermally induced transformation to be considerably suppressed below $K_c$. Then, activation by an applied stress becomes viable.

Estimation of the conditions required for toughening can be obtained by examining the free energy of the total system, before and after transformation. Toughening will be permissible if, as noted above, an applied hydrostatic tension and shear are needed to yield an incremental energy decrease due to transformation. The total mechanical energy change of the system following transformation, under applied stress $\sigma$ (dilational) and $\tau$ (deviatoric) is:

$$\Delta U = -\left[ e^{T}_p \sigma + \tau p^{T}/2 \right]$$

while $e^T_p$ and $\tau$ are the dilational and deviatoric transformation strains; $p^T$, $\tau$ are the stresses within the inclusion after transformation, given (for a spherical particle) by:

$$p^T = -\left(1+\nu_1\right)/2E_m \left(1-2\nu_1\right)p/E_p$$

$$\tau = \left(1+\nu_1\right)/E_p + \left(1+\nu_1\right)/E_m \left(4-5\nu_1\right)/7 \left(1-5\nu_1\right)$$

By considering the total free energy of the system following transformation,

$$\Delta F = \Delta U + \Delta S - \Delta F_0$$

where $\Delta F_0$ is the difference in chemical free energy, per unit volume, between the two crystal structures and $\Delta S$ is the difference in surface energy, the critical transformation stresses $\sigma_{cT}$ and $\tau$ can be deduced by permitting $\Delta F$ to
incrementally decrease; this assumes that the thermodynamic driving force is a sufficient condition for transformation (i.e., appropriate nuclei pre-exist within the particles). Neglecting the difference in surface energy, since this is presumed to be small (at least, for incoherent particles), the critical applied stress for transformation, under uniaxial tension $\sigma_A / 3; \sigma_A^t \approx \sigma_A / 2$ becomes

$$\sigma_A \left[ \frac{2 + 2 \epsilon}{E_p} \right] = \frac{1}{\beta (1 + \epsilon)^2} \left( \epsilon^2 / 2 \right) \left( 7 - \frac{5 \epsilon}{1 + \epsilon} \right)$$

$$+ \left( \frac{1}{(1 + \epsilon)^2} \right) \left( 7 - \frac{5 \epsilon}{1 + \epsilon} \right) \left( 4 - \frac{5 \epsilon}{1 + \epsilon} \right)$$

$$- \Delta E_p / \left( E_p / \epsilon^4 \right)$$

(24)

where $\epsilon = \epsilon^t / E_p$ and $\beta = E_p / E_m$. As a first approximation, conditions which yield a positive $\sigma_A$ are likely to result in transformation toughening.

This approach can be tentatively extended to examine transformation zones around crack tips, by inserting the crack tip field relations for $\sigma_A$ and $\sigma_A^t$ into Eq. (21). This approach can only be regarded as highly approximate because significant effects are neglected; namely, stress gradient effects, particle interactions, crack surface relaxations and reverse transformations. Nevertheless, some useful insights are provided. The relation for the transformation zone radius $r_c$ at small particle volume fractions (for $\nu_p = 0.2$) is:

$$r_c \left( \frac{E_p}{E_m} \right)^2 = 4 \epsilon$$

(25)

$$\left[ \frac{\cos(\theta/2)/\left( 1 + 1.25 \sin(\theta/2) \cos(3\theta/2) \right) / \epsilon^4}{\beta (1 + \epsilon^2)^2 - 3.5 \epsilon + 0.34 \epsilon^2} \right]^2$$

Some typical zones are plotted in Fig. 11. The transformation zone radius can subsequently be used to estimate the toughening increment:

$$\Gamma_T = \frac{\epsilon}{1 + \epsilon^2} \frac{\left[ 0.34 \epsilon^2 - \Delta E_p / \left( E_p / \epsilon^4 \right) \right]}{\beta (1 + \epsilon^2)^2 - 3.5 \epsilon + 0.34 \epsilon^2}$$

(26)

where $V_f$ is the volume fraction of particles. The central role of the chemical free energy in the resulting toughening signifies strong effects of temperature and of chemical composition: the toughness decreasing as the temperature increases (Fig. 12). The evident merits of an intrinsically high toughness host also emerge.

Further developments which take specific account of interaction effects and of the particle orientation can be envisaged using computer techniques, analogous to those recently employed for microcracking (see following section). Research on the influence of chemical composition on $\Delta E_p$ and hence, on $\Gamma_T$ is also needed; probably involving compositional probes within particles, using a scanning transmission electron microscope.

**Microcracking**

Stable microcracking is presumed to occur in materials that contain stresses (e.g., due to thermal contraction mismatch or transformation), which also have a sufficiently coarse microstructure.

The induction of stable microcracks within a crack tip process zone (Fig. 13) can be described through probabilistic microcrack formulations (such as Eq. 3) superimposed on the crack tip field equations. However, computer techniques are needed to take proper account of the strong interaction of the microcracks with the primary crack. Such an approach has recently been reported by Hoagland and Embury using a Green's function devised by Hirth et al. One important result of this analysis is the realization that
Fig. 13 A microcrack process zone induced in materials with large local residual stresses, indicating that microcrack densities and the resultant crack tip stress field.

Conceptually it is useful to consider the microcrack process zone as a dilatant, compliant zone that inevitably must yield a reduction of the tensile stress at the primary crack tip (Fig. 13). The effect is typified by solutions for a crack entering a low modulus inclusion. One microcrack toughening situation that can be addressed more positively is the toughening in the presence of second phase particles. A typical condition would be the incorporation of small particles that exhibit a larger thermal contraction mismatch strain (e = ΔαΔT) and γ_{int} is the fracture surface energy for particle microfracture. Note that, in the present approximation, a particle which contracts away from the matrix is necessary, in order that the strain energy of the system can be increased by the crack tip (tensile) stress field. Microfracture will then proceed, according to the present criteria, when ΔU > ΔS; while toughening can occur when p^A in Eq. (27) is positive (the particles must be intact prior to stress application). Inserting the hydrostatic component of the crack tip stress field.

\[ p^A = \frac{2K(1+\nu)\cos\theta/2}{3\pi\nu^2} \]

the transformation zone size and shape r_c(θ) can be approximately deduced. For the condition, \( \nu = 0.2 \), substitution of Eq. (28) into Eq. (27) gives;

\[ r_c = \frac{1}{2\pi} \left( \frac{-K}{E_m eT} \right)^2 \frac{\cos^2(\theta/2)(1+\nu)^2}{(3.6\nu(1+\nu)-\nu)^2} \]

where α = γ_{int}/E_m eT. The zone size can be converted into a toughening increment from the residual free energy in the transformation zone.

\[ \Gamma_T = \alpha \left[ (\Delta S - \Delta U) r_c(\theta) \right] \frac{W_f}{(4/3)\pi r_p^3} \]

where \( W_f \) is the volume fraction of particles, \( \Delta S \) is the strain energy in the absence of an applied stress and \( \theta = \pi/2 \). Combining Eqs. (27), (29) and (30), the toughening increment becomes;

\[ \Gamma_T = \alpha \left[ (\Delta S - \Delta U) r_c(\theta) \right] \frac{W_f}{(4/3)\pi r_p^3} \]

The maximum toughening clearly occurs when \( 3V_f(1+\nu) \). The particle size and the mismatch strain exert an influence through the α term in the denominator, in the sense that \( \Gamma_T \) increases as the particle size or the mismatch strain increase, provided that \( r_c \) is below the critical value for spontaneous (stress free) microcracking. These results provide useful perspectives for designing optimally tough ceramic systems, based on controlled microcracking.

**NON-DESTRUCTIVE DEFECT CHARACTERIZATION**

**Accept/Reject Criteria**

Accept/reject decisions based on a non-destructive measurement of scattering from a defect must recognize the probabilistic character of the problem. At least three probabilities enter the analysis: the failure probability.
given the defect dimensions (discussed above) \( \phi(a^0 | a) da \); the joint probability of identifying the defect type and of estimating its size. \( \phi(a, es | a) da es \); the a priori distribution of defect sizes, \( \phi(a) da \). These probabilities are combined and integrated to various inspection levels, \( a^* \), to obtain two interrelated probabilities: the false-accept probability \( \phi_A \) and the false-reject probability, \( \phi_R \): (32)

\[
\phi_A = \int_0^{a^*} \int_0^{es} \int_0^{es} \phi(a^0 | a) \phi(a) da \ (32)
\]

\[
\phi_R = \int_0^{a^*} \int_0^{es} \int_0^{es} \phi(a, es | a) \phi(a) da es
\]

where \( a^0 \) is the level of the applied tension in the volume element containing the defect. The inspection level \( a^* \) refers to the estimated defect dimension(s) selected for the rejection or acceptance of the component, e.g., all components with an estimated maximum dimension less than \( a^* \) are accepted and all components with an estimated dimension greater than \( a^* \) are rejected. The false-accept probability \( \phi_A \) is thus the probability that components accepted in accord with the specified inspection level will contain defects more severe than indicated by the estimate, and will actually fail in service (i.e., related to the failure probability). This probability decreases, of course, as the inspection level decreases (Fig. 14b). The false-reject probability \( \phi_R \) is the (related) probability that rejected components would, in fact, have performed satisfactorily in service, because the defect severity has been overestimated by the selected inspection level. This probability increases as \( a^* \) decreases (Fig. 14b). However, it is crucial to recognize that these probabilities are interrelated, i.e., they merely represent different ranges of integration of the same combination of probability functions (Eq. 32). This interdependence is exemplified in Fig. 14a, which is a typical plot relating the false-accept and false-reject probabilities: once one of these probabilities has been selected, the other probability, as well as the associated inspection level, are necessarily defined. It is now apparent from Fig. 14a that the inspection technique, or combination of techniques, that would be preferred is that which yields a curve as close as possible to the probability axes. For example, technique B is preferred over technique A, because the rejection of satisfactory components required to satisfy the failure probability requirements is much lower. Such curves thus represent a quantitative method for characterizing the failure prediction capabilities of various inspection techniques, for a given material and service condition.

Scrutiny of the available inspection methods pertinent to ceramics indicates that acoustic methods are preferred, because acoustic waves are appreciably scattered by all of the critical defect types encountered in structural ceramics. The most promising measurement algorithms and their future potential are thus briefly reviewed.
Acoustic Measurement Algorithms

Surface Waves - The most directly successful method is the use of surface acoustic waves to predict failure from surface cracks; in particular, the use of long wavelength, $\lambda >> a$, surface waves. In the long wavelength limit, the scattering of an acoustic wave (stress wave) by a crack is closely analogous to the interaction of the crack with an applied stress field. In particular, both the scattering coefficient $S$ and the strain energy release rate $J$ are related to the crack surface integral,

$$J = \int_{A} \sigma_{ij} \Delta u_{j}^{i} dA_{s}$$

where $\sigma_{ij}$ is the stress across the crack plane in the absence of the crack, $\Delta u_{j}^{i}$ is the displacement of the crack surfaces and $A_{s}$ is the crack surface area. Hence, it is straightforward to demonstrate that the scattering coefficient is directly related to the crack extension stress $\sigma_{C}$ by:

$$K_{C} = 2 \left[ \frac{(1-v)\lambda^{2}S\omega^{2}}{\pi^{2}f_{m}^{2}} \right]^{1/6}$$

(33)

where $\omega$ is the beam width, $n$ is the transducer efficiency and $f_{m} = 0.4$. This result is strictly correct when both the acoustic wave and the applied stress are normal to the crack plane, as exemplified by recent results summarized in Table I. However, since the coplanar $J$ criterion also appears to afford a reasonably satisfactory description of surface crack extension in ceramics, at least over the angular range of interest, the approach appears to be of general applicability. Surface waves also have the advantage that they propagate over curved surfaces, so that complex shapes can be readily probed.

Table I
Comparison of Measured Surface Crack Sizes With Those Predicted Using Long Wavelength Surface Acoustic Waves

<table>
<thead>
<tr>
<th>Sample</th>
<th>$\sigma_{F}$ Actual MPa</th>
<th>Acoustic a $\mu$m</th>
<th>$\sigma_{F}$ Acoustic MPa</th>
<th>$%$</th>
</tr>
</thead>
<tbody>
<tr>
<td>5kg: 1</td>
<td>338.45</td>
<td>56</td>
<td>350</td>
<td>3.3</td>
</tr>
<tr>
<td>2</td>
<td>365</td>
<td>56</td>
<td>367</td>
<td>0.54</td>
</tr>
<tr>
<td>10kg: 2</td>
<td>298.5</td>
<td>67</td>
<td>320</td>
<td>6.72</td>
</tr>
<tr>
<td>275.4</td>
<td>66</td>
<td>322</td>
<td>14.6</td>
<td></td>
</tr>
<tr>
<td>20kg: 1</td>
<td>159.22</td>
<td>274</td>
<td>158.4</td>
<td>0.52</td>
</tr>
<tr>
<td>2</td>
<td>179.13</td>
<td>262</td>
<td>159.7</td>
<td>12.17</td>
</tr>
<tr>
<td>3</td>
<td>189</td>
<td>255</td>
<td>164.2</td>
<td>15.1</td>
</tr>
</tbody>
</table>

Bulk Waves - The characterization of bulk defects is more complex. Information over a wide range of frequencies appears to be needed to obtain a highly probable defect type classification and hence, a size estimation. Appropriate techniques are available including: the scanning laser acoustic microscope $^{20}$ 200 MHz ZnO transducers $^{21}$ and conventional (2-50 MHz) transducers. Rapid scanning methods for defect location have also been developed. The most critical issue, therefore, concerns the appropriate choice of algorithms to obtain the most reliable defect characterization. A typical set of algorithms and their interaction are illustrated in Table II, using low and high frequency information as well as acoustic microscopy. This set has not yet been fully evaluated, so many redundancies may exist. Four algorithms are employed in this scheme: (i) long wave length scattering, (ii) intermediate wavelength Born approximation, (iii) high frequency scattering, and (iv) cross sectional information from acoustic microscopy. The impulse response functions (Fig. 15) are firstly used to determine whether the defect is a void or an inclusion; the void has an impulse response function (Fig. 15a) characteristic of the transducer, while inclusions have more complex functions (Fig. 15b). Thereafter, voids can be analyzed straightforwardly, using a variety of algorithms. For example, a long wavelength algorithm similar to that described for surface cracks may be employed. In the long wavelength limit, the scattered amplitude is related to the void volume $V$ by:

$$A = \frac{V \omega}{(4\pi)^{2/3}} \left[ 1 + \frac{1}{2\nu} + \frac{10(1-2\nu)}{7-5\nu} \right]^{2}$$

(34)

where $\omega$ is the frequency and $c$ is the elastic wave speed in the host. Inclusions are more difficult to analyze; the combined use of several algorithms is almost certainly required. For nearly spherical inclusions, the interpretation is relatively straightforward. For example, a combination of the long wavelength algorithm (which contains coupled volume and type information) and the Born approximation (which provides an independent estimate of the distance from the geometric center to the back face of the inclusion) can yield the requisite size and type information. A typical result, obtained for a 100 $\mu$m radius Si inclusion in Si, is illustrated in Fig. 16; wherein the joint probability of the defect type and size is plotted as a function of the estimated size. In order to obtain this result, six possible inclusion types were permitted to exist within the material (selected on the basis of detailed failure analyses conducted on this material). Alternatively, high frequency measurements displayed in the frequency domain would provide close estimates of the defect size and type.

FUTURE PROSPECTS

It is hoped that this paper conveys the impression that a positive start has been made in establishing the scientific framework for microstructural design with brittle materials. Certain rewarding research directions have emerged and several exciting near term, and more remote, prospects seem viable.

Further studies aimed at characterizing models of fracture from defects are very pertinent. The incisive combination of inputs from mechanics, materials and statistics demonstrated on the limited set of problems addressed thus far should provide some direction and scope for continued activity. Important defects not yet considered include: voids, crack arrays, sub-surface inclusions, surface crack arrays. Progress toward the comprehension of fracture from these defects could utilize existing (or marginally extended)
Fig. 15a Impulse response functions for defects in ceramics, a void in Si₃N₄.

Fig. 15b Impulse functions for defects in ceramics, a WC inclusion in Si₃N₄.
stress analyses coupled with advanced statistical methods and fracture mechanics solutions.

A very preliminary comprehension of toughening mechanisms has evolved from simplified thermodynamic analyses and computer simulations. Considerable advances could be anticipated through the coupled use of analytic and computer methods. These would include stress (or strain) based transformation or microcracking criteria, statistically distributed in accord with distribution functions inferred from critical experiments (e.g., acoustic emission amplitude distributions). Monte Carlo methods could then be used to study the evolution of process zones, with interaction effects accounted for using image stress solutions.

More immediate advances can be anticipated in ultrasonic flaw characterization. A comprehensive set of inversion algorithms already exist, and initial results imply that good estimates of defect size and type are possible, using combinations of these algorithms. Future prospects for devising effective accept/reject schemes pertinent to ceramics are thus very exciting.

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REFERENCES

28. A.G. Evans, to be published.
32. F.F. Lange, work performed at Rockwell International Science Center, to be published.
40. L.W. Kessler, work performed at Sonoscan.
42. W. Kohn and J.R. Rice, J. Appl. Phys., to be published.
43. J.H. Rosi and J.A. Krumhansl, Materials Science Center Report 2846, Cornell Univ.
SUMMARY DISCUSSION
(A. G. Evans)

Tom DerKacs (TRW): I noticed on the slide you had strength versus defect size you didn't have any defect smaller than about 100 microns. I also wondered what temperature that data was taken at. Was that room temperature data? And if so, what happens?

Tony Evans: Good question. It turns out that to achieve a strength of about 50,000 psi which is just about the limit of any design stresses for ceramic turbines, 100 micron defects is as small as you can get. 100 microns is about right. But those data were at room temperature. What happens when you take equivalent data at high temperature is that the silicon inclusion becomes less deleterious. Everything else more or less gets in the same category. But the silicon inclusion becomes less deleterious, it becomes more pliable and, therefore, less brittle. So, silicon turns out to be more like a void in really high temperatures. About 1,000 degrees centigrade. So, depending upon where the components see the stresses, because if it's a thermal shock then you need the results pertinent to about 900 degrees centigrade. And those results are incorporated. The (inaudible) rotations in the turbine blade, high temperature is also more appropriate.

John Schuldies (Airesearch): Just a general comment from those of us who are out in the industry and who have to use these components. I think perhaps it wouldn't be unrealistic to say in the past we weren't encumbered by too much knowledge, so we went ahead and used some of these things. And in some cases successfully and in other cases not, as we are all aware. I think there is maybe one point that should be made, that you have to look at the stress distribution in the particular component and its geometry. And it turns out in most cases the area of real concern is at or near the surface. And while we may be able to detect very, very small defects in the subsurface, we still got the near surface resolution problem to tackle. And if there was an area that those of us would like to see work being done in, it would be in tackling the near surface resolution problem and defect detection. Obviously, in a size range you have indicated now, that's critical for the bigger-size defects.

Tony Evans: You're right about the perturbation of the acoustic wave on the front face of the component. And therefore, the defect gets obscured by that front face.

John Schuldies: That's why we are pursuing things in acoustic microscopy and photoacoustic spectroscopy. We are interested in learning of the work at Stanford now that detects those things because when you look at the stress distribution in a component and you superimpose thermal and vibration and all that, high stress gradients are at the surface of the part.

Tony Evans: The surface or subsurface. That's right.

Paul Holler: I was wondering on one of your first slides showing two gaussian distributions for being satisfactory or not, and the abscissa was a defect size. So, I assume you have continuous distribution of your defect size versus more porous type. Yes? How do you get from these two well-separated gaussian probabilities for satisfactory or nonsatisfactory?

Tony Evans: They weren't gaussian. They were somewhat schematic.

Paul Holler: They were separated. It doesn't matter, they were --

Tony Evans: I agree with that. This is the slide he is referring to. He is wondering about why there is a separation. The separation comes from the measurement process. It's distinguishing large defects from small ones. And the data relates that distinction. The wider apart those two (inaudible) become. In fact, to get this, one has to make an assumption for a measurement of the distribution of existing defects. And in this case we took a Weibull distribution of defect size (inaudible) exponential function for the defect size distribution and used that information to compute --

Paul Holler: Still a uniform distribution of the defect?

Tony Evans: Still a uniform distribution of the defect.
Paul Holler: How do you get a uniform distribution of defects to this very well separated distribution for the probability to be satisfactory or not satisfactory?

Tony Evans: Because of the measurement. The measurement distinguishes the very large extreme of the distribution from the intermediate extreme, and the measurement allows those curves to separate out. If there is no measurement, you're right, they all come together in one curve.

John Richardson (Science Center): It also depends on the nature of the failure process. The failure process has a great deal of randomness, so they won't separate.

Tony Evans: What John is saying is if it's an extremely random process. In other words, if you have a defect of size $A$ and a fracture strength associated with that size so broad, then those would also be separated by a very narrow amount. But separation for a given size is not that broad and therefore, that allows you to make those curves.

Paul Holler: I still have not understood, but I don't want to hold you up.

Tony Evans: One more question. I'm afraid we're going to have to get the talks under way......Let's get back to the question that John made about surface versus volume. If you were to use surface wave technique to look at subsurface inclusions, then you concentrate on the distributions that are in the near surface which is sampled by the surface wave. If you look using a bulk wave method, then you use distribution which is pertinent to the bulk wave. It may be the same or it may be different. Anyway, I think we have to conclude. Many answers to these questions are better approached in the actual talks as they emerge this morning.

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