# TOUGHENING OF CERAMICS

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## ABSTRACT

This review highlights major advances that have occured in the last decade in the field of toughening of ceramics. The major advance both technologically and theoretically has been in the area of zirconia additions to ceramics or zirconia toughened ceramics (ZTC), There are a number of toughening mechanisms involved in such ceramics. For convenience five distinct mechanisms of toughening ceramics are identified; 1) crack deflection, 2) crack bowing, 3) fibre reinforcement, 4) microcracking and 5) transformation toughening. Experimental evidence and limited theoretical developments in these areas are presented.

## INTRODUCTION

The major drawback of conventional ceramics, when viewed as structural materials, is their low fracture toughness. In other respects, they possess many desirable properties: high hardness, chemical inertness, thermal stability and creep resistance. Consequently, there have been many attempts to improve the toughness of ceramics. This paper summarizes advances in this field since the review by Evans, Porter and Heuer (1977), with special emphasis on zirconia-toughened ceramics (ZTC).

A number of toughening mechanisms can operate simultaneously in these ceramics and there has been some debate as to which of these makes the most substantial contribution to the increase in toughness. Experimentally, it can be difficult to distinguish between the separate contributions when more than one toughening mechanism operates. There has also been some debate on the proper definition of the crack extension force. Consequently crack extension criteria and the concept of R-curve in ceramics will be presented first(section 2). This will be followed by a brief outline of zirconia toughened ceramics (section 3) and finally a more detailed account of theoretical and experimental developments of the various toughening mechanisms operative in ceramics will be presented (section 4).

## CRACK EXTENSION CRITERIA AND R-CURVE BEHAVIOUR

The crack extension criterion in ceramics has usually been considered as a classic examples of the Griffith energy balance. However recently fundamental questions concerning the concept of $K_{1c}$ or $G_{1c}$ have been raised particularly for the toughened ceramic materials. This is particularly the situation for materials exhibiting microcracking, transformation or fibre reinforced toughening where there is definite experimental evidence and theoretical arguments that indicate R-curve behaviour, or rising crack resistance with crack extension, occurs in ceramics. The common feature of materials and mechanisms that show this behaviour is that closure forces act on the flanks of an extending crack.

Munz (1983) has recently reviewed aspects of measuring fracture toughness values of brittle materials, particularly the role of test geometry and test method. He has also addressed the problem of measuring $K_{1c}$ in materials that undergo significant sub-critical crack extension, due to environmental effects, or display R curve behaviour. Because of the occurence of an R-curve in toughened ceramics it maybe useful to review this concept.

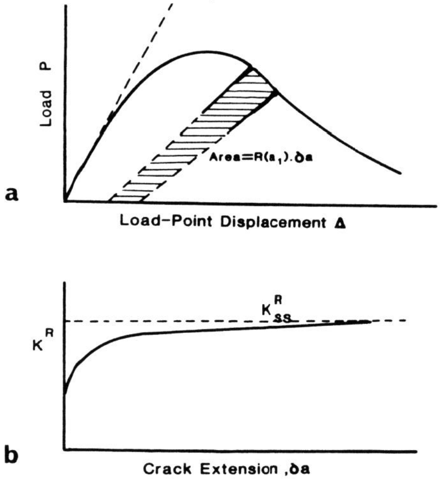

Fig. 1. a) Typical experimental observations of load versus load point displacement for stable crack extension fracture toughness test.
b) Asymptotic approach of $K^R$ to a steady state $K^R_{SS}$ value with crack extension.

The raw data which one derives from the experimental set-up to measure toughness consists of a record of load P versus load-point displacement, as in Fig. 1a. One can also measure the current crack length visually or one can deduce that crack length from the initial compliance during a partial unloading. Fig. 1a shows a partial unloading when the crack length is $a_1$, followed by another partial unloading for a slightly longer crack (of length $a_1 + \delta a$) as manifested by the slight increase in compliance (decrease in slope of the unloading line).

From this data one can derive two experimental measures of toughness. First, from the load and crack length one can calculate the nominal stress intensity factor, that is the stress intensity factor which one would expect for that value of load and crack length, if one were to ignore the presence of transformed inclusions. This nominal K is denoted by $K^R$. A typical plot of K versus crack extension $\delta a$, starting from some initial crack length $a_o$ is shown in Fig. 1b The second measure of toughness is the incremental work of fracture R, which one can derive from the incremental area under the load/displacement curve. For example the area shown in Fig. 1, divided by $\delta a$, gives the value of R for crack length $a_1$. And one could again plot R versus crack extension.

Now, one would normally expect to have,

$$R = \Gamma (K^R)^2 \qquad (1)$$

where $\Gamma$ is the usual factor $(1 - \nu^2)/E$ for plane strain. If that relation holds, the two measures of toughness may be said to be equivalent: we only need to measure one of them, either $K^R$ or R, whichever is the most convenient and we can then derive the other using Eq. (1). With transformation toughening, however, this relation does not hold, in general, so that the two measures are not equivalent, except for steady-state crack growth. This particular aspect is considered in more detail in section 4.

The significance of R-curves is that they represent the basic material property data which is required when one is conducting an analysis of the stability of cracking. Thus for an energy formulation of a crack stability analysis, actual experimental R-curve results, not R-curve results derived from experimental measurements of K using equation (1), should be used.

A common feature of ceramic materials that display a well developed R-curve is the occurence of a substantial residual crack mouth opening displacement during load-unload crack extension cyles. A number of approaches have recently been reported to adequately interpret this behaviour. Sakai and co-workers (1983) have used an energy principle of elastic-plastic fracture mechanics to explain the fracture behaviour of polycrystalline graphite. This approach enables the elastic and plastic (microcracking) components of the toughness to be separated. Whereas Mai and Hakeen (1983) measuring the R-curve of fibre reinforced cement adapt the compliance function to include an additional term that incorporates residual crack opening.

## ZIRCONIA TOUGHENED CERAMICS

Brittle ceramics may be toughened and strengthened by utilising the tetragonal-to-monoclinic phase transformation of zirconia ($ZrO_2$) particle dispersed on precipitated in a ceramic matrix†. As first pointed out by Garvie, Hannink and Pascoe (1975) the toughening originates from the volume and shape change associated with the transformation. Two mechanisms are generally recognised as imparting this improved toughness; martensitic transformation of zirconia particles about the crack tip, or nucleation and extension of matrix microcracks about particles transformed on cooling before loading. Detailed discussion of these toughening mechanisms is deferred until section 4.

Claussen (1983) has recently reviewed the range of materials that may be toughened by the addition of zirconia. For simplicity here we shall only mention two subsets of materials, namely partially stabilised zirconia ceramics (PSZ) and dispersed zirconia composite (DZC) ceramics. PSZ materials, are usually partially stabilised with magnesia (MgO), calcia (CaO), yttria ($Y_2O_3$) or rare earth oxides. They may be fabricated in a number of ways leading to coherent tetragonal precipitates in a cubic matrix, in the form of coarse grained polycrystals or large single crystals, or as fine grained, single phase tetragonal polycrystalline materials. The latter materials are usually made from Y-PSZ sintered in the tetragonal single phase field at modest temperatures (1400-1500°C) and require that the grain size is strictly controlled between 0.1-1 μm. The latter materials are probably some of the strongest ceramic materials ever made with strengths in excess of 2GPa reported (Tsukuma, Kubota and Tsukidate, 1983). A summary of the mechanical properties reported on PSZ materials is given in Table 1. Also listed in Table 1 is the toughness of the cubic stabilised zirconia (CSZ) the matrix phase of most PSZ materials with the exception of fine grained tetragonal materials.

DZC materials consist of non zirconia ceramics to which a dispersion of zirconia has been added. The form of the zirconia depends upon the manner of introduction, the size, matrix and location. Addition of zirconia may vary from conventional milling as with alumina-zirconia composites to in-situ high temperature reactions as in the case of mullite-zirconia. The size of zirconia particles in the DZC together with the modulus, thermal expansion and whether they reside within or at the grain boundary dictates which phase, tetragonal or monoclinic, is present in the sintered body. Only when metastable tetragonal particles are present is transformation toughening possible whereas with monoclinic zirconia microcrack toughening may occur. Other advantages resulting from the dispersion of zirconia in DZC ceramics include the inhibition of grain growth during sintering and scavenging of grain boundary phases. Both factors improve the strength of these materials. The properties of some DZC ceramics are listed in Table 2, also listed are the matrix properties. Notice in this table that the presence of tetragonal and monoclinic both improve the toughness but tetragonal imparts much higher strengths owing to surface compressive stresses generated on grinding.

TABLE 1

Composition, Fracture Toughness and Strength of Optimum PSZ Materials

| Material | Composition content | Tetragonal % | $K_{1c}$ MPa√m | Strength MPa | Grain Size μm |
|---|---|---|---|---|---|
| Mg-PSZ | 9 m/o MgO | 40 | 12 | 800 | 50 |
| Ca-PSZ | 7 m/o CaO | 40 | 7 | 650 | 80 |
| Y-PSZ | 2 m/o $Y_2O_3$ | 97 | 10 | 1800 | 0.5 |
| Y-PSZ | 2.5 m/o $Y_2O_3$ | 50 | 9 | 1400 | Single Crystal |
| CSZ (Y,Mg or Ca)* | | | 1.8 | 200 | 5 μm to Single Crystal |

TABLE 2

Composition and Properties of DZC Ceramics (Claussen, 1983)

| Matrix | $ZrO_2$ Content Vol % | Tetragonal Vol% | $K_{1c}$(matrix) MPa√m | Strength (matrix) MPa |
|---|---|---|---|---|
| $Al_2O_3$ | 15 | 20 | 10 (5) | 480 (550) |
| $Al_2O_3$ | 16 | 100 | 8 (5) | 1200 (500) |
| ZnO | 36 | - | 3 (1.5) | - |
| $ThO_2$ | 13 | - | 4 (1.5) | - |
| Spinel | 17.5 | 50 | 5 (2) | 500 (200) |
| Mullite | 23 | 30 | 5 (3) | 400 (270) |
| $Si_3N_4$ | 25 | - | 9 (5) | 950 (670) |
| SiC | 15 | - | 6 (7) | - |

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† Hafnia ($HfO_2$) behaves in a similar manner to zirconia. However Kriven (1983) has suggested a number of other ceramic materials that may also exhibit stress induced martensitic phase transformations.

* Cubic stabilised zirconia, matrix phase of cubic zirconia, composition depends upon stabiliser ( 14 m/o MgO, 12 m/o CaO or 12 m/o $Y_2O_3$).

In the following section the various methods of toughening ceramics are considered in isolation however it is realised that often the improved toughness of ZTC materials is due to more than one mechanism.

## CLASSIFICATION OF TOUGHENING MECHANISMS

A number of toughening mechanism have been investigated either theoretically or experimentally, or both. These include:

(i) Crack-front bowing (Green, 1983)

(ii) Crack-plane deflection (Faber and Evans, 1982)

(iii) Fibre reinforcement (Piggot, 1981, Fitzer, 1979)

(iv) Transformation toughening (Budiansky and coworkers, 1983, Marshall and coworkers 1983 & McMeeking & Evans, (1982)

(v) Microcracking (Kreher and Pompe, 1981, Faber, 1983).

The major progress since the review by Evans, Porter and Heuer (1977) has been in (iv) & (v), we shall concentrate primarily on these two in the present review.

It is convenient, both conceptually and for detailed theoretical analysis to divide the above toughening mechanisms into two groups, depending on the ratio W/L, where W denotes the range of the basic interaction involved, and L denotes the relevant microstructural length (mean free path between precipitates or microcracks, etc). For transformation toughening in PSZ, for example, we can distinguish two toughening mechanisms. There is first a short-range interaction associated with crack front bowing and crack-plane deflection, where the range of interaction W is comparable with, or less than, the average spacing L between transformed inclusions. The second toughening process is the stress-induced transformation of inclusions as the crack grows, and this is usually a long-range interaction, because the range W corresponds now to the width of the transformation zone, and (for cases of practical interest) this width is usually much larger than the average spacing L of transformable inclusions.

For PSZ it is relatively easy to estimate the separate contributions of these two mechanisms: in overaged PSZ there is no possibility of stress-induced transformation, hence extra toughness, relative to CSZ, is due to short-range interactions. [See Table 1.]

There are two reasons for the above division, (i) for long-range interactions a continuum representation may be used (eg. McMeeking and Evans (1982), Marshall et al, (1983), Budiansky et al (1983)). (ii) The R-curve behaviour associated with each of these mechanisms occurs over a length comparable with the range W of the interactions. Since W is the microstructural length L for the short-range interactions (such as crack twisting/deflections), it is not usually possible to observe the associated R-curve experimentally. On the otherhand, R-curve for long-range interaction is clearly seen.

The steady state toughness for quasi-static cracking (Fig. 1b) may then be written

$$K^R = K_c^o + \Delta K(S.R.) + \Delta K(L.R.) \tag{2}$$

where $K_c^\circ$ is the matrix toughness, $\Delta K(S.R.)$ and $\Delta K(L.R.)$ are the toughness increments associated with short range and long range interactions. As noted above, R-curve associated with $\Delta K(S.R.)$ is not usually detectable experimentally, so

$$K_c = K_c^\circ + \Delta K(S.R.) \tag{3}$$

Alternatively an energy viewpoint may be used, then

$$R = 2\gamma^\circ + \Delta R(S.R.) + \Delta R(L.R.) \tag{4}$$

where $2\gamma^\circ$ is the matrix toughness and $2\gamma_c = 2\gamma^\circ + \Delta R(S.R.)$. In the following subsections developments in the estimation of both short range and long range interactions will be outlined.

### i) Crack bowing

Crack bowing about second phase particles in a matrix poses very difficult theoretical questions which have not been completely addressed. The problem is further compounded by the presence of residual stresses or interaction stresses due to mismatch of thermal and elastic properties and interfacial adhesive forces. However elegant experimental observations by Green, Nicholson and Embury, (1979a,b) have demonstrated that the concept of crack bowing suggested by Lange, (1970) following the line tension analogy for pinned dislocations, is correct. Green (1983) has recently reviewed quasi-theoretical treatments of crack bowing and suggested that the critical parameters are the ratio of the particle size to interparticle spacing and the relative toughness of the matrix and inclusions.

The maximum toughening effect occurs when the interparticle spacing is comparable to the inclusion diameter, and the inclusions are almost impenetrable, that is have a higher toughness than the matrix. For weak obstacles the toughness is predicted to increase four fold when the volume fraction of obstacles exceeds 25%, Green (1983).

### ii) Crack twisting and deflection

A schematic diagram of this toughening mechanism is shown in Fig. 2. This like the previous mechanism imparts a toughness increase because of short range interactions between the crack front and materials microstructure. The crack front may be twisted or deflected by either localised residual stress fields or fracture resistant second phases. Crack twisting and deflection by particles, Fig. 2 reduces the driving force and increases the surface area created, both factors contributing to a toughness increment. The resultant toughness of the material depends upon the volume fraction and shape, particularly the aspect ratio, of the second phase particles. Calculations of the anticipated toughness increments (Faber and Evans, 1983) show that the effect is maximised for rod or disk shaped particles with a two fold increase in stress intensity factor for rod shaped particles of aspect ratio 1:12. The interesting feature of these calculations is thatthe toughness increment saturates with approximately 10% volume fraction of disk or rods. Also it is predicted that this mechanism is temperature independent.

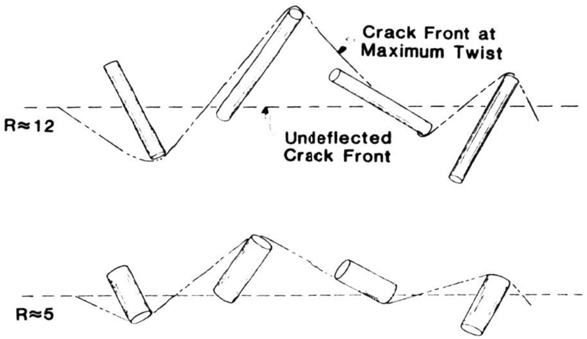

Fig. 2 Schematic of crack front deflection and twisting about rods in a matrix. R is the aspect ratio of the rods.

Evidence in support of crack deflection as a toughening mechanism is considered to be the observed increased toughness of zinc oxide with the addition of zirconia. Tetragonal or monoclinic zirconia additions both have the same effect on the observed toughness. The toughness of precipitated PSZ materials is always greater than CSZ materials,even at high temperatures where transformation toughening does not occur. This is readily observed with the highly ellipsoidal nature of tetragonal precipitates in Mg- and Y-PSZ. Another system that might have been anticipated to provide experimental evidence for this mechanism is the observed increase in toughness of silicon nitride with increasing volume fraction of the acicular phase. However the toughness increment appears to follow the rule of mixtures rather than saturate at modest volume fractions of the acicular β phase (Lange 1983).

### iii) Fibre reinforcement

Of all the toughening mechanisms the greatest improvement is possible with fibre reinforcement. Fibre reinforcement has elements of short range and long range interaction processes. The short range effects include crack deflection and bowing of the crack tip by the reinforcing fibres. The long range interaction processes might be considered ligment formation, fibre pullout and fracture behind the crack tip. Fibre pullout being of the major energy absorbing mechanisms as shown with fibre reinforced polymeric materials. Estimates for ΔR(L.R.) may be found in a number of texts (Kelly 1973, Piggot 1981) and have been critically appraised by Marston and coworkers (1974). No attempt is made to review theoretical developments in this field instead emphasis will be placed on recent experimental observations and the values of toughness that have been achieved in specific ceramic composite materials.

Some recent measurements of values obtained, usually work of fracture estimates, are listed in Table 3. These materials would all be anticipated to show profound R-curve effects. The measured values are two and three orders of magnitude higher than the matrix material although in many cases the strength maybe degraded. However recent studies by Prewo and Brennan (1981) show that with aligned and woven silicon carbide fibers in a glass ceramic matrix (of lower modulus) it is possible to improve both strength and toughness dramatically. One of the critical parameters with fiber reinforced ceramics is that the thermal expansion mismatch between matrix and fibres is minimised (Brekhovskikh and Gorodnev, 1980). This is because of the large thermal cycles experienced during fabrication or use, ΔT < 1500°C, coupled with the low toughness of the matrix and the importance of the interfacial shear stress for energy adsorption during fibre pullout. Currently carbon-carbon fibre composites are readily available and have provided some guidance for the development of high temperature (> 1200°C operating temperatures) ceramic-fibre composite materials (Fitzer, 1979). Future developments appear very optimistic with the recent availability of high quality alumina and silicon carbide fibres (Rice, 1984).

TABLE 3

Toughness of some Fibre Reinforced Ceramics

| Matrix | Fibre | Vol % | Fracture Toughness $Jm^{-2}$ | Ref. |
|---|---|---|---|---|
| Mullite | SiC coated carbon | 50 | 16,000 | Yasuda & Schlichting |
| $AL_2O_3$ | " " " " " | 40 | 10,000 | " " " |
| $Si_3N_4$ | Carbon | 30 | 4,770 | Guo et al |
| Carbon | Carbon | 60 | 5,000 | Zhao et al |
| Glass | SiC | 50 | 2,500 | Prewo & Brennan |

### iv) Transformation Toughening

The phenomenon of transformation toughening has long been known to occur in certain steels (the TRIP-steels; see Antolovich 1968), but current interest in this toughening mechanism derives from its possible application to ceramics, which was first recognized by Garvie, Hannink and Pascoe (1975)

The essential requirement for transformation toughening to occur is the presence in the material of inclusions which can undergo a stress-induced martensitic transformation from one crystal structure to another. The toughening occurs when the process of crack growth triggers the transformation of inclusions around the crack tip, leaving a wake of transformed inclusions.

The increased toughness can be explained from two viewpoints: First one could say that the system has to supply some energy to induce the inclusions to transform, and that this work of transformation is additional to the work of fracture for the matrix, so that the net work of fracture has been increased. That's the energy viewpoint. On the other hand one could also say that the transformed inclusions act as sources of internal stress which modify the nominal stress field and consequently which modify the load which must be applied to cause fracture. That is the residual stress viewpoint. A fundamental question, first raised by Evans and Heuer(1980) is whether these two viewpoints lead to the same measure of fracture toughness.

It has been argued theoretically (Rose 1984) and demonstrated experimentally (Swain and Hannink 1984) that the two experimental measures of toughness corresponding to the energy and to the residual stress viewpoints are not equivalent for initial crack growth, but become equivalent as cracking proceeds to steady-state conditions. Fig. 3a) shows the theoretical predictions of Rose (1984), based on a very simplified but conceptually useful representation of the transformation zone, while Fig. 3b) shows the experimental results of Swain and Hannink (1984) on Mg-PSZ. While the qualitative features are similar, there is at least one important difference which is not yet fully understood theoretically, namely the experimental value of R (the incremental work of fracture) decreases towards it seventual steady-state value, while $\Gamma(K^R)^2$ increases towards that same value.

The next important question is to derive a formula for the steady-state toughness, and it would appear at first sight that this is more easily done from the energy viewpoint. From dimensional consideration one would expect

$$R_{ss} = 2\,\gamma_c + 2\,h\,\Omega \qquad (5)$$

where $2\,\gamma_c$ is the matrix toughness plus toughness increment resulting from any short-range interactions, h is the height of the steady state zone (Fig. 4), and $\Omega$ has dimensions of energy per unit volume. It has been shown (Marshall, Evans and Drory, 1983, Budiansky, Hutchinson and Lambropoulous, 1983) that for dilating inclusions where $V_f$ is the volume fraction of inclusions, $e^T$ is the unconstrained volumetric dilation and $\sigma_c$ is the critical stress to nucleate the tranformation.

$$\Omega = V_f \sigma_c\, e^T \qquad (6)$$

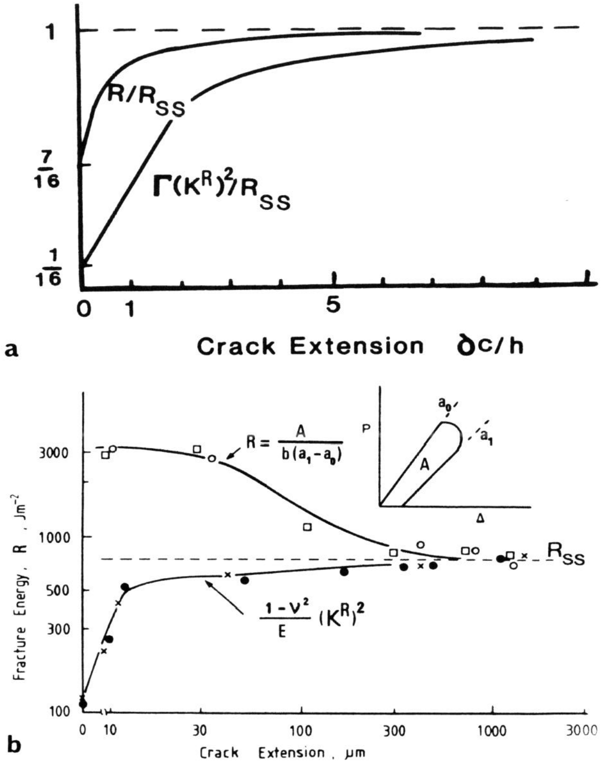

Fig. 3. a) Theoretical R-curves for PSZ material with $K^R \to 4K_c$. Notice $R > \Gamma(K^R)^2$ Rose 1984). b) Experimental $K^R$ and R curve for Mg-PSZ (Swain & Hannink, 1984).

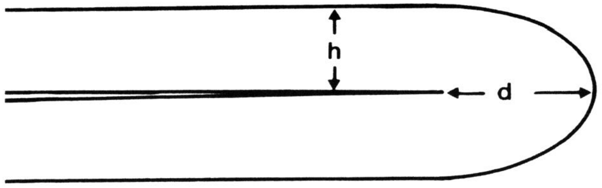

Fig. 4 Steady State transformation zone about quasi-static crack.

The residual stress approach, again on dimensional considerations (Evans, Heuer and Porter, 1977), leads to

$$K_{ss} = K_c + \Sigma\sqrt{h} \qquad (7)$$

where $\Sigma$ has dimensions of stress, but it is not simple to calculate. The real difficulty, however, is to calculate correctly the zone size h. The usual estimate (Marshall, Evans and Drory 1983, McMeeking and Evans 1983) leads to

$$h \propto (K_c/\sigma_c)^2 \qquad (8)$$

This predicts that the zone size (and therefore the toughness increase) becomes indefinitely large as $\sigma_c$, the critical stress for transformation, approaches zero, thus suggesting that the optimum value for $\sigma_c$ should be close to zero. But Rose (1984) has shown that the corect expression for his of the form

$$h \propto K_c/(\sigma_c - \psi)^2, \qquad (9)$$

where $\psi$ is a positive constant dependent on material parameters (volume fraction, transformation strain), and dependent also on the precise zone shape. The implication is that the optimum $\sigma_c$ is now some definite non-zero value.

Now, for a material such as PSZ, the critical stress $\sigma_c$ can be varied by varying the ageing time, so that the difference just noted has important practical implications in that Eq. (8) and Eq. (9) effectively lead to different conclusions as to what is the optimum ageing time.

The theoretical analyses assume that the transformation should occur at a specific stress $\sigma_c$ for all inclusions. However in PSZ systems, the propensity of inclusions to transform depends on their size (Hannink and coworkers, 1981). As there is always a range of inclusion sizes, there will also be a corresponding range of transformation stresses and hence a broadening of the maximum toughness rather than a sharp singularity as predicted by equation (8). Experimental evidence in support of the inclusion size dependence of the toughness of Ca-PSZ is shown in Fig. 5.

The energy approach to the toughening increment, equation 5, may be written in more thermodynamic fundamental terms by replacing $\sigma_c e^T$ by $\Delta G_{trfs}$ . That is

$$\Delta\gamma = 2h V_f \Delta G_{trfs} \qquad (10)$$

where $\Delta G_{trfs}$ is the energy per unit volume necessary to transform a precipitate. It is the sum of all the other energy terms, chemical energy, strain energy, surface energy, interaction energy, etc. Of these terms only the interaction energy and chemical energy terms are driving the transformation. With increasing temperature the chemical term approaches zero at the tetragonal to monoclinic equilibrium temperature, ∿1200°C for pure zirconia. The effect of this is that the toughness increment should decrease with increasing temperature which

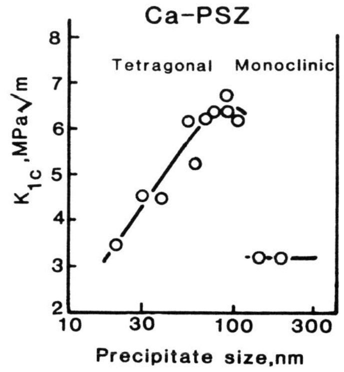

Fig. 5. The influence of precipitate size on the critical stress intensity factor of Ca-PSZ (Swain et al, 1983).

will reduce both $K_{1c}$ and zone size. A similar effect would be anticipated with yttria or rare earth oxides which decrease $\Delta G_{chem}$. An example of the temperature dependence of the toughness of Y-PSZ single crystals is shown in Fig. 6 (Ingel, 1982). Also shown in this figure is the temperature dependence of Y-CSZ single crystals. The difference

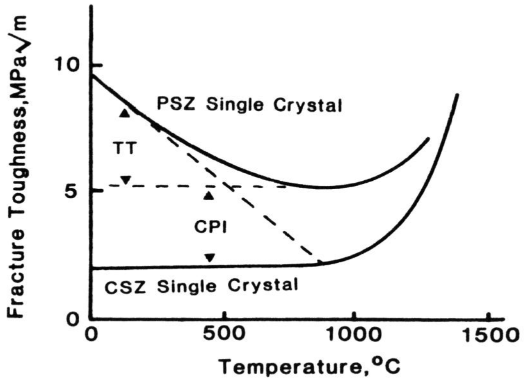

Fig. 6. Variation of the fracture roughness of Y-PSZ and Y-CSZ single crystals with temperature. TT and CPI are the toughness increments due to transformation toughening and crack preipitate interaction. Plastic deformation above 1000°C leads to the high temperature toughness results. After Ingel (1982).

between the two values at high temperature > 600°C may be attributed to crack precipitate interactions, bowing or deflection, that is short range interactions. Observations in Ca- and Mg-PSZ polycrtystalline materials support this and indicate that the transformation zone thickness decreases with increasing temperature (Swain, Hannink and Garvie, 1983).

As outlined in section 3, transformation toughening may also occur in fine grained Y-PSZ (Gupta and co workers, 1979). These materials are primarily tetragonal zirconia that maintain their stability because of yttria solubility in the tetragonal phase, their fine grain size (< 1$\mu$m), and matrix constraint. Observations of the strength and toughness dependence on yttria content are shown in Fig. 7. The theoretical modelling of these materials has essentially followed that of precipitated PSZ materials. However there are two major differences; namely, that the volume fraction of transformable phase approaches 100%, and there is no matrix within which the crack propogates, as in the precipitated systems, usually circumventing transformed precipitates. It is quite conceivable in these materials that the "matrix" toughness may change due to the transformation of that phase as the crack extends through the material. This is virtually the situation Antolovich addressed in 1968 with trip steels. Also recent observations suggest that considerable mirocracking accompanies transforming grains about the crack tip (Rühle, Claussen and Heuer, 1983).

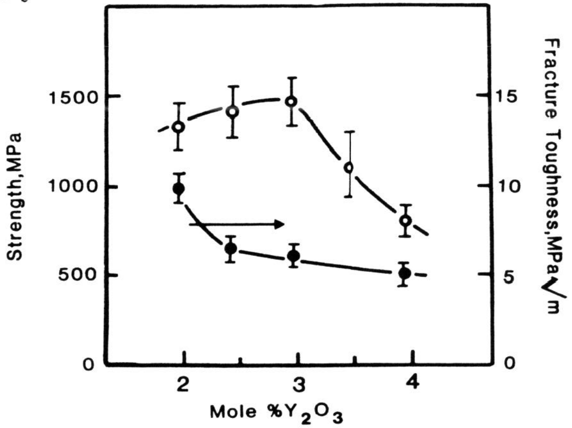

Fig. 7. Dependence of bending strength and $K_{1c}$ of fine grained Y-PSZ materials on yttria ($Y_2O_3$) content. The specimens were hot isostatically pressed at 1400°C and 150 MPa. After Tsukuma et al, 1983.

Transformation toughening is also observed in a range of zirconia dispersed ceramic composites (section 3), particularly alumina-zirconia. However because of the zirconia size distribution and the possibility of microcracking toughening as well as transformation toughening this area is still being investigated . Theoretical aspects of transformation and microcrack toughening will be considered in the following sub section. Before discussing microcracking a few closing words on transformation toughening are in order. To date, virtually all theoretical studies have only considered the pure dilatation component of the transformation of zirconia in an isotropic matrix. The consequences are that the transformation zone shapes are similar to the hydrostatic tension contours about the crack tip. Two recent anisotropic elasticity studies that considered the transformation in Y and Mg-PSZ single crystals showed that the shear component of the transformation distinctly modifies the zone shape (Porter, Evans and Heuer, 1979 and Ingel, 1982). A comparison of the hydrostatic tension contour and anisotropic contour, the latter based on the Westergaard anisotropic crack tip stress field and anisotropic tetragonal to monoclinic transformation for one of the six possible crack tip precipitate orientation interactions is shown in Fig. **8** for Mg-PSZ. Recent experimental observations do in fact support the anisotropic calculations (Swain & Clarke, 1984).

### v) Microcrack Toughening

Microcracking may occur for a number of reasons in ceramic materials. Usually it is the result of residual stresses present within the body after sintering. This source of stress may be the consequence of; thermal expansion anisotropy (TEA) in non-cubic symmetry materials, inclusions of a second phase with differing thermal expansion and elastic properties from the matrix, and transformation of a second phase. The common feature to all these systems is the presence of internal residual stresses before a macrocrack is introduced. Another distinction must be drawn between materials where these microcracks are present before or as a result of the stress field about an introduced macro-crack.

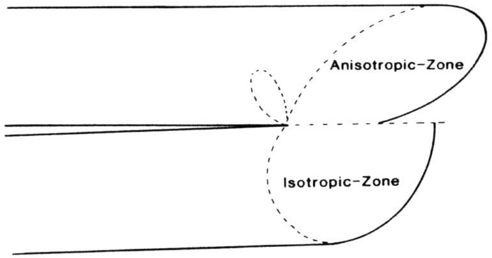

Fig. 8. Comparison of anisotropic versus isotropic predictions of tranformation zone about the crack tip.

Examples of various types of microcracking are shown in Fig. 9 . Fig 9a represents model systems, usually spherical inclusions in a glass (isotropic) matrix. Fig.9b idealises the situation in non-cubic symmetry materials with residual stresses acting on triple points at grain boundary junctions whereas Fig. 9c is a schematic of the conditions prevailing in ZDC materials with both misfitting and transformed zirconia inclusions. The latter situation occurs in alumina-zirconia composite systems. A statistical element is introduced into the problem through the variation of zirconia particle size and phase, and the alumina grain size (Davidge, 1981). Despite these compounding complexities, introduced going from model to commercially interesting materials, the mechanical behaviour follows a similar trend.

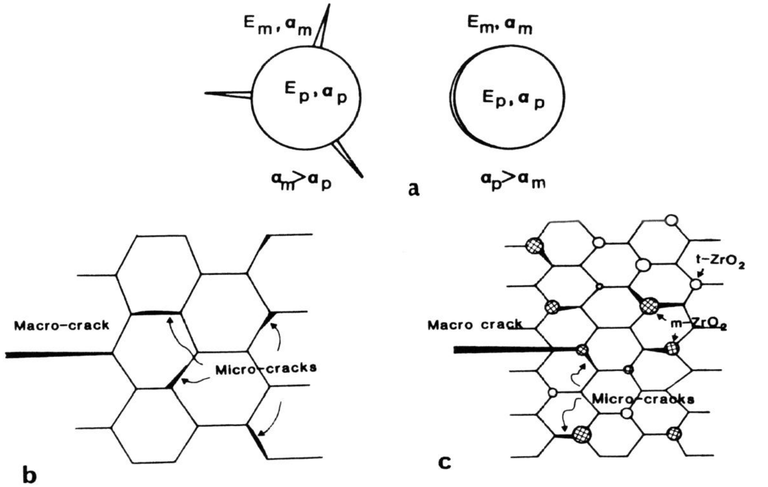

Fig. 9 a) Microcracking about isolated inclusions in a matrix with differing thermal expansion coefficients (α). b) Microcracking at grain boundaries in non cubic materials. c) Microcracking at grain boundaries of materials containing misfitting and transformed inclusions.

Turning first to model systems, Miyata and colleagues (1983) have studied the effect of glass bead additions to a glass matrix and varied the size, volume fraction and thermal expansion mismatch between the two constituents. The thermal expansion of the spheres was less than or equal to the matrix, generating compressive stresses within the spheres. Larger spheres with sufficient residual stresses formed microcracks upon cooling thus it was possible to distinguish between pre existing and crack field initiated microcracking on the strength and toughness of these materials. Some typical examples are shown in Fig. 10, they compare the effect of volume fraction and increasing thermal expansion mismatch (1→ 4) for constant size (300μm) glass bead additions. Glass bead compositions 3 and 4 lead to microcracking on cooling after fabrication whereas 2 microcracked in the crack tip stress field. The strength and volume fraction dependence of the latter material (2) are superior to the former (3 & 4).

Before turning attention to the theoretical interpretation and modelling of microcrack toughening let us consider two other types of microcracking, due to phase changes and TEA stresses. Alumina-zirconia composites are an example of a material with both stress systems present, however generally the grain size is kept small so that the TEA stresses do not lead to microcracking (and strength reduction). The effect of different sized monoclinic inclusions and volume fractions has been studied by Claussen (1976, 1977), the results are shown in Fig .12 These observations suggest that the critical zirconia particle size to initiate spontaneous microcracking on cooling lies between 1.25 and 3μms

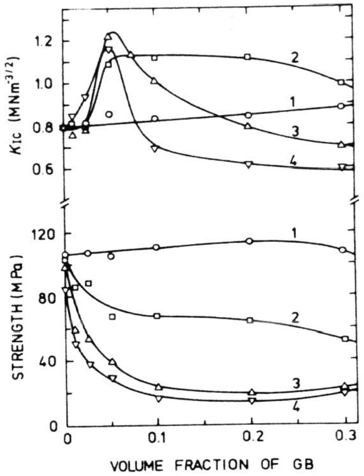

Fig. 10. Dependence of $K_{1c}$ and strength on the volume fraction of glass beads (300μm diameter) added to a glass matrix. The thermal expansion mismatch increases from 1 to 4. After Miyata and coworkers (1983).

Increasing the volume fraction as shown by Lange and Green (1981) is to increase the probability of zirconia particles agglomerating and the likelihood of microcracking on cooling. The similarity between these results and the model systems is striking.

The third area of microcracking due to TEA stresses has also received considerable attention in the last few years. Some controversy existed in this area because of testing techniques (Mussler, Swain and Claussen, 1982). However Rice and colleagues (1981) have recently summarised the evidence for both TEA and elastic anisotropic (EA) stresses on the grain sized dependence of single phased materials. Their results for alumina, titania ($TiO_2$) and niobium oxide are shown in Fig. 12. In all these cases TEA stresses are present and the toughness passes through a maximum with grain size.

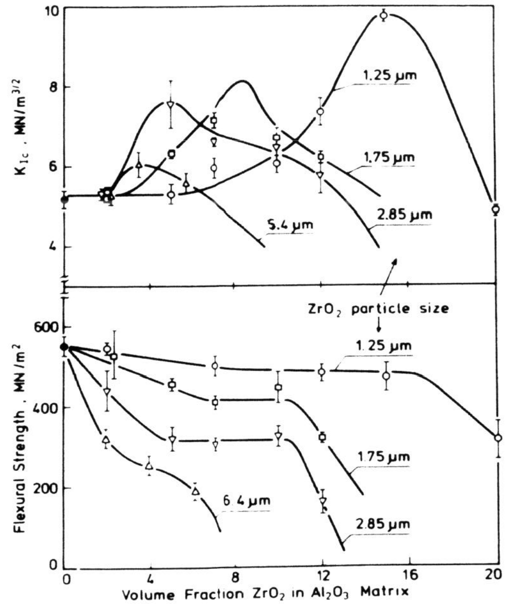

Fig. 11. Dependence of $K_{1c}$ and strength on the volume fraction and size of zirconia particle additions to alumina. After Claussen et al (1977).

There have been a number of attempts to theoretically model microcrack toughening, two of the most recent by Pompe and Krehar, (1981,83) and Evans (1983). The major differences between these two authors is that Pompe and Kreher consider only a steady state situation within which exist two well defined interaction zones whereas Evans considers both the initial development of a single interaction zone and then the steady state propogation of this zone. A consequence of the latter approach is that R-curve behaviour is anticipated in microcrack toughened materials because of crack tip shielding by the energy dissipative zone. There exists experimental evidence in support of this conclusion in alumina polycrystalline materials (Steinbrech and co workers, 1983).

Theoretical analysis of toughening due to microcracking follows a similar pattern to that due to transformation toughening, but the detailed calculations are now more complicated. In particular, during steady-state cracking, equation (4) is applicable, where again ΔR(L.P.) is 2hΩ. However at this time the estimation of the various terms contributing to ΔR(L.R.) have not reached a definitive stage.

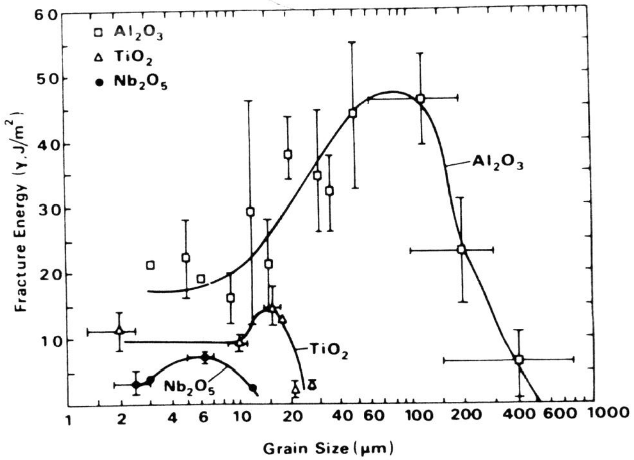

Fig. 12. Fracture toughness dependence of three non cubic oxide materials on grain size. After Rice et al (1981).

In contrast to pure transformation toughening, ΔR(S.P.) may now be negative. This is because the microcracks link up with the macrocrack. A common estimate of ΔR(S.R.) is

$$\Delta R(S.R.) = -2\gamma A_{mc} \qquad (11)$$

where $A_{mc}$ denotes the area fraction of microcracks across the fracture surface. This estimate effectively assumes that the increased work of fracture associated with crack-plane deflection due to interactions with non coplanar microcracks, is cancelled by a decrease resulting from interactions with co-planar microcracks. This simple estimate is unlikely to be accurate for large area fractions, ($A_{mc} > 0.2$), but at present, there is no better theoretical estimate.

When estimating $\Omega$ , the work dissipated per unit volume in the microcrack wake, we must distinguish between microcracking associated with (i) pre-existing centres of dilatation (for example thermal expansion mismatch or pre-transformed zirconia (monoclinic) inclusions),and (ii) stress induced centres of dilatation. Case (i) is the simpler one to analyse theoretically, and we shall only note an expression for $\Omega$ that has been derived for that case.
Kreher and Pompe (1981) use as basic parameters, i) a critical stress $\sigma_{mc}$ for microcracking, and ii) a damage parameter $\omega$ given by

$$\omega = kN(2c)^3 \tag{12}$$

where N is the number of microcracks per unit volume, c is the effective average radius of the microcracks viewed as circular cracks, k is an adjustable constant which depends on the actual shape of microcracks and is chosen so that the effective modulus of microcracked material is given by

$$E_{eff} = E(1 - \omega) \tag{13}$$

iii) The volume fraction of transformed inclusions, $V_f$, or of regions under compression due to thermal expansion mismatch. The point is that microcracking only occurs in the remaining portion $(1-V_f)$ of material.
iv) The stress free dilatation $e^T$ due to transformation or thermal expansion mismatch of a misfitting inclusion. Then Kreher and Pompe obtain the following expression (slightly rearranged) for $\Omega$ ,

$$\Omega = (1-V_f)\frac{\sigma_{mc}}{2E_{eff}}\left\{1-\frac{5}{2}\left(\frac{Ee^T}{3\sigma_{mc}}\right)^2 \cdot \frac{V_f(1+V_f)(1-\omega)}{2-\omega(1-V_f)}\right\} \tag{14}$$

This expression highlights the significance of the volume fraction of non damaged material, microcracks initiation stress and any dilatational strain.

Faber and Evans (1983) have given an alternative expression for $\Omega$ , derived by an extension of the method used by Budiansky, Hutchinson and Lambropoulos (1983) for transformation toughening. At present it is not clear whether their expression is equivalent to that of Kreher and Pompe (1981).

Finally, returning to equation (5), current estimates of the zone size h suffer from the limitation noted earlier in the context of transformation toughening, namely that these estimates do not correctly account for the perturbation of the nominal stress field due to the presence of the microcracked zone. It will be clear from the above remarks that we do not yet have a definitive theory of microcracking.

## CONCLUSIONS

During the last decade tremendous advances have been made both conceptually and practically in the development of tough ceramics. The most spectacular advances have been in the field of transformation toughening based upon zirconia containing systems. However more importantly these advances have lead to the possibility of multiple toughening mechanisms operating simultaneously in a ceramic thereby greatly improving its properties.

The level of theoretical understanding of these toughening mechanisms has improved dramatically over the last few years. Again the greatest advances have been with transformation toughening and the paper by Budiansky, Hutchinson and Lambropoulus (1983) is an excellent example of this development. However despite these advances a number of simplifying assumptions have had to be introduced including omission of the shear component of the transformation and in the case of Y-PSZ (fine grained) the fact that the matrix changes upon transformation. There appears to be some conflicts between various attempts to model microcrack toughening and these problems are further exaggerated with the presence of both microcracking and transformation toughening.

Future work both practically and theoretically will be attempting to incorporate multiple toughening mechanisms in the same material. The recent advances in fibre reinforcement with the availability of quality ceramic fibres will undoubtedly spur further developments in this field. With such a variety of toughening mechanisms available the challenge to design both strong and tough ceramics will intensify.

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