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Comparison of Indentation and Notched Bar Toughness of TZP Ceramics: Relevance to Models of the Fracture Process

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SUMMARY

Indentation and notched bar (SENB) toughness were determined for two compositions, sintered using different schedules designed to develop different toughness.

Experimental results indicate that the indentation technique tends to give higher values of toughness than SENB specimens with narrow notches. This is attributed to the extensive transformation that takes place in the region of the indentation.

The effect of notch width in tetragonal zirconia polycrystalline (TZP) materials was examined and found to be pronounced; measured K_{Ic} values varied between ~7 and 17 MPa m$^{1/2}$ for notch widths between 0·1 and 1 mm. X-ray diffractometry (XRD) was used to determine the extent of the tetragonal to monoclinic phase transformation on fracture surfaces. These data, correlated with the K_{Ic} data, permit an experimental assessment of the toughening increment, ΔK^{T}_{Ic}, due to the transformation, to be made. The experimental value shows good agreement with the theoretical model of Seyler and his co-workers, based on the deviatoric component of the shear transformation.

A modified equation permits a conservative estimate of the toughness to be made, if the extent of transformation is known.

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1. INTRODUCTION

The simplification of fracture toughness measurement in ceramic materials by the introduction and refinement of the indentation technique[1] has led to its application on materials that were not widely available when the technique was developed.

Tetragonal zirconia polycrystalline (TZP) ceramics based on the Y_2O_3–ZrO_2 system have a structure that can consist of 100% tetragonal grains retained metastably, at room temperature. Many favoured compositions used for engineering applications contain 10–20% of the cubic phase or of a non-transformable tetragonal phase, t', produced from the high temperature cubic phase on cooling.[2]

High voltage transmission electron microscopy (TEM) studies[3] of TZP fracture in a thin foil have shown that some tetragonal grains do not transform in the crack tip stress field and its wake. In addition, certain workers[4] have reported relatively low levels for the $t \rightarrow m$ transformation measured using X-ray diffractometry (XRD) methods.

The literature also indicates a growing distinction between the toughening mechanism of partially stabilised zirconia (PSZ) and TZP materials. This is related to the relative importance of the hydrostatic and deviatoric components of the transformation,[5–7] an aspect that will be explored further in this paper.

Fracture toughness measurements employing indentation techniques have been used on TZP materials by a number of researchers.[4,8–11] In contrast, notched bar (SENB) techniques have been applied by relatively few researchers[12] who worked with mixed-oxide powders.

The importance of notch width in notched bar (SENB) specimens has been widely recognised in studies of alumina.[13,14] In addition a number of other materials have been shown to have K_{Ic} dependence on notch width, including silicon nitride, silicon carbide, porcelain and glass.[15] Zirconia ceramics with a PSZ structure showed an increase in K_{Ic} of 38% with increase in notch width from 0·2 mm to 1·2 mm.[16] In the study by Munz on aluminas, K_{Ic} increased from 4 MPa m$^{1/2}$ with a 50 μm notch to approximately 5·7 MPa m$^{1/2}$ with notches in the range 0·3–1 mm (43% increase). It has been found that SENB specimens with sharp cracks have K_{Ic} values which are equal to those of bars with a notch tip radius of 0·08 mm[15] or a notch width of 50 μm,[14] but the minimum K_{Ic} may be conservatively estimated, by extrapolation, to be at zero notch width.[16] In the present study the effect of notch width in TZP was investigated because of the complicating factors relating to the transformation in the region of both a static crack and one propagating under an applied stress.

A consequence of the passage of a crack is the development of a transformed layer and thus the extent of transformation in TZP fracture surfaces has been determined in order to correlate this parameter with K_{Ic} values.

2. EXPERIMENTAL

Homogeneous co-precipitated Y_2O_3–ZrO_2 powders 2Y and 2·5Y, Toyo Soda Manufacturing Co., Tokyo, Japan, with 2 and 2·5 mol% Y_2O_3 were uniaxially pressed in a 42-mm die at 25 MPa. The discs were sintered using a range of firing schedules, cut into bars and notched for testing. The bars were then annealed for 10 min at 1100°C in order to minimise transformation effects due to machining. The geometry of the bars is listed in Table 1.

The specimens were broken in 3-point bending in an Instron testing machine with a cross-head speed of 0·1 mm min^{-1} and a load cell full scale deflection of 1 kN. K_{Ic} values were calculated using the equation of Richerson.[17]

TABLE 1
Single Edge Notch Bar Geometry

Dimension	*Size/mm*
Width	5
Breadth	2·5
Notch depth	1·5
Span	19
Notch width	0·1, 0·5, 1

Indentation fracture toughness measurements were performed with a load of 10 kg on a Zwick microhardness tester and the indentation diagonals and crack lengths were measured directly. Five to ten indentations were performed on each specimen. Indentations with loads above 10 kg were made using a Vickers hardness tester and were again measured directly. The equation of Anstis *et al.*[1] was used to compute K_{Ic} from the length of the indent diagonals and the crack length, with values of the elastic modulus determined by a sonic method.

Fracture surfaces were X-rayed to determine the monoclinic phase content using a Philips diffractometer with Cu–Kα radiation. The nature of the monoclinic phase in the fracture surface is discussed elsewhere.[7]

3. RESULTS

Table 2 shows the toughness as determined by the notched beam technique (with different notch widths) and the indentation techniques for the different compositions and sintering conditions used in this study. It is clear that toughness tends to increase with sintering temperature and is higher for the 2 mol% Y_2O_3 composition than for the 2·5 mol% composition. Both SENB and indentation techniques show the same trend for the change in toughness with sintering and composition. However, the individual values of toughness vary strongly as a function of notch width and differ between the SENB technique and the indentation technique.

TABLE 2

Composition/mol% Y_2O_3	*Sintering treatment/°C: h*	*SENB K_{Ic}/MPa $m^{1/2}$*			*Indentation K_{Ic}/MPA $m^{1/2}$*
		0·1	*0·5*	*1*	
2	1 675: 2	7·67	15·91	17	10·0
2·5	1 675: 2	6·88	13·40		8·9
2	1 575: 0·5	6·99	13·71	17	9·3
2	1 400: 2	6·44	12·69	16	9·0
2·5	1 400: 2	5·56	10·11		5·7

Of particular note is the observation that SENB toughness with a 0·1 mm notch is considerably lower than the indentation toughness for the 2 mol% Y_2O_3 sintered at 1675°C.

The test bars with the 1 mm wide notches showed considerable variation between individual specimens (averaging 5 MPa $m^{1/2}$), whilst the scatter in the K_{Ic} values for test bars with 0·1 mm notches is <0·4 MPa $m^{1/2}$. The increase in notch width causes the measured K_{Ic} values to increase by more than 100%, indicating the importance of this experimental variable to the measurements.

Following the XRD analysis of the fracture surfaces and the determination of the monoclinic content, it became possible to plot the increase in toughness as a function of the extent of the monoclinic transformation directly. These results are shown in Fig. 1.

It is apparent that the toughness measured is dependent on the width of the notch and that specimens with similar sintering schedules exhibit less transformation to the monoclinic phase with the narrow (0·1 mm) notches when compared to the 0·5 mm notches.

For the 0·1 mm and 0·5 mm notch width fracture toughness data there is a linear relationship between K_{Ic} and the monoclinic content.

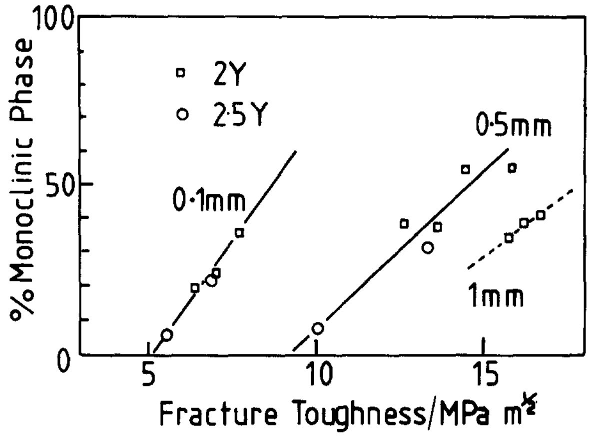

Fig. 1. Percentage monoclinic phase in fracture surface vs toughness for three notch widths, 0·1, 0·5 and 1 mm, and two compositions (□, 2Y; ○, 2·5Y).

Extrapolation of this line for the 0·1 mm notch width specimens to the K_{Ic} axis indicates a toughness for tetragonal material of about 5 MPa m$^{1/2}$ where no transformation occurs.

In order to obtain a set of data over a range of notch widths, test bars with 1 mm wide notches were made. However, no further conclusions can be drawn from the data on this specimen configuration.

4. DISCUSSION

4.1. Comparison of SENB and indentation techniques

The most surprising result of this study has been the discovery that SENB specimens with 0·1 mm notches have K_{Ic} values considerably smaller than the indentation K_{Ic} values for the same materials. It is not yet clear whether further reduction in notch width (for example to 40 μm) will result in a further reduction in K_{Ic}. The evidence from other studies[13] indicates this to be the case. Further, the study of Haberko and Pampuch[4] while it is not directly comparable, showed a minimum value of K_{Ic} by indentation of ~2 MPa m$^{1/2}$. This may be partly attributable to the low value of the Young's modulus, E, indicative of the higher porosity ceramic used in their study.

A 6 mol% Y_2O_3 material examined in conjunction with the present work proved to have an indentation toughness of 2·2 MPa m$^{1/2}$, and yet after abrasion showed no evidence of the presence of transformable tetragonal material. However, there could well be a lowering in toughness with an increase in yttrium oxide concentration not related to the transformation process. A 3 mol% Y_2O_3 material sintered at low temperature to produce a fine grain size, and therefore in a relatively untransformable condition, has a toughness of 3·9 MPa $m^{1/2}$. These results indicate that K_{Ic} values of certain TZP ceramics may be even lower than the present study indicates. If a value of 4 MPa $m^{1/2}$ is assumed to be the toughness of tetragonal zirconia of low yttrium oxide content in the absence of the transformation, the data in Fig. 1 indicate that the maximum toughness measured by the notched beam technique on a specimen undergoing 100% transformation would be of the order of 12·5 MPa $m^{1/2}$. However, it is clear that such extensive transformation rarely occurs in materials based on the homogeneous co-precipitated powders used in this study. The 2Y material sintered at 1675°C for 2 h has a grain size of the order of 1·5–2 μm but only undergoes 40% transformation in the fracture surfaces with a 0·1 mm notch.

From the results and the preceding discussion it is apparent that the toughness of TZP materials determined by indentation is overestimated when extensive transformation occurs. This may be due in part to the induced transformation taking place in regions of the ceramic adjacent to the indent. Such transformation together with the accompanying volume expansion would tend to close the radial cracks and inhibit their propagation, resulting in overestimation of the toughness. By contrast, cracks in annealed SENB specimens with fine notches may begin to propagate in the tensile stress field before there is a sufficient stress intensity to initiate transformation ahead of the crack tip. Thus the 'material-independent' constant for well behaved materials in the indentation equation of Anstis *et al.*[1] clearly requires revision where transformation takes place and the condition of the metastable material in the region of the crack requires quantitative characterisation.

4.2. The effect of notch width on K_{Ic} in TZP

The effect of increase in notch width on measured K_{Ic} in TZP, compared to that in studies of other ceramic materials (including PSZ), is very large where extensive transformation takes place. Over the range of notch widths studied, increases in K_{Ic} of the order of 120% are observed. This is three times the increase observed for aluminas and PSZ materials. The size of this increase must be related to the induced transformation in the notch tip zone prior to crack initiation and is analogous to excessive plastic yielding due to overload during crack initiation in metals.

The large increase in the scatter of K_{Ic} results with increase in notch width suggested that fracture initiation at the base of large notches may be controlled by both the transformation and statistical variations in the flaw population associated with the notch. As the notch width is increased, the flaw distribution in the base of the notch dominates the load required to initiate failure and, hence, gives rise to large variability in the results. Furthermore, the increased radius of curvature of the notch will allow a large volume under stress in the notch tip, which on transformation can develop transformation-induced flaws, caused by the link up of microcracks associated with the transformation, or by their interaction with pre-existing processing flaws.

4.3. Estimation of K_{Ic} increment due to transformation

Earlier work indicated that the shear component of the transformation dominates in the fracture toughness increment of TZP ceramics.[7] Seyler and his co-workers[6] have analysed this problem and suggested that the toughening increment for transformation can be determined by

$$\Delta K_I^T = \frac{E\gamma^T}{(1-\nu^2)} w^{1/2} 0{\cdot}21 \,(\sin 2\phi)$$

where γ^T is the shear strain, E is Young's modulus, w is the zone diameter, ϕ is the angle between the crack plane and the shear direction, and ν is Poisson's ratio. This relation can be used to predict the toughening increment for a fully transformed zone (assuming all grains are favourably oriented).

Using

$E = 203$ GPa (determined using a longitudinal resonance technique)

$\nu = \sim 0{\cdot}32$ (Ingel *et al.*[9])

$w = 2\ \mu$m (the maximum grain size of the material observed by SEM)

$\phi = 45°$ (to maximise the expression)

$\gamma^T = 0{\cdot}08$ (Rühle *et al.*[18])

$\Delta K_I^T = 5{\cdot}37$ MPa m$^{1/2}$

This relatively simple analysis compares quite well with the increment estimated from experimental data in this study, which is

$$\Delta K_I^T = 7{\cdot}5 \text{ MPa m}^{1/2} \text{ (for 100\% transformation)}$$

There is also a hydrostatic and a microcracking contribution to the toughening[19] which would make up for the conservativeness of the theoretical prediction. Seyler's expression can therefore be modified to take account of the incompleteness of the transformation by introducing a term for the extent of the transformation, V_f.

Thus

$$\Delta K_{\mathrm{I}}^{\mathrm{T}} = 0{\cdot}21\,\frac{E\gamma^{\mathrm{T}}}{1-\nu^2}\,w^{1/2}\,.\,V_{\mathrm{f}}$$

The $(\sin 2\phi)$ term is omitted since only favourably oriented grains are observed to transform.

If the fracture toughness in the absence of transformation is assumed to be 4 MPa $m^{1/2}$ for materials in the compositional range 2–3 mol% Y_2O_3, it is possible, using the above expression, to make a conservative estimate of the toughness of the material. It requires a knowledge of the grain size and the extent of transformation in the fracture surface, which can be determined by XRD and SEM of MOR specimens.

The modified expression is similar in form to that of Evans:[5]

$$\Delta K = 0{\cdot}2G\gamma^{\mathrm{T}} V_{\mathrm{f}} w^{1/2}$$

where G is the shear modulus.

5. CONCLUSIONS

1. The indentation technique for determining K_{Ic} is inappropriate for TZP materials and overestimates the toughness by at least 25% where substantial transformation occurs on fracture.
2. There is a considerable dependence of SENB K_{Ic} values on the width of the notch. The narrow (0·1 mm) notch used in this study gives K_{Ic} values lower than those given by indentation techniques.
3. There is a linear relationship between toughness increment and the extent of transformation, which can be predicted by the expression

$$\Delta K_{\mathrm{I}}^{\mathrm{T}} = 0{\cdot}21\,\frac{E\gamma^{\mathrm{T}}}{(1-\nu^2)}\,w^{1/2} V_{\mathrm{f}}$$

when the zone size (w) is considered to be of the same dimension as the grain size of the transforming material.

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