Evaluation of Fracture Toughness and R-Curve Behaviour of Y-TZP Ceramics

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(Received on August 8, 1988; accepted in the final form on November 18, 1988)

The fracture toughness of tetragonal zirconia polycrystals containing 2 and 3 mol% Yttria (Y-TZP) were evaluated by 4 measuring techniques which included (1) Double Cantilever Beam (DCB), (2) Vickers indentation technique by direct measurement of radial cracks or Single Indentation Technique (SIT), (3) from the strength dependence of the Vickers indentation load and crack length of the so called Modified Indentation Technique (MIT), and (4) Single Edge Pre-crack Beam (SEPB). There were considerable differences of K_{IC} value between the different techniques with these being exaggerated for 2 mol% Y-TZP material. It was found that SEPB and DCB techniques gave the most conservative and consistent estimates of K_{IC}. The SIT technique was very sensitive to the indentation load for 2Y-TZP, estimates of K_{IC} decreasing with increasing load. It was found that K_{IC} increased with the ratio of transformation zone size to crack length. Recommendations for determination of K_{IC} of TZP materials are made. R-Curve behaviour and K_{IC} in various environments were determined by the DCB technique. The monoclinic content of different specimen surfaces including as-sintered, as-polished and fracture surface have also been analysed by X-ray diffraction.

KEY WORDS: fracture toughness; Y-TZP; K_{IC} - value; transformation zone; R-Curve.

1. Introduction

Yttria-tetragonal zirconia polycrystals (Y-TZP) have been considered as one of the most promising candidate “high-tech” ceramic materials for structural applications because of their extremely high strength ("1 600 MPa) and excellent fracture toughness ("22 MPa m^{1/2}). As a consequence the availability of literature on these materials has recently multiplied. Some excellent reviews of Y-TZP materials are also available. However evaluation and comparison of fracture toughness testing techniques for these materials, which exhibit a martensitic transformation, is limited. In particular, clarification of R-Curve behaviour of Y-TZP and determination of K_{IC} in different environments have not yet been addressed.

Fracture toughness (K_{IC}) is a critical mechanical property parameter which characterizes the material’s resistance to crack propagation or to damage (including mechanical, thermal shock or stress-corrosion damage). Therefore, it is a very important parameter for high-tech ceramics being considered for structural components. K_{IC} is generally considered an independent parameter of materials, but the value of K_{IC} determined for a given material is a function of environment, loading rate, and specimen geometry. Hence the K_{IC} values determined by various testing techniques are often considerably different. To date there are a number of techniques proposed for determining K_{IC} of ceramics. Several excellent reviews of fracture toughness testing techniques of ceramics have recently been published.

On the basis of indentation fracture mechanics, the principal indentation techniques for determination of K_{IC} in ceramics have been developed. These are:

1) the direct crack measurement indentation technique or single indentation technique (SIT),
2) indentation-strength technique (IST),
3) combining 1) with 2), that is, where both indent crack length and strength of indented specimen are measured for calculating K_{IC}, or the so called modified indentation technique (MIT).

Evans has critically commented on the three techniques. Unfortunately experience indicates that the K_{IC} value obtained is either overestimated or underestimated, and different K_{IC} values are obtained by different K_{IC} expressions for the SIT analysis. The single edge notch beam (SENB) technique is a simple and easy method to use under different environments and temperature, the value of K_{IC} determined, however, varies strongly with notch width and notch radius in the specimen. Various chevron notch beam techniques are now theoretically well developed, but the complex specimen shape limits their application. The double torsion (DT) technique has an inadequate theoretical basis because of crack front curvature, relatively complex experimental device and problems associated with machine relaxation particularly for determining slow crack growth. The double cantilever beam (DCB) technique is a feasible, reliable and multi-purpose method: however, it is only used for verification purposes because of complex specimen shape, relatively large specimen size and preparation time. Recently a new technique based on the SENB technique, which
takes advantage of the indentation technique, has been developed,\textsuperscript{29,31} Vickers indents are made on the specimen surface along a line which is perpendicular to the specimen tensile axis. Radial cracks emanating from those indents will link up upon stressing. In addition the sub-surface median cracks will link and then unstably propagate (pop-in) forming a uniform subsurface pre-crack that is further stably extended. The stressing system used to stably propagate these median cracks consists of loading regions either side of the indented line in compression, the so called "bridge indentation method",\textsuperscript{29} This specimen with such pre-cracks is then tested by the SENB technique. The $K_{\text{ic}}$ value determined by this method will be a conservative estimate. It has been named the "bridge indentation"\textsuperscript{(29)} technique or "Single Edge Pre-crack Beam" (SEPB) technique. To date no standard test method for estimating $K_{\text{ic}}$ of ceramics is established.

The primary objectives of the present work are:

1) to evaluate the fracture toughness of Y-TZP containing 2 and 3 mol\% yttria using various testing techniques including DCB, SIT, MIT and SEPB techniques in order to find which will give the most reasonable and reliable (conservative) estimates of $K_{\text{ic}}$

2) to determine $K_{\text{ic}}$ of Y-TZP in different environments, and

3) to determine and observe R-Curve behaviour of Y-TZP.

2. Experimental Procedure

DCB specimens were prepared from homogeneous co-precipitated $\text{Y}_2\text{O}_3-\text{ZrO}_2$ powder containing 2 and 3 mol\% $\text{Y}_2\text{O}_3$ which was produced by Toyo Soda Manufacturing Co. (TOSOH) in Japan. Plates $\sim 21 \times 100 \times 5$ mm were pressed at 25 MPa in a steel die, then iso-pressed at 200 MPa and sintered at 1500°C for 1 h. The final specimen size was $3.5 \times 15 \times 68$ mm. Fig. 1(a) shows the configuration of the DCB specimen. After machining, these specimens were annealed at 700°C for 30 min, and then polished. A pre-crack was introduced with the testing machine, the specimen was then annealed again in order to minimize transformation effects due to polishing and introducing the pre-crack. The temperature of 700°C for annealing was chosen as this temperature exceeds the reverse monoclinic to tetragonal transformation temperature ($A_T$) but is less than the temperature at which creep occurs. Load and corresponding crack increment were simultaneously recorded with an $X-Y$ recorder by a resistance-track technique in order to determine the R-Curve behaviour. The resistance track technique consisted of a parallel series of narrow lines (20 $\mu$m wide) of Cr metal that had been evaporated onto the specimen surface ahead of the anticipated crack path. The load at failure of specimen was used for calculating $K_{\text{ic}}$. $K_{\text{ic}}$ was also determined at $-65 \sim -70$°C (in an ethanol–dry ice mixture), in water and silicone oil by the DCB technique for investigating the effect of environment on the fracture toughness of Y-TZP ceramics.

MIT specimens, $3.5 \times 6.5 \times 40$ mm in dimensions, made from the ‘arms’ of the fractured DCB specimens, were also annealed after machining. The indentation load was 40 kg and breaking of the specimen was performed at a crosshead speed of 0.05 mm/min, with the outer span of the 4-point bending jig being 30 mm and inner span 15 mm. The specimen generally breaks from one indent, leaving two survivors on the surface from which the final length of indent radial crack after fracture, $C_m$, can be measured with an optical microscope. The fracture load was used for calculating the fracture strength of an indentined specimen, $\sigma_m$. These values of $C_m$ and $\sigma_m$ are then used for calculating $K_{\text{ic}}$. Fig. 1(d) is a schematic drawing of the MIT technique.

SIT specimens were also taken from polished DCB specimen fragments. The indentation load was varied from 10 to 80 kg in 10 kg increments. The purpose was to observe the influence of load on $K_{\text{ic}}$.

SEPB specimens were prepared from the same starting powder as the DCB specimens, and identical forming and sintering conditions. The specimen size was $3 \times 4 \times 40$ mm. Some of them were also taken from fragments of DCB specimens. After grinding, two sides were polished in order to more easily observe the crack and to ensure that surface compressive stresses introduced by grinding did not locally arrest the crack. The procedure for introducing a pre-crack was as follows:

1) Place 3 or 4 Vickers indents on the top surface of the specimen along a line (see Fig. 1(b)). Indentation load, $P_r$, depends upon the material being examined. In the present work, $P_r$ was 30 kg.

2) Exert a compressive stress with the bridge loading device to the specimen (see Fig. 2) in order to promote indent median/radial crack propagation to form a deep pre-crack in the specimen.

3) Release the compressive stress when the pre-crack reaches $1/3$–$1/2$ the depth of the specimen, and measure the pre-crack length with an optical microscope.
Finally the pre-cracked specimen is broken following the normal SENB technique in 3-point bending. The span was 16 mm, crosshead speed 0.05 mm/min. Fig. 1 shows a schematic of the four specimen testing techniques. The following expressions were used for calculating $K_{le}$ in the present work, and nearly all the symbols are shown in Fig. 1,

(a) DCB

$$K_{le} = \frac{Pa}{[(1-\nu^2)H]^1/2} \left[(1+\delta/a)^9 + 0.41(\delta/a)^6\right]^{1/2}$$

(b) MIT

$$K_{le} = A\sigma_0 C_m^{1/2} + B$$

(c) SIT

$$K_{le} = 0.016(E/H)^{1/4}P(C)^{3/2}$$

(d) SEPB

$$K_{le} = \frac{1.5PLa^{1/2}[1.99-a(1-a)(2.15-3.93a+2.7a^2)]}{W^{3/2}B^{1/2}(1+2a)(1-a)^{3/2}}$$

where, 

- $a$: Pre-crack length
- $A, B$: Empirical constants, $A=2.02, B=0.68$
- $E$: Young's modulus
- $H$: Vickers hardness
- $I$: The moment of inertia of the cantilever beam, $I=bb^3/12$
- $L$: Span of three point bend rig
- $a$: $a=\alpha W$
- $\delta$: The characteristic web length, $\delta=(4\,W/\ell)^{1/4}$
- $\nu$: Poisson's ratio.

We have also determined the transformation zone size around the impression on the indented specimen under various indentation loads from 10 to 80 kg with an optical microscope. The [111] peaks from CuKα X-ray diffraction (XRD) were used to determine the ratio of monoclinic to tetragonal phase. Values of the monoclinic content were determined for the as-sintered, polished and fracture surfaces.

### 3. Results and Discussion

The average and standard deviation of all fracture toughness results are listed in Table 1.

Table 2 is the $K_{le}$ data determined by the DCB technique in different environments. The observations in Table 2 are for steady state values of crack propagation after more than 4 mm of crack extension in the same sample. The value of the $K_{le}$ in air and silicone oil are comparable as would be expected for region III of the $V-K$ diagram. The variation of $K_{le}$ with indentation load by the SIT is shown in Fig. 3.

Table 3 lists the monoclinic content for the fracture surface, as-sintered surface and as-polished surface of these materials, respectively. Fig. 5 is an optical photograph which shows the transformation zone around the indentation of a polished specimen surface. A comparison of the dimensions of the crack length, indentation diagonal and transformation zone size about the indentation are shown in Fig. 6 for the 2Y- and 3Y-TZP materials. These results clearly show similar slope for the transformation zone size and indentation diagonal ($a = Pt^{2/3}$) whereas the crack length as anticipated has a steeper slope ($a = Pt^{2/3}$).

| Environments | $K_{le}$ (MPa m$^{1/2}$) |
|--------------|-------------------------|
|              | 2Y-TZP                  | 3Y-TZP                  |
| Room temperature air | 5.61                    | 4.00                    |
| -65 ~ -70°C    | 7.70                    | 5.36                    |
| Distilled water | 5.22                    |                         |
| Silicone oil  | 3.90                    |                         |
and transformation zone size cross at a load of $\sim 30$ kg. 

$K_{ic}$ values for Y-TZP determined by the four measuring techniques were considerably different.

The single indentation technique was very sensitive to indentation load for 2Y-TZP, for which estimates of $K_{ic}$ decreased sharply with increasing indentation load. Its $K_{ic}$ varied by a factor of 1.9 (from 9.11 to 4.80) for loads from 10 to 80 kg. However, the variation was less obvious for the 3Y-TZP material.

A possible reason for the load dependence of $K_{ic}$ is that the tetragonal phase in 2Y-TZP is more metastable and hence transformable than 3Y-TZP. Evidence for this is available in Table 3 in which the monoclinic phase content on the fracture surface of 2Y-TZP is very high, having reached 39.7%, whereas the content for 3Y-TZP is only 18.3%. This greater metastability of the 2Y-TZP leads to the formation of a larger transformation zone (for 2Y-TZP) about the indentation which inhibits the extension of the radial cracks. This situation is highlighted in Fig. 6, in which we observe a significant dependence of measured $K_{ic}$ on the ratio of transformation zone size to crack length. When the zone size is greater than or comparable to the radial crack length the fracture toughness is grossly overestimated. This feature will be taken up later. These results indicate that for the SIT method the median load to enable comparison with the generally accepted more reliable DCB technique varies with stabilizer content. For the materials in question this load is $\sim 80$ kg for 2Y-TZP and 30 to 40 kg for the 3Y-TZP, respectively. It is generally accepted that the SIT is a more approximate or qualitative estimate of $K_{ic}$ that is particularly useful during processing and optimization of materials, although it is very attractive because of its simplicity. In a previous study of the SIT method by Matsu- moto20 several $K_{ic}$ expressions were used to calculate $K_{ic}$ of Y-TZP and Ce-TZP, respectively. His results for the two materials were very different and followed a similar trend to those outlined here. He recommended that the analyses by Evans and Charles6 and Anstis et al.20 were the most conservative.

Table 3. Monoclinic content of Y-TZP.

| Surface determined | 2Y (Mon %) | 3Y (Mon %) |
|--------------------|------------|------------|
| As-sintered        | 11.6       | <1         |
| As-polished        | 4.1        | <1         |
| Fracture surface   | 39.7       | 18.3       |

Fig. 4. $R$-Curves of 2 and 3 mol% Y-TZP at different temperatures measured using the DCB technique.

Fig. 5. Photograph of the transformation zone around a 20 kg Vickers indentation of a 2Y-TZP specimen.

Fig. 6. Load dependence of the size of the radial cracks, transformation zone size and indentation diagonal for 2Y (a) and 3Y-TZP (b) materials. Note: The crossover of the radial crack lengths and transformation radius with indentation load for the 2Y-TZP material.
ever, no details were given as to the influence of indentation load and whether crack stability was associated with an independent assessment by the DCB method mentioned.

The value of $K_{ic}$ determined by the MIT technique was larger than those given by the other three techniques. These observations are contrary to Masaki's results\(^5\) whose MIT values for $K_{ic}$ of Y-TZP were somewhat smaller than the SIT values. Overestimating the $K_{ic}$ by the MIT technique is probably due to the problem associated with the transformation zone about the indentation as outlined above for the SIT method.

With the SEPB technique one obtains the lowest $K_{ic}$ value, but it agrees with DCB values of $K_{ic}$ very well. This is a conservative result. This approach is very satisfactory as the method is based on the SENB technique which has a strong dependence on the width of the notch. It was reported\(^9\) that the SENB $K_{ic}$ value for 2Y-TZP decreased by a factor of 2.5 (from 17 to 6.99 MPa m\(^{1/2}\)) with decreasing notch width from 1 to 0.1 mm. There is evidence\(^9\) that further reduction of notch width will result in a further reduction of $K_{ic}$. The SEPB technique can now be regarded more accurately as a modified SENB which has minimal notch width. These $K_{ic}$ determined using this technique approaches the most conservative and reliable estimation. To date the stably extended DCB technique is recognized to be a most reliable technique for determining $K_{ic}$ of ceramics. But the SEPB technique is not only simpler and cheaper than the DCB technique, but is also more suitable for application at high temperature. However, there are some problems with the SEPB technique that necessitate further study. One is the influence of residual stress at the mouth of the pre-crack of the specimen on the $K_{ic}$ value. The present authors have obtained evidence that the $K_{ic}$ value of an annealed specimen (3Y-TZP) after introducing a pre-crack is slightly higher (5.5 %) than for the unannealed specimen. This is considered to be associated with the deformation and debris generated during indentation for crack initiation. After annealing because of the reverse monoclinic to tetragonal transformation much of the wedging influence would have decreased leading to a reduced contribution for the pre-existing stress intensity factor and hence a higher $K_{ic}$. Another important factor is the influence of the ratio $(a/w)$ of pre-crack depth $(a)$ to specimen depth $(w)$ on the $K_{ic}$ value. It was found that a high ratio would result in lower $K_{ic}$ values because of reduced stresses (wedging) about the initial row of indentations. However, the very low ratios will also result in lower $K_{ic}$ value because of $R$-Curve behaviour of Y-TZP. Therefore, an appropriate ratio for Y-TZP is in the range 0.2 to 0.6.

The $R$-Curve behaviour of Y-TZP shown in Fig. 4 and determined by the DCB technique shows some interesting trends. At low temperatures (–65 to –70°C) the $R$-Curves are less extensive in terms of the range of $K$ or crack extension distance before attaining a steady state value. By contrast, at room temperature the crack extension to achieve a steady state value is greater than 1 mm. A similar degree of crack extension before achieving a steady state value has been observed in a Ce-TZP material.\(^{30}\) In the latter instance this was rationalized by the larger transformation zone size and observed microracking occurring ahead of the crack tip as well as transformable bridging grains behind the crack tip. It is not anticipated that such a mechanism would be operative for the fine grained Y-TZP materials examined here because of the much smaller transformation zone sizes about the crack tip (<4 μm for the 2Y-TZP). An alternative explanation for the longer crack extension at room temperature before attaining a steady state value may be that moisture assisted stress corrosion cracking is taking place. This would assist in the initiation of crack growth at a much lower stress intensity factor than $K_{ic}$. With continued monotonic loading of the DCB specimen the crack velocity and the $K$ value increases before achieving a steady state $K_{ic}$ value, as observed. Such behaviour would also explain the lower $K_{ic}$ value measured for the 3Y-TZP material in water.

The $K_{ic}$ values at sub-zero temperatures are much higher than those at room temperature. This is because the thermodynamic driving force for the martensitic tetragonal to monoclinic transformation increases with decreasing temperature.\(^{30}\)

The indentation load dependence of the SIT measurement particularly for the 2Y-TZP material requires some additional comments. This dependence is associated with the transformational compressive stresses about the indentation site. It is possible to estimate the magnitude of such stresses if the crack lies completely within the transformation zone size. The basis for such an estimation is similar to that used to determine compressive residual stresses in thermally tempered glass. Marshall and Lawn\(^7\) have provided a simple theoretical basis to estimate such stresses. The stress intensity factor for a radial crack emanating from a pointed indenter may be written as the sum of the loading term, $K_L$, and a crack closing force, $K_R$, namely

$$K_{ic} = K_L + K_R \quad \text{................................(5)}$$

which may be written as

$$K_{ic} = \chi P(a/w - \sigma_{R}(\sigma/E)^{1/2}) \quad \text{................................(6)}$$

where, $\chi=0.016$ $(E/H)^{1/2}$

$\sigma_{R}$: surface compressive stress.

Using the values of $P$, $H$, $E$, $c$ and $K_{ic}$ for 10 and 20 kg indentation loads in 2Y-TZP, leads to values of $\sigma_{R}$ of 450 and 290 MPa, respectively. These values are considerably less than the anticipated surface compressive stress generated by the tetragonal to monoclinic transformation, namely $\sigma_{R} \approx 1140$ MPa, calculated using:

$$\sigma_{R} = \frac{\Delta VP/E}{3(1-\nu)} \quad \text{................................(7)}$$

with typical values of $V_{t}=0.3$, $\Delta V=0.04$ and $\nu=0.3$. There are a number of possible reasons for this large
discrepancy, including the free surface effect enabling much of the transformation dilation to be released normal to the surface as well as the decrease in the amount of transformation with distance from the edge of the indentation. Katagiri et al.28 have used a Raman microprobe technique to plot contours of the extent of transformation about a Vickers hardness impression in a range of Y-TZP materials. They observed that the extent of transformation ($V_f$) decreases from $\sim 0.6$ adjacent to the impression to only $\sim 0.1$ at one third of the diagonal distance from the edge of the impression. A more rigorous fracture mechanics analysis that incorporates such compressive stress gradients is required to more completely rationalize the results shown in Fig. 3.

4. Conclusions

Four methods have been used to determine the fracture toughness of two Y-TZP materials. The relative reliability of these techniques using the DCB method as the standard varied with composition. The SEPB technique is perhaps the most simple and reliable method for determination of $K_{IC}$ of these materials. It has two minor deficiencies, namely, inability to simply monitor any $R$-Curve behaviour (as is the situation for the DCB technique) and the possible presence of residual stresses due to the indentation impressions. The results for both materials were comparable with the DCB values.

The SIT value for $K_{IS}$, particularly for the more metastable 2Y-TZP, was found to be a very sensitive function of the indentation load. The appropriate indentation load is dependent upon the relative sizes of the transformation zone and radial cracks about the indentation impression. We recommend a ratio of zone size to crack length less than 0.5. However, because of its simplicity this technique is still an excellent means of qualitatively ranking the toughness of transformation toughened ceramics.

The MIT approach appears to suffer from many of the problems associated with the SIT procedure outlined above and again overestimates $K_{IC}$.

$R$-Curve behaviour was observed with the DCB technique and this was more extensive at room temperature than at $-65 \sim -70^\circ C$. The most extensive $R$-Curve behaviour at room temperature was associated with subcritical crack growth due to moisture assisted stress corrosion cracking.

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