A METHOD FOR FRACTURE TOUGHNESS TESTING OF CERAMICS - READY FOR STANDARDISATION

R. J. Damani* and R. Danzer*

In an European round robin programme performed by ESIS TC6, five different methods of fracture toughness measuring were compared. Of these a notched beam method (SENB-S) was found to deliver the most reliable results. However, if specimens were prepared with over-dimensioned notches, systematically high values of fracture toughness were determined. A simple explanation of this behaviour is offered. It is shown that fracture toughness may be determined accurately and reproducibly provided the initiating notch has a tip radius of the size of microstructural defects. A method to produce such sharp notches (the SEVNB method) is briefly described. Preliminary results of collaborative evaluations in the framework of ESIS TC6 and VAMAS activities confirm that this method is particularly suited to provide a standard for the fracture toughness testing of ceramic materials.

INTRODUCTION

Although a lack of toughness is the most important deficiency of ceramic materials, a commonly accepted method of fracture toughness measuring has up to now not been established. An ESIS TC6 round robin program (Primas (1), Gstrein (2) and Primas (3)) testing various methods of fracture toughness measurement on different ceramic materials concluded that none were ready for standardisation: the scatter of results was too large within one laboratory as well as between different laboratories.

Generally, fracture toughness is measured by loading a cracked specimen to the point where the crack instably propagates. At this point the stress intensity factor K_I, defined as $K_I = \sigma Y \sqrt{\pi a}$ (where σ is the stress amplitude in an uncracked body at the location of the crack, Y is a geometric factor and a is the length of the crack), equals the fracture toughness K_{Ic}. Consequently K_{Ic} can be measured if the load amplitude, the geometry factor and the crack length can all be determined at the moment of instability.

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* Department of Structural and Functional Ceramics, University of Leoben, A-8700 Leoben, Austria

The production of a well defined crack is a severe problem in fracture toughness testing in ceramics. If the crack is produced by an indent, its shape is not well known (resulting in an uncertain geometry factor) and, since the crack opening near the crack tip is very small, the crack length can not easily be measured. Furthermore, as a consequence of the plastic deformation zone underneath the indent internal stresses occur which are not well known, but add to the applied stresses. Thus, Y, a and even σ can not be exactly determined. These uncertainties result in imprecise testing results for K_{Ic} and indendation methods should only be used for comparitive purposes. If sharp cracks are produced by loading a chevron notch or by bridging a small crack produced by an indent (Amin (4) and Nishida (5)), the crack geometry is not exactly known and the crack length can not precisely be measured. If cracks are simulated by a sharp notch produced by a thin grinding wheel, the length, geometry and loading of the notch are well defined, but the stress concentration at the notch tip is weaker than the that of a crack, which leads to a systematic overestimation of the fracture toughness (Munz (6) and Wang (7)).

An additional problem occurs due to the increasing toughness of many ceramics with the extension of a crack (*R*-curve behaviour): if tests are made on specimens containing cracks which stably propagate – either during the production of the crack or during the test – the toughness may increase and some value on the *R*-curve is measured. This applies to any testing procedure using cracks produced by indents, bridged indents or chevron notches. The shape of the *R*-curve depends on the material investigated and therefore the measured toughness value depends on the method and on the material. To measure the smallest possible toughness value and for a fair comparison of materials a method has to be found where stable crack propagation is negligible. From all methods investigated in the ESIS TC6 round robin only the method using saw cuts fulfils this condition.

Indeed, of all the methods investigated the Single Edge Notch Beam method with a Saw cut notch (SENB-S) ((4) and (6)) delivered the most reproducible results, both within and between laboratories ((1), (2) and (3)). However, it has been observed that if notches are cut too thick, the values of fracture toughness are determined systematically too high. The deviation increases with the notch width (Damani (8)) (Figure 1). In the following, this effect is explained by the occurrence of small microstructural flaws which act as cracks and are influenced by the stress field around the notch. This model allows the definition of a critical notch width. For thinner notches, long cracks can be approximately described by the notch. For wider notches, the stress concentration of the notch is too low to simulate a crack and the fracture toughness is overestimated.

MODEL TO DESCRIBE THE INFLUENCE OF THE NOTCH WIDTH

It is assumed that ahead of the tip of a notch of length a, there exists at least one small crack resulting from the microstructure (e.g., non-sintered grain boundary) or from machining (8) (Figure 2). The stress intensity factor of this crack at the point of instability (i.e., fracture toughness, K_{Ic}) can be approximated by

$$K_{\mathrm{Ic}} \approx K^{*}_{\mathrm{Ic}} \cdot \tanh\left(2Y\sqrt{\delta a/\rho}\right), \qquad (1)$$

where δa is the length of the microcrack, ρ is the radius of the notch tip, and Y is the appropriate geometry factor for the notch crack configuration (8). K^{*}_{Ic} is the critical mode I stress intensity factor of a specimen containing a sharp crack of length $a + \delta a$. Note that $\tanh(x) \rightarrow 1$ for $x \rightarrow \infty$, and therefore, $K_{\mathrm{Ic}} \rightarrow K^{*}_{\mathrm{Ic}}$ for $\delta a >> \rho$: if the microcrack is larger than the notch root radius, the stress field around it is not greatly influenced by the presence of the notch, hence the notch-crack configuration can simply be regarded as a macrocrack of length $a + \delta a$ and standard evaluation techniques of fracture toughness will yield valid results. In general, since δa is much smaller than a, the total crack length may be approximated by a. This approximation is applied in the following.

A critical notch root radius ρ_c can be defined for which the overestimation of the fracture toughness by K^{*}_{Ic} is less than say 5 %, corresponding to the scatter of toughness data. Thus, according to Eq. 1:

$$\rho_c \approx 1.2\, Y^2\, \delta a\,, \qquad (2)$$

and therefore ρ_c approximately equals δa. This simple theory can be used to explain the SENB-S results of the ESIS TC6 round robin ((1), (2) and (3)), see fitting curves in Figure 1. It is interesting to note that δa was always about the grain size irrespective of whether it was estimated from microstructural analysis, see e.g. Figure 3, or by data fitting.

PRODUCING SHARP NOTCHES

From the previous chapter, it can be concluded that the notch root radius for SENB-S fracture toughness testing should be of the order of magnitude of the mean grain size or smaller. Therefore, there is a need for a simple and reliable method to produce notches with a root radius of a few micrometers or even less. A simple method which seems to fulfil the above criterion was recently described by Nishida et al (9), and has provided the impetus for extensive ESIS and VAMAS round robin evaluations. Interestingly, to the authors' best knowledge, the basic idea of this method was first mentioned in an East German patent from 1981 (DDR Patentschrift (10)) but was never generally applied in the West. A brief description is given below.

A notch of between 100 μm to 150 μm width is ground into a small beam using a conventional thin diamond cutting wheel. Then the tip of this notch is sharpened by careful polishing with a conventional razor blade (of the European type; they are ground to a finer cutting angle than some American types) and diamond paste thinned with a liquid. A diamond grit size of 0.1 µm has been proven to be suitable, although trials in other laboratories have been successfully conducted with grit sizes of up to 1 µm. The first notch acts as a guide for the razor blade and provides the greater part of the depth required. In principle, it is convenient, but not necessary. The process can be done by hand, but better results are achieved by using an automated oscillating polishing jig. Using this method it is possible to reproducibly achieve notch tip radii of less than 3 µm even in hard materials, see Figure 4, (Damani (11)).

SEVNB TESTING AND RESULTS

In the polished notch modification of the SENB-S method – the Single Edge V-Notched Beam (SEVNB) – a simple rectangular beam is notched as described above and then loaded to fracture in bending. Most tests are made with standardised bending specimens (3 x 4 x 45 mm^3), but other geometries are acceptable provided thin beam theory is applicable. Loading is done typically in 3 or 4-point bending; in our laboratory a 20/40 mm 4-point bend configuration is generally applied. The size of the notch (i.e., the assumed crack length, *a*) may be determined before by microscopy. Testing and evaluation are conducted according to the standard procedures for the SENB-S method (11).

SEVNB-trials were performed in the last three years by a large number of laboratories. Collaborative activities have been organised by Jakob Kübler in the framework of VAMAS and ESIS preliminary results are reported by Kübler (12). In a current round robin data from 36 laboratories have already been collected on tests with silicon nitride and alumina ceramics. Other materials are still under investigation. Test results clearly exhibit reduced scatter, both within and between laboratories. When viewed in the framework of previous investigations by ESIS TC6 these results are not surprising since, if the thin notch is prepared properly and is thin enough to simulate a crack: the crack length and geometry are easy to define and measure; the point of instability is also easily defined; residual stresses from preparation are minimal; the crack flanks are stress free and there should be no process zone ahead of the notch tip. Consequently values measured should be towards the beginning of any *R*-curve.

CONCLUSIONS

1. The SEVNB fracture toughness test is simple and practicable enough to be applied in most laboratories: all required parameters are easily defined or determined and sharp notches are quickly made.

2. The method provides reliable results with exceedingly low scatter for ceramic materials with a grain diameter of greater than about 2 µm. For finer materials the scatter may remain low but the fracture toughness may be overestimated.

3. The method should give a toughness value at the beginning of any R-curve due to the traction free starting crack surfaces. This enables a fair comparison of different materials.
4. The SEVNB method is ideally suited as a standard for the fracture toughness testing of ceramic materials.

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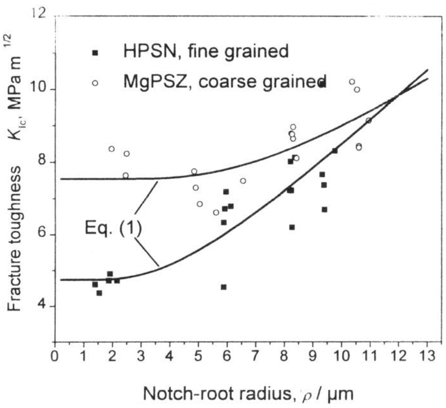

Figure 1 SENB-S fracture toughness testing results

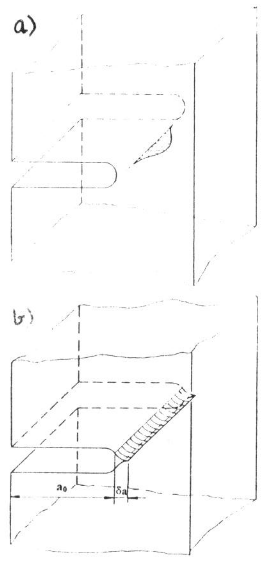

Figure 2 Cracks ahead of the notch tip: a) half penny, b) straight through edge crack

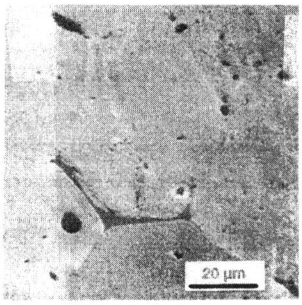

Figure 3 Non sintered grain boundary in MgPSZ which may act as notch tip cracks

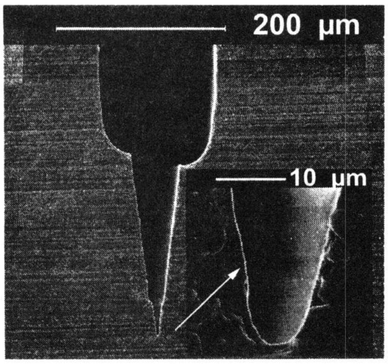

Figure 4 Sharp notch in a bending beam of HPSN. Inset: notch tip