

Polished Notch Modification of SENB-S Fracture Toughness Testing

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Abstract

In fracture toughness testing it is common for reasons of simplicity and reproducibility to use notches to approximate sharp cracks. However, a dependence of measured fracture toughness (K_{Ic}) on notch-root radius is observed. This can be explained as a consequence of the interaction of a distorted stress field with material flaws in front of a notch. A relationship to quantify this effect is presented and examined. It is shown that to measure true fracture toughness sharp notches of the size of microstructural features are required. A simple method to make very sharp notches is presented. Fracture toughness values determined with sharp-notched samples are compared with the results of experiments with conventional sawn-in notches. It is shown that sharp notches deliver considerably lower, more accurate and reproducible values of K_{Ic} for materials with fine microstructures. These values are thought to lie at the beginning of any R-curve. © 1997 Elsevier Science Limited.

1 Introduction

The very low plasticity of ceramic materials at room temperature allows the application of linear elastic fracture mechanics (LEFM) in quantifying and describing their fracture characteristics. One principal assumption of LEFM is that fracture initiates from an atomically sharp two-dimensional crack.¹ However, in fracture toughness testing it is common for reasons of simplicity and reproducibility to use notches to approximate such cracks.

[†]In Damani *et al.* K_{Ic} and K_{Ic}^{app} were termed K_T and K_{Ic}^{meas} , respectively.

Hence, because of the lower stress concentration ahead of a notch, as compared to a truly sharp crack, a greater force is required to cause a notch tip crack to extend. This deviation increases with increasing notch width and results in the commonly reported dependence of fracture toughness (K_{Ic}) on notch-root radius.²

In a previous paper, a theory to quantify the notch-root radius effect was presented. It was assumed that crack-like flaws already exist and are stochastically distributed in the microstructure. These flaws can act as fracture initiating cracks ahead of the notch-root. Fracture occurs, if the local stress intensity factor, as influenced by the notch, exceeds a critical value. The true crack tip stress intensity factor as seen by a small crack in front of a notch, K_I can be related to the apparent stress intensity factor calculated for a sharp crack, with the length of the notch, commonly calculated using standard formulae, K_I^{app} , by

$$K_I \approx K_I^{app} \cdot \tanh(2Y\sqrt{\delta a/\rho}), \quad (1)$$

where δa is the size of the fracture-initiating defect and Y is an appropriate geometric correction factor which ranges between $2/\pi$ for penny shaped starting cracks and 1.12 for edge cracks, as detailed in Damani *et al.*³ The same relationship holds for the critical value of the stress intensity factor, K_{Ic} , when the maximum force before fracture is used to calculate $K_I^{app†}$. Note that for $\delta a \geq \rho$, $\tanh(2Y\sqrt{\delta a/\rho})$ tends to unity. Thus, to measure true values of fracture toughness K_{Ic} the notch-root radius should be equal to or smaller than the size of the fracture initiating flaws. In general, this flaw size is related to the microstructure, and especially often to the grain size. For coarse-grained materials with grain size greater than $100 \mu\text{m}$, notches sharp enough to measure true fracture

toughness can be easily machined. For fine-grained materials this is hardly possible with conventional techniques if the advantages of using notches are to be retained. In the following section a simple method to make just such notches is described. Fracture toughness measured with these sharp notches is compared to values from standard saw-cut experiments and the validity of the approximation (1) is examined.

2 Experimental procedures

Two sets of experiments were made. Small beam samples were prepared from the same five materials in both cases: alumina (Al_2O_3) with mean grain diameter of about $10\ \mu\text{m}$ and relatively high porosity; tetragonal zirconia polycrystal (TZP) with sub-micron grains and very little porosity; magnesia partially stabilised zirconia (MgPSZ) with mean grain diameter of about $40\ \mu\text{m}$ and numerous open grain boundaries; hot pressed silicon nitride (HPSN) with low porosity and needle-like grains of up to $3\ \mu\text{m}$ in their long axis; and sintered silicon carbide (SSiC) with a bi-modal microstructure consisting of isolated large grains of about $30\ \mu\text{m}$ diameter surrounded by a highly porous region, which sit in a fine-grained low porosity matrix of about $7\ \mu\text{m}$ grain-size.

In the first set of experiments fracture toughness tests were carried out in four-point bending using the single edge notch bend – saw cut (SENB-S) method. Notches of different notch-root radii were cut into the small beam samples using a high-speed rotary saw. Notch width and depth were measured using an optical microscope on both sides of a sample and then averaged. Fracture toughness was calculated from the maximum load before failure. The results of these tests exhibited a dependency on the notch-root radius. These results have been comprehensively expressed in the report of an ESIS Round Robin⁴ as well as in Damani *et al.*

In the second set of experiments, described here, small beam samples with very fine notches were used. The procedure for introducing sharp notches is an adaptation of that used by Nishida *et al.*⁵ It essentially involves double notching: first a guiding notch of between 100 and $150\ \mu\text{m}$ width is sawn into a small beam sample to a relative notch depth, α , of about 0.45 ($\alpha = a/W$, where a is the notch depth and W is the height of the beam) then the tip of this pre-notch is extended to $\alpha \approx 0.5$ and sharpened by careful polishing with a conventional razor blade and diamond paste suitably thinned

with alcohol. A fine paste was chosen to avoid causing scratches larger than intrinsic material defects. The diamond paste used here had an average grain size of $0.1\ \mu\text{m}$. It was found that any commercially available razor blade is suitable. Care should be taken that the second, sharp notch is deep enough to be out of the zone of influence of the pre-notch. According to eqn (1) the second notch should be at least about half as deep as the first notch is wide.

The process was easily quasi-automated by mounting the sample on a railed carriage driven (oscillated) by an electric motor (Fig. 1). A spring-mounted blade was lowered into the pre-notch which acted as a stabilising guide. Thinned diamond paste was applied in the pre-notch and on the blade. The blades had to be exchanged during polishing to achieve the sharpest possible notches. It was possible to easily and quickly introduce notches of root radius less than $5\ \mu\text{m}$ in all the materials. Figure 2 shows a relatively good double notch with notch tip diameter less than $5\ \mu\text{m}$.

3 Results

Results from both series of tests are shown in Fig. 3. The lines show the best fit of relation (1) with K_{Ic} and δa as free parameters. In the case of TZP fitting was also carried out with a fixed value of δa . The value of Y was fixed as 1.12 or $2/\pi$, whichever seemed most appropriate. The dashed lines, as reported in Damani *et al.*, refer to the best fit to data from the first set of experiments (saw-cut notches) only. They have been extrapolated to provide a prediction of fracture toughness that should be determined from the sharp notch experiments. The solid lines show the best fit to all data. The fitted values of K_{Ic} and δa are summarized in Table 1.

It is clearly seen that for all materials except MgPSZ the polished notch modification of the SENB method delivered lower values of fracture toughness than the saw cut method, although for alumina the difference was marginal. For MgPSZ the results were surprisingly high, although, in absolute terms the deviation from the predicted value is still small. It is possible that the transformation of toughening precipitates ahead of the notch tip during sawing, i.e. before they can contribute to resisting crack initiation and propagation, may be responsible.

The greatest difference in values measured using saw-cuts and sharp notches is, as would be expected,

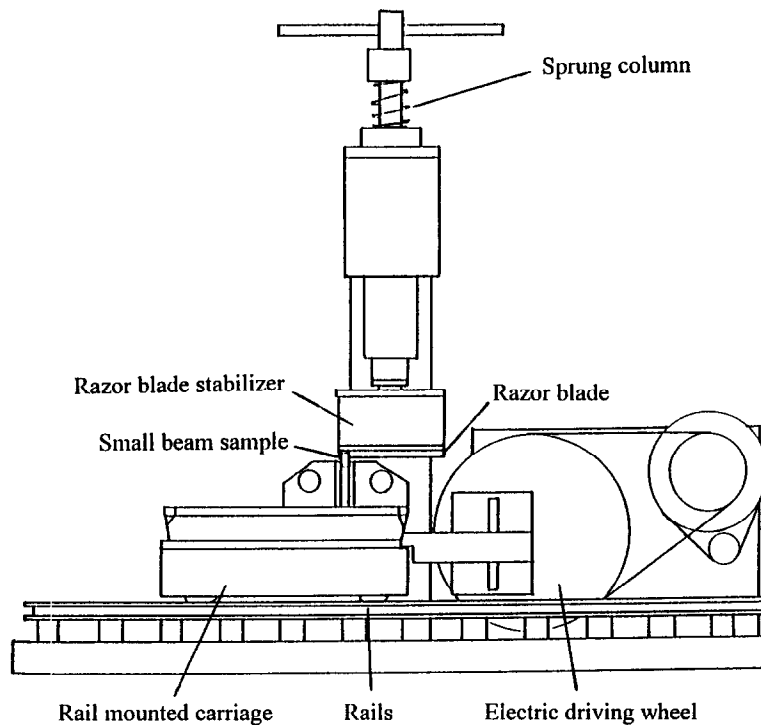


Fig. 1. Quasi-automated rail mounted jig for the polishing-in of sharp notches into small beam ceramic samples.

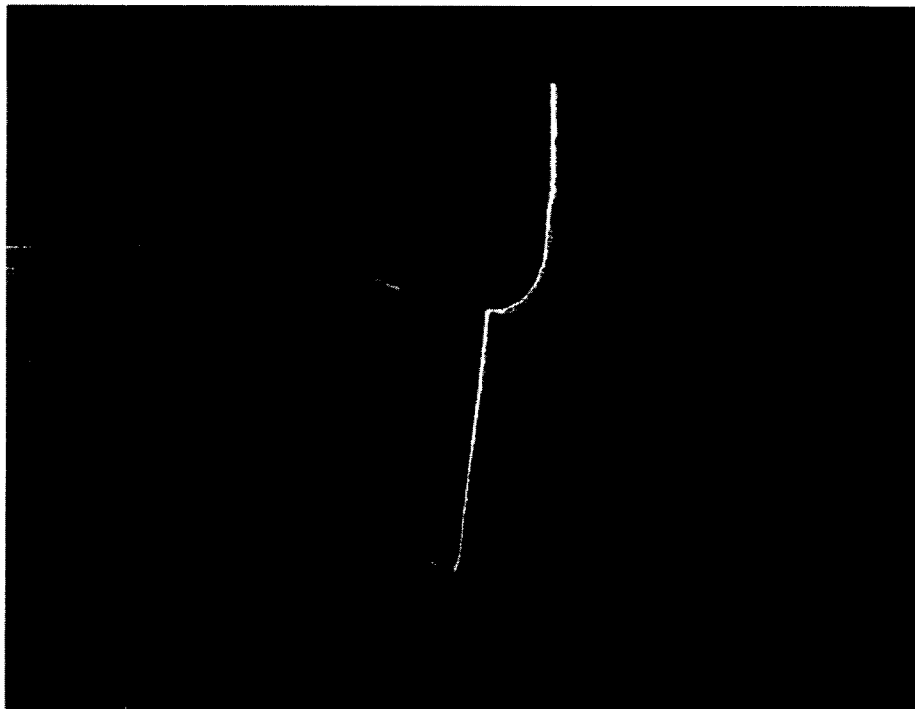


Fig. 2. Section view of a typical sharp (double) notch in HPSN. The notch-root radius is approximately two micro-metres.

ted from eqn (1), seen in the sub-micron grain-sized TZP material. Two kinds of fit of eqn (1) were made for the saw-cut experiments (Table 1): once with K_{Ic} and δa as free parameters, and a second time with fixed value of fracture initiating defect size $\delta a = 10 \mu\text{m}$, as might be expected from scratches

from the diamond grit of the saw blades. The measured values for sharp notches are in between the two extrapolated predictions. In any case, it is clearly seen that even notches of diameter of less than $100 \mu\text{m}$ may lead to gross over-estimation of fracture toughness when applied to very fine materials.

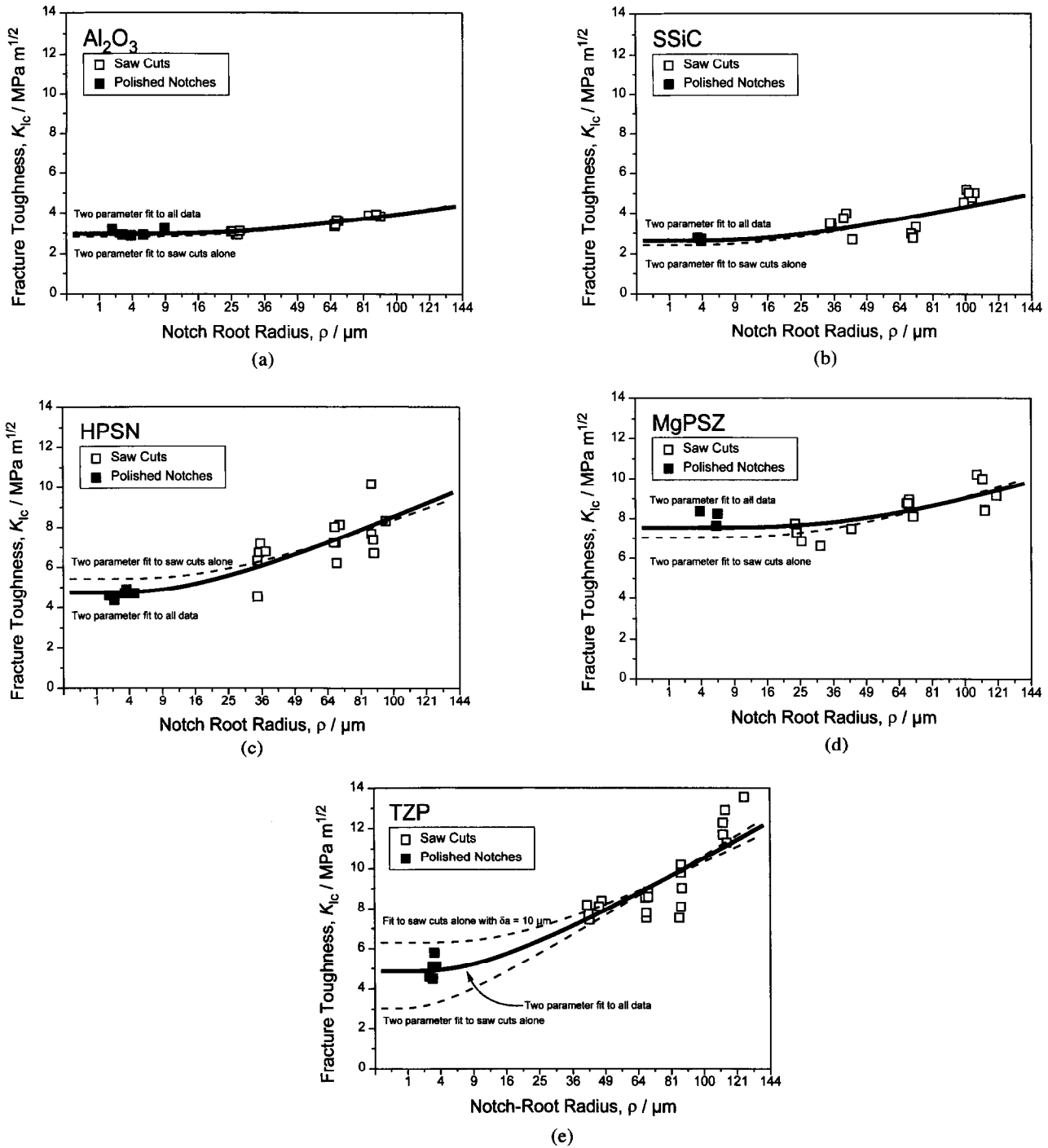


Fig. 3. Graphs of results of fracture toughness tests with varying notch width for all materials: (a) for Al_2O_3 ; (b) for SSiC; (c) for HPSN; (d) for MgPSZ and (e) for TZP.

Table 1. Summary of the results of the best fit of eqn (1) to measured data and mean values of fracture toughness determined by sharp notch experiments

	Fixed parameters	Fit to saw-cut data only		Fit to all data		Mean measurement $K_{Ic}/\text{MPa m}^{1/2}$
		$K_{Ic}/\text{MPa m}^{1/2}$	$\delta a/\mu\text{m}$	$K_{Ic}/\text{MPa m}^{1/2}$	$\delta a/\mu\text{m}$	
Al_2O_3	$Y = 1.12$	2.8	16	3.0	20	3.04
SSiC	$Y = 2/\pi$	2.4	24	2.6	31	2.70
HPSN	$Y = 1.12$	5.4	12	4.7	8	4.66
MgPSZ	$Y = 1.12$	7.1	21	7.5	29	8.07
TZP	$Y = 1.12$	3.0	2	4.9	5	5.02
TZP	$Y = 1.12,$ $\delta a = 10 \mu\text{m}$	6.3	—			

The outstanding feature of all the tests is the exceptionally low scatter in the values measured. This is particularly striking in the case of HPSN, where the scatter of values from saw-cut experiments alone was very high.

In the case of the three non-transforming materials (Al_2O_3 , SiC and HPSN) the measured values of fracture toughness correlate very well to the values predicted by the extrapolated best fit of eqn (1) to the data from saw-cut experiments. For the transforming materials the deviation is greater, and the scatter is higher, but the trend remains good. Further weight is lent to the relationship (1) since the mathematically optimized fitting parameter δa , indicating the size of the fracture-initiating defect, corresponds well to the size of microstructural features actually observed.

Another important aspect of the presented method may be its application to materials which exhibit *R*-curve behaviour. Increasing crack resistance as a crack grows may be partly responsible for the high scatter of fracture toughness values determined with different methods, since different measurement conditions could lead to different parts of the *R*-curve being measured. With the sharp notch method it is certain that the values obtained are equal to, or at least very close to the initial value of the crack resistance curve, R_0 , i.e. the minimum value of fracture toughness. This value may have special significance in describing material characteristics since natural flaws are small and, hence, virgin materials are naturally also at the beginning of the *R*-curve.

4 Concluding remarks

The polished notch modification of the SENB method of fracture toughness testing has been shown to be relatively quick and practicable in standard laboratories. The equipment required is simple and cheap, and the overall experimental effort is low.

The method provides accurate and reproducible results for both non-transforming and transforming materials. The scatter of results is particularly low and this can be understood in terms of the presented theory. In an ESIS Round Robin on fracture toughness testing the SENB – *S* method was seen to exhibit the lowest scatter both within and especially between laboratories. It is expected that the polished notch modification of this method will result in a still further decrease in scatter.

The results confirm the validity of the continuum mechanics based expression presented here and in Damani *et al.* for the explanation of the notch-root radius dependence of measured values of fracture toughness. It has also been clearly demonstrated that the use of even relatively thin notches ($\rho < 50 \mu\text{m}$) can still lead to gross over-estimation of the fracture toughness of very fine grained low porosity materials. An accurate estimation of the true fracture toughness value may be obtained by fitting the presented relationship to data from wide-notch experiments.

Since the notch is polished in, the crack flanks should be free of tractions, there should be almost no crack-front transformation zone, and negligible residual stresses. The values of fracture toughness obtained from this modified SENB method are therefore close to R_0 , i.e. they are at the beginning of any *R*-curve. Thus, a lower limit of toughness, 'true' K_{Ic} is determined. Since fracture usually initiates from small natural flaws, this value may be of greater significance for use as a basis for safe design than values on the rising or plateau part of the *R*-curve as determined by many other methods.

Of particular technological importance is the suitability of this method, justified by the above mentioned advantages, for the standardisation of ceramics fracture toughness testing.

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