Fractography and Fracture Toughness Measurement

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Abstract

Using a variety of advanced ceramic materials, a comparison has been conducted of fracture toughness test methods using the single edge vee-notch beam method and the surface crack in flexure method, the latter restricted to optical fractography. Good agreement has been found between the two methods on materials which were amenable to the SCF method. It has further been shown that the SEVNB method can produce reliable results on materials to which the SCF method is not readily applicable.

Introduction

Over the past three decades a number of methods for fracture toughness determination have been proposed, and of these, three methods were initially standardized as ASTM standards within document ASTM C1421. They were the single-edge pre-cracked beam method (SEPB), the chevron notched beam method (CNB) and the surface crack in flexure method (SCF). All are now ISO standards for advanced ceramics. These methods were considered to possess good crack geometries with well-defined stress intensity factors based on sound fracture mechanics principles. They had the additional advantage that they could be conducted on conventional flexural strength test-pieces, and thus were economical in terms of material quantity and fabrication ease. However, each of them has some limitations for the average laboratory. In the SEPB test, there is a need to create a popped-in pre-crack, typically from one or more indentations, with an acceptable (valid) geometry, using a bridge pre-cracking method. This requires a pre-cracking fixture with good alignment and appropriate compliance, operator skill and experience, and some adjustment of conditions to suit the properties of the test material. In the CNB test, the ideal notch shape is readily achieved by diamond sawing, but controlled initiation of a sharp crack from the notch tip can be problematic in some materials. A stiff load train is highly desirable to maximize the chances of obtaining controlled crack growth and valid results. In the SCF method, results are critically dependent on being able to obtain a defined pseudo-elliptical crack by indentation and to identify clearly its boundary. This works reasonably well in fine-grained materials, but in many technical materials problems of crack surface roughness make the easy delineation of the crack boundary impossible, even with technique adjustments intended to reveal it more clearly. However, its key advantage is that test results are a close approximation to short-crack toughness relevant to small flaws and cracks, rather than to long cracks. Additionally, each of these methods can be biased differently if the material shows any R-curve effect. Thus, each of the methods has its own operational complexities and limitations [1].

In order to overcome some of these issues, a development of the traditional, but not generally accepted single-edge notched beam (SENB) method, but with a sharpened notch tip more representative of a sharp crack was first proposed in Japan [2] by employing a specially sharpened diamond slitting wheel in which the blade edge was honed to a radius of 10 μm or less. Such a blade, unfortunately, has a very limited life, and although it was found to give good results, it was not a practical option for routine tests. A technique involving honing the notch with a razor blade and diamond paste was first put forward in a patent [3], and later developed in Japan [4] and then in Europe [5]. Using a conventionally sawn notch as a guide, a standard razor blade is reciprocated across a batch of test-pieces, deepening the notch and sharpening it. Typically, the notch would be roughed out with 6 μm diamond paste, and finished with a fresh blade and 3 μm diamond paste or finer. The degree of sharpening achievable appears to depend on the consistency of the reciprocating motion (a carefully constructed machine is better than attempting this by hand) and on the grain structure of the material. The release of individual large grains in an alumina, for example, will limit the sharpness of the notch compared with the achievable effect in a fine-grained silicon nitride or yttria-stabilised zirconia (Y-TZP), where a root radius of 2 μm can be obtained under optimum conditions. It is then argued that the sharp honed notch is much more like a crack than a blunt sawn notch, but after fracture as in the SEPB method, the fracture toughness can be determined using the same equations. Kübler [6, 7] organised an ESIS/VAMAS[1] round robin to evaluate the consistency of the method, and the closeness of approach to true sharp-crack toughness values. Using a 99.8% alumina (grain size >10 μm), a 99.9% alumina (grain size 1.7 μm), a gas-pressure sintered silicon nitride, a sintered silicon carbide, and a Y-TZP, a comparison was developed between this single-edge vee-notched beam (SEVNB) technique and the other three techniques listed above which had been the subject of a previous round robin on the same materials. Despite the novelty of the technique to most of the participants, the results overall were remarkably consistent, both in terms of within-lab and between-lab variations. Participants made the notches either by hand, or using a simple reciprocating machine, and mostly achieved good results, although not unexpectedly the machine-produced notches were generally much sharper than the hand-produced ones. In the aluminas and Y-TZP, there was some subcritical crack growth before failure which could be detected by slight non-linearity of the force-displacement plots obtained by some participants [8]. This implies that at peak force, the crack length is longer than the notch length by an amount that needs to be determined fractographically. This is not always straightforward, and requires some skill at interpreting the appearance of the fracture faces adjacent to the notch, especially in the coarser grained materials. Correction for the crack extension gives a higher toughness than that calculated from the original notch. This behaviour, which is also seen in the SEPB and SCF test methods, is found to be test environment dependent, as might be expected. Fett [9] has reviewed the issue of initiation of a crack from the root of the notch, and has provided a means of correcting the apparent toughness results obtained if crack extension is ignored.

Despite these small uncertainties, a standardized procedure was developed, first in Europe (CEN TS 14425-5) but since becoming international (ISO DIS 23146). During this process there was a call to demonstrate further the equivalence of the SEVNB method to other methods. This paper reports some comparative evaluations with the SCF method on a variety of material classes.

Test procedures

Test-pieces for SEVNB tests were either standard 4 x 3 x >45 mm flexural test bars or half-bars from previous tests. They were notched by machine on a 3 mm wide face to a depth of 0.6 to 1.0 mm using the procedure described above. Notch tip radii were examined microscopically for acceptability, and generally were found to be less than 10 μm, within the guidelines set by the standards. At least five test-pieces were used.

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[1] European Structural Integrity Society / Versailles Agreement on Advanced Materials and Standards

Test-pieces for SCF tests were half-bars from previous tests. They were indented on a 4 mm wide face using a Knoop diamond on a standard Vickers hardness machine at force levels derived from masses of 5 kg and/or 10 kg. The indentation long diagonal was measured, from which the minimum depth of indentation face removal was computed (≥ 5 times the indentation depth, which is one thirtieth of the long diagonal length: ASTM C1422, ISO 18756). This amount of material was removed by light hand-held grinding on a 20 µm diamond lap, monitoring the removal depth using a micrometer. At least five test-pieces were used.

The test-pieces were fractured in one of several four-point flexure jigs with 40/20 mm, 20/10 mm, or 20/6.7 mm spans in an Instron 4505 universal testing machine. Cross-head displacement speeds of 0.5 mm/min (40 mm span) or 0.2 mm/mm (20 mm span) were used. Peak fracture forces were determined. Notch depths at both ends of the notch on both sides of the fractured SEVNB specimens were measured using a calibrated X-Y stage on a Nikon measuring microscope. SCF pre-cracks were identified fractographically and their dimensions were measured directly on the same microscope, or on photographs captured using this microscope and calibrated using a certified stage graticule at the same magnifications.

Test results on high-purity alumina

Three batches of Vitox®[2] test-pieces previously subjected to a test-house evaluation of toughness using a non-standard sawn-notch single edge notched beam (SENB) method were tested. SEVNB notches could be readily produced with a tip radius of less than 5 µm. SCF pre-cracks could be readily detected in the optical microscope using normal illumination because of reflectivity differences between the different zones of the fracture surface. The pop-in crack, which is dominantly transgranular in appearance, appears brightly speckled, while a subcritical growth zone appears dark as a result of its intergranular nature (Fig. 1). The initial propagation zone during the fracture test appears bright, turning duller as the crack accelerates. The boundary between the subcritical growth and propagation zones was thus clearly defined, and taken as the true flaw shape for toughness analysis. Results are summarized in Table 1. There is good agreement between the SEVNB and the SCF methods. The reported SENB values were, by contrast, distinctly lower.

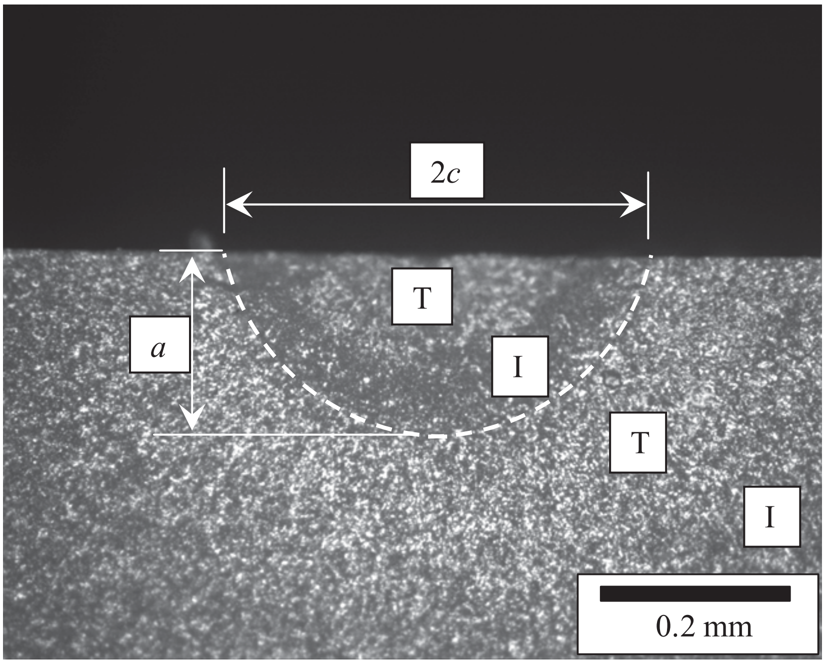

Figure 1: HK10 pre-crack in Vitox® alumina, normal illumination, showing zones of different reflectivity and measurements taken (I = intergranular region, T = transgranular region, pre-crack size 2*c* by *a*)

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[2] Morgan Advanced Ceramics Ltd, Rugby

Table 1: Comparison of SENB, SEVNB and SCF

Batch	Apparent fracture toughness, MPa $m^{1/2}$		
	Original SENB	SEVNB	SCF, HK5
Batch 1	2.65 ± 0.04	3.80 ± 0.81	3.86 ± 0.01
Batch 2	2.61 ± 0.02	3.75 ± 0.25	3.69 ± 0.17
Batch 3	2.50 ± 0.27	3.34 ± 0.50 (3.52 ± 0.29 removing one outlier)	3.74 ± 0.14

An investigation into the SENB fracture surfaces showed evidence of significant subcritical crack growth before fast fracture. Two examples of this are shown in Fig. 2. The effective extension of the notch (~ 0.4 mm) is sufficient to explain the difference (~ 0.9 MPa $m^{1/2}$) between SENB and SEVNB/SCF results. There was no evidence for any similar extension from the SEVNB notches. It is unclear why the crack extension occurs in SENB; it may possibly have been a result of pre-existing machining flaws at the notch root and a slow stressing rate.

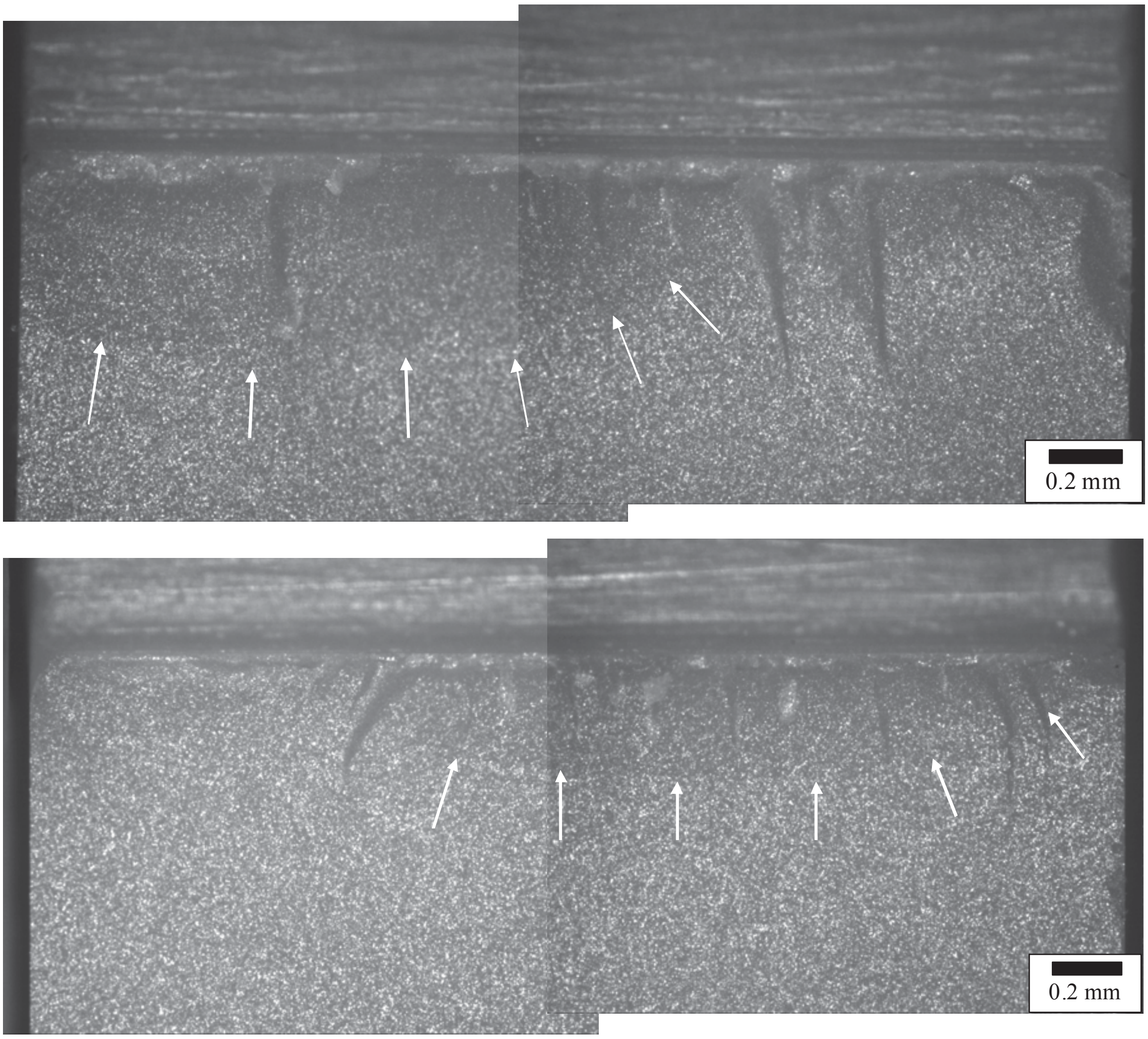

Figure 2: Subcritical crack growth zones seen in alumina SENB test-pieces from (upper) batch 1, (lower) batch 2. The wake hackle suggests severe machining damage at the notch root.

Test results on zirconia-strengthened alumina (ZTA)

An equivalent set of tests was performed on an experimental ZTA material. In this case the SCF pre-cracks did not display the same convenient features as in alumina alone, and it proved to be more difficult to be certain of the pre-crack boundaries. Some guess-work was required, so in this case, the SCF tests were repeated using indentation with the test-piece tilted lengthwise at an angle of about 1°, as recommended by the standards. This should produce a pop-in pre-crack at an angle to the final fracture plane, making the boundary easier to see, but not significantly affecting the stress intensity factor. Figure 3 shows the effect of tilting on optical visibility. The test results are shown in Table 2, and demonstrate close equivalence of the two methods with a reduction in scatter with indentation tilting. There was no evidence of significant subcritical growth in either method, either fractographically or in force-displacement behaviour.

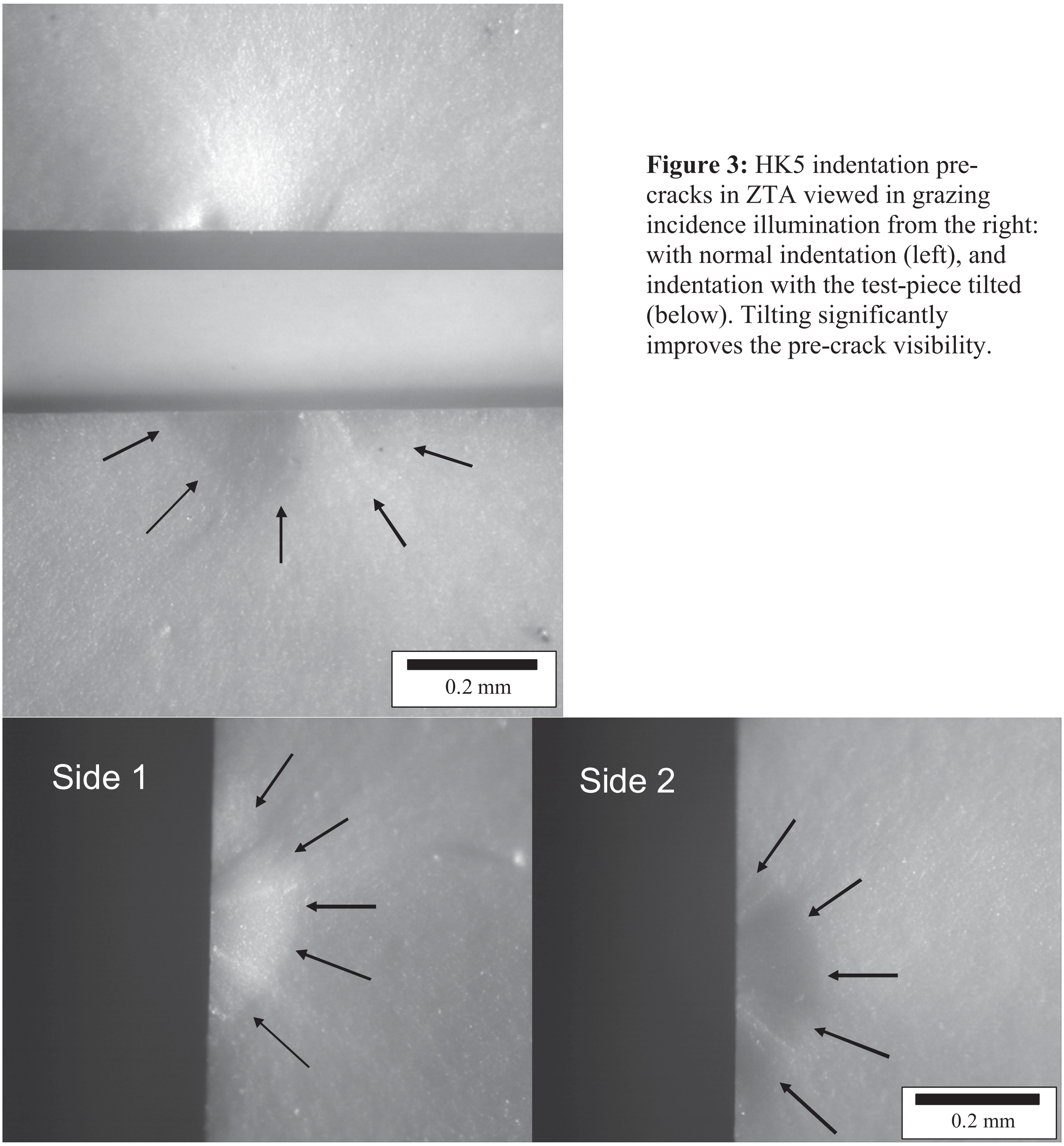

Figure 3: HK5 indentation pre-cracks in ZTA viewed in grazing incidence illumination from the right: with normal indentation (left), and indentation with the test-piece tilted (below). Tilting significantly improves the pre-crack visibility.

Table 2: Fracture toughness determinations on ZTA

Batch	Apparent fracture toughness, MPa $m^{1/2}$		
	SEVNB	SCF, HK5, normal indentation, estimated pre-crack boundary	SCF, HK5, angled indentation
ZTA	4.22 ± 0.49	3.98 ± 0.33	4.13 ± 0.21

Test results on armour ceramics

In a separate exercise, a comparison was made between the SEVNB and SCF methods on a range of ceramic armour materials. These materials do not all have a fine grained microstructure, but provide a wider range of characteristics and toughness levels to test out a method comparison. The materials are listed in Table 3.

Table 3: Ceramic armour materials evaluated[3]

No.	Type	Grade and source
1	SiC	PS 10777, Morgan Advanced Ceramics, USA
2	SiC	PS 5000, Morgan Advanced Ceramics, USA
3	SiC	EKASiC T (batch 1), ESK, Germany
4	SiC	EKASiC T (batch 2), ESK, Germany
5	B_4C	Hot-pressed, Cercom, USA
6	Al_2O_3	96%, white, ETEC, Germany
7	Al_2O_3	Sintox FA, 95%, pink, Morgan Advanced Ceramics, UK
8	WC/W_2C	Hot-pressed, Cercom, USA (evaluated in [10])
9	Si_3N_4	Ceralloy 147A, hot-pressed, Ceradyne, USA
10	SiC	SIKA, Saint-Gobain, USA

Standard 3 x 4 x >45 mm test-pieces were machined, and notched on a 3 mm wide face for the SEVNB tests, made over 40/20 mm spans in four-point flexure. The broken halves were then subjected to HK5 indentations on a 4 mm wide face for the SCF tests, using both a 1° lengthwise tilt of the test-pieces, and also providing a small deliberate in-plane twist. The indentation long diagonals were measured and the depths of required surface removal were determined in the usual way. For the particularly hard materials, a 60 µm diamond lap was used initially, transferring to a 20 µm lap for the final stages of removal. They were then tested over a 20/6.7 mm spans in four-point flexure. Initially, it was found that HK5 pre-cracks proved difficult to see in materials 3, 4, 9 and 10, while materials 5 and 6 did not fail from the pre-cracks. The tests on all materials were therefore repeated using HK10 indentations, but generally this resulted in no improvement in optical visibility. Examples of the readily visible and indeterminate pre-cracks are shown in Figs. 4 and 5 respectively. No attempts so far have been made to employ reflective metallic coatings or scanning electron microscopy for pre-crack identification because of the additional time and effort involved.

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[3] Materials supplied by Advanced Defence Materials Ltd. Test-pieces machined by Morgan Advanced Ceramics Ltd.

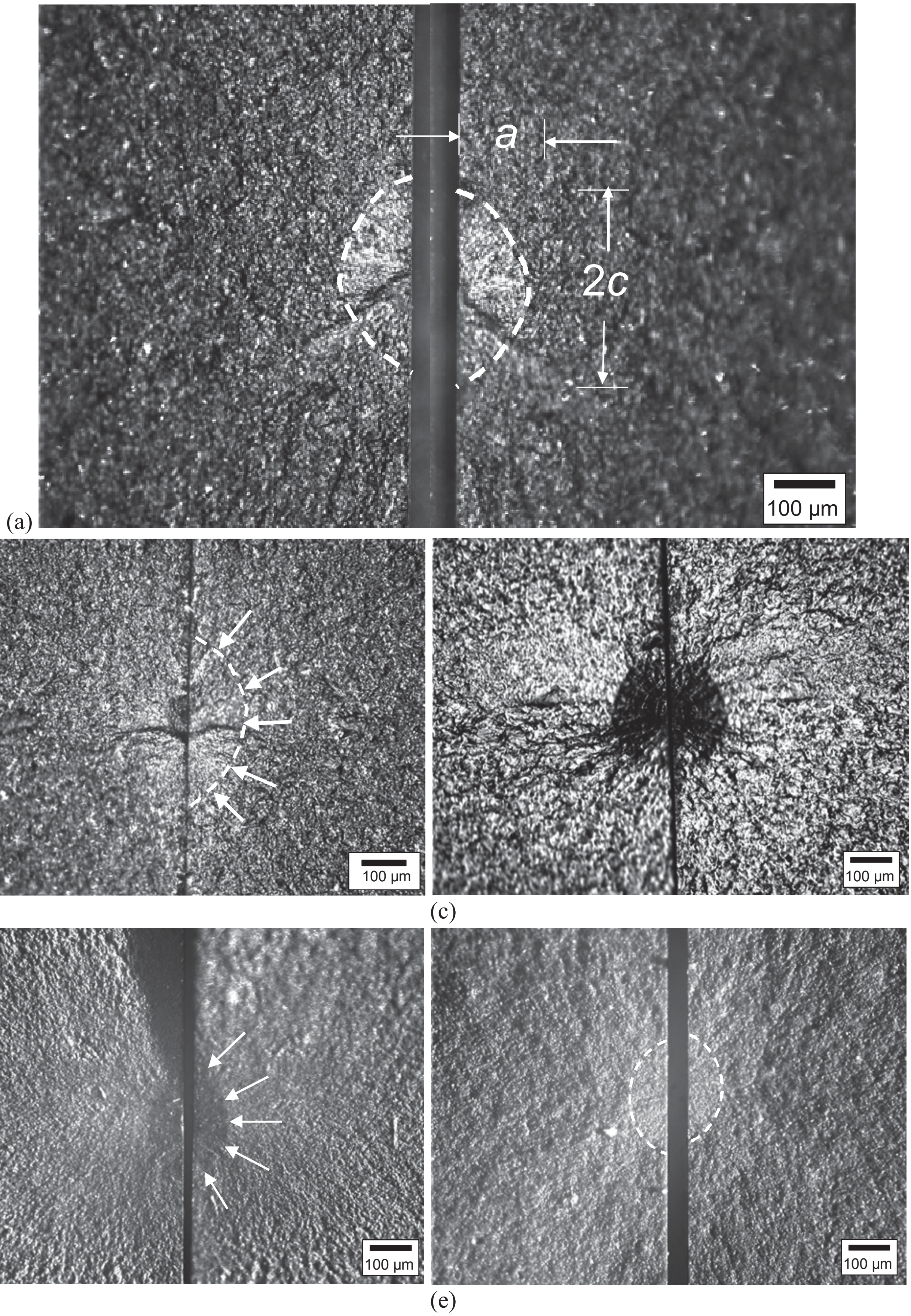
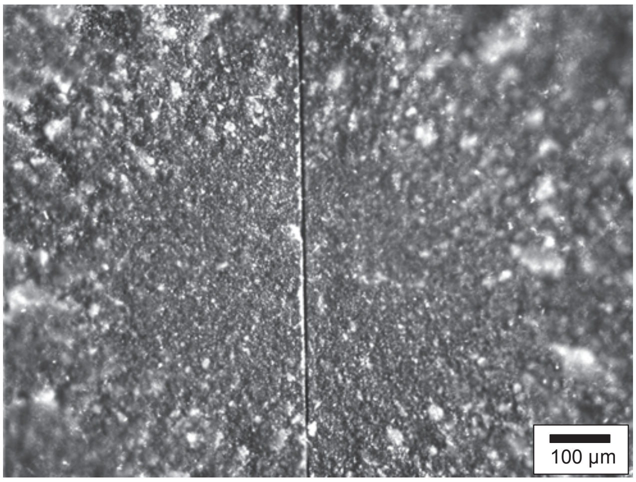


Figure 4: Examples of optical imaging of HK5 SCF pre-cracks in (a) material 3, EKASiC T SiC; (b) material 1, PS10777 SiC; (c) material 5, B_4C; (d), material 8, WC/W_2C; (e) material 9, Ceralloy 147A silicon nitride.



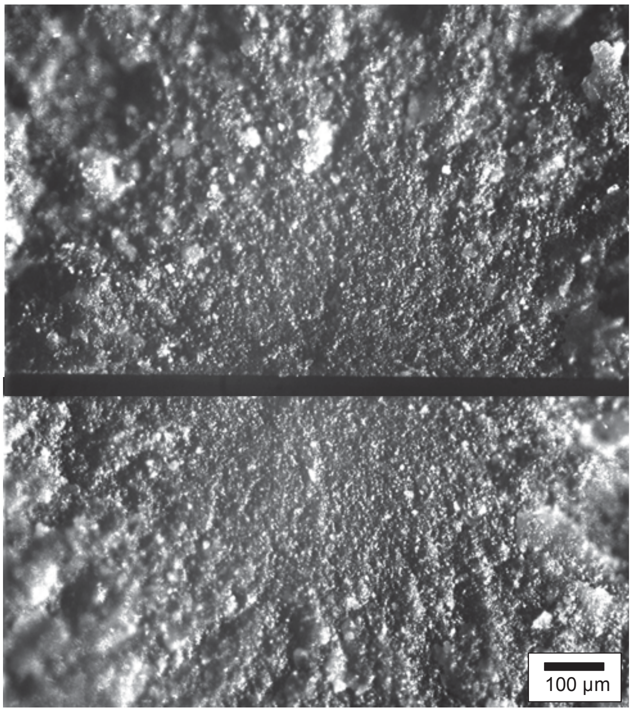


Figure 5: Indeterminate HK5 inclined SCF pre-crack in material 10, SIKA SiC using either (left) normal illumination or (right) grazing illumination.

The test results are shown in Table 4 and Fig. 6. There is generally a very good correlation between the SEVNB and the SCF data where the latter could be obtained. The spread of results in both cases is small, as has been previously recognized, although generally, there is a little more scatter with the SEVNB values.

Table 4: Results of fracture toughness tests

Material	Fracture toughness, MPa m$^{1/2}$		
	SCF - HK5	SCF - HK10	SEVNB
#1 PS10777 SiC	2.88 ± 0.10	2.97 ± 0.08	3.01 ± 0.32
#2 PS5000, SiC	2.78 ± 0.06	2.92 ± 0.19	2.70 ± 0.15
#3 ESK SiC(1)	3.59 ± 0.44	4.44 ± 0.47	4.42 ± 0.64
#4 ESK SiC(2)	4.61 ± 0.08	3.88 ± 0.09	4.42 ± 0.36
#5 B_4C	2.94 ± 0.09	2.96 ± 0.42	3.24 ± 0.16
#6 White alumina	- *	- *	3.71 ± 0.13
#7 Pink alumina	- *	- *	4.19 ± 0.30
#8 WC/W_2C	8.74 ± 0.27	7.25 ± 0.38	7.70 ±1.11
#9 Si_3N_4	3.56 ± 0.26	- **	4.17 ± 0.28
#10 SIKA SiC	5.9? **	- *,**	5.80 ± 0.23

* Did not fail from indentation flaw.

** Failed from flaw, but flaw boundary could not be clearly identified except possibly in one test-piece.

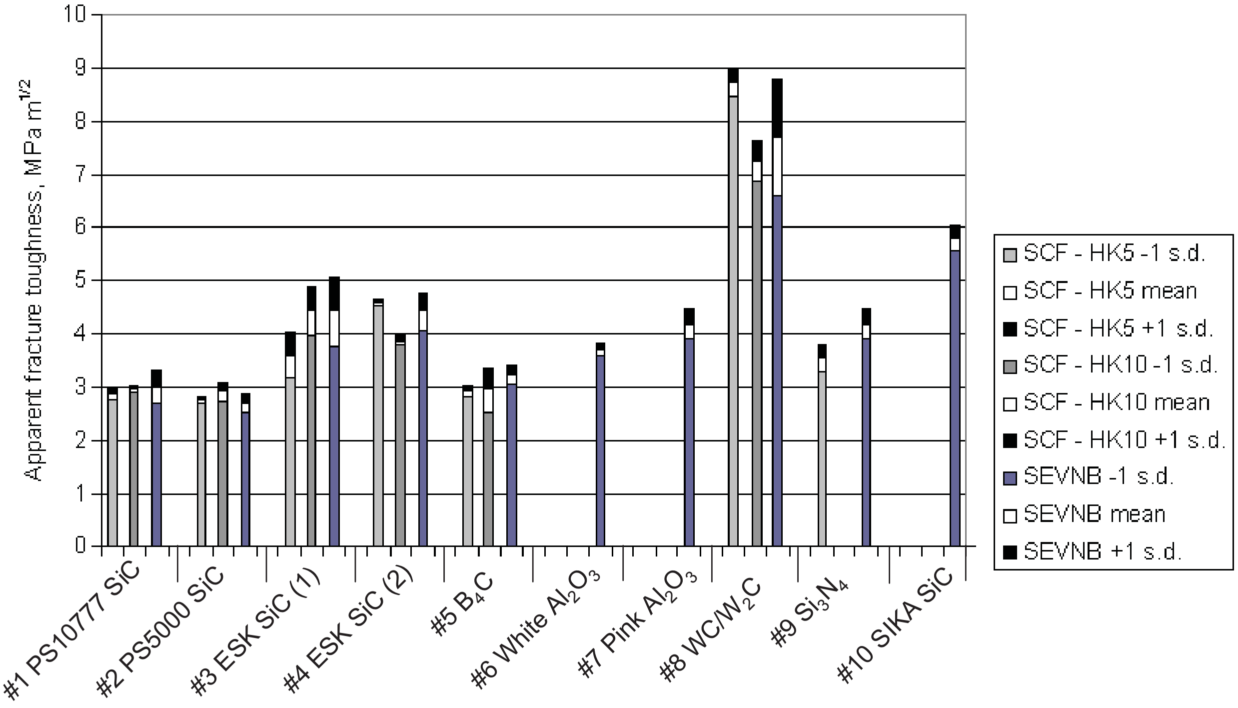


Figure 6: Schematic comparison of fracture toughness data.

Discussion

In all the test cases used in this work, a good agreement was found between the SEVNB and the SCF results for fracture toughness. In addition, the SEVNB method provides data which could not be readily be obtained by the SCF method using convenient optical fractography. This is promising, and provides evidence of the flexibility of the SEVNB method in terms of its usability on a wide range of materials types.

It is already acknowledged in the CEN and ISO standards that the application of the SEVNB method to certain very fine-grained materials may result in a small overestimate of fracture toughness because of a notch root radius problem. This has also been identified by Quinn *et al.* [11] in their evaluation of a fracture toughness reference material, SRM2100, based on a fine-grained silicon nitride, where an overestimate by the SEVNB method of about 0.1 MPa m$^{1/2}$ compared with other methods was established. However, this is small compared with the typical scatter of results, so it generally seems to be the case that this is not a problem for most technical materials. In addition, not accounting for a small subcritical crack extension from the notch root would result in a small conservative bias in fracture toughness value, the same as in the SCF method. In this work, in only the high-purity fine-grained alumina SCF pre-cracks could any evidence of subcritical growth be clearly seen. Any residual risks associated with atmospheric moisture could, of course, be controlled in both methods by testing in a dry atmosphere, or with oil in the notch or pre-crack.

The difficulties with identifying SCF pre-cracks in some of the materials probably relate to the nature of the microstructure. The coarser the grain size, the less planar the pre-crack becomes, and the rougher is the final fracture surface in relation to the indentation pre-crack size. It therefore becomes more difficult to distinguish the pre-crack boundary, as has already been identified [12]. Thus in materials 9 (HP silicon nitride) and 10 (SIKA silicon carbide), indenting on tilted specimens does not significantly assist optical identification of the pre-cracks. Further enhancement of the technique, e.g. using reflective coatings and/or the scanning electron microscope may assist. There is also, of course, the risk with tough materials that too much material has been removed in the polishing process, especially if the pre-cracks are significantly semielliptical rather than semicircular (e.g. tending to shallow ellipses or Palmqvist-like [13]).

The existence of lateral cracks under the indentation site has been considered as a potential interference to the SCF method [13, 14]. By keeping the indentation force used in this work to 100 N or less, the risk of development of deep lateral cracking is minimized [13]. In none of the examples in the present work could any interference from lateral cracking be seen, implying that any deep laterals had been successfully removed by the polishing process.

The failure to produce fracture-dominating SCF pre-cracks in the armour aluminas is not surprising. Most technical aluminas in large sizes are made from spray-dried granulates, followed by pressing and sintering. In this process, the gaps between granules often do not completely disappear, leaving cusped pores that act as fracture origins. These pores can be quite large. In the above tests the flexural strengths were only about 260 and 280 MPa for materials 5 and 6, respectively. In order to cause failure preferentially by an indentation pre-crack, this would have to be at least 400 µm wide and 100 µm deep, and would require an indentation of at least HK30 or higher, with significant risks of test-piece fracture. In contrast, in the high-purity fine-grained alumina materials, strong efforts have been made to eliminate such pores, and much smaller SCF pre-cracks dominate as strength-controlling defects.

Conclusions

A series of materials has been fracture toughness tested using both the SEVNB and the SCF methods to provide assurance of their equivalence. It has been found that this is indeed the case for examples where the SCF method can be safely employed using optical fractographic methods methods.

Acknowledgements

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