The measurement of K_{IC} in single crystal SiC using the indentation method

J. L. HENSHALL, C. A. BROOKES
Department of Engineering Science, University of Exeter, Exeter, Devon, UK

The basic factors affecting the fracture behaviour of commercially available ceramics are due to a combination of the primary material parameters and those associated with the microstructural form, e.g. grain size and nature of the grain boundaries. The present work has concentrated on investigating the underlying fracture toughness behaviour of SiC single crystals. This material was chosen because of the commercial importance of the various polycrystalline forms of SiC and the relative ready availability of reasonably sized single crystals. Measurements of K_{IC} values, determined using conventional three-point bend tests on notched beams in this material have been reported and discussed previously [1]. However, these results were for only one crack plane, $\{11\bar{2}0\}$, and involved considerable effort in the preparation of sufficient specimens. Also, it is only possible to measure K_{IC} for planes of the type $\{hki0\}$ using this method, because of the basic platelet form of these crystals. Therefore this study has examined the feasibility of using the indentation technique [2] to determine K_{IC} in SiC single crystals. This requires much more less complex experimentation and also affords the possibility of being able to use this method to study the orientation dependence of K_{IC} in a similar manner to that used to investigate anisotropy in indentation hardness behaviour [3, 4].

A single crystal of 6H-SiC was used for all the hardness and conventional K_{IC} results reported here. The particular polytype and orientation were determined using the Laue X-ray method. All the measurements were made under ambient conditions. Three-point bend tests, with a 6 mm span on single edge notched beams, SENB, orientated such that the plane of the notch was $\{11\bar{2}0\}$ and the crack propagation direction $\langle 1\bar{1}00\rangle$ were used for the conventional K_{IC} tests. Fig. 1 shows the geometry and orientation of the testpiece. All specimens were cut with a diamond saw; lapped with 13 μm SiC on a cast iron wheel; cleaned in hydrochloric acid; and then washed in alcohol. The notches were machined with an annular diamond saw to have a width of 240 μm and a tip radius of 120 μm. The tests were performed in a screw driven tensile test machine using a constant cross-head displacement rate of 0.05 cm min^{-1}.

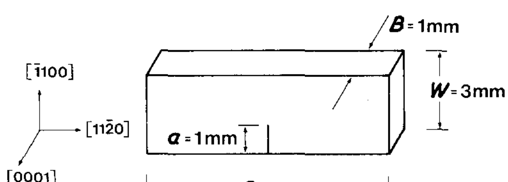

Figure 1 Geometry and orientation of the three-point single edge notched beam specimens.

The hardness indentations were all made on one particular SENB test piece after it had been fractured. The specimen was mounted in acrylic and ground and polished, finishing with 1 μm diamond paste, in the usual manner. A Leitz Miniload Hardness Tester was used with both Vickers and Berkovich indenters lowered at a rate of 0.2 mm sec^{-1} and a dwell time of 12 sec. The geometry and nomenclature are summarized in Fig. 2 but note that the orientation of

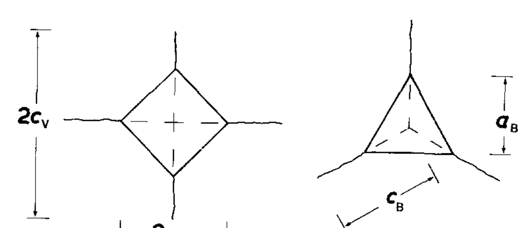

Figure 2 Geometry and nomenclature of indentations and cracks used to determine K_{IC} from indentations made with an applied load F. (a) Vickers indentations — square-based pyramidal indenter with an angle of 68° between the vertical axis of the indenter and each of its facets: $H_v = 0.4636\ F/a_v^2$. (b) Berkovich indentations — triangular-based pyramidal-type indenter with an angle of 65° between the vertical axis of the indenter and each of its facets: $H_B = 1.732\ F/a_B^2$.

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TABLE I Details of Vickers indentations and resultant measurements and calculated hardness and K_{IC} values. (Symbols have the same meaning as in Fig. 2.) All indentations were on the (0001) plane

Load F (N)	Angle between facet and $[11\bar{2}0]$	a_v (μm)	c_v (μm)	H_v (GPa)	K_{IC} (MPa m$^{1/2}$)
4.90	+15°	8.3	27.1	33.0	3.4
9.81	+15°	12.6	47.9	29.0	3.1
19.61	+15°	19.1	77.0	25.0	3.2
9.81	+ 5°	13.3	48.1	25.6	3.2
9.81	0°	12.6	48.7	28.5	3.0
9.81	− 5°	12.6	47.9	28.5	3.1
9.81	−15°	12.4	46.3	29.4	3.2

a given indenter was determined by its facets, rather than the diagonals, with respect to a particular crystallographic direction on a given crystal plane.

In all the SENB K_{IC} tests, fracture occurred catastrophically without visible departure from linearity on the load–time curves. The value of K_{IC} was calculated from the formula of Srawley and Brown [5], i.e.

$$K_{IC} = \frac{3P_f L a^{1/2}}{2BW^2}[A_0 + A_1(a/W) + A_2(a/W)^2 + A_3(a/W)^3 + A_4(a/W)^4] \quad (1)$$

where $A_0 = 1.9 + 0.0075(L/W)$, $A_1 = -3.39 + 0.08(L/W)$, $A_2 = 15.4 - 0.2715(L/W)$, $A_3 = -26.24 + 0.2815(L/W)$, $A_4 = 26.38 - 0.145(L/W)$, and P_f is the failure load, L the bending span, and a, B and W are defined in Fig. 1. It had been shown previously for SiC that this sawn notch is sufficiently sharp at the crack tip to act as a natural crack [6]. Thus the average value of K_{IC} for this crystal was determined as 3.4 ± 0.2 MPa m$^{1/2}$.

There have been several analyses proposed for the determination of K_{IC} from cracks around Vickers indentations [2, 7, 8]. It was found in the present series of tests that, regardless of whether the cracks were Palmqvist or median, the analysis of Evans and Charles [2] provided the most reliable and consistent results – in agreement with the observations of Lankford [9]. Consequently, K_{IC} values were derived from the measurements of Vickers indentations on the basal plane using Evans and Charles' analysis [2] i.e.

$$K_{IC} = 0.129\left(\frac{c_v}{a_v}\right)^{-3/2}\frac{H_v a_v^{1/2}}{\Phi}\left(\frac{E\Phi}{H_v}\right)^{2/5} \quad (2)$$

where a_v, c_v and H_v are defined in Fig. 2, E is the Young's modulus (=460 GPa [10]), and Φ a constaint factor = 2.57. Table I lists the values of a_v, c_v, H_v and K_{IC} for the Vickers indentations in the orientations and for the loads shown. There was no difference between the two different directions corresponding to cracks forming on $\{11\bar{2}0\}$ and $\{1\bar{1}00\}$. The average value for K_{IC} for this orientation was 3.2 ± 0.2 MPa m$^{1/2}$, which agrees well with the SENB value of 3,4. As can also be seen from this table there is no significant anisotropy of K_{IC}, although there is possibly a minimum value at 15° to $\{11\bar{2}0\}$. It is known, however, that there is a small but significant anisotropy in the Knoop hardness on the basal plane between $\langle 11\bar{2}0\rangle$ and $\langle 1\bar{1}00\rangle$ [4] and that for the most consistent results on single crystal surfaces the symmetry of the indenter should reflect that of the indented plane [3]. Therefore, it was decided that the Berkovich indenter [11], with its three-fold symmetry, would be the most appropriate choice. However, this indenter has not been previously used to determine K_{IC} values and therefore its application required further examination.

Now, since $H_v \propto F/(a_v)^2$, Equation 2 can be re-written as:

$$K_{IC} = \alpha_v \frac{F^{0.6} a_v^{0.8}}{c_v^{1.5}} \quad (3)$$

where α_v is a constant = 0.002 14 for SiC and the Vickers indenter, with F in N, a and c in m and K_{IC} in MPa m$^{1/2}$. Table II lists the values of the corresponding parameters for the Berkovich indentations, i.e. a_B, c_B, and $F^{0.6}a_B^{0.8}/c_B^{1.5}$, which is virtually constant with a mean value of 2240 ± 30. Thus, similar to Equation 3 we can

TABLE II Details of Berkovich indentations and resultant measurements and calculated hardness and K_{IC} values. (Symbols have the same meaning as in Fig. 2). All indentations were on the (0 0 0 1) plane except for the last one (*) which was on the $(1\bar{1}00)$ plane.

Load F (N)	Angle between facet and $[11\bar{2}0]$	a_B (μm)	c_B (μm)	H_B (GPa)	$\frac{F^{0.6}a_B^{0.8}}{c_B^{1.5}}$	K_{IC} (MPa m$^{1/2}$)
2.94	0°	11.9	21.9	36.0	2150	3.1
4.90	0°	16.7	31.0	30.6	2267	3.3
9.81	0°	25.7	51.5	25.8	2267	3.3
19.61	0°	36.5	81.9	25.5	2267	3.3
4.90	15°	16.3	33.2	31.9		2.9
4.90	30°	16.3	29.5	32.0		3.5
9.81	30°	25.0	53.7	27.1		3.0
19.61	30°	36.2	84.0	25.9		3.1
4.90*	90°	16.9	32.5	29.9		3.1

write:

$$K_{IC} = \alpha_B \frac{F^{0.6}a_B^{0.8}}{c_B^{1.5}} \quad (4)$$

where a value of $\alpha_B = 0.001\,43$ yields an average value for K_{IC} on the $\{11\bar{2}0\}$ planes of 3.2 MPa m$^{1/2}$ and particular values for the different loads in the range 3.1 to 3.3 MPa m$^{1/2}$, as detailed in Table II. As can also be seen from this table, K_{IC} was determined using Equation 4 for Berkovich indentations on the basal plane with the cracks on $\{1\bar{1}00\}$ planes for loads of 4.9, 9.8 and 19.6 N as 3.2 MPa m$^{1/2}$. This confirms that there is no anisotropy in K_{IC} between $\{11\bar{2}0\}$ and $\{1\bar{1}00\}$ planes. This is despite a slight anisotropy in the observed hardness values with the 4.9 and 9.8 N loads. The results for the orientation giving cracks along planes at 15° to $\{11\bar{2}0\}$ again suggest that the value of K_{IC} for this plane may be slightly lower, i.e. ~10%, but this does lie within the experimental scatter. Berkovich indentations were also made on a $\{1\bar{1}00\}$ plane with one of the cracks forming in the $\{11\bar{2}0\}$ plane. The hardness and K_{IC} values were essentially the same as for the indentations on (0 0 0 1).

One of the major aims of the present work was to investigate the possibility of using the hardness technique to investigate variations in K_{IC} with orientation, and at a later stage with temperature. All that is required to do this is to be able to detect changes rather than absolute values. The precision of the present method, i.e. ~5%, is certainly sufficient to allow the detection of significant changes. This is very similar to the way that the basic hardness test is used and has much the same advantages, i.e. small specimens, speed, cost, etc. However, the more surprising factor is the very good absolute accuracy of the method. The achievement of results agreeing to within 5% is as good as could be expected from any two different techniques of determining K_{IC}, particularly in small ceramic specimens. Thus it can be safely concluded that the formula of Evans and Charles [2] for the Vickers indenter and Equation 4 of this paper allow the determination of K_{IC} to an absolute accuracy of ~5%. However, even with this very good precision it has not proved possible to determine whether there is any anisotropy of K_{IC} for $\{hki0\}$ type planes, since the observed lower values for orientations of cracks at 15° to those on $\{11\bar{2}0\}$ still lie within the experimental scatter.

The average value of K_{IC} for this crystal of 3.3 MPa m$^{1/2}$ corresponds to a G_{IC} value of 23 J m^{-2}. This has been interpreted as being primarily controlled by the thermodynamic surface energy and the surface roughness effect [1]. In view of a lack of marked anisotropy in K_{IC}, the slightly greater G_{IC} values obtained on polycrystalline SiC of ~30 to 40 J m^{-2} are almost certainly caused mainly by an enhanced surface roughness.

It is possible to estimate the energy expended in crack formation i.e. $\sim G_{IC}\pi c_v^2$ for median cracks with the Vickers, to the energy expended in plastic deformation i.e. $\sim Fa_v/3.5$. For the combination of loads and orientations detailed in Table I, this ratio is effectively constant with an average of $0.46\% \pm 0.01\%$. This clearly demonstrates that most of the energy is absorbed by plastic deformation and that the cracking is incidental and should not significantly affect the hardness values. This is also supported by many hardness results on other materials in the authors' laboratory, although there is some debate concerning the contribution of cracking to the measured hardness in SiC and other ceramics [12].

It is apparent from the above results that the Berkovich indenter can also be used to determine K_{IC}. The general formula, corresponding to that of Evans and Charles' [2] for the Vickers is:

$$K_{IC} = 0.0392 \left(\frac{c}{a}\right)^{-3/2} \frac{H_B a_B^{1/2}}{\Phi} \left(\frac{E\phi}{H_B}\right)^{2/5} \quad (5)$$

The above formula should apply regardless of whether the initial cracks are radial, Palmqvist or median. Any lateral or conchoidal cracking occurs most frequently at the higher loads and can render the measurement of indentation dimensions effectively impossible. Observations of this cracking, both visually and using acoustic emission, would suggest that this type of cracking is formed generally on removal of the indenter, when secondary tensile stresses are generated. The main advantage of the Berkovich indenter is that it is capable of determining anisotropy effects on planes with three-fold or six-fold symmetry, e.g. Brookes [13]. Thus, in this case, it has been possible to detect the slight hardness anisotropy in the basal plane and also show that there may be a very slight decrease in K_{IC} on the planes at 15° to $\{11\bar{2}0\}$.

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