NPL

National Physical Laboratory

A NATIONAL MEASUREMENT GOOD PRACTICE GUIDE

No. 9

Palmqvist Toughness
for Hard and
Brittle Materials

Measurement Good Practice Guide No. 9

Palmqvist Toughness for Hard and Brittle Materials

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Abstract: The Palmqvist toughness method is widely used for measuring the fracture toughness of hardmetals. Fracture toughness values are required to control quality, for new material assessment and for design in product development. There is no standard for this test method. This document, therefore, includes good laboratory practice for measuring Palmqvist toughness in hardmetals. It is applicable to indentation tests at room temperature. Technical input to the document has been provided by an industrial steering group including representatives of industrial users and producers of a wide range of products that are both made from and use hardmetals.

Various technical exercises were conducted in association with the production of this document to quantify the uncertainties associated with Palmqvist toughness measurements on hardmetals with a range of hardnesses. Also, published literature on the toughness of hardmetals has been reviewed from the point of view of measurement issues.

Additional work was conducted to validate the original version of this good practice document, through a VAMAS collaboration. An edited version of this document has been produced which will form the basis of an ISO Standard.

In contrast, the test is somewhat unsatisfactory for most ceramic materials with problems of identification of the crack tip, poor quality indentations and crack geometry, uncertain subsurface crack shape, a wide range of formulae for calculating toughness, and large potential errors in determining toughness. Alternative methods of determining toughness are recommended.

Palmqvist Toughness for Hard and Brittle Materials

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Foreword

Good test methods are those which enable a user or manufacturer to clearly discriminate between different materials. Palmqvist tests for toughness are widely used because of their perceived apparent simplicity. However, there are no standards for their use. There are a number of different ways in which a value of Palmqvist toughness can be obtained including different methods of surface preparation. This NPL Good Practice Guide (GPG) provides a recommended method for hardmetals* which can be used as a technical precursor to a common standard. There is also an annex which discusses the application of the method to ceramic materials, where technical problems make the method rather unsatisfactory for standardised measurements.

These guidelines were originally prepared in 1998 as part of project CAM4 within the Advanced Materials Programme, an underpinning materials measurement research activity supported by the UK Department of Trade and Industry (DTI). This version (January 2008) is an updated edition taking account of new VAMAS work and ISO committee activity. The GPG describes a method for measuring the Palmqvist toughness of hardmetals and cermets at room temperature by an indentation method. The guidelines recommend good practice to minimise levels of uncertainty in the measurement process. The procedure has been validated through underpinning technical work within the CAM4 and previous DTI materials measurement programmes.

Additional work was undertaken to validate the original good practice document, through a VAMAS international collaboration in 2002-2004. An edited version of this document has been produced which will form the basis of an ISO Standard. ISO TC114/SC4 is currently reviewing a standardised procedure based on this guide.

Terminology - There is a range of terms used for this type of material, including especially cemented carbides and/or cermets as well as hardmetals. The word 'hardmetal' has been used in this document. It includes all hard materials based on carbides that are bonded with a metal. In ISO 3252 Terminology 'hardmetal' is stated to be 'a sintered material characterised by high strength and wear resistance, comprising carbides of refractory metals as the main component together with a metallic binder phase'. 'Cemented carbide' is synonymous with 'hardmetal'. A 'cermet' is defined as 'a sintered material containing at least one metallic phase and at least one non-metallic phase generally of a ceramic nature'.

Project industrial advisory group (1998)

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1.   General background

1.1   Fracture toughness methods

Hardmetal products can fracture from crack-like defects that develop in service through, for example, wear or fatigue processes. Consequently, fracture toughness is an important parameter that influences the strength of hardmetals components. Fracture toughness values are usually required for one or more of three reasons:

- for product design and performance assessment;

- for materials selection;

- for quality control.

Their fracture toughness, expressed either as a stress intensity factor with units MN m$^{-3/2}$ or as a fracture surface energy with units J m^{-2}, increases with increasing Co content and with increasing WC grain size. The range of values for typical WC/Co hardmetals is from 7 to 25 MN m$^{-3/2}$ [1-31].

When applied unqualified to hardmetals the term 'toughness' can have several meanings:

- **Plane strain fracture toughness, K_{Ic}** (MN m$^{-3/2}$) - a value obtained from tests on specimens with appropriate geometries for plane strain conditions and containing a well defined geometry of crack. There is no standard method for hardmetals and different organisations use different test methods for introducing the pre-crack.

- **Strain energy release rate (or work of fracture), G** - an alternative expression for toughness, often obtained by converting plane strain toughness, K, to G (*i.e. $G = K^2/E(1-\nu^2)$, E* is Young's modulus and ν is Poisson's ratio). It has units of J m^{-2}. Again there is no standard method.

- **Palmqvist toughness, W** - a value obtained by measuring the total length of cracks emanating from the four corners of a Vickers hardness indentation. For a given indentation load the shorter the crack the tougher the hardmetal. There is no standard for the test and the results are very sensitive to methods of surface preparation.

- Finally, 'toughness' is also widely used, in a loose sense, to describe a perceived resistance to dynamic impacts. This is neither standardised nor quantified, but is clearly important for many industrial applications of hard materials. Also, principally for hardmetals, it may be more realistically assessed through either fatigue tests or high-rate strength tests, rather than a conventional fracture toughness test.

There is a general inverse trend of hardness against fracture toughness. Both plane strain fracture toughness data (K_{Ic}) obtained from tests on a wide range of WC/Co hardmetals at NPL [1] and published data from Sandvik Hard Materials [2] show a general trend of decreasing toughness

with increasing hardness (Figure 1). However, there are few data for grades with harness above 1800 HV30, and a very wide spread for grades with hardness less than 1200 HV30.

There is no ISO standard test method specifically for measuring the toughness of hardmetals, primarily because of the practical difficulty of introducing stable pre-cracks into these tough but hard, high-modulus materials (a prerequisite for a valid toughness test). While there are some options for lower toughness grades of material, there is a particular need for a suitable test for grades with toughness values greater than about 15 MN m$^{-3/2}$ that are even more difficult to pre-crack. One route to overcoming the pre-cracking problem has been the development of tests based on the 'chevron notch short rod' (CNSR) method, which has been standardised by ASTM as E1304 and B771, but this requires specialist equipment. Also, recent research published by the Plansee group [28] has shown that controlled cracks can be introduced by using a very stiff pre-cracking rig.

Different groups and organisations in the hardmetal community have evaluated a range of techniques [1-31] with varying outcomes, as follows:

- **SEPB** **Single-Edge Pre-cracked Beam.** Beam tested in flexure with a sharp crack on the tensile side. Hardmetals are difficult to pre-crack. Wedge indentation and fatigue (including in compression from a notch) have proved successful in some cases. A new method based on a stiff loading system has been used in recent work.

- **SENB** **Single-Edge Notched Beam.** Beam with a notch of finite tip radius. Results depend on the notch width and on the method of preparation. Not generally recommended for hardmetals (nor most ceramics).

- **SEVNB** **Single-Edge Vee-Notch Beam.** Beam containing a notch with a sharpened tip by diamond honing. Validated for many ceramics. More work needed for hardmetals to confirm requisite notch sharpness and methods of obtaining it.

- **SCF** **Surface Crack in Flexure.** Beam containing a small semi-elliptical flaw introduced by indentation and with surface damage removed. Such pre-cracks are difficult to make in hardmetals, and it is not possible to remove the damage without removing the pre-crack. Not recommended.

- **IF** **Indentation fracture.** Also known as the Palmqvist toughness test. Relies on measurement of lengths of cracks from indentation corners. Works reasonably well for hardmetals in the toughness range 8 – 16 MN m$^{-3/2}$ provided that the surface is free from residual stresses. Tougher materials produce few or inconsistently sized cracks. Evaluated in the 2002-4 VAMAS exercise and included in this Guide.

- **IS** **Indentation Strength.** Rectangular beam pre-cracked with an indentation and subsequently fractured. Damage and residuals tresses associated with indentation have a strong influence on the result. Not recommended for hardmetals.

- **CNB** **Chevron Notched Beam.** Controlled crack initiation from the chevron tip is difficult in hardmetals. Not recommended.
- **CNSR** **Chevron Notched Short Rod.** Chevron notched machined into one end of a rod,

which is then split open lengthwise. Commercial equipment is available (Terratek/Dijon). Need to be careful with residual stresses. ASTM standards in place (E1304 and B771). Evaluated in the 2002-4 VAMAS exercise and results are included in this Guide.

Of all these methods, the most economical and superficially user-friendly for the manufacturing industry is the Palmqvist test, which is the subject evaluated in detail by NPL through international round robin activities, and which forms the subject matter of the guide.
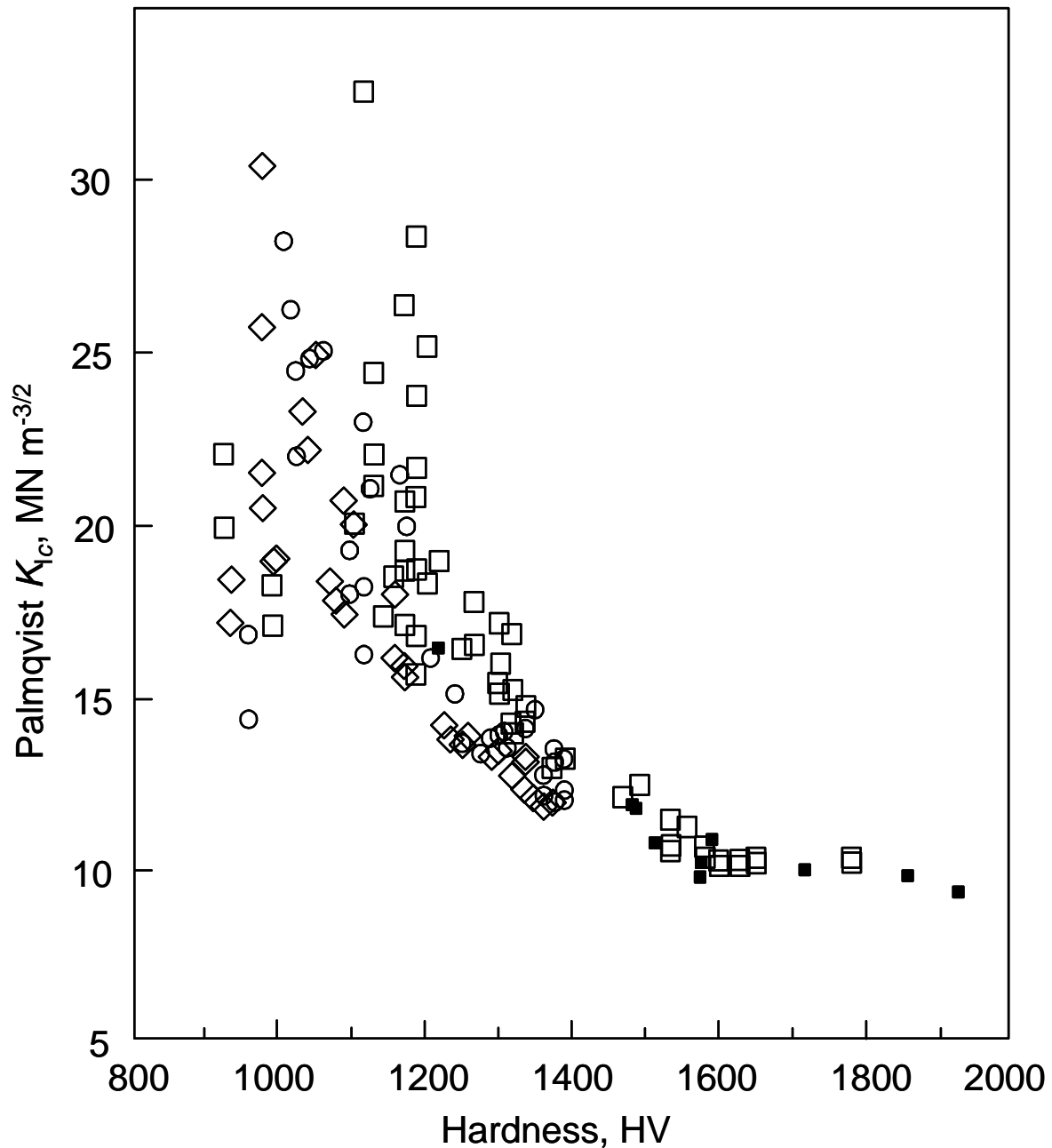
Figure 1 - *Variation of toughness with hardness for WC/Co hardmetals.*

1.2 'Palmqvist' toughness

There is a considerable body of published information on Palmqvist toughness tests for hardmetals [3-5, 7-31]. Palmqvist toughness, *W*, is a toughness value obtained by measuring the crack lengths at the corners of a Vickers indentation. It can be evaluated by making indentations either at a single load, usually 30 kgf, or from the inverse of the slope of a plot of crack length against load for a range of applied loads. There is no standard method for measuring the crack lengths, either of the methods shown in Figure 2 can be used. For hardmetals, the crack depth profile is normally of the Palmqvist type, *i.e.* independent shallow arcs emanating from each indentation corner. Measurement of surface crack length is, however, open to operator error. It is widely recognised that test surfaces should be carefully prepared to remove the effects of residual surface stresses [7]. The test also has a poor fracture mechanics pedigree because of uncertainties associated with residual stresses introduced by the indentation.

Palmqvist Toughness, W_G, Definitions

Hardness:

$$H = \frac{1.8544\ P}{[(d_1 + d_2)/2]^2}$$

Indentation force, *P*:

usually 30 kgf for hardmetals

Figure 2 - Schematic diagram and definitions for the Palmqvist test method.

There are two methods for calculating the toughness value:

Method 1 - Ratio of indent load to crack length

W_G is calculated from the ratio of indent load, P, to total crack length, T (either by using the inverse of the slope of a plot of crack length against load or using the crack length from the indentation at a single load), where T is measured by one of the methods shown in Figure 2.

$$W_G = \frac{P}{T} \qquad (1)$$

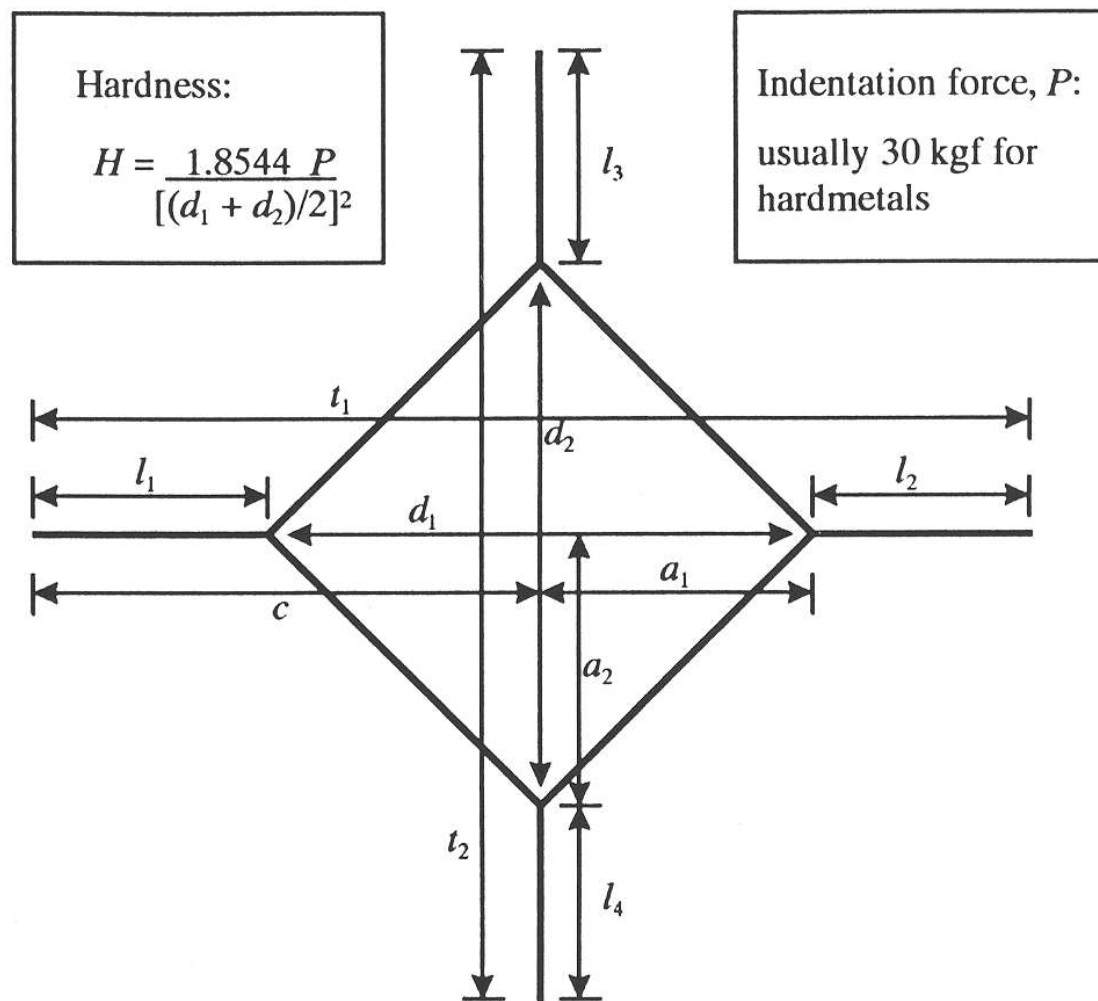W_G is expressed in units of N mm^{-1}, which is equivalent to J m^{-2} (*i.e.* strain energy release rate).

Method 2 - Calculated value of toughness

W_K is obtained from W_G using the following formula [3-5]:

$$W_K = A\sqrt{H}\,\sqrt{W_G} \qquad (2)$$

where A is an empirical constant with a value of 0.0028, H is the hardness in N mm^{-2} and W_K is expressed in MN m$^{-3/2}$.

For conventional hardmetals a good correlation is claimed between K_{Ic} and W_K [4]. This has been confirmed by comparison with validated tests according to an agreed standard method [6]. Research at NPL has compared Palmqvist toughness measurements on a range of WC/6% Co hardmetals with plane strain K_{Ic} values obtained by an NPL recommended procedure [6]. Figure 3 shows the comparison and the agreement is quite good between K_{Ic} values of about 10 and 15 MN m$^{-3/2}$. For low values the Palmqvist method overestimates the toughness, in agreement with recent work published by Gartner *et al.* [31], possibly because:

i)   the true lengths of cracks in hard fine-grained hardmetals (*i.e.* low toughness) are difficult to measure accurately and the length is underestimated giving a higher apparent toughness;

or   ii)   the annealing treatment of 800 $^{\circ}$C for 1 h is not adequate to fully relieve residual stresses.

In some published work temperatures of 850 $^{\circ}$C for 2 h have been used. In the absence of further systematic work on temperatures and times of annealing, especially for the finer-grained harder materials, then 2 h at 800 $^{\circ}$C would seem to be more likely to produce stress-free surfaces than 1 h at 800 $^{\circ}$C.

or   iii)   expression (2) is not applicable in the case of long cracks where the crack shape may be approaching that of a half-penny, as in many low toughness ceramic materials. Another expression for calculating W_K may be more appropriate (see Appendix D).

Toughness Comparison
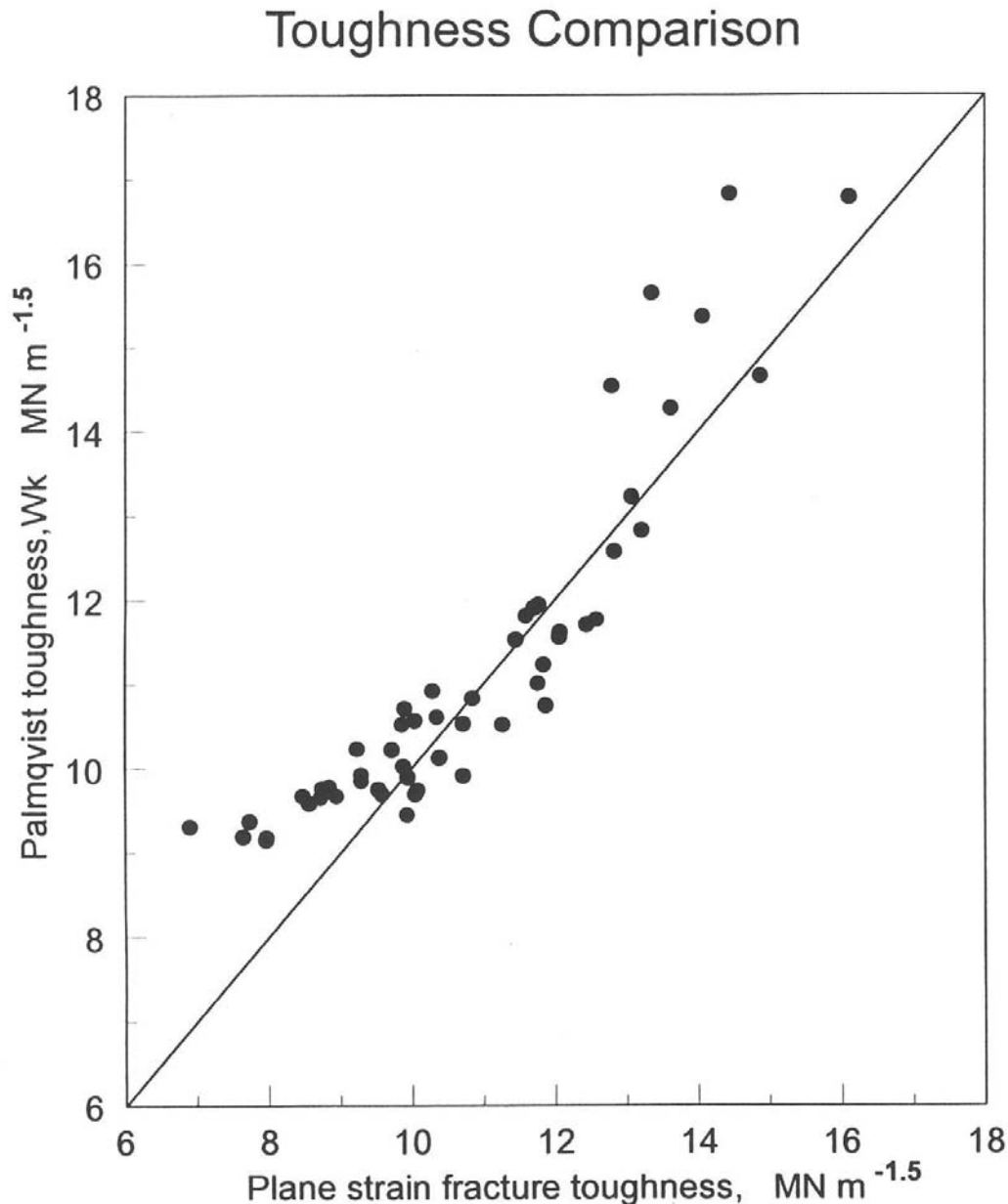
Figure 3 - *Comparison of Palmqvist toughness values with plane strain fracture toughness measurements on WC/Co hardmetals. See also the work of Gartner et al. [31].*

For all values the Palmqvist data show more scatter than the plane strain data and this is especially large for high toughness hardmetals. In fact, at high toughness values it becomes very difficult to obtain cracks at the corners of an indentation even when the applied loads are as high as 60 kgf. It is impractical to use loads higher than 30 kgf on a regular basis because of the cost of damage to the diamond indenter.

One advantage of the Palmqvist method is that parallel measurements are made of sample hardness, which is required for quality control purposes. The crack length, and thus toughness measurements, do not therefore require much more effort and can yield equally useful material characterisation data provided the measurements are obtained carefully in line with the methods proposed in this Good Practice Guide.

1.3 VAMAS interlaboratory evaluation

An interlaboratory exercise was conducted to generate underpinning technical information on toughness tests for hardmetals [28, 29]. More than 10 industrial organisations participated, either by correspondence, supply of materials or by conducting tests. Eight organisations were able to complete Palmqvist tests and two completed short-rod chevron notch tests; however, only three organisations were able to provide single edge beam data. Good statistics were obtained on the Palmqvist data that enabled a quantitative assessment of uncertainties to be performed for this relatively simple test. Single-edge pre-cracked beam data were thought to be closest to the 'true' value, and most of the short rod chevron notch tests data compared reasonably well with these results. However, care was needed in test-piece preparation to ensure a good correlation between data from the Palmqvist tests and the single-edge pre-cracked beam results. A number of VAMAS participating organisations supplied materials for Palmqvist validation tests [28, 29]. The materials tests are described in Table 1.

Table 1   Material Properties

Material	Density Mg m^{-3}	Coercivity kA m^{-1}	Magnetic moment μT m^3 kg^{-1}	HRA	HV30[+]
B1	-	-	-	-	1778
B2	-	-	-	-	1626
H1	14.77	31.0	0.86	93.3	1810
H2	14.50	19.9	1.45	92.0	1592
H3	14.19	14.5	2.02	89.9	1364
TCM10 (P2)	6.6[††]	-	-	-	1636
K313 (K1)	14.81	23.8	0.94	92.7	1726
K420 (K2)	12.38	11.1	1.31	91.4	1486
K3560 (K3)	14.38	4.9	1.44	85.9	1028

[+] NPL values   [††] Source values

Research at NPL [27] has shown that the uncertainties associated with the Palmqvist test are generally about $\pm$ 1.5 MN m$^{-3/2}$ for calculations of toughness from the formula, $W_K = A\sqrt{}$ HV $\sqrt{W_G}$. These values of uncertainty can be compared with an estimated uncertainty of about $\pm$ 0.5 MN m$^{-3/2}$ for plane strain toughness tests on more conventional fracture toughness test-pieces [28]. Thus, toughness data quoted for very fine grained hardmetals must be considered very carefully since there are no standard methods. Differences of less than 50 N mm^{-1} (W_G), 1.5 MN m$^{-3/2}$ (W_K) or 0.5 MN m$^{-3/2}$ (K_{Ic}) are not likely to be significant.
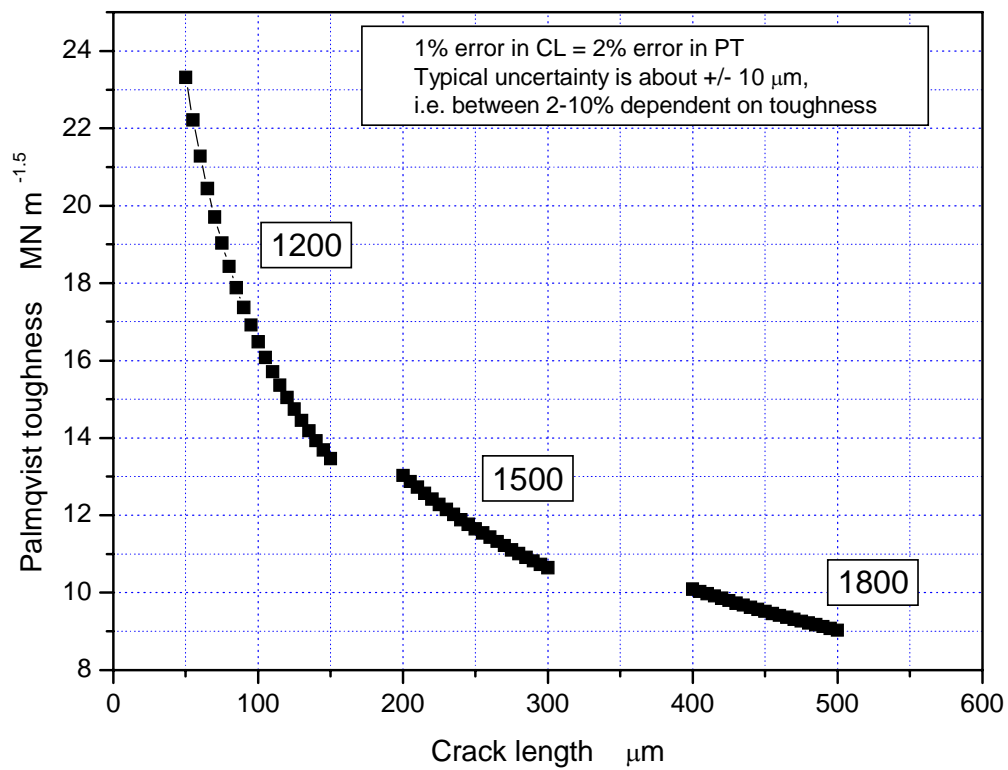
Figure 4 - Effect on Palmqvist toughness of differences in crack length at constant hardness.
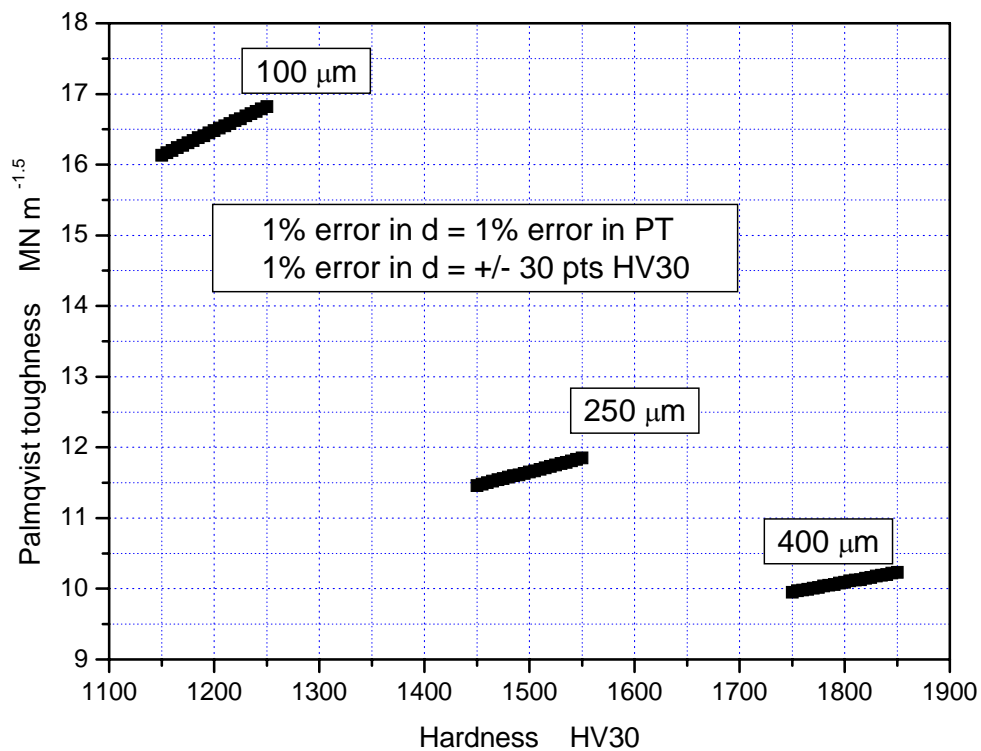
Figure 5 - Effect on Palmqvist toughness of differences in hardness at constant crack length.

The uncertainty in Palmqvist measurements that can arise from two sources, measurement of hardness and measurement of crack length, is demonstrated in Figures 4 and 5, where plots are shown of the range of Palmqvist values that can be obtained assuming either a constant hardness or a constant crack length. Clearly crack length is a more important measurement issue, especially for crack length values of less than 200 µm. For this reason, participants were asked to measure crack length only (although some participants also provided information on hardness as it was relatively easy to obtain at the same time as measuring crack length) and Palmqvist toughness values were calculated using the mean values of hardness obtained at NPL.

For each material about 14-15 test-pieces were prepared (using the principles outlined in the NPL Good Practice Guide) for circulation to potential participants for Palmqvist measurements. Each sample was indented at HV30 (plus HV100 for K3560) and the results obtained at NPL are plotted as crack length against hardness in Figure 6 and Palmqvist toughness, W_K, against hardness in Figure 7. These plots give a visual indication of spread in values for each material.

The NPL data are plotted against the Plansee SEPB data as cluster plots in Figure 8. The agreement is good, even for the tough grade K3560, which showed a high standard deviation in crack length. In fact there was probably better agreement between the Palmqvist data and the SEPB results than between the short rod and the SEPB methods.
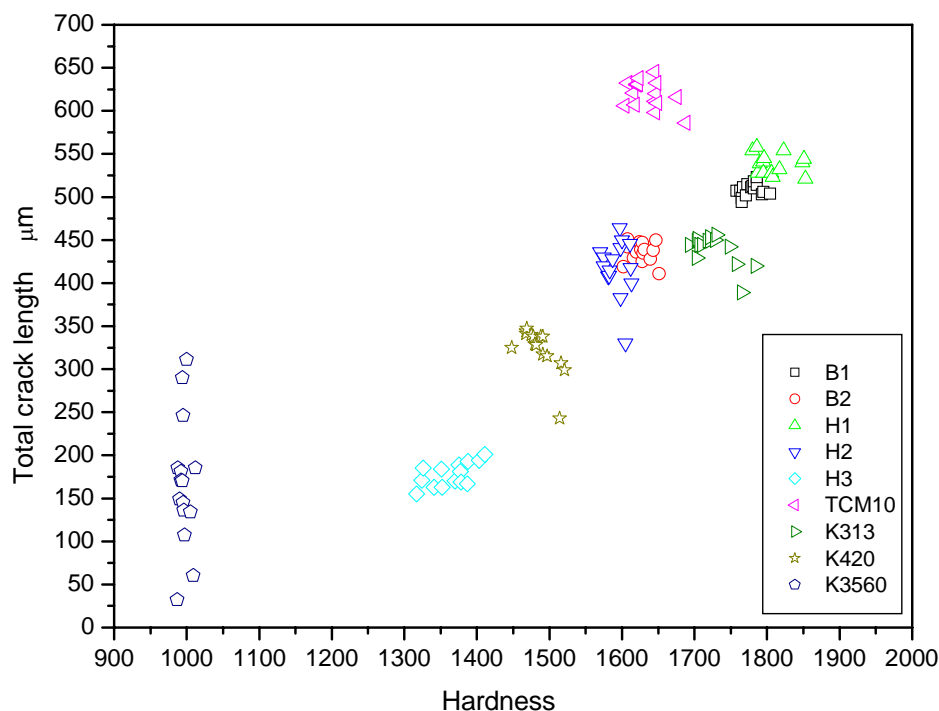
Figure 6 - *NPL data – crack lengths.*
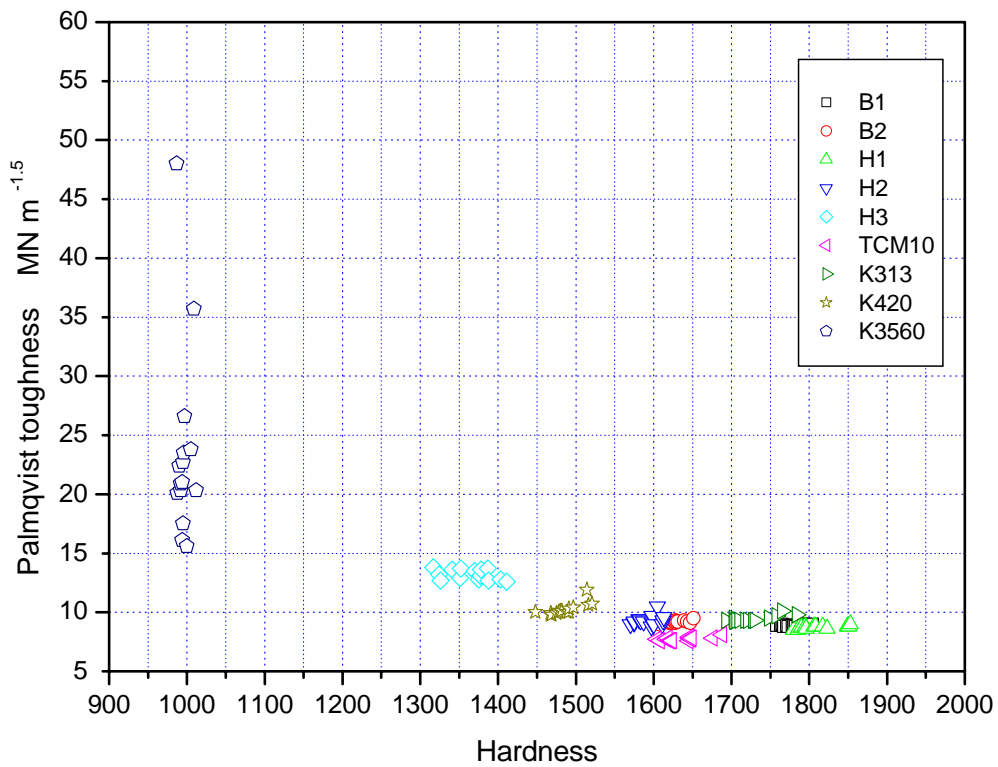
Figure 7- *NPL data – Palmqvist toughness.*
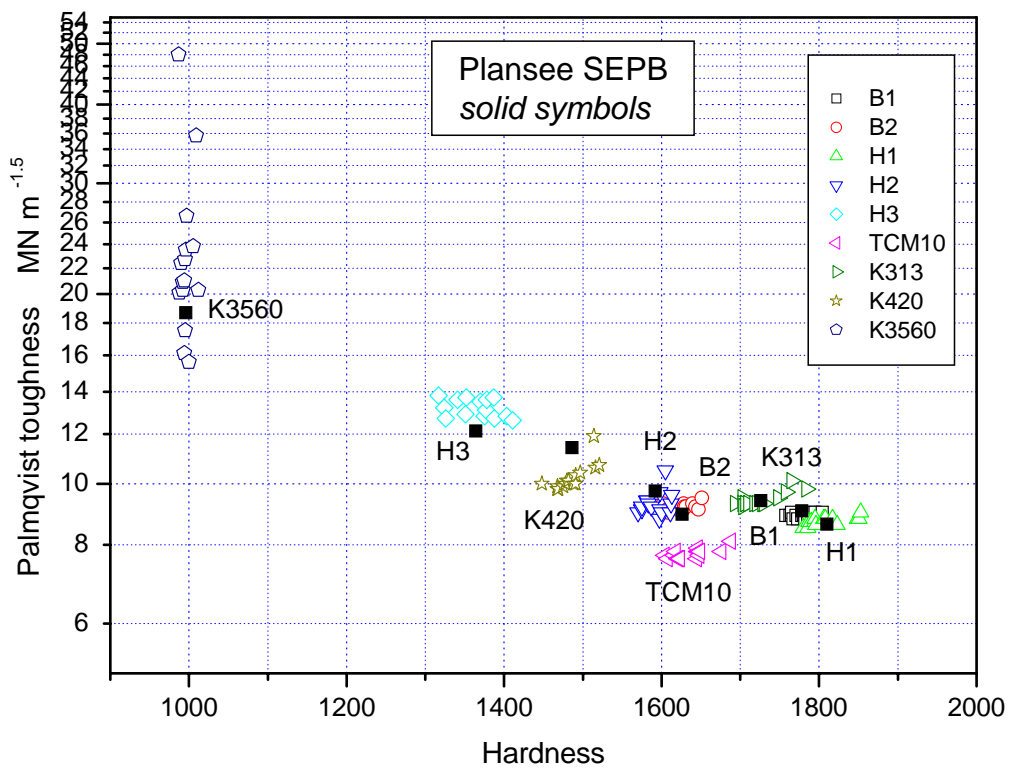
Figure 8 - *Comparison of Plansee SEPB and NPL Palmqvist data (logarithmic scale).*

A coefficient of variation (CV) was calculated for each material in each of the different groups of measurements, and this CV is shown in Figure 9 plotted against hardness for the different measurement strategies. The coefficient of variation decreases with increasing crack length (increasing hardness, decreasing toughness). It was significantly higher when participants were allowed to prepare their own surfaces (including some that were not annealed), but typically varied from 1% to 10% respectively, over a hardness range of 1800-1200 (HV30). The uncertainty expressed as the coefficient of variation in crack length CV_L, for the measurements on the NPL prepared surfaces can be written as a function of hardness

$$Log_{10} \ \ CV_L \ = \ a \ - \ bH \tag{7}$$

where *a* and *b* are constants having values of 2.73 and 0.00125. Typically this corresponds to a standard deviation (SD) of about ± 50 μm at a mean crack length of 150 μm at a Vickers hardness of about 1000 HV100 with a calculated W_K of about 22 ± 4 MN m$^{-3/2}$. Partial differentiation of expression (2) shows that the fractional uncertainty in W_K is equal to half the fractional uncertainty in crack length, *i.e.* equivalent to about 15% at HV1000 and 1% to 2% at HV1000.
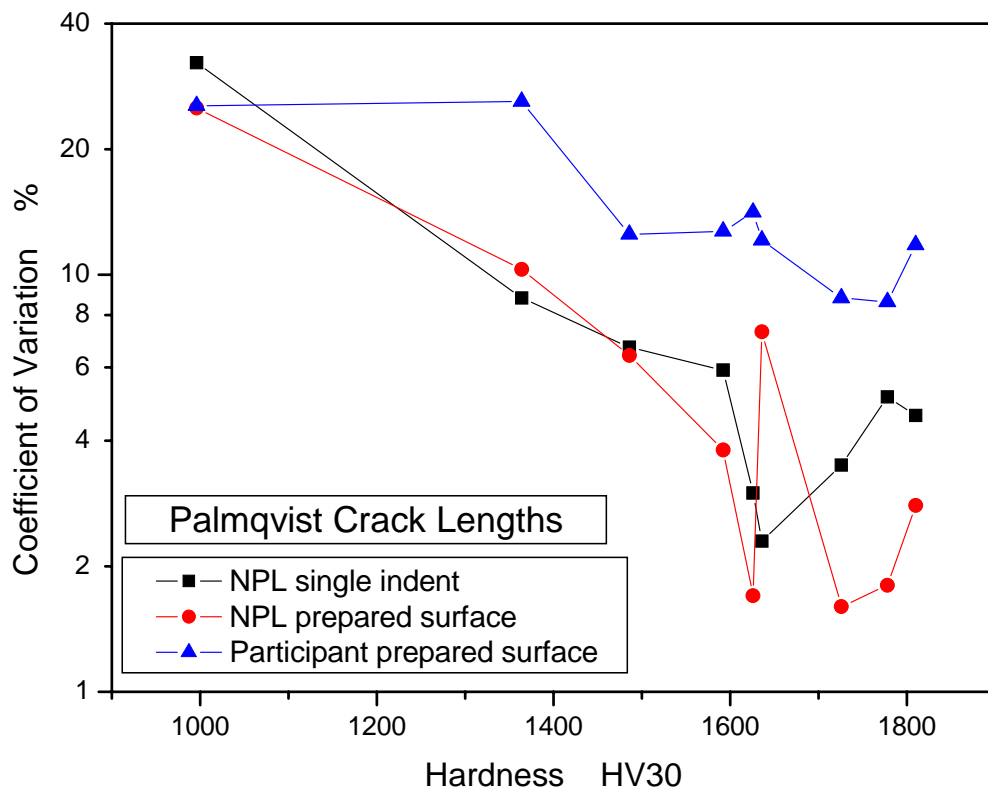
Figure 9 - *Effect of hardness and surface preparation method on coefficient of variation of Palmqvist crack lengths.*

2. Scope of this Guide

- These guidelines are intended for use with metal bonded carbides and carbonitrides (normally called hardmetals, cermets or cemented carbides).

- *Different considerations apply when using indentation tests for ceramic materials owing to different crack geometry and different measurement issues. Appendix D provides a discussion of the problem, and advises the use of alternative methods.*

- The test procedures proposed in these guidelines are intended for use at ambient temperatures, but can be extended to higher or lower temperatures by agreement.

- The tests should be performed in a normal laboratory air environment. They are not intended for use in corrosive environments, such as strong acids or seawater.

- These guidelines apply to a measurement of toughness, called Palmqvist toughness, calculated from the total length of cracks emanating from the corners of a Vickers hardness indentation.

- These guidelines have been validated by the interlaboratory exercise described above [28, 29].

3. Symbols and units

For the purpose of these guidelines the following nomenclature applies:

Symbol	Designation	Units
K_{Ic}	Plane strain fracture toughness	MN m$^{-3/2}$
G	Strain energy release rate	J m^{-2}
W_G	Palmqvist toughness	N mm^{-1}
W_K	Palmqvist fracture toughness	MN m$^{-3/2}$
HV P	Vickers hardness at load P (kgf)	kgf mm^{-2}
P	Indentation load	N (kgf)
T	Total crack length	mm
d	Indentation diagonal mean value	mm
d_1, d_2	Indentation diagonal individual values	mm

4.   Test-pieces and sample preparation

4.1   Test-piece size and sampling

Any test-piece shape can be used provided that it can be prepared with a flat surface and a flat opposing face for making the indentation. Hot mounting in a press gives flat and parallel faces. Cold mounting does not.

Diamond slicing or electro-discharge machines are convenient to use for this purpose. However, the surfaces must then be polished. It is recommended that 0.2 mm of material is removed before the final polish to ensure that material typical of the bulk is tested. For example, the ISO Vickers Hardness Test for hardmetals (ISO 3878) specifies removal of 0.2 mm. It has also been suggested, in a dissertation by M Heinonen (UMIST) [18], that the test-piece should be at least as thick as ten times the crack length. Thinner test-pieces may not give representative results because the stress state will be dependent on the amount of material supporting the indentation and its associated cracks. It can be convenient to mount the test-pieces in cold-setting or hot-setting resins to directly provide flat and parallel faces (see Appendix B). However, if the test-pieces are to be subsequently annealed to remove surface residual stresses then this can be a disadvantage since the test-piece has to be removed from the mount to put it in the annealing furnace (typically 800 ^{o}C for 1 h in vacuum).

4.2   Surface preparation

It is essential to prepare a surface which is flat so that the indentation is of regular geometry. It is recommended that the flatness is confirmed after the indentation is made by measuring the diagonal of the Vickers indentation in orthogonal directions. If the diagonals differ by more than 1% the surface is not flat and the test should be declared invalid.

Grinding should be done wet with metal-bonded 40 μm diamond-impregnated discs since silicon carbide wheels introduce larger residual stresses than diamond. The grinding stage produces a planar surface which then needs to be polished. The recommended sequence of diamond abrasives is at least 30 μm, followed by 6 μm and 1 μm. Napless cloths should be used for the final stages.

This process will produce stress-free surfaces if the final polishing stages are sufficiently long to remove all grinding damage. However, it is difficult to prove that this is the case without extensive comparisons of results from as-polished and polished/annealed test-pieces.

4.3   Surface condition

It has been shown that surfaces free of residual stress are required for consistent results [7]. No polishing procedure can guarantee a stress-free surface without tedious systematic measurements which are not feasible on a regular basis. At NPL all test-pieces are polished so that the microstructure can be observed and then annealed at 800 ^{o}C for 1 h in vacuum following the studies published by Exner [7]. The newer grades of material with very fine WC grain sizes (less than about 0.8 μm as measured by the linear intercept technique on polished and etched

sections) developed in recent years since Exner's work are likely to have even higher surface residual stresses. It may be that longer annealing times or even higher temperatures are required for these materials. In the absence of further work it would probably be sensible to recommend 2 h at 800 ^{o}C. The annealing stage adds to the complexity of the sample preparation process but ensures that the surface is free from residual stresses. If measurements are performed on as-polished surfaces without annealing this must be indicated in the test report.

5.   Apparatus

The indentations should be introduced into the test-piece of interest using test machines calibrated to National Standards. The shape of the indentation should be checked regularly for damage to the indenter tip. The diagonal and crack dimensions can be measured using a microscope attached to the indentation test machine or separately but it should have been calibrated against a standard. Current practice at NPL is given in sections 6.1 and 6.2.

5.1   Indentation

At NPL indentation is carried out on a Vickers hardness testing machine in accordance with BS 427:1990 method for Vickers hardness test and for verification of Vickers hardness testing machine. A NAMAS certified diamond indenter is used.

5.2   Indentation and crack measurement

Indent diagonals and cracks are measured using a NAMAS accredited Reichert Univar microscope. The image is projected onto a projection screen which has been calibrated using a stage graticule traceable to national standards.

6   Procedure and conditions of testing

6.1   Indentations

Indentations should be made in a deadweight hardness machine which is calibrated at least annually. The recommended procedure is to make indentations using a Vickers diamond indenter at one load rather than a series of loads. The indentations can be made at 30 kgf or 60 kgf. However, 30 kgf is recommended. Two indentations should be made initially and the toughness values for each indentation compared. If they are within the measurement uncertainty associated with the procedure (see section 7.6 - Analysis) the two measurements are considered satisfactory. If they differ by more than this uncertainty a third indentation is made and the result reported as an average with an associated standard deviation. If the two measurements are within the estimated measurement uncertainty then an average value of the two measurements is reported without a standard deviation.

It is also possible to make the measurement of *W* by indenting with a series of loads and plotting the total crack lengths obtained against the load for each indentation. If this method is used to obtain a value for W_G and W_K then it must be noted in the test report.

6.2   Indentation and crack length measurements

It is recommended that the indentation diagonal and crack lengths are measured optically at a magnification of at least x500. Alternatively, the optical system of the Vickers hardness machine can be used (x100) as this has been shown to give equivalent results (see Appendix A). The magnification used should be calibrated for each measurement session using a traceable grid.

Either take photographs of the indentation and cracks or project the image onto a measurement screen if a suitable microscope is available. Measure both indentation diagonals. Record both values. *If the diagonals differ by more than 2 mm at x500 magnification the test should be repeated because of a lack of flatness of the test-piece.*

There are two methods for measuring the crack length. The results are independent of the method (see Appendix B). Either method can be used.

Method A:

Measure crack tip to crack tip for both diagonal directions. The total crack length is the sum of both these values minus the sum of the indentation diagonals (see Figure 2). *If the magnification is x500 this method is impractical because the crack tip to crack tip distance is usually too large to include in one image.*

Method B:

Measure individual crack lengths at x500 from indentation corner to crack tip for each of the four cracks. Sum to give a total crack length. If the crack root does not coincide with the tip of the indentation diagonal measure the crack length from where the crack initiates along the edge of the indentation.

6.3   Test validity

If there is more than one crack emanating from the indentation corner the indentation should be ignored as measurement is invalid.

If the total crack length is less than 40 μm the test should be considered invalid. *For a 60 kgf load this corresponds to a toughness value, W_G, of 7360 N m^{-1} (or 25 MN m$^{-3/2}$ for W_K) for a material with a hardness of 1100 HV60. These materials are likely to have coarse structures and the individual crack lengths at each indentation corner will be no more than one or two grains long. This is too short to be confident that the crack is sampling a representative volume of hardmetal.*

If the indentation diagonals differ by more than 4 μm for an indentation load of 30 kgf the surface is not sufficiently flat and the test is invalid.

7. Analysis

7.1 Vickers hardness

Take the average value of the two diagonals in mm and convert to a true value, d, in mm by dividing by the calibrated value for the magnification. The Vickers hardness, HV, is given by

$$HV = \frac{1.8544\,P}{d^2}$$

where P is the load in kgf and d is the average indentation diagonal in mm. Express as HV30 or HV60 corresponding to the load used in kgf.

The hardness number should be rounded to the nearest 5.

7.2 Toughness

Two different values for toughness can be calculated, Palmqvist toughness, W_G, and Palmqvist fracture toughness, W_K (see introduction).

$$W_G = \frac{P}{T} \quad \text{(N mm}^{-1}\text{ or J m}^{-2}\text{, 1 N mm}^{-1} = 1000 \text{ J m}^{-2}\text{)}$$

where for method 1 (simple indentation load) P is the load in N and T is the total crack length in mm and for method 2 (multiple loads) P/T is the inverse of the slope of a plot of total crack length against load.

$$W_K = A\,\sqrt{HV}\,\sqrt{W_G} \quad \text{(MN m}^{-3/2}\text{)}$$

where   A is a constant of value 0.0028
and   HV is the Vickers hardness in N mm^{-2} (*i.e.* 9.81 x numerical value of HV hardness number) and W_G is in N mm^{-1}.

Calculate both values and report with a mean value if two indentations/sample are made and a mean value and standard deviation if three or more indentations/sample are made.

The results should be reported to three significant figures only.

8. Test report

It is recommended that the proforma given in Appendix A is used for the test report which shall contain at least the following information:

- reference to this guide

- material type and condition

- test-piece identification

- location and direction of sampling of test-piece

- surface preparation method

- load of indentation

- analysis method

- results in tabular or computer file format

- validity assessment.

9. References and other related procedural documents

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10.   Acknowledgements

Appendix A - Report proforma

Palmqvist Toughness Tests on Hardmetals and Cermets Measurement Report

(Typical inputs are included for guidance.)

A.1   Material

Reference	Description	Comment
Material	Source Identifier Composition Coercivity Magnetic moment Form Heat treatment/processing	*Marshalls Hard Metals* *MHM15C* *WC/6%Co* *50 kA m^{-1}* *0.80 $\mu Tm^3 kg^{-1}$* *Rectangular block* *As-sintered*
Test-piece Information	Size Polished area	*40 x 5 x 20 mm* *40 x 20 mm*
Procedure	Applicable standard(s)	*

*Refer to this guide and other documents as appropriate.

A2   Testing organisation

Organisation:	*XXXXX*
Name:	*YYYYY*
Signature:	
Date:	*15 March 1997*

Indentation parameters:

Reference	Description	Comment
Surface Preparation	Polished only **or** Polished and annealed	*Polished and annealed at 800 °C for 1 h in vacuum*
Test Procedure	Method 1 Single indentation load Method 2 Multiple indentation loads Number of indents Environment Crack length measurement method Indent diagonal measurement method	*30 kgf* *Not used* *3* *Room temperature, air* *Optical, x515 (calibrated)* *Optical, x515 (calibrated)*
Test Validity	Consistency of indentation shape Total crack length > 40 μm Multiple corner cracks	*Yes, valid test* *Yes, valid test* *No, valid test*

Results and analysis: (*Use spreadsheet*)

Individual readings (*For 3 samples, 3 measurements/sample*)	Indent diagonal** μm			Hardness* HV30	Total crack length**, μm	Palmqvist Toughness**	
Sample code	d_1	d_2	mean			W_G N mm^{-1}	W_K MN m$^{-3/2}$
MHM15Ca1	*204*	*204*	*204*	*1335*	*162*	*1820*	*13.7*
MHM15Ca2	*203*	*203*	*203*	*1350*	*168*	*1750*	*13.5*
MHM15Ca3	*206*	*204*	*205*	*1325*	*155*	*1900*	*13.9*
MHM15Cb1	*203*	*205*	*204*	*1335*	*132*	*2230*	*15.1*
MHM15Cb2	*206*	*204*	*205*	*1325*	*143*	*2060*	*14.5*
MHM15Cb3	*204*	*204*	*204*	*1335*	*128*	*2300*	*15.4*
MHM15Cc1	*204*	*204*	*204*	*1335*	*160*	*1840*	*13.8*
MHM15Cc2	*203*	*205*	*204*	*1335*	*142*	*2070*	*14.6*
MHM15Cc3	*203*	*203*	*203*	*1350*	*138*	*2130*	*14.9*

*   Rounded to nearest 5
**   To three significant figures

Mean values:

Sample Code	Hardness* mean value HV30	Palmqvist Toughness**			
		W_G, N mm^{-1}	S.D.	W_K MN m$^{-3/2}$	S.D.
MHM15C	*1335*	*2010*	*± 190*	*14.4*	*± 0.70*

*   Rounded to nearest 5
**   To three significant figures, S.D. - standard deviation, if appropriate

Appendix B - Measurement case studies at NPL

Two exercises were conducted at NPL for the original edition of this document to examine the measurement uncertainty associated with Palmqvist toughness tests.

1. Measurements on WC/Co and Ti(C,N) cermets were compared using different methods and reported in 'The Palmqvist test method for Ti-based cermets and WC-Co hardmetals'; NPL Report DMM(A)33; October 1991; M.G. Gee, L.A. Lay and B. Roebuck.

2. Additional work was performed on the surface preparation method. The effects of different polishing methods for Palmqvist toughness tests on a WC/6%Co hardmetal were evaluated in work not yet formally reported, L.A. Lay and E.G. Bennett - 1997.

The outcome of these two exercises can be summarised as follows.

B.1   Report DMM (A) 33

The following questions were addressed for tests on a range of hardmetals and cermets:

*   For one operator, one method -
    what is the uncertainty in indentation size and crack length?
*   For several operators, same indentation or independent indentations -
    what is the uncertainty in indentation size and crack length?
*   What are the effects of annealing?
*   What is the level of discrimination in Palmqvist toughness values?

Several methods were used to measure the indentation diagonals and crack lengths:

1. Using the microscope on the Vickers hardness machine at x100. Tip to tip crack length minus the indentation diagonal for crack length.
2. Using an optical microscope at x500, separate from the indentation. Individual cracks were measured from each crack root.
3. Using a high resolution field emission scanning electron microscope (SEM) to measure crack lengths and indentation diagonals.

It was concluded that:

*   Measurements at low indentation loads, less than 10 kgf result in large variations in crack lengths and are not recommended.

*   Indentation diagonals can be measured to ± 2 μm for one operator and ± 4 μm for several operators. This was similar both for x500 and x100 (optical) magnifications. The SEM gave higher uncertainties due to the difficulty of seeing

the indentation corner in an SEM image.

- For one operator the crack length can be measured to ± 20 μm using optical methods. The uncertainty was similar when different operators measured the crack length.

These uncertainties can be converted to Palmqvist toughness values as follows:

Nominal hardness HV30	Uncertainty in			Nominal Palmqvist toughness		Uncertainty in	
	Indentation diagonal ± μm	Hardness ± HV30	Crack length ± μm	W_G, N mm^{-1}	W_K, MN m$^{-3/2}$	W_G, ± N mm^{-1} (%)	W_K, ± MN m$^{-3/2}$ (%)
1000	2	20	20	7660	24.3	2750 (36)	4.9 (20)
1000	4	40					
1250	2	25	20	2900	16.7	550 (19)	1.3(8)
1250	4	50					
1500	2	30	20	1310	12.3	110 (8)	0.5 (4)
1500	4	60					
1750	2	35	20	670	9.5	30 (4)	0.2 (2)
1750	4	70					
2000	2	50	20	380	7.6	10(2½)	0.1 (1)
2000	4	100					

- The measurements using the optical microscope at x100 on the Vickers hardness machine and x500 on a separate microscope gave similar results within the measurement uncertainty.

- The crack lengths measured using the SEM were systematically higher by about 10%. The implications are plotted below in Figure B1.
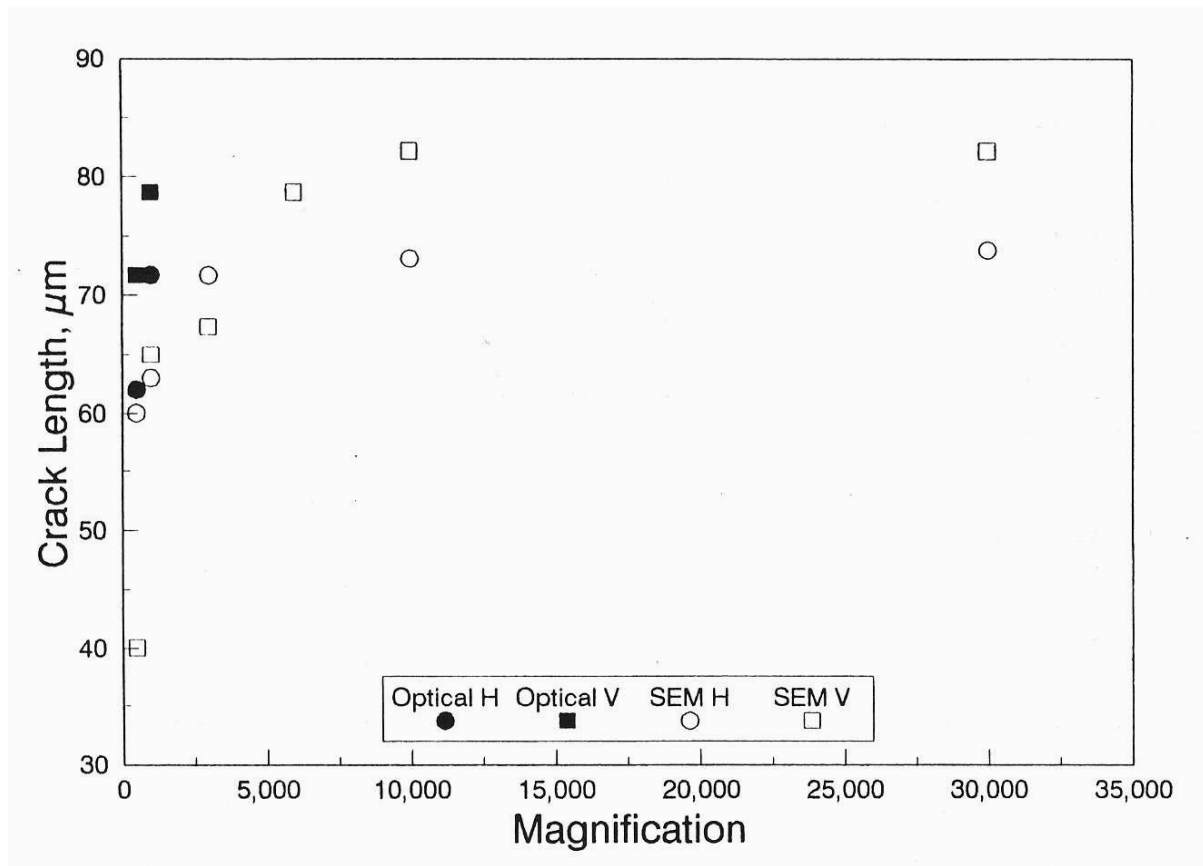
Figure B1 *Crack length measured using optical and FESEM methods on a 15 kgf indentation. H corresponds to horizontal cracks and V for vertical cracks.*

- Surface preparation has a very significant effect on the Palmqvist toughness values, as found by Exner [7]. Annealing removes compressive residual stresses.

- The ability of the Palmqvist method to differentiate between materials depends on the hardness. For HV2000 the uncertainty is $\pm$ 10 N mm^{-1}, for HV1500 it is $\pm$ 110 N mm^{-1} and for HV1250 it is $\pm$ 550 N mm^{-1}.

- Palmqvist toughness values calculated from single high load indentations are as effective as parameters calculated from analysis of a series of indentations at different loads.

B.2   Additional work on surface preparation

Two polishing methods were examined, one targeted to maximise residual compressive stresses and one to minimise stresses.

Minimise surface stresses			Maximise surface stresses		
30 μm	Tin lap	30 min	40 μm	Diadisc	5 min
6 μm	Tin lap	30 min	30 μm	Diadisc	5 min
6 μm	Napless cloth	30 min	10 μm	Diadisc	5 min
1 μm	Napless cloth	30 min	1 μm	Napless cloth	30 min
1 μm	Napped cloth	10 min	1 μm	Napped cloth	10 min

Samples were either resin mounted or unmounted and cracks were measured tip-to-tip or individually. Samples were then re-examined after annealing. The results were as follows:

Sample	Action	Crack length, μm	Palmqvist toughness, W_K, MN m$^{-3/2}$
	Polishing Method		
KRM2	Minimise stress	433	13.4
KRM3	Maximise stress	305	16.0
KRM4	KRM3 repolished to minimise stresses	435	13.4
KRM2	KRM2 annealed		13.4
	Measurement		
KRM1	Tip-to-Tip	407	13.9
KRM1	Individual	409	13.8
	Mounting		
KRM1	Resin mounted	408	13.8
KRM6	Unmounted	401	13.8

It can be seen that the polishing technique that maximised stresses gave a much higher toughness value than the technique to minimise stresses. The latter method gave the same result as in samples that were annealed, indicating that it is possible to polish samples to produce surfaces free from residual stress. However, the preparation time was extremely long.

The same toughness value was obtained in unmounted as in mounted specimens and it did not matter whether the crack was measured tip-to-tip or individually.

Appendix C - Literature resumé

There is a considerable body of scientific literature concerned with Palmqvist toughness tests for almost 40 years on Hardmetals and Cermets (see Section 9 for references.) This Appendix reviews this literature from the point of view of issues concerned with the measurement method.

PALMQVIST - 1957, 1962

Developed the original experimental approach to measure crack length as a parameter corresponding to toughness. Recognised the importance of surface preparation.

DAWIHL and ALTMEYER - 1964

Used crack lengths to measure surface residual stresses.

EXNER - 1969

Exner proposed use of a parameter, *W*, (*P/a*, *i.e.* slope of the load/crack length plot), for a measure of Palmqvist toughness.

Exner demonstrated that surface preparation is critical in producing repeatable values for Palmqvist toughness. He showed that compressive residual stresses are introduced by grinding, which are higher for SiC wheels than for diamond. These compressive stresses result in high apparent values of Palmqvist toughness. Annealing at 800 $^{\circ}$C or prolonged polishing removes the residual stresses and give more representative values of Palmqvist toughness. Exner also reviewed earlier work by Palmqvist and Japanese workers which had equally shown the variability in results that could occur from different surface preparation techniques.

He also concluded that the optimum measurement method involved making measurements of crack length at different loads and the inverse of the slope of the crack length/load plot was a measure of toughness. This avoids standardisation of specific loads at which to do the test, since the more tough grades of hardmetal require higher loads to produce significant cracks. He claimed that individual cracks could be measured with an uncertainty of $\pm$ 2 µm which is considerably smaller than has been found in measurements at NPL.

ALMOND and ROEBUCK - 1973, 1979, 1981

SEM observations of the indentation process showed that at least 50% of the cracking that occurs happens during unloading of the indenter. This work also showed that cracking during indentation is a continuous event. A crack is formed which moves ahead of the indenter as it pushes into the material and is closed up behind at the crack root as the indenter moves over the cracked region.

PETERS - 1979

The coefficient of variation of crack lengths was found to range from 3.5 to 8.5%. A very extensive polishing routine was used to prepare stress-free surfaces involving 2 h of polishing with different, successively finer, grades of diamond paste.

WARREN and MATZKE - 1981

Gives a derivation for the expression used to calculate a K_{Ic} value from the hardness and P/a value from Palmqvist tests. Values for P/a were obtained in general from single load tests with $P > 20$ kg. None of the materials were annealed. All were polished to 1 μm diamond.

NIIHARA, MORENA and HASSELMAN - 1982

Median vent crack formulae for calculating K_{Ic} values were not appropriate for use with indentation cracks in hardmetals because the cracks are of the Palmqvist type. Developed an approach to calculate K_{Ic} from $K_{Ic} = A(HW_G)^{1/2}$, where A is a constant.

SHETTY, WRIGHT, MINCER and CLAUER - 1985

Evaluated the Warren and Matzke and the Niihara *et al.* expressions for calculating a value for fracture toughness, K_{Ic}:

$$K_{1c} = A(HW_G)^{1/2}$$

Provided an analysis for the derivation of the value of the coefficient A in the above expression.

SINGH and RAMAKRISHNAN - 1985

Concluded that standardisation of the polishing procedure was necessary for obtaining repeatable results for Palmqvist toughness.

LAUGIER - 1989

Noted that for tough grades of hardmetal the corner cracks are very short compared with the indentation diagonal. He recommended that valid tests from reliable measurements of crack length required that $(d + 2c)/d > 1.3$ where c is the length of one corner crack and d is the indentation diagonal.

A rule of thumb was developed for polishing surfaces to remove residual stresses. The time required to polish any grinding grooves was measured and polishing was then continued for a further, equal, period.

HEINONEN - 1996

Presents a convenient summary of equations for calculating toughness from Palmqvist parameters. Good micrographs of Palmqvist crack profiles obtained by staining cracks. Recommends that test-pieces should be at least as thick as ten times the crack length.

SCHUBERT, NEUMEISTER, KINGER AND LUX - 1997

A very thorough investigation of the use of the Palmqvist test method for measuring the toughness of fine and very fine grained hardmetals. They developed a polishing routine (without using an annealing stage) to prepare stress-free surfaces. The uncertainty in measurement of both hardness and crack length was estimated as:

Property	Experienced single operator (repeatability)	Different operators/ Different measurement techniques (reproducibility)
Hardness	± 20 HV30	± 30 HV30 (<2000) ± 50 HV30 (>2000)
Crack length	± 20 μm	± 20 μm

ROEBUCK AND BENNETT – 2005

VAMAS report on extensive interlaboratory comparison of Palmqvist method with valid K_{Ic} measurements. Uncertainties were quantified and method/procedures recommended.

ROEBUCK, HAMANN AND BENNETT - 2005

Assessment of the Palmqvist method for tough hardmetals, limitations and images of short cracks in coarse-grained materials.

GARBER, SCHLEINKOFER AND GREIF – 2005

Demonstrate that a new design of stiff pre-cracking apparatus can generate controlled cracks of known geometry in a range of hardmetals leading to valid K_{Ic} measurements.

Appendix D - Application of the indentation crack length test to ceramic materials

D.1   Introduction

Although the execution of the test is similar to that for hardmetal materials, there are a number of very significant differences that come into play when dealing with ceramic materials. The principal ones are:

- Hardmetals are opaque and of metallic appearance, and cracks are relatively easy to see on a polished surface. Ceramics, on the other hand, may vary between highly and semi-translucent, usually with some porosity, and optical observation is hampered by internal light scattering.

- Hardmetals are typically indented at HV30, whereas only a few fine-grained ceramics can tolerate more than an HV10 indentation without flaking or spalling around the indentation because of the generally lower toughness.

- When there is more than about 2 volume % porosity, indentation tends to result in crushing rather than the required wedging deformation, and cracks are thus shorter or not present, leading to radical overestimates of toughness.

- The microstructures of ceramics are very varied and the micromechanisms of fracture are generally different to those observed in metals. Many ceramics are deliberately coarse grained. Fractures often occur along grain boundaries, giving meandering, sometimes branching, cracks rather than the relatively straight cracks seen in most hardmetals.

- The crack geometry is not often the same as Palmqvist cracks which are restricted to four shallow lobes. Instead, in low toughness ceramics the crack may be half-penny shaped (Figure D1), while in materials with greater toughness, the geometry can be intermediate between half-penny and Palmqvist. This introduces uncertainty of the choice and validity of the equation to be used for calculating toughness.

- The fracture mechanical basis for calculating fracture toughness has uncertainties and there is a wide choice of equations that could be used.

This annex discusses some of these issues, and proposes conditions under which indentation fracture toughness may have some value for ceramic materials.
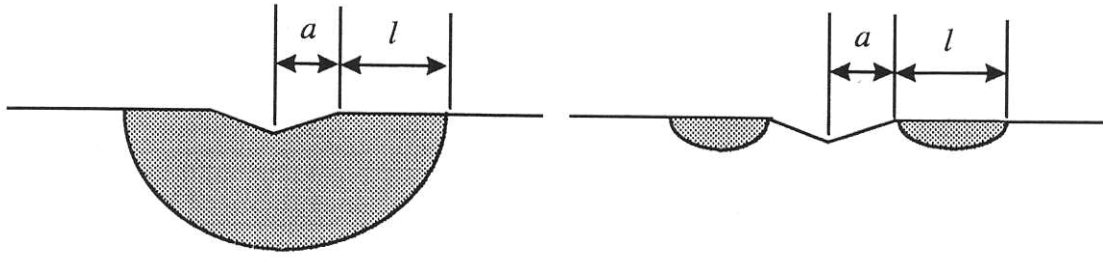
Figure D1 *Schematic diagram of (left) half-penny and (right) Palmqvist cracks typically developed in ceramic materials.*

0.2 mm

50 µm
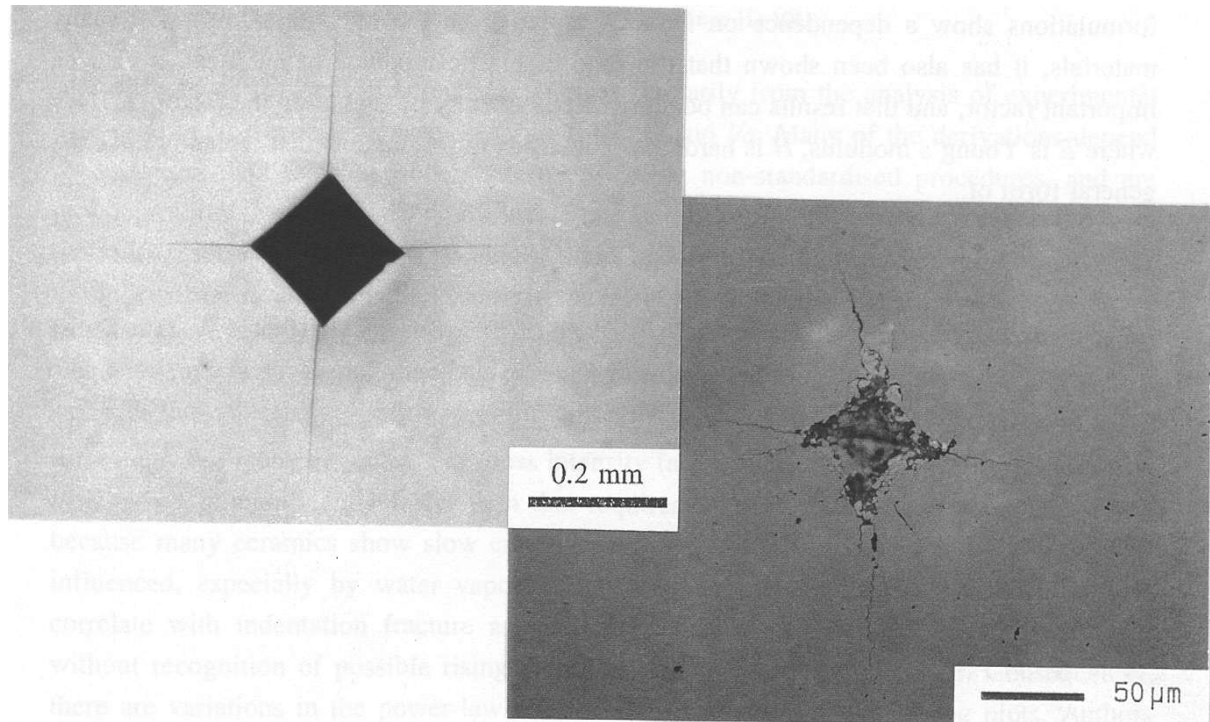
Figure D2 *An illustration of the experimental problem of applying the test to ceramic materials: (left) a tungsten carbide hardmetal indented at 50 kgf showing clearly developed straight cracks, and (right) a sintered silicon carbide of low fracture toughness indented at only 5 kgf showing a poor quality of indentation, and meandering and bifurcating cracks.*

D.2   Theoretical basis

D.2.1   Introduction

A large number of different formulae have been derived to relate the crack half-length to a toughness parameter, usually K_c. These formulae are usually based on an estimate of the stress field developed under a Vickers indenter, coupled with a correlation of indentation crack lengths with K_c determined by other methods to provide dimensional factors. Unfortunately, it is generally held that the different formulae are subject to large potential errors for different reasons, and it is by no means clear which formula, if any, is 'correct'. Generally speaking none of them can have general validity, because different ceramics behave in different ways regarding crack growth behaviour, and this is reflected in the `calibration' data employed.

D.2.2 Indentation theory

It has been shown that, for a material with a well-defined toughness not displaying any significant R-curve behaviour (rising apparent toughness with increasing crack length) over the dimensions of the crack, the surface crack length c (from the indentation centre) is proportional to the indenting force P to the 2/3 power. This relationship has been demonstrated theoretically for a penny-shaped crack wedged at its centre [D.1]. Alternative formulations show a dependence on l (crack length from the indentation tip). For many materials, it has also been shown that the ratio of elastic modulus to hardness is also an important factor, and that results can be normalised typically by including the factor $(E/H)^{1/2}$ where E is Young's modulus, H is hardness. This leads to formulae which typically have the general form of:

$$K_c = \beta_0 (E/H)^{1/2} (P/c^{3/2})$$

but with some variations in the numerical values of the powers. The factor β_0 is a fitting parameter which permits the 'correct' value of K_c to be obtained. Some typical equations with their β_0 values are shown in Table D.1. Note that in most of the literature the half-diagonal length $a = d/2$ is used.

The differences between these equations arises primarily from the analysis of experimental data used to fit the dependences of K_c on E/H, c/a and l/a. Many of the derivations depend on historical data that have been determined using non-standardised procedures, and are probably unreliable. The indentation test is one in which the crack length is measured after the indentation is made. It is a nominally stable crack driven by a hoop tensile stress while the indentation is under load and, after removal of the indenting force, the crack continues to be wedged by the plastically deformed zone under the indentation and may continue to grow. The stress field rapidly decays with distance from the centre of the indentation so that the driven crack has entered a region of the stress field which is no longer sufficient to move it forward. For many ceramics, the stress intensity factor K just insufficient to permit a crack to lengthen is significantly lower than that required to cause fast fracture, K_c. This arises because many ceramics show slow crack growth behaviour which is also environmentally influenced, especially by water vapour. In contrast, most experimental K_c data used to correlate with indentation fracture are from larger-crack, fast fracture tests, and usually without recognition of

possible rising crack resistance (R-curve) behaviour. Consequently, there are variations in the power-law relationships determined from log-log plots. Authors have tended to look for the best fits of their formulae to data for a variety of materials but, because of uncertainties, have ignored ancillary factors such as Poisson's ratio and indenter/test-piece friction. Different relationships have been found when restricting the range of *c/a* because different classes of materials are excluded, *e.g.* because the crack geometry tends to be part-way towards the Palmqvist type. Some analyses deal with the potential effects of subcritical crack growth and surface pre-stressing by preparation techniques, which is most marked in transformation strengthened zirconia materials [*e.g.* D.12] but can exist in all brittle materials [D.13]. Surface stresses have the effect of restricting the length of the crack at the surface, but not necessarily beneath the surface. Reliance on surface crack length measurement can then lead to an overestimate of toughness.

Table D.1: Example half-penny geometry indentation toughness equations

Equation	Source
$K_c = 0.036\ E^{0.4}\ P^{0.6}\ a^{-0.7}\ (c/a)^{-1.5}$	Marshall and Evans [D.2]
$K_c = 0.0264\ E^{0.5}\ P^{0.5}\ a\ c^{-1.5}$	Miyoshi *et al.* [D.3], JIS R1607-1990 [D.4]
$K_c = 0.016\ (E/H)^{0.5}\ (P/c)^{1.5}$	Anstis *et al.* [D.5]
$K_c = 0.010\ (E/H)^{0.667}\ P\ c^{-1.5}$, half-penny $K_c = 0.015\ (E/H)^{0.667}\ P\ c^{-1.5}\ (l/a)^{-0.5}$, Palmqvist	Laugier [D.6] Laugier [D.7]
$K_c = 0.0711\ H_v\ a^{0.5}\ (E/H_v)^{0.4}\ (c/a)^{-1.5}$, $c/a \geq\approx 2.5$ $K_c = 0.035\ (H_v/\varphi)\ a^{0.5}\ (E\varphi/H_v)^{0.4}\ (l/a)^{-1/2}$, $c/a \leq\approx 2.5$	Niihara *et al.* [D.8] $\varphi \approx 2.7$
$K_c = 0.035\ (E/H_v)^{0.25}\ P\ c^{-1.5}$	Tanaka [D.9]

Note: It is often the case in these equations that $H_v = (2P\ sin\theta)/d^2$ (the hardness based on the true surface area of the indentation of diagonal $d = 2a$ made with an indenter of included face angle 2θ, = 136° for a standard Vickers indenter) is used interchangeably with force and indentation size. It should be noted that some analyses use mean pressure hardness, $H = 2P/d^2$, derived from the projected area of the indentation, while in others it is left to the reader's imagination. A rationalisation of equations has been proposed by Ponton and Rawlings [D.10].

Ceramics often contain pores between 0.1 μ and 10 μm across, frequently up to 5 vol% in many sintered products. On loading an indentation into such a material, the pores collapse and the material locally crushes with restricted on no development of the desired radial cracking. Results can be misleading or impossible to obtain.

It should also be noted that for Palmqvist type cracks, or cracks intermediate between classical Palmqvist and half-penny shapes, the equations developed in the ceramic literature are different from those conventionally used for hardmetals, notably in their use of the dimensionless ratio (*E/H*). This arises in part from different approaches to the analysis, and to the use of different data in the fitting of constants. In particular, the range of values of E for hardmetals is small compared with that which can be used for ceramics.

D.2.3 Using the formulae

The accuracy of calculation of K_c using any one of the formulae is likely to be in error by possibly ± 30%, as stated by some of the authors, and this is in addition to experimental errors of crack length measurement. Caution must be used in using any particular formula because of possibly restricted validity, notably of *c/a* ratio. The only method of reducing this potential error is for systematic work to be done to determine the true relationship on a single class of material over a wide range of indenting force, taking all known factors into account. Even so, this test still does not have the fracture mechanics pedigree of tests with well-defined stress-free initial crack shapes which can be analysed mathematically. **Consequently, the test cannot be recommended if accurate fracture toughness data are required.**

D.3   Experimental issues

If the reader intends to pursue making indentation fracture toughness measurements, this section is intended to give some guidance concerning good practice in the preparation of test samples, methods of making the crack length measurements, and the criteria needed for the measurements to be acceptable.

D.3.1 Surface preparation

The first requirement is to prepare a good polished surface so that the cracks are visible against a plain scratch-free background. A common problem is that the early stages with coarse grinding grits can cause sub-surface damage, so that grain tear-out occurs during later polishing stages. In addition it is necessary to eliminate any compressive stresses introduced during the early stages of preparation, since these have the effect of restricting crack length.

The first step in preparing a test-piece is to obtain a planar surface. If an as-received surface is being prepared, the outer zone should be removed since it may not be representative of the bulk of the material, having been exposed to the atmosphere during sintering. Then, a series of increasingly fine diamond abrasives is used on suitable cloths to obtain a polished surface. A final chemical polishing stage may be used to produce a superior finish.

Planar grinding may be carried out using bonded diamond discs, or a slurry of loose diamond abrasive on a platen. The former method is quicker, but introduces stresses and produces sub-surface damage. The use of loose abrasives on a shock-absorbing platen minimises the introduction of stresses and helps to prevent sub-surface damage and is therefore preferable. A sequence of 30 μm, 6 μm, 1 μm and ¼ μm diamond grits gives good results with most ceramics. It is generally accepted that the thickness of the layer to be removed should be at least half the grit diameter used for the preceding step. The removal of material can be monitored by measuring the size of a Vickers hardness diagonal before and after a polishing stage. The depth removed is about one seventh of the reduction in the mean diagonal. A lap such as a tin/plastic composite can be used for the first stage, and napless cloths are suitable for the other stages. The use of napped cloths is not advisable for polishing ceramics, since they can cause tear-out. A

final chemical polishing stage using alkaline colloidal silica (such as Mastermet from Buehler) on a napless cloth gives excellent results with many ceramics, and helps to reduce residual stresses.

An alternative method of reducing stresses in the surface is by annealing. However it may be necessary to determine an appropriate annealing schedule by trial and error because each material will behave differently. Clearly, the annealing process must not change the microstructure or cause oxidation or any other type of surface degradation, such as thermal etching. On balance it seems preferable to use polishing methods which, although slower, minimise the introduction of stresses in the first place.

D.3.2   Surface coatings

Most ceramics are optically translucent to varying degrees, and it can be very difficult to see the cracks because they are masked by internal diffuse reflections. It has been suggested that a surface coating of gold or gold alloy (about 20 to 40 nm thick, *i.e.* not resolvable optically) before making the indentations will make the cracks more visible [D.11]. However, this could affect indentation behaviour by influencing the friction between indenter and test-piece, leading to an error in hardness measurement. Coating after indentation has been used to improve surface reflectance and to mask internal reflections, but unless the coating is very thin very narrow crack tips can be bridged.

D.3.3   Indentation quality

It is convenient to use a standard Vickers hardness (or micro-hardness) tester, but it is also possible to use a suitable strength testing machine equipped with a standard Vickers indenter. The diamond indenter must be of good quality, with no chips, especially on the edges, and must be aligned so that the indentations produced are symmetrical with no loss of definition of the corners by chipping, flaking or sideways displacement of grains. **This is essential because all formulations require values of H or a as well as c.**

The load required depends on the composition and microstructure of the ceramic. In all ceramics a threshold force must be exceeded before any cracking is observed. This is typically of the order 5 N to 10 N (*i.e.* HV0.5 to HV1), although may be much lower in some very brittle materials. This threshold force is influenced by surface finish, increasing as the quality of the surface improves. Generally, forces a little above the threshold produce shallow Palmqvist cracks, and higher forces form `half-penny' cracks, the transition occurring at a *c/a* ratio of about 2.5. The load applied should thus be sufficient to give a *c/a* ratio of at least 2.5 to achieve half-penny type cracks, though this condition cannot be achieved in all ceramics. Typical loads quoted in the literature for testing different types of ceramic are given in Table D.2. In some cases these ranges include low values which give only Palmqvist cracks.

To ensure no interference between indentations they should be positioned with centres no less than about 4*c* apart [D.11].

Table D.2: Typical indentation forces used for ceramics

Ceramic type	Force, N (9.80665 N = 1 kgf)
Silicon nitrides and sialons	10 - 400
Silicon carbides	5 - 200
Aluminas	30 - 500
Zirconias	30 - 500
Zirconia toughened aluminas	50 - 500
Glass-ceramics	50 - 200
Ceramic matrix composites	50 - 300

D.3.4   Radial crack quality

If the indenter is well-aligned and its motion is perpendicular to the test-piece surface, the four cracks originating from the indentation corners or very close to them should be fairly straight and roughly equal in length. Some materials do not yield such ideal crack patterns; cracks may meander, be very variable in length, or appear to halt at pores. In such cases it is simply not feasible to make meaningful measurements. In some marginal cases, more than four cracks might be produced, or the main radial cracks may bifurcate. Such indentations should be ignored. Even in a well-behaved material, the crack lengths may vary by ± 15 % about the average value.

The presence of significant amounts of porosity in the material tends to make the individual crack lengths more variable. Cracks can be deflected towards pores, and be stopped by them. In some cases radial cracks may be prevented from developing. Ideally the test should be used only on materials of low (< 2%) and fine-scale porosity.

D.3.5   Measuring crack lengths

If the indentations are made using a standard hardness tester, the cracks can be measured using the microscope fitted to the machine. Alternatively, the test-piece will need to be transferred to a microscope with a calibrated stage micrometer. The measurements should be made as soon as possible after withdrawal of the indenter owing to the possibility of slow crack growth. Penetrant dyes have been used to make the cracks more visible, but this technique carries the risk of causing crack growth. Some workers have applied a drop of silicone oil or immersion oil to the indentation immediately after unloading in order to minimise slow crack growth effects from moisture in the atmosphere [D.5, D.12] and thus to restrict slow crack growth. Adding oil before indenting may change the frictional contact between indenter and test-piece.

For many ceramics, the application of a thin reflective metal coating aids the visibility of the crack, although within the resolution of an optical microscope, the true tip may be significantly further from the indentation than can be readily seen, since the crack opening displacement is very small. The use of dark-field illumination, or simply a restricted aperture diameter with the

crack tip in the region not directly illuminated, can help to identify the true crack tip.

Alternatively, a scanning electron microscope can be used with calibrated magnification and/or stage movement. Although the SEM has a high resolution, it can still be difficult to track a crack, especially when a coating has to be applied to the specimen to aid electron conduction.

Comparisons have been made between optical crack length measurements and results obtained by scanning electron microscopy, using a glass-ceramic specimen [D.11]. The SEM results tended to identify slightly longer crack lengths (in accordance with NPL experience), but the difference was no more than about 4%. The conclusion is that the accuracy of optical measurements is acceptable, except for small loads (less than HV5) which produced short cracks.

D.3.6 Subsurface crack shape

Since the interpretation of indentation fracture toughness measurements depends on knowledge of sub-surface crack shapes, numerous studies have been carried out on this topic. A number of techniques have been used, but the most popular is by step-wise polishing away of the indentations. The depth removed is monitored by measuring the reduction in indentation diagonal, and the crack shape is inferred by observing the surface crack pattern (Figure D3). For example, when polishing away the first few micrometres of surface if the four surface cracks remain discrete, separated from the indentation, classical Palmqvist type cracking is indicated. If the four cracks disappear completely before the indentation tip, they are fairly shallow and of the type seen in hardmetals. If the cracks remain visible after the indentation has disappeared they are deeper and of type intermediate between Palmqvist and half-penny geometries. If the cracks remain continuous across the indentation position with increasing depth of removal, but shrink in overall size, they are of half-penny type. Studies reported in the literature include those on a glass-ceramic [D.13], zirconia-toughened alumina [D.6], yttria-stabilised zirconia [D.14, D.15], sintered silicon carbide [D.16], alumina [D.17] and hot-pressed silicon nitride [D.18]. Figure D4 shows the influence of indentation force on the development of cracks in an yttria stabilised zirconia.

Another method involves using saturated aqueous lead acetate solution [D.19]. Before indenting the indenter is dipped into the solution, which flows into the crack during loading. The specimen is dried and then fractured, and the fracture surface is examined using backscattered scanning electron microscopy, using atomic number contrast from the lead to reveal the crack shape. There is a risk of enhancing crack growth by the presence of the solution. An alternative method used for ceramics is the application of penetrating dyes [D.11], but again there is a risk of enhancing slow crack growth.
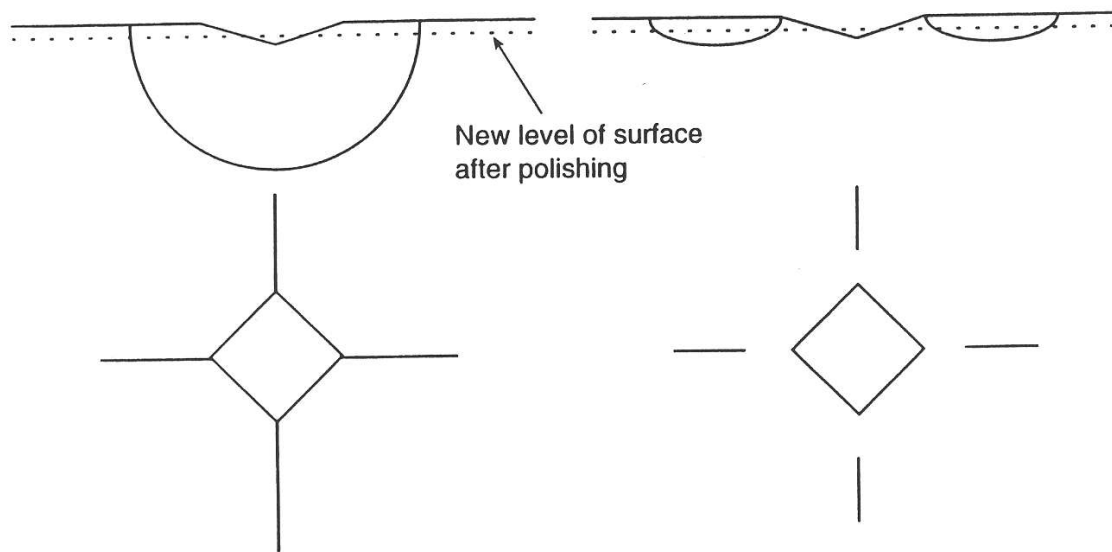
Figure D3 *The geometry of the subsurface (left) half-penny and (right) Palmqvist cracks can be determined by polishing the surface away and viewing whether the cracks become disconnected from the indentation.*
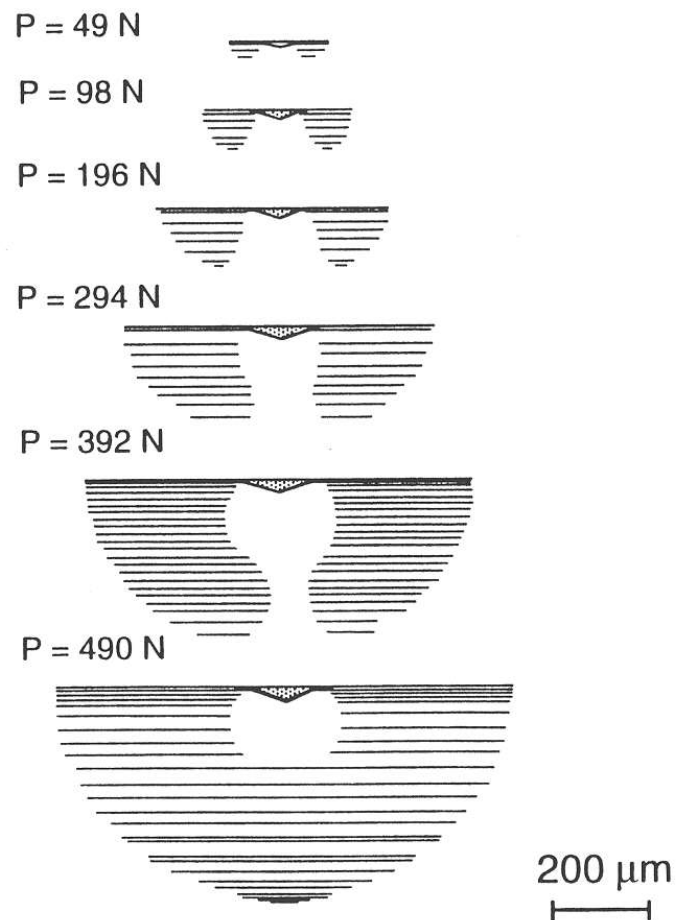

Figure D4 *The influence of indentation force on subsurface radial crack geometry as determined from a serial sectioning method. After [D.15].*

The results from these studies can be generally summarised in the following way:

(a)   when a ceramic of high fracture toughness such as hot-pressed silicon nitride is tested using a low load, shallow Palmqvist cracks are formed;

(b)   with increasing load the Palmqvist cracks become deeper;

(c)   at higher load there may be a transition to half-penny type cracks;

(d)   when a ceramic of low fracture toughness such as alumina is tested at moderate loads (10 N to 100 N), the cracks are generally of the half-penny type;

(e)   when a ceramic of moderate hardness but high fracture toughness, such as transformation strengthened zirconias, is tested, the cracks are of Palmqvist type, even at high loads.

D.4   Comparison of indentation fracture with other methods

D.4.1   General findings

The literature contains a large number of citations of the use of the indentation method, a significant proportion of them for situations in which the test or the equations used are clearly invalid. There are examples where the apparent toughness K_c is indentation load dependent [see discussion in D.20], despite the fact that the equations are intended to remove such dependence.

The VAMAS round robin conducted in 1989 compared the indentation fracture method (IF, the generalised term for the indentation crack length method) with the single-edge pre-cracked beam (SEPB) method and the indentation strength (IS) method on both a silicon nitride and a zirconia-alumina composite [D.21, D.22]. The results for the IF method showed a very large variation between participants: for the silicon nitride at $P = 196$ N, the scatter was $\pm 15\%$; for the zirconia-alumina the scatter was $\pm 20\%$ at $P = 490$ N and $\pm 27\%$ at 294 N. Some of the participants found a significant indentation force dependence while others did not. By comparison, the other methods had much smaller variations between participants, suggesting that the IF method had larger potential scatter and bias amongst users, and thus poor value compared with the alternatives, even assuming the calculation equation employed was 'correct'.

D.4.2   Alternative toughness tests using indentation

D.4.2.1   Indentation strength (IS) test

This uses the radial/median indentation crack to 'pre-crack' a flexural strength test-piece [D.23]. The flexural strength is determined using the conventional flexural stress equation and inserted into the formula:

$$K_c = 0.59\,(E/H)^{1/3}(\sigma_f P^{1/3})^{3/4}$$

where σ_f is the flexural strength, P is the indentation force. The constant term is, as with

indentation fracture, determined by data-fitting. This equation takes care of the crack pre-stressing caused by the indentation, and the crack length *c* does not need to be measured. This means that if the test-piece is annealed to remove the pre-stressing by the plastically deformed zone, the above equation is invalid, and a more conventional approach based on a shallow unstressed half-penny surface flaw needs to be used. Implicit in either analysis is the fact that the crack developed is of half-penny type, which may not always be the case, and that R-curve effects are ignored. In the above equation, the same data fitting exercise is involved in its initial calibration as for indentation fracture.

An experimental evaluation of the technique in comparison with the single-edge pre-cracked beam method was made through the same VAMAS study [D.21, D.22] as for the indentation fracture method. Using a silicon nitride, it was found that although the apparent toughness for a given indentation force was remarkably consistent between laboratories, it was also significantly indentation force dependent, changing from 5.6 to 6.0 MPa m$^{1/2}$ at 49 N force to 6.5 to 7.0 MPa m$^{1/2}$ at 294 N force. A similar result was obtained for a zirconia/alumina composite material. The origins of this effect are not clear, but may be associated with R-curve behaviour over the indentation size range, or factors associated with the indentation plastic zone size[*]. Thus although superficially this test has some highly desirable characteristics, notably on having low scatter as a result of removing the human element of crack length (but not of hardness) determination, it may be no better in achieving accurate results than the indentation fracture test.

D.4.2.2 Surface flaw in flexure method

This method is similar to the previous test, but in this case the surface zone associated with the indentation is removed by light abrasion to leave a nominally stress-free, approximately elliptical surface crack [D.24]. After determining the apparent strength of the test-piece, the fracture toughness is calculated from the pre-crack dimensions as observed and measured fractographically. Some small developments (indenting at a slight angle to the surface, using a Knoop indenter rather than a Vickers indenter) improve the fractographic visibility of the pre-crack and make the method more user-friendly [D.25]. On many materials, notably coarser-grained ones, this still requires some skill to interpret fractographically the boundary of the pre-crack, but has been found to be reliable in a VAMAS round robin [D.26]. One of the materials in the round robin, a Y_2O_3-TZP, developed Palmqvist cracks, the depth of which could be enhanced on one side by angling the indentation. The results obtained were in excellent agreement with those from pre-crack beam tests. On the basis of the round robin and some additional work it has become one of the methods in ASTM C1422 as well as BS EN ISO 18756.

Although more complicated than the indentation-strength method, this test appears to have wider validity from a fracture mechanics view-point in that it uses a stress-free pre-crack of known size and shape, backed by well-defined analytical equations [D.27]. It is the recommended small-crack method for materials which develop half-penny cracks under indentation, with possibilities for use on some Palmqvist cracking materials. However, it does not work on all materials, notably some coarser grained ones in which well-defined pre-cracks do not develop, or in which the pre-crack size cannot readily be distinguished by fractography [D.28].

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[*] Note: Although the participants were asked to check fractographically that fractures were initiated from the indentation cracks, no checks were made that the cracks were fully half-penny at both indentation loads, and hence that they satisfied the validity criteria for the test.

D.4.2.3 Pre-cracked beam method

A larger, mostly pre-stress free crack can be introduced into a strength test-piece by making a suitable indentation in the face of the test-piece, similarly to the IS method above, but then propagating the indentation crack by controlled compressive loading of the test-piece. The indented test-piece is placed on a flat anvil with the indentation over a gap in its surface, and loaded with a second anvil on its opposite face until there is an audible 'pop-in' of the crack. Alignment of the loading is crucial in obtaining a straight-fronted crack. The test-piece is then broken in three- or four-point bending, and the length of the pre-crack determined from the fracture surface. Standard fracture mechanics equations then relate the fracture toughness to the pop-in crack length. Any pre-stressing introduced by initial indentation is essentially removed on loading the test-piece in bending because the crack opens before propagating through the remainder of the test-piece. Details of the test may be found in references D.4, D.21, D.22. It has been shown to give consistent results between participants in VAMAS and ESIS round-robins (*e.g.* D.21, D.22). It has being developed as a standard method in ASTM C1422 and BS EN ISO 15732.

D.5   Conclusions

The following detailed conclusions can be drawn:

1.   Although the test is easy to perform and there is no doubt that indentation crack length does correlate inversely with toughness, the measurement is an unreliable indicator of toughness in absolute terms.

2.   Crack geometries can range from shallow Palmqvist type to semicircular (half-penny) type, with intermediate stages.

3.   Surface crack lengths are dependent on the surface preparation method, on the observation method and on the interpretation of crack lengths.

4.   Crack tip visibility can be poor unless means of improving contrast are employed, such as the use of reflective coatings.

5.   Unless the test material is particularly fine-grained and homogeneous, there is a large scatter of results that is material type and microstructure dependent. The test is inappropriate for materials with more than about 2% porosity.

6.   There is a wide variety of equations that have been used to calculate K_c values from crack lengths, and there are fracture mechanical assumptions behind them which may not be upheld in a given class of ceramic.

7.   Any apparent difference in indentation crack lengths between different materials can be considered significant only if it is outside the scatter of crack lengths obtained on each material, which can be typically as high as ± 15%. Consequently, the test is not a good discriminator of materials, but might be used in a statistical way by a single user to

identify differences in toughness between materials of similar type. If the test is to be used in this way it is suggested that:

a)   the material should be fine-grained and of very low or zero porosity to give long straight radial cracks (*c/a* should be greater than 2.5);

b)   there should be minimal residual stresses resulting from grinding stages prior to final surface finishing;

c)   the surface finishing procedure should avoid grain pluck-out;

d)   the indenting force should be high enough to give half-penny cracks without loss of integrity of the diamond impression;

e)   consistent conditions of measurement should be established, together with a consistent criterion for location of the apparent crack tip.

8.   If accurate numerical values of toughness are required, alternative methods should be sought. These can be based on indentation, but only as a means to produce a small pre-crack. The best method for small flaws shown to apply satisfactorily to many ceramics is the surface flaw in flexure method [D.27] in which the residual stress field caused by indentation is removed by polishing before fracture testing, and the indentation crack size is measured after fracture by fractographic methods.

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