VAMAS

Technical Working Area 21
Mechanical Tests for Hardmetals

HARDNESS TOUGHNESS TESTS: VAMAS INTERLABORATORY EXERCISE

Report No. 48
ISSN 1016-2186
March 2005

[VAMHMtoughness/BM]

VAMAS

The Versailles Project on Advanced Materials and Standards supports trade in high technology products through international collaborative projects aimed at providing the technical basis for drafting codes of practice and specifications for advanced materials. The scope of the collaboration embraces all agreed aspects of enabling science and technology - databases, test methods, design standards, and materials technology - which are required as a precursor to the drafting of standards for advanced materials. VAMAS activity emphasizes collaboration on pre-standards measurement research, intercomparison of test results, and consolidation of existing views on priorities for standardization action. Through this activity, VAMAS fosters the development of internationally acceptable standards for advanced materials by the various existing standards development organizations.

• Canada • France • Germany • Italy • Japan • UK • USA • EC •

Technical Working Area 21
Mechanical Tests for Hardmetals

HARDMETAL TOUGHNESS TESTS: VAMAS INTERLABORATORY EXERCISE

B Roebuck and E G Bennett
National Physical Laboratory
Teddington
Middlesex TW11 0LW, UK
e-mail: bryan.roebuck@npl.co.uk
eric.bennett@npl.co.uk

Report No. 48
ISSN 1016-2186
March 2005

[VAMHMtoughness/BM]

Contents

Hardmetal Toughness Tests:
VAMAS Interlaboratory Exercise

B Roebuck and E G Bennett
NPL, UK

Summary

Rationale

Optimised use of hardmetals requires good data on fracture toughness. Hardmetals are low ductility materials and fracture can limit performance. Various test methods for obtaining toughness values are in current use and an improved understanding of the differences will underpin a sensible choice of technique.

Objective

To evaluate hardmetal toughness tests through an investigation of three different test methods (chevron notched bars, single edge precracked beams and Palmqvist tests) on nine different hardmetals.

Interlaboratory Exercise

An interlaboratory exercise was conducted to generate underpinning technical information on well characterised materials that works towards good practice for toughness tests for hardmetals. It was planned to provide a wider understanding of the benefits of short rod chevron notched tests vis a vis single edged precracked/notched beams and provide guidance on the allowable range for useful Palmqvist tests.

Outcome

Eight organisations were able to complete Palmqvist tests and two completed short rod chevron notch tests; but only three organisations were able to provide single edge beam data. Good statistics were obtained on the Palmqvist data that will support the dissemination of good testing practice. Single edge precracked beam data was thought to be closest to the "true" value and most of the short rod chevron notch test data compared reasonably well with these results. However, care was needed in testpiece preparation to ensure a good correlation between data from the Palmqvist tests and the single edge precracked beam results.

Following circulation of this report of the interlaboratory exercise, an ISO Technology Trends Assessment document is planned, as a first step in recommending appropriate suitable test methods that will have wider acceptance across industries that make and use hardmetals.

Foreword

Hardmetal* fracture toughness values are required for a number of reasons; for example:

- for product design and performance assessment.
- for materials selection.
- for quality control.

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 hardmetal components. Fracture toughness increases with increasing Co content and increasing WC grain size. There is no ISO international standard test method for measuring toughness specifically for hardmetals, primarily because of the difficulty of introducing stable precracks into these tough but hard materials. There is a particular need for a suitable test for materials with toughness values greater than about 15 MN $m^{-3/2}$, that are even more difficult to precrack (a prerequisite for a valid toughness test). The ASTM have developed tests based on the "chevron notch short rod" method, ASTM E1304 and ASTM B771, and this is one of the test methods being assessed in the current VAMAS exercise.

Different groups and organisations in the hardmetal community have evaluated a range of techniques [1-17] including:

- SEPB Single Edge Precracked Beam. Beam with sharp crack on tensile surface. Hardmetals are difficult to precrack. Wedge indentation and fatigue (including in compression from a notch) have proved successful in some cases. A new method based on the use of stiff loading system has been used in the current exercise.

- SENB Single Edge Notched Beam. Beam with notch. Results depend on notch width and method of preparation. Not generally recommended for hardmetals.

- SEVNB Single Edge V-notched Beam. Beam containing notch with sharpened tip - diamond honed. Validated on ceramics. More work needed on hardmetals to confirm requisite notch sharpness.

- SCF Surface Crack-in Flexure. Beam containing small semicircular flaw introduced by indentation and damage removed. It is not possible to remove damage in hardmetals and leave a useful precrack. Not recommended.

- IF Indentation Fracture. Palmqvist toughness test - crack lengths at indentation corners. Works reasonably well for hardmetals in toughness range 8-16 $MNm^{-3/2}$ provided that the surface is free from residual stresses. Tougher materials produce few or inconsistently sized cracks. Being evaluated in current VAMAS interlaboratory comparisons.

---

* 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 'hardmetals' 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' in 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".

- IS — Indentation Strength. Rectangular beam with indentation; subsequently fractured. Damage and residual stresses associated with indentation have strong influence on result. Not recommended for hardmetals.

- CNB — Chevron Notched Beam. Crack initiation difficult in hardmetals. Not recommended.

- CNSR — Chevron Notched Short Rod. Commercial equipment available – Terratek/Dijon. Need to be careful with residual stresses. ASTM standards in place (E1304 and B771). Included in current VAMAS interlaboratory comparison.

1 Symbols and units

For the purpose of this report the following nomenclature applies:

Symbol	Designation	Units
K_{Ic}	Plane strain fracture toughness	MN $m^{-3/2}$
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

2 VAMAS Intercomparison Plan

Initially fifteen organisations showed an interest in participation and materials supply:

National Physical Laboratory	UK	Sandvik Hard Materials	UK
Dymet Alloys	UK	Marshalls	UK
Teledyne Advanced Materials	USA	Kennametal	USA
Plansee Tizit	Austria	BAM$^+$	Germany
Harditalia	Italy	Baildonit	Poland
Boart Longyear	Germany	United Hardmetals	Germany
Hughes Christensen	USA	Hilti	Liechtenstein
University of Catalunya (UPC)	Spain		

Some organisations have changed their names since the start of the exercise. The original names have been kept for this report.

$^+$ BAM – Bundesanstalt für Materialforschung und-prufung.

Not every organisation that originally agreed to take part were able to complete their measurements by the date of this current report.

Additional/Dissemination Groups:

British Hardmetal Association Research Group
USA Cemented Carbides Association
Japan Cemented Carbides Association

2.1 INTERCOMPARISON PLAN

A subset of the participating organisations supplied materials for tests in the form of rectangular bars or rods, dependent on test method to be evaluated. Further subsets of these were used for Palmqvist tests.

NPL coordinated materials supply and preparation of Palmqvist testpieces. Participating organisations were sent two sets of samples for Palmqvist tests, one already indented and one with as-ground surfaces. Participants polished, indented and measured crack length by their own procedures on this second set as well as measuring the first set. Participants were not asked to measure hardness because NPL values were to be used in the analysis. However, some organisations did measure hardness and these are reported where appropriate. Table 1 gives the material supply and testing matrix for each organisation. Table 2 gives the material types that were originally offered. In practice, four organisations provided nine hardmetals, including one (Ti(C,N) based. Table 3 gives some properties of these nine hardmetals (Data supplied by Hughes Christensen and NPL). In-house tests were performed by appropriate organisations on suitable sets of material.

Table 1 Initial Materials Supply and Testing Matrix

Organisation	Offer of Material Supply Y/N	Palmqvist Tests Y/N	In-house Tests Y/N
NPL	N	Y^{+}	Y^{++}
BAM	N	Y^{+}	Y^{+}
Dymet*	Y(3)	Y^{++}	N
Sandvik Hard Materials*	Y(2)	Y^{++}	Y^{++}
Kennametal	Y(3) ✓	Y^{++}	Y^{++}
Baildonit	Y(2) ✓	Y^{+}	N
United Hardmetals*	Y(2)	Y^{++}	Y^{++}
Hilti	N	Y^{++}	N
Harditalia	Y(3) ✓	Y^{+}	N
Boart Longyear*	Y(2)	Y^{+}	Y^{++}
Teledyne*	Y(2)	Y^{+}	Y^{+}
Plansee	Y(2) ✓	Y^{+}	Y^{+}
Marshalls	N	Y^{++}	Y^{++}
Hughes Christensen	N	Y^{+}	Y^{+}

* Materials not supplied in time for exercise.
$^{+}$ Tests successfully completed.
$^{++}$ Tests not completed.
✓ Supplied materials

Table 2   Material Types – Original Offer

Organisation	Code	Material	Organisation	Code	Materials
Sandvik HM	HM1 HM2	Ultrafine Fine/alternative binder	Baildonit*	B1 B2	6% Co, submicrometre 10% Co, submicrometre
Teledyne	T1 T2	Medium, low cubic, 10-11%Co As above + Ruthenium	Harditalia*	H1 H2 H3	Low Co Med Co High Co
Plansee*	P1 P2	Submicrometre hardmetal Cermet (Palmqvist only)	Kennametal*	K1 K2 K3	6% Co, small gs, low cubics 8½%Co, med gs, high cubics 9½%Co, large gs, no cubics
Dymet	D1 D2 D3	5% Co; fine/medium; 9% cubics 7½% Co; medium; 20% cubics 6% Co; fine/medium	United Hardmetals	U1 U2	10% Co, 0.8 μm gs 3½% Co, 0.8 μm gs
Boart Longyear	BL1 BL2	7½% Co; medium; 20% cubics High/medium Co; coarse WC			

* Materials used in interlaboratory exercise.

Table 3   Material Properties – Hughes Christensen and NPL Measurements

Material	Density $Mg\ m^{-3}$	Coercivity $kA\ m^{-1}$	Magnetic moment $\mu T\ m^3\ kg^{-1}$	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

2.2 TEST SCHEDULE

All materials were tested for Palmqvist toughness, but only subsets were tested by in-house methods. Only a limited number of organisations were able to complete tests by methods other than Palmqvist.

The test schedule required some organisations to prepare materials for collaborating companies to test. The dimensional requirements for appropriate in-house tests are given in Table 4.

Table 4 In-house Testpiece Requirements

Organisation	Test**	Dimensions, mm*
NPL	SEB	2 × 5 × 35 (min)
BAM	SEB	3 × 4 × 45
Boart Longyear	Terratek short rod	nominal 10 ϕ × 15 long
United Hardmetal	SEB	6 × 10 × 20
Teledyne	Terratek short rod^{+}	nominal 12.7 ϕ × 19 long
Kennametal	SEB	3 × 6 × 45
Sandvik HM	SEB	2 × 5 × 35
Marshalls	Terratek short rod	nominal 12.7 ϕ × 19 long
Hughes Christensen	Terratek short rod^{+}	nominal 12 × 12 × 18
Plansee	SEB^{+}	nominal 40 × 5 × 2

* 3 testpieces/material grade
** SEB - Single edge beam, notched or precracked
$^{+}$ Organisations able to complete test.

Materials for Palmqvist tests were taken from the materials supplied for in-house tests.

In practice only two organisations were able to perform chevron notch short rod tests and three organisations completed SEB tests (Table 5). Other organisations attempted in-house tests but were unable to complete them because of problems with the test method, facility availability or testpiece dimensions being slightly out of specification.

Table 5 Test Outline (Excluding Palmqvist tests)

TESTING ORGANISATION		TESTPIECE GEOMETRY		
		SEB	SR1	SR2
NPL+	SEPB 2×5×35 mm	B(2) K(3) H(3)		
BAM	SEVNB 3×4×35 mm* 2×5×40 mm**	B(2) K(3) H(3)		
Boart Longyear+	SR2 10ϕ×15 mm			H(3) B(2) K(3) P(1)
United Hardmetals+	SEPB 6×10×20 mm	B(2) K(3) H(3)		
Teledyne	SR1 12.7ϕ×19 mm		H(3) B(2) K(3)	
Kennametal+	SEPB 3×6×45 mm	B(2) K(3) H(3)		
Sandvik Hard Materials+ (CERMeP)	SEPB 2×5×35 mm	B(2) K(3) H(3)		
Marshalls+	SR1 12.7ϕ×19 mm		H(3) B(2) K(3)	
Hughes Christensen	SR1 12.7ϕ×19 mm		H(3) B(2) K(3)	
University of Catalunya	SEPB 2×5×40 mm	B(2) K(3) H(3)		
Plansee	SEPB 2×5×40 mm	B(2) K(3) H(3)		

+ Unable to complete tests
* Preferred
** Supplied

Testpiece design

SEPB - Single Edge Precracked Beam
SEVNB - Single Edge V-notched Beam
SR1 - Short Rod, 12.7 mm ϕ × 19 mm
SR2 - Short Rod, 10 mm ϕ × 15 mm

Material Supply

B - Baildonit
P - Plansee
K - Kennametal
H - Harditalia

Number in brackets – number of hardmetals.
5 off each material to be supplied.

2.3 INSTRUCTIONS FOR PALMQVIST CRACK LENGTH MEASUREMENT

Each pack for participants contained nine polished sections, one from each grade of material together with a reporting table. In the central region of each sample was a HV30 Vickers indent. The sample of grade K3560 had an additional HV100 indent made, which is situated between the HV30 indent and one of the corners of the sample.

Step 1 For all samples except grade K3560, measure the total length of the cracks produced at the corners of the HV30 indent. For sample of grade K3560, measure the total length of the cracks produced at the corners of the HV100 indent. Enter the measured values in a table provided.

Step 2 For all samples except grade K3560; place 3 HV30 indents into the polished face of the specimen. Ensure that these are sufficiently spaced so that cracks produced do not overlap. Measure the total length of the cracks produced for each of the indents and enter the values into the table. The following stage is optional for those who can use 100 kgf for Vickers indentation. For grade K3560, place 3 HV100 indents into the polished face of the specimen. Measure the total length of the cracks produced for each of the indents and enter the values into the table.

Step 3 Polish the opposite face of each of the specimens. Please provide details of the polishing routine and any other preparation technique that is used.

Step 4 For all samples except grade K3560; place 3 HV30 indents into the face of the specimen polished by your in-house routine. Please ensure that these are sufficiently spaced so that cracks produced do not overlap. Measure the total length of the cracks produced for each of the indents and enter the values into the table. The following stage is optional for those who can use 100 kgf for Vickers indentation. For grade K3560, place 3 HV100 indents into the polished face of the specimen. Measure the total length of the cracks produced for each of the indents and enter the values into the table.

Step 5 Please provide details as to how the crack lengths were measured.

NB Any other comments/observations you care to make would be appreciated and can be annotated below the table.

Further information on the Palmqvist test method is given in Appendix A.

3 General background to toughness tests

The results of fracture toughness test methods can be expressed either as a stress intensity factor, K_{Ic}, MN $m^{-3/2}$ or as a fracture surface energy, J m^{-2}. The range of K_{Ic} values for typical WC/Co hardmetals is from 7 to 25 MN $m^{-3/2}$. For hardmetals there is a general inverse trend of hardness against fracture toughness.

When applied unqualified to hardmetals ‘toughness’ can have several meanings:

a) 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 ISO standard method for hardmetals. Different organisations use different test methods for introducing the precrack. However, the ASTM have developed a “chevron notch short rod” test (ASTM E1304 and B771).

b) 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. $G = K^2/E(1-v^2)$, E is Young's modulus and v is Poisson's ratio). It has units of J m^{-2}. Again there is no standard method.

c) 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.

d) Finally, toughness is also widely used, in a loose sense, to describe the empirical relation between 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.

3.1 PALMQVIST TOUGHNESS

Palmqvist tests are widely used to assess the toughness of hardmetals since only small amounts of material are used and the difficulties of precracking conventional toughness testpieces are bypassed [18,19]. However, the method has yet to be standardised and until it has there will always be some doubt in comparing data published from different sources. The method is particularly sensitive to the testpiece preparation procedure [5,6]. There is a considerable body of published information on Palmqvist toughness tests for hardmetals [1-17], and a good practice guide has been written by NPL [20]. The residual stresses, which affect crack lengths in the Palmqvist test, are likely to be more significant in the fine grained hardmetals. Currently an annealing treatment of 800 ^{o}C for 1h is generally used for Palmqvist tests following the method outlined by Exner [5]. This procedure should be re-examined, however, for very fine grained hardmetals. Testpieces may need to be annealed at higher temperatures to ensure complete removal of residual stresses. However, for newer grades of hardmetal, especially with very fine structures or alternative binder phase alloys, the use of higher annealing temperatures could produce changes in structure [21,22]. A good review of the effects of residual stresses was provided by Yohe [23]; this reference also addressed the issue of residual stresses in Short Rod toughness tests and concluded that the effects were significant and required careful consideration.

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 1 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 [5]. The test also has an uncertain fracture mechanics pedigree because of uncertainties associated with residual stresses introduced by the indentation.

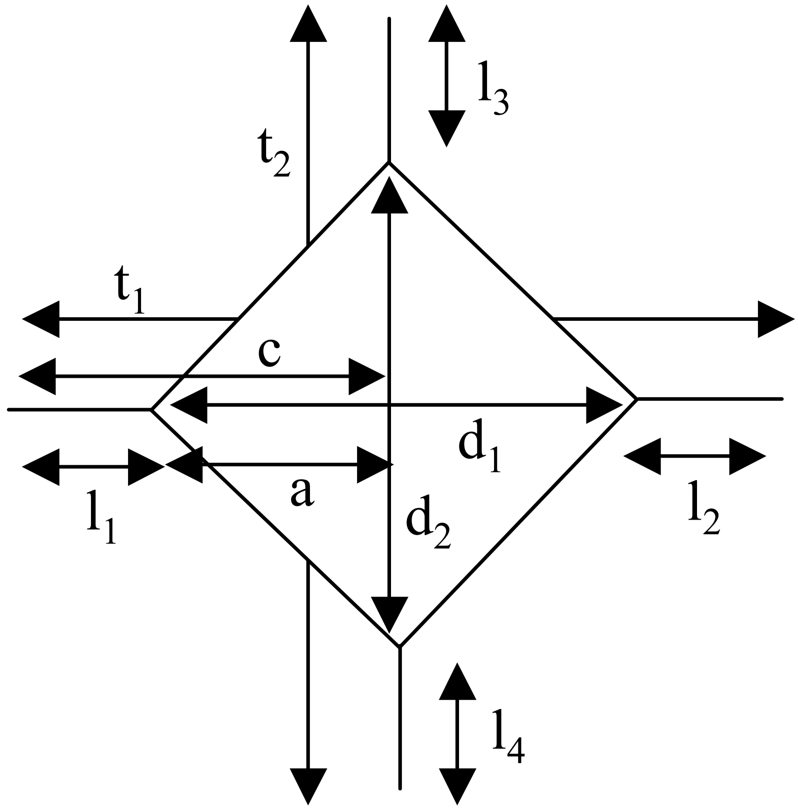

Figure 1 Schematic diagram and definitions for Palmqvist Test Method.

Hardness:

$$H = 1.8544\ P / ((d_1 + d_2)/2)^2$$

Indentation Force : P ; usually 30 kgf

Alternative methods measuring crack length and toughness, W:

a) Measure crack tip to crack tip, $t_1 = (\ell_1 + \ell_2 + d_1)$, $t_2 = (\ell_3 + \ell_4 + d_2)$
   Measure indentation diagonals, d_1, d_2
   $W_G = P/[(t_1 - d_1) + (t_2 - d_2)]$
b) Measure crack lengths ℓ_1 to ℓ_4 from crack tip to indentation corner
   Total crack length, $T = \ell_1 + \ell_2 + \ell_3 + \ell_4$ or $(t_1 - d_1) + (t_2 - d_2)$
   $W_G = P/(\ell_1 + \ell_2 + \ell_3 + \ell_4)$.

Note: Parameters c and a are widely used in the literature for calculating indentation toughness values for ceramic materials.

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 1.

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

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 [1-3]:

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

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 $m^{-3/2}$. Following the analysis discussed by Warren and Matzke [1], Shetty *et al*. [2] and Spiegler *et al.* [3], where A is a constant of value 0.0028, HV is the hardness (in N mm^{-2}) at a load of 30 kgf and W_G is already defined in expression (1).

In principle the crack length can be measured in one of two ways:

1. *from indent corner to crack tip (for each corner and then summing the value)*
2. *from crack tip to crack tip minus the indent diagonal (and summing orthogonal values).*

At NPL method 1 is used.

Research at NPL [24] has shown that the uncertainties associated with the Palmqvist test are generally about ± 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 ± 0.5 MN $m^{-3/2}$ for plane strain toughness tests on more conventional fracture toughness testpieces [25].

In summary, 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.

For conventional hardmetals a good correlation is claimed between K_{Ic} and W_K [2]. This remains to be confirmed by validated tests according to an agreed standard method. Research work at NPL compared Palmqvist toughness measurements on a range of WC/6% Co hardmetals with plane strain K_{Ic} values obtained by an NPL recommended procedure using the wedge indentation method [4]. The agreement was quite good between K_{Ic} values of about 10 and 15 MN $m^{-3/2}$. For low values the Palmqvist method overestimated the toughness, 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 ^{o}C for 1 h is not adequate to fully relieve residual stresses

In some published work temperatures of 850 ^{o}C for 2 h have been used. Clearly further systematic work on temperatures and times of annealing, especially for the finer-grained harder materials would seem to be required.

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. Other expressions for calculating W_K may be more appropriate [20].

For all values the Palmqvist data show more scatter than the plane strain data and this is especially large for high toughness hardmetals, (Fig 2). 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 60-100 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. Although there is scope perhaps, for using indenters manufactured from polycrystalline diamond (PCD).

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 additional effort and can yield equally useful material characterisation data provided the measurements are obtained carefully.

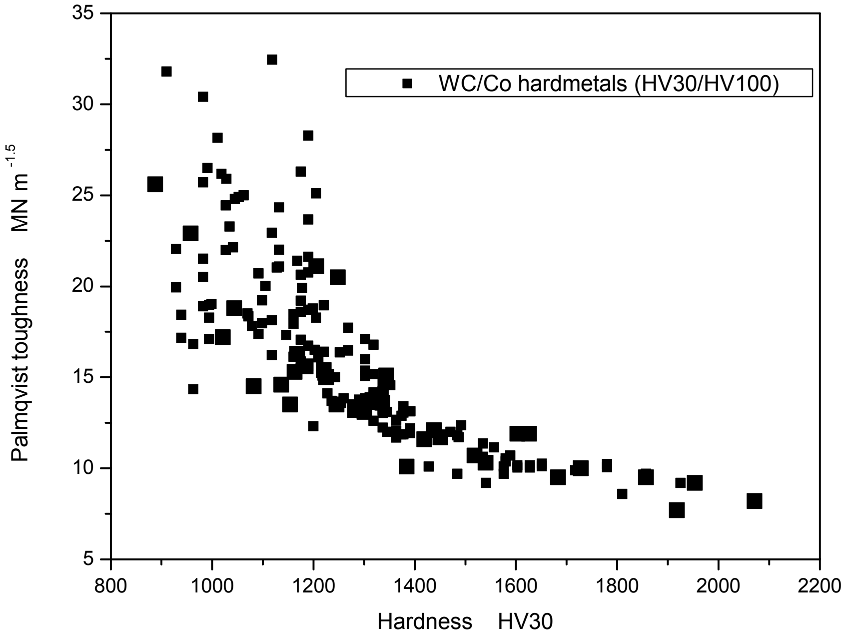

Figure 2 Palmqvist toughness values across a range of hardness values for WC/Co hardmetals (unpublished work – NPL).

3.2 SEB FRACTURE TOUGHNESS TESTS

Tests in which beams or rods, notched or precracked, are fractured in bending, are preferred by the conventional fracture mechanics community, since these arrangements allow better control over loading and stress state at the crack tip, ensuring that 'valid' K_{Ic} values are obtained. The ability to obtain load-displacement data from these tests also permits subcritical crack growth and other factors that may compromise the value obtained to be better accounted for. While both specimen preparation and the tests themselves are expensive, the toughness values obtained are valuable as benchmarks against which to compare other methods more suitable for routine use in industry.

A variety of methods have been developed for fracture toughness tests using a rectangular beam (SEB) testpiece. The key objective is to develop a stress-free crack of known geometry that can be loaded to fracture and the fracture load is then used, together with the known crack length, to calculate fracture toughness. For precracked SEB (single edge beam) testpieces the value of K_{Ic} is related to the nominal applied bending moment, M, and crack length, a, by:

$$K_{Ic} = \frac{6M}{BW^2}\sqrt{\pi a}\, f(a/W) \qquad (3)$$

where, a is the crack length, B and W are the testpiece width and height respectively and f(a/W) is fourth order polynomial. Tabulated values for f(a/W) appear in reference books for specimens of standard dimensions.

Fracture toughness results can be calculated using the compliance derived f(a/W) and the Brown and Srawley 3 pt bending formula for L/W = 4 where:

$$f(a/W) = 1.09 - 1.73\,(a/W) + 8.2\,(a/W)^2 - 14.2\,(a/W)^3 + 14.6\,(a/W)^4 \qquad (4)$$

The crack lengths are generally measured on the fracture face after testing to failure.

For precracked single edge cracked beam (SEPB) specimens, the value of K_{Ic} is thus related to the nominal applied bending load, P, and crack length, a, by

$$K_{Ic} = \frac{PS}{BW^{1.5}} f(a/W) \qquad (5)$$

where B and W are the testpiece width and height, respectively, and f(a/W) is a polynomial, S is the total span in a 3 pt bend test and the distance between the inner and outer rollers in a 4 pt bend test.

A variety of approaches to develop stress-free cracks in SEB tests have been published, or are in the process of development, including:

- Wedge indentation (developed at NPL)
- Diamond notching (developed for ceramics, adopted by BAM in current interlab exercise)
- Chevron notch beam
- Stiff precracking machine, to grow a crack from a chevron notch (adopted by Plansee in current interlab exercise)
- Precracking of notch in compression fatigue; followed by extension fatigue of crack to known geometry in tension (adopted by UPC in current interlab exercise).

For the NPL wedge indentation method the cracks can easily be seen if they are stained by the coolant fluid used when grinding away the plastic indentation after precracking with the wedge indenter. Previous work has shown that this stain had no effect on the measured value of K_{Ic} [25]. Five testpieces of each batch are usually precracked using the wedge indentation method at NPL, which has been shown to produce stress free cracks of controlled length. NPL were unable to use this method in the current exercise, due to problems with the availability of appropriate test equipment.

Plansee have developed a new in-house stiff precracking machine that uses a piezoactuator to drive controlled crack growth in a SEB testpiece which has a narrow starter notch. This machine allowed stable crack growth to a depth of about W/2. There was some concern over crack front curvature – differences in crack length measured at the surface and at the centre of the testpiece gave K_{Ic} values that differed by about 1-2%. Expression (4) – the Srawley function – was used for calculating K_{Ic} values from the mean crack length (measured at 5 equidistant points across the testpiece width B) and fracture load. Further work is being conducted at Plansee to investigate the effects of surface preparation (grinding/lapping/polishing) on the extent of crack curvature and its effect on calculated values of K_{Ic}.

UPC used the compression fatigue method to introduce stress free cracks with a simple geometry. A notch of 2.4 mm in depth was machined in the middle of 3 × 6 × 45 mm bars using a diamond disc of 0.3 mm thickness. The root of the notch was shaped by a razor blade with diamond paste until the final notch had a depth of 2.6-3.0 mm and a notch radius of about

15-45 μm. A sharp crack was then nucleated at the root of the notch by compressive fatigue cycling in four-point bending. In doing so, a sinusoidal wave with a frequency of 10 Hz and maximum compressive peak stress of 565-1975 MPa (and load ratio of 10) was applied. However, the induced cracks were extremely small (less than 100 μm). Thus, they were further extended under far-field cyclic tensile loads (at R = 0.1) until final lengths were about 0.5 mm. Finally, fracture toughness values were determined by testing the precracked samples to failure under constant loading rate, between 200 and 400 N/s, and using the stress intensity factor given by Tada, Paris and Irvine [26]. Two specimens were employed for each hardmetal grade. Crack length was measured at 5 equidistant points along the crack front and a mean value taken. Some interesting features regarding compressive fatigue precracking in bending, as compared to compressive fatigue precracking in axial loading, are: less restriction regarding specimen dimensions and testing set-out; the requirement for lower applied nominal loads; insignificant residual stresses (further research is in progress). A relative shortcoming is the fact that "sharper" notches are requested in order to avoid potential fatigue failure from "natural" flaws in the "unnotched" – but subjected to tensile load-side.

BAM, for this exercise, chose to use the diamond notch method developed for ceramics. In each case five testpieces and two dummy specimens were mounted on a holder. The dummies are used to protect the specimens during sawing and polishing the notch. Each holder was mounted on a diamond saw. A starter notch (depth 1.3 mm) was sawn into all specimens. After this the holder was fixed in a vice and the starter notch was filled with diamond paste (at first 6 μm, by the end 1 μm) and oil. A razor blade was put into the starter notch and a light force was applied. The V-notch was controlled periodically with an optical microscope. At the end of this process the specimens were removed from the holder and cleaned with acetone in a small powerful ultrasonic bath. All specimens were checked optically. The V-notch tip was photographed with a magnification of ×300 and the V-notch radius was measured.

An Instron testing machine capable of keeping a uniform crosshead speed was used to fracture the notched testpieces. This machine was capable of measuring the true load at rupture to better than ± 1%. A 4-point flexure loading fixture (inner span 20 mm, outer span 40 mm) was used, fulfilling the requirements of EN 843-1: Determination of flexural strength. The specimens were placed with the V-notch down on the outer rollers and were loaded with a crosshead speed of 0.5 mm/min. The fracture load was recorded. The fracture tests were performed in air at room temperature. After the fracture test the depth of the V-notch was measured by observing the fracture surface using a calibrated microscope with a magnification ×50 on three evenly distributed points.

The fracture toughness was calculated using the following expression, with $S = (S_1 - S_2)$, where S_1 and S_2 are the outer and inner span lengths respectively.

$$f(a/W) = \frac{3(\alpha)^{0.5}}{(1-\alpha)^{1.5}}\left(1.9887 - 1.326\alpha \;-\left(\frac{\alpha(1-\alpha)(3.49 - 0.68\alpha + 1.35\alpha^2)}{(1+\alpha)^2}\right)\right) \quad (6)$$

3.3 CHEVRON NOTCH SHORT ROD TESTS

ASTM standard test method E1304, “Standard Test Method for Plane Strain (Chevron-Notch) Fracture Toughness of Metallic Materials”, significantly simplifies measurement of plane strain fracture toughness. E1304 allows five different specimen geometries. These include specimens of round (rod), square, and rectangular (bar) cross section with a length to diameter ratio (W/B) of 1.45 or 2.00. A groove is machined in one end of the short rod or short bar specimen parallel to the plane of the precrack, to provide a loading surface where the grips in the test machine can apply a load to the two specimen halves. Thus, fatigue pre-cracking is not required, with substantially lower testing costs. An important part of the specimen preparation procedure is the machining of the chevron slots in the specimen blank. These narrow slots (typically 0.2 mm) can be machined with a diamond slitting saw. When the loading grips are moved apart, an opening load is applied to the two specimen halves. Transducers in the test system monitor the magnitude of the opening load as well as the specimen mouth opening displacement. The test record plots mouth opening displacement vs mouth opening load. As opening load is increased, specimen mouth opening displacement increases as the test specimen is deformed elasticly, until the load reaches a point at which a natural crack is initiated at the tip of the triangular ligament (chevron) that joins the two specimen halves. As the load increases further, the crack length increases. The triangular shape of the fracture surface area dictates that as the crack length increases, the width of the crack front also increases. This widening of the crack front requires more energy to grow the crack, resulting in a stable, steady-state, natural crack. At a crack length known as the “critical crack length” (a_c), the load required to advance the crack passes through a maximum, and less load is then required to advance the crack. This critical crack length is geometry dependent and therefore a known length for the test specimen. In the simplest form of the test, one can simply measure the maximum load, and, knowing the critical crack length, then calculate plane strain fracture toughness for that test specimen.

A valid plane strain fracture toughness test requires a minimum specimen size to assure that the crack front is subject primarily to plane strain conditions. The minimum valid size for a short rod test specimen is one where the minimum “B” dimension is half the minimum “B” dimension for a compact tension (E399) test specimen of the same material. This minimum short rod test specimen is only 3% of the volume of the equivalent compact tension test specimen. Not only does this smaller size reduce significantly the amount of material required to perform a test, but it allows fracture toughness tests on materials where limited section thickness is available for a test specimen. In addition, the small size allows local measurement of fracture toughness.

Two organisations, Hughes Christensen and Teledyne, were able to perform short rod tests for this VAMAS exercise.

4 Results and Discussion

A full set of all the results provided by participants is given in Appendix B, including:

- SEB data returned by three participants (Plansee, BAM and UPC).
- Chevron Notch Short Rod data returned by two participants (Hughes and Teledyne).
- Palmqvist data obtained at NPL on all samples provided to participants.
- Palmqvist data returned by participants.

4.1 SEB FRACTURE TOUGHNESS TEST

Three sets of results were obtained from single edge beam tests:

- Plansee - use of stiff precracking machine.
- BAM - use of diamond notching technique developed for ceramics.
- UPC - crack initiation by fatigue of notched samples in compression. Some difficulties due to small size of testpieces.

Other organisations attempted this method, but were unable to provide data.

- NPL - wedge precracking; equipment not available at the time of the exercise.
- CERMeP - Lack of confidence with notching method.

The results from the use of the Plansee stiff precracking machine are given in Table 6 (mean of 3-4 tests; complete details are given in APPENDIX B) and this data is plotted against hardness in Fig 3. This data set was considered to be the reference data set with which to compare the alternative precracking techniques.

Table 6 Plansee SEPB results

Sample	K_{Ic}* MN $m^{-1.5}$	HV30 NPL value	Sample	K_{Ic}* MN $m^{-1.5}$	HV30 NPL value
B1	9.1	1778	H3	12.1	1364
B2	8.9	1626	K313	9.4	1726
H1	8.6	1810	K420	11.4	1486
H2	9.7	1592	K3560	18.7	996

* Mean of 4 tests; standard deviation is about 0.05-0.15 MN $m^{-3/2}$ for all hardness values (about 1% of mean value).
The Ti(C,N) cermet sample (TCM10) was not tested.

The UPC results from the compression fatigue initiation method are given in Table 7 and Fig 4. The results are in good agreement with those reported by PLANSEE; thus, both sets seem to be suitable as reference values for comparison of other techniques.

Table 7 UPC SEPB results

Sample	K_{Ic} (MN $m^{-1.5}$)	HV30 NPL value	Sample	K_{Ic} (MN $m^{-1.5}$)	HV30 NPL value
B1	9.22 ± 0.13	1778	H3	12.03 ± 0.14	1364
B2	9.99 ± 0.13	1626	K313	9.27 ± 0.09	1726
H1	8.90 ± 0.18	1810	K420	11.67 ± 0.07	1486
H2	9.96 ± 0.20	1592	K3560	18.93 ± 0.11	996

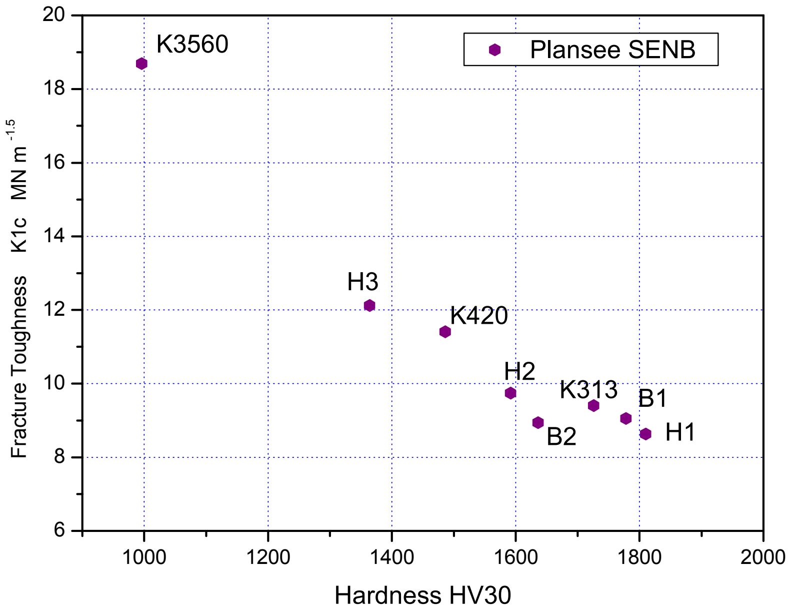

Fig 3 Plansee SEPB test results.

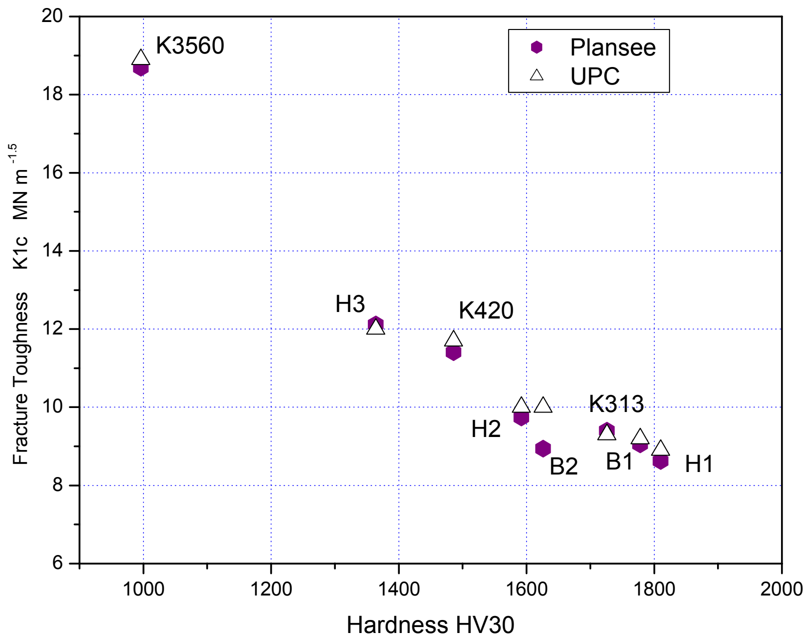

Fig 4 Comparison of UPC and Plansee SEPB data.

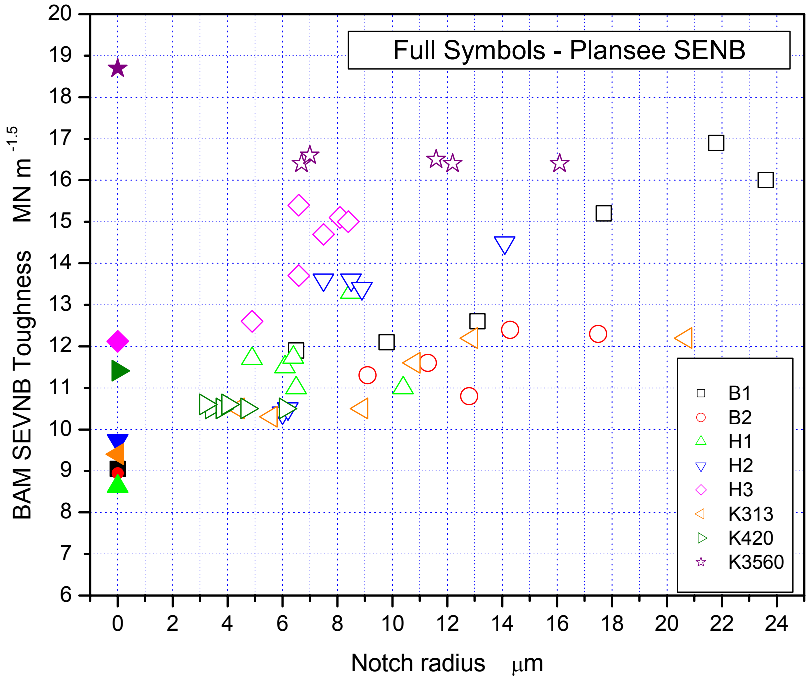

Fig 5 BAM SEVNB test results.

The results from the BAM SEVNB tests (APPENDIX B) are shown plotted against the radius of the diamond notch in Fig 5.

Figure 5 also includes the Plansee SEB test data, where it was assumed that the notch radius was 0 µm for the purpose of comparison. The BAM data fall into two types

- one set with a clear dependence of K_{Ic} on notch radius, with the result from the smallest radius approaching that of the Plansee result.
- one set that appears to be independent of the notch radius (K3560 and K420). In this case the results are lower than the Plansee data.

Thus, it would appear that none of the diamond notch results are valid and this technique for precracking requires further research before it could be widely recommended for hardmetals.

4.2 CHEVRON NOTCH SHORT ROD TESTS

The results from the chevron short rod tests from Teledyne and Hughes (APPENDIX B) are summarised in Table 8 and compared in Fig 6.

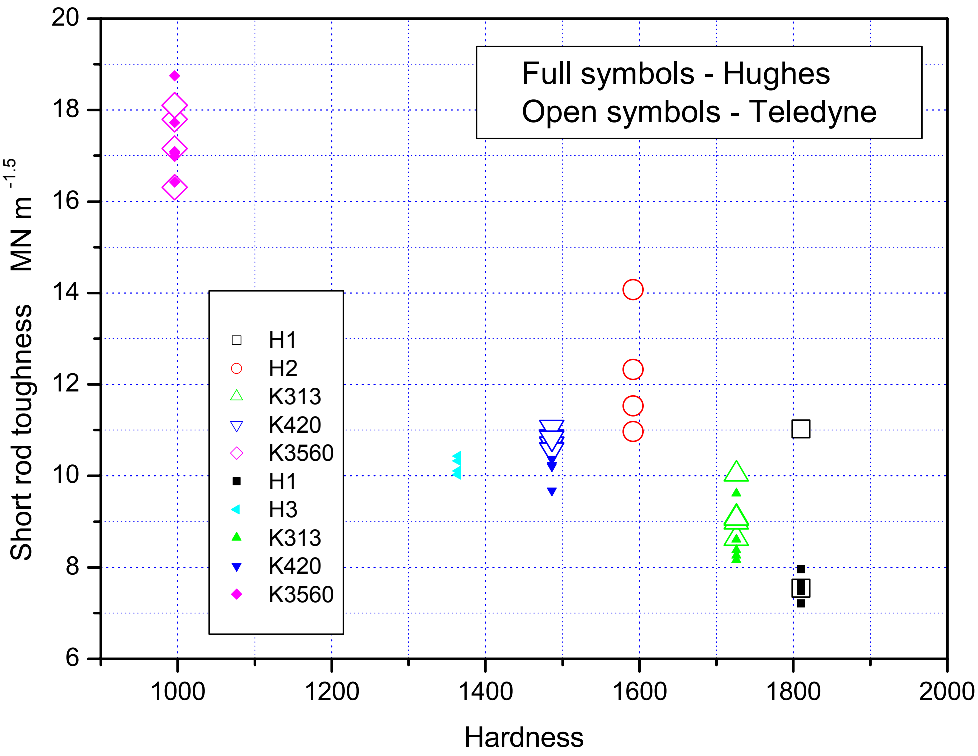

Fig 6 Comparison of chevron notch short rod test results.

Table 8 Mean values for chevron notch tests

Sample*	Teledyne K_{IcSR} MN m$^{-1.5}$	Comments	Sample*	Hughes K_{IcSR} MN m$^{-1.5}$	Comments
H1 (3)	9.9^{+}	2 samples – diameter too big for valid test.	H1 (5)	7.5	
H2 (4)	12.2		H2 (5)	7.9^{++}	But all considered invalid. p factor too high.
H3	-	All samples diameter too big for tests.	H3 (5)	10.2	
K313 (5)	9.2		K313 (5)	8.6	
K420 (6)	10.8		K420 (5)	10.2	
K3560 (5)	17.5		K3560 (6)	17.3	

* Numbers in brackets – number of tests thought to be valid.
\+ Mean of two values of about 7 and one of 11 MN m$^{-1.5}$. If the latter is excluded then agreement with Hughes data is better.
++ If K_{DL} value (12.0 MN m$^{-1.5}$) is used then the agreement with the Teledyne data is improved.

The standard deviation of the Hughes results was about 0.2-0.8 MN $m^{-1.5}$ with the higher values associated with the softer grades. The mean values of the chevron notch short rod data from Hughes are shown plotted against Palmqvist data (mean values also obtained by Hughes) in Fig 7. In every case the short rod data are lower than the Palmqvist results, although for the toughest grade, K3560, the difference was quite small. The chevron notch results from Hughes and Teledyne are compared with the Plansee SEPB results in Fig 8. There was reasonable agreement between the short rod values and the Plansee SEPB data although the uncertainties for the short rod data were much larger than those from the SEPB method. Also, three grades K3560, H3 and H1 gave significantly lower values and one grade, H2, significantly higher values. The reasons for these differences are not known.

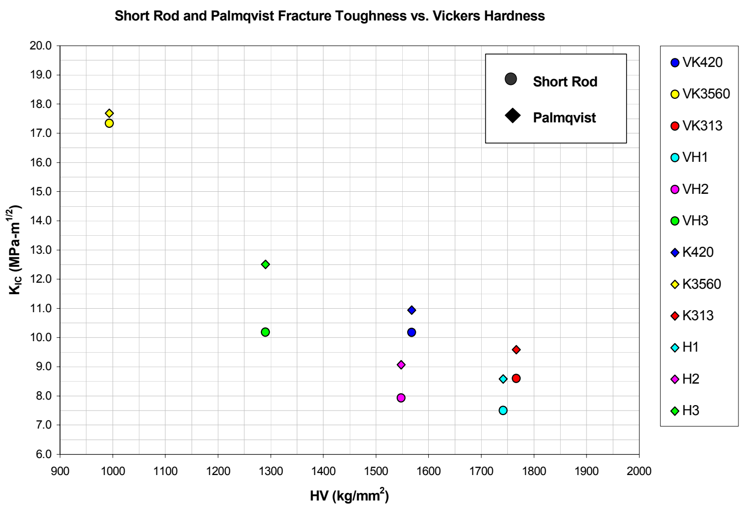

Fig 7 Comparison of Hughes chevron notch short rod data and Palmqvist results.

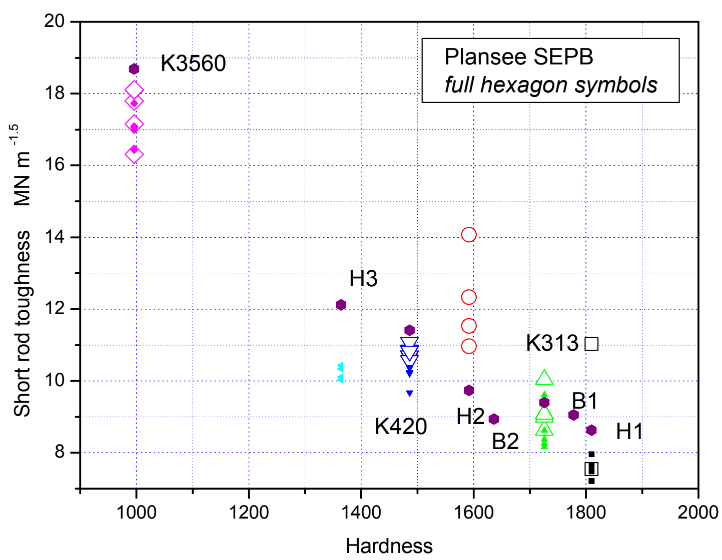

Fig 8 Comparison of Plansee SEVNB and chevron notch short rod test results.

4.3 PALMQVIST TESTS

The Palmqvist results are summarised in 4 parts in Tables 9-12, including:

- Table 9 – Measurements performed by NPL and Hughes on all samples (mean values).
- Table 10 – data on the single indents made at NPL.
- Table 11 – data on tests on the polished surface provided by NPL.
- Table 12 – data on tests made on surfaces prepared by participants.

Table 9 Mean values of Palmqvist crack lengths and toughness values – NPL prepared surface.

NPL and Hughes data (mean values; * HV100 – K3560)

Sample	HV30	**NPL** crack length μm	Toughness W_K MN $m^{-3/2}$	HV30	**Hughes** crack length μm	Toughness W_K MN $m^{-3/2}$
B1	1778	509	8.9	1779	503	8.9
B2	1626	436	9.2	1574	444	9.0
H1	1810	538	8.7	1741	533	8.6
H2	1592	419	9.3	1548	430	9.0
H3	1364	178	13.2	1290	183	12.6
TCM10	1636	620	7.7	1580	611	7.6
K313	1726	439	9.4	1767	456	9.4
K420	1486	323	10.2	1568	310	10.7
K3560	996*	167	21.2	993*	223	18.3

Table 10 NPL indent – Data from different organisations

Sample	HV30 NPL value	**Cermep** crack length μm	Toughness W_K MN $m^{-3/2}$	HV30 NPL value	**Plansee** crack length μm	Toughness W_K MN $m^{-3/2}$
B1	1778	498	9.0	1778	499	9.0
B2	1626	438	9.2	1626	424	9.3
H1	1810	544	8.7	1810	541	8.7
H2	1592	436	9.1	1592	446	9.0
H3	1364	186	12.9	1364	160	13.9
TCM10	1636	636	7.6	1636	640	7.6
K313	1726	442	9.4	1726	450	9.3
K420	1486	328	10.1	1486	345	9.9
K3560	996*	28	51.8	996*	279	16.4

* HV100

Sample	HV30 NPL value	**Teledyne** crack length μm	Toughness W_K MN $m^{-3/2}$	HV30 NPL value	**Hughes** crack length μm	Toughness W_K MN $m^{-3/2}$
B1	1778	512	8.9	1778	492	9.0
B2	1626	440	9.1	1626	437	9.2
H1	1810	556	8.6	1810	539	8.7
H2	1592	496	8.5	1592	460	8.9
H3	1364	200	12.4	1364	186	12.9
TCM10	1636	648	7.6	1636	621	7.7
K313	1726	440	9.4	1726	443	9.4
K420	1486	348	9.8	1486	353	9.8
K3560	996*	276	16.5	996*	121	24.9

* HV100

Table 10 (continued)   NPL indent – Data from different organisations

Sample	HV30 NPL value	**Baildonit** crack length µm	Toughness W_K MN $m^{-3/2}$	HV30 NPL value	**UPC** crack length µm	Toughness W_K MN $m^{-3/2}$
B1	1778	434	9.6	1778	492	9.0
B2	1626	406	9.5	1626	448	9.1
H1	1810	483	9.2	1810	568	8.5
H2	1592	431	9.1	1592	420	9.3
H3	1364	154	14.2	1364	172	13.4
TCM10	1636	606	7.8	1636	648	7.6
K313	1726	462	9.2	1726	412	9.7
K420	1486	315	10.3	1486	288	10.8
K3560	996*			996*	188	20.0

* HV100

Sample	HV30 NPL value	**NPL** crack length µm	Toughness W_K MN $m^{-3/2}$	HV30 NPL value	**BAM** crack length µm	Toughness W_K MN $m^{-3/2}$
B1	1778	509	8.9	1778	508	8.9
B2	1626	436	9.2	1626	440	9.1
H1	1810	538	8.7	1810	547	8.7
H2	1592	419	9.3	1592	468	8.8
H3	1364	178	13.2	1364	164	13.7
TCM10	1636	620	7.7	1636	634	7.6
K313	1726	439	9.4	1726	460	9.2
K420	1486	323	10.2	1486	349	9.8
K3560	996*	167	21.2	996*	291	16.1

* HV100

Table 11 NPL prepared surface – Data from different organisations

Sample	HV30 NPL value	**Cermep** crack length µm			Mean crack length µm	Toughness W_K MN $m^{-3/2}$
B1	1778	490	504	498	497	9.0
B2	1626	438	430	430	433	9.2
H1	1810	518	550	542	537	8.7
H2	1592	424	412	420	419	9.3
H3	1364	156	158	186	167	13.6
TCM10	1636	638	640	634	637	7.6
K313	1726	424	448	456	443	9.4
K420	1486	318	326	322	322	10.2
K3560	996*					

* HV100

Sample	HV30 NPL value	**Plansee** crack length µm			Mean crack length µm	Toughness W_K MN $m^{-3/2}$
B1	1778	493	492	501	495	9.0
B2	1626	432	424	428	428	9.3
H1	1810	529	519	537	528	8.8
H2	1592	389	409	413	404	9.4
H3	1364	143	160	150	151	14.3
TCM10	1636	640	638	636	638	7.6
K313	1726	430	417	458	435	9.5
K420	1486	322	310	310	314	10.3
K3560	996*	288	296	332	305	17.5

* HV100

Table 11 (continued) NPL prepared surface – Data from different organisations

Sample	HV30 NPL value	**Teledyne** crack length µm			Mean crack length µm	Toughness W_K MN $m^{-3/2}$
B1	1778	508	516	512	512	8.9
B2	1626	436	444	452	444	9.1
H1	1810	548	556	560	555	8.6
H2	1592	436	440	452	443	9.0
H3	1364	188	188	192	189	12.8
TCM10	1636	620	624	608	617	7.7
K313	1726	448	464	440	451	9.3
K420	1486	352	348	348	349	9.8
K3560	996*	248	280	244	257	17.1

* HV100

Sample	HV30 NPL value	**NPL**[+] crack length µm			Mean crack length µm	Toughness W_K MN $m^{-3/2}$
B1	1778	+	See Table B1	+	509	8.9
B2	1626	+	+	+	436	9.2
H1	1810	+	+	+	538	8.7
H2	1592	+	+	+	419	9.3
H3	1364	+	+	+	178	13.2
TCM10	1636	+	+	+	620	7.7
K313	1726	+	+	+	439	9.4
K420	1486	+	+	+	323	10.2
K3560	996*	+	+	+	167	21.2

* HV100

[+] Data taken from means of 16 tests (Appendix B, Table B1)

Table 11 (Continued) NPL prepared surface – Data from different organisations

Sample	HV30 NPL value	**Hughes** crack length µm			Mean crack length µm	Toughness W_K MN $m^{-3/2}$
B1	1778	496	502	511	503	8.9
B2	1626	432	464	436	444	9.1
H1	1810	539	528	533	533	8.8
H2	1592	437	426	426	430	9.2
H3	1364	189	178	183	183	13.0
TCM10	1636	618	618	598	611	7.8
K313	1726	457	460	451	456	9.3
K420	1486	310	322	299	310	10.4
K3560	996*	248	192	228	223	18.4

* HV100

Sample	HV30 NPL value	**Baildonit** crack length µm			Mean crack length µm	Toughness W_K MN $m^{-3/2}$
B1	1778	494	480	473	482	9.1
B2	1626	420	424	431	425	9.3
H1	1810	490	518	532	513	8.9
H2	1592	413	434	424	424	9.2
H3	1364	193	172	168	178	13.2
TCM10	1636	606	582	630	606	7.8
K313	1726	427	466	431	441	9.4
K420	1486	319	329	308	319	10.3
K3560	996*				0	

* HV100

Table 11 (Continued) NPL prepared surface – Data from different organisations

Sample	HV30 NPL value	**UPC** crack length µm			Mean crack length µm	Toughness W_K MN $m^{-3/2}$
B1	1778	500	496	512	503	8.9
B2	1626	428	444	408	427	9.3
H1	1810	556	564	568	563	8.5
H2	1592	396	392	384	391	9.6
H3	1364	132	136	144	137	15.0
TCM10	1636	736	744	764	748	7.0
K313	1726	456	436	448	447	9.4
K420	1486	288	276	264	276	11.0
K3560	996*	192	180	164	179	20.5

* HV100

Sample	HV30 NPL value	**BAM** crack length µm			Mean crack length µm	Toughness W_K MN $m^{-3/2}$
B1	1778	502	506	505	504	8.9
B2	1626	452	442	422	439	9.2
H1	1810	543	531	534	536	8.7
H2	1592	434	429	408	424	9.2
H3	1364	168	170	162	167	13.6
TCM10	1636	592	622	633	616	7.8
K313	1726	433	426	455	438	9.4
K420	1486	307	308	320	312	10.4
K3560	996*					

* HV100

Table 12 Organisation prepared surface – Data from different organisations

Sample	HV30 NPL value	**Cermep** crack length µm			Mean crack length µm	Toughness W_K MN $m^{-3/2}$
B1	1778	398	406	398	401	10.0
B2	1626	330	296	246	291	11.3
H1	1810	378	388	378	381	10.4
H2	1592	388	382	384	385	9.7
H3	1364	156	150	156	154	14.2
TCM10	1636	526	538	490	518	8.5
K313	1726	428	394	412	411	9.7
K420	1486	296	296	294	295	10.7
K3560	996*					

* HV100

Sample	HV30 NPL value	**Plansee** crack length µm			Mean crack length µm	Toughness W_K MN $m^{-3/2}$
B1	1778	521	507	494	507	8.9
B2	1626	395	383	393	390	9.7
H1	1810	522	513	515	517	8.9
H2	1592	371	373	375	373	9.8
H3	1364	148	160	150	153	14.2
TCM10	1636	622	636	648	635	7.6
K313	1726	464	478	460	467	9.1
K420	1486	340	349	342	344	9.9
K3560	996*	286	320	207	271	18.6

* HV100

Sample	HV30 NPL value	**Teledyne** crack length µm			Mean crack length µm	Toughness W_K MN $m^{-3/2}$
B1	1778	540	536	544	540	8.6
B2	1626	508	488	496	497	8.6
H1	1810	572	568	568	569	8.5
H2	1592	512	508	512	511	8.4
H3	1364	300	276	260	279	10.5
TCM10	1636	604	596	608	603	7.8
K313	1726	544	544	504	531	8.6
K420	1486	388	368	360	372	9.5
K3560	996*	248	272	328	283	16.3

* HV100

Table 12 (Continued) Organisation prepared surface – Data from different organisations

Sample	HV30 NPL value	Hughes crack length µm			Mean crack length µm	Toughness W_K MN $m^{-3/2}$
B1	1778	503	498	493	498	9.0
B2	1626	415	415	429	420	9.4
H1	1810	542	536	530	536	8.7
H2	1592	403	406	426	412	9.4
H3	1364	172	192	206	190	12.7
TCM10	1636	584	553	499	545	8.2
K313	1726	432	426	378	412	9.7
K420	1486	257	305	231	264	11.3
K3560	996*	228	284	257	256	17.1

* HV100

Sample	HV30 NPL value	Baildonit crack length µm			Mean crack length µm	Toughness W_K MN $m^{-3/2}$
B1	1778	469	441	462	457	9.4
B2	1626	399	406	396	400	9.6
H1	1810	483	494	480	486	9.2
H2	1592	322	340	343	335	10.4
H3	1364	182	172	186	180	13.1
TCM10	1636	515	536	529	527	8.4
K313	1726	434	413	417	421	9.6
K420	1486	308	294	301	301	10.6
K3560	996*					

* HV100

Table 12 (Continued) Organisation prepared surface – Data from different organisations

Sample	HV30 NPL value	UPC crack length µm			Mean crack length µm	Toughness W_K MN $m^{-3/2}$
B1	1778	492	500	496	496	9.0
B2	1626	436	420	404	420	9.4
H1	1810	548	560	564	557	8.6
H2	1592	380	388	384	384	9.7
H3	1364	120	128	132	127	15.6
TCM10	1636	760	724	740	741	7.1
K313	1726	456	408	436	433	9.5
K420	1486	284	296	260	280	11.0
K3560	996*	156	164	176	165	21.3

* HV100

Sample	HV30 NPL value	BAM crack length µm			Mean crack length µm	Toughness W_K MN $m^{-3/2}$
B1	1778	488	486	486	487	9.1
B2	1626	406	422	426	418	9.4
H1	1810	420	526	514	487	9.2
H2	1592	369	405	417	397	9.5
H3	1364	150	160	150	153	14.2
TCM10	1636	570	626	641	612	7.8
K313	1726	448	453	443	448	9.3
K420	1486	273	237	287	266	11.3
K3560	996*					

* HV100

The uncertainty in Palmqvist measurements that can arise from two sources, measurement of hardness and measurement of crack length, is demonstrated in Figs 9 and 10, 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 testpieces 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 given in Table B1 (APPENDIX B) and plotted as crack length against hardness in Fig 11 and Palmqvist toughness, W_K, against hardness in Fig 12. 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 Fig 13 and as mean values in Fig 14. 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.

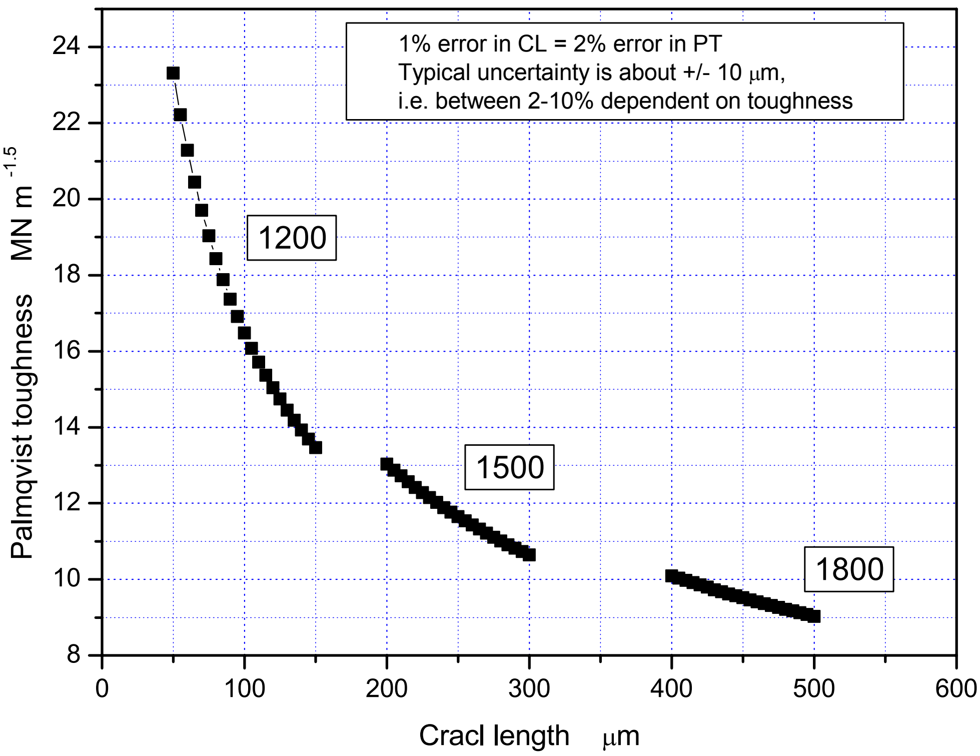

Fig 9 Effect of differences in crack length at constant hardness.

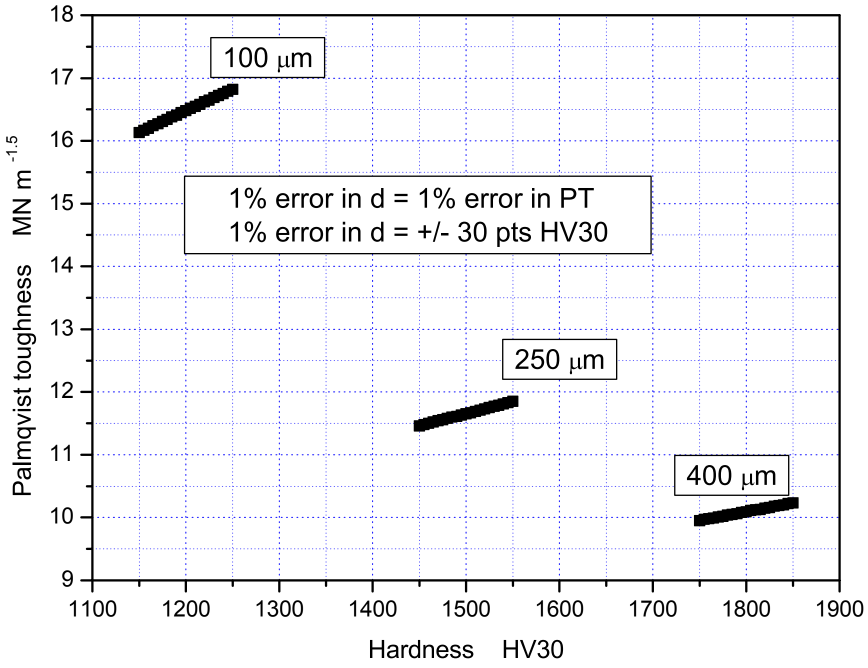

Fig 10 Effect of differences in hardness at constant crack length.

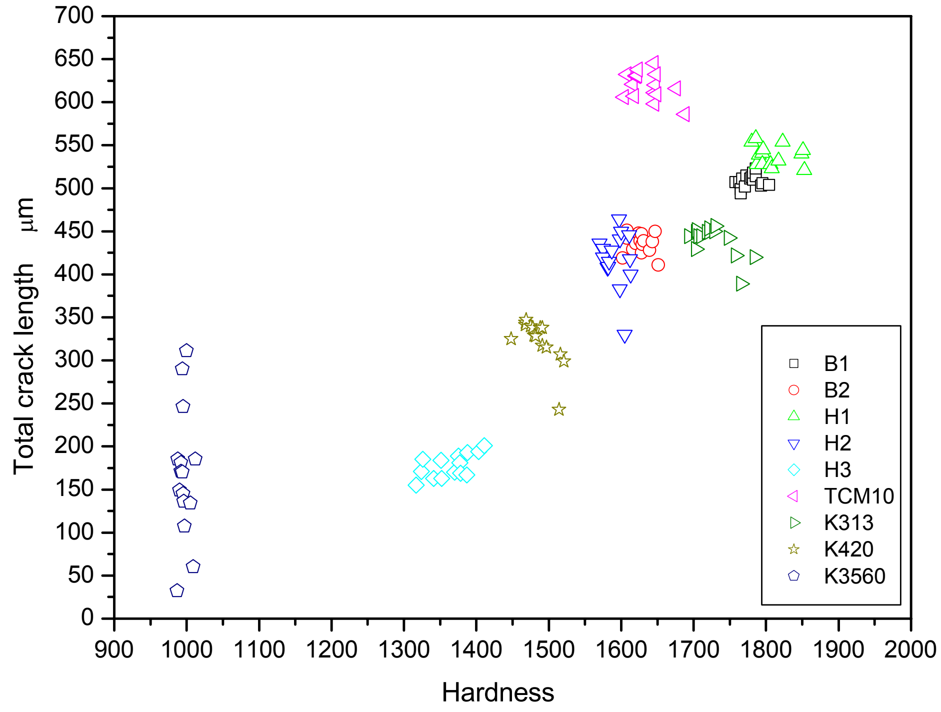

Fig 11 NPL data (single indent) – crack lengths.

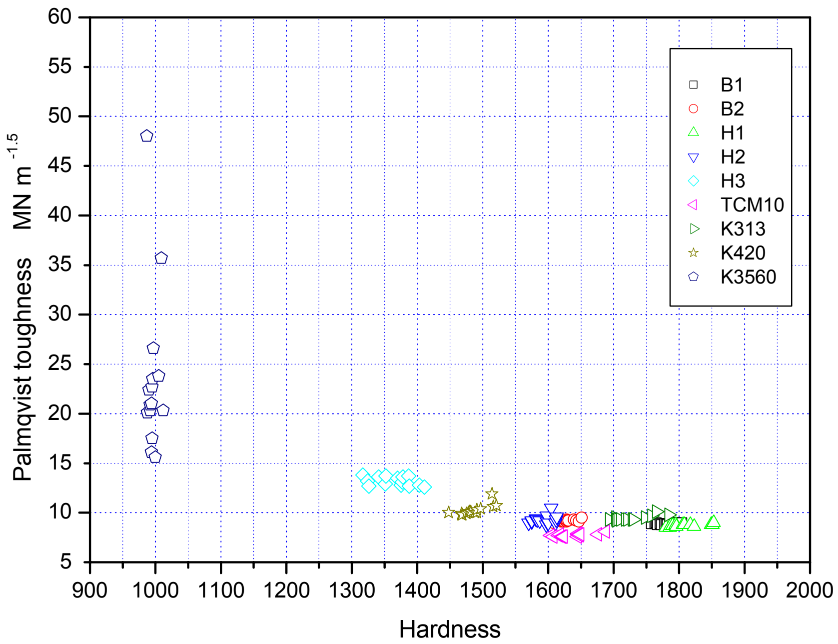

Fig 12 NPL data (single indent) – Palmqvist toughness.

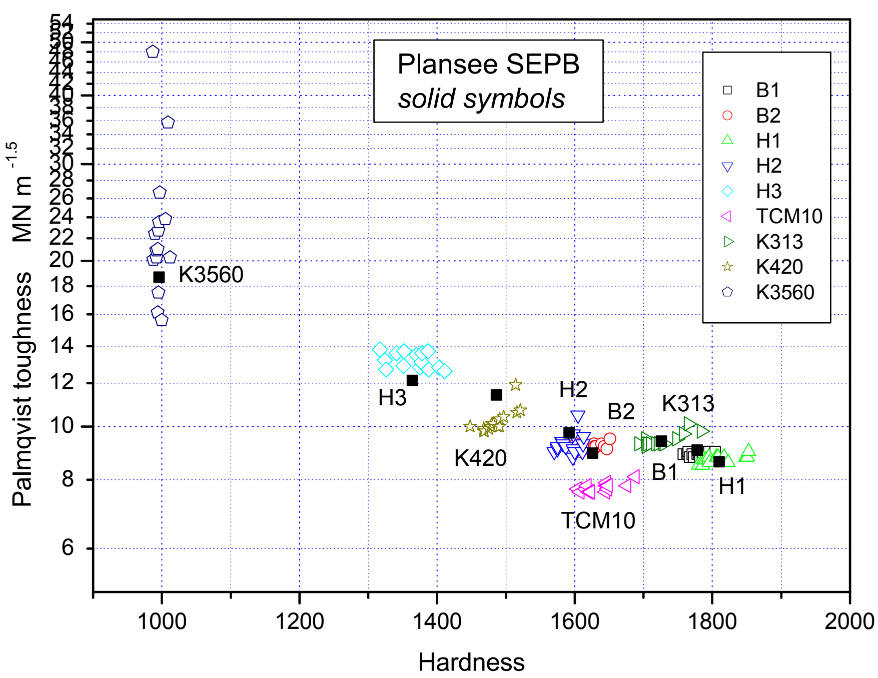

Fig 13 Comparison of Plansee SEPB and NPL Palmqvist data (log scale).

A comparison of results from all the participating organisations is shown in Figs 14-16, where the three figures show

Fig 14 - data on the NPL single indent.
Fig 15 - data on the NPL polished surface.
Fig 16 - data on the surface polished by the participating organisation.

NB: Hardness data is HV100 for the softest hardmetal; HV30 for the remainder in Figs 14-16.

Each figure has three plots:

- crack length against hardness.
- Palmqvist toughness against hardness.
- expanded plot of the full toughness/hardness graph for the harder grades.

A coefficient of variation (CV) was calculated for each material in each of the different groups of measurements (Table 13) and this CV is shown in Fig 17 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 1-10% 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

$$\text{Log}_{10}\ CV_L = a - bH \tag{7}$$

where a and be 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 1-2% at HV1000.

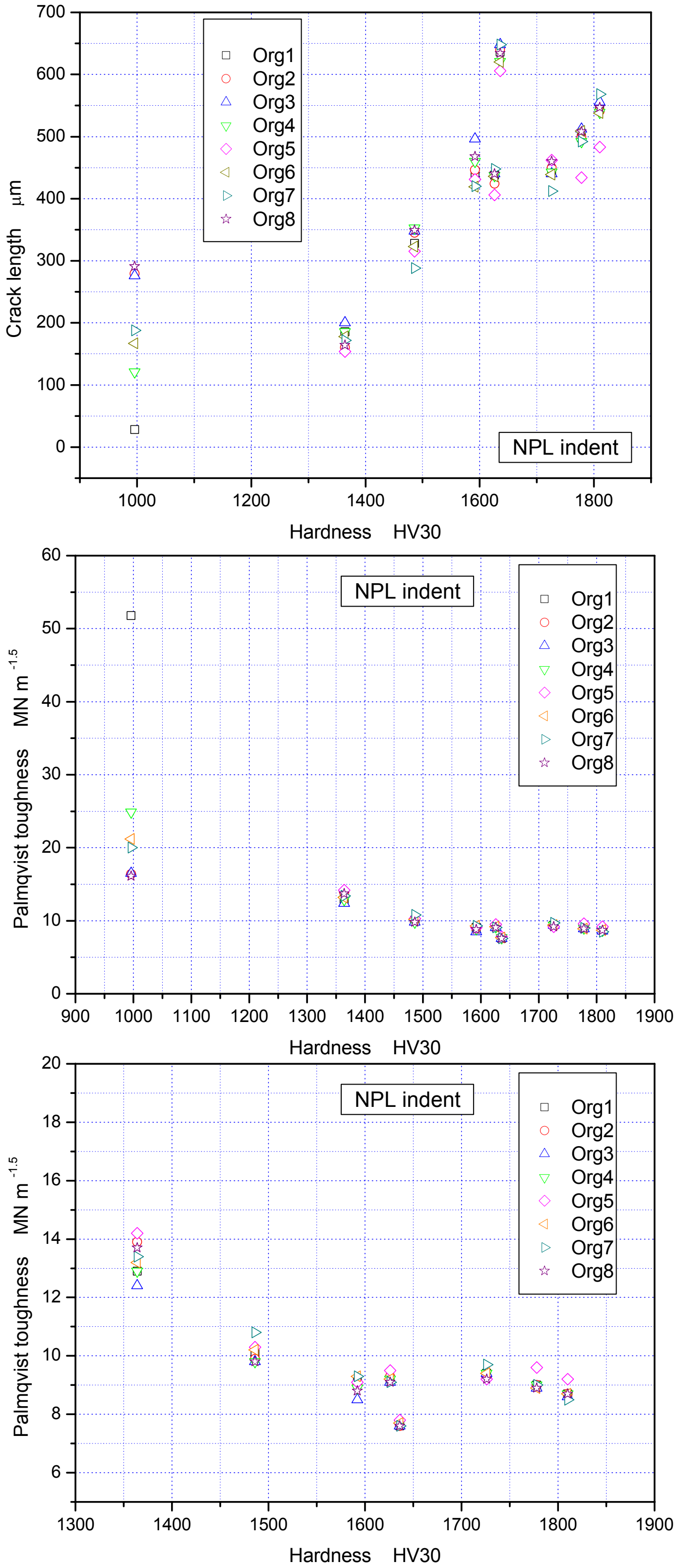

Fig 14 NPL single indent.

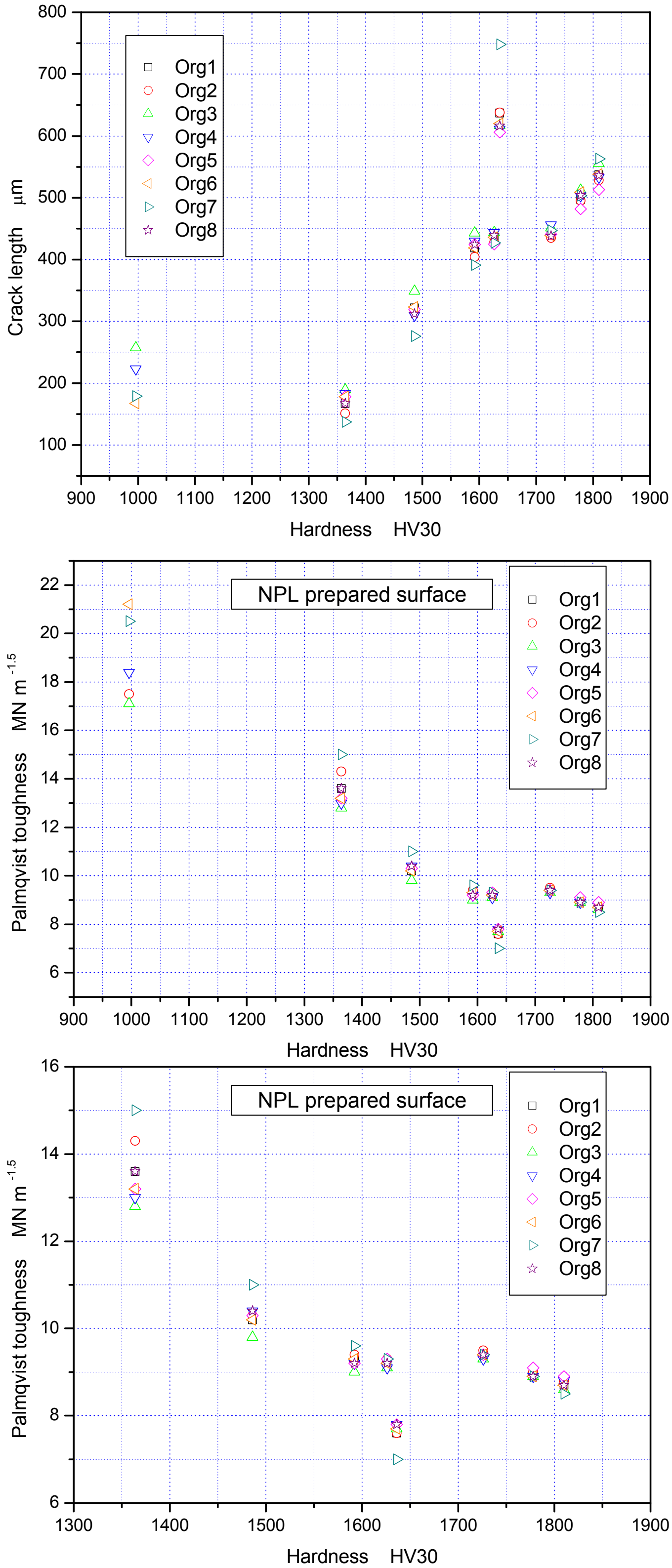

Fig 15 NPL polished surface.

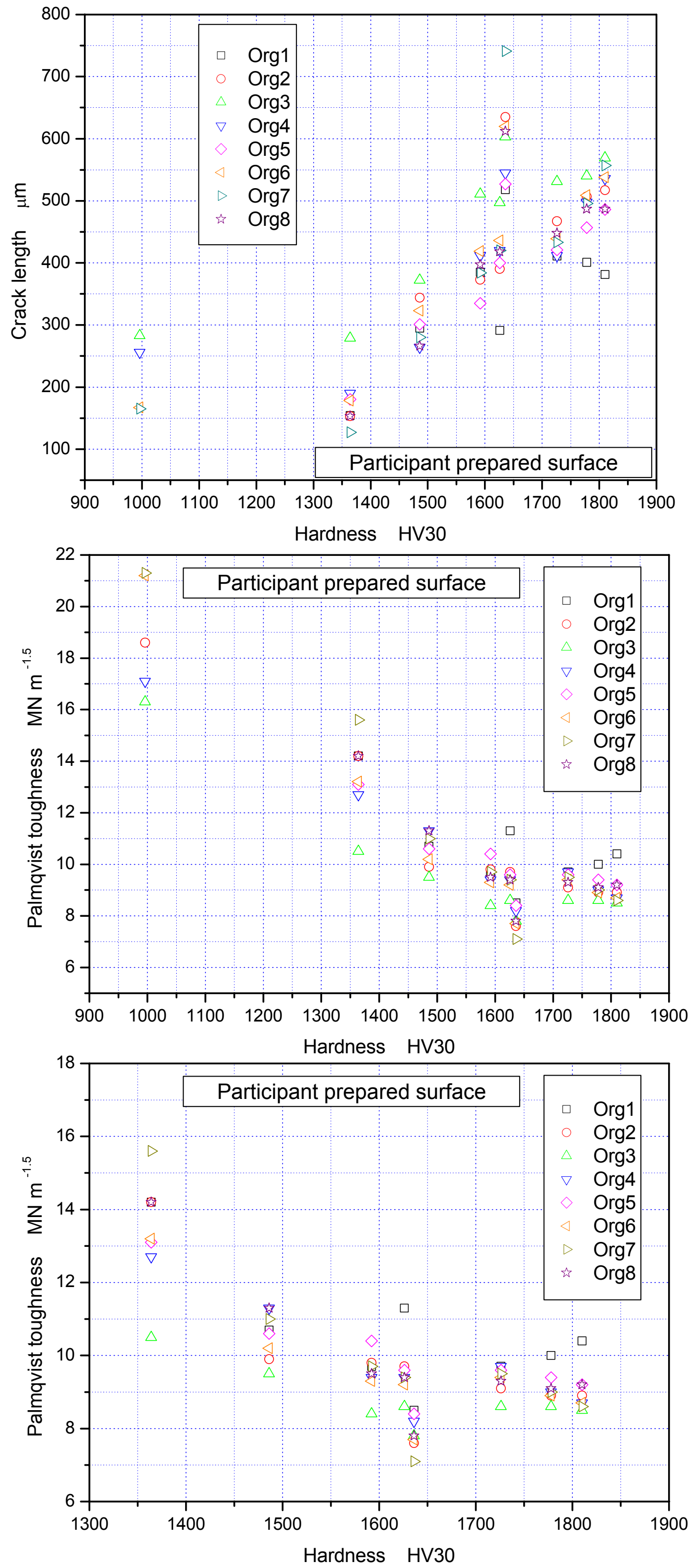

Fig 16 Organisation polished surface.

Table 13 Coefficient of variation (CV) of Palmqvist Crack Lengths

Sample	HV	CV, % NPL single indent	CV, % NPL prepared surface	CV, % Organisation prepared surface
K3560	996	32.2	25.1	25.4
H3	1364	8.8	10.3	26
K420	1486	6.7	6.4	12.5
H2	1592	5.9	3.8	12.7
B2	1626	3	1.7	14.1
TCM10	1636	2.3	7.3	12.1
K313	1726	3.5	1.6	8.8
B1	1778	5.1	1.8	8.6
H1	1810	4.6	2.8	11.8

Table 14 Ranking of Crack Lengths – Anneal or Not

B1	B2	H1	H2	H3	TCM10	K313	K420	K3560	Mean ranking	Organisation	Ordered ranking	Organisation	Anneal (Y) or (N)
1	1	1	4	4	1	1	4		2.3	CERMeP	2.3	CERMeP	N
6	2	4	2	2	7	7	7	5	4.3	Plansee	3.1	Baildonit	Y
8	8	8	8	8	4	8	8	6	8.0	Teledyne	3.7	BAM	N
5	5	5	6	7	3	2	1	3	4.4	Hughes	4.0	UPC	N
2	3	2	1	6	2	3	5		3.1	Baildonit	4.3	Plansee	Y
4	6	7	3	1	8	4	3	1	4.0	UPC	4.4	Hughes	N
3	4	3	5	3	5	6	2		3.7	BAM	6.1	NPL	Y
7	7	6	7	5	6	5	6	2	6.1	NPL	8.0	Teledyne	Y

The measured crack lengths for each testpiece were ranked by organisation, Table 14, to see if there was a systematic effect of annealing. The lower the ranking number the shorter the crack length (implying the greater the effect of residual stress.) The results were not obviously clear cut although there were more Ns in the top half of the rank (i.e. shorter crack lengths corresponding to testpieces that had not been annealed) than in the lower half.

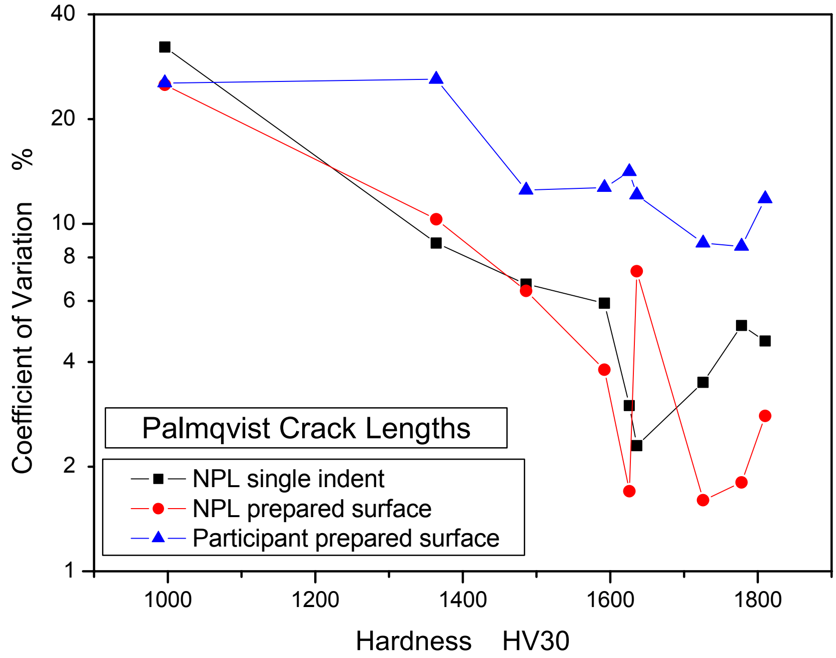

Fig 17 Effect of hardness and surface preparation method on coefficient of variation of Palmqvist crack lengths.

5 Conclusion

An interlaboratory exercise was conducted to generate underpinning technical information on toughness tests for hardmetals. The results will enable good practice for toughness tests to be developed.

More than ten 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 have enabled a quantitative assessment of uncertainties to be performed for this relatively simple test. Single edge precracked beam data was thought to be closest to the "true" value and most of the short rod chevron notch test data compared reasonably well with these results. However, care was needed in testpiece preparation to ensure a good correlation between data from the Palmqvist tests and the single edge precracked beam results.

Following circulation of this report of the interlaboratory exercise, an ISO Technology Trends Assessment document is planned, as a first step in recommending appropriate suitable test methods that will have wider acceptance across industries that make and use hardmetals.

6 References

1. Warren, R. and Matzke, H.J. Indentation testing of a broad range of cemented carbides, *Proc 1st Int. Conf. on Science of Hard Materials*, edited by Viswanadham, Rowcliffe and Gurland, Plenum Press, New York, 1981, 563-82.

2. Shetty, D.K., Wright, I.G., Mincer, P.N. and Clauer, A.H. Indentation fracture toughness of WC-Co composites, *J. Mater. Sci.*, 1985, **20**, 1873-82.

3. Spiegler, R., Schmauder, S. and Sigl, L.S. *J. Hard Mater.* 1990, **1**(3), 147-158.

4. Almond, E.A. and Roebuck, B. A Recommended Practice for a Plane Strain Fracture Toughness Test on Hardmetals, NPL Report DMA(B)4, February 1979.

5. Exner, H.E. The influence of sample preparation on Palmqvist's method for toughness testing of cemented carbides. *Trans. Met. Soc. AIME*, 1969, **245**, 677-683.

6. Viswanadham, R.K. and Venables, J.D. A simple method for evaluating cemented carbides. *Met. Trans. A.*, 1977, **8A**, 187-191.

7. Peters, C.T. The relationship between Palmqvist indentation toughness and bulk fracture toughness for some WC-Co cemented carbides. *J. Mater. Sci.*, 1979, **14** (7), 1619-1623.

8. Niihara, K., Morena, R. and Hasselman, D.P.H. Evaluation of K_{Ic} of brittle solids by the indentation method with low crack-to-indent ratios. *J. Mater. Sci. Lett.*, 1982, **1**, 13-16.

9. Laugier, M.T. Palmqvist cracking in WC-Co composites. *J. Mater. Sci. Lett.*, 1985, **4** (2), 207-210.

10. Singh, S. and Ramakrishnan, P. Palmqvist toughness of cemented carbide alloys. *Int. J. Refract. Hard Met.*, 1985, **4** (1), 27-30.

11. Laugier, M.T. Palmqvist indentation toughness in WC-Co composites. *J. Mater. Sci. Lett.*, 1987, **6**, 897-900.

12. Laugier, M.T. Palmqvist toughness in WC-Co composites viewed as a ductile/brittle transition. *J. Mater. Sci. Lett.*, 1987, **6**, 768-770.

13. Shin, Y., Cao, W., Sargent, G. and Conrad, H. Effects of microstructure on hardness and Palmqvist fracture toughness of WC-Co alloys. *Mater. Sci. Eng.*, 1988, **A105-A106**, 377-382.

14. Zhang, S.H. and Liu, Y.X. Palmqvist indentation fracture toughness of WC-Co cemented carbide alloys. *Modern Developments in Powder Metallurgy, **Vol 19***, Orlando, Florida, USA, June 1988. Metal Powder Industries Federation, USA. 33-41.

15. Laugier, M.T. Validation of the Palmqvist indentation approach to toughness determination in WC-Co composites. *Ceram. Int.*, 1989, **15** (2), 121-125.

16. Heinonen, M. Comparison of Fracture Toughness Values for Cermet Materials, MSc Thesis, 1996, UMIST, Manchester, UK.

17. Roebuck, B. and Almond, E.A. Deformation and fracture processes and the physical metallurgy of WC/Co hardmetal. *Int. Mater. Rev.* 1988, **33**(2), 90-110.

18. Palmqvist, S. Metod att bestämma segheten hos spröda material, särsskilt hårdmetaller. *Jernkontorets. Ann.*, 1957, **141**, 300.

19. Palmqvist, S. Riβbildungsarbeit bei Vickers-Eindrücken als Maβ für die Zähigteit von Hartmetallen. *Arch. Eisenhutternwes.*, **33**, 1962, 629-634.

20. Roebuck, B., Bennett, E.G., Lay, L.A. and Morrell, A. NPL(GPG)009, June 1998. The Measurement of Palmqvist Toughness for Hard and Brittle Materials.

21. Schubert, W.D. personal communication, 2004.

22. Schubert, W.D, Neumeister, H., Kinger, G. and Lux, B. Hardness to toughness relationship of fine-grained WC-Co hardmetals, 14th International Plansee Seminar, V4, Reutte, Austria, May 1997 and Refractory Metals and Hard Materials, 16, 1988, 133-142.

23. Yohe, W.

24. Gee, M.G., Lay, L.A. and Roebuck, B. NPL DMM(A)33, October 1991. The Palmqvist Test Method for Ti-based Cermets and WC/Co Hardmetals.

25. Almond, E.A. and Roebuck, B. Precracking of Fracture-Toughness Specimens of Hardmetals by Wedge Indentation, Met. Technol., 1978, 5, 92-99.

26. Tada, Paris and Irvine. Stress Analysis of Cracks Handbook, St Louis, Paris Productions Inc, 1973, 2.13-2.15.

27. Luis Lanes Fatigue paper(s).

7 Acknowledgements

This document was written with the support of the DTI Materials Programme. Thanks are also due to international members of VAMAS TWA 21 for participation and comments and to participants in the Europe PM Association Hard Materials Group (EHMG) WINTEREV seminar at NPL in February 2004 on Toughness Testing for Hardmetals.

APPENDIX A

PALMQVIST TESTS
Information from NPL Good Practice Guide – 1998 and from University of Vienna (Professor W-D Schubert)

A1.1 TESTPIECES AND SAMPLE PREPARATION

A1.1.1 TESTPIECE SIZE AND SAMPLING

Any testpiece 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 electrospark 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, University of Manchester Institute of Science and Technology, that the testpiece should be at least as thick as ten times the crack length. Thinner testpieces 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 testpieces in cold-setting or hot-setting resins to directly provide flat and parallel faces. However, if the testpieces are to be subsequently annealed to remove surface residual stresses then this can be a disadvantage since the testpiece has to be removed from the mount to put it in the annealing furnace (typically 800 ^{o}C for 1h in vacuum).

A1.1.2 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 testpieces. The main body of this appendix is taken from the NPL Good Practice Guide which recommends annealing at 800 ℃ for 1h in vacuum before making Palmqvist indentations. However, since the publication of the Guide Professor W-D Schubert's Group at the University of Vienna have *conducted extensive studies of the properties of very fine grained hardmetals, often containing alloy binder-phase. Because of the possibility of ageing in reactions in the binder-phase during an annealing step this group developed a mechanical polishing route prior to conducting indentation tests to minimise residual stresses (Fig A1 gives some representative results) and thin must be considered as an alternative in good testing procedure.*

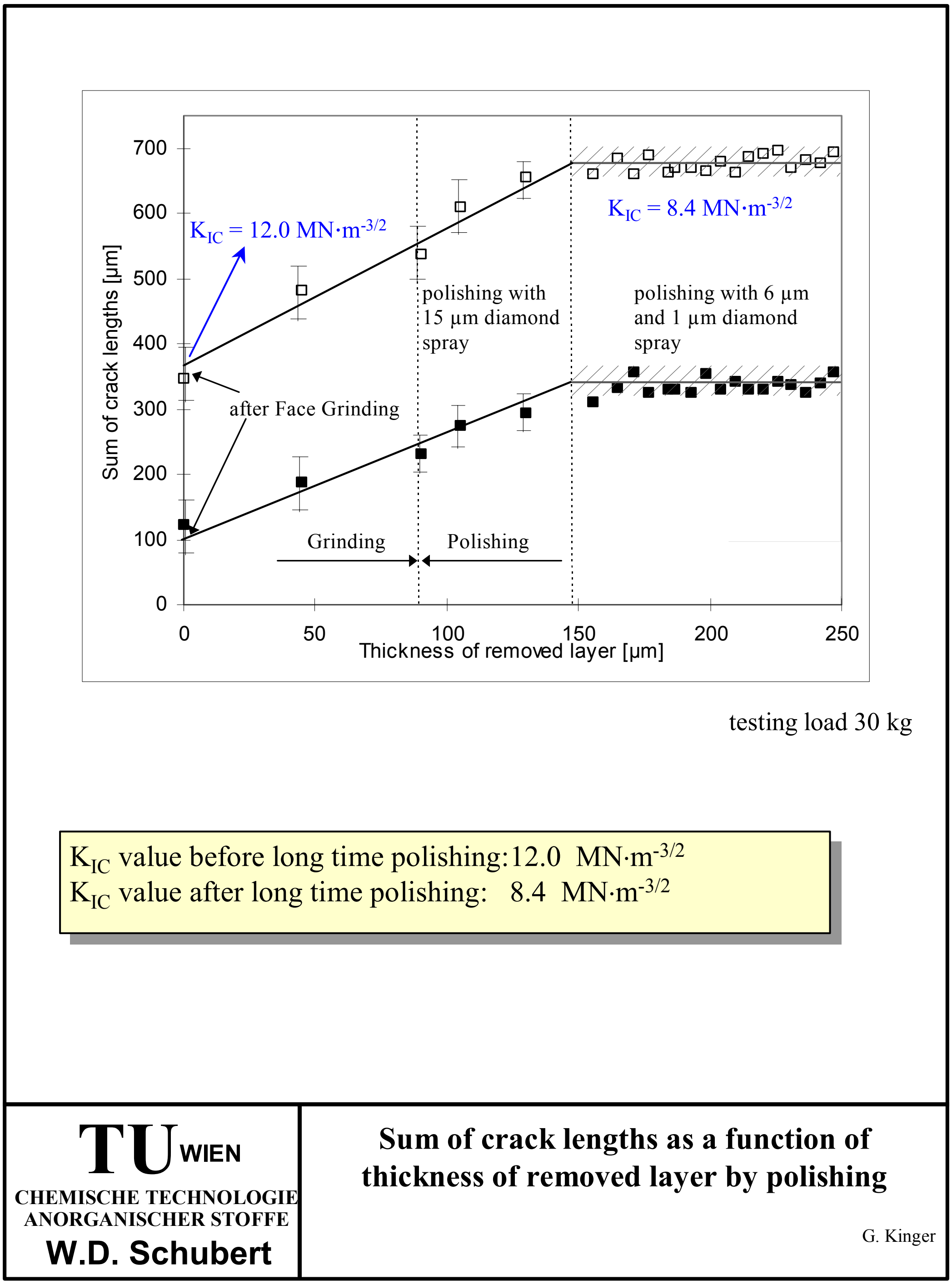

Fig A1 Effect of surface layer removal by polishing (Courtesy Prof W-D Schubert – TU Wien)

A1.1.3 SURFACE CONDITION

It has been shown that surfaces free of residual stress are required for consistent results. No polishing procedure can guarantee a stress-free surface without tedious systematic measurements which are not feasible on a regular basis. At NPL all testpieces are polished so that the microstructure can be observed and then annealed at 800 ^oC for 1h in vacuum following the studies published by Exner. 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 2h at 800 ^oC. 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 an anneal this must be indicated in the test report.

A.2 APPARATUS

The indentations should be introduced into the testpiece 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 4.3.1 and 4.3.2.

A2.1 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.

A2.2 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.

A.3 PROCEDURE AND CONDITIONS OF TESTING

A3.1 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, 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.

A3.2 INDENTATION AND CRACK LENGTH MEASUREMENTS

It is recommended that the indentation diagonal and crack lengths are measured optically at a magnification of at least ×500. Alternatively, the optical system of the Vickers hardness machine can be used (×100) as this has been shown to give equivalent results. 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 ×500 magnification the test should be repeated because of a lack of flatness of the testpiece.*

There are two methods for measuring the crack length. The results are independent of the method. 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. *If the magnification is ×500 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 ×500 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.

A3.3 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 to be of a lower reliability. *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 about 4 μm for an indentation load of 30 kgf the surface is not sufficiently flat and the test is invalid.

A4 ANALYSIS

A4.1 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.

A4.2 TOUGHNESS

Two different values for toughness can be calculated, Palmqvist toughness, W_G, and Palmqvist fracture toughness, W_K.

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

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.

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

where A is a constant of value 0.0028
and HV is the Vickers hardness in N mm^{-2} (i.e. 9.81 × 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.

APPENDIX B

Toughness Tests for Hardmetals

Detailed Results

Table B1	-	**NPL** Palmqvist data on all samples supplied to participants (includes HV30 and HV100 on material K3560).
Tables B2 – B8	-	Data returned by Participants

B2	**Cermep**	(Palmqvist)
B3	**Plansee**	(Palmqvist and SENB)
B4	**Teledyne**	(Palmvist and Short Rod)
B5	**Hughes Christensen**	(Palmqvist and Short Rod)
B6	**Baildonit**	(Palmqvist)
B7	**UPC**	(Palmqvist and SENB)
B8	**NPL**	(Palmqvist summary)
B9	**BAM**	(Palmqvist and SENB)

Table B1 NPL Palmqvist Measurements on all Samples Prepared for Participants

Material B1

Sample Number	Participant	Hardness HV30	Crack length µm	W_k MN $m^{-3/2}$
1	Archived	1782	518	8.8
2	Hughes Christensen	1771	502	8.9
3	Marshalls	1763	507	8.9
4	Sandvik Hard Materials	1767	511	8.8
5	Kennametal	1780	513	8.9
6	Archived	1758	507	8.9
7	Universitat Politecnica	1778	511	8.9
8	Konrad Friedrichs KG	1765	494	9.0
9	Archived	1804	504	9.0
10	Baildonit	1782	510	8.9
11	Plansee Tizit	1765	499	8.9
12	Boart Longyear	1795	506	9.0
13	United Hardmetals	1786	523	8.8
14	Teledyne	1786	514	8.9
15	Archived	1773	515	8.8
16	BAM	1793	503	9.0
	Mean Value ± sd	**1778 ± 13**	**509 ± 7**	**8.9 ± 0.1**

Material B2

Sample Number	Participant	Hardness HV30	Crack length µm	W_k MN $m^{-3/2}$
1	Konrad Friedrichs KG	1651	411	9.5
2	Teledyne	1643	438	9.2
3	Plansee Tizit	1628	425	9.3
4	Archived	1602	419	9.3
5	Archived	1639	428	9.3
6	Archived	1608	451	9.0
7	Kennametal	1616	429	9.2
8	Hughes Christensen	1626	439	9.2
9	United Hardmetals	1631	439	9.2
10	Boart Longyear	1624	448	9.1
11	Universitat Politecnica	1647	450	9.1
12	Baildonit	1629	435	9.2
13	Archived	1620	440	9.1
14	BAM	1620	436	9.2
15	Marshalls	1628	447	9.1
16	Sandvik Hard Materials	1608	442	9.1
	Mean Value ± sd	**1626 ± 14**	**436 ± 11**	**9.2 ± 0.1**

Table B1 (Continued) NPL Palmqvist Measurements on all Samples Prepared for Participants

Material H1

Sample Number	Participant	Hardness HV30	Crack length μm	W_k MN $m^{-3/2}$
1	Plansee Tizit	1795	541	8.7
2	Kennametal	1853	521	9.0
3	Baildonit	1795	528	8.8
4	Hughes Christensen	1780	554	8.5
5	Konrad Friedrichs KG	1790	539	8.7
6	Sandvik Hard Materials	1808	523	8.8
7	Universitat Politecnica	1851	544	8.8
8	Teledyne	1796	545	8.6
9	Archived	1849	540	8.8
10	United Hardmetals	1817	532	8.8
11	Boart Longyear	1823	554	8.6
12	BAM	1786	558	8.5
13	Archived	1787	528	8.7
14	Marshalls	1806	528	8.8
	Mean Value ± sd	**1810 ± 25**	**538 ± 12**	**8.7 ± 0.1**

Material H2

Sample Number	Participant	Hardness HV30	Crack length μm	W_k MN $m^{-3/2}$
1	BAM	1600	450	9.0
2	Universitat Politecnica	1605	330	10.5
3	Teledyne	1597	464	8.8
4	Boart Longyear	1611	446	9.0
5	Hughes Christensen	1570	436	9.0
6	Sandvik Hard Materials	1575	420	9.2
7	Archived	1613	400	9.6
8	Konrad Friedrichs KG	1598	383	9.7
9	Archived	1583	415	9.3
10	Plansee Tizit	1581	408	9.4
11	Kennametal	1587	428	9.2
12	Marshalls	1582	409	9.4
13	Archived	1612	418	9.3
14	United Hardmetals	1598	441	9.1
15	Baildonit	1575	430	9.1
	Mean Value ± sd	**1592 ± 14**	**419 ± 32**	**9.3 ± 0.4**

Table B1 (Continued) NPL Palmqvist Measurements on all Samples Prepared for Participants

Material H3

Sample Number	Participant	Hardness HV30	Crack length µm	W_k MN $m^{-3/2}$
1	Baildonit	1370	170	13.5
2	Kennametal	1403	194	12.8
3	Sandvik Hard Materials	1411	201	12.6
4	Marshalls	1375	189	12.8
5	Konrad Friedrichs KG	1351	184	12.9
6	Teledyne	1326	185	12.7
7	Plansee Tizit	1378	169	13.6
8	Archived	1377	181	13.1
9	Hughes Christensen	1388	193	12.7
10	Archived	1387	167	13.7
11	Boart Longyear	1324	171	13.2
12	BAM	1317	155	13.8
13	Universitat Politecnica	1352	163	13.7
14	United Hardmetals	1341	163	13.6
	Mean Value ± sd	**1364 ± 30**	**178 ± 14**	**13.2 ± 0.4**

Material TCM10

Sample Number	Participant	Hardness HV30	Crack length µm	W_k MN $m^{-3/2}$
1	Archived	1676	616	7.8
2	Baildonit	1649	609	7.8
3	Universitat Politecnica	1621	630	7.6
4	Archived	1688	586	8.1
5	Archived	1646	611	7.8
6	Archived	1646	598	7.9
7	Hughes Christensen	1621	631	7.6
8	Boart Longyear	1604	606	7.7
9	Marshalls	1608	632	7.6
10	Teledyne	1645	645	7.6
11	United Hardmetals	1648	632	7.7
12	BAM	1622	631	7.6
13	Sandvik Hard Materials	1623	638	7.6
14	Plansee Tizit	1647	620	7.8
15	Kennametal	1616	621	7.7
16	Konrad Friedrichs KG	1618	607	7.8
	Mean Value ± sd	**1636 ± 24**	**620 ± 16**	**7.7 ± 0.1**

Table B1 (Continued) NPL Palmqvist Measurements on all Samples Prepared for Participants

Material K313

Sample Number	Participant	Hardness HV30	Crack length μm	W_k MN $m^{-3/2}$
1	Kennametal	1721	449	9.3
2	Universitat	1784	420	9.8
3	Archived	1765	389	10.1
4	Archived	1758	422	9.7
5	Hughes Christensen	1748	442	9.5
6	Konrad Friedrichs KG	1710	444	9.3
7	Sandvik Hard Materials	1704	451	9.2
8	BAM	1730	456	9.3
9	Teledyne	1693	444	9.3
10	United Hardmetals	1703	429	9.5
11	Boart Longyear	1721	453	9.3
12	Plansee Tizit	1706	444	9.3
13	Baildonit	1718	449	9.3
14	Archived	1728	450	9.3
15	Marshalls	1703	449	9.3
	Mean Value ± sd	**1726 ± 26**	**439 ± 18**	**9.4 ± 0.3**

Material K420

Sample Number	Participant	Hardness HV30	Crack length μm	W_k MN $m^{-3/2}$
1	BAM	1475	340	9.9
2	Boart Longyear	1491	317	10.3
3	Teledyne	1488	338	10.0
4	United Hardmetals	1480	329	10.1
5	Politecnica	1516	307	10.6
6	Archived	1514	243	11.9
7	Archived	1521	299	10.7
8	Konrad Friedrichs KG	1468	344	9.8
9	Archived	1448	325	10.0
10	Marshalls	1482	328	10.1
11	Materials	1491	338	10.0
12	Kennametal	1468	341	9.9
13	Hughes Christensen	1469	347	9.8
14	Plansee Tizit	1478	338	10.0
15	Baildonit	1497	315	10.4
	Mean Value ± sd	**1486 ± 20**	**323 ± 26**	**10.2 ± 0.5**

Table B1 (Continued) NPL Palmqvist Measurements on all Samples Prepared for Participants

Material K3560

Sample Number	Participant	Hardness HV30	Crack length μm	W_k MN $m^{-3/2}$
1	Plansee Tizit	1034	0	N/A
2	Marshalls	990	0	N/A
3	Hughes Christensen	1070	0	N/A
4	Baildonit	1043	0	N/A
5	Konrad Friedrichs KG	1061	0	N/A
6	Archived	1016	0	N/A
7	Sandvik Hard Materials	999	0	N/A
8	Kennametal	1007	0	N/A
9	BAM	1052	0	N/A
10	Boart Longyear	1043	0	N/A
11	United Hardmetals	1025	0	N/A
12	Teledyne	999	0	N/A
	Mean Value ± sd	**1028 ± 26**		

Material K3560

Sample Number	Participant	Hardness HV100	Crack length μm	W_k MN $m^{-3/2}$
1	Archived	1009	60	35.7
2	Universitat Politecnica	1005	134	23.8
3	Archived	1012	185	20.3
4	BAM	995	145	22.7
5	Boart Longyear	994	170	21.0
6	United Hardmetals	1000	311	15.6
7	Teledyne	992	181	20.3
8	Kennametal	987	32	48.0
9	Sandvik Hard Materials	990	149	22.4
10	Archived	988	185	20.1
11	Marshalls	992	171	20.9
12	Hughes Christensen	995	246	17.5
13	Konrad Friedrichs KG	997	107	26.6
14	Plansee Tizit	994	290	16.1
15	Baildonit	996	136	23.5
	Mean Value ± sd	**996 ± 7**	**167 ± 75**	**21.2 ± 8.3**

Table B2

CERMeP

RESULTS

CERMeP
Palmqvist Crack Length Measurements

Sample code	NPL HV30 indent	Total crack length, µm HV30 indents made into NPL polished surface			Total crack length, µm HV30 indent made into surface polished by participant		
	Indent 1	Indent 1	Indent 2	Indent 3	Indent 1	Indent 2	Indent 3
B1	498	490	504	498	398	406	398
B2	438	438	430	430	330	296	246
H1	544	518	550	542	378	388	378
H2	436	424	412	420	388	382	384
H3	186	156	158	186	156	150	156
TCM 10	636	638	640	634	526	538	490
K313	442	424	448	456	428	394	412
K420	328	318	326	322	296	296	294
	NPL HV30 indents	HV100 indent crack lengths made in NPL polished surface			HV100 indent crack lengths made in surface polished by participant		
K3560	28	-	-	-	-	-	-

Comments or observations here

- On the NPL polished surface, the total crack length is almost the same from your and our indent.
- Concerning the surface polished at CERMeP, the total crack length is always smaller than on the NPL polished surface. It is sometimes due to an indent which generates 4 cracks smaller than the cracks of the other indents of the same sample.
- To check the reproducibility of our measurements, other indents have been performed on 3 samples. (Results are presented in Annex.)

Details of in-house polishing routine

- Hot embedding in a glass fibre resin.
- Polishing in 5 steps:
  - - Diamond tray of 75 µm (with a metallic binder)
    3 min at 2 daN, 300 rot/min.
  - - Diamond tray of 30 µm (with an organic binder)
    7 min at 6 daN, 350 rot/min.
  - - Diamond tray of 10 µm (with an organic binder)
    7 min at 7 daN, 350 rot/min.
  - Finishing on a cloth with diamond suspension (3 µm)
    7 min at 4 daN, 350 rot/min.
  - Finishing on a cloth with diamond suspension (1 µm)
    7 min at 4 daN, 350 rot/min.

Before making the new indents, the resin was taken off. A saw-cut was made in the resin and the sample was recovered.

Details of measurement of crack lengths.

The crack lengths have been measured with an optical microscope. The magnification used was ×500. The objectives contain a graduated rule to be able to measure the crack length. A conversion table enabled the crack length to be calculated for the magnification used. Each crack was measured just one time.

Table B3

Plansee

RESULTS

PLANSEE
Palmqvist Crack Length Measurements

Sample Code	NPL HV30 Indent	Total Crack Length, µm HV30 indents made into NPL polished surface			Total Crack Length, µm HV30 indents made into surface polished by participant		
	Indent 1	Indent 1	Indent 2	Indent 3	Indent 1	Indent 2	Indent 3
B1	499	493	492	501	521	507	494
B2	424	432	424	428	395	383	393
H1	541	529	519	537	522	513	515
H2	446	389	409	413	371	373	375
H3	160	143	160	150	148	160	150
TCM 10	640	640	638	636	622	636	648
K313	450	430	417	458	464	478	460
K420	345	322	310	310	340	349	342
	NPL HV100 indents	HV125 indents made into NPL polished surface			HV125 indents made into surface polished by participant		
K3560	279	288	296	332	286	320	207

Comments or observations

HV100 is not possible with our Hardness tester. We used instead HV125.
Problem with HV125 indents.
Crack length on sample K3560 not well distributed. At one corner there was no crack or a significant short crack. In this case the total crack length was reduced. The same problem occurred with tougher grades (H3) at HV30.
In these cases the higher value in total crack length seems more reliable.
Additionally, we observed some smaller cracks around the K3560 indents. These are not included in our measurement.
We think that the Palmqvist toughness is not very reliable for such tough grades.

Details of the in-house polishing routine

After grinding with a fine (10 µm) diamond grinding wheel the samples were polished with diamond polishing paste (Struers DP-P 3 µm) – time: 4 minutes. After this, the samples were annealed at 800 ^{o}C/2 hours in vacuum. In some cases we observed an annealing structure on the surface and repolished slightly for 1 minute.

Details of measurement of the crack lengths

We measure the crack lengths in an optical microscope (magnification 500). We see the Palmqvist-method as a coarse approximation therefore we did not work out a better method of crack length measurement (SEM etc).
For precise toughness measurements we use the K_{IC}-method described by L Sigl (1985) with modified crack initiation.

PLANSEE
SEPB Results

Material	Code	Fracture Toughness K_{IC} MN $m^{-3/2}$	Standard Deviation
B1	1	8.90	
	2	8.95	
	3	9.15	
	4	9.18	
Mean		**9.05**	**0.14**
B2	1	8.77	
	2	9.04	
	3	8.98	
	4	8.97	
Mean		**8.94**	**0.12**
H1	1	8.76	
	2	8.40	
	3	8.66	
	4	8.69	
Mean		**8.63**	**0.16**
H2	1	9.47	
	2	9.98	
	3	9.66	
	4	9.86	
Mean		**9.74**	**0.22**
H3	1	12.04	
	2	12.31	
	3	12.01	
	4	12.12	
Mean		**12.12**	**0.14**
K313	1	9.23	
	2	9.47	
	3	9.43	
	4	9.45	
Mean		**9.40**	**0.11**
K420	1	11.37	
	2	11.45	
	3	11.42	
	4	11.42	
Mean		**11.41**	**0.03**
K3560	1	18.62	
	2	18.77	
	3	18.67	
Mean		**18.69**	**0.08**

Table B4

Teledyne

RESULTS

TELEDYNE
Palmqvist Crack Length Measurements

Sample Code	NPL HV30 Indent	Total Crack Length, μm HV30 indents made into NPL polished surface			Total Crack Length, μm HV30 indents made into surface polished by participant		
	Indent 1	Indent 1	Indent 2	Indent 3	Indent 1	Indent 2	Indent 3
B1	512	508	516	512	540	536	544
B2	440	436	444	452	508	488	496
H1	556	548	556	560	572	568	568
H2	496	436	440	452	512	508	512
H3	200	188	188	192	300	276	260
TCM 10	648	620	624	608	604	596	608
K313	440	448	464	440	544	544	504
K420	348	352	348	348	388	368	360
	NPL HV100 indents	HV125 indents made into NPL polished surface			HV125 indents made into surface polished by participant		
K3560	276	248	280	244	248	272	328

Comments or observations

Details of the in-house polishing routine

The samples were prepared for polishing by hot mounting in resin. Each sample was placed in a clamped, levelling holder. The samples were then ground and polished as follows:

~45 sec on a Struers 20 μm diamond grinding disc.
2.5 min on an allegro disc with Struers 6 μm diamond suspension.
5 min on a silk polishing pad with Struers 6 μm diamond suspension.
5 min on a silk polishing pad with Struers 1 μm diamond suspension.

After polishing the samples surfaces were suitable for metallographic examination.
The samples were then removed from the mounting material and cleaned with alcohol. They were then placed in a tube furnace and annealed for 2 hours at 850 ^{o}C in hydrogen to relieve surface stresses. The samples were cooled in an argon atmosphere to room temperature and then removed to testing.

Details of measurement of the crack lengths
i.e. imaging technique (microscope, electron microscope) magnification, image analyser.

The sample crack lengths were measured on an optical microscope with a Boeckler VIA-150 video measuring system at 500× magnification.

TELEDYNE – Short Rod Data
VAMAS FRACTURE TOUGHNESS ROUND ROBIN TEST RESULTS:
ASTM-B09.06 TASK FORCE ON FRACTURE TOUGHNESS OF CEMENTED CARBIDES

Material Code	Sample No	Diameter B (in)	Length W (in)	Slot Depth (a_o) in	Cord Angle θ (deg)	Slot Thickness τ (in)	K (Max) MPa	Correction Factor ρ	Comments
H1	4492	.4993	.7427	.265	58	.015	7.340	No data	Bad graph
H1	4493	.5004	.7462	.250	58	.015	7.540	-.037	
H1	4496	.5005	.7456	.252	58	.015	11.03	-.43	
H1	4497	.5027	.7498	.251	58	.015	11.03	-.103	
H1	4498	.5022	.7502	.248	58	.015	No data	No data	Diameter too big
H1	4499	.5028	.7502	.255	58	.015	No data	No data	Diameter too big
H2	4555	.5027	.7452	.250	58	.015	11.51	No data	Bad graph
H2	4562	.5014	.7423	.251	59	.015	11.68	No data	Bad graph
H2	4563	.5045	.7504	.251	58	.015	12.33	-.113	
H2	4564	.5008	.7419	.251	58	.015	14.07	-.259	
H2	4565	.5019	.7433	.258	58	.015	11.53	-.266	
H2	4554	.5004	.7396	.260	58	.015	10.97	-.270	
H3	4732	.5044	.7453	.253	58	.015	No data	No data	Diameter too big
H3	4738	.5078	.7509	.265	58	.015	No data	No data	Diameter too big
H3	4739	.5058	.7467	.268	59	.015	No data	No data	Diameter too big
H3	4741	.5054	.7500	.259	58	.015	No data	No data	Diameter too big
H3	4742	.5068	.7520	.247	58	.015	No data	No data	Diameter too big
H3	4744	.5047	.7463	.252	58	.015	No data	No data	Diameter too big
K313	5460	.5001	.7507	.252	57	.015	8.880	No data	Bad graph
K313	5461	.4997	.7510	.251	58	.015	8.630	-.075	
K313	5463	.5000	.7505	.253	58	.015	9.090	-.093	
K313	5465	.5000	.7507	.250	58	.015	8.990	-.045	
K313	5466	.5000	.7510	.254	58	.015	10.04	+.021	
K313	5467	.5003	.7505	.252	58	.015	9.070	-.074	
K420	5603	.4998	.7498	.250	58	.015	10.69	-.033	
K420	5618	.5002	.7498	.250	58	.015	10.69	-.037	
K420	5619	.4999	.7506	.248	58	.015	10.86	+.018	
K420	5620	.5001	.7515	.254	58	.015	11.07	-.088	
K420	5623	.5000	.7499	.249	58	.015	10.57	-.041	
K420	5624	.5000	.7498	.251	58	.015	10.83	-.076	
K3560	5722	.4999	.7500	.255	59	.015	17.80	-.060	
K3560	5724	.4999	.7504	.247	58	.015	18.10	-.098	
K3560	5726	.5000	.7504	.251	58	.015	18.30	No data	Bad graph
K3560	5728	.4999	.7505	.260	57	.015	16.31	-.040	
K3560	5729	.4999	.7511	.259	57	.015	17.16	-.035	
K3560	5730	.5000	.7507	.249	58	.015	18.11	+.036	

Table B5

Hughes Christensen

RESULTS

HUGHES CHRISTENSEN
Palmqvist Crack Length Measurements

Sample Code	Total Crack Length (μm)						
	NPL HV30	HCC HV30 on NPL polish			HCC HV30 on HCC polish		
	Indent	Indent 1	Indent 2	Indent 3	Indent 1	Indent 2	Indent 3
B1	492	496	502	511	503	498	493
B2	437	432	464	436	415	415	429
H1*	539	539	528	533	542	536	530
H2*	460	437	426	426	403	406	426
H3*	186	189	178	183	172	192	206
TCM10	621	618	618	598	584	553	499
K313*	443	457	460	451	432	426	378
K420*	353	310	322	299	257	305	231
	NPL HV30	HCC HV100 on NPL polish			HCC HV100 on HCC polish		
K3560*	121	248	192	228	228	284	257
	* grades for which corresponding short rod (SR) samples were supplied.						

Comments or observations

Wilson Tukon tester used to apply 30 Kgf test load. Wilson Rockwell tester used to apply 100 Kgf load.

Note: Sample B1 has two “bad” indentations which were not used. See sample container for diagram.

Details of the in-house polishing routine

As received pieces were polished by hand on a 40 μm diamond “dimple” pad. This was followed by 30 μm diamond lapping film, 3 μm diamond lapping film, and 0.5 μm film.

Details of measurement of the crack lengths
i.e. imaging technique (microscope, electron microscope) magnification, image analyser.

Cracks were measured using an eyepiece filar on our Tukon tester. Stage micrometer made by Buehler was used to calibrate the eyepiece filar and 32× objective.

Note: Diagonal measurements for Vickers hardness values were done using image analysis system on Buehler microhardness tester.
(Did not think of using this until after crack lengths were already done by the other method.)

HUGHES CHRISTENSEN

Additional Information

Sample Code	Average	Vickers	Palmqvist
	Total crack, µm	HV30	K_{IC}, MN $m^{-3/2}$
B1	499	1779	9.0
B2	432	1574	9.1
H1	535	1741	8.6
H2	426	1548	9.1
H3	187	1290	12.5
TCM10	584	1580	7.8
K313	435	1767	9.6
K420	297	1568	10.9
HV100			
	240	993	17.7

Note: K_{IC} calculated from the following formula
$K_{IC} = W_K = A\ (HV)^{1/2}\ W_G{}^{1/2}$
A = 0.0028
HV = Vickers hardness value in N/mm^2
W_G = P/T in N/mm
P = Load in Newtons
T = Total crack length in mm

HUGHES CHRISTENSEN

Vickers Hardness Indentations

Sample	Indentation Diagonal (µm)							Average	
	1	2	3	4	5	6	7		
	NPL indent	NPL Polish			HCC Polish				
B1	177	173	177	177	178	179	180	µm	HV30
	176	174	176	176	177	177	179	177	1778
B2	185	188	190	188	188	188	190	µm	HV30
	184	188	190	188	187	190	188	188	1574
H1	178	178	176	178	178	180	179	µm	HV30
	180	180	177	178	180	181	179	179	1741
H2	190	190	187	190	191	190	192	µm	HV30
	189	187	187	190	190	191	190	190	1548
H3	207	206	208	207	208	208	210	µm	HV30
	206	206	208	208	208	210	207	208	1290
TCM10	187	188	187	188	188	188	185	µm	HV30
	187	189	189	189	189	188	185	188	1580
K313	181	182	184	180	173	172	168	µm	HV30
	181	183	181	179	175	174	171	177	1767
K420	197	193	194	197	170	182	187	µm	HV30
	197	197	190	199	166	185	183	188	1568
K3560	243	432	429	427	427	438	437	µm	HV100
	243	437	429	430	433	435	430	432	993
								µm	HV30
								239	978

NPL indent	NPL polish	HCC polish
1785	1806	1749
1634	1563	1565
1736	1759	1726
1549	1565	1530
1304	1296	1279
1591	1568	1588
1698	1688	1876
1433	1463	1739
978	1000	987

Notes: NPL Indent = HCC measurement of NPL indentation on NPL polished side.
NPL Polish = HCC measurement of HCC indentation on NPL polished side.
HCC Polish = HCC measurement of HCC indentation on HCC polished side.

VK420 - Short Rod Results

											Hardness				
	Density	Hc	Sigma*	Def	P	T	K_{DL}	p	K_{ICSR}	Cc	Hra				
Number	g/cm^3	Oe	emu/g	emu/g	in	in	MPa √m	factor	MPa √m	Dim	1	2	3	4	Ave
5414	12.3780	140	13.00	12.36	0.750	0.500	11.18	0.000	10.36	0.927	91.45	91.35	91.35	91.50	91.4
5615	12.3763	140	13.09	12.45	0.749	0.500	11.28	-0.025	10.24	0.931	91.35	91.35	91.40	91.45	91.4
5616	12.3774	138	13.12	12.48	0.749	0.500	10.99	-0.070	9.68	0.948	91.30	91.40	91.40	91.30	91.4
5617	12.3770	139	13.14	12.50	0.749	0.500	11.16	-0.037	10.21	0.950	91.35	91.35	91.25	91.35	91.3
5621	12.3737	139	13.09	12.45	0.750	0.500	11.06	-0.019	10.38	0.957	91.40	91.50	91.45	91.35	91.5
Average	12.38	139					11.13		10.18						91.4
Std Dev	0.002	1					0.11		0.29						0.1
95% Cl	0.002	1					0.14		0.35						0.1
Max	12.38	140					11.28		10.38						91.475
Min	12.37	138					10.99		9.68						91.3
Range	0.004	2					0.29		0.70						0.2
Count	5	5					5		5						5

VK3560 - Short Rod Results

Grade: VK3560														
	Density	Hc	Sigma*	Def	P	T	K_{DL}	p	K_{ICSR}	Cc	Hra			
Number	g/cm^3	Oe	emu/g	emu/g	in	in	MPa √m	factor	MPa √	Dim	1	2	3	Ave
5744	14.3714	61.3	14.37	13.67	0.749	0.500	18.51	-0.050	17.09	0.972	85.8	86.0	85.8	85.9
5746	14.3808	61.7	14.43	13.73	0.749	0.500	18.27	-0.038	16.99	0.967	85.8	86.0	86.1	86.0
5743	14.3780	61.7	14.38	13.68	0.749	0.500	18.49	0.047	18.75	0.969	85.7	85.7	85.6	85.7
5751	14.3742	61.4	14.41	13.71	0.749	0.500	18.47	-0.012	17.73	0.972	86.0	86.2	85.9	86.0
5740	14.3749	61.5	14.43	13.73	0.749	0.500	18.10	-0.015	16.43	0.922	85.9	86.0	85.9	85.9
5741	14.3783	61.5	14.39	13.69	0.750	0.500	18.20	-0.036	17.06	0.972	85.8	86.0	85.9	85.9
Average	14.38	62					18.34		17.34					85.9
Std Dev	0.00	0					0.17		0.80					0.1
95% Cl	0.00	0					0.18		0.84					0.1
Max	14.38	61.7					18.51		18.75					86.0
Min	14.37	61.3					18.10		16.43					85.7
Range	0.01	0.4					0.41		2.32					0.4
Count	6	6					6		6					6

VK313 - Short Rod Results

Grade: VK313															
	Density	Hc	Sigma*	Def	P	T	K_{DL}	p	K_{ICSR}	Cc	Hra				
Number	g/cm^3	Oe	emu/g	emu/g	in	in	MPa √m	factor	MPa √m	Dim	1	2	3	4	Ave
5474	14.8066	299.2	9.39	8.92	0.750	0.500	9.60	?	?	0.967	92.80	92.70	92.70	92.70	92.7
5464	14.8052	299.9	9.41	8.94	0.750	0.500	9.50	-0.048	8.37	0.925	92.75	92.65	92.75	92.75	92.7
5475	14.8087	297.3	9.39	8.92	0.750	0.499	9.65	0.043	9.61	0.955	92.80	92.60	92.65	92.75	92.7
5470	14.8017	299.5	9.40	8.93	0.750	0.500	9.42	-0.071	8.26	0.944	92.75	92.70	92.80	92.75	92.8
5469	14.8106	297,8	9.38	8.91	0.750	0.499	9.50	-0.068	8.16	0.921	92.65	92.70	92.75	92.85	92.7
5478	14.8111	300.1	9.42	8.95	0.749	0.500	9.40	0.000	8.61	0.916	92.80	92.90	92.75	92.85	92.8
Average	14.81	299					9.51		8.60						92.7
Std Dev	0.00	1					0.10		0.59						0.0
95% Cl	0.00	1					0.10		0.73						0.0
Max	14.81	300.1					9.65		9.61						92.8
Min	14.80	297.3					9.40		8.16						92.7
Range	0.01	2.8					0.25		1.45						0.1
Count	6	6					6		5						6

VH1 - Short Rod Results

Grade: VH1															
	Density	Hc	Sigma*	Def	P	T	K_{DL}	p	K_{ICSR}	Cc	Hra				
Number	g/cm^3	Oe	emu/g	emu/g	in	in	MPa √m	factor	MPa √m	Dim	1	2	3	4	Ave
4515	14.7797	390.9	8.61	8.17	0.747	0.499	8.60	-0.067	7.64	0.952	93.30	93.35	93.35	93.40	93.4
4514	14.7746	387.4	8.69	8.25	0.743	0.499	8.25	-0.059	7.48	0.963	93.90	93.35	93.55	93.70	93.6
4494	14.7714	388.1	8.65	8.21	0.750	0.499	8.63	-0.132	7.21	0.962	92.80	93.20	93.25		93.1
4513	14.7709	389.5	8.64	8.20	0.750	0.500	8.41	0.000	7.96	0.946	93.40	93.20	93.10	93.60	93.3
4512	14.7774	391.6	8.63	8.19	0.741	0.498	8.41	-0.118	7.21	0.972	92.65	93.00	93.25	93.30	93.1
Average	14.77	390					8.46		7.50						93.3
Std Dev	0.00	2					0.16		0.32						0.2
95% Cl	0.00	2					0.19		0.39						0.3
Max	14.78	391.6					8.63		7.96						93.625
Min	14.77	387.4					8.25		7.21						93.1
Range	0.01	4.2					0.38		0.75						0.6
Count	5	5					5		5						5

VH2 - Short Rod Results

Short Rods																
Grade: VH2																
	Density	Hc	Sigma*	Def	P	T	K_{DL}	p	K_{ICSR}		Cc	Hra				
Number	g/cm³	Oe	emu/g	emu/g	in	in	MPa √m	factor	MPa √m		Dim	1	2	3	4	Ave
4570	14.5006	247.5	14.63	13.92	0.745	0.498	12.50	-0.361	invalid	7.75	0.970	92.50	92.25	92.00	91.95	92.2
4567	14.5035	252.5	14.44	13.74	0.741	0.500	11.81	-0.324	invalid	7.81	0.978	92.20	91.80	92.25	92.25	92.1
4569	14.4906	252.7	14.41	13.71	0.745	0.500	12.08	-0.357	invalid	7.44	0.958	91.60	91.55	91.95	92.00	91.8
4568	14.4853	246.2	14.70	13.99	0.743	0.499	11.60	-0.281	invalid	8.14	0.976	91.90	92.05	91.85	92.15	92.0
4566	14.5048	249.2	14.59	13.88	0.739	0.497	12.08	-0.255	invalid	8.53	0.947	91.55	92.15	91.80	91.95	91.9
Average	14.50	250					12.01			7.93						92.0
Std Dev	0.01	3					0.34									0.2
95% Cl	0.01	4					0.42									0.2
Max	14.50	252.7					12.50									92.175
Min	14.49	246.2					11.60									91.8
Range	0.02	6.5					0.90									0.4
Count	5	5					5									5

VH3 - Short Rod Results

Grade: VH3															
	Density	Hc	Sigma*	Def	P	T	K_{DL}	p	K_{ICER}	Cc	Hra				
Number	g/cm^3	Oe	emu/g	emu/g	in	in	MPa √m	factor	MPa √m	Dim	1	2	3	4	Ave
4751	14.1905	179.6	20.28	19.32	0.738	0.500	11.37	-0.089	10.03	0.968	89.80	89.30	89.80	89.70	89.7
4748	14.1868	177.9	20.35	19.39	0.741	0.500	11.16	-0.032	10.43	0.965	89.80	89.50	89.70	89.95	89.7
4752	14.1879	181.8	20.16	19.21	0.739	0.500	11.15	-0.057	10.11	0.962	90.00	90.05	90.00	89.85	90.0
4749	14.2017	192.1	19.81	18.87	0.743	0.500	10.80	-0.038	10.03	0.966	90.10	90.20	90.20	90.15	90.2
4760	14.1861	179.0	20.32	19.36	0.738	0.499	10.86	-0.025	10.33	0.976	89.85	89.80	89.85	89.60	89.8
Average	14.19	182					11.07		10.19						89.9
Std Dev	0.01	6					0.24		0.18						0.2
95% Cl	0.01	7					0.29		0.23						0.3
Max	14.20	192.1					11.37		10.43						90.163
Min	14.19	177.9					10.80		10.03						89.7
Range	0.02	14.2					0.57		0.39						0.5
Count	5	5					5		5						5

Table B6

Baildonit

Results

Baildonit
Palmqvist Crack Length Measurements

Sample Code	NPL HV30 Indent	Total Crack Length, μm HV30 indents made into NPL polished surface			Total Crack Length, μm HV30 indents made into surface polished by participant		
	Indent 1	Indent 1	Indent 2	Indent 3	Indent 1	Indent 2	Indent 3
B1	434	494	480	473	469	441	462
B2	406	420	424	431	399	406	396
H1	483	490	518	532	483	494	480
H2	431	413	434	424	322	340	343
H3	154	193	172	168	182	172	186
TCM 10	606	606	582	630	515	536	529
K313	462	427	466	431	434	413	417
K420	315	319	329	308	308	294	301
	NPL HV100 indents	HV100 indents made into NPL polished surface			HV100 indents made into surface polished by participant		
K3560	-	-	-	-	-	-	-

Comments or observations

We have not computed the sum of crack length for sample K3560 because:

- on a polished surface of sample prepared by NPL we had observed unclear cracks
- on a polished surface of sample prepared by our laboratory we had observed cracks not only at corners of indentation.

Details of the in-house polishing routine

1. Grinding: from a sintered surface should be ground off at least 0.5 mm but not more than 1.0 mm – using a diamond disc.
2. Polishing: using beech wood and diamond with a grain size smaller than 0.5 μm.
3. Heating: 1.5 hour at 900 °C – for avoidance of stresses which can arise during grinding and polishing.
4. Performing the Vickers indentations according to ISO 3878.

Details of measurement of the crack lengths
i.e. imaging technique (microscope, electron microscope) magnification, image analyser.

Using a microscope: magnification ×200.

Table B7

Universitat Politècnica de Catalunya (UPC)

Results

Universitat Politècnica de Catalunya, UPC
Palmqvist Crack Length Measurements

Sample Code	NPL HV30 Indent		UPC HV30 Indents	Total Crack Length, µm HV30 indents made into NPL polished surface			Total Crack Length, µm HV30 indents made into UPC polished surface		
	Hardness	Crack Length µm	Hardness	Indent 1	Indent 2	Indent 3	Indent 1	Indent 2	Indent 3
B1	1830	492	1680	500	496	512	492	500	496
B2	1610	448	1550	428	444	408	436	420	404
H1	1760	568	1760	556	564	568	548	560	564
H2	1550	420	1550	396	392	384	380	388	384
H3	1280	172	1330	132	136	144	120	128	132
TCM 10	1550	648	1550	736	744	764	760	724	740
K313	1680	412	1680	456	436	448	456	408	436
K420	1430	288	1430	288	276	264	284	296	260
	NPL HV100 indents		UPC HV100 indents	HV100 indents made into NPL polished surface			HV100 indents made into surface polished by participant		
K3560	900	188	900	192	180	164	156	164	176

Comments or observations

Ratio between half diagonal (indentation) and mean Palmqvist crack length is below unity for all materials studied, except for grades H3, K420 and K3560. Additionally, in the latter grade there are some corners of the HV100 indent where cracks do not appear.

Details of the in-house polishing routine

	Time (min)	Disc	Medium
Step 1 (grinding)	15	TBW grid-abrade magnetic diamond disc (68 µm)	Water
Step 2 (grinding)	15	TBW grid-abrade magnetic diamond disc (30 µm)	Water
Step 3 (polishing)	20	Wood disc	30 µm diamond paste + water
Step 4 (polishing	20	Wood disc	6 µm diamond paste + water
Step 5 (polishing)	20	Wood disc	3 µm diamond paste + water

Details of measurement of the crack lengths
i.e. imaging technique (microscope, electron microscope) magnification, image analyser.

Conventional optical microscopy at 10× and 40× magnifications

Universitat Politècnica de Catalunya, UPC
SEPB Measurements

Sample	K_{Ic} ($MN\ m^{-1.5}$)	HV30 NPL value	Sample	K_{Ic} ($MN\ m^{-1.5}$)	HV30 NPL value
B1	9.22 ± 0.13	1778	H3	12.03 ± 0.14	1364
B2	9.99 ± 0.13	1626	K313	9.27 ± 0.09	1726
H1	8.90 ± 0.18	1810	K420	11.67 ± 0.07	1486
H2	9.96 ± 0.20	1592	K3560	18.93 ± 0.11	996

Table B8

National Physical Laboratory

Results

National Physical Laboratory
Palmqvist Crack Length Measurements

Sample Code	NPL HV30 Indent	Total Crack Length, µm		
		HV30 indents made into NPL polished surface		
		Indent 1	Indent 2	Indent 3
B1				
B2		**See Table B1**	**See Table B1**	**See Table B1**
H1				
H2				
H3				
TCM 10				
K313				
K420				
	NPL HV100 indents	HV100 indents made into NPL polished surface		
K3560		**See Table B1**	**See Table B1**	**See Table B1**

Comments or observations

All samples annealed at 800 °C for 1h in vacuum. Slow cooling (18h) to room temperature.

Details of the in-house polishing routine

Samples mounted in phenolic resin and prepared on Abramatic polishing machine.

Grinding using 220 µm fixed diamond abrasive to remove 200 µm of surface. Grinding for 1 minute, 65 µm fixed diamond abrasive. Grinding for 1 minute, 20 µm fixed diamond abrasive. Lapping for 10 minutes using 6 µm diamond on Petrodisc-M wheel. Polishing for 5 minutes using 6 µm diamond abrasive, DP-Pan cloth. Then 3 µm diamond abrasive, DP-Pan cloth. Finally, 1 µm diamond abrasive, DP-Pan cloth. Samples annealed as above.

Details of measurement of the crack lengths

Indent diagonals and crack length measured using KS400 image analysis system. Images obtained using ProgRes camera with a resolution of 1000 by 700 pixels. Leica DMXRE microscope used at magnification of ×500 or ×1000 where appropriate.

Table B9

BAM

Results

BAM
Palmqvist Crack Length Measurements

Sample Code	NPL+ HV30 Indent	Total Crack Length, µm HV30 indents made into NPL polished surface			Total Crack Length, µm HV30 indent made into surface polished by participant		
		Indent 1	Indent 2	Indent 3	Indent 1	Indent 2	Indent 3
B1	508	502	506	505	488	486	486
B2	440	452	442	422	406	422	426
H1	547	543	531	534	420	526	514
H2	468	434	429	408	369	405	417
H3	164	168	170	162	150	160	150
TCM 10	634	592	622	633	570	626	641
K313	460	433	426	455	448	453	443
K420	349	307	308	320	273	237	287
	NPL HV100 indent	HV100 indents made into NPL polished surface			HV100 indent crack lengths made in surface polished by participant		
K3560	291						

Comments or observations

+ Only samples TCM10 and K3560 fulfilled requirements for plane parallelism. All other results of NPL HV30 indents were considered as invalid.
- Before placing 3 HV30 indents into the polished face the opposite side of the polished surface were finished by grinding to receive coplanar faces.

Details of the in-house polishing routine

The specimens were prepared in the usual way for ceramics, with PM5 Auto Lap Precision Lapping and Polishing Machine with PP5 GT Polishing Jig from Logitech.

The final stage of polishing using SF-1 suspension has proven especially suitable and allows the production of a polished surface about completely free of defects (cracks and chip-out).

Grinding and Polishing

Step	Surface	Force (gf)	Speed (rpm)	Abrasive grain size	Time (h)
1	Cast-Iron	4426	23	Al_2O_3 20 µm	8
2	Cast-Iron	4426	23	Al_2O_3 9 µm	3
3	Cast-Iron	4426	23	Al_2O_3 3 µm	2
4	MD SUBA X	1500	49	SF1 (SiO_2 0.04 µm)	3
5	Polyurethan	2370	49	SF1 (SiO_2 0.04 µm)	3
6	MD SUBA X	2370	49	SF1 (SiO_2 0.04 µm)	12

Details of measurement of the crack lengths
i.e. imaging technique (microscope, electron microscope) magnification, image analyser.

Crack lengths were measured optically at a magnification of ×500 using microscope Axiotech 25HD.

BAM
Individual Palmqvist Measurements

Surface polished by NPL (following grinding on opposite side to achieve adequate flatness)

Sample No		HV30	Total crack length mm	W_G Nm m^{-1}	W_K MN $m^{-3/2}$
B1 4337	Ind 1	1775	0.502	586	8.95
	Ind 2	1785	0.506	582	8.94
	Ind 3	1745	0.505	583	8.84
	Mean value			**584**	**8.91**
	St. Dev			**2**	**0.05**
B2 4391	Ind 1	1600	0.452	651	8.95
	Ind 2	1590	0.442	666	9.02
	Ind 3	1610	0.422	697	9.29
	Mean value			**671**	**9.09**
	St. Dev.			**19**	**0.15**
H1 4438	Ind 1	1795	0.543	542	8.65
	Ind 2	1795	0.531	554	8.75
	Ind 3	1815	0.534	551	8.77
	Mean value			**549**	**8.72**
	St. Dev.			**5**	**0.05**
H2 4667	Ind 1	1590	0.434	678	9.11
	Ind 2	1585	0.429	686	9.14
	Ind 3	1600	0.408	721	9.42
	Mean value			**695**	**9.22**
	St. Dev.			**19**	**0.14**
H3 4695	Ind 1	1365	0.168	1752	13.56
	Ind 2	1350	0.170	1731	13.41
	Ind 3	1320	0.162	1817	13.58
	Mean value			**1766**	**13.52**
	St. Dev.			**36**	**0.08**
TCM10 5011	Ind 1	1615	0.592	497	7.86
	Ind 2	1610	0.622	473	7.65
	Ind 3	1615	0.633	465	7.60
	Mean value			**478**	**7.70**
	St. Dev.			**14**	**0.11**
K313 5458	Ind 1	1680	0.433	680	9.37
	Ind 2	1680	0.426	691	9.45
	Ind 3	1690	0.455	647	9.17
	Mean value			**672**	**9.33**
	St. Dev.			**19**	**0.12**
K420 5579	Ind 1	1500	0.307	959	10.52
	Ind 2	1480	0.308	956	10.43
	Ind 3	1470	0.320	920	10.20
	Mean value			**945**	**10.38**
	St. Dev.			**18**	**0.13**

BAM
Individual Palmqvist Measurements

Surface polished by BAM

Sample No		HV30	Total crack length mm	W_G Nm m^{-1}	W_K MN $m^{-3/2}$
B1 4337	Ind 1	1755	0.488	603	9.02
	Ind 2	1805	0.486	606	9.17
	Ind 3	1795	0.486	606	9.14
	Mean value			**605**	**9.11**
	St. Dev			**1**	**0.06**
B2 4391	Ind 1	1615	0.406	725	9.49
	Ind 2	1610	0.422	697	9.29
	Ind 3	1615	0.426	691	9.26
	Mean value			**704**	**9.35**
	St. Dev.			**15**	**0.10**
H1 4438	Ind 1	1830	0.420	701	9.93
	Ind 2	1805	0.526	560	8.81
	Ind 3	1795	0.514	573	8.89
	Mean value			**611**	**9.21**
	St. Dev.			**64**	**0.51**
H2 4667	Ind 1	1625	0.369	798	9.98
	Ind 2	1615	0.405	727	9.50
	Ind 3	1610	0.417	706	9.35
	Mean value			**743**	**9.61**
	St. Dev.			**39**	**0.27**
H3 4695	Ind 1	1335	0.150	1962	14.19
	Ind 2	1325	0.160	1839	13.69
	Ind 3	1335	0.150	1962	14.19
	Mean value			**1921**	**14.03**
	St. Dev.			**58**	**0.24**
TCM10 5011	Ind 1	1615	0.570	516	8.01
	Ind 2	1645	0.626	470	7.71
	Ind 3	1645	0.641	459	7.62
	Mean value			**482**	**7.78**
	St. Dev.			**25**	**0.17**
K313 5458	Ind 1	1725	0.448	657	9.34
	Ind 2	1755	0.453	650	9.36
	Ind 3	1715	0.443	664	9.36
	Mean value			**657**	**9.35**
	St. Dev.			**6**	**0.01**
K420 5579	Ind 1	1500	0.273	1078	11.15
	Ind 2	1495	0.237	1242	11.95
	Ind 3	1485	0.287	1025	10.82
	Mean value			**1115**	**11.31**
	St. Dev.			**92**	**0.47**

BAM
Results of Fracture Toughness – SEVNB Test

Additional Code	Sample ID	Thickness mm	Width mm	Notch radius μm	F_{max} N	Notch depth mm	K_{Ic} MPa $m^{1/2}$
B1	4364	2.88	5.75	23.6	705	1.412	15.98
	4365	2.88	5.75	21.8	749	1.398	16.86
	4366	2.88	5.75	17.7	681	1.381	15.21
	4367	2.89	5.76	13.1	568	1.384	12.63
	4368	2.89	5.76	9.8	542	1.400	12.13*
	4369	2.89	5.75	6.5	534	1.386	11.91*
B2	4422	2.87	5.75	17.5	533	1.440	12.27
	4423	2.88	5.75	14.3	538	1.452	12.40
	4424	2.88	5.75	12.8	459	1.483	10.75
	4425	2.88	5.75	9.1	488	1.448	11.25*
	4426	2.88	5.75	11.3	504	1.446	11.61
H1	4467	2.90	5.76	10.4	488	1.417	10.96
	4470	2.90	5.75	4.9	506	1.467	11.68*
	4473	2.91	5.75	6.1	510	1.431	11.54*
	4476	2.92	5.75	6.5	491	1.417	11.01*
	4479	2.91	5.74	6.4	517	1.429	11.73*
	4480	2.91	5.75	8.5	588	1.430	13.29*
H2	4625	2.90	5.77	14.1	642	1.439	14.51
	4626	2.90	5.76	7.5	600	1.442	13.63*
	4627	2.90	5.76	8.5	598	1.439	13.57*
	4628	2.91	5.76	6.0	460	1.444	10.44*
	4629	2.91	5.75	6.2	462	1.448	10.53*
	4630	2.91	5.76	8.9	585	1.464	13.38
H3	4725	2.91	5.75	4.9	574	1.357	12.56*
	4726	2.91	5.76	6.6	615	1.399	13.66*
	4727	2.91	5.77	7.5	664	1.396	14.67*
	4728	2.91	5.76	6.6	694	1.388	15.35*
	4729	2.90	5.76	8.1	681	1.386	15.09*
	4730	2.90	5.76	8.4	683	1.367	15.00*
K313	5401	3.02	6.01	12.9	561	1.646	12.20
	5402	3.02	5.99	10.8	527	1.648	11.56
	5403	3.02	5.99	8.9	482	1.621	10.45*
	5404	3.02	6.00	5.6	471	1.643	10.27*
	5405	3.02	5.99	4.4	481	1.637	10.49*
	5406	3.02	5.99	20.7	557	1.647	12.21
K420	5530	3.02	5.99	6.1	522	1.441	10.48*
	5531	3.02	6.00	4.7	508	1.509	10.46*
	5532	3.02	5.99	3.4	502	1.525	10.46*
	5533	3.02	5.99	3.8	507	1.519	10.52*
	5534	3.02	5.99	3.2	511	1.521	10.61*
	5535	3.02	6.00	4.0	514	1.509	10.59*
K3560	5686	3.01	6.00	16.1	816	1.440	16.37
	5687	3.02	6.00	12.2	813	1.456	16.36
	5688	3.01	6.01	11.6	819	1.458	16.49
	5689	3.02	5.99	6.7	819	1.442	16.44*
	5690	3.00	6.00	7.0	826	1.431	16.56*

* Considered valid by BAM: i.e. notch radius is small enough.