

**A NATIONAL MEASUREMENT
GOOD PRACTICE GUIDE**

No. 20

**Mechanical Tests
for Hardmetals**

Measurement Good Practice Guide No. 20

Mechanical Tests for Hardmetals

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Abstract: Methods for the measurement of the mechanical properties of hardmetals at ambient temperatures are discussed. Current standards are assessed together with good measurement practice, including both routine procedures and research requirements. A comprehensive "property review" is included together with a summary of key microstructural parameters and their measurement. Comments are given on the discriminability of different mechanical test methods.

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Mechanical Tests for Hardmetals

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Executive Summary

Hardmetals, cermets and other sintered hard materials are used in a very diverse range of applications. They face such a wide array of stress/temperature/environmental combinations that a simple correlation of one-parameter tests and service performance is generally not viable. Also, the cost of in-the-field testing for every possible new development of structure or processing route is generally prohibitive. Consequently, laboratory benchmark testing is vital, in particular for:

- **Characterising structural effects**
- **Comparing materials**
- **Quality control**
- **Quantification of properties**

Industry and supporting research organisations now use a wide variety of tests, some of which are standardised and of long standing pedigree, for example, density measurements. Others which are thought to be relevant are fairly well understood, but have some limitations, such as bend testing (transverse rupture tests). Finally there are research or in-house methods, such as impact and corrosion testing, which are generally not covered by widely agreed procedures.

This guide examines the different types of laboratory - scale tests. It does not cover performance testing such as that used by the larger companies and users in cutting or rock drilling trials. It excludes comment on techniques for characterising raw materials and concentrates on tests for sintered products.

Because of the importance to hardmetal industry of quality control testing the main body of the guide concentrates on four ambient temperature mechanical tests:

- **Hardness**
- **Abrasion**
- **Bend strength**
- **Fracture toughness**

Detailed discussion of these key properties is preceded by a "property review" that succinctly assesses most of the important properties that control performance. However, many of these tests and properties are not yet well understood, and these are covered more briefly in a separate section to the main body of the guide. The key microstructural parameters are also reviewed as microstructure is so important in controlling materials properties and thus influences the interpretation of the results of test methods. Thus, in summary, the guide comprises sections on

- **Structure**
- **Property review**
- **Ambient mechanical tests**
- **Brief guide to additional tests**

Also included is a section on test method discriminability, since an understanding of this aspect of testing allows users or manufacturers to differentiate between materials types.

1 Hardmetal Test Methods

1.1 Standards

Hardmetal test methods and parameters of interest can be classified as follows in Table 1.

Table 1 - Classification of Test Methods

<i>Structure</i> Composition* Microstructure* Grain or phase size Magnetic properties Coercivity* Moment/Saturation Porosity* Surface integrity	<i>Environmental</i> Corrosion Erosion
<i>Physical</i> Density* Thermal expansion Thermal diffusivity Electrical resistivity	<i>Mechanical</i> Hardness* Hot hardness Strength Bend* Compression* Fatigue Abrasion* Impact Residual stress Toughness Indentation toughness Plane strain toughness Stiffness* Creep

The list is dominated by mechanical requirements, which clearly reflects hardmetals' importance in general engineering applications. The methods for which there are published standards in place are starred (*) and listed in Table 2. ISO or ASTM standards are commonly used in Europe and N America. In Japan, many indigenous standards have also been developed. This list however only refers to ISO and ASTM documents.

Probably the most important aspect of testing is the characterisation of structure from which all else follows. For industry the primary need is the production of products with consistent properties and for this a hierarchy of test methods are used, mostly concerned with the use of the ISO standards.

Table 2 - Hardmetal Test Method Status

Wide usage:			
Microstructure		ISO 4499	
Porosity / C content		ISO 4505	
Magnetic coercivity		ISO 3326	
Density		ISO 3369	
Hardness (Vickers)		ISO 3878	
Hardness (Rockwell)		ISO 3738 pts 1 and 2; ASTM B294	
More restricted usage:			
Bend strength		ISO 3227	
Compression strength		ISO 4506	
Abrasion		ASTM B611	
Fracture toughness		No standard	
Palmqvist toughness		No standard	
Co content / composition		ISO 4503; ISO 4883; ISO 3909	
Stiffness (Young's modulus)		EN 23312	
Research/Grade Development:			
<i>Mechanical</i>		<i>Physical</i>	
Corrosion	No standard	Expansion	No standard
Erosion	No standard	Diffusivity	No standard
Fatigue	No standard	Resistivity	No standard
Impact	No standard	Grain size	No standard
Hot hardness	No standard		

The larger companies provide representative data on grades from many of these tests and the “Directory and Handbook on Hardmetals and Hard Materials” by K.J.A. Brookes (International Carbide Data) published at regular intervals gives comparative tables. The Handbook also contains useful comparative property graphs, usually plotted against cobalt content. However, in many cases this gives rise to considerable scatter because the WC grain size is omitted. The measurement of WC grain size is not yet covered by an international standard. The ISO committee plans to modify ISO 4499 to introduce a standard by 2009. Those properties which do

not show much scatter for a particular value of Co content, such as density, thermal expansion coefficient or Young's modulus, are clearly insensitive to changes in WC grain size.

It is significant that many of the properties which have a strong influence on the performance of hardmetal products, such as corrosion, fatigue, impact and high temperature strength and toughness, are often measured but not by standardised test methods. Equally of relevance is the cost of performing these tests. The preparation of suitable test-pieces for measuring the mechanical properties of hardmetals and similar hard materials is always expensive because of the necessity to use diamond grinding/lapping procedures and the requirement for stress-free surface preparation. At present, as industrial practice stands, probably 5-10% of the production costs are those related to measurement requirements in the finished product. Consequently testing for properties over and above those already provided by current standards is undertaken only when driven by specific customer/producer agreements supported by good technical arguments which demonstrate a clear relationship between the property of interest and performance. This does not preclude individual companies developing new grades and products by using as wide an array of testing methods as possible in a laboratory environment in order to characterise fully a new product in the development stage.

Thus, it can be summarised that the use of hardmetals is a mature technology. Industry is currently well served by a range of baseline established standards which, if properly followed with good attention to correct quality procedures, will ensure consistent products, although even with these standards, it is essential to have a full understanding of the effects of test method variables. For example, the effects of test-piece preparation grinding stresses can change apparent bend strength by up to 100%. The most important gaps are probably a standard for magnetic moment measurements, the requirement for agreed methods to quantify WC grain size, and improved techniques for benchmark laboratory testing which are more closely related to performance than the existing mechanical tests of hardness and bend strength.

1.2 Discriminability

Choosing an appropriate mechanical test method for characterising the properties of WC/Co hardmetals either for quality control or for use in design is not straightforward because in practice the application areas for these materials are extremely wide, for example, metal cutting, wear parts, seals, punches, dies and forcing tools.

Good test methods are those which can clearly enable the user or manufacturer to discriminate between different materials. The ability of tests to discriminate can be assessed by comparing the range of typical values of a test for a given range of materials with the measurement uncertainty for that test.

An attempt has been made [1] to assess the ability of a number of standard and non-standard tests such as hardness, abrasion wear, bend strength and fracture toughness, to discriminate effectively between different materials, by evaluating the range of likely results, R , the uncertainty of measurement, U , and the number of intervals of discrimination over the range of these tests.

These are summarised in Table 3 and Fig 1.

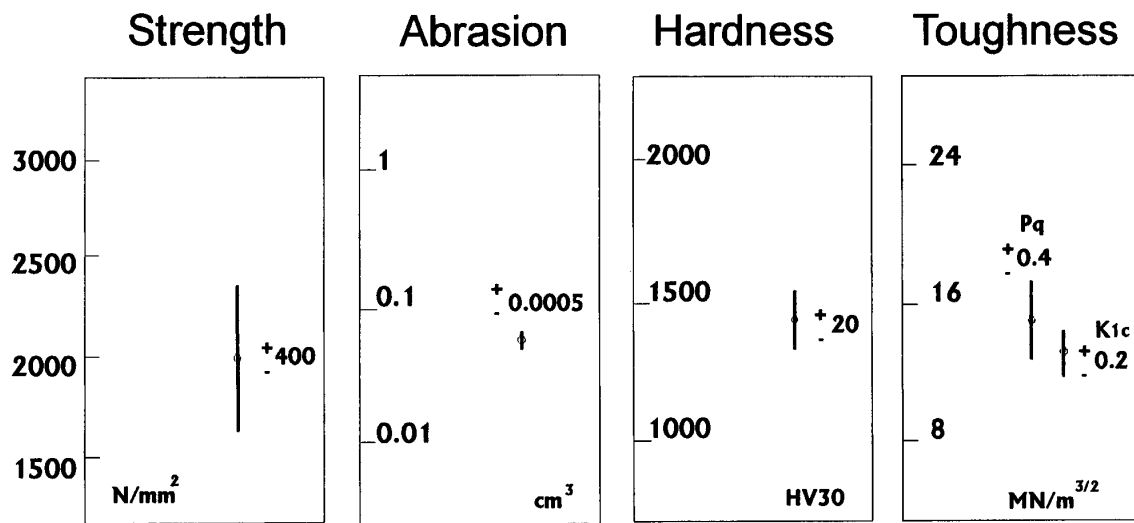


Figure 1 Discriminability of test methods for hardmetals.

Table 3 - Test Discrimination

	Bend strength N mm^{-2}	Abrasion resistance, loss in volume, cm^3	Hardness HV30	Toughness, $\text{MN m}^{-3/2}$	
				Palmqvist, W_K	Plane strain, K_{Ic}
Range, R	1500 (1500-3000)	100 (0.01-1)	1000 (1000-2000)	8 (8-16)	16 (8-24)
Uncertainty	± 400	± 0.0005	± 20	± 0.4	± 0.2
Uncertainty (fraction of R)	$R/4$	$R/2000$	$R/50$	$R/20$	$R/40$
Intervals*	4	2000	50	20	40

*Discriminability measure

The concept of discriminability is defined in Table 3 as the number of intervals across the measurement range. The more intervals the better the test is able to differentiate between different materials.

On this basis it can be seen that the bend test is a poor discriminator of materials whereas the abrasion test is extremely good. Hardness and toughness tests are considerably better than the bend test. Plane strain toughness tests are more discriminating than Palmqvist tests. These issues are discussed in more detail in Section 3.

2. Property review

Hardmetals have unique combinations of properties which make them one of the most useful tool materials produced by man. Many of their properties can be flexibly varied by straightforward changes in composition (*e.g.* volume fraction binder phase or hard phase grain size). The range of properties can be summarised as follows:

- **COMPRESSIVE STRENGTH**

Compressive strength values of 4 - 8 kN mm⁻² make them the strongest commercial materials available, as indicated by their use as anvils for diamond manufacture and in hot rolls for metallic materials.

- **TENSILE STRENGTH**

Less than compressive strength, typically 2 - 3 kN mm⁻², but still much stronger than most other materials. Processing route is very important. Hot Isostatic Pressing (HIP) or SINTERHIP is required for the highest values of strength. SINTERHIP is a combination of sintering and HIPing in one process.

- **HARDNESS**

Can be varied between 1000 HV and 2000 HV by changing cobalt content and WC grain size. Like many other properties of hardmetal there is the flexibility to design specific hardness values by changing composition. As hard at the higher end as most hard ceramic materials, yet much tougher.

- **TOUGHNESS**

Difficult to measure quantitatively as there is no agreed standard. Typical values of plane strain fracture toughness lie in the range 7 - 25 MN m^{-3/2}. These values are much higher than for similar hard materials like ceramics which have toughnesses of 2 - 8 MN m^{-3/2}.

- **FATIGUE STRENGTH**

Few data are available to judge properties but this is likely to be very good as this property usually correlates with strength.

- **HIGH TEMPERATURE STRENGTH**

Excellent, although decreases steadily with increasing temperature. Sensitive to choice of hard phase. Cubic carbide additions such as (Ta, Nb)C and (Ta, Ti)C increase strength significantly.

- LOW TEMPERATURES

Hardmetals retain a good combination of properties at temperatures down to as low as that of liquid nitrogen. Hardness increases by about 20 - 30% with a concomitant decrease in toughness.

- IMPACT STRENGTH

Not well understood, probably related to tensile strength and fracture toughness. No standard test method available.

- ABRASION RESISTANCE

In severe abrasion environments it is the most wear resistant material available. This is because it has a unique combination of strength, hardness and toughness compared with other hard materials.

- STIFFNESS

It is one of the stiffest materials known, after diamond, with a Young's modulus about three times that of steel and six times that of aluminium.

- DENSITY

Hardmetals are very dense if they contain significant amounts of WC, up to 15 Mg m^{-3} is possible. Lower densities can be tailored by using higher Co contents or cubic carbides and carbonitrides for the hard phase, down to $4 - 5 \text{ Mg m}^{-3}$.

The high density endows hardmetal with a very satisfying feel when handling products but if specific properties (*i.e.* properties per unit weight) are required it is a disadvantage.

- SURFACE FINISH

Hardmetals can take a superb mirror finish, a consequence of their defect-free, fine-scale, structure. They have a deep grey lustre that is particularly attractive.

- COEFFICIENT OF FRICTION

Hardmetals have a relatively low coefficient of friction, about 0.2, in unlubricated tests in contact with steel, as compared with typical steel against steel values of about 0.4. There is little systematic data of the effects of surface roughness, composition variations and temperature effects.

- COATINGS

Hardmetals easily take a variety of coatings, *e.g.* electroless, electroplate, chemical, physical, vapour or spray deposited. Both wear resistance and cosmetic coatings of many types are cheap and effective.

- SHAPE

Because hardmetals are manufactured by a powder metallurgy route there are few restrictions

on shape and mass production technologies are possible. However, post-manufacture shape changes can be expensive if extensive diamond grinding is required.

- **DIMENSIONAL STABILITY**

Their use as high quality slip gauges indicates exceptional dimensional stability, provided that grinding stresses are stress-relieved. The microstructures are inherently stable since they are formed by an equilibrium process at very high temperatures and are cooled to room temperature relatively slowly.

- **EDGE RETENTION AND SHARPNESS**

The fine scale of the microstructure and the high strength of the hardmetal enables very sharp edges and corners to be manufactured which have myriad uses in cutting and shaping processes (paper, wood and glass, for example).

- **CORROSION RESISTANCE**

Corrosion resistance in non-acids is excellent for all hardmetals. However, if a cobalt binder phase is used they can be prone to dissolution in even mildly acidic environments. This can be mitigated by using Ni-base or alloy binder-phases which are inherently more corrosion resistant.

- **JOINING**

Brazing to metallic components is relatively straightforward as hardmetals are readily wetted if clean, but care has to be taken to minimise thermal residual stresses because the thermal expansion coefficient is quite low, about half that of high speed steel. Welding is not possible but adhesives can be used in some circumstances if care is taken with surface preparation.

- **THERMAL EXPANSION AND CONDUCTIVITY**

The thermal conductivity of standard WC/Co hardmetals is about twice as high as that of high speed steel with values in the range of $100 \pm 20 \text{ Wm}^{-1} \text{ K}^{-1}$. Thermal expansion values are typically $5 - 6 \times 10^{-6} \text{ K}^{-1}$ for WC/Co hardmetals. Both conductivity and expansion can be tailored by changing the volume fraction of binder-phase and type of carbide/carbonitride hard phase.

- **ELECTRICAL AND MAGNETIC PROPERTIES**

Hardmetals usually have transition metal binder phases and consequently are slightly ferromagnetic, with coercive force values between $5 - 30 \text{ kA m}^{-1}$, depending on the binder-phase content. Non magnetic hardmetals can be made using Ni or alloy binder-phases as an alternative to the more usual cobalt. Electrical conductivity is good with values of resistivity in the range $160 - 220 \text{ n}\Omega\text{m}$. Higher values of up to $1000 \text{ n}\Omega\text{m}$ can be produced if cubic carbides such as TiC are substituted for the WC.

3 Structure

3.1 Main features

WC/Co hardmetals are generally manufactured to comprise two phases WC and Co. WC/Co is a pseudo-binary section in a three component system (W, C and Co). If the processing conditions are such that the carbon balance is either too high or too low then other phases are present in the structure *i.e.* graphite if the C is high and eta-phase (an M_6C or $M_{12}C$, where M is generally Co_nW_n) if the C content is low. The structure of two phase WC/Co hardmetals, Fig. 2, is defined by three key parameters:

- Grain size of the WC phase:
WC is a stoichiometric phase, generally with no internal features, although occasional small defects (sometimes C or Co inclusions) are present inside a grain.

ISO 4499 will shortly (2009) provide a standard for measurement. The mean linear intercept technique is recommended [2]. Coercivity measurements provide a useful NDE technique to estimate grain size.

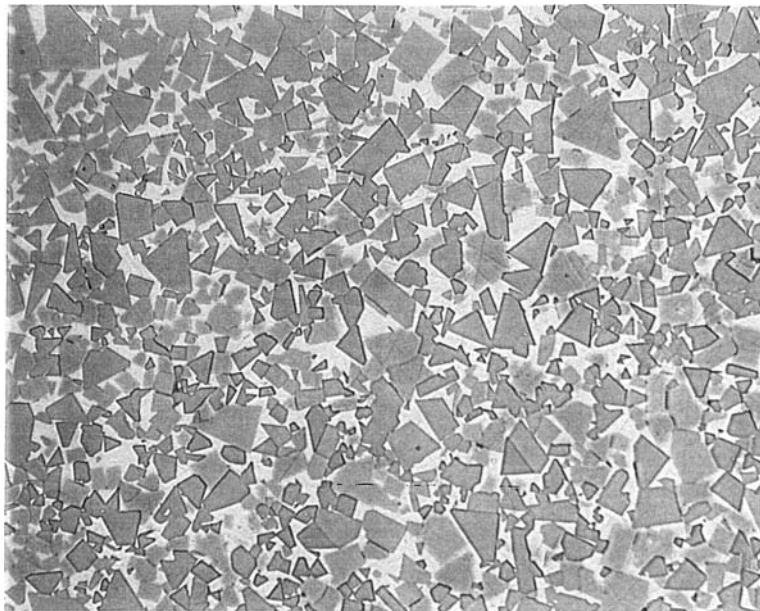


Figure 2 Structure of a typical WC/Co hardmetal; WC grains dark-grey, Co-light grey.

- Volume fraction of the Co binder-phase:
Weight fraction is used to specify hardmetal composition.

Volume fraction can be calculated from the weight fraction using density values or measured directly by metallography.

- Composition of the Co binder-phase

The binder-phase is a Co-W-C alloy and the alloy content of the Co matrix can be varied.

The composition (i.e. W and C content) can be calculated from magnetic and lattice parameter measurements.

3.2 Composition and Phase Size

The composition of WC/Co hardmetals is conventionally specified in terms of how much Co is present in wt% and a nominal figure for the grain size of the WC. The amount of alloying elements (W and C) present in the Co binder-phase can, in principle, be determined by measurements of a combination of properties such as magnetic moment (saturation) and lattice parameter. In practice, however, this is made difficult both by the solution of small amounts of Fe, Ni or other elements in the Co and by the requirement to measure accurately the Co content of the hardmetal.

3.2.1 WC Grain size

Measurement of the WC grain size (d_{WC}) is not standardised, although ISO 4499 will shortly provide a method based on the intercept technique. The required methods require care, and this is particularly the case for very fine grained materials. The results of grain size measurement methods frequently depend on the method of preparation and examination of samples [2-4].

The use of linear intercept distributions (Heyn method) is a common method by which the grain size and distribution of the phases in composite hard materials is measured. The distribution is generally plotted on cumulative probability paper (to linearise the plot) with the abscissa plotted as log (intercept distance). The distribution is obtained by counting individual intercept lengths (at least 200) on a randomly placed line laid across a polished and etched microstructural image. Area counting, by measurement of the number of grains per unit area, (Jeffries method) can also be used. This can be performed using optical microscopes for some coarser-grained WC hardmetals but for many materials the grain size is too fine to be resolved optically and so scanning electron microscopes (SEM) must be used [3,4]. In very fine grained hardmetals the average grain size can be very small, less than 0.5 μm for the arithmetic mean linear intercept, and good quality images are difficult to obtain in conventional SEMs. However, it has been shown that a high resolution instrument which uses a field emission electron beam can provide acceptable images [5]. Recent research indicates that orientation imaging techniques based on electron backscatter diffraction (EBSD) and the use of standardised methods will soon provide a more accurate method [6,7].

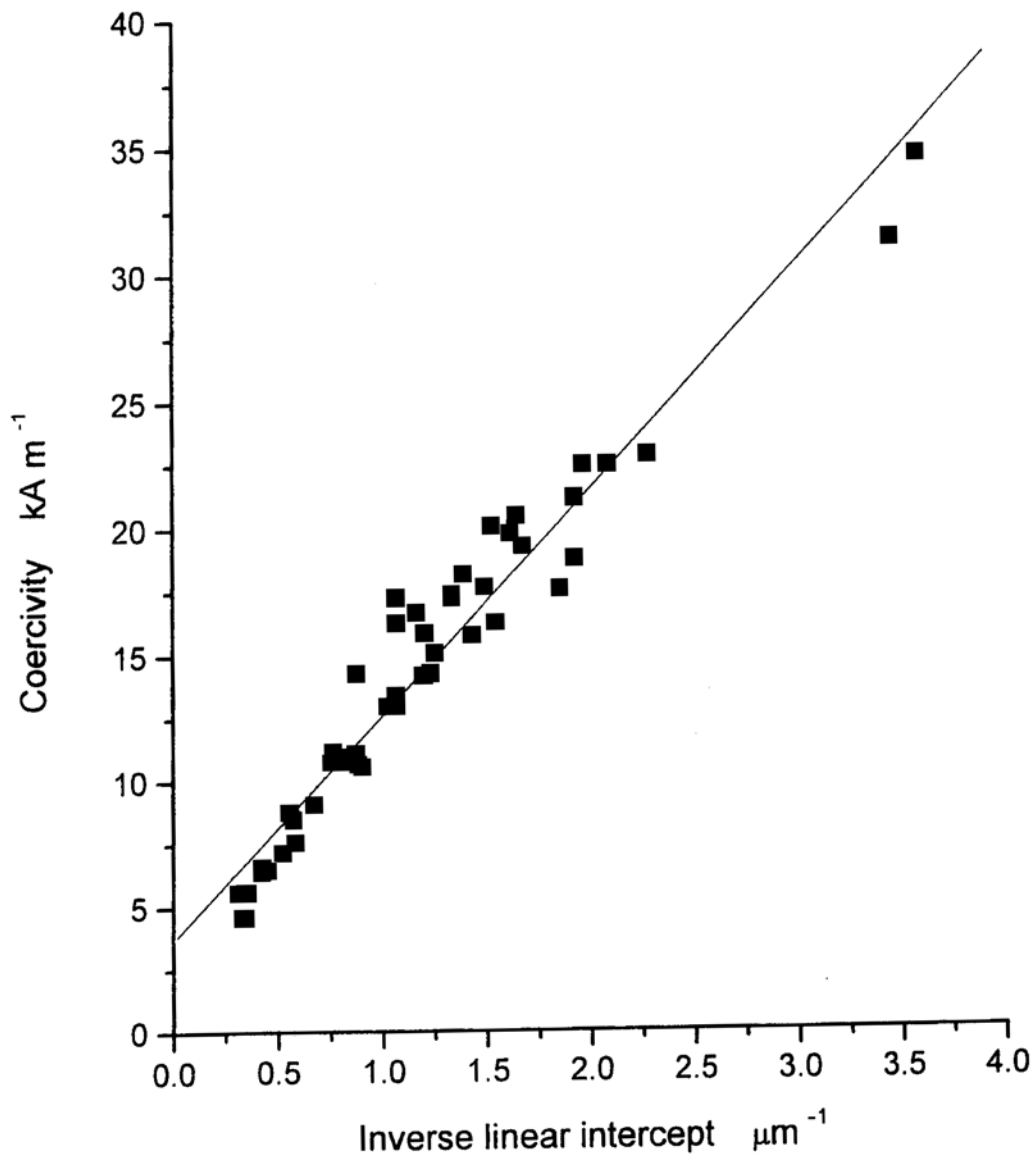


Figure 3 Coercivity-inverse grain size correlation.

The most widely used non-destructive method for the assessment of grain size measures coercive force, which is covered by a standard, ISO 3326. The coercive force required to demagnetise a WC/Co hardmetal is primarily related to the Co/WC interphase area, because magnetic domain walls are pinned by these phase boundaries. The Co/WC interphase area is inversely related to the WC grain size. For smaller values of the latter, the interphase area increases and consequently so does the coercivity. However, coercivity also changes with variations in Co composition and this should be allowed for in empirical equations relating WC grain size to coercivity [8]. Furthermore, difficulties can arise when trying to correlate coercivity with grain size due to effects of differences in cooling rate from the sintering temperature. This results in changes to the composition of the binder-phase which can have a significant effect on coercivity. For example, in low carbon content hardmetals it is possible to precipitate Co_3W in the binder-phase on ageing of the hardmetal after accelerated cooling. This procedure can result in very significant changes in coercivity. Additionally, there may be effects on coercivity due to the

phase boundaries between the hexagonal and face centred cubic phases in individual binder regions. Also, deformed hardmetals show a significant increase in coercivity due to deformation of the binder-phase, including the creation of phase boundaries, twins and stacking faults, which can be at least partially removed on subsequent annealing. Finally, the role of WC/WC contiguity must also be acknowledged since it can be seen that two materials of different contiguity but similar grain size will have different interphase areas and hence different coercivities. Contiguity is a measure of the proportion of each WC grain that is in contact with adjacent contiguous WC grains [5]. Fig. 3 shows a typical correlation between WC inverse grain size and coercivity for a range of WC/6 wt% Co hardmetals.

3.2.2 Co phase size

Measurement of the size (d_{Co}) of the Co binder-phase regions is also not standardised. It can be measured directly using the intercept procedure on a micrograph by counting a sufficient number of Co regions. The average is called the Co mean free path. Alternatively it can be calculated from a measurement of the WC grain size and knowledge of the Co volume fraction from the following empirical relation [5]:

$$d_{Co} = d_{WC} (0.1 + 2V_{Co})$$

Accurate measurement of d_{Co} is difficult because the Co regions are much smaller than the WC grains. A scanning electron microscope is required to image the structure. Frequently magnifications of greater than x20k are needed to image the Co regions, which are often smaller than 0.1 μm , particularly in the fine and very fine grained hardmetals.

3.2.3 Composition (Co-W-C binder-phase)

Measurements of lattice parameter can be made with a X-ray diffractometer using $\text{CuK}\alpha$ radiation on the surface of test-pieces which have been etched to remove WC in Murakami's reagent for a half hour at 60 °C. The inter-planar spacings of the {200} and {311} planes of fcc cobalt are measured together with the spacings for the {1011} and {1122} planes of tungsten carbide powder scattered on the surface. Measurement of the interplanar spacings of the tungsten carbide are carried out as an internal standard.

The results of the lattice parameter measurements show that the range of values in the two- phase WC/Co region is between about 0.3545 and 0.3570 nm. The measurement uncertainty is about ± 0.0002 nm.

Work at NPL [9] has shown that the lattice parameter of the fcc phase can be expressed as:

$$a = a_0 + Qm_w + Rm_c \quad (2)$$

where a_0 , Q and R are constants with values of 0.3548 nm, 0.00036 and 0.0012 respectively. The atomic percentages of W and C are given by m_w and m_c respectively.

If the lattice parameter of the binder-phase with a known tungsten content can be measured, then data on the variation of fcc cobalt lattice parameter with separate additions of tungsten and carbon can be used to estimate the carbon content by subtracting the calculated change corresponding to a given tungsten content from the total change in lattice parameter.

The W content can be calculated from measurements of magnetic moment (see section 3.3.1).

3.3 Density

- ***Density measurements are covered by a standard, ISO 3369. It is a practical quality control measure that can be obtained accurately and with little difficulty.***

Density is a very important quality control measurement which is routinely measured to enable manufacturing companies to establish a core database for every grade produced. Most hard materials manufactured by a liquid-phase sintering route essentially have zero porosity and the density measurement thus provides information on the structure of two or multi-phase materials.

Density in essentially pore-free materials is dependent on the volume fraction of each phase present and their composition. In WC/Co hardmetals for a given Co content the volume is also sensitive to the total C content, because as the C content is lowered the Co binder phase increasingly contains more W in solution. The WC grain size does not affect density.

The measurement is covered by a standard, ISO 3369. In general, careful measurements can be made fairly repeatably with a resolution of at least 1 part in 500. This makes the test particularly discriminating. For example, the densities of WC/6wt% Co and a WC/9wt% Co hardmetals are about 15 and 14.7 Mg m⁻³ respectively. The density of pure WC is 15.65 Mg m⁻³ (from lattice parameter measurements) and that of pure Co is 8.95 Mg m⁻³. Thus, for a composite of 6 wt% Co/94% WC the theoretical density is 14.98 Mg m⁻³. Fig. 4 shows theoretical values of density for WC/Co hardmetals for two compositions of the Co binder phase; pure Co and a Co-10 wt% W alloy.

In practice, the effects of additional elements such as Fe or Ni in the binder and TiC and/or Ta(Nb)C as an additional hard phase must also be taken into account if an exact correlation between structure and density is attempted.

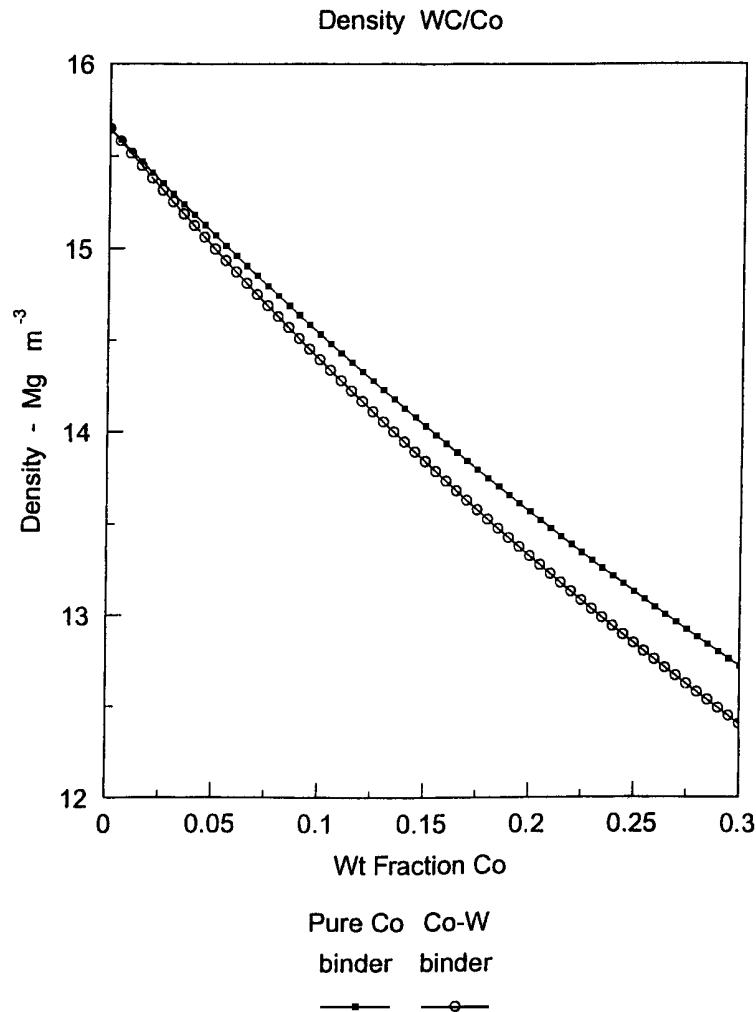


Figure 4 Density and Co weight fraction.

3.4 Magnetic measurements

3.4.1 Introduction

Magnetic measurements are vital for controlling the consistency of hardmetal products. They comprise:

- **Moment (saturation)**
Used for quality control and to estimate Co-W-C binder-phase composition
- **Coercivity**
Used for quality control and to estimate WC grain size

The use of non-destructive methods to assess consistency of structure is very important because hardmetals have microstructures on a very fine scale which are difficult to inspect. Also, they are strong and not easy to test mechanically. However, hardmetals contain cobalt which is ferromagnetic. Consequently measurements of magnetic properties are widely used by industry to assess consistency. Both coercive force and magnetic saturation (moments) are measured for this purpose. If interpreted correctly, they can be linked to the cobalt composition and hence overall properties and performance.

3.4.2 Magnetic saturation (moment)

Tungsten carbide/cobalt hardmetals generally contain 5-20 wt% cobalt as a binder-phase. The cobalt binder-phase contains both tungsten and carbon in solution. The amount of tungsten dissolved in the cobalt binder-phase can be assessed by measurement of the magnetic saturation or magnetic moment of the hardmetal because the saturation value of cobalt decreases linearly [9,10] with the addition of tungsten and is not affected by the amount of carbon in solution. The term ‘magnetic saturation’ is an abbreviation for the saturation (or maximum) value of magnetic induction that can be obtained in a test-piece in a strong magnetic field.

There is no standard for magnetic saturation (moment) measurements on hardmetals.

The values obtained give information which is related to the composition of the binder-phase. For proper interpretation it is necessary also to have additional data on the chemical composition, such as for example wt% of Co and wt% of additional contaminant elements such as Ni and Fe in WC based hardmetals.

Definitions

I_s	=	Magnetic moment/unit volume (intensity of magnetisation/unit volume), tesla
J_s	=	$4\pi I_s$, Saturation induction (polarisation), tesla
σ	=	Magnetic moment/unit weight (intensity of magnetisation/unit wt), tesla m ³ kg ⁻¹
σ	=	I_s/ρ where ρ is the density

Units

Magnetic moment	-	Wb m (Weber-metre)
Magnetic moment/unit volume	-	Wb m ⁻² (T, tesla)
Magnetic moment/unit mass	-	Wb m kg ⁻¹ or T m ³ kg ⁻¹

It is important not be confused between the saturation or moment values that can be quoted for hardmetals [11]:

- Often the value refers to the magnetic saturation or moment value of the hardmetal;
- Alternatively it can refer to the magnetic saturation or moment value of the Co binder-phase (Co-W-C) in the hardmetal;
- Magnetic moment is converted to magnetic saturation by 4π multiplication;
- Saturation values are sometimes expressed as a percentage of the maximum expected for a nominally pure Co binder phase.

The values can be expressed in several ways:

- as magnetic saturation of hardmetal or binder phase;

- as magnetic moment of hardmetal or binder phase;
- as percentage of the value that would be obtained for an essentially pure Co binder.

Tables 4 and 5 give examples of the different methods for expressing the magnetic moment data, in SI and pre-SI units.

In practical materials there is a range of magnetic saturation and moment values associated with a variation in the carbon content of the hardmetal, Fig. 5. The measurements in Fig. 5 were obtained with equipment at NPL that gives magnetic moment values smaller than theoretical (Tables 4 and 5), probably because the magnetising field is not strong enough to fully saturate the Co phase.

Table 4 - Typical ranges of magnetic saturation and moment values
SI units - $\mu\text{T m}^3 \text{kg}^{-1}$

	5 wt% Co	10 wt% Co	15 wt% Co
Hardmetal			
Magnetic saturation, $4\pi\sigma$	8.0 - 10.1	16.1 - 20.2	24.1 - 30.3
Magnetic moment, σ	0.64 - 0.80	1.28 - 1.61	1.92 - 2.41
Percentage saturation	80 - 100	80 - 100	80 - 100
Binder-phase			
Magnetic saturation, $4\pi\sigma$	160 - 202	160 - 202	160 - 202
Magnetic moment, σ	12.8 - 16.1	12.8 - 16.1	12.8 - 16.1
Percentage saturation	80 - 100	80 - 100	80 - 100

Table 5 - Typical ranges of magnetic saturation and moment values
pre-SI units - gauss $\text{cm}^3 \text{g}^{-1}$

	5 wt% Co	10 wt% Co	15 wt% Co
Hardmetal			
Magnetic saturation, $4\pi\sigma$	80-101	161-202	241-303
Magnetic moment, σ	6.4-8.0	12.8-16.1	19.2-24.1
Percentage saturation	80-100	80-100	80-100
Binder-phase			
Magnetic saturation, $4\pi\sigma$	1600-2020	1600-2020	1600-2020
Magnetic moment, σ	128-161	128-161	128-161
Percentage saturation	80-100	80-100	80-100

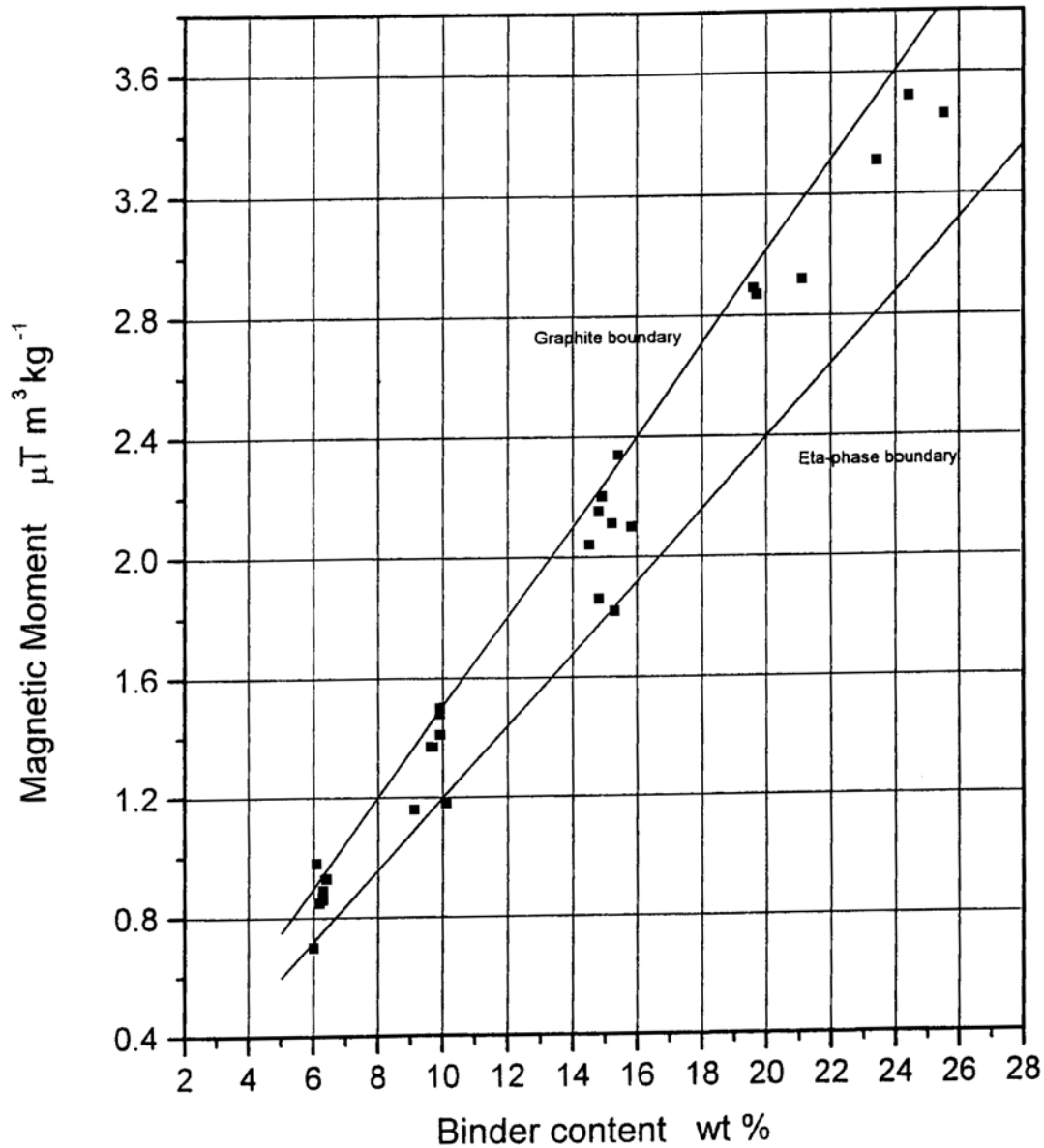


Figure 5 Magnetic moment of WC/Co hardmetals plotted against Co content.

In most hardmetals the binder contains W and C in solution. Presence of C does not affect the cobalt magnetic saturation and moment values, but W decreases them. The decrease can be represented by [11],

$$4\pi\sigma_B = 4\pi(\sigma_{Co} - 0.275m_W) \quad (3)$$

where $4\pi\sigma_{Co}$ is $202 \mu T m^3 kg^{-1}$ and m_W is the wt% W in the Co binder-phase.

When $4\pi\sigma_B$ attains a value of about $160 \mu T m^3 kg^{-1}$ eta-phase begins to form and so the range $202-160 \mu T m^3 kg^{-1}$ for the magnetic saturation of the cobalt phase defines the two-phase field in WC/Co hardmetals. This expression can be used to either calculate the W content of the binder-

phase from the magnetic moment or vice versa:

Thus, if a 6% Co hardmetal has a magnetic saturation value of $11 \mu\text{T m}^3 \text{ kg}^{-1}$

then $4\pi\sigma_B = 11/0.06 = 183.3 \mu\text{T m}^3 \text{ kg}^{-1}$

and therefore

$$m_W = \frac{202 - 183.3}{4 \times \pi \times 0.275} = 5.4 \text{ wt \%}.$$

The previous discussion has assumed that the binder-phase in WC/Co hardmetals is a Co-W-C alloy. In practice other elements can be present, particularly Fe, which can have an effect on the magnetic moment which makes precise calculations difficult.

For example, the equation relating the change in magnetic moment of the binder phase with W additions:

$$\sigma_B = \sigma_{Co} + A m_W \quad (4)$$

is probably acceptable for elements other than W with the values for A shown in Table 6 [12].

Table 6 - Magnetic moment coefficients

Element	A $\text{T kg}^{-1}/\text{wt\% addition}$
W	-0.28
Cr	-0.60
Fe	+0.13
Mo	-0.24
Ni	-0.11

It is probably reasonable to assume that in dilute Co-W-X alloys that the effects of different elements on A are additive. No data for V or Ta could be found in the literature. These are important elements which could be present in small amounts in the binder phase.

For example in a typical hardmetal the binder might contain about 2 wt% of Fe. The effect of Fe on magnetic moment is opposite to that for W at about half the strength. This leads to a correction of about +1% on the wt% W figure calculated for the magnetic moment, assuming a Co-W binder.

3.4.3 Magnetic coercivity measurements

There is a standard for coercivity measurements for hardmetals, ISO 3326.

The relationship between magnetic coercivity and arithmetic mean linear intercept was given in Fig. 3 (section 3.1.1), which shows a plot of the coercivity values against grain size for 6 wt% Co hardmetals [8]. These results can be characterised by a straight line given by:

$$K = a + b d_{WC}^{-1} \quad (5)$$

where K is the coercivity in kA m^{-1} , a and b are constants and d_{WC} is the WC arithmetic mean linear intercept in μm . The linear relation is consistent with the arguments given in the introduction for a physically based relation between d_{WC} and K .

The results in Fig. 3 showed more scatter than might be expected from the equipment used to make the coercivity measurements. It is possible that this scatter is due to a combination of several of the effects of microstructure on coercivity discussed in the introduction.

The linear regression fit for all the data did not give particularly good predictions for materials with grain sizes greater than about $2 \mu\text{m}$. Consequently a bilinear fit was performed with data for coercivities less than 12.5 kA m^{-1} treated separately from data from materials with $K > 12.5 \text{ kA m}^{-1}$. The resulting expressions were as follows:

$$\begin{aligned} \text{For } K < 12.5 \text{ kA m}^{-1} \quad K &= 179 + 10.9 d_{WC}^{-1} \\ \text{For } K > 12.5 \text{ kA m}^{-1} \quad K &= 6.71 + 7.39 d_{WC}^{-1} \end{aligned} \quad (7)$$

The initial work on 6 wt% Co hardmetals has recently been widened to include a more extensive range of Co contents from 6-25% [13]. The results were grouped into 6%, 10%, 15%, 20% and 25% Co batches, and plots of coercivity against inverse grain size gave the following fitted values for the coefficients (Table 7):

Table 7 - Coercivity - grain size coefficients (see eqn. 5)

wt% Co	a	b
6	1.74	10.61
10	2.10	8.63
15	1.89	6.55
20	1.94	5.14
25	2.85	3.53

Assuming a linear relation between the coefficients a and b and wt% Co allows an expression to be written for the variation of coercivity, K , with grain size, d for $6 < \text{Co} < 25$ wt% as follows:

$$K = (c_1 + d_1 W_{\text{Co}}) + (c_2 + d_2 W_{\text{Co}})/d \quad (8)$$

where W_{Co} is the wt% Co in the hardmetal and $c_1 = 1.44$, $c_2 = 12.47$, $d_1 = 0.04$ and $d_2 = -0.37$.

Expression (8) can be plotted as a property map, Fig. 6, bearing in mind the constraint that it has not been extensively validated for coercivity values greater than about 15 kA m^{-1} , *i.e.* equivalent to WC grain sizes (arithmetic linear intercept) less than about $0.8 \mu\text{m}$. Additional work is needed to evaluate the finer grained materials. A property map using Co wt% content as the variable can also be plotted from expression (8). This is shown in Fig. 7 for values of linear intercept WC grain size of $5 \mu\text{m}$, $2 \mu\text{m}$, $1 \mu\text{m}$, $0.75 \mu\text{m}$ and $0.5 \mu\text{m}$, respectively.

It is also necessary to be aware that the coercivity also varies with carbon content and W content of the binder-phase. The materials in the evaluation exercise contained a range of carbon contents and this has not been allowed for in the construction of the property maps. It has been assumed that the compositions are approximately in the centre of the two-phase field. An estimate of uncertainty due to this source showed [13] that the calculated size varied by about $\pm 20\%$ across the two-phase field, whereas the measured variation is probably $\pm 10\%$. The difference is thus probably due to variation in composition. Further work would be needed to refine expression (8) to allow for this effect.

A further aspect that is clear from inspecting the property maps is that the coercivity measurements are much less sensitive to changes in grain size as the mean value increases beyond about $3 \mu\text{m}$.

It is also possible that microstructural instabilities in low C hardmetals or residual stresses could affect measurements of coercivity through changes in internal strain and composition. These effects contribute added uncertainty to the correlation between grain size and coercivity and need to be systematically examined.

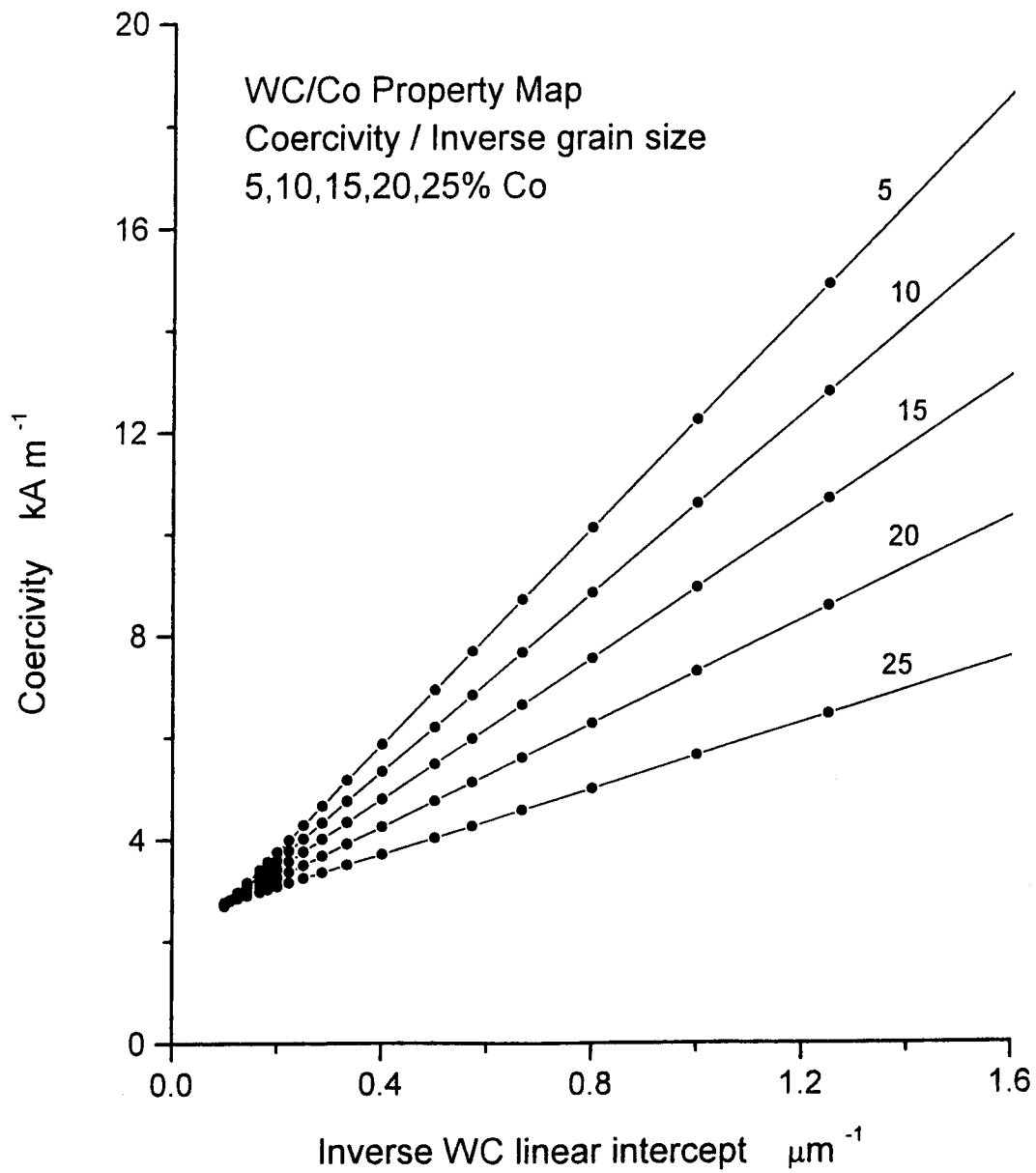


Figure 6 Coercivity/inverse grain size property map.

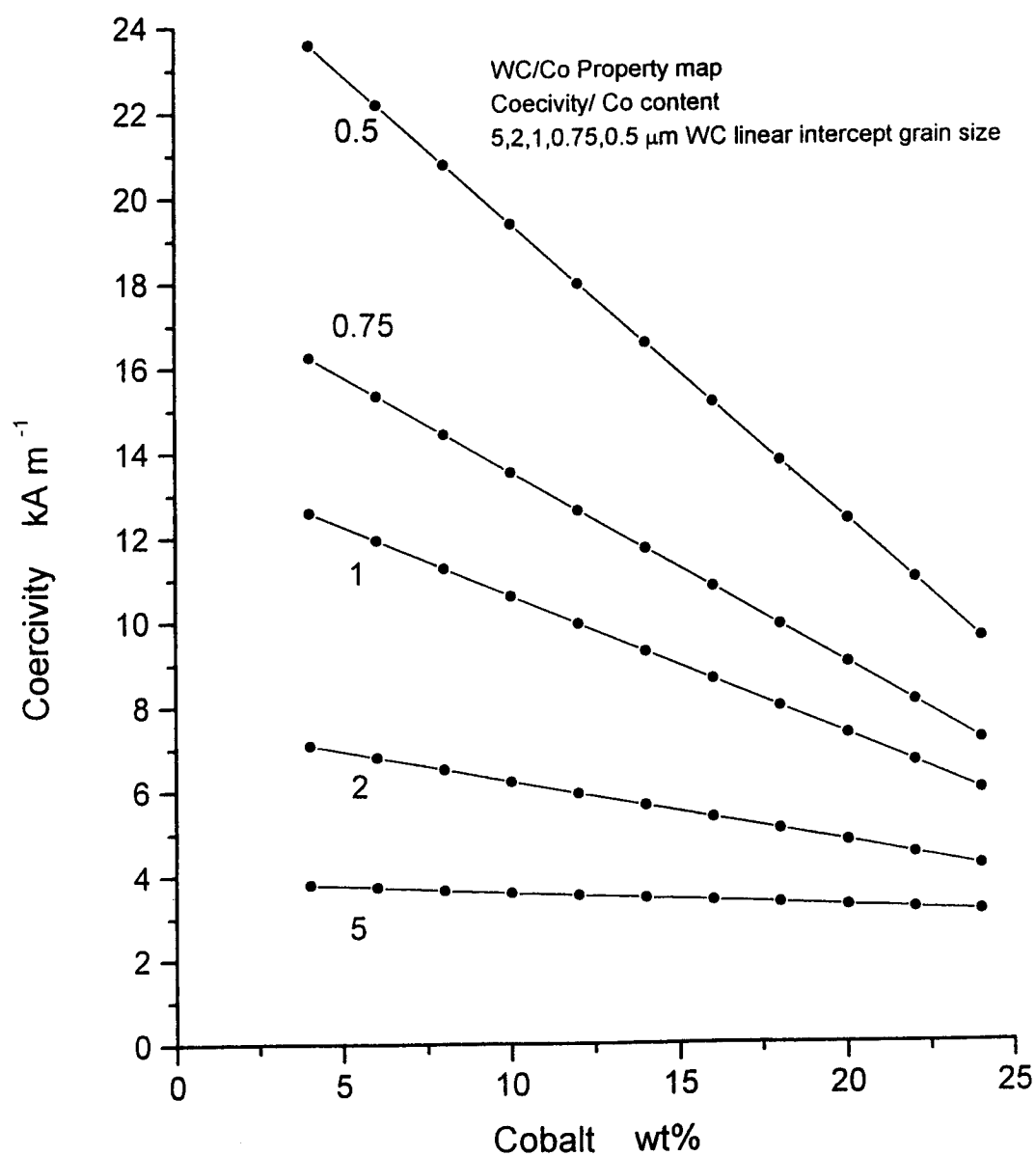


Figure 7 Coercivity/Co content property map.

4 Mechanical tests at ambient temperature

4.1 Hardness

Hardmetals, as their name implies, are very hard materials with hardness values generally of greater than 1000 HV30. The hardness test is, in principle, relatively simple to apply and is therefore widely used to characterise the strength of hardmetals. However, there are two methods in use around the world, based on Vickers and Rockwell indenters, and it is not straightforward to compare results from the two methods. Also, industrial requirements are driving the hardness of hardmetal products to values greater than 2000 HV30 and this is raising important issues of measurement for the relatively small indentation sizes that result from tests on these materials. Models have been developed to relate internal structure to hardness and their use has considerable implications regarding measurement methods. The models also allow "property maps" to be derived that relate structural parameters such as WC grain size and Co content to hardness. The maps allow hardness values of products to be specified by their manufacture from well characterised powders.

There are two standard methods for measurement of hardness:

- HV30 Vickers (Europe) ISO 3878
- HRA Rockwell A (Japan, N America) ISO 3738 pts 1 and 2; ASTM 3294

Full test details are given in the relevant standards. Rockwell A measures indentation depth under load (typically 60 kgf). Vickers measures indentation area after removal of load (typically 30 kgf).

Other laboratory methods are being increasingly investigated, largely based on the use of depth-sensing techniques, like the Rockwell A method but with more sensitivity on depth measurements and using the full load/displacement trace to measure hardness and other related parameters (*e.g.* modulus) rather than indentation depth. The Knoop test, using a rhomboidal pyramidal indenter, is sometimes used on hard brittle materials because there is less cracking associated with the indentation than in Vickers test. However, there is generally less damage to hardmetals compared with, say, ceramics, other than the single cracks emanating from indentation corners, so Knoop tests are not commonly reported for hardmetals.

Hardness tests are performed at NPL on polished and annealed (800 °C for 1 h in a vacuum) test-pieces using an accredited Vickers hardness machine with a test mass of 30 kg. The indentation diagonals are measured using a calibrated optical microscope at 515x. The estimated measurement uncertainty is $\pm 1 \mu\text{m}$ on a typical indentation diagonal of 200 μm . This

corresponds to about ± 20 pts on a typical hardness of 1400 HV30. Comparative studies using standard Vickers Hardness machines have shown that 40 pts is typical scatter to be expected from operator to operator variation. Test-piece surfaces should be perpendicular to the indenter axis to produce symmetrical indentations.

Hardness measurements on conventional hardmetals are reasonably independent of load in the range 5-50 kgf. However, at smaller loads and, especially below 1 kgf, the apparent hardness increases for reasons not yet fully explained - possibly due to differences in the extent of the plastic strain field and the precise details of the associated deformation mechanisms, or possibly due to inherent measurement limitations. This effect is larger for harder materials. Consequently, it is to be expected that hardmetals with HV30 > 1800 will show a strong dependence of hardness on load when small applied loads are used. Therefore, Vickers hardness measurements at loads greater than about 20 kgf should always be used for purposes of comparison. It has been recently recognised that reference materials, of a similar hardness to the required measurements, can be very useful for calibration purposes. A fine-grained HIPed hardmetal has been used by NIST in the USA as a reference material for HV1 measurements.

4.1.1 Test method comparison

Fig. 8 shows a comparison of Vickers and Rockwell A values for straight commercial WC/Co hardmetals from three sources. It is not possible to make conversions for different combinations of hardmetals and binder phases because the methods are intrinsically different. Manufacturers generally produce in-house comparison plots for different types of hardmetal.

4.1.2 High hardness hardmetals

It is difficult to make comparisons of hardness values on the newly-developing very fine grained hardmetals because of a number of factors:

- The use of a Rockwell indenter in USA and Japan, compared with predominantly a Vickers indenter in Europe;
- The occasional use of microhardness testing equipment (with applied loads < 1 kgf);
- Lack of a standard surface preparation method;
- Problems associated with the measurement of smaller indentations.

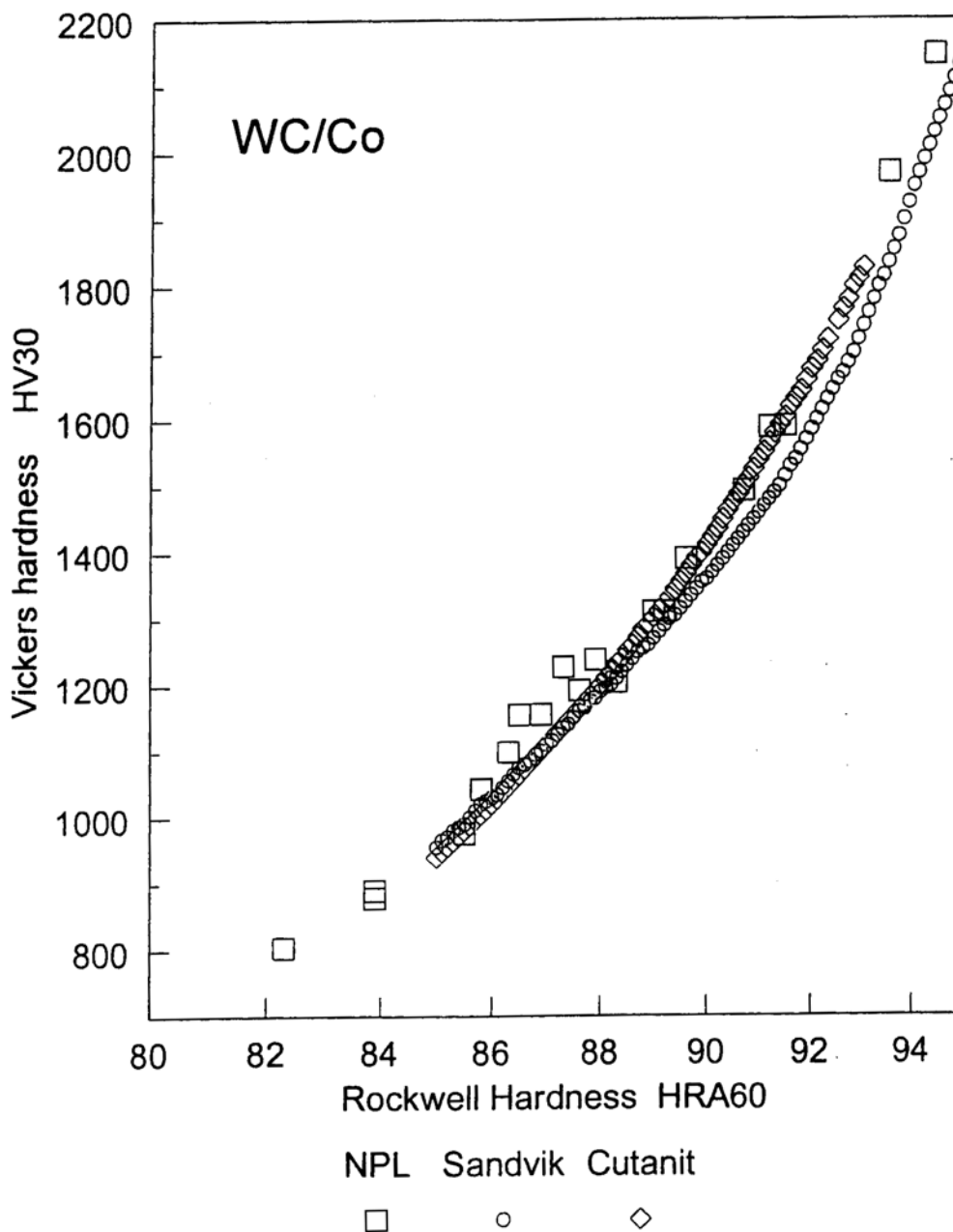


Figure 8 Comparison of Rockwell and Vickers Hardness Measurements on WC/Co.

There is no internationally agreed scale comparison for conventional materials between the Rockwell A and Vickers tests; consequently comparison of reported values for hardnesses above 1750 (HV30) and 93.0 (HRA) should be considered with care. ISO TC119/SC4 is currently developing a recommended comparison [14].

In Vickers tests the problem of measurement can be illustrated by reference to Table 8 which shows indentation diagonals in micrometres for a range of applied loads at different values of hardness. For loads of 0.5 kgf the changes in diagonals are very small, only about 3 μm in 20 μm

between 1900-2500 HV0.5. The uncertainty in measurement, that is typically about 1-2 μm , depends on the magnification at which the measurements are made [15]. Clearly better discrimination is obtained if a load of 30 kgf is used. Even so, at a practical level it is likely that an uncertainty of ± 100 will be associated with hardness values greater than 2000. There is a need for more accurate methods for measurement of the indentation, perhaps by using non-contacting scanning devices or image analysis which would minimise errors associated with human eye discrimination.

Table 8 - Indentation sizes

Hardness HV	Diagonal length, μm , at indentation force of:			
	0.5 kgf	2 kgf	10 kgf	30kgf
1600	24.1	48	108	186
1700	23.4	47	104	181
1800	22.7	45	101	176
1900	22.1	44	99	171
2000	21.5	43	96	167
2100	21.0	42	94	163
2200	20.5	41	92	159
2300	20.1	40	90	156
2400	19.7	39	88	152
2500	19.3	39	86	149

Finally, for very fine grained materials the method of surface preparation could be a source of uncertainty since the development of residual stresses by grinding and their effect on strength is influenced by grain size [16, 17]. It would be sensible to use the approach advocated for Palmqvist testing and anneal the surfaces before making the hardness measurements. However, a treatment of greater than 1200 °C for 1 h in a vacuum would probably be required for all materials to ensure that dislocation structures within the carbide phase are fully recovered. Further research is clearly needed.

4.1.3 Hardness Models and Measurements

The applicability of the Hall-Petch equation for hardness predictions:

$$H = E_1 + F_1 d_{\text{WC}}^{-1/2} \quad (9)$$

has been examined [4] for materials which are nominally 6 wt% Co (approximately 10 vol%). In Fig. 9 the hardness values have been plotted against the average value of the inverse of the square root of the arithmetic mean linear intercept size.

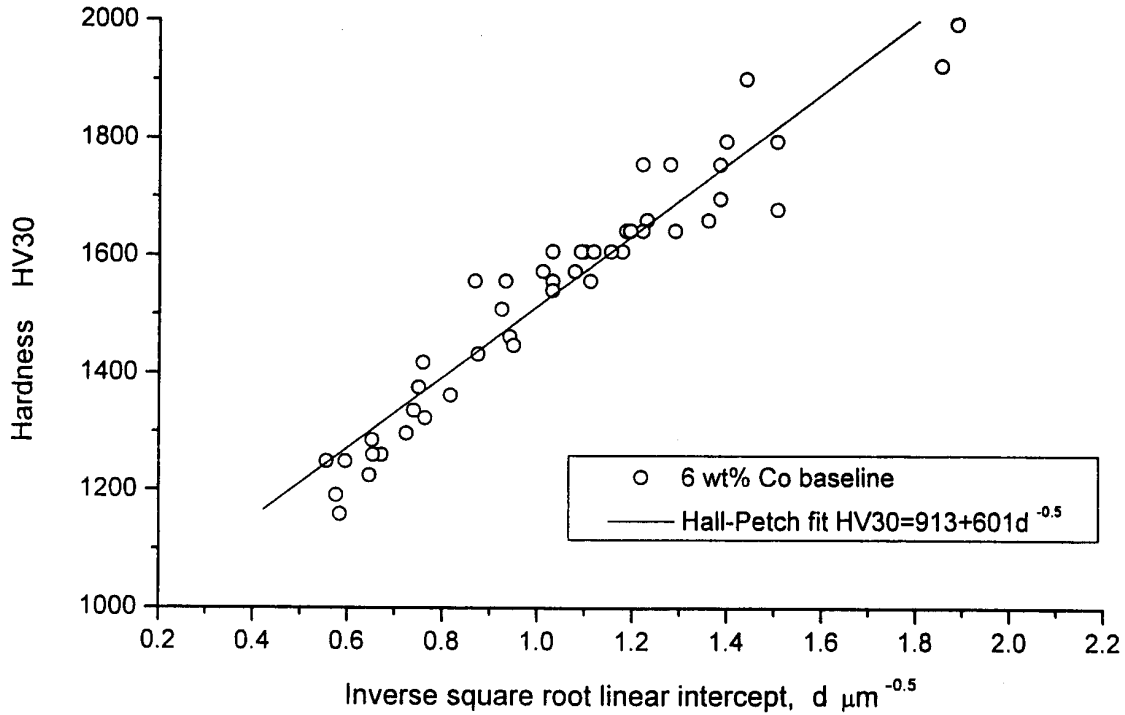


Figure 9 Hardness plotted against inverse square root of linear intercept WC grain size, with a linear data fit.

Least squares regression analysis was used to fit a linear plot to the data and the result is shown in Fig. 9. The values for the constants E_1 and F_1 are 913 and 601 respectively.

The experimental data extend to a value of about $1.9 \mu\text{m}^{-0.5}$ for the inverse square root of d_{WC} . This corresponds to a value of about $0.3 \mu\text{m}$ for the arithmetic mean linear intercept size. In order to predict what hardness might be obtained in materials with grain sizes less than this value it is necessary to extrapolate the fits shown in Fig. 9 to a value of arithmetic mean linear intercept corresponding to about $0.1 \mu\text{m}$. Careful hardness measurements from materials with grain sizes less than $0.3 \mu\text{m}$ (linear intercept) are clearly needed for evaluation of the prediction capability of the model.

A wider range of hardness data for a more extensive set of Co contents have also been obtained and have been plotted against $d^{-0.5}$ and used to generate values for the coefficients E_1 and F_1 [13]. Regression analysis allowed an expression to be written for the variation of hardness, H , with grain size, d , for $6 < \text{Co} < 25 \text{ wt\%}$ as follows:

$$H = (888 - 9.9W_{\text{Co}}) + (229 + 532 \exp\{-(W_{\text{Co}} - 6)/6.7\})/\sqrt{d} \quad (10)$$

Expression (10) can be plotted as a property map, Fig. 10, bearing in mind it is not validated extensively for the finer grained materials. A property map using Co wt% content as the variable can also be plotted from expression (10). This is shown in Fig. 11 for different values of the WC linear intercept grain size.

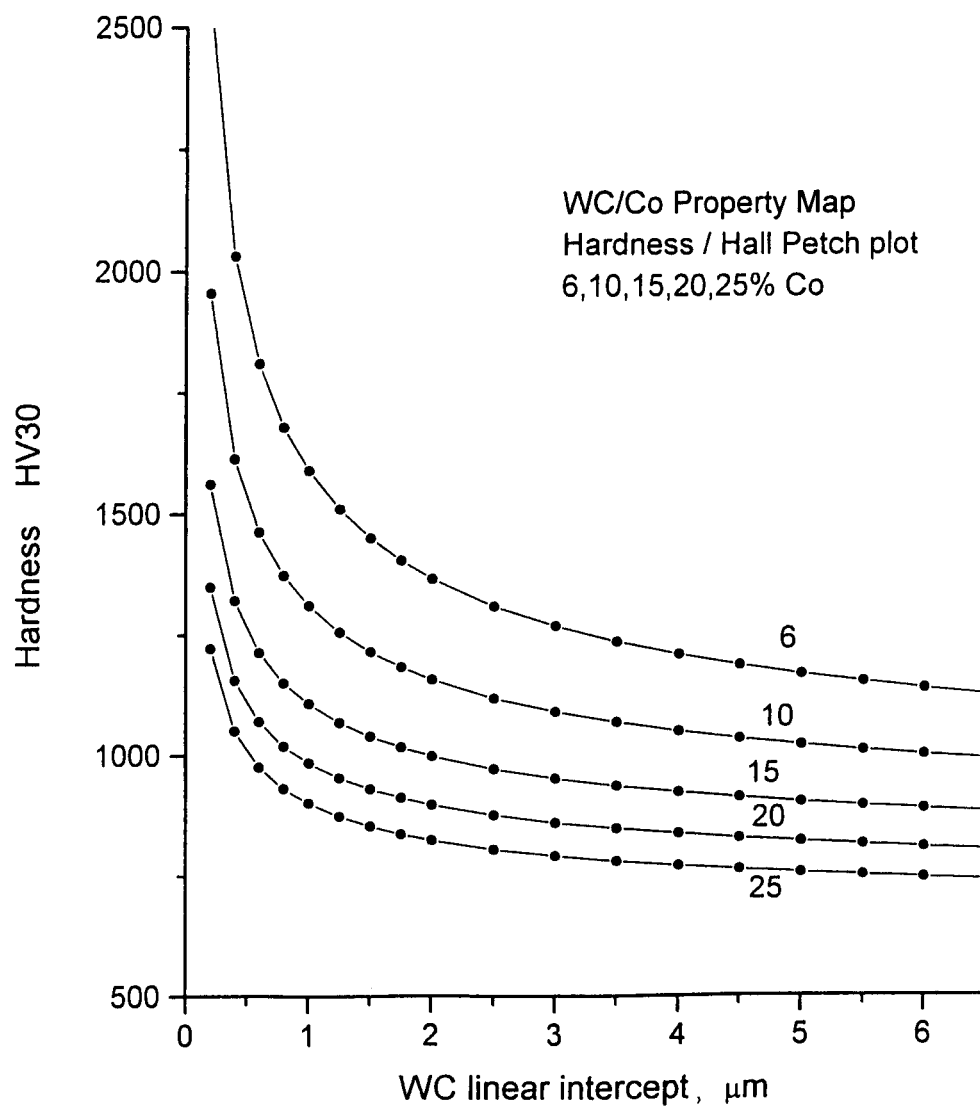


Figure 10 Hardness/grain size property map.

Recent research [18] indicates that an improved model may be usefully developed based on plots of HV30 vs. $\log d$ as opposed to $d^{-1/2}$, but more measurements need to be obtained on materials with mean intercept grain sizes less than $0.2 \mu\text{m}$.

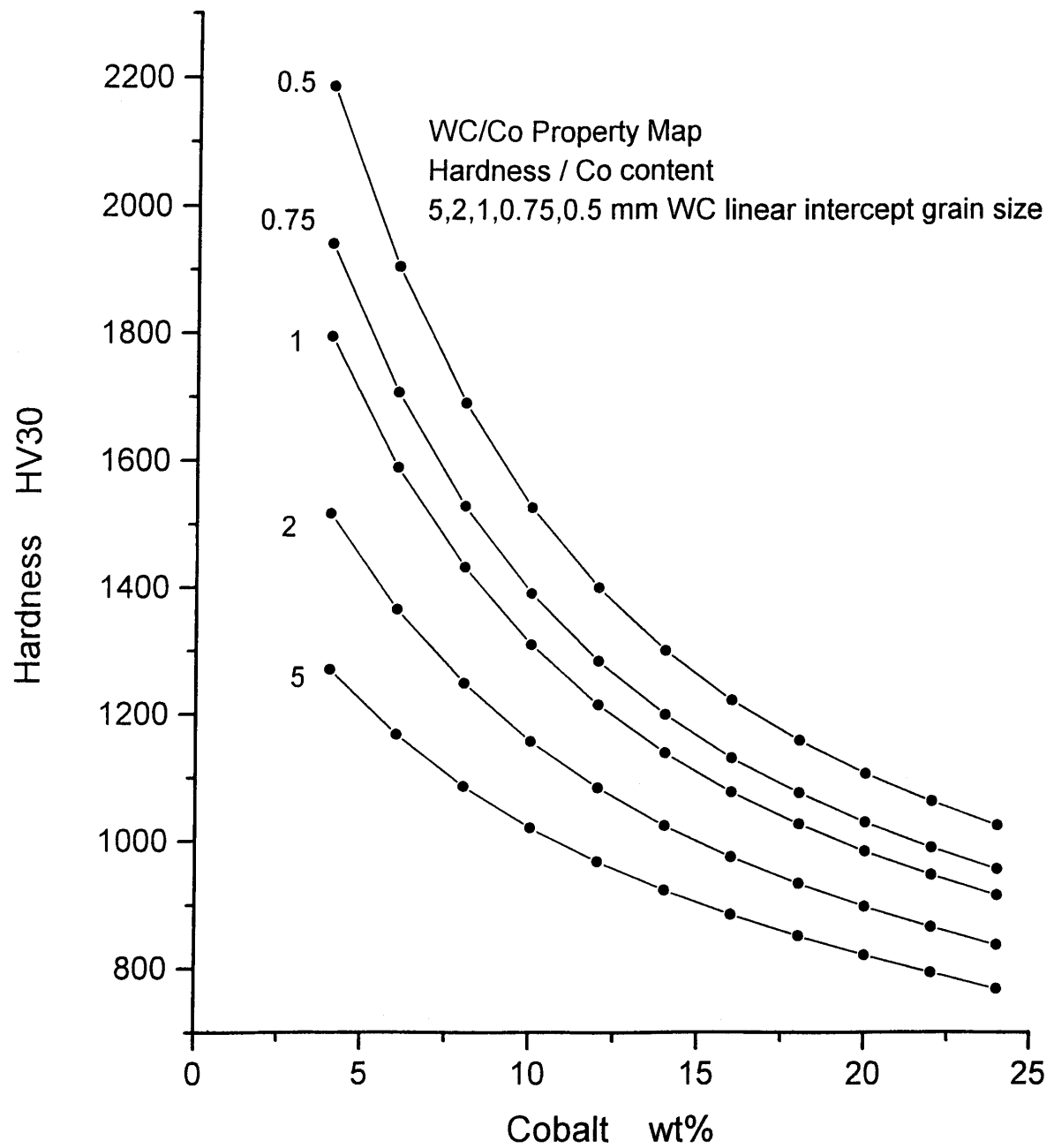


Figure 11 Hardness/Co content property map

4.2 Abrasion Tests

Many hardmetal applications make use of their extremely good abrasion resistance. In fact, few materials can resist wear as well as hardmetals. For this reason a laboratory test is required that gives repeatable and reproducible results and that is sensitive to changes in hardmetal structure. Historically the ASTM B611 abrasion test has proved to be the most ubiquitous, although other tests are being considered (such as a rubber wheel test and a ball cratering test). The most important issues are

- **Abrasive medium**
- **pH of test medium**

Al₂O₃ has proved to be a useful abrasive medium and is recommended because it gives results that are more sensitive to structural changes compared with other abrasive materials such as SiO₂. SiO₂ is more relevant to applications but is not sufficiently abrasive to give as sensitive results as Al₂O₃ in a reasonable period of test.

The pH of the test medium must be controlled because hardmetal is a composite of an acid-sensitive phase (Co binder) and an alkali-sensitive phase (WC).

Abrasion resistance can be measured using an ASTM standard - ASTM B611, Fig. 12. The results are expressed in terms of a volume loss for a given number of revolutions of the wheel. It is thus necessary to measure the density of the test-piece and convert mass loss (measured using a sensitive balance) to a volume loss. The tests are repeatable and sensitive to changes in structure.

The abrasion tests performed according to the ASTM B611 standard are generally made on a 40 x 20 x 5 mm diamond-ground hardmetal test-piece. The abrasive required by the standard is Al₂O₃. This produces severe wear in order to generate measurable wear scars in a reasonable period. The measurements of loss in mass are made using a sensitive balance. The uncertainty in these measurements can be as low as ± 0.2 mg in an overall sample mass of about 60 g. The density is obtained by measurements of weight in air and water, and these can be measured with an uncertainty of about ± 0.01 Mg m⁻³.

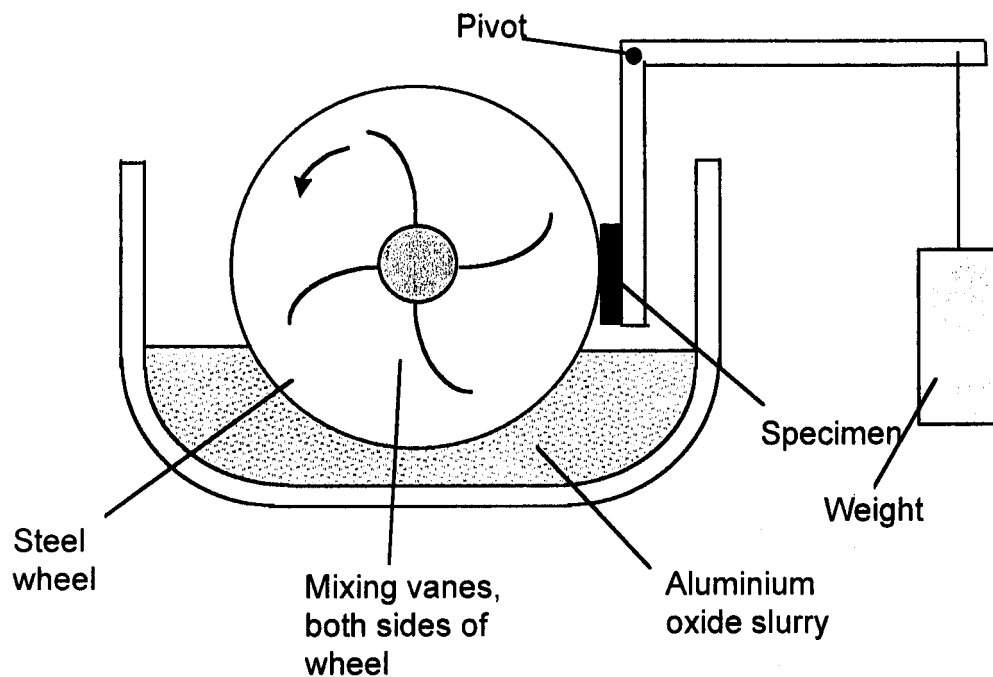


Figure 12 Test configuration for ASTM B611.

A small amount of work has been done on the effects of different abrasive carrier pH values. Very acid or very alkaline environments result in enhanced wear rates. In the ASTM B611 standard test, water at ambient temperature is specified to carry the Al_2O_3 abrasive. At NPL triple distilled water of pH value 5.4 is used for each test. The pH of the water does not change significantly during the test. This pH level is slightly acidic due to dissolved CO_2 , *e.g.* a specimen left standing for 1 month in the water showed signs of a brown corrosion product - probably a cobalt hydroxide. Fig. 13 shows the relation between abrasion resistance and hardness (HV30) for a range of WC/6 wt% Co hardmetals with different WC grain sizes and Co contents.

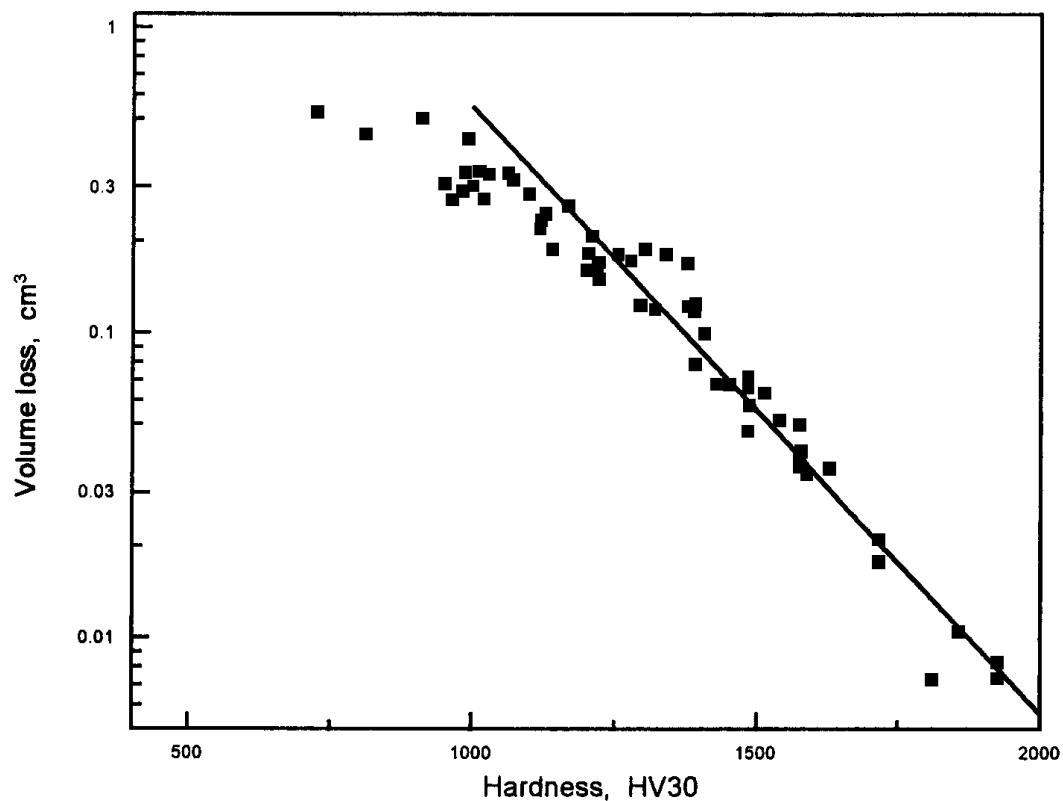


Figure 13 Abrasion resistance (volume loss) plotted against hardness, HV30.

It was found that the following expression fitted the volume loss, V_L , to hardness, H . The straight line in Fig. 13 corresponds to a linear regression fit to the data for the following expression:

$$V_L = g \exp [hH] \quad (11)$$

where V_L is the volume loss in cm^3 , H is the hardness (HV30-kgf/mm² implied), g and h are constants having the values 50.4 and -0.0046 respectively.

However it is important to note that the empirical relation in expression (11) is not particularly helpful in accurately predicting V_L from HV30 since the uncertainty in measurement of HV30 at a value of, say 1450 HV30, is typically ± 25 points. This is equivalent to a value of V_L of 0.064 ± 0.007 , compared with a standard deviation from the measurement itself of about ± 0.0005 to $\pm 0.001 \text{ cm}^3$.

The use of Al_2O_3 may not be representative of practical abrasion situations where SiO_2 is a more likely abrasive particle. Consequently tests have been performed on a range of hardmetals using SiO_2 as an abrasive. The results are shown in Fig. 14.

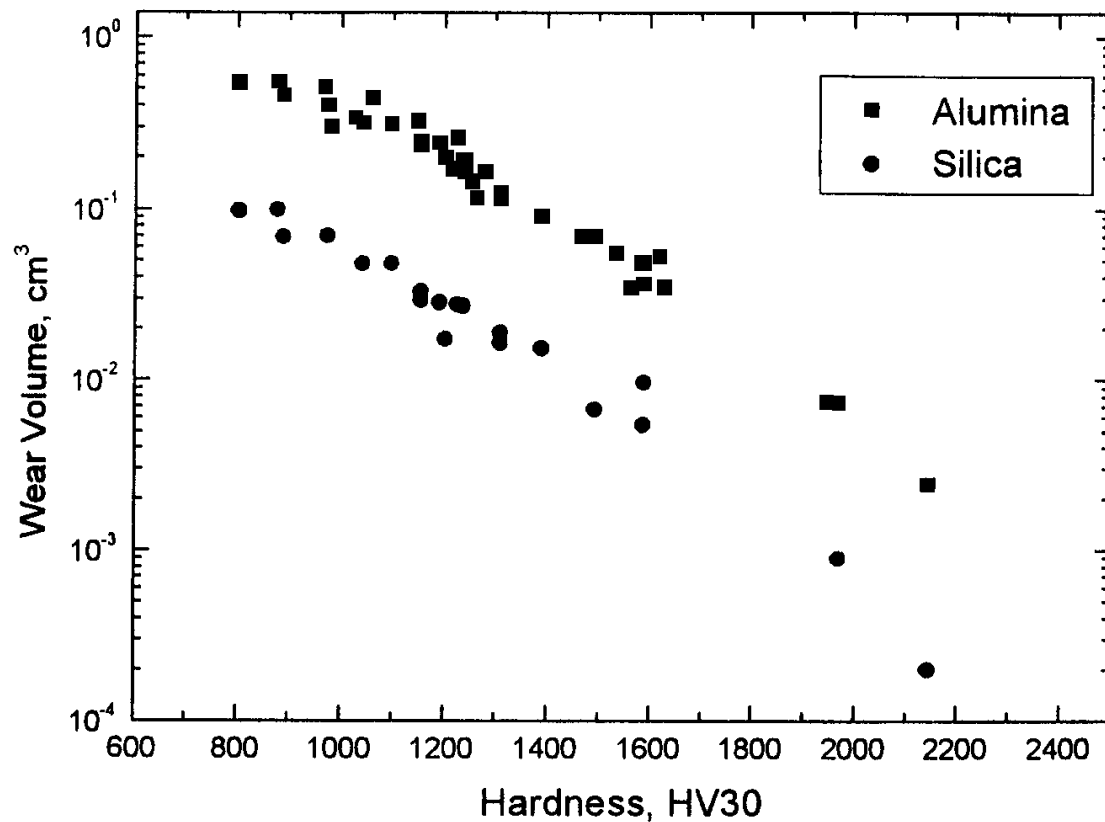


Figure 14 Effect of SiO_2 and Al_2O_3 abrasives on abrasion resistance.

It can be seen that the trends when using SiO_2 are identical to those observed when using Al_2O_3 , but the mass loss is nearly $\times 100$ less. Thus there is no advantage to using SiO_2 and the use of Al_2O_3 is recommended as this gives a greater mass loss and thus a better ability to discriminate between different materials.

4.3 Bend Tests

Like other hard materials hardmetals are difficult to test in tension. Therefore a bend test is used, as for ceramics, in order to generate strength data that can be used to rank materials and for design.

It is important to be aware of several important issues when attempting to perform and interpret strength tests on hardmetals and these are discussed in detail in this section of the guide. The two most important parameters are:

- **Testpiece geometry**
- **Testpiece surface preparation.**

The ISO standard geometries are all acceptable for use but it is important to recognise that different geometries can give different strength values, by a factor of up to 50%. Generally, the shorter the test rig span the higher the measured strength for a given material.

Test-piece surface preparation can have an even bigger effect than geometry through the manufacture the test-pieces. The increase can be as much as 100% and is larger the higher the hardness of the material.

The ISO standard recommends five test-pieces of compressive residual stresses introduced through the grinding process to pieces be broken, but ten is more acceptable if the results are to be analysed in depth, and as many as thirty could be required for a Weibull and fractographic analysis to characterise defect origins [15].

Until recently the standard for bend tests, ISO 3327, allowed two geometries for three-point tests on rectangular test pieces, nominally:

- 5 x 6 x 14.5 (span) mm
- 5 x 5 x 30 (span) mm

A range of alternative test geometries has been investigated in an international round robin, organised through VAMAS (an international activity aimed at developing pre-standardisation technical documents in the standards area to promote harmonisation) and as a result a new geometry (a round test-piece) has recently been added to the ISO standard.

This section of the guide gives considerable detail on the VAMAS exercise in order to illustrate the complexity of interpretation necessary to fully understand the results of bend tests on hardmetals. A guide to bend testing for ceramics and hardmetals has recently been completed [19] that gives additional background to the issues associated with bend tests on hard, brittle materials.

4.3.1 VAMAS interlaboratory exercise

The standard for transverse rupture tests (TRS, bend tests) for Hardmetals, ISO 3327, was re-evaluated to take account of developments in the understanding of strength measurements, the increasing desire for more relevant data to material quality and design, and not least, to work towards a test which can give results comparable to test data obtained on competing materials, such as ceramics and cermets.

In North America a military standard MIL-STD-1942A for flexural tests on ceramics has been adopted by the ASTM as C1161. In Europe a new CEN standard for bend tests on ceramics, EN843-1, has been developed, similar to the ASTM standard. In Japan there is a relevant standard JIS R 1601. An ISO standard will also shortly be published. The geometries associated with these standards are summarised in Table 9*. None of these geometries are compatible with ISO 3227. The usefulness of testing round specimens needed to be considered because many new hardmetal products are manufactured from rod. Also work at NPL indicated that notched bend tests can give useful data. Finally, puzzling very high strength data have been obtained in bend tests on ultra-fine grained hardmetals.

Table 9 - Standard geometries for bend testing

Standard	Type	Width <i>b</i> (mm)	Thickness <i>h</i> (mm)	Total span 3 pt, 4 pt (mm)	Inner span 4 pt (between central rollers) (mm)
MIL STD 1942A ≡ ASTM C1161-1990	A	2	1.5	20	10
	B	4	3	40	20
	C	8	6	80	40
JIS 1601	-	3	3	30	10
CEN EN843-1	A	2	2.5	20	10
	B	4	3	40	20

* An important feature is the effort that has been involved in minimising errors of measurement, allowing the evolution of a testing geometry in which the fracture stress error is typically less than 2%.

Existing standards, prior to the VAMAS exercise, are summarised in Tables 10 and 11.

Three-point (3 pt) testing, rectangular test-pieces

The information in Table 11 is taken from four standards documents which contained information of relevance: EN843-1 (flexure testing of ceramics), ISO 3327 (TRS of hardmetals), ASTM B406 (TRS of cemented carbides) and JIS 026-1983 (TRS of cemented carbides - CCTMAS, Japan). The information in the table relates only to ground test-pieces.

Four-point (4 pt) testing, rectangular test-pieces

The standards for testing hardmetals do not include the option of 4 pt tests. However, the ASTM conducted a comparative exercise in the 1980s and the EN standard for ceramics allows 4 pt tests, Table 11. Table 11 contains some information of a comparative nature from these sources, used to assist in planning the current interlaboratory exercise. Test-piece preparation is essentially the same as that given in Table 10 for 3 pt test comparisons.

The following comments taken from the ASTM B09.06 exercise on 3 pt vs 4 pt tests, and additional notes from the Japanese standards document JIS 026-1983 were also taken into account.

ASTM B09.06:

- 4 pt TRS results are significantly lower than those measured by 3 pt tests;
- The use of 4 pt test gave no improvement in test data scatter compared with 3 pt tests;
- No significant difference was noted between 3 pt tests using a 10 mm diameter cemented carbide ball or a 6.35 mm diameter cemented carbide roll for the central loading point.

JIS 026-1983:

The document indicates that preliminary tests were conducted to compare the Japanese test-piece design (24 x 8 x 4) with the ISO type A design (35 x 5 x 5) and type B design (20 x 6.5 x 5.25). The following comments have been selected:

- ISO type B is recommended to be excluded because the data scatter was larger and bigger loads were required (and hence more damage to jigs).
- The use of balls as load points was excluded.
- No difference was noted between tests on ground compared with as-sintered test-pieces, therefore both are allowed.

The types and numbers of test-pieces examined in the VAMAS exercise are given in Table 12 and the geometries in Fig 15. All test pieces were horizontally ground to ISO 3327, 0.2 mm chamfer (45°), out of squareness $\pm 2^\circ$, ± 0.01 mm tolerance, cylindrical load supports and load points with loading rates of $\sim 1500 \text{ N s}^{-1}$. Loads were measured to $\pm 1\%$. Information on materials and their sources are given in Table 13.

Table 10 - Standards (3pt tests)

	Information Source			
3 PT TESTS	ISO 3327	ASTM B406	JIS026-1983	EN 843-1 (ceramics)
Specimen type, Nominal dimensions (Length x width x height) (mm)	A 35 x 5 x 5 B 20 x 6.5 x 5.25	19 x 6.25 x 5	A 35 x 5 x 5 J 24 x 8 x 4	A 25 x 2.5 x 2 B* 45 x 4 x 3
Support distance (Nominal)	A 30 mm B 14.5 mm	14.3 mm	A 30 mm J 20 mm	A 20 mm B 40 mm
Surface condition	Longitudinally ground, (all four faces), Remove 0.1 mm, no pass _ 0.01 mm, roughness <0.4 $\mu\text{m Ra}$	Longitudinally ground (top and bottom), no pass _ 0.0127 mm, 0.381 μm roughness	Longitudinally ground (4 faces), remove 0.1mm 0.165 roughness (JIS B0601)	Longitudinal grinding, very specific - see standard for details Alternatively - application matched
Parallelism	_ 0.01 mm	_ 0.0254 mm	_ 0.02 mm	-
Out of squareness	Not specified	< 2°	Not specified	< 5°
Chamfer	45°, 0.15-0.2 mm	45°, _ 0.25 mm	45°, _ 0.2 mm	45°, 0.12 mm
Measurement tolerance	± 0.01 mm	± 0.02 mm	± 0.01 mm	± 0.01 mm
Visual inspection	Must be free from cracks & defects	Eliminate obviously damaged test-pieces	Reject obviously defective test-pieces or if failure >3 mm from load pt	Include all results even if failure away from central zone
Number	_ 5	5	_ 5	10-30 (30 required for Weibull analysis)
Load	$\pm 1\%$	$\pm 1\%$	$\pm 1\%$	$\pm 1\%$
Load or stress rate	_ 200 $\text{Nmm}^{-2}\text{s}^{-1}$	_ 1500 Ns^{-1}	_ 800 Ns^{-1}	May be material dependent - record value
Load support	Cylinders	Cylinders	Round bars	Rollers
Load point	Cylinders or ball	Ball	Round bar	Rollers
Comments				Fractography recommended
Results	Round to 10 N/mm^2	-	Round to 10 N/mm^2	-

* EN843 test-piece B is equivalent to ASTM C1161-1990 type B (ceramics)

Table 11 - Standards (4 pt tests)

4 pt. tests	ASTM GTE Sylvania Technical Memorandum ASTM Subcommittee B09.06	EN843-1 Ceramic flexural tests
Specimen type, nominal dimensions, mm (length x width x height)	ISO 3327 - A 35 x 5 x 5	A 25 x 2.5 x 2 B 45 x 4 x 3
Outer span support distance	27.94 mm	A 20 mm B 40 mm
Inner span support distance	9.27 mm	A 10 mm B 20 mm
Load support	Cemented carbide cylinders	Rollers
Load points	Cemented carbide cylinders	Rollers
Number	_ 6	10-30

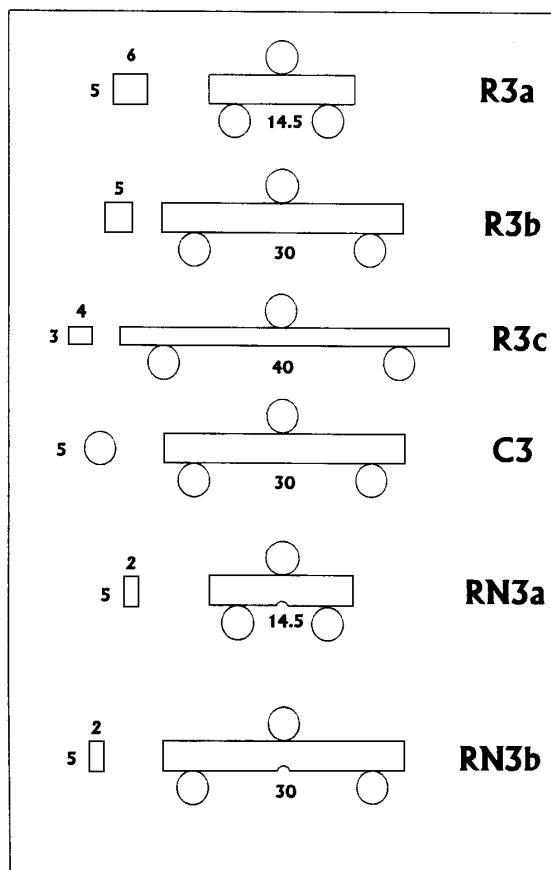
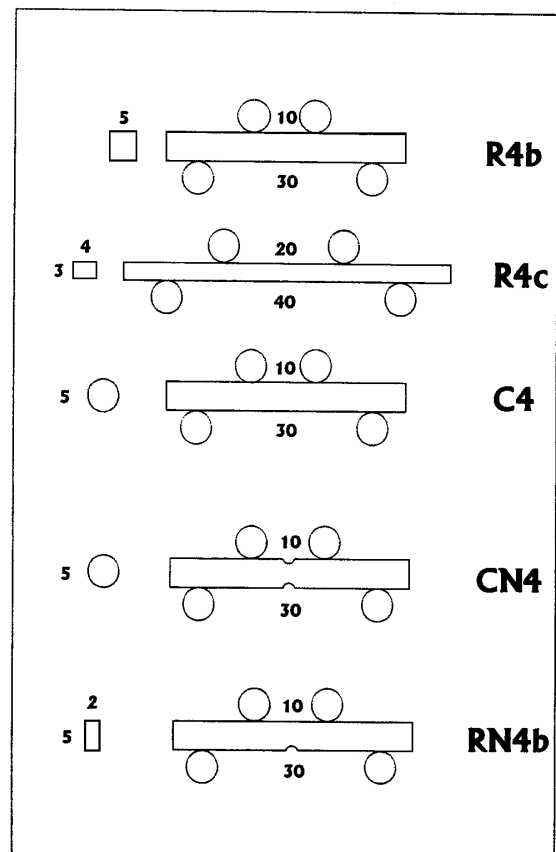
Hardmetal Bend Tests**Three Point Tests****Hardmetal Bend Tests****Four Point Tests****Figure 15** Schematic diagram of bend test geometries.

Table 12 - Options and numbers of test-pieces

Test type- test-piece size (mm)	Test category	Material category				
		1 WC/Co U fine*	2 WC/Co fine*	3 WC/Co/CC fine/medium*	4 Cermet fine*	5 WC/Co coarse/medium*
Rectangular (3 pt) ^a 15 x 6 x 5 30 x 5 x 5 40 x 3 x 4	[C1] ^a [C2] ^a [C3] ^a	30 20 20	30 20 20	30 20 20	30 20 20	30 20 20
Rectangular (4 pt) 30(10) ^b x 5 x 5 40(20) ^b x 4 x 3	[D1] ^c [D2] ^c	20 20	20 20	20 20	20 20	20 20
Notched (3 pt) 15 x 2 x 5 30 x 2 x 5	[E1] ^d [E2] ^d	12 12	12 12	12 12	12 12	12 12
Notched (4 pt) 30(10) x 2 x 5	[E3] ^d	12	12	12	12	12
Round (3 pt) 30 x 5 mm Ø	[F1] ^e	20	20	20	20	20
Round (4 pt) 40(20) x 5 mm Ø	[F2] ^e	20	20	20	20	20
Round & notched (4 pt) 30 x 5 mm Ø	[F3] ^e	12	12	12	12	12

^a C1, Existing ISO 3327 Type B for baseline; C2, C3, ISO Type A and ceramic Type B for comparison with C1.

^b 30(10), 30 mm full support span, 10 mm loading point span; 40(20), 40 mm full support span, 20 mm loading point span.

^c D1,D2, two different spans; which are based on ISO 3327 and ceramic Type B.

^d E1,E2, notched test-pieces; E1 based on ISO 3327 Type B; E2 based on ISO 3327 Type A; E3, 4 pt notched, based on ISO 3327 Type A.

^e F1, 3 pt, based on ISO 3327 Type A; F2, 4 pt, based on Ceramic Type B; F3, 3 pt, notched; for comparison with E2.

* WC grain size

Table 13 - Materials and sources

Category	Nominal Type	Source
1	WC/Co; _ 6% Co fine grained HV30 - 1800 to 1900	Teledyne Advanced Material
2	WC/Co; 11% Co fine/medium grained HV30 - 1500 to 1600	Boart RC Sandvik
3	WC/Cubic Carbide/Co; 16% Co HV30 - 1500 to 1600	Kennametal
4	Ti(C,N) cermet HV30 - 1500 to 1600	Sandvik
5	WC/Co; 12% Co medium/coarse grained HV30 - 1000 to 1200	Sandvik American National Carbide

The interlaboratory exercise included tests on both round and notched test-pieces. Previous work at NPL (16) had shown that the bend strength of hardmetals is determined by two factors:

- The distribution of large defects (pores *etc.*), greater in size than approximately 20 μm ;
- The intrinsic strength of the average microstructure, termed limiting strength.

Further work (1, 14) has shown that the limiting strength can be measured using a notched bend test-piece. This test method has several advantages; as follows:

- The scatter is considerably reduced;
- Much lower loads are required to break test-pieces, *i.e.* less damage to jigs and perhaps less expensive test machines;
- The strength values obtained are higher (because failures from large defects are excluded).

The notched tests performed to date have been on modified ISO 3327 test-pieces (type B), *i.e.* 14.5 mm span x 2 mm wide x 5 mm high (3 pt) with a 1 mm deep notch with a 0.5 mm notch radius. The interlaboratory exercise examined 4 pt notched and 3 pt tests on different geometry specimens (Table 12).

4.3.2 VAMAS data analysis

The data for all the tests are given in sets of tables in two VAMAS reports [21, 22] written

following the completion of the interlaboratory tests.

The results were plotted as sets of data ranked against strength. The rank value (R) was calculated from:

$$R = (n_i - 0.5)/N \quad (12)$$

where n_i is the i^{th} value of strength and N is the total number of tests.

Preliminary conclusions from the ranked plots showed that:

- There was good agreement between laboratories testing a given geometry and material.
- Surface preparation had a very significant effect on strength for some of the materials tested. In particular, annealing reduced strength by relieving compressive residual stresses introduced during grinding.
- Nominal strengths differed by a factor of up to two depending on the geometry used.
- Notched test-pieces gave less scatter except in material processed to contain few macroscopic internal defects.

In order to compare strength values from the different geometries and materials several methods for analysing the data were examined:

- By using Weibull analysis on all of the results in a set to investigate whether strength follows the stressed volume systematically (section 4.3.2.1).
- Examination of notched test-pieces (section 4.3.2.2).
- Through Weibull analysis to obtain a "characteristic strength" value σ_0 , (section 4.3.2.3).
- By averaging the top 3-4 values in the ranked distribution (section 4.3.2.3).

The effects of annealing on strength values was also investigated by plotting ranked values of strength and comparing them with the results from the as-ground test-piece (section 4.3.3).

4.3.3 Weibull analysis

The strength values obtained in bend tests are controlled by the mechanism of failure initiation. There are two possibilities

- Initiation from defects such as pores, flaws, large WC grains;

- Microstructure-initiated (in the absence of macroscopic defects).

A full interpretation of bend test data thus requires fractography to correlate scatter in strength values with the failure mechanism (section 4.4).

It is fairly common practice to plot the results of bend tests on other brittle materials such as ceramics using a Weibull distribution of strengths based on an assumption that only one failure mechanism is present and that the failure probability, P , is a function of the stress, σ given by:

$$P = 1 - \exp\{-(\sigma/\sigma_0)^m\} \quad (13)$$

where σ_0 and m are constants, with m being called the Weibull modulus. For results that show a high scatter m takes a low value. The failure probability, P_i , of the i^{th} test in ranked order is defined as:

$$P_i = 1/(N + 1) \quad (14)$$

where N is the total number of tests

It is not generally recommended that values of m are derived from tests with a population, N , less than about 30 because of the large uncertainties that arise in taking double logarithms. From equation (13):

$$\ln(\ln(1/(1-P))) = m \ln \sigma - m \ln \sigma_0 \quad (15)$$

therefore plotting $\ln(-\ln(1-P))$ against $\ln \sigma$ should result in a straight line of slope m and intercept $m \ln \sigma_0$. Although insufficient tests were performed to permit an accurate analysis representative data from the exercise are plotted in accordance with equation (15) in Fig. 16. No attempt was made to fit a straight line to any of the plots, because, in particular for the plain specimens there is clearly considerable curvature, in addition to there only being 12 data points. However, it can be seen that the data from the notched specimens show less scatter, equivalent to higher m values (about 12) and the data from both kinds of test appear to fit the same population at high values of σ , thus indicating a similar mechanism of failure, one of microstructural initiation rather than one of large-defect initiation.

When the left hand side of expression (15) is equal to zero then:

$$m \ln \sigma = m \ln \sigma_0 \quad (16)$$

and σ is characterised by a representative σ_0 value, the "characteristic strength" where the plot cuts the horizontal line for $\ln \ln(1/(1-P))$ equal to zero.

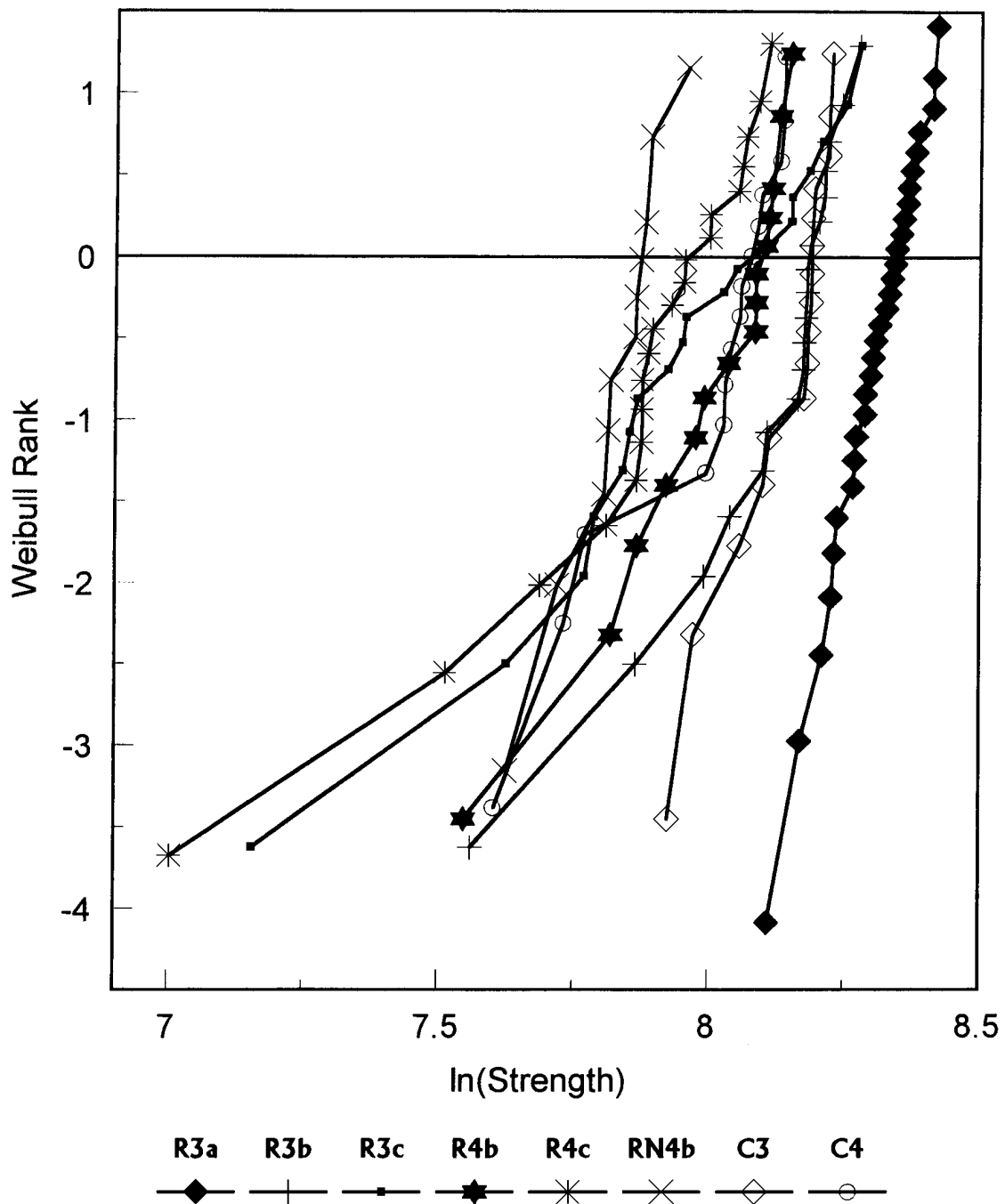


Figure 16 Weibull plots of ranked strength values from different test-piece geometries.

It is recognised that the reliability of Weibull analysis is poor on small test-piece numbers, and increasingly so for low values of Weibull modulus [23]. However, the principles of the analysis permit some correlations to be made between the different testing geometries, and thus provide a better basis for discussing the results than using arithmetic mean strengths and standard deviations.

Because of the difficulty of analysing mathematically whether data sets belonged to mixed or single mode Weibull behaviour, best fit lines were drawn by eye through the Weibull plots [22] and approximate values for the Weibull modulus were calculated from these lines. It has been shown [24] that for different values of m different numbers of test specimens are required such that sufficiently accurate values of m can be estimated to allow sensible extrapolations to be made from the data. For example [24]:

m	Number of test-pieces*
7	70
20	10

* to give accurate estimates for a value of 10^{-6} for the strength probability.

It was found that there were four categories of plot in the VAMAS exercise [22]:

1. Those with high values of m ($\geq$ about 20), for example for the R3b geometry Sandvik (medium/coarse) material;
2. Those with low values of m ($\leq$ about 10), for example for most of the Ti(C,N) cermet materials;
3. Those with intermediate m values ($20 > m > 10$);
4. Those which have two distinct parts to the plot, *e.g.* R4c - Boart fine, R4c - Teledyne, R4b-Kennametal. This has been observed previously in tests on hardmetals [24] and was then attributed to failures from two different types of originating defects, classed as Type A and Type B:

Type A - microstructure initiated

Type B - gross defect (pore, inclusion, large WC grain, *etc*)

For Category 1 plots above, it was therefore assumed that the failures were microstructure initiated. For Category 2 plots, failures were generally defect initiated [22]. For Category 3 plots, the failure initiation sites needed to be individually classified. The VAMAS project had insufficient resources to characterise the fracture surfaces in every test-piece (over 1000). However, many of the Ti(C, N) cermets were examined. These were analysed by comparing the

stress at the position of the fracture site with the inverse of the square root of defect area (by fitting an ellipse to the shape of the defect) following a procedure discussed in previous work [17, 24]. For large defects it was found that there was a correspondence between stress and a parameter with units of $\mu\text{m}^{-1/2}$, which is indicative of a fracture mechanics correlation; but for small defects the strength attains a limiting value [25].

Weibull analysis was used to reference the mean strength values to a common level to see if strength followed the stressed volumes or areas in a systematic way [21]:

$$\bar{\sigma}_{\text{nom}} = \bar{\sigma}_{\text{ref}} \left\{ \frac{V_{\text{ref}}}{V \Sigma V} \right\} \text{ or } \bar{\sigma}_{\text{ref}} \left\{ \frac{A_{\text{ref}}}{A \Sigma A} \right\} \quad (17)$$

where σ_{nom} is the mean nominal strength of a test population, σ_{ref} is the mean strength of a reference volume or area which has been uniformly stressed; V_{ref} and A_{ref} are the relevant reference volumes or areas, V and A are the actual stressed volumes or areas and ΣV and ΣA are stress volume and stress area integrals respectively given by

$$\Sigma V = \int \left(\frac{\sigma(V)}{\sigma_{\text{nom}}} \right)^m \frac{dV}{V} \quad (18)$$

$$\Sigma A = \int \left(\frac{\sigma(A)}{\sigma_{\text{nom}}} \right)^m \frac{dA}{A} \quad (19)$$

ΣV and ΣA have the following values for 3 pt and 4 pt rectangular and round test-pieces (Table 14).

Table 14 – Stress-volume and stress-area integrals*

Geometry	ΣV	ΣA
3 pt Rectangular, R3b	$\frac{1}{2(m+1)^2}$	$\frac{1}{2(m+1)^2} \left\{ \frac{1+f+m}{1+f} \right\}$
4 pt Rectangular, R4b	$\frac{m+2}{4(m+1)^2}$	$\frac{m+2}{4(m+1)^2} \left\{ \frac{1+f+m}{1+f} \right\}$
3 pt Round, C3	$\frac{1}{\pi(m+1)} \cdot A(m)$	$\frac{m+2}{2\pi(m+1)} \cdot A(m)$
4 pt Round, C4	$\frac{m+2}{2\pi(m+1)} \cdot A(m)$	$\frac{(m+2)^2}{4\pi(m+1)} \cdot A(m)$

*Where f is h/b and $A(m)$ is a function given by:

$$A(m) = \left(\Gamma\left(\frac{m+1}{2}\right) \Gamma\left(\frac{3}{2}\right) \right) / \left(\Gamma\left(\frac{m+4}{2}\right) \right) \quad (20)$$

This can be approximated by a power law:

$$A(m) = 1.26 m^{-1.3} \quad (21)$$

The integrals in Table 14 can then be used to examine the ratios of 3 pt to 4 pt strength values predicted by values of m measured from real data sets and compared with measured ratios of 3 pt to 4 pt strength.

For example from (17), for both rectangular (R3b and R4b) and round (C3 and C4) test-pieces:

$$\text{On a volume basis:} \quad \frac{\sigma_{3\text{pt}}}{\sigma_{4\text{pt}}} = \frac{(V_4 \Sigma V_4)^{1/m_4}}{(V_3 \Sigma V_3)^{1/m_3}} \quad (22)$$

$$\text{On an area basis:} \quad \frac{\sigma_{3\text{pt}}}{\sigma_{4\text{pt}}} = \frac{(A_4 \Sigma A_4)^{1/m_4}}{(A_3 \Sigma A_3)^{1/m_3}} \quad (23)$$

where m_3 and m_4 are the Weibull moduli from 3 and 4 pt bend tests respectively. Weibull moduli, obtained from fits to all the data, and mean values for each material type and test geometry were compared with values for the ratio of 3 pt/4 pt strength, and the predicted ratios of 3 pt/4 pt strength from the Weibull moduli [22].

The results indicated that

- It was not possible to separate predicted strength ratios assuming area or volume defect distributions. The σ_3/σ_4 ratio was the same for both defect types, for R3b/R4b and for C3/C4.
- The predicted σ_3/σ_4 ratio for rectangular test-pieces was much higher than for the σ_3/σ_4 ratio for round test-pieces.
- The R3/R4b and C3/C4 measured strength ratios were similar for the conventional hardmetals but different for the Ti(C,N) cermet.

Thus the Weibull analysis was probably not appropriate for the conventional WC/Co grades since the analysis predicted different σ_3/σ_4 ratios, independent of test-piece type. However, for the Ti(C, N) cermet, where it was known that many of the failures were initiated by volume-defects, the measured ratios for rectangular and round test-pieces were different, as predicted by the Weibull analysis. However, the values of the σ_3/σ_4 ratio predicted by the analysis for the

rectangular test-pieces (1.86, 1.73), were much higher than that measured (1.25 [22]).

4.3.4 Notched bend tests

For this test developed at NPL rectangular test-pieces were ground on all sides to produce 20 x 5 x 2.5 mm bend specimens. These were then V-notched [17] on one of the 20 x 2.5 mm faces to a nominal depth of 1 mm using a profiled diamond resin-bonded wheel, removing material at about 0.002 mm per pass. The radius of the root of the finished notch is 0.5 mm and the included angle between the faces of the notch is 90°. The notched test-pieces are annealed, prior to testing, at 800 °C for 1 h in a vacuum to relax ground-in residual stresses.

Notched bend tests were originally performed in a test jig with a span of 14.5 mm in line with the current standard for bend tests at room temperature, approximately 20 °C, with the load applied at a rate of about 50 N s⁻¹. In more recent work this approach has been extended to test in a 4 pt bend jig with outer span 30 mm and inner span 10 mm with a similar V-notch in 2 x 5 x 40 mm test-pieces.

The nominal stress, σ_A , in an un-notched specimen is given by:

$$\sigma_A = \frac{6M}{BW^2} \quad (24)$$

where M is the bending moment, B is the width and W is the height.

In the notched specimens the stress at the root of the notch, σ_B , is given by:

$$\sigma_B = K \frac{6M}{BY^2} \quad (25)$$

where K is a stress concentration factor and Y is the thickness (height) of the ligament beneath the notch.

Thus:

$$\sigma_B = K \frac{W^2}{Y^2} \sigma_A \quad (26)$$

For the 20 x 5 x 2.5 mm test-pieces K was 2.45 [17] and W/Y was 1.25.

Therefore:

$$\sigma_B = 3.82 \sigma_A \quad (27)$$

Typical ranked strength results are shown in Fig. 17. The ranking was obtained by using the formula in expression (12).

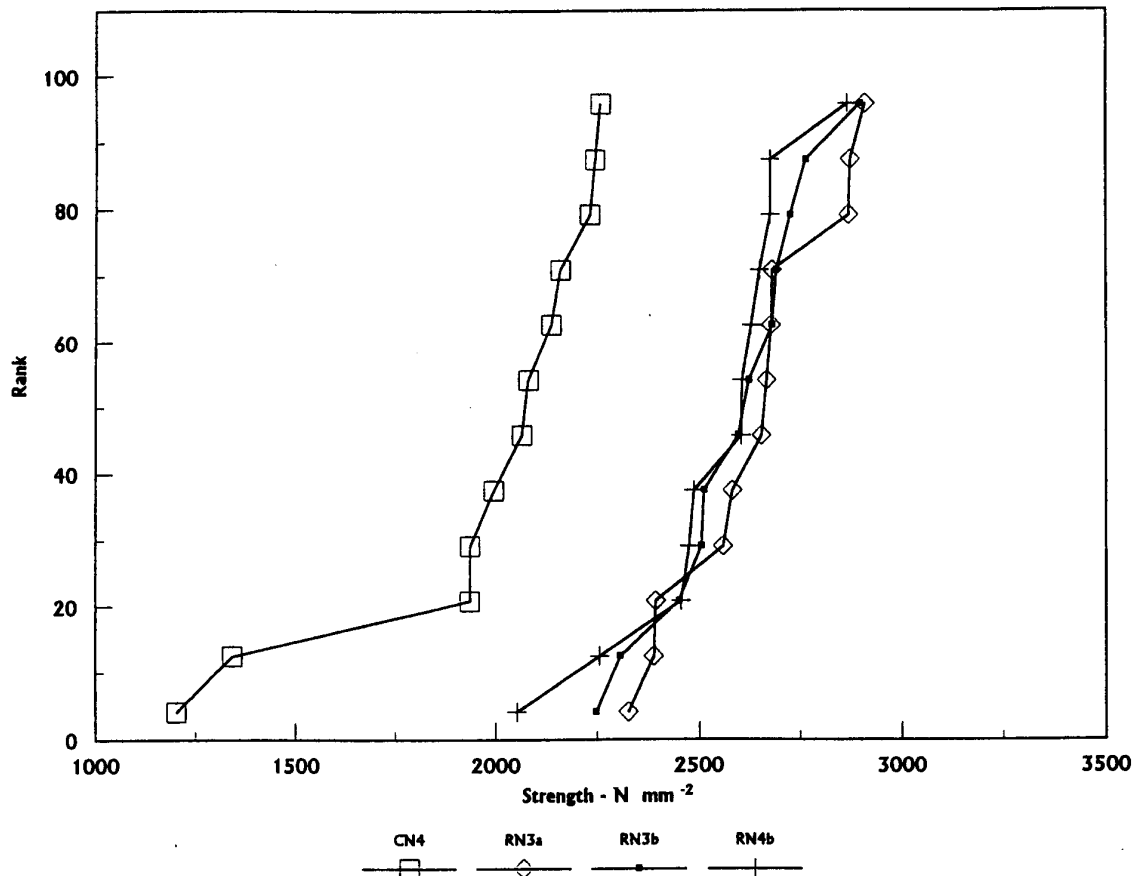


Figure 17 Ranked notched bend test results.

The data are plotted in a ranked format to give an indication of spread in strength values and to make it easier to identify test-pieces which give "outlier" values either at the top or bottom of the distribution. In general, however, most of the notched bend test results fitted a linear plot of rank against strength and are usually sufficiently tightly classed to allow most samples to be discriminated from each other. The average typical standard deviation is about 6%, and mean strengths range from about 2700 - 3600 N mm⁻².

4.3.5 Representative values of strength

The upper section of the Weibull ranked plots, which is clearly Type A for bimodal plots but could be Type A or Type B (section 4.3.2.1) for single mode fractures, allows a characteristic σ_0 value to be calculated. This was chosen to be a strength parameter, characteristic of the material and geometry, by which the results from all the tests might be compared, as opposed to taking the arithmetic mean of all the tests. This value is more likely to be representative of the underlying structure. These σ_0 values are shown plotted in Fig. 18 for all the geometries and materials.

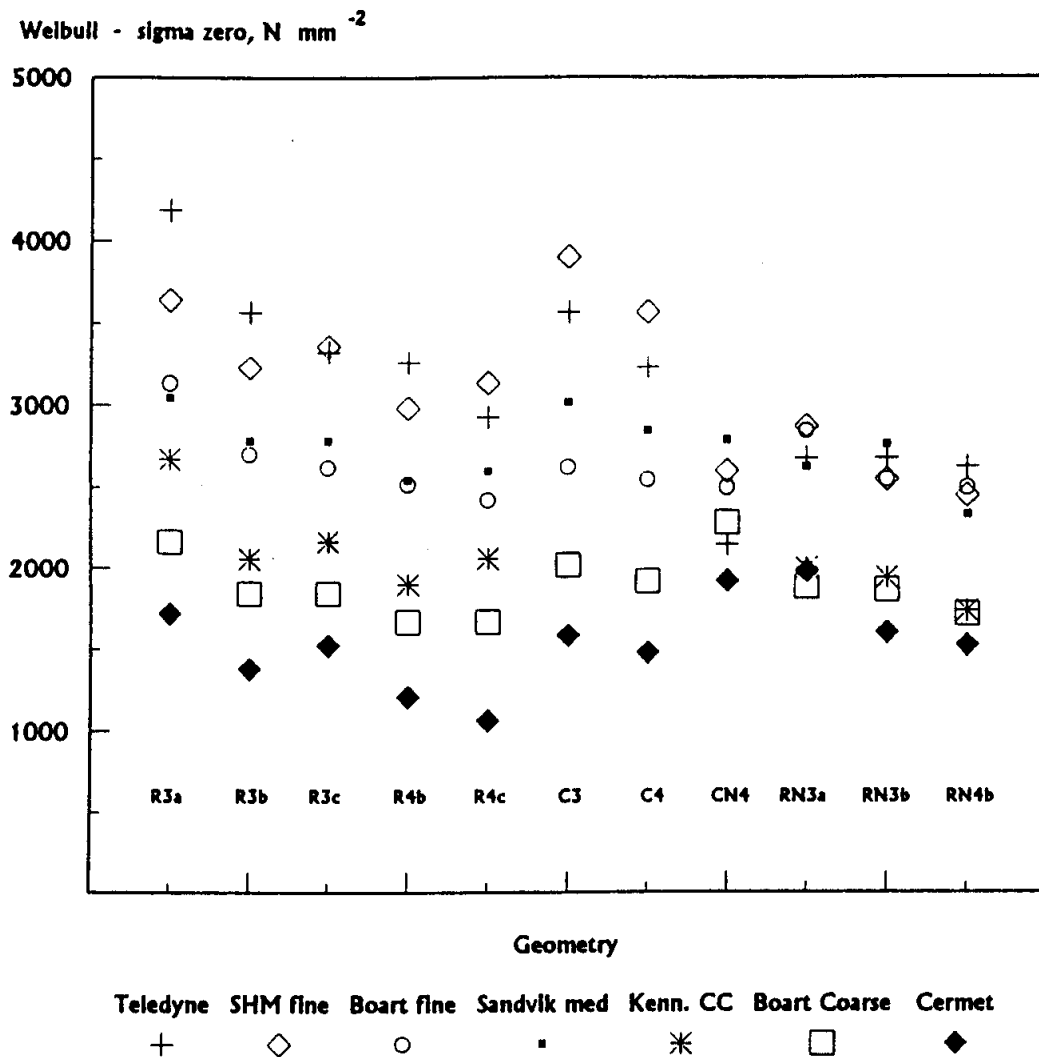


Figure 18 Representative σ_0 strength values for tests from different geometries.

An alternative method for calculating representative strength values was adopted by averaging the top 3 or 4 highest values of strength in each category. The trends between different materials and geometries were very similar whichever method of plotting was used, whether a Weibull σ_0 value or a mean strength value for the highest strength test-pieces in each population.

A number of observations can therefore be made from the data shown in Fig. 18:

- The materials were ranked similarly by all the unnotched geometries;
- Geometry R3a produced the highest strength values;
- The three-point bend geometries produced higher strength values than the four-point bend geometries, *i.e.* R3b > R4b, R3c > R4c, C3 > C4;

- The 5 mm x 5 mm rectangular cross-section test-pieces produced higher strengths than the 4 mm x 3 mm test-pieces (*i.e.* R3b and R4b > R3c and R4c respectively);
- The circular test-pieces, C3 and C4, gave similar strength values to the rectangular test-pieces, R3b and R4b, of similar span to depth geometries;
- For the WC hardmetals the strengths (calculated from the notch geometry) from the notched test-pieces are generally lower than those from unnotched test-pieces;
- However, for the Ti(C,N) cermet, the notched strengths were as high or higher than strength values obtained in unnotched tests.

There are a number of possibilities for the differences observed in Fig. 18 and the following issues were examined

- Friction stresses
- Wedging stresses

4.3.6 Friction stresses

If the supporting rollers cannot easily rotate then friction stresses are set up in the outer tensile fibres of the beam which require an additional bending moment to deflect the test-piece to the curvature it would have in the absence of friction. This means that the calculated stress in the beam is higher than it really is. The correction factor, CF_f , expressed as a percentage is given by [19]:

$$CF_f \% = \{ \mu / (S / h - \mu) \} . 100 \quad (28)$$

where μ is the coefficient of friction, S is one half the full span in 3 pt bend and the distance between the inner and outer rollers in 4 pt bend and h is the depth of the test-piece. A value of 0.2 was used for μ .

4.3.7 Wedging stresses

Timoshenko [26] has shown that a concentrated load on a beam produces "wedging stresses" under the point load which modify the stress distribution on the tensile surface. The correction factor, CF_w , is of the same sign as the friction correction factor (*i.e.* stress is over-estimated using the nominal formula), and is given by

$$CF_w \% = \left(\frac{2h}{3\pi d} \right) . 100 \quad (29)$$

where d and h are the same as above. However, this correction only applies to the position on

the tensile surface directly opposite the point load. Baratta *et al.* [27] have shown that the correction factor differs as the point of interest moves away from the position directly beneath the load application point, and at some positions it takes on the opposite sign. Thus, it is very difficult to correct for the wedging stress in a general sense as test-pieces can fail at random positions along the tensile surface. They do not always fail at the position of nominal maximum stress. However, for the purposes of correcting the nominal stress values the above formula was used for the 3 pt tests.

A similar situation occurs in 4 pt bend and FE has been used [28] to examine the variation in stress along the test-piece for different span to depth values. For the geometries used in the current exercise, this results in quite small correction values in the 4 pt bend tests.

In summary, the following correction factors were thus used to recalculate representative strength values for all the materials and all the geometries (Table 15).

Table 15 - Correction factors

Geometry	Correction factor, % friction CF_f	Correction factor, % wedging CF_w
R3a, RN3a	- 16	- 14
R3b/C3/RN3b	- 7	- 7
R3c	- 3	- 3
R4b/C4/RN4b/CN4	- 11	+ 2
R4c	- 6	0

The corrected values of the characteristic strengths were compared with the uncorrected characteristic strength values and it was shown [22] that the strength values for the unnotched test-pieces after correction were very similar, indicating that in some of the geometries true stresses are some 10-30% less than those calculated using the nominal beam formula.

The strength values of the rectangular notched test-pieces were not brought together any closer by the correction factors since they were quite close anyway. The values were just reduced by a factor of about 20%. There was, however, a significant difference between the circular notched test-piece and rectangular notched test-piece results. For most of the materials the strength values from the circular notched test-pieces were higher than those measured on the rectangular specimens. The reason for this is not known.

In general, the notched test-pieces gave lower strength values than the unnotched test-pieces and this was probably due to surface compressive residual stresses in the unnotched test-pieces since the notched test-pieces were annealed before testing. The difference was greatest for the harder, fine grained WC/Co hardmetals.

Thus, in summary, differences in nominal strength in the unnotched test-pieces could be due to differences in friction and wedging stresses. Using nominal correction factors the strength values from the different geometries agreed reasonably well. Better agreement would depend on specific FE analysis for the different test-pieces and more accurate figures for the coefficient of friction. The use of strain gauged test-pieces would allow some of these correction factors to be quantified more accurately.

4.3.8 Effects of annealing

It was shown in the VAMAS reports [21, 22] that annealing unnotched test-pieces of different geometries at 800 °C for 1 h in vacuum reduced the strength significantly. Fig. 19 shows comparative plots of representative values of some of the annealed and unannealed strengths, taking the top 3 or 4 values. (Geometries tested are given in Table 16.)

Table 16 - Geometries of annealed test-pieces

Material	Geometry of annealed test-pieces	
	First VAMAS Report [21]	Second VAMAS Report [22]
Teledyne (UF)	R3b, C3, C4	R3a
Boart (F)	-	-
Sandvik HM (F)	-	-
Sandvik Cermet	R3b, C3	R3a
Kennametal CC	-	-
Sandvik (M/C)	R3b, C3, C4	-
Boart (C)	R3b, R4b, C3, C4	R3a, R3c

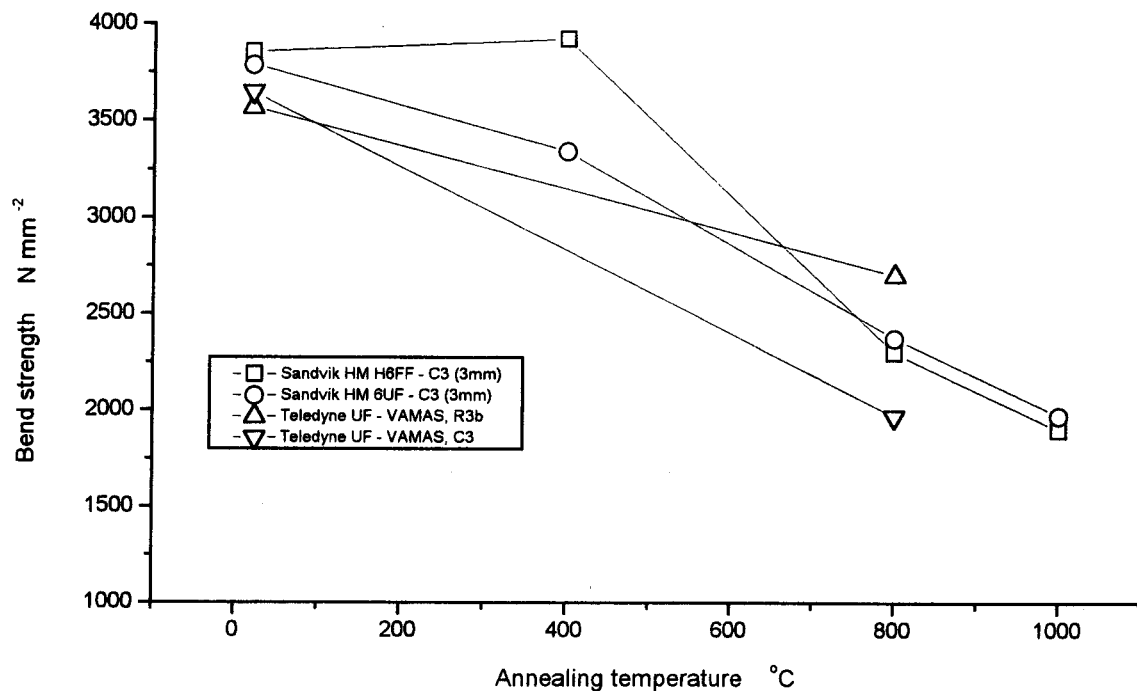


Figure 19 Effects of annealing on strength.

The results in Fig. 19 show that annealing the as-ground test-pieces reduced the strength. The amount of reduction in strength was dependent on the material type, annealing temperature and the geometry. There are two possible reasons for the reduction:

1. The as-ground surfaces contain compressive residual stresses which are removed when the samples are annealed;
2. The as-ground surfaces contain small cracks which are held closed by the residual compressive grinding stresses. After the sample is annealed at this low temperature the cracks become stress-free and act as small defects, thus reducing strength.

Further work is needed to evaluate which of these two mechanisms (or both together) is responsible for the reduction in strength that occurs when the samples are annealed.

The reduction in strength for the Ti(C, N) cermet material was much smaller than for the other materials except for the round test-piece. This is consistent with the fact that even for the highest strength values subsurface volume distributed defects were observed.

The highest reductions in strength on annealing occurred in the Teledyne (UF) hardmetal. This could mean that the compressive residual stresses are higher in finer-grained material or that finer-grained hardmetals are more susceptible to the formation of small surface cracks.

4.3.9 Fractography

In order to examine some possible reasons for "outlier" strength values and in order to characterise fracture sources, broken test-pieces are generally examined by scanning electron microscopy. A recent NPL guide [29] to fractography of hard materials has been written and should be consulted for detailed guidance on the interpretation of fracture origins in bend test-pieces.

Fracture, for most of the notch bend test-pieces, was microstructure-initiated. The fracture process is clearly not instantaneous. There is an initiation and growth process whereby weak points in the microstructure - such as WC/WC interfaces or cracked WC grains - link through fracture of Co ligaments until a crack has been formed that is of a critical size to propagate quickly. It is possible to calculate the size of this critical defect for each of the materials tested by notch bend [25].

A schematic diagram is shown in Fig. 20 of three possible sources of fracture in the test:

- a) Microstructure-initiated. A deformation zone grows to form a critical semicircular crack of diameter $2C$. To aid discussion the term "characteristic length" is given to the value of C .
- b) Surface defect-initiated. A semicircular defect is present at the surface at the notch root. The diameter of the defect is $2a$. At fracture a crack grows to diameter $(2C + 2a)$ and initiates fast fracture at this point.
- c) Subsurface defect-initiated. A circular defect is present below the surface of the notch root. The diameter of the defect is $2a$. At fracture a crack grows to diameter $(2C + 2a)$ and initiates fast fracture at this point.

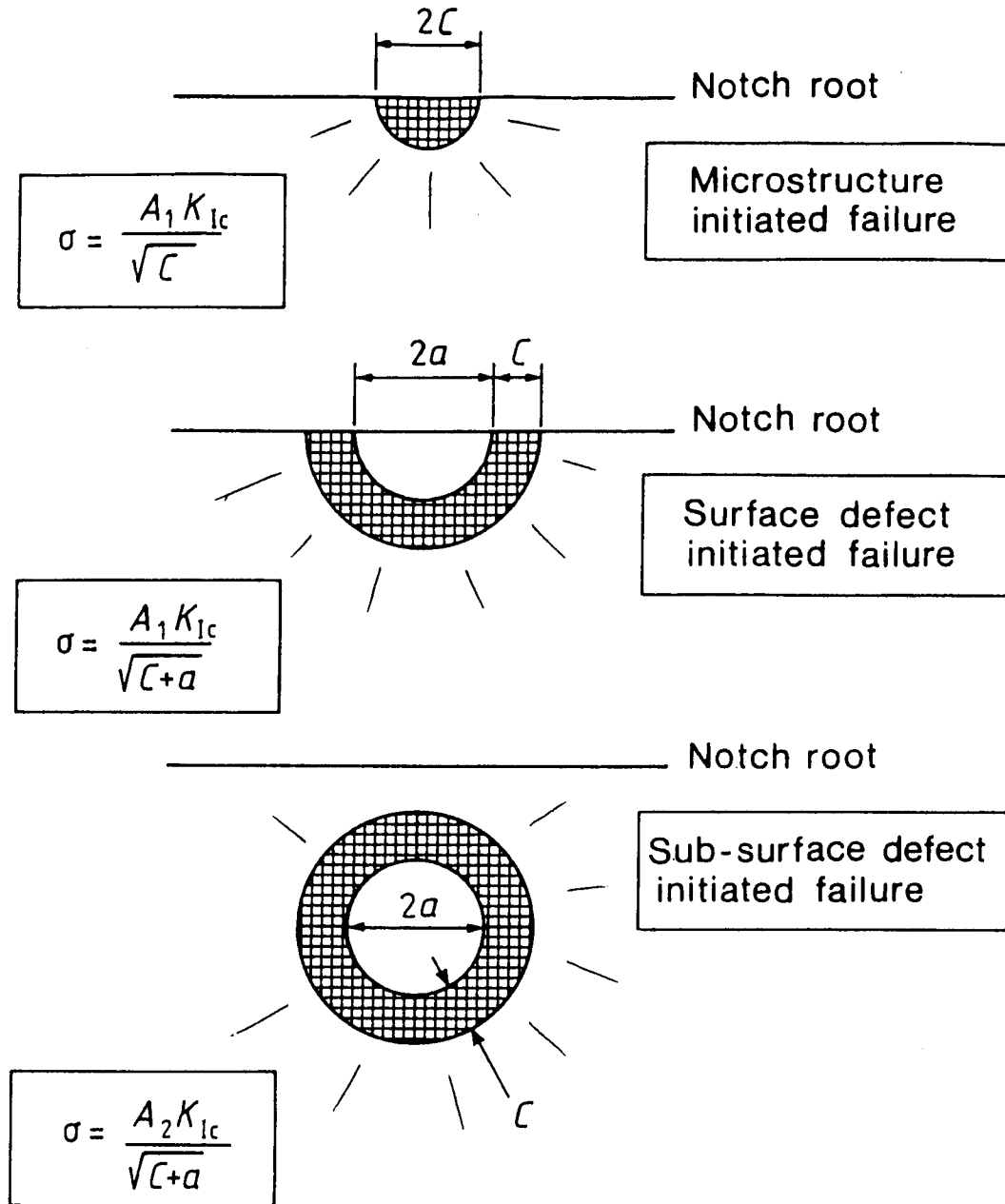


Figure 20 Schematic diagram of failure source geometries.

The failure stresses for each of these three cases are given by the standard fracture mechanics formulae relating failure stress, σ , to fracture toughness, K_{Ic} .

a)
$$\sigma = \frac{A_1 K_{Ic}}{\sqrt{C}}$$

(30)

b)
$$\sigma = \frac{A_1 K_{Ic}}{\sqrt{a+C}}$$

(31)

c)
$$\sigma = \frac{A_2 K_{Ic}}{\sqrt{a+C}}$$

(32)

where A_1 and A_2 are constants and K_{Ic} is the plane strain fracture toughness.

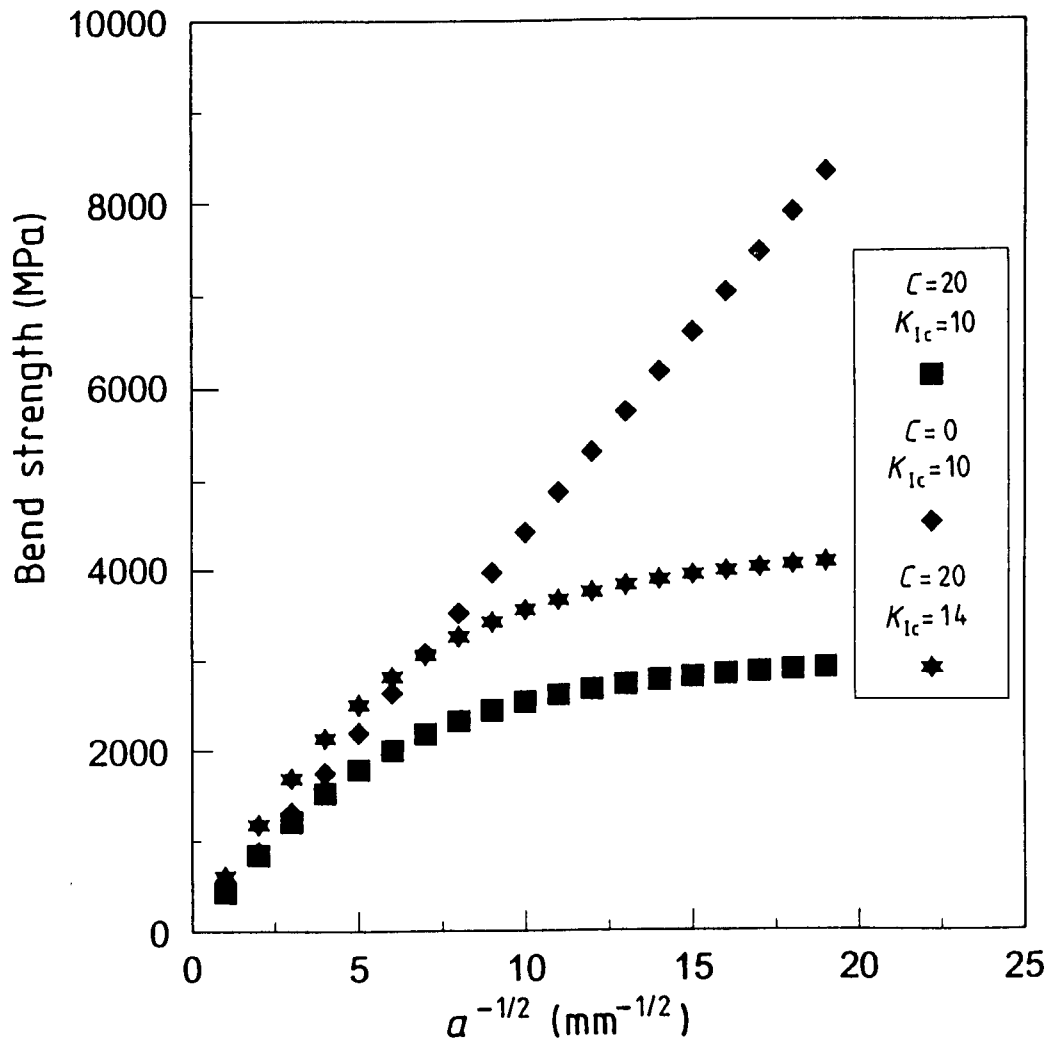


Figure 21 Theoretical plot of failure stress against characteristic length.

A plot of expression (31) is shown in Fig. 21 for various combinations of K_{Ic} and C [25]. If there were no deformation (crack initiation) process, to grow a defect from diameter a to $a + C$, then strength would continue to increase as the defect size decreased (as shown in Fig. 21). However, previous work at NPL has shown that in bend tests the strength reaches a "limiting" value and that beyond this value there is no increase in strength as the defects present in the material decrease in size. Clearly expression (31) gives theoretical support to this experimental observation, since it predicts that strength reaches an asymptotic value as the defect size, a , becomes equal to or less than the size of the characteristic length, C .

4.4 Fracture toughness

Many hardmetal products 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 residual strength of hardmetal components. Fracture toughness increases with increasing Co content and increasing WC grain size. However, there is no standard test method for obtaining accurate values of fracture toughness. This is particularly important for materials with toughness values greater than about $15 \text{ MN m}^{-3/2}$ that are difficult to pre-crack (a prerequisite for a valid toughness test). The different available test methods are about to be examined in a VAMAS interlaboratory exercise. Until the results of this project have been analysed it is difficult to give detailed recommendations on toughness testing.

The different options are therefore discussed without a preferred method being indicated.

4.4.1 Fracture toughness measurement methods

There are several methods for the measurement of fracture toughness, **none of which are standardised**. The results are usually expressed in $\text{MN m}^{-3/2}$. The range of values for typical WC/Co hardmetals is from 7-25 $\text{MN m}^{-3/2}$.

Toughness is a term which would benefit from a tighter usage controlled by carefully defined terminology. When applied to hardmetals as an unqualified word it can have several meanings:

- Plane strain fracture toughness, K_{Ic} - a value obtained from tests on a specimen with a well defined geometry of crack. There is no standard method for hardmetals and different organisations use different test methods. The result is expressed in $\text{MN m}^{-3/2}$. Fracture toughness is defined as the resistance to propagation of a pre-crack of known size; for plane strain fracture toughness this is given by:

$$K_{Ic} = Y\sigma_f \sqrt{c} \quad (33)$$

where Y is a geometrical parameter, c is the crack length and σ_f is the applied stress normal to the crack surfaces.

- Strain energy release rate, G - an alternative expression for toughness, usually obtained by converting plane strain toughness, K, to G $\{G = K^2/E (1 - \nu^2)\}$, where ν is the Poissons ratio}. It has units of J m^{-2} . Again there is no standard method.
- Palmqvist toughness, W_G - a value obtained by measuring the crack lengths at the corners of

a Vickers indentation (see section 4.4.1.1). It can be evaluated by making indentations either at a single load or from the inverse of the slope of load against crack length for a range of applied loads. It is expressed in N m^{-1} , which is equivalent to J m^{-2} . There is no standard method for measuring the crack lengths. This can be performed, a) - by taking away the indentation diagonals from the crack tip to tip measurement or b) - by measuring each crack from root to tip individually. However, it is widely recognised that surfaces on which the measurements are made should be annealed to remove the effects of residual stresses.

- Palmqvist fracture toughness, W_K - a value obtained from W_G using the formulae [30-32]:

$$W_K = A\sqrt{HV} \sqrt{W_K} \quad (34)$$

where m is a constant, HV is the hardness and W_K is expressed in $\text{MN m}^{-3/2}$ (see section 4.4.1.1). For conventional hardmetals a good correlation is claimed between K_{Ic} and W_K [30], but this remains to be confirmed by validated tests according to an agreed standard method.

- Finally, toughness is also widely used, in a loose sense, to describe the empirical relation between perceived resistance to dynamic impact or impacts. This is neither standardised or quantified.

There is a general inverse trend of hardness against fracture toughness. Plane strain fracture toughness data (K_{Ic}) obtained from tests on a wide range of WC/Co hardmetals at NPL are shown in Fig. 22 together with a wide range of published data from Sandvik Hard Materials [33] plotted against hardness. The two sets of data are not strictly comparable because of the lack of an agreed test method. However, both show the general trend of decreasing toughness with increasing hardness. There are few data above 1800 HV30. Clearly, it is therefore difficult to predict from currently available data whether plane strain fracture toughness will continue to decrease with decreasing grain size or will reach an asymptotic value as discussed previously [9].

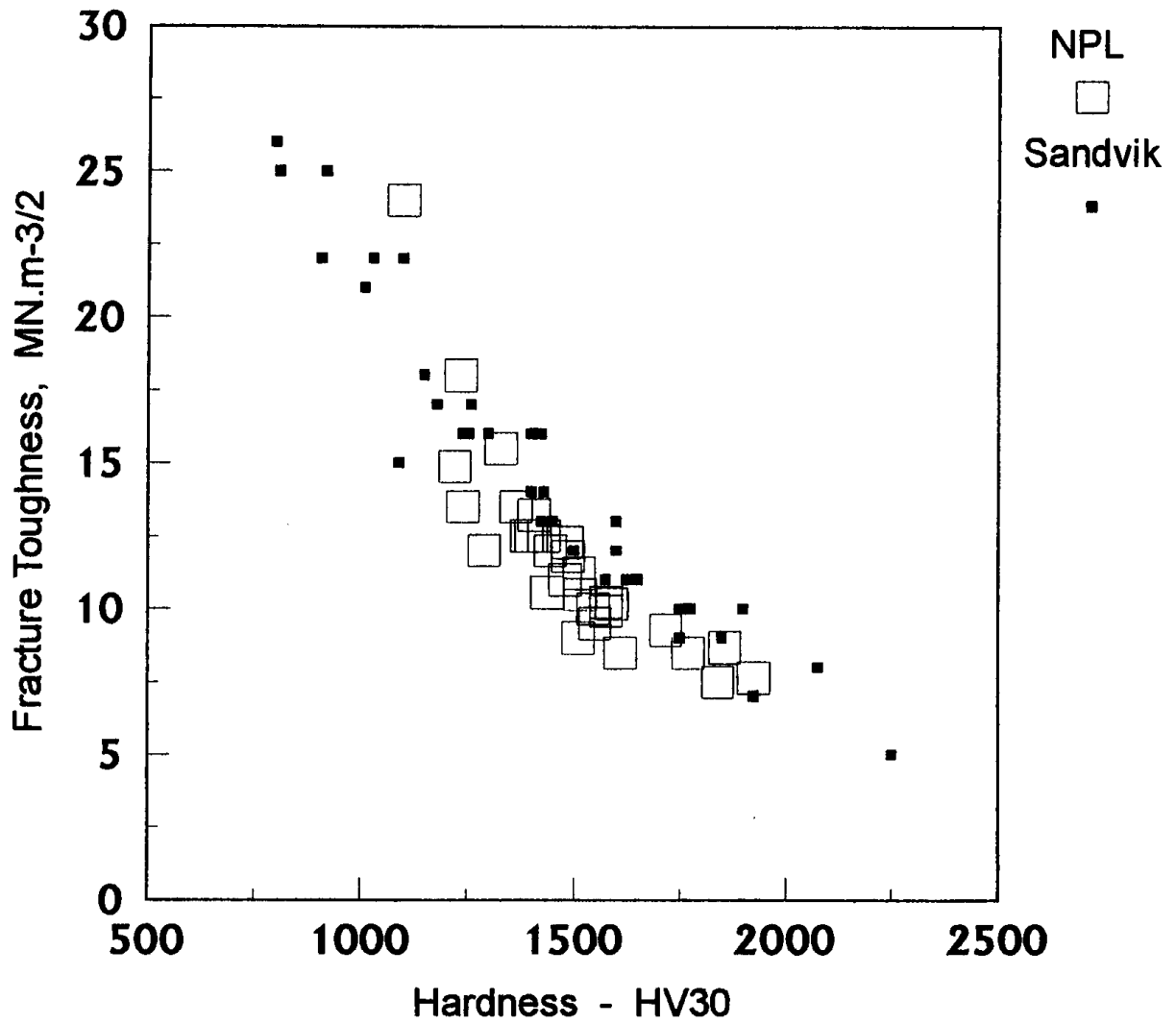


Figure 22 Fracture toughness variation with hardness, HV30.

For low toughness materials there are many possible test methods [34] 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. To be evaluated in VAMAS interlaboratory exercise.
- **SENB** Single Edge Notched Beam. Beam with notch. Results depend on notch width and method of preparation. Not 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. To be evaluated in VAMAS interlaboratory exercise.
- **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 $10\text{-}16 \text{ MNm}^{-3/2}$ provided that the surface is free from residual stresses. Tougher materials produce few or inconsistently sized cracks. To be evaluated in VAMAS interlaboratory exercise.
- **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. Need to be careful with residual stresses. To be evaluated in VAMAS interlaboratory comparison.

The IF (Palmqvist) and SEPB tests are discussed in more detail in the next Sections 4.4.1.1 and 4.4.1.2.

4.4.2 Palmqvist toughness tests (indentation fracture (IF))

Palmqvist tests are widely used to assess the toughness of hardmetals since only small amounts of material are used and the difficulties of pre-cracking conventional toughness test-pieces are bypassed. 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 test-piece preparation procedure [35,36]. 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 °C for 1h is generally used for Palmqvist tests following the method outlined by Exner [35]. This procedure should be re-examined, however, for very fine grained hardmetals. Test-pieces may need to be annealed at higher temperatures to ensure complete removal of residual stresses.

A Good Practice Guide for Palmqvist tests has been produced [37] and this section highlights important issues.

At NPL Palmqvist indentation results are generally obtained by making two indentations, each at a load of 30 kgf (294.3N), using an accredited Vickers Hardness Machine with a mass of 30 kg on polished surfaces of each test-piece of interest. The indentations and cracks are measured on an optical microscope at a calibrated magnification of x515. Hardness values are obtained from the indentation diagonals. The estimated uncertainty in diagonal is $\pm 1 \mu\text{m}$ and in total crack length $\pm 20 \mu\text{m}$ [36]. A schematic diagram is shown in Fig. 23.

Two methods can be used for calculating Palmqvist toughness

- W_G - in this method the toughness is calculated from the ratio of indent load, P , to total crack length, T

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

(35)

- W_K - in this method the toughness is calculated as follows

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

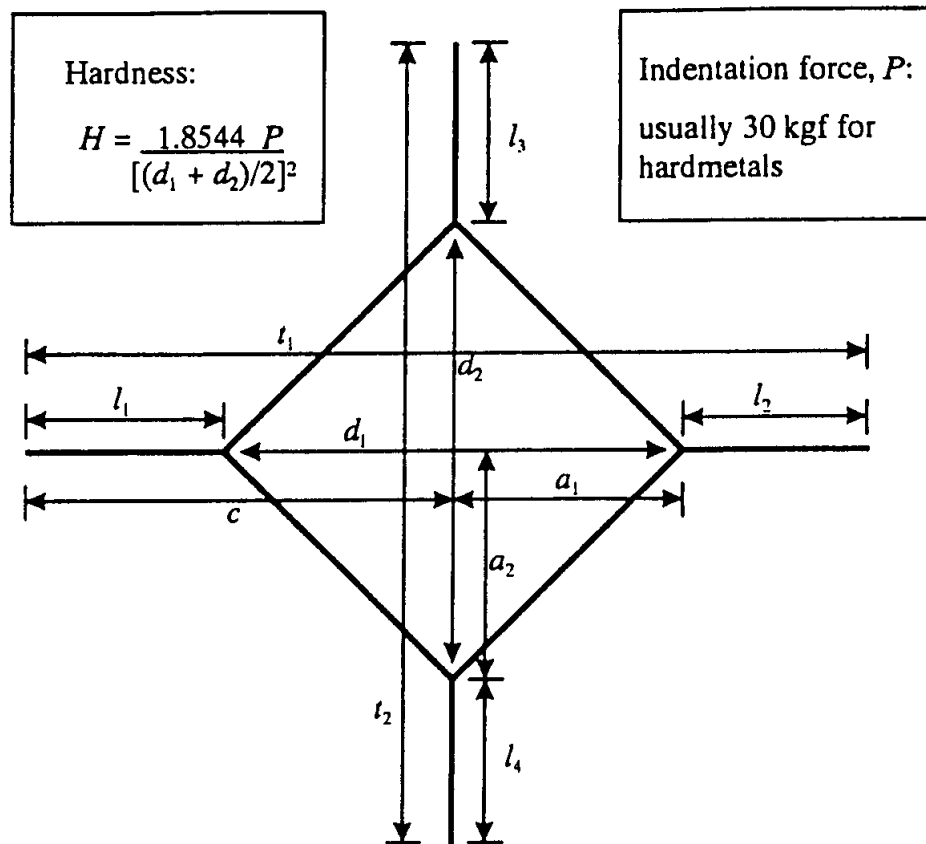
(36)

following the analysis discussed by Warren and Matzke [30], Shetty *et al.* [31] and Spiegler *et al.* [32], 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 (35).

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.

Palmqvist Toughness, W_G , Definitions

Alternative methods measuring crack length and toughness, W :

- Measure crack tip to crack tip, $t_1 = (l_1 + l_2 + d_1)$, $t_2 = (l_3 + l_4 + d_2)$
Measure indentation diagonals, d_1 , d_2
 $W_G = P/[(t_1 - d_1) + (t_2 - d_2)]$
- Measure crack lengths l_1 to l_4 from tip to indentation corner
Total crack length, $T = l_1 + l_2 + l_3 + l_4$ or $(t_1 - d_1) + (t_2 - d_2)$
 $W_G = P/(l_1 + l_2 + l_3 + l_4)$

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

Figure 23 Schematic diagram and definition for Palmqvist Test Method.

Recent work [36, 37, 38, 39] has shown that the uncertainties associated with the Palmqvist test generally range from about ± 0.5 to $0.1 \text{ MN m}^{-3/2}$, dependent on hardness in the range 1400-2000 HV30 respectively. At lower hardness levels, the standard deviation increases significantly for calculations of toughness from the formula, $W_K = A\sqrt{HV}\sqrt{W_G}$. These values of uncertainty can be compared with an estimated uncertainty of about $\pm 0.1 \text{ MN m}^{-3/2}$ for plane strain toughness tests on more conventional fracture toughness test-pieces [37-39].

In summary, toughness data quoted for hardmetals must be considered very carefully since there are no standard methods. Differences of less than 50 N mm^{-1} (W_G), $1.5 \text{ MN m}^{-3/2}$ (W_K) or $0.5 \text{ MN m}^{-3/2}$ (K_{Ic}) are not likely to be significant in low hardness materials.

4.4.3 SEPB fracture toughness tests

For pre-cracked SEPB (single edge notched beam) test-pieces the value of K_{Ic} is related to the nominal applied bending moment, M , and crack length, a , by:

$$K_{Ic} = \frac{6M}{BW^2} \sqrt{\pi a} f(a/W) \quad (37)$$

where, a is the crack length, B and W are the test-piece 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 are calculated using the compliance derived $f(a/W)$ and the Brown and Srawley 3 pt bending formula for $L/W = 4$ where:

$$f(a/W) = 1.09 - 1.73(a/W) + 8.2(a/W)^2 - 14.2(a/W)^3 + 14.6(a/W)^4 \quad (38)$$

The crack lengths are generally measured on the fracture face after testing to failure. The cracks can easily be seen because 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 has no effect on the measured value of K_{Ic} . The test method can be performed along guidelines recommended in a NPL report [40].

Five test-pieces of each batch are usually precracked using the wedge indentation method, which has been shown to produce stress free cracks of controlled length. Other techniques are permissible such as fatigue crack growth or the square indentation method [41]. For pre-cracked single edge cracked beam (SEPB) specimens, the value of K_{Ic} is related to the nominal applied bending moment, M , and crack length, a , by:

$$K_{Ic} = \frac{PS}{BW^{1.5}} f(a/W) \quad (39)$$

where B and W are the test-piece 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.

In tests at NPL a 4 pt bend rig is sometimes used with values for S of 9.8 mm, for W of about 7 mm and for B of about 2 mm. This is a non-standard geometry. Consequently, the correct value to use for $f(a/W)$ is not known. Three functions were examined, f_1 , f_2 and f_3 , with the following values:

$$f_1 = \frac{3(\alpha)^{1/2} (1.99 - (\alpha - \alpha^2)) (2.15 - 3.93\alpha + 2.7\alpha^2)}{2(1 + 2\alpha)(1 - \alpha)^{3/2}} \quad (40)$$

$$f_2 = 3(\alpha)^{1/2} (1.99 - 2.47\alpha + 12.97\alpha^2 - 23.17\alpha^3 + 24.8\alpha^4) \quad (41)$$

$$f_3 = \frac{3(\alpha)^{1/2}}{(1 - \alpha)^{3/2}} \left(1.9887 - 1.326\alpha - \left(\frac{\alpha(1 - \alpha)(3.49 - 0.68\alpha + 1.35\alpha^2)}{(1 + \alpha)^2} \right) \right) \quad (42)$$

where $\alpha = a/W$.

f_1 is the polynomial used in the current ASTM standard for 3 point bend tests on metallic materials, f_2 is that used in an earlier standard for 3 point bend tests and f_3 is the value to use when the stress state is one of pure bend.

Secondly, toughness values calculated from the use of the Brown and Srawley compliance function (expression (40)) gave typical mean value and standard deviations of $14.0 \pm 0.5 \text{ MN m}^{-3/2}$. Although the number of tests is low the results are reasonably consistent. The uncertainty associated with measurement of crack length and load gives an uncertainty of about $\pm 0.5 \text{ MN m}^{-3/2}$ which is comparable with the calculated standard deviations. The values are substantially higher, by about 30%, than the values calculated from the indentation.

4.4.4 Comparison of methods

The effects of two different test methods (Palmqvist and Single Edge Cracked Beam, SEPB) on fracture toughness results are shown in Fig. 24 [39]. Features to note are:

- At all values of toughness the Palmqvist results show more scatter;
- At low values the Palmqvist data are higher than the SEPB data, probably because of the difficulty in measuring the Palmqvist crack lengths;
- At intermediate values of toughness the agreement is reasonable;
- At high values of toughness it is difficult to obtain good Palmqvist toughness data because frequently cracks are not produced at the indentation corners, or if they are, they are ill-

formed and difficult to measure.

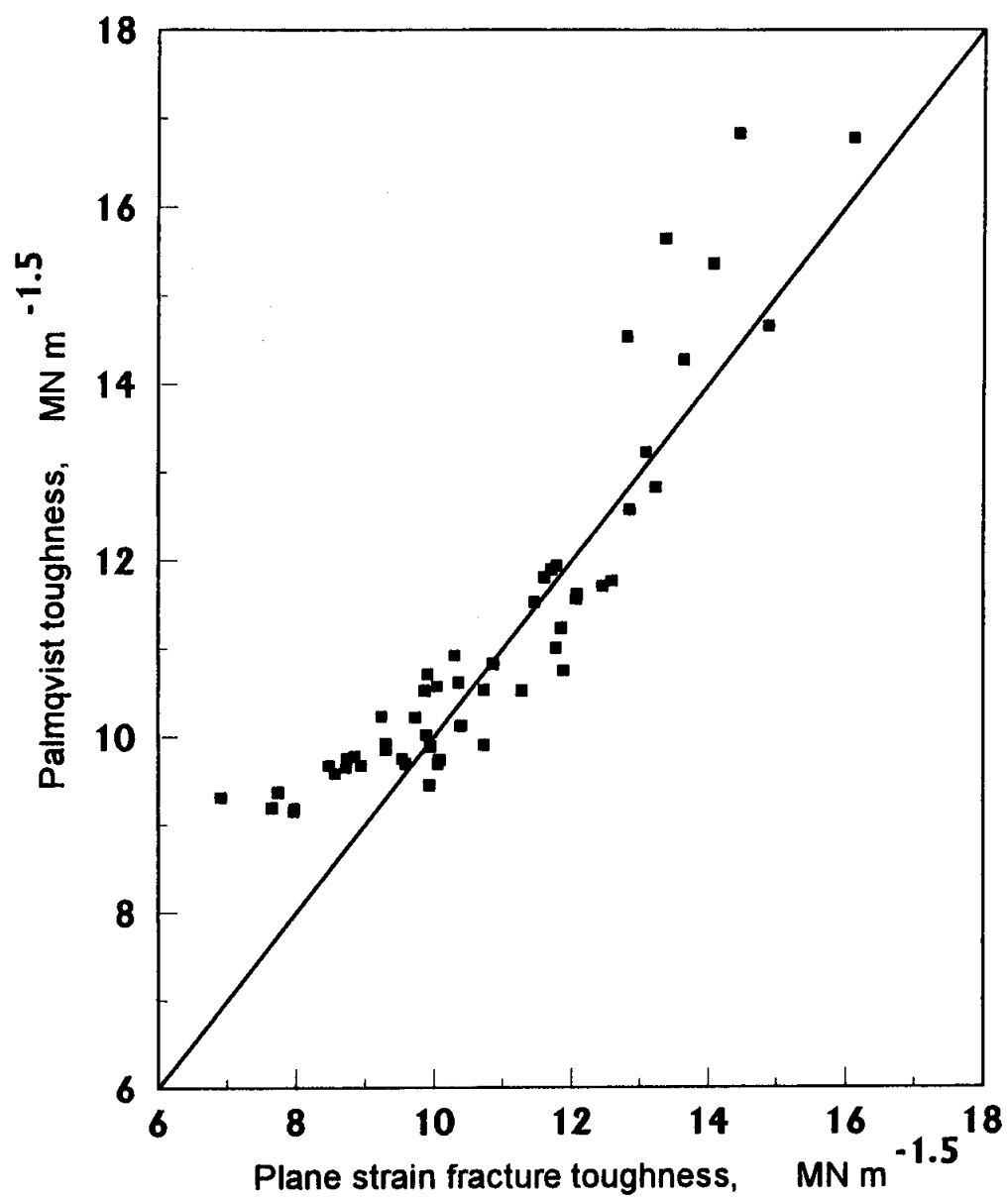


Figure 24 Comparison of Palmqvist and plane strain toughness data.

5 Additional tests

Apart from the four test methods discussed in the main body of this guide the property review in Section 2 lists the following mechanical test requirements

- Stiffness*
- Fatigue strength
- High temperature properties*
- Impact strength
- Compressive strength*

** The remainder of the guide addresses the starred test methods, but in less detail than was the case for hardness, abrasion, strength and toughness. These methods are less well researched and it is not yet appropriate to give detailed guidance. Fatigue and impact tests are so little understood for hardmetals that they have been omitted completely. There is on-going work at NPL to investigate these two test methods to remedy this gap in test method understanding.*

5.1 Stiffness

The elastic properties of hardmetals are important to their performance since they control the extent of localised deflection in many components, *i.e.* long narrow gundrills, cutting tools, knives etc. WC/Co hardmetals are very stiff and Young's Modulus values lie in the range 450-650 kN mm⁻². Hardmetals are elastically isotropic, being made from small equiaxed particles of transition metal carbides. WC/Co hardmetals especially are typical composites in that their elastic properties can be tailored by varying the binder-phase volume fraction. The higher the amount of Co binder-phase the lower the stiffness.

There is a standard for the measurement of stiffness, EN 23312.

This standard uses the resonant frequency of round or rectangular rod at least 60 mm long (see below). Round test-pieces should be 6 mm in diameter and rectangular bars, 6 x 8 mm. Density (ρ) is also measured and Young's Modulus (E) is calculated from:

$$E = 4 \times 10^{-9} L^2 \rho f^2 \quad (43)$$

where f is the resonant frequency and L is the test-piece length.

Alternative methods have been developed for ceramics (summarised in Good Practice Guide No. 19 - R Morrell, June 1999, [34]). These include:

- **Flexure of a beam**

For results with less than 1% uncertainty, beams need to be long and thin, *e.g.* 1 x 10 x 600

mm, and strain should be preferably measured with strain gauges rather than deflection transducers. Transverse as well as longitudinal gauges allow Poisson's ratio (ν) to be measured as well as Young's Modulus (E).

- **Beam resonance (impact excitation)**

For ceramics results with better than 1% uncertainty can be obtained from measurements of the frequency of vibrating or vibrated beams. Disc test-pieces can also be used and these provide data to measure E and ν .

- **Ultrasonic time of flight**

By using suitable transducers longitudinal and transverse mode waves can be detected which allows the calculation of values for E , ν and G (shear modulus). This technique provides results with better than 1% uncertainty for reasonably sized (20 mm long x 5 x 5 mm cross section test-pieces) hardmetal test-pieces.

5.1.1 Effect of Co content on Young's Modulus

A resonance method was used by Doi *et al.* [42] to evaluate the effect of Co content (volume fraction, V_{Co}^f) on the Young's Modulus (E). Doi found that WC grain size had little effect on the results but increasing Co content decreased E substantially. Examination of the data in Doi's paper gives the following expression for E in terms of V_{Co}^f

$$E = 708 - 821V_{Co}^f + 412(V_{Co}^f)^2 \quad (44)$$

This expression is plotted in Fig. 25.

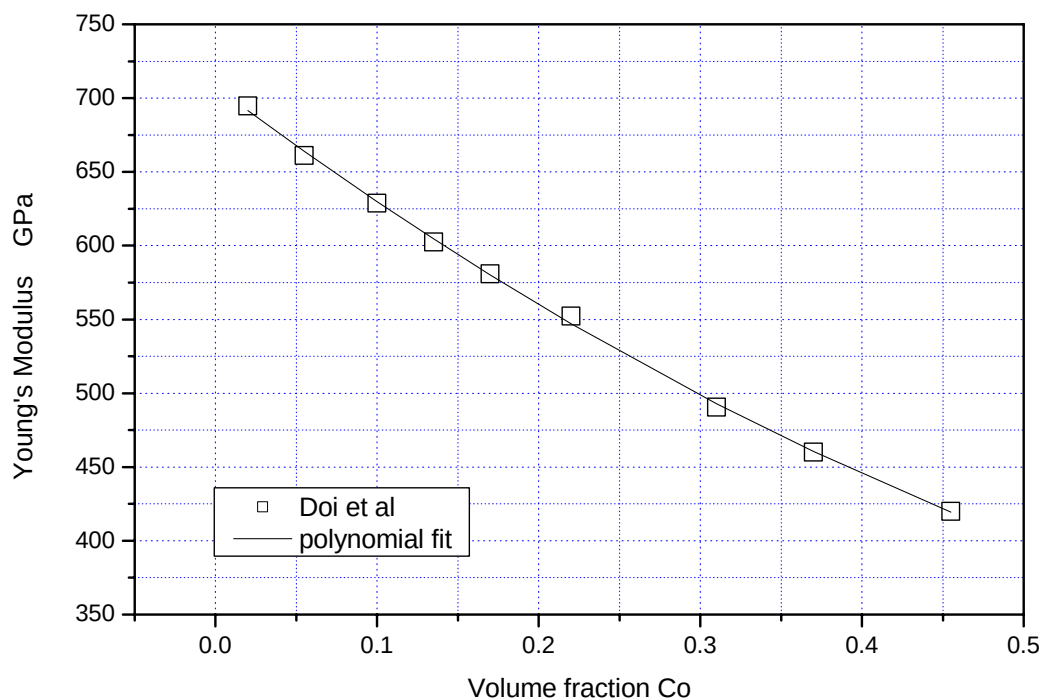


Fig. 25 Effect of Co volume fraction on the Young's modulus of WC/Co hardmetals.

5.2 Compression Tests

There is a standard for compression testing of hardmetals, ISO 4506.

The standard recommends cylindrical test-pieces with complex geometries which are quite large, about 40 mm long by 8 mm diameter (gauge length). These require large testing machines, as WC/Co hardmetals have typical compressive strengths of 4-8 kN mm⁻², i.e. about 300 kN to fracture. At these high loads the high stored energy in the test machine results in explosive fractures which are damaging to the test machine and anvils. It is thus a costly test and is not widely performed.

To minimise some of these problems tests at NPL have been performed on small cylindrical test-pieces, 8.74 mm long by 5.00 mm diameter. A hard-beam rig is used and the test-pieces deformed at a strain rate of about 10⁻⁵ s⁻¹. The test-pieces are strain-gauged and are generally compressed to a point just short of fracture (without an explosive failure because the stiff machine/rig stores less energy), characterised by a sudden load drop and one or more loud cracks, to determine their relative yield strengths and ductility at fracture. Experience has shown that a further small application of load past this point results in total fracture of the specimen [43].

Recently published work by Getting *et al.* [44] used a compression test fixture with thin lead disks inserted between the test-piece and the anvils (manufactured from hardmetal), to distribute loads evenly, and a binding ring to prevent premature failure from corners. Even so, failure was catastrophic, unlike the NPL stiff system, because of the large amount of stored energy in the system. Strain was also measured in these tests using strain gauges. Comparative strength data were obtained for many grades from different manufacturers and it was shown that the Ramberg-Osgood constitutive equation fitted these data very well up to 2% strain. At higher strains internal microcracking produced deviations from the theoretical fit. The Ramberg-Osgood expression relates total strain, ε_T , to applied stress, σ , by

$$\varepsilon_T = \sigma / E + (\sigma / a)^b$$

where E is the Young's modulus and a and b are constants.

5.3 High temperature properties

There are no standard tests for the high temperature mechanical properties of hardmetals.

Many hardmetals have excellent high temperature properties and this underpins their use as cutting and milling tools, saws, rolls and drills. However, it is particularly difficult to measure their high temperature strength because they are extremely strong (gripping systems are therefore problematical). Also, in many cases tests have to be performed in a vacuum or inert environments because they are not oxidation resistant (particularly WC/Co).

High temperature properties can be categorised as follows:

- **Short term strength, either creep or uniaxial**
- **Fracture toughness**
- **Thermal fatigue**

Each set of properties has different test method requirements. Most of these are still at the laboratory stage and have not reached the position where standards can be written. More intercomparison work remains to be performed before test methods can be harmonised to give agreed international test procedures. For this reason brief descriptions of the different test methods are given with an emphasis on the test parameters. Stiffness and its variation with temperature is also likely to be important but has not been studied extensively [45, 46]. It is likely that resonance methods [47] could be of most practical use, provided that test-pieces can be prevented from oxidation.

5.3.1 Short term strength and creep

Several test techniques have been used to measure short term strength and creep properties including:

- | | |
|-----------------------------------|-------------------------|
| ● Bend [48-54] | ● Hot hardness [55, 56] |
| ● Compression [46, 50, 52, 57-62] | ● Tension [63, 64] |

Bend tests have been perhaps the most extensively used method to evaluate high temperature properties. The test system must be able to withstand the high temperatures of operation, and for example Graphite, Molybdenum and Sintered Silicon Carbide fixtures have all been used to 1300 °C. Also, even when load and displacement transducers are engineered well enough to work effectively at high temperatures load-displacement traces are generally non-linear and the elastic equations that are used to calculate strength are invalid. Various techniques have been developed to obtain stress-strain data from non-linear load-displacement traces but these are complex and not yet validated.

Displacement should be measured at the test-piece if possible, rather than from any machine/test system ram movement. It is also difficult to compare results from different test systems because

different test-piece geometries have been used, *i.e.* usually rectangular bars with dimensions ranging from 4 x 8 x 24 mm to 5 x 2 x 20 mm to 4 x 2 x 30 mm to 7 x 3.5 x 35 mm, in either 3 point or 4 point bend rigs. It was generally found that mechanical behaviour was elastic at test temperatures up to 600 °C with increasing amounts of plasticity as the temperature was raised above this value. As plasticity increases properties become more and more strain-rate dependence. Therefore this parameter should ideally be controlled during the test and reported. Hardmetals, especially those based on WC/Co, can be prone to oxidation. Therefore it is necessary to conduct tests in a vacuum or inert atmosphere (argon).

For high temperature compression tests, again there are no standards, and tests have been reported on both cylinders (from 5 mm diameter by 8 mm long to 9 mm diameter by 11 mm long) and rectangular parallelepipeds (1 x 1 x 4 mm to 4 x 4 x 8 mm). In hot compression tests properties can be sensitive to test-piece dimensions because of constraint and friction effects at the test-piece ends. In addition to tests in vacuum and argon other inert gas mixtures have been used, for example CO + N₂, to minimise oxidation effects. Deformation, particularly if elastic effects are to be evaluated, should always be corrected for machine compliances and ideally displacements should be measured on the test-piece rather than at some other point (crosshead) in the test system. Measurements of diameter can also be used on cylindrical test-pieces to give direct strain measurements, provided that an optical path and suitable filters are available to image the test-piece [62]. Push rods and platens for use at high temperature must also be chosen with care to avoid shape changes, fracture and chemical interaction, especially at very high temperatures. Single crystal alumina is a typical example at >1000 °C and sialon has been used successfully in lower temperature tests (<1000 °C) at NPL.

Properties at high temperatures are probably most sensitive to the temperature of test. Very often, precise details of how this is measured are omitted from published papers. Also temperature can vary along the test-piece [62] and it is important to evaluate the extent of this and maintain any variation within reasonable limits, say better than ± 5 °C at 1000 °C.

Hot hardness tests have also been used for estimating the high temperature mechanical properties of a range of hardmetals. However, again the test requires test-pieces to be in a vacuum or inert environment. It is also a relatively time consuming test because the conventional method requires indentations to be measured after testing. An analysis was developed to allow creep data to be obtained from indentation creep experiments [55], *i.e.*

$$\frac{d\varepsilon}{dt} = \frac{1}{D} \frac{dD}{dt} = \frac{d \ln D}{dt} \quad (46)$$

where D is the indentation diameter, ε is strain and t is time. The test is likely to become more widely used when depth sensing systems become available that allow the depth displacements associated with plastic indentation to be measured directly in-situ at temperature, avoiding the requirement to measure indentations (that are sometimes difficult to find) after the test.

In principle, tension tests can overcome some of the difficulties of grips/platens and localised

deformation analyses associated with compression and bend tests. However, care has to be given to alignment as relatively small cross-section test-pieces (e.g. 1 x 3 mm) have been used. Also, the measurement of strain is just as difficult at high temperatures on tensile test-pieces as it is in other modes of test.

Some of these difficulties in short term strength tests have been overcome by the development of a miniature test system [65, 66] at NPL that allows tension, compression, yield strength and creep data to be obtained quickly on rectangular test-pieces (typically 2 x 1 x 40 mm in dimensions) that are heated using a d.c current in an environmental box through which argon is flushed to prevent oxidation (Fig. 26).

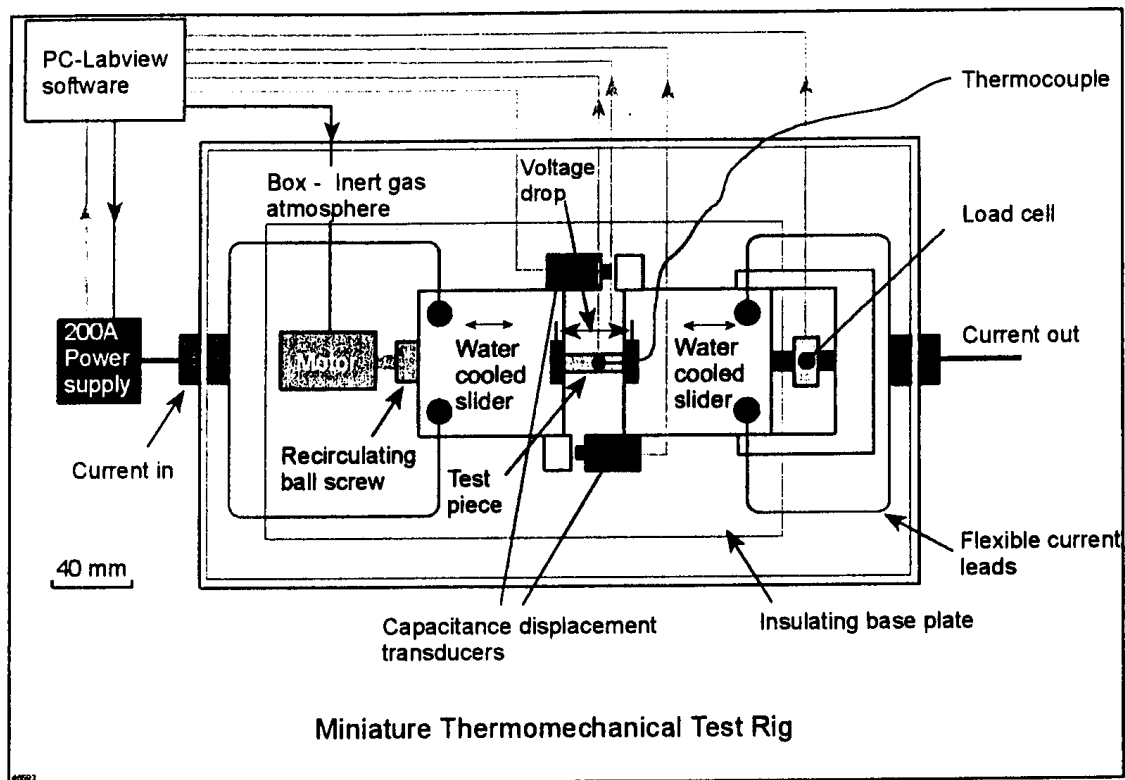


Figure 26 Schematic diagram of miniature thermomechanical test system.

5.3.2 Fracture toughness

High temperature fracture toughness data has been reported from bend tests [67-69], but by three different methods, characterised by differences in the test-piece geometry, methods of pre-cracking and analysis of results. All methods involve measuring the load / displacement trace. It is important, if possible, to measure displacement on the test-piece rather than at some other point in the test system. A good method is given by Warren [68] where three transducers are used (two for reference, one for difference) in contact with the bottom tensile surface of the bend test-piece. The three different pre-cracking methods included:

- direct pre-cracking using the bridge method [70];
- chevron notch method [71, 72];
- electro-discharge machined notch.

All the same constraints that apply to direct strength tests are also applicable, *i.e.* controlled atmosphere to inhibit oxidation and careful temperature measurement.

Tests usually indicate plane strain behaviour at low temperatures with increased plasticity and stable crack growth as temperature increases. The method of analysis is thus critical to the fracture toughness parameter measured and there was little consistency between the different reported methods of analysis. Clearly there is scope for discussion and harmonisation of practice in precracking methods, test-piece geometries and analysis of load/displacement traces to give agreed high temperature toughness values.

5.3.3 Thermal fatigue

Many hardmetal components fail by a process of thermal fatigue, *i.e.* hot rod rolls develop cracks in the roll gap that require expensive roll regrinds. This is a particularly difficult property to measure because of the high strength of the materials, the low strains to failure and the environmental sensitivity puts high demands on suitable testing rigs.

Recently two groups in the UK/USA [65, 66] and Germany [73] have reported data from thermal fatigue tests using different test techniques. The UK/USA collaboration used the NPL ETMT referenced in Section 5.3.1 and found that the test system discriminated well between different grades in S-N fatigue behaviour with load cycles superimposed on temperature cycles or with either load and temperature constant with the other parameter cycling. Discriminating data were also reported by the German group using a halogen lamp to heat long rods of hardmetal 80 x 3 x 4 mm in dimensions. The tests were done in air however, whereas the ETMT allowed tests in Argon. No frequency dependence of S-N data was found between 2 and 22 Hz. S-N data at different temperatures were generated and fatigue behaviour was shown to be temperature sensitive. Both test systems require considerable numbers of test-pieces to generate statistically relevant S-N data for all the different test conditions. There is clearly scope for accelerated test methods where perhaps loads are step-ramped to derive data for material comparisons, as opposed to S-N data for fatigue design.

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