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## Hårdmetall – Palmqvist hårdhetsprovning (ISO 28079:2009, IDT)

## Hardmetals – Palmqvist toughness test (ISO 28079:2009, IDT)

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Den internationella standarden ISO 28079:2009 gäller som svensk standard. Detta dokument innehåller den officiella engelska versionen av ISO 28079:2009.

The International Standard ISO 28079:2009 has the status of a Swedish Standard. This document contains the official version of ISO 28079:2009.



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# Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 28079 was prepared by Technical Committee ISO/TC 119, *Powder metallurgy*, Subcommittee SC 4, *Sampling and testing methods for hardmetals*.

# Introduction

Good test methods are those which enable a user or manufacturer to clearly discriminate between different materials.

Fracture toughness values are required for three reasons:

a) for product design and performance assessment;

b) for selection of materials;

c) for quality control.

A specific International Standard for the toughness of hardmetals [1)] has not been developed to date, primarily because of the difficulty of introducing stable precracks into these tough but hard materials. However, Palmqvist tests for toughness are widely used because of their perceived apparent simplicity. Cracks are formed at the corners of Vickers hardness indentations and these can be used to calculate a nominal surface toughness value. This value is sensitive to the method of measurement and to the method of surface preparation of the sample. This International Standard outlines good practice to minimize uncertainties due to these issues.

There are several possible methods for the measurement of the fracture toughness of hardmetals. The results can be expressed either as a stress intensity factor, in $MN{\cdot}m^{-3/2}$, or as a fracture surface energy, in $J{\cdot}m^{-2}$. The range of values for typical WC/Co hardmetals is from 7 $MN{\cdot}m^{-3/2}$ to 25 $MN{\cdot}m^{-3/2}$. There is a general inverse trend of hardness against fracture toughness (see [1] and [2] in the Bibliography).

When applied unqualified to hardmetals, “toughness” can have several meanings.

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

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

c) Palmqvist toughness, $W$, is a value obtained by measuring the total length of cracks emanating from the four corners of a Vickers hardness indentation. For a given indentation load, the shorter the crack, the tougher the hardmetal.

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

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

There is a considerable body of published information on Palmqvist toughness tests for hardmetals (see [5] to [29] in the Bibliography). Palmqvist toughness, $W$, is a toughness value obtained by measuring the crack lengths at the corners of a Vickers indentation. It can be evaluated by making indentations either at a single load, usually 30 kgf, or from the inverse of the slope of a plot of crack length against load for a range of applied loads. For hardmetals, the crack depth profile is normally of the Palmqvist type, i.e. independent shallow arcs emanating from each indentation corner. The measurement of surface crack length is, however, open to operator error. It is widely recognized that test surfaces are carefully prepared to remove the effects of residual surface stresses (see [8] in the Bibliography). The test also has a poor fracture-mechanics pedigree because of the uncertainties associated with residual stresses introduced by the indentation.

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

This International Standard is based on a “Good Practice Guide for the Measurement of Palmqvist Toughness” published by the UK National Physical Laboratory in 1998. This International Standard recommends good practice to minimize levels of uncertainty in the measurement process. The procedure has been validated through underpinning technical work within the VAMAS [2)] framework (see [29] in the Bibliography). An interlaboratory exercise was conducted to generate underpinning technical information on toughness tests for hardmetals. More than ten industrial organizations participated, either by correspondence, supply of materials or by conducting tests. Eight organizations were able to complete Palmqvist tests. Good statistics were obtained on the Palmqvist data that enabled a quantitative assessment of uncertainties to be performed for this relatively simple test. Single-edge precracked beam data was thought to be closest to the “true” value, and the mean values from the Palmqvist test data compared reasonably well with these results. However, care was needed in test piece preparation to ensure a good correlation between data from the Palmqvist tests and the single-edge precracked beam results.

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

# Hardmetals — Palmqvist toughness test

## 1 Scope

This International Standard specifies a method for measuring the Palmqvist toughness of hardmetals and cermets at room temperature by an indentation method. This International Standard applies to a measurement of toughness, called Palmqvist toughness, calculated from the total length of cracks emanating from the corners of a Vickers hardness indentation, and it is intended for use with metal-bonded carbides and carbonitrides (normally called hardmetals, cermets or cemented carbides). The test procedures proposed in this International Standard are intended for use at ambient temperatures, but can be extended to higher or lower temperatures by agreement. The test procedures proposed in this International Standard are also intended for use in a normal laboratory-air environment. They are not intended for use in corrosive environments, such as strong acids or seawater.

## 2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 3878, *Hardmetals — Vickers hardness test*

## 3 Symbols and units

For the purposes of this document, the following symbols and units apply.

| Symbol | Designation | Unit |
|---|---|---|
| $A$ | Constant of value 0,002 8 | — |
| $d$ | Indentation-diagonal mean value | mm |
| $d_1$, $d_2$ | Indentation-diagonal individual values | mm |
| $E$ | Young's modulus | N·mm$^{-2}$ |
| $F$ | Indentation load (toughness calculations) | N |
| $G$ | Strain-energy release rate | J·m$^{-2}$ |
| $H$ | Hardness | kgf·mm$^{-2}$ |
| HV($P$) | Vickers hardness at load $P$ (kgf) | kgf·mm$^{-2}$ |
| $K_{\text{Ic}}$ | Plane-strain fracture toughness | MN·m$^{-3/2}$ |
| $\ell_n$ | Crack length at indent corner | mm |
| $P$ | Indentation load (Vickers hardness method) | kgf |

| Symbol | Designation | Unit |
|---|---|---|
| $T$ | Total crack length | mm |
| $t_n$ | Tip-to-tip crack length | mm |
| $W_G$ | Palmqvist toughness | $N \cdot mm^{-1}$ or $J \cdot m^{-2}$; |
| | | $1\ N \cdot mm^{-1} = 1\ 000\ J \cdot m^{-2}$ |
| $W_K$ | Palmqvist fracture toughness | $MN \cdot m^{-3/2}$ |
| $\nu$ | Poisson's ratio | — |

# 4 Test pieces and sample preparation

## 4.1 Test piece size and sampling

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

Diamond slicing or electrospark discharge machines are convenient to use for this purpose. However, the surfaces shall then be polished. It is recommended that 0,2 mm of material be removed before the final polish to ensure that material typical of the bulk is tested. For example, the ISO Vickers hardness test for hardmetals (see ISO 3878) specifies the removal of 0,2 mm.

NOTE It has also been suggested, in a dissertation by M. Heinonen (UMIST) [19], that the test piece should be at least as thick as ten times the crack length. Thinner test pieces might not give representative results because the stress state will be dependent on the amount of material supporting the indentation and its associated cracks. It can be convenient to mount the test pieces in hot-setting resins to directly provide flat and parallel faces. However, if the test pieces are to be subsequently annealed to remove surface residual stresses then this can be a disadvantage, since the test piece has to be removed from the mount to put it in the annealing furnace (typically 800 °C for 1 h in a vacuum).

## 4.2 Surface preparation

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

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

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

## 4.3 Surface condition

It has been shown that surfaces free of residual stress are required for consistent results (see [8] in the Bibliography). No polishing procedure can guarantee a stress-free surface without tedious systematic measurements which are not feasible on a regular basis. Usually, test pieces are polished so that the microstructure can be observed and then annealed at 800 °C for 1 h in a vacuum, following the studies published by Exner [8].

NOTE The newer grades of material with very fine WC grain sizes (less than about 0,8 µm as measured by the linear intercept technique on polished and etched sections) developed in recent years since Exner's work are likely to have even higher surface residual stresses. It might be that longer annealing times or even higher temperatures are required for these materials. It would probably be sensible to recommend a higher temperature, but future research is needed to confirm this recommendation. The annealing stage adds to the complexity of the sample preparation process, but ensures that the surface is free from residual stresses.

If measurements are performed on as-polished surfaces without an anneal, this shall be indicated in the test report.

# 5 Apparatus

## 5.1 General

The indentations shall be introduced into the test piece of interest using test machines calibrated according to national standards. The shape of the indentation should be checked regularly for damage to the indenter tip. The diagonal and crack dimensions can be measured using a microscope attached to the indentation test machine or separately, but it shall have been calibrated against a standard.

## 5.2 Indentation

Indentation shall be carried out on a Vickers hardness testing machine in accordance with the method in ISO 3878 for the Vickers hardness test and for verification of the Vickers hardness testing machine. A certified diamond indenter should be used and accredited by a national body.

## 5.3 Indentation and crack measurement

Indent diagonals and cracks should be measured using an accredited microscope. The image can be projected onto a screen which has been calibrated using a stage graticule traceable to national standards.

# 6 Procedure and conditions of testing

## 6.1 Indentations

Indentations shall be made in a deadweight hardness machine which is calibrated at least annually. The recommended procedure is to make indentations using a Vickers diamond indenter with one load, rather than a series of loads. The indentations should be made at 30 kgf or, if no cracks are visible at this load, at 100 kgf. Sometimes, even 100 kgf will not provide cracks. Until further research is conducted, this International Standard recommends that measurements at loads greater than 100 kgf are not valid. Two indentations shall be made initially and the toughness values for each indentation shall be compared. If they are within the measurement uncertainty associated with the procedure (see Clause 8), the two measurements are considered satisfactory. If they differ by more than this uncertainty, a third indentation shall be made and the result reported as an average with an associated standard deviation. If the two measurements are within the estimated measurement uncertainty, then an average value of the two measurements is reported without a standard deviation.

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

## 6.2 Indentation and crack length measurement

It is recommended that the indentation diagonal and crack lengths be measured optically at a magnification of at least ×500 (see Figure 1). The magnification used shall be calibrated for each measurement session using a traceable grid.