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Advanced technical ceramics – Monolithic ceramics – Mechanical properties at room temperature – Part 4: Vickers, Knoop and Rockwell superficial hardness

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Advanced technical ceramics - Mechanical properties of monolithic ceramics at room temperature - Part 4: Vickers, Knoop and Rockwell superficial hardness

Céramiques techniques avancées - Propriétés mécaniques des céramiques monolithiques à température ambiante- Partie 4: Essais de dureté Vickers, Knoop et Rockwell surperficiel

Hochleistungskeramik - Mechanische Eigenschaften monolithischer Keramik bei Raumtemperatur - Teil 4: Härteprüfung nach Vickers, Knoop und Rockwell

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EUROPEAN COMMITTEE FOR STANDARDIZATION
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Management Centre: rue de Stassart, 36 B-1050 Brussels

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Contents

Foreword

This document (EN 843-4:2005) has been prepared by Technical Committee CEN/TC 184 “Advanced technical ceramics”, the secretariat of which is held by BSI.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by December 2005, and conflicting national standards shall be withdrawn at the latest by December 2005.

EN 843 *'Advanced technical ceramics – Mechanical properties of monolithic ceramics at room temperature* ' consists of six parts:

Part 1: *Determination of flexural strength*

Part 2: *Determination of Young’s modulus, shear modulus and Poisson’s ratio*

Part 3: *Determination of subcritical crack growth parameters from constant stressing rate flexural strength tests*

Part 4: *Vickers, Knoop and Rockwell hardness tests*

Part 5*: Statistical analysis*

Part 6: *Guide for fractographic investigation*

This document supersedes ENV 843-4:1994.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Slovakia, Slovenia, Spain, Sweden, Switzerland and United Kingdom.

1 Scope

This part of EN 843 defines conditions for conducting, and provides guidelines concerning the value that may be ascribed to the results of, standard hardness tests when applied to advanced monolithic technical ceramics. It is assumed that the calibration and test procedures employed are exactly those for metallic materials. This European Standard refers to Rockwell A, Rockwell N-scale, Vickers, and Knoop hardness testing, as described in existing international standards.

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.

EN ISO 6507-1, *Metallic materials — Vickers hardness test — Part 1: Test method (ISO 6507-1:1997)*

EN ISO 6507-2, *Metallic materials — Vickers hardness test — Part 2: Verification of testing machines (ISO 6507-2:1997)*

EN ISO 6507-3, *Metallic materials — Vickers hardness test — Part 3: Calibration of reference blocks (ISO 6507-3:1997)*

EN ISO 6508-1, *Metallic materials — Rockwell hardness test — Part 1: Test method (scales A, B, C, D, E, F, G, H, K, N, T) (ISO 6508-1:1999)*

EN ISO 6508-2, *Metallic materials — Rockwell hardness test — Part 2: Verification and calibration of testing machines (scales A, B, C, D, E, F, G, H, K, N, T) (ISO 6508-2:1999)*

EN ISO 6508-3, *Metallic materials — Rockwell hardness test — Part 3: Calibration of reference blocks (scales A, B, C, D, E, F, G, H, K, N, T) (ISO 6508-3:1999)*

EN ISO/IEC 17025, *General requirements for the competence of testing and calibration laboratories (ISO/IEC 17025:1999)*

EN ISO 23878*, Hardmetals — Vickers hardness test (ISO 3878:1983)*

ISO 4545, *Metallic materials — Hardness testing — Knoop test*

ISO 4546, *Metallic materials — Hardness test — Verification of Knoop hardness testing machines*

ISO 9385, *Glass and glass-ceramics — Knoop hardness test*

ISO 14705, *Fine ceramics (advanced ceramics, advanced technical ceramics) — Test method for hardness of monolithic ceramics at room temperature*

OIML-36, *Verification of indenters for hardness testing machines*.[1)]

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[1] This international recommendation is available from the International Organization of Legal Metrology (OIML), 11, rue Tugot, 75009, Paris, France).

3 Terms and definitions

For the purposes of this European Standard, the following terms and definitions apply.

3.1
hardness
resistance displayed by a material to penetration by a hard indenter of defined geometry and forced into the test surface in a prescribed manner

3.2
hardness number
hardness calculated in a specified hardness test, usually without units specified, derived from the depth of penetration of the indenter or lateral dimension of the indentation, and the applied force

3.3
hardness indenter
hard device of defined geometry, and for the purposes of testing ceramics usually fabricated from single-crystal diamond

NOTE Types of hardness test are defined in clause 5, 6, and 7 for Vickers, Knoop and Rockwell tests respectively.

4 Significance and use

4.1 General points

The three types of test defined in clauses 5, 6 and 7 have been standardised for metallic materials, and are widely used as a guide to the state of thermal treatment or work-hardening. In advanced technical ceramics they are also widely used, especially to describe materials for applications in a wear environment. Whereas in a metal a hardness test is a measure of the yield stress, in a brittle material the deformation tends not to be homogeneous. In addition to plastic flow, there is usually some cracking and fragmentation occurring, the extent of which has a marked effect on the apparent hardness and the ability to perform meaningful measurements.

A hardness test on a range of widely differing ceramics will enable them to be ranked in order of resistance to localised penetration, which may be correlated with other behavioural characteristics of similar type, e.g. abrasive wear or erosion resistance. Such an interpretation may not be possible if materials show similar characteristics because the discrimination shown by hardness tests may be inadequate.

Uses beyond this application should be viewed with caution. It is, for example, recommended that hardness tests are not used as a pass/fail criterion in a specification. The potential differences between observers and/or test machines, as explained below, are too great for high levels of confidence in the test results, leading to possible dispute between parties to the specification.

4.2 Verification of test equipment

Hardness standard test blocks are usually supplied with the test machine. It is imperative that they be used for checking the machine behaviour and, in the case of Vickers and Knoop tests, also the visual criteria being employed by the operator for measurement. The test block should also be used to ensure that the indenter is free from chips or cracks which might easily develop when used on very hard materials. Very high hardness calibration blocks are recommended when testing ceramics.

The test force for hardness measurements on ceramics may not be the normal one for which the test machine has previously been calibrated. If this situation occurs, it is desirable to carry out checks that the intended force is actually being applied to the test surface for the required period of time.

Direct verification of test equipment is described in EN ISO 6507-2 (for Vickers tests), ISO 4546 (for Knoop tests) and in EN ISO 6508-2 (for Rockwell N-scale tests). The procedure involves calibration of force and of the reading system. This document does not deal directly with such issues.

Indirect verification of test equipment ensures that the equipment is reading correctly at the time of use, by providing a check on the quality of indentations, particularly whether there is damage to the indenter and whether the indenter is correctly aligned. It can also provide a check on the reading criterion being used by the operator. Indirect verification is normally conducted using certified reference blocks, and by ensuring that the indenter being used has previously been certified as being geometrically within the tolerances defined in the above standards.

Calibration of standard reference blocks is described in EN ISO 6507-3 (for Vickers tests), and in EN ISO 6508-3 (for Rockwell N-scale tests). There are currently no CEN or ISO standards for Knoop test block verification and calibration. Verification of the geometry of indenters is dealt with in OIML-36.

4.3 Conversion of hardness numbers to other scales

Whereas for metallic materials there are conversion tables to convert between various hardness numbers on particular alloy types, there is no equivalent for ceramic materials. Since ceramics tend to show a strong force dependence of hardness characteristics, it is highly unlikely that there could be a unique relationship between hardness values determined using different forces or different types of indenter.

NOTE Attempts to convert hardness numbers from one scale to another are strongly discouraged.

5 Test method: method A: Vickers test

5.1 Principle

A hardness test in which a square-based sharp pyramidal diamond indenter having specified face angles is forced into the test-piece surface under a defined force, held for a defined duration and removed. The indentation diagonal lengths are measured, the mean result calculated, and this value then employed to calculate a hardness number which is equivalent to the mean force per actual unit area of indenter surface contacting the test surface (no units are given, but kgf/mm^2 are implied):

$$\mathrm{HV}\,F = 1{,}8544\; F/d^{2} \tag{1}$$

where

HV F is the Vickers hardness number at applied force F (expressed as the mass in kg from which F is derived), and

where

d is the mean length of the diagonals of the indentation (expressed as mm).

The Vickers test for metallic materials is described in detail in EN ISO 6507-1 for applied forces derived from masses of 0,2 - 100 kg, in EN ISO 23878 for hardmetals, and in ISO 14705 for advanced technical ceramics.

NOTE 1 Other references to the Vickers test method, including microhardness tests may be found in references [1] and [2] of the Bibliography.

In accordance with ISO 14705 and for the purposes of this document the use of SI units is preferred. In the use of equation (1), when F is expressed in newtons (= mass in kg $\times$ 9,807) and d is expressed in mm, the hardness HV is then computed in MPa. The results shall be expressed as, for example: 15,0 GPa HV 1,0 for a test under a force of 9,807 N derived from a mass of 1 kg.

Unless otherwise agreed between parties, for the purposes of testing advanced monolithic technical ceramics, the applied force shall be that derived from a mass of 1,0 kg (i.e. 9,807 N), i.e. test type HV1,0.

NOTE 2 If this force results in excessive cracking, or indentations which are not readable (see 5.4), then a lower force is permitted. A higher force is permitted if there is minimal cracking associated (see 5.4).

5.2 Machine calibration

5.2.1 Direct verification

Ensure that the test machine is constructed and has been directly verified in conformity with EN ISO 6507-2. Using a metallic reference block certified in accordance with EN ISO 6507-3, make at least three indentations at a test force of HV 1,0. Ensure that the correct cross-hair positioning and zeroing criteria are used (Figure 2). Calculate the hardness using equation (1) and ensure that the mean value matches the certified value. If the apparent hardness does not match the certified value to within the uncertainty envelope, the test machine should be serviced and re-verified before use.

5.2.2 Indirect verification

Using a high hardness reference test block certified in accordance with EN ISO 6507-3, make an indentation at a test force of HV1,0 (unless otherwise agreed, see 5.1). Check that the indentation is regular in shape and shows no damage to the indenter. If damage is suspected, change the indenter and repeat. Measure the diagonal lengths to the nearest 0,2 μm using either the machine graticule or micrometer stage, or using a separate microscope with a micrometer stage. If the indentation is asymmetric, check the alignment of the machine or the indenter. Calculate the Vickers hardness in accordance with equation (1). If there is a small difference (≤ 2 μm in diagonal length) between the test block calibrated value and the observed value of hardness, check the visual reading criteria employed, and adjust them appropriately. If there is a large difference (> 2 μm in diagonal length), first check the function of the machine, particularly that the applied force is correctly calibrated.

NOTE 1 The use of hardmetal or ceramic reference blocks is recommended, especially to provide appropriately small indentations when testing advanced technical ceramic materials.

NOTE 2 It may be helpful to mark the position of the calibration indentation with a pen if the measurement of the diagonal lengths is made on a separate microscope.

NOTE 3 Adjustment of reading criterion may be necessary before beginning a measurement session on test-pieces. The use of a reference block is considered to be a useful method of getting the eye accustomed to making measurements before testing the test-piece.

5.3 Test piece

The test piece surface and the support surface shall be parallel to obtain symmetrical indentations (see Figure 1, schematic 7 for limit of asymmetry). Irregular shaped fragments or components may be mounted in mounting plastic for the purposes of this test. The thickness of the test piece should be at least five times the distance that the radial cracking extends from the centre of the indentation, or ten times the depth of penetration, whichever is greater. Polish the surface to be tested until it is scratch-free in the region in which indentations are to be made, as observed at the magnification used for measuring the indentation size.

NOTE The surface quality of ceramic test-pieces may affect the results. The test-piece should be neither thermally nor chemically etched to reveal grain structure, as this can obscure the corners of indentations. Note should also be made that it is possible that surface stresses produced by machining and polishing may affect the indentation size. The test should preferably be performed on test-pieces prepared with prolonged polishing such that at least 20 μm has been removed with an abrasive grit size of less than 3 μm, or which have been annealed (but not thermally etched) if not polished as above. If annealing is used, the optimum annealing temperature should be established by experiment as that which results in a maximum size of indentation, or a minimum hardness.

5.4 Test procedure

Place the test piece in the test machine and make at least 5 indentations spaced apart by at least five times the length of the radial cracking (see Figure 1, schematic 3). The indentations shall be randomly positioned, and shall not be positioned over any particular microstructural feature.

NOTE 1 In coarse-grained or some multiphase materials the size of the indentation may be similar to or smaller than the grain size or other microstructural features of the test material. The test then loses the averaging element normally required for assessment of polycrystalline materials, and a larger spread of results is obtained. Any bias towards preferential positioning of the centre of the indenter at particular microstructure features will produce a bias in the test results. For material comparison purposes, where possible it is advisable to ensure that the indentation diagonal size is at least five times the average grain size of the test material.

Inspect the shape of the indentations for regularity, and reject any that show suspected irregular shape, displacement of one or more corners, loss of one or more corners or excessive radial cracking (see Figure 1, schematics 6 to 14). If the indentations are asymmetric (see Figure 1, schematic 7), the surface is not adequately flat or perpendicular to the axis of the machine, and the test piece should be re-mounted or re-polished as appropriate. Repeat the tests until at least five acceptable indentations are produced. Measure the lengths of both diagonals of each indentation to the nearest 0,2 µm using the criteria appropriate to the test machine and the guidance shown in Figure 2.

NOTE 2 Most ceramics are translucent under the conditions of observation of the indentations in Vickers (and Knoop) tests. This results in very poor contrast at the corners of the indentations compared with metallic materials, and there are consequent difficulties in placement of measuring crosswires. Some experience may be needed by an operator in order to develop a consistent criterion for measurement.

If damage to the indenter has occurred during the course of the tests, replace the indenter and repeat the reference block tests and the tests on the test-piece.

Calculate the HV1,0 hardness in GPa or the hardness number for each indentation according to equation (1), calculate the mean result and the standard deviation.

5.5 Accuracy and uncertainties

The principal errors arising in a Vickers hardness test on advanced monolithic technical ceramics vary in magnitude according the size of the indentation, and thus the indentation force used. The Vickers diamond geometry was originally chosen because natural cleavage planes of the diamond were employed. Variations in geometry between indenters are therefore small, and can usually be ignored except when indentations are of less than 20 µm diagonal length where the tip and edges near the tip may be variable between indenters. In particular, the edges may have flats up to 1 µm across on them, and this has the effect of cutting the corners off the indentation. The error that this introduces is insignificant if the indentation is larger than about 30 µm, but increases rapidly in importance as the size is reduced.

Determination of the diagonal lengths using cross-wires or other device attached to the instrument relies on the operator positioning them at the "true" opposing corners of the indentation. There is a subjective element in performing this task which increases with poor optical contrast and reducing size of the indentation. The possible errors can be reduced by experience, and by consistent use of high-hardness, preferably ceramic or hardmetal, test blocks to familiarise the eye at the start of measurement sessions. In this way any systematic measurement bias can be reduced. In a round-robin exercise on high-alumina ceramics (reference [3], Bibliography), it was found that when two individuals measure the same set of indentations on different measurement equipment, a poor correlation was obtained unless the true sizes of the indentations varied by more than ± 1 µm. It follows that, discounting differences between machines, it cannot be guaranteed that any two observers will agree that one material is significantly harder than another unless the average indentation sizes are systematically smaller by more than 1 µm. Thus even if it is possible to measure the indentation diagonal length to an apparent precision of 0,1 µm, or the optical resolution limit if larger, the ability to discriminate between materials is limited to an order of magnitude greater in size. Errors of this size assume significance when the indentation size is less than about 20 µm. In addition there is the actual scatter in indentation sizes as a result of local microstructure variations such as grain size, grain orientation, secondary phase content, microcracking, porosity, etc. In a very uniform and homogeneous, hard, fine-grained material, the scatter in actual indentation sizes may be less than the potential measurement errors, and thus not be discernible. In a less-homogenous material, the true indentation size may vary significantly. In such a case, the mean result may be determined by the choice of measurement position, deliberate or inadvertent. The certainty of mean result can be improved only by increasing the number of indentations, but the possibility of a human bias remains. The discrimination between inhomogeneous materials is poorer than for homogeneous ones.

The use of thin metal coatings or alternative optical techniques (such as Nomarski interference techniques) for improving the contrast of indentations prior to visual measurement is not allowed by EN ISO 6507. In this document, the use of coatings is allowed for translucent or transparent ceramics provided that it is less than 0,2 µm thick such that the dimensions of the indentation are essentially unaffected. Nomarski interference techniques distort the image, and shall not be used.

The use of the scanning electron microscope is not recommended for a number of reasons. The principal ones are that the topographic contrast produced by an indentation is not great, that the edges and corners are not always clearly defined, and that the actual magnification of the image requires careful calibration and checking for distortion in both directions.

In summary, the systematic and material inhomogeneity errors may be minimised by employing the highest possible measurement force consistent with no chipping or displacement of corners of the indentation. Under such conditions, the discrimination between materials is greatest. Tests at HV1,0 represent an optimum force in terms of the range of materials which can give acceptable indentations. At greater forces, problems with quality of indentations can make measurement impossible for many materials, even though errors may be proportionately smaller. Even so, the possible errors contribute typically ± 0.7 GPa in hardness (± 70 in hardness number) as a confidence level. Microhardness tests are subject to much larger overall errors, typically ± 2.0 GPa in hardness (± 200 in hardness number) (10 –15 %) can be expected at HV0,2, and greater at lower forces, and should not be used for any test required for a specification.

6 Test method: Method B: Knoop test

NOTE The notes appearing under the Vickers test (clause 5) apply also to the Knoop test.

6.1 Principle

A hardness test similar to the Vickers test, but where an elongated indentation is produced by a rhombic-based sharp diamond indenter having specified face angles. Only the long diagonal length of the indentation is measured, and the result is calculated as the mean force per unit projected area of indentation (no units are given but kgf/mm^2 are implied):

$$\text{HK}\,F = 14{,}229\,F/d^{2} \qquad (2)$$

where

HK F is the Knoop hardness number at applied force F (expressed as the mass in kg from which F is derived),

and

d is the length of the long diagonal of the indentation in mm.

Knoop hardness tests are normally conducted at applied forces derived from masses of 2,0 kg or less. The test is described in ISO 4545 for metallic materials, in ISO 9385 for glass and glass-ceramics and in ISO 14705 for advanced technical ceramics.

NOTE 1 The Knoop test may be applied more satisfactorily than the Vickers test in circumstances where the criteria for acceptability of Vickers indentations are not met, but those for Knoop indentations are. Further information on the Knoop test may be found in references [4] and [5] of the Bibliography.