VAMAS

Technical Working Area 3

CERAMICS

GUIDELINES FOR CONDUCTING HARDNESS TESTS ON ADVANCED CERAMIC MATERIALS

September 1990

VAMAS Technical Report 8

Versailles Project on Advanced Materials and Standards
Canada, CEC, FRG, France, Italy, Japan, UK, USA

VAMAS Report 8 September 1990

NATIONAL PHYSICAL LABORATORY

Division of Materials Metrology

GUIDELINES FOR CONDUCTING HARDNESS TESTS ON ADVANCED CERAMIC MATERIALS

by

R Morrell

Summary

This document has been prepared to propose the type of wording that may be employed in the development of a standard defining the usefulness of hardness testing of ceramics. It has been written in a style and format suitable for immediate discussion by standards committees. It follows from a VAMAS hardness testing round-robin conducted in 1987-8 on two high-alumina ceramics in which 25 participants undertook an intensive measurement exercise designed to evaluate systematic and random errors of measurement in using Rockwell Superficial HR45N, Vickers HV1.0 and HV0.2, and Knoop HK0.2 tests.

ISSN 1016 - 2186

National Physical laboratory
Teddington, Middlesex, TW11 0LW, UK

M. K. Hossain

Signature of Superintendent

Date 26th. September 1990.

R Morrell

Signature of principal author

Date 18 September 1990

Approved on behalf of Director, National Physical Laboratory, by Dr M K Hossain, Superintendent, Division of Materials Metrology.

VAMAS TWA3 - CERAMICS

GUIDELINES FOR CONDUCTING HARDNESS TESTS ON ADVANCED CERAMIC MATERIALS

1. Scope

This part of XXXXXXX (Standard designation) provides guidelines concerning the conducting of, and the value that may be ascribed to the results of, standard hardness tests for metallic materials when applied to advanced technical ceramics. It is assumed that the calibration and test procedures employed are exactly those for metallic materials. This standard refers to Rockwell A, Rockwell Superficial (N-scale), Vickers, and Knoop hardness testing, as described in the following documents:

ISO 716, 1079, 1355, 3738 (Rockwell testing);
ISO 146, 640, 3878 (Vickers testing);
ASTM E384, C730, C849 (Knoop testing);
OIM R-I-36 (Verification of indenters).

2. Definitions

Hardness: The resistance displayed by a material to penetration by a hard indenter of defined geometry and loaded in a prescribed manner.

Hardness number: The 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.

Hardness indenter: A hard device of defined geometry, usually fabricated from single-crystal diamond.

Rockwell hardness test: A hardness test as performed according to ISO 716 and 1079, wherein a sphero-conical diamond indenter is initially loaded onto the test-piece surface under a small force (the minor load), normally derived from a mass of 3 kg, a displacement scale is set to zero, and then a larger force (the major load) is applied and then removed. The net displacement of the indenter is recorded. Tests that have been employed for ceramic materials include Rockwell "A" (ISO 716 and 3738, major load derived from a total mass of 60 kg), and Rockwell Superficial "N" (ISO 1079, major load derived from total masses of 45 kg, 30 kg or 15 kg). The displacement is converted by the measuring machine to a hardness scale of 0 - 100, where 100 represents zero penetration:

$$\text{HRA, HRN} = 100 - \delta$$

where δ is the depth of penetration under the major load measured in micrometres (HRN) or in units of two micrometres (HRA).

Vickers hardness test: A hardness test in which a square-based sharp pyramidal diamond indenter having specified face angles is loaded 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):

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

where HV(P) is the hardness number at applied load P (expressed as the mass in kg from which P is derived), and where d is the mean length of the diagonals of the indentation (expressed as mm). The Vickers test is described in detail in ISO 146 for applied loads derived from masses of 0.2 - 100 kg, and in ASTM E384 for applied loads derived from masses of 1.0 kg downwards.

Knoop hardness test: 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):

$$HK(P) = \frac{14.229\,P}{d^2}$$

where HK(P) is the hardness number at applied load P (expressed as the mass in kg from which P is derived), and d is the length of the long diagonal of the indentation in mm. Knoop hardness tests are normally conducted at applied loads derived from masses of less than 1.0 kg. The test is described in detail in ASTM E384.

3. Use of hardness tests on advanced technical ceramics

The three types of test defined in Para. 2 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 of the material, 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 ceramic materials 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 is 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. Important points in hardness testing of advanced technical ceramics

When applied to advanced ceramic materials, the following factors need to be observed:

(1) The brittle nature of ceramics results in cracking from the indentation. Radial cracking, especially from the corners of HV1.0 - HV30 tests, can result in difficulties of identifying the corners clearly for measurement. In some cases, there is sub-surface lateral cracking, visible in transparent or translucent materials. This cracking may result in the loss of material in the form of chips from around the

indentation. Such indentations must be ignored.

(2) As a consequence of (1), it may be necessary to limit the applied force in Vickers and Knoop tests to a level where a minimum of cracking occurs. Generally it is found that only on high-strength, tough, fine-grained materials can HV10 tests be performed. HV30 tests are generally unacceptable. HRA tests are similarly only possible on fine-grained, tough materials. If the material is not sufficiently tough or strong, the test-piece may be fractured, risking damage to the indenter and machine. HR45N, HR30N and HR15N tests are generally possible on most dense ceramic materials without risk of gross fracture. Local fracturing around indentations is not normally a problem with Rockwell testing because the depth measurement is made under the minor load before such fracturing occurs on complete unloading.

(3) Unlike many metallic materials, ceramics tend to show a marked load dependence of hardness number, which becomes particularly evident when a test force derived from a mass of less than 1.0 kg is used in HV and HK tests. It is critically important that the test load is appended to each test result, and that no attempt is made to compare results at different test loads in order to make a choice of material or to test to a specification.

(4) In Rockwell Superficial tests, hard ceramics give test results with hardness numbers in excess of 70. This corresponds to a penetration of 30 µm. When the hardness exceeds 90, the test lacks adequate discrimination between materials because the differences in penetration depth recorded become small. Although test results might appear consistent, the contracted scale is very insensitive to differences between materials.

(5) In Vickers and Knoop tests at low loads, the small size of the indentations means that measurement errors can be large. Optical resolution is also a fundamental limitation. For materials comparison purposes, preference should be given to tests at higher loads where this is possible.

(6) Many ceramics contain porosity which may be distributed uniformly or unevenly (e.g. porous patches). A hardness test will tend to compact the pores in the immediate vicinity of the area of contact of the indenter, giving a lower hardness than for an area which is pore-free. Care should be taken that the positioning of the indenter for hardness measurements is random, and not selective, although clearly large obvious pores need to be avoided.

(7) For all tests the test-piece shall have parallel flat faces so that it does not rock or move during indentation. If necessary, it may be mounted in mounting resin for microhardness tests. The thickness of the test-piece shall be at least five times the the distance that the radial cracking extends from the centre of the indentation, or ten times the depth of penetration, whichever is greater. The indentations in a test-piece shall be spaced a distance apart greater than at least five times the distance that any radial cracks extend from the centre of the indentations, and shall not be closer than this distance from a free edge of the test-piece. In this way, there is little risk that the microstructure is affected by a neighbouring indentation, and the risk of catastrophic fracture is remote.

(8) The test load selected 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 load is actually being applied to the test surface for the required period of time.

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

Verification of test equipment is described in ISO 146 (Vickers), ISO 716 (Rockwell A), ISO 1079 (Rockwell N), ASTM 384 (Vickers and Knoop microhardness), and calibration of standard reference blocks is described in ISO 640 (Vickers), ISO 3738 (Rockwell A), ISO 1355 (Rockwell N). There are currently no ISO standards for Knoop test verification and calibration.

(10) Most ceramic materials are translucent under the conditions of observation of the indentations in HV and HK 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.

(11) In microhardness tests, 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 for 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, it is advisable to use indentation loads large enough such that the indentation diagonal size is at least five times the average grain size of the test material.

(12) The surface quality of ceramic test-pieces may affect the results. For Rockwell tests, surface finish is unimportant, since the minor load applied tends to obliterate any localised features before the actual measurement is made. In Vickers and Knoop testing it is imperative that the surface of the test-piece is polished to a metallographic finish free from scratches and with a roughness of less than 0.5 µm R_a (less than 0.1 µm R_a for tests at loads derived from masses of less than 1.0 kg). The test-piece shall 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 in a given microhardness test. 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) after polishing. If annealing is used, the optimum annealing temperature must be established by experiment as that which results in a maximum size of indentation, or a minimum hardness.

5. Errors in hardness measurements on advanced ceramic materials

5.1 Rockwell tests

Since the measurement is performed generally on a purpose-built machine, no operator errors are involved in the measurement itself. It is essential that the test-piece is flat and parallel faced such that it presents a stable surface for the measurement. The principal error that can arise results from the geometric form of the diamond indenter not being perfectly spherical at its tip. Calibration with a high-hardness test-block is essential, and visual inspection of the quality of the indentation with a microscope is highly desirable. The diamond should be changed if there is any doubt about its quality.

The Rockwell scale is rather contracted at high hardness levels compared with the HV or HK scales in which the reverse is true. The scatter in hardness number about the mean result is typically ± 1 in a homogeneous ceramic material, which at a hardness numbers of 75 and 90 correspond to ± 4% and ± 10% respectively in depth of penetration, and to similar values of supported area and thus of mean supported stress.

Systematic errors of typically ± 1 in hardness number between different test machines and/or diamond indenters are commonly found.

The combined effect of these errors is that for a small number of indentations a difference of about ± 1 in hardness number between two materials can be considered to be the minimum significant difference measurable with a given diamond indenter and machine, rising to ± 1.5 when using different machines.

At least 5 indentations must be made.

5.2 Vickers hardness tests

The principal errors arising in a Vickers hardness test on advanced technical ceramics vary in magnitude according the size of the indentation, and thus the indentation load 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 at microhardness levels where the tip and edges near the tip may be variable between indenters. In particular, the edges 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 measuring instrument relies on the operator placing 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 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, 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 [1]. 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 to an accuracy of ± 0.1 μm, limited by optical resolution, the discrimination is at least an order of magnitude larger. 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, porosity, etc. In a very uniform and homogeneous 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 only be improved by increasing the number of observations, but the possibility of a human bias remains. The discrimination between materials is poorer than for a homogeneous material.

In summary, as demonstrated by [1], the systematic and material inhomogeneity errors may be minimised by employing the highest possible measurement load consistent with no chipping or displacement of corners of the indentation. Under such conditions, the discrimination between materials is greatest. Tests at HV1.0 or greater load are to be preferred to microhardness tests in cases where great importance is to be placed on the hardness number measured. Even so, the possible errors contribute typically ± 70 as a confidence level to the mean hardness number. Microhardness tests are subject to much larger overall errors, and typically ± 200 (10-15%) can be expected at HV0.2, and greater at lower loads.

At least 10 indentations must be made for loads derived from masses of less than 1.0 kgf, 5 for greater loads.

5.3 Knoop tests

The Knoop test is conducted in the same manner to the Vickers test, except that only the long diagonal is measured. Since the indentation is much shallower than the Vickers indentation, the angle of intersection of the indentation surfaces with the original surface is small, and the optical contrast this produces in the conventional measurement system is poorer than in the Vickers case. Possible measurement errors and biases are thus much larger, and as the round-robin exercise [1] demonstrated, the fractional error in the test results is similar to or greater than that for Vickers tests at the same load. Thus although the HK test is sometimes recommended as being more benign to the material and covering more microstructure because of the greater length of the diagonal than in the Vickers case, the human element cancels out any advantages. The tendency for generating cracks at the tips of the long diagonal provides additional visual uncertainty for the observer.

In summary, since Knoop tests are generally conducted at loads derived from masses of less than 1.0 kg, the errors in measurement could be ± 100 (typically 5 - 8%) in hardness number rising to ± 250 (typically 10 -15%) at HK0.2. At least 10 measurable indentations must be made.

5.4 Improving measurement resolution of indentations

The use of thin metal coatings or alternative optical techniques for (such as Nomarski interference techniques) for improving the contrast of indentations prior to visual measurement is not allowed by the existing standards. The use of coatings has some value, but care should be taken that the coating is less than 0.2 µm thick such that the dimensions of the indentation are essentially unaffected. Nomarski interference techniques distort the image, and should not be used.

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 clearly defined, and that the actual magnification of the image requires careful calibration and checking for distortion in both directions.

6. Use of hardness tests for other purposes

Hardness tests have been used to generate cracks for the purposes of determining fracture toughness, for example by measuring radial crack length, or fracturing the test-piece using the crack as an induced defect. There are some uncertainties in the physical basis for the evaluation of fracture toughness in these tests, and considerable errors in the measurement of crack length, a similar problem to that of measurement of indentations. There are no standardised procedures.

7. Report

In addition to the information required under the particular ISO or ASTM standard used, the following shall be reported.

(a) Details of the preparation of the surface of the test-piece, including grinding and polishing schedule, annealing temperature and time.

(b) Details of the calibration of the instrument using high-hardness certified test blocks.

(c) Individual results, the mean and the standard deviation.

(d) A reference to this standard in respect that the test procedure takes cognisance of the recommendations made.

Reference

1. D M Butterfield, D J Clinton, R Morrell, "The VAMAS Hardness Test Round-Robin on Ceramic Materials", VAMAS Report No. 3, National Physical Laboratory, Teddington, Middlesex, TW11 0LW, UK, April 1989.

Other standards cited

ISO 146:1984 Metallic materials - hardness test - verification of Vickers hardness testing machines HV0.2 - HV100.

ISO 640:1984 Metallic materials - hardness test - calibration of standard test blocks to be used for Vickers hardness testing machines HV0.2 - HV100.

ISO 716:1984 Metallic materials - hardness test - verification of Rockwell hardness testing machines Scales A-B-C-D-E-F-G-H-K.

ISO 1079:1989 Metallic materials - hardness test - verfication of Rockwell superficial hardness testing machines (scales 15N, 30N, 45N, 15T, 30T, 45T).

ISO 1355:1989 Metallic materials - hardness test - calibration of standardised test blocks to be used for Rockwell superficial hardness testing machines (scales 15N, 30N, 45N, 15T, 30T, 45T).

ISO 3738-1:1982 Hardmetals - Rockwell hardness test (scale A) - Part 1 test method.

ISO 3738-2:1982 Hardmetals - Rockwell hardness test (scale A) - Part 2 preparation and calibration of standard test blocks.

ISO 3878:1983 Hardmetals - Vickers hardness test.

OIM R-I-36 Verification of indenters for hardness testing machines. (OIM = Organisation Internationale de Metrologie Legale)

ASTM E384:1984 Standard test method for microhardness of materials.

ASTM C849:1981 Standard test method for Knoop indentation hardness of ceramic whitewares.

ASTM C730:1985 Standard test method for Knoop indentation hardness of glass.