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Sintered metal materials, excluding hardmetals — Determination of apparent hardness and microhardness

Matériaux métalliques frittés, à l'exclusion des métaux-durs — Détermination de la dureté apparente et de la microdureté

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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 4498 was prepared by Technical Committee ISO/TC 119, *Powder metallurgy*, Subcommittee SC 3, *Sampling and testing methods for sintered metal materials (excluding hardmetals)*.

This first edition of ISO 4498 cancels and replaces: ISO 4498-1:1990, and ISO 4498-2:1981.

Introduction

Sintered metal materials generally have a porous structure. Therefore, they can be understood as composite metal/pore materials. That is why this International Standard describes two procedures to determine their hardness:

— Procedure 1 for the macro hardness (this is the apparent hardness);

— Procedure 2 for the micro hardness (this is the hardness of the metallic phase only).

Tests in Procedure 1 determine Vickers, Brinell and/or Rockwell macrohardnesses — their acronyms are: HV, HB, and HR. These tests determine the apparent hardness (macrohardness) of the materials because indentations generally include both the solid phase and a number of pores. The usual test forces applied to an indenter are from 10 N to 2 000 N.

The apparent hardness value is often used as an expression of the mechanical strength of the material as a whole; it is usually lower than that of a solid material of the same composition and metallurgical condition. However, this does not imply that the functional characteristics (for example wear resistance) are necessarily inferior to those of an equivalent full-density material.

The apparent hardness is a macrostructural property. It characterises the material taken as a whole.

Tests in Procedure 2 determine the Vickers and/or Knoop microhardnesses of the material — their acronyms are: HVa, HKa [1]. The usual test forces applied to an indenter are from 0,147 N to 1,960 N for Vickers, and 0,981 N for Knoop.

The microhardness is a microstructural property used to control chemical composition, heat treatment or surface treatment. For these purposes, it is necessary to ensure that hardness test indentations are small enough not to include any visible pores, but only the solid phase.

1) Where a is the test load, in kilograms.

Sintered metal materials, excluding hardmetals — Determination of apparent hardness and microhardness

1 Scope

This International Standard specifies methods of hardness testing of sintered metal materials, excluding hardmetals.

1.1 Procedure 1 determines the apparent hardness of the whole material.

Procedure 1

— applies to sintered metal materials which have either not been subjected to any heat treatment, or which have been heat treated in such a way that the hardness is essentially uniform to a depth of at least 5 mm below the surface,

— applies to the surfaces of sintered metal materials which have been treated in such a way that the hardness is not uniform in the section to a depth of 5 mm below the surface,

— therefore applies to materials in which the hardness is obtained essentially by surface enrichment by carbon, or by carbon and nitrogen (for example, and by carburising, carbonitriding, nitrocarburising or sulphidising), and

— applies to materials which have been induction hardened.

1.2 Procedure 2 determines the microhardness of the metal phase.

Procedure 2

— applies to all types of sintered metal materials.

— is used, in particular, to determine the hardness profile of case-hardened or carbonitrided materials according to the method described in ISO 4507.

— also applies to any sintered metallic materials which have been subjected to surface treatments such as electrodeposited plating, chemical coating, chemical vapour deposition (CVD), physical vapour deposition (PVD), laser, ion bombardment, etc. To determine the microhardness of treated surfaces, Procedure 2 applies.

NOTE However, it should be noted that international agreement has not yet been reached on a number of factors involved in microhardness testing. Nevertheless, the parameters defined in Procedure 2 are important enough to enable a considerable measure of standardisation of extensively used practices.

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 4507:2000, *Sintered ferrous materials, carburized or carbonitrided — Determination and verification of case-hardening depth by a micro-hardness test.*

ISO 4516:2002, *Metallic and other inorganic coatings — Vickers and Knoop microhardness tests*

ISO 6506-1:1999, *Metallic materials — Brinell hardness test — Part 1: Test method*

ISO 6507-1—[2)], *Metallic materials — Vickers hardness test — Part 1: Test method*

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

3 Apparatus

Procedure 1: Vickers, Brinell and Rockwell hardness testing machines and test methods meeting the requirements of ISO 6506-1, ISO 6507-1 and ISO 6508-1, respectively.

Procedure 2: Vickers and Knoop microhardness testing machines and test methods meeting the requirements of ISO 4516.

4 Sampling and preparation of test pieces

4.1 Since the apparent hardness of a sintered material is affected by density, which can vary throughout a part, the position of the hardness indentations, for the purpose of quality control, shall be agreed between the parties.

4.2 The sintered metal surface shall be clean, smooth and flat to obtain well-defined hardness indentations. Test samples will have anvil support surfaces filed or ground flat wherever practicable, so as to prevent burrs from affecting results. This is particularly important when determining Vickers and Brinell hardness. Emery paper of 180 to 240 grit is acceptable for grinding. It is generally found sufficient to clean the surface with a suitable solvent. If not, the surface may be lightly polished, provided that laboratory measurements have shown that the influence of such polishing is insignificant.

NOTE This polishing may be carried out, for example, by using metallographic paper or a 6 µm diamond paste.

4.3 Microhardness can be measured either on the surface of a part or on a cross-section of the part normal to the surface. For microhardness determinations, it is necessary to ensure that the surface is smooth enough to allow measurement of the indentation diagonal length accurately. The sample may then be chemically cleaned, electrochemically or mechanically polished to reveal porosity. Mechanical polishing should involve minimum local heating or working, so as not to affect hardness.

Previous impregnation of the part with a thermosetting resin can be beneficial, if the part has more than 8 % open porosity. The surface to be measured shall be flat and smooth. Indentations should have sharp edges in order to carry out accurate diagonal measurement. The thickness of the test piece shall be greater than 1,5 times the length of the impression diagonal.

4.4 Surface curvature introduces a certain error in determining microhardness, which increases as the radius decreases. On convex surfaces, higher hardness values and, on concave surfaces, lower hardness values, than the actual values are obtained. If the Vickers hardness test (apparent hardness or microhardness) has to be performed on a curved surface sample, the influence of the curvature will have to be compensated for by correction factors (see: ISO 6507-1 and ISO 4516).

4.5 The measurement of microhardness shall not be valid if the test surface is not perpendicular to the indenter axis. Non-perpendicularity will be probable with isotropic materials, if one leg[3)] of the diagonal is noticeably longer than the other leg[2)] (Vickers or Knoop microhardness). The specimen for microhardness testing shall be positioned on the supporting table, or presented in such a way that the test surface is perpendicular to the direction of the test force, otherwise the indentation will be distorted. This position shall be maintained during the entire test.

2) To be published. (Revision of ISO 6507-1:1997)

3) The leg is the distance from the center of the indentation to the outer corner.

5 Test procedures

5.1 Procedure 1 — Determination of apparent hardness

5.1.1 The tests shall be made in accordance with the requirements of ISO 6506-1, ISO 6507-1 or ISO 6508-1, but also with the additional requirements given in 5.1.2 to 5.1.5.

5.1.2 The hardness class to which a test piece belongs shall be determined by Vickers hardness testing using a test force of 49,03 N (HV5). The test conditions shall then be selected from Table A.1 according to the class determined. Details of the conditions for the Rockwell test are given in Table A.2.

In some cases, particularly with solution-hardened PM materials, it is advantageous to determine hardness values on the HRB scale using a hardmetal ball indentor. In these cases, results are then denoted by HRB and are used up to a maximum value of HRB 115.

If, after the initial HV5 test, there is any doubt as to the hardness class to be chosen, the lower class shall be selected.

When a material specification covers more than one hardness class, the test shall be conducted under the conditions appropriate to the lower hardness limit given in the specification.

5.1.3 For some test pieces, it will be necessary to use smaller test forces than those specified in Tables A.1 and A.2, in order to meet the requirements of ISO 6506-1, ISO 6507-1 or ISO 6508-1. This will be particularly so

— for thin test pieces,

— for test pieces of small cross-sectional areas,

— when the designated test area is very small, and

— when the test piece or its mount is likely to be distorted.

When such test conditions are necessary, the details shall be agreed between the customer and the supplier. It should be noted that, in these circumstances, the scatter of the results will be greater than under normal test conditions and that the value obtained will be less representative of the state of the material since the indentation will be very small.

5.1.4 When determining Vickers hardness, an indentation is not valid if

— it does not have clearly defined corners,

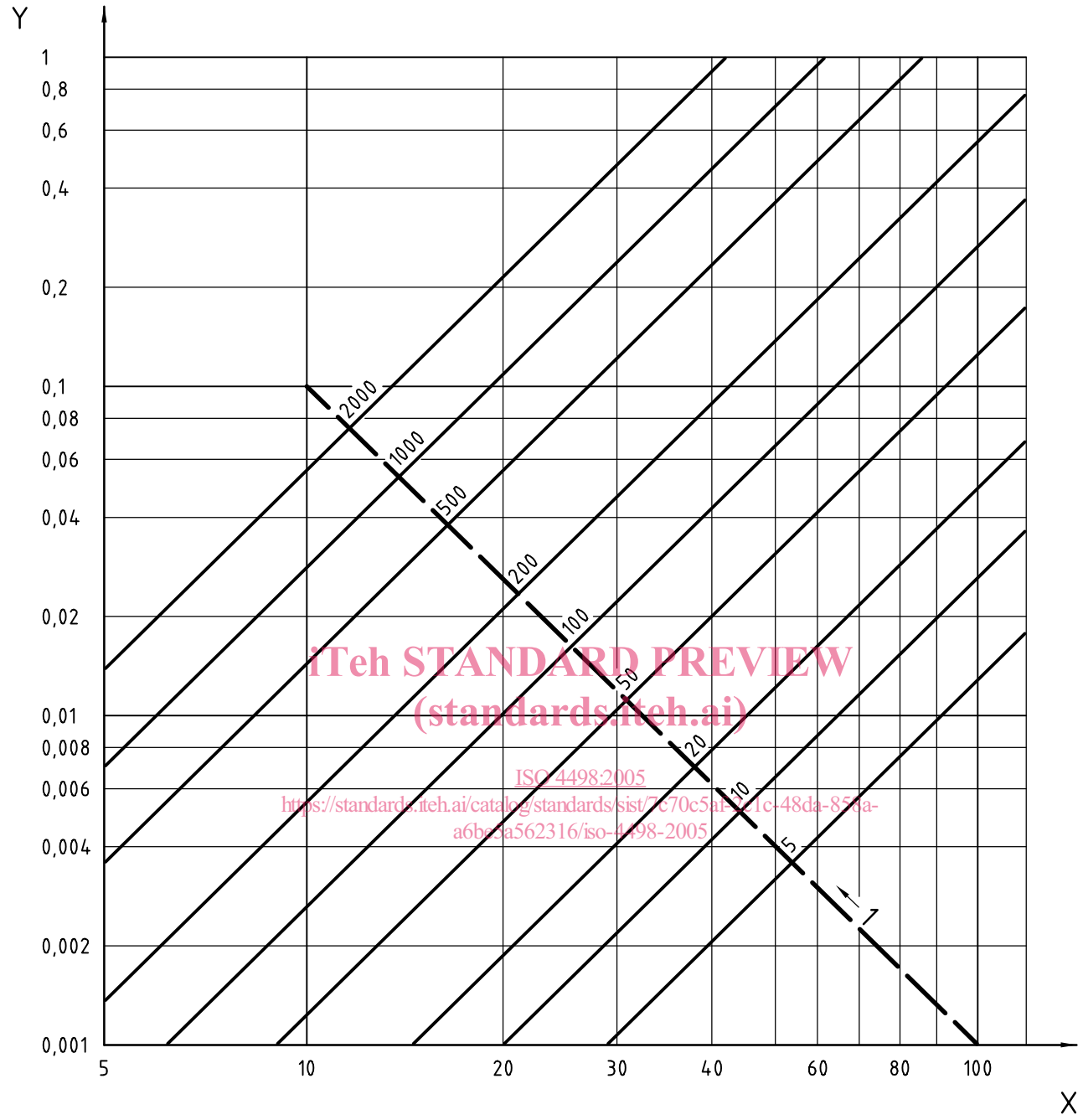
— the edges are distorted, (inwards or outwards), or

— the lengths of the diagonals are substantially different.

5.1.5 Five valid indentations shall be made and the corresponding hardness values calculated (or simply read in the case of Rockwell testing). Another procedure for treatment of results is permitted, by agreement between the customer and the supplier.

5.2 Procedure 2 — Determination of microhardness

5.2.1 When determining the microhardness of surface-treated material (as described in 1.2), reference shall be made to ISO 4516 for test conditions (precautions, load, velocity and direction of application of the force). Figure 1 gives an indication of the force to be used as a function of the thickness of material which has undergone surface modification by a method listed in 1.2.



Speed of application of the force: 15 µm/s to 70 µm/s

Application time of the force: 10 s to 15 s

Key

1 coating hardness

x coating thickness

y test force

Figure 1 — Relation of maximum applicable test force to modified thickness (Vickers indenter)

5.2.2 When determining the microhardness of the metal phase, the use of the test forces in Table A.3 is recommended for Vickers. In the case of Knoop, 0,981 N is the most commonly used test force.

These are forces currently used in powder metallurgy. The test force shall be chosen in order to correspond to a diagonal length that is large enough for an acceptable accuracy of reading to be achieved (e.g. a length between 20 µm and 30 µm) but also small enough for the requirements of measurement of metal-phase microhardness to be satisfied. The test force shall be applied to the indenter for between 10 s and 15 s.

Lower test forces may be required in order to define local properties of the microstructure. When such test conditions are necessary, all details, including the metallographic preparation of the test specimen, shall be agreed between the customer and the supplier.

5.2.3 The position of indentations shall be chosen with care in the following manner:

a) In relation to the distance between the edges of the metal phase and neighbouring pores: The distance between these edges and the centre of an indentation shall be at least 2,5 times the diagonal of the indentation (in the case of Knoop: 2,5 times the smaller diagonal of the indentation). In the case of coatings, each corner of an indentation shall be at least half the length of a diagonal apart from the edge of the coating or from a pore.

b) In relation to the edge of the test piece: The distance between this edge and the centre of the indentation shall be at least 2,5 times the indentation diagonal (50 µm for Knoop).

c) The shortest distance between the centres of two adjacent indentations shall be at least 2,5 times the diagonal of the larger indentation.

d) In the case of coatings, the four edges of the indentation shall be of equal length within 5 %, and additionally for Vickers hardness tests, the two diagonals shall be of equal length within 5 %.

e) The indentation depth, which can be calculated as a function of the length of a diagonal, shall not exceed one-third of the thickness of the layer to be characterised.

For HV, $t \approx \frac{d' + d''}{14}$ and for HK, $t \approx \frac{d}{30}$

5.2.4 An indentation is not valid

— if it does not have clearly defined corners,

— if the edges are distorted, inwards or outwards,

— or, for Vickers microhardness, if the lengths of the diagonals are substantially different, or

— if there is evidence of material collapse adjacent to the indentation.

Results which appear abnormally low as compared with the results on neighbouring indentations shall be discarded, because this might be due to the presence of an invisible underlying pore very close to the impression under study.

5.2.5 In general, at least five valid indentations shall be made and measured within the prescribed area.

5.2.6 Vibration due to external factors shall be avoided.

6 Expression of results

6.1 Apparent hardness

Report the arithmetical mean of the five valid determinations rounded to the nearest whole number. Hardness values shall not be converted from one scale to another, nor used to derive values for mechanical strength.