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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

Reference number
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Foreword

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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 second edition cancels and replaces the first edition (ISO 4498:2005), of which it constitutes a minor revision.

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 macrohardness (this is the apparent hardness);

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

Tests in Procedure 1 determine Vickers, Brinell and/or Rockwell macrohardnesses; their acronyms are: HV, HBW 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 characterizes the material taken as a whole.

Tests in Procedure 2 determine the Vickers and/or Knoop microhardnesses of the material; their acronyms are: HVa and 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

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

1.2 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 by carburizing, carbonitriding, nitrocarburizing or sulfidizing), and

— applies to materials which have been induction hardened.

1.3 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 in accordance with the method described in ISO 4507, and

— 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, an 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 standardization 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, *Sintered ferrous materials, carburized or carbonitrided — Determination and verification of case-hardening depth by a micro-hardness test*

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

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

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

ISO 6508-1, *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 can 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, and electrochemically or mechanically polished to reveal porosity. Mechanical polishing should involve minimum local heating or working, so as not to affect hardness. The sample for nickel-alloyed sintered steels can be smoothly etched before measuring the microhardness. This smooth etching of the sample will detect the softer areas of nickel-alloyed sintered steels in order to eliminate them from measurement. This leads to a more precise test result.

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[2)] 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.

5 Test procedures

5.1 Procedure 1 — Determination of apparent hardness

5.1.1 The tests shall be carried out 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 (HV 5). 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 (powder metallurgy) materials, it is advantageous to determine hardness values on the HRB scale using a hardmetal ball indenter. In these cases, results are then denoted by HRB and are used up to a maximum value of HRB 115.

If, after the initial HV 5 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

— on thin test pieces,

— for test pieces of small cross-sectional area,

— 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

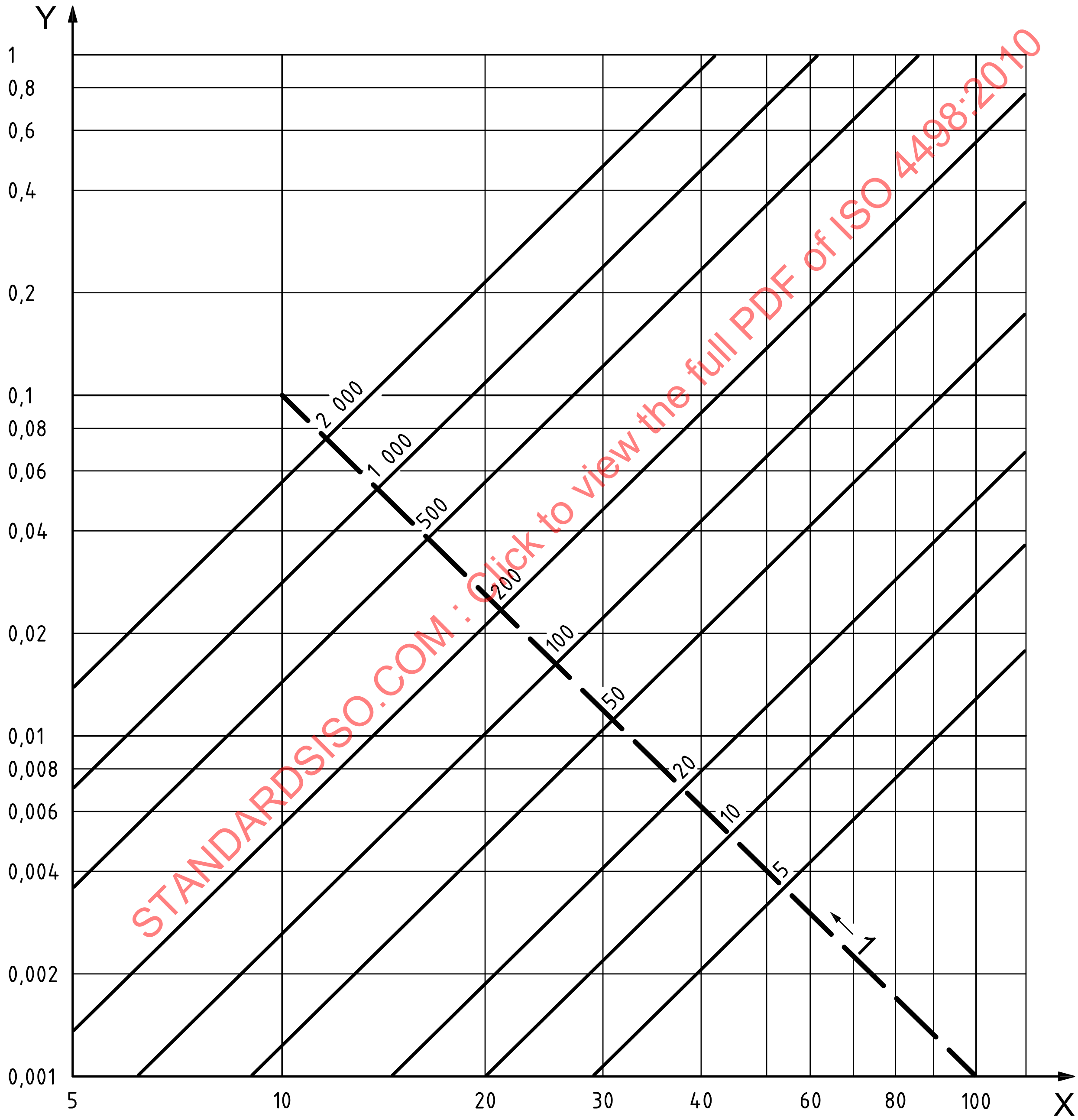
— the lengths of the diagonals are substantially different.

2) The leg is the distance from the centre of the indentation to the outer corner.

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 1.3), reference shall be made to ISO 4516 for test conditions (precautions, load, velocity and direction of application of the force). Figure 1 shows an indication of the force to be used as a function of the thickness of material which has undergone surface modification by one of the methods listed in 1.3.



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, in HV
X coating thickness, in µm
Y test force, in N

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 microhardness. In the case of Knoop microhardness, 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 away 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 characterized. (For a definition of the symbols used, see Table A.4.)

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),

— 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 arithmetic 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.

6.2 Microhardness

6.2.1 Report the arithmetic 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.

6.2.2 The microhardness symbol (HV for Vickers, HK for Knoop) shall be preceded by the value determined and shall be followed by a number indicating the test load (in newtons × 0,102)[3] and a second number indicating, in seconds, the duration of application of the test load, if this is other than 10 s to 15 s (e.g. 585/HV 0,1/20).

6.2.3 Table A.4 gives the appropriate symbols and designations for microhardness values.

7 Repeatability and reproducibility

7.1 Vickers apparent hardness

No public information is available at present, but work is progressing to prepare a precision statement.

7.2 Rockwell apparent hardness

See Table B.1 which gives information produced by MPIF[4] on a range of materials.

7.3 Vickers microhardness

Limited information is available at present; see Tables B.2 and B.3.

7.4 Precision statement

Interlaboratory evaluations conducted by MPIF[4], using a slightly different protocol and reported in MPIF 51[5], give the following precision statement.

The repeatability (r) and reproducibility (R) measurements were determined in 1994 according to ASTM E691. The test sample was prepared from heat treated FL-4605. One Knoop and one Vickers microhardness indent were placed into the surface of the test sample and then measured by 12 participating laboratories.

The mean hardness value was HK 701,1 with a repeatability of 22,4 and a reproducibility of 76,0. Duplicate Knoop hardness results from the same laboratory should not be considered suspect at the 95 % confidence level, unless they differ by more than 22,4. For the same test specimen, Knoop hardness results from two different laboratories should not be considered suspect at the 95 % confidence level, unless they differ by more than 76,0.

3) Therefore, an indicated number 0,1 (for instance) corresponds to 100 g.

4) MPIF: Metal Powder Industries Federation (USA).

5) MPIF 51: 1994, *Determination of Microhardness of Powder Metallurgy Materials*.

The mean hardness value was HV 715,7, with a repeatability of 42,9 and a reproducibility of 177,8. The Vickers hardness results from the same laboratory should not be considered suspect at the 95 % confidence level, unless they differ by more than 42,9. For the same test specimen, the Vickers hardness results from two different laboratories should not be considered suspect at the 95 % confidence level, unless they differ by more than 177,8.

8 Test report

The test report shall include the following information:

a) a reference to this International Standard;

b) all details necessary for the identification of the test sample;

c) the result obtained, with the appropriate symbol and test conditions in accordance with ISO 6506-1, ISO 6507-1, ISO 6508-1 or ISO 4516;

d) any operation not specified by this International Standard or regarded as optional;

e) details of any occurrence which may have affected the results.

Annex A
(normative)

Test conditions and test loads as well as symbols and designations for microhardness values

Table A.1 — Determination of hardness test conditions on a test piece after having determined its Vickers macrohardness class using a 49,03 N test force (HV 5)

Hardness class (HV 5)	Test conditions
15 to 60	HV 5 HBW 2,5/15,625/30 HRH
> 60 to 100	HV 5 HBW 2,5/31,25/15 HRH HRF
> 100 to 200	HV 5 HBW 2,5/62,5/10 HRF HRB
> 200 to 400	HV 10 HBW 2,5/187,5/10 HRA HRC
> 400	HV 20 HBW 2,5/187,5/10 HRA HRC

Table A.2 — Conditions for Rockwell hardness test

Rockwell hardness	Type of indenter	Preliminary test force	Total test force
HRA	Diamond cone 120°	98,07 N	588,4 N
HRB	Ball 1/16 inch (1,587 5 mm)	98,07 N	980,7 N
HRC	Diamond cone 120°	98,07 N	1 471,0 N
HRF	Ball 1/16 inch (1,587 5 mm)	98,07 N	588,4 N
HRH	Ball 1/8 inch (3,175 mm)	98,07 N	588,4 N

Table A.3 — Recommended test loads for Vickers microhardness

Microhardness	Test load g	Force N	Length of diagonal, in µm, for a microhardness of 100	200	500	1 000
HV 0,05	50	0,490	30,4	21,5	13,6	9,6
HV 0,1	100	0,981	43,0	30,4	19,3	13,6
HV 0,2	200	1,960	60,8	43,0	27,2	19,3

Table A.4 — Microhardness: symbols and designations

Symbol	Measuring unit	Designation: Vickers	Designation: Knoop
F	N	Test force in newtons	Test force in newtons
d	µm	Arithmetic mean of the two separately measured diagonals d' and d'': $d = \frac{d' + d''}{2}$	Length of the larger diagonal
HV	—	Vickers hardness number: $\frac{0{,}102 \times F}{A_v} = 1{,}854 \times 10^6 \times \frac{0{,}102 \times F}{d^2}$ where A_v is the surface of indentation, in mm²	—
HK	—	—	Knoop hardness number: $\frac{0{,}102 \times F}{A_k} = 14{,}229 \times 10^6 \times \frac{0{,}102 \times F}{d^2}$ where A_k is the unrecovered projected area of indentation, in mm²[a]

[a] The areas corresponding to the measured diagonal(s) of the indentation are given in tables provided by the manufacturers of the testing machines and instruments.

Annex B
(informative)

Repeatability and reproducibility

Table B.1 — Precision of Rockwell apparent hardness readings on purpose-made parts

Material (MPIF)	**Density** g/cm³	**Hardness** (average)	**Repeatability** (r) (95 % confidence limits)		**Reproducibility** (R) (95 % confidence limits)	
			one reading	average of five readings	one reading	average of five readings
Copper zinc alloy 20 % Zn, 2 % Pb (CZP-2002)	7,92	HRH 82,5	1,7	0,8	2,2	1,9
Plain iron (F-0000)	6,74	HRF 63,4	4,0	1,8	4,4	3,5
Copper steel 2 % Cu, 0,8 % C (FC-0208)	6,63	HRB 70,8	4,5	2,0	5,7	4,9
Copper infiltrated steel 20 % Cu, 0,8 % C (FX-2008)	7,45	HRB 86,4	4,3	1,9	4,9	4,0
Heat-treated low-alloy steel 2 % Ni, 0,5 % Mo, 0,5 % C (FL-4605-HT)	6,90	HRB 107,2[a]	1,9	0,8	3,1	2,8
Heat-treated low-alloy steel 2 % Ni, 0,5 % Mo, 0,5 % C (FL-4605-HT)	6,90	HRC 34,6	2,2	1,0	3,1	2,7
Heat-treated copper steel 2 % Cu, 0,8 % C (FC-0208-HT)	6,29	HRB 97,1[a]	3,1	1,4	4,4	3,9
Heat-treated copper steel 2 % Cu, 0,8 % C (FC-0208-HT)	6,29	HRC 18,7	4,2	1,9	5,1	4,3
Heat-treated nickel steel 2 % Ni, 0,8 % C (FN-0208-HT)	6,89	HRB 105,3[a]	2,9	1,3	4,1	3,6
Heat-treated nickel steel 2 % Ni, 0,8 % C (FN-0208-HT)	6,89	HRC 30,5	3,8	1,7	4,6	3,8

[a] HRB scale with carbide-ball indenters 1/16 inch (1,587 5 mm) diameter.

The repeatability (r) and reproducibility (R) of readings were determined according to ASTM E691. The reproducibility (R) of readings predicts how closely one laboratory will approximate performing another Rockwell hardness test on the same PM specimen. If the laboratories tested two different specimens, even if they were from the same lot, then larger differences between the two test results would be expected.