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Standard Test Method for Hardness Testing of Cemented Carbides[1]

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1. Scope

1.1 This test method covers the hardness testing of cemented carbides by use of the Rockwell hardness tester with the Rockwell A scale (diamond indenter and 588.4 N (60 kgf) load) in the range of Rockwell A80 and above. Also covered are the procedures for the testing and selection of diamond indenters, the management and traceability of the four levels of standard test blocks, the acquisition of secondary standard test blocks, and the making and calibration of working standard test blocks.

1.2 The Rockwell hardness tester is a convenient and reliable means of measuring the hardness of cemented carbides. A hardness value is obtained easily, but it is subject to considerable error unless certain precautions are observed.

1.3 Test Methods E 18 shall be followed except where otherwise indicated in this test method.

1.4 The values stated in SI units are to be regarded as standard. The values given in parentheses are for information only.

1.5 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

E 18 Test Methods for Rockwell Hardness and Rockwell Superficial Hardness of Metallic Materials[2]

E 29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications[3]

2.2 *ISO Standard:*

3738-2 Hardmetals—Rockwell hardness test (Scale A)—Part 2, Preparation and calibration of standard test blocks[4]

3. Significance and Use

3.1 Rockwell hardness is one of the more important properties used to evaluate cemented carbides. For compositional groups of cemented carbides, hardness is an indication of wear resistance and toughness. Lower hardness grades usually indicate less wear resistance but greater toughness. For a specific grade of cemented carbide, hardness is an indication of the metallurgical quality of the material. In no case is hardness the only property to be considered in evaluating cemented carbides.

4. Apparatus

4.1 *Scale*—All hardness tests shall be made on the regular (as opposed to superficial) Rockwell tester, using a 588.4 N (60 kgf) load (Rockwell A scale).

4.2 *Effect of Vibration*—The Rockwell hardness tester should be located in a vibration-free area in order to avoid erroneous results. If this is not possible, the tester shall be mounted so as to minimize vibrations, since vibrations tend to cause erratic readings.

4.3 *Indenter*—The standard indenter shall be selected, in accordance with the Annex to this test method, from diamond cone indenters specified for Rockwell A scale use and in conformance with Test Methods E 18.

4.3.1 The indenter, and an indentation made with it, in hardened steel or cemented carbide should be examined optically at approximately 50-diameter magnification for defects, conformance to shape, and mounting of the diamond. Examination should be made when selecting an indenter, occasionally during use, and whenever some event may be suspected of having damaged the diamond or its mounting.

4.4 *Anvils*—Select an anvil suitable for the specimen to be tested. The shoulder of the screw and the mating surface of the anvil should be clean. Seat the anvil securely. For the best accuracy, flat test pieces should be tested on a flat anvil of approximately 6-mm (¼-in.) diameter. The bearing surface of this anvil, with a Rockwell C hardness of at least 60, shall be polished smooth and be free of pits and heavy scratches. The test piece should be supported suitably, with the test surface perpendicular to the line of travel of the indenter. Dust, dirt, grease, or scale should not be allowed to accumulate on any part of the apparatus, as this will affect the results.

4.5 *Test Blocks*—Secondary standard test blocks or working standard test blocks that have been prepared and calibrated in

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[2] *Annual Book of ASTM Standards*, Vol 03.01.

[3] *Annual Book of ASTM Standards*, Vol 14.02.

[4] Available from American National Standards Institute, 11 W. 42nd St., 13th floor, New York, NY 10036.

accordance with the Annex to this test method shall be used.

5. Test Specimens

5.1 *Size of Specimens*—A minimum thickness of 1.6 mm (1⁄16 in.) is recommended. With thinner specimens, breakage may occur, resulting in damage to the anvil, the indenter, or both. Specimens that have enough overhang to cause imbalance shall be supported properly. The 6-mm (¼-in.) anvil will support flat test specimens up to approximately 113 g (¼ lb) and will also support the standard test blocks recommended previously.

5.2 *Preparation of Test Specimens*—The finish of the test surface is of major importance. The surface to be tested should be prepared to obtain a roughness of Ra ≤0.2 µm (8 µin.) A coarser finish will provide a wider range of readings. Preparation shall be conducted in such a way that alteration of the surface due to heat or cold-working is minimized. A 220-grit medium hardness resinoid bond diamond wheel, downfed 0.01 mm (0.0005 in.) per pass with abundant flow of coolant, should provide the desired surface. The thickness of the layer removed from an as-sintered surface to be tested shall be not less than 0.2 mm (0.008 in.).

5.3 The surfaces of the test specimen shall be flat and parallel within one part per hundred parts in general practice, but within one part per thousand parts when critical comparisons are being made. The surface in contact with the anvil shall be free of any irregularity (for example, a previous hardness indentation). Taper that results in the test surface not being normal to the axis of the indenter, or irregularity that causes instability during the test, will result in error.

5.4 When determining the hardness of a test specimen with a curved surface, the radius of curvature shall not be less than 15 mm (9⁄16 in.). If less, then a flat surface at least 3-mm (⅛-in.) wide shall be prepared on which to conduct the test, and there shall be an opposite flat surface such that the specimen conforms to the requirements of 5.2 and 5.3. If the test surface is curved or the opposite surface must be supported in a V-anvil, the repeatability and reproducibility limits of 8.2 and 8.3 may not apply.

5.5 *Preparation of Mounted Carbides*—Remove mounted carbides from the steel body by heating or some other convenient method. All braze metal or other bond material shall be removed from both the test surface and the opposite face. The specimen should then be prepared as described in 5.1 through 5.4.

6. Procedure

6.1 Procedures that are not described in this test method shall conform to those of Test Methods E 18.

6.2 Disregard the first two readings after an indenter has been newly mounted.

6.3 Limit the speed of applying the major load so that the movement of the weights is completed in 4 to 6 s, with no test piece on the testing equipment and with the machine set to apply a major load of 60 Kg. Verification should be by direct observation of the weight motion, if visible.

6.4 Do not permit the time of maintaining the major load after the motion of the needle or the changing of the digital readout has ceased to exceed 2 s. Removal of the major load should be gradual by operating lever in manual machines or by motor in automatic machines, and should not exceed two additional seconds. On manual machines, abrupt actuation of the major load trip lever may affect the hardness value obtained. Abrupt actuation of the major load removal lever will significantly affect the hardness value obtained.

6.5 The Rockwell A hardness value is read after the major load has been removed and while the minor load is still applied.

6.6 The distance between the centers of any two adjacent indentations, and the distance between the center of any indentation and the edge of a test specimen, shall be at least 1.5 mm (0.06 in.).

6.7 Hardness should be read or estimated to the nearest 0.1 HRA. Calculations should be carried to two decimal places.

6.8 Make two trial determinations of the hardness of the test specimen. This action also reassures that the indenter is seated properly.

6.8.1 Select the standard test block having a value closest to the trial hardness of the test specimen. Determine the Rockwell A hardness at three points on the block.

6.8.2 If the arithmetic mean of the three determinations differs from the certified hardness value of the standard test block by more than± 0.5 HRA, check the diamond indenter and the testing equipment, and eliminate the cause of the error. Repeat the determinations.

6.8.3 If the arithmetic mean of the three determinations differs from the certified hardness value of the standard test block by ±0.5 HRA or less, record the difference, giving due regard to the algebraic sign. This difference will be used to correct the arithmetic mean of the hardness of the test specimens.

6.8.4 Determine the Rockwell A hardness of the test specimen, with determinations at three or more locations chosen at random, or as dictated by the purpose of the test.

6.8.5 Calculate the arithmetic mean of the hardness determinations. Apply the correction determined as in 6.8.3, giving due regard to the algebraic sign.

6.8.6 Report the corrected arithmetic mean of the hardness determinations, rounded in accordance with Practice E 29[5] to the nearest 0.1 HRA.

7. Report

7.1 Report the following information:

7.1.1 All details necessary for identification of the test specimen,

7.1.2 The corrected mean hardness,

7.1.3 The range of hardness determinations,

7.1.4 The number of hardness determinations,

7.1.5 The smallest division of readout or graduation of the hardness test machine and whether it is digital or analog,

7.1.6 The identification and original source of calibration for the standard test blocks used,

[5] When the second decimal place is less than 0.05, leave the first decimal place unchanged. When the second decimal place is more than 0.05, increase the first decimal place by 0.1. When the second decimal place is exactly 5 and the first decimal place is odd, increase the first decimal by 0.1. If the first decimal place is even, leave it unchanged.

7.1.7 A reference to this test method, and

7.1.8 Details of any deviations from this test method, of optional procedures used, and of any conditions and occurrences that may have affected the results.

8. Precision and Bias [6]

8.1 The following statements regarding the repeatability and reproducibility of hardness (HRA) measurements of cemented carbide test specimens shall apply only within the hardness range established for the indenter in accordance with A1.8.2 or A1.8.3. See Table A1.1.

8.2 The repeatability limit (r) is 0.3 HRA. On the basis of test error alone, the difference in absolute value of two test results obtained in the same laboratory on the same test specimen will be expected to exceed 0.3 HRA only approximately 5 % of the time. The repeatability standard deviation (S_r) is 0.1 HRA.

8.3 The reproducibility limit (R) between or among laboratories is 0.4 HRA when each has calibrated its machine, indenter, and operator system with a standard test block that has itself been calibrated to the same superior test block used to calibrate the test blocks of the other laboratories. On the basis of test error alone, the difference in absolute value of the test results obtained in different laboratories on the same test specimen will be expected to exceed 0.4 HRA only approximately 5 % of the time. The reproducibility standard deviation (S_R) is 0.14 HRA.

8.4 Neither the data of the interlaboratory study nor theoretical considerations suggest a bias in this test procedure.

8.5 If the test specimens are of a hardness substantially outside the hardness ranges of the standard test blocks on which the indenter has been performance tested, and if interlaboratory reproducibility is critical, the same indenter and standard test blocks should be used by each laboratory.

9. Keywords

9.1 cemented carbides; hardness; indenters; Rockwell hardness test; Scale A; test blocks

[6] The statements of repeatability and reproducibility in this section are based on an interlaboratory study conducted by the Cemented Carbide Producers Association.

ANNEX

(Mandatory Information)

A1. PREPARATION, CALIBRATION, AND CONTROL OF STANDARD TEST BLOCKS AND SELECTION OF SCALE A INDENTERS USED IN THE PERFORMANCE OF THE PROCEDURES OF THIS TEST METHOD

A1.1 *Scope and Field of Application*—This Annex specifies the control of master, primary, secondary, and working standard test blocks. It specifies the preparation and calibration of primary, secondary, and working standard test blocks. It also specifies the procedure for selecting indenters having the required precision from standard Scale A indenters. Both test blocks and indenters complying with this Annex are required for Rockwell hardness testing of cemented carbides by the procedures of this test method.

A1.2 *Hierarchy and Availability of Standard Test Blocks:*

A1.2.1 Secondary standard test blocks, and the calibration or recalibration services for secondary standard test blocks, are available from the authorized calibrating agency. To provide traceability to the master standard test blocks, the Cemented Carbide Producers Association (CCPA) has released Set 2, master standard test blocks, to the authorized calibrating agency.[7]

A1.3 *Master Standard Test Blocks:*

A1.3.1 Of three sets of five master standard test blocks, Set 1 is retained by the CCPA. Set 2 has been released by the CCPA to Wilson Instruments, so that Wilson Instruments may serve as the calibrating agency for secondary standard test blocks traceable through primary standard test blocks to the set of master standard test blocks. Set 3 is retained by the Secretariat of ISO/TC 119.

A1.3.2 The sets of master standard test blocks retained by the CCPA and ISO/TC 119 shall be kept as permanent standards and shall be used only when calibration of a new master standard is required.

A1.3.3 Blocks retained by ISO/TC 119 shall be retained as unused international standards. Only in the event of destruction of some or all of Set 1 retained by the CCPA, and then only by majority vote of the members of ISO/TC 119/SC4, shall they be released to be used for the calibration of new master standard test blocks. They shall then be returned to ISO/TC 119.

A1.3.4 The markings and internationally agreed upon hardnesses and standard deviations of three sets of the five master set blocks are given in Table A1.2, Table A1.3, and Table A1.4.

A1.3.5 The blocks shall not be reground, and, to ensure that this has not been done, their thickness is to be measured and recorded at the time of the calibration.

A1.4 *Primary Standard Test Blocks:*

A1.4.1 Primary standard test blocks shall be prepared and

TABLE A1.1 Control of Test Blocks

Level	Name	Control	Available
1	Master (M)	CCPA	Not applicable, see text
2	Primary (P)	CCPA	Not applicable, see text
3	Secondary (S)	User	Calibrating agency
4	Working (W)	User	Various sources

[7] The Cemented Carbide Producers Association has authorized Wilson Instruments, 6 Emma St., Binghamton, NY 13905, to be the calibrating agency.

TABLE A1.2 Set 1 Retained by CCPA

Marking	Hardness	Standard Deviation	Thickness, in.
2 Series I	85.70	0.07	0.260
6 Series II	88.64	0.07	0.270
8 Series III	91.08	0.06	0.280
2 Series IV	91.59	0.04	0.304
8 Series V	92.80	0.05	0.280

TABLE A1.3 Set 2 Released to Calibrating Agency

Marking	Hardness	Standard Deviation	Thickness, in.
4 Series I	85.68	0.08	0.260
10 Series II	88.63	0.08	0.270
6 Series III	91.06	0.06	0.280
12 Series IV	91.62	0.04	0.304
5 Series V	92.81	0.08	0.280

NOTE 1— The standard deviation is calculated on 40 indentations made in such a way as to cover the whole test surface.

TABLE A1.4 Set 3 Retained by the Secretariat of ISO/TC 119

Marking	Hardness	Standard Deviation	Thickness, in.
6 Series I	85.69	0.07	0.260
5 Series II	88.56	0.07	0.270
1 Series III	91.06	0.06	0.280
14 Series IV	91.60	0.06	0.304
6 Series V	92.79	0.08	0.280

calibrated in compliance with ISO 3738-2.

A1.4.2 Primary standard test blocks shall be reserved for use by the calibrating agency to calibrate secondary standard test blocks.

A1.5 *Secondary Standard Test Blocks:*

A1.5.1 Secondary standard test blocks shall be prepared and calibrated in compliance with ISO 3738-2.

A1.5.2 Secondary standard test blocks shall be used to calibrate working standard test blocks, or they may be used to verify Rockwell hardness testing machines (Scale A) and indenters for testing hard metals when a higher degree of confidence is desired than may be possible with working standard test blocks.

A1.5.3 Secondary standard test blocks shall have a nominal diameter of 45 mm and a nominal thickness of 8 mm. The bottom face shall be chamfered 0.8 mm by 45°.

A1.6 *Preparation, Calibration, and Marking of Working Standard Test Blocks:*

A1.6.1 Working standard test blocks may be prepared by the user.

A1.6.2 Working standard test blocks shall be prepared and calibrated against secondary standard test blocks. They should be used for routine hardness testing, so that the test surfaces of the secondary hardness test blocks are preserved for test block calibration and other critical hardness testing.

A1.6.3 Working standard test blocks may be prepared in sets of from two to five (depending on the hardness ranges of test pieces to be measured), so as to have some or all of the following nominal hardnesses: 93, 92, 91, 88.5, and 85.5 HRA.

A1.6.4 Each block shall be marked permanently on the periphery with the letter "W" and an appropriate code such that it can be related unmistakably to a record of its most recent calibration.

A1.6.5 Working standard test blocks shall conform to the following conditions: all working standard test blocks shall comprise hardmetals composed substantially of tungsten carbide and cobalt without other carbides or with less than a total of 1 % (m/m) of other carbides (for example, of titanium, tantalum, and niobium); the composition and structure shall be chosen to provide the desired hardness; and free carbon and etaphase shall be absent.

A1.6.6 Working standard test blocks should be not more than 45-mm (1.75-in.) diameter and not less than 5-mm (0.2-in.) thickness. The bottom face shall be chamfered 0.8 mm (1/32 in.) by 45°.

A1.6.6.1 Working standard test blocks shall be ground on both flat faces.

A1.6.6.2 The face on which indentations are to be made shall have had a minimum of 0.35 mm (0.014 in.) ground off of the as-sintered surface and shall have a surface finish of Ra ≤0.2 µm (8 µin.), and it may be polished. The maximum deviation in flatness of the surfaces shall not exceed 0.010 mm (0.0004 in.). The bottom of the blocks shall not be convex. The maximum deviation in parallelism shall not exceed 1 part per 2500 parts.

A1.6.7 A secondary standard test block traceable to a CCPA master standard test block shall be chosen that has a hardness nearest to that of the working standard test block, and ten indentations shall be made on its test surface. The arithmetic mean of the ten results shall be calculated to the nearest 0.01 HRA and subtracted from the certified hardness of the secondary standard test block. The result is the correction for the given combination of testing machine and indenter.

A1.6.8 If the correction is greater than 0.3 HRA absolute, the machine and indenter shall be examined to ascertain the cause, and the test shall then be repeated.

A1.6.9 If the correction is ≤0.3 HRA absolute, ten indentations shall be made on the test surface of the candidate working standard test block, and the standard deviation of the results shall be calculated.

A1.6.10 Standard deviation, s,

$$= \sqrt{\frac{\Sigma(x - \bar{x})^2}{n - 1}} \qquad \text{(A1.1)}$$

where:

x = the individual hardness result,
$\bar{x}$ = the arithmetic mean of hardness results in the sample, and
n = the number of hardness results in the sample.

A1.6.11 If the standard deviation is greater than 0.10, HRA, the block shall be discarded, but it shall be recorded if it is not greater than 0.10 HRA. The arithmetic mean of the ten results shall be calculated to the nearest 0.01 HRA and corrected for the error of machine and indenter obtained above. The results shall be rounded off to the nearest 0.01 HRA and shall be recorded as the hardness of the working standard test block.

A1.6.12 When calibrating a number of working standard

test blocks of the same nominal hardness in an uninterrupted series, the correction may be determined only at the commencement of the series, unless there is reason to believe that the correction may have altered.

A1.6.13 The thickness of the secondary standard test block shall be measured and recorded.

A1.6.14 The test surfaces of the working standard test blocks shall not be reground unless they are then recalibrated against secondary standard test blocks.

A1.7 *Test Report:*

A1.7.1 Supply a test report or certificate with every secondary or working standard test block, and include the following information:

A1.7.1.1 A reference to this test method, ASTM B 294,

A1.7.1.2 All details necessary for identification of the test block,

A1.7.1.3 The hardness as determined in accordance with this Annex,

A1.7.1.4 The standard deviation of hardness readings over the test surface,

A1.7.1.5 The thickness of the block and the date on which it was calibrated,

A1.7.1.6 Identification of the block from which it was calibrated,

A1.7.1.7 The name of the institution, association laboratory, or individual responsible for the calibration, and

A1.7.1.8 Any deviations from the procedures of this test method, or a statement that there have been no deviations.

A1.8 *Indenter Selection:*

A1.8.1 Select suitable indenters from standard Scale A indenters.

A1.8.2 Conduct a performance test of the indenter. Make at least three indentations on each of a series of five cemented carbide standard test blocks having the nominal hardnesses 85.5, 88.5, 91, 92, and 93 and conforming to the specifications of this Annex. Calculate the arithmetic mean of the hardness readings taken on each block. Determine the difference between the mean hardness of each block and its certified hardness, retaining the plus or minus signs. Tabulate these differences in the order of increasing hardness of the blocks. Calculate the algebraic difference between adjacent differences in the table and divide by 2. The absolute value of the resulting quotient is the maximum expected interpolation error when the indenter is used to measure the hardness of a test piece having a hardness between the hardnesses of the standard test blocks from which the quotient resulted. An indenter for which all of the quotients are 0.15 or less shall be accepted for use within a hardness range extending from the actual hardness of the hardest standard test block plus 0.3 HRA to that of the softest minus 0.3 HRA.

A1.8.3 An indenter discarded by the criteria of A1.8.2 may be accepted for use over a narrower hardness range. From the data generated in A1.8.2, select a subset of adjacent standard test blocks for which the acceptance criteria of A1.8.2 is met. The indenter may be accepted for use within a hardness range extending from the actual hardness of the hardest in the subset, plus 0.3 HRA, to that of the softest in the subset, minus 0.3 HRA.

A1.8.4 Include an example of the calculations and decisions for A1.8.2 and A1.8.3.

A1.8.5 Record the serial number of the indenter and the HRA range over which it has been approved for use, as well as the data of the selection test. The record should be posted, with indenters flagged in a manner such that they are used only within the approved range.

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