National Institute of Standards & Technology

Certificate

Standard Reference Material® 1893

Copper Microhardness Test Block (Knoop)

Lot # 09

Serial No. SAMPLE

This Standard Reference Material (SRM) is intended for use as a primary standard in calibrating Knoop-type microhardness testers and is certified for mean Knoop hardness values at loads of 0.245 N, 0.490 N, and 0.980 N (0.025 kgf, 0.050 kgf, and 0.100 kgf, respectively). A unit of SRM 1893 consists of a square test block of electrodeposited bright copper on an AISI 1010 steel substrate. The test block measures 1.35 cm on each side, is approximately 1.5 mm thick, and is mounted in a thermosetting epoxy. Five indentations were made on the SRM's polished surface for each load at positions illustrated in Figure 1. The mean Knoop hardness value and the corresponding expanded uncertainty for the mean of the five indentations made for each load tested are presented in Table 1. Knoop hardness values are reported as Knoop hardness numbers (HK) in units kgf/mm^2 and in SI units of gigapascals (GPa). Each SRM was individually measured and bears a serial number imprinted on the side of the epoxy mount.

Table 1. Certified Mean HK and Expanded Uncertainties

Load		Mean HK	
N	(kgf)	GPa	(kgf/mm^2)
0.245	(0.025)	SAMPLE	(SAMPLE)
0.490	(0.050)	SAMPLE	(SAMPLE)
0.980	(0.100)	SAMPLE	(SAMPLE)

Expiration of Certification: The certification of **SRM 1893** is valid indefinitely, within the measurement uncertainty specified, provided the SRM is handled, stored, and used in accordance with the instructions given in this certificate (see "Instructions for Handling, Storage, and Use"). Accordingly, periodic recalibration or recertification of this SRM is not required. Aside from indentation, any physical damage or other alteration of the surface of the specimen, including all processes that remove surface material such as repolishing, will invalidate the certification.

Maintenance of SRM Certification: NIST will monitor this SRM over the period of its certification. If substantive technical changes occur that affect the certification, NIST will notify the purchaser. Registration (see attached sheet) will facilitate notification.

The coordination of the production and the technical measurements leading to the certification of SRM 1893 were performed by D.R. Kelley of the Thin Film and Nanostructure Processing Group of the NIST Metallurgy Division.

Statistical analysis of the data was performed by N.F. Zhang of the NIST Statistical Engineering Division.

Support aspects involved in the issuance of this SRM were coordinated through the NIST Measurement Services Division.

Frank W. Gayle, Chief
Metallurgy Division

Robert L. Watters, Jr., Chief
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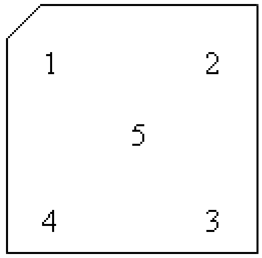
Gaithersburg, MD 20899
Certificate Issue Date: 04 September 2012
Certificate Revision History on Last Page



SRM 1893 Serial No.: SAMPLE
Calibrated by: D.R. Kelley

Figure 1. Positions of Indentations

NIST Specimen Preparation: The starting material for this SRM was a flat sheet of electroformed copper, 23 cm × 46 cm × 2 mm, over a steel substrate. The copper sheet/steel substrate combination was cut into coupons that were mounted and highly polished to provide flat and parallel surfaces suitable for Knoop microindentations. After polishing, the 0.1 µm layer of gold was electroplated over the coper to prevent corrosion. Finally, the samples were indented and the Knoop hardness measured.

Certification Procedure: The Knoop microindentations are located in the center and near the four corners of the block as illustrated in Figure 1. These indentations were made in accordance with the Knoop hardness test principle, in which a diamond indenter, in the form of a rhombic-based pyramid, is forced into the surface of a test piece followed by measurement of the long diagonal length of the indentation left in the surface after removal of the test force. The full test force was applied for 12.5 seconds with one set of five indentations made for each load. The certified mean HK of this block at three different loads are given in Table 1. Analysis of variance (ANOVA) of all data collected led to the treatment of load and position as statistically significant factors.

Hardness values for this SRM were obtained using a dedicated, calibrated hardness tester. The loading mechanism of the hardness tester was calibrated with a miniature precision load cell that was calibrated with NIST-certified weights. The indentation sizes were measured on an optical microscope using a filar micrometer and/or an image analyzer calibrated with a NIST-certified stage micrometer. When using the 10 X filar micrometer for measuring the indentation size, the total magnification was 1000 X using a 100 X dry objective lens with a numerical aperture (NA) of 0.90. For measurement with the image analyzer, the microscope 100 X dry objective lens was used with a 1 X video camera lens.

The HK is obtained by dividing the applied force to the indenter by the projected area of the permanent indentation made by the diamond indenter. The value is computed from the following equation (taking into consideration the Knoop indenter geometry shown in Figure 2):

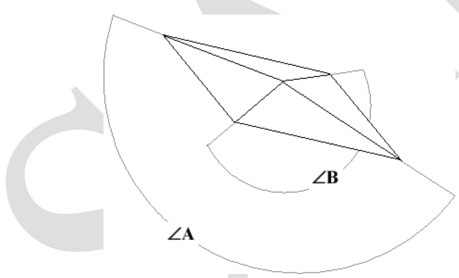


Figure 2. Knoop indenter geometry showing ∠A, the included longitudinal edge angle, and ∠B, the included transverse angle

$$HK = \frac{2 \cdot \tan\left(\frac{\angle A}{2}\right)}{\tan\left(\frac{\angle B}{2}\right)} \frac{P}{d^2} \quad (1)$$

Where ∠A and ∠B are the included angles for the indenter as shown in Figure 2 above, P is the applied force to the indenter, and d is the length of the long diagonal of the indentation. For the ideal indenter, ∠A = 172°30' and ∠B = 130°0', thus, transforming Equation 1 into:

$$HK = \frac{14.229 \quad P}{d^2} \quad \text{kgf/mm}^2 \qquad (2)$$

where P is expressed in kgf and d in mm.

Since the units of gram-force (gf) and micrometers (µm) are normally used in this field, the constant in Equation 2 can be modified to accommodate the conversion factors to ease use during computation. The equation for Knoop hardness, still expressed in kgf/mm^2, becomes:

$$\mathrm{HK} = \frac{14229\ \mathrm{P'}}{d'^2}\ \mathrm{kgf/mm}^2 \tag{3}$$

where P' is expressed in gf and d' in µm.

To express the HK in SI units of GPa, with P'' in Newtons and d in mm, the constant must be further modified to obtain

$$\mathrm{HK} = \frac{0.014229\ \mathrm{P''}}{d^2}\ \mathrm{GPa} \tag{4}$$

Discussion of Uncertainty: The uncertainty in the average certified value of the Knoop hardness measurement is expressed as an expanded uncertainty, U, at the 95 % level of confidence, and is calculated according to the method described in the ISO Guide [1]. The expanded uncertainty is calculated as $U = ku_c$, where u_c represents the combined effect of the individual sources of uncertainty listed in Table 2 and $k = 2$ is the coverage factor. These sources include uncertainties due to deviations of the NIST standardizing tester and indenter from the defined requirements for the hardness test (force, diagonal measurement, test cycle, and indenter shape). The value of u_c is individually calculated for each SRM unit and load. The expanded uncertainty, U, provides an indication of the precision of NIST's estimate of the true value of the average hardness of this SRM. The uncertainty should not be interpreted as the range in expected hardness values that would be measured across the test surface, nor is it a limit of acceptable hardness values for verifying the hardness equipment. Similarly, the uncertainty components listed in Table 2 contribute to the overall uncertainty in the certified average hardness value. These are not ranges of expected hardness results due to each source of uncertainty.

Table 2. Sources of Uncertainty for the Certified Average HK

Type	Uncertainty Source	Uncertainty in kgf/mm^2 (at the load listed)			Notes
		0.025 kgf	0.050 kgf	0.100 kgf	
A	Material Uniformity and Measurement Repeatability	Standard Deviation, s, of 15 Measurements			$s/\sqrt{15}$
B	Hardness Tester	0.569	0.361	0.270	u_{b_1} sources: load diagonal measurement indenter dwell time
B	Standardizing Indenter (bias)	–1.20	–1.20	–1.22	$u_{b_2} = h/\sqrt{3}$

For each load, one indentation is made at each of the five positions shown in Figure 1. The long diagonal of each indentation is measured three times. The average value for the diagonal length and the corresponding standard deviation are listed as information values in Table 3. The average of the 15 hardness values calculated using each diagonal length is used as the certified hardness value. The standard deviation for the average of the measurements of the five indents is calculated as the Type A uncertainty given by $s/\sqrt{15}$, where s is the sample standard deviation. For the Type B uncertainty, two components are considered. The first component is denoted u_{b_1} with its sources listed in Table 2. The second component, u_{b_2}, is due to indenter bias. The bias error is considered to have a uniform distribution with the magnitude of the bias corresponding to the height of such distribution, denoted as h. Therefore, the standard uncertainty is $u_{b_2} = h/\sqrt{3}$. The two components are combined using a root-sum-of-squares (RSS) calculation, to yield the standard uncertainty, $u_b = \sqrt{u_{b_1}^2 + u_{b_2}^2}$. Finally, the standard uncertainty of the mean, u_c, is a combination of the Type A and the Type B uncertainty, also by using RSS calculation.

Table 3. Information Values

Load		Length of the Long Diagonal	
N	(kgf)	Mean, µm	Standard Deviation, s, µm
0.245	(0.025)	SAMPLE	SAMPLE
0.490	(0.050)	SAMPLE	SAMPLE
0.980	(0.100)	SAMPLE	SAMPLE

INSTRUCTIONS FOR HANDLING, STORAGE, AND USE

The metallic block is durable but may be susceptible to tarnish or corrosion in an environment of high humidity and/or acidic sulfur or chlorine-bearing gases or liquids. Oils, fingerprints, or skin oils should be removed from the SRM before and after use. The SRM unit may be cleaned with ethyl alcohol and soft wipe materials. The surface polish should be protected from abuse. The blocks must **NEVER BE REPOLISHED**, as this will invalidate the certification.

This SRM is intended for use with microhardness testing machines whereby a Knoop indentation can be made and then measured with an optical microscope. When using this SRM, a **minimum of five indentations must be made** for comparison to the certified mean Knoop hardness value and expanded uncertainty.

When making new indentations in the block, special care should be taken to ensure that the loading rates and load duration are as prescribed by the applicable Knoop hardness test method standard. There should be no vibrations or impact imparted to the machine during the indentation cycle. The surface of the test block and the indenter must be clean and should not contain skin oils, which could alter the friction between the indenter and block surface. Indentations may be placed in any region of polished surface provided that they are not within 1.9 mm of any edge, since slight edge rounding from polishing can distort the indentation shape and affect the size. Guidelines for indentation spacing can be found in applicable Knoop hardness test method standards.

When measuring indentations, proper illumination and focus of the indentation tips are critical to obtain good clarity and contrast. The apparent indentation size will be affected by the magnification used since the numerical aperture of the objective lens establishes the resolution limits.

Magnifications should be checked by use of a calibrated stage micrometer. FSilar micrometers and image analyzing systems should be calibrated with stage micrometers. Proper use of filar crosshairs is essential. For best results, it is critical that the instructions of the hardness machine manufacturer and the applicable Knoop hardness test method standard be followed. Additional information on the preparation of this SRM can be found in references 2 and 3.

REFERENCES

[1] JCGM 100:2008; *Evaluation of Measurement Data – Guide to the Expression of in Measurement* (ISO GUM 1995 with Minor Corrections); Joint Committee for Guides in Metrology (JCGM) (2008); available at http://www.bipm.org/utils/common/documents/jcgm/JCGM_100_2008_E.pdf (accessed Sep 2012); see also Taylor, B.N.; Kuyatt, C.E.; *Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results*; NIST Technical Note 1297; U.S. Government Printing Office: Washington, DC (1994); available at http://www.nist.gov/pml/pubs/index.cfm (accessed Sep 2012).
[2] Kelley, D.R.; Johnson, C.E.; Lashmore, D.S.; *Electroformed Microhardness Standards*; Proceedings 37th Meeting of the Mechanical Failure Prevention Group, Cambridge University Press, pp. 55-58 (1984).
[3] Kelley, D.R.; Johnson, C.E.; Lashmore, D.S.; *Fabrication and Certification of Electroformed Microhardness Standards*; Proceedings of IMS/ASTM Meeting, ASTM Special Technical Publication 889, pp. 186-195 (1985).

Certificate Revision History: 04 September 2012 (Certification procedure updated; editorial changes); 20 July 2011 (Original certificate date).

Users of this SRM should ensure that the Certificate in their possession is current. This can be accomplished by contacting the SRM Program: telephone (301) 975-2200; fax (301) 948-3730; e-mail srminfo@nist.gov; or via the Internet at http://www.nist.gov/srm.