National Institute of Standards & Technology

Certificate

Standard Reference Material® 1894a

Vickers Microhardness of Copper

Serial No.: SAMPLE

This Standard Reference Material (SRM) is intended for use as a primary standard in calibrating Vickers-type microhardness testers and is certified for mean Vickers hardness values (HV) at loads of 0.245 N, 0.49 N, and 0.98 N (0.025 kgf, 0.050 kgf, and 0.100 kgf, respectively). A unit of SRM 1894a 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 1750 µm 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 Vickers HV and the corresponding expanded uncertainty for the mean of five future indentations for each load tested are presented in Table 1. Vickers hardness values are reported as Vickers HV in units of kgf/mm^2 and SI units of gigapascal (GPa). Each SRM was individually measured and bears a serial number imprinted on the epoxy mount.

Table 1. Certified Mean Vickers HV and Expanded Uncertainty

Load		Mean HV	
N	(kgf)	GPa	(kgf/mm^2)
0.245	(0.025)	SAMPLE	(SAMPLE)
0.49	(0.050)	SAMPLE	(SAMPLE)
0.98	(0.100)	SAMPLE	(SAMPLE)

Expiration of Certification: The certification of **SRM 1894a** 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 1894a were performed by D.R. Kelley of the Thin Film and Nanostructure Processing Group of the NIST Metallurgy Division. This SRM was fabricated by D.R. Kelley and C.E. Johnson and certified by H.B. Gates of the Electrochemical 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

Gaithersburg, MD 20899
Certificate Issue Date: 04 September 2012
Certificate Revision History on Last Page

Robert L. Watters, Jr., Chief
Measurement Services Division

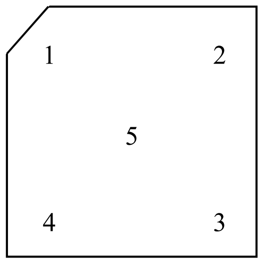

Figure 1. Position of Indentations

SRM 1894a Serial No.: SAMPLE
Calibration Date: SAMPLE
Calibrated by: H.B. Gates

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 Vickers microindentations.

NIST Certification Procedure: The Vickers 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 Vickers hardness test principle, in which a diamond indenter, in the form of a right pyramid with a square base, is forced into the surface of a test piece followed by measurement of the two diagonal lengths of the indentation left in the surface after removal of the test force. The certified mean Vickers HV of this block at three different loads are shown in Table 1. Measurements on randomly selected blocks have established that the HV is uniform across the surface of each block.

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 500 X using a 50 X dry objective lens with a numerical aperture (NA) of 0.80. For measurement with the image analyzer, the microscope 50 X dry objective lens was used with a 1 X video camera lens.

The Vickers HV is computed from the following equation [2]

$$\mathrm{HV} = \frac{2\mathrm{P}\sin(\alpha/2)}{d^2} = \frac{1.8544\,\mathrm{P}}{d^2} \quad \mathrm{kgf/mm^2} \tag{1}$$

where P is the indenter load in kilogram-force, d is the average of the two diagonal lengths in millimeters, and α is the face angle of the ideal indenter, which is 136°.

Since the units of gram-force (gf) and micrometers (µm) are normally used in this field, the constant in equation 1 can be modified to accommodate the conversion factors to ease use during computation. The equation for Vickers HV, still expressed in kgf/mm^2, becomes

$$\mathrm{HV} = \frac{1854.4\,\mathrm{P'}}{d'^2} \quad \mathrm{kgf/mm^2} \tag{2}$$

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

To express the Vickers HV in SI units of GPa, the constant must be further modified to obtain

$$\mathrm{HV} = \frac{18.185\,\mathrm{P}}{d^2} \quad \mathrm{GPa} \quad \text{or} \quad \mathrm{HV} = \frac{18185\,\mathrm{P'}}{d'^2} \quad \mathrm{GPa} \tag{3}$$

Discussion of Uncertainty: The uncertainty in the certified value of the hardness measurement is expressed as an 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 is intended to represent, at the level of one prediction standard deviation for the **mean of five future observations**, the combined uncertainty due to material variability and measurement uncertainty. The coverage factor, k, is determined from the Student's t-distribution corresponding to the 4 degrees of freedom and 95 % confidence.

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 before and after use of the SRM. 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 Vickers indentation can be made and then measured with an optical microscope. When using this SRM, a **minimum of five indentations must be made** for accurate comparison to the certified mean HV 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 Vickers 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 Vickers 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. A total magnification of 400 X or higher is recommended.

Magnifications should be checked by use of a calibrated stage micrometer. Filar 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 Vickers 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 Uncertainty 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://physics.nist.gov/Pubs/ (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, Issue 889, pp. 186–195 (1985).

Certificate Revision History: 04 September 2012 (Certification procedure updated; editorial changes); 10 October 2003 (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.