National Institute of Standards & Technology

Certificate

Standard Reference Material® 1909

Vickers Microhardness of Nickel

Serial No.:

This Standard Reference Material (SRM) is intended primarily for use in calibrating Vickers-type microhardness testers and is certified for mean Vickers hardness values (HV) at a load of 9.81 N (1.000 kgf). SRM 1909 consists of a 1.35 cm square test block of electrodeposited bright nickel, approximately 750 μm thick, on an AISI 1010 steel substrate, mounted in a thermosetting epoxy. Five indentations were made on the polished surface of the SRM at positions illustrated in Figure 1. Mean Vickers HV with the corresponding expanded uncertainty for the mean of five future indentations is presented in Table 1. Hardness value is reported in gigapascal (GPa) and kgf/mm^2. The SRM was individually measured and bears a serial number imprinted on the side of the epoxy mount.

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.

Expiration of Certification: The certification of this SRM is deemed to be indefinite, provided the SRM is stored, handled, and used in accordance with this certificate (see *Storage and Handling*). 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.

NIST Specimen Preparation: The starting material for this SRM was a flat sheet of electroformed nickel, 23 cm × 46 cm × 1 mm, over a steel substrate. The nickel sheet/steel substrate combination was cut into coupons that were mounted and highly polished to provide flat and parallel surfaces suitable for Vickers microindentations.

Storage and Handling: 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 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.

The support aspects involved in the issuance of this SRM were coordinated through the NIST Standard Reference Materials Program by B.S. MacDonald of the NIST Measurement Services Division.

Carol A. Handwerker, Chief
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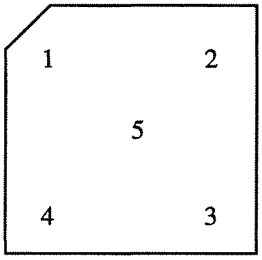
Certificate Issue Date: 10 October 2003

John Rumble, Jr., Chief
Measurement Services Division

Table 1. Certified Mean Vickers HV and Expanded Uncertainty, *U*, for the Mean of Five Future Indentations

Load		Mean HV	
N	(kgf)	GPa	(kgf/mm^2)
9.81	(1.000)		



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Calibration Date:
Calibrated by: H.B. Gates

Figure 1. Position of Indentations

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 ASTM E 384-99 Test Method for Microhardness of Materials [2]. The certified mean Vickers HV of this block is shown in Table 1.

The requirements of ASTM E 384-99 for separate verification of microhardness machines and calibration of standardized hardness test blocks, annexes A1 and A2, were followed with the exception that only one group of five indentations was made. 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]

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

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

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

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

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

INSTRUCTIONS FOR USE

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 appropriate standard, ASTM E 384-99 [2]. 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 ASTM E 384-99 [2].

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 and is in accordance with the ASTM E 384-99 [2] for an indentation diagonal length of less than 76 μm.

Magnifications should be checked by use of a calibrated stage micrometer. Also 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 appropriate test method, ASTM E 384-99 [2], be followed scrupulously. Additional information on the preparation of this SRM can be found in References [3-4].

REFERENCES

[1] *Guide to the Expression of Uncertainty in Measurement*, ISBN 92-67-10188-9, lst Ed., ISO, Geneva, Switzerland, (1993); see 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 from http://physics.nist.gov/Pubs/.

[2] ASTM E 384-99, Standard Test Method for Microhardness of Materials, ASTM Annual Book of Standards, Vol. 3.01 (2000).

[3] 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).

[4] 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).

Users of this SRM should ensure that the certificate in their possession is current. This can be accomplished by contacting the SRM Program at: telephone (301) 975-6776; fax (301) 926-4751; e-mail srminfo@nist.gov; or via the Internet http://www.nist.gov/srm.