National Institute of Standards & Technology

Certificate

Standard Reference Material® 2829

Vickers Microhardness of Steel

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 at a load of 4.90 N (0.500 kgf). A unit of SRM 2829 consists of a steel block, approximately 3.2 cm in diameter and 1 cm in height. Five indentations were made within the inscribed square on the SRM's polished surface at positions illustrated in Figure 1. The mean Vickers hardness value and the corresponding expanded uncertainty for the mean of the five indentations made are presented in Table 1. Vickers hardness values are reported as Vickers hardness numbers (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 top surface.

Table 1. Certified Mean Vickers HV and Expanded Uncertainty

Load		Mean HV	
N	(kgf)	GPa	(kgf/mm^2)
4.90	(0.500)	SAMPLE	SAMPLE

Expiration of Certification: The certification of **SRM 2829** 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 2829 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.

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Certificate Revision History on Last Page

Figure 1. Position of Indentations

SRM 2829 Serial No.: SAMPLE
Calibrated by: D.R. Kelley

NIST Specimen Preparation: A lot of uncalibrated hardness blocks was purchased from a commercial vendor. A nominal hardness value of 800 kgf/mm^2 was requested from the manufacturer. One test block was randomly selected from the lot and a test pattern of eleven rows and eleven columns of indents was performed in the block's test area. Each indentation was measured manually once. For the lot to be accepted, the resultant test data complied with the requirement of having a hardness measurement standard deviation no greater than 3 % of the mean value. Finally, each of the remaining blocks was certified according to the procedure described below.

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 full test force was applied for 12.5 seconds with one set of five indentation made. The certified mean HV of this block is given in Table 1. Analysis of variance (ANOVA) of all data collected lead to the conclusion that position is not a statistically significant factor.

Hardness value for this SRM was 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 using an image analyzer calibrated with a NIST-certified stage micrometer. When using the image analyzer, the microscope's 200 X dry objective lens was used with a 1 X video camera lens.

The HV is computed from the following equation:

$$\mathrm{HV} = \frac{1.8544\ \mathrm{P}}{d^2} \quad \mathrm{kgf/mm^2} \tag{1}$$

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 1 can be modified to accommodate the conversion factors to ease use during computation. The equation for 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 HV in SI units of GPa, with P'' in Newtons and d in millimeters, the constant must be further modified to obtain

$$\mathrm{HV} = \frac{0.0018544\ \mathrm{P''}}{d^2} \quad \mathrm{GPa} \tag{3}$$

Discussion of Uncertainty: The uncertainty in the average certified value of the Vickers 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. 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 Hardness Value

Type	Uncertainty Source	Uncertainty in kgf/mm^2 (at 0.500 kgf load)	Notes:
A	Material Uniformity and Measurement Repeatability	Standard Deviation, *s*, of 15 Measurements	$s/\sqrt{15}$
B	NIST's Hardness Tester	2.07	u_{b_1} sources: load measurement of length indenter dwell time
B	NIST Standardizing Indenter (bias)	0.10	$u_{b_2} = h/\sqrt{3}$

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 Diagonal	
N	(kgf)	Mean µm	Standard Deviation, *s*, µm
4.90	(0.500)	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 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.

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.

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://www.nist.gov/pml/pubs/index.cfm (accessed Sep 2012).

Certificate Revision History: 10 September 2012 (Certification procedure updated; editorial changes); 21 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.