HARDMEKO 2004
Hardness Measurements Theory and Application in Laboratories and Industries
11-12 November, 2004, Washington, D.C., USA

A STANDARD REFERENCE MATERIAL FOR VICKERS HARDNESS OF CERAMICS AND HARDMETALS

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Abstract – Standard Reference Material (SRM) 2831 was developed to improve Vickers hardness testing of Ceramics and Hardmetals. It may be used with conventional hardness testing machines that make indentations that are measured with an optical microscope. The SRM is a hot-isostatically pressed tungsten carbide with 12 % cobalt disk which has five indentations made at a load of 9.8 N (1 kgf). Each SRM is individually certified for the size of each of the 5 indentations, the average diagonal length (≈ 35.0 μm), and the average hardness HV1. The HV1 is nominally 15 GPa which is in middle of the hardness range for most ceramics and cutting tool carbides.

Keywords Vickers hardness, ceramics, standard reference material, tungsten carbide

1. INTRODUCTION

One attribute of most ceramics is that they are very hard. Conventional Knoop or Vickers hardness test methods are most commonly used to quantify hardness, but Rockwell and even Mohs hardness methods are occasionally utilized. Depth sensing, nano, or instrumented hardness testing will play important roles in the future, but they are most commonly used as research tools at present. Conventional Knoop or Vickers hardness data are important for commerce and materials specifications and still have value for research.

One would suppose that applying the common diamond pyramid hardness tests to ceramics would be simple, but there are many pitfalls, controversies, and surprises. Unfortunately, many in the ceramics field have been cavalier with their hardness testing and unconcerned with uncertainty or experimental error. In many instances the test load or other key information or the experimental uncertainty are simply not reported. One problem is that despite the fact that every Vickers hardness standard in the world defines Vickers hardness HV as load / contact area as shown on the left side of Fig. 1, there are many in the ceramics field who use load / projected surface area as shown on the right side of Fig. 1. As a consequence, many reported Vickers hardness numbers have little or no value since neither the test load nor the equation used are identified.

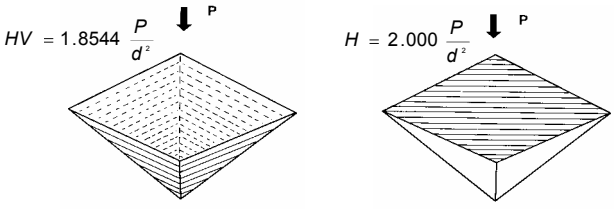

Fig. 1. The alternative hardness equations in use by the ceramics community. HV (on the left) is standardized and is preferred for all conventional hardness purposes. P is the indentation load and d is the average diagonal size.

A Versailles Advanced Materials and Standards (VAMAS) round robin project led by the National Physical Laboratory in 1989 underscored many of these problems [1]. Even with common specimens and specified procedures, hardness uncertainties of 15 % were obtained and sometimes they were much greater. A key recommendation was that ceramic reference materials should be prepared along with improved test method standards.

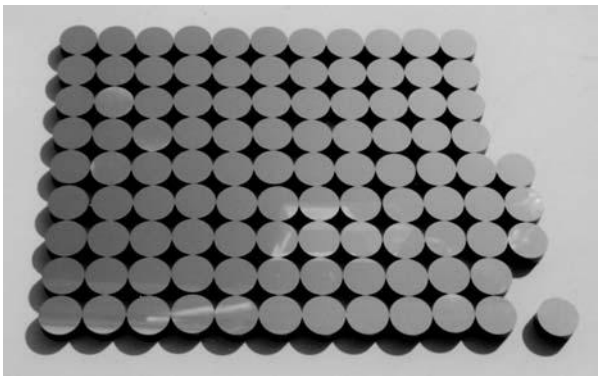

Fig. 2. The SRM 2831 hardness disks.

In response to this need, NIST began work on Knoop and Vickers SRM's in 1992. Vickers SRM 2831 was finished in 2003 (Fig. 2) after several other standards and two other SRM's including the Knoop Ceramic SRM 2830 had been completed.[a] The delay was actually beneficial since the laborious scanning electron microscope methodology used in the early 1990's for the Knoop SRM was replaced by a more modern and efficient digital optical technology that became available in 2000. Preliminary findings on prototype batches of the two SRM's have been reported previously [2-3].

[a] SRM's 2830 and 2831 area available from the SRM Customer Service Office, NIST, srmorder@nist.gov, (+001) 301 975 6776.

The SRM 2830 and 2831 work was part of a coordinated international effort to improve the quality of ceramic hardness data and testing procedures. This multifaceted effort included the preparation of standard test methods, the preparation of complementary reference materials, and the execution of round robins. Rigorous, high quality test standards tailored to the special requirements of ceramics were prepared by ASTM International, the European Committee for Standardization (CEN), and the International Organization for Standardization (ISO). Several major round robins were conducted under the auspices of the VAMAS program and other organizations. Consensus approaches emerged through this international cooperation, to the extent that the national and regional standards (Table 1) are almost identical. A consensus on an ISO test method was quickly reached, a draft easily prepared, and a formal standard approved in a remarkably short time in 2000.

SRM 2831 supports all these Vickers standard test methods. Table 1 also lists the Knoop standards for which SRM 2830 may be used.

Table 1 Standard Test Methods for Ceramics. The primary indentation loads are listed. Several standards recommend use of a range of loads for an ISE evaluation.

Standard	Method	Test Load (N)	Year adopted	NIST SRM
ASTM C 1326 [4]	Knoop	19.6, 9.8	1996	2830
ASTM C 1327 [5]	Vickers	9.8	1996	2831
ASTM C 849 [6]	Knoop	9.8	1976	2830
CEN ENV 843-4 [7]	Vickers Knoop Rockwell	9.8 19.6, 9.8 45	1994, 2000	2831 2830
JIS R 1610 [8]	Vickers	9.8, 98.	1991	2831
ISO 14705 [9]	Vickers Knoop	9.8 19.6, 9.8	2000	2831 2830

2. CERAMIC HARDNESS

Although the same Vickers and Knoop hardness methods that are used for metals may be applied to ceramics, some nuances about ceramics make them more difficult to test than metals. Fig. 3 shows some of the problems with ceramics. Many are transparent or translucent and the indentation tips and diagonal lengths are more difficult to see clearly. Ceramic indentations also are much smaller than those metals and smaller indentations are more difficult to measure. Cracking around the indentations, especially at the tips, pose another set of problems. Some researchers feel that any cracking invalidates an indentation while others feel cracking is integral to the indentation process. Uncracked Vickers indentations cannot be made at all in some ceramics and nearly all glasses, unless very low indentation loads are used (e.g., < 5 N for some silicon carbides). At these loads, the hardnesses are on a steep portion of the indentation size effect (ISE) curve, whereby hardness rapidly decreases with load, so that hardnesses comparisons are difficult.

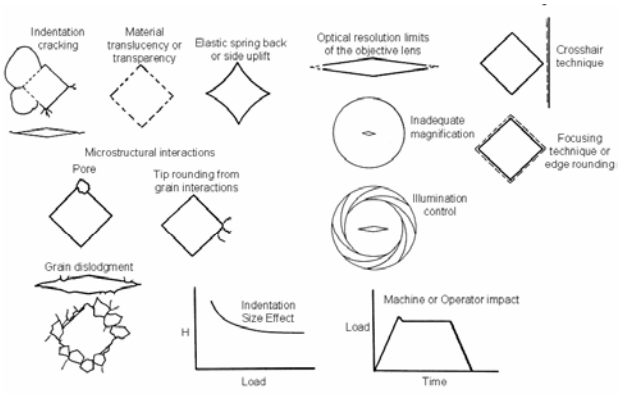

Fig. 3. Common problems in ceramic hardness testing.

Hardness is a measure of resistance to penetration by a specified indenter under specified loading conditions. In ceramics, the process of forming a permanent impression is controlled not merely by classical plastic deformation, but also by densification, displacement, and fracture. Densification is important since porosity is often present in sintered ceramics. Densification may also occur in some glasses and even nearly fully-dense ceramics with traces of microporosity. Cracking around indentations is not merely an interference, but is a critical component of the indentation process. Some micro fissuring or microfracture is almost always underneath or in the vicinity of a ceramic or glass indentation.

Our primary goal was to prepare hardness reference disks that could be used to verify proper conventional hardness machine operation and operator technique. It was decided at an early stage to choose a dense material that did not crack, was opaque, and which could take a good polish. Although several ceramics such as silicon nitride were considered, a commercial tungsten carbide was selected for the Vickers reference material.[b]

3. MATERIAL

Most structural ceramics for which hardness is important have hardnesses in the 10 GPa to 20 GPa range. It was decided that the SRM should have a hardness of about 15 GPa. A fully dense, fine-grained tungsten carbide[c,d] with nominal 12 % volume fraction cobalt was selected. The material was hot isostatically pressed and was obtained in the form of 25.4 mm diameter disks that were 9.3 mm thick. These were polished using conventional 1 μm diamond paste. Eleven prototype disks were acquired in 1993 and used for preliminary analyses and the round robins. A

[b] A commercial silicon nitride was used for Knoop SRM 2830 since there was much less cracking with the Knoop indenter.

[c] Kennametal Grade kf 312, Latrobe, PA.

[d] Certain commercial equipment, instruments, or materials are identified in this paper to specify adequately the experimental procedure. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

subsequent main lot of one hundred and eight disks was acquired in 1995 for the SRM 2831 production series.

Figs. 4 and 5 show scanning electron microscope (SEM) images of a typical 9.8 N indentation (≈ 35 μm diagonal size) and the fine microstructure. The microstructure was analysed with a sequential etching procedure in accordance with ASTM B 657 [10] using scanning electron micrographs at 1500 X. Only tungsten carbide and the cobalt binder phase were detected. The mean grain size of the tungsten carbide measured in accordance with ASTM E 112 (mean linear intercept) [11] using 5000 X SEM photos blown up by a factor of two, was 0.52 μm ± 0.29 μm (1 std. dev.). Representative images across the surface on one disk showed there were no microstructural variations.

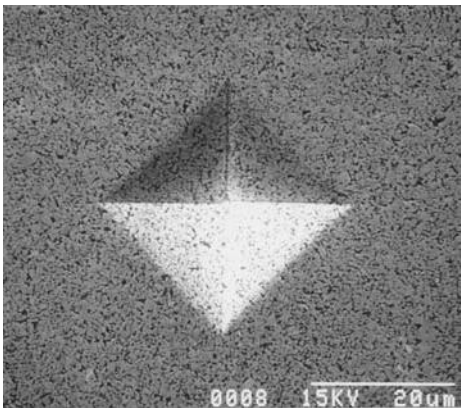

Fig. 4. SEM image of a 9.8 N indentation that is ≈ 35 μm in size. Microstructural interactions affect the tip shapes and the top tip is especially difficult to discern.

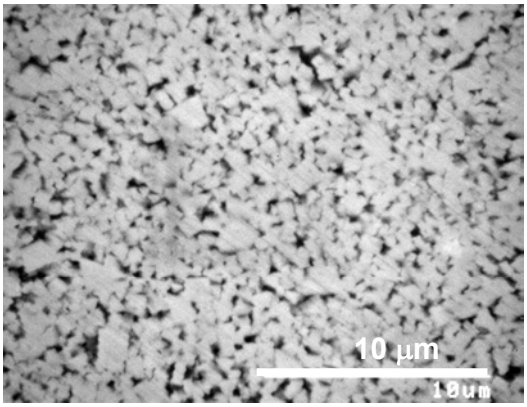

Fig. 5. SEM close-up of the microstructure of SRM 2831.

This tungsten carbide corroded if left exposed to water, fingerprint oils, or salts for any length of time greater than a few days. The corrosion appeared as a thin white haze over portions of the polished surface. Micro pitting also occurred in some areas. Experiments were done with different tissue papers, anticorrosion papers, plastic bags, and envelopes. All of the protective wrappings work well if the surface is clean to begin with. SRM 2831 is delivered wrapped in a simple white tissue. This tissue may be replaced. Ethanol or other alcohols that have some water solubility should not be used to clean the disks. Methanol is very effective and is recommended on the SRM certificate.

Several disks were repolished to remove the corrosion haze or to remove small handling scratches. Minor damage could be removed with a light repolish of 0.5 μm diamond paste with lubricating oil for 1 min. Moderate damage could be removed with 9 μm and 0.5 μm diamond paste with oil lubricant for between 1 to 5 min. The hardness was unchanged. It was discovered that although the corroded disks may have had had their surfaces restored to a fine polish, the corrosion film rapidly recurred. Evidently the effects of the corrosion contaminated the disks beneath the surface along the cobalt phase. Repolishing is not recommended for the certified SRM 2831 disks since it will alter the NIST-made reference indentations.

4. TEST PROCEDURES

4.1 Hardness testing machine and diamond indenter

All specimens were indented using a hardness machine[e] dedicated exclusively to this project. The machine used a dashpot-controlled lever system and was mounted on a vibration-damping table. The diamond indenter had dimensions close to the ideal. The manufacturer measured two face angles with a sin bar apparatus and certified the average angle to be 135° 57′ 13.″ The tip offset was certified as 0.19 μm. The indenter angles were checked at NIST on an optical comparator. This is less accurate and precise than the sin bar apparatus, but we nonetheless measured all face angles and even the edge angles for the diamond, and estimated that the average face angle was 135° 53′. These angles are well within the 136° ± 15′ that is specified in ASTM standard E 384 [12] for calibration grade indenters.

4.2 Indentation Load

An indentation load of 9.8 N was chosen for the SRM since this load is specified in the Vickers world standards and it is close to or on the hardness plateau (load independent hardness) of the indentation size effect curve. This load is available on most common hardness testing machines that have an indenter and a measuring microscope. As large an indentation load as possible should be used to make a large indentation in order to diminish the overall hardness uncertainty due to problems in precise tip location and diagonal length determination. On the other hand, the indentation load should not be so high as to cause cracking.

The indentation load and timing cycle were verified by NIST using a load transducer with an accuracy and precision of better than 0.05 % to ensure compliance with ASTM E 384. The load applied by the indenter on the hardness machine was within 0.1 % of the selected 9.801 N (1.000 kgf). A NIST traceable reference kilogram mass was used to calibrate the load transducer. The gravitational constant was 9.801 m/s^2 where the machine was located. A stopwatch was used to monitor the timing. Each prototype disk received ten or more indentations. All production disks received at least five indentations arranged in a pattern as shown in Figure 6.

[e] Tukon Model 300, Wilson Division of Instron, Canton, MA.

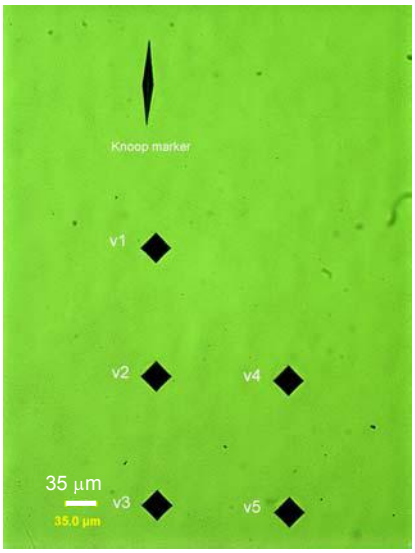

Fig. 6. Bright field optical micrograph with a green filter of the Vickers indentation pattern with a single Knoop marker.

4.3 Diagonal length measurement –Calibrated SEM

Most of the errors in hardness measurements arise from measuring the indentation size. Although most microhardness machines can measure diagonals to 0.1 µm or 0.2 µm, in practice difficulty in determining tip locations and the subjectivity of the viewer can easily lead to uncertainties of up to ± 1 µm in tip location [13,14]. Proper illumination is critical, especially the adjustments of the microscope diaphragms. Ideally the microscope should have both field and aperture diaphragms. Optical resolution limits are much less of an issue for Vickers indentations than for Knoop indentations due to geometry of the indentation corners.

A scanning electron microscope (SEM) was initially used to measure the nominally 35 µm diagonal lengths in prototype SRM 2831 disks. The high magnification and depth of field capabilities of the SEM aided the determination of the exact tip location. Magnifications up to 5000 X were used to study some tips. All indentations were photographed for diagonal measurements at a nominal magnification of 1500 X. Precise calibrations in both the horizontal and vertical directions were established using a NIST SEM 484d calibration line standard with a length of 55.19 µm ± 0.22 µm (95 % confidence level, coverage factor of 2). The calibration was in accordance with ASTM E 766 [15]. The disks were demagnetized prior to measurement as a precaution. The repeatability of these readings was determined by examining the same indentations on several specimens on different occasions. Although the repeatability of Vickers diagonal length measurement was sometimes as good as 0.1 µm (1σ, standard deviation) for a good indentation, on average the repeatability was within 0.4 µm (1σ) or 1.0 % of the diagonal size. Much of this uncertainty was traced to the poor contrast and definition of the top indentation corner in the SEM photographs such as illustrated in Fig. 4. Under optimum conditions, the process of installing the specimen in the SEM,

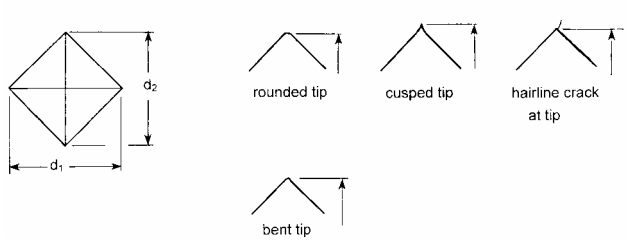

Fig. 7. The hard tungsten carbide grains caused slight irregularities to some indentation tips.

photographing the five indentations, and photographing the magnification standard at the beginning and end of each session required approximately 20 min per disk.

The SEM examinations also revealed that the indentation tips were sometimes rounded or distorted as shown in Fig. 7 due to the ≈ 0.5 µm hard tungsten carbide grains.

Experienced observers in our laboratory could match the SEM Vickers diagonal length measurements when using normal procedures with conventional optical microscopes and 40 X or 50 X objectives. The 1994 round robin results described below also confirmed that observers in other laboratories matched the SEM readings when using good optical microscopy practice. In 2002, when intensive work on SRM 2831 resumed at NIST, we confirmed that readings from a conventional research grade reflected light optical microscope[f] matched the calibrated SEM readings. The optical microscope had been retrofitted with a new high-resolution digital camera[g] with matching length measuring computer software. Repeated measurements with several SRM 2831 disks confirmed that the average SEM diagonal length for five indentations matched the average optical diagonal length to within 0.1 µm or better. A single horizontal or vertical diagonal length reading could differ by as much as a few tenths of a micrometer due to the differences in the mode of viewing and the precise shape of the indentation and its tips.

Hence, it was decided to forgo the complex, but very precise and accurate calibrated SEM procedure in favour of a faster, more efficient, and equally accurate and precise procedure using a modern digital camera on a high quality optical microscope.

4.3 Diagonal length measurement –Optical Microscope

The research optical microscope was capable of magnifications in excess of 1000 X when a 100 X objective lens with a 0.8 numerical aperture was used in conjunction with 10 X eyepieces. Images were further magnified when captured by the 1600 x 1200 pixel resolution digital camera chip and displayed on a high-resolution 485 mm (19 in) diameter computer monitor[h]. However, the images of the edges and tips of the indentations were not sharp at the highest magnifications due to poor contrast between the edges and the polished surface. Superior, sharper images were

[f] Leica Model DMRM, Wetzlar, Germany.

[g] Diagnostics Instruments, Spot Insight camera, 3 color mosaic mask CCD chip 11.8 mm x 8.9 mm, 1600 x 1200 pixels, Sterling Heights, MI.

[h] Hitachi Model CM 771 SVGA flat monitor, 1600 x 1280 pixels, with 0.22 mm pitch.

obtained with a 40 X, long working distance objective that had a 0.6 numerical aperture. The microscope also had built-in 1.25 X and 1.6 X auxiliary magnifier lenses. Ultimately it was determined that the best viewing mode (ease of use, best accuracy and repeatability) was with the 40 X objective in conjunction with the internal 1.6 X auxiliary magnifier. When displayed on the computer monitor, the 35 μm indentations appeared 68 mm wide or about 350 to 365 pixels for an approximate magnification of 2000 X. The length measuring software that came with the camera enabled us to measure diagonal lengths to within several pixels, corresponding to real length differentials of 0.1 μm to 0.2 μm. The digital software also allowed close-up imaging of portions of the image. We viewed every tips at up to 5700 X apparent magnification on the high resolution computer monitor.

A master optical stage micrometer[i] was used to check the magnification every single session on the optical microscope. This particular stage micrometer was certified by the NIST calibration laboratory to have an remarkable accuracy uncertainty of better than 0.01 % for the paired lines that were used in this project. Lines spaced 140 μm apart were used to set the overall magnification factor and was projected as 1435 pixels on the computer monitor. Hence one pixel corresponded to 0.1 μm. Special care was taken to use to use the computer software's thinnest measuring line (1 pixel wide) running to the same sides of the stage micrometer marker lines. It was very reassuring that the calibration and magnification factors were invariant over the entire period of several months that the measurements were made, attesting to the stability of the microscope. It was also confirmed that there were no magnification variations within the field of view, or in the vertical and horizontal directions. The consistency and stability of the optical microscope was a refreshing change compared to our experiences with the SEM with its day-to-day variations, variations in the field of view, drift, etc. Accurate and precise readings could be made much faster with the optical microscope. Nevertheless, it was very gratifying that the optical readings were verified by the calibrated SEM measurements. The SEM also revealed important information about tip shapes and microstructural interactions.

Three modes of illumination as shown in Figs. 8 to 10 were evaluated in a series of experiments in which the same indentations were measured numerous times on different days in order to establish measurement repeatability. Bright field reflected light illumination with a green filter was first evaluated. Dark field illumination and also differential interference contrast (DIC) modes were also evaluated. Both the 100 X and 40 X objectives were used The field and aperture diaphragms were properly adjusted for optimum illumination, contrast, and clarity and the stage micrometer was used to verify the magnifications.

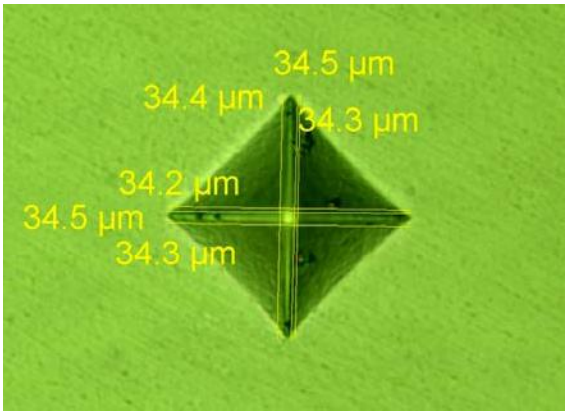

Fig. 8 Bright field image taken with the 40 X objective. The camera software enabled multiple length measurements to be made at the same time for comparison.

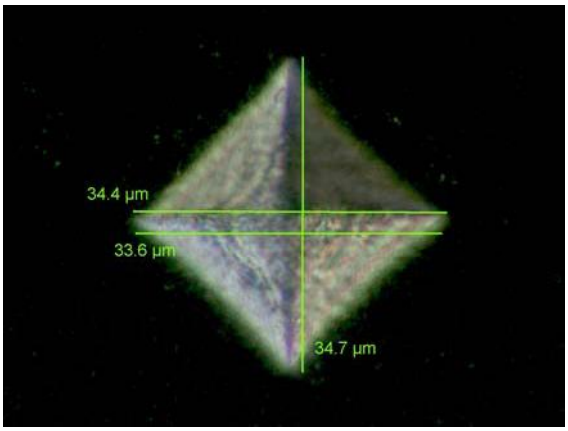

Fig. 9. Dark field image of a different indentation taken with the 100 X objective. Notice the fuzzy edges and blurred tips.

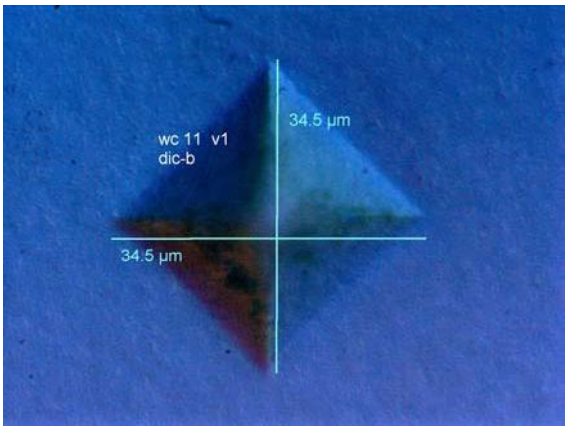

Fig 10. Differential interference contrast image. Although these images were the easiest and most pleasing to view, they were subject to size distortions.

[i] WILD, model 31045, Heerbrugg, Switzerland.

Space limitations here preclude a lengthy discussion and full statistical treatment of all the optical and SEM measurements, but it was determined that the optimum method (best precision and accuracy, ease of use and focussing, etc.) was with reflected bright field illumination with the 40 X objective, a green filter, and normal practice with the field and aperture diaphragms (Fig 8). This matches what most users have with their conventional micro hardness indentation machines that have 40 X or 50 X objectives lenses. This difference was that our reference indentations were imaged with a high quality research metallographic microscope, captured with a high-resolution digital camera[j], and measured with excellent computer software.

The horizontal and vertical diagonal lengths were measured and an average value used to compute the Vickers hardness. The indentations, which were nominally 35 μm in size, were quite symmetric and the two diagonal lengths usually matched to within 0.1 μm to 0.3 μm and only occasionally were as much as 0.6 μm to 0.85 μm different.

The dark field images (Fig. 9) did not produce sharp edges or tips at any magnification. Diagonal length measurements tended to underestimate the correct lengths by as much as 0.4 μm.

The differential interference contrast images (Fig. 10) were the most intriguing. Sharp images of the sides and tips could be obtained, even with the 100 X objective. The colored images were aesthetically pleasing to view. The problem was that inconsistent diagonal readings were obtained that depended on the positioning of the interference prisms. Readings could under or overestimate true lengths by as much as 0.5 μm.

5. RESULTS ON THE SRM 2831 DISKS

5.1 Disk uniformity

A variety of experiments were conducted on all eleven prototype and six production disks to check disk uniformity. All results showed that the disks had uniform hardness. Any variations were comparable to our measurement repeatability precision for the method. As an example of the within-disk uniformity, one Vickers prototype disk 1 had groups of five indentations at three different locations. The within-group repeatability was 0.7 μm (0.7 %)[k] and the between-group reproducibility was 0.7 μm (0.7 %)[l]. Similarly, groups of five indentations were placed in two or more locations in eight Vickers prototype specimens. The groups were in the center of the disks or within 2 mm to 4 mm from an edge. For the eight Vickers prototypes, the overall mean diagonal length was 34.3 μm (15.5 GPa hardness). Repeatability within a specimen was 0.7 μm (0.7 %). One of the production disks had over 100 indentations placed in six spokes that radiated outward from the disk center to the rim. No systematic hardness variation was detected.

5.2 Disk-to-Disk uniformity

Although these was little or no variation within a disk, experiments with both prototype and production disks confirmed there were small but statistically significant differences between disks. As a consequence, each production disk was individually measured and certified. Each has five NIST indentations in the middle. The certificate for each disk states the size of each indentation, the average size, and the average disk hardness. Fig. 11 shows the variation in hardness of the 108 production disks.

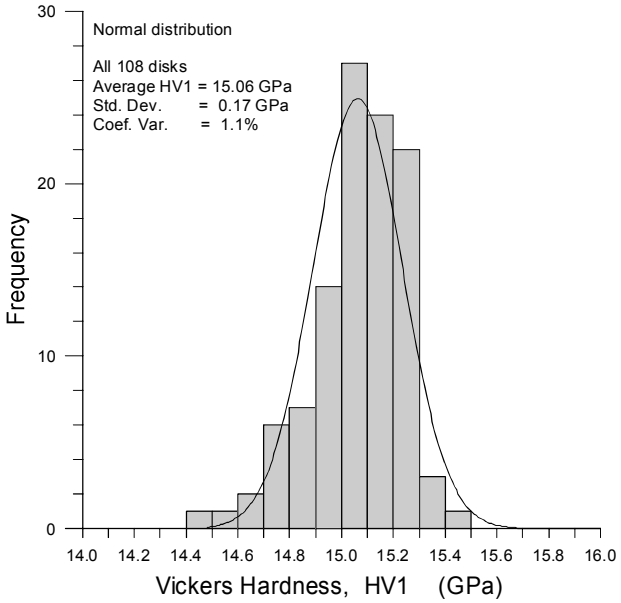

Fig. 11. The variation in average HV1 for all SRM 2831disks.

5.3 Other SRM Requirements

ASTM E 384 [12] has surface roughness and parallelism requirements for hardness reference blocks. The surface parallelism shall be better than ± 0.0005 mm/mm, and the R_A surface roughness shall not exceed 0.1 μm. The SRM 2831 disks had a parallelism of ± 0.00002 mm/mm and a R_A surface roughness of 0.051 μm.

5.4 SRM Uncertainty analysis

Space limitations preclude a detailed discussion of the uncertainty analysis. It is discussed in detail on the SRM certificate. An analysis of variance of all data collected (including many measurements for repeatability estimation) confirmed that the within-disk variation was negligible and the between-disk variation was significant. A test for the homogeneity of the variances for the five indentations in each disk showed that the variances were not statistically

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[j] The optics of the research metallographic microscope, and the high resolution digital camera and matching monitor have more resolution than most RGB video cameras mounted on hardness machines that may have only 600 x 480 resolution.

[k] This is the repeatability as defined by ASTM Standard E 691 [16] of the diagonal lengths *within* a group of five indentations at the 95 % confidence level. It is 2.8 x the root mean square average of the standard deviations of diagonal lengths in three groups of five indentations in one specimen. The second number, in parenthesis is the coefficient of variation in percent.

[l] This is the reproducibility at the 95 % confidence level *between* groups of five indentations on one specimen.

significantly different. We note that the uncertainty in the average diagonal size for one indentation is actually less (0.707 times) than the uncertainty for an individual diagonal length measurement, since the average is the result of two readings (the horizontal and vertical diagonals).

In summary, the uncertainty for an individual indentation size is ± 0.32 μm (± 0.9 % of the diagonal size) at a 95 % confidence level. The uncertainty for the average indentation size is only ± 0.14 μm (± 0.4 %). The uncertainty for the average Vickers hardness, HV1, is ± 0.29 GPa (40 kgf / mm^2, or 1.9 % of the average hardness).

Ninety-six disks were made available for sale. Every one has been indented and measured at NIST. Twelve disks were set aside for archival purposes, had cosmetic defects, or had an indentation with an unsatisfactory tip.

7. ROUND ROBINS THAT USED SRM 2831

7.1 NIST 1994 Round robin

An international round robin was conducted among eleven industrial and research laboratories to evaluate the suitability of the prototypes disks as SRM's. The participants were given one disk with a group of five NIST made indentations. The participants were asked to measure these indentations. They also made and measured five new indentations. They were given a blank steel specimen as a control. Preliminary findings of these results have been reported earlier [2].

The results are shown Fig. 12 where the laboratory mean diagonal lengths were normalized by the NIST calibrated SEM readings in order to factor out the slight disk-to-disk variability. In most cases, the laboratory measured diagonal lengths differ by less than 5 % from the NIST value. This m means the hardness will be within ± 10 % of the NIST value. It was very reassuring that the three participating laboratories that certify metal hardness blocks did extremely well (MPA, Materials Prufung Amt, Dortmund, Germany; Wilson, USA; and the Metals Division, NIST, USA). Their readings of the NIST indentations and their new indentations concurred with the NIST-Ceramics Division reference data.

The round robin did identify some problems. Laboratory 7's own indentations had the same size as the NIST indentations, but all were measured much too large (9 %). Evidently their microscope had a faulty length calibration factor. Their Vickers indents into a control steel block were also 4.3 % larger than normal. Laboratory 1 used three different observers, who were reasonably consistent with their diagonal length readings. Their own indentations were much too small, however, suggesting a load calibration fault.

The results are tabulated in Table 2. The mean diagonal length, d, is the average of the laboratory averages. Eleven labs participated overall, but laboratory 7's results and laboratory 1's own indentation size results were deleted as outliers in accordance with ASTM standard E 691 [16]. The precision estimates are in two categories: the within-laboratory scatter or "repeatability" and the between-lab scatter or "reproducibility." The standard deviation, 95% confidence level, and coefficient of variation are listed. These are conservative estimates of precision since the labs received different prototypes disks that had some hardness variability as discussed previously.

The precision uncertainty estimates in Table 2 have been incorporated into ASTM C 1327, the test method standard for Vickers hardness of ceramics.

In general, the participants were pleased with the SRM prototypes. With reasonable care, participants should be able to obtain diagonal lengths within ± 5 % of the NIST values (± 10 % in hardness) for Vickers indents at 9.8 N (1 kgf).

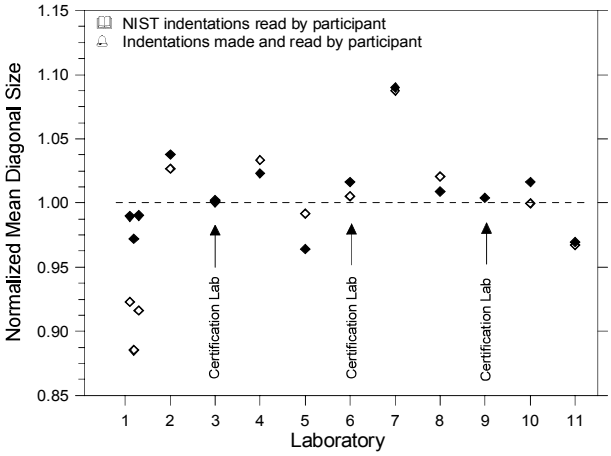

Fig. 12. Results for the 1994 NIST coordinated round robin. Mean diagonal lengths for five indentations normalized by the NIST SEM reference lengths. The three certification laboratories did extremely well.

Table 2 Results of the 1994 NIST round robin for the SRM 2831 prototype disks

Indentations*	# of Labs	Mean d (μm)	Within-lab Repeatability			Between-lab Reproducibility		
			Std. Dev. (μm)	Exp. Unc.[3] (μm)	COV %[4]	Std. Dev. (μm)	Exp. Unc.[3] (μm)	COV %[4]
NIST's	10[1]	34.5	0.2	0.6	0.6	1.1	2.9	3.0
Participant's	8[2]	34.6	0.2	0.6	0.6	1.0	2.7	2.8

1 Results of one lab deleted due to high h statistic (between-lab deviation) [16].
2 Results of two labs deleted due to high h statistic [16].
3 Expanded uncertainty with a coverage factor of 2.8, corresponding to a 95 % confidence level
4 Coefficient of variation, in percent.

7.2 CERANORM 1998 Round robin

This was a European laboratory only exercise [17,18] designed to validate the procedures and requirements in CEN ENV 843-4 [7]. Six prototype SRM 2831 Vickers hardness disks were loaned to the National Physical Laboratory (NPL, Teddington) and to the Federal Office for Materials Testing (BAM, Berlin). These were used along with eighteen other ceramics or hard metals including Knoop SRM 2830 silicon nitride disks and several candidate reference hardness materials prepared by the Fraunhofer Institute for Ceramic Technology and Sintering Materials (IKTS, Dresden).

Vickers hardness was measured at 9.8 N indentation load and the SRM 2831 disks performed well and the participating laboratories had some of their best results with the tungsten carbide disks. The within-laboratory and between-laboratory hardness precisions were 1 % to 2 % in every case, and most laboratories obtained HV1 values in good agreement with the certified values although two other laboratories were as much as 7 % in error, suggesting they had equipment or calibration problems. This project was instrumental in the refinement and conversion of the 1994 prestandard ENV 843-4 [7] into a full Euronorm EN 843-4 in 2000.

9. CONCLUSIONS

Vickers hardness SRM's that are tungsten carbide disks have been prepared that have a nominal hardness HV1 of 15 GPa. The SRM work was done in tandem with standardization projects in ASTM, CEN, and ISO. Prestandardization work on the SRM aided the work on the test method standards, and vice versa. SRM 2831 supports and complements numerous world standards and may be used with ceramics, hardmetals, carbides, or any hard material. The indentation sizes are certified to within 1 % and the hardness to within 2 % at the 95 % confidence level.

Two round-robins confirmed that the SRM's are suitable as reference materials and furnished valuable information for precision and bias statements in two ASTM ceramic hardness standards.

ACKNOWLEDGEMENTS

The authors would like to thank Joseph Fu, Chris Johnson, and Nien Fan Zhang of NIST; Dr. T. Polzin of MPA, Dortmund; and Dr. R. Morrell of NPL, United Kingdom for their measurements, assistance, and analysis. The U. S. Department of Energy, Office of Transportation Technologies through Oak Ridge National Laboratory supported the Vickers hardness test method standardization work.

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