Certificate of Analysis

Standard Reference Material® 2830

Knoop Hardness of Ceramics

Block Serial Number:

This Standard Reference Material (SRM) is intended for calibrating hardness testers. It is certified for the average diagonal length and the average hardness value. The SRM consists of a hot-isostatically pressed silicon nitride block with five NIST indents, made at a load of 19.6 N (2 kgf) in the center of the polished face. Each SRM is individually calibrated and bears a serial number scribed on the opposite, unpolished face of the block.

The SRM is hot-isostatically pressed silicon nitride, Norton Advanced Ceramics Grade NBD-200, fabricated in the form of a 22 mm (7/8") diameter bearing ball which has been ground and polished to provide a very flat, parallel, high quality surface for Knoop indents, with a final thickness of 9.54 mm ± 0.01 mm. The hardness is uniform across the polished surface. Five indents were made in the center of each block by a dedicated hardness testing machine.
The certified average diagonal length and the average hardness for this block are shown in Table 1.

Table 1: Certified Diagonal Length and Hardness

Average Diagonal Length, d	Average Diagonal Length Uncertainty*	Average HK2 Hardness**	Average HK2 Uncertainty
μm	0.6 μm (0.42 %)		0.12 GPa (0.86 %)

* The uncertainty of the average diagonal length for five indentations.
** The first number is the average hardness in units of GPa. The second number, in brackets, is the average hardness expressed as a dimensionless number. The average hardness should be calculated by averaging the five individual hardness results, and should not be calculated from the average diagonal size.

HK2 designates hardness at an applied load of 19.6 N (2 kgf). The uncertainties are at the 95 % confidence level. The numbers in parenthesis are two times the coefficient of variation expressed as a percentage.

Period of Certification: There is no need for periodic recertification assuming proper storage, handling, care, and use of this SRM. However, repolishing the surface, or other mechanical damage to the surface will invalidate the certification.

This SRM was prepared and certified by R.J. Gettings, G.D. Quinn, A.W. Ruff, and L.K. Ives of the NIST Ceramics Division. The assistance of J. Fu of the NIST Precision Engineering Division with the SEM calibration procedures is gratefully acknowledged.

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

The technical and support aspects involved in the preparation, certification, and issuance of this SRM were coordinated through the Standard Reference Materials Program by N.M. Trahey and R.J. Gettings.

Gaithersburg, MD 20899
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Thomas E. Gills, Chief
Standard Reference Materials Program

Storage and Handling: The ceramic block is very durable and not susceptible to corrosion, but the surface polish should be protected from abuse. Oils, fingerprints, or skin oils should be removed before indentation. The SRM unit may be cleaned with ethyl alcohol and soft tissues. If necessary, mild rubbing with a rubber eraser on the end of a pencil can be used to dislodge a tenacious contaminant. The blocks should **never be repolished**, as the size or raised edges of the NIST indentations would be altered.

Certification Procedures: The indents are located in the center of the block and are evenly spaced with the exception of the first indent as illustrated in Figure 1. The indents were made in accordance with ASTM E 384-89, Test Method for Microhardness of Materials [1], and the ASTM C 1326-96 Standard Test Method for Knoop Indentation Hardness of Advanced Ceramics [2]. In particular, the requirements of E 384 for separate verification of microhardness machines and calibration of standardized hardness test blocks, sections 14.1 and 17 were followed with the exception that only one group of five indentations has been made. Measurements of selected blocks have established that hardness is uniform across the block face.

These indents were examined and each photographed at a nominal magnification of 600 x in a scanning electron microscope (SEM). Selected indentation tips were also photographed at 1500 x or 3000 x. Indent sizes were then measured from the SEM micrographs. Precise magnifications were established for every block by using an SRM 484d Scanning Electron Microscope Magnification Standard. The polished surface and the indentations of each certified block were also inspected by an optical microscope at magnifications of 100 x and 400 x. Additional details on the preparation of this SRM can be found in References 3, 4, and 5.

The Knoop hardness of each indentation is computed:

$$HK = \alpha \frac{P}{d^2}$$

where P is the indent load in N (or kgf), d is the diagonal size in mm, and α is the indenter constant, which for an ideal Knoop indenter with angles of 172° 30' and 130° 00', is 14.229. The α for the NIST indenter used for this SRM was 14.240. The hardness was calculated using a load of 19.627 N (2.0025 kgf) which was the average load value during the SRM production. The gravitational constant was 9.801 m/sec^2 on the grounds of NIST.

Discussion of Uncertainties: The uncertainty estimates were made in accordance with the NIST and ISO Guides [6,7]. Uncertainties were either evaluated through statistical means, Type A, or by other means, Type B. Overall uncertainties were estimated by summing the variances of individual sources of uncertainty, with the exception of the uncertainty in HK wherein the variance in diagonal length was weighted by a factor of four in accordance with the law of propagation of error. Uncertainties listed in Table 1 and discussed below are at the 95 % confidence level (2σ) in each instance. The values in parenthesis are the uncertainties as a percentage and are **two** times the coefficient of variation. Uncertainties for the average values of d and HK were estimated by dividing the uncertainty for an individual measurement by the square root of five, which is the number of indentations measured.

The uncertainty in the length of an individual indent made by NIST was estimated by a series of repeatability experiments on a prototype lot of specimens to be 1.5 μm (1.08 %). The total uncertainty is the square root of the sum of squares of the systematic uncertainty (0.40 %) associated with the certified SRM 484d length standard and the random uncertainties associated with photographing, interpreting, and measuring (1.00 %) the indent length.

The variation in indentation sizes within an SRM block was calculated from the pooled results for all specimens made in the SRM production run and was estimated to be 1.34 μm (0.94 %). This variation is comparable to the uncertainty of the measurement method. A two way analysis of variance of all SRM production run results indicated that there probably is a block-to-block variation in diagonal lengths and hardness, but that within a block, the indentation size as an effect was not statistically significant. In other words, within a single block there was no statistically significant variation in indent size.

The uncertainty in the **average** diagonal length for the five indentations was estimated from the total population of indents in the SRM set, and is $0.94\ \%/\sqrt{5} = 0.42\ \%$, where the sample size is five.

The uncertainty in the average HK is for a sample size of five indentations and includes the components of uncertainty in the load, P, (0.25 %) which includes a repeatability uncertainty ($\pm$ 0.06 %) and a drift uncertainty ($\pm$ 0.24 %); the indenter constant, α, ($\pm$ 0.31 %); and the diagonal length, d, ($\pm$ 0.94 %).

Instructions for Use: This SRM is intended to be used with all hardness and microhardness testing machines whereby a Knoop indent can be made and then measured with an optical microscope.

The user must observe and measure the lengths of the five NIST indentations in order to verify that the test machine optics and the length measuring apparatus are optimized. The NIST indentations are located in the center of the polished surface. The indent long diagonal should be measured to within 0.0005 mm with an optical microscope. The apparent indent size will be affected by the magnification used since the objective lens numerical aperture (NA) sets the resolution limits. A total magnification of 400 x or higher is recommended and is in accordance with the Standard Test Method for Knoop Indentation Hardness of Advanced Ceramics, which requires objective lenses with NA's from 0.65 to 0.90. Magnifications of 400 x or 500 x are commonly available with most hardness machines, many of which have 40 x or 50 x objectives and 10 x eyepieces. Magnifications less than 400 x are not recommended since the indentation lengths will be underestimated by 1 μm to 2 μm or more. Magnifications should be checked by the use of a calibrated stage micrometer.

The Knoop indent is long and the tips slender, which can make precise determination of the tip location difficult. It is critical that the instructions of the hardness machine manufacturer and the appropriate standard test methods be followed scrupulously. Proper illumination and focus of the tips is critical to obtain good clarity and contrast. Proper use of filar crosshairs is essential.

The microstructure of the SRM is uniform and contains tiny inclusions which appear white in a reflected-light microscope. These are very helpful to aid focussing and to serve as reference points near the indent tips as illustrated in Figure 2. The grain size of the material is of the order of 1 μm. Occasionally a grain or grains near the tip will dislodge and cause the indent edge to be slightly irregular as shown in Figure 2. This is unavoidable, but is much less a problem with this SRM than for many ceramics. In a few instances, a tiny crack may extend from the tip of the indent as shown in Figure 2, which may create some uncertainty as to where the tip ends. Should the detected feature wander or meander off the axis of the indent, it is safe to assume that it is a crack, and the feature should not be read as the indent tip. The certified indentation diagonal lengths do not include the length of the crack, if such exist.

Producing New Indentations: The user can also make new indentations in the block but special care should be taken to ensure that the loading rates and load duration are as prescribed by the appropriate standard. There shall be no vibrations or impact imparted to the machine during the indentation cycle. The surface must be clean and should not contain skin oils which could alter the friction between the indenter and the block surface. Indentations may be placed in any region of the polished surface **provided that they are not within 1 mm of the rim,** since slight edge rounding from the polishing can distort the indentation shape and affect the size. The indents should not be made too close to each other, should lateral cracks (which may form in some ceramics) cause interference. The indentation centers should be no closer than 1.5 x the diagonal length.

Note: In a few rare instances, a block may contain a cluster of small inclusions or a faint black spot. If observed, it is recommended that indentation not be placed in such areas, although they may be harmless. A few blocks may have tiny scratches, which should also be avoided.

REFERENCES

[1] ASTM E 384-89 Standard Test Method for Microhardness of Materials, ASTM Annual Book of Standards, Vol. **3.01**, (1989).

[2] ASTM C 1326-96 Standard Test Method for Knoop Indentation Hardness of Advanced Ceramics, ASTM Annual Book of Standards, Vol. **15.01**, (1996).

[3] Gettings, R.J., Quinn, G.D., Ruff, A.W., and Ives, L.K., "New Hardness Standard Reference Materials (SRMs) for Advanced Ceramics," Ceram. Eng. and Sci. Proc., Vol. **15**, #5, pp. 717-826, (1994).

[4] Gettings, R.J., Quinn, G.D., Ruff, A.W., and Ives, L.K., "Development of Ceramic Hardness Reference Materials," pp. 617-624 in *New Horizons for Materials*, Ed. P. Vincenzini, Proceedings of the 8th World Ceramic Congress, CIMTEC, Florence, Italy, July, 1994, Techna, Florence, (1995).

[5] Gettings, R.J., Quinn, G.D., Ruff, A.W., and Ives, L.K., "Hardness Standard Reference Materials (SRMs) for Advanced Ceramics," Proceedings of the 9th International Symposium on Hardness Testing in Theory and Practice," Dusseldorf, Germany, 23-24 November, 1995, VDI, Dusseldorf, VDI, Berichte 1194, pp. 255-264, (1995).

[6] Taylor, B.N. and Kuyatt, C., "Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results," NIST Tech. Note 1297, U.S. Government Printing Office, Washington, D.C., (1994).

[7] *Guide to the Expression of Uncertainty in Measurement*, ISBN 92-67-10188-9, 1st Ed. ISO, Geneva, Switzerland, (1993).

Figure 1 Five Knoop indentations are located in the middle of the block.

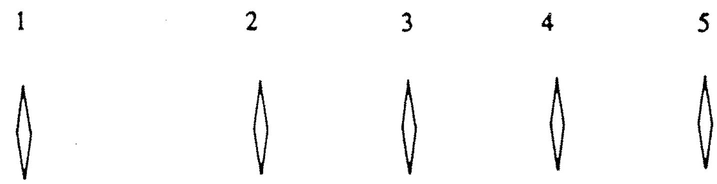

Figure 2 Details of the indentation tips.