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A STUDY OF THE DIRECTIONAL HARDNESS IN SILICON*

A. A. GIARDINI, *U. S. Army Signal Engineering Laboratories, Fort Monmouth, New Jersey.*

ABSTRACT

The directional hardness of single crystal silicon has been investigated both by a method of peripheral grinding on oriented thin circular specimens and by Knoop microindentations. Grinding hardness has been evaluated in the principal zones [100], [110] and [111]. Knoop Hardness Numbers are given for planes (100), (110) and (111) with indentations extended over 90° azimuths. Experimental results are compared with published data on diamond, which has the same crystal structure.

INTRODUCTION

Several workers (1, 2, 3, 4, 5, 6, 7) have made both qualitative and quantitative studies on the directional variation of grinding hardness in diamond. A review of the literature, however, has failed to yield any record of previous work on the anisotropy of hardness in silicon. Because of the structural similarity and bonding dissimilarity between diamond and silicon, a comparison of the relative hardness vectors of the two materials is desirable. The results of a hardness investigation on planes in the [100], [110] and [111] zones in silicon are presented herein.

Both grinding and indentation hardness have been evaluated. The former has been carried out by a method of peripheral (zonal) grinding on oriented thin disks of single crystal silicon, whereas the latter was performed with a Knoop microindenter with impressions made on the flat surfaces of the circular specimens.

Considering the difficulties encountered in quantitative hardness studies, the results obtained by the peripheral grinding technique have been found to be pleasingly systematic and reproducible. Microindentation data also can be reasonably correlated with structural symmetry, but on a statistical basis.

The advantages and disadvantages of the peripheral grinding technique compared to the usual procedure of grinding on fixed planes are as follows.

Advantages

(a) All planes in a given zone may be simultaneously evaluated relative to each other, with an automatic time integration of results.
(b) Selected zones can be easily examined and compared.
(c) Mounting, orientation and measuring procedures are greatly simplified.
(d) Experimental conditions can be easily varied and easily reproduced.
(e) The symmetrical nature of grinding hardness can be evaluated directly.

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Disadvantages

(a) Relatively large crystal specimens are needed.
(b) Accurate, oriented disks are required.
(c) The possibility of evaluating various grinding azimuths on given planes is restricted.

The silicon used in this study was obtained from the U. S. Army Signal Engineering Laboratories, Fort Monmouth, New Jersey, and is of transistor-grade purity. Specimens were cut from two boules, both of which were elongated (pulled) parallel to [111]. Although some spinel-type twinning was present in both boules, specimens were cut from areas free of recognizable twinning. The latter was determined both by visual inspection of ground surfaces and by x-ray diffraction

SPECIMEN PREPARATION

As previously mentioned, crystallographically oriented thin disks of single crystal silicon have been used for this study. Specimen orientation was carried out by the Laue method of x-ray diffraction. The apparatus and technique have been described earlier (8). Orientational error of specimens was maintained within ±30 minutes of arc.

Oriented, flat and parallel wafers were cut from boules with a precision diamond saw. A small diamond core drill was designed and constructed in order to cut true circular disks of constant diameter from the wafers. An accurate drill press was used to operate the core drill. The diametrical variation among disks amounted to less than ±0.5%.

A total of six disks were cut for quantitative investigations, two each of the following orientations: the normal to the circular face of the disk, (a) parallel to the [100] zone axis, (b) parallel to the [110] zone axis, (c) parallel to the [111] zone axis. In order to eliminate small cleavage irregularities which developed along the edges of the disks during core drilling, all were simultaneously cemented on a lapping block with a thin film of Canada balsam and carefully ground until free of noticeable imperfections. The same procedure was repeated for the opposite side of the disks. A machinist's micrometer was used to maintain parallelism, and therefore, correct orientation. The thickness of all disks was 0.165 cm. ±0.001. The diameters were 1.05 cm. ±0.005.

The same specimens have been used for both the peripheral grinding and microindentation studies.

EXPERIMENTAL APPARATUS FOR PERIPHERAL GRINDING

The experimental technique used for this part of the study consists of peripheral grinding on oriented silicon disks using a flexible, bonded

SiC abrasive cloth. The disks were coaxially mounted on the end face of a machined brass spindle by means of a special centering jig. Dopping wax was used as the cementing agent. The spindle, with disk, was then mounted in a Jacobs chuck which in turn was mounted on a 10″ machinist's lathe. Centering of the disk in the lathe was accomplished by use of shims and a dial indicator. During the grinding operation the disks were rotated at 68 rpm.

A $\frac{1}{2}$ inch wide strip of 240 grit SiC abrasive cloth moving in the direction opposite to the peripheral motion of the disk was used as the grinding medium. A flat spring, equipped with a pair of parallel rollers and mounted on the lathe tool holder, served as a flexible backing for the abrasive and as a constant pressure regulator. The diameter of each roller was 0.25 cm. giving a disk to roller diametrical ratio of approximately 4 to 1. Although a higher ratio (smaller roller diameter) would be desirable, the dimensions constitute a practical compromise between an acceptable minimum contact area between the disk and abrading medium, and a smooth movement of the abrasive cloth over the rollers. The abrasive feed was controlled by a 1,725 rpm motor geared to 2.9 rpm. The latter provided an average rate of feed of 35 cm. per minute for a 15 minute grinding period.

Prior to grinding operations, the abrasive strip and rollers were aligned parallel to the axis of the disk and spindle, and centered directly below the center of the disk. A compensating force was placed on the spring to counterbalance the pull of the abrasive cloth moving over the rollers. An additional force of 200 grams was then applied to the system. The operational contact surface between the disk and the abrasive strip was empirically determined to be $0.01 \text{ cm.}^2 \pm 10\%$.

The above values for experimental variables such as disk surface speed, abrasive feed, abrasive grit size and grinding force, were fixed on the basis of available equipment, disk dimensions, positive contact between disk and abrasive, loading of the abrasive cloth, and the relative hardness of the material under investigation.

In order to interpret properly and record hardness data, the orientation of the principal crystallographic directions and direction of rotation were directly scribed on the exposed flat surface of the disks. The disks were photographed in situ before grinding and at regular intervals during grinding operations. Maximum and minimum diametrical measurements and their respective orientations were determined during these intervals by use of a micrometer which could be read to ±0.0005 inch. The accuracy of this orientational procedure was found to be approximately ±3°. Figure 1 provides a view of the grinding apparatus.

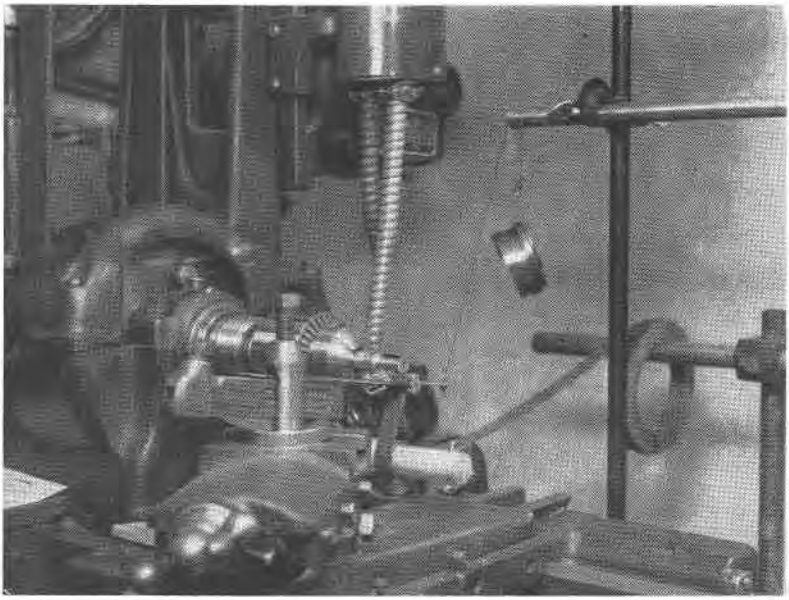

FIG. 1. A view of the experimental peripheral grinding hardness apparatus.

EXPERIMENTAL DATA FROM PERIPHERAL GRINDING

The experimental data obtained from peripheral grinding are given in graphical form in the following three ways:

(1) Radial reduction curves are presented for the three crystallographic zones investigated, namely, [100], [110] and [111]. The orientations, grinding azimuths and relative hardness (in terms of reduction in disk radii*) are included in the presentation. Data are given for a grinding period of 15 minutes and a total time of 30 minutes. No corrections have been made between the two grinding periods for the initial distortion of the circular periphery due to the directional hardness, and for the progressive reduction of peripheral surface area with decreasing diameter of the disk. The surface area reduction was determined by measuring the circumference on enlarged photographs of the disks and found to be approximately 6% for the first 15 minute grinding interval and 8% for the second 15 minute period. The 2% difference represents the resultant effect of the initial out-of-round shape which the disk assumes early in the first grinding period and the progressive reduction

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* Since all experimental variables are held approximately constant in this work, disk radii are proportional to the hardness constant, K, defined by Denning (5) in his studies of the hardness vectors in diamond.

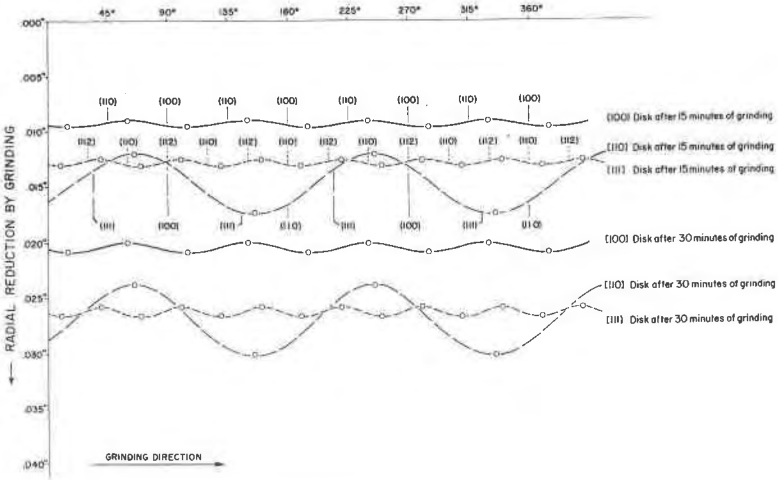

FIG. 2. Radial reduction curves for the [100], [110] and [111] zones in silicon. The indices represent forms rather than specific faces.

of the peripheral speed. Data presented in each curve are the averages obtained from two separate specimens of the same orientation. All specimens have been exposed to the same experimental conditions. The grinding curves are given in Fig. 2.

(2) Data are presented in terms of the per cent weight loss resulting from peripheral grinding for a 30 minute period for each of the three zones investigated. Data from each of the two disks for each of the three zonal orientations are individually represented. Averaged values are also indicated. It can be seen that the variance of the individual measurements is no greater than ±5% from the respective averaged values. These data are given in Fig. 3.

(3) Principal relative grinding hardness vectors are given in Fig. 4 in stereographic form. Vector magnitudes are plotted in linear units. The point of origin for each vector corresponds to its reference plane. Because of symmetry considerations (*m3m*), data are presented only in one stereogram quadrant. A vector presentation of Knoop Hardness Numbers, to be discussed later, also is included in Fig. 4. Due to the nature of the microindentation, all Knoop vectors are given as bidirectional, parallel to the long diagonal of the indenter. Again, because of symmetry, data are presented for only one quadrant.

Experimental conditions for all data described above are as follows: (a) average surface speed of the disks during the first 15 minutes of grinding was approximately 220 cm./minute, (b) the average surface speed of the disks during the second 15 minutes of grinding was approximately 210 cm./minute, (c) the rate of feed of the 240 grit SiC abrasive cloth averaged 35 cm./minute, (d) the grinding pressure during all grinding cycles was 20 kg./cm.$^2 \pm 10\%$.

SOURCES OF ERROR IN PERIPHERAL GRINDING

Sources of error in grinding hardness research are numerous, difficult to control, and difficult to correct. No theoretical corrections have been derived in this present study; however, most of the recognized sources of error have been minimized by careful and repeated measurements. A listing of several sources of error and their approximate limits is given in Table 1.

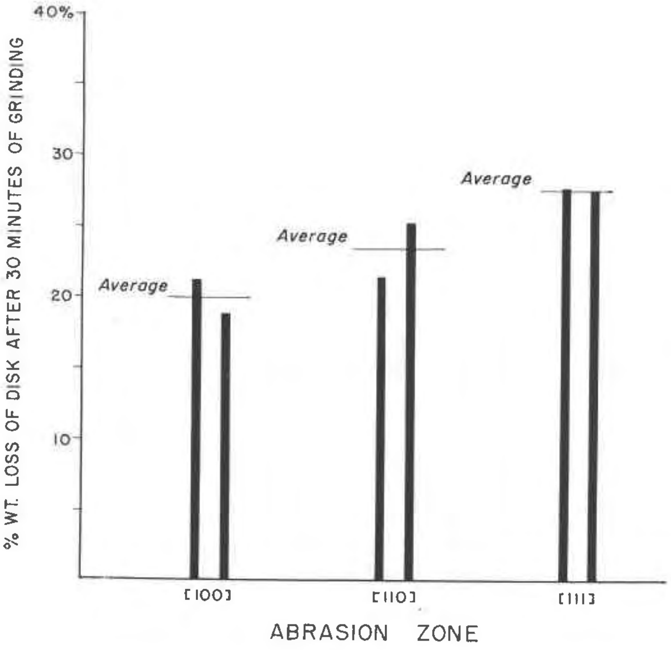

FIG. 3. The weight lost by each of the [100], [110] and [111] zonal disks of silicon during a 30 minute grinding period.

The total accountable variance amounts to $\pm 15.4\%$. This value represents the sum of the limits. An indication of the actual degree of error in the reported experimental data can be obtained, however, from the general agreement of data both from the individual disks of zonal pairs and by comparison of weight loss and radial reduction measurements.

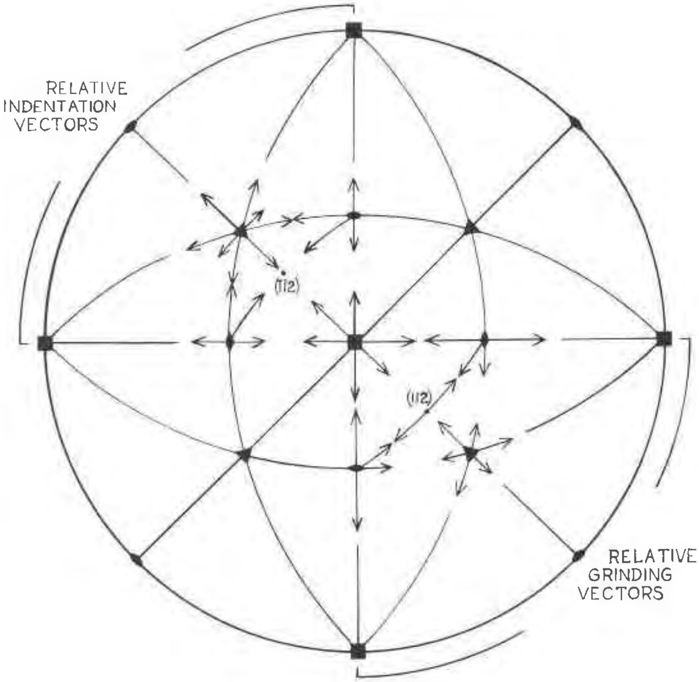

FIG. 4. A composite stereographic presentation of both peripheral grinding and micro-indentation relative hardness vectors. Magnitudes are plotted in linear units based on a common relative scale derived from maximum and minimum values. See Tables 2, 3, 4 and 6 for relative numerical values. KHN vectors are pictured as bidirectional parallel to the long diagonal of the indentation.

TABLE 1. SOME SOURCES OF ERROR ENCOUNTERED IN GRINDING EXPERIMENTS AND OBSERVED LIMITING VALUES

Sources	Limits
Surface speed of the silicon disks	± 1.0%
Rate of feed of the abrasive cloth	± 0.05%
Grinding contact area	±10.0%
Micrometer measurements	± 0.15%
Disk orientational error	± 0.5%
Orientational error in hardness vectors	± 3.33%
Weight loss measurements	± 0.2%
Time of the grinding periods	± 0.15%
Non-uniformity of the grinding force	±?

Variations show an approximate maximum of ±5%. The latter undoubtedly could be reduced through refinements in apparatus and procedure.

Microindentation Experimental Apparatus

A Bergsman micro-hardness tester, model number 4-418, equipped with a certified Knoop point, was used for the indentation part of this study. The Bergsman instrument was mounted on a Vickers projection microscope. The latter was set on rubber pads to minimize the effect of building vibrations.

The indenter load (100 grams) and optical system (Spencer 1.25 N. A. oil immersion objective and a Bausch and Lomb filar eyepiece) were chosen to produce the "ideal" 500–600 filar unit length of indentation image. The filar graduations of the eyepiece were calibrated with a Bausch and Lomb stage micrometer. The loading time for indentations was approximately 10 seconds.

The flat surfaces of the cylindrical peripheral grinding specimens were used for the indentation study. The latter were given an optical quality polish with 0–2 micron diamond paste. The specimens were then mounted in lucite cylinders. Indentation azimuths were positioned by rotation of the graduated microscope stage. Light figure reflections from the roughened edges of the disks were used to orient the indentation azimuths with respect to principal crystallographic directions.

Microindentation Experimental Data

A total of 100 indentations were made on each of the planes (100), (110) and (111). Groups of ten indentations were made on azimuths spaced at 10° intervals. A larger azimuth range is unnecessary because of the high symmetry.

Almost all indentations were accompanied by fracturing, and in some cases by octahedral micro-cleavage on the (111) plane, and fine cracking across the narrow tips of the indentations. The fracturing generally occurred on either or both sides of the indentation long diagonal in approximately half circular or elliptical shape. The "radii" of these fractures in some cases were as much as the full length of the indentation. In general, however, the shape of most indentations with respect to the long diagonal were of perfect apparent symmetry. Data are reported only for those of the latter quality.

No special correlation was apparent between the occurrence and severity of fracturing (and cracking), and crystallographic orientation. Although undoubtedly related to the mechanics of producing the indentation, these structural disruptions also probably reflect the distribution of micro-defects in the silicon.

The experimental data, in terms of Knoop Hardness Numbers for a 100 gram load, are presented in Tables 2, 3 and 4. Each of the values given represents the arithmetic average of ten consecutive indentations along a given azimuth. As previously mentioned, a composite stereographic presentation of peripheral grinding and microindentation data is given in Fig. 4.

TABLE 2. KNOOP HARDNESS NUMBERS (0.1 Kg LOAD) FOR THE (001) PLANE OF SILICON

(SEE FIG. 4)

Azimuth*	Average KHN_{100}	Av. Variance of 10 Indentations	Limiting Variance
0°	960	±1.0%	±1.1%
Towards $(\bar{1}01)$†	950	0.4	0.7
20°	960	0.3	0.7
30°	960	0.5	1.0
40°	965	0.3	0.7
50°	970	0.5	1.0
Towards $(\bar{1}\bar{1}1)$†	—	—	—
60°	980	0.5	1.0
70°	960	0.4	0.9
80°	970	0.5	0.9
90°	965	0.4	0.7
Towards $(0\bar{1}1)$†	—	—	—
	Overall average = 964 ± 0.5%		

* The limit of absolute collective orientational error for all azimuths on a given plane is estimated at ±5°; the relative error is ±0.25°.

† The long diagonal of the indentations are toward the respective planes indicated. See Fig. 4.

SOURCES OF ERROR IN MICRO-HARDNESS TESTING

Several of the factors influencing micro-hardness testing have been analyzed and discussed by Bergsman (9). In this study, care was exercised wherever possible to minimize sources of error and to empirically evaluate limits. The latter are given in Table 5.

No attempt has been made to evaluate the effects of specimen surface preparation and the observed fracturing, cleavage and cracking on KHN values.

DISCUSSION OF RESULTS

The hardness of silicon on the Mohs scale is 7. The results of this study have shown a significant directional variation of grinding hardness con-

TABLE 3. KNOOP HARDNESS NUMBERS (0.1 Kg LOAD) FOR THE $(\bar{1}01)$ PLANE OF SILICON
(SEE FIG. 4)

Azimuth*	Average KHN_{100}	Av. Variance of 10 Indentations	Limiting Variance
Towards (001)*	940	±0.6%	±1.4%
10°	955	0.5	1.0
20°	945	0.6	1.7
30°	960	0.5	1.0
40°	965	0.7	1.5
50°	980	0.8	1.5
Towards $(\bar{1}\bar{1}2)$*	—	—	—
60°	970	0.7	1.5
70°	980	1.0	1.7
80°	965	1.1	1.7
Towards $(\bar{1}\bar{1}1)$*	980	0.5	1.0
	Overall average = 964 ± 0.7%		

* See Table 2.

sistent with the *m3m* symmetry of silicon, and a reasonable statistical correlation of KHN values with atomic arrangement.

The greatest variation of grinding hardness was found to exist in the [110] zone. The maximum variance of microindentation hardness was observed on the (110) and on the (111) planes. For both grinding and indentation, the higher relative hardness values were obtained when

TABLE 4. KNOOP HARDNESS NUMBERS (0.1 Kg LOAD) FOR THE $(\bar{1}\bar{1}1)$ PLANE OF SILICON
(SEE FIG. 4)

Azimuth*	Average KHN_{100}	Av. Variance of 10 Indentations	Limiting Variance
Toward (001)*	970	±0.5%	±1.2%
10°	940	0.7	1.4
20°	940	0.5	0.9
30°	945	0.5	0.8
40°	960	0.3	0.5
50°	940	0.6	1.4
Toward $(\bar{1}01)$*	970	0.6	1.2
70°	945	0.6	1.2
80°	935	0.4	1.0
Toward $[\bar{1}10]$*	935	0.7	1.4
	Overall average = 948 ± 0.5%		

* See Table 2.

TABLE 5. SOME SOURCES OF ERROR ENCOUNTERED IN MICROINDENTATION EXPERIMENTS AND OBSERVED LIMITING VALUES

Sources	Limits
Calibration of the filar eyepiece	±1.6% limiting variance, ±1.1% av. of 12 meas.
Loading time of indentation	±5%
Inter-azimuth orientation	±1%
Crystallographic-azimuth orientation	±5.5%

resultant forces were directed into planes of greater atomic density. Similarly, the lower values were observed against planes of lesser density.

A summary of the relative directional hardness in silicon for the principal planes and azimuths is given in Table 6. Although at first glance a general inconsistency may appear to exist between similar grinding and indentation vectors, consideration of the resultant force directions with respect to crystal structure and bonding vectors at once reveals a correlation which, in view of experimental differences and difficulties, is surprisingly good.

The relative grinding hardness of the principal planes and similar azimuths in diamond, as reported by Slawson and Kohn (3), and Denning (5, 6, 7) is given in Table 7. Also indicated are the Knoop Hardness Numbers reported by Peters and Knoop (10), and Wolff *et al.* (11).

A comparison of the relative grinding hardness values for diamond with those currently observed in silicon shows a decided difference between certain of the principal vectors. Whereas (001) toward (111), and (101) in the direction toward (001), are the two hardest grinding directions in diamond; (101) in the direction toward (001), and (001) in the direction toward (101) are the two hardest in silicon. No difference in the two vectors could be resolved in diamond and essentially no difference was observed for the two hardest in silicon.

Further observed differences between the two materials are apparent in the principal azimuths on the (111) plane, and the softest grinding vector. Whereas little difference appears to exist between (111) toward (101), and (111) toward (001) in diamond; (111) toward (001) is a moderately hard vector in silicon, and (111) toward (101) is the softest vector. Grinding on plane (101) toward (111) is the softest vector in diamond.

The above differences in relative grinding hardness between diamond and silicon are compatible with the differences in morphology (surface energy), and microcleavage (as determined by light figure studies) which have been observed by Wolff (12, 13).

TABLE 6. THE RELATIVE GRINDING AND INDENTATION HARDNESS ON THE PRINCIPAL PLANES OF SILICON IN THE SPECIFIED AZIMUTHS
(SEE FIG. 4)

Grinding on Plane	In the Direction Toward	Descriptive Relative Hardness	Comparative Numerical Hardness*
(a) (101)	(001)	Hardest	2.0
(b) (001)	(101)	Slightly less hard than (a)	1.95
(c) (112)	(101)	Third hardest	1.55
(d) (101)	(112)	Fourth hardest†	1.50
(e) (001)	(111)	Same as (d) within experimental error†	1.50
(f) (111)	(001)	Same as (d) within experimental error†	1.50
(g) (101)	(111)	Fifth hardest	1.2
(h) (111)	(101)	Softest	1.05

Indentation on Plane	Long Diagonal ± Toward‡	Average KHN$_{100}$
(i) $(\bar{1}01)$	$(\bar{1}\bar{1}1)$	980
(j) (001)	$(\bar{1}\bar{1}1)$	980
(k) $(\bar{1}01)$	$(\bar{1}\bar{1}2)$	980
(l) $(\bar{1}\bar{1}1)$	(001)	970
(m) (001)	$(\bar{1}01)$ and $(0\bar{1}1)$	approx. 955
(n) $(\bar{1}\bar{1}1)$	$[\bar{1}10]$	935
(o) (001)		overall av. 964
(p) $(\bar{1}01)$		overall av. 964
(q) $(\bar{1}\bar{1}1)$		overall av. 948

* Radial reduction for the softest direction (unity) divided by the radial reduction for the specified direction.

† No significant difference could be observed between vectors (d), (e) and (f) above; however, the hardness from (e) decreases in the direction of peripheral grinding whereas the hardness increases from (f).

‡ The orientation of indentation azimuths are correct to ±5°.

There are insufficient data available on diamond for a comparison of Knoop Hardness Values.

Another point worthy of notice from this study is the ability of the peripheral grinding technique to provide a direct and clear evaluation of the symmetrical nature of grinding hardness. This effect is nicely illustrated by Fig. 2. As expected for materials of class *m3m*, two-, three- and four-fold symmetries are displayed by the respective principal zones. The symmetry of the maximum and minimum hardness vectors with respect to crystallographic directions as a function of peripheral grinding direction also has been confirmed during the course of this investigation. Since this type of zonal hardness test can be made quite easily and

TABLE 7. THE RELATIVE HARDNESS OF THE PRINCIPAL PLANES OF DIAMOND IN THE SPECIFIED AZIMUTHS

Grinding on Plane	In the Direction Toward	Descriptive Relative Hardness
(a) (101)	(001)	Hardest*
(b) (001)	(111)	Hardest*
(c) (001)	(101)	Soft
(d) (111)	(001)	Hardest in (111) (Slawson & Kohn)
(e) (101)	(111)	Softest
(f) (111)	(101)	Hardest in (111) (Denning)
Indentation on Plane	**Long Diagonal Parallel to**	**Average KHN**
(111)	?	av. KHN_{1Kg} 8200–8500 (Knoop and Peters)
(111)	?	KHN_{100g} 8820 ± 1380 (Wolff *et al.*)

* Of equal hardness within experimental error.

quickly, it should prove of value with respect to the basic question of the centro-symmetrical nature of grinding hardness.

ACKNOWLEDGMENTS

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