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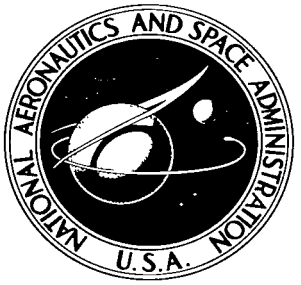

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OPTICAL ALIGNMENT OF SILICON CRYSTALS

by

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College Park, Md.

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ABSTRACT

The principles of optical alignment of crystals, specifically silicon, are discussed. A relatively simple apparatus is described which allows crystal orientation to within $\pm 2^{\circ}$ or better, depending on the crystal plane. Data are presented and optical reflection patterns are given which identify not only the usually given $\{100\}$, $\{110\}$, and $\{111\}$ planes, but also the $\{311\}$ and $\{122\}$ planes. The usefulness of the latter two is pointed out when crystal orientations in more than one direction are required. The relationship between reflectograms and stereographic projection of crystal planes, and the latter's use for identifying optical reflections, are pointed out. An Appendix gives some useful hints on cutting such crystal wafers as required in solar cell and transistor technology, and in the measurement of Hall effect, resistance, and electron spin resonance.

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INTRODUCTION

The use of optical reflections from etched crystal surfaces for crystal orientation has been known for some time. The literature is fairly extensive and appears to go back to the beginning of this century. McKeehan (Reference 1), Potter and Sucksmith (Reference 2), Weerts (Reference 3), and Chalmers (Reference 4) discuss optical orientation of metal single crystals; orientation of single crystals of silicon and germanium is the subject of more recent papers (References 5, 6, 7). The method depends on preferential etching of crystal surfaces. The etchant apparently creates microscopic pits in the crystal surface such that each pit is bounded by some crystallographic planes. A parallel beam of light incident on such an etched surface will suffer reflection from each of the crystallographic planes in the etch pits, and will produce a characteristic light pattern (reflectogram). This light pattern is used in orientation of the crystals. Optical reflection patterns have been reported for silicon and germanium (References 5, 6, 7); this paper presents a more extensive discussion of observed patterns for silicon, with measurement and identification of the optical reflections $\{311\}$, $\{221\}$, and $\{110\}$, in addition to the usually given reflections of $\{100\}$, and $\{111\}$.

METHOD OF REFLECTION MEASUREMENT

The preparation of crystals for optical orientation has been discussed in detail elsewhere (References 6, 7). We have not studied the effect of etchant and conditions of etching on the reflection pattern. Silicon crystals were cut by a No. 100 grit diamond wheel, ground with a No. 80 or 100 aluminum oxide cloth, then etched for two minutes at approximately 100 °C in a 10% (by weight) NaOH solution. The crystals were mounted on a simple goniometer which allowed rotation about two mutually perpendicular axes; a third axis of rotation would have been desirable. Figure 1 illustrates the optical setup and goniometer.

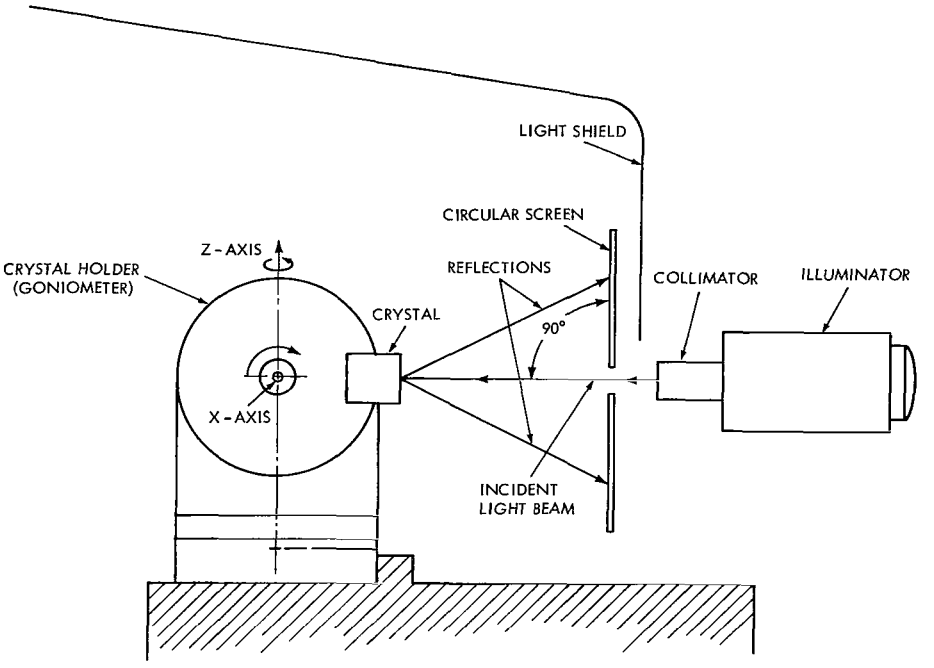

Figure 1—Schematic diagram of optical crystal orientation system.

A collimated beam of light (2 to 3 mm in diameter) strikes the crystal surface; reflections from the surface appear on a screen whose plane is perpendicular to the light beam. Measurements are made with a zone axis parallel to one of the axes of the goniometer, usually, the "z" axis. Measurements are then made on the reflections of a given zone, which will all fall on a straight line. This procedure permits direct determination of angles between planes from the goniometer readings. If, for example, the <110> zone axis of the crystal is made to coincide with the "z" axis of the goniometer, and the crystal is oriented in the <100> direction (i.e., the incident light beam is parallel to the <100> direction), then the zone of reflections will consist of the planes {100}, {113}, {112}, {111}, {221}, etc.* We shall quote briefly other zone axes and their related zones which are needed to interpret the observed data. For a crystal oriented in the <100> direction, and the goniometer axis parallel to the <100> zone axis, the low index zone reflections will be {100}, {012}, {013}, and {011}. Finally, a crystal oriented in the <111> direction, and a <110> zone axis parallel to the "z" goniometer axis, will have a zone {111}, {221}, {331}, and {110}, again selecting only low index planes.

RESULTS

Figure 2 is a diagrammatic representation of a reflection pattern obtained from a <100> oriented silicon crystal with a <110> zone axis parallel to the "z" axis of the crystal holder

*A table of zones and angles between crystallographic planes of the cubic system along selected zones can be found in "Crystal Orientation Manual," E. A. Wood, Columbia University Press, New York, 1963.

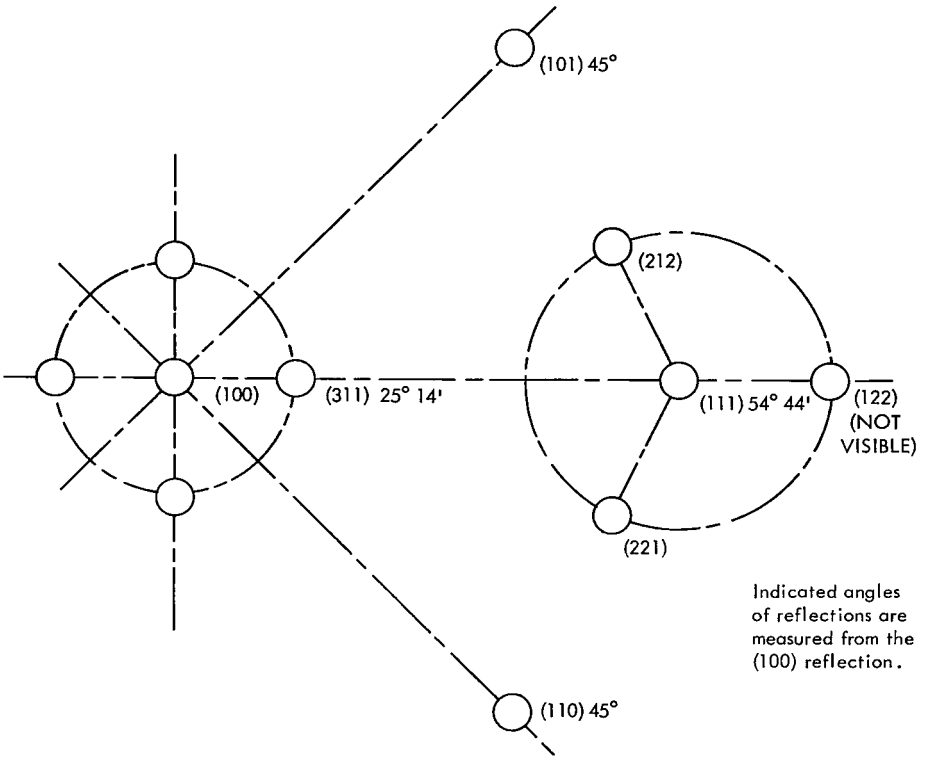

Figure 2—Optical reflection pattern from {100} surface of silicon.

(Figure 1). The reflection pattern centered around the {100} reflection clearly exhibits the four-fold symmetry of the <100> direction. We further see along a straight line the reflections {311} and {111} belonging to the same zone. Measurements made on these reflections are shown in Table 1, columns 1 and 2. Angular separation between planes is shown in column 3. In all cases a representative sample of individual angular readings is given with its average. Column 4 gives the theoretical value of the angular separation of a {100} plane from a given plane closest to the observed average value. (Tables of angles between planes of cubic crystals are given in Wood's "Crystal Orientation Manual"). We have thus been able to identify the reflections designated by the letters "B", "D", and "E" in Figure 3 as {311}, {111}, and {110} planes, respectively. As expected, the {111} reflection exhibits threefold symmetry although only two of the three {122} reflections could be seen. Since the visible {122} reflections are not contained in the particular zone used for identification of above planes, identification was made on another crystal oriented in the <111> direction. This is discussed below. Measurement of reflection "E" required a change of the zone axis from <110> to <100> as outlined in the section, "Method."

Figure 4 shows the reflection pattern obtained from a silicon crystal oriented along a <111> direction, with a <110> zone axis parallel to the "z" axis of the goniometer. Identification was made of reflections designated by the letters "$\bar{G}$", "F", "G", and "H". The results are summarized in Table 1. With the crystal in this orientation, we were able to obtain a measurement of reflection

"G" and to correlate it with a {122} plane. It is therefore reasonable to assume that the reflections showing threefold symmetry about {111} in Figure 4 are indeed due to {122} planes.

We have mentioned the fourfold symmetry of the pattern in Figure 2 and the threefold symmetry of the pattern in Figure 4. No measurements were made to determine the angular separation of the {311} and {221} reflections in the plane of the screen, because such measurements would

Table 1

Experimentally Determined Separation Between Crystallographic Planes in Silicon.

Goniometer readings of reflections, θ* (degrees)**			Angular separation between reflections (degrees)	Calculated separation between planes (degrees)
θ_A {100}	θ_B		AΛB	{100} Λ {311} = 25° 14'
1.3°	337.3°		24.0°	
2.3	339.2		23.1	
1.3	337.3		24.0	
0.8	336.6		24.2	B = {311}
ave. 1.4°	ave. 337.6°		ave. 23.8°	
θ_A	θ_E		AΛE	{100} Λ {110} = 45°
358.2°	313.2°		45.0°	
0	315.0		45.0	
2.0	317.0		45.0	
0.8	315.5		45.3	E = {110}
ave. 360.2°	ave. 315.2°		ave. 45.1°	
θ_A	θ_D		AΛD	{100} Λ {111} = 54° 44'
			54.7°	
—	—		55.4	
			54.9	
			55.0	D = {111}
			ave. 55.0°	
θ_F {110}	θ_G	$\theta_{\bar{G}}$	FΛG	{110} Λ {122} = 19° 28'
314.5°	293.0°		21.5°	
314.5		335.5°	21.0	
314.5		334.5	20.0	
314.5	292.5		22.0	G = {122}
ave. 314.5°	ave. 292.7°	335.0°	ave. 21.1°	
θ_F	θ_H		FΛH	{110} Λ {111} = 35° 16'
			35.0°	
—	—		36.0	H = {111}
			ave. 35.5°	

*All readings ±2°.
**Subscripts refer to lettered points in Figure 3.

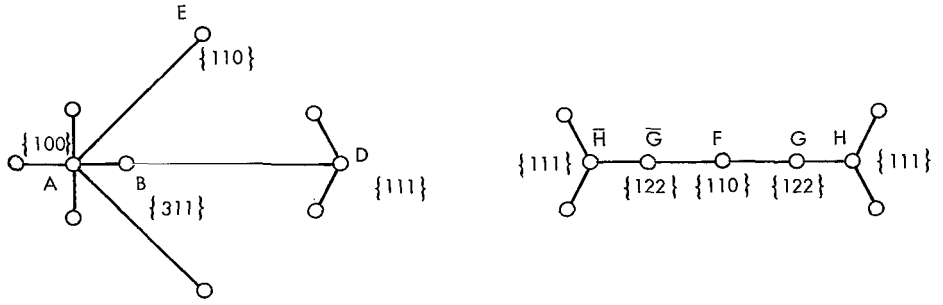

Figure 3—Diagram for identification of optical reflections used in Table 1.

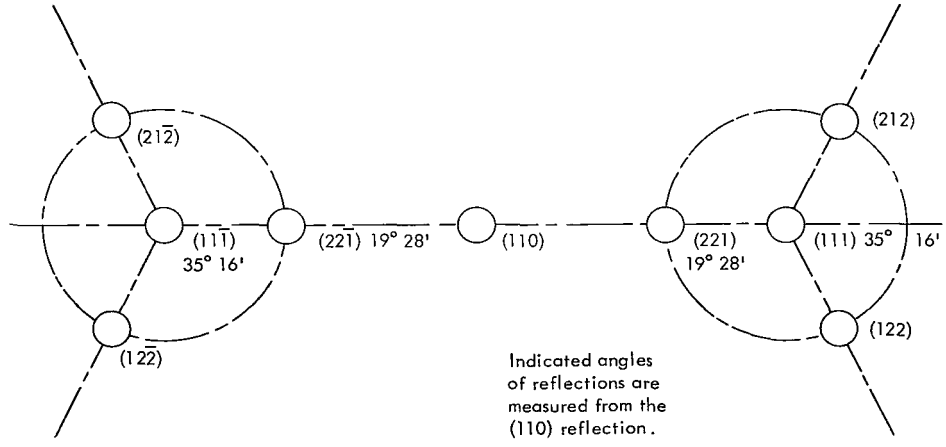

Figure 4—Optical reflection pattern from {111} surface of silicon.

have required a third goniometer axis. Visual examination of the reflection patterns was considered as adequate proof of the 90° and 120° spacings of the {311} and {211} reflections, respectively, on the screen.

Conclusion

Comparison of the calculated angular separations of planes and those determined by the optical method shows that orientations can be made to within ±2°. Presumably, a better optical system and more careful control of the etching process would result in more sharply defined reflection patterns and in turn more accurate angular readings. The optical orientation method with the present setup and techniques is not as accurate as the Laue method (3 cm specimen-to-film distance) but proved sufficient for our purpose (orientation of silicon samples for electron spin resonance). The advantage of the optical method is its obvious convenience.

The optical reflection pattern bears a striking resemblance to the stereographic projection of planes in the cubic system. Some consideration will show that there is indeed a one-to-one

correspondence between the light "spots" as produced by the etched crystal on a screen, and the "dots" of the stereographic projection (See, for example, Figure 2 in "Crystal Orientation Manual" mentioned previously). The relative separation of the planes on the stereographic projection is, however, different from that of the reflectogram. Nonetheless, the stereographic projection has been found extremely useful in identifying optical reflections and in orienting crystals in a given direction. The Appendix briefly describes the wafering of a floating zone single silicon crystal (oriented in the <111> direction) with the wafer surface parallel to {100}.

Optical orientation of silicon crystals with the type of optical system described has been found to be quite adequate for use in electron spin resonance experiments of silicon donors and radiation defects in silicon. For use in either checking the orientation of silicon solar cells or cutting silicon blanks for solar cells, the method is more than adequate. Similarly, the method should prove useful in Hall effect and resistance measurements of semiconducting crystals.

Goddard Space Flight Center
National Aeronautics and Space Administration
Greenbelt, Maryland, June 28, 1967
120-33-01-11-51

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Appendix A

Brief Procedure for Cutting {100} Wafers* from Floating-Zone Silicon

Floating-zone silicon is always grown with the axis of rotation of the boule in the {111} direction which, in silicon, is perpendicular to the {111} plane. Since the angle between the {111} and {100} planes is rather large (54° 44')**, care must be exercised in wafering the crystal in order to obtain as many slices as possible.

After an initial rough {100} cut is made across the end of the boule, the crystal is precisely aligned in the {100} direction and a precision {100} cut is made. Without changing the orientation of the crystal relative to the saw blade, the crystal is moved forward and a second precise {100} cut is made parallel to the first. The distance between the two cuts is determined by the dimensions of the slices one expects to cut.

After this second {100} cut, the boule is rotated 90° so that a cut may be made perpendicular to the first {100} cut, in the {110} plane. The positions in which these cuts are made will also be determined by the expected dimensions of the slices. In order to get two 10 x 20 mm slices on the elliptical {100} face of the boule, the two {110} cuts were made 20 mm apart and positioned on the {100} face so as to yield the maximum number of slices.

The section of the boule which was cut off by the {110} cuts was removed from the goniometer and mounted on a section of steel ground stock in order to cut the slices. The crystal was mounted on the larger of the two {110} faces, and the {100} face was aligned with the precision ground edge of the steel block. After three cuts were made mutually perpendicular to both the {110} and {100} faces, to determine the width (10 mm) of the slices, the crystal was rotated 90°, so that the saw cut was exactly parallel to the {100} surface, after which slices 1/2 mm (~.020") thick, oriented in the {100} plane, were cut.

*The wafers here described were 10 x 20 x 1/2 mm, with the 10 x 20 mm surface coincident with the {100} plane and the 1/2 x 10 and 1/2 x 20 mm surfaces with the {110} planes.

**Since the angle between the {111} plane and the {122} plane is also 54° 44', it is important not to confuse the {122} and {100} planes. The {100} plane is distinguished by the readily apparent fourfold symmetry of the reflection pattern.

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