



Standard Test Methods for Determining the Orientation of a Semiconductive Single Crystal¹

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1. Scope

1.1 These test methods² cover techniques for determining the crystallographic orientation of a surface which is roughly parallel to a low-index atomic plane in single crystals used primarily for semiconductor devices.

1.2 Two types of test methods are covered as follows:

1.2.1 *Test Method A, X-ray Diffraction Orientation*—This test method may be used for the orientation of all semiconductive single crystals.

1.2.2 *Test Method B, Optical Orientation*—This test method is limited in application at the present time to elemental semiconductors.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* Specific hazard statements are given in Section 6.

2. Referenced Documents

2.1 ASTM Standards:

E82 Test Method for Determining the Orientation of a Metal Crystal³

E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods⁴

2.2 American National Standard:

B74.10 Specifications for Grading of Abrasive Microgrits⁵

2.3 Other Standard:

Code of Federal Regulations, Title 10, Part 20, Standards

¹ This test method is under the jurisdiction of ASTM Committee F-1 on Electronics, and is the direct responsibility of Subcommittee F01.06 on Silicon Materials and Process Controls.

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² DIN 50433 is an equivalent method. It is the responsibility of DIN Committee NMP 221, with which Committee F-1 maintains close technical liaison. DIN 50433, Testing of Inorganic Semiconductor Materials: Determining the Orientation of Monocrystals; Part 1 with an X-ray Goniometer, and Part 2 by the Light-figure Method, is available from Beuth Verlag GmbH, Burggrafenstrasse 4-10, D-1000 Berlin 30, Federal Republic of Germany.

³ Annual Book of ASTM Standards, Vol 03.01.

⁴ Annual Book of ASTM Standards, Vol 14.02.

⁵ Available from American National Standards Institute, 11 West 42nd St., 13th Floor, New York, NY 10036.

for Protection Against Radiation⁶

3. Significance and Use

3.1 The orientation of semiconductor crystals and wafers as determined by these test methods is an important materials acceptance requirement because the orientation controls various parameters of semiconductor devices fabricated from the material.

3.2 *X-ray Diffraction Test*—The X-ray test method is non-destructive and yields the more precise measurement of orientation; however, use of the equipment requires compliance with stringent safety regulations.

3.3 *Optical Orientation Test*—The optical test method requires etching the specimen and is therefore destructive of polished wafer surfaces. This test method is less precise than the X-ray test; however, the apparatus required is less complex.

TEST METHOD A—X-RAY DIFFRACTION ORIENTATION

4. Summary of Test Method

4.1 The atoms of a single crystal form a periodic three-dimensional array in a crystal lattice structure whose units may be considered as lying in a series of parallel planes of equal perpendicular spacing, d , as shown in Fig. 1. When a beam of parallel, monochromatic X rays of wavelength λ is incident upon the planes, diffraction (reflection) occurs when the path difference of the X rays between adjacent planes is an integral number, n , of wavelengths. When this geometrical condition is satisfied, the reflections from the various planes of the series are exactly in phase and the diffracted beam is of maximum intensity. Thus, as shown in Fig. 1, the diffracted beam of X rays will possess maximum intensity when the angle of incidence, θ , of the beam with the reflecting planes, the X-ray wave-length, λ , the atomic interplanar spacing, d , and the order of the diffraction, n , simultaneously have values that obey Bragg's law as follows:

$$n \lambda = 2d \sin \theta$$

The reflecting planes are more commonly defined in terms of their Miller indexes (h , k , l). The Miller indexes are the

⁶ Published in Federal Register, Nov. 17, 1960. Available from Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402.

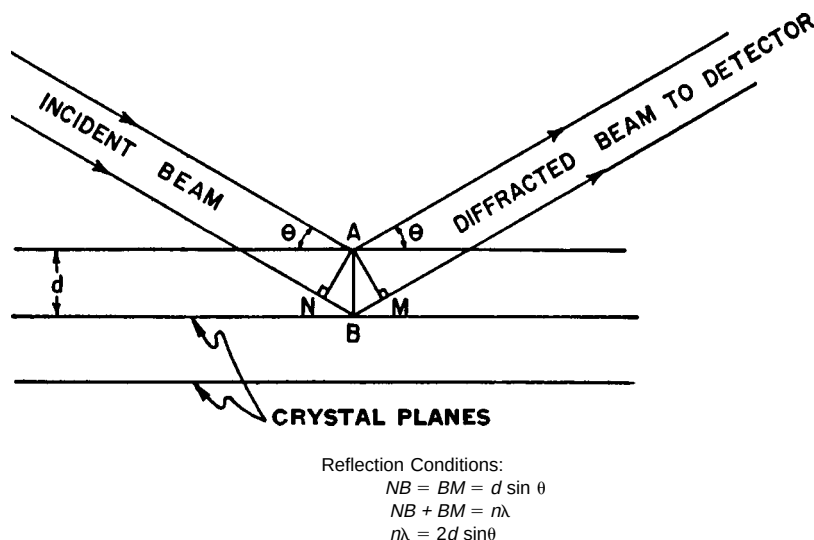


FIG. 1 Geometrical Reflection Conditions for X Rays from a Single Crystal

smallest integers proportional to the reciprocals of the intercepts of the plane on the three crystal axes of unit length. Thus, for any lattice structure where the periodicity is represented by a cubic unit cell of side length (lattice parameter), a , the lattice spacing, d , of a set of parallel atomic planes, may be written as follows:

$$d = a / \sqrt{h^2 + k^2 + l^2}$$

The angle, θ may then be found from the following modified form of Bragg's law for cubic lattice structures:

$$\sin \theta = n\lambda \sqrt{h^2 + k^2 + l^2} / 2a$$

In the diamond cubic structure, to which silicon and germanium, the Group IV semiconductors, belong, and in the zinc blende structure, to which gallium arsenide and the other Group III-Group V semiconductors belong, the following general rule gives the commonly observed reflections: h , k , and l must be all even or all odd with the further restriction that when h , k , and l are all even, then $h + k + l$ must be divisible by four. Values of θ for various low-order reflections, h , k , l , are given in Table 1 for silicon, germanium, and gallium arsenide.

4.2 The orientation of a single crystal surface is the crystallographic plane, described in terms of its Miller indices, with

TABLE 1 Bragg Angles, θ , for the X-ray Diffraction of $\text{CuK}\alpha$ Radiation in Semiconductive Crystals (Wavelength, $\lambda = 1.54178\text{\AA}$)

Reflecting Planes h, k, l	Silicon $a = 5.43073\text{\AA}$ ($\pm 0.00002\text{\AA}$) ^{A, B}	Germanium $a = 5.6575\text{\AA}$ ($\pm 0.00001\text{\AA}$) ^{A, B}	Gallium Arsenide $a = 5.6534\text{\AA}$ ($\pm 0.00002\text{\AA}$) ^{A, B}
111	14°14'	13°39'	13°40'
220	23°40'	22°40'	22°41'
311	28°05'	26°52'	26°53'
400	34°36'	33°02'	33°03'
331	38°13'	36°26'	36°28'
422	44°04'	41°52'	41°55'

^A a = Lattice parameter values.

^BReferences for lattice parameter values:

Silicon—Bond, W. L., and Kaiser, W., *Journal of Physics and Chemistry of Solids*, Vol 16, 1960, p. 44.

Germanium—Greiner, E. S., *Journal of Metals*, Vol 4, 1952, p. 1044.

Gallium Arsenide—Giesecke, G., and Pfister, H., *Acta Crystallographica*, Vol 11, 1958, p. 369.

which the surface is ideally coincident. In semiconductive single crystals, where the cross-sectional plane of the crystal or the surface of a wafer cut from the crystal usually corresponds within several degrees to a low index crystallographic plane, such as a {100} or {111} plane, the orientation is frequently described in terms of the maximum angular deviation of the low index crystallographic plane from the mechanically prepared surface. Other crystallographic planes may be found with respect to this low index plane by using a table of angles between crystallographic planes of a cubic crystal. Such a table appears in Test Method E 82.

4.3 The angular deviation of a reflecting plane from a prepared reference surface is found by determining two components of the deviation in two planes perpendicular to each other and to the reference surface. Each of the deviation components represents a setting of the crystal lattice that satisfies Bragg's law for the particular X-ray beam configuration. The reference surface itself must be perpendicular to the plane of the incident and reflected beams. Initially, an orientation determination is made at any arbitrary position of the crystal. Then the crystal is rotated 90° of arc about the normal to the reference surface (the normal lying in the plane of the incident and reflected beams) and a second orientation determination made. The crystal is rotated another 90° of arc (in the same direction) and a third orientation determination is made. The crystal has now been rotated 180° of arc with respect to the starting point. The crystal is then rotated another 90° of arc (that is, 270° of arc from the starting point) and a fourth orientation determination is made.

4.4 These four measurements can be combined to determine both the instrument error and the two components of the angular deviation, α and β , between the reference surface under investigation and the desired crystallographic plane. This information facilitates slicing of the crystal along the desired plane or alternatively the determination of the maximum angular deviation.

5. Apparatus

5.1 X-Ray Apparatus, commercially available, utilizing a