SEMI MF26-0714
TEST METHODS FOR DETERMINING THE ORIENTATION OF A SEMICONDUCTIVE SINGLE CRYSTAL

This Standard was technically approved by the Silicon Wafer Global Technical Committee. This edition was approved for publication by the global Audits and Reviews Subcommittee on May 12, 2014. Available at www.semiviews.org and www.semi.org in July 2014; originally published by ASTM International as ASTM E26-63T; previously published February 2011.

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1 Purpose

1.1 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.

2 Scope

2.1 These test methods 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.

2.2 Two types of test methods are covered as follows:

2.2.1 *Test Method A, X-ray Diffraction Orientation* — This test method may be used for the orientation of all semiconductive single crystals. The X-ray test method is nondestructive and yields the more precise measurement of orientation; however, use of the equipment requires compliance with stringent safety regulations.

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

NOTICE: SEMI Standards and Safety Guidelines do not purport to address all safety issues associated with their use. It is the responsibility of the users of the Documents to establish appropriate safety and health practices, and determine the applicability of regulatory or other limitations prior to use.

3 Referenced Standards and Documents

3.1 *SEMI Standards and Safety Guidelines*

SEMI C28 — Specifications for Hydrofluoric Acid

SEMI C30 — Specifications Hydrogen Peroxide

SEMI C40 — Specification for Potassium Hydroxide, 45% Solution

SEMI C43 — Specification for Sodium Hydroxide, 50% Solution

3.2 *ANSI Standard*[1]

ANSI B74.10 — Specifications for Grading of Abrasive Microgrits

3.3 *ASTM Standards*[2]

ASTM D5127 — Standard Guide for Ultra-Pure Water Used in the Electronics and Semiconductor Industries

ASTM E82 — Standard Test Method for Determining the Orientation of a Metal Crystal

ASTM E177 — Standard Practice for Use of the Terms Precision and Bias in ASTM Test Methods

[1] American National Standards Institute, 25 West 43rd Street, New York, NY 10036, USA; Telephone: +1.212.642.4900, Fax: +1.212.398.0023, http://www.ansi.org

[2] ASTM International, 100 Barr Harbor Drive, West Conshohocken, PA 19428-2959, USA; Telephone: +1.610.832.9585, Fax: +1.610.832.9555, http://www.astm.org

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3.4 *Other Standard*

Code of Federal Regulations, Title 10, Part 20, Standards for Protection Against Radiation[3]

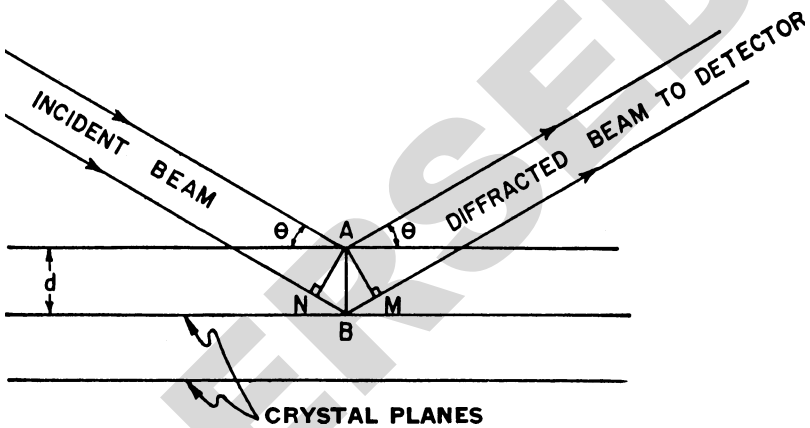
NOTICE: Unless otherwise indicated, all documents cited shall be the latest published versions.

4 Test Method A — X-Ray Diffraction Orientation

4.1 *Summary of Test Method*

4.1.1 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 Figure 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 Figure 1, the diffracted beam of X rays possesses 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:

$$n\lambda = 2d\sin\theta \tag{1}$$



#1 Reflection conditions are as follows: NB = BM = $d\sin\theta$, NB + BM = $n\lambda$, $n\lambda = 2d\sin\theta$.

Figure 1
Geometrical Reflection Conditions for X-rays from a Single Crystal

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

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

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

$$\sin\theta = \frac{n\lambda\sqrt{h^2 + k^2 + l^2}}{2a} \tag{3}$$

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

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4.1.1.3 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 to 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.

Table 1 Bragg Angles, θ, for X-ray Diffraction of CuK_α Radiation in Semiconductive Crystals (λ = 1.54178 Å)

Reflecting Planes h, k, l	*Silicon* *a = 5.43073 Å (±0.00002 Å)*[#1, #2]	*Germanium* *a = 5.6575 Å (±0.00001 Å)*[#1, #3]	*Gallium Arsenide* *a = 5.6534 Å (±0.00002 Å)*[#1, #4]
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′

#1 *a* = lattice parameter value.

#2 Bond, W. L., and Kaiser, W., *J. Phys. Chem. Solids* **16**, 44 (1960).

#3 Greiner, E. S., *J. Metals* **4**, 1044 (1952).

#4 Giesecke, G., and Pfister, H., *Acta Crystallographica* **11**, 369 (1958).

4.1.2 The orientation of a single crystal surface is the crystallographic plane, described in terms of its Miller indices, with 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 ASTM E82.

4.1.3 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 (i.e., 270° of arc from the starting point) and a fourth orientation determination is made.

4.1.4 These four measurements are 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.

4.2 *Apparatus*

4.2.1 *X-Ray Apparatus* — Commercially available, utilizing a copper-target tube, gives satisfactory results. The X-ray beam is collimated by means of a slit system to give parallel rays, and the X-rays are passed through a thin nickel filter to give a beam that is essentially monochromatic. The specimen is placed in a holder so that the surface under investigation contains the axis about which the specimen is rotated in satisfying the Bragg law conditions. Rotations about this axis are measured on a scale in degrees and minutes of arc. The holder must also permit rotation of the specimen about a normal to the surface under investigation. The Bragg angle corresponding to the particular X-ray wavelength used, the material under investigation, and the particular family of planes being oriented must be known from Table 1 or be determined. A suitable detector, such as a Geiger counter, is positioned so that the angle between

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