

SEMI M93-0923

TEST METHOD FOR QUANTIFYING BASAL PLANE DISLOCATION DENSITY IN 4H-SiC BY X-RAY DIFFRACTION TOPOGRAPHY/IMAGING

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1 Purpose

- 1.1 Basal plane dislocations (BPDs) are detrimental for devices manufactured on 4H-SiC substrates, especially for bipolar devices for power electronics.
- 1.2 Most BPDs are transformed into threading edge dislocations (TEDs) when growing epitaxial layers on top of 4H-SiC substrates, but a small fraction is also transferred into the epitaxial layer or may result in the formation of stacking faults (SFs), both being critical defects potentially causing device failure.
- 1.3 The density and distribution of BPDs are therefore important parameters describing the quality of a substrate. The specification of these parameters helps to select the appropriate material for a certain task.
- 1.4 The quantification of BPDs is possible by X-ray diffraction topography (XRT)—also known as X-ray diffraction imaging (XRDI). This Test Method provides information on the procedure to measure the BPD density using this technique and can be used for acceptance testing.

2 Scope

- 2.1 This Test Method covers the determination of BPD density on round test slices and commercial 4H-SiC substrates using XRT/XRDI.
- 2.2 The BPD density is used as a measure of the crystallographic perfection of a crystal.
- 2.3 This Test Method describes the requirements of the sample quality, the procedure to obtain a topogram suitable for BPD density measurement and to calibrate the results against results from etch pit data.

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 Limitations

- 3.1 This Test Method requires a surface condition free of subsurface damage and is not applicable if subsurface damage is present. Surface defects such as scratches or grinding marks will typically result in an overestimation of the BPD density.
- 3.2 The presence of other defects such as stacking faults may affect the quantification.
- 3.3 The BPD density is assumed to be constant along the sample normal within the volume probed, which depends on the (hkil) reflection used (between 20 μm and the full wafer thickness).
- 3.4 This Test Method is not applicable to substrates with epitaxial layers.
- 3.5 This Test Method is only applicable to wafers with nominal [0001] surface orientation and an off-orientation of between 3° and 10°.

3.6 The accuracy of the results is limited by the calibration procedure and thus by the quality of the reference data.

4 Referenced Standards and Documents

4.1 SEMI Standards and Safety Guidelines

SEMI M55 — Specification for Polished Monocrystalline Silicon Carbide Wafers

SEMI M59 — Terminology for Silicon Technology

SEMI M81 — Guide to Defects Found in Monocrystalline Silicon Carbide Substrates

SEMI M83 — Test Method for Determination of Dislocation Etch Pit Density in Monocrystals of III-V Compound Semiconductors

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

5 Terminology

5.1 Terms, acronyms, and symbols relating to compound semiconductor material technology are defined in SEMI M10, SEMI M55, and SEMI M59.

5.2 Definitions

5.2.1 *basal plane dislocation (BPD)* — dislocation with a propagation perpendicular to the [0001] crystallographic direction.

5.2.2 *curvature correction* — variation of the goniometer angles of an X-ray topography machine to compensate for local changes in the lattice plane orientations.

5.2.3 *defect-selective etching (DSE)* — etching of a crystalline material with an etchant that has different etching rates in the vicinity of a crystallographic defect than for the perfect lattice, leaving etch pits or hillocks that can be observed and counted by microscopic investigation.

5.2.4 *edge exclusion Δd* — region within a certain distance Δd from the edge of the wafer which is excluded from defect counting.

5.2.5 *off-orientation* — the tilt angle between the crystallographic c-axis and the wafer surface normal.

5.2.6 *stacking fault (SF)* — two-dimensional crystallographic defect resulting from a disorder of the normal stacking sequence of the crystal.

5.2.7 *threading edge dislocation (TED)* — dislocation with a propagation parallel to the [0001] crystallographic direction and a Burgers vector perpendicular to that.

5.2.8 *topogram* — image obtained by XRT/XRDI measurements.

5.2.9 *X-ray diffraction imaging (XRDI)* — imaging of the intensity of a diffracted X-ray beam.

5.2.10 *X-ray diffraction topography (XRT)* — imaging of the intensity of a diffracted X-ray beam.

6 Summary of Test Method

6.1 Samples for this Test Method should comply with the specification for surface defects according to SEMI M55. The backside preparation shall be good enough that no surface defects are visible in the topograms.

6.2 The wafer is placed on the sample stage of the X-ray topography machine.

6.3 The goniometer angles are adjusted for a Bragg reflection of the desired lattice and aligned for the sample.

6.4 A topogram of the full wafer is recorded.

6.5 The topogram is divided into a square grid. A value for each square grid cell is obtained by image analysis. The local BPD density is obtained by applying a calibration curve obtained from comparison to data from etch pit counting.

7 Apparatus

7.1 *X-Ray Topography Machine* — Able to generate wafer maps by scanning over the sample area.

7.1.1 *Goniometer* — Able to set detector and source angle such that a suitable reflection can be measured.

7.1.2 *Motorized Sample Stage* — Featuring a scan range covering the full wafer area.

8 Safety Precautions

8.1 *X-Radiation*

8.1.1 It is absolutely necessary to avoid personal exposure to X-rays.

8.1.2 It is the responsibility of the user to make sure that the equipment and the conditions under which it is used meet all applicable regulations.

9 Test Specimens

9.1 *Surface*

9.1.1 For measurements with radiation with a penetration depth less than the wafer thickness, the surface of the wafer requires a CMP finish on the measured face.

9.1.2 If the penetration depth of the applied radiation exceeds the wafer thickness, particularly in the case of measurements in transmission geometry, a mechanically polished surface on both sides is sufficient as long as no grinding marks are apparent in the topograms.

9.2 *Off-Orientation*

9.2.1 The offcut direction of the wafer must be in the [11-20] direction.

9.2.2 Any deviation from the offcut direction results in a discrepancy between etching and XRT results. A maximum of deviation of 3° is considered acceptable.

9.2.3 The off-orientation of the test specimen must be within $\pm 0.5^\circ$ of the off-orientation of the wafers used for calibration.

9.3 *Lattice Curvature*

9.3.1 Lattice curvature must be small enough that homogeneous image contrast can be achieved.

9.3.2 The upper limit depends on the properties of the XRT machine in use (beam divergence, beam dimensions, curvature correction strategy).

10 Calibration and Standardization

NOTE 1: The absolute accuracy of the procedure described in § 10.1 is limited by the accuracy of the reference data from etch pit counting (§ A1-2.1). In order to achieve best possible reproducibility between different systems/labs, it is recommended to use the same reference data for recalibration according to § 10.1 or to use a Golden Sample according to § 10.2.

10.1 *Calibration Against Etch Pit Data*

10.1.1 Measurement by XRT yields data of the BPD line length integrated over the penetration depth of the X-rays. The relevant information for judging the impact on epitaxial layers, however, is the number of intersection points between BPDs and the sample's surface, as indicated by etch pits obtained from a suitable technique. This is the physical background for the necessary calibration against DSE.

10.1.2 Select a set of at least three wafers that fulfill the requirements of § 9. The off-orientation of the wafers must be within a range of 1° ; smaller variations result in more accurate results. The local regions with the lowest BPD density should have a BPD density close to 0 cm^{-2} . The highest BPD density to which the measurements are applicable is limited by the maximum local BPD density of the calibration wafers.

10.1.3 Measure these wafers by XRT according to the Procedure described in this Test Method.

10.1.4 Carry out a defect-selective etch on the wafers after completion of the XRT measurements. For etching, an epi-ready surface fulfilling the specification in SEMI M55 is required. The measurement parameters must be chosen

in a way that undercounting due to etch pit overlapping is negligible. A suggested procedure for industry-standard highly n -doped 4H-SiC substrates is etching in KOH melt at 520°C for 12 minutes. For other dopant concentrations, different etching parameters might be necessary. Other etching parameters may be mutually agreed on between the parties involved.

10.1.5 Identify BPD-related etch pits based on their shape (SEMI M83) and count them (SEMI M81). Then, determine their local density on a grid identical to that used for the XRT analysis.

10.1.6 The chosen grid size a_F must be suitable. Grid sizes between 1 and 3 mm are recommended. When using a grid size below 2 mm, data binning or smoothing is recommended for DSE data (refer to Appendix 2). For XRT data, this is only recommended if the measurement is carried out in reflection geometry under strongly absorbing conditions (e.g., using the 11-28 reflection of Cu $K\alpha$ radiation), otherwise the measurement in transmission will already cause a smoothing of the data.

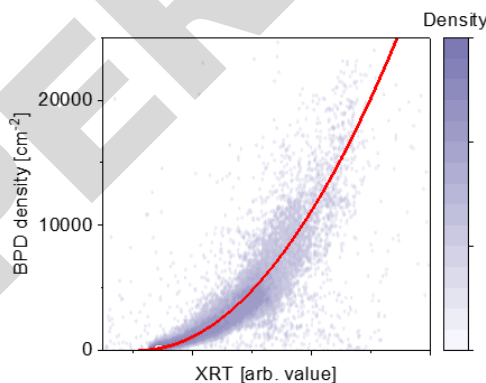
10.1.7 The coordinate system of both measurements must be identical. If necessary, transform the coordinate system (translation and rotation) such that the spatial deviation is minimized. The error should be below 0.5° in rotation and 1 mm in translation in any direction (refer to Appendix 2).

10.1.8 Plot the BPD density from etch pit counting over the respective values determined by XRT.

10.1.9 The calibration curve is obtained by fitting a polynomial function to the cloud of data points.

10.1.10 If an overlapping of etch pits cannot be excluded for a BPD density above a certain limit, the evaluation may be limited to this upper value. The measurements based on this calibration are then only valid for BPD densities below this value.

10.1.11 The correlation coefficient for each of these calibration curves should be not less than 0.5. A higher value is desirable and will result in a more accurate calibration. For high-quality reference data and an appropriate image evaluation algorithm, values above 0.8 can be achieved. A good correlation is only achieved when the measurement conditions and analysis routine for the XRT analysis were chosen well and when the quality of etch pit counting is sufficiently good.



#1 The calibration curve was obtained by fitting a parabolic equation to the data points.

Figure 1

Calibration Curve Showing a 2D Kernel Density Representation of the Correlation Between the Value Generated from XRT Measurements on the X-Axis and the BPD Density Determined from KOH Etching on the Y-Axis

10.2 Standardization Against a Golden Sample

10.2.1 Due to the nondestructive measurement approach of XRT, a Golden Sample can be defined which is measured in different laboratories.

10.2.2 Select a Golden Sample with a known BPD distribution defined as a Standard.