SEMI M63-0915
TEST METHOD FOR MEASURING THE Al FRACTION IN AlGaAs ON GaAs SUBSTRATES BY HIGH RESOLUTION X-RAY DIFFRACTION

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1 Purpose

1.1 The purpose of this Test Method is to describe the use of high resolution X-ray diffraction (HRXRD) as a means of measuring the Al fraction in an AlGaAs epilayer on GaAs substrates, by defining the experimental method and explaining the analysis.

2 Scope

2.1 This Test Method addresses measurement of the composition of unrelaxed, undoped AlGaAs epilayers on <001>-oriented GaAs substrates.

2.2 Principles are given for a standardized measurement method.

2.3 There are differing parameters for analysis already widely in use, either for historical reasons or from local values (e.g., of GaAs lattice parameter) which are appropriate to that user. As such, examples of analysis are given but a single standardized analysis is avoided.

2.4 With the standardized measurement method and a guide for standardized reporting of results, it should be possible to reproduce measurements at different sites, using suitable equipment.

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 does not apply to substrates which are cut to crystallographic planes other than (001).

3.2 The substrates shall have an off-orientation of no more than 15° to the (001) crystallographic planes.

3.3 For determination of Al fraction using direct calculation from peak separation, the layer(s) shall be more than 300 nm thick.

3.4 While thickness of the epilayer can be obtained from high resolution diffraction data, such analysis is not covered in this test method.

4 Referenced Standards and Documents

4.1 None.

5 Terminology

5.1 Refer to SEMI's Compilation of Terms (COT) for a list of the most current terms and their definitions.

5.2 *Abbreviations and Acronyms*

5.2.1 *FWHM* — full width at half maximum (maximum = peak of the curve)

5.2.2 *HRXRD* — high resolution X-ray diffraction

5.3 *Definitions*

5.3.1 *2Theta (2θ)* — the axis which alters the angle of the detector to the incident beam direction.

5.3.2 *ω-2θ* — a scanning axis where the detector angle (2θ) is scanned at twice the rate of the sample axis (ω).

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5.3.3 *arc second* — there are 3600 arc seconds in 1° angle.

5.3.4 *Bragg angle* — the angle for diffraction of X-rays from a set of crystallographic planes, as defined by Bragg's Law:

$$\lambda=2d\sin\theta \quad (1)$$

where λ is the wavelength of X-rays, d is the spacing between adjacent crystallographic lattice planes and θ is the angle between the planes and the X-ray beam when diffraction occurs.

5.3.5 *Chi* — the axis which tilts the sample about an axis in the plane of the sample and in the diffraction plane.

5.3.6 *Omega (ω)* — the axis which alters the angle between the incident beam direction and the sample surface.

5.3.7 *Phi* — the axis which rotates the sample about the normal to the surface.

5.3.8 *Poisson ratio, ν* — an elasticity parameter which describes how a material expands or contracts in one direction if compressed or extended in the orthogonal direction. It is composition-dependent.

5.3.9 *rocking curve* — scan of diffracted intensity measured as the angle between the incident beam and the sample diffracting planes is changed, going through a diffraction peak. The peak may be only a few arc seconds wide in a HRXRD measurement.

5.3.10 *scattering plane* — the plane containing the incident and diffracted X-ray beams.

5.3.11 *Vegard's Law* — this law states that the lattice parameter of a ternary alloy epilayer $A_xB_{1-x}C$ varies linearly with composition, x, between the lattice parameters of the two component binary alloys AB and AC; that is, between $0 \le x \le 1$.

6 Summary of Method

- Instrument Alignment
- Sample Alignment
- Sample Optimization
- Measurement
- Analysis

7 Apparatus

7.1 *High Resolution X-ray Diffractometer*

7.1.1 The incident beam divergence should be comparable with the intrinsic full width at half maximum (FWHM) from a GaAs substrate under a plane wave. For Cu radiation and the 004 reflection from a <001>-oriented sample, this should be ~12 arc seconds. A true double crystal system has a larger beam divergence but will measure the intrinsic FWHM of the sample, which would be <~12 arc seconds with Cu radiation.

7.1.2 There must be a means of mounting the sample stress-free. Vacuum methods must not be used.

8 Safety Precautions

8.1 X-rays are ionizing radiation and, as such, are hazardous to health. National and local regulations and rules must be adhered to, and good practice followed.

9 Test Specimen

9.1 The sample is an epilayer of $Al_xGa_{1-x}As$ on a GaAs substrate; specifically, an unrelaxed, undoped AlGaAs epilayer on a <001>-oriented GaAs substrate.

10 Preparation of Apparatus

10.1 The diffractometer should be aligned with the beam incident within 1 mm of the center of rotation of the sample axis.

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10.2 The sample axis, Omega, must be capable of angular steps of 2 arc seconds or less.

10.3 The 2Theta axis need not be movable, provided that the detector can be placed at twice the Bragg angle for diffraction and collect the diffracted beam over the whole range of the scan.

11 Calibration and Standardization

11.1 The measurement relies on the wavelength of the radiation used, which is usually very well known, and the accurate measurement of angle. Angle has a natural calibration in a full circle, if the X-ray instrument used has a full circle on Omega.

11.2 There is no need for standard samples in order to calibrate the measurement or analysis. However, users may wish to have "golden" samples available to check repeatability of the measurement on a given X-ray tool.

12 Procedure

12.1 The sample shall be mounted such that any off-orientation is directed perpendicular to the direction of the incident beam.

12.2 If the sample is off-orientation, adjust Chi by the nominal size of the off-orientation, in the direction which will bring the diffracting crystallographic planes perpendicular to the scattering plane.

12.3 If there is no off-orientation, place the major flat or notch 90° counter-clockwise from the incident beam direction (Figure 1).

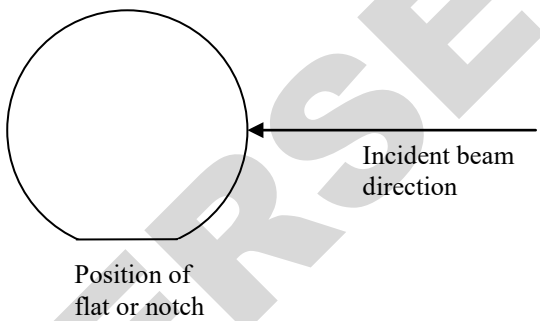

Figure 1
Orientation of Wafer Relative to Incident X-ray Beam, in the Absence of Off-Orientation

12.4 The surface of the sample shall be within 1 mm of the center of rotation of the sample axis, which shall coincide with the incident X-ray beam.

12.5 Adjust the detector position to $2\theta = 66.046°$ and the sample position to $\omega = 33.023°$.

12.6 Mount the sample in a stress-free manner. The success of this can be assessed by measuring the FWHM of the rocking curve of the substrate, after optimization (see ¶ 12.10). The FWHM should be less than 30 arc seconds.

12.7 If the FWHM is too large:

12.7.1 Try remounting the sample, in case the mounting caused stress;

12.7.2 Try reducing the beam size, in case there is an issue of sample curvature; and

12.7.3 Try rotating the sample 45° about its surface normal, in case there is an off-orientation issue.

12.8 If the FWHM remains large, there is an issue with the sample; for example, poor quality.

12.9 Locate the Bragg diffraction peak from the substrate by rocking Omega about the Bragg angle until strong diffracted intensity is observed.

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