

Technical Note

APPLICATION OF X-RAY DIFFRACTION TECHNIQUES TO THE SEMICONDUCTOR FIELD

SHIGERU MUNEKAWA

Rigaku Corporation, Tokyo, Japan

Atoms or molecules that compose a substance are generally arranged at a distance of 0.1 nm to 0.5 nm from one another. When such a substance is irradiated with X-rays having a wavelength roughly equivalent to the interatomic or intermolecular distance, the X-ray diffraction phenomenon will take place. X-ray diffraction is widely used in the semiconductor field because it is nondestructive and yields crystal structure information relatively easily in an atmospheric environment. Table 1 shows a summary of the application of X-ray diffraction techniques to the semiconductor device manufacturing process. Among various types of equipment used for this purpose, the thin film X-ray diffractometer employs an optical system (Fig. 1) specifically designed for the studies of thin films that are relevant to recent new materials. This diffractometer therefore allows measurement of thin film samples. The measurement has hitherto been considered impracticable. Generally, when the sample thickness becomes less than 1000nm the number










of crystal lattices that contribute to X-ray diffraction will decrease resulting in a drastic reduction in the X-ray diffraction intensity and a rise of the background. To cope with this, the thin film diffractometer is so designed that the incident X-rays are fixed at a small angle of 3° to 5° so as to lengthen the optical path along which the X-ray beam passes through the thin film. This increases the X-ray diffraction intensities. Also, a monochromator is mounted in front of the detector in order to reflect only the diffracted X-rays, to remove scattered X-rays emitted from the sample and to reduce the background.

As a measurement example, Fig. 2 shows a diffraction pattern of a sample formed by deposition of a 10 nm Au thin film on a Si single crystal substrate.

As examples of applications to the semiconductor field, Fig. 3 and Photo 1 show data obtained with the reflection method small angle scattering goniometer and with the double crystal topographic camera (listed in Table 1) respectively.



Table 1 Application of X-ray diffractometry to the semiconductor device manufacturing process.

Seed crystal		
Single crystal ingot		Crystal orientation measurement
Wafer		Crystal orientation measurement
Etching		Lattice constant precision measurement (absolute method)
Rapping		Lattice defect observation GaAs composition ratio
Epitaxial		Thin film analysis
Exposure		Lattice constant precision measurement (relative method)
Process treatment		IC superfine working
Cutting		
Bonding		
Package		IC micro area analysis Observation of defects in IC micro area

		Purpose	Application range	Equipment
Sample	Single crystal	Crystal orientation measurement	Si,GaAs GGG	Rigaku/Crystal orientation measuring system Rigaku/X-ray Laue camera
		Lattice defect observation	LiNbO ₃ , LiTaO ₃ diamond	Rigaku/X-ray Lang camera Rigaku/Double crystal topographic camera
		Lattice constant absolute measurement	Ferrite, quartz and other single crystals	Rigaku/Lattice constant precision measuring system (Bond method)
		Lattice constant relative measurement	GaAlAs on GaAs single crystal	Rigaku/Lattice constant precision measuring system (double crystal method)
		Quantitative analysis	GaAs composition ratio	Rigaku/GaAs X-ray stoichiometric system
	Polycrystal	Fine working on IC	VLSI	Rigaku/X-ray generator for X-ray exposure
		Qualitative and quantitative analysis	For analysis, High melting-point material on Si	Rigaku/X-ray diffractometer Rigaku/Thin film X-ray diffractometer
		Qualitative analysis of thin film	Co-Ni film on film, Lens coating material	Rigaku/Fully automatic pole figure diffractometer Rigaku/Small angle scattering goniometer (double crystal method)
		Thin film crystal system determination	Memory material	Rigaku/Reflection method small angle scattering gonio,eter
		Thin film texture (preferred orientation)	Various thin films on semiconductors	
	Thin film particle size measurement			
	Qualitative analysis of micro areas	IC precipitates, deposits	Rigaku/Microdiffractometer (PSPC/MDG)	
	Xray absorption fine structure	Si fine structure	Rigaku/X-ray EXAFS measuring system	
	Observation of micro defect in matter	Micro defect on bipolar IC pacakge	Rigaku/Microfocus X-ray fluoroscopic system (MRS)	

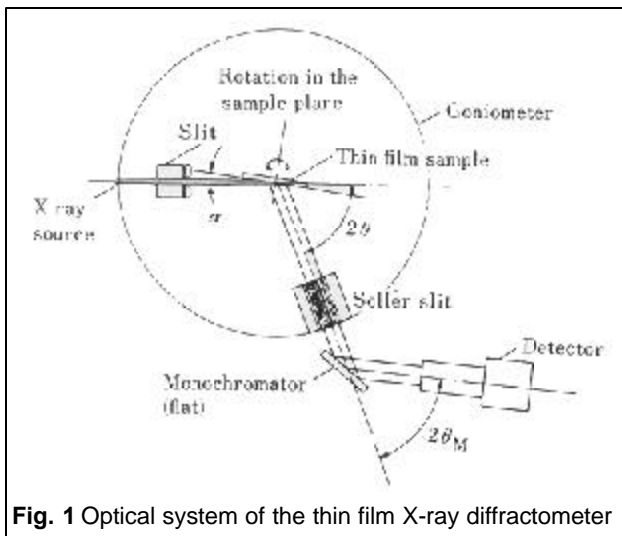


Fig. 1 Optical system of the thin film X-ray diffractometer

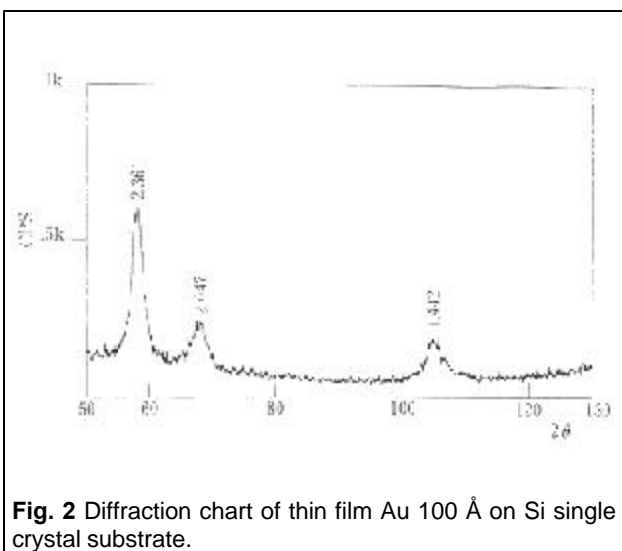


Fig. 2 Diffraction chart of thin film Au 100 Å on Si single crystal substrate.

Example of Measurement with the Thin Film X-ray Diffractometer System

Fig. 2 shows the measurement example of a gold thin film (thickness: approx. 10nm) on a silicon single crystal substrate. Au (110), Au (200), and Au (220) which are unobservable with the conventional X-ray diffractometer were easily detected.

[Measurement condition]

Target: Cr
 X-ray condition: 40 kV, 50 mA
 Slit: Divergence slit (DS) 0.6 mm
 Incident X-ray angle: $\alpha=3^\circ$

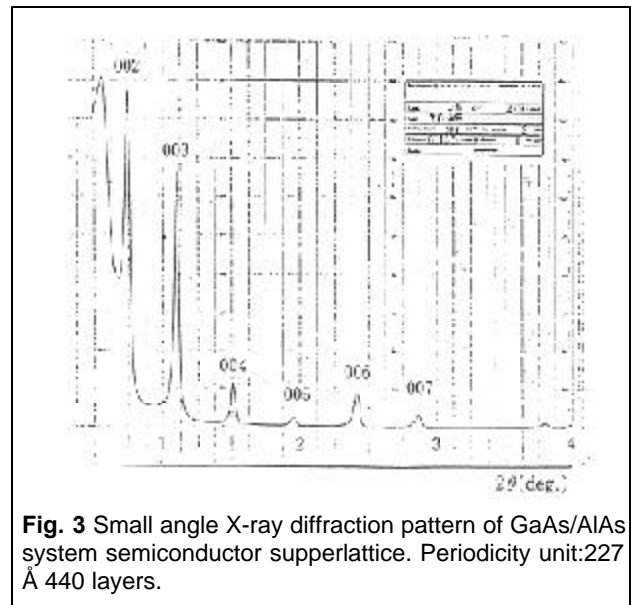


Fig. 3 Small angle X-ray diffraction pattern of GaAs/AlAs system semiconductor superlattice. Periodicity unit: 227 Å 440 layers.

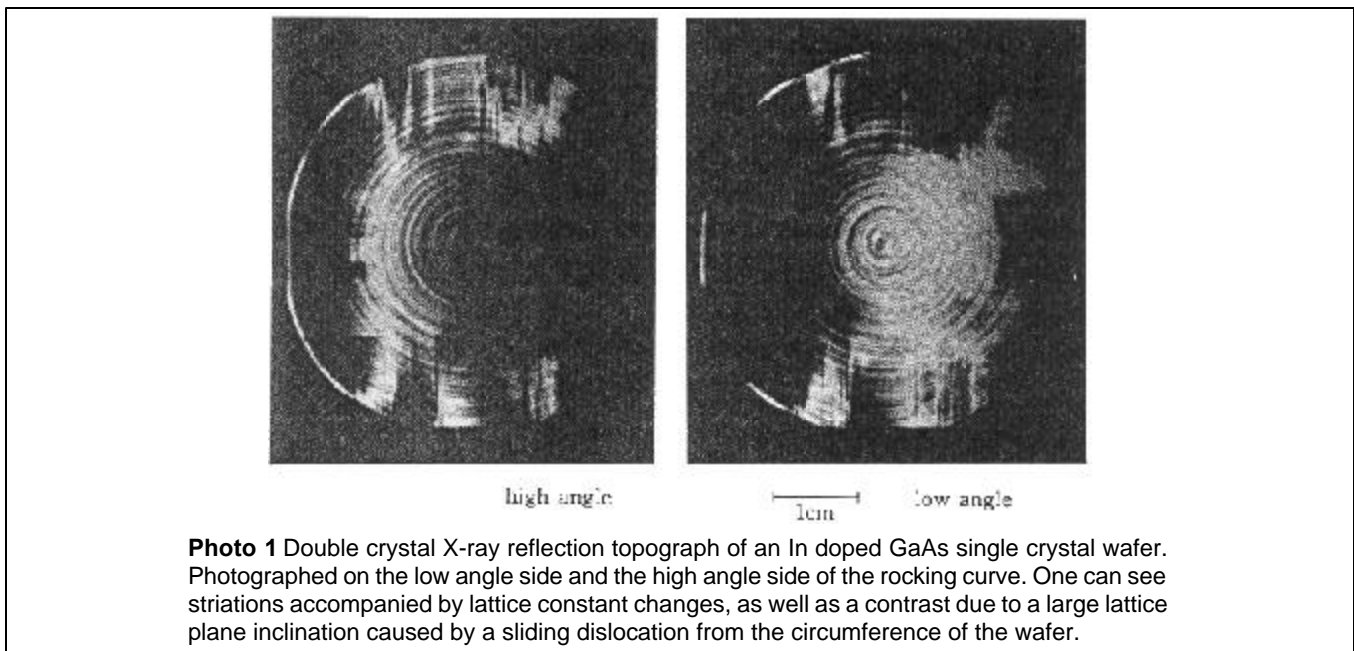


Photo 1 Double crystal X-ray reflection topograph of an In doped GaAs single crystal wafer. Photographed on the low angle side and the high angle side of the rocking curve. One can see striations accompanied by lattice constant changes, as well as a contrast due to a large lattice plane inclination caused by a sliding dislocation from the circumference of the wafer.

Example of Measurement with the Reflection Method Small Angle Scattering Goniometer

This is a 2-axis ($2\theta, \theta$) small angle scattering goniometer combined with a Kratky U slit and a diffracted beam monochromator. It features a capability of obtaining good signal-to-noise ratio data at low angles. Fig. 3 shows a measurement example of a GaAs/AlAs system semiconductor. A superlattice pattern can be clearly seen in this example.

[Measurement condition]

X-ray-condition: Cu 40kV, 200mA

Slit: ES: 70 μ m, RS: 0.14mm

SS: 6mm

Scanning speed: 0.125 $^\circ$ /min

[Data]

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Example of Measurement with the Double Crystal Topographic Camera

It can easily be seen in the topographic image that the incidence of X-rays monochromated by the first crystal on a nearly perfect single crystal (2nd crystal) at a Bragg angle yields diffracted X-ray intensities from lattice defect areas differ from those from the surrounding perfect areas. The double crystal topographic camera employs the (+, -) parallel arrangement optics. The first and second crystals set up the asymmetrical and symmetrical reflection plane, respectively. Then an image is photographed on a photographic dry plate. By arranging the same interplanar spacing about the two crystals [e.g., Si (511) and Si (330) planes], it is possible to obtain an extremely narrow rocking curve. Likewise, by installing the sample at the second crystal position, it is possible

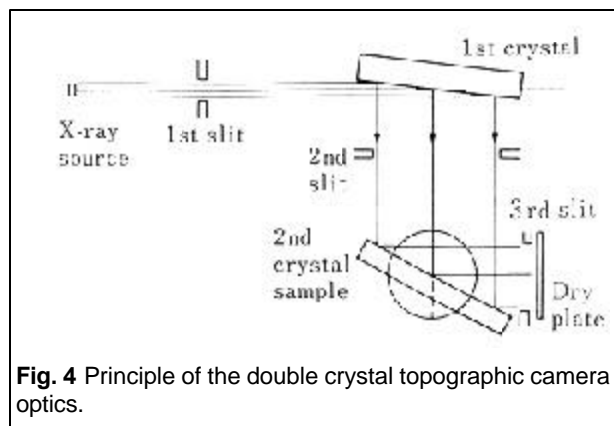


Fig. 4 Principle of the double crystal topographic camera optics.

to observe a very small lattice distortion ($\Delta d/d = 10^{-5} \sim 10^{-7}$, an inclination of about 0.1 second, etc.) or a point defect. Photo 1 shows a double crystal topographic photograph of an In doped GaAs crystal. While the lattice plane inclination in the wafer plane is as small as several seconds or so, an intense contrast (large lattice plane inclination) is seen on the circumference portions of the wafer.

[Data]

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