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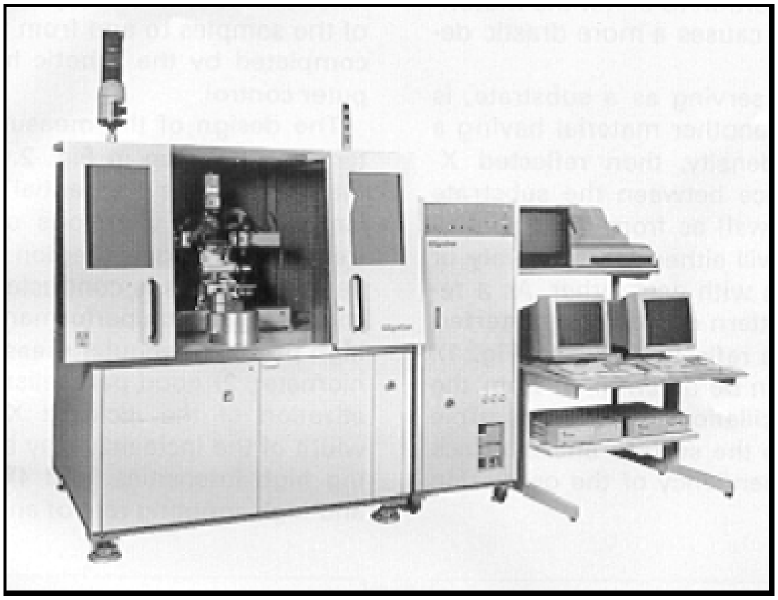
Product Information

GRAZING INCIDENCE X-RAY REFLECTOMETER GXR^2



1. Introduction

In recent years, thin film materials have been applied to various sorts of electronic devices and are now playing a central role in high technology materials. Because the characteristics of devices largely depends on the single and multilayered film thicknesses, density, surface and interface roughness, the method of evaluating these relevant quantities is critical for control of the conditions of films manufacturing. Against such a backdrop, intensive studies using X-ray reflectometry have been progressing and broadening their applications. This method is such that the X-ray beam is made to impinge onto a material's surface at grazing angles of incidence in order to analyze the reflected X-ray intensity profile with respect to the angle of incidence. Parameters of this film structure are then determined.

In parallel with the broadening application of thin film materials evaluation by this technique, there has been a growing demand for measuring instruments geared for rapid processing of routine work. Introduced here is a system called the Grazing Incidence X-ray Reflectometer GXR^2, which has been specifically developed to meet such requirements.

2. Applications

- This system permits non destructive, non-contact measurements of film thicknesses ran-ging from several tens to several thousands Å.
- Assessment of the surface roughness and the interface width (defined by roughness and mutual dispersion/diffusion) can be made.
- If the film composition is already known, assessment of the film density can be made.
- Structural assessment can be made of both monolayer and multilayered films.
- This method is applicable to a wide range of materials regardless of crystalline nature, including semiconductor thin films, superconductors, magnetic substances, metals, macromolecular thin films and so on.

3. Principle

When X-rays are applied to a material's flat surface at grazing angles of incidence, total reflection will occur at or below a certain angle, θc. This angle is exceedingly small and is referred to as the critical angle. In the case of Cu Kα radiation, the critical angle is 0.22° for silicon on a glass plate, 0.42° for nickel, and 0.57° for gold. The angle varies depending upon the electronic density of the material. The higher the incident X-ray angle relative to the critical angle, the deeper the X-rays transmit into the material. With a material whose surface is ideally flat, the reflectivity suddenly decreases at angles above θc in proportion to θ^{-4}. If the material surface is rough, it causes a more drastic decrease in reflectivity.

If such a material, serving as a substrate, is evenly overlaid with another material having a different electronic density, then reflected X-rays from the interface between the substrate and the thin film as well as from the free surface of the thin film will either constructively or destructively interfere with each other. As a result, an oscillation pattern due to X-ray interference will appear on a reflectivity curve (Fig. 1). The film thickness can be determined from the periodicity of this oscillation. It is also possible to get information on the surface and interface from the angular dependency of the oscillation pattern's amplitude.

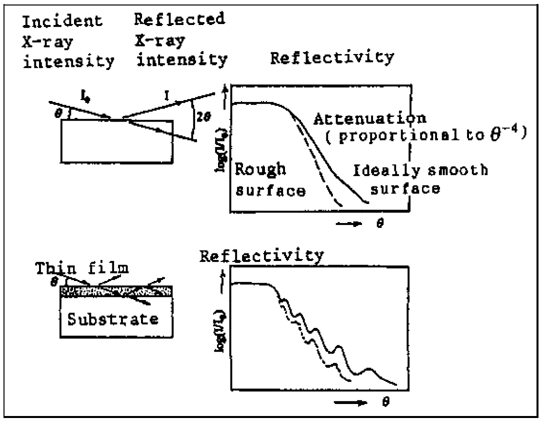


Fig. 1 Principle of X-ray Reflectivity Measurement

4.System Configuration and Features

The Grazing Incidence X-ray Reflectometer GXR^2 is so designed that the operator has only to insert the sample. The system will take care of all the rest of the operations automatically. It is a handy tool designed and dedicated to routine work.

The GXR^2 is comprised of a sample feeding elevator, a robotic handler, a central unit in the which the measurement optical system is arranged, an X-ray generator power supply, a controller containing stepping motor driver controllers and the counting circuitry, and a computer system. All these units are incorporated in a radiation enclosure equipped with a safety interlocking mechanism.

Up to ten samples can be inserted in a magazine holder. Each sample is mounted on a pallet. The magazine is set onto an elevator located inside a front lower door of the central unit, After the door is closed, feeding and removing of the samples to and from the sample stage is completed by the robotic handler under computer control.

The design of the measurement optical system can be seen in Fig. 2. For the reflectivity measurement, it is essential to accurately detect large intensity variations occurring in an extremely low angular region where grazing incidence of X-rays is conducted. Thus the system should have such performance constraints as 1) high precision angular measurement with a goniometer, 2) good parallelism and monochromatization of the incident X-rays, 3) a narrow width of the incident X-ray beam but still allowing high intensities, and 4) a low background and high counting rate of an X-ray detector.

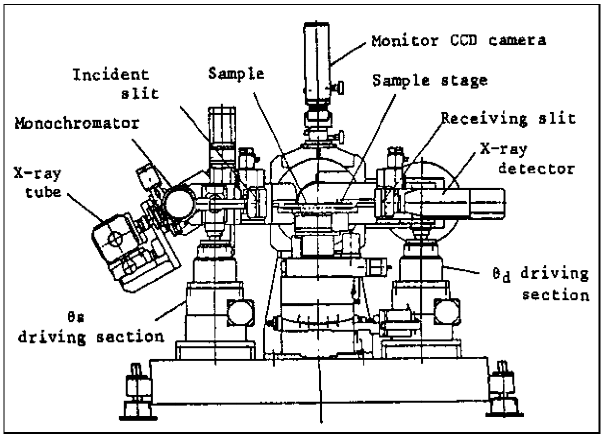


Fig. 2 Measurement Optical system

The goniometer is designed in a “θ/θ” mode, such that its θs optics (X-ray tube, monochromator, incident slit, and related adjusting mechanisms) are mounted on one moving arm, and its θd optics (receiving slit and X-ray detector) are held on a second arm. The θs and θd axes coaxially rotate by means of carefully preloaded precision bearings.

Rotational driving is made with a tangential screw system fine rotation mechanism through stepping motor drivers. Usually, step scanning is performed at about 0.001°, with an angle measuring precision better than 10^{-4}°.

For the X-ray source, a 2 kW sealed X-ray tube equipped with a Copper target is used. The X-rays from the point focus orientation impinge on a Ge (111) monochromator, which employs an asymmetric cut to compress the beam width to be 80 μm. The use of such a monochromator designed to compress the beam width allows a shorter dimension for the optical system. While dispersing the $K\alpha_1$ and $K\alpha_2$ X-rays, very little loss in intensity is observed in the X-ray source. This makes it possible to obtain highly intense X-ray beams. The removal of the $K\alpha_2$ is made with the incident slits, and the beam width and length are adjusted at the same time. The receiving slit, placed in front of the X-ray detector, limits X-ray scattering from the sample. In the standard configuration both slits are 0.05mm wide and 2 mm long.

A scintillation counter is employed as the detector. Its background is excellent -0.1 cps or less. To cope with high counting rates, a counting loss correction is provided to maintain linearity over a range up to 800,000 cps. Thus, a 6-order dynamic range is assured.

For alignment of the measurement optical system, adjustments are made regarding the ω_M and $2\theta_M$ rotations of the monochromator and the positions of the incident and receiving slits so that only the Cu $K\alpha_1$ passes through the axes center of rotation. All of the alignment conditions are adjustable by remote control with the aid of stepping motors.

Optimum X-ray beam wavelength dispersion is achieved and maintained by the above-mentioned optical system. The overall dispersion for the natural line width of the Cu $K\alpha_1$ radiation ($\Delta\lambda/\lambda=3.8 \times 10^{-4}$) still gives 0.1 mrad in the divergence angle and over 400,000cps intensity when combined with the receiving slit.

The sample stage is set at the point of intersection of the θs/θd axes and the X-ray beam. A sample may be placed directly onto the pallet which serves as the sample holder plate. No adhesive, vacuum, or clamp is needed. The axes of the sample stage may be separated according to their functionality. One group is the adjusting axes required for the selection of the measurement location on the sample. These included the sample's rotation in the plane and forward/backward and sideways translations. A combination of these allows selected positioning anywhere on a sample up to 6 inches in diameter. The other set of axes is the adjusting axes required for the so-called “halving” (Sample axis = θ axis adjustment) for the sample's upward/downward positioning and adjustment in two orthogonal rotational directions for centering on the reference point. This adjustment is designed to make the sample plane coincide with the reference plane formed by the θs/θd axes and the X-ray beam. Each axis of the sample stage is driven by a stepping motor under computer control.

The features of the Grazing Incidence X-ray Reflectometer GXR^2 may be summarized as follows:

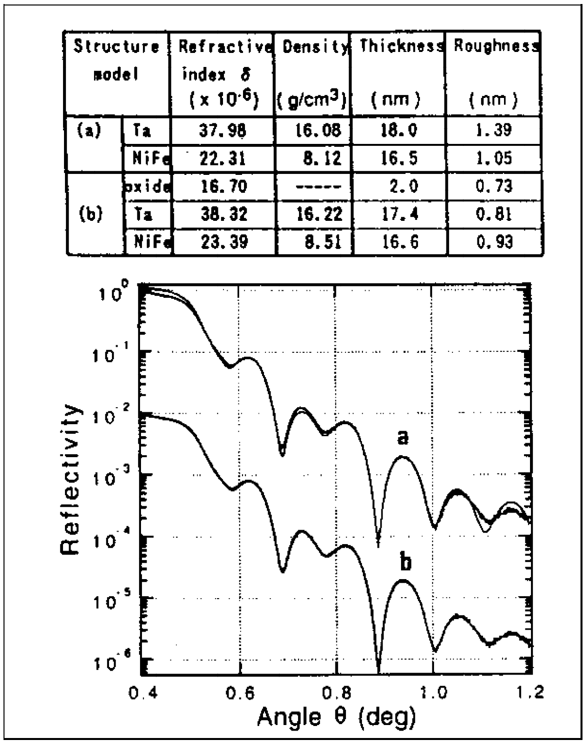
- The X-ray optical system is of a vertical type where the sample is held horizontally. Therefore, there is no need for clamping or adhesive. The sample is free from contamination or distortion.
- With electrically driven adjusting axes, coupled with computer control, automatic and remote-controlled operations can be made safely and easily.
- A 6 inch sample can be handled, and the rotation in the plane and the X-Y stage movement make it possible to conduct area map measurements.
- The use of the tangent bar system goniometer assures high precision measurements.
- The use of a monochromator that can compress the X-ray beam width enables designing of a shorted optical system, leading to a higher intensity X-rays beam.
- The detector counting system used features low backgrounds and is able to cope with high counting rates. A 6-order dynamic range is provided.

5. Analysis Software

An oscillation pattern appearing on the reflectivity curve represents the film structure with high sensitivity. Accordingly, by analyzing this oscillation pattern, it is possible to evaluate the film thickness, surface roughness, and interface width of thin films.

The analysis software includes 1) the Fourier analysis method [1], 2) simulation calculations, 3) curve fitting by the least squares methods, and 4) X-ray optical constants (δ, β) calculations [2].

The Fourier analysis methods is initiated by subtraction of the mean reflectivity from the



Structure model		Refractive index δ (x 10⁻⁶)	Density (g/cm³)	Thickness (nm)	Roughness (nm)
(a)	Ta	37.98	16.08	18.0	1.39
	NiFe	22.31	8.12	16.5	1.05
(b)	oxide	16.70	-----	2.0	0.73
	Ta	38.32	16.22	17.4	0.81
	NiFe	23.39	8.51	16.6	0.93

Fig. 3 Comparison of the fitting results between when the surface film oxide was ignored (a) and when it ws given consideration (b) in the case of Ta/NiFe/SiO_2.

measured reflectivity. After normalization, the oscillation component is extracted. The film's refractive index is used for angular correction. Fourier transformation is carried out to obtain the film thickness from the oscillation periodicity. This method is simple and allows determination of the film thickness with fairly high precision. Moreover, improved results can be obtained when the preliminary Fourier transformation results are used for simulation calculations.

The simulations calculation procedure is based on a recursion formula [3], established by Parratt, and is combined with theory which takes roughness [4] into account. It is software for creating simulated reflectivity patterns to permit comparisons between measured data and the simulation results on the CRT. By this method, it will be impossible to obtain good agreement with a bad theoretical model. For instance, when a material is susceptible to oxidation, it is absolutely necessary to set up a model which includes a surface film oxide.

When a relatively good agreement is reached between the simulation results and the measured data, parameter refinement can be applied to attempt to adjust the parameters used in the simulation calculations to optimize the fit between the two. A non-linear least squares method [5] is employed for the fitting.

Figure 3 shows an example of analysis by the fitting method [6]. The sample is a stacked film of Permalloy (Ni-Fe alloy) and Ta on a glass substrate. With respect to the experimental data (dotted curve), the two solid lines show the results of fitting with a model (a) where the Ta surface film oxide was ignored and (b) where it was considered. In the case of the model ignoring the film oxide, some disagreement is seen between the experimental and the calculated patterns. When the film oxide is considered, an excellent agreement is noted between the experimental and calculated patterns over the entire angular region.

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