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Local thickness measurements using reflectivity of X-rays in the dispersive angle mode

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Abstract

The multiple beam interference of parallel layers and/or the Bragg peaks of multilayers are measured in the angle-resolved dispersive mode with a position sensitive proportional counter. The specimen is illuminated by a divergent X-ray beam produced by a sealed linear focus tube. The arrangement permits either to obtain spectra in a short time and to eliminate any movement during measurement. Furthermore, this configuration has the advantage of analysing a small surface area of the specimen (less than 1 mm^2). Consequently, a scanning of a layer or a multilayer is possible. Both examples concerning a deposited layer and a multilayer will be given, showing variation in thickness according to the distance between the sputtering or evaporation system and the sample holder.

Introduction

Surface coating and surface treatment of materials are now widely developed as techniques for modification of surface properties because there is a great commercial promise [1,2], especially when assisted by implantation. In this context, new techniques have been developed to get accurate measurements of the structures on an atomic or medium range scale.

Grazing X-ray reflectometry (GXRR) is one of these new techniques which combine surface sensitivity and small penetration depth. It is a non-destructive technique which is well suited in thin layer characterization (Parratt [3], 1954). A monochromatic and quasi-parallel X-ray beam is sent at grazing incidence onto the surface of a flat sample. The specularly reflected beam intensity is recorded versus the grazing angle θ of the incident X-ray beam with respect to the average plane of the illuminated surface. This technique has been widely applied to the study of surfaces, thin layers and multilayers, because it allows measurements of thicknesses, electron densities, periodicity and roughness of multilayered samples.

The reflectometry method has developed with high accuracy goniometers because of very small angles to be measured. An example is the high quality reflectometer with all remote controlled movements built at the Institut d'Optique d'Orsay (Nevot [4], 1978). Its angular mechanical accuracy is 1 second of arc. The surface alignment is critical and recording the reflectivity curve $I(\theta)$ with stepping motors needs quite a long time. Furthermore the surface flatness of the studied sample must be perfect and the illuminated area in the vicinity of the critical angle θ_c is of the order of 1 cm^2 .

We have developed a new reflectometer working in the angular dispersive mode, easy to use and fast in its running, even if its accuracy is not as good as a classical θ - 2θ diffractometer. It has the following important advantage: only a small surface area of the sample is illuminated by the X-ray beam.

Principle of the set-up.

The principle of our new set-up has already been described elsewhere [5]. It is based on the simultaneous recording of the reflected beams in a large angular range with a position-sensitive detector (see figure 1).

Figure 1 : Principle of the set-up in the angular dispersive mode : (F) X-ray line focus; (E) sample; (C) knife edge; (M) monochromator; (D) counter.

The sample E, which has a small area, is hit by the X-ray beam produced by a sealed X-ray tube with a linear focus F (of apparent size $8 \times 0.04 \text{ mm}^2$) in the plane perpendicular to the sample surface. A knife edge C, very close to the sample surface, is used for absorbing the useless primary beam and for determining the angular origin at one and the same time.

The X-ray beam is reflected by a polished Ge (111) crystal which acts as a monochromator and selects a characteristic wavelength of the X-ray beam. The linear focus can be considered as a linear set of point sources, each of them giving a different incident angle onto the sample. It should also be observed that any fluorescence coming from the sample is eliminated by the monochromator.

The scattering profiles are recorded on a one-dimensional position-sensitive proportional counter which allows short counting times.

There is no mechanical movement during the recording of a spectrum. Furthermore, fluctuations of the filament current do not have to be taken into account during the measurement because of the accumulation procedure.

The sample-counter distance can also be adjusted in order to get both the required angular range and a good accuracy. For example it is easy to obtain in the same spectrum the total reflection and a set of interference fringes in the case of a thin layer, or several Bragg peaks and their secondary peaks in the case of a multilayered sample. The angular resolution of the spectrometer is 12 seconds of arc and its energy resolution of 5 eV.

One of the main advantages of this spectrometer working in the angle-resolved dispersive mode with a linear X-ray focus is that the surface area of a small sample illuminated by the X-ray beam is less than 1 mm^2 . In fact it is a linear surface of about $50 \mu\text{m}$ in width and having the size of the sample in the X-ray direction (typically 5 mm) which is analysed. So, different measurements can be made on a same sample in order to check the fabrication device, for example the homogeneity of the thickness of a layer or the periodicity of a multilayer.

Local measurement of a TiN layer

When a sample is made of a single layer of uniform thickness t with a refractive index η higher than the one of the substrate on which it is deposited, the intensity curve $I(\theta)$ display oscillations which are the Kiessig fringes [6]. This is an interference phenomenon between the beam partially reflected by the air-layer interface and the beam reflected by the layer-substrate interface. The angular position and the order of these interference fringes provide means to determine the layer thickness t . For example two maxima of the fringes of integer order m and n corresponding to angles θ_m and θ_n are related to t by the following formula derived from the Snell-Descartes

relationship :

$$t = \frac{\lambda}{2} \left(\frac{m^2 - n^2}{\theta_m^2 - \theta_n^2} \right)^{1/2} \quad (1)$$

Fig. 2 shows a concrete example of the full potentiality of our reflectometer with two local measurements of a TiN sputtered layer on a 8" silicon wafer. The wafer has been cut and two samples of small surface area taken, one in the centre of the wafer and the other at the edge. Measurements of their thicknesses, as determined by formula (1) give 428 Å and 393 Å respectively, with an accuracy of 2 Å. Such a difference is clearly seen in the figure. It is well understandable that with a larger area illuminated by the X-ray beam, as common for a standard goniometer, the reflectivity curve would not have displayed such well contrasted fringes, and addition of different elementary set of fringes could lead to a general blurring of the scattering pattern.

Figure 2 : reflectivity curves of a TiN layer with Kiessig fringes, in the centre of the wafer and at the edge. The accumulation time is 2 min for each curve.

Local measurement of multilayers

For a multilayer, the classical Bragg formula must be corrected for refraction and for interference order p . It becomes :

$$p \lambda = 2 d \sin \theta [1 - \delta / \sin^2 \theta] \quad (2)$$

where δ is the real part of the refractive index η of the multilayer.

An example of a multilayered sample is shown in Fig. 3. It is a stack of 50 bilayers of around 40 Å of Si and 60 Å of Ti, elaborated by magnetron sputtering and having a thickness variation of about 15% between the centre and the edge of the 2" silicon wafer [7]. Such a multilayer is a good candidate for a local measurement with our reflectometer. The sample we measured had a rectangular shape (1 x 3 cm²) as indicated in the inset of the figure. The distance between the two measurements was 1 cm and the accumulation time only 1 min. The period of the multilayer as determined with the 3 Bragg peaks is 104 ± 0.5 Å for the dashed curve and 96 ± 0.4 Å for the full curve.

Figure 3 : two local measurements of the periodicity of a Ti/Si multilayer, at 1 cm from each other. The accumulation time is only 1 min.

Another example of a multilayer is shown in Fig. 4. It was elaborated in an ion-sputtering evaporator and consists of 12 bilayers of gold and nickel (average thickness of 73 Å). The distance between the target and the sample holder (circle of 10 cm in diameter which rotates during the deposition) is around 30 cm. Two local measurements of the periodicity, performed on two small area samples, one in the centre of the sample holder and the other one at the edge, indicate values of 75 Å for the former and 71 Å for the latter; that means a variation of about 5%. Such a value is in full agreement with the 30 cm distance between the target and the sample holder of 10 cm in diameter, because a deposited layer has a thickness proportional to the square of the target-sample distance. The difference in distance between the centre and the edge being 0.8 cm.

Figure 4 : two local measurements of the periodicity of the Au/Ni multilayer elaboration, one in the centre and the other one at the edge. The accumulation time is 2 min.

Conclusion

Different advantages of this reflectometer working in the angular-dispersive mode have been shown in this paper. The arrangement permits to obtain spectra in a short time and to eliminate any movement during measurement. Furthermore, this configuration has the advantage of analysing a small surface area of the specimen (less than 1 mm²). Consequently, a scanning of a layer or a multilayer is possible. Both examples concerning a deposited layer and a multilayer have been given, showing variation in thickness according to the distance between the sputtering or evaporation system and the sample holder.

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