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Two-channel X-ray reflectometer

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Abstract

The two-channel X-ray reflectometer is proposed providing an increase in accuracy and sensitivity especially to nanoscale oxide layers. The reflectometer has two independent measuring channels controlled by a processor and the beam-splitting and spectral selection device based on a row of semitransparent plates of pyrolitic graphite. Results of reflection curve measurements in a relative mode are presented for an Ni film and GaAs monocrystal. © 2000 Elsevier Science B.V. All rights reserved.

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1. Introduction

Increasing production and application of thin film, multilayer structures, and other kinds of planar objects demand for more quick, accurate, and reliable methods of their characterization. Optical ellipsometers and X-ray reflectometers allowing noncontact measurements directly in air conditions meet some of these demands [1–4]. However, at a scale of thicknesses of ~ 1 nm and less they do not provide necessary reliability and accuracy. In this work we describe a new measurement scheme – the two-channel X-ray reflectometer (TCX-reflectometer) – capable of improving drastically thin film and surface layer metrology and give a more detailed information on the surface layer structure.

2. Description of the TCX-reflectometer

General view of the TCX-reflectometer measuring scheme is shown in Fig. 1. Standard X-ray diffractometer DRON-3M ("Burevestnik") with a horizontal-type goniometer was chosen as a work platform. A copper anode tube with a focus projection $8 \times 0.04\,\mathrm{mm}^2$ was used as an X-ray source. Distances from the main axis of goniometer O_1 to the focal spot and to receiving slit 9 were 330 and 225 mm, respectively. A prototype of this reflectometric scheme, where a single monochromatic line is selected after passing through a receiving slit, was described earlier in Ref. [5]. The essence of our cardinal improvement of this arrangement lies in setting on the arm support 15 a beam-splitter and spectral selector (splitter-selector based on semitransparent (0 0 0 1) pyrolitic graphite (see Fig. 2). The first monochromator along the X-ray path was a plate of pyrolitic graphite 46 μm thick with $0.8\,\mathrm{cm}^2$ in area. This plate was fixed at the Bragg

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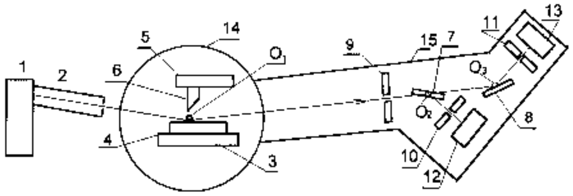

Fig. 1. General view of the X-ray optical scheme of TX-reflectometer. (1) X-ray tube; (2) collimator; (3) sample holder; (4) sample; (5) linear movement device; (6) X-ray shield; (7) semitransparent monochromator; (8) replacable monochromator; (9–11) slits; (12, 13) detectors; (14) rotation table; (15) rotation arm support.

angle (13.2°) for Cu K_α radiation. It had peak diffraction reflectivity $R_{dif} = 22\%$ for Cu K_α doublet, which in total external reflection measurements may be considered a single characteristic line, and transparency T equal to 85% of Cu K_β line. The second monochromator was replacable. In experiments described below we used 1 mm thick pyrolitic graphite. To appreciate the possibility of further increasing the number of analyzed spectral lines, up to three semitransparent plates 50–75 μm thick were installed in a row. If all the three plates were tuned to the Cu K_α line the sum R_{dif} and T for Cu K_β were equal to 47% and 52%, respectively, that clearly demonstrates the possibility of multichannel mode.

Specially developed electronic block was used for simultaneous data acquisition from two scintillation counters. The block consists of power supply for photomultipliers, processor and memory modules, and two amplitude discriminators. It is able to work autonomously and transmit data to the computer upon completion of the planned measurement cycle.

3. Measurements in relative mode

Advantages of simultaneous data acquisition in two spectral bands are evident enough. One may receive twice as more data during a single Θ–2Θ scan and check the results of calculations for different spectral lines. However if intensity results of reflectivity measurements are determined from the ratio $I^r(\Theta)/I_0$, where I^r (Θ) and I_0 are specularly reflected at the grazing angle Θ and incident intensity of the X-ray beam for each spectral line, respectively, the inherent errors of the one-channel scheme is preserved. It is mainly due to uncontrollable change of illumination along the sample surface at small angles when the size of its projection on the plane normal to the incident beam is comparable with an HWHM of the X-ray beam.

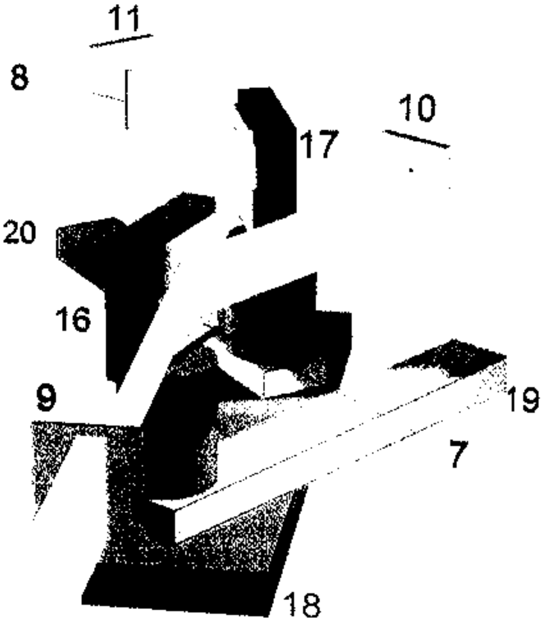

Fig. 2. Beam-splitting and spectral selection block. (7, 8) semitransparent monochromators; (9–11) slits; (16, 17) rotation monochromator heads; (18) monochromator support; (19, 20) detector supports.

If the shield 6 is made of highly absorbing material and the distance between a sample surface and shield's edge is less than the collimator slit width, the change of luminosity is determined by the brightness variation of the focus projection and its geometrical position with respect to sample surface and the shield. In the case functions describing spatial variations of the paraxial incident beams should be identical for any pair of spectral lines within an accuracy of a factor $g = I_{m1}/I_{m2}$, where I_{m1} and I_{m2} are the experimentally measured maximums of intensity for chosen spectral lines. Hence, dividing angle dependence of reflected intensity, for instance for Cu K_α-radiation, by angle dependence

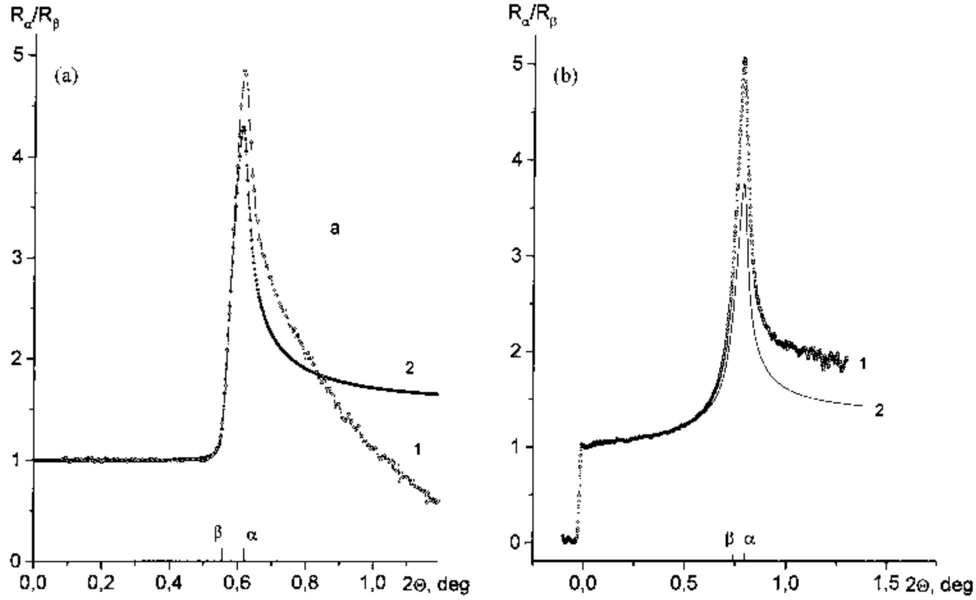

Fig. 3. Angle dependencies of relative reflectivities for monocrystal GaAs (a) and Ni film (b) (1) experiment; (2) theoretical calculation; α, β – critical angles for Cu K_α and Cu K_β lines.

of reflected intensity for the second spectral line (e.g. Cu K_β) one should receive the ratio $D(\Theta) = gR_1(\Theta)/R_2(\Theta)$ which is free from three basic sources of errors: uncontrollable variation of luminosity, current instability of an X-ray generator, uncontrollable variation of effective reflection area. Note that independence of $D(\Theta)$ from current instability gives an opportunity to regulate the X-ray flow during the angle scanning to minimize statistical errors.

Results of $R^\alpha(\Theta)/R^\beta(\Theta)$ measurements from Θ to 2Θ scan of 300 nm GaAs layer on GaAs grown by molecular beam epitaxy 900 nm and Ni film on glass are shown in Figs. 3a and b, respectively. The size L of both samples in the plane of incidence was 10 mm. It gave unfavourable measuring conditions because the size of the sample surface projection $L\sin(\Theta)$ over a wide range of angles of total external reflection was comparable with the HWHM of the incident beam. As one can see from Fig. 3b the experimental data ideally fit the results of calculation with using of Fresnel formula in the angle range from $\Theta = 0$ to the critical angle $\Theta_c(\mathrm{CuK}_\beta) = 0.278°$. In the range $\Theta_c(\mathrm{Cu\,K}_\beta) - \Theta_c(\mathrm{Cu\,K}_\alpha)$ experimental curve $D(\Theta)$ goes up over the calculated curve. It is well explained by the presence of ~ 1 nm oxide layer that should increase the absolute value of $R(\Theta)/\mathrm{d}\Theta$ near the Θ_c and, respectively, give higher values of $R^\alpha(\Theta)/R^\beta(\Theta)$ ratio. From the practical point of view, it is important that it clearly demonstrates high sensitivity of $D(\Theta)$ curves to the presence of very thin layers with X-ray optical parameters different from those in the volume. Furthermore, it gives the possibility of computer calculation of the depth dependencies of concentration using physical constants that will be a more reliable optical measurement based on optical parameters.

4. Conclusions

The proposed TCX-reflectometer offers the following advantages as compared to the traditional one-channel schemes.

1. Higher accuracy, sensitivity, and time effectiveness is provided. This is achieved due to (a) simultaneous registration of data for two

spectral lines; (b) transmission of registered spectral lines through the common X-ray path from the source to the receiving slit; (c) selection of the spectral lines at the end of the X-ray path between a receiving slit and radiation detectors.
2. The X-ray splitter–selector block based on semitransparent plates of pyrolitic graphite provides effective selection of characteristic lines and can be easily modified, if necessary, to measure simultaneously more than two spectral lines.
3. Owing to unique mode of relative measurements additional advantages are achieved: (a) one of the basic errors of the one-channel reflectometry – uncontrollable variation of the X-ray illumination of the sample surface during its rotation at small grazing angles – is excluded; (b) statistical errors of counting can be easily controlled by regulation of the X-ray tube current in the process of measurements; (c) limitations for the sample size and interval of scanned angles are considerably diminished.

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