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HAL Id: jpa-00249206
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Classification

Physics Abstracts

78.70C — 81.70C

X-ray characterization of semiconductor surfaces and interfaces

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(Received 19 November 1993, Revised 9 March 1994 and 3 June 1994, accepted 6 June 1994)

Abstract. — A method for the characterization of surfaces and interfaces on a nanometer scale by combining specular and non-specular glancing incidence X-Ray scattering is presented. The application of the method to crystalline silicon, polycrystalline silicon, and a thin gold layer on a silicon substrate is reported.

Introduction.
The surfaces and interfaces of multilayer structures play an important role in modern technology. The thickness of the layers is decreasing continuously and is for some application already in the order of nanometers. Therefore the measurements of the layer thickness and the characterization of interfaces is an important issue. The thickness and the interface properties of thin layers can be obtained by measuring specular and non-specular X-ray reflectivity [1]. The specular wave reflected from a rough layer represents the coherent component of the entire reflected wave. This component can be described using the coherent approximation of the reflection process, which only includes the point properties of the rough interface, i.e. its root mean square roughness. In order to investigate the in-plane correlation in a rough interface a measurement of the incoherent component of the reflected wave is required. The incoherent contribution to the reflected wave is represented by the non-specular (diffuse) wave.

We present a model for the coherent reflectivity and the differential cross-section of the non-specular scattered wave from a multilayer with rough interfaces. The angular distribution of the non-specular component is closely connected with the fractal properties of the rough interfaces. From comparison of the theoretical results with experimental data we obtain a set of interface parameters, namely the root mean square roughness, the lateral correlation length and the fractal dimension of the interface. We show that our method allows to characterize the roughness of surfaces and interfaces of semiconductors and metallic layers on an nanometer scale.
Theory.

The interaction of X-rays with a rough layered system is described by the Distorted-Wave-Born-Approximation (DWBA) (for details see Ref. [2]). Within this approach the differential cross-section for the scattering from the initial state \( |1\rangle \) into the final one \( |2\rangle \) is calculated. Both states are represented by the wave fields propagating in a sample with flat interfaces. This formalism allows to calculate the intensity of both specular and non-specular components of radiation reflected from a single rough interface [3]. The DWBA can be applied if the roughness profile of the surface is sufficiently «rapid», i.e. when the in-plane correlation length \( \Lambda \) is sufficiently small with respect to the X-ray coherence length.

The coherent (specular) complex reflectivity of a rough surface equals that of the flat one multiplied by an attenuation term \( \exp(-\sigma^2Q_\perp Q'_\perp) \) where \( \sigma \) denotes the root-mean-square roughness of the surface, \( Q \) and \( Q' \) are the wave vector transfers from the incident to the reflected beams in vacuum and in the sample, respectively [3, 4]. As shown in figure 1a, the components of \( Q \) parallel and normal to the interface are called \( Q_\parallel \) and \( Q_\perp \). Since \( Q'_\perp \) is purely imaginary for incidence angles below the critical angle of total reflection (absorption is neglected), no influence of the surface roughness on the coherent reflectivity can be observed in this angular range.

The differential cross-section \( d\sigma/d\Omega \) which is the probability density for the scattering of an X-ray quantum into a certain solid angle is given by [3]

\[
\left( \frac{d\sigma}{d\Omega} \right)_1 = \frac{k^4}{16 \pi^2} |1 - n^2|^2 |T_1 T_2|^2 \frac{S}{|Q_\perp|^2} \exp(-\sigma^2|Q_\perp|^2) \times
\]

\[
\times \int_S dx \exp(-iQx)[\exp(|Q_\perp|^2 C(x)) - 1] \quad (1)
\]

\( T_1, T_2 \) are the complex transmitivities of a flat surface for the incident state \( |1\rangle \) and the final state \( |2\rangle \), respectively. The refractive index is \( n \), the absolute value of the wave vector \( k \) is calculated from the wave length of the X-rays \( \lambda \) by \( k = \frac{2\pi}{\lambda} \) and \( S \) is the irradiated sample area. The in plane correlation function of the roughness profile \( C(x) \) is assumed to be given by

\[
C(x) = \sigma^2 \exp\left(-\frac{x^2}{\Lambda^2}\right). \quad (2)
\]

Fig. 1. — a) Definition of \( Q \). b) Schematic illustration of measurements of specular reflectivity \((\theta/2\theta \text{ scan})\) and nonspecular reflectivity (rocking curve) in \( Q \) space.
C(x) depends on the root mean square roughness \( \sigma \), the in-plane correlation length \( \Lambda \) and on the fractal dimension \( D = 3 - h \) of the surface. We use \( h = 1 \) for all our calculations of the diffuse scattered intensity. In reference [5] it has been shown that this is a good approximation for surfaces with a fractal dimension less than 2.5. Equation (1) can also be used if the rough surface is covered by a thin film which has a constant thickness. For this case \( T_1 \) and \( T_2 \) are replaced by the complex transmitivities of the surface film [6].

From the measured coherent (specular) and the incoherent (non-specular) X-ray reflectivity as functions of the incidence angle \( \theta_1 \) and the exit angle \( \theta_2 \) the surface parameters \( \sigma \) and \( \Lambda \) are obtained by a fitting procedure. The reflected intensity \( I_m(\theta_1, \theta_2) \) is calculated using

\[
I_m(\theta_1, \theta_2) = I_{nc} \left[ \int_{\Omega_{ap}} (\frac{d\sigma}{d\Omega})_l d\Omega + F(\theta_1, \theta_2) R_C(\theta_1) \right] + I_0
\]

where \( I_{nc} \) is the incoming intensity flux (in cps/cm\(^2\)) and \( I_0 \) is the background noise of the detector. \( \Omega_{ap} \) is the angular aperture of the detector receiving slit, \( R_C \) the coherent reflectivity, and \( F(\theta_1, \theta_2) \) is an instrumental function describing the shape of the primary beam and the receiving slit. The surface parameters (e.g. \( \sigma \) and \( \Lambda \)) are obtained by a least-square fit of the calculated intensity to the measured intensity.

Experiments.

Most X-ray reflectivity experiments reported in the literature were performed using synchrotron radiation [7-9]. In this paper we demonstrate the application of this technique using standard, laboratory sized equipment. All X-ray reflectivity curves were obtained using a Philips high resolution diffractometer equipped with a 2 kW fixed anode tube. We used Cu K\( \alpha_1 \) radiation \((k = 4.078 [\text{Å}^{-1}])\), a four crystal X-ray monochromator and a 0.2 mm wide receiving slit at a distance of 65 mm of the sample. The specular reflectivity is measured by a \( \theta/2 \theta \) scan which is schematically shown in figure 1b. In this scan \( \mathbf{Q} \) is perpendicular to the sample surface while its length is varied from 0 to \( \mathbf{Q}_{\text{max}} \). Measurements of the diffuse scattering were performed by varying the angle of incidence \( \theta_1 \) while \( \theta_1 + \theta_2 \) is fixed (rocking curve or transverse scan). In such a scan the absolute value of \( \mathbf{Q} \) is kept constant while \( \mathbf{Q} \) is rotated with respect to the surface. For glancing incidence, \( \mathbf{Q}_{\perp} \) is almost constant while \( \mathbf{Q}_{\parallel} \) is varied (Fig. 1b).

Results and discussion.

Figure 2 shows specular and nonspecular reflectivity of an (100) oriented silicon wafer. The calculated specular reflectivity (full curve) in figure 2a fits the measured (dots) well, although the diffuse contribution is not taken into account. Figure 2b shows the rocking curve of this sample at \( \mathbf{Q}_{\perp} = 0.142 \text{[Å}^{-1}] \) (dots) and a computer simulation (full line with squares). The parameters used for the calculations are given in table I. The measured curve is shown smoothed (full line with circles), too. The absence of diffuse scattered radiation around \( \mathbf{Q}_{\parallel} = 0 \) in this rocking curve supports the neglect of the diffuse scattering within the calculation of the specular reflectivity. Assuming a background noise of \( I_0 = 0.5 \) [cps] we find that at \( \mathbf{Q}_{\parallel} = 0 \) the specular reflected intensity exceeds the diffuse scattered intensity by more than 2 orders of magnitude. The intensity of diffuse scattered radiation is very close to the background noise, so only the smoothed measured curve clearly exhibits a maximum of diffuse scattering at \( \mathbf{Q}_{\parallel} = -0.002 \text{[Å}^{-1}] \). Therefore the value obtained for the in-plane correlation length \( \Lambda \) is extremely unreliable. This is expressed by a large uncertainty of this value in table I. If we ignore the smoothed measured curve and attribute all off-specular intensity to the
Fig. 2. — Silicon wafer surface: a) Measured (dots) and simulated (full curve) X-ray reflectivity. b) Measured (dots), smoothed measured (full curve with circles) and simulated (full curve with squares) X-ray rocking curve at $Q_\perp = 0.142 \text{ [Å}^{-1}]$.

Table I. — Parameters obtained from the fits shown in figures 2 and 3

<table>
<thead>
<tr>
<th>Sample</th>
<th>$\Lambda$ [Å]</th>
<th>$\sigma$ [Å]</th>
<th>$d$ [Å]</th>
<th>$\rho$ [g cm$^{-3}$]</th>
</tr>
</thead>
<tbody>
<tr>
<td>Si</td>
<td>$70 \pm 40$</td>
<td>$6.5 \pm 1$</td>
<td>n.a.</td>
<td>n.a.</td>
</tr>
<tr>
<td>Poly-Si</td>
<td>$210 \pm 50$</td>
<td>$45 \pm 5$</td>
<td>$40 \pm 30$</td>
<td>$1.6 \pm 1$</td>
</tr>
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</table>

background noise, we conclude that the in-plane correlation length $\Lambda$ is larger than approximately 3000 Å. For such large values of $\Lambda$ the diffuse scattering is basically a delta function hidden in the specular peak.

The reflectivity of a 4000 Å thick polycrystalline silicon film on a crystalline silicon substrate is shown in figure 3a. Since interference fringes are not observed it was not possible to determine the thickness of the layer. For the simulated reflectivity (dashed curve) diffuse scattering was not taken into account yielding a pronounced difference between the calculated and the measured intensity for $Q_\perp$ between 0.05 and 0.15 [Å$^{-1}$]. From the rocking curve measured at $Q_\perp = 0.071$ [Å$^{-1}$] which is shown figure 3b, it is evident that in this range diffuse scattering dominates. The simulated reflectivity including diffuse scattering at specular conditions is shown in Figure 3a, too (full curve). The parameters for the surface of the polycrystalline silicon obtained from a fit are given in table I.

Maxima of the diffuse scattering are found at $Q_\parallel \approx \pm 4 \times 10^{-4}$ [Å$^{-1}$]. These so-called «Yoneda wings» [10] are caused by maxima of $T_1$ or $T_2$ in equation (1), which appear when $\theta_1$ or $\theta_2$ is equal to the critical angle, respectively. In order to get the shape of the left Yoneda wing in figure 3b, we had to assume a thin surface layer with correlated interfaces. However, this assumption does not necessarily mean that a real surface layer exists but rather that the density profile in the surface region cannot be described by a simple Gaussian roughness. From X-ray reflectivity measurements we can not get information on the chemical composition. We can not even deduce that there exists a distinct surface layer. It is possible that the surface
roughness is extremely non-Gaussian so that a single interface model assuming a random Gaussian interface is not appropriate. In such a case the assumption of another roughness model which can be done by the introduction of an interface layer may reduce the difference between the measured and calculated intensity.

Another rocking curve from this sample, which has been taken at $Q_{\perp} = 0.043 \, [\text{Å}^{-1}]$ is plotted in figure 4. Using the same set of parameters obtained from the measurements in figure 3, we obtain good fit for the incoherently scattered intensity.

If present, interference fringes can be used to determine the layer thickness and interface roughness with high accuracy. The reflectivity of a gold layer on a silicon substrate is shown in
Fig. 5. — 1000 Å thick gold layer on silicon: a) Measured and simulated X-ray reflectivity. The calculation is shifted upwards by a factor of 10. b) Measured (dots) and simulated (full curve) X-ray rocking curve \( Q_\perp = 0.171 \, [\text{Å}^{-1}] \).

Table II. — Parameters obtained from the fits shown in figure 5.

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Layer Thickness [Å]</td>
<td>1047 ± 7</td>
</tr>
<tr>
<td>( \sigma_{\text{Surface}} ) [Å]</td>
<td>12 ± 1</td>
</tr>
<tr>
<td>( \Lambda_{\text{Surface}} ) [Å]</td>
<td>250 ± 60</td>
</tr>
<tr>
<td>( \sigma_{\text{Substrate-Layer}} ) [Å]</td>
<td>4.5 ± 2</td>
</tr>
</tbody>
</table>

Figure 5a. A rocking curve of this sample measured for \( Q_\perp = 0.171 \, [\text{Å}^{-1}] \) is given in figure 5b, showing the contribution from coherent and incoherent scattering in the measured intensity. The curve in figure 5b, which fits the experimental curve very well was calculated using the layer thickness, the roughness of the silicon-gold interface and the surface roughness which are given in table II. The oscillations found around \( Q_\parallel = 0 \) are specular interference fringes reflecting the size of the probe within the Q-space. The roughness of the Si-Au interface is found to be slightly smaller than the value obtained for pure silicon. However, within the experimental error these values are equal.

Conclusions.

We have shown that the surfaces of semiconductors and metals can be well characterized on a nanometer scale using coherently and incoherently scattered glancing incidence X-rays. The experimental results can be interpreted within the DWBA approximation. Using this approach we obtain the values for surface parameters (r.m.s. roughness, in-plane correlation length). From the interference fringes the thickness of surface layers is determined.

Acknowledgment.

This work is supported by the « Fonds zur Förderung der wissenschaftlichen Forschung in Österreich », the BCR project « NAMIX », the EC project « TEMPUS », Philips Analytical X-Ray BV. We are grateful to Dr. Noll at AMS for supplying some of the samples.
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