

Direct determination of the phase of the reflection amplitude by means of standing x-ray waves

I. A. Vartan'yants, M. V. Koval'chuk, V. G. Kon, A. M. Nikolaenko,
and I. Yu. Kharitonov

A. V. Shubnikov Institute of Crystallography, Academy of Sciences of the USSR

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A method for directly determining the phase of the reflection amplitude by means of standing x-ray waves has been implemented experimentally. The method is based on a measurement of the x-ray reflection and the photoelectron emission at large deviations from the Bragg angle. The solution of the phase problem has made it possible to unambiguously determine the structure of the real surface layers of single crystals. This new approach is illustrated by the example of a silicon crystal after ion implantation.

In any experiment which is based on the scattering of radiation by condensed matter, the measured quantity is an intensity, from which one can determine only the modulus of the scattering amplitude. In order to obtain comprehensive information about the structure of the scattering object, however, it is also necessary to know the phase of this amplitude. This problem arises in particular in the study of the structure of the surface layers of single crystals by means of x-ray diffractive scattering. In this case the depth profile of the structure of the surface layer can be found directly from the angular dependence of the amplitude and phase of the reflected wave by means of Fourier transforms. A standard x-ray diffraction experiment cannot tell us the phase of the scattering amplitude.

For this purpose, indirect methods are widely used in x-ray structural analysis. These indirect methods are based on, for example, probabilistic estimates of the phase invariants from an analysis of the experimental distributions of the moduli of the structure amplitudes for a large number of reflections.¹ In addition, recent years have seen the rapid development of direct methods for determining phases by means of multiwave diffraction,²⁻⁴ standing x-ray waves,^{5,6} etc. The efficiency of these methods is increasing thanks to progress in x-ray optics and the use of synchrotron radiation.

In this letter we report the first demonstration of the possibility of directly determining the profile of structural changes of a surface layer by means of standing x-ray waves. Specifically, the determination is based on simultaneous measurements of the reflection coefficient for x rays and the yield of x-ray photoelectrons from a small depth over the entire angular range near the Bragg peak, including both the central part of the peak and its "tails."

Structural changes in a crystal are described by two parameters: $u(z)$, which is the average displacement of atoms from the positions corresponding to an ideal crystal; and $f(z) = \exp[-W(z)]$, the static Debye-Waller factor. The latter factor incorporates a random scatter in the positions of atoms around the position corresponding to the mean displacement.

The idea underlying the method was presented in Ref. 7 and can be outlined as follows: We assume that the thickness (L) of the damaged layer in the sample satisfies the condition $L \ll L_{\text{ex}}$, where L_{ex} is the extinction length (this condition can always be satisfied by choosing a sufficiently high reflection order). In this case the reflection of x rays by the damaged layer is kinematic, and the reflection amplitude $R(\Delta\theta)$ for σ -polarized radiation is given by

$$R(\Delta\theta) = - \frac{i\pi\chi_h}{\lambda\sin\theta_B} \int_0^{\infty} dz f(z) \exp(iqz - i\varphi(z)) = |R(\Delta\theta)| \exp(i\Phi_R(\Delta\theta)). \quad (1)$$

Here $\varphi(z) = \mathbf{h}u(z)$, $q = (4\pi/\lambda)\cos\theta_B\Delta\theta$, and $\chi_h = \chi_{rh} + i\chi_{ih}$ are the Fourier components of the polarizability of the crystal, \mathbf{h} is a reciprocal-lattice vector, λ is the wavelength of the radiation, and θ_B is the Bragg angle. According to expression (1), outside the region of total reflection the amplitude $R(\Delta\theta)$ is essentially the Fourier component of the function $F(z) = f(z)\exp[-i\varphi(z)]$. This function and thus the structure of the damaged layer can be reconstructed by simply using inverse Fourier transforms. Here, however, we need to know both the modulus and the phase of $R(\Delta\theta)$.

The modulus of the amplitude $R(\Delta\theta)$ is found from the tails of the curve of the angular dependence of the reflection coefficient, from the formula

$$P_R(\Delta\theta) = K |R(\Delta\theta)|^2. \quad (2)$$

Here K is a polarization factor. In a two-crystal measurement arrangement, this factor is $K = (1 + \cos^3 2\theta_B)/(1 + \cos 2\theta_B)$. To determine the phase, we make use of the angular dependence of the secondary emission from a small emission depth, L_{yi} ($L_{yi} \ll L$), e.g., photoelectron emission. At large deviations from the Bragg angle,

this curve is described by

$$I(\Delta\theta) = 1 + P_R(\Delta\theta) + 2K|R(\Delta\theta)| \frac{|\chi_{ih}|}{\chi_{i0}} f(0) \cos[\Phi_R(\Delta\theta) + \varphi(0) - \omega], \quad (3)$$

where ω is the phase of the complex quantity χ_{ih} . By simultaneous measuring reflection curves $P_R(\Delta\theta)$, as in (2), and the secondary emission $I(\Delta\theta)$, as in (3), we can thus directly determine the modulus $|R(\Delta\theta)|$ and the phase $\Phi_R(\Delta\theta)$ of the reflection amplitude $R(\Delta\theta)$ over the entire angular range. The values of $f(0)$ and $\varphi(0)$ are found from the central parts of the $P_R(\Delta\theta)$ and $I(\Delta\theta)$ curves. Using inverse Fourier transforms, we then find the actual structure of the damaged layer.

In the present study we have illustrated this method in the example of determining the profile of distortions of the surface layer in silicon crystals after the implantation of B^+ ions (energy of 100 keV, dose of 10^{15} cm^{-2}) and a subsequent annealing ($T = 800^\circ\text{C}$, $t = 10 \text{ min}$) in an oxidizing atmosphere. In the experiments we used the (400) reflection of $\text{Cu } K\alpha$ radiation. We measured curves of the photoemission and the x-ray reflection of the samples both in the region of total reflection (Fig. 1) and at

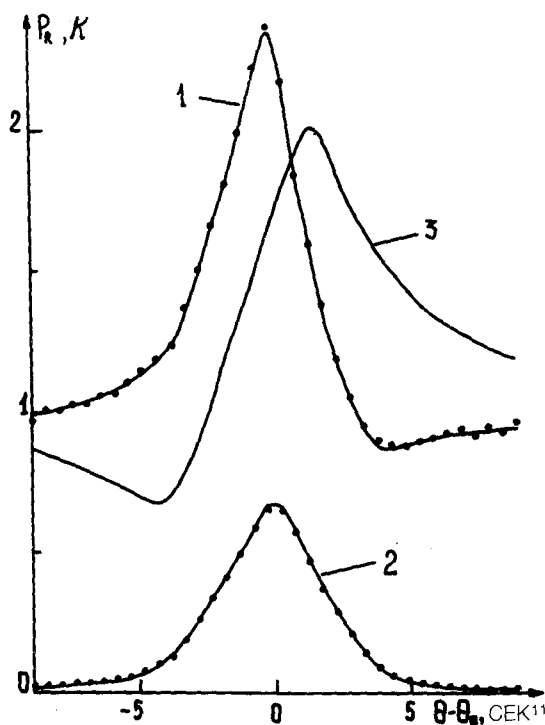


FIG. 1. 1,2—Curves of (1) the photoemission and (2) the x-ray reflection from the test sample in the region of the total reflection; 3—photoemission of an ideal silicon crystal; solid lines—calculated from the dynamic theory.

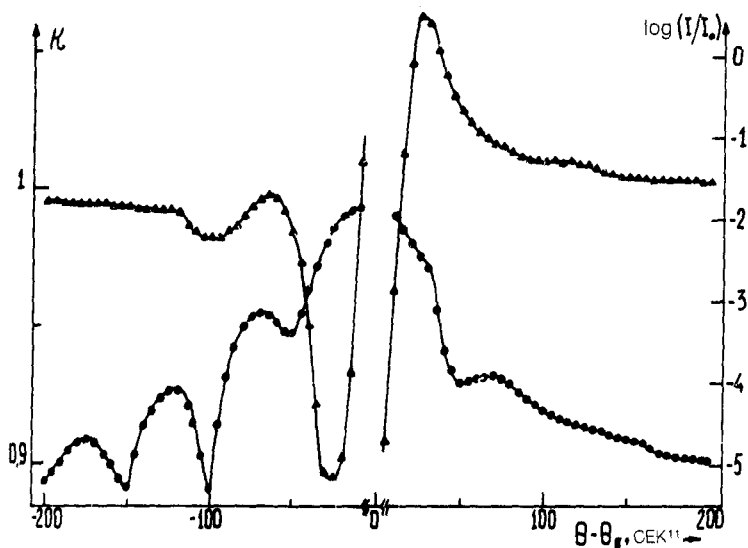


FIG. 2. Curves of (Δ) the photoemission and (\circ) the x-ray reflection of the test sample at large deviations from the Bragg angle. The x-ray-reflection curve was recorded by the method of three-crystal, x-ray diffractometry.

large deviations from the Bragg angle (Fig. 2). The photoelectron emission was measured by a gas-filled proportional counter with a resolution $\approx 15\text{--}20\%$ (Ref. 5). We detected electrons with energies from 5.9 keV to 6.4 keV, from a small emission depth ($L_{yi} \approx 1000 \text{ \AA}$).

To solve our problem, we first solved the problem of measuring a small secondary-emission useful signal (amounting to 10 counts/s on the tails of the photoemission curve), by developing an efficient buildup system⁵ which made it possible to build up the useful signal over many hours. It should also be noted that the reflection curves recorded in a standard two-crystal arrangement contain the sum of coherent and incoherent scattering channels⁸⁻¹⁰ and are thus unsuitable for theoretical analysis. The incoherent part of the intensity is made up of both diffuse scattering in the test sample (this scattering is slight in our case) and thermal diffuse scattering in the monochromator, which is at its greatest on the tails of the reflection curve. In measuring the tails of the reflection curve we accordingly used a three-crystal arrangement, which made it possible to distinguish the coherent intensity as the intensity of the main peak.^{9,10} In the photoemission measurements, however, we used a two-crystal arrangement, since the incoherent contribution from the monochromator is negligible in this case.

Figure 2 shows tails of experimental photoemission and x-ray-reflection curves. In its central part, the photoemission curve (Fig. 1) is similar to the inverted curve from an ideal crystal. It coincides with the theoretical curve with $\varphi(0) = 2.3$ and $f(0) = 0.9$. Figure 3 shows profiles of the deformation, $\Delta d(z)/d = \partial u/\partial z$, and of the Debye-Waller factor $f(z)$ found by the method described above. We see from this

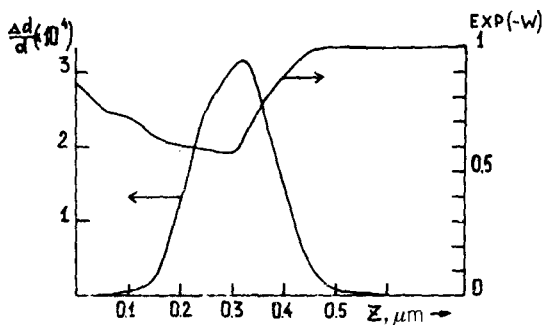


FIG. 3. Profiles of the deformation $\Delta d/d(z)$ and of the static Debye-Waller factor $f(z) = \exp(-W)$.

figure that the deformation in the damaged layer is positive and reaches a maximum at a depth $L \approx 0.3 \mu\text{m}$. This figure correlates with both the range of B^+ ions in silicon ($\approx 0.3 \mu\text{m}$) and the theory for defect formation in these crystals.¹¹ Although the calculations were carried out in the limit $L_{yi} = 0$, incorporating the actual emission depth does not alter the results, according to our analysis, and the error does not exceed 10–15%.

In summary, simultaneous measurements of the tails on the curves of the photoelectron emission and the x-ray reflection make it possible to directly solve the phase problem in a study of damaged surface layers of single crystals. In other words, it is possible to completely determine the complex reflection amplitude and thereby find the distortion profile itself.

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