Angstrom-level resolution in x-ray-diffraction study of crystal surface structure

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It is possible to analyze the structure of extremely thin surface layers of a crystal, with thicknesses on the order of a few interatomic distances. The structure of the surface of a germanium crystal under a film ($\sim 1\,\mu$m thick) of another material has been studied for the first time.

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Conventional x-ray-diffraction techniques can be used to study the structure of crystal layers at depths comparable to the extinction depth, which usually runs between 1 and 10 μm. Away from the Bragg-angle region, x rays penetrate much farther into a crystal, to a depth determined by ordinary absorption: 10–100 μm. The depth of the source of purely diffraction emission in the case of Bragg reflection falls off with increasing deviation from the Bragg angle, $\Delta\theta$. This circumstance can be exploited under certain conditions to obtain direct information about the structural quality of thin surface layers. The method of three-crystal x-ray spectrometry[1] makes it possible to work quite far from the Bragg angle, so that exceedingly thin crystal layers can be studied.

In this letter we report the use of three-crystal x-ray spectrometer to study the intensity of the main peak of germanium single crystals as a function of the angular deviation $\Delta\theta$ up to $\sim 6000''$, which is nearly 10^3 times the width of the Bragg peak. The range of values of $\Delta\theta$ over which measurements are possible was extended substantially by accumulating data with a special system using a multichannel analyzer. This extension of the measurement range makes it possible to study outer layers with thicknesses of only a few angstroms.

Some typical curves are shown in Fig. 1. Despite the large deviations from the Bragg angle—deviations which exceed the half-width of the Bragg peak by nearly three orders of magnitude—the diffraction peak undergoes no change in shape. Its broadening amounts to only 10% of the widths of the corresponding peaks at small deviations from the Bragg angle. Figure 2 shows $I(\Delta\theta)^2$ vs $\Delta\theta$, where I is the measured intensity of the peak (the area under it). In ideal crystals, without any structural damage in the surface layers, this plot should be a straight line, so that deviations from a straight line give us information about the depth of the transition layer. It can be seen from these curves that the intensity $I(\Delta\theta)^2$ changes sharply with increasing $\Delta\theta$.

To calculate the width of the transition layer we adopt the following model: The decrease in the scattering strength of the crystal is determined by the static Debye-Waller factor $e^{-W(z)}$, where the axis runs into the crystal. This factor can be approxi-

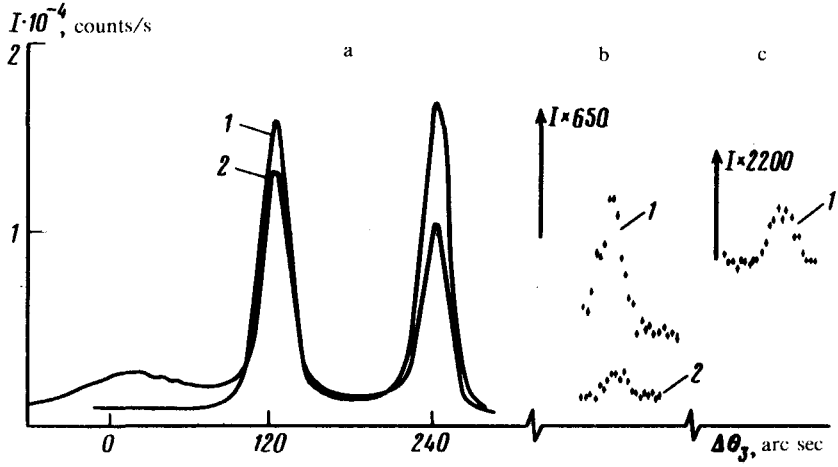

FIG. 1. Intensity of the x-ray beam reflected from the crystal vs the angular position of the analyzer crystal, $\Delta\theta_3$. 1: Germanium single crystal (the [111] reflection). 2: The same sample after the deposition of an aluminum film 1.2 μm thick. a—The deviation from the Bragg angle is $\Delta\theta = -120''$; b—$\Delta\theta = -4000''$; c—$\Delta\theta = -6000''$. Only the main peaks are shown for curves b and c.

mated by

$$e^{-W(z)} = ae^{-\kappa z} + b. \tag{1}$$

In this case we have the following simple analytic expression for the function $I(\Delta\theta)^2$ (normalized to a unit value at $\Delta\theta = 0$):

$$I(\Delta\theta)^2 = \frac{C(2+C)}{1+(\kappa/\psi)^2} + 1, \tag{2}$$

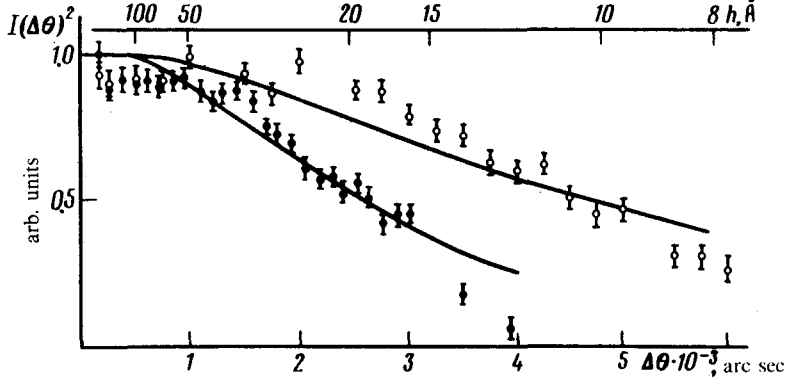

FIG. 2. The intensity $I(\Delta\theta)^2$ vs the angle $\Delta\theta$. The upper scale gives the depth of the layer being analzyed vs $\Delta\theta$. Open circles—experimental data for a germanium single crystal; filled circles—for the same sample, after deposition of an aluminum film; curves—theoretical, calculated from Eq.(2) with the parameter values giving the best fit.

where $\psi = (4\pi \cos\theta_B \Delta\theta)/\lambda, \Delta\theta$ is the angle through which the crystal is rotated, λ is the x-ray wavelength, and

$$C = a/b = e^{\Delta W} - 1, \quad \Delta W = W(\infty) - W(0).$$

The parameter $l = 1/\kappa$ is a measure of the depth of the transition layer. By varying the parameters κ and C, we can fit the theoretical curves to the experimental results.

We studied a germanium crystal which had been subjected to a chemical and mechanical polishing, followed by a deep polishing etching. We then studied the same crystal after depositing on it an aluminum film 1.2 ± 0.2 μm thick. We found that the transition layer is ~ 10 Å thick for the original crystal and thickens to ~ 20 Å for the crystal covered by a film.

The increase in thickness can be attributed to a partial penetration of aluminum into the germanium crystal. Further evidence for a degradation of the quality of the surface layers in this case comes from the appearance of a weak diffusion peak on the reflection curves.

These results thus demonstrate that it is possible to analyze the structure of extremely thin surface layers of a crystal, with thicknesses down to a few interatomic distances. The unique resolution attainable in the x-ray method has been realized experimentally. It has also been shown possible to study crystal layers covered with thick films of another material. The relative simplicity of this experimental technique raises the hope that it will find widespread practical use.

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