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Measurement of step heights on a crystal surface using synchrotron phase contrast imaging

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An experimental and theoretical study of surface step heights on a basal-faceted sapphire ribbon grown using the Stepanov's method was carried out. Obtained results were compared with atomic force microscopy data. We established that the step heights on the order of $1\,\mu$m could be determined using simple in-line phase-contrast imaging setup.

Keywords: synchrotron radiation, phase contrast, microrelief, sapphire, Stepanov's method.

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1. Introduction

Sapphire ribbons faceted by surface (0001) do not contain any steps in ideal conditions. However, if the thickness decreases slightly from the middle to the edges of the ribbon, steps are formed on its surface. As the ribbon surface deviates from the basic plane by angles of less than 1°, the structure of its surface changes. Segments of the singular facet are interrupted by steps, the density of which is low; but the vicinal facet of the ribbon becomes not smooth. For the relief surface, a minor difference of optical properties is specific, which is one of the causes for the technical drawbacks in the optical raw materials.

Micromorphology is studied by the methods of optical microscopy. In particular, Nomarski microscopy, multi-beam interferometry and phase-contrast light microscopy to reflection are very sensitive to detection of a small relief. For the substances whose surface withstands making replicas, reflective electron microscopy (REM) is used. The entire surface of the specimen may be studied by the method of scanning electron microscopy (SEM). However, the REM method is of damaging nature, and the SEM method has relatively low resolution. It should be added to this that the analysis of morphology of the large surfaces by method of atomic-force microscopy (AFM) — is a rather difficult task.

One of the relatively new methods to assess the microinhomogeneity in the crystals is the method of phase contrast imaging (PCI) in synchrotron radiation (SR) [1]. The parallel beam of X-ray radiation that passed through the specimen with heterogeneous electron density may form an image in the space behind the specimen, if the change in the phase shift causes the occurrence of interference pattern. Besides, coherence is the necessary condition to observe interference. Computer simulations calculate distribution of intensity for a certain model of an object. Adjusting the calculated curves for the profiles in the detector, the object parameters are specified [2,3]. Compared to the above methods, PCI has undoubted advantages. Contrary to the light microscopy, the PCI method is used to identify small heterogeneities in the volume of nontransparent objects regardless of their thickness, removing the limitation caused by short focus distance of the optical lens. Compared to the methods of X-ray or electron microscopy, which does not allow obtaining statistically significant information, PCI provides a researcher with quite a large area recorded in a pixel detector by specimen movement. Besides, the small size of the detector pixel determines high resolution.

Thanks to the fact that the structure of steps on the vicinal surface of the basal-faceted (BF) ribbon is simple, and the density of the steps is low, the ribbons are a convenient model object for the PCI simulation. This paper identified the patterns in the phase contrast change, the height of the microstep was assessed, and this result was compared to another independent method — AFM.

2. Experiment

SR source Pohang Light Source (PLS) in Pohang city operates at electron energy 3.0 GeV. PLS has beamlines 6 C and 9 D to obtain PCI and topographs. On beamline 6 C the insertion device is a wiggler, and a monochromator identifies a narrow peak at the specified energy from the range of 23–50 keV. Thanks to large distance from the wiggler to the specimen (35 m) and small vertical size of the source ($29\,\mu$m), the length of spatial coherence makes several dozens of microns.

The image recording system has a design of a light microscope projecting the enlarged luminescent image from the scintillation screen to the matrix of charge coupled devices (CCDs). Maximum resolution in the vertical plane is determined by the size of CCD-chip pixel, magnification, and screen quality. PC-images were obtained in the radiation with wavelength of $\lambda = 0.054$ nm. Detector PCO Edge (PCO AG, Kelheim, Germany) with pixel size $6.5 \times 6.5\,\mu\text{m}^2$ and resolution 2560×2160 of pixels recorded images enlarged with the help of the lens 20×. Scintillation screen YAG : Ce practically had no damages.

The BF-ribbon was grown from the melt using Stepanov method with the growth speed of 1 mm/min to the size of $10 \times 0.4 \times 100$ $(W \times H \times L)\,\text{mm}^3$. Disorientation of the ribbon surface relative to the basic facet made several angular minutes. The scheme of the experimental setup of the PCI method requires no other elements except for the detector, monochromator and SR source.

3. Results

Figure 1 presents PCIs of one and the same area of the sapphire ribbon recorded on the detector at various distance from the specimen. The image (*a*) ($z = 20$ cm) clearly shows the complex relief of the surface. However, at the close (*b*) distance ($z = 8$ mm) the surface relief blends with the background.

The period of PCI coherent oscillations may be assessed using the radius of the first Fresnel zone: $r_1 = (\lambda z)^{1/2} = 0.66\,\mu$m, where $\lambda = 0.054$ nm and $z = 8$ mm. The radius of irregularity in the lower part of Figure 1, *b* is equal to $6.5\,\mu$m. And it is by an order more r_1. The period of oscillations in this case is smaller than the resolution of the available solid-state detectors. As z increases from the millimeter to the centimeter range of distances at the edges of irregularity, the period of intensity oscillations increases. And the small oscillations still blend to each other, only the first ones remain, forming a wide dark rim around the light middle. Patterns of micropore contrast formation were studied, for example, in papers [4,5]. However, the phase contrast of microscopic steps on the surface of crystals was not studied previously.

The objects of observation selected are the features located in the central region of the specimen section in Figure 1, *a*. Figures *1* and *2* indicate accordingly the round and wavy boundaries of the specific black and white contrast. In the second case we deal with the image similar to other images of identical steps, which follow the shape of the crystallization front. The nature of figure *1* is possibly related to the local superheating of the surface caused by hitting of the hot molybdenum particle into this point at a certain distance from the crystallization front.

Direct measurement of round boundary relief *1* was made using atomic-force microscope „Integra Aura" (NT-MDT, Zelenograd, Moscow). Figure 2 presents the relief of the surface (*a*) and the profile (*b*) of the figure *1*, which turned out to be a cavity. Note that PCI defines it immediately by

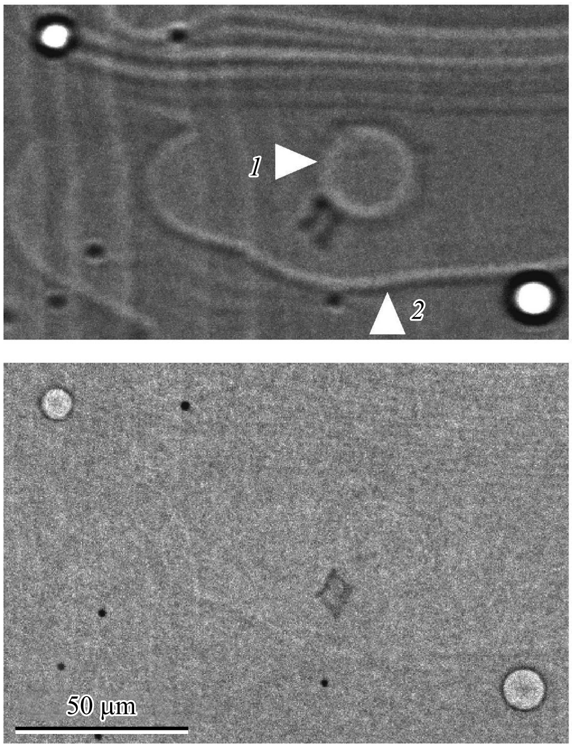

Figure 1. Phase-contrast images of sapphire ribbon surface obtained at various distances, specimen–detector z: *a*) $z = 20$ cm, *b*) $z = 8$ mm.

contrast orientation. From the curve it follows that the edges of the cavity have the appearance of slightly inclined steps with height of $1.24\,\mu$m.

In the PCI method the step looks like a black and white band. The step height only manifests itself through contrast. For proper comparison of the experimental PCI with the calculation, it is necessary to process with caution the experimental profile of intensity. However, parasitic noise prevents generation of strong useful signals. Signal-to-noise ratio for boundary *2* was higher than for boundary *1*. Experimental images were recorded on the CCD-matrix and saved in TIFF, 16 bit format with the range of values from 0 to 65536. TIFF-files were converted into the matrix of numbers, from which the fragments intended for modeling were cut. Measurement of intensity profiles along the lines perpendicular to the selected contrast band was made on the pictures obtained from matrices again.

Experimental distribution of intensity across boundary *2* in Figure 1, *a* is shown with a black line in Figure 3. One can see that deviations of the minimum and maximum from the average value are approximately equal. This is the way it should be based on PCI theory from the step. It can be shown that the relative intensity on the step image in the parallel and monochromatic radiation is described by function

$$I/I_0 = 1 + \varphi[S(x/x_0) - C(x/x_0)],$$
$$\varphi = K\delta t, \quad x_0 = (\lambda z/2)^{1/2}. \quad (1)$$

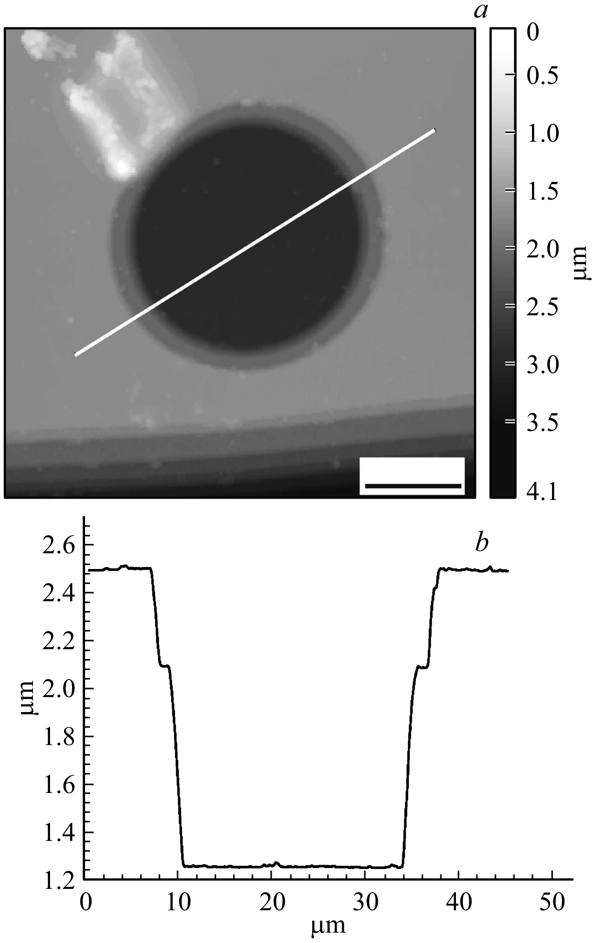

Figure 2. *a*) Image of sapphire ribbon surface by AFM method. Scale $10\,\mu$m. *b*) Profile of step thickness in the same place of the specimen.

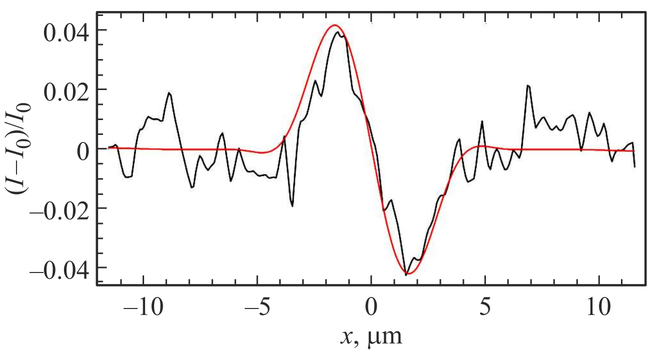

Figure 3. Profiles for distribution of intensity across phase-contrast image of microstep: experimental (black color) and theoretical (red color).

Here $S(x)$ and $C(x)$ — sine- and cosine-integrals of Fresnel, z — distance from the object to the detector, φ — wave phase shift on the step. The curve of theoretical contrast has multiple oscillations. However, the experimental image is not quite clear. It does not show fine details visible on calculated curves. This fact makes to assume that apart from the resolution of the detector and the size of the source, it is necessary to take into account other factors of image distortion, for example, vibrations. In process of every experiment it is necessary to study the degree of radiation coherence at model objects. However, a specific user may not increase this degree in any way.

To account for all factors in theoretical simulations, the intensity distribution was convolved with Gaussian function of the given half-width, which was known from the analysis of multiple other experimental data at this beamline. Gaussian half-width is equal to $3\,\mu$m. In this case the theoretical contrast is equal to 0.2φ. Comparing to the experiment, we get that $\varphi = 0.2$. At $E = 23\,$keV the value of the refractive index decrement for sapphire is $\delta = 1.532 \cdot 10^{-6}$ [6]. As a result, we identify the step height $t = 1.12\,\mu$m from the simple estimate. Taking into account the fact that the surface of the ribbon consists of the steps formed by smooth sections of singular facet with width of dozens of microns [7], the height of the adjacent steps in Figure 1, *a* may be assumed to be nearly equal.

4. Conclusion

Therefore, the studied section of the ribbon contains microsteps with the height of order of $1\,\mu$m, which corresponds to the result of the AFM method. The AFM method works with the support of the rather complex equipment. At the same time we managed to determine that small height using the extremely simple PCI method, i.e. in transmission geometry. The stepped structure of the growth surface is specific not only for sapphire ribbons, but for other materials as well. The described study discloses a new aspect of PCI use for quantitative analysis of morphology of almost perfect crystals.

Conflict of interest

The authors declare that they have no conflict of interest.

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