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Study of the three-dimensional distribution of defects in crystals by synchrotron radiation diffraction tomography

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Abstract

The three-dimensional (3D) distribution of defects was investigated in several crystals by synchrotron radiation diffraction tomography. This experimental procedure combines white-beam synchrotron radiation section topography with electronic image recording and computer data processing. A scan involving many section topographs is performed with the same Bragg reflection, resulting in a stack of section topographs containing the 3D information.

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1. Introduction

X-ray topography is a widely used method for the visualization of crystal defects in single crystals [1,2]. In projection topography, a two-dimensional (2D) image of the three-dimensional (3D) sample is produced; thus, good spatial resolution (μm range) is obtained along the crystal plate surface, whereas depth information is lost. By restricting the incident beam to width around 10–20 μm, i.e., in section topography [3], good resolution is obtained along the crystal depth, but at the cost of a drastic reduction of the investigated volume.

Several approaches were proposed to solve this problem. Manual tracing of the images obtained with adjacent slit positions [4,5,10] yielded the 3D geometry of a small part of the crystal. The use of different azimuthal setting or different reflections on projection topographs gave rise to the method of stereo-pairs [6]. More recently, ''topo-tomography'' was shown to produce a 3D image of defects in crystals [7,8]. This approach consists in recording many images in the same Bragg reflection, while turning the sample around the diffraction vector. The usual computed algorithm is then used to reconstruct the 3D image.

Another possibility for 3D reconstruction of the defect distribution in a crystal is diffraction tomography: it consists of collecting a complete stack of section topographs covering the whole volume of the crystal. Neglecting contrast mechanisms other than the direct one, a plausible approximation when the absorption factor μt, where t denotes the crystal thickness and μ the linear absorption coefficient, is not much larger than unity, each point in the volume of the crystal corresponds to a definite point on one of the section topographs. The aim of the present work is to test the capabilities of this method when renewed, compared with Lang's original pencil-and-paper approach of 1957 [4], through the use of modern tools, viz. synchrotron radiation, electronic recording of the images and their digital processing.

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2. Experimental method

The experiments were carried out at the ID19 beamline at the ESRF. The white beam of synchrotron radiation from a wiggler was incident through a narrow slit, 10 μm wide, on the single-crystal sample set in transmission geometry. The reflection was adjusted so that its diffraction plane was horizontal, and the diffracted beam was centered on a FReLoN camera [9] set to 6 μm pixel size. The exposure time for each section topograph was about 10 s with a source current of 90 mA. The Bragg angle was about 10°. The sample was moved in a horizontal direction close to the scattering vector, with a step equal to the width of the slit. A section topograph was recorded by the detector at each step.

A scan resulted in a set of section topographs. All the images were first processed to normalize them to the same machine current in the storage ring, eliminate glitches, adjust the brightness and make elementary geometrical corrections. Two types of presentations were made of the resulting set. The first is an animation, where all the 2D sections are viewed in an almost continuous succession like a video. The second type of presentation is a reconstruction of the 3D image of the crystal. The reconstruction is performed by stacking the section topographs, like a deck of cards, in the computer memory. By processing this virtual 3D image it is possible to measure the size of the visible defects, to rotate the image in order to view it from different angles, and to reconstruct arbitrary sections of the image.

3. Experimental results

Several crystals, featuring different kinds of defect structure, were investigated. All of them were previously studied and their defect structure was basically known. They are:

(a) A 1 mm thick plate of quartz with (1 1 −2 0) orientation: the reflection with the main contribution was 2 −2 0, at a photon energy of about 28 keV and μt of about 0.2.
(b) A 0.5 mm thick plate of artificially grown diamond with (0 0 1) orientation. The plate was set nearly perpendicular to the beam, the reflection with the main contribution was 2 2 0, at a photon energy of about 28 keV and μt of about 0.01

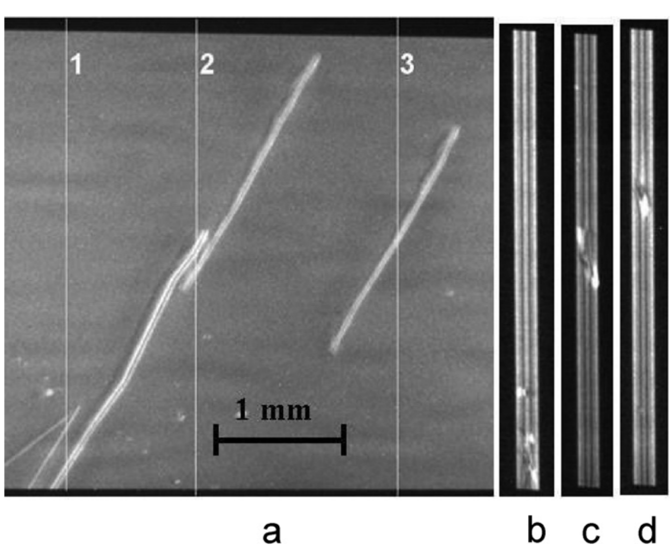

Fig. 1. Projection topograph of the quartz crystal (a) and three section topographs, taken at the positions marked on the projection topograph by vertical lines (b–d).

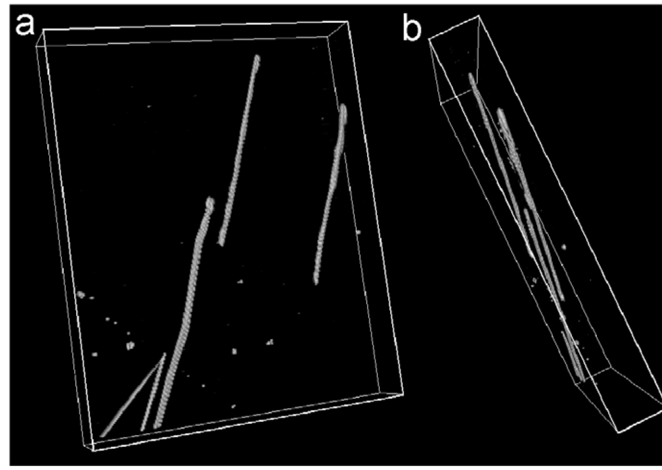

Fig. 2. 3D reconstruction of the quartz crystal viewed from the front (a) and from the side (b).

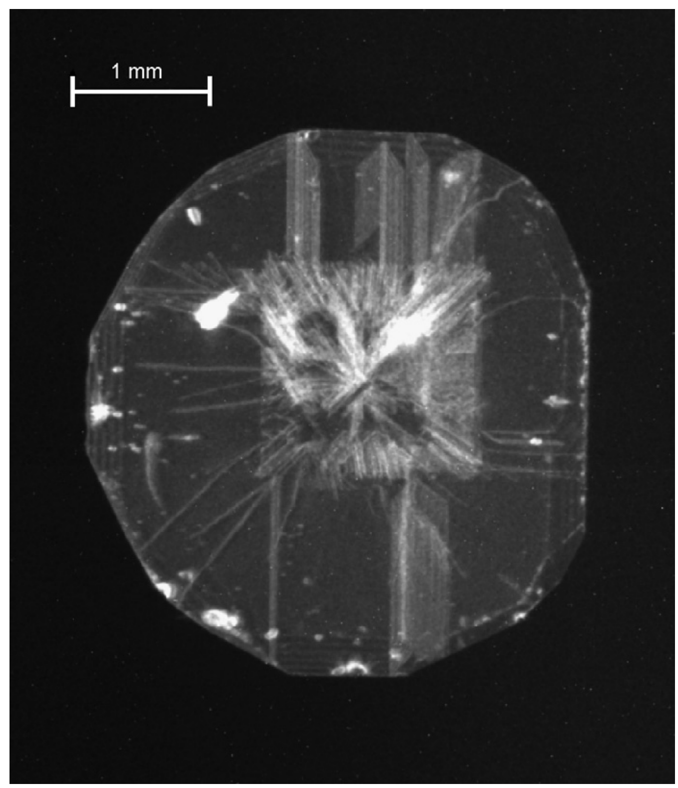

Fig. 3. Projection topograph of the diamond crystal.