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NEUTRON DIFFRACTION TOMOGRAPHY:
A UNIQUE, 3D INSPECTION TECHNIQUE FOR
CRYSTALS USING AN INTENSIFIER TV SYSTEM*

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Abstract

This paper describes the application of phosphor-intensifier-TV techniques to neutron topography and tomography of crystals. The older, analogous x-ray topography using wavelengths $\approx 1.5\text{\AA}$ is widely used for surface inspection. However, the crystal must actually be cut in order to see diffraction anomalies beneath the surface. Because $1.5\text{-}\text{\AA}$ thermal neutrons are highly penetrating, much larger and thicker specimens can be used. Also, since neutrons have magnetic moments, they are diffracted by magnetic structures within crystals. In neutron volume topography, the entire crystal or a large part of it is irradiated, and the images obtained are superimposed reflections from the total volume. In neutron tomography (or section topography), a collimated beam irradiates a slice (0.5-10 mm) of the crystal. The diffracted image is a tomogram from this part only. A series of tomograms covering the crystal can be taken as the specimen is translated in steps across the narrow beam. Grains, voids, twinning, and other defects from regions down to 1 mm in size can be observed and isolated. Although at present poorer in resolution than the original neutron and film methods, the TV techniques are much faster and, in some cases, permit real-time viewing. Two camera systems are described: a counting camera having a 150 mm $^6\text{Li-ZnS}$ screen for low-intensity reflections which are integrated in a digital memory, and

a 300-mm system using analog image storage. Topographs and tomograms of several crystals ranging in size from 4 mm to 80 mm are shown.

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Introduction

The investigation of the surfaces of crystals by x-ray diffraction (x-ray topography) is a well-established technique in solid state physics and metallurgy.¹ Film methods, with and without intensifiers, and television techniques have been used.^{2,3} The latter techniques make possible real-time observations, even of crystal growth. The observations are limited to surfaces or thin sections of metals, semiconductors, etc., because of the small depth of penetration of the low-energy x rays ($\lambda \approx 1 \text{ \AA}$) which must be employed for diffraction.

Neutrons of comparable wavelength (thermal neutrons), on the other hand, are highly penetrating for most materials throughout the periodic table and have been used for many years for neutron radiography, particularly for heavy elements as well as for hydrogenous materials. Film and television techniques have been used.⁴ When applied to topography, neutron penetration makes possible observations of volume diffraction from crystals several centimeters thick. Also, since the neutrons have magnetic moments, they are reflected from magnetic domains in materials and provide the only means for viewing these structures below the surface. Nuclear reactors provide intense beams of thermal neutrons which, when made monochromatic by Bragg reflection from a single crystal, are used to illuminate the experimental specimen.

The experimental setup is sketched in Fig. 1, along with the main features of the detection system. This figure is about ten years old and illustrates the phosphor screen-mirror lens system we envisaged for the neutron counting camera we were developing for collecting neutron diffraction data for crystal structure determinations.^{5,6} This mirror-lens system, invented by Bouwers in the Netherlands and Maksutov in Russia, was being successfully used in medical x-radiography to give 12-in. diameter imaging areas. Tests conducted with members of the Oude Delft Company (Aerojet-Delft) indicated that the operation as a neutron counter sensitive to single events would be marginal with the mirror-lens coupled intensifiers and camera tubes available at that time. We abandoned the idea of the Bouwers system.

We chose an intensifier (150-mm-diameter fiber optic input with a 25-mm-diameter fiber optic output) coupled to a secondary electron conduction camera tube shown in Fig. 2.⁷ The neutron phosphor of $^6\text{Li-ZnS}$ is coupled directly to the fiber optic input. Sufficient light is produced so that single events are well above the camera noise. The counting efficiency is approximately 20% for 0.5-mm-thick screen. Since the distribution of scintillation pulse heights from the multicrystalline phosphor is broad, some events fall below the discriminator threshold and are not counted. The coordinates of each event counted are converted to pulse amplitudes, and the count is stored in the memory of a 20K channel (200×100) two-parameter pulse height analyzer. The diffraction pattern consisting of many discrete spots or, in topography, the image of a single spot is built up in the memory of the analyzer or on the screen of a storage scope.

For crystals larger than 1 mm in minimum dimension, the spots show size, shape, and contrast related to the diffraction efficiency throughout the crystal when the whole crystal is in the beam. These images are volume topographs which reveal macroscopic internal features about the crystal, such as large grains, voids, etc. An example of a volume topograph of a sucrose crystal is shown in Fig. 3.⁶ A misoriented region was found as the crystal was rotated through 1.7°. Figure 4 shows a topograph from a large ferrite crystal taken with the crystal still in the platinum crucible. In a matter of seconds, the quality of the crystal inside the crucible was determined by noting whether odd-shaped, small reflections flashed up on the monitor at angles different from the main reflections as the crystal was rotated in the beam.

Although we had used the technique of locating certain features in crystals by illuminating isolated regions and had achieved a crude tomography,⁷ it was Doi et al.^{9,10} and Schlenker et al.¹¹ who, with film techniques for detection, first used narrow-slit collimators to virtually slice the crystal with the neutron beam. These film methods, which sometimes require hours of exposure, can produce higher-resolution (0.1 mm) images than the TV techniques (~0.5 mm), but they are not capable of giving real-time information.

The experimental arrangement for the diffraction tomography is shown in Fig. 5. The crystal is translated in steps across the beam while the Bragg angle is maintained. At each position a tomogram is obtained in counting times of two to ten minutes. A series of tomograms for a 13 mm × 43 mm lead crystal are shown in Fig. 6.⁸

The figures shown thus far were taken with the counting camera and the digital storage in the 200 × 100 channel analyzer.

Recently we obtained an x-ray imaging system based on the optical scheme shown in Fig. 1. This system is the Oude-Delft Delcalix mirror-lens system with a 300-mm-diameter screen, an image intensifier, and a lens-coupled isocon camera tube. We replaced the x-ray phosphor with a 300-mm-diameter neutron phosphor^{12,13} and have begun to use it for tomography. The diffraction images are observed and photographed directly on the monitor or after integration on the target of a PEP-400 storage tube.

Figure 7 shows seven 6-mm slices spaced 1 cm apart of a 30 mm × 80 mm metal crystal. The spots are end views of pencil-like grains which extend the length of the sample. Figure 8 shows another view of these reflections which is a volume topograph of neutrons reflected from the whole crystal and projected onto the plane of the detector. Figure 9 shows the use of the collimating slits to isolate two grains within the crystal. The top picture is a topograph of the whole crystal. By placing the slits in front of the crystal and moving the crystal across the beam, the region causing the two reflections shown in the bottom picture was isolated. In Fig. 10 a reflection from a 20 mm × 20 mm portion of another metal sample showed the dark spot seen in the bottom picture. When the crystal was rotated 10°, the reflection shown in the upper picture appeared. By "dodging" the sample with a cadmium wand while watching the monitor, the upper reflection was eliminated, and therefore the region from which it arose was located.

Summary

The technique of neutron diffraction tomography (or neutron section topography) has been described. Both a counting camera and an integrating

camera were used with thermal neutrons from the Oak Ridge Research Reactor to demonstrate in real time or with short exposures of 0.5 to 100 s the inspection of crystals for voids, misoriented regions, and grains. We believe that the technique will be valuable for inspection of monocrystalline objects being developed for high strength, such as high speed rotors or turbine blades, or for other crystals used in electronics, such as ferrites. Because the technique can be real-time, and since the neutrons are highly penetrating, the observation of crystal growth within a high temperature furnace is a distinct possibility.

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Figures

Fig. 1. Original ORNL conception of a neutron counting camera.

Fig. 2. Block diagram of a 150-mm-diameter intensifier camera with directly coupled phosphor and fiber optic coupled camera tube. Coordinates of the neutron scintillations are determined by time-to-pulse-height conversion.

Fig. 3. Topographs of a sucrose crystal revealing a misoriented region. Several display modes for the digital data are shown.

Fig. 4. Topograph from a large ferrite crystal taken with the crystal still in the crucible.

Fig. 5. Experimental arrangement for tomography.

Fig. 6. Series of tomograms taken every 0.8 mm of a 13 mm x 45 mm lead crystal.

Fig. 7. Tomograms of a 30 mm x 80 mm metal crystal, showing ends of pencil-like grains which extend the length of the specimen.

Fig. 8. Volume topograph of the same crystal as Fig. 7.

Fig. 9. Illustration of tomographic technique to isolate individual grains. Top: topograph of whole sample. Bottom: reflection from a region which was isolated with a cadmium slit.

Fig. 10. Identification of region in a 5-mm-thick crystal by "dodging" with a cadmium wand to eliminate the small reflection (top) which "fits" the hole in the bottom reflection. Difference in reflection angle was 4° .

FIG 1

Fig. 2 Block diagram of system.

0.0°

0.2°

0.35°

0.69°

1.2°

1.5°

1.7°

TOPOGRAPHY VERSUS ROTATION(T20)

BOTTOM

HORIZONTAL SECTIONS THROUGH PEAK (021)

TOP

BOTTOM
INTENSITY CONTOUR (021)

ISOMETRIC VIEW (021)

INTENSITY PROFILES (021)
HORIZONTAL UPPER
VERTICAL LOWER

Fig. 3. Topographs of a 4 x 4 x 4 mm sucrose crystal, showing misoriented portion (dark segment within round image).

Fig. 4 Reflection of part of a large ferrite crystal taken in crucible in which it was grown. Small reflections are from platinum crystallites formed during heating. Platinum crucible is shown in inset.

Fig. 5. Experimental set up for tomography.

Fig. 6. Diffraction tomographs of lead crystal taken at 0.8 mm intervals with 2-min exposure and 0.4-mm slits.

Fig.7.

FIG. 8.

FIG. 9

FIG 10.