ELECTRON-OPTICAL REGISTRATION OF X-RAY DIFFRACTION

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ELECTRON-OPTICAL REGISTRATION OF X-RAY DIFFRACTION

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An important element of any X-ray structural study is the effective registration of the X-ray diffraction pattern. The registration methods now in use—photographic and ionization—have been brought to a high degree of perfection, and their further development is unlikely to improve their capabilities in any fundamental way. A serious shortcoming of these methods is the long duration of the registration process. In a number of cases, registration of the diffraction pattern takes many tens of hours. Even when the specimen under study gives a bright diffraction pattern and highly efficient ionization detectors are used, the duration of registration of a considerable part of the diffraction pattern is measured in minutes.

At present no way is apparent to greatly reduce the registration time by further increasing the brightness of X-ray sources. Sharp-focus X-ray tubes with a rotating anode have already been created, with brightness close to the limiting value. Pulsed X-ray sources also have their own substantial limitations. Thus, the creation of new methods for registering an X-ray diffraction pattern is an important practical task.

New possibilities for registration have appeared in connection with the development of electron-optical converters (EOCs) (1-3). The idea of the method is very simple—the amplification of the light image of X-ray diffraction obtained on a fluorescent screen. Data on the practical application of EOCs for registering X-ray diffraction patterns have been published in (4).

We assembled an installation of this kind. Its schematic is shown in Fig. 1. A collimated beam of X-rays falls on the specimen 3. The diffracted beams fall on the fluorescent screen 5. The image obtained on this screen is projected by the optical system 6 onto the photocathode of the electron-optical converter 7. The amplified image of the diffraction pattern is photographed from the output screen of the EOC by an ordinary camera 8. The source of X-radiation 1 was a URS-50I unit with a BSV-3 tube (with a molybdenum anode). The collimator was a capillary made of lead glass, 0.3 mm in diameter and 15 mm long. The specimen–film distance was 25–30 mm. The zinc sulfide-based fluorescent screen emitted in the green region of the spectrum. The optical system that transferred the image from the fluorescent screen to the EOC screen consisted of two Jupiter-3 lenses, with an aperture ratio of 1:1.5 and a focal length of 50 mm. The system captured approximately 3% of the light, had a resolution better than 25 lines per mm, and introduced no noticeable spatial distortions. A disadvantage of this optical system was its small field of view. The system correctly transmitted the intensity of the object' s luminescence only within a circle of radius 8-10 mm. The total resolution

Fig. 2. Laue pattern of a single crystal of $Pb(NO_3)_2$. Exposures: *a* —1250 sec.; *b* —10 sec.

Fig. 3. Debye pattern of KCl powder. Exposures: *a* —1200 sec.; *b* —5 sec.

Fig. 4. X-ray photograph of polyethylene (oriented film). Exposures: *a* —900 sec.; *b* —5 sec.

the entire system was limited by the poor resolution of the fluorescent screen. The system practically did not resolve two reflections falling on the screen at a distance of 0.7-1 mm from one another. Photography from the output screen of the EOC onto 36-mm film with a sensitivity of about 400 GOST units was carried out from a distance of 25 cm. The efficiency of light collection in this case did not exceed 0.1%. A considerably higher efficiency (and, correspondingly, shorter exposures) can be obtained by using fiber optics.

Radiographs recorded with the aid of the EOC (Figs. 2b, 3b, 4b) were compared with radiographs recorded by the ordinary method (Figs. 2a, 3a, 4a), for which purpose the X-ray film was placed in the position of the fluorescent screen (5 in Fig. 1). The exposure times were selected so that the two methods produced negatives of equal density. The reduction in exposure estimated in this way reached 120-240 times. Radiographs photographed from the EOC were distinguished by poor resolution (reflections separated by an angular distance of ~2° from one another were not resolved). The small size of the field of view of the optical system leads to incorrect transmission of the relative intensities of the reflections (or rings in Debyegrams) (a sharp decrease in intensity with distance from the central beam). Measurement of the coordinates of the reflections showed the presence of geometrical distortions in photographs from the EOC, and the error in measuring the coordinates of a reflection from these photographs in one case reached 12%. In other cases the error did not exceed 5%. The principal cause of these distortions is apparently anisotropic deformation of the photographic film during development. When radiographs are recorded with the aid of the EOC, there is also a certain reduction in contrast (this is seen in Fig. 4).

Fig. 1. Diagram of the setup for recording an X-ray diffraction pattern with the aid of an EOC. 1 —X-ray tube, 2 —capillary made of lead glass, 3 —specimen on a rotating holder, 4 —trap for the primary beam, 5 —fluorescent screen, 6 —optical system for image transfer, 7 —electron-optical converter, 8 —camera

Fig. 1. Diagram of the setup for recording an X-ray diffraction pattern with the aid of an EOC.

Figure 1: Fig. 1. Diagram of the setup for recording an X-ray diffraction pattern with the aid of an EOC.

The radiographs shown in Figs. 2–4 are clearly observed visually on the output screen of the EOC and can be projected in the usual way onto any external screen with magnification by several times. This makes it possible within a few minutes to set the specimen in the required position, determine the presence of texture and its axis, assess the quality of the crystal, grain sizes, etc. It is also possible to observe the kinetics of structural transitions in solids with characteristic times on the order of seconds.

In the present version, the setup does not make it possible to accelerate the recording of weak radiographs, for which the ordinary photographic method requires times on the order of several hours (or more). In these cases the illumination produced on the photocathode proves to be less than its background illumination.

The spatial resolution of the apparatus can be sharply improved by using a thinner fluorescent screen, and the field of view can be increased to the required dimensions by using high-aperture objectives with focal lengths of $\sim$ 100 mm. A significant increase in the brightness of the image on the photocathode of the electro-optical converter can be obtained by the method described in work ([4]), and by using x-ray tubes with a higher specific load on the focus.

The method cannot yet compete with the photographic method and, still less, with the ionization method in the accuracy with which the geometry and intensity of the diffraction pattern are determined. There exists, however, a broad class of problems whose solution requires rapid recording of the diffraction pattern, for which a relatively low accuracy in determining the positions of reflections and only a qualitative determination of their intensities are sufficient. In this case electro-optical recording can already now be very useful.

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