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A New High Resolution Small-Angle X-Ray Camera

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A novel small-angle x-ray camera, utilizing the Borrmann effect in the 220 Laue reflection from germanium to form the incident beam, has been designed and tested. A test pattern of amorphous carbon showed angular resolution of 0.8 milliradian. In principle, an improvement of resolution by an order of magnitude is possible.

Introduction

A thin single crystal diffracting in the Laue position astigmatically images the source at a distance from the crystal equal to that of the source from the crystal. [2]

The focusing is not dependent on the spectral purity of the source to a first approximation. If the source is made small (i.e., a microfocus tube) and the optical path length made large, conditions obtain which are of interest for small-angle scattering; the scatterer is placed close to the crystal between it and the detector and a beam stop employed at the focus. Since crudely monochromatized radiation suffices in most small-angle scattering work, the lack of wavelength discrimination in the above arrangement may be exploited to gain intensity.

Realization of such a scattering camera is, of course, crucially dependent on the perfection of the crystal. The problems of preparing a suitably thin ($\mu_l t < 1$) crystal and subsequently mounting it in some strain-free fashion appear somewhat formidable. The requirement that the crystal be thin may be avoided by employing a crystal of sufficiently high perfection that it exhibits a strong Borrmann effect. Such crystals have recently become readily available and have been used in the device described below. The intensity loss in the crystal used ($\mu_l t \approx 40$) is, however, quite appreciable compared to the thin crystal case so that, when intensity requirements are severe, efforts toward realization of the thin crystal case should be considered.

Description of Apparatus

The collimating crystal used was of germanium [3] approximately 20 × 5 mm in aspect and 1 mm thick. It was cut according to figure 1 for Laue diffraction from the 220 planes.

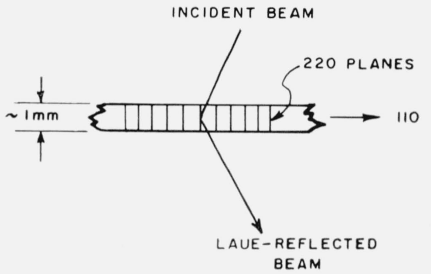

FIGURE 1. *Orientation of* Ge *crystal.*

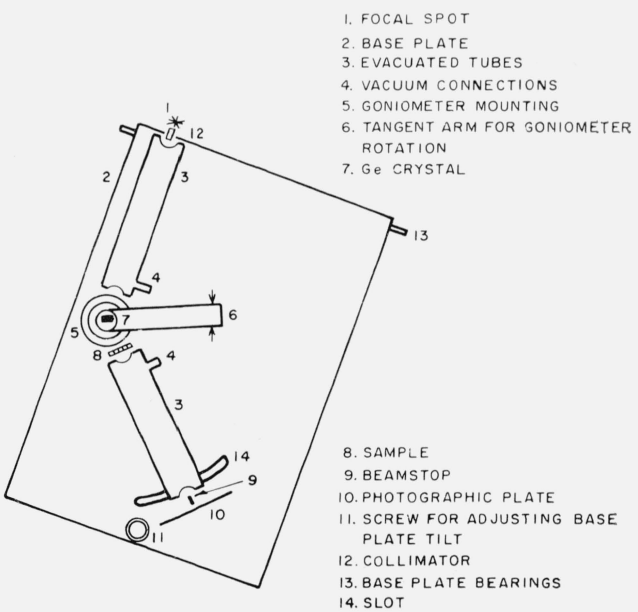

FIGURE 2. *Schematic diagram of apparatus.*

The camera is shown schematically in figure 2. After it left the focal spot (1), the beam was collimated by a lead tube (12) whose aperture was just sufficient to permit illumination of the entire crystal at a distance of 50 cm from the focal spot. The beam entered an evacuated tube (3) whose ends were fitted with Mylar windows (1.9 × 1.3 cm). The germanium crystal (7) was cemented to a brass

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[2] A. Guinier and J. Tennevin, Acta Cryst. **2**, 133 (1949).
[3] The crystal was furnished by H. Cole of International Business Machines, Thomas J. Watson Research Center, Yorktown Heights, N.Y. The authors are most grateful to Dr. Cole for his assistance.

plate which had an aperture of 1.5 × 0.5 cm. The latter was provided with a pin by which it was held in a goniometer head of the type used in single crystal diffractometry. The goniometer head was supported by a closely fitted dry bearing (5) whose angular position could be sensitively adjusted by means of a tangent arm (6). The sample (8) was held in an apertured brass plate with Mylar films on both sides between which the sample was contained. The sample holder was supported by a second evacuated tube (3) at the exit window of which was placed the primary beam stop (9). The beam stop was a piece of clock-spring mounted edgewise to the beam. Its dimensions were 0.05 × 0.6 cm. It was mounted in a fashion which permitted fine rotation about the beam axis and micrometer driven translation in the plane of dispersion of the instrument. The photographic plate (10) was placed close to the beam stop in the orientation shown.

The components were all supported by adjustable riders mounted on commercial optical bench sections, with the exception of the collimator and goniometer head. The entire apparatus rested on ¾-in. aluminum jigplate which could be pivoted about a horizontal bearing (13) on the x-ray unit by the screw (11) in order to adjust the camera to a 6° takeoff angle.

The front section of the optical bench, and the goniometer bearing, were rigidly bolted to the base plate after alinement with the x-ray beam. The back section of the optical bench, which could be pivoted around the goniometer bearing, rode in a slot (14) allowing adjustment of the angle made with the front section within a 10-deg range about 135°.

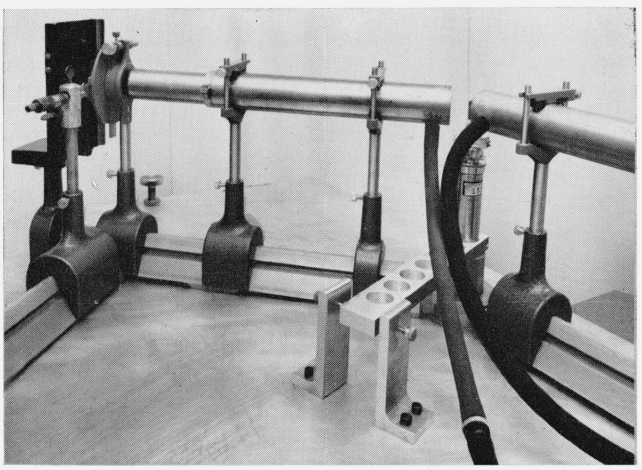

FIGURE 3. *Photograph of camera.*

Figures 3 and 4 show photographs of the camera and goniometer head mounting respectively.

A Jarrell-Ash Microfocus x-ray unit with the electron gun furnishing a nominal 100 μ spot focus was used as the source of CuKα radiation.

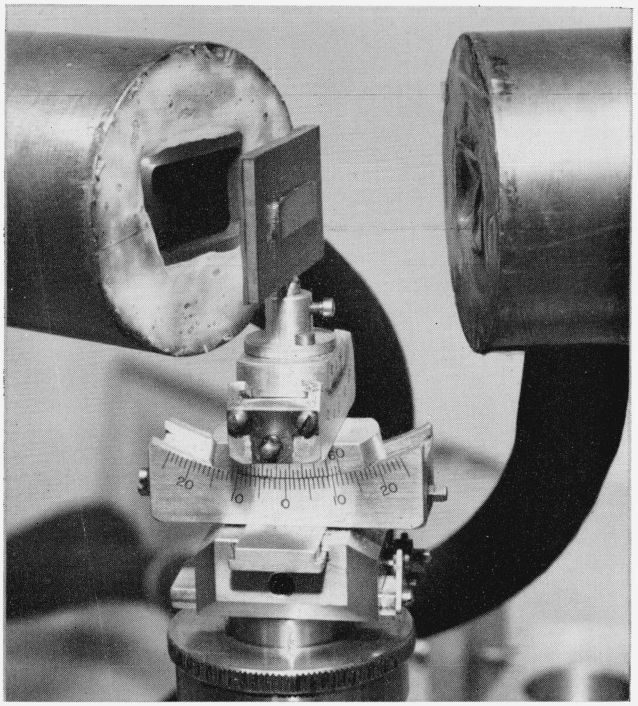

FIGURE 4. *Photograph of goniometer head mounting.*

Adjustment and Operation of the Camera

All components were alined with a proportional counter in place of the photographic plate. Once the 220 reflection was found, a series of 1-min exposures (Ilford G film) were made with the crystal at slightly different angles to allow reflection by its different parts. No significant differences were observed. A photometer trace of one of these exposures is shown in figure 5.

A photographic plate placed close behind the crystal was exposed. The components of the α doublet were well resolved and, as expected from dispersion considerations, separated by approximately 0.6 mm. With this pattern roughly centered in the crystal aperture, the β components and remote continuum effects were automatically excluded. Should a situation arise in which the α_1, α_2 wavelength difference were troublesome, α_2 could easily be rejected by further restricting the horizontal aperture.

The beam stop was inserted and adjusted to give the minimum reading with proportional counter and rate meter. Final adjustment was made with the aid of photographs.

Sample Run

A sample of "lamp black", amorphous carbon, was used because of its known scattering ability at small angles. The sample was exposed under the following conditions:

Exposure time—6 hr at 50 kV, 1.4 mA
Sample to film distance—450 mm
Sample thickness—0.4 mm.

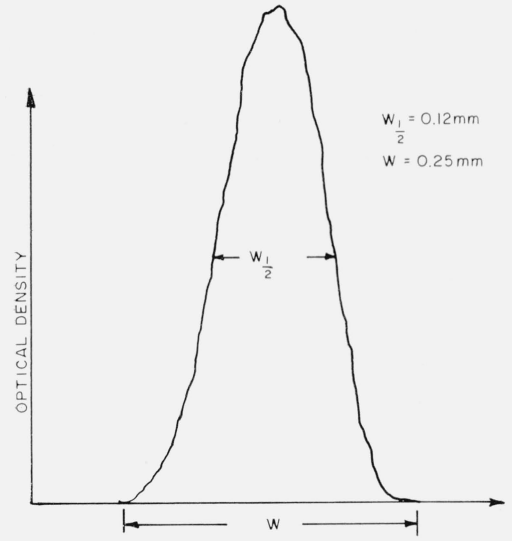

FIGURE 5. *Photometer trace of 220 reflection.*

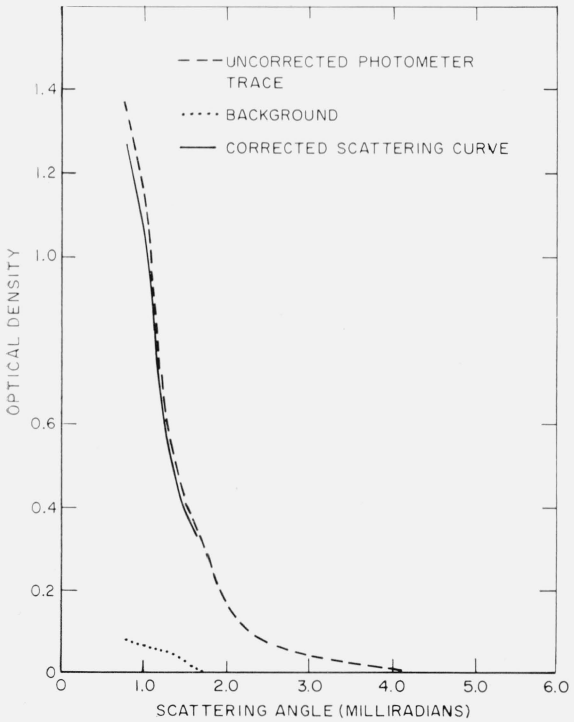

FIGURE 6. *Scattering curve of carbon sample (6 hr exposure).*

A background exposure was made on the same film under the same conditions. Some spillover can be observed but is at most 6 percent of the scattered intensity at the smallest angles. This is partly air scattering, partly fluorescence from the beam stop (which can be eliminated by plating the latter). There is practically no slit scattering, since the only limitation on the beam is in its height, due to the windows of the evacuated tube. The background scattering obtained from a photometer curve and corrected for sample absorption, is shown in figure 6, together with the corrected and uncorrected scattering curves for the carbon sample.

A resolution of about 1900Å was achieved in this experiment, corresponding to a scattering angle of $\sim 8\times10^{-4}$ radians.

Discussion

Considering the simplicity of the apparatus, its performance is excellent. The low cost—about $1,500—also adds to its attractiveness.

By increasing the source-crystal and crystal-detector distances (which would not entail the entire loss of intensity normally associated with increased distance), and/or decreasing the size of the focal spot, increased resolution is possible. Certainly 1-m distances are easily achieved, and a nominal 40 μ focus is available for the Jarrell-Ash unit; the combined effects would give an increase in resolution by a factor of 5, i.e., to 10,000 Å, or better if some of the beam stop spillover can be eliminated.

The beam formed by Borrmann effect is highly polarized. This may offer difficulties in the interpretation of some scattering measurements. On the other hand, there may be circumstances in which such a polarized source may be exploited.

Finally, the large spatial separation of the Kα doublet at the exit face of the crystal while introducing a severe requirement of specimen homogeneity in scattering experiments, suggests the possible utility of the device as a "Laue Monochromator" for diffraction studies. In such an application the entire device could be vastly scaled down with a corresponding gain in intensity. In particular with a 50 μ focal spot, an equal aperture at the crystal and 5 cm distance between the two, CuKα_1 would be satisfactorily isolated.

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