Low Angle X-Ray Diffraction with Long Wavelengths

BURTON HENKE AND JESSE W. M. DUMOND
California Institute of Technology, Pasadena, California
(Received January 30, 1953)

THE use of long wavelengths in the investigation of submicroscopic structures by low angle diffraction methods is of considerable interest for the following reasons. First, the effects of multiple scattering, multiple refractions,[1] and of electron density fluctuations within the particles become or can be made vanishingly small for these wavelengths. This makes the direct application of a relatively simple theory of low angle diffraction[2] more feasible. Second, the larger angles of scattering for the long wavelengths open up to experimental observation the very important central portion of the diffraction pattern. And finally, the sizes of the particles and the thinness of the sample mountings appropriate to these wavelengths permit direct comparison studies with the electron microscope. These provide valuable complementary information as to the nature of packing and other gross features of the material. For larger particles, these studies would permit a dependable verification by means of the electron microscope of the low angle diffraction method.

Very few workers have attempted investigations of this sort in the long wavelength region.[3] Perhaps this has been because of the necessity of working in vacuum and with very low x-ray production efficiencies. In order to meet such problems a new type of diffraction unit has been developed at this laboratory which is designed to operate at such wavelengths as Cr-*K*, Al-*K*, Cu-*L*, O-*K*, and C-*K* lines (2.3, 8.3, 13.3, 23.6, and 44.5 angstroms, respectively).

The central feature of the instrument is the use of a nearly cylindrical totally reflecting mirror, which forms a point-focused image of the source. (See Fig. 1.) This mirror is ground and

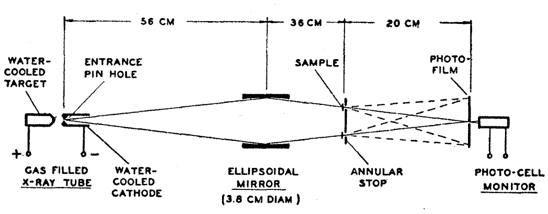

FIG. 1. Sketch of a high intensity, lowangle diffraction unit for soft x-radiation. A large solid angle of x-ray beam is totally reflected and point-focused by an ellipsoidal mirror, ground and polished from Pyrex. Reflection angles are very nearly equal to the critical angle of reflection for the desired line radiation so that all harder background radiation is essentially "cut off."

polished into an ellipsoidal section from Pyrex. In this way a solid angle of radiation is used which is from one hundred to several thousand times that of comparable pinhole geometries.

The principal problem involved in the effective monochromatization of long wavelengths is the suppression of the continuous background radiation. This, however, rapidly diminishes in comparison with the associated characteristic radiation for the longer wavelengths.[4] Effectively complete suppression of this background has been possible by a combination of methods. A specially designed gas-filled x-ray tube has been developed to give a high intensity of x-radiation in the direction of 180° from that of the electron beam by placing the entrance pinhole inside the cathode focusing cup. Soft continuous radiation in this "back" direction should be a minimum. Next the mirror is constructed of such diameter as to present angles of reflection only at the critical angle for the desired line radiation. In this way all radiation that is harder is effectively "cut off." Then the soft component that remains is rapidly absorbed by the filter action of an appropriately chosen substrate on which the sample is supported. Spectrographic checks of the radiation at the focal point have revealed no effective background.

A preliminary study has been made at 13.3A of the same Dow Latex sample which has been investigated by others of this laboratory[5] at the Cu *K*-1.54A. One of these diffraction photographs (ten-hour exposure) is shown in Fig. 2. Measurements

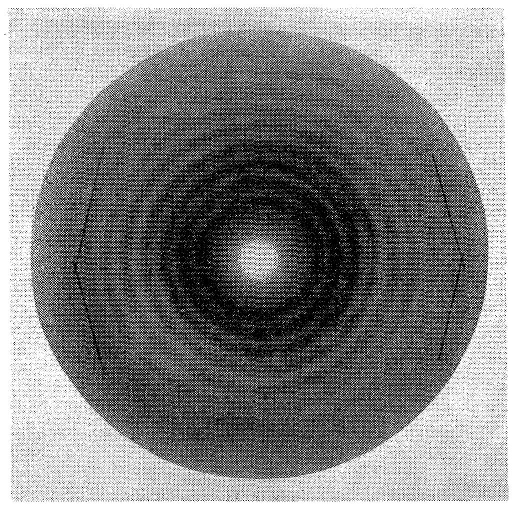

FIG. 2. A low angle diffraction photograph of the Dow Latex spherical particles taken with Cu-*L* 13.3A radiation. The sample-to-film distance is 20 cm. The separation of each pair of short, straight, parallel scratches is 1.88 cm. A few preliminary measurements of the rings indicate a particle diameter of 2720±20A.

based on the Guinier formula for a homogeneous system of spheres randomly spaced yields a value of 2720±20A for the diameters, which is in good agreement with that at the shorter wavelength.

A report on this work, including studies of the Dow Latex at other wavelengths along with parallel measurements with the electron microscope, will be forthcoming.

We wish to acknowledge the generous support of this research by the U. S. Office of Naval Research. We would also like to acknowledge the very valuable assistance of Mr. Herbert Henrikson on the mechanical layout of the instrument.

1 R. von Nardroff, Phys. Rev. 28, 240 (1926).
2 A. Guinier, Ann. Phys. 12, 161 (1939).
3 K. L. Yudowitch, J. Appl. Phys. 20, 174 (1949).
4 E. C. Unnewehr, Phys. Rev. 22, 529 (1923).
5 Danielson, Shenfil, and DuMond, J. Appl. Phys. 23, 860 (1952).

Resolution of the "Photon Difference Method"

J. GOLDEMBERG* AND L. KATZ
Physics Department, University of Saskatchewan, Saskatoon, Saskatchewan, Canada
(Received February 2, 1953)

SINCE its formulation[1,2] the "photon difference method" has been used extensively in betatron work for obtaining cross sections from activation curves. The resulting cross sections so obtained are, in general, quite smooth and present the usual resonance shape of photonuclear reactions. Recently, however,[3–7] experiments have been performed in which the neutrons emitted in the photonuclear reactions are detected directly. These activation curves are usually more complex, since in some cases there are several isotopes present as well as neutrons from (γ, $2n$) and (γ, np) processes. Thus the cross sections obtained from the "photon difference method" sometimes present very "odd" shapes and occasionally show two different peaks. Since this method of solution is liable to give rise to damped oscillations in the cross-section curve if small errors are present in the activation curves, some questions may arise as to the validity of interpreting the humps in the high energy side of the cross-section curves as contributions from the (γ, $2n$) and (γ, np) reactions.[3–6]

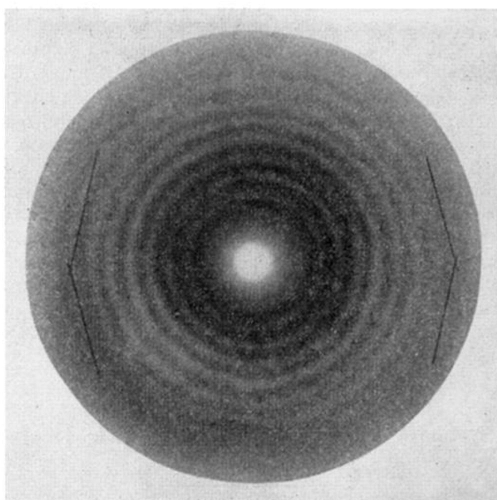

FIG. 2. A low angle diffraction photograph of the Dow Latex spherical particles taken with Cu-*L* 13.3A radiation. The sample-to-film distance is 20 cm. The separation of each pair of short, straight, parallel scratches is 1.88 cm. A few preliminary measurements of the rings indicate a particle diameter of 2720±20A.