

Newkirk J B. The observation of dislocations and other imperfections by X-ray extinction contrast. *Trans. Met. Soc. AIME* 215:483-97, 1959.
[Metallurgy and Ceramics Research Dept., General Electric Co., Schenectady, NY]

An X-ray diffraction method is described by which certain atomic-scale defects in crystals may be directly photographed and their structures analyzed. Examples are presented which include (1) the direct images of line dislocations in Si and LiF, and (2) a method by which their Burgers vectors may be experimentally determined. [The SCI® indicates that this paper has been cited in at least 110 publications since 1959.]

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"In the late 1940s and 1950s, the study of crystal dislocations was very popular among solid-state physicists and research physical metallurgists. Diffuse X-ray effects were being used to describe populations of dislocations. At that time, no one suspected that a single isolated dislocation could be imaged in an X-ray photograph.

"C.S. Barrett was then a consultant to the General Electric Research Laboratory on the subject of precipitation and phase transformations. He suggested and demonstrated the so-called Berg-Barrett (B-B) X-ray method¹ as a tool for the microstructural characterization of various materials. I applied the method first to a coarse-grained alloy of iron plus four percent silicon which was being used extensively in transformer cores. It soon became clear that the B-B method was capable of visualizing what were suspected to be clusters of dislocations.

"The 'breakthrough' B-B picture was made with a single crystal of silicon which had been twisted at high temperature by W.C. Dash,² also at General Electric, to produce a controlled array of what he had proved were undecorated dislocation loops. Small dark spots appeared in precisely the same places as did surface pits when the crystal was later etched. This correlation proved that undecorated single dislocations in silicon could register as spots in B-B topographs.

"At this same time, J.J. Gilman and W.G. Johnston³ (also working at the General Electric Laboratory) were conducting intensive and highly productive optical microscopy studies of dislocations in LiF. Using some of their crystals, I found that individual dislocations emerging upon the crystal surface could be seen as dark spots in B-B photographs. One day, I noticed that the intensities of the spots varied systematically with the sets of diffracting planes that were chosen to make the B-B topographs. By comparing images of the same dislocations in topographs made with different diffracting planes, I deduced a consistent criterion for dislocation visibility. That criterion was independently and concisely expressed by other authors (for a summary description, see reference 4) as $f \cdot b = 0$ for no contrast, in interpretation of electron micrograph images of line dislocations, where f represents the diffraction vector and b the Burgers vector.

"Early in 1958, A.R. Lang,⁵ from Bristol University, England, gave a seminar at the General Electric Laboratory in which he described a sophisticated transmission scanning X-ray method which he had developed. Well-resolved lines, which were presumed to be line dislocations, were commonly present in his topographs. However, neither proof of their identity as dislocations nor a relation between their Burgers vectors and their visibility was demonstrated at that time.

"Upon returning to Bristol, Lang reviewed his topographs and confirmed the $g \cdot b$ criterion for image contrast. Thereafter, many investigators invented and developed several ingenious X-ray topography techniques, producing images with improved resolution, contrast, and angular sensitivity.^{6,7}

"Probably the reason this paper has been so often cited is because it combined a relatively simple laboratory technique with the first conclusive proof that single isolated crystal dislocations could be visualized and analyzed using X rays. The paper has not received any other form of special recognition. For a recent publication, see reference 8."

1. Barrett C S. A new microscopy and its potentialities. *AIME Trans.* 161:15-64, 1945. (Cited 100 times.)
2. Dash W C. Copper precipitation on dislocations in silicon. *J. Appl. Phys.* 27:1193-5, 1956. (265 cites since 1956.)
3. Gilman J J & Johnston W G. Observations of dislocation glide and climb in lithium fluoride. *J. Appl. Phys.* 27:1018, 1956. (135 cites since 1956.)
4. Amelinckx S. *The direct observation of dislocations.* New York: Academic Press, 1964. p. 128.
5. Lang A R. Direct observations of individual dislocations by X-ray diffraction. *J. Appl. Phys.* 29:597-8, 1958. (185 cites since 1958.)
6. Newkirk J B. X-ray diffraction microscopy. *J. Metals* 14:661-5, 1962.
7. Newkirk J B & Wenzel I H, eds. *Direct observation of imperfections in crystals: proceedings of a technical conference held in St. Louis, Missouri, March 1-2, 1961.* New York: Interscience, 1962. 617 p.
8. Tanner B K & Bowen D K, eds. *Characterization of crystal growth defects by X-ray methods.* New York: Plenum Press, 1980. 589 p.