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Dynamical Diffuse Scattering from Impure and Deformed MgO Crystals

By Roger Chang

North American Aviation Science Center, Thousand Oaks, California, U.S.A.

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The transmission electron diffraction patterns of several impure and cold worked MgO crystals are presented showing in particular the Kikuchi lines and Kikuchi bands. The fine structures and the broad and curved Kikuchi lines associated with slight cold-working can be qualitatively understood as the result of multi-beam diffraction effects. The existence of deficient and asymmetrical Kikuchi lines within an excess band is anomalous and may possibly be interpreted as due to Bragg scattering of the relatively intense diffuse beams according to Gjønnes and Watanabe.

Introduction

The dynamical diffuse scattering from nearly perfect MgO crystals obtained by burning magnesium metal was recently discussed by Watanabe and Gjønnes (Watanabe & Gjønnes, 1965; Gjønnes & Watanabe, 1966), who reported Kikuchi lines accompanied by a fine structure (particularly for the strong reflections) and very strange diffuse scattering patterns (instead of the Kikuchi lines) when three or more Bragg reflections were excited. The present paper reports the dynamical diffuse scattering of impure and slightly cold worked MgO crystals.

Experimental

MgO crystals (Norton) containing 93 and 300 ppm of iron were used. Cleaved plates about 5 × 5 × 1 mm in dimensions were heated in air to 1400 °C for two hours then either slowly cooled in the furnace or rapidly cooled in air. Some of the specimens were plastically deformed by compression (along two opposite rectangular faces) to a total strain of about two per cent. The plates were then hand polished to appropriate thicknesses for acid thinning, which was done with a moving jet of hot phosphoric acid until puncture. Thin sections near the puncture were collected, washed and transferred to an electron microscope (AEI model EM-6) for transmission studies. The thicknesses of these specimens varied from 3000 to 5000 Å with faces nearly parallel to the cube plane. 100 kV electron beam with a resolution of a few volts was used. The specimens can be tilted to a maximum of 6–7° in the microscope but the actual tilt used in these studies was never more than 2°.

Fig. 1 shows the Kikuchi lines and bands of a MgO crystal (300 ppm Fe, slowly cooled from 1400 °C) excited for the 040 Bragg reflection. All the Kikuchi lines (deficient and excess) and bands parallel to both the [100] and [010] directions can be indexed. Attention is directed to the anomalous deficient line nearly in the middle of the 200 Kikuchi band (marked by arrowhead). This point will be discussed further in the latter part of the paper.

Fig. 2 shows the Kikuchi lines and bands of a second MgO crystal (300 ppm Fe, slowly cooled from 1400 °C) excited for the 020 Bragg reflection. Attention is again directed to the intensity anomaly inside the 200 Kikuchi band (marked by arrowhead). All the Kikuchi lines and bands can be readily indexed.

Fig. 3 shows the diffraction pattern of a third MgO specimen (93 ppm Fe, slowly cooled from 1400 °C). The fine structure associated with the 400 and 600 Kikuchi lines (marked by arrowheads) is evident.

Fig. 4 shows the Kikuchi lines from a fourth MgO specimen (93 ppm Fe, air cooled from 1400 °C, deformed by compression to a total strain of 2%). Here the Kikuchi lines are not only broad but show slight curvature.

Discussion

Both the Kikuchi lines and bands are observed in impure and cold worked MgO. The anomalous deficient line within the excess 200 Kikuchi band shown in both Figs. 1 and 2 is worth particular attention. One might say that it is a special feature of the fine structure associated with the Kikuchi bands. The theoretical treatment of Kainuma (1955) suggests that when the thickness of the specimen under investigation becomes thin, an oscillating modulating term (last term in the square bracket of equation (104) of Kainuma's paper) will come into play. However, this oscillating term can predict only broad sinusoidal changes in intensity within the band but will not predict the existence of any sharp deficient line. It may possibly be interpreted in terms of fragmentary displacement of the (400) deficient Kikuchi line by a 200 reciprocal lattice vector due to Bragg scattering of the relatively intense diffuse beams suggested by Gjønnes & Watanabe (1966). It is noteworthy, however, that the deficient line shown in Figs. 1 and 2 is located slightly off center of the band, suggesting possibly the presence of another excess line symmetrically positioned with respect to the deficient line.

The fine structure shown in Fig. 3 can be interpreted qualitatively as the result of multi-beam diffraction effects discussed recently in some detail by Gjønnes &

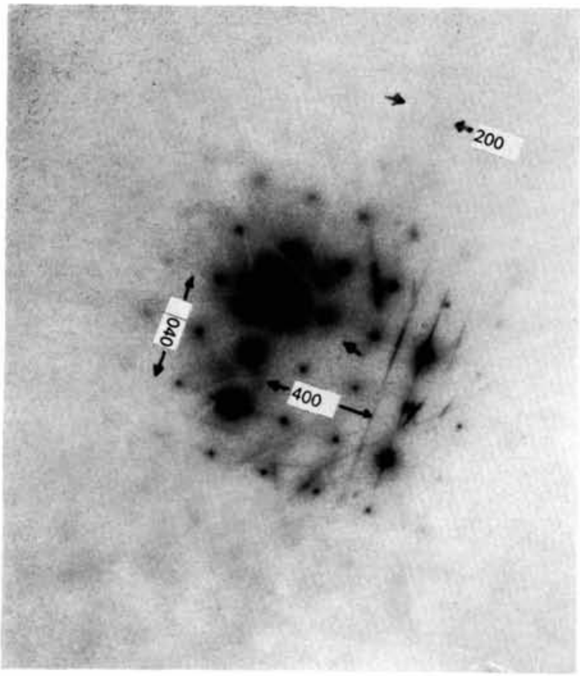

Fig. 1. Electron diffraction pattern of MgO crystal no. 1 (300 ppm Fe, slowly cooled from 1400°C).

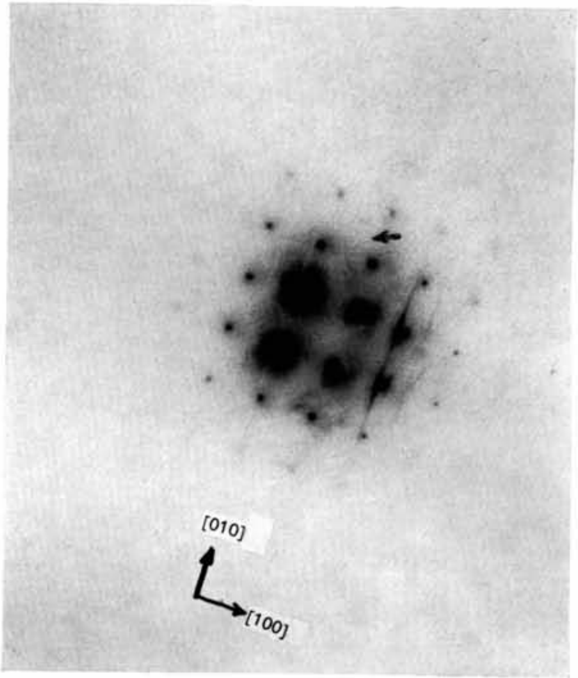

Fig. 2. Electron diffraction pattern of MgO crystal no. 2 (same as crystal no. 1).

Fig. 3. Electron diffraction pattern of MgO crystal no. 3 (93 ppm Fe, slowly cooled from 1400°C).

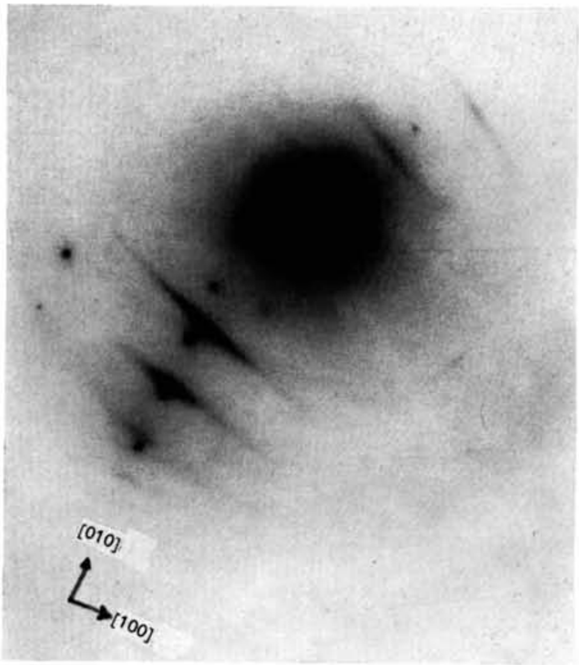

Fig. 4. Electron diffraction pattern of MgO crystal no. 4 (93 ppm Fe, air-cooled from 1400°C and cold-worked by compression 2%).

Watanabe (1966). The curvature of the broad Kikuchi lines shown in Fig. 4 can therefore be interpreted as the overlap of the broadened fine structures seen in Fig. 3. The interpretation is qualitative, however, and any quantitative analyses of the diffraction patterns must await further theoretical understanding of the dynamic diffuse scattering of electrons.

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The Darwin Dynamical Theory of X-ray Diffraction*

By Bernard Borie

Metals and Ceramics Division, Oak Ridge National Laboratory, Oak Ridge, Tennessee 37830, *U.S.A.*

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A simple alternative to the Ewald–von Laue dynamical theory of X-ray diffraction is described. Several of the more important features of dynamical diffraction, including anomalous transmission, diffraction in asymmetric Laue geometry, and the properties of the dispersion surface, are derived. The method involves the solution of a system of difference equations similar to those first solved by Darwin. The formalism of electromagnetic theory is avoided, and the result is achieved with no loss in rigor. In addition to its greater simplicity, the theory seems to be easier to modify to account for small deviations from perfect periodicity, which are difficult to account for in terms of the Ewald–von Laue treatment.

Introduction

The central problem of X-ray crystallography has traditionally been, given a specific array of centers of scattering factor f, to combine the amplitudes and phases of the resultant scattered waves in order to recover the diffraction pattern associated with the array. Neither quantum mechanics nor electromagnetic theory is normally invoked. The crystallographer simply takes f to be the ratio of the wave scattered by an atom to that scattered by a classical electron, and leaves its computation to the theoretical physicist. All of the electromagnetic and quantum theory of the problem is contained in the calculation of f.

The Ewald (1916)–von Laue (1931) dynamical diffraction theory is a departure from this custom. Here, in order to obtain the total wave field inside a perfect crystal, one solves Maxwell's equations in a medium with a periodic, time dependent, complex dielectric constant. The treatment is elegant but rather involved.

We here show that that is not necessary, that all of the features of dynamical diffraction including the anomalous aspects of the Borrmann effect are recoverable with the usual tools of X-ray crystallography. No electromagnetic theory is used. The result is achieved by simply solving in Laue geometry the difference equations first solved by Darwin (1914) in Bragg geometry.

It has previously been shown (Borie, 1966) that at the precise Bragg angle for the symmetrical Laue case, such a procedure leads to the vanishing of the linear absorption coefficient and the anomalous behavior of the refractive index associated with the Borrmann effect. In this paper we compute the wave field for an arbitrary direction of incidence. Diffraction in asymmetric Laue geometry is discussed. We examine the behavior of the wave field in the immediate vicinity of the Bragg reflection for the symmetrical Laue case, and we derive the properties of the dispersion surfaces. The result is identical with that of the Ewald–von Laue theory.

Fresnel diffraction in transmission

A preliminary to writing the Darwin difference equations is to calculate the wave scattered by a single plane of scattering material. A family of such planes is then assembled to form a crystal, and the combination of the amplitudes and phases of the scattered waves is expressed by the difference equations.

This is conventionally done in reflection, or Bragg geometry (James, 1950), as illustrated in Fig. 1. The xy plane is populated by a uniform distribution of

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