

Lamellar Intergrowth of Orthoenstatite and Clinoenstatite [(Mg, Fe)SiO₃] Studied by Lattice Imaging

By W. F. MÜLLER

Institut für Kristallographie, Frankfurt a.M., Germany (BRD)

(Received 29 April 1974)

Deformation experiments under high static confining pressure and observations on tectonically deformed specimens have shown that orthoenstatite (space group *Pbca*) can be transformed to clinoenstatite (*P2₁/c*) by shear stress (for literature, see Coe, R. S. [*Contr. Mineral. Petrol.* (1970), **26**, 247–264]). The specimen used for the present study was prepared by ion-thinning from an orthoenstatite sample deformed at 800°C and 5 kb confining pressure at a strain rate of 10⁻⁴s⁻¹. Previous high voltage (650 kV) transmission electron microscopy had revealed submicroscopic lamellae of orthoenstatite (OE) and untwinned clinoenstatite (CE) joined along (100) planes [Coe, R. S. & Müller, W. F. (1973). *Science*, **180**, 64–66]. The transmission electron microscope technique of one-dimensional lattice imaging has been applied now to the same specimen using a JEM 100 B instrument equipped with a side-entry goniometer. Lattice fringes with spacings of 18.2 Å and 9.1 Å corresponding to the (100) planes of OE and CE, respectively, make it possible to distinguish easily the two polymorphs. The lamellae of CE consisted of an even number of lattice units along *a**. This is due to their mode of formation: The shear transformation causes an halving of the (100) spacing of OE, thereby producing twice the number of unit cells of CE [Coe, R. S. (1970); Morimoto, N. & Koto, K. (1969). *Z. Kristallogr.* **129**, 65–83]. The lamellae of OE and CE are sometimes only one and two (100) spacings, respectively, in width.

Theoretical Basis of Diffraction Contrast of Lattice Imperfections in Transmission Electron Microscopy

By A. HOWIE

*Physics Institute, University of Aarhus, Denmark**

(Received 5 June 1974)

The dynamical theory of diffraction, both for X-rays and electrons, was formulated many years ago but for several decades found only limited application in the analysis of diffraction patterns because of the difficulty in obtaining specimens whose defect content was known with sufficient precision. This situation was transformed with the advent of real-space techniques, such as transmission electron microscopy and X-ray topography, which allowed the dynamical theory to be applied to local regions in the vicinity of individual defects instead of to the diffraction pattern coming from the specimen as a whole. As a consequence there has, in the electron case, been an intimate relation between the continuing improvement of the instrumental technology by which these real space images are obtained and the development of the dynamical theory for imperfect crystals which is essential for their interpretation. What may be described as the established theory (see, for example, Hirsch, P. B., Howie, A., Nicholson, R. B., Pashley, D. W. & Whelan, M. J. [*Electron Microscopy of Thin Crystals* (1965). London: Butterworth] was based on a series of approximations suitable for a transmission instrument with a resolution of about 20 Å at an energy of 100 keV operating on ~2000 Å thick films of structures with relatively small unit cells. These approximations, discussed in detail by Howie, A. & Basinski, Z. S. [*Phil. Mag.* (1968), **17**, 1039–1063] include the *deformable-ion approximation* (appropriate for slowly varying strains), the *strong-beam approximation* (appropriate to small unit cells where, with careful choice of orientation, only a very small number of diffracted beams need be considered) and the *column approximation* (appropriate when the lateral spreading of the waves in the crystal is small compared with the resolution achieved). In addition, it is assumed that the small-angle inelastically scattered electrons accepted by the aperture

* Permanent address: Cavendish Laboratory, Cambridge, England.

preserve the image contrast effects built up by elastic scattering {for a discussion see Cundy, S. L., Howie, A. & Valdré, U. [*Phil. Mag.* (1969). **20**, 147–163]}. An enormous number of observations have been analysed in great detail with this theory and new information about the structure of dislocations, stacking faults, precipitates and point-defect agglomerates obtained. It is not in general possible from the observations to work backwards to deduce the structure of a completely unknown defect. Instead, images must be computed for a whole range of possible models and diffraction conditions and compared with observation. All of the above approximations are to some extent invalid for the analysis of the high-resolution results now being obtained as a result of improvements in the instrumental stability and electron optical performance. So far the most successful theoretical developments have been in the use of greatly increased numbers of diffracted beams, even several hundred, to analyse the lattice images from complex oxides with large unit cells [Allpress, J. G., Hewat, E. A., Moodie, A. F. & Sanders, J. V. (1972). *Acta Cryst.* **A28**, 528–536; Cowley, J. M. & Iijima, S. (1972). *Z. Naturforsch.* **27a**, 445–451]. The processing of the large number of equations involved is made possible with modern computers and in principle this method could be applied to any defect problem by placing it in an artificially periodic structure. Greatly improved resolution of defects in simple structures has followed from the development of the weak-beam method [Cockayne, D. J. H., Ray, I. L. F. & Whelan, M. J. (1969). *Phil. Mag.* **20**, 1265–1270]. These images can be analysed with usually less than six diffracted beams and a large number of columns. When the column approximation is relaxed, the computed effect can sometimes be quite appreciable [Howie, A. & Sworn, C. H. (1970). *Phil. Mag.* **22**, 861–864] but so far, possibly because of the convergent illumination normally used, experimental confirmation of these effects is still lacking. A further problem requiring more detailed examination is the use of the deformable ion approximation for small radiation damage clusters, where strain contrast and phase contrast (due to dilatation) are often present together. It may be possible to get useful information from out-of-focus images in these cases (see work by Wilkens and his colleagues at this conference) but the contribution of inelastically scattered electrons will have to be considered. A considerable contribution from these electrons to the background intensity in weak beam images has recently been measured (P. L. Gai – private communication). Although these developments are perhaps the ones of most immediate relevance for transmission studies of defects, it should be noted that the dynamical theory of electron diffraction is currently being tested in many other instruments. Rather detailed information about the elastic and inelastic scattering in perfect crystals can be obtained from high-voltage electron microscopy, in particular the critical-voltage effect {see, for example, Lally, J. S., Humphreys, C. J., Netherell, A. J. F. & Fisher, R. M. [*Phil. Mag.* (1972). **25**, 321–343]}. The scanning electron microscope has opened up a number of new applications, in particular the influence of dynamical diffraction effects on backscattering and the possibility of detecting defects near the surface of bulk crystals by using this effect {see, for example, Spencer, J. P., Humphreys, C. J. & Hirsch, D. B. [*Phil. Mag.* (1972). **26**, 193–213]}. The dependence of the backscattering from a crystal on the incident-beam orientation has now been observed over a wide energy range and may provide a long awaited theoretical connexion between the structural studies made in the electron microscope and those possible at much lower energies in Auger equipment [Rusch, T. W., Bertino, J. P. & Ellis, W. P. (1973). *Appl. Phys. Lett.* **23**, 359–360].

J. Appl. Cryst. (1975). **8**, 213

Kinematical Theory of Images from Polycrystalline and Random-Network Structures

BY O. L. KRIVANEK AND A. HOWIE

Cavendish Laboratory, Cambridge, England

(Received 5 June 1974; accepted 6 August 1974)

Kinematical electron microscope images to be expected from the Polk continuous random-network model and from a polycrystalline cluster have been computed. Dark-field images of both models show qualitatively similar speckle patterns. Fringes are observed in bright-field images of thin polycrystals ($t \lesssim 40\text{\AA}$), but they disappear in thicker samples and are not observed in any of the Polk-model images. The experimental observation of fringes therefore cannot be explained by the current theory, possibly because it does not take account of the angular spread of illumination. Except perhaps in very thin specimens, it seems impossible at present to draw any reliable conclusions about the local atomic arrangement in amorphous materials by simple examination of electron micrographs.

1. Introduction

Renewed discussion and some controversy about the structure of amorphous solids has arisen from recent

applications of high-resolution electron microscopy to these materials. Rudee (1971) suggested that the $\sim 15\text{\AA}$ diameter bright spots which he observed in dark-field images of amorphous Ge originated from crystallites