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Potentialities and Limitations of High-Resolution Electron Microscopy of Crystal Lattices and their Imperfections

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The resolving power of modern electron microscopes is better than 0·3 nm. This enables images to be formed which show detail at the level of interatomic distances. However the relatively strong scattering of electrons means that when images from crystals are obtained, the image detail is not generally easy to interpret in terms of crystal structure. Recently much attention has been paid to devising experimental methods of forming images which will both utilise the high resolving power of the microscope, and at the same time produce images whose detail can be interpreted in terms of structure in a relatively straightforward manner. The method of lattice fringe images has been of particular importance in this area. By allowing a number of diffracted beams from a crystal to interfere in the image plane, an interference pattern or lattice fringe image is formed. For regions of perfect crystal, the details of the fringe image are sensitive to parameters such as the number of contributing diffracted beams, the lens focal conditions, the diffraction geometry and the foil thickness. The reasons why these parameters influence the images can be understood from geometrical optics and from the theory of dynamical scattering. It is therefore of importance for the interpretation of lattice fringe images to be able either to take into account the influence of these parameters on the image (*e. g.* by image processing) or to determine under what conditions their influence can be neglected. While both of these approaches are feasible, to date most attention has been paid to the latter, *viz.* specifying the experimental conditions for which a straightforward relationship exists between image and object. In the case of specimens which can be treated as phase objects, Cowley, J. M. & Moodie, A. F. [*Proc. Phys. Soc.* (1960). 378–384] gave an analytical relationship between the image contrast and the projected charge density of the object, valid under certain conditions of defocus and resolution, and applicable both to periodic and non-periodic objects. More recently this relationship and its limiting conditions have been studied for periodic objects by comparing computer-simulated lattice fringe images (taking into account dynamical scattering effects and lens aberrations) with maps of the projected charge density {for a review see Allpress, J. G. & Sanders, J. V. [*J. Appl. Cryst.* (1973). **6**, 165–190]}. In this way it has been possible to define limits on foil thickness, lens defocus and resolution within which straightforward image interpretation is valid. These studies have given theoretical support and guidance to important experimental studies in the field of complex oxides and related structures. At present the computer simulation studies apply to specific crystal structures, but hopefully the criteria for image interpretation obtained from them can be generalized to give guidelines for a wider range of structures. In the case of imperfect crystals. establishing the conditions for straightforward image interpretation has proved to be more elusive. In many fringe images, deviations from perfect periodicity (*e.g.* terminating fringes, fringe bending) are observed. If a one-to-one correspondence between fringes and projected charge distribution (or lattice planes) is assumed, then detailed models of lattice defects can be constructed. In this way the lattice strain has been measured, on a scale of interatomic spacings, around defects identified from the fringe images as end-on dislocations [Parsons, J. R. & Hoelke, C. W. (1969). *J. Appl. Phys.* **40**, 866–872] and edge-on G. P. Zones [Philips, V. A. (1973). *Acta Met.* **21**, 219–229]. While such interpretations may be valid, there are a number of important factors which require consideration: – (i) It has been demonstrated experimentally by Cockayne, D. J. H., Parsons, J. R. & Hoelke, C. W. [*Phil. Mag.* (1971). **24**, 139–153] that an image showing a terminating fringe does not necessarily imply that there is a terminating lattice plane at the corresponding point in the object. (ii) It has been shown experimentally (Cockayne, Parsons & Hoelke, 1971) that even gross features of images such as the presence or absence of terminating fringes can be influenced by diffraction geometry (*e.g.* small changes in the beam direction can entirely change the appearance of the image). Despite these difficulties, calculated images [Cockayne, D. J. H. (1970). D. Phil. Thesis. Univ. of Oxford] indicate that for defects in specific orientations (*e.g.* dislocations viewed end-on) this dependence of the fringe detail upon experimental parameters may be negligible, enabling straightforward interpretation of the images to be carried out. However it is important to note that: – (i) To date, these calculations involve approximations which become increasingly important with increasing resolution. Further calculations are required in this area. (ii) In most experimental studies, it is not possible to determine the nature and orientation of the defect by standard contrast techniques (foils too thin for normal contrast experiments; lack of Kikuchi lines). Consequently, if a straightforward image interpretation requires that the defect be in a specific orientation, there may be difficulty in ensuring this. In many cases, the defects likely to be

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present in a lattice may be constrained by geometrical or other factors, making it possible to overcome the difficulty outlined above. This appears to be the case in many studies of defects in complex oxides. However in the case of dislocations in metals and semiconductors, this difficulty cannot be ignored. At a lower level of resolution, a method has been developed for using weak diffracted beams to study lattice defects {the weak-beam method [Cockayne, D. J. H., Ray, I. L. F. & Whelan, M. J. (1969). *Phil. Mag.* **20**, 1265–1270]}. In this method the diffracted beam acts as a probe, having increased scattering in regions of large lattice distortion. By this means, the ability to locate defect cores and to characterize defect geometries has been improved in resolution by an order of magnitude over previous diffraction contrast techniques. Although with standard high-resolution microscopes the method is limited experimentally to a resolution of approximately 1·5 nm (because of the low image intensity and long exposure times), there is experimental evidence that developments in high-brightness electron sources will improve the resolution attainable. The method has been applied to a wide range of problems in crystal defect studies, and the limitations on image interpretation have been investigated in some detail. The parameter of incident-beam convergence angle appears to be important in damping the dependence of the image upon defect depth and foil thickness. This is relevant to use of the weak-beam method for obtaining accurate size-density determinations of small point-defect clusters. The possibility of the increased image resolution enabling experimental measurements of the parameters of dislocation core models to be made is being investigated. Initial theoretical and experimental results indicate that this method may provide an experimental means for differentiating between models of defect cores.

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Diffraction Contrast of Small Point-Defect Agglomerates as Studied by Transmission Electron Microscopy (TEM)

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Point Defect Agglomerates (PDA) are frequently observed in particle-irradiated or quenched metals. The sizes of the PDA are often so small that their shape cannot be resolved directly with the electron microscope. In the paper methods are outlined by which the shape (cavities, Frank dislocation loop or perfect dislocation loop) and nature (clusters of vacancies or interstitials) can be determined by means of an electron microscopical analysis. The PDA can be subdivided roughly into two groups: PDA with and without strain field in the surrounding crystal matrix, respectively. In the first case the TEM images are determined mainly by the strain field rather than by the (atomistic) structure of the PDA. The identification of such PDA requires in general three steps [Wilkens, M. (1970). *Modern Diffraction and Imaging Techniques in Materials Science*, Edited by S. Amelinckx *et al.*, p. 233. Amsterdam: North-Holland; Rühle, M. (1969). *Radiation Damage in Reactor Materials*, Vol. 1, 113. Vienna: IAEA; Wilkens, M. (1974). In *Proc. Int. School on Electron Microscopy, Erice*, Edited by U. Valdré and E. Ruedl. Luxembourg: C. I. D.]: (i) imaging the PDA under different, well defined diffraction conditions, (ii) calculations of the contrast figures for relevant models of PDA, and (iii) comparison of the observed and calculated contrast figures. For the first time, this concept was applied successfully for PDA in neutron-irradiated and ion-bombarded f.c.c. materials [Rühle, M., Wilkens, M. & Essmann, U. (1965). *Phys. Stat. Sol.* **11**, 819; Rühle, M. & Wilkens, M. (1967). *Phil. Mag.* **15**, 1075; Rühle, M., Häussermann, F. & Rapp, M. (1970). *Phys. Stat. Sol.* **39**, 609.], the PDA were revealed as Frank dislocation loops. The experimental observations in b.c.c. and h.c.p. materials suggested that the PDA are perfect dislocation loops. In these cases for the determination of the loop plane and the Burgers vector contrast calculations (step ii) have to be done for a very large number of different loop configurations. Therefore, for particular diffraction conditions specially adopted approximation methods for the calculation of the contrast figures of small PDA were developed [Wilkens, M. & Rühle, M. (1972). *Phys. Stat. Sol.* (*b*) **49**, 749]. and applied successfully to PDA in b.c.c. and h.c.p. materials [Häussermann, F., Rühle, M. & Wilkens, M. (1972). *Phys. Stat. Sol.* (*b*) **50**, 445; Föll, H. Wilkens, M. (1974). To be published]. Among small defects without strain field (*e.g.*, cavities, incoherent precipitates, disordered zones, *etc.*) the diffraction contrast of cavities is of special interest. In this case the sign and the size of the contrast depend very sensitively on the focusing conditions of the objective lens [Rühle, M. (1972). *Radiation-Induced Voids in Metals*, Edited by J. W. Corbett and L. C. Ianniello, p. 255. USAEC; Rühle, M. & Wilkens, M. (1972). *Proc. 5th Europ. Reg. Conf. on Electron Microscopy, Manchester*, p. 416; Rühle, M. & Wilkens, M. (1974). *Proc. 8th Int. Congress on Electron Microscopy, Canberra*]. This is particularly important for cavities which are very small compared to the thickness of the specimen. By imaging small cavities (in a thin foil) in the defocused mode further information on the internal structure of the cavities can be obtained [Rühle, M. & Wilkens, M. (1974). Submitted for publication].