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Introduction

The plane matching (PM) model of grain boundary structure was originally proposed by Pumphrey (1) in order to explain certain transmission electron microscopy (TEM) observations of periodic contrast features (2,3) at grain boundaries. The basic premise of this model is that the matching of a set of close-packed planes across a grain boundary results in a low energy configuration. Since the matching is brought about by a periodic array of defects, it is responsible for a characteristic structure within the boundary.

Subsequent applications (4) of the PM model to the description of grain boundary structures have met with success. Nevertheless each of these studies has had to rely heavily upon indirect evidence (viz. electron diffraction information) due to the limited spatial resolution of the involved TEM techniques. The purpose of the present article is to demonstrate an exact verification of plane matching conditions at a grain boundary utilizing high resolution electron microscopy. The proof is based upon a direct image of the mismatched atomic planes.

Optical Analogue

Periodic structural features observed within grain boundaries by TEM are given a simple optical representation in the PM theory. (1) Idealizing the set of mismatched atomic planes on both sides of a boundary as a pair of optical gratings, the boundary region itself is depicted by their overlap (Fig.1). Consequently the observed linear contrast features at the boundary may be visualized as a Moiré effect.

In general the spacings d_1 and d_2 of the two optical gratings need not be the same, and as shown in Fig. 1, when misoriented by an angle θ the overlapping gratings give rise to a Moiré fringe pattern of characteristic (5) spacing:

$$d = \frac{d_1\, d_2}{(d_1^2 + d_2^2 - 2d_1 d_2 \cos\,\theta)^{1/2}}$$

Furthermore the rotation angle (ρ) between the Moire fringes and the fringes of grating 1 may be computed as: (5)

$$\sin\,\rho = \frac{d_1\, \sin}{(d_1{}^2 + d_2{}^2 - 2d_1 d_2\, \cos\,\theta)^{1/2}}$$

It should be pointed out that unlike typical Moire fringe images, PM defects do not change image character with the operating reflection. Both the spacing and orientation of PM lines remain constant when imaged with any reciprocal lattice vector ($\underline{g}$) in either bordering grain. It has been postulated (1) that the high density Moiré bands are actually images of the localized strain fields due to relaxation effects at positions of high atomic density in the interfacial region. Hence although they obey the geometrical relationships of a purely optical Moiré, PM lines have distinct structural characteristics.

Experimental Procedure

An alloy of 90.5 at % Al and 9.5 at % Zn was fabricated under Ar atmosphere, cast in a chilled crucible and homogenized at 420°C for 72 hrs. Following both hot and cold rolling to a final thickness of approx. 5 mils, the material was solution annealed (15 mins at 400°C) and then aged at 180°C for 30 mins. to encourage grain boundary precipitation.

Foils were prepared in a double jet polisher using a nitric acid-methanol electrolyte at -25° to -30°C. These were immediately fitted into ± 45° biaxial tilt cartridge and viewed in a Siemens Elmiskop 102 operated at 125 kV.

Regions of precipitate/matrix interfaces at grain boundaries (satisfying $d_1 \neq d_2$) were surveyed and imaged in a tilted illumination mode. The objective aperture included only one strongly diffracted beam from each of the regions to be imaged as well as the transmitted beam.

Results

The lattice image shown in Fig 2 (a) is an example of the type of micrograph used for analysis. Diffraction conditions are shown in Fig. 2 (b). The figure includes simultaneous images of the (200) planes from matrix [1] the $(11\bar{2}0)$ planes of the rhombohedrally distorted (6) Zn-rich precipitate (P) and the (200) planes (at much lower visibility) from matrix [2]

Using the highly visible fringes in grain [1] as a magnification standard, their spacing (6) (2.02 Å) was measured from enlarged micrographs to within ± 0.01 Å. By comparison the precipitate fringes were found to be spaced 2.09 ± 0.01 Å, indicative of a higher Zn content. The rotation angle θ between matrix [1] and precipitate (P) fringes was determined from both lattice image and SAD (Fig. 2(b)) measurements to be 9.2 ± 0.1.° Applications of the Moiré formulae given above allowed the measured spacing (d) and rotation angle (ρ) of the boundary structural lines to then be compared with the calculated values (see also Fig. 1), as follows:

$$d \text{ meas.} = 12.2 \pm 0.5 \text{ Å} \qquad d \text{ calc} = 12.4 \pm 0.2 \text{ Å}$$

$$\rho \text{ meas.} = 106.5 \pm 0.5^\circ \qquad \rho \text{ calc} = 107 \pm 1^\circ$$

These results are in excellent agreement. At this level of resolution, tilting experiments failed to reveal the presence of any other structural lines within the boundary region.

The structural nature of the apparent Moiré fringes is revealed in Fig. 3 which is a high magnification image of the boundary region in Fig. 2(a). Here it is seen that continuity of lattice planes across the boundary is for the most part preserved. However some degree of mismatch is observed within the high density Moiré bands as evidenced by the presence of terminating fringes (arrowed). These terminations furthermore suggest that misorientation dislocations with a Burgers vector equal to the interplanar spacing may be associated with the misfit lines, in agreement with the PM theory. (4)

Discussion

In order to apply other models of grain boundary structure to the example shown here, it would be necessary to know the precise crystallographic description of the boundary. (7) Unfortunately this requires the presence of Kikuchi lines in electron diffraction patterns, (8,9) a condition which is obviously prevented by the severe restrictions on foil thickness ($\lesssim$ 400 Å) necessary for lattice imaging.

Nevertheless, as shown in this study, the enhanced resolution afforded by lattice fringe imaging clearly provides an advantage in grain boundary structural analysis. Not only can the local misorientation of atomic planes be measured with high accuracy using the technique; but the details of mismatch accommodation can be directly observed. In Fig. 3 the presence of terminating fringes within regions of high optical density indicates that strain is taken up within regions of high atomic density. This and the excellent agreement between experimental and computed values of d and ρ are a consistent indication of plane matching conditions.

Conclusions

A direct (real space) verification of plane matching at a grain boundary interphase interface has been obtained using high resolution transmission electron microscopy. Analysis shows that the observed pattern of grain boundary lines is a structural Moiré, with strain accommodation occurring at boundary regions of high atomic density.

Acknowledgements

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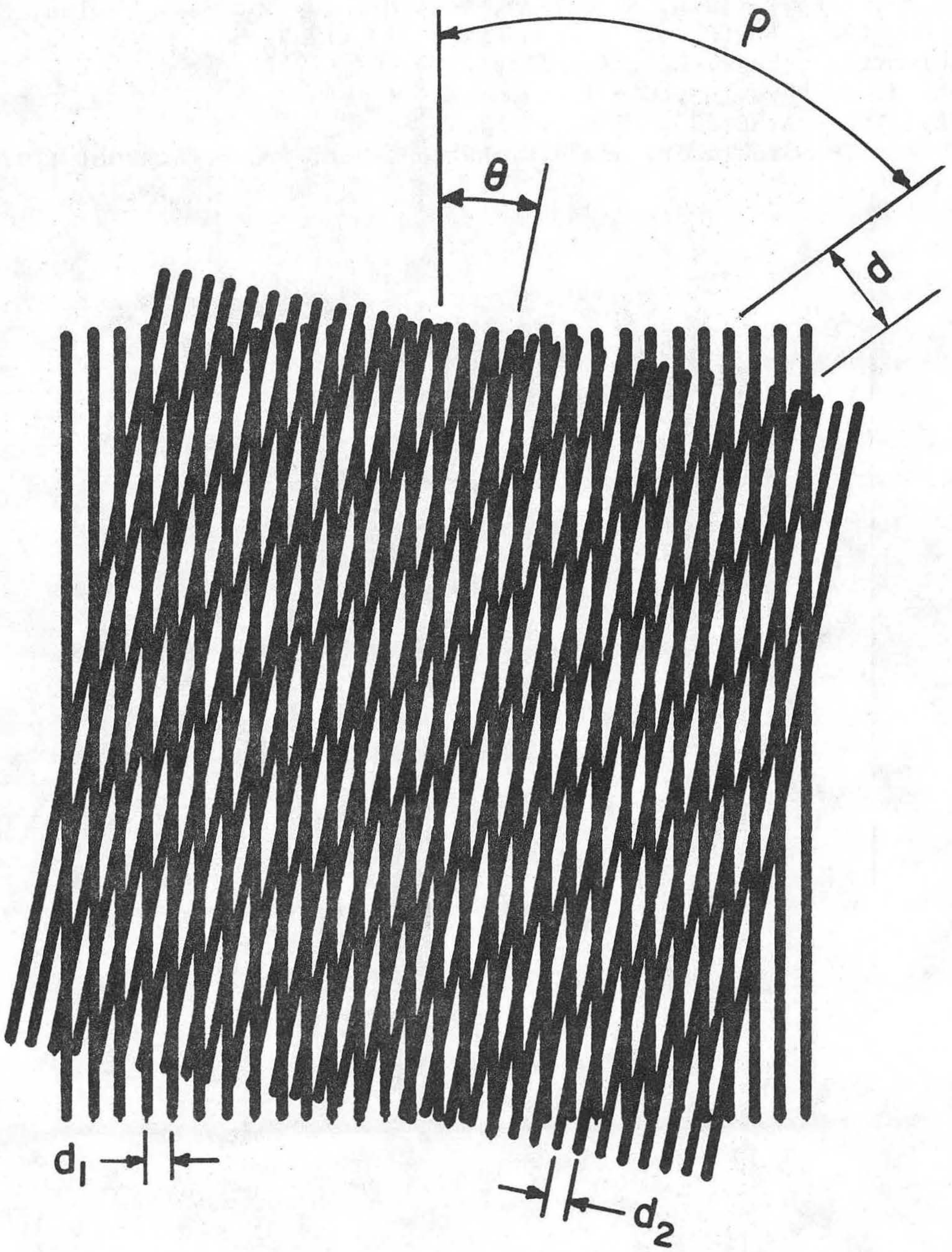

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FIG. 1

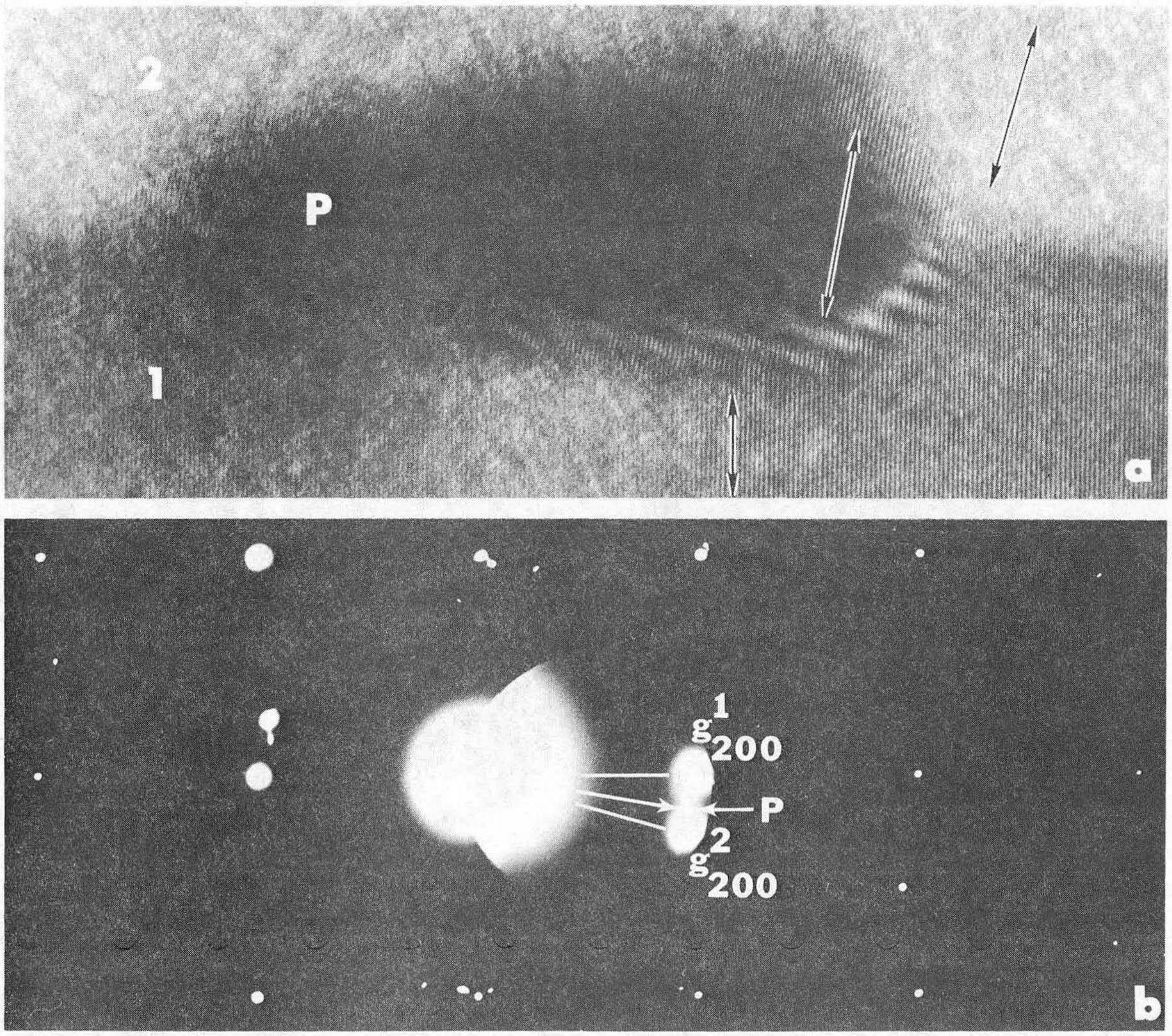

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FIG. 2

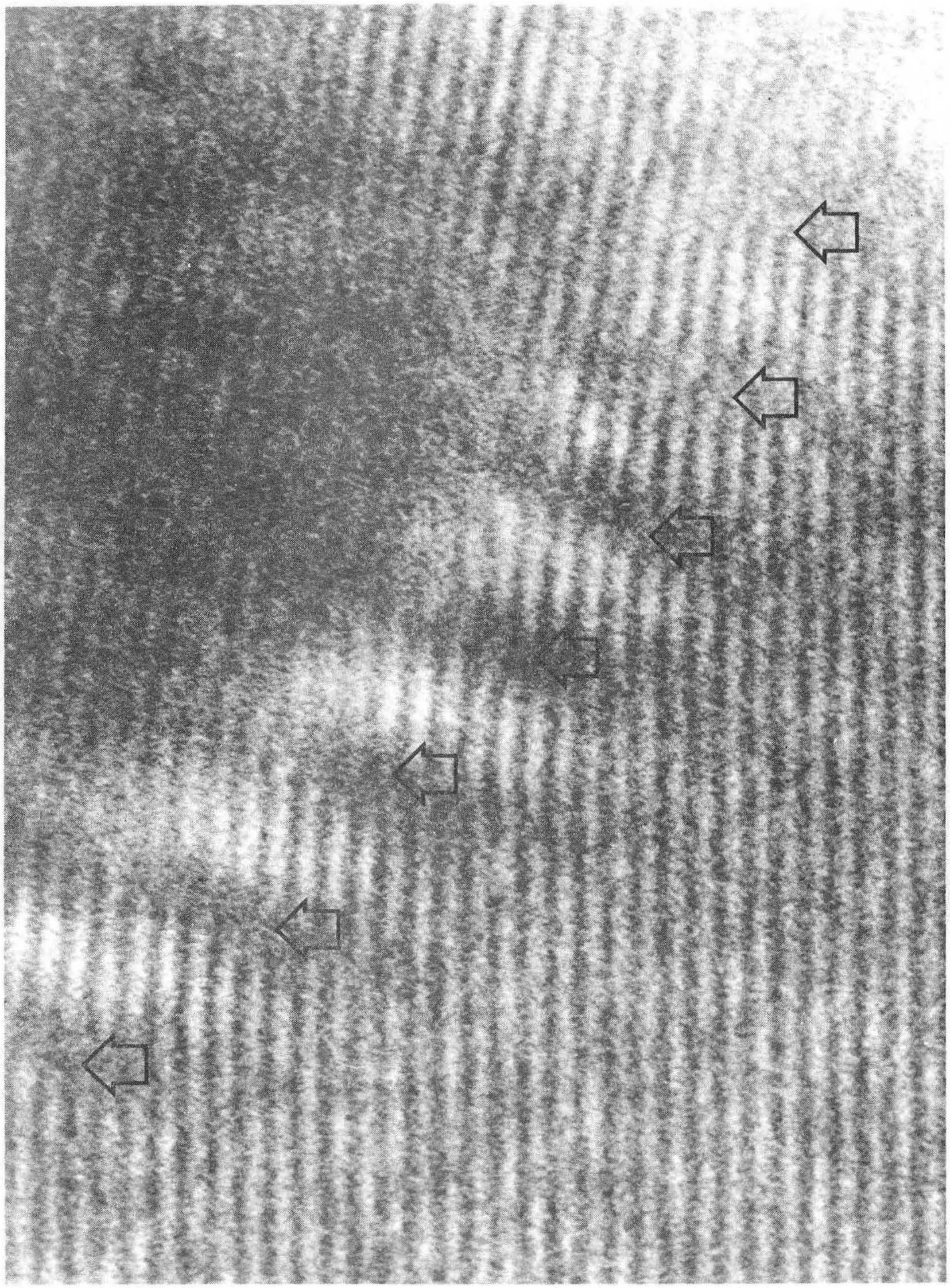

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FIG. 3

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