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“Optimisation” of imaging conditions for weak beam studies of dislocation core structures in Ti_3Al

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Abstract

The imaging conditions for weak beam (WB) studies of dislocations in Ti_3Al with intermediate voltage transmission electron microscopes have been derived following a theoretical examination of the traditional criteria for WB-microscopy. The cores of $\langle 2c+a \rangle$ -dislocations in room temperature deformed Ti_3Al have been characterised in a case study with 300kV electrons by experimental WB-microscopy, which used imaging conditions not satisfying the traditional WB criteria. Image simulations facilitated the determination of the corrected dislocation dissociation widths for dissociated edge character dislocations. A superpartial separation of 19.5nm has been determined from experimentally observed image peak separations of $(21 \pm 1)\text{nm}$ for the edge dislocation cores.

1. Introduction

The deformation behaviour of the intermetallic phase Ti_3Al is not very well understood on a fundamental level. Dislocation core effects are thought to be of importance in understanding the deformation behaviour of this promising material. The dark field weak beam (WB) technique of transmission electron microscopy (TEM) allows the direct observation of partial dislocations down to separations of about 1nm and has been used to deduce planar fault energies [1]. However, the interpretation of DFWB images is not always straightforward. Image artefacts like multiple image peaks from a single dislocation can result in misleading conclusions about dissociation reactions and planar fault energies [2,3,4]. Additionally the effects of elastic anisotropy and the difference between dislocation image peak positions and the real positions of the dislocation cores have to be corrected for in order to obtain accurate values of planar fault energies [5,6]. The work presented here is part of an ongoing programme investigating the influence of interstitial and Nb additions on the deformation microstructure in Ti_3Al . The purpose of this paper is to derive the "optimum" imaging conditions for WB studies of dislocations in Ti_3Al with intermediate voltage TEM, and to demonstrate the importance of image simulations to ensure correct interpretations of WB image contrast. In the next section we firstly report the requirements of WB-microscopy and then relate them to the study of dislocation cores in Ti_3Al with modern TEM. This is followed by a case study of WB-microscopy of dislocations in Ti_3Al .

2. Requirements for weak beam-microscopy

The criteria, which have to be satisfied to obtain high resolution WB images of dislocations, which are free of artefacts, have been derived for 100 kV electrons and can be summarised as following:

- (1) geometrical deviation $s_g > s_{\text{crit}} \geq 0.2 \text{ nm}^{-1}$;
- (2) dynamical deviation $w_g = s_g s_g > w_{\text{crit}} \geq 5$;
- (3) no other beams near their respective Bragg position, i.e. no other reflections significantly excited ;
- (4) use $(g \cdot b) \leq 2$ [7,8].

Criterion (1) controls the fringe spacings and the image width of strain contrast images obtained with the weak diffracted beam g , whereas criterion (2) should be satisfied in order to minimise the interaction of the weak g -beam with the transmitted beam. These two requirements for WB-microscopy apply only to the imaging diffracted beam g and the transmitted 0-beam, thus they can be regarded essentially as two-beam criteria. Satisfaction of criterion (3) prevents significant interaction of the weak g -beam with other diffracted systematic and non-systematic beams. Satisfaction of the fourth criterion avoids the complication of image interpretation by multiple image peaks from a single dislocation, which can occur for larger values of $(g \cdot b)$, even when nearly kinematical imaging conditions are obtained by satisfying the criteria (1) to (3), which is particularly significant for superdislocations with large total Burgers vectors.

Satisfying the criteria (1) to (3) for low order reflections leads to the classic $(g, 3g)$ WB-imaging condition for Cu and many other metals at 100kV. However, most modern TEM operate at optimum accelerating voltages between 200kV and 400kV, and the effects of the decrease in wavelength λ for the faster electrons on the above criteria have to be considered.

The Ewald sphere for a reflection g becomes flatter with decreasing electron wavelength. Thus, at least as far as systematic row reflections are concerned, criterion (3) becomes harder to satisfy. Moreover, imaging conditions (g, ng) with increased values of n are required to produce in sufficiently large geometrical deviations satisfying criterion (1). An additional effect of the increase in accelerating voltage is the increase of the extinction distances ξ_g for a given material, which means that for higher accelerating voltages criterion (2) will be satisfied for correspondingly smaller values of s_g than for lower voltages.

The intensity in the WB image decreases with increasing dynamical deviation w_g , as the diffracted intensity I_g is proportional to $\{1/w_g\}^2$. The image width decreases with increasing geometrical deviation s_g . Thus, it can be concluded that the optimum WB-imaging conditions (g, ng) for a given reflection and material will always be a compromise between minimisation of image width and adequate intensity for a given TEM system.

3. Results

3.1 Examination of the weak beam imaging conditions for Ti_3Al

The imaging conditions (g, ng) of WB-microscopy for a given reflection g in Ti_3Al are fully characterised by the corresponding systematic row intersection n , which in practice can be identified from the diffraction pattern. The relationships between the deviations s_g and w_g and the parameter n , i.e.

$$s_g = |g|^2 (\lambda/2) (n-1) \quad (6a)$$

and

$$w_g = \xi_g s_g \quad (6b),$$

have been used to derive the imaging conditions (g, ng) required to satisfy the criteria (1) and (2) for Ti_3Al at a range of accelerating voltages, which are summarised in table 1. Most of the imaging conditions listed in table 1 are markedly different from the classical $(g, 3g)$ WB imaging condition derived for Cu and 100 kV electrons [8]. For instance, for $g=0002$ imaging conditions $(g, 12g)$ and $(g, 6g)$ are required to satisfy the WB criteria (1) and (2) when 300 kV electrons are used. For this reflection it is virtually impossible to obtain proper WB imaging conditions satisfying criterion (1), which would allow for the highest resolution of image detail in strain contrast images. Consider on the other hand the systematic row $g=4220$ for 300 kV electrons, imaging conditions closer to the classic $(g, 3g)$, namely, $(g, 5g)$ and $(g, 2g)$, satisfy criteria (1) and (2) respectively, which should be easily achieved in practice.

As can be seen from table 1, the electron wavelength has an important influence on the WB imaging conditions as predicted in section 2, and as the accelerating voltage increases the value of n required to satisfy criterion (1) increases for the systematic row under consideration. The increased values of the extinction distance for faster electrons counteract this effect on the dynamical deviation to some extent but not completely, since the values of n required to satisfy criterion (2) increase slightly with increasing accelerating voltage.

The imaging conditions for WB microscopy in Ti_3Al are different from those commonly quoted for pure metals such as Nb, Cu or Ni, because of the relatively large lattice parameters and the relatively small scattering amplitudes of the former lighter metallic compound, which both produce large values of ξ_g .

3.2 Weak beam imaging of $\langle c \rangle$ -component dislocations

Ti_3Al deforms by dislocation slip. The deformation behaviour is generally dominated by prismatic slip of $\langle a \rangle$ -dislocations with Burgers vectors $b=1/3\langle 2110 \rangle$. Strain with components in $\langle c \rangle$ -directions is accommodated by slip of $\langle 2c+a \rangle$ -dislocations with Burgers vectors $b=1/3\langle 2116 \rangle$ on pyramidal planes,

where they occur in the dissociated form of superpartials with parallel Burgers vector $b=1/6\langle -2116 \rangle$ bounding a ribbon of antiphase [9].

As our example of WB-microscopy we chose to study $\langle 2c+a \rangle$ -dislocations in stoichiometric polycrystalline Ti_3Al , which had been deformed in compression at room temperature to true strains of about 3% in the as-cast state. A Philips CM30 operating at 300 kV was used for the experimental TEM work. The detailed analysis of the dislocation image contrast has been limited to images obtained for the $g=0002$ reflection. $\{0001\}$ was chosen, because it has the shortest extinction distance of all the reflections in Ti_3Al , which in principle allows us to obtain narrow strain contrast images of $\langle c \rangle$ -component dislocations. Furthermore, using the reflection $g=0002$ allowed for an easy distinction between $\langle a \rangle$ -dislocations and $\langle c \rangle$ -component dislocations in Ti_3Al .

3.2.1 Experimental observations

The defects shown in fig.2 have been characterised by a combination of (g, b)- and trace analysis. Their Burgers vectors have been identified to lie parallel to $[-1-126]$, whereas their line directions were found to lie close to $[-1100]$ for the widely dissociated long dislocation depicted in fig.2f), and close to $[-1-126]$ for the narrowly dissociated short dislocation dipoles of figs 2a) to 2e). Thus, the deformation microstructure in the room temperature deformed Ti_3Al comprised two distinct subsets of $\langle 2c+a \rangle$ -dislocations, namely widely dissociated edge dislocations and narrowly dissociated screw dislocation dipoles.

If image peak positions and separations are to be measured accurately from the micrographs, narrow image peaks are required. For that reason it is desirable to satisfy criterion (1), which for $g=0002$ in Ti_3Al at 300 kV requires a value of $n \geq 12$ (table 1). In addition effects of the free surfaces on the dislocations have to be avoided by measuring peak separations of line segments located centrally in sufficiently thick regions of the foil [10]. Changes of the line directions of dislocations upon approaching the free surfaces can clearly be seen for both the edge and screw dislocations (fig.2), indicative of surface effects [11].

None of the imaging conditions used to obtain the micrographs of fig.2 satisfy criterion (1). Nevertheless, reasonably narrow images, which allowed four image peaks to be discerned for each of the short screw dipoles in fig.2, were produced for values of $n > 4$. The imaging condition with $n=6.30$ satisfies criterion (2) and results in a very uniform background apart from the surface speckle. Thickness fringe contrast is absent in fig.2e), but is observed for the lower deviation WB-images (figs.2a)-d)).

The image peak separations were measured from micrographs such as that of figs. 2e) and 2f) in order to achieve highest accuracy. For the near screw character defect (projected line direction nearly parallel to $[0002]$) image peak separations of $(2.5 \pm 0.3)nm$ have been determined from the central part of the defect line in fig.2e). For the near edge character defect (projected line direction close to $[-1100]$) we deduced from fig 2f) image peak separations in the central region of the foil ranging from $(21 \pm 1)nm$ to $(25 \pm 1)nm$ respectively.

3.2.2 Contrast Simulations

The WB contrast of $\langle c \rangle$ -component dislocations in the Ti_3Al foils has been simulated for 300 kV using the dynamical theory of electron diffraction, the non-column approximation and assuming anisotropic elasticity theory. These calculations employed 6 reflections from the systematic row and used the elastic constants of $\alpha-Ti$, as the complete set of elastic constants of Ti_3Al is not yet known. This makes attempts to deduce planar fault energies from the observation of partial dislocation separations questionable. The simulated intensity profiles for imaging conditions with $3.88 \leq n \leq 6.30$ in fig.3 displayed two well separated image peaks of different heights corresponding to the dislocation cores of two edge character $\langle c+a/2 \rangle$ -superpartials separated by 19.5nm. The image peak width and the maximum peak intensity decreased with increasing deviation s_g , as predicted in section 2 and 3. A comparison of the image peak positions for the stronger and the weaker image peaks and the resulting image peak separation Δ_{obs} with the actual positions of the two superpartials and their separation Δ used in the model foils revealed the following:

- i) there was a sideways shift of the individual image peak with respect to the corresponding dislocation core position, which was qualitatively but not quantitatively the same for each of the partials,
- ii) the image peak separations Δ_{obs} , 23.4nm, 22.7nm, 22.0nm and 21.4nm for figs.2a)-d) respectively, were larger than the actual dislocation separation Δ of 19.5nm for all imaging conditions simulated, but they decreased towards the true value with increasing deviation s_g .

The sideways translation of image peaks from dislocations was dependent on the foil thickness. This effect has been reported and discussed in an earlier study for non-column approximation simulations and parallel illumination as used here[12]. The stronger image peak was shifted to a greater extent than the

weaker peak, which gave rise to the discrepancy between Δ_{obs} and Δ . The image peak separations for the widely dissociated edge character $\langle 2c+a \rangle$ -dislocations (fig.2f) overestimated the dislocation separation by up to about 20% (fig.3a).

Fig.4 shows 6-beam simulations of the contrast for a symmetrically dissociated $\langle 2c+a \rangle$ -dipole of screw character, consisting of four $\langle c+a/2 \rangle$ -superpartials separated by 2.5nm. The simulations for $g=0002$ displayed four well separated peaks, a stronger and a weaker one for each of the dissociated $\langle 2c+a \rangle$ -dislocations constituting the dipole (figs.4a) and 4c)). Upon reversal of the sign of g the relative peak heights swapped for each of the superdislocations. The simulated intensity profiles for $g=000-2$ displayed only one dominant broader peak with two or more significantly weaker peaks to either side of it (figs.4b) and 4d)), because the image peaks from the partials at the centre of the dipole configuration overlap significantly. The image peak positions in the simulations for both $g=0002$ and $g=000-2$ shifted to opposite sides with respect to the actual positions of the superpartial dislocation positions for each of the dissociated $\langle 2c+a \rangle$ -dislocations. The image peak separations Δ_{obs} overestimated the actual superpartial dislocation separations $\Delta=2.5\text{nm}$ for the screw dipole.

4. Discussion

The "optimum" WB-imaging conditions in our case study were obtained for $n=6.30$, which corresponds to values of deviation $s_g=0.097\text{nm}^{-1}$ and $w_g=5.60$ respectively. Thus, the imaging condition satisfies criterion (2), but fails to satisfy criterion (1). Nevertheless, dislocation images with narrow image peaks and a uniform background without thickness fringes were produced (fig.2e). Thickness fringes result from the interaction of the transmitted 0-beam and the diffracted g -beam. The absence of thickness fringes in the WB-image therefore indicates, that the aim of criterion (2), the minimisation of interaction between the 0- and the g -beam, has been achieved. For crystal tilts further away from the Bragg orientation for $g=0002$ than for $n=6.30$ image intensity became too low for recording. Taking into account the limits for practical exposure times set by the mechanical and electronic stability of the TEM and the sensitivity of the standard recording system we used, the above "optimum" conditions for WB-imaging are the best compromise between image width minimisation and the retention of image intensity for $g=0002$ in Ti_3Al and 300kV electrons. A similar study to ours based on observations for a nickel base alloy concluded, that for intermediate accelerating voltages in the range of 200kV to 400kV "optimum" WB-imaging conditions are obtained for a value of $s_g \geq 0.14\text{nm}^{-1}$ instead of $s_g \geq 0.20\text{nm}^{-1}$ [13]. We propose that criterion (2) should be used in preference to criterion (1) for WB-microscopy at intermediate voltages, because the satisfaction of criterion (2) is a more stringent requirement than the satisfaction of criterion (1). This can be understood in terms of the discussion of the influence of the shortened wavelength for faster electrons on the criteria (1) and (2) (see sections 2 and 3.1).

The image simulations for the edge and screw character $\langle 2c-a \rangle$ -dislocations reproduced the general contrast morphology of the experimental images very well. The quantitative agreement between simulated image peak separations and the experimentally observed image peak separations was very promising for the broader dissociated edge dislocation, but unsatisfactory for the screw dipole. This indicates that the dislocation core model for the edge dislocation is more realistic than that for the screw dislocation dipole. This is hardly surprising, because the planar arrangement of the four screw $\langle c+a/2 \rangle$ -dislocations is likely to be unstable with respect to dislocation arrangements spread out over two or more pyramidal planes resulting in S-type and Z-type dipole configurations. For the edge $\langle 2c+a \rangle$ -dislocation in room temperature deformed Ti_3Al a dissociation width of 19.5nm was deduced from the matching of the experimental dislocation contrast with image simulations. In an earlier study an image peak separation of about 20nm has been reported for dissociated $\langle 2c+a \rangle$ -dislocations [14]. The preliminary results presented in figs.3 and 4 showed that the image peak separations in WB-images of dissociated $\langle 2c+a \rangle$ -dislocations in Ti_3Al are larger than the actual dislocation separations of the model defects for the imaging conditions and foil geometries used. Thus, erroneous antiphase boundary energies would be derived for the pyramidal plane in Ti_3Al if the image peak separations rather than the the actual dislocation separations as deduced from image matching were used. The image simulations assisted the correct interpretation of the experimental WB-dislocation images obtained for the "optimum" imaging conditions without satisfying criterion (1).

5. Conclusions

- 1) The WB-imaging conditions required to satisfy the traditional WB-criteria for Ti_3Al at intermediate voltages have been derived following the theoretical predictions of Cockayne's treatment.

- 2) For "optimised" WB-microscopy of Ti_3Al at intermediate voltages the satisfaction of criterion (1) can be relaxed and narrow images can be obtained for deviations of about $s_g \geq 0.10 \text{ nm}^{-1}$, whereas the satisfaction of criterion (2) remains a more stringent requirement.
- 3) Detailed image simulations based on observations from carefully conducted experimental WB-microscopy can greatly assist the development of more realistic dislocation core models in this intermetallic material.

Fig.1: WB micrographs of dissociated $\langle 2c+a \rangle$ -dislocations in Ti_3Al for imaging conditions a) $n=3.88$, b) $n=4.20$, c) $n=5.20$ d) $n=6.09$, e) and f) $n=6.30$ with $g=0002$ and a beam direction near $[-1-450]$ for 300kV electrons.

Fig.2: Simulated WB intensity profiles of dissociated edge $\langle 2c+a \rangle$ -dislocation for $g=0002$ at 300kV and a) $n=3.88$, b) $n=4.20$, c) $n=5.20$ and d) $n=6.30$. Partial dislocation positions marked.

Fig.3: Simulated WB intensity profiles of a dissociated screw $\langle 2c+a \rangle$ -dipole for $n=6.30$ at 300kV for a) $g=0002$ and b) $g=000-2$. Partial dislocation positions marked.

Table 1: Imaging conditions ng required to satisfy criteria (1) / (2) for low order reflections in Ti_3Al at various accelerating voltages

reflection g	accelerating voltage in kV				
	120	150	200	300	400
0002	7g/5g	8g/5g	10g/5g	12g/6g	14g/7g
20-20	8g/3g	9g/4g	11g/4g	14g/4g	16g/7g
20-21	7g/4g	8g/4g	9g/4g	11g/5g	13g/5g
22-40	3g/2g	4g/2g	4g/2g	5g/2g	6g/2g
22-42	3g/2g	3g/2g	3g/2g	4g/2g	5g/2g

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