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Identification of planar defects in $D0_{19}$ phases using high-resolution transmission electron microscopy

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ABSTRACT

An analysis based on crystal symmetry and high-resolution transmission electron microscopy (HRTEM) is presented as a general methodology to identify planar defects on the basal planes of $D0_{19}$ compounds. As a starting point, the possible (close-packing preserving) planar defects are classified in accordance with their visibility, and the magnitude of the displacement vector on prismatic projections. Analysis of experimental HRTEM images, obtained under two different viewing conditions, followed by matching with simulated images, enables unambiguous identification to be made. The methodology is applied to planar defects observed in $D0_{19}$ Co_3W , which are identified as π rotations around $[0001]$. To the best of our knowledge these defects have not been identified before in $D0_{19}$ phases.

§ 1. INTRODUCTION

In the last two decades, extensive fundamental research has been made on structural intermetallics aiming at an implementation of these compounds in the field of transportation and service at high temperature (Pestman *et al.* 1991, Horton *et al.* 1995, Veyssire and Saada 1996, Vitek *et al.* 1996, Koch *et al.* 1997, Yoo and Fu 1997, Liu and Maziasz 1998, Yamaguchi *et al.* 2000). The high strength, good oxidation resistance and high melting point displayed by many non-cubic intermetallics make this class of materials potentially suitable for such applications. However, because moderate ductility at ambient temperatures is also demanded, research has focused mainly on the non-cubic structures closely related to cubic ones. Examples are compounds crystallizing with the hexagonal $D0_{19}$ structure (e.g. Ti_3Al), the tetragonal $D0_{22}$ structure (e.g. Al_3Ti , Ni_3V and Al_3V) and the tetragonal L1_0 structure (e.g. TiAl). The promising $D0_{19}$ has been one of the non-cubic structures most intensively investigated. In fact, this ordered hcp structure is very similar to the well-known cubic L1_2 structure of, for example, Ni_3Al , which is already being used in a variety of cast and wrought applications, some of them with high-temperature requirements (Stoloff and Liu 1996, De Hosson 1997, Nicholson and Nutting 1961). Nevertheless, the lack of ductility has invariably limited the application of non-cubic compounds and their alloys. Therefore, a better understanding of the mechanical behaviour of such systems, compared with systems based on cubic intermetallics and cubic metals, is still necessary. Classification and

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identification of extended defect structures are essential for that purpose, as these defects critically influence the ductility of non-cubic compounds (Kantha *et al.* 1994).

This work presents a general methodology of identification of planar defects in $D0_{19}$ phases based on crystal symmetry and high-resolution transmission electron microscopy (HRTEM). The planar defects occurring on the basal planes of $D0_{19}$ structures are classified in accordance with the projected displacement and with the contrast effects induced in HRTEM images. Because of the projective nature of HRTEM, information from two different viewing directions is generally required for unambiguous identification. An experimental demonstration is performed for the case of planar defects observed in Co_3W with a $D0_{19}$ structure.

§ 2. PLANAR DEFECTS IN $D0_{19}$ PHASES

The atomic arrangement in the (0002) planes of the $D0_{19}$ structure, based on the composition X_3Y , is shown in projection in figure 1. Open and full circles represent X and Y atoms respectively. Large circles represent atoms in the A layer, the plane of the paper, and small circles represent atoms in the B layer, immediately below and above the A layer. The sequence ABAB... corresponds to the $D0_{19}$ structure. Displacements preserving the close packing on the basal plane of the $D0_{19}$ structure can generate antiphase boundaries (APBs), complex intrinsic stacking faults (CISFs) and a superlattice intrinsic stacking fault (SISF), where $\mathbf{f}_{APB} = \frac{1}{6}\langle \bar{1}2\bar{1}0 \rangle$, $\mathbf{f}_{CISF} = \frac{1}{6}\langle 01\bar{1}0 \rangle$ and $\mathbf{f}_{SISF} = \frac{1}{3}\langle \bar{1}100 \rangle$ (Fagot *et al.* 1975, Umakoshi and Yamaguchi 1981, Yamaguchi *et al.* 1981a,b). Displacement vectors are also represented in figure 1.

Figure 1. Atomic arrangement in the (0002) planes of a $D0_{19}$ structure (and underlying hcp lattice). The open and full circles represent X and Y atoms respectively. The large circles represent atoms in the A layer, the plane of the paper, and the small circles represent atoms in the B layer, immediately below and above the A layer. Displacements preserving the close-packing are indicated: $\mathbf{f}_{APB} = \frac{1}{6}[\bar{1}2\bar{1}0]$, $\mathbf{f}_{CISF} = \frac{1}{6}[01\bar{1}0]$ and $\mathbf{f}_{SISF} = \frac{1}{3}[\bar{1}100]$.

All displacements of the $\frac{1}{3}\langle\bar{1}100\rangle$ family result rigorously in the same fault and can only be distinguished by identification of the dislocations bounding the planar defect. The configuration resulting from the APB displacement indicated in figure 1 can equivalently be generated by a $\frac{1}{2}[10\bar{1}0]$ vector. In the $L1_2$ structure, because of the ABCABC... stacking, the same types of vector would result in different APB configurations. However, in the $D0_{19}$ case, only identification of the bounding dislocations allows distinction between the two types of translation. In practice, the latter displacement is unlikely to occur by shear, owing to its large magnitude, but it may result from growth. Displacements perpendicular to the basal plane ($\mathbf{f} = \frac{1}{2}[0001]$) lead to extrinsic faults, which at least theoretically can also have a superlattice or complex (incorrect order at the C plane) nature. When the production of these extrinsic faults is accompanied by shear at the basal plane level, the resulting defects have both an extrinsic and an intrinsic character. Complex faults with $\mathbf{f} = \frac{1}{6}\langle 01\bar{1}3\rangle$ are formed when the displacement at the basal plane is equivalent to the passage of a Shockley dislocation ($\mathbf{b} = \frac{1}{6}\langle 01\bar{1}0\rangle$). A superlattice fault with $\mathbf{f} = \frac{1}{6}\langle 2203\rangle$ results when the displacement corresponds to the glide of a superpartial dislocation ($\mathbf{b} = \frac{1}{3}\langle\bar{1}100\rangle$). As in the SISF case, all displacements of the $\frac{1}{6}\langle 2203\rangle$ type result in the same fault.

§ 3. HIGH-RESOLUTION TRANSMISSION ELECTRON MICROSCOPY ANALYSIS

HRTEM imaging of planar defects has the following prerequisites:

- (i) The crystal on either side of the defect must be aligned to a low-index orientation to allow structural imaging with atomic (or nearly atomic) resolution.
- (ii) The defect itself must lie parallel to the incident beam, that is an edge-on configuration is essential.

In the case of planar defects on the basal plane of $D0_{19}$ structures, these requisites can only be met for $\langle 11\bar{2}0\rangle$ and $\langle 10\bar{1}0\rangle$ viewing directions. Typically, HRTEM analysis of planar defects is carried out by imaging along one suitable direction, preferably with acquisition of a through-focal series, followed by computer simulation and image matching. Nevertheless, because of the projective nature of HRTEM imaging, coherent planar defects can be invisible along certain viewing directions. This provides the means for a different identification approach, comparable with the visibility criterion used in conventional transmission electron microscopy (TEM).

The visibility of planar defects in HRTEM images is determined by the component of the displacement vector perpendicular to the viewing direction. However, in ordered structures, projected displacements equivalent to lattice vectors of the underlying disordered structure can only be detectable if disruption of long-range chemical order gives rise to sufficient intensity contrast. For example, in the case of an APB (or a CISF) on the basal plane of the $D0_{19}$ structure, it is essential to differentiate pure-X atomic columns from X/Y atomic columns, when viewing along $\langle 11\bar{2}0\rangle$ and $\langle 10\bar{1}0\rangle$ directions (see figure 1). On the other hand, detection of defects having superintrinsic character does not require chemical contrast, because the projection of the displacement vector is never equivalent to a vector of the underlying hcp lattice. Based on a sufficient chemical contrast assumption, the planar defects described in figure 1 are expected to display the general visibility conditions listed in table 1.

As can be seen in table 1, distinction between the planar defects cannot be based solely on the visibility criterion, but differentiation between the three types of fault

Table 1. Defect visibility and projected displacements along different viewing directions in HRTEM imaging (see figure 1). Quantification of one projected displacement can be enough to pinpoint an APB or a CSF (underlined displacements).

	Defect visibility ^a and projected displacement along the following viewing directions					
	$[2\bar{1}\bar{1}0]$	$[10\bar{1}0]$	$[11\bar{2}0]$	$[01\bar{1}0]$	$[\bar{1}2\bar{1}0]$	$[\bar{1}100]$
APB	V <u>$\frac{1}{2}[01\bar{1}0]$</u>	NV $\frac{1}{3}[\bar{1}2\bar{1}0]$	V <u>$\frac{1}{2}[\bar{1}100]$</u>	V $\frac{1}{6}[\bar{2}110]$	NV 0	V $\frac{1}{6}[\bar{1}1\bar{2}0]$
CSF	V $\frac{1}{3}[01\bar{1}0]$	V $\frac{1}{6}[\bar{1}2\bar{1}0]$	V <u>$\frac{1}{6}[\bar{1}100]$</u>	NV 0	V <u>$\frac{1}{6}[10\bar{1}0]$</u>	V $\frac{1}{6}[\bar{1}1\bar{2}0]$
SISF	V $\frac{1}{3}[01\bar{1}0]$	NV $\frac{1}{3}[\bar{1}2\bar{1}0]$	V $\frac{2}{3}[\bar{1}100]^b$	NV $\frac{1}{3}[\bar{2}110]$	V $\frac{1}{3}[\bar{1}010]$	NV 0

^a V, visible; NV, not visible.
^b Projected displacements of $\frac{2}{3}\langle 10\bar{1}0 \rangle$ are not distinguishable from $\frac{1}{3}\langle 10\bar{1}0 \rangle$.

can be attained with additional quantification of the projected fault vector. Generally, identification of planar defects on basal planes of D0₁₉ structures requires imaging along one of the $\langle 11\bar{2}0 \rangle$ directions and one of the $\langle 10\bar{1}0 \rangle$ directions (the mandatory 30° of tilting are achievable with most high-resolution microscopes). In some particular situations, however, the quantification of one projected displacement can be enough to pinpoint an APB or a CISF (underlined displacements in table 1). Nevertheless, distinction between purely intrinsic defects and defects having also an extrinsic component always requires further image simulation at the level of the disrupted region.

§ 4. EXPERIMENTAL DETAILS AND SIMULATION METHODS

A Co–W alloy containing 20 at.% W was prepared in an arc furnace, where commercially pure Co and W were melted together in a water-cooled Cu bed under a protective Ar atmosphere. The alloy was encapsulated in an evacuated quartz tube and heat treated at 900°C for 170 h followed by a water quench. TEM samples were prepared by grinding, dimpling and ion milling 3 mm discs to electron transparency. A JEOL 4000 EX/II, operating at 400 kV (spherical aberration coefficient, 0.97 ± 0.02 mm; defocus spread, 7.8 ± 1.4 mm; beam semiconvergence angle, 0.8 mrad) was used for the HRTEM work. To achieve a reasonable contrast level, the grey scale of the digitized experimental HRTEM images was expanded to the full range. High-resolution image simulations were performed using the MacTempas multislice software (Kilaas 1991), supported by CrystalKit. The lattice parameters of the hexagonal cell used to simulate the bulk structure were $a = 0.512$ nm and $c = 0.412$ nm (Magnelli and Westgren 1938). The supercells containing planar defects were constructed by the periodic continuation technique (Williams and Carter 1996) and their dimensions were based on the bulk unit-cell parameters. The objective aperture diameter used to obtain the experimental and simulated images was 13.1 nm^{-1} . The Debye–Waller factor and absorption coefficient used in the simulations (for both elements) were 0.005 nm^2 and 0.1 respectively.

§ 5. RESULTS AND DISCUSSION

The heat treatment induced a homogeneous precipitation of Co_3W plates on the $\{111\}$ planes of the (W) supersaturated fcc matrix. The resulting morphology, characteristic of Widmanstätten precipitation, is known to occur in the Co–W system (Adkins *et al.* 1960). The Co-rich matrix presents a hcp stacking (ϵ) in regions adjacent to the Co_3W plates and an fcc stacking (α) in intermediate regions. The interfaces are largely coherent and the phases have the following orientation relationship: $(0001)_{\text{Co}_3\text{W}} // (0001)_{\epsilon} // \{111\}_{\alpha}$; $\langle 11\bar{2}0 \rangle_{\text{Co}_3\text{W}} // \langle 11\bar{2}0 \rangle_{\epsilon} // \langle 110 \rangle_{\alpha}$. As can be seen in figure 2, the Co_3W plates exhibit frequent planar defects on (0001) planes. A

Figure 2. Many-beam image taken along $[11\bar{2}0]//[1\bar{1}0]$ featuring a typical Co_3W plate embedded in a fcc matrix (α). The Co-rich matrix presents a hcp stacking (ϵ) in regions adjacent to the Co_3W plates. The Co_3W and ϵ phases are being viewed along a $\langle 11\bar{2}0 \rangle$ direction and α is being viewed along a $\langle 110 \rangle$ direction. The precipitate plates often exhibited planar defects lying on the basal planes.

general analysis of the $\text{fcc} \rightarrow \text{D0}_{19} \text{Co}_3\text{W}$ transformation and a description of the defect formation mechanisms can be found elsewhere (Carvalho *et al.* 2000).

The chemical contrast in Co_3W images can be assessed from figure 3. This figure presents image simulations for planar defects having $\mathbf{f}_{\text{APB}} = \frac{1}{6}[\bar{1}2\bar{1}0]$ and $\mathbf{f}_{\text{CISF}} = \frac{1}{6}[01\bar{1}0]$ and being viewed respectively along $[2\bar{1}\bar{1}0]$ and $[10\bar{1}0]$. In $\langle 10\bar{1}0 \rangle$ projections, because the spacing between the atomic columns (0.128 nm along $\langle 11\bar{2}0 \rangle$ directions) is smaller than the point resolution of the microscope (0.165 nm), the structure is not completely resolved. Nevertheless, there is always a direct correspondence between intensity maxima and columns of either mixed Co and W atoms or pure Co atoms, and such chemical contrast reveals the shear displacement associated with the defects (figures 3(e) and (f) at a glancing angle). On the other hand, the chemical contrast on the simulated $\langle 11\bar{2}0 \rangle$ projections depends strongly on defocus and thickness (figures 3(b) and (c)), and under some of the simulation conditions the defects are hardly detectable (figure 3(b)). In practice, the planar defects running along the plate length invariably presented discernible segments on a broader observation field, owing to the sensitive dependence of the chemical contrast on the variations in thickness and defocus. Subsequently, the local experimental imaging conditions could be optimized for maximum chemical contrast. This enabled the identification method to be used with reliability.

Figure 4 presents a Co_3W plate bounded by ε and containing two planar defects parallel to (0001). When imaged along $[11\bar{2}0]$, these defects induced a strong contrast

Figure 3. (a) A projection of the Co_3W atomic structure viewed along $[2\bar{1}\bar{1}0]$, where an APB on a basal plane was generated by a displacement $\mathbf{f}_{\text{APB}} = \frac{1}{6}[\bar{1}2\bar{1}0]$. (d) A projection of the atomic structure viewed along $[10\bar{1}0]$ where a CISF on a basal plane was generated by a displacement $\mathbf{f}_{\text{CISF}} = \frac{1}{6}[01\bar{1}0]$. (b), (c), (e), (f) Image simulations for different sample thicknesses t and defoci Δf .

Figure 4. Part of a Co_3W ($D0_{19}$) lamella containing two stacking faults being bound by ϵ (hcp). The left image was obtained along $[1\bar{1}20]$ and the right image was obtained at the same region but along the adjacent $[01\bar{1}0]$ direction. In the low-magnification images, white open arrows indicate the position of the two stacking faults. The defects exhibit no contrast in the $[01\bar{1}0]$ image.

variation whereas, along $[10\bar{1}0]$, only a residual effect could be detected. The projected displacements, measured on the $[11\bar{2}0]$ image, correspond to $\frac{1}{3}[1\bar{1}00]$ or $\frac{2}{3}[1\bar{1}00]$, that is 0.148 or 0.256 nm for $a_{\text{hcp}} = 0.256$ nm. A comparison of the projected displacement and visibility with the conditions presented in table 1 enables us to assign a superlattice intrinsic character to the defects. It should be noted that the intrinsic component of complex faults could be associated with the same visibility and displacement conditions but not on consecutive viewing directions, one of the $\langle 11\bar{2}0 \rangle$ type and the other of the $\langle 10\bar{1}0 \rangle$ type. Similar results were obtained in all HRTEM contrast experiments performed on Co_3W plates exhibiting planar defects on (0002) planes.

Figure 5 presents magnified details of the images in figure 4. The insets show the results of image simulation, obtained for a sample thickness of 8 nm and a defocus of

Figure 5. HRTEM images of the planar defect shown in figure 4. The left image was obtained along $[11\bar{2}0]$ and the right image was obtained in the same region but along the contiguous $[01\bar{1}0]$ direction. The insets were simulated with a defocus of -12.0 nm, a thickness of 8 nm and beam tilt of 0.6 mrad: (a) SISF; (b) π rotation around $[0001]$. The white vertical bars indicate the extra periodicity induced along $[0001]$ by the beam tilt. In the $11\bar{2}0$ simulations, the bright regions correspond roughly to the atomic columns, with pure Co columns displaying enhanced brightness. In the $[01\bar{1}0]$ simulations, the white regions correspond only to pure Co columns.

–12 nm. The simulations have shown that the unexpected periodicity along [0001], present in the [11 $\bar{2}$ 0] experimental image, must have been produced by antisymmetric phase shifts induced in the diffracted beams by a slight off-axis misalignment of the incident beam. Indeed, residual beam tilt could be expected since a voltage-centre alignment, and not a coma-free alignment, was performed before obtaining the experimental images. The best matches for the bulk regions were obtained by allowing a beam tilt of 0.6 mrad in the simulations.

In figure 5(a), the image simulations were carried out using as proposed defect model a SISF. The lack of contrast in the [10 $\bar{1}$ 0] image and the projected displacement [11 $\bar{2}$ 0] in the image were correctly simulated. However, the extension of the contrast variation observed in the region of the defect is not fairly reproduced in the [11 $\bar{2}$ 0] simulation. In fact, the contrast disruption caused by the defect in the experimental [11 $\bar{2}$ 0] image seems smaller than the contrast disruption that would be produced by a SISF, as shown in the simulated image. Furthermore, the extra periodicity along [0001] induced by beam tilt (indicated by white vertical bars) is out of phase with the experimental periodicity on the left side of the fault. A plane less seems to be involved in the defect and thus a fault resulting from a $\frac{1}{6}\langle 2203 \rangle$ displacement (see § 2) should result in a better match. The insets in figure 5(b) are image simulations for such a defect under the same viewing conditions. In this case, the extent of the perturbation was properly simulated in the [11 $\bar{2}$ 0] image and a fair match was obtained. Residual beam tilt thus turned out to be beneficial because it emphasized the partly extrinsic nature of the defect.

The bright fringe associated with fault (figures 4 and 5) could not be reproduced by optimization of parameters such as defocus, beam tilt or crystal tilt. Selective grooving during sample preparation does not seem to be the cause of such intensity variation, since the effect was strictly confined to two atomic layers. Chemical heterogeneity could have induced the observed intensity change. Nevertheless, image simulations carried out with Co local enrichment showed that a Co excess as high as 30 at.% would be required to justify such behaviour. Moreover, the same level of intensity variation would have to be observed in the [10 $\bar{1}$ 0] images, which is not the case. The contrast feature observed at such rigid-body translation interface is hence most likely to be a Fresnel fringe, as Fresnel-like features are expected to occur at places where the mean inner potential changes rapidly.

A large number of the frequent planar defects occurring on the (0002) planes of the Co_3W phase were analysed in the present study. None of these defects presented an extension along [0001] compatible with a SISF nature, and all were unambiguously assigned to the defect type simulated in figure 5(b). To the best of our knowledge, these partly intrinsic and partly extrinsic superlattice defects have not been identified before in $D0_{19}$ phases. The crystallographic changes produced in the $D0_{19}$ structure can most easily be described by a π -rotation around [0001] (Carvalho *et al.* 2000) and for this reason such defects have been called π rotation faults (π RFs). The difference in atomic arrangement between the SISF ($\mathbf{f}_{\text{SISF}} = \frac{1}{3}[\bar{1}100]$) and the π RF ($\mathbf{f}_{\pi\text{RF}} = \frac{1}{6}[2203]$), when viewed along [11 $\bar{2}$ 0], can be evaluated in figure 6. The atomic columns are either of pure X or of alternating X and Y. The SISF displacement at the basal plane changes A into C and B into A positions, and the displacement involved in a π RF brings planes A back to their original position. A, B and C planes possess an $\text{L}1_2$ type of bonding configuration. Schematically, a π RF can be obtained by removing plane C (or equivalently plane B) from a SISF. As a result, the π RF causes less disruption of the correct bonding configuration than a SISF does and is

Figure 6. Atomic arrangements of a SISF and a π RF viewed along $[11\bar{2}0]$. The atomic columns are either of pure Co or of alternating Co and W. The broken lines indicate the position of the planar defects. Plane stacking is indicated by the side of the arrangements. Schematically a π RF can be obtained by removing plane C from a SISF.

probably energetically more favourable than the SISF (De Mol Van Otterloo and De Hosson 1997). One may raise the question of whether the π RF is equivalent to an extrinsic fault in D0_{19} . Indeed an extrinsic-type fault can transform to an intrinsic loop by the nucleation and sweep of a Shockley partial dislocation. As in the SISF case, the displacement vector becomes $\frac{1}{6}\langle 2203 \rangle$. However, the π RF cannot be formed by simple shear and is introduced during the growth of the Co_3W plates by mistakes in the displacive mechanism of the $\text{fcc} \rightarrow \text{D0}_{19}$ Co_3W transformation (Howe and Mahon 1989, Carvalho *et al.* 2000).

In summary, a general HRTEM method for identification of planar defects in D0_{19} phases is presented. Two different viewing conditions are required for an unambiguous identification of the intrinsic component. The complete characterization (determination of the extrinsic component) can be attained with image simulation at the level of the disrupted region. To the best of our knowledge the partly extrinsic and partly intrinsic superlattice defects observed in Co_3W , and identified as π RFs, have not been reported before.

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