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Characterization of complex planar faults in FeAl(B) alloys

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Complex planar faults were observed by diffraction contrast transmission electron microscopy in a B2 FeAl based alloy containing Ni and B. The comparison of experimental images to simulated ones revealed the detailed structure of these faults that lie on {001} planes with both in-plane and out-of-plane components of the displacement vector. It was deduced that these defects form by segregation of (B-Al vacancies) complexes on a<100> dislocations, leading to various defect structures depending on the screw or edge character of the dislocation.

1. Introduction

Alloys based on the FeAl iron aluminide intermetallic compound have been widely studied over the last three decades [1–4]. Such alloys present good mechanical properties as well as oxidation and corrosion resistance associated with a reasonable cost. However, these alloys suffer from low ductility at room temperature due to grain boundary brittleness and show a decrease in creep resistance at temperatures higher than 600 °C. Efforts to improve their mechanical properties include microalloying, grain size reduction and oxide dispersion strengthening [5]. Crimp and Vedula [6] noticed that adding boron to binary alloys changes the mode of fracture from intergranular to transgranular, thus improving ductility. In addition, they observed that the microstructure of boron doped FeAl alloys shows a considerable amount of planar features when observed by Transmission Electron Microscopy (TEM) [6]. Similar features were further observed in FeAl based alloys directly following the processing [7] of after plastic deformation [8]. The modification of dislocations and antiphase boundary (APB) structures due to the segregation of vacancies was reported in FeAl B2 alloys [9,10]. It was shown that the yield stress anomaly of FeAl compounds could be related to such interactions between vacancies and dislocation [11]. More generally, the structure of APBs in connection with vacancies and/or solute segregation and their role on the mechanical properties have been studied in various ordered intermetallic compounds such as B2 - CuZn [12], $D0_3$ - Fe_3Al [13] and $L1_2$ - Ni_3Al [14,15].

The present study focuses on defects observed in B2 - FeAl alloys, identified as planar defects lying on {001} planes and characterized by a displacement vector of the type $\mathbf{R} = \frac{a}{2}\langle 010\rangle$. Yoshimi et al. [16] first recognised that this type of defect, which they labelled Complex Planar Faults (CPF), is associated with the presence of boron. In the meantime, some debate occurred on the exact nature of these CPF, first considered as stacking faults (SF) [6,8] they were also associated with precipitates [7]. Finally, a detailed analysis of TEM contrast revealed that CPF present both characteristics of SF and APB defects [17]. Additionally Z contrast microscopy showed that the fault is aluminium depleted and it was suggested that CPF are associated with B segregation [17]. This finding was confirmed by field ion microscopy and 3D atom probe analysis [18,19]. It is worth noting that these defects were observed only in B containing FeAl based alloys with various compositions: Fe-35 at.% Al-0.5 at.%B [16], Fe-35 at.%Al-100 ppm.B [20], Fe-40 at.%Al-0.7 at.% C-0.5 at.%B [21] Fe-40 at.%Al-3.8 at.%Ni-150 ppm.B [19] and Fe-40 at.%Al-2.7 at.%Ni-150 ppm.B (present study and [20]). To summarize the characterization of the CPF as it is now stated, one can describe them as planar faults lying on {001} planes of the B2 structure and presenting both characteristics of SF and APB. The associated displacement vector of the fault is of the type $\mathbf{R} = \frac{a}{2}\langle 010\rangle$ lying in the plane of the defect, though it has been

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argued that this vector must present some out of plane component [21]. The purpose of the present paper is to characterise CPF in FeAl based B2 compounds more precisely, using conventional diffraction contrast in TEM. The comparison of experimental images to computer-simulated images has allowed us to test various configurations and to quantify the actual components of the displacement vector of the defect. The final objective of this study is to propose a structural model for the CPF and to gain some insight into the comprehension of their formation mechanisms.

2. Material and methods

The FeAl alloy used in this study, was kindly provided to us by Anna Fraczkiewicz from The School of Mines, Saint Etienne, France. The alloy was obtained by fusion under controlled atmosphere (argon) from purified electrolytic iron (C < 10 ppm, S < 10 ppm, O < 10 ppm, N < 10 ppm), aluminium of 99.99% purity and nickel of 99.99% purity. The alloy was homogenised by six successive meltings, during which pure boron was added to the melt, and finally cast in the form of a small rectangular ingot. The alloy was studied as cast and presents a single phase B2 structure with quite large grains (grain size of approximately 1 mm). The chemical composition was measured by inductively coupled plasma – mass spectroscopy analysis that gives (in at %): Fe-56.9, Al-40.6, Ni-2.7 and 150 ppm B. The alloy will be labelled FeAlNiB throughout this paper.

Samples for TEM observations were prepared by mechanical polishing until reaching a 150 μm thick foil approximately and then electropolished by the twin jet method in a Tenupol 5 with an electrolyte consisting of 30% nitric acid and 70% methanol, at −15 °C and 10 V. The TEM characterization of the defects was made in conventional diffraction contrast using a transmission electron microscope JEOL 2010 operating at 200 kV and a double tilt specimen holder. The orientation of the selected grains was determined by searching for three low index zone axes in diffraction mode and plotting them onto a stereographic projection. This was necessary to avoid any ambiguity in the determination of geometrical and structural parameters needed for TEM image simulations. Several images of the same defect were then taken using a wide range of diffraction vectors (**g**) in bright field mode and close to the Bragg condition. The conventional bright field TEM images obtained in this work were compared to image simulations that were calculated using the multi beam image simulation program Cufour from Schäublin and Stadelmann [22]. The generic data used for the simulation presented in Figs. 3–5 are given in Table 1. The specific data such as geometry and defect configuration is detailed in the results section. The deviation from the exact Bragg condition was determined for each image from the selected area diffraction pattern and is given (in terms of excitation error amplitude $\mathbf{s_g}$) in the corresponding image captions.

3. Results and discussion

3.1. Observation of CPF using conventional TEM

TEM observations of the FeAlNiB alloy in the as-cast state indicate a high density of dislocations and the presence of planar faults, as visible in the general view from Fig. 1. Two types of dislocations are currently observed from the B2 structure of the alloy, namely rectilinear edge dislocations with $\mathbf{b} = a\langle 100\rangle$ and tangled superdislocations with $\mathbf{b} = a\langle 111\rangle$. Several planar defects corresponding to the CPF as described above are visible in this field. They correspond either to isolated extended planar faults marked with a white cross, or to more complex arrangements of faulted bows as indicated by black arrows in Fig. 1. The distribution of dislocations and CPF appears somehow uneven.

One CPF was selected in another area for a more precise analysis of the contrast in order to determine its configuration. The characterization was made by tilting experiments and using different diffracting vectors in two-beam bright field images in the TEM as showed in Fig. 2. Stereographic analysis of the defect shows that it lies on (010) plane and that the bounding dislocations are aligned along $\mathbf{u} = [100]$ direction. The visibility of the defect was analysed depending on the diffraction vector. The defect is completely invisible using $\mathbf{g} = 100$. The defect is fully visible with a 001 diffraction vector, while only the bounding dislocations show contrast with 002. In the same picture, Fig. 2b, one can also notice that both bounding dislocations present the same contrast with black and white lobes on the same side of the dislocation line at the foil emergence, as indicated with white circles. This shows that the two dislocations are of the same sign and do not constitute a dislocation dipole. Since the contrast of both dislocations is very similar, they may have the same Burgers vector. These images, and other bright field and dark field images not shown here, demonstrate that the defect corresponds to two edge partial dislocations with Burgers vector parallel to [001]. The planar fault itself exhibits features of both APB type contrast, or π type contrast, when imaged with superlattice 001 diffraction vector and SF type contrast, or α type, when imaged with fundamental $10\overline{1}$ vector. Thus, this defect actually corresponds to the CPF as proposed earlier by Yoshimi et al. [16] and Pang at al [17].

Based on this classical contrast analysis, the displacement vector of the fault, **R**, which is connected to the Burgers vector of bounding dislocations, should be $\mathbf{R} = \frac{a}{2}[001]$. However this conclusion is contradicted by the experimental image with $\overline{1}10$ diffraction vector (Fig. 2e) which shows a clear contrast for the fault despite the fact that this diffraction vector should lead to $\alpha = 2\cdot\pi\cdot\mathbf{g}\cdot\mathbf{R} = 0$. Some significant residual contrast is also visible arising from the dislocation lines. This may be explained by the fact that even if $\mathbf{g}\cdot\mathbf{b} = 0$, the secondary condition for the full extinction ($\mathbf{g}\cdot\mathbf{b}\wedge\mathbf{u} = 0$) is not fulfilled. Nevertheless such dislocation residual contrast would be limited to the vicinity of the dislocation line and could not explain the extended fault contrast as observed in Fig. 2e. In 1986, Baker and Gaydosh [7] initially noticed a similar strong residual contrast of CPF under diffraction conditions for which $\alpha = 0$. This was ascribed to a precipitation-like defect. Similar residual contrast was also clearly visible in various images from other studies [8,17]. However, this contrast was not explained and the displacement vector of the fault **R** was considered to lie in the fault plane.

The usual analysis of experimental TEM images obtained under conventional or even weak-beam diffraction contrast mode is often not sufficient to quantify the observed defects. Previous studies demonstrated that image simulations may be necessary in many cases, e.g. for accurate measurement of dissociation width [24], analysis of special contrast effects [25] or even the clear identification of dislocation configuration [26]. In order to clarify the origin of the observed contrast, experimental images of CPF were

Table 1
Input parameters used in simulations.

Elastic constants [23]: $C_{11} = 181.1$; $C_{12} = 113.7$; $C_{44} = 127.1$ GPa
Beams used for calculation: $-\mathbf{g}$, 0, $+\mathbf{g}$, $+\mathbf{2g}$
(except for the image with $\mathbf{g} = 010$ for which a strict two beam calculation - 0, $+\mathbf{g}$ - was used)