DOI: 10.1017/S1431927605504835
Microsc Microanal 11(Suppl 2), 2005

Tomographic Dark-Field TEM Observation of Ordered and Disordered Precipitates in Ni–Al–Ti Alloy

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Dark-field TEM (DF-TEM) is useful for observing lattice defects, grain boundaries, ordered domain structures, etc. However, the DF-TEM has not been utilized for electron tomography since the DF-TEM image contrast is highly sensitive to the direction of the incident electron beam. On the other hand, we found an effectiveness of a new DF-TEM tomography method controlling the tilt axis of the specimen and the direction of the incident electron beam [1]. In this study, we carried out tomographic DF-TEM observation of ordered and disordered precipitates in Ni–Al–Ti alloy [2, 3] to clarify their three-dimensional (3D) microstructures.

A solution-treated Ni–8.5 at.% Al–5.4 at.% Ti alloy was aged at 1213 K for 0.75 h to form cuboidal $L1_2$-ordered precipitates in the $A1$-disordered matrix (Fig. 1(a)). When the $A1/L1_2$ two-phase microstructure is aged at a lower temperature, 1023 K, $A1$-disordered precipitates newly appear in the $L1_2$ precipitates (Fig. 1(b)). In the course of further ageing at 1023 K, the $A1$-disordered precipitates coarsen with plate- or rod-like shapes (Fig. 1(c)), and then some of the $A1$ precipitates finally reach the $A1$ matrix surrounding the $L1_2$ precipitates (Fig. 1(d)).

In this study, the alloy specimen shown in Fig. 1(d) was placed on the specimen holder where the [001] direction is parallel to the tilt axis of the holder and the $L1_2$ superlattice reflection at $hkl = 001$ is excited for the DF-TEM imaging. While obtaining a tilt series of DF-TEM images at intervals of 2^o of the tilt angle, fine adjustments of the diffraction condition were carried out using a beam tilt function in order to optimize the image contrast (Fig. 2). A 3D image of the $A1/L1_2$ two-phase microstructure was reconstructed from the tilt series using the IMOD software [4]. Figure 3 shows the 3D image of the $A1$-disordered precipitates in the $L1_2$ precipitate viewed from three different directions. The $A1$ precipitates are in the form of plates with surfaces parallel to {100} planes and not in contact with the $A1$ matrix. This result suggests that the $A1$ precipitates are formed as if the phase separation, $L1_2 \rightarrow (L1_2 + A1)$, took place in the $L1_2$ precipitates.

In summary, the obtained result demonstrates that the DF-TEM imaging can be utilized in the electron tomography. By using this imaging method, the applicability of the electron tomography to crystalline materials will be expanded.

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[3] M. Doi et al., Superalloys 2004 (Proc. 10th Int. Symp. on Superalloys), TMS, (2004) 109.
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[5] This work was supported in part by the Grant-in-Aid for Scientific Research B from the Japan Society for the Promotion of Science, Grant-in-Aid for Exploratory Research from the Ministry of Education, Culture, Sports, Science and Technology and a grant from the Interdisciplinary Graduate School of Engineering Sciences, Kyushu University, Japan.

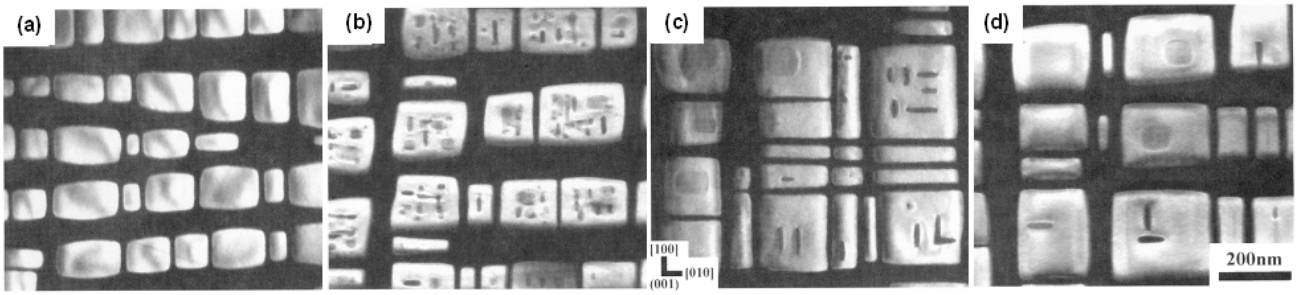

Fig. 1. TEM images showing the precipitation of $A1$ (dark areas) and $L1_2$ phases (bright areas) in Ni–8.5 at.% Al–5.4 at.% Ti alloys aged at 1213 K for 0.75 h (a), and subsequently aged at 1023 K for 12 (b), 48 (c) and 192 h (d).

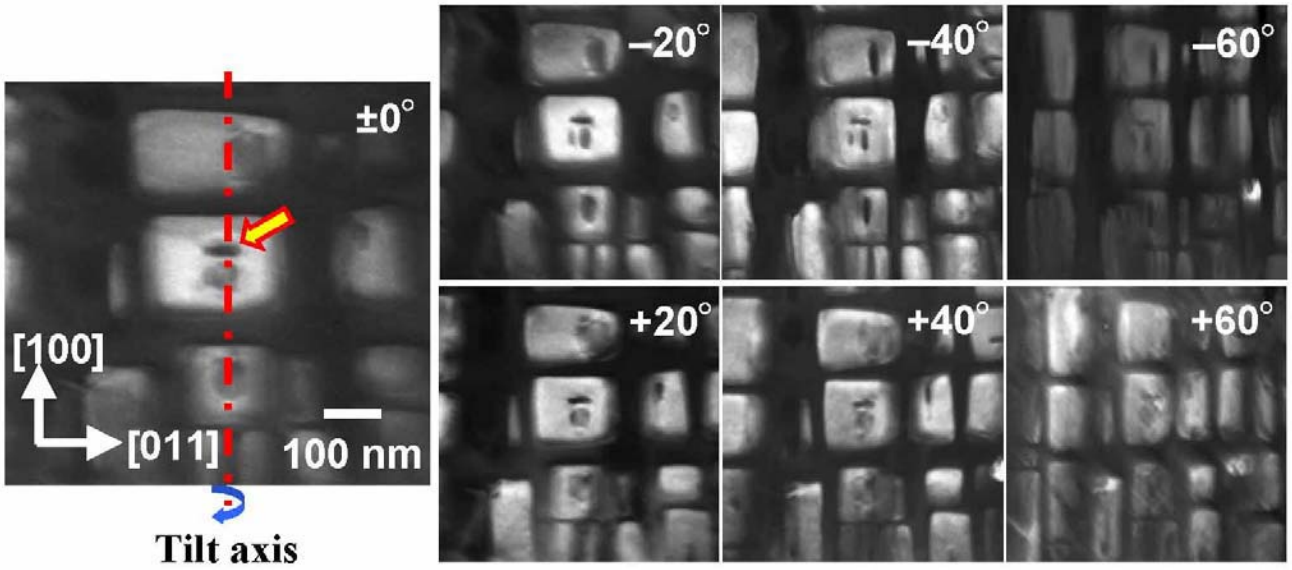

Fig. 2. Part of a tilt series of DF-TEM images of the $L1_2$-ordered phase. The $A1$-disordered precipitates formed in the $L1_2$ precipitates, as indicated by the arrow, are clearly visible for all the images.

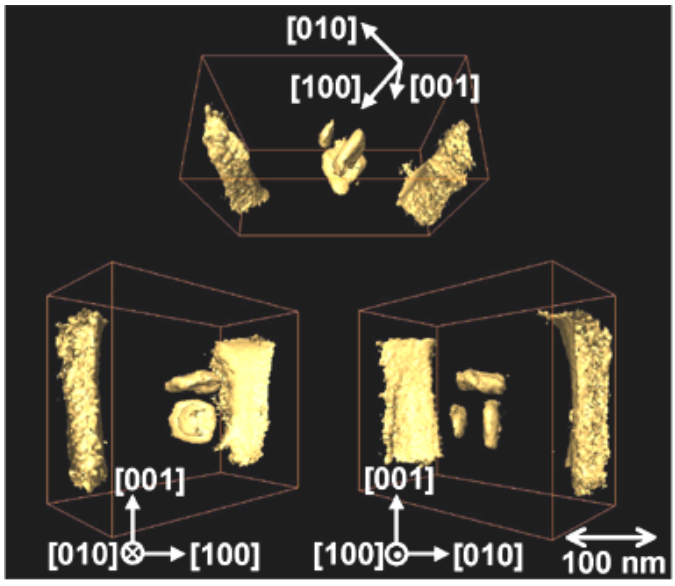

Fig. 3. Reconstructed views of the $A1$-disordered phase in the $L1_2$ precipitate made from the tilt series in Fig. 2. The dark area corresponds to the $L1_2$ precipitate.

https://doi.org/10.1017/S1431927605504835 Published online by Cambridge University Press