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Tracking the path of dislocations across ordered Al_3Zr nano-precipitates in three dimensions

W. Lefebvre,* N. Masquelier, J. Houard, R. Patte and H. Zapolsky

Groupe de Physique des Matériaux, UMR CNRS 6634, Université de Rouen, BP 12, Avenue de l'Université, 76801 Saint Etienne de Rouvray, France

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We report on the shear–bypassing transition of chemically ordered Al_3Zr nanoprecipitates. Observation of complex combinations of lattice translations, revealed by scanning transmission electron microscopy, is used as the signature of precipitate shearing during cold deformation. A method is proposed to build a three-dimensional atomic model of sheared particles from a set of three atomic scale projections. An estimation of the antiphase boundary energy of the Al_3Zr structure is achieved via the comparison of experimental findings to a model of precipitation hardening.

Keywords: Dislocation; Precipitation; Antiphase domains; HAADF

Dislocation–obstacle interactions dominate the plasticity of crystalline materials and are hence one of the major concerns in materials science. In the case of precipitation hardening [1–6], as dislocations approach precipitates, they experience attraction/repulsion forces that can significantly retard their glide. Such forces derive from a number of factors: (i) mismatch between the elastic properties of the two phases; (ii) coherency strains between the precipitate and matrix; and (iii) changes in the core structure of dislocations as they penetrate precipitates. In addition, when precipitate structures exhibit chemical order, this order may also contribute to hardening. Consequently, the theoretical description of the precipitation hardening phenomenon [5] requires a consideration of both the elastic properties of matrix and precipitate and the precipitate's nature, volume fraction and size distribution. This set of parameters will determine the mechanism of motion of dislocations across precipitates: shearing or bypassing.

The present work proposes a detailed investigation of the shearing–bypassing transition in an Al–Zr alloy system of technological interest, in which precipitation hardening is achieved by the formation of nanometre-size Al_3Zr–$L1_2$ precipitates, highly stable upon ageing [7–12]. This stability is due to the low solid solubility of Zr in aluminium, the small precipitate/matrix misfit strain and the sluggish diffusion of Zr in aluminium [7–12]. In the metastable $L1_2$ structure, Zr atoms occupy the summits of a face-centred cube whereas Al atoms' positions are at the centre of the faces. The semi-coherent $D0_{23}$ tetragonal equilibrium phase, currently forming at higher ageing and/or longer ageing times [13], was not observed in investigated materials.

The interaction between dislocations of the Al lattice and shearable Al_3Zr–$L1_2$ precipitates raises several questions. Slip systems active in aluminium at low temperature mainly consist in a combination of $\frac{1}{2}\langle 1\bar{1}0\rangle$ Burgers vectors dislocations with $\{1\,1\,1\}$ glide planes. These slip systems will induce the formation of antiphase boundaries (APBs) inside shearable precipitates. The energy required to disrupt the chemical order via the creation of APBs is expected to induce an increase in strength [5]. However, it is not clear whether the contribution of chemical order dominates for small precipitate radii. It is, however, remarkable that, for the $L1_2$ structure, an APB would disappear if two successive dislocations of identical Burgers vectors shear the precipitate along the same plane, which would be the case if only a single slip system were active. Careful three-dimensional (3-D) observations of sheared precipitates are expected to reveal whether such a coupling between dislocations exists. Beyond the particular case of Al_3Zr, the aim of this study is to provide an experimental investigation on a model system in order to compare experimental findings to theoretical predictions. Indeed, it was suggested [12,14] that in Al-based alloys precipitation

* Corresponding author. Tel.: +33 232955141.; e-mail: williams.lefebvre@univ-rouen.fr

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strengthening by $L1_2$ (Al_3Zr) phase at room temperature can occur by means of either precipitate shear or bypass, depending on the precipitate size. However, no experimental evidence of precipitate shear has been given.

The methodology applied in this study takes advantage from the observation of APBs of the $L1_2$ structure. For this purpose, precipitates are observed at the atomic scale by high angle annular dark field scanning transmission electron microscopy (HAADF-STEM) [15–19]. Use of such a technique enables the direct visualization of Zr-rich columns in the $L1_2$ structure of chemically ordered Al_3Zr precipitates. Eventual translations of Zr-rich columns induced by precipitate shearing can hence be revealed by this technique.

An Al–0.1 at.% Zr alloy was produced by continuous casting from an Al–10 wt.% Zr master alloy and pure aluminium alloy (1050). A precipitation heat treatment at 400 °C during 48 h was applied. The heat treated samples were then deformed by cold rolling up to a section reduction of 85%, corresponding to a true strain of ~61.5%. TEM samples were prepared from disks (diameter: 3 mm; thickness: 100 μm) by standard twin-jet polishing in a 2/3 methanol, 1/3 nitric acid solution at −30 °C. HAADF-STEM observations were performed on a JEOL ARM 200F microscope equipped with a Cs-probe corrector and Shottcky field emitter operating at 200 kV. The following parameters were used for the acquisition of HAADF-STEM images: probe diameter of 0.1 nm, objective aperture semi-angle of 22.5 mrad and detector half-collection angle between 40 and 150 mrad.

The structure of the precipitates was characterized by HAADF-STEM before and after the cold deformation. Before deformation, the round shaped precipitates exhibited a "perfect" $L1_2$ structure (i.e. without any APB) and were coherent with the Al matrix. Very few DO_{23} precipitates were visible along some grain boundaries. The size distribution of $L1_2$ precipitates after heat treatment at 400 °C for 48 h (prior to deformation) leads to a precipitate mean radius $\langle r \rangle$ of 2.26 ± 0.70 nm. Precipitates observed after deformation are visible in Figure 1. The absence of interface dislocations indicates that precipitates remain coherent with the Al matrix. The structure of the smallest precipitates (Fig. 1a and b) is rather complex and strongly deviates from a perfect $L1_2$ structure. Whereas the smallest precipitates clearly show defects corresponding to APBs of the $L1_2$ structure, the larger precipitates are free of those defects. More generally, over the population of 250 precipitates analysed by HAADF STEM, only precipitates smaller than a value in the range 2.4–2.8 nm are sheared.

Three distinct projections of the precipitate of Figure 1a have been recorded (see Fig. 2a–c). Based on these images, a 3-D model of the $L1_2$ precipitate has been built. For the sake of clarity, the successive shears leading to our atomic model of the deformed precipitate are detailed in Figure 3. In order to build the atomic model of the particle, an initial model was proposed to fit the [0 0 1] experimental projection. The set of dislocations and glide planes was then refined to fit both [0 0 1] and [−1 0 3] directions. Final refinement of the model was then achievable thanks to the third recorded projections. However, it was not necessary here since the agreement between experimental projection and the projection of the model particle along

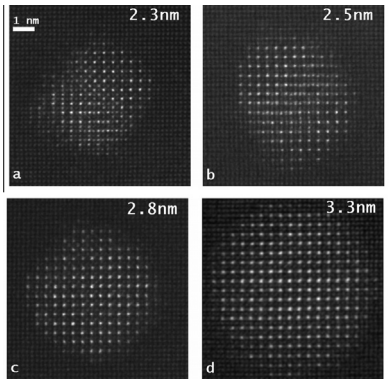

Figure 1. HAADF STEM image of different Al_3Zr precipitates of the same crystal recorded along a [0 0 1] orientation of Al. Precipitates in (a) and (b) are sheared whereas precipitates in (c) and (d) are not. All images are displayed with the same scale. The precipitate radius is indicated for each image.

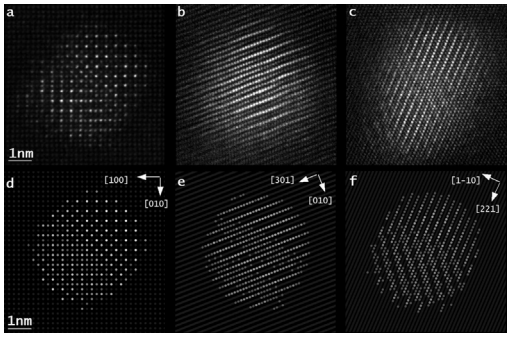

Figure 2. HAADF STEM images of the same sheared Al_3Zr precipitate recorded along three distinct orientations: (a) [0 0 1], (b) [−1 0 3] and (c) [−1 −1 4]. (d), (e) and (f) are the respective projections of the 3-D atomic model of the precipitate for each orientation. The brightness of columns in the 3-D model is proportional to the amount of Zr atoms in the column.

[−1 −1 4] was already satisfying. Projections of the same atomic model are visible in Figure 2d–f, together with the experimental ones. The similarity between the experimental observations and the projections of the model precipitate indicates that the APBs observed are the consequence of successive shears of $\frac{1}{2}\langle 1\,\bar{1}\,0\rangle$ Burgers vectors dislocations gliding on distinct $(\bar{1}\,1\,1)$ planes. A direct indication of these experimental findings is that the APBs cannot result from the beginning of a $L1_2$ to $D0_{23}$ transition. Beyond this first conclusion, the relevance of the method used in Figure 2 relies on the fact that one has complete access to the set of dislocations and glide planes involved during the deformation of the precipitate investigated. Our analysis reveals that the observed translations are perfectly consistent with the glide of five dislocations along three distinct (1 1 1) planes. The first, second and third (1 1 1) planes cross the precipitate at different heights. It is hence obvious that, in the case of this particle, successive shears do not occur in the same plane and that distinct slip systems are involved in a set of parallel planes at different depth of the particle. No particular coupling between similar dislocations was revealed here.

The observations displayed in Figure 1 clearly indicate that precipitates are either shearable or not depending on their size. More explicitly, a critical radius