

Regular article

In-situ investigation of the evolution of annealing twins in high purity aluminium

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ARTICLE INFO

Article history:

Received 22 February 2018

Received in revised form 20 April 2018

Accepted 21 April 2018

Available online xxxx

Keywords:

Aluminium

Annealing twins

In-situ annealing

EBSD

ABSTRACT

With focus on annealing twins, the microstructural evolution of cold rolled high purity aluminium was characterized in-situ during annealing using the electron backscatter diffraction technique. It was found that annealing twins developed during recrystallization. Many but not all of the twins were gradually removed during grain growth. The grain boundary energies of all the boundaries in a network associated with the twins are estimated and reasons why most twins disappear while a few remain are discussed.

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Annealing twins are frequently observed in face center cubic (FCC) metals with low or medium stacking fault energies (SFEs), i.e. Cu [1], Ag [2], and Ni [3–6]. Formation of annealing twins in these metals results in grains with a variety of orientations that can eventually grow and thus significantly affect the microstructures and textures [7]. Annealing twins in these metals have also been considered to improve properties such as corrosion [8,9] and fatigue resistance [10] due to the low energy of twin boundaries, which is the basis for grain boundary engineering [11].

Annealing twins in Al and Al alloys with high SFEs, however, are much rarer; and in fully recrystallized Al as well as in Al annealed to grain growth, hardly any twins are observed. This is particularly interesting as several authors have observed many annealing twins in the early stages of recrystallization. Key examples of this include: Berger et al. [12,13] observed a twin-relationship between the deformed and recrystallized nuclei during in-situ annealing of Al by high-voltage electron microscopy; Xu et al. [14] characterized nuclei forming during annealing beneath a microhardness tip in an Al crystal and detected nuclei with twin relationships to the deformed matrix; Humphreys and Ferry et al. [15] found formation of annealing twins related to particle-stimulated nucleation in an Al-Si alloy. It is thus a puzzle what happens with

the annealing twins formed early in the recrystallization process, how and why do they disappear. An investigation of this is the purpose of the present work.

Recently, in a preliminary investigation, Wu et al. [16] reported removal of annealing twins in Al-0.3%Mg during annealing. However, the evolution of twins and the nearby grain boundary regions were not investigated in their work. In the present study, the microstructural evolution of a cold rolled high purity Al during annealing was followed by electron backscatter diffraction (EBSD) using an in-situ heating stage in a scanning electron microscope (SEM), which allows direct observation of the evolution of annealing twins at the sample surface. It is reported that annealing twins develop more frequently at the surface than in the bulk [15]. This is considered an advantage for the present investigation.

The starting material was 99.996% purity Al cold rolled to a reduction of 25% in thickness. A sample 5 mm long (along rolling direction, RD), 3 mm thick (along normal direction, ND) and 1 mm wide (along transverse direction, TD) was cut from the rolled sheet. The RD-ND section of the sample was ground, followed by electropolishing. It was observed that the microstructure of the deformed sample is composed of very large original grains (average size of ~1 mm) containing many deformation-induced low angle boundaries (see Fig. 1a). In-situ heating was performed with a home-made heating stage in a FEI Nova 400 thermal field emission gun SEM. The sample was annealed by two heating units at the two ends of sample, and the temperature was measured by a thermocouple directly touching the backside of the sample opposite to the EBSD detector. The annealing schedule to the maximum

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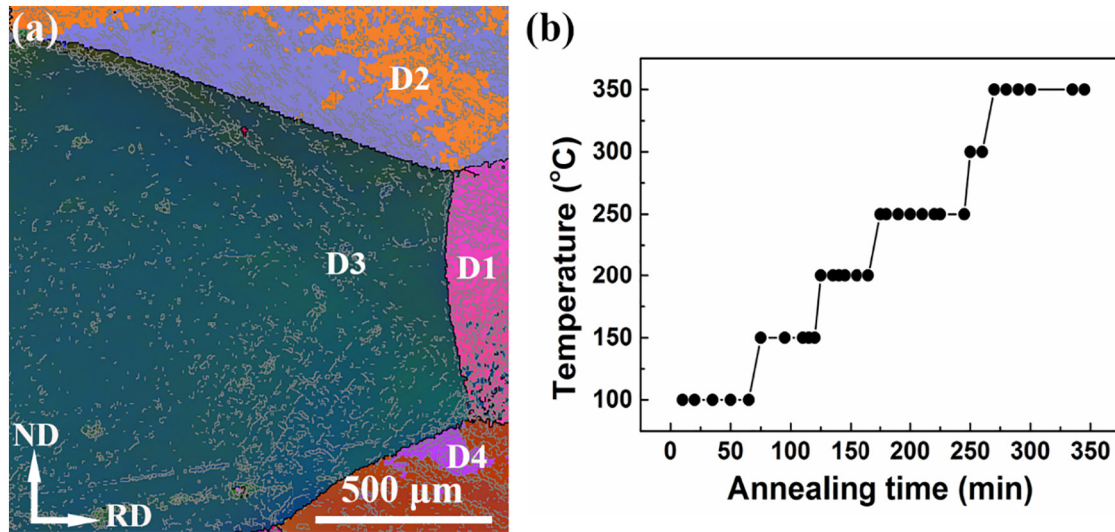


Fig. 1. (a) EBSD map of the deformed microstructure. The gray, black, and white lines in the map show boundaries with misorientation above 2° , 15° and twin boundaries, respectively. (b) The annealing sequence.

annealing temperature of 350°C and a total annealing time of approximately 6 h is illustrated in Fig. 1b. The microstructure of the sample at each annealing step was characterized by EBSD using a step size of $5\ \mu\text{m}$ to compromise between a fast acquisition time ($\sim 5\ \text{min}$) and a large sampling area ($\sim 1\ \text{mm} \times 0.8\ \text{mm}$). Twin boundaries were identified having a maximum deviation of 1.5° from the $\Sigma 3$, $60^\circ/\langle 111 \rangle$ misorientation relationship, and are shown as white lines in Figs. 2–4.

Fig. 2a shows an EBSD map of the sample after annealing at 150°C . Compared to the map in the deformed state, the observed area is shifted about $800\ \mu\text{m}$ to reveal the entire grain D1. Both annealing twins and recrystallizing grains are found to form within D1, whereas the grains D2–D4 have not yet started to recrystallize. Within D1, two twin boundaries named TB1 and TB2 with lengths of tens to hundreds of micrometers, respectively, are observed separating recrystallizing grains. Two “regular twins” labeled T3 and T4 with approximately parallel straight boundary traces have developed within the recrystallized grain R5, with one end of the twins attached to the grain boundary of R5.

Several mechanisms may have led to the formation of these twins and twin boundaries: as can be seen from Fig. 2b, the two grains, R1 and R2, are twin orientation-related; and both grains have a $\langle 111 \rangle$ type misorientation to the deformed matrix. A similar orientation

relationship is observed between the two grains R3 and R4 (separated by TB1) and the deformed matrix. In previous works [17,18], nuclei misoriented to the deformed matrix by a rotation around a common $\langle 111 \rangle$ axis have been reported, and in other works [12,13,19], it is observed that when a nucleus is formed, it may continue to grow by twinning. Both these observations may well explain the presence of TB1 and TB2 in our work. However, as each of the recrystallized grain separated by TB1 and TB2 shares a common $\langle 111 \rangle$ twinning plane with the deformed matrix, it is not possible to conclude which grain developed first.

The regular twins, T3 and T4, have an orientation within the spread of the deformed grains, D1 and D2, respectively (see Fig. 2c), and are contained within the recrystallized grain R5. There are also near twin relationships between R5 and both T3 and T4 as well as between R5 and both D1 and D2 ($50\text{--}52^\circ\langle 111 \rangle$). Because of the similarity in orientation between the twin and the deformed grain, it is very likely that the twins nucleated from the deformed grains and R5 formed by growth twinning.

As the main aim of the present work is to follow the evolution of twins as a function of recrystallization and grain growth, it is an advantage that we in the investigated area have twin boundaries separating recrystallized grain, i.e. TB1 and TB2 as well as boundaries surrounding “regular twins” i.e. T3 and T4.

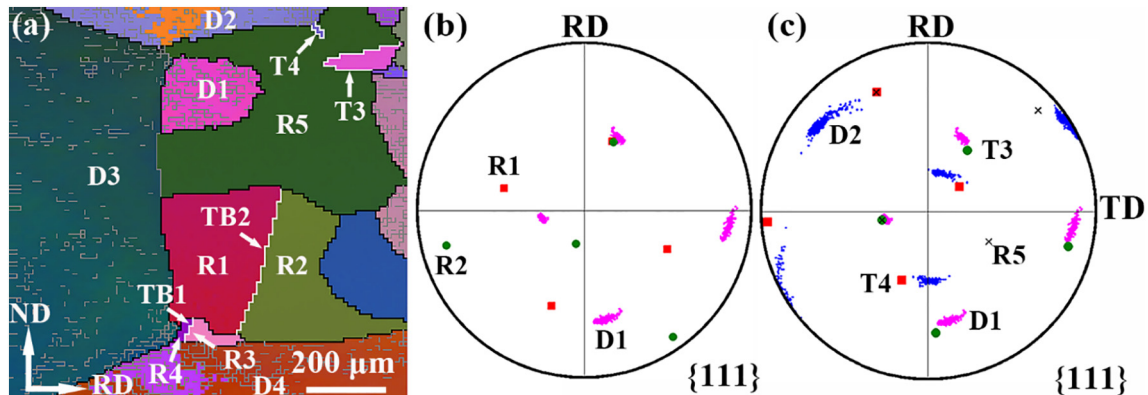


Fig. 2. (a) EBSD map of the microstructure after annealing at 150°C for 40 min. The gray, black and white lines in the map indicate grain boundaries above 2° , 15° and twin boundaries, respectively. (b) $\{111\}$ pole figure of the deformed grain D1 plus the grains R1 and R2. Pink dots represent the orientations of the deformed grain D1; red squares and green circles represent the grain R1 and R2, respectively. (c) $\{111\}$ pole figure of the deformed grains D1, D2, the recrystallizing grain R5 and the annealing twins T3 and T4. Pink and blue dots represent the orientations of the deformed grain D1 and D2, respectively; while green circles, red squares and black crosses represent the T3, T4 and R5, respectively. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

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