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Free surface effects on the recrystallization of compressed, stable, Al-Mn single crystals

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ABSTRACT

The free surface effect on the grain crystallography and morphology during recrystallization has been studied in stable Goss {110}⟨001⟩ and brass {110}⟨112⟩ - oriented single crystals of Al-1%wt.Mn alloy. The samples were plane strain compressed up to a strain of about 0.5 to develop a homogeneous structure comprising two sets of symmetrical microbands and then lightly annealed. The variation of recrystallization after annealing was then analysed by optical microscopy and SEM/EBSD on different cross-sections. The results are discussed in terms of the misorientation angle and axis distributions. The free surface provokes high frequency nucleation leading to a small grain size compared to internal sections of the bulk sample. However, the free surface and surface 'quality' do not modify the orientations of the recrystallized grains, i.e. similar groups of recrystallized grain orientations were observed for all sections. This leads to a strong misorientation relation between deformed/recovered area orientations and the limited number of groups of recrystallized grain orientations, basically the same as those observed during bulk recrystallization of similar crystals.

1. Introduction

The free surface is known to influence recrystallization experiments. It has been established that the presence of a free surface may lead to effects not observed within bulk samples, e.g. dislocation-surface interactions, relaxation of long range stresses, decreased growth rate of small grains, etc. [1–5]. This causes difficulties when comparing nucleation and grain growth in the central regions of bulk samples with those on or near the free surface, particularly during in-situ recrystallization experiments in scanning (SEM) and transmission (TEM) electron microscopes [6,7].

The influence of the free surface and surface quality on the recrystallized grain orientations is very difficult to analyse in polycrystalline metals and alloys, but the issue can be simplified by using single crystals of stable orientations. Some previous studies on the influence of the free surface on recrystallization in plane strain compressed (PSC) single crystals led to ambiguous results. In the case of bulk samples the free surface stimulates nucleation with a high density leading to finer grains as compared to the sample centre [8,9]. During annealing of TEM thin foils both free surfaces delayed nucleation and the growth of recrystallized grains is hindered, e.g. [4,10,11]. At a given temperature recrystallized grains nucleate preferentially in the thicker regions of thin foils and grain boundaries (the recrystallization front) can move forward only if the foil thickness and stored energy are sufficiently large [2,12], and the size of the as-deformed cell structure is sufficiently small [13].

It has also been established that during annealing of stable single crystals only limited groups of recrystallized grain orientations were observed [9,14] and the new grain orientations in thin foils were always enclosed within the orientation spread measured in the centre of bulk samples [14]. This non-random distribution of grain orientations, with a preferred orientation relationship across the recrystallization front [9,14,15], suggests that new orientation formation during annealing can be controlled by the orientations identified in the deformed state so that the influence of the free surface on recrystallization texture would be only small or negligible.

There is also another aspect of the free surface influence on recrystallization. It is well known that recrystallized grains can nucleate from residual surface damages introduced by sample cutting or abrading of the specimen surfaces. This means that the 'surface quality' is an important factor, which influences the intensity of nucleation and grain growth. Even though earlier studies were based on experiments with poor statistics (small number of recrystallized grains) [16–25] it is widely accepted that the orientations arising from surface damage differ from those inside bulk samples.

The subject of the present study is to analyse the orientations of

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recrystallized grains that nucleate in different sample sections, i.e. the orientations measured near the free surface (polished or roughened with an abrasion paper) are compared to those at ¼ sample depth and in the sample centre. The microstructural and textural analyses are simplified by the choice of a simple binary Al-1%wt.Mn alloy in the solid solution state. Stable single crystals of {110}⟨112⟩ and {110}⟨001⟩ orientations were deformed in channel-die to moderate strains; they develop homogeneous, dislocation microstructures and textures. This approach to the problem makes its description simpler and clearer since the stability of the orientation during deformation enables one to precisely define the orientation relationship which appears when the 'primary nucleus' is formed. Another important aspect is that the deformation of stable orientation crystallites (up to large strains) practically does not lead to the appearance of high-angle boundaries. In the absence of high-angle boundaries, the conventional mechanism of primary nucleation, based on the presence of pre-existing nuclei in the deformed structure, cannot be accepted as valid for explaining a texture change during recrystallization.

The basic research technique employed is scanning electron microscopy equipped with an electron backscattered diffraction (SEM/EBSD) facility. The results are discussed in terms of the misorientation angle and axis distributions. This enables one to precisely define the orientation relationship when the 'primary nucleus/grain' is formed.

2. Experimental

2.1. Material

The Al-1wt.%Mn alloy was obtained by melting 99.996% aluminium with high purity manganese. Single crystals were grown in a graphite mould using a horizontal solidification method from polycrystalline bars welded to an oriented seed crystal. After solidification the alloy crystal was homogenised and solutionised by a treatment of 48 h at 630 °C and air cooled. In this state, used for cold plane strain compression (PSC) tests, no precipitates were visible by high resolution SEM and none were found after deformation. However, if the cold deformed samples were aged 10 min (or longer) at 450 °C then some micron sized precipitates of Al_6Mn could be detected. It is concluded that the cold deformation was carried out in the solid solution state. The Goss (110)[00-1] and brass (110) [1-1-2] - oriented samples were carefully cut from the bars using a wire saw to dimensions of 10 mm (height) × 10 mm (width) × 10 mm (length). Crystal orientations were checked by X-ray diffraction using an X Pert PW 1830 diffractometer equipped with ATC-3 goniometer; all samples were oriented within 5° of the ideal positions.

2.2. Sample Processing and Characterization

Teflon™ lubricated samples were plane strain compressed (PSC) (Fig. 1a) at room temperature (293 K) to a reduction of 40% (logarithmic strain of 0.5). At this strain level a clear homogeneous microband structure with relatively weak spread of the initial orientation, was observed. To investigate the recrystallized grain orientations and interrelation between deformed and recrystallized textures the as-deformed samples were annealed to > 90% of recrystallized fraction on the sample surface but significantly less recrystallization within the sample interior. Therefore, the samples were annealed in an air furnace (for 480 s at 423 °C (brass) and at 413 °C (Goss)) and then water quenched. The annealing conditions were chosen in such a way that the recrystallized grains were observed on all analysed sections, but do not go beyond primary recrystallization. To solve this issue several attempts were performed. Applying low temperatures and/or short annealing times lead to a small part of recrystallized fraction on the surface, but the recrystallized grains did not appear in deeper sample sections. On the other hand higher temperatures and/or longer annealing times caused complete recrystallization across the entire sample volume. However, these annealing conditions can trigger the secondary stage of recrystallization on the sample surfaces which change the orientation of already recrystallized grains. Finally, we decided to show the results of experiments performed under the optimum conditions, as described above.

Prior to annealing, the samples were mechanically ground on all faces with 5000 grid SiC paper and electropolished. The influence of the section depth on recrystallized grain orientations and grain growth were mostly investigated in the planes perpendicular to TD and ND, i.e. in the ND/ED and ED/TD sections, where: ND, ED and TD are the normal, extension and transverse directions, respectively. A complementary study was made on the ND/TD plane. In each case entire areas of the surface, ¼ and ½ width/depth sections, (Fig. 1b), were analysed in the annealed samples by optical microscopy of anodized surfaces. Since the surfaces have undergone different mechanical states during deformation, they are denoted "top" for the compression face, "side" for the die-contact face and "end" for the free end face. In order to determine the effect of 'surface quality' (roughness) on the crystallography of recrystallization an additional test was performed: a deformed sample of the {110}⟨112⟩ orientation was scratched (on all faces) using 240 grit SiC paper prior to annealing at 420 °C for 50 s.

All microstructures and textures were characterized by a Jeol 6500F or FEI Quanta 3D SEM, using backscattered electrons at 20 kV in order to reveal the crystallographic contrast. Both SEMs were equipped with facilities for EBSD. To obtain good statistics orientations of a few hundred grains were measured, except for some central areas, where much larger grains were observed. In the case of sections perpendicular to TD the entire surface of the sample section was analysed, but in sections perpendicular to ND only half of the sample surface was characterized, as shown in Fig. 1b. For the sake of clarity each series of sections was visualized by the same colour coding, i.e. IPF || X. Notwithstanding, it leads to a small inconveniency since the matrix colour changes due to a sample reference system change. However, despite this disadvantage the colour coding used here allows to highlight the difference between the deformed and recrystallized states in the best possible way.

The as-deformed samples were also examined by TEM using a 200 kV Philips CM20 operating at the nominal voltage of 200 kV. The thin foils were prepared by a twin-jet technique using a standard solution composed of 30% nitric acid and 70% methanol, at −35 °C and a voltage of 18–22 V.

3. Results

3.1. Deformed State

The aim was to generate a homogeneous deformation microstructure throughout the samples but this turned out to somewhat overambitious since regions of both homogeneous and slightly heterogeneous deformation structures were found. The heterogeneities were two diffuse macroscopic shear bands starting in the sample corners and crossing the samples at 40°–45° to ED. The rest of the sample underwent homogeneous deformation as described below.

3.1.1. The Homogeneous Deformation Microstructure and Orientation Stability

The evolution of deformation and recrystallization microstructures and textures of Goss and brass-oriented single crystals of Al and its alloys has been extensively analysed in the past, e.g. [8,9,14,15,26–29]. For a clear correlation of the as-deformed structure with the morphology of growing grains, it is important to remember that, in the case of the brass (110) [1-1-2] orientation, microband formation could be directly correlated with the dislocation activity on the (111) and (11$\bar{1}$) planes along [10$\bar{1}$] and [011] directions, respectively (Fig. 1c). In the case of the Goss (110)[00-1] orientation two pairs of co-planar slip systems of (111)([01$\bar{1}$] + [10$\bar{1}$]) and (11$\bar{1}$)([011] + [101]) type are