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Microstructural refinement of Al before compression with oscillatory torsion process

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ABSTRACT

Purpose: The present study is aimed at a quantitative description of microstructural parameters as a average subgrain size, subgrain width and length of Al deformed by using compression test at ε= 0.8 with the next annealing. Conventional compression followed by annealing as a preliminary stage for compression with oscillatory torsion should contribute to refining grain size.

Design/methodology/approach: Al samples were compressed in room temperature to a true strain of 0.8 and next annealed at 250 °C for 1 min and 1h. Structural investigations were conducted by using light microscopy (LM) and transmission electron microscopy (TEM).

Findings: Compression leads to the formation of elongated subgrains bounded by microbands. The average subgrain size increase steadily with annealing time. The aspect ratio (R) of the grain length and the grain width is high initially especially for the as compressed materials and decreasing significantly after annealing. On annealing the deformed material, dislocation recovery occurs, leading to reductions in dislocation density both at grain boundaries and within the grains. Some of the dislocations arrange to form small eqiuaxed subboundaries.

Research limitations/implications: An increase of the contribution of grain boundary (by using method of preliminary refining structure of Al) to the deformation process by compression with oscillatory torsion may influence on more refining Al structure than compression with oscillatory torsion only.

Originality/value: Contributes to research on deformation procedure to achieve a refine structure of metals.

Keywords: Nanomaterials; Al; Microstructure; Severe plastic deformation

MATERIALS

1. Introduction

A specific feature of pure aluminum deformed via severe plastic deformation (SPD) methods is the grain refining problem to a nanometric scale [1-3]. The application of equal channel angular extrusion (ECAE) methods results in a grain size reduction to ~ 1μm [4]. Pure aluminum deformed by cyclic extrusion compression (CEC) refining to about 1.2-1.4μm [5]. hydrostatic extrusion allows to grain reduction about 700 nm [6]. Compression with oscillatory torsion ensures obtaining a structure (at selected parameters) with a mean grain size ≈ 1.6μm. Should be noted that most of the subgrains in this SPD procedure are sometimes free of dislocation in the interiors. It has been suggested that the structure is a result of recovery. Perpendicular cross section shows mixture of both equiaxed and elongated subgrains. (The effects of Al refining after mentioned test will be described elsewhere).

The using of SPD methods show the capacity for structural reconstruction in spite of a low (room) initial temperature of the material. Deformation process which accumulate considerable structural defects cause the partial recrystalization. Moreover, the process of adiabatic material heating during the deformation, favors the recrystalization phenomenon. For this reason the grain refining effect is much slower.

The structural changes of Cu presented in [7, 8] indicate, that the introduction of additional loading to the compression process, in the form of torsional moment, induces a quicker activation of different slip systems. In consequence different slip system are active. This phenomenon occurs in a much larger number of grains than in the case of samples deformed monotonically. Therefore, a subgrain structure (with micrometric scale) consisting of equiaxial subgrains is often observed in the substructure processed by compression with oscillatory torsion.

The investigations presented in [5, 9] prove that, a frequent change of the deformation path leads to high non-uniformity of deformation, which leads to the occurrence of thick deformation bands passing through a lot of grains.

Heavy cold worked metals by rolling, torsion or compression assure shear band effect too. As present in [10], the shear bands have been observed in high purity Al after high deformation degrees and within the grains with possess specific orientations. Additionally, they are favored by deformation at low temperatures. The early stages of the deformation process of pure Al lead to an almost homogeneous cell microstructure. With increase of the imposed strain, the occurrence of a single walled dense dislocation walls and microband is observed. After higher deformation degress (~ 30% in reduction) these cell blocks become very flat and have a great tendency to form compact clusters of microbands which the background for the formation of the shear bands. The mutually crossing of macro- and microbands forms rectangular or rhombohedral shape of grains. The formation of nearly equiaxial grains may be due to by using significant deformation [11], but annealing after deformation changes grains shape, as a results dynamic process of structure recovery.

The shear bands' areas are those elements of the substructure which can be controlled in order to obtain a fine-grain structure where an equiaxial grain structure of a nanometric size can be formed. In [12] argued that shear bands are privileged places of new grain nucleation, since they are built of double dislocation walls of a dimension not exceeding 0.5μm, and the misorientation inside microbands can even reach 15^o. Application of preliminary deformation initial structure by compression with next annealing should contribute to refining grain size. An increase of the contribution of grain boundary to the deformation process, and the grain boundary state may influence on refining Al structure.

The premise for an attempt at the initial grain refining was the research conducted in [13], where compression tests were performed for Fe of a diverse grain size. Structural research showed that for the grain size from 20 μm to 98 μm, the mechanism of deformation (uniform deformation) did not change, the intense shear bands' development was observed only where the grain size considerably decreased, amounting to $d \approx 300$ nm. The quantitative examination shows that the width of the generated shear bands (carrying a nanogranular structure) depends on initial grain size. This finding can be explained by the possibility of easier reorientation of a large amount of small grains into orientations responsible for shearing, whereas for the group of large grains the mechanism is hindered.

X. Huang and N. Hansen studied the microstructue of fine grained pure copper (grain size, 3.8 μm) and coarse (66.7μm) after tension at room temperature, they concluded that the grain size seems to have an effect on the evolution of microstructural parameters, e.g. boundary spacing and misorientations across the dislocation boundaries. The spacing between the dislocation boundaries appears to be smaller in the fine grained sample when compared with the coarse grained sample [14].

Microstructure of pure aluminum (99.9%) deformed by (equal channel angular drawing (ECAD) was studied by U. Chakkingal et al. [15]. They reported that the absence of high angle boundaries in the microstructure is therefore likely to be due to the extremely large initial grain size (2000μm) of the sample and the fact that the plastic deformation involved in ECAD is very uniform.

The purpose of the present study is an attempt at refining initial grain of an Al (99.7%) through the application of conventional compression followed by annealing. Prepared in this way materials will be initial state for compression with oscillatory torsion processing.

2. Experimental details

The material used was aluminum (99.7% purity). The original rods were annealed at temperature 500^oC/ 5h. After annealing average grain size was D=100μm (Fig.1). The bars with dimension 18 mm diameters and 36 mm length were compressed (by using teflon washer) in room temperature to a true strain of 0.8 and next annealed at 250^oC for 1 min and 1h. Light microscopy (LM) and transmission electron microscope (TEM) were used to examine the microstructure of specimens. For optical microscopy, metallographically polished samples were electrolitically etched with a 5% aqueous solution of HBF_4. For TEM, longitudinal sections were taken from the 0.8 value of sample radius. Foils were prepared using conventional techniques by electropolishing using: 600 ml CH_3OH, 340 ml C_4H_9OH, 60 ml 60% $HClO_4$. TEM observations were carried out at 100 kV.

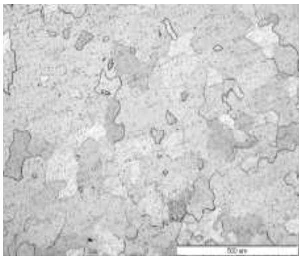
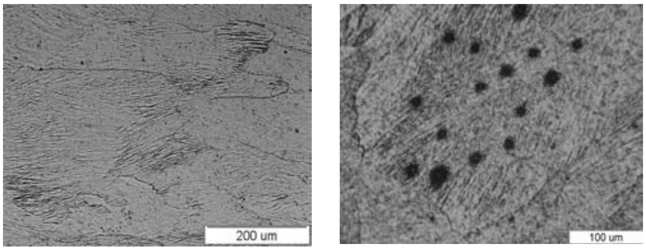


Fig. 1. Al microstructure after annealing at 500 ^oC/ 5h

Quantitative analyses were determined from TEM images. For quantitative analysis of grain size distribution more than 200 grains were measured for each specimen. Grain size is defined by the equivalent circle diameter. The (R) is a ratio of the grain length and the grain width. Hardness test were performed to characterize mechanical property by using Vickers Hardness testing ($HV_{0.05}$).

3. Results of researches

Optical microscopy investigations of Al revealers well defined slip bands and clusters of slip lines (Fig. 2) after compression test. The mean value of microhardnes was μHV5= 32.8. Observed on fig.3 elongated subgrains are family of microbands. (The nature of shear determines the evolution of this microstructure type).



a) b)

Fig. 2. a) Microstructure of Al after deformation at ε=0.8, b) μHV5= 32.8

Two types of microbands are present. The first type consist of large dislocation density (very narrow and elongated) and second type as a thin subgrain almost free of dislocation as present on Fig. 3.

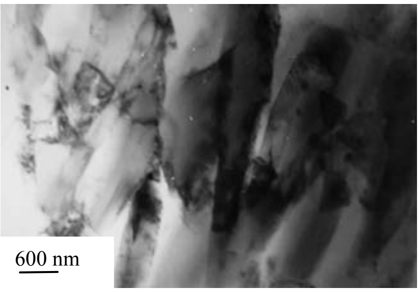


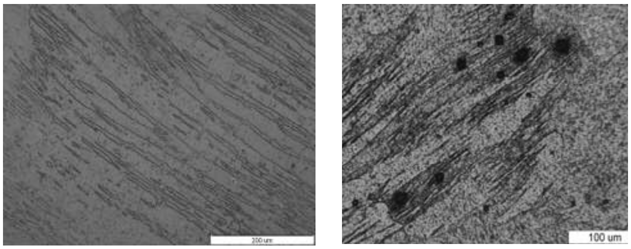
Fig. 3. Development of grains structure in Cu deformed at ε=0.8

The obtaining data from TEM measurement are summarized in Table 1. The value of (R) for compressed sample sharply increase, that is, the shape becomes more lamellar (see Table 1).

Table 1.
Structural parameters deformed and annealed Al

Material state	Average grain size (μm)	Grain width (μm)	Grain length (μm)	Grain aspect ratio R
As compression	1.38	0.95	2.85	2.99
1min at 250^{o}C	1.19	0.86	1.59	1.84
1h at 250^{o}C	1.69	1.25	2.29	1.83

In compressed and next annealing samples at 250^{o}C/ 1min there is no distinct differences in LM microstructures compared to samples compressed only (Fig.4). The microhardness having an average value of =30.1 μHV5 and is a not bit smaller compared to compressed sample. On annealing the deformed material, dislocation recovery occurs, leading to reductions in dislocation density both at grain boundaries and within the grains. During this recovery stage, some of the dislocations arrange to form small eqiuaxed subboundaries (Fig. 5).



a) b)

Fig. 4. a) Microstructure of Al after annealing at 250^{o}C/ 1min, b) μHV5= 30.2

For this reason the subgrain width and length are slightly small compared to sample compressed only. Moreover recovery process divide elongated grains lead to some reduction of grain length (see Fig. 5), for this reason the grain aspect ratio R tends towards a value 1.8 (Table 1).

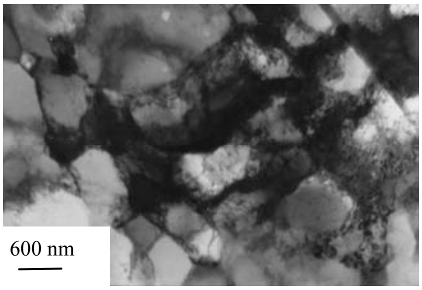
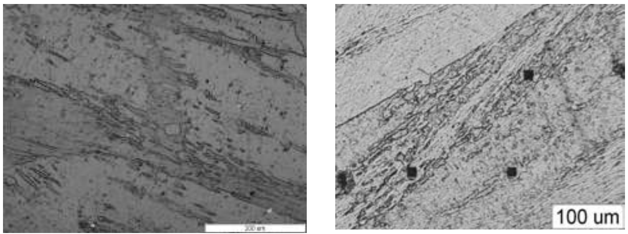


Fig. 5. Microstructure of Al after deformation followed by annealing at 250^{o}C/ 1min. Dislocations arrange to form small equiaxed subboundaries

It has been shown on fig.6 that shear bands are privileged places of new grain nucleation.



a) b)

Fig. 6. a) Microstructure of Al after annealing at 250^{o}C/ 1h, b) μHV5= 27.6. Equiaxed grain structure formed inside shear bands

At sufficiently longest times of annealing (1h at 250^{o}C) the grain size tends to increase, so the overall effect is an increase in grain width and grain length. Recovery enable formation equiaxed subgrains by the coalescence process (Fig. 7,8). Moreover recovery treatment produces a decrease in the dislocation density. The hardness of annealed materials decrease and reach value μHV5= 27.6.

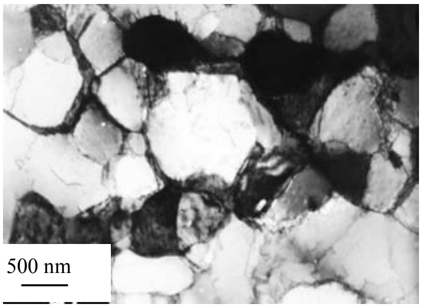


Fig. 7. Microstructure of Al after annealing at 250°C/ 1h, with equiaxed subgrains

Recovery treatment moreover influence on increasing fraction of equiaxed subgrains and a increasing population of elongated subgrains.

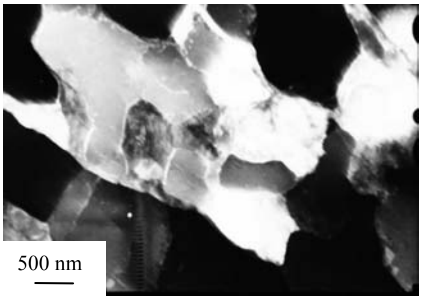


Fig. 8. Microstructure of Al after annealing at 250°C/ 1h with low and high value of misorientation between subgrains and subgrains free of dislocations (dark field)

4. Conclusions

Compression leads to the formation of largely elongated subgrains bounded by microbands. The aspect ratio (R) of the grains is about 2.99 and is high initially especially for the as compressed materials and decreasing significantly after annealing times, but never reaching the value corresponding to a truly equiaxed state. The aspect ratio of the grains are about 1.8.

The annealing reduces the hardness of material and makes the grains grow in the size. But for annealing at 250°C/ 1min average value of subgrain size is 1.19 as a results of recovery (some of the dislocations arrange to form small eqiuaxed subboundaries).

An increase of the contribution of grain boundary (yielding by using presented procedure) to the deformation process by compression with oscillatory torsion may influence on refining Al structure.

Acknowledgements

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