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Grain Size and Grain Boundary Analysis of Aluminum Compacts with/without 10 wt.% of Nickel Powder Processed by Equal Channel Angular Pressing

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The aim of this study is to consolidate the Al-Ni powders using the equal channel angular pressing method in order to achieve improved structural and mechanical properties. Thus, both alloying ability at low temperature, as a result of consolidations of ultrafine grained alloy by equal channel angular pressing method, and the average grain size and grain boundary angle have been studied and the effect of Nickel has been investigated. Severe plastic deformation and consolidation process with 15 passes of equal channel angular pressing, having route C, have been carried out at 200 °C using pure aluminum and aluminum with 10 wt.% of nickel. Electron backscatter diffraction was used to obtain scanning electron microscopy images and to determine the average grain size and average grain boundary angle. Density measurements of samples have been carried out using the Archimedes principle and the microhardness distributions have been obtained using the HV 0.5 method.

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PACS/topics: powder, aluminum and nickel, ECAP, consolidation, EBSD

1. Introduction

Low cost, lightness, resistance against corrosion, good ductility and aesthetic appearance are the most crucial properties of the Al alloys. Due to these properties, research of these alloys has been undergoing in the academic field and the potential for commercialization of these alloys has increased [1].

A considerable amount of methods and techniques have been developed to improve the properties of Al and its alloys. In particular the parameters of high purity Al have been tried to be improved with the help of such mechanisms as synthesis of solid solutions, precipitation hardening/aging, strain hardening and grain refining. Furthermore, numerous researches have been done to produce the extraordinarily fine grains (below 1 μm) with severe plastic deformation (SPD) process [2–4].

A number of studies have been done to produce metallic materials containing ultrafine grain structure using SPD process [5]. A lot of techniques have been discovered to be used in SPD process. The most effective ones, described in scientific studies, are based on equal channel angular pressing (ECAP) [6, 7], equal channel angular extrusion [8], ECAP with back-pressure [9] and ECAP with torsion [10] methods.

The aim of these methods is to obtain ultrafine grain (UFG) structure [11] and due to obtained UFG, according to Hall Petch equation [12, 13], to obtain high mechanical properties caused by the fine grains. When grains reach the fine form, high angle orientation of grains with respect to each other directly helps to prevent shear mechanism and increase dislocation density at the grain boundaries [14].

With the obtained UFG form, in methods similar to ECAP, the addition of the alloying elements to the structure enables one to have a composite structure. This way, grain investigation and composition development can be achieved at the same time and in the same structure [15].

In this study, it is aimed to obtain ultrafine grain structure using ECAP method in pure Al and Al with 10 wt.% of Ni powders. It is also aimed to obtain Al-Ni composite structure in the consolidated form.

2. Experimental study

In the study Al powders (Alfa Aesar) with average grain size of 13.74 μm and 99.5% purity and Ni powders (Alfa Aesar) with average grain size of 11.50 μm have been used. The average grain sizes of the powders were determined with Malvern laser particle size analyzer.

In this study the ECAP process was performed by using route C which has 15 passes (Fig. 1) for pure Al powder and the mixture of Al and Ni powders with 10 wt.% of Ni added into pure Al. Al and 10 wt.% Ni powders have been mixed for 15 min. in a closed container. Mixed powders have been poured into L shaped channel ECAP form from the open channel space and then packing process with 190 MPa pre-pressing has been performed.

The pressed powders have been heated to 200 °C together with the form. At this temperature samples have been transferred into the intersection canals, which have been positioned at 90° angle to the ECAP form, with pressure increasing at a constant rate of 1 mm/s. By using the ECAP method, the dispersion and consolidation

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of 10 wt.% of Ni powders in Al material and the effects of Ni powders added to Al matrix have been analyzed.

The dispersion and effect on grain size of Ni have been examined using scanning electron microscopy (SEM), electron back scatter diffraction (EBSD) analysis and X-Ray diffraction. The JEOL JSM-6060LV model was employed for SEM analysis. EDAX TSL EBSD device was used for EBSD analyses with 60 nm step interval. For X-ray diffraction analyses APD 2000 PRO XRD has been used.

With the aim of determining the average grain size and the average grain angle in EBSD analyses, the samples have been polished with 6 μm, 3 μm and 1 μm diamond paste after grinding with sandpaper with 1200 grit size. Later to be able to obtain a better super finishing, surfaces have been polished with colloidal silica suspension with grain size of 0.25 μm. Finally, the surfaces have been polished by putting them into Struers lectoPol-5 electrolytic polishing device in the solution of 5% perchloric acid ($HClO_4$), 15% butoxyethonat ($C_6H_{14}O_2$), 60% ethanol and 20% of water and keeping them in it at 22 °C and 39 V for ten seconds.

Polished surfaces have been preserved by keeping them in the silica gel in order to prevent oxidation and to keep them clean. Images have been obtained from electrons reflected from the surface and falling on detector as a result of electron bombardment under 70° angle in EBSD device.

XRD analyses have been done using Cu K_α radiation in the range of 5–70° and using 0.04° step interval. Archimedes principle has been used in density measurements of produced samples. Microhardness values have been determined in Shimadzu HMV2 microhardness measurement apparatus by applying 50 g weight with HV 0.5 techniques.

In this study, route C of the ECAP process has been applied as pressing and operation route (Fig. 1a). Metallographic and micro hardness samples were obtained from material produced by ECAP process (Fig. 1b). Samples for route C are rotated 180° after single pass. It is noted that route C type and channel intersection angle are the most effective methods for the grain refinement [16].

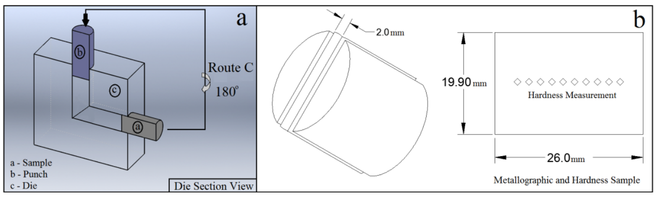

Fig. 1. ECAP process (a), metallographic and micro hardness sample (b).

3. Results and discussion

EBSD analyses of pure Al and Al+10 wt.% Ni powders after 15 passes in ECAP can be seen in Fig. 2a and b, respectively. As it is seen, with the addition of 10 wt.% of Ni an observable refinement has occurred in grains at the same transition rate. Grain refinement and changes in grain angle in the structures have been demonstrated in Fig. 3. When Fig. 3 is examined carefully it can be seen that while the average grain size of pure Al is 1.73 μm (Fig. 3a), the average grain boundary angle is 23.95° for pure Al (Fig. 3b). For Al+10 wt.% Ni alloy (Fig. 3d) the average grain size is 1.56 μm (Fig. 3c) and the average grain boundary angle is 27.71°.

It is thus demonstrated that the average grain size for Al+10 wt.% Ni alloy has been refined considerably and that grain boundary angle was increased significantly. Addition of 10 wt.% of Ni, as a result of SPD process, has increased dislocation density and has caused formation of thin and high-angle grains by preventing shear mechanism of Al grains [14]. This has been shown based on the fact that the disorientation between the grains has increased with the addition of 10 wt.% of Ni. Thus, while the amount of high-angle grain boundaries for Al is 53.18% after 15 passes, it has been found to be 62.91% for Al+10 wt.% Ni alloy after 15 passes.

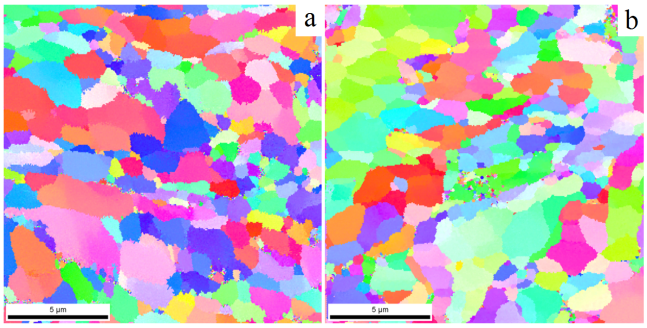

Fig. 2. EBSD images of (a) pure Al, (b) Al+10 wt.% of Ni.

Optical microscope and SEM images for pure Al and Al+10 wt.% Ni samples are presented in Fig. 4. When the images of samples produced of pure Al powder are examined (Fig. 4a and b), one can clearly see in Fig. 4a the density of deformation lines after the 15 ECAP passes applied.

As it can be seen in Fig. 4c and d, Ni particles have decreased the deformation line sizes to micro size after 15 ECAP passes with Al+10 wt.% Ni powders. Thus the optical microscope images show that Ni addition has resulted in a homogenous dispersion in the micro structure (Fig. 4c).

Comparison of relative density-hardness measurements of the samples produced with pure Al and Al+10 wt.% Ni is shown in Fig. 5a. In Fig. 5b the average grain sizes and the average grain boundary angles are compared for samples produced of pure Al and of Al+10 wt.% Ni. The XRD diagram of Al+10 wt.% Ni alloy after 15 ECAP passes is shown in Fig. 5c. While Al and Ni peaks have been obtained in the XRD diagram, no intermetallic phases have been identified in the structure of samples produced under ECAP conditions (at 200 °C).

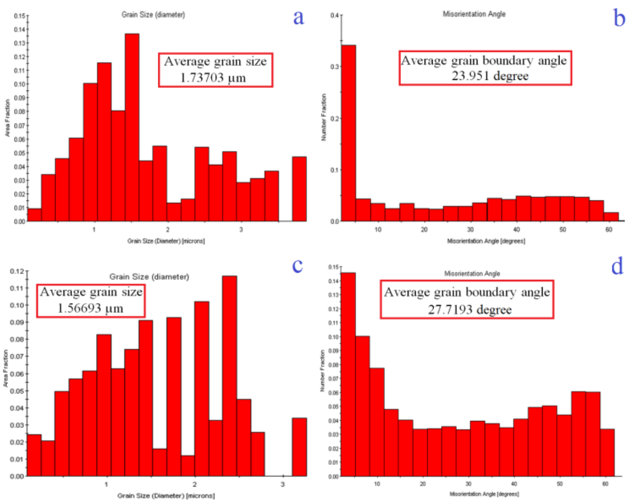

Fig. 3. Average grain sizes of (a) pure Al, (c) Al+10 wt.% Ni, average grain boundary angles of (b) pure Al and (d) Al+10 wt.% Ni.

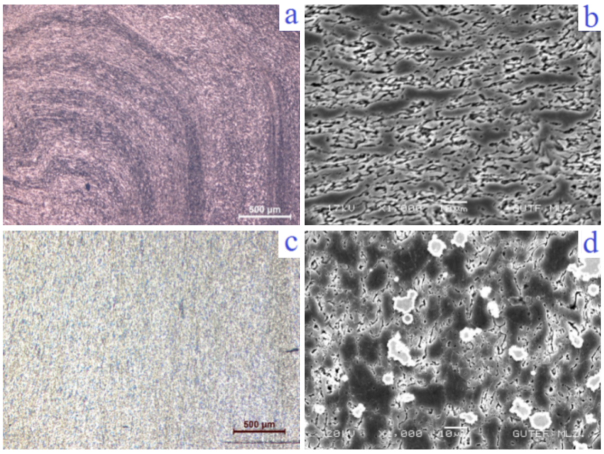

Fig. 4. Optical images of (a) pure Al, (c) Al+10 wt.% Ni, SEM images of (b) pure Al, (d) Al+10 wt.% Ni.

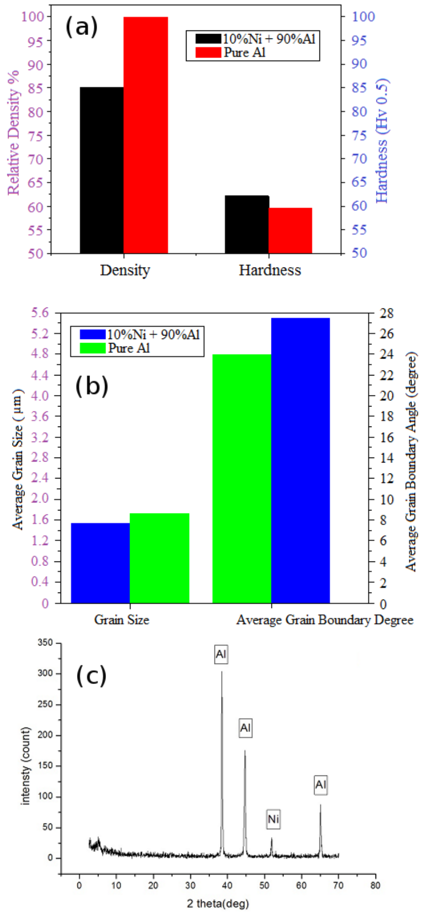

Fig. 5. (a) Relative density-hardness measurement, (b) average grain size and average grain boundary angle of pure Al and Al+10 wt.% Ni samples. Figure (c) shows XRD scan of Al+10 wt.% Ni.

The examination of Fig. 5 shows that samples containing Al+10 wt.% Ni have a smaller fine grain sizes than those of samples containing Al, increased average grain boundary angle and the increased amount of dislocations. Al+10 wt.% Ni alloy containing finer grains has higher hardness values than Al [12, 13]. Since there is no complete dissolution between Al and Ni in the structure containing Al+10 wt.% of Ni after the ECAP process, high dislocation rates in materials as a result of SPD mechanism have a direct effect on the decrease of density.

4. Conclusions

In this study, the effects of SPD process on micro structure and mechanical properties for pure Al and Al+10% Ni alloys produced using ECAP methods at 200 °C are studied. The results are summarized below:

- Finer grain size has been obtained with consolidation in pure Al and Al+10 wt.% Ni alloy as a result of SPD process containing 15 passes.
- Finer grain size structure and higher grain boundary angle have been obtained in 10 wt.% Ni alloy after 15 passes, compared to pure Al. While high-angle grain boundary amount has been 53.18% for pure Al, it has been increased up to 62.91% with the addition of 10 wt.% of Ni.
- While 99.9% density was reached with pure Al after 15 passes, with the addition of 10 wt.% of Ni the density of the alloy has decreased to 84.99%. Hardness values have been found to be 59.62 HV and 62.16 HV for pure Al and Al+10 wt.% Ni alloy, respectively.
- While deformation lines have been seen in macro images of pure Al with a severe deformation process, in Al matrix containing 10 wt.% Ni, deformation line sizes have decreased because particles have decreased their deformation line sizes in macro size.

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