Microstructure control using severe plastic deformation

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Abstract

The process of severe plastic deformation (SPD) was applied to Al alloys in order to demonstrate that it is possible to control the morphology and distribution of second phase particles using SPD. Equal-channel angular pressing (ECAP) is used for the SPD process and transmission electron microscopy is used for the microstructural observations. It is shown that precipitate particles are severely deformed or fragmented by the shear strain introduced by the ECAP process. It is also shown that the particles may even be dissolved in the matrix when the strain becomes intense. However, the change in the particle size and morphology are different depending on the alloy system.

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1. Introduction

The process of severe plastic deformation (SPD) is gaining great interest in material science because it is useful to refine microstructures to the submicrometer or nanometer levels [1]. The grain size in polycrystalline metallic materials is reduced significantly using the SPD process and, therefore, subsequent enhancements of mechanical properties such as the strength and/or ductility including advent of superplasticity have been reported [2–4]. In addition, the SPD process is effective to control the size and distribution of second phase particles. They may be fragmented to smaller sizes or dissolved into the matrix due to strong shear created by SPD. The precipitation process during aging may be accelerated due to the formation of large amount of lattice defects through SPD. In this paper, experimental results are introduced to show changes in the morphology of second phase particles following application of SPD. Examples are shown from two Al-based alloys as Al–Mg–Si and Al–Ag, both containing a fine dispersion of precipitates but behaving different ways with SPD.

The microstructural evolution is examined using transmission electron microscopy. In this study, equal-channel angular pressing (ECAP) is adopted for the SPD process.

2. Experimental procedures

2.1. ECAP

It is well known that the ECAP is a common process in SPD. As illustrated in Fig. 1, a rod-shaped sample is pressed through channels with equal cross sections meeting at an angle of \( \Phi \) [5,6]. The pressing is repeatable because there is no change in the cross-sectional dimension unlike other conventional deforming processes such as rolling, extrusion and drawing. Apparently, strain is accumulated in the sample as pressing is repeated. An important feature is that the ECAP process is applicable to bulk materials and this should be different from the powder metallurgy using ball-milling techniques. The ECAP process has been used in a wide range of materials but among them Al alloys have been often processed by ECAP.

In this study, ECAP was conducted at room temperature using a die, having a channel angle \( \Phi \) of 90° and an angle for the outer arc of curvature \( \Phi \) of 20°, which create an equivalent strain of \( \approx 1 \) for one passage through the die [6].
The sample was rotated by 90° about the longitudinal axis in the same sense between consecutive passes where this is generally designated as processing route BC [7].

2.2. Materials and microscopy

An Al alloy containing 0.57 wt% Mg and 0.37 wt% Si was melted in air and cast into an ingot with dimensions of $17 \times 55 \times 120 \text{mm}^3$. This composition is equivalent to Al–0.9 wt% Mg$_2$Si in the quasi-binary system. The cast ingot was homogenized at 843 K for 24 h and cut into bars with dimensions of $15 \times 15 \times 120 \text{mm}^3$. They were swaged into rods with diameters of 10 mm and cut to lengths of $\sim 60 \text{mm}$ for use in ECAP. All samples were solution treated at 848 K for 1 h and subsequently quenched into iced water. They were aged at 473 K for 30 h, the condition which corresponds to the peak-aged condition.

An Al–10.8 wt% Ag alloy was also prepared from high-purity (99.99%) Al and high-purity (99.9%) Ag using the same casting procedure as the Al–0.9 wt% Mg$_2$Si alloy. The ingot was homogenized at 753 K for 24 h and swaged at room temperature to obtain rods with diameters of 10 mm and lengths of $\sim 60 \text{mm}$ for ECAP. These rods were solution treated at 823 K for 1 h, followed by quenching in iced water. Aging was undertaken at a temperature of 473 K for 10 h, which corresponds to the peak-aged condition.

Microstructures were examined using conventional transmission electron microscopy (H-8100, 200 kV), high-resolution electron microscopy (JEM-4000EX, 400 kV) and analytical electron microscopy (TECNAI-20, 200 kV and JEM-2010FEF, 200 kV). The Vickers microhardness was measured for the samples subjected to ECAP. The average values were obtained from 10 measurements on each sample.

3. Results and discussion

3.1. Hardness change

Fig. 2 plots the Vickers microhardness against the number of pressings for (a) the Al–0.9 wt% Mg$_2$Si alloy and (b) the Al–10.8 wt% Ag alloy. For the former alloy, the hardness increases as the number of pressings increases but it reaches a maximum after four passes and thereafter the hardness decreases with a further increase in the number of pressings.
number of pressings. However, for the latter alloy, the hardness continuously increases with increasing number of ECAP passes and there is no decrease in the hardness at least for up to 12 passes. The hardness behavior of the Al–0.9 wt% Mg2Si alloy is quite similar to the report of Murayama et al. [8] on an Al–3.7 wt% Cu alloy. Electron microscopy observation revealed that the precipitate particles in the Al–3.7 wt% Cu alloy disappeared due to dissolution in the matrix during repetitive pressings through ECAP.

3.2. Microstructural change

Fig. 3 shows dark field images of the Al–0.9 wt% Mg2Si alloy: (a) before ECAP, (b) after one pass, (c) after four passes and (d) after eight passes. The corresponding microstructure changes are shown in Fig. 3.

Fig. 4. Bright field images with SAED patterns of Al–10.8 wt% Ag alloy: (a) after aging for 10 h at 473 K and after (b) one pass, (c) four passes and (d) eight passes of ECAP following aging for 10 h at 473 K.
selected area electron diffraction (SAED) patterns are also included. The dark field images were taken from a diffracted beam indicated by the arrows in the SAED patterns. Bright field images taken before ECAP confirmed that the particles consisted of the three orthogonal variants having rod shapes with the longitudinal axes lying along the $<100>$ orientation of the matrix, and these particles were identified as the metastable $\beta'$ phase. Before ECAP, bright contrasts appear continuously in certain lengths as in Fig. 3(a), but after one pass these lengths become discontinuous although aligned with the original lines as in Fig. 3(b), thereby suggesting that the rod-shaped particles are sectioned into several parts. After four passes shown in Fig. 3(c), the sectioned parts become separated although a few alignments remain. Most of the particles become round and distributed randomly after eight passes as shown in Fig. 3(d). It is noted that the density of the particles is decreased and this is probably due to a coherency loss between the particles and the matrix to some dissolution of particles during the process of repetitive pressings. This microstructural change appears to be consistent with the hardness change with respect to the number of pressing. In particular, the decrease in hardness for more than four passes can be due to loss of coherency with the matrix and disappearance of particles through dissolution into the matrix as reported by Murayama et al. [8] on the Al–3.7 wt% Cu alloy.

Fig. 4 shows bright field images of the Al–10.8 wt% Ag alloy together with SAED patterns (a) after aging for 10 h at 473 K but before ECAP and (b) after one pass, (c) after four passes and (d) after eight passes of ECAP following the same aging treatment. All images were taken from the [110] orientation of the matrix. The precipitation of a second phase occurred in the matrix and these precipitates correspond to the metastable $\gamma'$-phase as they are known to form in the direction parallel to the matrix {111} planes [9]. These $\gamma'$-phase precipitates are distorted after one pass of ECAP and the distortion becomes more pronounced with increasing number of ECAP passes.

Fig. 5 shows (a) an annular dark field image and the corresponding X-ray maps with (b) the Al $K_\alpha$ line and (b) the Ag $L_\alpha$ line of the Al–10.8 wt% Ag alloy, where all image and maps were taken in the scanning transmission electron microscopy (STEM) mode. The sample was subjected to four passes of ECAP after aging for 10 h at 473 K. The $\gamma'$-precipitates exhibit bright contrast in Figs. 5(a) and (c) because they are rich in Ag and dark contrast in Fig. 5(b) because they are depleted in Ag. It is again demonstrated that the precipitates were heavily deformed by ECAP and, as indicated by arrows in Figs. 5(a) and (c), there is clear evidence of an intense shear, dividing the precipitate into two parts. A close inspection of Fig. 4(c) further reveals that there are regions of Ag depletion around the precipitates. Such an Ag depletion was observed for the as-aged sample [9] and it seems that there is less effect of the severe deformation due to ECAP on the presence of the Ag-depleted zone near the precipitates. It seems also that the severe deformation does not create dissolution of Ag into the matrix unlike the cases observed in Al–0.9 wt% Mg$_2$Si and Al–3.7 wt% Cu alloys [8].

Lattice images observed by high resolution electron microscopy of the Al–10.8 wt% Ag alloy are shown in Fig. 6 where (a) is after aging for 10 h at 473 K but before ECAP and (b) after one pass and (c) after four passes. All images were taken with the beam incidence parallel to the [110] direction of the matrix. In Fig. 6(a), for the image before ECAP, dark contrasts are periodically arrayed along the interphase boundary between the $\gamma'$ precipitate and the matrix and this must be due the coherency strain formed by the lattice mismatch. Nevertheless, the
interphase boundary is smooth and lies parallel to the (1 1 1) plane of the matrix. After ECAP for one pass, as shown in Fig. 6(b), the interphase boundary is now curved and is no longer parallel to the (1 1 1) plane of the matrix. The deviation of the interphase boundary from the (1 1 1) plane is more prominent and the boundary exhibits a zigzag nature. It is important to note that a rotation of the \( \gamma' \) precipitate occurs with respect to the matrix but no rotation occurs in the crystal orientation of the \( \gamma' \) precipitate with respect to that of the matrix.

This study has shown that ECAP has different effects on the morphology of the second phase precipitates formed in the matrix. For the Al–0.9 wt% Mg\(_2\)Si alloy, the \( \beta' \) precipitates are fragmented and lose coherency with the matrix and some fraction disappears due to the dissolution in the matrix. These observations are consistent with the hardness behavior with respect to the number of ECAP passes: the hardness reaches a maximum and decreases with a further increase in the ECAP passes. For the Al–10.8 wt% Ag alloy, the \( \gamma' \) precipitates are heavily deformed by the shear strain and occasionally sheared across the precipitates. However, the crystal orientation remains the same between the precipitate and the matrix. It appears that no dissolution occurs for the Al–10.8 wt% Ag alloy.

4. Summary and conclusions

1. For the Al–0.9 wt% Mg\(_2\)Si alloy, the metastable rod-shaped \( \beta' \) particles were fragmented into round-shaped particles through shear straining by ECAP. These round particles were aligned along the longitudinal directions of the rod-shaped particles after one pass in ECAP but they became separated and randomly distributed on subsequent passes. There was also a corresponding decrease in the density of the particles, which appears to be due to a dissolution of particles with increasing numbers of passes. The maximum hardness was reached after four passes of ECAP and the hardness decreased with further passes; this is consistent with the microstructural observations.

2. For the Al–10.8 wt% Ag alloy, the hardness continuously increased with increasing numbers of ECAP passes. The metastable \( \gamma' \)-phase in the form of plate-like precipitates was heavily deformed by ECAP. No dissolution of Ag into the matrix occurred but Ag-depleted zones remained present after severe plastic deformation through ECAP.

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