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Influence of equal-channel angular pressing on aging precipitation in 7050 Al alloy

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ABSTRACT

7050 Al alloy was successfully processed by equal-channel angular pressing (ECAP) at room temperature (RT). The effect of ECAP on the subsequent aging precipitation behavior was investigated by using transmission electron microscopy (TEM) and high resolution transmission electron microscopy (HRTEM). The results reveal that the kinds, spatial distribution and sizes of precipitates in the unECAPed and the ECAPed samples are different. ECAP accelerates the process of aging precipitation and results in the broadening of precipitate size distribution. ECAP can produce not only deformation heat but also internal defects such as excess vacancies and high density of dislocations when the sample passes through the main deformation zone. Deformation heat can lead to pre-precipitation, forming a small amount of GPII zones during ECAP processing. Strain-induced excess vacancies make solute segregation along dislocations by the mechanism of nonequilibrium segregation. High density dislocations mainly accelerate the process of aging precipitation. Besides, dislocations also induce the competition between homogeneous precipitation and heterogeneous precipitation on dislocations due to the flow of solutes and vacancies towards dislocations.

1. Introduction

Severe plastic deformation (SPD) is an effective method of producing metals and alloys with ultrafine-grained and/or nanocrystalline structures [1,2]. Among various SPD techniques currently available, ECAP is one of the most attractive methods to impose extremely large plastic strain to bulk materials [1,2]. It is well known that the strengthening mechanism by ECAP is grain refinement strengthening and strain hardening. Moreover, previous studies [3,4] have shown that only one pass of ECAP is enough to improve the strength of Al alloys. More recently, there has been a growing interest in post-ECAP aging treatment for age-hardenable Al alloys to achieve a combination of strengthening from grain refinement, strain hardening and precipitation hardening [3–7].

Some researchers [8–11] have referred to the influences of plastic deformation on precipitation in age-hardenable Al alloys. Plastic deformation not only promotes the aging precipitation processes [8–10], but also forms the dynamic precipitation of GP zones during deformation [10,11]. The accelerated kinetics is mainly concerned with excess vacancies and a high density of dislocations which are generated during deformation. On the one hand, excess vacancies increases solute diffusion since there exist solute-vacancy binding energies in Al alloys [12]. On the other hand, dislocations act as nucleation sites for precipitates and provide pipe-diffusion paths [8,10].

Although the influences of plastic deformation on precipitation in age-hardenable Al alloys have been investigated [8–11], besides the influence of the vacancy and dislocation on precipitation, there may also exist other mechanisms, such as the influence of temperature rise on precipitation. Moreover, the effect of plastic deformation on aging precipitation is extremely interrelated with experimental processes (such as deformation parameters and heat treatment conditions). In the paper, therefore, we study the influence of SPD on aging precipitation in 7050 Al alloy by experimental process which is different from previous studies. A 7050 Al alloy sample was subjected to ECAP immediately after solution treatment, and then artificial aging at 160 °C for 6 h. The kinds, spatial distribution and sizes of precipitates were analyzed by using TEM and HRTEM. In addition, the mechanism of the effect of ECAP on aging precipitation was discussed in detail.

2. Experimental

The 7050 Al alloy was chosen for investigation and the nominal chemical composition of the alloy is (wt%):

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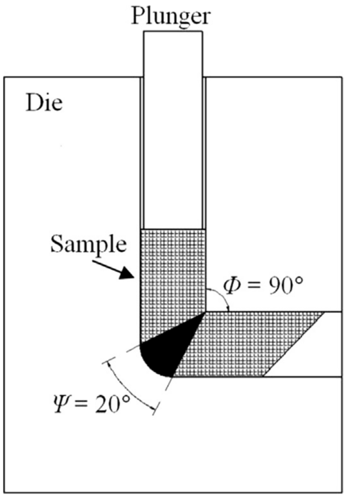

Fig. 1. Schematic illustration of the ECAP die. The main deforming zone is indicated in the black area.

Table 1
Details of ECAP and heat treatment processes used in current study.

Sample	Processing
ECAPed	Solution treatment (475 °C/1 h) + ECAP (one pass at RT) + aging treatment (160 °C/6 h)
unECAPed	Solution treatment (475 °C/1 h) + aging treatment (160 °C/6 h)

Al–6.29Zn–2.22Mg–2.28Cu–0.15Zr, with a small amount of Fe, Si, Mn, Ti and Cr. The cast ingot was homogenized at 460 °C for 24 h, and then hot rolled to a 30 mm thin plate, following by cutting into short billets with the dimension of 20 mm × 20 mm × 60 mm. These billets were solution treated at 475 °C for 1 h and subsequently water quenched. The ECAP processing was conducted at RT immediately after water quenching through a die with a plunger speed of 6 mm/s. The ECAP die has the angle of intersection of the two channels $\Phi = 90°$ and the angle subtended by the arc of curvature at the point of intersection $\Psi = 20°$, as shown in Fig. 1. The billet and the die were well lubricated using MoS_2-containing grease. The number of ECAP pass is one. For comparison purposes, both the unECAPed and the ECAPed samples were aged at 160 °C for 6 h. The full details of these two processes are summarized in Table 1. All TEM samples were subsequently prepared by mechanical grinding and punching into 3 mm disks in diameter. The disks were finally thinned using twin jet electropolishing with an electrolyte of 30% nitric acid and 70% methanol at 15 V at −25 °C. Microstructure characterization was carried out by Tecnai F30 G^2 TEM operated at 300 kV. For overviewing the grain size and grain size distribution of the alloy, electron back-scattered diffraction (EBSD) was conducted by using HKL channel 5 equipped in a Zeiss Supra 55 scanning electron microscope (SEM). The precipitate size distributions of the unECAPed and the ECAPed samples aged at 160 °C for 6 h were obtained by using VNT QuantLab-MG software.

3. Results

3.1. Microstructures after solution treatment

Fig. 2 presents an EBSD band contrast map and a bright-field (BF) TEM image showing the typical microstructure of hot-rolled 7050 Al alloy which was solution treated at 475 °C for 1 h and then water quenched to RT. One can see from the EBSD band contrast map in Fig. 2(a) that, although there exist elongated grains of 2–8 μm in width and 10–24 μm in length due to hot rolling, a lot of equiaxial grains of about 2–5 μm have been formed after the solution treatment. Fig. 2(b) shows grain size distribution histogram from Fig. 2(a). The grain equivalent diameter ranges from 2 μm to 17 μm. Fig. 2(c) shows some equiaxial grains of about 4 μm in diameter observed by TEM, and the grains have low dislocation density.

3.2. Microstructures after ECAP

Fig. 3 presents the TEM micrographs with low and high magnification, revealing the microstructure after ECAP at RT for one pass. The subgrains were apparently elongated along the shear direction. The size of the elongated grains ranges from 100 to 500 nm in the longitudinal direction, and is about 100 nm in width, as shown in Fig. 3(a). High density of dislocations was homogeneously generated in the ECAPed alloy, as shown in Fig. 3(b). Fig. 3(c) is a typical HRTEM image along <011>$_{Al}$ direction showing the morphological and crystallographic features of the GPII zones [13]. Some planar precipitates are fully coherent with the Al matrix, parallel to $\{111\}_{Al}$ planes, with the thickness of about 1–3 times of $\{111\}_{Al}$ atomic plane spacing and the length of about 4–7 nm. Additionally, weak diffraction streaks are observed along $\{111\}_{Al}$ in the Fast Fourier transformation (FFT) spectrum of Fig. 3(c), indicating the GPII zones formed during ECAP processing.

3.3. Microstructures of the samples after aging treatment

Fig. 4 shows the characterization of the precipitate microstructure of the unECAPed and the ECAPed samples aged at 160 °C for 6 h Fig. 4(a) and (b) present BF TEM images of the two samples. In the unECAPed sample, fine dispersed disk-shaped precipitates with

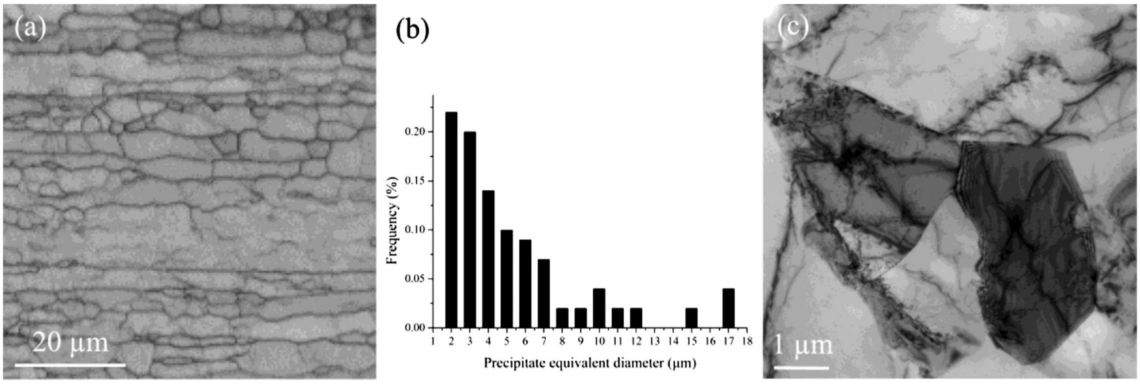

Fig. 2. EBSD band contrast map (a), grain size distribution histogram (b) and BF TEM micrograph (c), showing the grains of the 7050 Al alloy after solid solution treatment.