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Mechanical Spectroscopic Study of Equal-Channel Angular Pressed Al-Ni Eutectic Alloy

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Abstract. In this study, temperature dependence of internal friction, tanδ, of ECAPed Al-Ni eutectic alloy was measured using a dynamic mechanical analysis (DMA) to discuss the recrystallization behavior of the alloy. The temperature dependence of internal friction of 8 passed samples shows a large peak at around 200 °C on heating, while this peak disappears on the subsequent cooling stage. Moreover, smaller peak was observed for 2 passed samples. Therefore, it can be concluded that the origin of the peak comes from the recrystallization of α-Al.

Introduction

It has recently become possible at least on the laboratory scale to fabricate ultrafine grained (UFG) metallic materials with mean grain sizes smaller than 1-2 μm [1]. A lot of researches have been made to obtain UFG metallic materials with a favorable strength-toughness balance so far. Severe plastic deformation (SPD) is known to produce UFG structure [2-4], and the most popular process to fabricate UFG structure by SPD under laboratory conditions is equal channel angular pressing (ECAP) [5, 6]. Figure 1 shows a schematic diagram of the ECAP process. In case of route A, the specimen is not rotated between repetitive pressings. On the other hand, in case of route Bc, the sample was rotated by 90° about the longitudinal axis in the same sense between consecutive passes, as shown in Fig. 1. Microstructural changes by ECAP in Al and Al alloys have been studied at various temperatures [7]. In our previous study [8], the effect of texture evolution on tensile properties of ECAPed Al-Ni eutectic alloy was studied using two strain introduction methods.

Fig. 1 Schematic diagram of the ECAP process.
route Bc and route A. It was found that, fine Al$_3$Ni particles of about 300 nm were homogeneously dispersed in the matrix, and the specimens showed no clear anisotropy in tensile properties after ECAP processing by route Bc. On the other hand, after ECAP processing by route A, eutectic textures containing $\alpha$-Al and Al$_3$Ni fibrous dispersoids had a highly anisotropic distribution and were demonstrated to have significantly anisotropic tensile properties [8].

Usually, with deformation increasing, the lattice distortion energy increases, and the driving force of recrystallization increases, which causes the recrystallization softening temperature to descend [9]. Recrystallization behavior of SPDed pure Cu by multi-directional forging is reported by Nakao et al. [10]. It is reported that thermal stability of the SPDed Cu samples decreased with increasing cumulative strain. Effect of SPD on internal friction of Fe-26at%Al and Fe-26at%Al-5at%Cr alloys was studied by Pavlova et al. [11]. However, recrystallization behavior of ECAPed Al-Ni eutectic alloy is still unclear. In this study, temperature dependence of internal friction of ECAPed Al-Ni eutectic alloy was measured using a dynamic mechanical analysis (DMA) to discuss the recrystallization behavior of the alloy.

**Experimental Procedures**

Rod shaped Al–5.7 mass-%Ni alloy was prepared by casting at 850 °C using high purity Al and Al–20 mass-%Ni ingots. Cylindrical pieces were machined to give rod shaped specimens with a diameter of 10 mm and a length of 60 mm. The ECAP procedure was conducted with a pressing speed of 0.33 mm s$^{-1}$ at room temperature using MoS$_2$ as a lubricant. In the present study, ECAP was conducted by routes Bc and A, as explained above. Repeated pressings of the same specimen were carried out using up to 8 passes through the die, since in the ninth pass cracks were observed on the surfaces of specimens. A strain of 8 would be introduced into specimens after ECAP of eight passes through the die. Following the ECAP procedure, a small coupon was prepared from the as pressed Al–Ni rod specimens by electrical discharge machining. Examination of the specimens was carried out using a Hitachi S–3000 scanning electron microscope (SEM). Detailed descriptions of the sample preparation can be found in our previous work [8].

Internal friction (tan$\delta$) of the specimens was measured using a dynamic mechanical analysis (DMA) (NETZSCH DMA242C) [12, 13] in tensile mode between 0 °C and 400°C. A sinusoidal dynamic tensile stress was applied to the specimen and the resultant displacement was measured, as shown in Fig. 2. A dynamic stress ($\sigma_0$) amplitude of 5MPa was applied. The static stress (mean stress) was set to 1.3$\sigma_0$ with a constant stress of $\sigma_{\text{const}} = 0.3\sigma_0$ and a maximum of $\sigma_{\text{max}} = 2.3\sigma_0$. A dynamic stress frequency of 1Hz was selected. Tan$\delta$ was calculated from the phase lag between the applied stress and the resultant strain.

![Fig. 2 Schematic discription of the DMA measurement.](image-url)
Results and Discussion

Figures 3 (a) to (f) show the microstructures of Al-Ni alloy samples before and after 2, 4, 6, 8 passes of route Bc and 6 passes of route A, respectively. The eutectic lamellar microstructure with two phases is found for the virgin sample, as shown in Fig. 3 (a). Grey contrasted phase is the $\alpha$-Al matrix, and the other one is $\text{Al}_3\text{Ni}$ intermetallic alternating with the $\alpha$-Al matrix. As can be seen from Figs. 3 (b) to (f), fine microstructures are observed for severely deformed samples. Moreover, the ECAPed specimens by route Bc have no obvious anisotropy distribution trend of $\text{Al}_3\text{Ni}$ particles, while eutectic structure in route A sample had a highly anisotropic distribution.

![Fig. 3 Microstructures of ECAPed samples. (a): 0pass, (b): 2passes, route Bc, (c): 4passes, route Bc, (d): 6passes, route Bc, (e): 8passes, route Bc, and (f): 6passes, route A.](image)

The temperature dependence of internal friction, $\tan\delta$, of 8 passed Al-Ni alloy samples deformed by route B$_c$ is shown in Fig. 4. The measurement was made both on heating and on cooling. As can be seen, a large peak is found at around 200 °C on heating, while this peak disappears on the subsequent cooling stage, as shown in Fig. 4. Therefore, the origin of the peak may come from the recrystallization of $\alpha$-Al.

![Fig. 4 The temperature dependence of internal friction, $\tan\delta$, of 8 passed Al-Ni eutectic alloy samples (#1 and #2) deformed by route B$_c$ method.](image)
the effect of number of ECAP pass on the internal friction peak was studied, and results are shown in Fig. 5. It is found that a smaller peak was observed for 2 passed sample. This phenomenon will be discussed later.

In our previous study [8], it was found that fine Al₃Ni particles were homogeneously dispersed in the matrix after ECAP processing by route Bc, and the specimens showed no clear anisotropy in tensile properties. On the other hand, eutectic textures in the samples after ECAP processing by route A containing α-Al and Al₃Ni fibrous dispersoids had a highly anisotropic distribution and showed significantly anisotropic tensile properties [8]. Therefore, annealing behavior of the ECAPed samples by route A is different from that of by route Bc. Figure 6 shows plots of internal friction against temperature for the ECAPed samples by route A. As can be seen, internal friction peak is also found at around 200 °C even for ECAPed samples processed by route A. Moreover, the height of internal friction peak is found to increase with increasing the number of ECAP pass. In this way, annealing behavior of the ECAPed samples by routes A and Bc is similar.

In this study, internal friction peak is found at around 200 °C on heating stage, and the height of internal friction peak increases with amount of deformation increasing. To discuss the annealing behavior of ECAPed sample quantitatively, peak temperature and height of internal friction peak, \( \Delta_{\text{max}} \), are defined. Definitions of peak temperature and height of internal friction peak, \( \Delta_{\text{max}} \), are illustrated in Fig. 7.

Figure 8 (a) shows the variation of peak temperature with number of passes for samples after ECAP by routes Bc and A. It is seen that the peak temperature is not strongly affected by the number of passes and strain introduction method. Height of internal friction peak, \( \Delta_{\text{max}} \), is plotted against the
number of ECAP pass is shown in Fig. 8 (b). It must be noted here that the height of internal friction peak, \( \Delta_{\text{max}} \), increases uniformly with the number of ECAP pass, although notable difference between route A and route Bc cannot be recognized. Therefore, it can be concluded that the origin of the peak comes from the recrystallization of \( \alpha \)-Al.

**Summary**

In this study, equal channel angular pressing (ECAP) was conducted for Al-Ni eutectic alloy using two strain introduction methods, route Bc and route A. Then, temperature dependence of internal friction, \( \tan \delta \), of ECAPed was measured using a dynamic mechanical analysis (DMA) to discuss the recrystallization behavior of the severe plastic deformed (SPDed) alloy. It is found that the ECAPed samples show an internal friction peak at around 200 \(^\circ\)C on heating. However, this peak disappears on the subsequent cooling stage. The height of internal friction peak increases uniformly with the number of ECAP pass. Therefore, it can be concluded that the origin of the peak comes from the recrystallization of \( \alpha \)-Al.

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