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Annealing behavior of 5083 Al alloy deformed at cryogenic temperature

YOUNG BUM LEE
Department of Materials Science and Engineering, Kookmin University, Seoul 136-702, Korea

DONG HYUK SHIN*, WON JONG NAM
Department of Metallurgy and Materials Science, Hanyang University, Ansan, Kyunggi-Do 425-791, Korea
E-mail: wjnam@kookmin.ac.kr

Generally, the refinement of grain size in polycrystalline materials improves ductility as well as strength. Earlier works have shown that severe plastic deformation (SPD) techniques, such as equal channel angular pressing (ECAP), accumulative roll bonding (ARB), and severe torsional straining (STS), are effective in manufacturing ultrafine grained (UFG) materials [1–5]. One of the distinguishing features of UFG materials is the lower ductility than conventional grain sized materials. This unusual behavior could be explained in terms of the transition of deformation mechanisms from dislocation activity to grain boundary-related deformation [6, 7]. Meanwhile, according to Wang *et al.* [8], the excellent mechanical properties of high strength and ductility can be successfully obtained in pure Cu by deformation at liquid nitrogen temperature and annealing. They suggested that the duplex microstructure, consisting of recrystallized ultrafine grains and large coarsened grains, would give a good combination of strength and ductility, since the coarsened grains would supply sufficient deformability with pronounced strain hardening while the large volume fraction of ultrafine grains would maintain high strength. Additionally, the suppression of dynamic recovery during the deformation at cryogenic temperature would enhance the accumulated dislocation density compared with that of a material deformed at room temperature. Accordingly, the application of a cryogenic rolling process to materials would require less plastic deformation to achieve ultrafine grains than other SPD processes.

In the present work, the effects of annealing temperature and time on microstructure and mechanical properties of a 5083 Al alloy deformed at cryogenic temperature are investigated.

The material used in this work was commercial 5083 Al alloy annealed at 540 °C for 2 hr, to give equiaxed grains, with a mean size of about 90 μm. Cryogenic rolling was performed on plates, which were 10 mm in thickness, by dipping them into liquid nitrogen for at least 15 min before each rolling pass. To investigate the effect of annealing temperature, the rolled sheets with 85% reduction were annealed in the temperature range 150 ∼ 300 °C for an hour. For tensile tests, the sheets annealed at various temperatures were machined into specimens of the 25 mm gauge length. Uniaxial tensile tests were conducted with an initial strain rate of 3×10^{-3}/s on an INSTRON machine operating at the constant crosshead speed. Additionally, to investigate the effect of annealing time, the Vickers microhardness was measured under a load of 200 gf. For a detailed understanding, a transmission electron microscope (TEM) was used to analyze thin foils obtained from the materials annealed at various temperatures. Thin foils parallel to the transverse cross section of the sheets were prepared by utilizing a conventional jet polishing technique in a mixture of 75% methanol and 25% HNO_3 at a temperature of −30 °C.

The annealing at 150 °C (Fig. 1) leads to a small decrease in strength and a considerable increase in uniform elongation through the recovery process. Elongated substructure boundaries have recovered into subgrain boundaries that exhibit a distinct boundary contrast, without a noticeable change in the dimension of dislocation substructures. Meanwhile, the increase of annealing temperature to 200 °C causes only the reduction in the aspect ratio of the subgrains. The formation of transverse boundaries within subgrains and the slight increase in subgrain width during recovery would contribute to the reduction in the aspect ratio of the subgrains. Since the small grain size, including the subgrain width, would inhibit the formation of dislocation cells within grains during tensile deformation [9], the slight increase in mean free path for the movement of dislocations, without a significant decrease in dislocation density, should not cause any significant change in mechanical properties. It is interesting to note that nearly equiaxed grains, with a size similar to the subgrain width of about 200 nm, are observed at the annealing temperature of 200 °C (Fig. 2a). Although the recrystallization temperature was found to be 264 ∼ 335 °C from the differential scanning calorimeter curve (at a heating rate of 5 °C/min) in Fig. 3, annealing for an hour enhanced the occurrence of recrystallized grains at 200 °C. Although the existence of equiaxed grains, free from dislocation substructures, is known to improve ductility, the small amount of equiaxed grains produced during annealing at 200 °C was not anticipated to improve ductility effectively in the present case. The annealing at 250 °C for 3 min, Fig. 2b and c, results in a similar microstructure to annealing at 200 °C for an hour, i.e., bands of equiaxed grains and elongated subgrains, and non-recrystallized coarse grains as observed

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*Author to whom all correspondence should be addressed.

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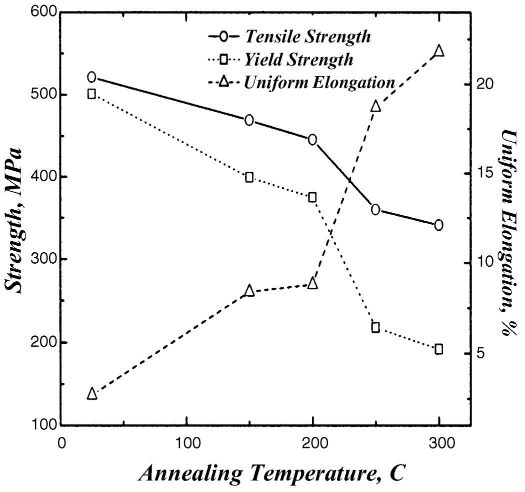

Figure 1 The effect of annealing temperture on mechanical properties, such as tensile strength, yield strength and uniform elongation in 5083 Al alloy, cryo-rolled with 85% reduction.

by Morris *et al.* [10]. With an increase in the annealing time to 9 min, an increase in the volume fraction of equiaxed grains, the coarsening of equiaxed grains within bands and an increase in the width of bands, are observed (Fig. 2d). Further annealing up to 1 hr leads to the formation of a duplex microstructure, consisting of equiaxed coarse grains and non-recrystallized grains (see Fig. 2e). Most equiaxed grains, 1.5 ∼ 2 μm in a diameter, show the characteristics of recrystallized and coarsened grains, free from dislocations, while a few equiaxed grains still retain a high dislocation density. The presence of coarse grains implies that the growth of equiaxed grains also occurs during the annealing at 200 °C for an hour. It is interesting to note that the non-recrystallized grain, marked B in Fig. 2e, consists of equiaxed grains of about 100 nm and elongated subgrains as shown in Fig. 2f.

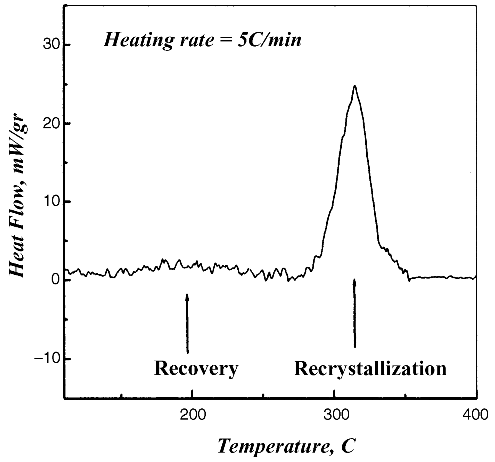

Figure 3 DSC curve for the 5083 Al alloy, cryo-rolled with 85% reduction.

Hardness deceases with increases in both temperature and time, with significant changes occurring between 150 and 300 °C (Fig. 4). It is well known that the rate of recrystallization is influenced by annealing temperature and time. In Fig. 4, lower hardness at high temperatures reflects the larger fraction of equiaxed grains. Thus, longer time is required at lower temperatures to cause the same changes as high temperature with short time. The steady hardness values above 300 °C indicates that the coarsening of recystallized grains has little influence on hardness for the annealing temperature of 300 ∼ 400 °C. Therefore, it is obvious that the hardness in the annealed samples is predominantly influenced by the fraction of recrystallized grains rather than the coarsening rate of recrystallized grains.

The significant change in mechanical properties occurs for annealing temperatures of 200 ∼ 250°C in Fig. 1. Uniform elongation significantly increases from 9 to 19% as annealing temperature increases from 200

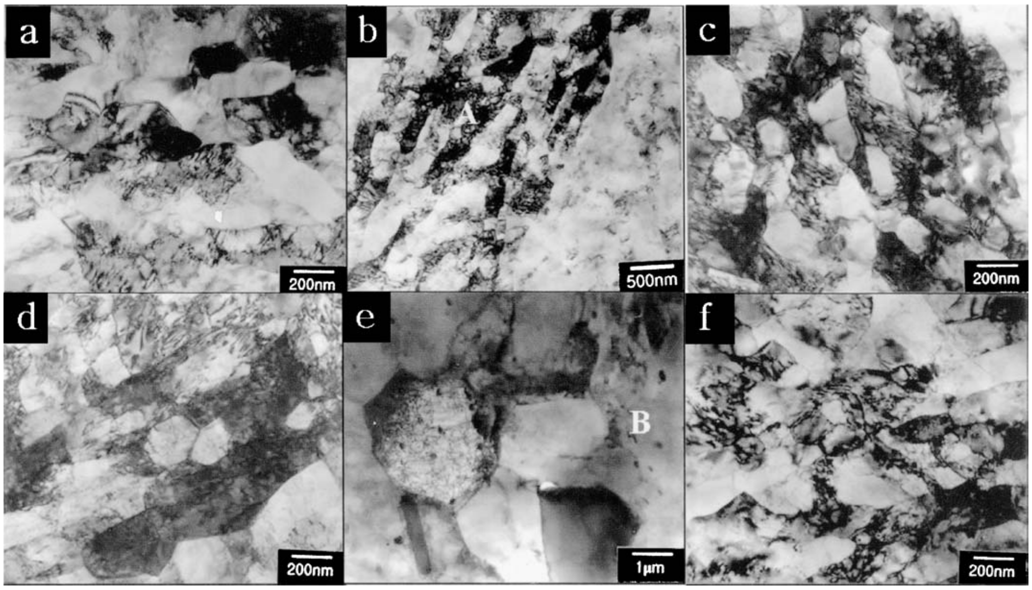

Figure 2 TEM micrographs of 5083 Al alloy, cryo-rolled with 85% reduction and annealed at various temperatures; (a) at 200 °C for 1 hr, showing the presence of equiaxed grains of about 200 nm in diameter, (b) at 250 °C for 3 min, showing bands of equiaxed grains and subgrains, and non-recrystallized coarse grains, (c) at 250 °C for 3 min, showing a mixture of equiaxed grains and subgrains (an enlargement of A in (b)), (d) at 250 °C for 9 min, (e) at 250 °C for 1 hr, showing the presence recrystallized grains of 1.5 ∼ 2 μm in diameter and non-recrystallized grains, and (f) at 250 °C for 1 hr, showing elongated and equiaxed grains in the non-recrystallized region (an enlargement of region B in (e).

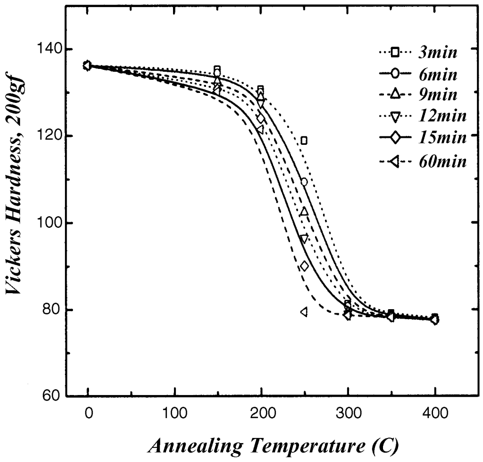

Figure 4 The effect of annealing temperature and time on hardness of 5083 Al alloy, cryo-rolled with 85% reduction.

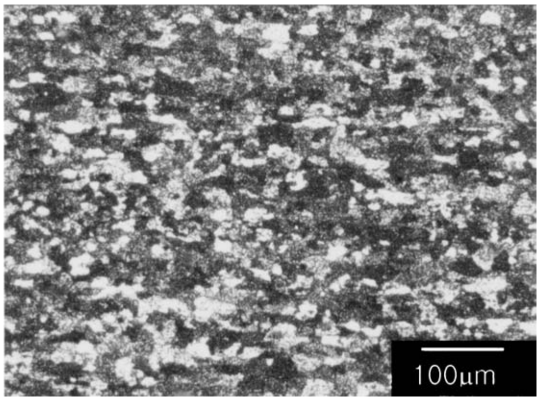

Figure 5 Optical micrograph of 5083 Al alloy, cryo-rolled with 85% reduction and annealed at 300 °C for 1 hr.

to 250 °C, while TS drops rapidly by 19%. Although the quantitative measurement of the volume fraction of recrystallized grains has not been performed in this work, it is evident that high uniform elongation in materials annealed at 250 °C could be attributed to the increased amount of recrystallized and/or coarsened grains. Meanwhile, the material annealed at 300 °C for an hour consists of recrystallized and coarsened grains of 20 μm (Fig. 5), and exhibits a low tensile strength of 340 MPa and a high uniform elongation of 22%.

From the above results, it can be concluded that mechanical properties of 5083 Al alloy deformed at cryogenic temperature and annealed are significantly influenced by the volume fraction of recrystallized grains rather than the coarsening of recrystallized grains.

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