Effect of annealing temperature on tensile behavior of 5052 Al alloy deformed at cryogenic temperature

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Recently, deformation at cryogenic temperature has attracted much attention as one of the effective processes to produce ultrafine grained (UFG) materials. The suppression of dynamic recovery during deformation at extremely low temperatures is expected to preserve a high density of defects generated by deformation, which can act as the potent recrystallization sites. Accordingly, the cryogenic deformation would require less plastic deformation for achieving ultrafine grains, compared to the severe plastic deformation (SPD) processes [1–5] at ambient or elevated temperatures. For instance, Wang *et al.* [6] obtained UFG pure copper by applying cryogenic rolling (cryo-rolling). Further, they reported that the bimodal microstructure of ultrafine grains and relatively coarse grains obtained by cryo-rolling followed by flash annealing resulted in the excellent combination of high strength (>400 MPa) and ductility (65% total elongation). They suggested that the bimodal microstructure, consisting of recrystallized ultrafine grains and large coarsened grains, would be an appropriate microstructure for a good combination of mechanical properties, since coarsened grains would sustain sufficient deformability with pronounced strain hardening whereas the large volume fraction of ultrafine grains could maintain high strength. Thus, it is expected that the application of cryogenic rolling to 5052 Al alloy would require less plastic deformation in achieving ultrafine grains with high strength than other SPD processes.

The distinguishing features of UFG materials during tensile deformation are the smaller strain hardening and the lower ductility than coarse grained ones. According to recent work [7–10], the small strain hardening is related to the annihilation kinetics of extrinsic grain boundary dislocations introduced by SPD processes and the number of dislocations necessary to deform ultrafine grains. Meanwhile, a small strain hardening region of the engineering stress–strain curve is often observed in 5052 Al alloy deformed at cryogenic temperature and annealed at low temperatures. Furthermore, ductility, as an important parameter of UFG materials, is significantly influenced by annealing temperature. Since microstructural evolution with annealing temperature, such as recovery and recrystallization, would enhance the variation of tensile behavior in engineering stress–strain curves, it is necessary to examine the relationship between microstructure and tensile behavior in engineering stress–strain curves.

In view of the foregoing, the present work was carried out to investigate tensile behavior in terms of engineering stress–strain curves, in conjunction with microstructural evolution during annealing of 5052 Al alloy deformed at cryogenic temperature.

The material used in this work was commercial 5052 Al alloy plate annealed at 813 K for 2 h, with equiaxed grains of 65 μm. Cryogenic rolling was performed by dipping plates into liquid nitrogen for at least 15 min before each rolling pass. To investigate the effect of annealing temperature, rolled sheets with 85% reduction were annealed in the temperature range of 423–573 K for an hour. Tensile tests were conducted on the annealed sheets, machined to the ASTM subsize specimen of the 25 mm gauge length, at an initial strain rate, 3×10^{-3}/s. Strain was measured using an extensometer with a 25 mm gauge length. For a detailed understanding of the microstructural evolution during annealing, a transmission electron microscope (TEM) was used to analyze thin foils obtained from the samples 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 243 K.

The representative engineering stress–strain curves of annealed 5052 Al alloy are shown in Fig. 1. The stress–strain curves can be classified into two groups, according to their shapes. The first one shows a typical continuous yielding behavior for the specimens annealed below 473 K. It is well known that the presence of excess mobile dislocations causes the occurrence of continuous yielding in engineering stress–strain curves. The engineering stress–strain curves of the second group show that continuous yielding disappears and the formation of a yield plateau, where flow

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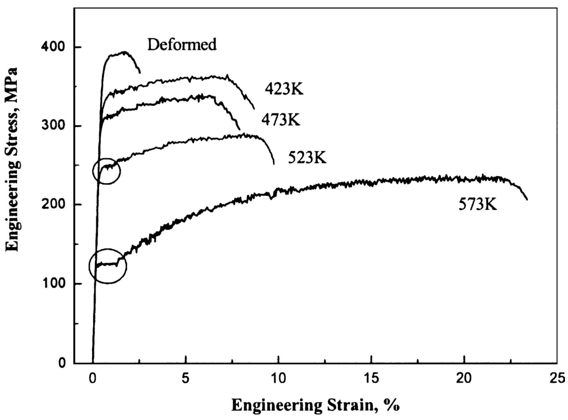

Figure 1 Engineering stress–strain curves of 5052 Al alloy, deformed 85% at cryogenic temperature, with various annealing temperatures of 423–573 K.

stress is independent of a strain, starts to appear. It is interesting to note that as annealing temperature increases, the yield plateau becomes more prominent and progressively expands up to an annealing temperature of 573 K (circled area in Fig. 1). This phenomenon must be related to microstructural evolution, occurring during annealing at temperatures above 523 K. Hayes *et al.* [11] reported that the occurrence of the large yield point elongation in stress–strain curves contributed to the increase of tensile elongation for annealing temperatures above 473 K in Al–3Mg alloy deformed by equal channel angular extrusion. Generally, the occurrence of yield point elongation is closely related to the annihilation of mobile dislocations and the presence of a considerable amount of coarse equiaxed grains. Although a quantitative analysis regarding the dislocation density was not performed, the annihilation of mobile dislocations during annealing above 473 K as recovery would be responsible for the disappearance of continuous yielding. Additionally, the expansion of the yield plateau with increasing annealing temperature in Fig. 1 indicates that the extent of the yield plateau in stress–strain curves is significantly influenced by the volume fraction of coarse equiaxed grains. Accordingly, the formation of nearly small equiaxed grains, less than 200 nm in a diameter, during annealing at 473 K (Fig. 2b) could not contribute to the occurrence of a yield plateau, due to the small amount and grain size. However, the increased volume fraction of equiaxed and coarsened grains at 523 K (Fig. 2c) enhances the formation of a yield plateau, and fully recrystallized grains with grain size of 20 μm (Fig. 2d) expand the yield plateau up to a strain of 1.2% in Fig. 1. From the above, it is obvious that large total elongation above annealing temperatures of 523 K can be attributed to the increased amount of equiaxed and/or coarsened grains.

Meanwhile, it is interesting to note that total elongation for annealing at 423 K is found to be larger than for annealing at 473 K in Fig. 1. Generally, ductility, such as total elongation and reduction in area, increases with annealing temperature through some loss of dislocations, subgrain growth and recrystallization. Therefore, these softening processes could not properly explain that phenomenon. Thus, it is necessary to carry out a close examination of microstructures annealed at 423 and 473 K. The typical microstructure for annealing at 423 K in Fig. 2(a) shows the formation of subgrains including the rearrangement of dislocations, as recovery. Meanwhile, annealing at 473 K enhances the formation of a bimodal microstructure, consisting of elongated subgrains and small equiaxed grains, less than 200 nm (Fig. 2b). The reduction of the aspect ratio in elongated subgrains, compared with the 423 K annealing, can be attributed to the formation of transverse boundaries within subgrains and a slight increase in subgrain width. The slight increment of mean free path, subgrain width, for the movement of dislocations would increase ductility (total elongation) to some degree. Therefore, it is obvious that the formation of small

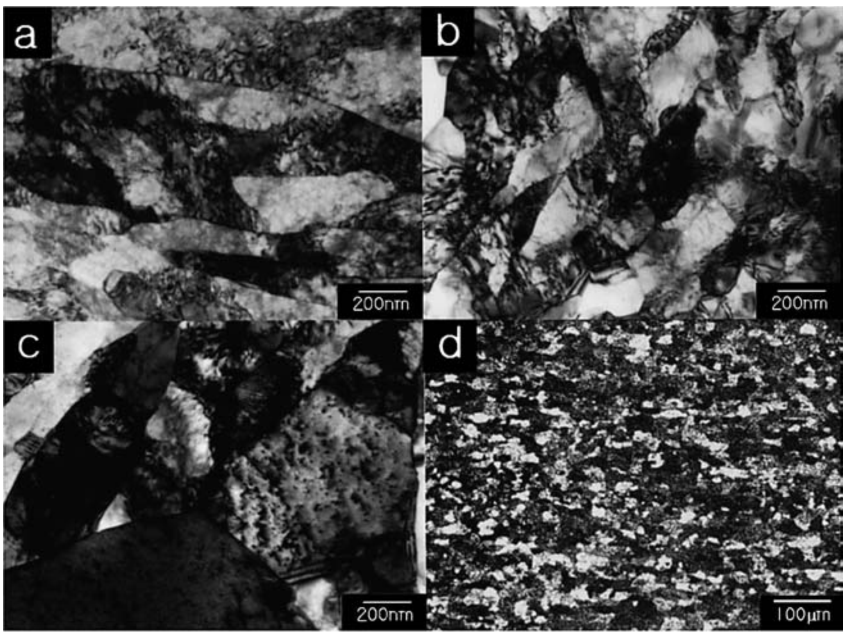

Figure 2 Microstructures of 5052 Al alloy, deformed 85% at cryogenic temperature and annealed at various temperatures for an hour: (a) TEM micrograph, annealed at 423 K, (b) TEM micrograph, annealed at 473 K, (c) TEM micrograph, annealed at 523 K, showing the presence of equiaxed coarse grains, and (d) optical micrograph, annealed at 573 K.

equiaxed grains, less than 200 nm, reduces ductility. However, the reason for the decreased total elongation for annealing at 473 K is still unclear. Although the authors do not have the full answer at the moment, the transition of deformation mechanisms from dislocation activity to grain boundary-related deformation in small equiaxed grains [12, 13] seems the most probable explanation.

Therefore, it can be concluded that the disappearance of continuous yielding and the formation of a yield plateau in engineering stress–strain curves at annealing temperature above 523 K is closely related to the annihilation of mobile dislocations during annealing and the increased amount of coarse equiaxed grains. In addition, the total elongation of the specimen annealed at 473 K was found to be smaller than that annealed at 423 K, due to the formation of small equiaxed grains, less than 200 nm, at 473 K.

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