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ULTRAFINE GRAINED MATERIALS

The improvement of strength and ductility in ultra-fine grained 5052 Al alloy by cryogenic- and warm-rolling

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Abstract The effects of deformation temperatures and post-deformation annealing on mechanical properties, in conjunction with microstructural evolution in the 5052 Al alloy, were investigated. The combination of cryogenic-rolling with warm-rolling effectively increased tensile strength and yield strength without the decrease of ductility through the formation of ultra-fine grains with dynamic recovery in the 5052 Al alloy. And static annealing, as a post-heat treatment, enhanced the ductility. Therefore, ultra-fine grained 5052 Al alloy with high strength and a moderate level of ductility could be made by the combination of cryogenic-rolling with warm-rolling and the additional static annealing process.

Introduction

Aluminum alloys, especially 5*xxx* Al alloys, have been studied extensively, since they provide good properties, such as high fatigue strength, excellent corrosion resistance, and formability. Furthermore, the improvement of strength and formability in Al alloys will accelerate the applications to industry as a promising structural material for substituting steels [1–5]. This improvement can be achieved by the application of severe plastic deformation (SPD), which manufactures ultra-fine grained (UFG) materials with the grain size less than 1 μm.

Earlier works have shown that some SPD processes, such as equal channel angular pressing (ECAP) [6–9], cyclic extrusion compression (CEC) [10], high-pressing [11–15], and accumulative rolling-bonding (ARB) [16, 17], are effective in manufacturing UFG materials. The distinguished features of UFG materials are the higher strength, the lower strain hardening, and the lower ductility than conventional grain sized materials. Although many works have been carried out to improve the limited ductility of UFG materials [18–23], the ductility still remains as a mechanical property to be overcome.

Meanwhile, the deformation at cryogenic temperature is also recognized as one of the effective processes to produce UFG materials [24–28]. The suppression of dynamic recovery during deformation at low temperatures preserves a high density of dislocations generated by deformation, which can act as potent recrystallization sites during post-deformation annealing. The larger driving force for recrystallization, due to the larger density of accumulated dislocations, would lower recrystallization temperature or accelerate recrystallization during annealing. Accordingly, the cryogenic deformation would require less plastic deformation for achieving ultra-fine grains, compared to SPD processes. Among these SPD processes, an ARB process is known to have a potential for continuous production of materials in bulk. The repeated rolling at warm temperatures yields ultra-fine grains and high strength after several repetition cycles, through the microstructural evolution, including work hardening, the subdivision of grains, and dynamic recovery. Additionally, the combination of cryogenic-rolling with warm-rolling [26], whose temperature range is similar to that of an ARB process, was found more effective than a single cryogenic-rolling process or an ARB process in improving mechanical properties of a 5052 Al alloy. However, none of above methods has clearly solved the limited ductility problem in UFG materials.

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The post-deformation annealing, accompanying static recovery or static recrystallization, is very effective on improving ductility with losing its high strength. Since Al alloys produced by an ARB process, the deformation at cryogenic temperature or the combination of cryogenic-rolling with warm-rolling, show a very high strength, Al alloys might maintain a high strength level even though the occurrence of softening during the post-deformation annealing. Thus, it is anticipated that the post-deformation annealing after the combination of cryogenic-rolling with warm-rolling would result in the excellent combination of high strength and good ductility.

In view of the aforementioned, the present investigation was carried out to investigate the effect of deformation temperatures on tensile properties, in conjunction with microstructural evolution in a 5052 Al alloy. Additionally, the annealing behavior of 5052 Al alloys rolled at different deformation temperatures, paying attention to the microstructural evolution and the corresponding mechanical properties was also examined.

Experimental procedures

The material used in this work was a commercial 5052 Al alloy. The chemical compositions of Al alloy are 2.50 wt% Mg, 0.40 wt% Fe, 0.25 wt% Si, 0.25 wt% Cr, 0.10 wt% Cu, 0.01 wt% Zn, and 0.10 wt% Mn. The material was annealed at 613 K for 2 h and then quenched in water, resulting in a grain size of 65 mm.

To compare microstructures and mechanical properties of samples deformed at different rolling temperatures, the plates, 8 mm in thickness, were rolled with 80% reduction at cryogenic temperature (cryo-rolling) or at room temperature (cold-rolling). In addition, the combination of cryogenic and warm-rolling was carried out as cryogenic-rolling with 55% reduction followed by warm-rolling with 56% reduction (total reduction of 80% in thickness). Cryogenic-rolling was performed by dipping plates into liquid nitrogen for at least 15 min before each rolling pass. Subsequently, cryo-rolled sheets were heated at 448 K for 5 min to homogenize the temperature and rolled at 448 K. Tensile specimens were taken after each pass and tensile tests were conducted on the specimens, which were machined to the ASTM subsize form of 25 mm gauge length at the initial strain rate, 3×10^{-3}/s on an INSRTON machine at room temperature. To investigate the effect of a post-deformation annealing, the different rolled sheets with 80% reduction were annealed at 448 K for 48 h. Vickers hardness (200 g) was measured to keep track of the hardness variation. For a detailed understanding of the microstructural evolution, a transmission electron microscope (TEM) was used. Thin foils, parallel to the transverse cross section of the sheets, were prepared by utilizing a twin-jet polishing technique using a mixture of 75% methanol and 25% HNO_3 at a temperature of 243 K.

Results and discussion

The effect of deformation temperatures

Tensile properties in Fig. 1a reflect the characteristics of microstructures evolved during the deformation at different rolling temperatures. Cryo-rolling with 80% reduction led to the increase of ultimate tensile strength (UTS) from 212 to 350 MPa, while cold-rolling with the same reduction resulted in UTS of 325 MPa. The higher TS in samples received from cryo-rolling implies that cross-slip or climb of dislocations, associated with dynamic recovery, was effectively suppressed during cryo-rolling and therefore the dislocation density remained high [18, 24–28]. Meanwhile, the high UTS of 452 MPa could be achieved in the sample deformed at cryogenic temperature with 55% reduction and subsequently warm deformed at 448 K with 56% reduction. This indicates that the combination of cryo-rolling and warm-rolling was more effective to increase strength than cryo-rolling alone. The increment of UTS by the application of warm-rolling at 448 K on the sample received cryo-rolling with 55% reduction was 132 MPa, while the cryo-rolling with the same reduction increased only 30 MPa of UTS. Generally, the increase of a rolling temperature accelerates softening due to dynamic recovery or dynamic recrystallization rather than work hardening due to plastic deformation. The variations of YS with deformation strain in Fig. 1b resembled the trend of UTS in Fig. 1a, except for the rapid increase of YS in a low-strain region.

To understand this unusual mechanical behavior, the microstructure of a sample, cryo-rolled and warm-rolled at 448 K (Fig. 2a), was examined, compared with the microstructure deformed at cryogenic and room temperatures (Fig. 2b, c). Cryogenic deformation with 80% reduction (Fig. 2b) led to the formation of parallel bands of elongated substructures, 0.05–0.15 μm in width and 0.4–0.8 μm in length, containing high density of dislocations. The microstructure of a cold-rolled sample (Fig. 2c) showed the similar trend to that of a cryo-rolled sample, except for a slight decrease of dislocation density due to the occurrence of dynamic recovery. However, in spite of the increase of the deformation temperature, the microstructure of a sheet, cryo-rolled and warm-rolled at 448 K (Fig. 2a), resembled the microstructure deformed at cryogenic temperature, consisting of parallel bands of elongated substructures, 0.10–0.15 μm in width, with dislocations.

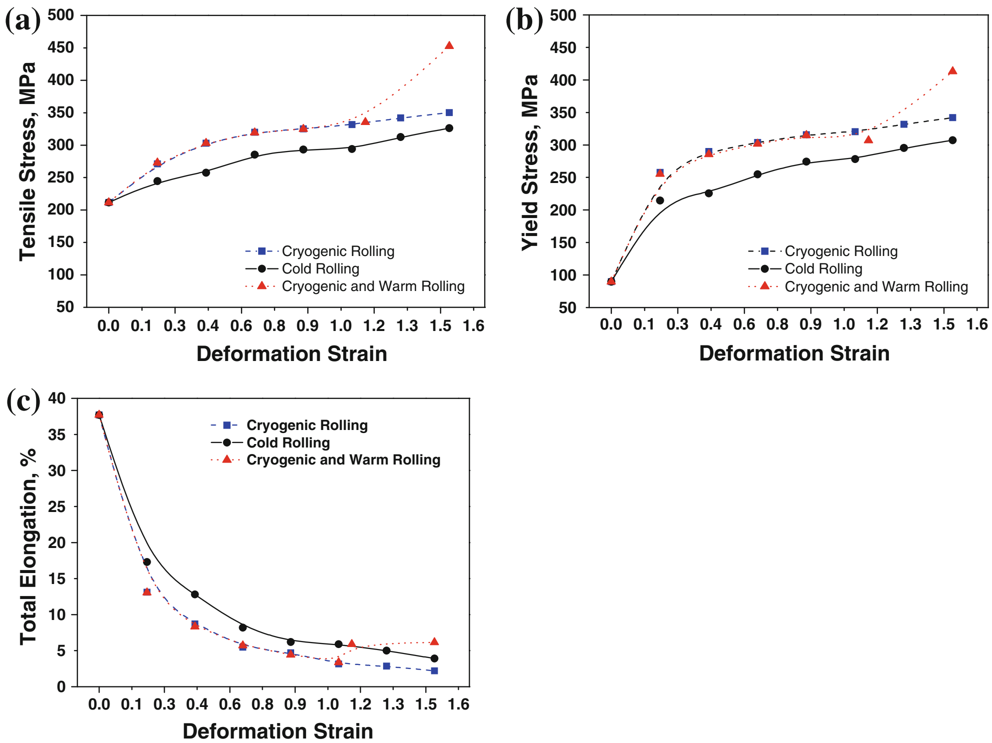

Fig. 1 The variations of mechanical properties as a function of deformation strain under different deformation temperatures in Al 5052 alloys; **a** tensile stress, **b** yield stress, and **c** total elongation

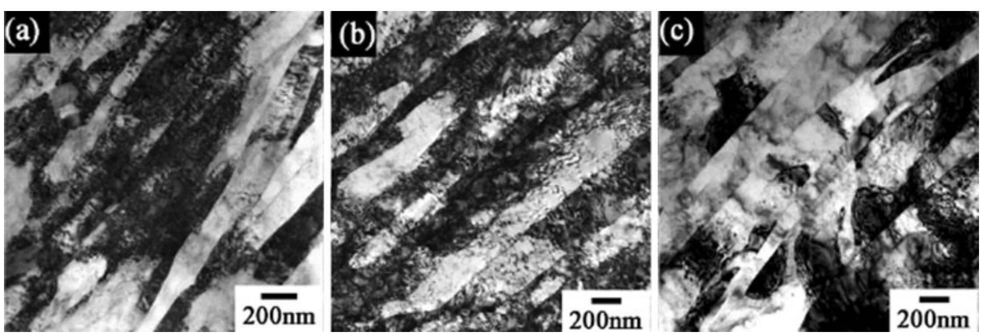

Fig. 2 TEM micrographs, showing the effects of deformation temperatures, such as **a** cryogenic- and warm-rolling, **b** cryogenic-rolling, and **c** cold-rolling, of 5052 alloys deformed with 80% reduction

The observed differences in microstructures were the presence of fine precipitates, about 50–100 nm in diameter (indicated as arrows in Fig. 3a, b), and the smaller density of dislocations due to dynamic recovery during warm-rolling. Thus, it is expected that this unusual increase of UTS in the sheet, cryo-rolled and warm-rolled at 448 K, can be attributed to the formation of fine precipitates.

To investigate the effect of warm-rolling on the formation of precipitates, measured hardness of samples by cryo-rolled and subsequently annealed at 448 K compared with that of cryo-rolled and warm-rolled sample in Fig. 4. Static annealing for 20 min on a sample cryo-rolled with 80% reduction increased hardness from 105 Hv (cryo-rolled) to 112 Hv. This increase of hardness would be attributed to the formation of precipitates during static annealing. Meanwhile, the decrease of hardness for the further annealing at 448 K would be attributed to the softening process such as static recovery and coarsening of precipitates. However, the hardness value of 135 Hv in a sample, cryo-rolled and warm-rolled at 448 K, is too high to be explained with the contributions of work hardening during warm-rolling and of the formation of precipitates during inter-pass annealing of warm-rolling, compared with 112 Hv in a sample cryo-rolled and subsequently annealed at 448 K.

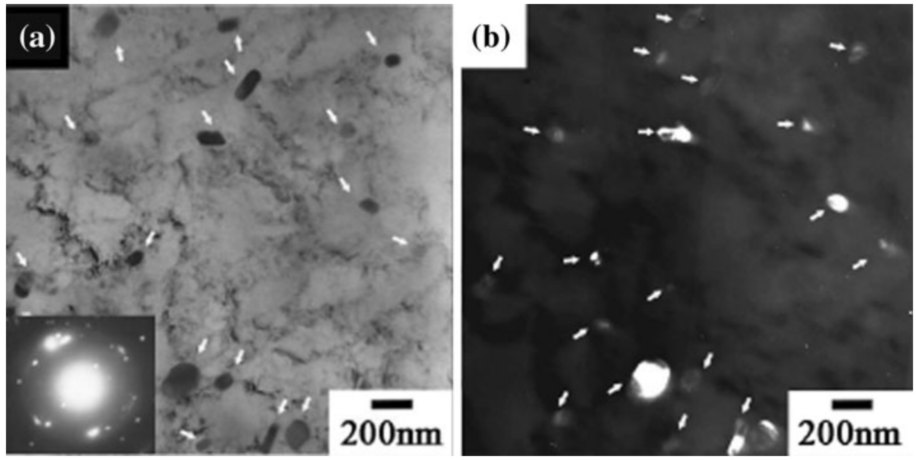

Fig. 3 TEM micrographs of a 5052 alloy deformed at cryogenic temperature with 55% reduction and subsequently warm deformed with 56% reduction; **a** bright field image with SAD pattern (*arrow* indicates precipitates) and **b** dark field image

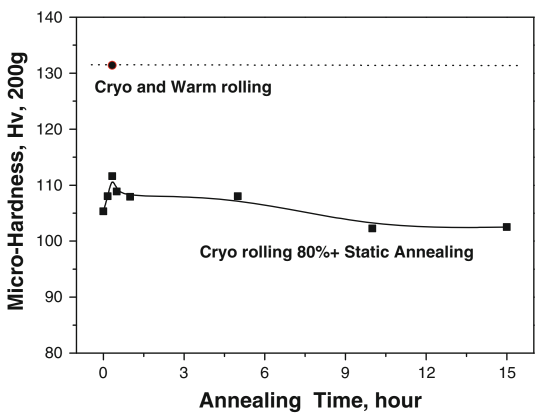

Fig. 4 The variations of micro-hardness as a function of annealing time in deformed Al 5052 alloys

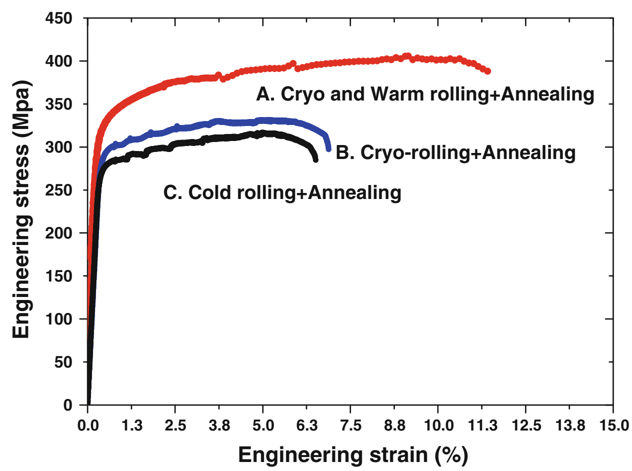

Fig. 5 The engineering stress–strain curves of Al 5052 alloys deformed at different temperatures and subsequently annealed at 448 K for 48 h

From the above discussion, it seems most probable that this remarkable increase of strengths and hardness in a sample cryo-rolled and subsequently annealed at 448 K would be caused by the fine precipitates produced during warm-rolling at 448 K rather than during static annealing.

In general, total elongation, as a parameter of ductility, exhibits the reverse trend to the variation of strengths. Total elongation of a cryo-rolled sample decreased more rapidly than that of a cold-rolled sample, reflecting the higher work hardening rate in a cryo-rolled sample. Additionally, total elongations of samples, cryo-rolled and cold-rolled with 80% reduction, dropped from 37.7% to 2.2% and 3.9%, respectively. In contrast, the combination of cryogenic and warm-rolled sample maintained the level of 6.2%, in spite of high UTS of 452 MPa.

The effect of static annealing

Figure 5 shows the engineering stress–strain curves of Al 5052 alloys deformed at different temperatures and subsequently annealed at 448 K for 48 h. The cryo-rolled and annealed sample showed YS of 295 MPa, UTS of 331 MPa, and an elongation to failure of 6.8% (curve B). Meanwhile, cold-rolling and annealing decreased both YS and UTS, and decreased its elongation to failure to 6.5%. Curve C produces the similarly shaped stress–strain curve to the deformation at cryogenic temperature with 80% reduction. However, cryo-rolled and warm-rolled sample at 448 K for 48 h dramatically increased its elongation to failure (curve A). It is also clear that annealing at 448 K for 48 h yielded higher strengths and ductility than other samples. Ultimate tensile strength in curve A is 405 MPa, which is much higher than that in curve B (331 MPa) and curve C (315 MPa). Furthermore, curve A showed that annealing at 448 K for 48 h produces an elongation to failure of 12%. This elongation is much higher than those of cryo-rolled and cold-rolled samples, which are 6.8% and 6.5%, respectively.

The above-mentioned mechanical behaviors are closely related with the microstructural evolution during annealing of deformed 5052 Al alloys. Figure 6 shows annealed

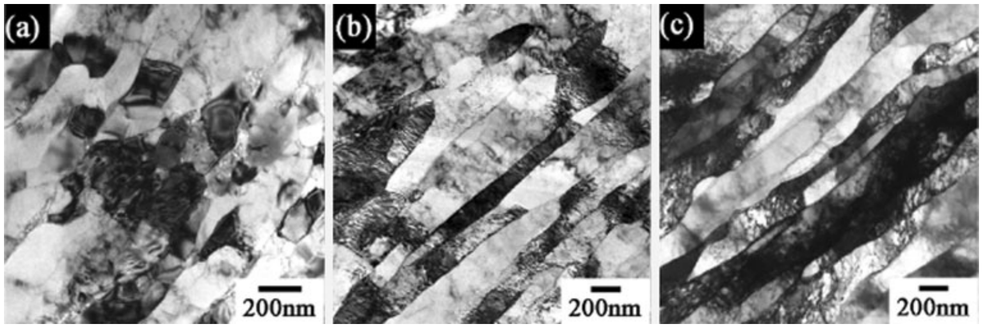

Fig. 6 TEM micrographs of 5052 alloys, annealed at 448 K temperature for 48 h after **a** cryogenic- and warm-rolling, **b** cryogenic-rolling, and **c** cold-rolling

microstructures after cryogenic and warm-rolling, cryo-rolling or cold-rolling. The annealing of a cryo-rolled sample (Fig. 6b) led to the formation of subgrains and the rearrangement of dislocations including some loss of dislocations, as recovery proceeded. Many of elongated substructure boundaries recovered into subgrain boundaries exhibiting the distinct contrast, without a noticeable change in the substructure dimension. Accordingly, UTS decreased from 350 to 331 MPa by annealing. Tensile properties reflect this microstructural changes during static annealing at 448 K for 48 h. This small decrease of UTS by annealing would be attributed to a little change of dimension of dislocation substructures during the annealing, while the rearrangement of dislocations during recovery would be responsible for the increase of total elongation from 2.2% to 6.8%.

It is interesting to note that the reduction of the aspect ratio in subgrains is observed in a cold-rolled and annealed sample (Fig. 6c). The formation of transverse boundaries within subgrains due to the rearrangement of dislocations or the slight increase of the width of subgrains would become a reason to reduce the aspect ratio of subgrains during annealing of cold-rolled samples. However, the reduced aspect ratio of sugrains caused little variation of mechanical properties, as shown Fig. 5 (curve C). The slight increase of dislocation mean free path associated with the increase of the subgrain width did not cause any significant change in tensile properties.

In contrast, as shown in Fig. 5 (curve A), a tensile elongation to failure significantly increased to 12%, compared with 6.2% in a deformed state. Comparing to the increasing rate of elongation, the decrease of UTS due to annealing was not large as about 10%. These behaviors would be related to the formation of equiaxed grains. Figure 6a showed the presence of nearly equiaxed grains, whose size is similar to the subgrain width (100–200 nm). Hayes et al. [29] reported that the Al–3Mg alloy deformed by equal channel angular extrusion (ECAE) showed the rapid increase of tensile elongation for the annealing temperature above 448 K, accompanying the occurrence of yield point elongation which is closely related to the presence of considerable amount of equiaxed dislocation-free grains. Although there was no yield point elongation in curve A in Fig. 5, the presence of equiaxed dislocation free grains would be helpful in the improvement of tensile elongation. Thus, the large amount of equiaxed grains produced during annealing as well as the reduction of internal stresses and dislocation density was anticipated to improve ductility effectively in the present case.

From the above-mentioned results, it is expected that the post-deformation annealing after the combination of cryo-rolling with warm-rolling would become a useful method to achieve the good combination of elongation and high strength. Additionally, this work has the meaningful information that the good mechanical properties can be achieved only by rolling and annealing processes.

Conclusions

The effects of deformation temperatures and post-deformation annealing on mechanical properties, in conjunction with microstructural evolution in a 5052 Al alloy, were investigated.

(1) The combination of cryogenic-rolling with warm-rolling was found more effective than a single cryogenic-rolling or cold-rolling in improving mechanical properties of a 5052 Al alloy.
(2) Warm-rolling at 448 K after cryogenic-rolling was found to significantly improve mechanical properties. This notable increase of ultimate tensile strength would be attributed to the formation of fine precipitates during warm-rolling at 448 K.
(3) The high ductility of a 5052 Al alloy, received from cryo-rolling with warm-rolling and subsequently annealed at temperature of 448 K for 48 h, would be attributed to the formation of fine equiaxed grains (100–200 nm) as well as the reduction of dislocation density.

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