Mechanical properties and microstructure of 6061 aluminum alloy severely deformed by ARB process and subsequently aged at low temperatures

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Abstract. In order to clarify the aging behavior in ultrafine grained (UFG) Al alloys, a commercial Al-Mg-Si alloy was severely deformed by accumulative roll-bonding (ARB) process and subsequently aged at 100°C or 170°C. The age-hardening behavior and microstructure change during aging were investigated. At 170°C, age-hardening was observed in solution treated (ST) specimens, but solution-treated and ARB-processed specimens were not hardened by aging. On the other hand, the hardness of the both ST specimen and ARB-processed specimen increased by aging at 100°C. From TEM observation, it was found that the ARB-processed specimen had an ultrafine lamellar boundary structure and the structure was kept during aging at 170°C and 100°C. In the ST specimen aged at 170°C, fine precipitates were observed within coarse grains. In the specimen ARB-processed and subsequently aged at 170°C, coarser precipitates were observed within ultrafine grains and on grain boundaries. It was considered that the reason why the hardness of the specimens ARB-processed and subsequently aged did not increase was coarsening of precipitates. In the specimens aged at 100°C, obvious precipitates were not observed, but clusters Mg and Si seemed to form during the aging, leading to the increase in the hardness of the specimen. From the results, it was suggested that aging at low temperatures could improve mechanical properties of Al alloys through combining grain refinement and precipitation hardening.

1. Introduction
Age-hardening is known as a conventional method to increase strength of commercial aluminum alloys. For example, in commercial Al-Mg-Si alloys, Mg₂Si precipitates formed during aging improve strength of the alloys. Grain refinement is also a conventional method to increase strength of metals. Recently, it has been reported that severe plastic deformation (SPD) processes can fabricate ultrafine grained (UFG) microstructures having mean grain sizes smaller than 1µm and the UFG metals exhibit high strength [1-4]. Thus, it is expected that the combination of age-hardening with ultra grain refinement would improve mechanical properties of Al alloys greatly.

Age-hardening behavior in the ultrafine grained (UFG) materials is probably more complex than that in conventional coarse grained metals, because the UFG materials fabricated by deformation process have many defects such as vacancy, dislocation and grain boundary which must affect precipitation behavior. In the present study, therefore, to clarify age-hardening behaviors of the UFG metals, a commercial Al-Mg-Si alloy (6061 Al alloy) was severely deformed by accumulative roll-bonding (ARB) process [1] and subsequently aged, and its mechanical properties and microstructure were investigated.
2. Experimental procedure
A commercial Al-Mg-Si alloy (JIS 6061 Al alloy) was used in this study. The chemical composition of the alloy is shown in Table 1. Sheets of the material were solution treated at 530°C for 4 hr and then quenched into water immediately. The specimen at this stage is called the solution treated (ST) specimen hereafter. The solution-treated sheets were severely deformed by the ARB process at room temperature (RT) up to 6 cycles. As 50% rolling reduction per one ARB cycle was used in the present study, the 6-cycle ARB corresponds to an equivalent strain of 4.8. The ARB-processed specimens as well as the ST specimen were aged at 100°C or 170°C for various periods of time. The temperature of 170°C is a conventional aging temperature in commercial heat treatments.
In order to investigate aging behaviors, hardness of the specimens aged at each temperature for various periods was measured by Vickers hardness test with a load of 1kN for 10s. Microstructures of the specimens ARB-processed and subsequently aged was observed from the transvers direction (TD) of the sheets by transmission electron microscopy (TEM). The specimens for the microstructural observations were mechanically polished and then electropolished in a solution of 30% \( \text{HNO}_3 \) + 70% \( \text{CH}_3\text{OH} \) at 11 volt and -30°C.

Table 1. Chemical composition of the aluminum alloy studied (mass%).

| Si  | Fe  | Cu  | Mn  | Mg  | Cr  | Zn  | Ti  | Al  |
|-----|-----|-----|-----|-----|-----|-----|-----|-----|
| 0.59| 0.45| 0.28| 0.02| 1.01| 0.23| <0.01| 0.04| Bal.|

3. Results and discussion
Figure 1 shows a TEM micrograph of the specimen ARB-processed at RT by 6 cycles. An ultrafine lamellar boundary structure elongated to the rolling direction (RD) with a boundary spacing of approximately 200 nm was observed. The lamellar structure has also been reported in commercial pure aluminums severely deformed by the ARB process [5]. The UFG specimen fabricated by ARB and the ST specimen were aged at 100°C or 170°C.

![Fig.1 TEM microstructure of the specimen ARB-processed by 6 cycles.](image)

Figure 2 (a) shows the change in Vickers hardness during aging at 170°C of the ST specimen and the 6-cycle ARB specimen as a function of the aging time. It was found that the hardness increased by the ARB process from 52HV to 131HV. The hardness of the ST specimen increased from 52HV to 104HV by aging for 3 x 10^4 s at 170°C. On the other hand, the hardness of the ARB specimen hardly increased by aging at 170°C. The hardness slightly decreased above aging time of 10^5 s. However, the hardness of the ARB-processed specimen was still higher than that of the ST specimen aged for the same period.

Figure 2 (b) shows the hardness change during aging at 100°C of the ST specimen and ARB-processed specimen as a function of aging time. It was found that the hardness of the both specimens increased monotonously with increasing aging time. Furthermore, the increase in hardness was similar between two kinds of specimens. The increase in hardness by aging at
100°C for 2 x 10\(^5\) s was 27HV and 28HV for the ST specimen and the ARB specimen, respectively. These results indicate that the age-hardening behavior at 170°C was significantly affected by the UFG microstructure fabricated by the ARB process and the amount of age-hardening was reduced by ARB. On the other hand, the grain refinement by the ARB process hardly affected the age-hardening behavior at 100°C.

![Fig.2](image1.png)  
**Fig.2** Changes in Vickers hardness during aging of the ST specimen and the specimens ARB-processed by 6 cycles as a function of aging time at (a) 170°C and (b) 100°C.

To clarify microstructural changes of the matrix during heat treatment (aging), the microstructures of the specimens ARB-processed and subsequently aged were observed by TEM. Figure 3 shows TEM micrographs of the specimens ARB-processed by 6 cycles and then aged at (a) 170°C for 3 x 10\(^4\) s and (b) 100°C for 2 x 10\(^5\) s. Both specimens showed lamellar boundary structures similar to those observed in the as-ARB-processed specimen (Fig.1). It seemed that grain coarsening did not occurred during aging at 170°C or 100°C and small lamellar spacing was maintained even after aging. A grain growth during annealing at 150°C in an ARB processed commercial purity Al (1100 Al, purity of 99%) has been reported previously [5]. Thus, it is concluded that the UFG microstructure fabricated by ARB in the 6061 Al alloy is stable during aging even at 170°C compared to the pure aluminum.

![Fig.3](image2.png)  
**Fig.3** TEM micrographs of the specimens ARB-processed by 6 cycles and then aged at (a) 170°C for 3 x 10\(^4\) s and (b) 100°C for 2 x 10\(^5\) s.

In order to investigate the precipitation behaviors, the microstructures within the matrix grains after aging were observed by TEM. The TEM micrographs of the ST specimen and the ARB-processed specimen after aging at 170°C for 3 x 10\(^4\) s are shown in Fig.4. In the aged ST specimen (Fig.4 (a)), very fine precipitates dispersed homogeneously within the matrix grain. Figure 4 (b) and (c) show the TEM micrographs of the ARB-processed specimen after aging. In Fig.4(b), rod-like precipitates were observed in the matrix grains. The length of the rod-
like precipitates was larger than that of the fine precipitates in the aged ST specimen (Fig.4(a)), though the aging condition was the same. Coarse precipitates indicated by arrows in Fig.4 (b) and (c) were also observed on grain boundaries. The results indicate that the precipitates in the ARB specimen having the UFG microstructure coarsened faster than those in the coarse grained specimen (the ST specimen) under the same aging condition at 170°C. Coarsening of precipitates is disadvantageous for age-hardening because coarsening leads to decreases in number of precipitates and distance between precipitates. Thus, it is considered that the reason for the lack in age-hardening at 170°C in the ARB processed specimen (Fig.2(a)) is the precipitation coarsening.

Figure 5 shows TEM micrographs after aging at 100°C for $2 \times 10^5$ s for (a) the ST specimen and (b) the ARB-processed specimen. In both specimens, obvious precipitates could not be observed. However, it was confirmed that the hardness of the specimens increased after aging (Fig.2). In Al-Mg-Si alloys, the formation of Mg-Si clusters at 373K and 363K was reported by 3 Dimensional Atom Probe (3DAP) analysis [6, 7]. Thus, it is expected that the
increase in hardness observed in the present study was also attributed to the Mg-Si clusters. The result that the similar age-hardening behavior was observed in both ST and ARB specimens at 100°C (Fig.2(b)) indicates that the effect of the UFG microstructure on cluster formation is perhaps small in aging at low temperatures.

From the results shown above, it is suggested that aging at low temperatures is preferable to realize higher strength in the UFG 6061 Al alloy fabricated by the ARB process through combining grain refinement strengthening and age hardening.

4. Summary
A commercial Al-Mg-Si alloy (6061 Al alloy) was severely deformed by the ARB process up to 6 cycles (equivalent strains of 4.8) at RT after a solution treatment. The ST specimen and the ARB-processed specimen were subsequently aged at 170°C or 100°C, and age-hardening behaviors and changes in microstructure were investigated. Main results obtained are summarized below.

(1) Hardness of the ST specimen increased by aging at 170°C. On the other hand, hardness of the ARB-processed specimen decreased by aging at 170°C.
(2) During aging at 100°C, both the ST specimen and ARB-processed specimen showed age hardening. The amount of increase in hardness of the ARB-processed specimen was similar to that of the ST specimen.
(3) The as-ARB-processed specimen showed a lamellar boundary structure elongated to RD and having a mean lamellar spacing of approximately 200 nm. It was found that the matrix microstructure was kept during aging at 170°C as well as 100°C.
(4) In the ST specimen aged at 170°C, fine precipitates were observed within the matrix grains. In the specimen ARB-processed and subsequently aged at 170°C, precipitates were observed in matrix grains as well as on grain boundaries. The size of the precipitates in the ARB-processed specimen was larger than that in the ST specimen.
(5) In both the ST specimen and the ARB-processed specimen, precipitates were not observed by TEM observation after aging at 100°C. Formation of Mg-Si clusters was considered to be the reason for the hardening at 100°C.

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