Effect of Cold Rolling on Cluster(1) Dissolvability during Artificial Aging and Formability during Natural Aging in Al-0.6Mg-1.0Si-0.5Cu Alloy

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Abstract: Natural aging after solution treatment has a negative effect on the precipitation strengthening of Al–Mg–Si alloys since Cluster(1) formed at a room temperature cannot be dissolved or transformed into precipitates during artificial aging at 170 °C. In this study, cold rolling is focused on as an alternative solution to pre-aging, which is a conventional method to prevent Cluster(1) formation. It is known that excess vacancies are necessary for cluster formation. Cold rolling suppresses cluster formation because excess vacancies disappear at dislocations introduced by cold rolling. In addition, it is expected that cold rolling accelerates the precipitation behavior because the diffusion of solute atoms is promoted by introduced lattice defects. The transition of Cluster(1) was evaluated by Micro Vickers hardness tests, tensile tests, electrical conductivity measurements and differential scanning calorimetry analyses. Results showed the negative effect of natural aging was almost suppressed in 10% cold-rolled samples and completely suppressed in 30% cold-rolled samples since Cluster(1) dissolved during artificial aging at 170 °C due to lowering of the temperature of Cluster(1) dissolution by cold rolling. It was found that the precipitation in cold-rolled samples was accelerated since the hardness peak of 10% cold-rolled samples appeared earlier than T6 and pre-aged samples.

Keywords: Al–Mg–Si alloy; natural aging; cold rolling; differential scanning calorimetry

1. Introduction

In the automobile industry, 6000 series aluminum alloys are utilized as body sheet panels. The panels are strengthened during heat treatment of the paint-baking process due to precipitation strengthening. The overall precipitation sequence of 6000 series aluminum alloys is mostly believed as follows [1,2]:

Supersaturated solid solution → Clusters → β"(L) → β'(Q') → β

L and Q' are precipitates containing Cu. T6 treatment, which is a thermomechanical treatment with artificial aging immediately after solution heat treatment, is a desirable thermomechanical treatment for precipitation strengthening since the precipitates are densely formed by this treatment. However, in industrial production, it is difficult to conduct artificial aging immediately after solution heat treatment, and there might be several weeks’ interval between finalizing heat treatment at material manufacturers and the paint-baking process at automobile manufacturers. While the alloys are left at a room temperature for storage and shipping, the precipitation phenomenon called natural aging occurs. Natural aging adversely affects precipitation behavior during subsequent artificial aging by inhibiting and delaying precipitation strengthening [3–11], known as the negative effects of natural aging in those alloys. The cause of negative effects is clusters formed during natural aging. Two types of clusters that are formed in 6000 series alloys are known [4–11]. One is Cluster(1), formed at a room temperature, and the other is Cluster(2), formed at 70 °C or higher. Cluster(2) is the main strengthening factor during the initial
artificial aging and can be transformed into precipitates during artificial aging, thus it does not adversely affect precipitation behavior. However, it is known that Cluster(1) has high stability and does not dissolve or transform into precipitates during artificial aging at 170 °C. The composition and structural characteristics of Cluster(1) and Cluster(2) have been recently investigated by 3D atom probe (3DAP). Serizawa et al. [5] reported that since the Mg/Si ratio of the larger sized Cluster(2) was close to a value identical to the β'' phase, Cluster(2) easily transformed into the β'' phase. Cluster(1) has been studied in particular to illuminate why it has high thermal stability. Aruga et al. [4,9] reported that the Mg/Si ratio of Cluster(1) is affected by the Mg/Si ratio in the alloys’ compositions, and the Si rich clusters are difficult to transform into the β'' phase or to dissolve during short term artificial aging because the bonding of Si-Si atoms is thermally stable. On the other hand, Poznak et al. [10] reported that at high alloys’ Mg/Si ratios, Cluster(1) is thermally stable and slowly grows during artificial aging. It is also reported that at sufficiently low alloys’ Mg/Si ratios, artificial aging promotes the dissolution of Cluster(1), particularly that of higher Mg concentrations. The formation of clusters requires solute atoms and excess vacancies. The formation of Cluster(1) during natural aging consumes solute atoms and excess vacancies and makes them scarce. Zhu et al. [11] reported that a lot of vacancies are tied with solute atoms in Cluster(1), which is the reason for the high stability of Cluster(1). As described above, various studies have shown the high thermal stability of Cluster(1), and it is found that Cluster(1) inhibits the precipitation behavior during artificial aging due to deplete solute atoms and excess vacancies. Therefore, it is important to prevent the formation of Cluster(1) during natural aging.

A typical method to prevent the formation of Cluster(1) is pre-aging [3,4]. By aging at around 100 °C immediately after solution heat treatment, Cluster(2) is formed, and the formation of Cluster(1) during subsequent natural aging is suppressed.

In this study, cold rolling is focused on replacing pre-aging to prevent the negative effects of natural aging. In general, industrial pre-aging requires keeping the furnace at about 100 °C to conduct the heat treatment of the products. In contrast, for cold rolling, passing through the rolls only a few times of is sufficient. Comparing these processes, the energy for cold rolling in industrial production is lower than that for pre-aging, thus it can be an attractive method. Masuda et al. reported that the amount of Cluster(1) formed during aging below 70 °C for 7.2 ks and 778 ks decreased by cold rolling [6]. The reason for this decrease is that the distance between dislocations is reduced due to the increase in dislocation density caused by cold rolling, and excess vacancies disappear at the dislocations [6,8]. They also found that the precipitation behavior during artificial aging was accelerated by cold rolling. However, even 3% cold rolling did not completely suppress the negative effects of natural aging. On the other hand, further suppression of cluster formation and acceleration of precipitation behavior in 90% cold-rolled alloys have been reported recently [9]. Therefore, it is expected that the negative effects of natural aging will be completely suppressed at an appropriate reduction ratio higher than 3%.

It has been reported elsewhere that plastic deformation before artificial aging affects precipitation behavior [8,12–20]. This is due to the effect of lattice defects introduced by plastic deformation. Dislocations can serve as nucleation sites for precipitates and promote diffusion of solute atoms by pipe diffusion [8,12,13]. However, it has been reported that precipitates become coarse, and low density of number in alloys facilitated plastic deformation [12,15]. On the other hand, it has been reported that precipitates become finer in Al-Mg-Si alloys containing Cu [21–24]. The effect is also exhibited in alloys with 1% deformation applied [23,24]. Therefore, adding Cu to alloys that are designed to undergo plastic deformation is suitable.

This study aims to investigate the effect of cold rolling 10% and 30% on Cluster(1) formation during natural aging and its transition during artificial aging. The effects were evaluated by Micro Vickers hardness tests, tensile tests, electrical conductivity measurements and differential scanning calorimetry analyses. A comparison of mechanical
properties was also made between the cold-rolled alloys and the pre-aged alloys, which is a conventional solution.

2. Experimental Procedure

An AA6011 alloy with the chemical composition Al–0.59%Mg–0.98%Si–0.51%Cu–
0.11%Fe–0.14%Cr (wt.%) was studied. The hot-rolled alloy (t = 15 mm) was put through
annealing at 400 °C for 1 h and cold rolling. This process was repeated twice to achieve
the respective thickness (t = 1.43, 1.10 and 1.00 mm). Solution heat treatment at 560 °C for
30 min was conducted in a salt bath. Subsequently, the alloys were quenched in ice water
before they were cold rolled to achieve thickness of 1 mm. The reduction ratio of each alloy
after solution heat treatment was 30%, 10% and 0%. Each cold-rolled sample is referred
to as 30CR or 10CR sample. The natural aging was carried out at a room temperature
for 2 weeks after cold rolling. As reference samples, pre-aging at 100 °C for 3 h in an oil
bath was put through for some 0% cold-rolled samples. After those, the artificial aging
at 170 °C for different aging times was applied in another oil bath. Figure 1 shows each
thermo-mechanical process. Samples that were artificially aged immediately after solution
heat treatment are called T6 samples.

![Figure 1. Schemes of thermo-mechanical process (a) T6 and NA processes, (b) CR and CR + NA processes and (c) PA and PA + NA processes.](image)

The mechanical properties were evaluated by Micro Vickers hardness tests and tensile
tests. Micro Vickers hardness tests were carried out on the rolled surface with a con-
stant force of 200 g and dwell time of 15 s using MMT-X manufactured by Matsuzawa.
The measurements were taken five times per sample and the average value was used as
the measurement value. The graphs show 95% confidence intervals. Tensile tests were
conducted at a room temperature at a strain rate of 1.85 × 10⁻³ s⁻¹ using an Autograph
AG-X plus manufactured by Shimadzu. The specimens for tensile tests were punched
into the shape shown in Figure 2. The electrical conductivity measurements and differ-
ential scanning calorimetry (DSC) analysis were conducted to reveal the precipitation
behavior of samples. The electrical conductivity was measured once for one sample using
AutoSigma 3000 manufactured by GE. It is a device that measures electrical conductivity
of non-magnetic metals using electromagnetic induction. The electrical conductivity it
measures has the following relationship with electrical resistivity [20]:

\[
EC [\% IACS] = \frac{1.724}{\rho [\mu \Omega m]}
\]

The samples of DSC analysis were cylindrical with masses of 40 mg. DSC experiments
were put through an argon flow with heating rate of 10 °C/min using Rigaku DSC8230.
Samples were measured relative to a thermodynamically inert reference sample of pure
aluminum with equal geometry.
DSC8230. Samples were measured relative to a thermodynamically inert reference sample. Comparing 30% cold-rolled samples with or without natural aging, there is little difference in hardness after 0.3 ks of artificial aging time.

3. Results and Discussion

Figure 3 shows aging curves of T6, NA, 10CR + NA and PA + NA samples during artificial aging at 170 °C and a comparison of electrical conductivity after each thermomechanical process. Cluster formation generally lowers electrical conductivity because clusters have more electron scattering capacity than solute atoms [25]. The increase in hardness and decrease in electrical conductivity of the A.Q. sample during natural aging are due to Cluster(1) formation. The A.R. (as-rolled) sample also hardened and decreased electrical conductivity during natural aging, thus Cluster(1) is expected to form even after cold rolling. On the other hand, both hardness and electrical conductivity of PA samples did not change during natural aging; pre-aging prevents formation of Cluster(1). Since hardness of NA samples changed little until 1.8 ks of artificial aging time, Cluster(1) obstructed the initial precipitation behavior. In contrast, hardness of PA + NA samples increased steadily in the early stage of artificial aging, thus pre-aging is effective in preventing the negative effects of natural aging. However, the peak time of the PA + NA hardness curve is same as that of NA, and it is the longer side than that of T6. Therefore, even the precipitation behavior of PA + NA samples is later than that of T6. On the other hand, the peak of 10CR + NA is 10.8 ks of artificial aging time; the same as that of T6.

Figure 4 shows aging curves of 10CR, 10CR + NA, 30CR and 30CR + NA samples during artificial aging at 170 °C. Comparing 30% cold-rolled samples with or without natural aging, there is little difference in hardness after 0.3 ks of artificial aging time. Concerning 10% cold-rolled samples, hardness of 10CR + NA samples is only slightly lower than that of 10CR after 0.3 ks of artificial aging time. The hardness of CR + NA samples decreased during artificial aging before 0.3 ks, thus there is a possibility that Cluster(1) formed during natural aging dissolved during artificial aging at 170 °C.
Concerning 10% cold-rolled samples, hardness of 10CR + NA samples is only slightly lower than that of 10CR after 0.3 ks of artificial aging time. The hardness of CR + NA samples decreased during artificial aging before 0.3 ks, thus there is a possibility that lower than that of 10CR after 0.3 ks of artificial aging time. The hardness of CR + NA samples decreased during artificial aging before 0.3 ks, thus there is a possibility that lower than that of 10CR after 0.3 ks of artificial aging time. The hardness of CR + NA samples decreased during artificial aging before 0.3 ks, thus there is a possibility that lower than that of 10CR after 0.3 ks of artificial aging time. The hardness of CR + NA samples decreased during artificial aging before 0.3 ks, thus there is a possibility that lower than that of 10CR after 0.3 ks of artificial aging time.

Figure 5 shows the results of the DSC analysis of samples without AA (artificial aging) or with AA for 0.3 ks. The main endothermic and exothermic peaks of the A.Q. sample are indicated in Figure 5a [1,14,19]. The peak of cluster formation disappeared from DSC curves of NA, 10CR + NA and 30CR + NA samples, indicating that cluster formation occurred during natural aging. Regarding PA samples, since the peak of cluster formation did not disappear from the DSC curve, clusters formed during pre-aging. There is no difference between the curves of PA and PA + NA. Therefore, similar to the hardness test, the results show that pre-aging prevented the effect of natural aging. The temperature of peaks of CR samples is lower than that of samples without cold rolling. It is reported that the diffusion coefficient of solute atoms affects the behavior of precipitation and dissolution [26]. In addition, it is known that the lattice defects such as dislocations and grain boundaries promote diffusion [27]. For these reasons, the peaks of DSC curves in CR samples are considered to shift to the lower temperature. The hardness of CR samples peaked in shorter time for the same reason. It is worth noting that the peak of cluster dissolution was shifted to the lower temperature side.

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Table 1 shows the peak temperatures of clusters dissolution of NA and CR + NA samples. The temperatures indicate when the cluster dissolution peak had the lowest heat flux. The largest cause of the negative effect of natural aging on the precipitation behavior is that Cluster(1) did not dissolve during artificial aging at 170 °C. In fact, the peak
temperature of the NA sample is 40 °C higher than the temperature of artificial aging. However, the peak temperatures of CR + NA samples decreased by cold rolling.

**Table 1.** Peak temperature of cluster dissolution of NA, 10CR + NA and 30CR + NA samples.

| Sample                  | Peak Temperature [°C] |
|-------------------------|-----------------------|
| NA                      | 217.2                 |
| 10CR + NA               | 186.5                 |
| 30CR + NA               | 183.4                 |

Figure 6 shows the enlarged cluster dissolution peak of NA, 10CR + NA and 30CR + NA samples without AA or with AA for 0.3 ks. Table 2 shows the amount of endothermal heat by cluster dissolution. These were calculated from area bounded by a straight line drawn horizontally from the point just before the start of the endothermic and the DSC curves. For samples without AA, the amount of heat decreased as the reduction ratio increased. It indicates that Cluster(1) formation during natural aging was suppressed by cold rolling. 30CR + NA samples aged artificially for 0.3 ks did not have a cluster dissolution peak, thus it is considered that Cluster(1) dissolved during artificial aging at 170 °C for 0.3 ks. For NA samples, the amount of heat decreased greatly by artificial aging for 0.3 ks. However, the main decrease was in the area below 170 °C, while the dissolution peak remained largely above 170 °C. For 10CR + NA samples, there was only a slight decrease in the amount of heat. Since the peak temperature of 10CR + NA samples was almost the same as that of 30CR + NA samples, the small decrease in the amount of heat is considered to be due to the fact that it took time for Cluster(1) dissolution during artificial aging. It is also the reason why the hardness of 10CR + NA samples was slightly lower than that of 10CR samples. The decrease in Cluster(1) formation was also considered to be a major factor that prevented the negative effect of natural aging in 10CR + NA samples.

**Figure 6.** Enlarged cluster dissolution peaks of DSC curves in (a) NA and NA + AA for 0.3 ks samples, (b) 10CR + NA and 10CR + NA + AA for 0.3 ks samples and (c) 30CR + NA and 30CR + NA + AA for 0.3 ks samples.

**Table 2.** Amount of endothermal heat by cluster dissolution in NA, 10CR + NA and 30CR + NA samples without artificial aging (AA) or with AA for 0.3 ks (J/g).

|                  | Without AA | AA for 0.3 ks |
|------------------|------------|---------------|
| NA               | 5.37       | 1.70          |
| 10CR + NA        | 1.03       | 0.66          |
| 30CR + NA        | 0.85       | -             |

The results of the hardness test and electrical conductivity measurement show that Cluster(1) was formed during natural aging even after cold rolling. However, 10CR + NA
samples were hardly affected by the negative effects of natural aging, and 30CR + NA samples were not affected at all. This lack of effect is due to the fact that cold rolling lowered the temperature of Cluster(1) dissolution, allowing the samples to dissolve during artificial aging at 170 °C. The 30CR + NA sample suppressed completely the negative effect of natural aging, but hardening of 30CR + NA samples during artificial aging was smaller than that of 10CR + NA samples. On the other hand, there is only a slight decrease in hardness due to natural aging of the 10CR + NA samples, and they mostly suppressed its negative effect. With these results, 10% rolling is considered to be better condition.

Figure 7 indicates the results of the tensile tests of T6, NA, 10CR + NA and PA + NA samples with and without artificial aging for 10.8 ks. For cold-rolled samples, tensile tests were conducted only on better condition 10CR + NA samples. The 10CR + NA samples had the lowest elongation in the tests at each artificial aging time. This caused higher dislocation density by cold rolling. It is expected that the elongation becomes lower as the reduction ratio increases. For the 10.8 ks aged samples, since the 10CR + NA sample was harder than the PA + NA sample in the hardness test, the UTS of the 10CR + NA sample was expected to be higher than that of the PA + NA sample, but it was comparable. This is because working hardening did not occur so much in the 10CR + NA sample during the tensile test due to high dislocation density by cold rolling. For the samples without artificial aging, the yield stress of the 10CR + NA sample is the highest. Since this alloy is intended to be heat-treated for paint-baking after forming, lower yield is required before artificial aging for easy processing. Therefore, there is an issue with ease of processing alloys cold-rolled more than 10% in industrial production. The yield stress is also expected to increase as the reduction ratio increases.

![Figure 7. Tensile properties of (a) T6, NA, 10CR + NA and PA + NA samples aged artificially for 10.8 ks and (b) NA, 10CR + NA and PA + NA without artificial aging.](image)

Cold rolling suppressed cluster formation and lowered the temperature of Cluster(1) dissolution. As shown in the results of DSC measurements, cold rolling also accelerated the precipitation behavior in Al–Mg–Si alloys. This is because the diffusion of solute atoms is promoted by lattice defects introduced by cold rolling. It was reported that the growth rate of precipitates on the dislocation was too large due to particularly fast diffusion [8,12]. As shown in Figure 3a, since the peak hardness of the 10CR + NA sample appeared earlier than the others, it is considered that its precipitation was more advanced compared at same aging time for 10.8 ks. However, cold rolling decreased the number density of precipitates because cluster formation was suppressed. Results show that the precipitates become coarse and low density by cold rolling [12,15]. In the results of hardness tests, the amount of hardening during artificial aging was smaller for the 30CR sample than for the 10CR sample. Since the precipitation strengthening decreases as the number density
of precipitates decreases [20], the strengthening decreases as the reduction ratio increases. Therefore, cold rolling at a 10% reduction rate is a favorable process to suppress the negative effects of natural aging for short aging times of around 10.8 ks.

4. Conclusions

In this study, the effect of cold rolling on Cluster(1) dissolvability and formability in Al-Mg-Si alloy was researched by evaluating the precipitation behavior of cold-rolled samples in order to illuminate whether cold rolling is effective in preventing the negative effect of natural aging. The evaluation was done by Micro Vickers hardness tests, tensile tests, electrical conductivity measurements and differential scanning calorimetry analyses. Cold rolling suppressed the amount of Cluster(1) formation since excess vacancies disappeared at dislocations introduced by cold rolling. Cold rolling lowered the temperature of Cluster(1) dissolution. This is probably because the diffusion was promoted by introduced lattice defects. The negative effect of natural aging was almost suppressed in 10% cold-rolled samples and completely suppressed in 30% cold-rolled samples, thus Cluster(1) in cold-rolled samples was supposed to dissolve during artificial aging at 170 °C. Cold rolling also accelerated the precipitation behavior. Since acceleration of precipitation leads to a decrease in the number density of precipitates, the strengthening during artificial aging became smaller as the reduction ratio increased. Therefore, the reduction ratio of 10% was better than 30%. The hardness of 10% cold-rolled samples reached the peak with artificial aging at 10.8 ks, earlier than samples without cold rolling. Thus, after a short artificial aging term of about 10.8 ks, the hardness of 10% cold-rolled samples had higher hardness than pre-aged samples.

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