Evaluation of AE Sources during Tensile Deformation of Al-Mg-Si Alloys with Different Heat Treatment

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Abstract
Acoustic emission (AE) behaviour during tensile deformation of Al-Mg-Si alloy has been investigated. We prepared some kinds of the Al-Mg-Si alloy with three different chemical compositions, consisting of magnesium and silicon balanced, excess silicon and excess copper alloys. As for the heat treatment, the specimens were solution-treated at 540°C for 1 hour, aged at 175°C for 30 min and finally aged at room temperature for 7 days (T4 treatment). The two cooling rates of water quenching and air-cooling after solution treatment were selected. As a result, the tensile property and AE behaviour were changed with the change of the cooling rate. From SEM observation, the fracture surface by the air-cooling after the solution treatment was covered with intergranular fracture because the ductility declines. The AE events of the air-cooled specimens during tensile deformation were detected in the elastic region. The source rise time was analyzed using the S₀ mode Lamb waveform of the AE, and appearances were also observed using the specimen also SEM, TEM and optical microscope.

In this paper, we focused on and estimated the AE sources observed during the elastic deformation. It was clear that the AE source was the micro-intergranular fracture depending upon the precipitates in the grain boundary of the copper excess specimen air-cooled.

Keywords: Al-Mg-Si alloy, AE during deformation, heat treatment, Lamb wave, waveform analysis

1. Introduction
Recently the weight of the car is lightening for CO₂ reduction as one of the global warming countermeasures. The Al-Mg-Si alloy (6000 series) is applied to automotive body panels to increase a mileage. This alloy is one of the heat-treatable aluminum alloys. The heat-treated aluminum alloy is strengthened by intended secondary-phase particles precipitated when heat treatments such as solution treatment, quenching and aging, were properly performed. However, it is reported that the intergranular fracture is easy to generate when the cooling rate after the solution treatment is low because of thick and large materials [1]. In fact, we have confirmed in the past study [2] that the intergranular fracture was generated due to air-cooling process after solution treatment in the Al-Mg-Si alloy with Cu additive and the characteristic acoustic emission (AE) behavior could be observed during the tensile deformation, especially in the elastic region. The AE events in the specimens due to water-quenching process after the solution treatment could not be observed in the elastic region but observed during the yielding.

In this paper, we prepared some kinds of Al-Mg-Si alloys with different additive elements, consisting of Mg and Si balanced, excess Si additive and Cu additive. The air- or water-cooling after the solution treatment were adopted. We will discuss the effect of the different additive element and the different cooling rates after the solution treatment on the mechanical property and the AE behavior during tensile deformation. Especially we will discuss the AE source detected in the elastic region using the AE waveform analysis [3], an optical, SEM and TEM observation.

2. Experimental Procedure

2.1 Specimen and heat treatment
Three types of specimens with different chemical compositions (A3, A4 and A8) were used in this study. The chemical compositions of these specimens are shown in Table 1. Specimen
A3 is with Mg and Si additives balanced for Mg$_2$Si precipitate forming, specimen A4 is with excess Si additive, and specimen A8 is with balanced and Cu additives. These specimens for tensile test were prepared using an electrical discharge machine (Brother HS-300). The shape of the specimen is shown in Fig.1. The tensile direction was vertical to the rolling direction of the Al-Mg-Si alloy plates.

The specimens were solution-treated at 540°C for 1 hour, air-cooled and water-quenched after the solution treatment, naturally aged for 7 days and aged at 175°C for 30 min (Bake hard treatment).

2.2 Tensile test and AE measurement

Tensile test was carried out using Instron-type machine (Shimadzu Autograph) at strain rate of $6.7 \times 10^{-6}$ s$^{-1}$ at ambient temperature.

Experimental setup for AE measurement during tensile test is shown in Fig. 2. We attached two AE sensors (Fuji Ceramics M5W) separated by 71 mm from the center of the specimen. AE measurement was made using 2-channel AE monitoring system to discriminate the grip noise. The threshold level was 40dB. The AE during the tensile test was transformed into the electrical signal by AE sensors, and then the signal was amplified by a constant gain of 40dB and passed through a band-pass filter of 100Hz to 20MHz (NF 9913). Finally AE waveforms were sent to the digital multi-recorder (Keyence GR-7000) in which the sampling time was 50ns, and were analysed off-line.

2.3 Optical, SEM and TEM observations

Surface appearance was observed after corroded in the solution with distilled water of 166ml, nitric acid of 20ml and hydrogen fluoride of 14ml using a digital optical microscope (Keyence VHX-600). Fracture surface was observed and qualitative EDX analysis was conducted using FE-SEM (Hitachi S-4700). Observation of precipitates and dislocations was conducted using TEM (Hitachi H-800) after a twin jet electro-polishing at -12.0°C on 10.5V in the solution with perchloric acid of 100ml, glycerin of 200ml and methyl alcohol of 700ml.

3. Results and Discussion
3.1 Tensile properties and fracture appearances

The tensile properties of each specimen are shown in Fig. 3. It was shown that the yield strength and the tensile strength of specimens air-cooled after the solution treatment were higher than those water-quenched. Especially the yield strength air-cooled was higher 43MPa in specimen A3, 32MPa in specimen A4 and 59MPa in specimen A8 than those water-quenched, respectively. The yield strength and the tensile strength in specimen A8 with excess Cu additive were much higher than those in the other. It was considered that the age hardening increased with Cu additive [4]. In contrast, the fracture strain in the specimens air-cooled were lower about 9% in specimen A3, about 13.5% in specimen A4 and about 12.7% in specimen A8 than those water-quenched, respectively. It was confirmed that the three specimens air-cooled showed very low ductility compared with those water-quenched, especially the lowest ductility in specimen A8 air-cooled because of the excess Cu additive.

The typical fracture surfaces in specimen A3, A4 and A8 are shown in Fig. 4. The fracture surface water-quenched was covered with large and deep transgranular dimples as shown in the lower parts of Fig. 4. The secondary-phase particles were not observed at the bottom of
the transgranular dimples. On the other hand, the fracture surface air-cooled was covered with intergranular fracture and the surface consisted of small and shallow intergranular dimples as shown in the upper parts of Fig. 4. A lot of secondary-phase particles in the size of about 0.5µm were observed at the bottom of the intergranular dimples as shown in Fig. 5. The fracture surfaces in specimens A3 and A4 water-quenched and air-cooled showed the similar aspect in specimen A8, respectively. The surface appearances in specimens A8 and A4 air-cooled are shown with the qualitative EDX analysis results in the grain boundary in Fig. 6. The typical grain boundary precipitation can be seen in the air-cooled specimens, although there is no precipitation along the grain boundary in the water-quenched specimens. The elements of the secondary-phase particles observed in specimen A8 air-cooled contain Al, Mg, Si and Cu in Fig. 6(a) that may form quadri-precipitate (Q phase) and Mg_2Si, whereas the elements observed in specimen A4 air-cooled contain Al, Mg and Si in Fig. 6(b) that may form ternary precipitate, Mg_2Si and excess Si.

These results were summarized as follows. A large number of secondary-phase particles including Al, Mg, Si and Cu elements were precipitated during air-cooling. Because the secondary-phase particles work as precipitate strengthening, the proof stress and the tensile strength show higher values. However, these precipitates generated along the grain boundaries lead easily to the intergranular cracking. As a result, the fracture surface in the specimens air-cooled is covered with small and shallow dimples.

3.2 AE activities during tensile test

The AE event rates during tensile test in specimen A3, A4 and A8 air-cooled and water-quenched are shown with the stress-displacement curves in Fig. 7 and 8, respectively. The

![Fig. 5 Typical example of intergranular fracture in the specimen A8 air-cooled](image)

![Fig. 6 Surface appearances in specimen (a) A8, (b) A4 air-cooled and the EDX analysis of the precipitates along the grain boundaries](image)

![Fig. 7 AE event rates during tensile test in specimen A3, A4 and A8 air-cooled with the stress-displacement curves. The number in the bracket shows the total event count.](image)
proportion of the event count detected at the displacement of 0.1 mm intervals to the total event count. The event rate in the air-cooled specimens is detected in the elastic region and around yield point in Fig. 7. The peak event rates in the elastic region that occupy the values of 40% to 50% shows the very high level compared to the peak rate around yield point. The AE event rate behavior in the elastic region was same in the air-cooled specimens regardless of the sort of the additive element. On the other hand, the event rate around yield point in the water-quenched specimens almost shows the peak rate of 10% to 15% in Fig. 8. The AE source at the peak of the event rate is considered to be due to the collective motion of dislocations. The event rate in all water-quenched specimens decreases with the deformation in progress, which is explained by decrease of mean free path of dislocations [5].

3.3 Source specification of AE events detected in the elastic region
Several experiments have been conducted to specify the source of AE events detected in the elastic region during tensile deformation in the air-cooled specimens.

The comparison of the median frequency distribution of the each waveform detected in specimen A8 air-cooled with that in specimen A8 water-quenched is shown in Fig. 9. The median frequency means an average frequency represented in a waveform. The waveform in

![Fig. 8 AE event rates during tensile test in specimen A3, A4 and A8 water-quenched with the stress-displacement curves. The number in the bracket shows the total event count.](image)

![Fig. 9 Comparison of the median frequency distribution of the each waveform detected in specimen A8 air-cooled with that in specimen A8 water-quenched](image)

![Fig. 10 Difference between the stresses at the peak event rate and at the elastic limit in the air-cooled specimens](image)
the elastic region is selected in the air-cooled and the in the plastic region is selected in the water-quenched because of no AE event in the elastic region. The higher frequency components in the water-quenched are more dominant than those in the air-cooled. It is suggested that the AE source in the elastic region is different from that during plastic deformation.

The difference between the stresses at the peak event rate and at the elastic limit in the air-cooled specimens is shown in Fig. 10. The AE event rate reaches to the peak before the elastic limit in all air-cooled specimen. It is surely demonstrated that we can detect AE events in the elastic region during tensile test of this aluminum alloys. The AE event in the air-cooled specimen A8 with Cu additive can be obtained easily in the elastic region because of the larger difference between the stresses at the peak event rate and at the elastic limit as shown in Fig. 10.

We tried to introduce the AE source rise time to characterize the AE source in the elastic region. The AE source rise time was analyzed using the estimation method proposed by T. Yasuda et al. [3] that was obtained using the AE waveform detected during martensitic transformation of Cu-Al-Ni shape memory alloy single crystal. The source rise time represents the rise time of the step function in the AE source. The relationship between the source rise times analyzed by AE waveform detected in specimen A8 air-cooled and water-quenched is shown with the stress-displacement curves in Fig. 11. The source rise time in the air-cooled is concentrated on 1.2~1.7µs in Fig. 11(a) but in the water-quenched is dispersed

(a) Specimen A8 air-cooled (b) Specimen A8 water-quenched

Fig. 11 Relationship between the source rise times analyzed by AE waveform detected in specimen A8 (a) air-cooled and (b) water-quenched with the stress-displacement curves

Fig. 12 Surface appearance (a) before the tensile deformation and (b) after the peak event rate in the elastic region. Photo (c) shows the photograph multiplied 5 times near the intergranular crack in the Photo (b)
in Fig. 11(b). The source rise time in the elastic region of the air-cooled is same as that in the final failure as shown in Fig. 11(a), so it is possible for the AE source in the elastic region to be the intergranular fracture because of the predominantly intergranular fracture in the final failure as mentioned previously. It is clear that the collective motion of dislocations have the extended source rise time in Fig. 11(b).

The surface appearance (a) before the tensile deformation and (b) after the peak event rate in the elastic region is shown in Fig. 12. We can find out the micro-intergranular fracture in the several parts of the surface as shown in Fig. 12(b). This agrees with the result of the source rise time and is considered to be one of the important evidences for the AE source in the elastic region.

4. Conclusions

The change of the mechanical properties and AE activities in the specimens air-cooled and water-quenched after the solution treatment has been examined during tensile test of Al-Mg-Si alloys with different additives. Especially, the experiment has been conducted in detail to specify the generation of the AE event in the elastic region and the AE source. The results obtained are as follows.

(1) The air-cooled specimen showed higher proof stress, tensile strength and lower ductility than the water-quenched one, which led to the intergranular fracture due to the precipitates segregated along the grain boundaries.

(2) In the case of the air-cooled specimens, the peak AE event rates were shown in the elastic region.

(3) The AE source rise time near the peak of the event rate agreed with that in the final failure.

(4) From the surface appearance observation after the peak event rate, the several micro-intergranular fractures could be found in specimen A8 air-cooled with Cu additive.

(5) As a result, the AE source in the elastic region was considered to be micro-intergranular fracture.

References