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Effects of Clusters and Precipitates on Tensile Properties in Al-Mg-Si Alloys

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Tensile tests and microstructural characterizations were carried out to clarify effects of clusters and of precipitates on tensile properties in Al-Mg-Si alloys. The alloys were isothermally aged at 373 K for 120 h and at 453 K for 20 h, in order to obtain the same hardness. It was revealed by transmission electron microscopy (TEM) that there were large numbers of rod-like β” phase precipitates along <100> directions in the 453 K aged alloy, while there was no β” phase precipitated in the 373 K aged alloy. Three-dimensional atom probe microscopy (3DAP) was therefore applied to characterize the 373 K aged alloy and confirmed the presences of Mg-Si clusters. Stress-strain curves showed that, in the case of 453 K aged alloy, the yield stress was higher and the total elongation was smaller than that of the 373 K aged alloy. TEM observation of 10 % tensile deformed samples showed that dislocations were pinned by β” precipitates in the 453 K aged alloy, while wavy dislocations and dislocation loops were observed in the 373 K aged alloy. These results indicate that clusters and precipitates give the different tensile properties, in particular, the yield stress and the elongation.

Keywords: *Al-Mg-Si alloy, tensile property, Mg-Si cluster, β”precipitate, TEM*

1. Introduction

For environmental and energy saving problems, automotive industries tends to use light materials to replace heavy materials. From this point of view, Al-Mg-Si alloy is a best candidate for automotive body applications, due to its formability, corrosion resistance and strength. For applying the alloy to an automotive body sheet which is manufactured by a pressing and a painting, therefore, a formability and age-hardening property at a paint-bake temperature are important. Aging process in Al-Mg-Si alloys have been studied by many researchers from the viewpoint of precipitation [1-6]. Edwards *et al*. observed the formation of Mg-Si clusters at temperatures lower than 343 K, while Mg-Si precipitates at temperatures ranging from 343 to 453 K in Al-Mg-Si alloys [1]. Murayama *et al*. [2, 3] and Serizawa *et al*. [4] reported the presence of Si clusters, Mg clusters and Mg-Si co-clusters using atom probe field ion microscopy (APFIM), high resolution transmission electron microscopy (HRTEM) and three-dimensional atom probe microscopy (3DAP). The formation processes of clusters and precipitates, such as β”, β’ and β (Mg_2Si), were also studied using differential scanning calorimetry (DSC) [4-6]. For practical applications of Al-Mg-Si alloys, influences of the formation of clusters and precipitates on mechanical properties are very significant subject but rarely considered with care.

In this study, influences of clusters and precipitates on mechanical properties in Al-Mg-Si alloys were investigated. Age-hardening behavior and tensile properties were examined, and microstructural observations were performed by TEM and scanning transmission electron microscopy (STEM).

2. Experiment

Al-Mg-Si alloy was prepared by Furukawa-Sky Aluminum Corp. for the experiment. The chemical composition of the alloy is listed in Table 1. The alloy was cast, homogenized at 803 K for 6 h, cold-rolled into 1 mm thickness and solution-treated at 823 K for 0.5 h in air. To investigate age-hardening behavior, the alloy was isothermally aged at 373 K or 453 K, and then Vickers hardness tests were carried out under an 1 kg load by MVK-G3, Akashi, Japan.

Table 1 Chemical composition (wt. %) of Al-Mg-Si alloy.

Cu	Si	Fe	Mn	Mg	Zn	Cr	Ti	Al
0.05	0.32	0.13	0.05	0.62	0.01	0.04	0.01	Bal.

Figure 1 shows the age-hardening behavior at 373 K and at 453 K. The 453 K aged alloy began to strengthen earlier but shows lower plateau than the 373 K aged alloy. Hereinafter, two particular alloys with different aging conditions exhibiting the same hardness about 70 Hv will be compared, namely the alloy isothermally aged at 373 K for 120 h and that at 453 K for 20 h, which are labeled as "373 K sample" and "453 K sample", respectively.

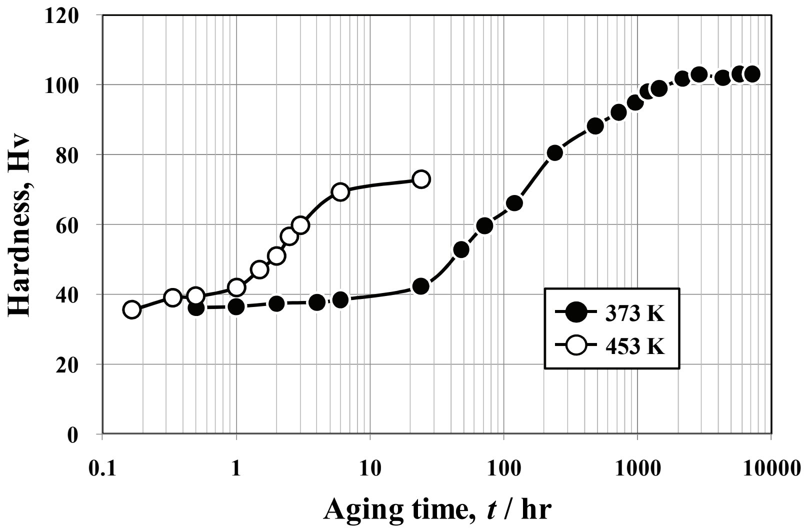

Fig. 1 The aging curves at 373 K and 453 K of Vickers hardness.

3DAP measurement was conducted by Three Dimensional Atom Probe (Oxford NanoScience Ltd., UK) to characterize Mg-Si clusters in 373 K sample. The analytical volume was 62 nm x 63 nm x 87 nm. DSC analysis was conducted in a temperature range from room temperature to 773 K at a heating rate of 20 $Kmin^{-1}$ by DSC8230 (Rigaku, Japan) to confirm the presence of Mg-Si precipitates in 453 K sample. Tensile tests of two samples were performed using Autograph AG-10TA (Shimadzu, Japan) with a tensile axis along the cold-rolling direction under a room temperature and a strain rate of 8.3×10^{-4} s^{-1}.

The specimens before and after tensile tests were cut and thinned down by mechanical polishing and twin-jet electro-polishing. TEM and STEM observations were carried out at 200 kV in JEM-2000EX/T (JEOL, Japan) and Tecnai-F20 (FEI, Netherland), respectively.

3. Results and discussions

Figure 2(a) and 2(b) show bright-field TEM images and selected area diffraction (SAD) patterns from 373 K sample and 453 K sample before the tensile test, respectively. Although the precipitates are not seen in Fig. 2(a), they are seen in Fig. 2(b) as many dot-like and strain contrasts. Therefore, it is considered that there are large numbers of rod-like precipitates along <100> in 453 K sample.

According to previous studies [1, 2], it is explained that clusters exist in 373 K sample, and that β" precipitates exist in 453 K sample.

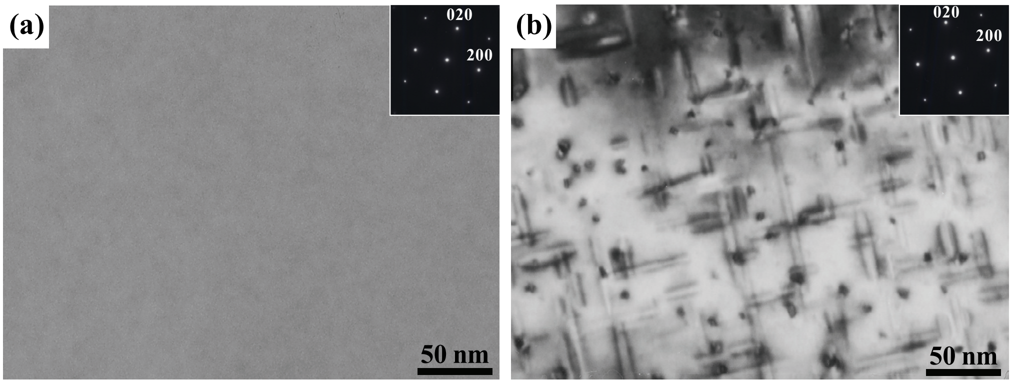

Fig. 2 TEM bright field images and SAD patterns of the grain in (a) 373 K and (b) 453 K samples.

A 3DAP result for 373 K sample is shown in Fig. 3. The blue and red dots indicate Mg and Si atoms, respectively. Typical condensed regions of Mg and Si atoms are indicated by arrows, which suggest the formation of Mg-Si clusters in the case of 373 K sample. Figure 4 shows a DSC curve for 453 K sample in a temperature range from 450 K to 775 K. The curve shows two exothermic peaks, B and D, and two dissolution cusps, A and C. According to previous studies [5, 6], these peaks and cusps are explained as follows: dissolution of β" precipitates at cusp A; formation of β' at peak B; dissolution of β' at cusp C; and formation of β at peak D. Based on the DSC result and the TEM observation, it is concluded that the rod-like precipitates in 453 K sample are β" precipitates. The regular arrays of dot-like contrasts in Fig. 2(b) are explained as cross-sectional image of the rod-like β" precipitates.

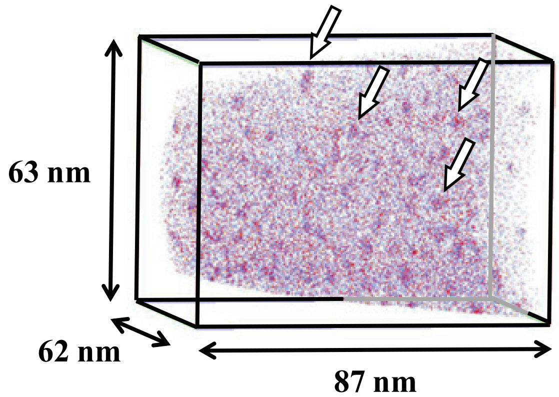

Fig. 3 Mg and Si atom map of 373 K sample. The clusters are recognized as white arrows.

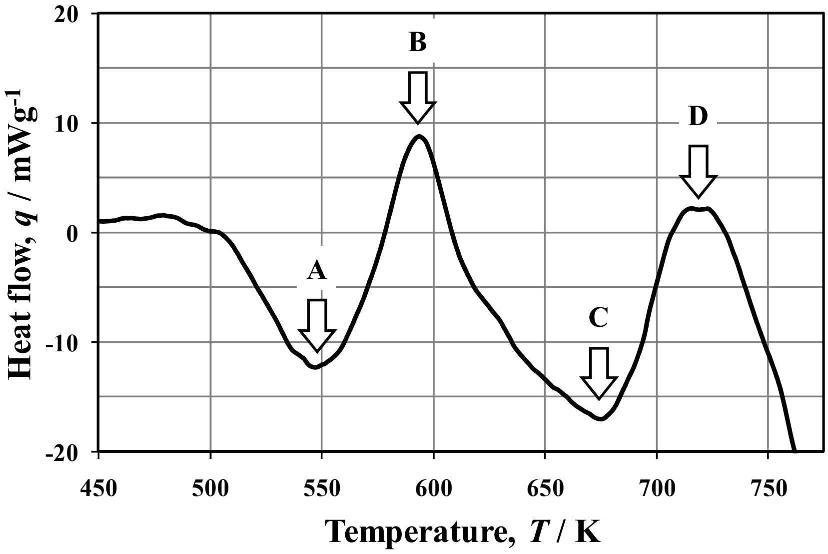

Fig. 4 DSC curves of 453 K sample.
Two exothermic peaks B and D and two dissolution cusps A and C are detected.

Figure 5 shows typical stress-strain curves of 373 K sample, 453 K sample and a solution treated (ST) sample. The maximal tensile stresses of 373 K sample and 453 K one are higher than that of ST sample. Although the microstructures of the 373 K and 453 K samples are different evidently, the maximal tensile stresses of these samples are almost the same, because these samples were aged in order to achieve the same hardness. On the other hand, the yield stress of these three samples are totally different, 172 MPa for 453 K sample, 145 MPa for 373 K sample, and 39 MPa for ST one. The total elongation of 373 K, 453 K and ST samples are 35.2 %, 21.6 % and 52.4 %, respectively. It is apparently shown that the effects of clusters, β” precipitates and solute atoms are different.

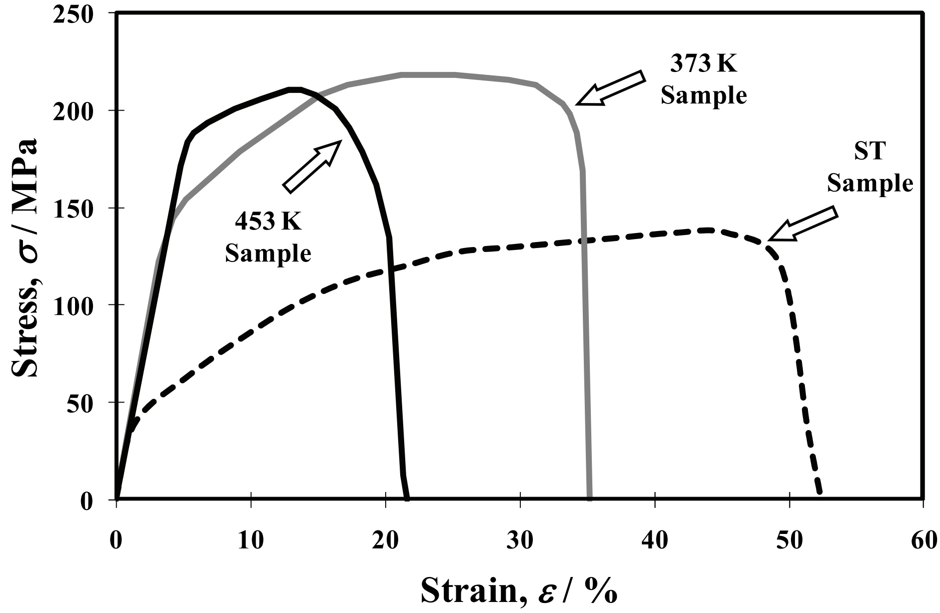

Fig. 5 Stress-strain curves of 373 K, 453 K and solution treated samples.

In order to clarify mechanisms of the different tensile properties, microstructures in 10 % tensile deformed specimens of 373 K, 453 K and ST samples were observed. Figure 6 shows a STEM bright-field image of 373 K sample after 10 % tensile deformation, in which wavy and loop-like dislocations are seen. A STEM bright-field image of 453 K sample after 10 % tensile deformation is shown in Fig. 7, where large numbers of dislocations interacting with β” precipitates are seen as indicated by arrows. Furthermore, it was found that wavy and loop-like dislocations rarely present in ST sample after 10 % tensile deformation by using STEM. Delmas et al. reported that precipitates, such as β” precipitates, in Al-Mg-Si alloy were sheared or by-passed by dislocations, and also mentioned that the formation of dislocation loops occurred

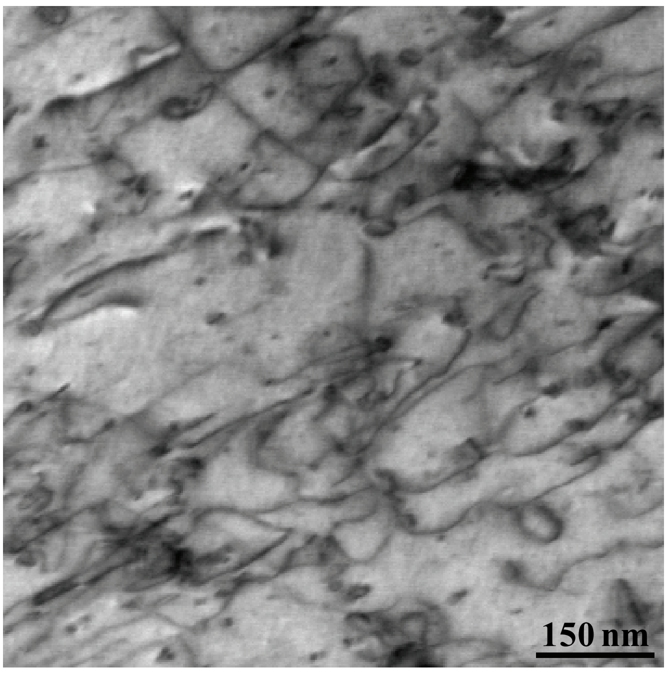

Fig. 6 STEM bright field image of 373 K sample. Wavy and loop-like dislocations are observed.

by cross-slip in case of the by-passing mechanism. When the shearing mechanism occurs, dislocations are bowed without forming dislocation loops around the precipitates [7]. In this study, in 373 K sample which has large numbers of clusters, many loop-like dislocations and wavy dislocations were observed. It is considered that by-passing and shearing mechanisms occurred in 373 K sample simultaneously. On the other hand, in 453 K sample, β" precipitates pinned the dislocations. It is assumed that the shearing mechanism or the Orowan mechanism occurs in 453 K sample, and that the yield stress is higher than ST sample.

From these results, it is concluded that the maximal tensile stress of Al-Mg-Si alloy with clusters or precipitates are higher than solute treated alloy by shearing and by-passing age-hardening mechanisms, and that the cross-slip makes the total elongation of Al-Mg-Si alloy with clusters larger than Al-Mg-Si alloy with precipitates.

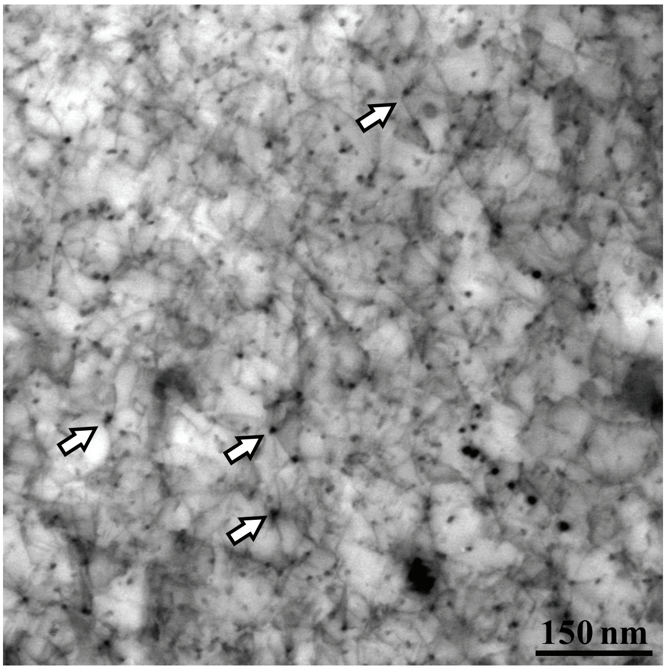

Fig. 7 STEM bright field images of 453 K sample. Pinned dislocations by β" precipitates are observed as shown by arrows.

4. Conclusion

In this study, in order to clarify the effect of clusters and precipitates of Al-Mg-Si alloy on mechanical properties, the investigation of the age-hardening behavior and the tensile property and the observation of the microstructure before and after tensile test by TEM and STEM were carried out. The main results are as follows.

(1) From the age-hardening behavior at 373 K and 453 K, the 453 K-aged sample began to strengthen earlier but weaker than the 373 K-aged sample.
(2) The TEM observation of 373 K sample showed that there was no precipitate, however, the 3DAP analysis of this one revealed the presence of Mg-Si clusters.
(3) It was recognized that there were large numbers of β" precipitates in 453 K sample by TEM and DSC.
(4) The stress-strain curves of 373 K, 453 K and ST samples were different. The yield stress of 373 K and 453 K samples were higher than that of ST sample and the total elongation of 373 K sample being larger than that of 453 K sample.
(5) TEM observation of 373 K and 453 K samples after 10 % tensile deformation showed that the dislocations were pinned by β" precipitate in 453 K sample and that there were wavy dislocations and loop-like dislocations in 373 K sample.
(6) It was concluded that the maximal tensile stress of Al-Mg-Si alloy with clusters or precipitates were higher than solute treated alloy by shearing and by-passing age-hardening mechanisms, and that the cross-slip made the total elongation of Al-Mg-Si alloy with clusters larger than Al-Mg-Si alloy with precipitates.

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