Effects of Ag on Ag-Hardening Behaviour of Al-Mg-Si Alloys

Y. Zou¹, K. Matsuda², T. Kawabata², Y. Himuro³, S. Ikeno²

¹ Venture Business Laboratory, Toyama University, Japan ² Faculty of Engineering, Toyama University, Japan ³ Furukawa Electric Co., Ltd., Japan

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Abstract

In this study, we have investigated the precipitation of AI-0.67mass%Mg-0.77mass%Si-0.5mass%Ag alloy and AI-0.67mass%Mg-0.77mass%Si alloy by hardness measurement, and transmission electron microscopic (TEM). The Ag-bearing alloy has higher aging-hardness than that of Ag-free alloy at the same aging condition. TEM observation result shows there is smaller size and higher number density of precipitate in Ag-bearing alloy than those of Ag-Free. The typical aging precipitate has similar lattice parameters with β " phase by HRTEM observation. It is thought that Ag can provide the nucleation sites of cluster or GP zones, resulting in finement of the size of precipitate and increasing the number density of precipitate.

1. Introduction

Al-Mg-Si alloys (6xxx) own medium-strength structures with good weldability, corrosion resistance and high damping capacity [1]. The main aging precipitates are β'' or β' phase in Al-Mg-Si base alloy or Al-Mg-Excess-Si alloy, which have been studied by many researchers [2-6]. In order to improve their properties, other elements have been also added in Al-Mg-Si alloys. About these metal elements, there are many reports about the addition of copper (Cu) into Al-Mg-Si alloys to enhance their mechanical properties, especially their ductility [7-14]. There are a few reports of the effect of silver (Ag) on the age-hardening of this alloy, and it provides the improvement of mechanical property. However the mechanism of its improvement keeps being a secret and we do not know whether it is same with the effect of Cu on precipitation of Al-Mg-Si alloy. In an Al-1.9Cu-0.3Mg-0.2Ag (at.%) alloy, L. Reich et al. [14] have reported that co-clusters of Ag and Mg atoms were presented at the early stage of aging. It is possible that Ag addition can affect the formation of clusters in Al-Mg-Si alloy that forms a lot of Mg-Si cluster (GP zone) at the early stage of aging. It will influence the growth of precipitates if both Mg-Si cluster and Mg-Ag cluster are formed.

In present study, we have investigated the hardening response in Al-1%Mg2Si-0.60%Si -0.5%Ag alloy during artificial aging at 443K, and tried to understand the effects of Ag addition on aging response by the way of HRTEM and EDS. As a comparison, the Ag-Free Al-1%Mg2Si-0.60%Si is also studied under the same heat-treatment condition.

The compositions of the alloys used in this study are Al-1%Mg2Si-0.60%Si-0.5%Ag and Al-1%Mg2Si-0.60%Si (in wt%), which are cold rolled to form 0.2mm thick sheets, solution treated for 60min at 848K and quenched in ice water. After quenching, alloys are artificially aged at 443K and the samples are studied at various aging time. The TEM samples are made by a twin-jet electrical polishing technique using a nitric acid-methanol solution. The micro Vickers hardness of each alloy is measured with a load of 0.987N and a holding time of 15 seconds. The TEM observation is taken by a Topcon EM-002B HRTEM operated at 120kV, and the energy dispersive X-ray spectroscopy (EDS) analysis is performed by a TEM-ECON equipped with EM-002B.



3. Results and Discussion

Figure1: Aging hardening curves of the Ag-bearing and Ag-free specimens aged at 443K.

Figure 1 shows the variation of hardness with aging time at 443K for Ag-bearing and Ag-free alloy, that indicates a very strong effect of Ag addition on hardening response. The aging hardness was increased with Ag addition. At the beginning of aging stage, the value of hardness has a more quickly increase for the Ag-bearing alloy than that of Ag-free.

Figure 2-4 shows the bright field images and the corresponding [001]_{Al} selected area diffraction patterns of Ag-bearing specimen aged at 443K for 32min (under-aging), 1000min (peak-aging) and 10000min (over-aging), respectively. A lot of fine dot-shaped and needle-shape precipitates were observed. It shows that there is a higher number density of precipitate in Ag-bearing alloy than that of Ag-free alloy corresponding to the same aging stage. The statistical result of the number of precipitates was given in Figure 5. From Figure 4(b), it is noted that there are obvious coarse precipitate (marked by arrow) expect for needle-shaped precipitates. These coarse precipitate is Si-particle and it was confirmed by EDS analysis. Figure 6 shows its image and EDS result.



Figure 2: Transmission electron micrographs of the specimens (a) Ag-bearing and (b) Ag-free aged at 443K for 32min. The corresponding selected-area diffraction pattern is given as an inset.



Figure 3: Transmission electron micrographs of the specimens (a) Ag-bearing and (b) Ag-free aged at 443K for 1000min. The corresponding selected-area diffraction pattern is given as an inset.



Figure 4: Transmission electron micrographs of the specimens (a) Ag-bearing and (b) Ag-free aged at 443K for 10000min. The corresponding selected-area diffraction pattern is given as an inset.



Figure 5: Changes of the number per unit area for Ag-bearing and Ag-free specimens aged at 443K.



Figure 6: (a) The coarse precipitate in Ag-free specimen aged at 443K for 10000min. (b) The EDS analysis result shows it is Si-particle.

Figure 7 shows the typical HRTEM images of precipitate for Ag-bearing sample with aging 32min, 1000min and 10000min, respectively. The arrangement of bright dots in the image of Figure 7(a) forms a parallelogram network having the spacings of 0.72nm and 0.64nm with an interior angle of 75°. In Figure 7(b), the sides of parallelogram are 0.76nm and 0.68nm with an interior angle of 73°. In Figure 7(c), the arrangement of bright dots is a parallelogram network with the spacing of 0.68nm and 0.78nm and interior angle of 72°. These parameters of three kinds of precipitate are very close to that of β ° phase [4].



Figure 7: Higher resolution TEM images of the cross sections of precipitates in Ag-bearing alloy aged at 443K

for (a) 32min, (b) 1000min and (c) 10000min.

Figure 8 shows the average diameter of end-on precipitate and length of precipitate for Ag-bearing and Ag-free sample at different aging stage. The result shows that the Ag-bearing sample has a smaller size than that of Ag-free sample at under-aging and peak-aging stages. But at the over-aging stage, the length of precipitate in Ag-bearing sample is longer than that of Ag-free sample. That is due to Si precipitation, which reduces the size of precipitate in Ag-free alloy.



Figure 8: Changes of diameter of end-on precipitate and length of precipitate for Ag-bearing and Ag-free specimens at different aging stage.

Experimental results show that there is smaller size of precipitate and higher number density of precipitate in Ag-bearing sample than that of Ag-free sample. It is the reason why Ag-bearing sample shows a higher hardness than the Ag-free sample. L. Reich et al. [14] have reported that co-clusters of Ag and Mg atoms were present at the early stage of aging. In this study, we presume some Ag atoms can also trap some moving Mg atoms by forming Mg-Ag cluster when Mg-Si clusters was formed by diffusion at the early stage of aging. These Mg-rich clusters can form complicated precipitate more easily than single Mg atom during the later ageing stage. Compared with Ag-free sample, these Mg-rich clusters caused by Ag actually increase the number of nucleation sites of precipitate.

4. Conclusions

In this study, we have investigated the precipitation of AI-0.67mass%Mg-0.77mass%Si-0.5mass%Ag alloy and AI-0.67mass%Mg-0.77mass%Si alloy by hardness measurement and transmission electron microscopy. The Ag-bearing alloy has higher aging-hardness than that of Ag-free alloy at the same aging condition. TEM observation result shows there is smaller size and higher number density of precipitate in Ag-bearing alloy than those of Ag-Free. The typical aging precipitate has similar parameters with $\beta^{\prime\prime}$ phase by HRTEM observation. It is thought that Ag can provide the nucleation sites of cluster or GP zones, resulting to fine the size of precipitate and increasing the number density of precipitate.

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