A R C H I V E S O F M E T A L L U R G Y A N D M A T E R I A L S
Volume 60 2015 Issue 2
DOI: 10.1515/amm-2015-0244

K. WATANABE*,♯, K. MATSUDA**, S. IKENO***, T. YOSHIDA****, S. MURAKAMI****

TEM OBSERVATION OF PRECIPITATE STRUCTURES IN Al-Zn-Mg ALLOYS WITH ADDITIONS OF Cu/Ag

OBSERWACJE WYDZIELEŃ W STOPACH Al-Zn-Mg MODYFIKOWANYCH Cu/Ag METODĄ MIKROSKOPII TEM

Al-Zn-Mg alloy has been known as one of the aluminum alloys with the good age-hardening ability and the high strength among commercial aluminum alloys. The mechanical property of the limited ductility, however, is required to further improvement. In this work, three alloys, which were added Cu or Ag into the Al-Zn-Mg alloy, were prepared to compare the effect of the additional elements on the aging behavior. The content of Ag and Cu were 0.2at.% and the same as, respectively. Ag or Cu added alloy showed higher maximum hardness than base alloy. The particle shape and rod shape precipitates were observed in all alloys peak-aged at 423K. According to addition of Ag or Cu, the number density of the precipitates increased higher than that of base alloy.
Keywords: Al-Zn-Mg alloy, aging, precipitates, TEM, Cu and Ag addition

Al-Zn-Mg jest stopem, który można efektywnie poddawać procesowi utwardzania wydzieleniowego. Spośród różnorodnych stopów aluminium charakteryzuje się bardzo wysoką wytrzymałością mechaniczną, jednak jego ograniczona plastyczność jest cechą wymagającą poprawy. W toku prac przygotowano trzy stopy: stop bazowy Al-Zn-Mg oraz stopy zawierające dodatki Cu lub Ag, dla porównania wpływu tych dodatków na starzenie się materiału. Zawartość Ag i Cu wynosiła odpowiednio 0,2% at. Stopy z dodatkiem Ag lub Cu wykazywały większą twardość maksymalną w porównaniu ze stopem bazowym. We wszystkich stopach starzonych w temperaturze 423K obserwowano charakterystyczne wydzielenia, a ich koncentracja była odpowiednio większa dla stopów z dodatkiem Ag lub Cu.

1. Introduction

The Al-Zn-Mg based alloys are widely used in aircraft and automotive industries for structure components because of their exceptionally high strength to density ratio in the fully aged condition [1]. The usual precipitation sequence for the Al-Zn-Mg alloys is generally described as follows [2]:

Supersaturated Solid solution → GP zones → metastable η'→stable η($MgZn_2$).

At higher levels of Mg, i.e., lower Zn/Mg ratio in the alloy, the T phase ($Mg_3Zn_3Al_2$ or Mg_{32}((Zn, Al)$_{49}$) may also form [3]. Two types of GP zones, the spherical morphology GP(I) zone and the plate-like morphology GP(II) zone, appear during ageing [4]. The η' phase is the main hardening precipitate [5]. The η phase has nine different orientation relationship between η phase and the matrix [6]. It is well known that the addition of Cu is beneficial to increase maximum hardness [7]. Similar effects are also observed for the alloy containing small amount of Ag [7]. The aim of this work is the fundamental research to know the effect of Cu and Ag addition to Al-6.0mass%(2at%)Zn-2.0mass%(2at%)Mg-0.3mass%(0.3at%)Si alloy on the age-hardening behavior and the precipitates.

2. Experimental procedure

Al-6mass%Zn-2mass%Mg-0.3mass%Si alloy was used in this study, which was called as base alloy. Base alloy with 0.49mass%Cu and 0.75mass%Ag, which was called as Cu-addition alloy and Ag-addition alloy, respectively. The alloys were cast with permanent mold. Sheets with 1.5 mm thickness and 15 mm width were made by hot extrusion and then rolled to sheets with 1.0 mm thickness. The solution heat treatment at 748K for 3.6ks in an air furnace and quenched into water. The aging treatment was performed at 423K and 473K in oil bath. The micro-vickers hardness was measured with Mitsutoyo HM-101(load: 0.98 N, holding time 15s). Tensile specimens were cut from sheets with 0.8 mm×6.0 mm cross-section and 17.5 mm gauge length. Thin specimens for TEM observation were prepared by electrolytic polishing method and microstructures were observed by Topcon EM-002B operated at 120kV.

* GRADUATE SCHOOL OF SCIENCE AND ENGINEERING FOR EDUCATION, UNIVERSITY OF TOYAMA, 3190 GOFUKU, TOYAMA, 930-8555, JAPAN
** GRADUATE SCHOOL OF SCIENCE AND ENGINEERING FOR RESEARCH, UNIVERSITY OF TOYAMA, TOYAMA, GOFUKU, TOYAMA, 930-8555, JAPAN
*** HOKURIKU POLYTECHNIC COLLEGE, 1289-1 KAWABARI, UOZU, TOYAMA, 937-0856, JAPAN
**** AISIN KEIKINZOKU CO., LTD. 12-3 NAGONOE, IMIZU, TOYAMA, 934-8588, JAPAN
♯ Corresponding author: ikenolab@eng.u-toyama.ac.jp

3. Results and discussion

The maximum hardness for these alloys aged at 423K are as follows: base alloy: 130HV, Cu-addition alloy: 157HV and Ag-addition alloy: 159HV. The age-hardening results for the alloys aged at 473K are as follows: base alloy: 74HV, Cu-addition alloy: 114HV and Ag-addition alloy: 128HV. The Cu or Ag addition alloys exhibit higher hardness than the base alloy aged at 423K and 473K. Fig. 1 shows TEM bright field images tree alloys peak-aged at 423K. The incident beam directions are parallel to the $[0\ 0\ 1]_{Al}$ direction. Fig. 1 (a) shows results of base alloy. There are rod shaped precipitates and particle shaped precipitates in base alloy. The longitudinal direction of rod shape precipitates are parallel to $<1\ 0\ 0>_{Al}$ and 45° or 27° from $<1\ 0\ 0>_{Al}$. SAED pattern was consist of some precipitates. These diffraction spots from precipitates can be indexed as the η' phase and η_1 phase. The morphologies of η' phase and η_1 phase are plates on $\{1\ 1\ 1\}_{Al}$ and $\{1\ 0\ 0\}_{Al}$ respectively[6]. The rod shape precipitates parallel to $<1\ 0\ 0>_{Al}$ in $[1\ 0\ 0]_{Al}$ corresponds to that of η_1 phase. The rod shape precipitates aligning with 45° or 27° from $<1\ 0\ 0>_{Al}$ in $[1\ 0\ 0]_{Al}$ corresponds to that of η' phase. The particle shape precipitates corresponds to that of η' phase or η_1 phase. The number density of η' phase higher than that of η_1 phase in base alloy.

Fig. 1 (b), (c) shows TEM bright field image in Cu-addition alloy and Ag-addition alloy peak-aged at 423K. The rod and particle shape precipitates were observed in Cu and Ag addition alloys. The precipitates of Cu or Ag addition alloy were finer than that of base alloy. The number density of the precipitates in Cu and Ag addition alloy was much higher than that of the base alloy. The same type of diffraction spot as base alloy was observed in Cu-addition alloy. The η' phase and η_x phase were observed in Cu-addition alloy from SAED pattern. In addition, the diffraction spot of GP(‡T) zone were observed in Cu-addition alloy [4].On the other hand, the diffraction spot of GP(‡T) zone and η_1 phase were not observe in Ag-addition alloy. Only the diffraction spot of η' phase was observed.

Fig. 2 shows TEM bright image obtained from three alloys over-aged at 473K. The coarse particle shape precipitates were observed in base alloy (Fig. 2 (a)). The diffraction spots of η_1 phase and T(T') phase were observed in base alloy. Fig. 2 (b) shows the results of Cu-addition alloy. The coarse precipitate and fine precipitates were observed in Cu-addition alloy. The diffraction spots of η_1 phase, T(T') phase and Q(Q') phase were observed in Cu-addition

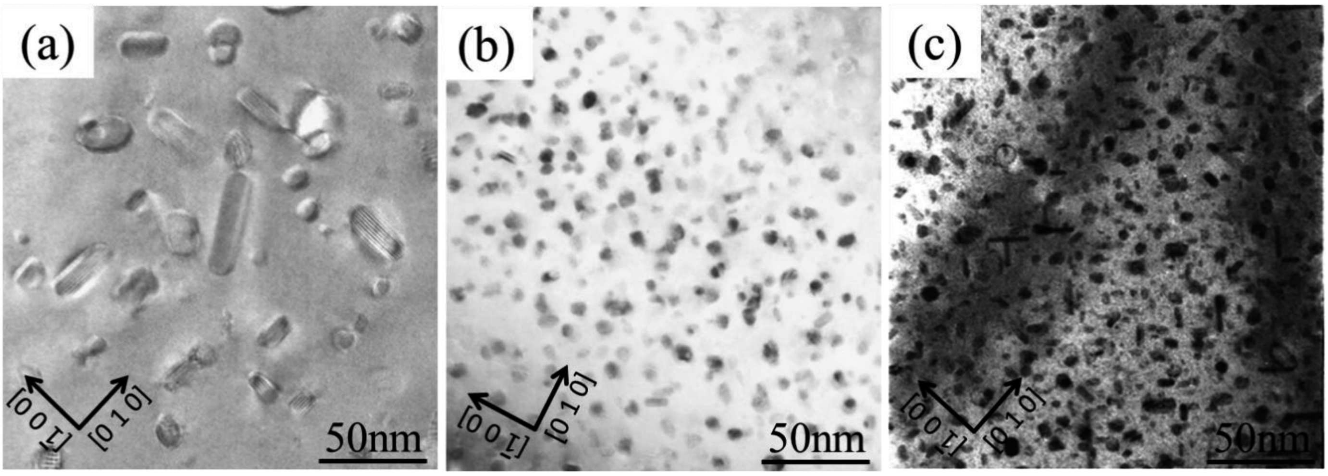

Fig. 1. TEM bright field images aged 60.0 ks at 423K. $[0\ 0\ 1]_{Al}$ projection. (a) base, (b) Cu-addition and (c) Ag-addition alloys

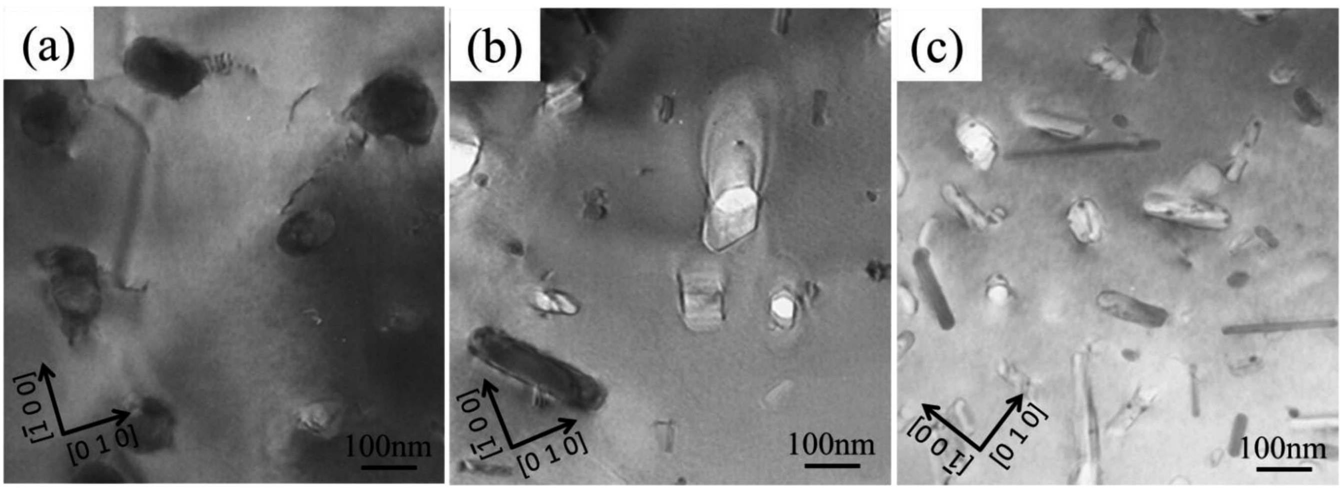

Fig. 2. TEM bright field images aged 60.0 ks at 473K. $[0\ 0\ 1]_{Al}$ projection. (a) base, (b) Cu-addition and (c) Ag-addition alloys

alloy. Fig. 2 (c) shows the results of Ag-addition alloy. The rod shape and particle shape precipitates were observed in Ag-addition alloy. The diffraction spots of η_1 phase and η' phase were observed in Ag-addition alloy.

4. Summary

The age hardening behavior and the microstructure of three different types of the Al-Zn-Mg alloys (Cu- and Ag-addition alloys and base alloy) have been investigated by means of TEM. The main results of the presented study are follows: Cu- and Ag-addition alloys exhibit higher hardness than base alloy. Cu- and Ag-addition alloy contain higher density of precipitates than reference base alloy. Cu- and Ag-addition alloys and reference specimen at peak aged conditions have been investigated by TEM and rot and particle shaped precipitates were observed. Analysis of the SAED patterns indicates that the base alloy contains η' phase and η_1 phase, the Cu-addition alloy contains GP(‡T) zone, η' phase and η_1 phase and the Ag-addition alloy contains the η' phase peak-aged at 423K. Analysis of the SAED patterns indicates that the base alloy contains η_1 phase and T(T') phase, the Cu-addition alloy contains η_1 phase, T(T') phase and Q(Q') phase and the Ag-addition alloy contains the η' phase and η_1 phase over-aged at 473K.

REFERENCES

[1] E.A. Starke Jr, J.T. Staley, Aerospace Sci. **32**, 131-172 (1996).
[2] N. Ryum, Z. Metallkde **66**, 338-345 (1975).
[3] E. Salamci, Mater. Sci. Tech. **20**, 859-863 (2004).
[4] L.K. Berg, J. Gjonnes, V. Hansen, X.Z. Li, M. Knutson-Wedel, G. Waterloo, D. Schryvers, L.R. Wallenberg, Acta Mater. **49**, 3443-3451 (2001).
[5] J.K. Park, A.J. Ardel, Met. Trans. **14A**, 1957 (1983).
[6] J. Gjonnes, Chr. J. Simensen, Act Metal. **18**, 881-890 (1970).
[7] Yoshio Baba, J. Inst. Met. **4**, 513-518 (1967).

Received: 20 February 2014.