

STM OBSERVATION OF LOCALIZED DEFORMATION NEAR GRAIN BOUNDARIES IN Al-1mass%Mg₂Si ALLOYS WITH VARIOUS AMOUNT OF COPPER

Fumitaka OHARA*, Yasuhiro UETANI**, Kenji MATSUDA***, Fujio SHINAGAWA***
and Susumu IKENO***

* Graduate School, Toyama University, Gofuku 3190, Toyama-shi, Toyama, 930-8555 JAPAN.

** Research Institute for Technology, Toyama Prefectural University, Kurokawa 5180,
Imizu-gun, Toyama, 939-0398 JAPAN.

*** Faculty of Engineering, Toyama University, Gofuku 3190, Toyama-shi, Toyama,
930-8555 JAPAN.

ABSTRACT Localized deformation near grain boundaries in age-hardened Al-1.0mass%Mg₂Si alloys containing up to 0.8mass% copper were investigated by scanning tunneling microscopy. Characteristics of surface topography of fold at grain boundary triple point in the alloys were almost similar to those of the Al-1.0mass%Mg₂Si alloy with 0.4mass% excess silicon. Two types of folds were observed: One developed with the progress of deformation and the another did not developed. The frequencies of fold formation decreased with copper addition as compared to the alloy without copper. Most of the folds did not developed for the alloys with 0.2 and 0.5mass% copper.

Keywords: *Al-Mg-Si alloy, copper addition, fold, intergranular fracture, scanning tunneling microscopy.*

1. INTRODUCTION

Drastic decrease in elongation followed by a marked intergranular fracture has occurred in the peak-aged Al-Mg-Si alloys, and localized deformation similar to grain boundary sliding has been observed during tensile deformation [1]. Such a localized deformation often accompanies with a fold generation at the grain boundary triple point, where a crack has been observed preferentially. It has also been shown that fold formation is largely influenced by the characteristics of the grain boundary causing a fold and the other two that constitute a triple point, i.e. by the orientations of each grain boundary plane against the tensile direction [2]. On the other hand, it has been known that addition of small amount of copper had increased the strength of Al-Mg-Si alloy [3,4]. In these alloys, however, there is few research about localized deformation near grain boundaries and therefore the correlation between fold formation and characteristics of grain boundaries has not been clarified yet.

So, in this investigation, surface topographies near grain boundaries due to localized deformation in Al-1.0mass%Mg₂Si alloys without and with a few amount of copper addition have been observed precisely using a scanning tunneling microscope. Based on the observations, the ductility of these alloys has been discussed.

2. EXPERIMENTAL PROCEDURE

Ingots of chemical compositions of Al-1.0mass%Mg₂Si alloy (base alloy), Al-0.98mass%Mg₂Si-0.21mass%Cu alloy (0.2%Cu alloy), Al-1.0mass%Mg₂Si-0.46mass%Cu alloy (0.5%Cu alloy) and Al-0.99mass%Mg₂Si-0.77mass%Cu alloy (0.8%Cu alloy) were cast in atmosphere using 99.99 mass% purity aluminum, 99.9mass% purity magnesium, silicon and electrolytic copper. These ingots were hot-rolled and then cold-rolled to plates of 0.9mm thickness. For micro-hardness measurements, small pieces cut from these plates were quenched into iced water immediately after

solution treatment at 848K for 3.6ks, then aged at 423K. The R-type tensile specimens of 0.8mm thickness and central width of 3.0mm [2] were made and aged at 423K for 600ks (corresponding to nearly peak-aged condition in age-hardening curves). After electrolytic polishing, scratch lines were drawn on the specimen surface using fine alumina particles both parallel and perpendicular to tensile direction. The specimens were deformed in tension to given strains and localized deformation near grain boundary triple point at the specimen surface was observed by scanning tunneling microscope (STM). Apart from STM observations, the tensile specimens with width of 6mm and gage length of 17.5mm were also made for fracture surface observation and measurement of uniform elongation. Both types of specimens were tensile tested at velocity of 1.67×10^{-4} m/s at room temperature.

The ratio of resolved shear stress on a grain boundary plane (τ_R) to the applied stress (σ_A), F was used to estimate the force imposed on each grain boundary plane. The F is given by the following expression :

$$F = |(\sin \lambda \sin \theta)(\sin \lambda \cos \theta \cos \phi - \cos \lambda \sin \phi)| \quad (1)$$

Where λ , θ and ϕ are the angles as shown in Fig.1, respectively. If F takes its maximum at $\phi = 0$ or 180° , it means that F will act on the direction generating a step at the grain boundary. If F also takes its maximum at $\phi = 90^\circ$, it will act on the direction generating displacements of scratch line at the grain boundary. The maximum value of F calculated from equation (1) ranges from 0 to 0.5 and is denoted as " F_{\max} " after this.

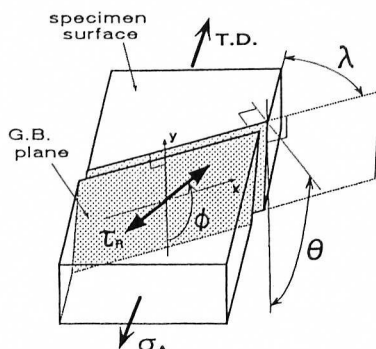


Fig.1 Schematic illustration of grain boundary. Direction of displacement has an angle ϕ on the grain boundary plane. The grain boundary at the specimen surface makes an angle λ to the tensile direction, and the grain boundary plane makes an angle θ to the specimen surface.

3. RESULT AND DISCUSSION

Fig.2 shows age hardening curves of the base alloy and the alloys with various amount of copper aged at 423K. It can be seen that the times to reach each peak hardness are generally same (~ 600 ks) and their values increase with increase in amount of copper addition. Nominal stress-strain curves of these alloys aged at 423K to nearly peak hardness are shown in **Fig.3**. From this figure, both 0.2% proof strength and ultimate tensile strength increase with the amount of copper addition in analogy with the changes in peak hardness. It should be mentioned that the uniform elongation increases from about 1% to 5% strain with increase in the copper addition up to 0.5mass%, while it decreases significantly in the 0.8%Cu alloy as compared with that of the 0.5%Cu alloy. **Fig.4** shows the fracture surfaces of these alloys and they are analogous in the mixed conditions of intergranular and transgranular fracture surfaces. It has been reported that the elongation in Al-Mg-Si alloy with excess silicon is improved by refinement of grain size to about $50 \mu\text{m}$ [5]. However, the grain size of the base alloy is about $600 \mu\text{m}$ and nearly equals to those of the alloys with various amount of copper. Therefore, the increase of the elongation in the alloys containing copper may not be caused by grain refinement.

Fig.5 shows a typical example of STM observation near grain boundary triple point at the specimen surface with increase of the amount of deformation in the base alloy. STM image at 0.4% strain is shown in **Fig.5(a)**. It can be seen that a fold generates so as to extend from grain boundary

A with parallel displacement to the specimen surface, and a hollow region is simultaneously formed due to a step at grain boundary B. With the progress of deformation, as shown in Fig.5(b), this fold develops and the hollow becomes deeper at 0.75% strain. The characteristics of topography of this fold has been the same as that of the alloy with excess silicon [6]. Schematic diagram for the magnitude and direction of F_{\max} at each grain boundary is shown in Fig.5(c). The values of F_{\max} at grain boundary A and B are 0.470 at $\phi=90^\circ$ and 0.473 at $\phi=180^\circ$, respectively, which indicate that F_{\max} at grain boundary A acts on the direction of a parallel displacement to the surface and the one

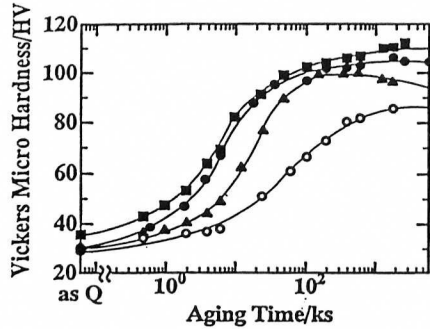


Fig.2 Micro-Vickers hardness as a function of aging time for the base alloy (○), the 0.2%Cu alloy(▲), the 0.5%Cu alloy(●) and the 0.8%Cu alloy(■) aged at 423K.

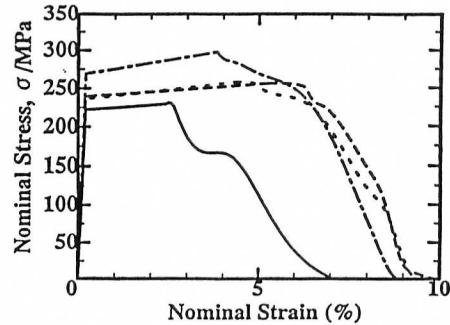


Fig.3 Typical nominal stress-nominal strain curves for the base alloy(—), the 0.2%Cu alloy(.....), the 0.5%Cu alloy(— —), the 0.8%Cu alloy(— · —) aged at 423K for 600ks (near peak-aged condition).

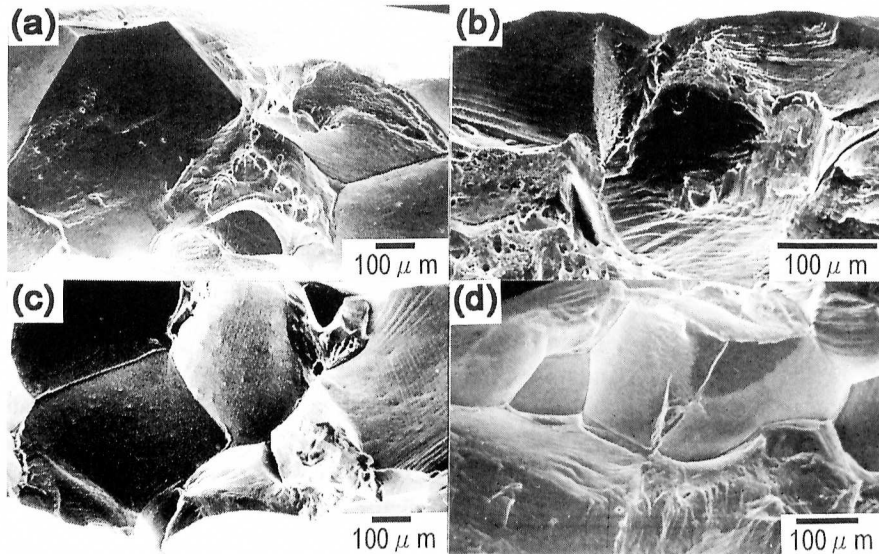


Fig.4 SEM images of fracture surface for (a)the base alloy, (b)the 0.2%Cu alloy, (c)the 0.5%Cu alloy and (d)the 0.8%Cu alloy aged at 423K for 600ks.

at grain boundary B acts on the direction generating a step. These results calculated are in good agreement with the result actually observed. At grain boundary C, the direction of F_{\max} is almost parallel to the specimen surface ($\phi=91.2^\circ$), but the displacement of scratch lines cannot be observed. This reason can easily be understood due to the small value of F_{\max} which is 0.048. The folds which had been generated from the grain boundary with parallel displacement, such as Fig.5,

could not be observed in the alloys with copper addition in this investigation. For the base alloy, in most of cases, the displacement of grain boundary causing a fold formation could be explained by the magnitude and direction of F_{max} at each grain boundary.

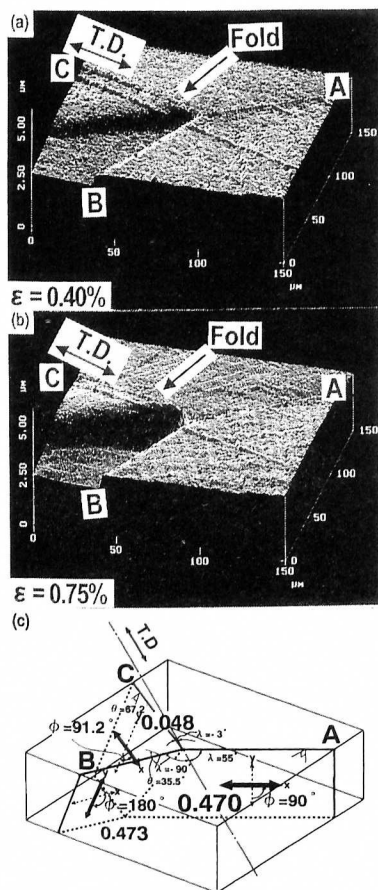


Fig.5 STM images at an elongation of (a)0.40%, (b)0.75% and (c)schematic diagram of three grain boundaries for the base alloy aged at 423K for 600ks : boundaries A and B accompany displacement of scratch line and a step, respectively. The arrows show the direction of the maximum resolved shear stress which is attained when F in eq.(1) takes its maximum (F_{max}). The values F_{max} of are also shown.

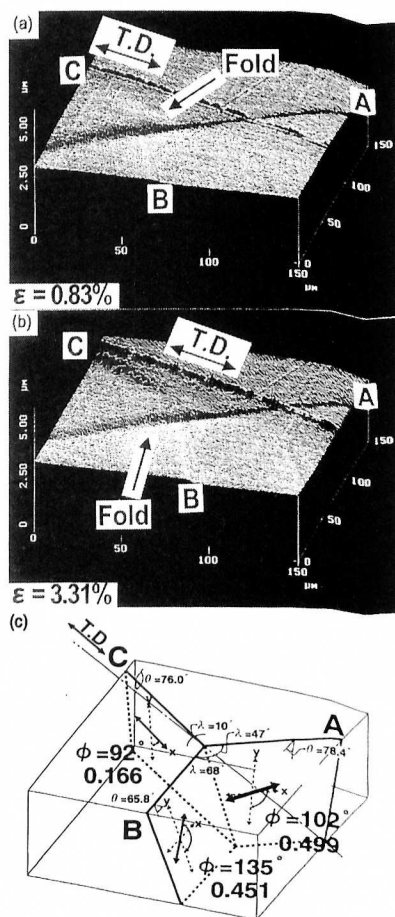


Fig.6 STM images at an elongation of (a)0.83%, (b)3.31% and (c)schematic diagram of three grain boundaries for the 0.5%Cu alloy aged at 423K for 600ks : boundary A accompanies a step. The arrows show the direction of the maximum resolved shear stress, and the values of F_{max} are also shown.

For the alloys with copper addition, as a typical example of the same observation as Fig.5, a STM image of the 0.5%Cu alloy at 0.83% strain is shown in Fig.6(a). It can be seen that a fold generates so as that a small step at grain boundary A extends into a grain. In this case, even if the amount of deformation increases to 3.31% strain, there has been a slight increase in the height of

the step and the fold scarcely develops as shown in Fig.6(b). In spite of a small shape of fold, the characteristics of topography of this fold has also been the same as that of the alloy with excess silicon [6]. From the schematic diagram for F_{\max} and ϕ at each grain boundary as shown in Fig.6(c), F_{\max} at grain boundary A takes a large value of 0.499 at $\phi=102^\circ$. It can be estimated that F_{\max} at grain boundary A acts on relatively near the direction of a parallel displacement to the surface. Nevertheless, the displacement of scratch lines cannot be observed at grain boundary A. Also at grain boundary B and C, the estimations obtained from F_{\max} are not in agreement with the observed results. As for the 0.5%Cu alloy, most of the folds observed have scarcely developed with the progress of deformation and their generations including those that had developed could not be explained by the magnitude and direction of F_{\max} at each grain boundary. In the case of 0.2%Cu alloy, significantly large portion of the folds observed did not develop in the same manner as Fig.6 and the fold formations could seldom be explained by the use of F_{\max} . In the 0.8%Cu alloy, most of folds developed like as those of the base alloy. But the estimations for the fold formation by F_{\max} have not been in agreement with the observation results in similar to those of the 0.5%Cu and 0.2%Cu alloys. Consequently, the tendency that the developed folds decrease with increase in the amount of copper addition up to 0.5mass% and they increased again by 0.8mass% copper addition, seems to be associated with the difference in uniform elongation between the base alloys and the alloys with copper addition as shown in Fig.3.

Results of SEM observations near grain boundary triple point just after peak strength for the base and the alloys with copper addition are shown in Fig.7. In these alloys, it is seen that a large steps has occurred at grain boundary A with a folds and also a significantly large crack has generated at the bottom of the step. Therefore, also in the base and the alloys with copper addition, it is thought that the site of crack leading to fracture may exist near the triple point neighboring a grain boundary with a large step and causing a more developed fold.

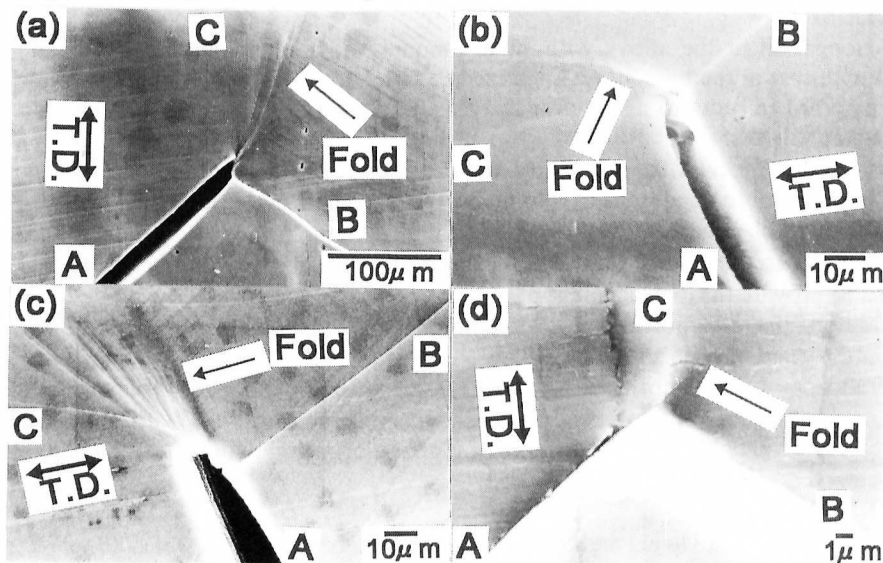


Fig.7 SEM images near a grain boundary triple point at specimen surfaces just after peak strength for (a)the base alloy, (b)the 0.2%Cu alloy, (c)the 0.5%Cu alloy and (d)the 0.8%Cu alloy.

Fig.8 shows the frequencies of fold formation against the increase of elongation for the various alloys investigated. The frequency is the percentage of the number of triple points having folds against the number of triple points within a gage area of 2×4 mm. It can be seen that the frequency for the base alloy increases with the increase of the amount of deformation and then keeps nearly constant after reaching about 40% at 2~3% strain. Against this, for the alloys with copper addition,

their frequencies saturate at about 30% and are lower than that of the base alloy. This means that the number of folds generated are few in the alloys with copper addition. Therefore, it is thought that the improvement of elongation for the 0.2%Cu and 0.5%Cu alloys is due to the decreases of frequency as compared with the base alloy, in addition to difficulties in development of folds with the progress of deformation. The reason that the elongation decreases in the 0.8%Cu alloy in spite of the same saturated frequency level of fold as the other alloys with copper, may relate to the same developed folds as the base alloy.

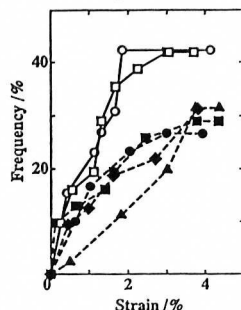


Fig.8 Frequencies of fold formation against the increase of elongation for the base alloy(○, □), the 0.2%Cu alloy(▲), the 0.5%Cu alloy(●, ■) and the 0.8%Cu alloy(◆).

At the present investigation, it has not been understood yet that the reason why a displacement of grain boundary cannot be explained by F_{\max} in the alloys with copper addition. Possibly, as the deformation within a grain differing from the base alloy by small amounts of copper addition seems more strongly to influence the localized deformation near grain boundaries than those of the base alloy, its influences on a displacement of grain boundary and a fold formation should be examined in detail.

4. CONCLUSION

The elongation in the alloys with small amounts of copper addition has been improved as compared with that in the base alloy. Localized deformation near grain boundary triple points were observed by STM in order to make clear the reason of the improvement of elongation. The results obtained are as follows:

- (1) The fracture surfaces of the base alloy and the alloys with copper addition by tensile test have consisted in the mixture conditions of intergranular and transgranular fracture.
- (2) From the STM observation, the characteristics of topographies of folds generated in these alloys have been similar to those of the alloy with excess silicon. The magnitude and the direction of F_{\max} has seemed to provide the displacement of grain boundary in the base alloy, while in the alloys with copper addition, the direction of F_{\max} has not been in agreement with that of a displacement observed at the grain boundary.
- (3) The frequency of fold formation has increased with the progress of deformation and then saturated at about 40% in the base alloy, while those in the all alloys with copper addition have saturated at lower value of near 30% in spite of higher elongation than that of the base alloy.

REFERENCES

- [1] S.Ikeno, K.Matsuda, Y.Uetani and S.Tada : J. Jpn. Inst. Light Metals, **38**(1988), 394.
- [2] Y.Uetani, H.Murase, K.Matsuda, H.Anada, S.Tada and S.Ikeno : J. Jpn. Inst. Metals, **58**(1994), 260.
- [3] Y.Baba and A.Takashima : J. Jpn. Inst. Light Metals, **19**(1969), 90.
- [4] K.Yokota, T.Komatsubara, T.Sato and A.Kamio : J. Jpn. Inst. Light Metals, **42**(1992), 149.
- [5] Y.Uetani, T.Katayama, K.Matsuda, K.Terayama and S.Ikeno : J. Jpn. Inst. Light Metals, **47**(1997), 154.
- [6] Y.Uetani, K.Ozaki, K.Matsuda, H.Anada, S.Tada and S.Ikeno : J. Jpn. Inst. Light Metals, **42**(1992), 400.