

EFFECTS OF Mm(MISCH METAL) ADDITION ON PRECIPITATION BEHAVIORS IN Al-Mg-Si ALLOYS WITH EXCESSIVE Mg.

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ABSTRACT The present work has been carried out focus on changes of ageing response and strengthening factors by Mm(Misch Metal) addition in Al-Mg-Si alloys with excessive Mg. With addition of Mm(Misch Metal), precipitation of $\beta(\text{Mg}_2\text{Si})$ phase was decreased and delayed. Also, formation rate of clusters decreased but heat-stability of precursor phases increased that may be reasons of why delayed ageing response. Especially, Although strengthening precipitate decreased in Mm added Al-Mg-Si alloy with most excessive Mg content, rapid changes of tensile properties were not observed, rather a little improved.

Keywords : *Al-Mg-Si Alloys, Excessive Mg, Mm(Misch Metal), Resistivity, Precipitation behaviors, Solid solution strengthening*

1. INTRODUCTION

There have been numerous studies to maximize potential properties by control of Mg_2Si precipitation in excessive Mg, Si contained Al-Mg-Si alloys which are promising materials in transport applications. Especially, the studies about high excessive Mg alloy may expect additional solid-solution strengthening by excessive Mg as like Al-Mg alloy, due to decrease of Mg_2Si phase solubility and possibility of $\beta(\text{Al}_3\text{Mg}_2)$ precipitation, have been limited as compared with excessive Si alloy. But, according to results of previous work in Al-high Mg($\geq 7\text{wt}\%$)-Mm(Misch Metal) alloys[1], it was possible to acquire solid solution strengthening, additionally, by restraint of β phase due to RE(Rare Earth elements) and Mm(Misch Metal) addition. So, if it may be possible to get rid of $\beta(\text{Al}_3\text{Mg}_2)$ precipitation though decrease of $\beta(\text{Mg}_2\text{Si})$ solubility is expected, it can expected that maximize of potential strength by solid solution strengthening due to increasing of Mg contents. So, the present works have been carried out focus on changes of ageing response and possibility of control on strengthening factors by Mm addition in Al- Mg-Si alloys with excessive Mg alloys.

2. EXPERIMENTAL PROCEDURE

The Al-Mg alloys were melted and cast in a high frequency melting furnace under Ar atmosphere using 99.9%Al, 99.9%Mg and 99.9%Si, and Mm. The alloy designs and the chemical compositions of these alloys are shown in Table 1. After all as-cast ingots were two step-homogenized at 773K for 4hr and 803K for 20hr in argon atmosphere, they were hot rolled to plates with 3mm thickness and cold rolled to 1mm thickness. All the specimens were solid solution treatment for 2hr at 723K followed by ice water quenching. The ageing treatments were carried out

at 453K in silicon bath, and room temperature tensile test was performed with ASTM E8 sub-mini size specimens by instron type tensile tester. For the resistivity measurement, plate specimen 1.0mm×2.0mm×80mm sized was used and electrical resistivity of specimen which was hold in infra-red furnace measured with heating from room temperature to 753K using SINKU-RIKO's TER-2000 electrical resistance measuring instrument. All the thin foil specimens for TEM analyses were electropolished in a solution of 40vol% HNO₃-60vol% CH₃COOH cooled to 233K at DC voltage of 18 to 20V. The thin foil specimens were examined by JEM200CX electron microscope operating at accelerating voltages of 160kV.

Table 1 Alloy design and chemical composition of Al-Mg-Si-(Mm) alloys with excessive Mg.

	Chemical Composition(wt%)						Remark
	Mg	Si	Fe	Mn	Zr	Al	
Al-2.0Mg-0.5Si	1.98	0.52	0.02	0.1	0.09	Bal.	Standard alloy
Al-2.0Mg-0.5Si- <i>0.1Mm</i> *	2.02	0.51	0.03	0.1	0.07	Bal.	Mm bearing alloy
Al-2.5Mg-0.5Si- <i>0.1Mm</i> *	2.54	0.56	0.02	0.1	0.1	Bal.	Excessive Mg alloy

*Mm(Misch metal) : Ce:50.9wt%, La:23.0wt%, Nd:15.1wt%, Pr:6.2wt%, etc

3. RESULTS AND DISCUSSION

3.1 Mechanical properties

Fig. 1 shows the hardness and ageing time curves in Al-Mg-Si-(Mm) alloys aged at 453K. Compared with free Mm alloy, maximum hardness increased in cases of Mm added alloys. Note that delayed ageing response was observed though the maximum peak hardness showed in Al-2.5Mg-0.5Si alloy as most excessive Mg alloy. Fig. 2 compares measurements of UTS (MPa/mm²) for Al-Mg-Si-(Mm) alloys with excessive Mg. Increment of UTS, about 40~50MPa, was observed

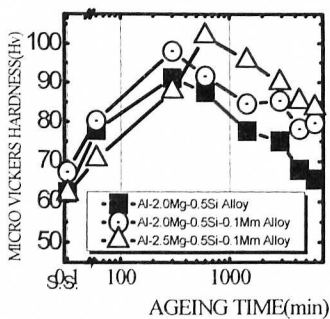


Fig.1 Changes of micro vickers hardness with ageing in Al-Mg-Si-(Mm) alloys.

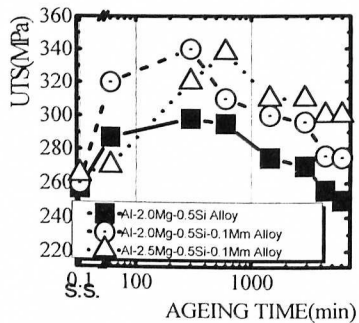


Fig.2 Changes of ultimated tensile strength with ageing in Al-Mg-Si-(Mm) alloys.

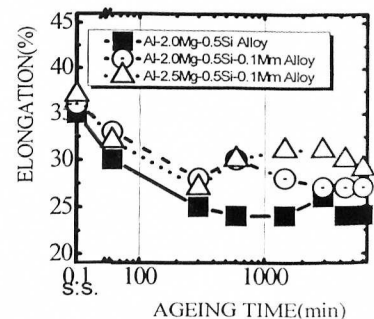


Fig.3 Changes of elongation with ageing in Al-Mg-Si-(Mm) alloys.

at peak ageing condition by Mm addition. But delayed ageing response, as like results of hardness test, was observed in Al-2.5Mg-0.5Si-0.1Mm alloy. Also, elongation with ageing in Fig. 3 improved with Mm addition in Al-Mg-Si alloy with excessive Mg. These behaviors, as delayed ageing responses, are good agreements with the results of Cho et al[2] in Rare earth elements added Al-Li alloys.

3.2 Microstructure

Photo. 1 showed the transmission electron micrograph in Al-Mg-Si-(MM) alloys with excessive Mg, aged for 5hr and 100hr at 453K, as peak and over ageing condition. Thanks to previous workers, we already have been known about various precipitates with ageing in Al-Mg-Si alloys. So, two precipitates, needle and rod type precipitates, in Photo.1 may confirm as $\beta''(\text{Mg}_2\text{Si})$ and $\beta'(\text{Mg}_2\text{Si})$ phases. In peak ageing condition, these phases were observed homogeneously

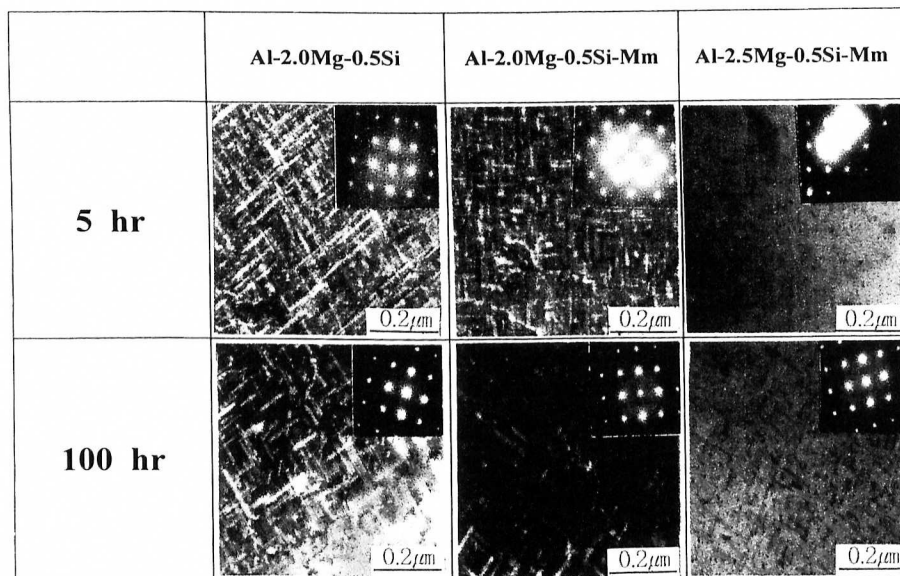


Photo. 1 SAD patterns and BF(DF) images of Al-Mg-Si-(Mm) alloys aged at 453K for 5hr and 100hr.(z=[001])

regardless of Mm added or not. But, amount and size of precipitation has a wide difference between free Mm and Mm bearing alloys. Namely, compared with free Mm alloy, more fine precipitate was observed, but streaks of matrix, which were a concern with existence of $\beta''(\text{Mg}_2\text{Si})$ phase, on SAD(Selected Area Diffraction) pattern became stronger in Al-2.0Mg-0.5Si-0.1Mm alloy. In Al-2.5Mg-0.5Si-0.1Mm alloy, rather weaker streak of matrix, compared with free Mm, more fine sized precipitates were observed what most excessive Mg contained. Also, in Al-2.0Mg-0.5Si-0.1Mm alloy, as compared with free Mm alloy, precipitates increased in quantity. On the contrary, in case of Al-2.5Mg-0.5Si-0.1Mm alloy, precipitates decreased in quantity. In case of overageing condition, aged 423K for 100hr, precipitates were grown with ageing regardless of Mm added or not. But, a tendency of precipitation feature as like precipitate size and amount, remain unchanged. Amount

of precipitates increased in Al-2.0Mg-0.5Si-0.1Mm alloy and decreased in Al-2.5Mg-0.5Si-0.1Mm alloy compared with free Mm alloy, as like a case of peak ageing condition. Especially, it is interesting to note that very small sized phases were observed homogeneously in Al-2.5Mg-0.5Si-0.1Mm alloy even though overageing condition.

From these results, it may be considered that, by addition of Mm, growth of precipitate has been suppressed considerably but amount of precipitates was different with Mg contents. About decreasing of precipitates in Al-2.5Mg-0.5Si-0.1Mm alloy, as most excessive Mg contained alloy, it may be estimated due to decreasing of β solubility with excessive Mg contents if the results of Lutts et al.[3] have been considered. Therefore, on bases of these considerations, we can now propose reasons to tensile properties changes in previous chapter. Namely, delayed ageing response and improvement of tensile property in Mm bearing alloys are connected with restraint of precipitate growing. Especially, in case of Al-2.0Mg-0.5Si-0.1Mm alloy, acceleration of precipitation may be reasons of why maximum value in results of UTS and elongation. But, it is noteworthy that improvements of tensile properties were observed, in spite of solubility decreasing of β phase in Al-2.5Mg-0.5Si-0.1Mm alloy. It is of common knowledge that predominant factor on strengthening in Al-Mg-Si alloys is precipitation strengthening by β phase. Practically, these are well known that $\beta''(\text{Mg}_2\text{Si})$ and $\beta'(\text{Mg}_2\text{Si})$ phases, as metastable phases having HCP structure, are most strong factors on strengthening in Al-Mg-Si alloys and the majority of researches have been concentrate upon how to get the best precipitation condition of $\beta''(\text{Mg}_2\text{Si})$ and $\beta'(\text{Mg}_2\text{Si})$ phases by various methods. Therefore, it is unavoidable think that the other factor had acted on strengthening. additionally, in Al-2.5Mg-0.5Mg-0.1Mm alloy if considered that improvements of UTS and microhardness compared with free Mm alloy. Of course, there are a lot of strengthening factors could be considered, but, according to present work till this time, just a little decrease of grain size was observed in Mm bearing alloys, typical differences could not be found against changes of precipitation behaviors. So, it may be estimated that additional increment of solid solution strengthening originated from restraint of β precipitate, act on strength increment in Al-2.5Mg-0.5Si-0.1Mm alloy.

Park and Cho[1] recently reported that restraint of $\beta(\text{Al}_3\text{Mg}_2)$ precipitation caused increment of solid solution strengthening in high Mg($\geq 7\text{w}\%$) contained Al-Mg alloys. So, even if other researches, just relate to control on precipitate condition, have been considered, it may be interesting result that shows possibility of strength increment by solid solution strengthening. To investigate changes of precipitation behaviors by Mm addition, in detail, electrical resistivity changes have measured.

3.3 Changes of electrical resistivity with heating

Though there are many application methods of electrical resistivity measurement, to take the valuable information about phase transformation, severe specimen preparing and measurements were needed. That is reason of why electrical resistivity is affected absolutely by temperature change and specimen size etc. sensitively. Especially, in present work that could not avoid thermal vibration factor, it was possible to analysis transformation of detail regardless of temperature and

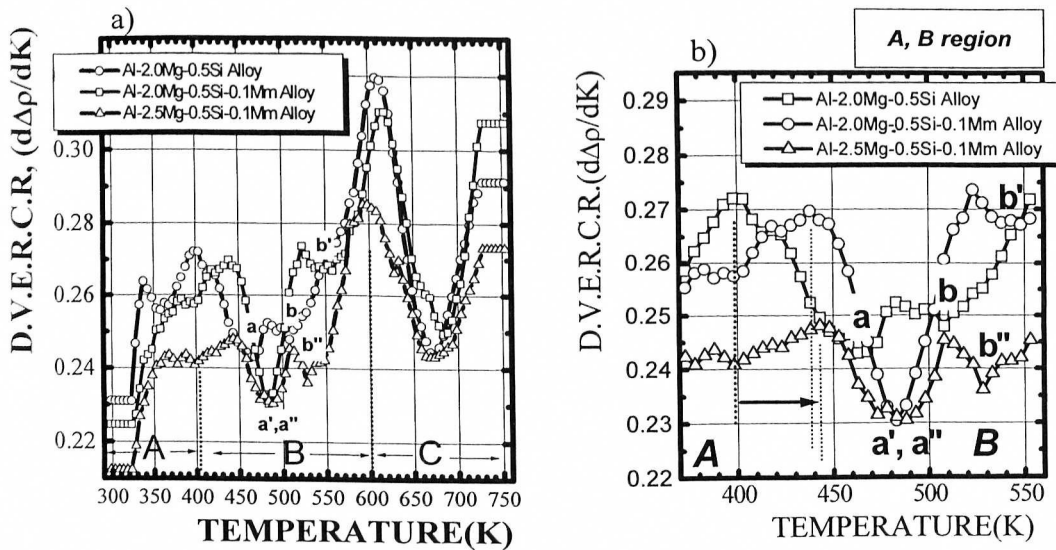


Fig. 4 Differentiate Values of Electrical Resistivity Change Ratio with heating in Al-Mg-Si-(Mm) alloys solid solution treated. b) Partial section near boundary at *A* and *B* temperature regions on a).

specimen size effects by the differentiation of resistivity change ratio, $\Delta R(((\rho_T - \rho_0) / \rho_0) \times 100)$: ρ_T is resistivity at each temperature and ρ_0 is resistivity at room temperature as standard temperature), to temperature as previous work.[1]. Fig. 4 compared differentiation values of electrical resistivity changes ratio(D.V.E.R.C.R.) with heating from room temperature to 753K after solid solution treatment in Al-Mg-Si-(Mm) alloys. As a whole, it was possible to divide into 3 temperature regions, as like *A*, *B* and *C*. by set region on the basis of free Mm alloys. According to many results of previous researcher, it can be considered that *A* temperature region, shaped upper peak, from room temperature to 400K, which was concerned with clusters and *B* temperature region, shaped down peak, from 400K to 600K, which was concerned with precipitation of β'' and β' phases and *C* temperature region, shaped upper peak, from 600K to 750K which was concerned with β precipitation. These results are in agreement with results of DSC analysis and electrical resistivity change worked by previous researchers. In case of free Mm alloy, splitting upper peak was observed in *A* temperature region, but, in case of Mm bearing alloys, peak development and splitting of upper peak were depressed. Considering report of G. A. Edwards et al.[4] who insisted that there were three type clusters in *A* temperature region, it may be estimated that splitting upper peak is associated with clusters of the different kind formations. Therefore, immature upper peak in Mm bearing alloys may be estimated that such formation process of cluster was depressed by Mm addition. Also, transition temperature from *A* temperature region to *B* temperature region moved to high temperature as like marked arrows in fig. 4 (b). So that it is possible to think that thermal stability of clusters increase by Mm addition. In *B* temperature region consisted successive small down peaks, as like *a* and *b*, two down peak as *a* and *b* in free Mm alloy, move to *a'*, *b'* in Al-2.0Mg-0.5Si-0.1Mm alloy and *a''*, *b''* in Al-2.5Mg-0.5Si-0.1Mm alloy that are located at more higher temperature. Especially, in case of Al-2.0Mg-0.5Si-0.1Mm alloy, most big under peak, *a'*, due to β'' precipitation, was observed but, in case of Al-2.5Mg-0.5Si-0.1Mm alloy, rather smaller under peak, *a''*, was observed compared with free Mm alloy's. In *C* temperature region related to stable β phase, just typical change of down peak in Al-2.5Mg-0.5Si-0.1Mm alloy, which was decreased against with free Mm alloy's, was observed. So that these are in agreements with previous results of TEM observations as photo.1.

Therefore, it is estimated that, by Mn addition, in Al-2.0Mg-0.5Si-0.1Mn alloy, tensile properties were increased due to increasing of β'' precipitation in quantity, as predominant phase on strength, and small sized β'' precipitate by depressing of growing. Although, about improvement of tensile properties in Al-2.0Mg-0.5Si-0.1Mn alloy compared with free Mn, because of decreasing of precipitates observed, clearly, in results of TEM observation and electrical resistivity measurements, it must be considered that the other factors, as like solid solution strengthening, act on tensile properties additionally. It is very interesting result if regard as new consideration to maximize tensile properties in Al-Mg-Si Alloys.

4. CONCLUSION

1. With addition of Mn, delayed ageing response observed and tensile properties and thermal stability of clusters, β'' phase and β' phase, have improved in Al-2.0Mg-0.5Si alloy as excessive Mn contained Al-Mg-Si alloy.
2. In case of 0.1wt%Mn added Al-2.0Mg-0.5Si alloy, precipitation of $\beta''(\text{Mg}_2\text{Si})$ phase, as the predominant strengthening phase, increased in quantity. That is why improvement of tensile properties with their fine size, but, in case of 0.1wt% Mn added alloy with more Mg contained, precipitation of $\beta''(\text{Mg}_2\text{Si})$ phase decreased in quantity to the contrary..
3. Considering of 0.1wt% Mn added alloy with more Mg contained. It was shown that in spite of decrease of precipitate, tensile properties increased, It may be estimated that the other factor, as like solid solution strengthening, additionally acted on strength by depressed β precipitation.

5. REFERENCES

- [1] S. D. Park, Light Weight Alloys for Aerospace ApplicationsIV (Pennsylvania : Publication of TMS, 1997),85-89
- [2] D. S. Chung, C. H. Lee and H. K. Cho : J. Korean Inst. of Met. & Mater, Vol. 35, No. 6(1997), 704-711
- [3] A. Lutts : Acta Met., 9(1961), 577
- [4] G.A.Edwards, G.L. Dunlop and M.J.Couper: The 4th international conference on Aluminum alloys