

Segregations and Precipitations in a Heat treated Al-Zn-Mg Alloy

M. Iqbal, M.A. Shaikh, W. Ahmad, M. Ahmad and K.L. Ali*

Radiation Damage Group, Nuclear Physics Division, Pakistan Institute of Nuclear Science & Technology, P.O. Nilore, Islamabad, Pakistan.

* Nuclear Materials Division, Pakistan Institute of Nuclear Science and Technology, P.O. Nilore, Islamabad, Pakistan.

Abstract

Segregation and precipitation of a locally prepared Al-10.0 wt% Zn-2.5 wt% Mg alloy was studied using scanning electron microscope and electron probe microanalyser. Samples were solution treated at various temperatures ranging from 450 to 550°C for 3h. Samples solution treated at 480°C for 3h were aged at 130 and 300°C from 1 to 72h. In the as cast alloy segregation of Mg and Zn at grain boundaries and liquid droplets within the grains was observed showing cellular/lamellar structure at higher magnification. Samples solution treated above 500°C have very sharp dendritic structure. Microhardness of the segregated areas was much higher than that of the normal matrix. Samples aged at 130°C show initial increase in hardness with increase in ageing time up to 24h after which hardness becomes constant. Hardness of samples aged at 300°C decreases with increase of ageing time up to 7h which may be due to formation of incoherent $MgZn_2$ precipitates. After 7h hardness remains constant.

Keywords

Segregation, precipitation, Hardness, Al-Zn-Mg alloy, electron probe microanalyser, cellular/lamellar structure.

Riassunto

Sono state studiate la segregazione e la precipitazione di una lega del Al 10% per peso - Zn 2.5% - Mg, localmente elaborata, utilizzando il microscopio elettronico a scansione e l'analizzatore a sonda elettronica. I campioni sono stati trattati in soluzione per 3 ore a diverse temperature tra i 450 ed i 550°C. Quelli trattati ai 480°C sono stati poi invecchiati ai 130 ed ai 300°C per 1 a 72 ore. Nella lega as cast, sono state osservate la segregazione del Mg e del Zn agli orli dei grani e varie goccioline di liquido dentro i grani stessi, nonché la presenza di una struttura cellulare/lamellare all'esame ad ingrandimento superiore. I campioni trattati a temperature superiori ai 500°C dimostrano una struttura dendritica molto acuta. La microdurezza delle parti segregate è molto più alta di quella della matrice normale. Dopo un aumento iniziale in funzione del tempo di invecchiamento durante le prime 24 ore, la durezza dei campioni invecchiati ai 130°C rimane costante, mentre quella dei campioni invecchiati ai 300°C rimane costante dopo un decremento iniziale fino alla settima ora, forse a causa della formazione di precipitati di $MgZn_2$ incoerenti.

Parole chiave

Segregazione, precipitazione, durezza, lega Al-Zn-Mg, analizzatore a sonda elettronica, struttura cellulare/lamellare.

INTRODUCTION

Al-Zn-Mg alloys are among the age hardenable and high strength aluminium alloys and these alloys have proved useful because of advantages of low density, high strength and good corrosion resistance. Their mechanical properties are greatly affected by changes in their microstructure [1-4] and variation in the concentration of alloying elements in the vicinity of grain boundaries has marked effect on their suscep-

tibility to stress corrosion cracking (SCC) which limits their usefulness in environments prone to SCC [5-6]. SCC of these alloys is dependent on precipitates formed at grain boundaries. It is thus desirable to investigate these phenomena in these alloys. The present paper describes experimental results of an investigation of as-cast and heat treated Al-Zn-Mg alloy.

EXPERIMENTAL

Al-Zn-Mg alloy was prepared from commercially available materials (Al-35 wt% Mg alloy, Al and Zn). The alloy contains 10.0 ± 0.5 wt% Zn and 2.5 ± 0.2 wt% Mg, the total content of impurities (Si+Fe+Ni+Cu) being about 0.4 ± 0.1 wt%. The alloy was prepared in an induction furnace under inert atmosphere. Two series of experiments were performed. In the first series, the samples were heat treated at 450, 480, 500, 530 and 550°C for 3h and quenched in water at room temperature. In the second series samples were first solution treated at 480°C for 3h, water quenched (at room tempera-

ture) and then aged at 130 and 300°C for different intervals of time ranging from 1 to 72h. As-cast and heat treated samples were polished on a lapping machine using silicon carbide paper of different grades and then on diamond paste down to $1/4 \mu\text{m}$ on appropriate cloth. Samples were then examined in the scanning electron microscope (SEM) Jeol JSM 35-CF and microanalysis was done using energy dispersive system, Link 860-2, attached with the microscope. Vicker's microhardness of the samples was measured using microindentation tester Leitz Miniload-2.

RESULTS AND DISCUSSION

Segregation

Examination of the as-cast alloy in SEM showed white contrast at the grain boundaries. Fine particles with white contrast were observed within the grains (fig. 1). Electron probe microanalyser (EPMA) results revealed segregation of Mg and Zn at the grain boundaries and in the fine particles. When viewed at higher magnification, the area of segregation appears to have a cellular and lamellar structure (fig. 2). Maximum concentration of Mg and Zn in the area of segregation was found to be 5.4 wt% and 88.9 wt% respectively. Since maximum solid solubility of Mg and Zn in Al is 17.4 wt% (at 450°C) and 70 wt% (at 443°C), respectively, and it is estimated to be approximately 2.0 wt% at 20°C for both Mg and

Zn [7], the observed segregation can be explained on the basis of rejection of solute and the instability caused by the undercooling results in altering the planar solid-liquid interface to cellular or dendritic interface [8]. The particles observed within grains are the liquid droplets.

In order to investigate the effect of heat treatment on the observed segregation in the as-cast alloy, samples were heat treated at different temperatures in the range 450-550°C for 3h. Treatment at 450°C causes diffusion of Mg and Zn inwards from the grain boundaries and concentration of Mg and Zn reduces at grain boundaries. Concentration of these segregants is observed to be minimum at 480°C and 500°C.

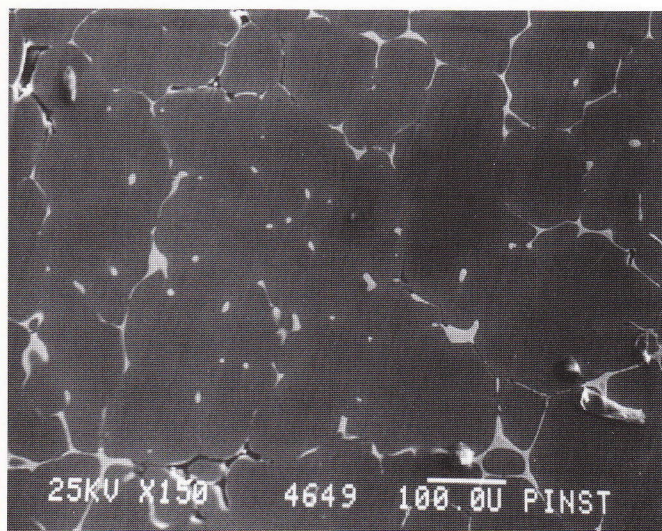


Figure 1: Segregation of Mg and Zn at grain boundaries and at fine particles within grains in the as-cast alloy

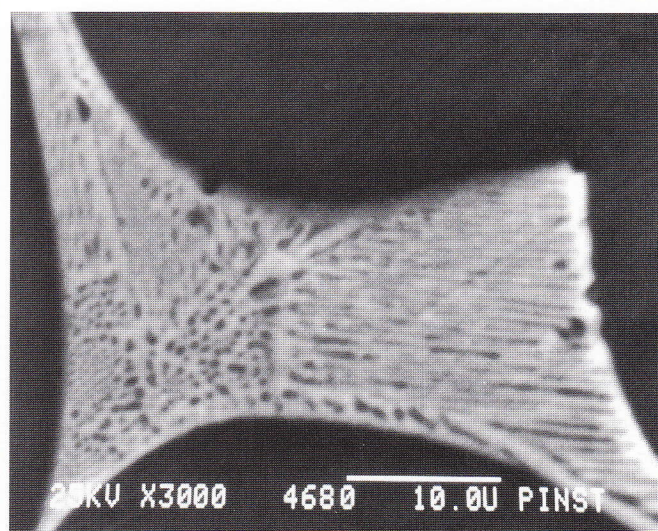


Figure 2: Cellular/lamellar/structure in the as-cast alloy

Even treatment upto 6h at these temperatures does not completely dissolve segregation. When the alloy is heated at 530 and 550°C, melting of grain boundaries occurs because of change in composition due to segregation which brings it to the liquid plus solid phase field. Compared with the as-cast material, cellular dendritic growth is more prominent and sharp in samples heat treated at temperatures higher than 500°C (fig. 3) which may be due to difference in cooling rate because as-cast material was cooled in air while heat treated samples were quenched in water. Microanalysis of the area of segregation showed that there is difference in elemental composition of the cell wall and within the cell. In the samples heat treated at 530 and 550°C maximum concentration of Mg and Zn in the area of bright cell wall was found to be 5.0 wt% and 86.0 wt% respectively. Inside the cell, in dark portion, Mg and Zn reduce to 1.4 wt% and 42.0 wt% respectively.

Although both Mg and Zn diffuse to the grain boundaries, it is observed that the diffusion of Zn is much more than that of Mg. This is due to the fact that migration energy of Zn-vacancy pair (0.45 eV) is much lower than that of Mg-vacancy pair (0.75 eV). Also, binding energy of Zn-vacancy pair (0.13 eV) is much lower than that of Mg-vacancy pair (0.25 eV) [9] which explains higher concentration of Zn compared to Mg.

Fig. 3 also shows another feature and it is the observation of two types of grain boundaries, one with large dendritic growth rate while others with very small rate of growth. The boundaries with very small growth rate are straight. The difference in growth rate may be due to the effect of misorientation of the grain boundaries. The boundaries with very small rate of dendritic growth may be small angle boundaries as these be-

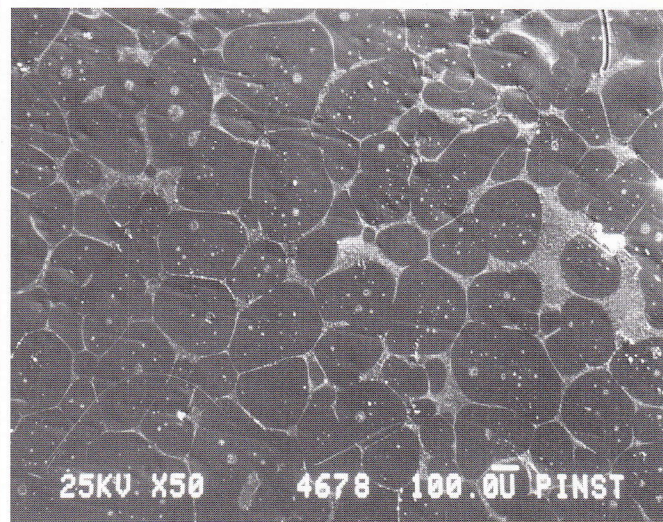


Figure 3: Segregation of Mg and Zn in the sample heated at 550°C for 3h

come pinned and remain straight [10].

The liquid droplets observed within grains have both spherical and non-spherical shapes (fig. 4a) suggesting that the solid-liquid interfacial energy is anisotropic. The size of these particles was found to range from 1 μm to 80 μm . Particles also show faceting. Some of the particles were fine while others were coarse showing dendritic growth. Fig. 4b shows one of such particles. The main stem is in the form of a circle from which the secondary and higher order arms come out.

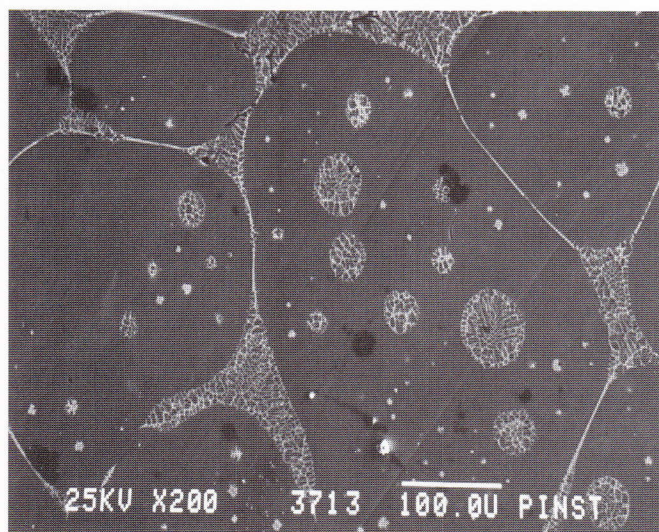


Figure 4a: Liquid droplets within the grains

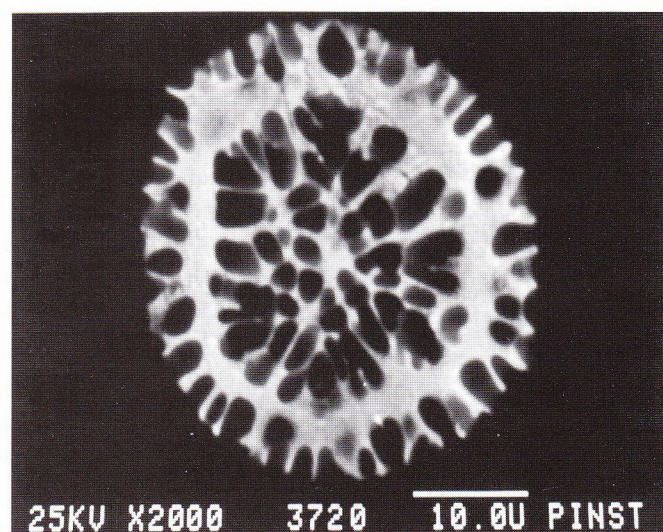


Figure 4b: Dendritic growth in a droplet

Precipitation

SEM examination of samples aged at 130°C up to 72h showed no precipitates at grain boundaries or within grains except the precipitates of impurities (FeAl_3 , $\text{Mg}_2\text{Si}/\text{Mg}_4\text{Si}$). In the samples aged at 300°C upto 72h three types of precipitates were observed (fig. 5): (i) The first type of precipitates gives dark contrast. These precipitates have both regular and irregular shapes and are present at grain boundaries as well as within grains. EPMA results show majority of them to be Mg_4Si while some Mg_2Si are also found. Mg_4Si are also observed in one of the starting materials, Al-Mg alloy. These precipitates have regular geometrical shape. (ii) The second

type of precipitates gives bright contrast in SEM. Most of these precipitates are present at grain boundaries and are observed to be long and coarse. Composition of these precipitates corresponds to FeAl_3 and these precipitates can easily be identified from their contrast, size and shape. These precipitates are formed due to Fe impurity present in the Al-Mg alloy which is the major starting material. (iii) The third type of precipitates are found to be of needle as well as spherical shape at grain boundaries. However within the grains these are of spherical shape and their density is very high and size is small. A large number of precipitates at grain boundaries and within grains have been analysed by EPMA and majority of these precipitates are found to be MgZn_2 type.

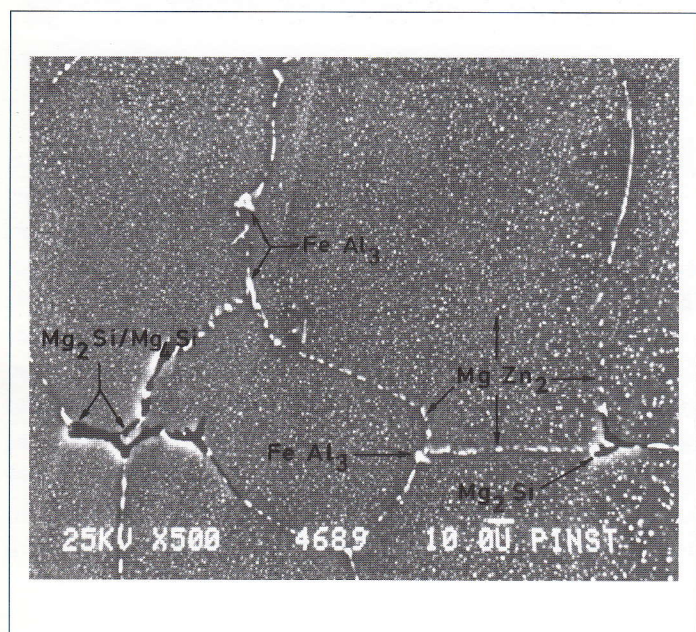


Figure 5: Precipitates in the sample aged at 300°C for 30 h

Hardness

Results of Vicker's hardness measurements show that hardness of solution treated sample (160 HV) is greater than that of as-cast sample (130 HV). This is due to solid solution hardening. In case of samples heat treated at higher temperatures (>500°C) measurement of hardness was also done on segregation areas. It was found that hardness is much higher in the areas of segregation than in the normal matrix (table 1). This is due to segregation of Mg and Zn in these areas. The difference is higher at lower temperature of treatment which is due to the fact that the cell structure in areas of segregation is fine whereas cell structure at higher tempera-

TABLE 1 - Vicker's microhardness of normal and segregation areas of heat treated samples

S.No.	Temperature/Time	Area	Hardness
1	480°C/3h	Normal	160
2	530°C/3h	Normal Segregation	150 274
3	550°C/3h	Normal Segregation	128 200

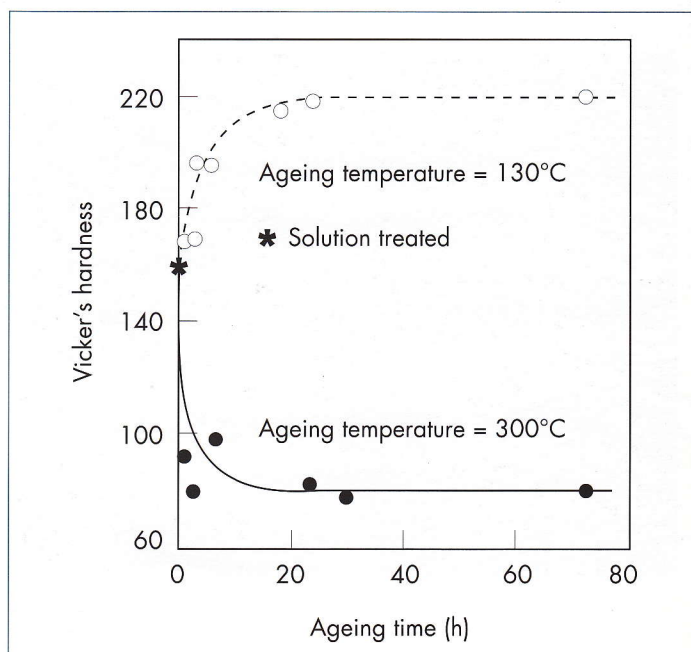


Figure 6: Microhardness of the alloy as a function of ageing time

ture is coarse. Change in hardness due to difference in microstructure produced because of difference in cooling has also been observed by Zhang et al [11] in Al-Cu alloys.

Fig. 6 shows variation of Vicker's hardness as a function of ageing time for ageing temperatures 130°C and 300°C. For samples aged at 130°C, hardness initially increases upto 24h of ageing after which it remains constant. Increase of hardness may be due to formation of certain precipitates which are too small to be observable in SEM. Examination of these

samples in TEM is yet to be carried out. SEM examination of samples aged at 300°C reveal precipitates at the grain boundaries and within grains as discussed above. In case of samples aged at 300°C, hardness initially decreases sharply up to 7h and then remains constant. The initial decrease may be due to incoherency of the precipitates $MgZn_2$, the density and size of which increases with ageing time. After about 7h of ageing, size and density remains constant and so hardness attains a constant value.

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