Evolution of intermetallics, dispersoids and elevated-temperature properties at various Fe contents in Al-Mn-Mg 3004 alloys

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Abstract

Nowadays, great interests are rising on aluminum alloys for the applications at elevated temperature, driven by the automotive and aerospace industries requiring high strength, light weight and low cost engineering materials. As one of the most promising candidates, Al-Mn-Mg 3004 alloys have been found to possess considerably high mechanical properties and creep resistance at elevated temperature resulted from the precipitation of a large number of thermally stable dispersoids during heat treatment. In present work, the effect of Fe contents on the evolution of microstructure as well as high temperature properties of 3004 alloys has been investigated. Results show that the dominant intermetallic changes from α-Al(MnFe)Si at 0.1 wt. % Fe to Al_6(MnFe) at both 0.3 and 0.6 wt. % Fe. In the Fe range of 0.1 to 0.6 wt. % studied, a significant improvement on mechanical properties at elevated temperature has been observed due to the precipitation of dispersoids, and the best combination of yield strength and creep resistance at 573K (300°C) is obtained in the 0.3% Fe alloy with finest size and highest volume fraction of dispersoids. The superior properties obtained at 573K (300°C) makes 3004 alloys more promising for high temperature applications. The relationship between the Fe content and the dispersoid precipitation as well as the materials properties has been discussed.

Keywords：Al-Mn-Mg 3004 alloys; Fe contents; Intermetallics; Dispersoids; Elevated temperature properties; Creep resistance.

1. Introduction

With the rapid development of automotive and aerospace industries, the demand for aluminum alloys working at elevated temperatures (~573 K (300°C)) is greatly increasing due to the lighter weight and lower cost. However, traditional precipitation hardened aluminum alloys, such as 2xxx, 6xxx and 7xxx, are hard to meet the strength requirement at elevated temperature due to the rapid coarsening of precipitates (overaging effect) [1, 2]. For instance, the instant yield strength (YS) of 6061 alloys at 588 K (315°C) can reach at 75 MPa but it rapidly decreased to 31 MPa after 100 hours

holding [1]. On the other hand, dispersoid strengthening is reported to be promising at elevated temperature. Hence, the addition of transition and rare earth elements either individual or combination of Sc with Zr, Ti, Gd, Sm, Er and Yb to wrought aluminum alloy has been explored, especially in 1xxx alloys to form coherent or semi coherent Al_3M dispersoids, which are thermally stable up to 573 K (300°C) [3-7]. However, the high cost of these elements limits their wide applications. Therefore, developing lower cost but thermally stable aluminum alloys at elevated temperature has been always an attractive industrial target.

The work hardening Al-Mn 3xxx alloys have been widely used in architecture and packaging industry because of the excellent formability and corrosion resistance [8, 9]. In 3xxx alloys, Mn is the major element and most of it will be dissolved into the aluminum matrix to form a supersaturated solid solution during the casting process. Therefore, dispersoids is likely to precipitate during the heat treatment due to the decomposition of supersaturated solid solution [10-14]. In the research works of Li *et al.* [12, 14], the partially coherent α-Al(MnFe)Si dispersoids were reported to form during the annealing process in AA3003 alloy, leading to an increase of mechanical properties at room temperature (RT). However, the improvement on mechanical properties was limited due to low volume fraction of dispersoids, which was reported to be 0.77 vol. % at peak condition [12]. Therefore, optimizing the dispersoid precipitation, such as the volume fraction and size to further improve the properties at both RT and elevated temperature, is the key solution in developing aluminum alloys for elevated temperature applications.

To date, limited work has been performed about the influence of heat treatment and alloying elements on the characters of dispersoids in 3xxx alloys. In the study of Huang *et al.*[13], it was found that the volume fraction of dispersoids decreased with increases of homogenization temperature in 3003 alloys. The effect of Mn and Si on the evolution of dipsersoids in 3003 alloy was studied by Muggerud et al. [11] and it was found that addition of Mn and Si could promote the precipitation of α-Al(MnFe)Si dispersoids and the volume fraction of dispersoids can reach to 2.3 vol. % with 0.99 % Mn and 0.48 % Si at fixed 0.5 % Fe (all alloy compositions are in wt. % unless indicated otherwise). In author's recent work [15], it was found that a higher volume fraction of dispersoids (~ 3 vol. %) can precipitate under proper heat treatment (648 K (375°C)/48h) in experimental Al-Mn-Mg 3004 alloy with 1.2% Mn and 0.6% Fe. Besides, the dispersoids were proved to be thermally stable at 573K (300°C) during long-term holding. Therefore, the elevated-temperature properties of 3004 alloys has been remarkable improved, indicating the great potential of 3004 alloys in the applications at elevated temperature.

In present work, a systematic study on the influence of Fe contents on the microstructure and dispersoids as well as the properties is carried out in order to fully develop the 3004 alloys. The mechanical properties and creep resistance at 573K (300°C) are evaluated and the relationship between the Fe content and the precipitation of stable dispersoids as well as the materials properties are discussed.

2. Experimental

2.1 Alloy preparation and heat treatment

Al-Mn-Mg 3004 alloys used in the present work were prepared with commercially pure Al (99.7%) and pure Mg (99.9%), Al-25%Fe, Al-50%Si and Al-25%Mn master alloys. The chemical compositions of experimental 3004 alloys with three different Fe contents were shown in Table 1. In each test, approximately 3 kg of material was prepared in a clay-graphite crucible by using an electric resistance furnace. The temperature of the melt was maintained at ~ 1023K (750°C) for 30 min. The melt was degassed for 15 min and then poured into the permanent mold preheated at 523K (250°C). The dimension of cast ingots was 30mm×40mm×80mm.

The precipitation temperature of dispersoids in 3004 alloy was reported to be around 613K (340°C) [15]. Hence, a precipitation treatment at 648K (375°C) up to 72 hours was selected to study the evolution of dipsersoids with various Fe contents. A programmable electric furnace with the temperature controller (± 2 K) and circulating air was used for the heat treatment. The heating rate was set as 5 K/min. After the precipitation treatment, the ingots were directly quenched into water at RT.

2.2 Alloy properties evaluation

The precipitation behaviour of dispersoids during the heat treatment was evaluated by the electrical conductivity (EC), microhardness and compression strengths. EC was measured using Sigmascope SMP10 electrical conductivity unit at RT and the average value of 3 measurements was taken for each sample. The Vickers microhardness tests were performed on NG-1000 CCD microhardness tester with a load of 200 g and a dwelling time of 20s on polished samples. The locations of indentation were within Al grains to determine the influence of dispersoid precipitation on matrix hardness. The average value of total 15 measurements was reported for each sample. The mechanical properties were obtained from the compression test performed on Gleeble 3800. Cylindrical specimens with a length/diameter ratio of 1.5 (15 mm length and 10 mm diameter) were machined and tested at elevated temperature (573K (300 °C)). The total strain of tested specimens was 0.2 and the strain rate was fixed at 10^{-3} s^{-1}. For the compression test, the specimen was first heated to 573K (300 °C) with a heating rate of 2 K/s and held for 3 minutes to stabilize followed by the compression deformation. The temperature of specimen was monitored by two thermocouples attached on the center of specimen. An average value of YS was obtained from 3 tests.

Besides, creep tests were performed at 573K (300 °C) for 100 hours. The creep specimens were the same size as the Gleeble samples. The creep tests were conducted on the compression condition with a constant load of 45 MPa. For each condition, 3 tests were repeated to confirm the reliability of results.

2.3 Microstructure observation

The microstructural features, including the intermetallics, dispersoids and grain structures were observed by optical and electron microscopes. In order to clearly reveal the dispersoids, the polished samples were etched in 0.5% HF for 30 seconds. A scanning electron microscope (SEM, JSM-6480LV) equipped with an energy dispersive x-ray spectrometer (EDS) and electron back scatter diffraction (EBSD) was used to identify the intermetallics and grain structure. A transmission electron microscope (TEM, JEM-2100)

operated at 200kV was used to observe the distribution of the dispersoids. EDS equipped on TEM was used to measure the chemical composition of dispersoids. The thickness of TEM sample was measured with electron energy loss spectroscopy (EELS). All the TEM images shown in the present work were taken from the area with the thickness of around 100 nm. The size and volume fraction of dispersoids as well as the volume fraction of intermetallics and particle free zone were measured using the image analysis software (Clemex PE 4.0). The volume fraction of dispersoids was calculated according to the model in the literature [14] and shown in Eq. (1):

$$V_v = A_A \frac{\overline{KD}}{\overline{KD}+t}(1-A_{PFZ})$$ (1)

where $\overline{D}$ is the average equivalent diameter of dispersoids, which is calculated according to the literature [14]; t is the TEM foil thickness; A_A is the area percentage of dispersoids from TEM observation; A_{PFZ} is the area percentage of the particle free zone (PFZ) from OM measurements; and $\overline{K}$ is the average shape factor of dispersoids.

3. Results and discussion

3.1 Evolution of intermetallics with various Fe contents

Figure 1 shows the as-cast microstructure of experimental alloys at various Fe contents. It can be found that the microstructure is composed of a dominant gray intermetallic with the morphology of Chinese script and minor black intermetallic in all three alloys. However, the chemical compositions of dominant intermetallics are different with various iron content, which is shown in Table 2. For gray Chinese script intermetallic, Mn and Fe are all detected in Alloys 1F, 3F and 6F while Si is only present in Alloy 1F. For black intermetallic, both Mg and Si are present in three alloys. According to the SEM-EDS results shown in Table 2 and literature [16-18], the gray Chinese script intermetallic is identified as α-$Al_{15}(MnFe)_3Si_2$ in Alloy 1F but $Al_6(MnFe)$ in both Alloy 3F and 6F, while the minor black intermetallic is Mg_2Si in all three alloys. Occasionally, a small volume of α-$Al_{15}(MnFe)_3Si_2$ can also be observed in Alloys 3F and 6F.

In order to further distinguish the Chinese-script intermetallics, EBSD is performed in present work and the results are shown in Figure 2. Figure 2a and 2c display the intermetallics in Alloys 1F and 6F while their corresponding simulated patterns and results are shown in Figure 2b-c and 2e-f, respectively. In EBSD, the mean angular deviation (MAD) between the experimental and the calculated patterns represents the accuracy of the solution given by the software (Channel 5). A smaller value indicates a closer match between the experimental and simulated patterns. Normally, a MAD value lower than 0.7 is considered to be desirable for an accurate solution [19]. As shown in Figure 2c and 2f, the MAD value for α-Al(MnFe)Si and $Al_6(MnFe)$ is 0.205 and 0.347, respectively, which is much lower than 0.7. Therefore, the intermetallics in experimental alloys, such as the dominant α-Al(MnFe)Si in Alloy 1F and $Al_6(MnFe)$ in Alloy 6F, have been further confirmed with the combined use of EDS and EBSD.

To preclude the possibility of occasional observations in Figure 1, Figure 3 shows the SEM-backscatter images and element mapping of intermetallics at low magnification in Alloys 1F and 6F. As shown in Figure 3a-b, the distribution of Si is uniform in almost all the dominant intermetallics in Alloy 1F, which is from the α-Al(MnFe)Si intermetallic. Though Si is also mapped in Alloy 6F (Figure 3d), its distribution is greatly different with most intermetallics and only small volume of Si present in the map. Compared with the intermetallics in Figure 3c, it can be found the present of Si in Figure 3d is mainly from the Mg_2Si intermetallic (black phase in Figure 3c), confirming that the dominant intermetallic is Al_6(MnFe) in Alloy 6F while α-Al(MnFe)Si in Alloy 1F .

Figure 4 shows the simulated Al-Mn-Mg-Si-Fe phase diagram at given chemical composition (Table 1) using Thermo-Calc software. It can be found that the final intermetallics vary with the Fe contents at fixed temperature. After solidification (893K (620 °C)), the intermetallic is α-Al(MnFe)Si when the Fe content is up to 0.11%. The intermetallic changes to Al_6(MnFe) with minor α-Al(MnFe)Si when the Fe content lower than 0.65%. In the present work, the Fe content in Alloy 1F is 0.10 %, which is located in the area of "Al + α-Al(MnFe)Si" while Alloy 3F with 0.31 % Fe and Alloy 6F with 0.58 % Fe are in the range of "Al + Al_6(MnFe) + α-Al(MnFe)Si", further confirming the formation of two different dominant intermetallics resulted from the various Fe contents in experimental alloys.

In addition to the type of dominant intermetallics with various Fe contents, the volume fraction of intermetallics also changes with Fe contents, which increases with increasing Fe contents. As shown in Table 3, the volume fraction of dominant intermetallics at as-cast condition increases from 1.05% at 0.1 % Fe (Alloy 1F) to 2.18% at 0.3% Fe (Alloy 3F) and further to 3.82% at 0.6 % Fe (Alloy 6F). However, only a modest increase for Mg_2Si is found, which is changed from 0.22% in Alloy 1F and to 0.23% in Alloy 3F and to 0.31% in Alloy 6F.

Figure 5 shows the intermetallic microstructure after the precipitation treatment (648K (375°C)/48h). Compared with the as-cast microstructure in Figure 1, the precipitation treatment does not cause any change for the intermetallic type, namely α-Al(MnFe)Si in Alloy 1F and Al_6(MnFe) in Alloys 3F and 6F, except their partial fragmentation. Besides, Mg_2Si is still present in all three alloys. The evolution of the intermetallic amount after 648K (375°C)/48h is summarized in Table 3. It can be found that there is a moderate increase on the dominant intermetallics in each alloy. For instance, the volume fraction of Al_6(MnFe) in Alloy 3F increases form 2.18% in as-cast condition to 2.51% after precipitation treatment. On the other hand, the volume fractions of Mg_2Si of three alloys are slightly decreased after precipitation treatment due to the diffusion of Mg and Si to the matrix.

3.2 Effect of Fe on the precipitation of dispersoids

Figure 6 shows the evolution of EC and microhardness of experimental alloys as a function of the holding time at 648K (375°C). As shown in Figure 6a and 6b, both EC and microhardness increase with prolonging holding time in all three alloys. At a fixed holding time, the values of EC increase with Fe contents. The microhardness of Alloy 3F exhibits the highest values and Alloy 6F yields the lowest microhardness values.

According the literature [11, 14, 20], the evolution of EC can be expresses as an indicator of element concentration in solid solution, which is expressed in the following equation:

$$1/EC=0.0267+0.032Fe_{ss}\%+0.033Mn_{ss}\%+0.0068Si_{ss}\%+0.003Mg_{ss}\%+0.0021particle\% \quad (2)$$

where, $Fe_{ss}\%$, $Mn_{ss}\%$, $Si_{ss}\%$ and $Mg_{ss}\%$ and particle% are all in the weight percent. As can be seen from Eq. (2), Si_{ss} and Mg_{ss} and particles have much less effect on EC than Mn_{ss} and Fe_{ss}, and almost of all Fe has been in the form of intermetallics during solidification, such as $Al_6(MnFe)$ and α-Al(MnFe)Si. Therefore, the change of EC during the precipitation treatment mainly depends on the concentration of Mn in solid solution. The increases of EC in all three alloys with the prolonging holding time of the precipitation treatment are due to the decreasing Mn in solid solution resulted from the decomposition of supersaturated solid solution. On the other hand, the increase of EC with increasing Fe content at a fixed time is likely resulted from various solute Mn concentrations in matrix due to different volume fractions of Mn and Fe contained intermetallics. As shown in Table 3, the volume fraction of intermetallics increases with increasing Fe content, leading to more consumption of Mn in intermetallics and therefore less solute Mn concentration in solid solution. Table 4 listed the estimated concentration of Mn in solid solution (C_{Mn}) from Eq. (2). It can be found the C_{Mn} decreases with increasing Fe content either at as-cast condition or after 648K (375 °C)/48h treatment.

Fig. 6b shows the evolution of microhardness during the precipitation treatment, in which the microhardness increases with prolonging holding time in all three experiment alloys. For instance, the microhardness increases from 55 HV at as-cast condition to 60.9 HV after 648K (375 °C)/72h in Alloy 6F, while it changes from 55.5 to 63.1 HV in Alloy 3F, indicating the dispersoid hardening effect during the precipitation treatment. Figure 7 shows the microstructure of experimental alloys after 648K (375°C)/48h. It can be found that most of dipsersoids formed in the grains while very few dispersoids located in the interdendrite grain boundaries, which is called the particle free zone (PFZ). Therefore, the hardness increases gradually during the precipitation treatment due to the continuous precipitation of α-Al(MnFe)Si dispersoids [11, 12, 15]. However, the influence of PFZ on microhardness is considered to be negligible because all the microhardness was measured within the grains.

However, the microhardness varies with Fe contents at a fixed holding time during the precipitation treatment and the highest value is always obtained in Alloy 3F. For instance, after 648K (375 °C)/48h the microhardness first increases from Alloy 1F (61.8 HV) to Alloy 3F (62.6 HV) but then decreases to Alloy 6F (60.5 HV) (Fig 6b). This can be explained by the different precipitation behaviour of dispersoids resulted from the various Fe contents. As shown in Figure 8, the dispersoids formed in Alloy 1F is larger than both Alloys 3F and 6F. The image analysis results (Table 5) show that the finest size and largest volume fraction of dispersoids are obtained in Alloy 3F. As shown in Table 5, the average equivalent diameter of dispersoids decreases from 74 nm in Alloy 1F to 62 nm in Alloy 6F and further to 52 nm in Alloy 3F, while the volume fraction of dispersoids increases from 2.55 % in Alloy 1F to 2.89 % in Alloy 6F and to the highest 3.14 % in Alloy 3F. Therefore, the significant contribution of dispersoid strengthening

results in the highest microhardness of Alloy 3F among three alloys studied. On the other hand, the solution strengthening can also contribute to the matrix hardness. As shown in Table 4, the Mn concentration in solid solution (C_{Mn}) after 648K (375°C)/48h is 0.78% in Alloy 1F and 0.59% in Alloy 6F, leading to less solid solution strengthening in Alloy 6. Although the dispersoids in Alloy 6 have finer size and higher volume fraction than in Alloy 1, a synergistic effect of C_{Mn} and dispersoids results in a higher microhardness in Alloy 1F than Alloy 6F.

As shown in Figure 8 and Table 5, the precipitation behavior of α-Al(MnFe)Sidispersoids is varing with Fe contents, which is related to their nucleation and growth mechanism. It is shown in Table 4, that the as-cast C_{Mn} decreases with increasing Fe content. Therefore, less dipersoids are expected to precipitate with increasing Fe contents. It is true in Alloys 3F and 6F, in which the volume fraction of dipsersoids decreases from Alloy 6F (0.6% Fe) to Alloy 3F (0.3% Fe). However, an exception happens in Alloy 1F (0.1% Fe), in which the volume fraction of dispersoids should be the highest. As a matter of fact, the volume fraction of dispersoids in Alloy 1F is lower than both Alloys 3F and 6F. This is probably due to the less nucleation sites of dispersoids during the precipitation treatment. According to the literature [21], Mg_2Si are the prerequisite and act as the potential nucleis for α-Al(MnFe)Si dispersoids. However, most of Si have been consumed to form as-cast α-Al(MnFe)Si intermetallic particles in Alloy 1F. Also, the α-Al(MnFe)Si dispersoids contain Si atoms thus a critical Si concentration is likely necessary for the dispersoid nucleaction. Conversely, as-cast $Al_6(MnFe)$ intermetallic particles in both Alloys 3F and 6F do not consume Si and more Si atoms are availible to form prerequisited Mg_2Si that promote the precipiptation of α-Al(MnFe)Si dispersoids during the precipitation treatment. Therefore, less Si is availible and less Mg_2Si forms in Alloy 1F for the nucleation of α-Al(MnFe)Si dispersoids, leading to a lower volume fraction of dispersoids. In fact, ΔC in Alloy 1F (Table 4) is lower than Alloys 3F and 6F, indicating the less Mn consumed for the precipitation of dispersoids and thus a lower volume fraction of dispersoids. Another possiblity is the relatively lower Fe available for the dispersoids in Alloy 1F compared with Alloys 3F and 6F, leading to the lower volume fraction of dispersoids in Alloy 1F. On the other hand, the dispersoids are easier to grow up once nucleated due to the higher Mn concentration in solid solution in Alloy 1F. With less nuclei, the size of dipersoids in Alloy 1F is bigger than Alloys 3F and 6F.

3.3 Effect of Fe on mechanical properties and creep resistance at elevated temperature

In order to evalute mechanical properties at elevated temperature, the compression strengths at 573K (300°C) have been tested after precipitation treatment (648K (375°C)/48h) and the results are shown in Figure 9. It can be observed that the YS at 573K (300°C) is relative high for all three alloys, indicating the remarkable strengthening effect of dipsersoidsat at elevated temperature. However, the YS and the compression strength at 0.1 strain vary with the iron contents. As shown in Figure 9, the YS increases from 78.5 MPa in Alloy 6F to 83.1 MPa in Alloy 1F and further to 86.6 MPa in Alloy 3F, while the compression strength increases from 88.2 MPa in Alloy 6F to 91.3 MPa in Alloy 1F and finally to 93.2MPa in Alloy 3F. It is worthwhile to mention

that the YS at 576K (300 °C) obtained in Alloy 3F can reach as high as 86.6 MPa, further confirming 3004 alloys after proper heat treatment for potential applications at elevated temperature.

Similar to the microhardness, the dispersoid precipitation and the solute concentration in solid solution are two major factors inflerencing the high temperature strengths. Besides, PFZ is reported to be deleterious to the mechanical properties [22, 23]. In the present work, the volume fraction of PFZ is also measured using image analysis. As shown in Figure 8 and Table 5, the area of PFZ in Alloy 3F is the smallest while it is the biggest in Alloy 6F. The volume fraction of PFZ is 25.9 % in Alloy 1F, 19.7 % in Alloy 3F and 30.1% in Alloy 6F, respectively, leading to the strengths increase from Alloy 6F to Alloy 1F and then to Alloy 3F. Therefore, the compression strengths at 573K (300°C) increases in the order of Alloy 6F, Alloy 1F and Alloy 3F under the combined effects of dispersoid precipitation, solid solution and PFZ.

For applications at eleveated temperature, the creep resistance is one of most important cosiderations in alloy selections [24, 25]. Therefore, the creep resistance at 573K (300°C) after 648K (375°C)/48h treatment has been assessed and the creep curves of three alloys are shown in Figure 10. It can be found that total strain in 100 hours during the creep test increases from Alloy 3F to Alloy 1F and to Alloy 6F, indicating that the creep resistance of Alloy 3F is better than 1F and 6F. The calculated minimum creep rates are 9.1×10^{-10} s^{-1} for Alloy 3F, 5.9×10^{-9} s^{-1} for Alloy 1F and 1.1×10^{-8} s^{-1} for Alloy 6F, respectively. The lowest creep rate is obtained in Alloy 3F, which has the best creep resistance at 573K (300°C).

According to the creep condition applied in the present work (45 MPa at 573K (300°C)), the creep behavior is mainly controlled by the glide and climb of dislocations [26]. Therefore, the creep resistance can be influenced by the factors that can have effect on the movement of dislocations, such as the grain boundary, solute atoms and dipsersoids. In the present work, the grain sizes in experimetnal alloys after 648K (375°C)/48h were measured using EBSD oritation mapping images and results shown the average grain sizes were 347 µm for Alloy 1F, 362 µm for Alloy 3F and 356 µm for Alloy 6F, respectively. It is evident that all the grains are relatively coarse (350-360 µm) in three experimental alloys and no major change are observed, indicating the negligible influence of grain size on the creep resistance. Therefore, similar to the evolution of hardness and strength, the highest volume of finer dipsersoids in Alloy 3F leads to the best creep resistance of Alloy 3F, while the the intergration of solute atoms and precipiation of dispersoids in Alloys 1F and 6F results in a slightly better creep resistance from Alloy 1F than 6F in a long-term holding [15, 27].

Similar to our previous work [15], a significant improvement on mechanical properties and creep resistance at elevated temperature has been achieved over a wide range of Fe contents studied (0.1 – 0.6%) due to the precipitation of dispersoids, which provides a great flexibility in the alloy design and manufacture process. Besides, a superior YS (86.6 MPa) and excellent creep resistance (9.1×10^{-10} s^{-1} minimum creep rate) at 573K (300°C) is achieved at the 0.3% Fe alloy in the present work, which is mainly benifited from the better dispersoid stengthening, such as the finest size and largest volume fraction of dispersoids among three alloys. Furthermore, the α-Al(MnFe)Sidipsersoids in 3004 alloy has been proved to be long-term thermally stable

at 573K (300°C) [15]. Therefore, it is likely to expect having stable and superior properties in the alloy with 0.3 %Fe during long-term thermal holding, confirming a strong potential of 3xxx alloys for applications at elevated temperatureas. It suggests that through optimizing the chemical composition, such as the Mn, Si and Mg contents, the mechanical properties and creep resistance at elevated temperature of 3xxx alloys can be further improved. Hences, a low cost solution for manufacturing aluminum products that can apply at elevated service temperature is envisaged using the common and economic alloying elements in 3xxx alloys.

4. Conclusions

In the present work, the influence of Fe contents on the evolution of microstructure and properties in Al-Mn-Mg 3004 alloys have been investigated, leading to the following conclusions:

(1) With increasing Fe contents, the dominant intermetallics change from α-Al(MnFe)Si at 0.1 % Fe to Al_6(MnFe) at both 0.3 wt. % and 0.6 wt. %Fe, which has been identified and confirmed by SEM and simulated phase diagram.

(2) A significant improvement on mechanical properties at elevated temperature after the precipitation treatment has been observed over a wide range of Fe contents studied (0.1-0.6 %), confirming the important strengthening effect of dispersoids of 3xxx alloys at elevated temperature.

(3) The best combination of microhardness, high temperature strength and creep resistance has been obtained in the intermediate Fe level (0.3 %) resulted mainly from the uniform distributed dipsersoids with finest size, highest volume fraction of dispersoids as well as the lowest volume fraction of particle free zone.

(4) The superior strength (YS of 86.6 MPa) and excellent creep resistance at 573K (300°C) achieved by the 0.3 % Fe alloy provide a strong potential of 3004 alloys for applications at elevated temperature with a low cost solution.

Acknowledgement

References

[1] J. G. Kaufman: *Properties of aluminum alloys : tensile, creep, and fatigue data at high and low temperatures*, ASM International ; Aluminum Association, Materials Park, Ohio; Washington, D.C.,1999.
[2] I. J. Polmear and M. J. Couper: *Metall. Trans. A*, 1988, vol. 19, pp. 1027-35.
[3] K. E. Knipling, R. A. Karnesky, C. P. Lee, D. C. Dunand and D. N. Seidman: *Acta Mater.*, 2010, vol. 58, pp. 5184-95.

[4] C. Booth-Morrison, D. C. Dunand and D. N. Seidman: *Acta Mater.*, 2011, vol. 59, pp. 7029-42.

[5] M. E. Van Dalen, D. C. Dunand and D. N. Seidman: *J. Mater. Sci.*, 2006, vol. 41, pp. 7814-23.

[6] M. E. Van Dalen, T. Gyger, D. C. Dunand and D. N. Seidman: *Acta Mater.*, 2011, vol. 59, pp. 7615-26.

[7] J. Lai, Z. Zhang and X. G. Chen: *Mater. Sci. Eng., A*, 2012, vol. 532, pp. 462-70.

[8] O. S. Es-Said, A. Zeihen, M. Ruprich, J. Quattrocchi, M. Thomas, K. H. Shin, et al.: *J. Mater. Eng. Perform.*, 1994, vol. 3, pp. 123-34.

[9] R. Kamat: *JOM*, 1996, vol. 48, pp. 34-38.

[10] Y. Li and L. Arnberg: *Essential Readings in Light Metals* John Wiley & Sons, Inc., Hoboken, NJ, USA, 2013, vol. 3, pp. 1021-27.

[11] A. M. F. Muggerud, E. A. Mørtsell, Y. Li and R. Holmestad: *Mater. Sci. Eng., A*, 2013, vol. 567, pp. 21-28.

[12] Y. J. Li, A. M. F. Muggerud, A. Olsen and T. Furu: *Acta Mater.*, 2012, vol. 60, pp. 1004-14.

[13] H.-W. Huang and B.-L. Ou: *Mater. Des.*, 2009, vol. 30, pp. 2685-92.

[14] Y. J. Li and L. Arnberg: *Acta Mater.*, 2003, vol. 51, pp. 3415-28.

[15] K. Liu and X. G. Chen: *Mater. Des.*, 2015, vol. 84, pp. 340-50.

[16] M. Dehmas, E. Aeby-Gautier, P. Archambault and M. Serrière: *Metall. Mater. Trans. A*, 2013, vol. 44, pp. 1059-73.

[17] Y. J. Li and L. Arnberg: *Mater. Sci. Eng., A*, 2003, vol. 347, pp. 130-35.

[18] D. T. L. Alexander and A. L. Greer: *Acta Mater.*, 2002, vol. 50, pp. 2571-83.

[19] K. Liu, X. Cao and X. G. Chen: *Metall. Mater. Trans. A*, 2013, vol. 44, pp. 682-95.

[20] Q. Zhao, B. Holmedal and Y. Li: *Philosophical Magazine*, 2013, vol. 93, pp. 2995-3011.

[21] L. Lodgaard and N. Ryum: *Mater. Sci. Eng., A*, 2000, vol. 283, pp. 144-52.

[22] Y.-L. Deng, Y.-Y. Zhang, L. Wan, A. Zhu and X.-M. Zhang: *Metall. Mater. Trans. A*, 2013, vol. 44, pp. 2470-77.

[23] T. Ogura, S. Hirosawa and T. Sato: *Sci. Technol. Adv. Mat.*, 2004, vol. 5, pp. 491-96.

[24] E. Arzt: *Res Mechanica*, 1991, vol. 31, pp. 399-453.

[25] K. E. Knipling, D. C. Dunand and D. N. Seidman: *Z. METALLKD*, 2006, vol. 97, pp. 246-65.

[26] G. E. Dieter: *Mechanical metallurgy*, McGraw-Hill, New York,1976.

[27] O. D. Sherby, A. Goldberg and O. A. Ruano: *Philosophical Magazine*, 2004, vol. 84, pp. 2417-34.

Figure captions:

Figure 1 As-cast microstructures of experimental alloys

Figure 2 - Intermetallics, EBSD patterns and simulated results for Alloys 1F and 6F:
(a), (b) and (c) is for α-Al(MnFe)Si from Alloy 1F (MAD = 0.205); and
(d), (e) and (f) is for Al_6(MnFe) from Alloy 6F (MAD = 0.347)

Figure 3 – SEM backscatter images (a and c) and element mapping for
Si distribution (b and d) in intermetallics in Alloys 1F and 6F

Figure 4 - Simulated Al-Mn-Mg-Fe-Si phase diagram using Thermo-Calc:
L-Liquid; α-Al_{15}(MnFe)$_3$$Si_2$; 6-$Al_6$(MnFe); 3-$Al_3$(MnFe)

Figure 5 - Microstructures of experimental alloys after 648K (375°C)/48 h (before etched)

Figure 6 - Evolution of EC (a) and microhardness (b) during the precipitation treatment

Figure 7 - Microstructure of experimental alloys after 648K (375°C)/48h (after etched)

Figure 8 - TEM images of dispersoids in experimental alloys after 648K (375°C)/48h

Figure 9 - Evolution of compression strengths at 573K (300 °C) after 648K (375°C)/48h:
(a) YS at 0.002 strain and (b) compression strength at 0.1 strain

Figure 10 - Creep curves of experimental alloys at 573 K (300 °C) after 648 K (375°C)/48h