Effect of grain refining and homogenizing treatment on Al-Fe-Mn-Si cast alloys

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Abstract. Al-Fe-Mn-Si alloys has showed wide application in aluminum industry mainly on rolled products used as food package and aluminum foil. Microsegregation is very common in as cast products, therefore homogenizing treatment is recomendable in order to reduce it before rolling through diffusion of segregated elements to aluminum-**a** phase. It was used AA8006 alloy with four titanium contents: 0,01%, 0,05%, 0,1% e 0,2% wt. The target is analyzing the titanium influence on grain refining with little solidification range alloy and diffusion of segregated elements. After obtaining, alloys were chacterized on optical microscope and SEM. Homogenizing treatment involved heating in a electrical furnace with 480, 520 and 560° C for 12hs. Both as-cast and homogenized samples showed a decreasing of grain size up to 0,05%wt Ti and increasing after this value due to the fade phenomena. Besides, it was observed phases transformations like Al(Fe, Mn) to Al(Fe, Mn, Si) that is favoured to low solubility of Fe, Si and Mn on aluminum.

Keywords: homogenizing, aluminum alloy, grain refining.

1. Introduction

The ability of knowing and find out the microstructure evolution of rolled products has become a great interest of aluminum industry. By joining together physically-based microstructural models for different elements of the process, the aim is predicting the formation of microstructure during direct chill (DC) or twin roll casting (TRC) and its development during homogenization and thermomechanical processing. (Alexander, 2002; Slamova, 2003). Al-Fe-Mn-Si has been applied widely for food package and housing hold foil (HHF) and its study became very important to evaluate productivity and quality of the final product. The necessity of using grain refiners as Al-Ti-B has been studied and the amount suitable to promotes the refining is still discussed. Thus, during the homogenization, determining the suitable amount of Al-Ti-B to promotes the grain refining and reducing of segregation is important.

Homogenization is a very important thermo process for non heat treatable and heat treatable wrought aluminum alloys. The aim of this process is eliminating microsegreagation, reducing manganese in solid solution and obtaining desirable size and distribution of constituent particles including fine dispersoids and coarse primary particles, which have strong influence on recristallization kinetics, texture development, grain size and mechanical properties of the alloy. An important phase transformation is (Fe, Mn)Al₆ to Al (Fe, Mn) Si during homogenizing of a few aluminum alloys if there is enough silicon content. Fe and Si addition promotes the precipitation of dispersoids and decreases the solubility of Mn in solid solution. (Yanjun, 2003; Slamova, 2000; Fogazzi, 2002)

The aim of this work is studying the grain refining of alloys with narrow solidification range and analyzing this effect on microsegregation formation because Al-Ti-B alloy must reduces the dendritic formation and then decreases the segregation. Besides, it was analyzed the phases tranformations, grain size evolution and particles distribution during homogenization in different temperatures.

2. Experimental procedure

It was used an Al-Fe-Mn-Si with chemical composition according to the table 1 determined with X-Ray Fluorescence equipment.

Table 1. Chemical composition of Al-Fe-Mn-Si alloy

Alloy	Si	Fe	Mg	Mn	Cu	Ti
AA8006	0.20	1.45	0.02	0.50	0.05	< 0.01

Ingots were melted and 4 billets were produced using a metallic mould with 4 different titanium content: 0.01, 0.05, 0.1 and 0.2% wt by adding Al-5Ti-1B master alloy at thixoforming laboratory UNICAMP/FEM. The samples were cutted and characterzed their macro and microstructure throughout anodyzing method that produces photos in color. Ascast samples were machined with dimensions 10x10mm and homogenized in a electrical furnace (Universidade Paulista – Laboratory of Materials Science) at temperatures 480, 520 and 560°C for 12 hours. After, grain size was measured to

characterize the evolution during heat treatment. Scanning electron microscope (SEM) (UNICAMP/FEM) JEOL was used in order to determine the phase tranformations (Fe, Mn)Al $_6$ to Al(Fe, Mn)Si and study the diffusion of elements as Mn through aluminum matrix.

This work only analyzed alloys with 0,01 and 0,05% wtTi.

3. Results and discussion

3.1. As-cast samples analyzis

Figure 1 shows the macrostructure of as-cast samples. It is possible to observe that 0.05% wtTi promotes the maximum refining effect. Addition of 0.2% wtTi generated the fade phenomenum in which the TiB₂ particles agglomerate lose their refining action. It is possible to realize that some points (b) shows ultra-refined grains, other points (d) with refined grains and the others with non-refined grains. Figure 2 shows the grain size according to the titanium content. It is clear that the maximum refining happens with 0.05% Ti. The refining effect occurs due to the presence of TiB₂ involved by TiAl₃ that promotes the high nucleation rate of aluminum- α . After 0.05% wtTi, the fading phenomenum happens. TiB₂ particles generate colonies and lose their refining effect. It can be observed in alloys with 0.1 and 0.2% wtTi.

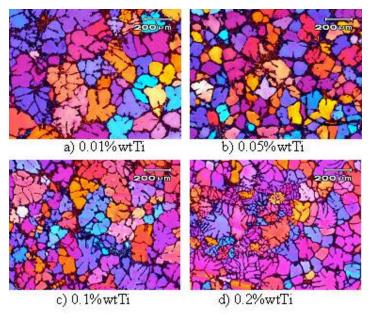


Figure 1. Macrostructures of Al-Fe-Mn-Si alloy with different titanium contents.

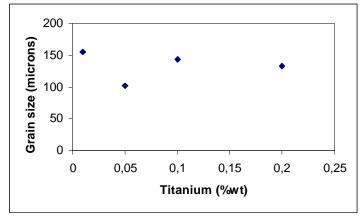


Figure 2. Grain size evolution with titanium content.

Figure 3 shows the microstructure of Al-Fe-Mn-Si alloy with different titanium contents. It is possible to observe the FeAl₃ and (Fe, Mn)Al₃ particles as chinese script dark color. Al (Fe, Mn) Si particles can also be observed in a few points and are lighter than the other phases. However, it will be clearer after homogenizing.

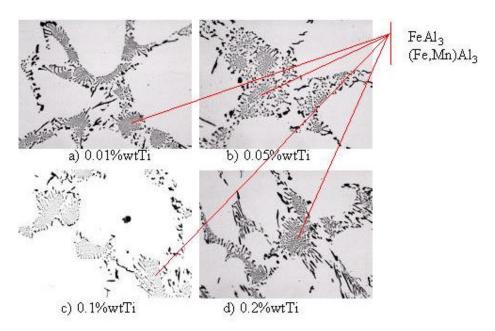


Figure 3. Microstructures of Al-Fe-Mn-Si alloy with different titanium contents. (magnification 500X)

3.2 Homogeneized samples analysis

Figure 4 shows the grain size evolution of Al-Fe-Mn-Si alloys according to the temperature after the homogenization treatment. It is possible to observe that the grain size increases as a function of the temperature. It was expected that the growing rate of the 0.05% wTi alloy was lower than for the others. Analyzing the figure 4, it can be observed that this alloy must have lower growing rate due to the lower dendritic feature, it means that the lower the grain size, the lower is its segregation. During homogenizing, there is diffusion of some elements as iron and manganese to inside aluminum matrix. After all temperatures tested, the alloy with 0.05% wtTi showed the lower grain size and lower growing rate.

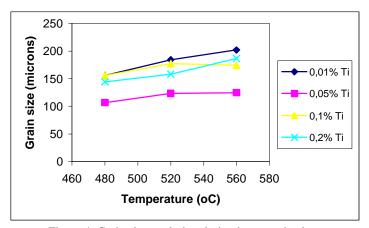


Figure 4. Grain size evolution during homogenization.

Figure 5 shows the macrostructure of Al-Fe-Mn-Si alloys according to the temperature after 12 hours of the homogenization treatment. The 0.05% wtTi alloy shows a different mophology than the others. Samples with 0.01, 0.1 and 0.2% wt Ti, shows emphasized dendritic features for all temperatures, it must allows some phases transformations throughout the grain boundaries. The low solid solubility of Mn in Al-matrix, 0.9% at. and Fe, 0.025% at. must favours the occurrence of phases transformations that will be discussed later after SEM analysis.

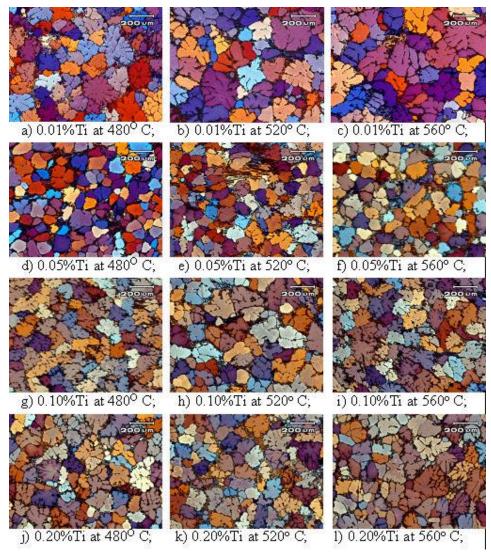
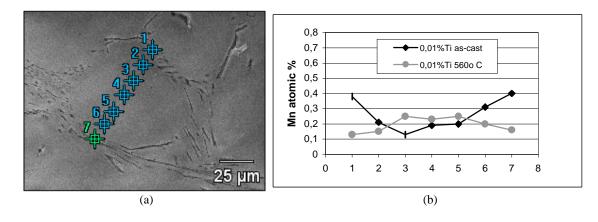
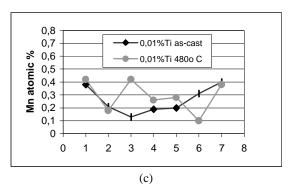


Figure 5. Macrostructures of Al-Fe-Mn-Si alloys after 12hs homogenization heat treatment at different temperatures.

3.3. SEM analysis

Figure 6(a) shows the microanalysis in seven different points throughout the grain. It was plotted the as-cast and 560° C homogenization conditions because it is clearer to observe the Mn diffusion phenomenum.





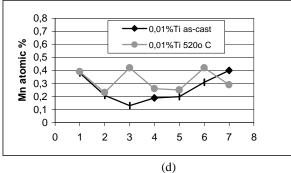


Figure 6. SEM analysis of AA8006 alloy with 0.01%Ti. Diffusion of Mn. (a) Points analized; (b) 560° C; (c) 480°C and (d) 520°C.

In truth, after 480°C (c) it is possible to observe the beginning of manganese diffusion but the maximum effect occurs at 560°C where the temperature is enough to do it. In the as-cast condition, it is possible to realize the high Mn concentration in the grain boundaries that is the result of the initial segregation during solidifying. After homogenizing, the tendency is the same chemical composition along of grain.

Figure 7 shows the diffusion of Mn across the grain. It was expected that alloys with 0,05% Ti in which the maximum refining was observed, would show lower amount of segregated elements in as-cast condition however, it was not observed. It is probably due to the low amount of manganese in the alloy that implies in a low amount of Mn segregated along the grain. Perhaps, if it was an alloy with higher maganese content, the effect of refining on the segregation would be observed easily.

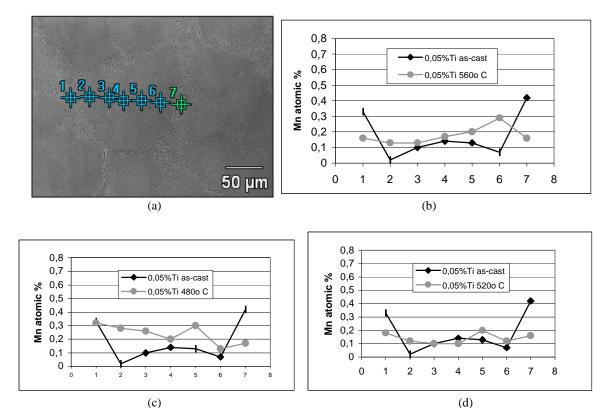


Figure 7. SEM analysis of AA8006 alloy with 0.05% Ti. Diffusion of Mn. (a) Points analized; (b) 560° C; (c) 480°C and (d) 520°C.

On the other hand, it is possible to observe the Mn diffusion is more homogeneous on alloys with 0,05% wtTi. Comparing figures 7 (c) and 6 (c) it is observed that alloy with 0,05% wtTi showed more Mn diffusion across the grain.

At 520°C, the Mn distribution is much more homogeneous in 0,05% wtTi alloy than with 0,01% wtTi. Probably due to the fact that the structure is less dendritic, the diffusion can become easier in alloys with 0,05% wtTi.

Some phases were identified before and after homogenizing. Figure 8 (a) shows the presence of a particle of (Fe, Mn) Al_n in the as-cast condition. Figure 7(b) shows a particle of (Fe, Mn, Si) Al_n after homogenizing.

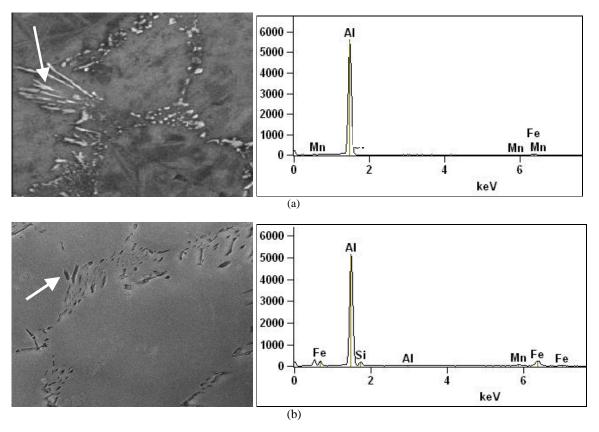


Figure 7. Phases identified in Al-Fe-Mn-Si alloys. (a) 0,01%Ti as-cast and (b) 0,01%Ti 560°C homogenizing.

The presence of silicon favours the occurence of phase transformation from (Fe, Mn) Al_n to (Fe, Mn, Si) Al_n during homogenizing and it could be observed, however, it is dependant of silicon content. The higher is the Si content, the higher is the phase transformation.

4. Conclusions

Al-Fe-Mn-Si alloys showed grain refining after addition of TiB₂ particles providing reduction of grain size even for alloys with narrow solidification range. The effect of refining did not result in effective reducing of segregated elements due to the low amount of manganese in this alloy that implied in difficulty to measurement. From 480°C homogenizing temperature it was possible to observe the diffusion of manganese to aluminum matrix and at 560°C the maximum diffusion was observed. Alloys with 0,05% wtTi showed more homogeneous Mn distribution along the grain than alloys with 0,01% wtTi. It was also possible to observe the presence of some intermetallic particles identified as (Fe, Mn) Al_n and (Fe, Mn, Si) Al_n that are typical for this alloy. Iron behaviour will be discussed in the next paper.

5. Acknowledgements

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6. References

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