# EFFECTS OF THE Fe/Mn WEIGHT RATIO AND COOLING RATE ON THE AREA FRACTIONS OF $\alpha$ -AlFeSi AND $\beta$ -AlFeSi PHASES IN Al-7.5Si-3.75Cu-0.5Mg-0.55Fe-xMn ALUMINUM ALLOY

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The effects of the Fe/Mn weight ratio (1,42 or 3,05) and cooling rate (0,1, 0,2, 0,5, or 0,6 °C/s) on the area fraction of  $\alpha$ -AlFeSi and  $\beta$ -AlFeSi intermetallic compounds in Al-7.5Si-3.75Cu-0.5Mg-0.55Fe-xMn aluminum alloy were studied. It was found that the difference between the formation temperature of the  $\beta$ -AlFeSi phase and the formation temperature of the eutectic Al-Si may determine the possibilities of  $\beta$ -AlFeSi elimination/reduction by increasing the cooling rate.

Keywords: aluminium alloy, cooling rate, thermal analysis, solidification, intermetallic compounds

## INTRODUCTION

Iron is a common impurity present in commercial Al-Si alloys [1-3] and has consistently emerged as the impurity element that may be the most detrimental to the mechanical properties of these alloys [4]. When this element is combined with the aluminum and other alloying elements, iron intermetallic compounds are formed. The iron-bearing compounds that form during solidification appear in a great variety of shapes and sizes and can generally be divided into three different morphologies:  $\beta$ -Al<sub>s</sub>FeSi-needles,  $\alpha$ -Al<sub>s</sub>(Fe,Mn)<sub>2</sub>Si<sub>2</sub>-Chinese scripts and polyhedral and/or star-like crystals. The amount, size and shape of these iron compounds depend largely on the cooling conditions and on the chemical composition, especially the level of iron in the melt [5].  $\beta$ -Al<sub>s</sub>FeSi-needles iron compounds have usually been considered most detrimental to the mechanical properties of Al-Si alloys because of the brittle features and stress concentration caused by the needle-like morphology. This kind of intermetallics is usually modified into Chinese script, which has been thought to be less detrimental to the alloy's mechanical properties because of its compact and globular morphology [6]. To reduce or eliminate the formation of the  $\beta$ -AlFeSi phase, several methods have been used, such as melt superheat [7], rapid solidification (a rapid cooling rate) [8], and chemical modification [9]. Chemical modification is the most widely used of these techniques [9].

The addition of manganese expands the thermodynamic stability region of the  $\alpha$ -AlFeSi phase, even at high iron contents [9]. In practice, the Fe/Mn weight ratio is considered a reference to control  $\beta$ -AlFeSi formation: as a rule, when the iron exceeds 0,45 wt. %, the manganese content should not be less than half of the iron content [10].

In a previous study [11] it was shown that Fe/Mn weight ratio that allows to avoid  $\beta$ -AlFeSi formation could depends of both parameters iron content and cooling rate.

The objective of this research was to study the effects of the cooling rate and the Fe/Mn weight ratio on the area fractions of the intermetallic phases in alloys with 0,55 wt. % iron and different manganese content.

## **EXPERIMENTAL PROCEDURE**

The alloys were prepared in a SiC crucible floor furnace. Commercial aluminum ingots of high purity (purity of 99,80 wt. %), metallic silicon with low calcium (purity of 99,62 wt. %), copper wire for electrical use (purity of 99,97 wt. %), iron wire (purity of 98,13 wt. %), electrolytic manganese flakes (purity of 99,54 wt. %), and magnesium (purity of 99,59 wt. %) were used as base materials. The chemical compositions of the alloys studied were determined by spark optical emission spectrometry and are shown in the Table 1.

The thermal analysis started with isothermal holding at 750 °C, followed by cooling at different rates: 0,1 °C/s (using a graphite cup covered with a ceramic fiber blanket), 0,2 °C/s (natural convection), 0,5 and 0,6 °C/s (forced convection by air). The experimental equipment was similar to that used by Belmares *et al.* [11]. The cooling rate,  $\phi_p$ , was calculated according to the crite-

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Alloy	Chemical composition / wt. %							
Alloy	AI	Si	Fe	Cu	Mn	Mg	Fe/Mn	
A: Fe/Mn weight ratio low	87,46	7,33	0,54	3,79	0,38	0,50	1,42	
B: Fe/Mn weight ratio high	87,74	7,43	0,52	3,60	0,17	0,54	3,05	

rion used by Narayanan, which specifies that  $\phi_{D}$  has to be measured during the formation of the dendritic network, in the range of 590 to 560 °C [12]. Once the thermal analysis experiments were completed, the samples were cross sectioned at the top, middle, and bottom. The samples were prepared for analysis in accordance with ASTM E3-0. To quantify the area fraction of the  $\alpha$ -AlFeSi +  $\beta$ -AlFeSi,  $\alpha$ -AlFeSi, and  $\beta$ -AlFeSi phases, 30 micrographs of each section of the samples were obtained using a scanning electron microscope (SEM) (Jeol®, model JSM-6300), using backscattering electron analysis with an amplification of 400X, for a total area of 58 mm<sup>2</sup> for each sample. The images were analyzed using the Pro Plus Image V.4.0 software. Identification of the phases was made according to their morphology. The area fractions reported for the phases studied were the averages of the measures of the three sections.

#### **RESULTS AND DISCUSSION**

Table 2 shows the cooling rates calculated using the criteria proposed by Narayanan [12], and the  $\alpha$ -AlFeSi and  $\beta$ -AlFeSi area fractions values. This table also shows the differences ( $\Delta T_{\beta-e}$ ) between the temperatures of  $\beta$ -AlFeSi ( $T_{\beta-AlFeSi}$ ) and eutectic Al-Si formation ( $T_{Al-Si}$ ). For reference, values of secondary dendritic arm space SDAS of the samples tested are also shown in Table 2. Figure 1 shows the sums of the area fractions of  $\alpha$ -AlFeSi +  $\beta$ -AlFeSi as a function of the cooling rate for both of the Fe/Mn weight ratios studied. These results show that as the cooling rate increases, the average area fraction of intermetallic compounds decreases. The sum of the area fraction of  $\alpha$ -AlFeSi +  $\beta$ -AlFeSi in the alloy with an Fe/Mn weight ratio of 3,05.

Figure 2 shows the experimental data corresponding to the fraction of  $\alpha$ -AlFeSi and  $\beta$ -AlFeSi as a function of the cooling rate for both of the Fe/Mn weight ratios studied. At any cooling rate, the area fraction of the phase  $\alpha$ -AlFeSi in the alloy with an Fe/Mn weight ratio of 1,42 was greater than that with an Fe/Mn weight ratio of 3,05, while the opposite trend was observed for the  $\beta$ -AlFeSi area fraction. As the cooling rate increased, the  $\alpha$ -AlFeSi area fraction decreased in both alloys. However, in the alloy with an Fe/Mn weight ratio of 1,42, a small area fraction of phase  $\beta$ -AlFeSi only occurred at a cooling rate of 0,22 °C/s, while in the alloy with an Fe/Mn weight ratio of 3,05, the  $\beta$ -AlFeSi phase was present at all cooling rates tested and was at a maximum at 0,28 °C/s, with lower  $\beta$ -AlFeSi area fraction values at cooling rates of 0,10 °C/s and 0,64 °C/s.

For the alloy with an Fe/Mn weight ratio of 1,42 (see Table 2),  $\beta$ -AlFeSi temperature formation could not be identified for any conditions. This result suggests that the  $\beta$ -AlFeSi phase might be formed simultaneously in a ternary eutectic with Al and Si; then,  $\beta$ -AlFeSi could have difficulty forming.

In the alloy with an Fe/Mn weight ratio of 3,05, the  $\beta$ -AlFeSi formation temperature (T<sub>B-AlFeSi</sub>) was detected before the eutectic Al-Si formation temperature  $(T_{Al-Si})$ at cooling rates of 0,28, 0,43, and 0,64 °C/s. Also it is observed in Table 2 that when the cooling rate increased,  $T_{\beta\text{-AIFeSi}}$  tended to decrease. These results suggest that the value of  $\Delta T_{\beta-e}~(T_{\beta-AlFeSi}\text{-}T_{Al-Si})$  could be considered a reference temperature for avoiding β-AlFeSi phase formation with increasing cooling rate. In other words, if  $\Delta T_{\beta\text{-e}} \left(T_{\beta\text{-AlFeSi}}\text{-}T_{\text{Al-Si}}\right)$  is lower, the  $\beta\text{-AlFeSi}$  will be less likely to precipitate, (case of alloy A: Fe/Mn weight ratio = 1,42), and therefore,  $\beta$ -AlFeSi will be suppressed at moderate cooling rates. When  $\Delta T_{\beta_{-e}}$  is higher, a high cooling rate is needed to avoid  $\beta$ -AlFeSi formation, as in the case of alloy B with an Fe/Mn weight ratio = 3,05, in which the  $\beta$ -AlFeSi phase was not eliminated even at a cooling rate of 0,64 °C/s. Higher cooling rates could be needed to eliminate or reduce further the area fraction of this intermetallic compound.

For example, Narayanan [12] studied an alloy Al-6Si-3.5Cu-1.2Fe-0.8 Mn-0.3 Mg (Fe/Mn weight ratio

φ <sub>D</sub> *	SDAS (µm)	$T_{\beta\text{-AIFeSi}}$	T <sub>AI-Si</sub>	$\Delta T_{\beta - e}^{**}$	f <sub>α</sub>	Error***	$f_{\beta}$	Error***				
A: (Fe/Mn weight ratio = 1,42)												
0,11	69,1		564,8		5,88	0,37	0,00	0,00				
0,22	51,8		564,8		4,69	0,32	0,06	0,02				
0,43	40,8		559,9		3,28	0,23	0,00	0,00				
0,65	15,1		571,9		2,42	0,36	0,00	0,00				
B: (Fe/Mn weight ratio = 3,05)												
0,10	71,0		557,7		4,48	0,24	1,12	0,13				
0,28	45,1	575,9	555,2	20,7	1,60	0,17	1,83	0,14				
0,43	38,6	565,0	560,0	5,0	1,10	0,18	1,68	0,19				
0,64	15,6	568,2	560,5	7,7	0,89	0,06	0,59	0,04				

Table 2 Experimental data obtained in this study

\* $\phi_{D}$ : Measure to 590 - 560 °C., \*\* $\Delta T_{\beta \cdot e} = T_{\beta \cdot AIFeSi} - T_{AI \cdot Si}$ , \*\*\*Error: Standard error of the mean.

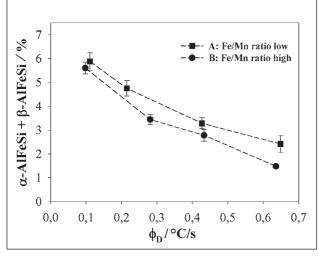


Figure 1 Area fraction of  $\alpha$ -AlFeSi +  $\beta$ -AlFeSi as a function of the cooling rate for the two alloys studied

of 1,50), for which  $\Delta T_{\beta-e}$  was estimated to be approximately 30 °C at a cooling rate of 2,5 °C/s. In such study,  $\beta$ -AlFeSi was observed even at high cooling rate as 16 °C/s. In another study, Belmares *et al.* [11] studied an alloy Al-7.1Si-3.25Cu-0.78Fe-0.5Mn-0.23Mg (with an Fe/Mn weight ratio of 1,56), and estimated a value of  $\Delta T_{\beta-e}$  of 10 °C at a cooling rate of 1,0 °C/s. The volume percentage of  $\beta$ -AlFeSi at this cooling rate was approximately 0,87 %, and decreased to 0,2 % when the cooling rate was increased to 3,5 °C/s. The findings of both of these studies are in agreement with the behavior observed in this study in the sense that it is difficult to eliminate or reduce  $\beta$ -AlFeSi formation by increasing the cooling rate when  $\Delta T_{\beta-e}$  increases.

## CONCLUSIONS

The sum of the area fraction of  $\alpha$ -AlFeSi +  $\beta$ -AlFeSi in the studied alloys diminished when cooling rate increase.

The difference between the temperature of formation of the  $\beta$ -AlFeSi phase (T<sub> $\beta$ -AlFeSi</sub>) and the temperature of formation of the eutectic Al-Si (T<sub>Al-Si</sub>) " $\Delta$ T<sub> $\beta$ -e</sub>" could determine the likelihood of reducing or eliminating the formation of  $\beta$ -AlFeSi by increasing the cooling rate. It was found that for an alloy A with an Fe/Mn weight ratio of 1,42, where  $\Delta$ T<sub> $\beta$ -e</sub>=0, the  $\beta$ -AlFeSi phase was suppressed at cooling rates of 0,11 °C/s or higher. However,  $\beta$ -AlFeSi phase formation was not eliminated by cooling rates in the range of 0,10-0,64 °C/s for an alloy with same iron content and an Fe/Mn weight ratio of 3,05, for which a value of  $\Delta$ T<sub> $\beta$ -e</sub> of 7,7 °C was observed for a cooling rate of 0,64 °C/s.

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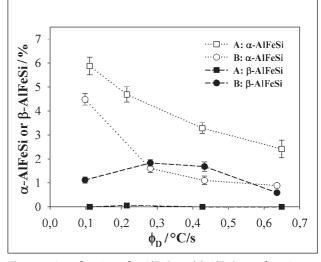


Figure 2 Area fraction of  $\alpha$ -AlFeSi and  $\beta$ -AlFeSi as a function of the cooling rate for the two alloys studied

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- Note: The responsible for English language is the lecturer from Cinvestav Saltillo, México