OPTIMIZING IRON REMOVAL IN SECONDARY ALUMINUM ALLOYS: ROLE OF MANGANESE, CHROMIUM, AND SILICON

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Abstract

Aluminum alloys are renowned for their exceptional strength-to-weight ratio, superior castability, excellent thermal and electrical conductivities, and remarkable corrosion resistance. This distinctive combination of properties makes aluminum alloys highly versatile, finding applications across various industries, including automotive, aerospace, packaging, electrical wiring and cables, construction, and consumer electronics. Additionally, aluminum's high recyclability positions it as a key material in supporting a circular economy. Secondary aluminum has garnered significant attention for its low energy consumption, reduced emissions, and excellent recyclability. The energy required for secondary aluminum production is just 5% of the energy needed for primary aluminum production. However, as aluminum alloy products reach the end of their service life, substantial quantities of scrap aluminum alloys enter the recycling stream. This process inevitably introduces impurity elements, such as Si, Fe, Mg, and Mn, which can adversely affect the mechanical properties of the resulting alloys. Impurity contamination can significantly reduce the economic value of aluminum scrap, often leading to its downcycling into lower-value products. Controlling the impurity content in aluminum scrap melts is very crucial. This study explores the use of alloying elements like Mn and Cr to enhance the efficient separation of Fe in Al-Si alloys through a gravity sedimentation technique. The efficiency of Fe-containing intermetallic particle phase separation is influenced not only by the concentrations of Mn, Cr, and Si but also by critical factors such as holding temperature and settling time. The CALPHAD method was employed using ThermoCalc to determine the appropriate temperature for the formation of impurity-rich intermetallic phases in the experimental alloys. The presence of Mn and Cr facilitates the formation of impurity-rich intermetallic phases, while high Si content not only lowers the solidification temperature of the α-Al phase but also broadens the temperature range for the formation of impurity-rich intermetallic phases and expedite their sedimentation. A significant decrease (>70%) in the concentration both Fe and Mn was observed within 1h in the presence of chromium in the high Fe containing aluminum alloy. Building on the results of lab-scale experiments, a series of large-scale trials was conducted, successfully replicating the findings observed at the laboratory level during production trials.

Introduction and Motivation

Aluminum alloys are widely used in automotive, packaging, and aerospace applications due to their properties like good corrosion resistance, high strength-to-weight ratio, good formability, and machinability, as well as excellent thermal and electrical conductivity [1]. Recycled aluminum is less expensive and has a lower carbon footprint compared to producing aluminum from bauxite ore [2]. The excellent scrap reuse capabilities of aluminum alloys make recycling very economically attractive. Aluminum scrap comes from various alloy sources and contains differing amounts of alloying elements. The heterogeneity of scrap compositions makes it challenging to regulate impurity levels and achieve the desired alloy components when recycling. Historically, impurities in recycled aluminum alloys have been managed by diluting scrap with pure primary aluminum metal. This allows the target alloy composition to be achieved while avoiding downgrading of the entire scrap batch. Besides copper, magnesium, manganese and silicon, iron is a major impurity resulting from both primary production and recycling processes [3]. Controlling iron and managing its effects remains an important concern in aluminum alloy production, especially for recycled Al-Si alloys. During solidification iron precipitates as Fe-rich intermetallic phase due to the low solubility of Fe in solid aluminum. In particular, the needle-like β-Al5FeSi intermetallic phase that forms in recycled Al-Si alloys promotes shrinkage pores and porosity defects, deteriorating mechanical properties.

Review of Related Work

Many methods have been employed to minimize the harmful effects of the β-iron phase, including adding neutralizing elements like Be, Sc, Sr, Mn, Cr, Co, RE, Mo, V and B which stimulates formation of less deleterious α-intermetallic

particles [4-12]. The addition of selected elements in the Al-Fe-Si alloy forms quaternary intermetallics compounds with high melting points. Because of the high density of these intermediate intermetallic phases, removal of iron-rich intermetallics by filtration or gravity sedimentation is also possible by optimizing melt composition, temperature and holding time to promote precipitation. Among other neutralizing elements, manganese has been extensively studied to suppress the formation of the β-intermetallic phase. Numerous investigation emphasizes the impact of Mn concentration on phase transformation kinetics, showing that a controlled Mn:Fe ratio is essential to achieving effective phase modification. The efficiency of iron precipitation depended heavily on the Mn/Fe ratio (up to 1.5) and processing temperature, especially for alloys with high iron contents. Moreover, the harmful β-Al5FeSi phase could not be fully eliminated using either a low Mn/Fe ratio with slow cooling or a high Mn/Fe ratio with fast cooling [4, 13-16]. Besides, excessive manganese additions introduce brittle phases in the microstructure. Chromium (Cr) has been explored as an effective agent for the removal of iron (Fe) impurities in aluminum alloys, primarily due to its ability to form Fe-Cr-based intermetallic phases with high density and low solubility in aluminum melts. Studies have demonstrated that Cr can selectively react with Fe to form stable compounds such as Al_7Fe_2Cr or $Al_{13}Cr_2$, which segregate from the melt and settle due to their higher density. Additionally, some studies suggest that Cr + Mn additions improve not only Fe removal but also the mechanical properties of the alloy by minimizing harmful Fe-rich phases. However, the use of Cr requires careful consideration of cost and potential environmental implications, making its application a subject of ongoing investigation. For die-cast alloys, melt composition and temperature can be optimized to minimize the harmful effects of iron by promoting controlled sludge (α-Al(FeMnCr)Si intermetallic) formation. The sludge factor (SF), which predicts sludge formation, is calculated based on the concentrations of iron, manganese, and chromium. An SF above 1.7 indicates the formation of coarse, dense, iron-rich phases with undesirable morphologies [17-20].

Technology Approach

Efficient removal of iron (Fe) from aluminum alloys requires a thorough understanding of the mechanisms governing impurity segregation and intermetallic phase evolution. In aluminum-silicon-iron (Al-Si-Fe) systems, high-density Fe-rich intermetallic compounds naturally settle at the bottom of the melt under the influence of gravity. To leverage this behavior, it is critical to precisely control melt conditions, including temperature, composition, and holding time, to maximize the segregation of these impurity phases and mitigate irons detrimental effects on the alloy's mechanical and corrosion properties. Fe removal is highly sensitive to melt holding temperature and the concentration of alloying elements, which directly influence the formation, growth, and sedimentation of Fe-rich intermetallics. Therefore, identifying optimal conditions for Fe segregation and understanding how other alloying elements affect the nucleation and stability of these phases are crucial for refining processes. This study investigates the role of alloying elements in Fe removal from both low and high Si containing aluminum alloys through a combination of thermodynamic simulation and experimental validation. Computational thermodynamic analysis is required to determine whether an alloy with Fe will form impurity rich intermetallics and at what temperature they will form. Calculations were performed on phases present as a function of temperature and composition using the software ThermoCalc [21] with the commercial database TCAL8. The results aim to establish a robust technological approach for impurity control in aluminum recycling and refining, enabling enhanced alloy performance and sustainability.

Experimental Procedure

Experimental alloys with varying silicon (Si) and iron (Fe) contents (low and high Si) were sourced from an industrial secondary aluminum producer. The manganese (Mn) and chromium (Cr) concentrations were adjusted by alloying the base material with commercial Al-20Cr and Al-25Mn master alloys. The chemical composition of the ingots was tested by Optical Emission Spectrometer (SpectroMaxx LMM-14, Kleve, Germany). Approximately 2 kg of the base alloy was remelted at temperatures >800°C in a muffle furnace. The required amounts of Mn or Cr were added, after which the melt was cooled to specific target temperatures in the furnace and held for a designated duration. For the low-Si alloy, after the experiment, the furnace was turned off, allowing the melt to cool rapidly within the furnace. The final compositions were analyzed by cutting the solidified melt longitudinally. For the high-Si alloy, specific amounts of the melt were extracted at various time intervals and poured into a graphite crucible for compositional analysis, while the remaining melt was left to cool inside the furnace. Samples for microstructure analysis were prepared using standard metallographic techniques.

Results and discussion

Removal of Fe from low Si aluminum alloy

Effect of Mn on Fe removal

Fig. 1 shows the Scheil–Gulliver solidification curves after 0.8% and 2% Mn addition to Al-0.8Si-0.75Fe(wt.%) alloy. It predicts that all the intermetallic phases precipitated out after the solidification of α-Al. A higher precipitation temperature than that of α-Al is necessary for the physical separation of any impurity rich intermetallic phases. When the Mn concentration is high, the Al_6Mn intermetallic phase solidifies first as the temperature decreases, followed by the solidification of the α-Al phase. This behaviour suggests that using high Mn concentrations alone is not a viable approach for Fe removal from wrought aluminum alloys via the precipitation of Fe-rich intermetallic phases.

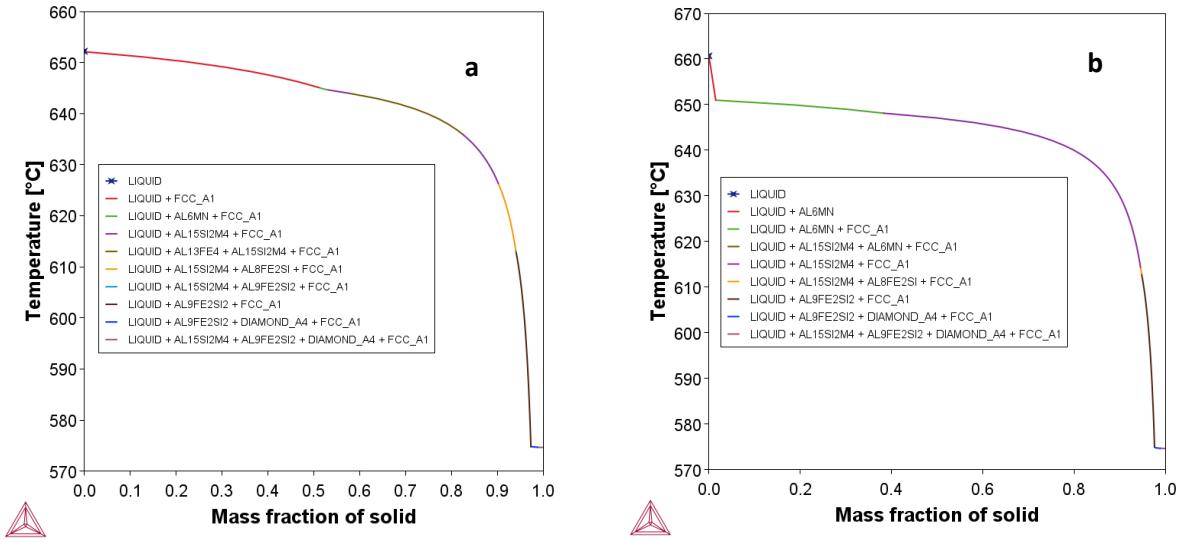

Fig. 1. The calculated Scheil-Gulliver solidification diagram with changing temperature in (a) Al-0.8Si-0.75Fe-0.8Mn alloy (b)Al-0.8Si-0.75Fe-2Mn.

Effect of Mn+Cr on Fe removal

Fig. 2a present the calculated solidification sequence of base alloy Al-0.8Si-0.75Fe-0.8% Mn after 1% Cr addition. The presence of both Mn and Cr promotes the formation of Fe-rich intermetallic phases in low Si alloys which suggests the possibility of removal of Fe/Mn via intermetallic precipitation-sedimentation route. α-AlFeMnCrSi is the primary phase appears during the solidification in the temperature range of 709º to 656ºC. Like Mn, the addition of Cr does not affect the α-Al phase formation temperature and, therefore, the holding temperature for the formation and separation of the Fe-rich intermetallic phases can be kept above the solidification temperature of α-Al phase. The variation of Fe and Mn contents during solidification could be understood more clearly by calculating the equilibrium concentration in the liquid in presence of Cr. Fig. 2b shows the variation in the iron and manganese in the liquid phase by varying the weight percentage of chromium addition at the holding temperature of 680ºC for the alloy Al-0.8Si-0.75Fe-0.8Mn-xCr (where x = 0-2%). The iron content in the liquid aluminum decreases significantly with increasing chromium according to the thermodynamic calculations. When Cr = ~0.9%, the equilibrium concentration of Fe was decreased from 0.75% to 0.35% at 680°C in the liquid phase (Fig. 2(b)). However, very minor decrease in Mn concentration has been predicted. These calculations demonstrate that the presence of Cr can significantly reduce the Fe content in the liquid phase of the alloy when maintained at temperatures between the liquidus and solidus. To verify this thermodynamic prediction, experimental validation was subsequently conducted.

Experiments were conducted using alloys with low Si content in the presence of Cr. Table 1 summarizes the chemical composition of the initial alloy and the percent reduction in Fe and Mn content after intermetallics sedimentation. As can be seen, there is a positive decrease after prolonged sedimentation time in the concentration of Fe and Mn in the separated alloy after Cr addition. However, the sedimentation of Fe-rich intermetallics was incomplete, with the majority of these particles distributed throughout the alloy, from the top middle regions to the bottom. Aluminum alloys with low silicon content exhibit relatively low fluidity at temperatures optimal for the sedimentation of impurity-rich intermetallics. This reduced fluidity hinders the effective removal of excess elements due to incomplete sedimentation. Ensuring high fluidity in the alloy system is crucial, as insufficient fluidity prevents the natural gravitational sedimentation of Fe/Mn-rich intermetallics that form during the process.

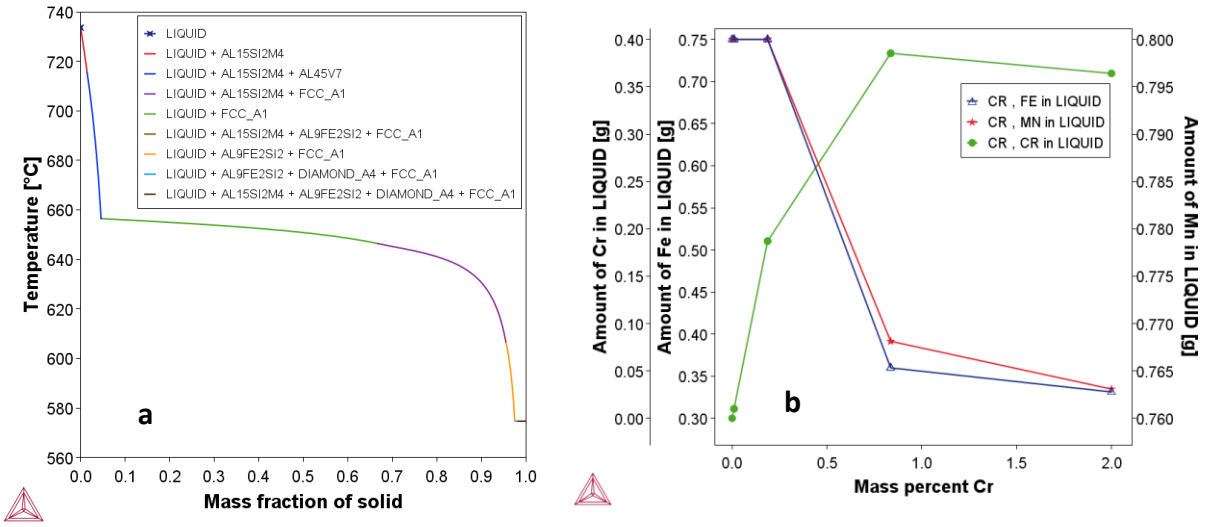

Fig. 2. (a) The calculated Scheil-Gulliver solidification diagram of Al-0.8Si-0.75Fe-0.8Mn-1Cr (wt.%) alloy and (b) Equilibrium concentration of Fe, Mn and Cr in liquid phase of alloy Al-0.8Si-0.75Fe-0.8Mn-xCr at 680ºC, calculated from equilibrium phase diagram.

Table 1: Effect of Cr on Fe/Mn removal from low Si containing Al-alloys (analysis of top portion of final alloy).

Initial Al- Alloy composition	Cr (%)	Tempº C (time)	Final Fe conc. (% Fe reduction)	Final Mn conc. (% Mn reduction)	Top portion Cr
Fe = 0.84%; Mn = 0.92%, Si = 0.8%	1.1%	660ºC (overnight)	0.67% (20)	0.69% (25)	0.32%
Fe = 0.73%; Mn = 0.9%, Si = 0.94%; Zn = 0.87%, Cu = 0.69%	0.85%	660ºC (overnight)	0.56% (23)	0.72% (20)	0.25%

Effect of Si on Fe removal

Low fluidity hinders the settling of impurity-rich particles, causing incomplete separation and impurity retention. Optimizing fluidity through alloy composition and processing is crucial for effective gravitational sedimentation. Silicon addition improves fluidity by lowering the melting temperature range, making it essential to determine the minimum Si concentration needed to facilitate the formation and sedimentation of Fe/Mn-rich intermetallic phases. The calculated equilibrium phase diagram for the Al-0.75Fe-0.8Mn alloy system with varying Si contents is presented in Fig. 3. The diagram indicates that the α-AlFeMnSi phase forms when the Si content in the melt system reaches a minimum of 5%. The calculated solidification sequences of Al-(5/7)Si-0.75Fe-0.8Mn (wt.%) alloys confirms that the presence of high Si not only decreased the solidification temperature of α-Al phase to 628ºC (5% Si) and 614ºC (7% Si) but also widens the formation temperature for impurity rich intermetallic phases. However, in the presence of both Mn and Cr, the α-intermetallic phase can form regardless of the Si content in the alloy. The CALPHAD calculation suggests that removal of Fe via intermetallic formation is highly dependent on Si level when only Mn is used.

Our previous sedimentation experiments with low-Si alloys containing Mn and Cr revealed that, although impurity-rich intermetallics formed in the low-Si alloy, their subsequent sedimentation did not occur due to the system's low fluidity. Therefore, to ensure successful sedimentation of the α-AlFeMnSi phase, the Si content in the alloy must be maintained at a certain level, regardless of the elements used for intermetallic formation. Preliminary experiments were carried out on A356 alloy with 5% Si to investigate the influence of silicon on the reduction of iron (Fe) and manganese (Mn) in the presence of 0.8% chromium (Cr). A notable reduction in the concentrations of both Fe and Mn was observed, accompanied by the accumulation of impurity-rich particles at the bottom of the alloy. Based on these findings, further experiments were conducted using high-Si alloys with varying concentrations of Mn and Cr to explore the effects in greater detail.

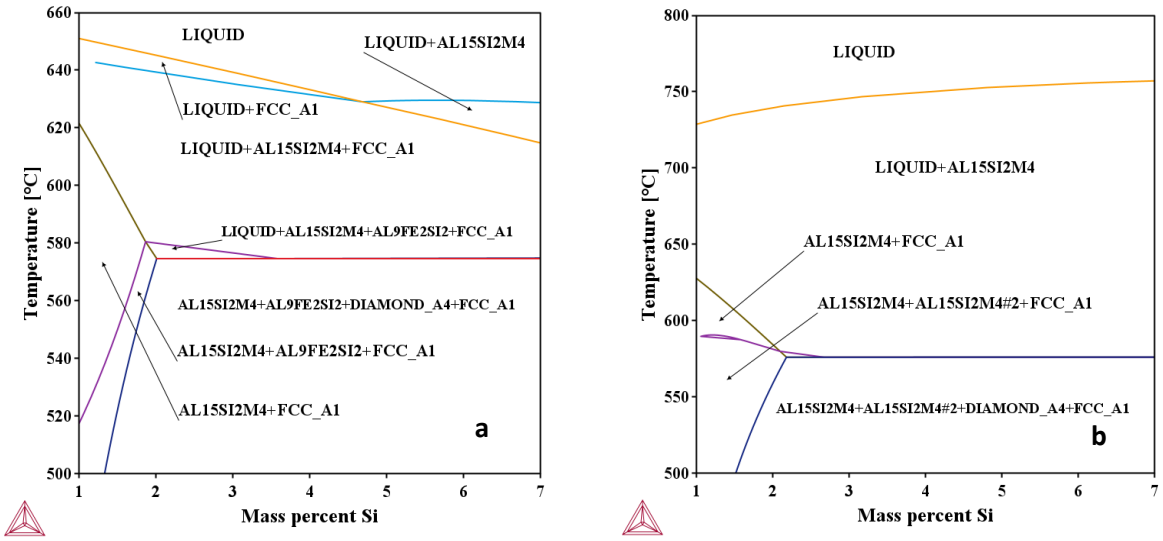

Fig. 3. Equilibrium phase diagram of (a) Al-0.75Fe-0.8Mn and (b) Al-0.75Fe-0.8Mn-0.8Cr alloy with different Si contents.

Removal of Fe from high Si aluminum alloy

Effect of Mn on Fe removal

The 3xx-series alloys 319 and A380 are two of the most important scrap-based high Si secondary alloys and are primarily used in engine blocks and cylinder heads. Their usage volume equals over 80% of all die-cast alloys. Taking this into account, the high-iron-content scrap A380 alloy was chosen for further investigation. The influence of manganese (Mn) was investigated first. The calculated phase diagram (Fig.4) for Al-7.5Si-3.5Cu-xFe-xMn alloy shows that α-Al(FeMn)Si primary intermetallics phase appear above the solidification temperature of α-Al (~600°C), suggesting that removal of Fe from high Si aluminum alloys via the Fe-rich intermetallic phase precipitation route is possible by adding manganese. The presence of high Mn concentration widens the temperature range and significantly enlarged the processing window for the precipitation of the impurity rich intermetallic phase.

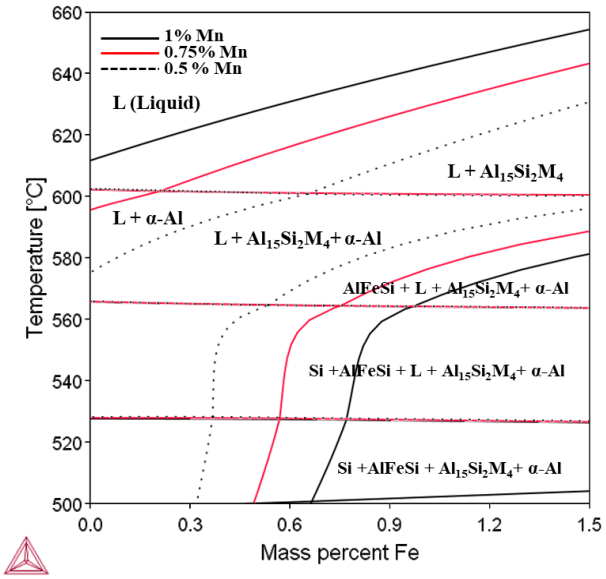

Fig. 4. Equilibrium phase diagram of Al-7.5Si-3.5Cu-xFe-xMn alloy with different Fe and Mn contents.

Based on CALPHAD results, a series of experiments were carried out by varying the Mn concentration from 0.5%-1.2% in the experimental alloy Al-7.5Si-3.5Cu-1.2Fe. In each experiment about 2 kg of alloy with known Mn content

was prepared and remelted at >800ºC and stirred to ensure complete dissolution. Subsequently the melt was cooled to predetermined temperature with the furnace cooling and held for certain time before sampling.
Table 2 present the changes in Fe and Mn concentrations in the alloy over time at holding temperatures of 630°C. The experimental data indicates that after the addition of Mn and subsequent holding, Mn and Fe concentrations in a high-Si alloy can be significantly reduced by maintaining different holding temperatures. The variation of Si was in the acceptable range. At a holding temperature of 630°C and an initial Mn content of 0.5%, the Fe and Mn concentrations decreased to 0.85% and 0.32%, respectively, within 3 hours—representing reductions of 30% and 36% from their initial values. When the initial Mn concentration was increased to 1.2% while maintaining the same holding period, the Fe and Mn levels further decreased to 0.56% and 0.46%, corresponding to reductions of 54% and 62%, respectively. Further lowering the holding temperature resulted in only a slight improvement in Fe and Mn reduction. Examination of the alloy cross-section confirms effective sedimentation, with impurity-rich intermetallic particles visibly clustered at the bottom.

Table 2. Removal efficiency of Fe and Mn from the Al-7.5Si-3.5Cu-1.2Fe alloy over time at a constant holding temperature of 630°C with varying Mn concentrations.

Time (min.)	0.5% Mn addition		0.7% Mn addition		1.2% Mn addition	
	%Fe	%Mn	%Fe	%Mn	%Fe	%Mn
60	1.22	0.47	1.06	0.65	0.66	0.54
120	1.05	0.36	0.92	0.54	0.61	0.50
180	0.85	0.32	0.72	0.42	0.56	0.46

Effect of Mn+Cr on Fe removal

Furthermore, the combined effect of Mn and Cr on Fe removal from the high-Si aluminum alloy through intermetallic sedimentation was also investigated. Prior to conducting the experiments, thermodynamic evaluations were performed using ThermoCalc software to guide the experimental setup. The equilibrium solidification curve of the alloys with adding Cr is shown in Fig. 5. Figure 5a and b show that the primary α-Al(FeMnCr)Si intermetallic phases were formed before the solidification of α-Al phase, with the addition of Cr. Furthermore, the initial formation temperature of impurity rich intermetallics significantly increased to 750°C and 780°C when Cr concentration increased from 0.3% to 0.8%, respectively. In other words, the higher the Cr content, the higher the initial formation temperature. Like Mn, the presence of high Cr concentration widens the temperature range and significantly enlarged the processing window for the precipitation and sedimentation of the impurity rich intermetallic phase.

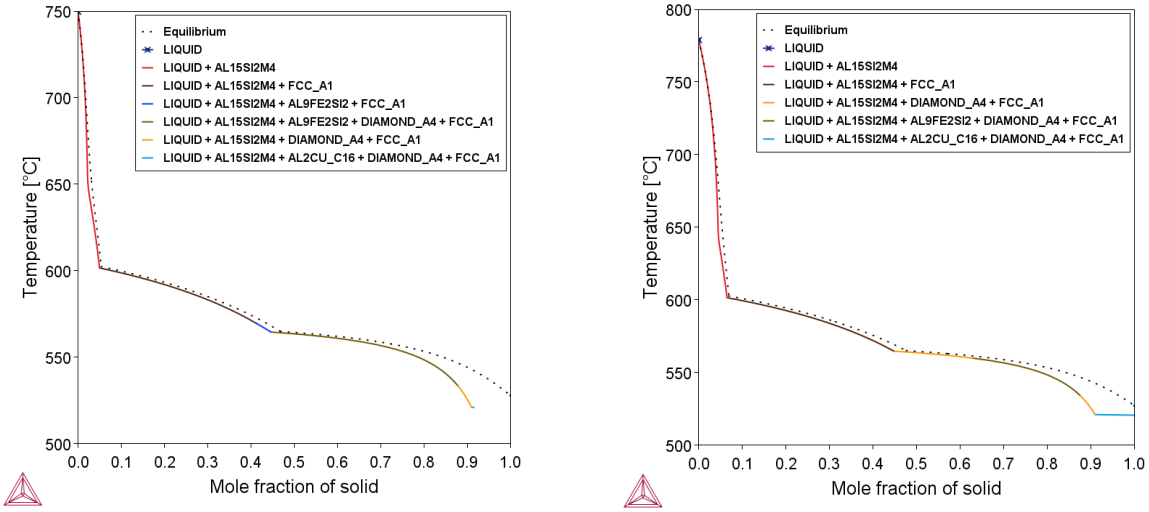

Fig. 5. The calculated Scheil-Gulliver solidification diagram of (a) Al-7.5Si-3.5Cu-1.2Fe-1.2Mn-0.3Cr and (b) Al-7.5Si-3.5Cu-1.2Fe-1.2Mn-0.7Cr (wt.%) alloys.

The variation of Fe and Mn contents in the liquid phase during solidification could be understood more clearly by calculating the equilibrium concentration in the liquid in presence of Cr. Fig.6 shows the expected effect of Cr addition

on the Fe and Mn levels in the liquid phase when the alloy is maintained at different holding temperatures of 630ºC and 615ºC. According to thermodynamic calculation, the Fe level can be reduced to 0.1% Fe irrespective of holding temperature when Cr is added up to 1% in the initial composition. At the same time, the Mn concentration can be reduced to 0.77% using the same concentration of Cr at 630ºC which suggests that addition of Cr is highly effective, most significantly for the reducing Fe in a high Si alloy. Chromium concentration significantly reduced in liquid with decrease in holding temperature. The small residual Cr concentration left in the melt would benefit the mechanical properties [22].

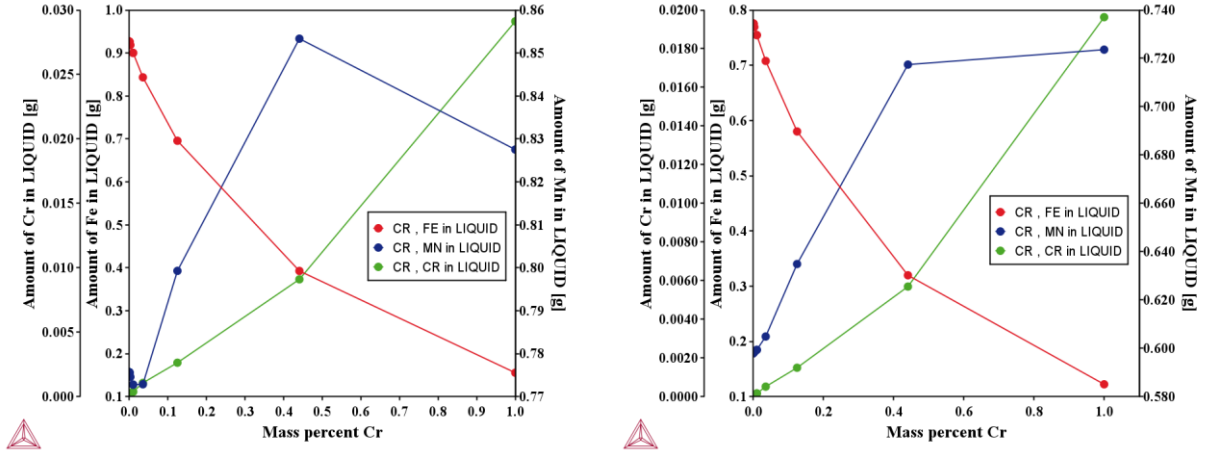

Fig. 6. Equilibrium concentration of Fe, Mn and Cr in liquid phase of alloy Al-7.5Si-3.5Cu-1.2Fe-1.2Mn-xCr during solidification at (a) 630ºC and (b) 615ºC, calculated from equilibrium phase diagram.

Based on thermodynamic predictions, the first series of experiments was conducted on an Al-7.5Si-3.5Cu-1.2Fe-0.1.2Mn alloy with varying Cr concentrations at 630°C for different durations. When 0.3% Cr added, the content of Fe and Mn in the alloy reduced to 0.61% and 0.51%, respectively in 1h of holding which is 51% and 58% lower than the initial values. A significant reduction in Fe (71%) and Mn (70%) was observed within 1 hour in the presence of 0.8% chromium in the alloy (Table 3). These results indicate that the simultaneous addition of chromium and manganese to high-silicon alloys has a synergistic effect on iron removal. When the holding temperature was reduced to 615°C, both Fe and Mn concentrations in the melt decreased to 0.29%, which is 76% lower than the values observed at 630°C. The further reduction in Fe and Mn with decreasing holding temperature can be attributed to the enhanced nucleation and growth rate of the sludge phase, driven by increased undercooling. This led to a more rapid diffusion of Mn and Fe, resulting in the formation of numerous large, high-density sludge phases during the holding process [23]. The cross-sectional analysis of the alloys demonstrates a well-defined accumulation of sedimented impurity-rich intermetallic particles at the bottom, indicating the potential industrial viability of the process. However, to fully realize this potential, the development of an effective filtration method to efficiently separate these intermetallic particles is essential.

Table 3. Removal efficiency of Fe and Mn from the Al-7.5Si-3.5Cu-1.2Fe-1.2Mn alloy over time at a constant holding temperature of 630°C with varying Cr concentrations.

Time (min.)	0.3% Cr addition			0.5% Cr addition			0.8% Cr addition		
	%Fe	%Mn	Residual Cr	%Fe	%Mn	Residual Cr	%Fe	%Mn	Residual Cr
60	0.60	0.51	0.12	0.48	0.47	0.10	0.35	0.37	0.18
120	0.48	0.43	0.06	0.45	0.40	0.09	0.30	0.32	0.15
180	0.49	0.40	0.05	0.44	0.38	0.08	0.30	0.31	0.14

Analysis of sedimented impurity rich intermetallic phases

SEM-EDS analysis was conducted on the sludge that settled on the bottom of the furnace in both experiments. The point EDS analysis (Fig.7) confirms the formation of α-Al(FeMn)Si intermetallic phase in the sludge formed when

Mn was used alone for Fe/Mn reduction whereas formation of α-Al(FeMnCr)Si was observed when Mn and Cr both were used for the reduction of Fe and Mn from high Si alloy.

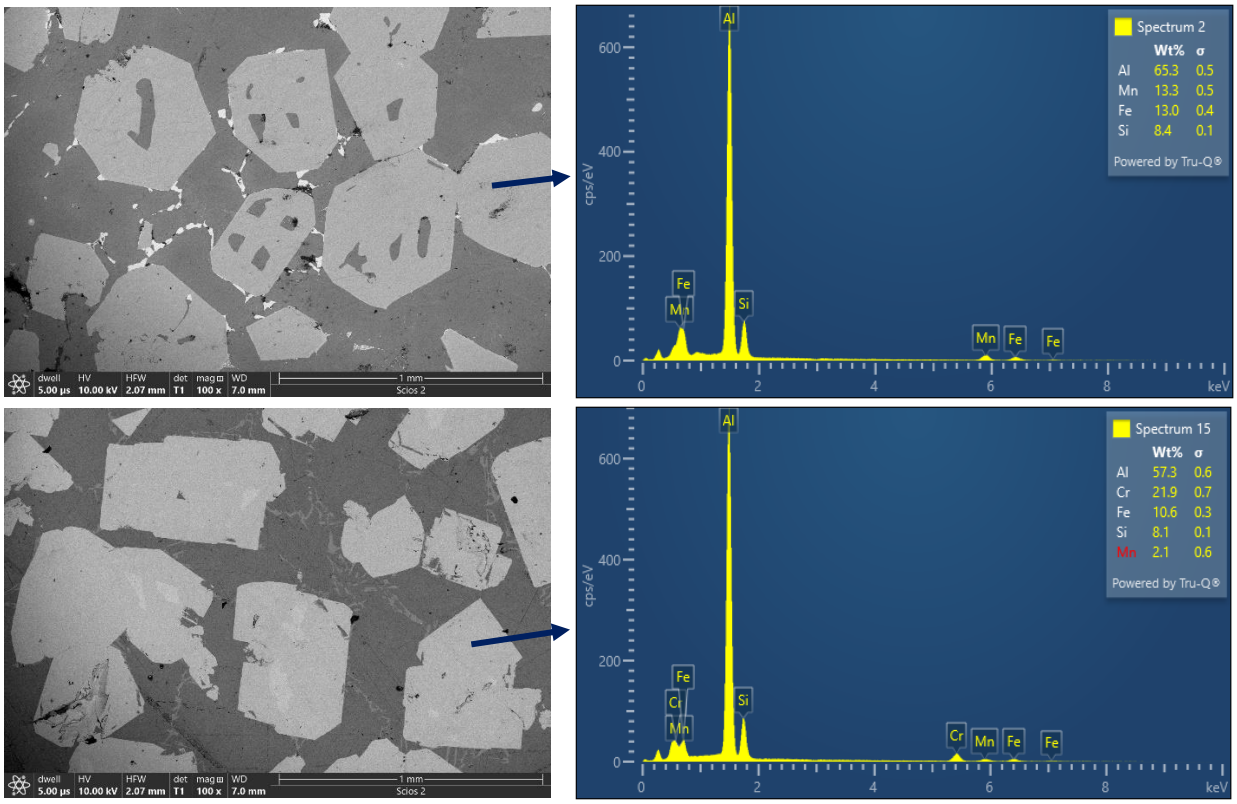

Fig. 7. SEM-EDS analyses of impurity rich intermetallic particles (a) only Mn (b) both Mn and Cr

Conclusions & Recommendations

The present study investigates the effect of different alloying element addition on the reduction of Fe content in low and high silicon aluminum alloys. The role of Mn, Cr and Si on removal of Fe through the Fe-rich intermetallics formation were explained based on CALculation of PHase Diagram (CALPHAD) thermodynamic simulations and experimental validation. Laboratory melt trials revealed that the intermetallic gravity sedimentation technique is ineffective for reducing Fe in low-Si aluminum alloys using both Mn and Cr, primarily due to the alloy's low fluidity. The addition of chromium significantly increases the precipitation temperature, facilitating the formation and accelerated sedimentation of Fe/Mn-containing phases in high-Si aluminum alloys. Additionally, reducing the holding temperature enables the growth of these phases, leading to more efficient removal of Fe and Mn through gravity sedimentation. A synergistic effect is observed when 1% Mn and 0.8% Cr are added, resulting in a 70% reduction of Fe and Mn in high-Si alloys containing over 1% Fe and Mn within 1 hour at a holding temperature of 630°C. To enhance process efficiency and yield, it is crucial to develop a suitable filtration system capable of effectively separating intermetallic particles formed during the sedimentation process, thereby ensuring the industrial viability of this method.

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