Evolution of Iron based intermetallic Phases in Al-7wt%Si hypoeutectic alloy

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Keywords: Al-Si-Fe alloys, High purity, Al-Si-Fe intermetallics, phase diagram

Abstract
This study has methodically characterized the iron based intermetallic phases evolving during solidification of Al-Si binary alloys as a function of solidification cooling rate and and composition of Fe in the alloys. Contrary to the predictions of the evolution of only the β (Al, Si, Fe) in these alloys by all commercial thermodynamic phase diagram simulation tools, the dominant phases were mostly the α (Al, Si, Fe) intermetallic phases and significantly vary in nature and type with the process parameters. The results from this study will further enable better design of the Al-Si alloys with an in-depth understanding of the evolution of the intermetallic phases and methodologies to prevent or modify the same in the final cast components.

Introduction
Al-Si alloys have significant applications in automotive, aerospace, defense and domestic sectors [1]. One of the detrimental impurities in these alloys is Fe which forms several forms of intermetallic phases such as binary phases with Al and ternary phases with Al and Si [2]. Thermodynamic phase diagram simulations reveal that very low concentrations of Fe in the order of 10 to 50 ppm is sufficient to alter the binary Al-Si system to a ternary Al-Si-Fe system [3]. The presence of certain Fe based intermetallic phases in solidified microstructure of cast components of Al-Si commercial alloys prove to be significantly detrimental to the mechanical properties and performance of the component [4, 5]. In this study, the Al-Si-Fe phases evolved during solidification of Al-7wt%Si alloy with 0.25 and 0.5 wt% Fe in them, respectively has been characterized with Scanning Electron Microscopy (SEM) coupled with Energy Dispersive X-Ray (EDX) analysis. The aim of the work is to verify the validity of predictions of these intermetallic phases evolving during solidification by commercial thermodynamic phase diagram software packages. Two cooling rates, namely, 1 and 5 °C.s\(^{-1}\) were used for solidification of the alloys.

Background
Most research [6, 7, 8, 9, 10, 11] on understanding the nature and structure of the Fe base intermetallic compounds in Al-Si hypoeutectic casting alloys (Al – 5 to 12.5 wt%Si) [1] have been carried out in complex multi-component commercial alloys. The prolific nature of Fe to form several complex intermetallic phases with most elements in the Al-Si commercial cast alloys throws more confusion than clarity on the nature of these phases observed in the commercial casting alloys. Rivlin et al [12] and Stefanis et al [13, 14] have presented an in-depth review of the intermetallic phases in the Al rich corner of the Al-Si-Fe ternary alloy phase diagram. However, there is no discussion presented on the evolution of the intermetallic phases during non-equilibrium solidification of Al rich Al-Si-Fe alloys which would have been more pertinent to the understanding the evolution of these intermetallic phases during solidification of commercial Al-Si casting alloys. Gupta [14] carried out diffusion couple experiments between pure solid Fe and liquid Al-12.5wt%Si eutectic alloy to observe and characterize the various Al-Fe-Si intermetallic compounds formed. The intermetallic phases obtain by Gupta [14] would all be terminal and near equilibrium phases and the list of intermetallic phases formed in such a diffusion couple reaction could not be assumed as comprehensive because of several Al-Si-Fe intermetallic phases that could evolve during non-equilibrium solidification conditions experienced during casting these alloys.

Predictions from thermodynamic simulations of solidification of Al-Si-Fe alloys using Scheil-Gulliver solidification criterion show only β-Al1.4FeSi phase as the Fe based intermetallic phase evolving as terminal phase for alloys with 0.05 to 2.3 wt% Fe in them [15, 3, 16, 17, 18, 19]. Figure 1 shows an isopleth of the Al-Si-Fe ternary phase diagram as simulated with the Pandat thermodynamic software with the PanAlB materials database [19], wherein the phase evolution with the addition of Fe to Al-7wt%Si alloy is shown. Figure 1 show that addition of trace levels of Fe to Al-7wt%Si is sufficient to enable the evolution of the β-Al1.4FeSi intermetallic phase during equilibrium solidification conditions. The two commercially important alloys of Al-7wt%Si with 0.25 wt% and 0.50 wt% Fe, respectively were chosen for this study and shown in Figure 1.

Figure 1: Isopleth of Al-Si-Fe ternary phase diagram showing the effect of Fe addition to Al-7wt%Si alloy. Also shown are the two experiment alloys selected for this study.
Experiments

Alloys were prepared and cast using 99.999% purity Al ingots, 99.9999% purity Si and Al-25%Fe master alloy. Figure 2 shows the schematic of the experimental matrix adopted for this study.

![Figure 2: Schematic of the experimental matrix](image)

A ceramic crucible with a coating of Boron Nitride was used to solidify the alloys at 1 °C.s⁻¹ and a steel casting mold was used for solidifying alloys at 5 °C.s⁻¹. Al was melted and held for 30 min at 820 °C followed by Si addition and holding time of 30 min; subsequently Fe was added and the alloy was held for an additional 60 min before it was cast into the ceramic crucible or steel mold. On-line temperature data during solidification was collected using two K-type thermocouples such that one was placed at the center (Tc) of the solidifying melt and the other at the outer edge (TE) of the melt in the same horizontal plane as the first thermocouple [20]; coupled with a laboratory data acquisition system. Specimens from the cast samples were sectioned around the thermocouple regions in the cast samples and prepared for metallographic analysis with a JEOL 7000 Scanning Electron Microscope (SEM) equipped with an Oxford instruments Energy Dispersive X-Ray Spectrometer (EDX).

Results and Discussion

Figure 3 is the typical back scatter electron image (BEI) obtained from the SEM for the Al-7wt%Si-0.25wt%Fe alloy solidified in the ceramic crucible at 1 °C.s⁻¹, showing the dark grey eutectic Si phase and the bright Fe rich intermetallic phases embedded in the Al matrix. In Figure 3, the only Fe based intermetallic phase observed was the α-Al₁₇Si₁₀Fe₂₀ whose stoichiometry was determined by EDX in the SEM and this phase is contrary to the one predicted by the Al-Si-Fe phase diagram shown in Figure 1. The morphology of the α-Al₁₇Si₁₀Fe₂₀ shown in Figure 3 was that of a Chinese script.

Figure 4 shows the typical BEI from the SEM for the Al-7wt%Si-0.50wt%Fe alloy for the two solidification conditions of 1 and 5 °C.s⁻¹ are shown in Figure 5 and Figure 6, respectively. In Figure 5, for the slower solidification rate, the β-Al₁₇Si₁₀Fe₂₀ intermetallic phase was solely observed in the solidified microstructure as predicted by the alloy phase diagram in Figure 1. In Figure 6, for the faster solidifying rate, the α-Al₁₇Si₁₀Fe₂₀ intermetallic phase was solely observed in the solidified microstructure, contrary to the phase diagram predictions in Figure 1.

The BEIs from the SEM for the Al-7wt%Si-0.50wt%Fe alloy for the two solidification conditions of 1 and 5 °C.s⁻¹ are shown in Figure 5 and Figure 6, respectively. In Figure 5, for the slower solidification rate, the β-Al₁₇Si₁₀Fe₂₀ intermetallic phase was solely observed in the solidified microstructure as predicted by the alloy phase diagram in Figure 1. In Figure 6, for the faster solidifying rate, the α-Al₁₇Si₁₀Fe₂₀ intermetallic phase was solely observed in the solidified microstructure, contrary to the phase diagram predictions in Figure 1.