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Precipitation of Fe-rich intermetallic phases in liquid Al-13.58Si-11.59Fe-1.19Mn alloy

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ABSTRACT

To contribute to the understanding of the Al–Fe–Mn–Si system, an exhaustive analysis of the intermetallic phases formed during thermal analysis experiments and specific isothermal treatments was carried out. In this work, three different phases were identified: the extensively reported β and α phases and the τ_h phase. The τ_h phase has been reported for the ternary Al–Fe–Si system, but not for the quaternary Al–Fe–Mn–Si system. Nonetheless, the present results suggest that the formation of the τ_h phase upon solidification of the alloy can be stabilized in the quaternary Al–Fe–Mn–Si system, with respect to the monoclinic θ-Al_3Fe phase in the ternary Al–Fe–Si system, due to the Mn addition. The τ_h phase was unambiguously identified with the help of complementary techniques such as SEM/EDS, X-ray diffraction and Mössbauer spectroscopy.

1. Introduction

Commercial Al–Si alloys always contain Fe impurities; this undesirable contamination comes either from the use of steel tools for melting and casting or from the use of scrap as the initial material. The Fe impurities have a detrimental effect on the mechanical properties and extrusion characteristics of these alloys. This is attributed to the formation of different Fe-containing intermetallic phases [1–4]. Around 17 different intermetallic phases with a general composition of Al–Fe and Al–Fe–Si have been summarized in the literature in Ref. [5].

The ternary Al–Fe–Si system has been widely studied; experimental data pertaining to its intermetallic phases and their relationships in the corresponding phase diagram have been summarized in well-documented review papers in Ref. [2,6–8]. However, for the improvement of the available thermodynamic databases, the study of this and other alloy systems is gaining importance in basic and applied research. Ravi and Wolverton [9] have made a critical comparison of two commercially available Al-alloy thermodynamic databases: Thermotech and Computherm. As a main result, they have suggested carrying out future improvements in both of these databases, based on their observation of differences in the intermetallic compounds involving Fe, Mn, Cr, and Zn. It has also been reported that the type of intermetallic phases formed strongly depends on the cooling conditions employed [10], the alloy's Fe and Mn content [11], and the presence of other alloying additions [12].

In the last few decades, investigations of the solid-state phase equilibrium [13,14] and the solid–liquid phase equilibrium at 727 °C [15] have been reported for the Al–Fe–Si system. Additionally, using a specific alloying composition in the Al–Fe–Si system, the hexagonal $Al_4Fe_{1.7}Si$ intermetallic phase has been found [16]; however, this phase has not been reported by other researchers [2,13–15]. The Al–Fe–Si phase diagram has also been evaluated by thermodynamic calculations [14,17]. Moreover, Davignon et al. [18] have investigated an isothermal section at 550 °C of the quaternary Al–-Fe–Mn–Si system. They have studied Al-alloys with both low and high Fe and Mn content to enhance the available data concerning the type, crystal structure, and chemical composition of the AlFeMnSi intermetallic phases.

It has been reported [3] that Mn is widely used as an alloying addition for Al–Fe–Si alloys to form the α phase with Chinese script

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Table 1
Onset reaction temperature, T_p, and EDS data for extracted intermetallic phases (wt%).

Temperature (°C)		AlFeMnSi intermetallic phase	Composition[a]				Formula	Lattice parameter (Å)
T_p	T_s		Al	Si	Fe	Mn		
601	586	β	63.43	14.85	19.26	2.44	$Al_{53}Fe_{7.8}MnSi_{11.9}$	–
723	708	α_h	53.78	11.89	26.77	7.56	$Al_{14.48}Fe_{3.48}MnSi_{3.07}$	$a = 12.38$, $c = 26.184$
817	802	τ_h	52.15	8.39	28.96	10.51	$Al_{10.1}Fe_{2.71}MnSi_{1.56}$	$a = 7.5019$, $c = 7.5868$

[a] Average of eight SEM/EDS analyses of the extracted particles for each alloy.

rather than plate-like phases, referred as β and δ phases, which are more detrimental to the alloy's mechanical properties than the α phase. Additionally, because Mn atoms can replace the Fe atoms in the crystal structure of the intermetallic phases [4,19], the solidification sequence of the aluminides in Al–Fe–Si alloys can be modified by the addition of Mn.

Mössbauer spectroscopy is a powerful technique that can be used to explain the formation of a specific intermetallic phase. Many authors have analyzed aluminum alloys with different amounts of Fe impurities. At Fe concentrations higher than the solid solution limit (~0.052 wt% Fe at 655 °C), many different phases have been elucidated using this technique, ranging from Fe clusters to a large variety of intermetallic phases including θ-$Al_{13}Fe_4$, Al_6Fe, β-AlFeSi, and γ-AlFeSi, as well as cubic (α_c) and hexagonal (α_h) α-AlFeSi phases [1,20–23].

The determination of the solid-state and solid–liquid equilibria in the Al–Fe–Mn–Si system will allow enhancement of the available data concerning the type, crystal structure, and chemical composition of the AlFeMnSi intermetallic phases. These aspects are needed to understand the solidification sequence, allowing control of the microstructure of the castings and hence improvement of the mechanical properties of the Al-alloys.

2. Experimental details

2.1. Materials

Hereinafter, all compositions are given in weight percentage unless otherwise specified. The alloy was prepared starting from Al ingots of commercial purity (99.77%); the main impurities present in these ingots were: 0.13% Fe, 0.05% Si, and 0.05% others. The added alloying elements were: Fe (99.98%), Mn (99.95%), and Si of commercial grade. The alloy's chemical composition, determined by inductively coupled plasma emission spectroscopy (ICP) with an Iris Intrepid spectrometer, was: 13.58% Si, 11.59% Fe, 1.19% Mn, and 73.59% Al; the main impurities present in this alloy were: 0.02% Zn, 0.014% Cu, 0.008% Ti, and 0.008% others.

2.2. Thermal analysis

To determine the solidification sequence and the onset reaction temperature, T_p, for the intermetallic phase formation (see Table 1), thermal analysis curves were obtained by employing an experimental device similar to the one used by Bäckerud et al. [10]. First, the alloy was melted and heated to 800 °C under ambient air conditions in a gas furnace. Subsequently, the melt was poured into a graphite cup having a wall thickness of 6 mm, which was preheated to 900 °C in an electric furnace; to achieve thermal equilibrium conditions, the melt was kept at 900 °C for 2 min in the graphite cup inside the electrical furnace. Finally, the sample was cooled down to room temperature at a rate of 1 °C/s.

The alloy's temperature was measured with two K-type thermocouples placed on the lid of the graphite cup, as shown in Fig. 1. The precision of the thermocouples was assessed by measuring the melting temperature of pure aluminum (659.9 °C). The temperature–time data were recorded using a 16-bit resolution DaqTemp 14A card linked to a computer.

2.3. Isothermal treatment

Based on the values of T_p obtained for the formed intermetallics (see Table 1), the alloy was subjected to an isothermal heat treatment at a temperature set ~15 °C below T_p, which hereinafter will be denominated as T_s. The purpose of this isothermal treatment was to allow the intermetallic particles to grow and settle down toward the bottom of the sample. Each sample (19 mm in diameter and 25 mm in height) was placed inside a graphite crucible having a wall thickness of 3 mm. A set of five crucibles, shown in Fig. 1, was heated in an electric furnace under ambient environment at 900 °C for 30 min, subsequently cooled down to T_s at 1 °C/min, and then held at T_s for 15 h. The furnace temperature was controlled with a K-type thermocouple placed inside one of the samples. At the end of the isothermal heat treatment, the samples were quenched in a water bath at ~20 °C to avoid the formation of additional intermetallic phases at lower temperatures. Due to the small size of the

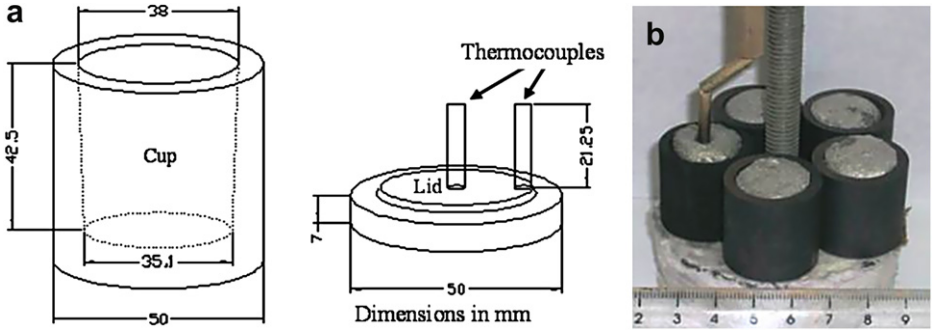

Fig. 1. a) Schematic illustration of the graphite cup and lid used for the thermal analysis experiments, and b) experimental setup used to carry out the isothermal treatment experiments.