



Evolution of Fe-rich phases in Mg melt and a novel method for separating Al and Fe from Al–Si–Fe alloys



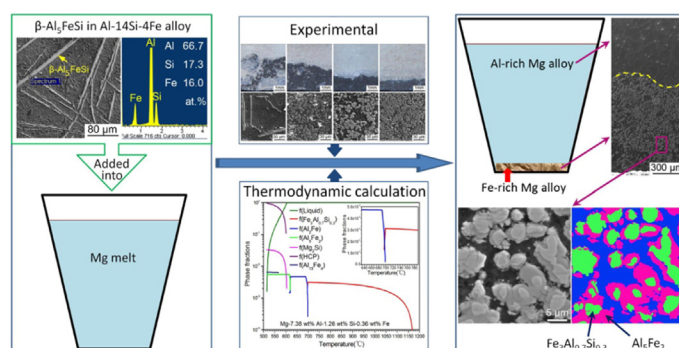
Tong Gao *, Zengqiang Li, Yaoxian Zhang, Jingyu Qin, Xiangfa Liu *

Key Laboratory for Liquid–Solid Structural Evolution and Processing of Materials, Ministry of Education, Shandong University, 17923 Jingshi Road, Jinan 250061, PR China

HIGHLIGHTS

- A novel method aiming at recycling Fe-rich Al–Si alloys by introducing them into Mg melt to separate Al and Fe is proposed.
- β -Al₅SiFe phase in Al–Si–Fe alloys can be designed evolving to Al-poor Fe₃Al_{0.7}Si_{0.3} by controlling the melting process.
- A separation layer rich of Fe can form at bottom while Al is released to the upper melt.

GRAPHICAL ABSTRACT



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ABSTRACT

The recycling of high Fe-contained Al–Si alloys is a green industry and how to separate Al and Fe is the key point therein. An efficient technique using Mg melt to separate Al and Fe is proposed in this study. By introducing Al–14 wt% Si–4 wt% Fe alloy into Mg melt and adjusting melting, holding and cooling parameters, the evolution behavior and separation performance of Fe-rich intermetallics were investigated. A separation layer with Fe-rich particles was obtained at the bottom of the cooled ingot. A meta-stable phase Fe₃Al_{0.7}Si_{0.3} was detected, which only exists above 699 °C confirmed by a thermodynamic calculation. Making β -Al₅SiFe phase from the initial Al–14 wt% Si–4 wt% Fe alloy transform to Fe₃Al_{0.7}Si_{0.3} in Mg melt is beneficial to achieve higher separation efficiency since the Fe₃Al_{0.7}Si_{0.3} phase is Al-poor, indicating that quantities of Al are released to the Mg matrix. Holding at 750 °C for 30 min followed by a further holding at 650 °C for 30 min is regarded most proper in this study. This work may be referred by relative study on the evolution of intermetallics by introducing one matrix alloy into another melt.

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1. Introduction

The rapid development of automotive, aeronautic, electronic and aerospace technologies in modern society strongly depends on the consumption of energy and materials [1]. In recent years, the concept of sustainable development and energy saving has been widely agreed

by humans [2]. Aluminum, as a recyclable material, has a significant influence on the national economic development [3–5]. Compared with primary electrolytic Al, the recycling and reusing of scrap Al is energy saving. However, Fe is one of the most common impurities in Al alloys as it may occur from the low-purity alloying materials, e.g. commercial Al and Si, and may also be introduced through the contamination of unprotected ferrous crucible, tools or equipment [6–8].

The repeating and circular melting of Al alloys results in the continuous accumulation of Fe, in some cases its content can be even higher

* Corresponding authors.

E-mail addresses: tgao@sdu.edu.cn (T. Gao), xfliu@sdu.edu.cn (X. Liu).

than 3 wt%. For instance, in die casting industries Fe is helpful to facilitate separation of the casting parts from the moulds, thus improving the surface quality of the products [9,10]. Therefore, it is not unusual for recommending Fe content to range from 0.8 wt% to 1.1 wt% in these alloys. Several rounds of re-melting the die casting products will lead to a quite high content of Fe in Al alloys. Another example is that for producing Al–Si based pistons, wear-resistant insert rings are usually applied in the piston head [11]. Besides, an infiltration procedure by putting the cast iron rings into Al melt is always essential before casting. Therefore, both the re-melting of pistons and the reuse of Al melt for infiltrating rings can also result in the enrichment of Fe element.

Since the solubility of Fe in Al is relatively low, the majority of Fe exists in the formation of intermetallic compounds [12]. Several Fe-rich phases have been reported in Al–Si alloys, e.g. needlelike β -Al₅SiFe (or β -Al₉Si₂Fe₂) or Chinese script morphological α -Al₁₅Fe₃Si₂ will precipitate depending on the composition and cooling conditions of the alloys [13–15]. It is widely known that the brittle β -Al₅SiFe phase has harmful effect on the mechanical properties of Al–Si alloys [16–18] due to its flake-like morphology with sharp interfaces acting as locations of high stress concentrations. Abouei et al. [19] pointed out that the formation of coarse β -Al₅SiFe particles initiate micro cracks and reduce the wear resistance of an Al–12 wt% Si alloy. Ma et al. [20] found that β -Al₅SiFe particles seriously affect the fracture behavior of Al–Si–Cu cast alloys.

It is of great significance to eliminate the harmful effect of β -Al₅SiFe on Al–Si alloys for industrial productions. One theory is to modify the morphology of β -Al₅SiFe phase through overheating the melt, increasing cooling rate or adding neutralized elements. Alizadeh et al. [21] reported that increasing cooling rate can refine the size and alter the morphology of Fe-rich intermetallics. Adding neutralized elements such as Be, Mn, Cr and V to modify β -Al₅SiFe phase is the most commonly used method [22–24]. For instance, a proper addition of Mn into Al–Si–Fe alloys leads to the precipitation of primary α -Al₁₅(Fe,Mn)₃Si₂ with a body centered cubic structure [25]. However, the method of overheating the melt will cause energy consumption, whereas increasing cooling rate is sometimes hard to achieve due to the limitation of industrial procedure, and the method by adding neutralized elements will lead to secondary pollution.

Another theory is to aggregate and remove β -Al₅SiFe phase. The usually applied methods contain Centrifugal Separation Method, Gravitational Sedimentation Method and Electromagnetic Separation Method. Basak et al. [26] reported that it is possible to eliminate β -Al₅SiFe phase by gravitational segregation after suitable heat treatment. Nafisia et al. [27] found that electromagnetic stirring can efficiently lead to the refinement of Fe-intermetallics. However, these methods also have nonnegligible disadvantages, e.g. the Centrifugal Separation Method and Electromagnetic Separation Method are complex and need ancillary equipment.

In fact, at present the most commonly used method in industries to reuse scrap Al–Si–Fe alloys is using primary Al melt for dilution. This method has quite low efficiency and only <30 wt% scrap ingots can be reused for each melting work. It is believed that the development of a proper way to recycle scrap Al alloys is of great significance.

In this study, a novel method by using Mg melt to separate Al and Fe was developed. By introducing Al–Si–Fe ingots into Mg melt and applying melt control technologies, Fe-rich intermetallics were found sinking to the bottom of the melt and evolving from Al-rich β -Al₅SiFe to Al-poor Fe₃Al_{0.7}Si_{0.3} phase. Through this procedure, Al can be released to the upper of the melt and thus can be recycled. Combining experimental results with a thermodynamic calculation, the influence of melting parameters on the separation efficiency of this method and the evolution mechanisms of Fe-rich phases were discussed. The obtained results in this paper provide an insight of the key scientific and technological points of this novel method. Also, they are expected to make contribution for the application of this method in industries.

2. Experimental

2.1. Materials and sample preparing procedure

The materials used in this paper include commercial purity Mg (99.8 wt%), commercial purity Al (99.8 wt%), commercial purity crystalline Si (99.9 wt%) and commercial purity Fe (99.9 wt%).

The Fe-rich alloy used in this paper is ternary Al–14 wt% Si–4 wt% Fe alloy, prepared according to following procedures: first, Al and Si were melted in a clean clay-bonded graphite crucible by high frequency induction furnace to 1100 °C then Fe was added in the melt. After holding for 10 min, the melt was poured into an iron chill mould.

For the separation experiments, i.e. to check the separation performance of Al and Fe elements by using Mg melt, the experiments were carried as follows: first, 80 g Mg ingot was melted under the protection of 1% RJ-2 flux (40–50 wt% MgCl₂, 35–45 wt% KCl, 5–8 wt% BaCl₂, 5–8 wt% CaF₂, 5–8 wt% NaCl&CaCl₂) in a BN ceramic crucible whose shape and dimensions are shown in Fig. 1, by using a resistance furnace to a designed temperature. The Al–14 wt% Si–4 wt% Fe alloy was added in the melt with the amount of 10 wt%. After that, the melt was regularly stirred for 15 min by a Φ 5 mm graphite rod to make the alloy totally melted. Then the melt was held for different holding time, i.e. 15 min, 30 min and 45 min in this study. Finally the crucible was cooled in the furnace or transferred into the air and cooled to room temperature. A deposition layer was obtained at the bottom of the crucible, as shown in Fig. 1.

2.2. Microstructure analysis

Metallographic specimens were all cut from the same position (the bottom center area, as marked by 'Sample' in Fig. 1) of the cooled ingots, then mechanically ground and polished by using MgO turbid liquid in standard routines. The microstructure observations were carried out by a Leica DM2700 High-Scope Video Microscope (HSVM) and field emission scanning electron microscopy (FESEM), i.e. a Hitachi SU-70 scanning electron microscope operated at 15 kV and linked with an energy dispersive X-ray spectrometry (EDX) attachment. The phase identification of the samples was detected by X-ray diffraction (XRD) with a D/max-RB diffractometer (Rigaku, Tokyo, Japan) using Cu K α radiation at 40 kV and 100 mA.

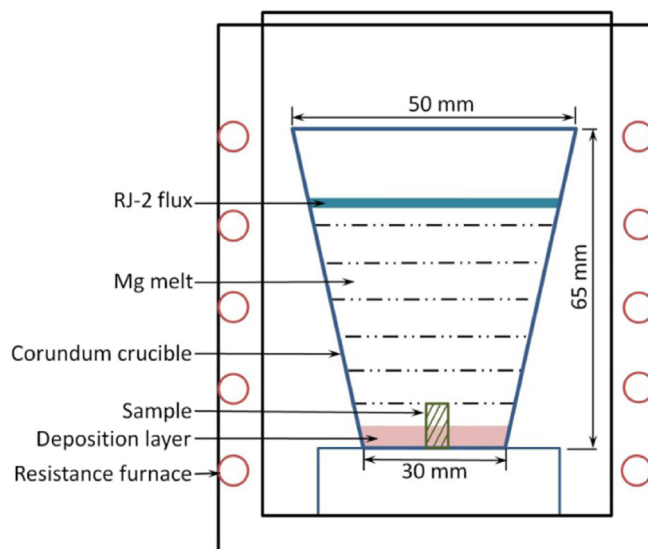


Fig. 1. Schematic diagram of the separation experiment setup.

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