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The synthesis of novel powder master alloys for the modification of primary and eutectic silicon crystals

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ABSTRACT

The effect of the high-energy synthesis of powder mixtures of aluminium granules, ferrophosphorous (Fe_2P) and strontium salt ($SrCO_3$) in a planetary mill was investigated on the structure of the resulting materials. Al–8Fe–2P, Al–10Sr and Al–10Sr–1P master alloys were synthesised, and the efficiency of these new alloys was demonstrated in the modification of an eutectic silicon in Al–12Si alloys and the complex modification of Al–18Si alloys.

1. Introduction

Currently, master alloys for the production of Al–Si alloys are prepared by melting aluminium and copper with strontium and phosphorus, respectively. In its pure form, strontium and phosphorus are unstable and pose significant risks to the environment. Moreover, the use of phosphor copper-based master alloys is inefficient due to the long and incomplete assimilation of aluminium melts.

Cu–P master alloys are often employed for the modification of hypereutectic Al–Si alloys [1–4]; however, the use of phosphorous Al-based master alloys [5–9] has recently increased. The production of phosphorous casting alloys is difficult and harmful to the environment because pure phosphorus is volatile and unstable. Nevertheless, unlike aluminium [5], the dissolution time of copper-based master alloys is sufficiently long, and more than 0.05% phosphorus can be introduced [1,2]. As previously demonstrated, the most effective eutectic silicon modifier is Sr [10,11]. Strontium is usually introduced into Al–Si alloys using Al–10Sr master alloys, which are obtained by melting aluminium and pure strontium. In the pure state, strontium is expensive and actively interacts with moisture. Cast strontium alloys contain a large amount of aluminide strontium coarse inclusions, which reduces its effectiveness [12]. Usually, modifying alloys are made from two separate master alloys, but, in a previous study [13], complex master alloys were obtained by melting two double alloys. The application of this type of multi-step synthetic method is time-consuming; thus, simple methods that do not require additional costs for the production of integrated master alloys from sustainable and environmentally friendly raw materials must be developed.

The following requirements are essential characteristics of master alloys: low melting point, maximum dispersion of intermetallic particles and uniform distribution within the volume and lack of large inclusions. The powder method of producing aluminium-based master alloys satisfies these basic requirements of master alloys. The aim of the present work was to synthesise powdered master alloys from simple stable chemical compounds of phosphorus and strontium (Fe_2P and $SrCO_3$) in a high planetary mill and to use the resulting materials for the modification of primary and eutectic silicon in Al–12Si and Al–18Si alloys.

2. Material and methods

Al–8Fe–2P, Al–10Sr and Al–10Sr–1P master alloys were prepared from Al (A99), ferrophosphorous (Fe_2P) and strontium salt ($SrCO_3$) powders in a 4-reel Retsch PM 400 centrifugal planetary mill. Tablets with a diameter of 25 mm and a height of 15 mm were pressed with a force of 1500 MPa from the powder mixtures. For structural analysis, microsections were produced using grinding and polishing installations (Struers Labopol-5 and Metkon, respectively).

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A413.2 (Al–12% Si) alloys and Si (99.7%) were used to melt Al–18Si alloys. Melting was carried out in graphite crucibles placed in a Nabertherm resistance furnace. The alloys were cast into $15 \times 30 \times 150\text{-mm}^3$ graphite moulds preheated to 250 °C.

A Neophot – 30 light microscope (LM) and an image analysis system consisting of an Axiovert 200MMAT and Axiovision 4.5 software were used for the qualitative and quantitative analysis of the microstructure. The microstructure and phase composition of the master alloys and modified alloys were examined with a TESCAN VEGA 3LMH scanning electron microscope (SEM) equipped with an energy dispersive X-ray (EDX) analyser XMAX-80. X-ray diffraction analysis was performed on a Bruker D8 Advance diffractometer using monochromatic radiation CuKα.

3. Results and discussion

Al–8Fe–2P, Al–10Sr and Al–10Sr–1P powder master alloys were synthesised in the present study. The optimal operating parameters of the high-energy planetary mill that provided minimal raw material losses and uniform dispersion microstructures were identified. The effect of the synthetic parameters, such as the rotational speed of the mill (ν), mass ratio of the grinding media to the material (q) and processing time (τ), on the phase transformation and dispersity of the alloys were studied. As q increased from 7 to 10, the synthesis time (τ) was cut in half, uniformly distributed inclusions of phosphide phases with an average size of 2 μm were obtained (Fig. 1a) and mixtures of aluminium and strontium salts (Fig. 1b, c) were produced. Fig. 1 shows the microstructure of the master alloys obtained under the optimal conditions. The phase composition of the material was investigated via X-ray analysis, and the results showed that the redistribution of phosphorus and the concomitant formation of a new aluminium phase (Al, Fe, P) occurred during the interfacial synthesis. Processing in the planetary mill contributes to the accumulation of a high amount of energy. This energy is sufficient for the formation of new phases, and even alloys. New ternary phase is most probably metastable, since there is no data on the existence of such a phase in the ternary Al–Fe–P system [14,15]. The maxima of intensity was detected in master alloy Al–8Fe–2P (Fig. 1d), which annot be identified by X-ray database. The formation of metastable phosphide phases based on aluminium in the master alloys will significantly expedite the assimilation of the melt during the modification process. Salt $SrCO_3$ prevents aluminium and

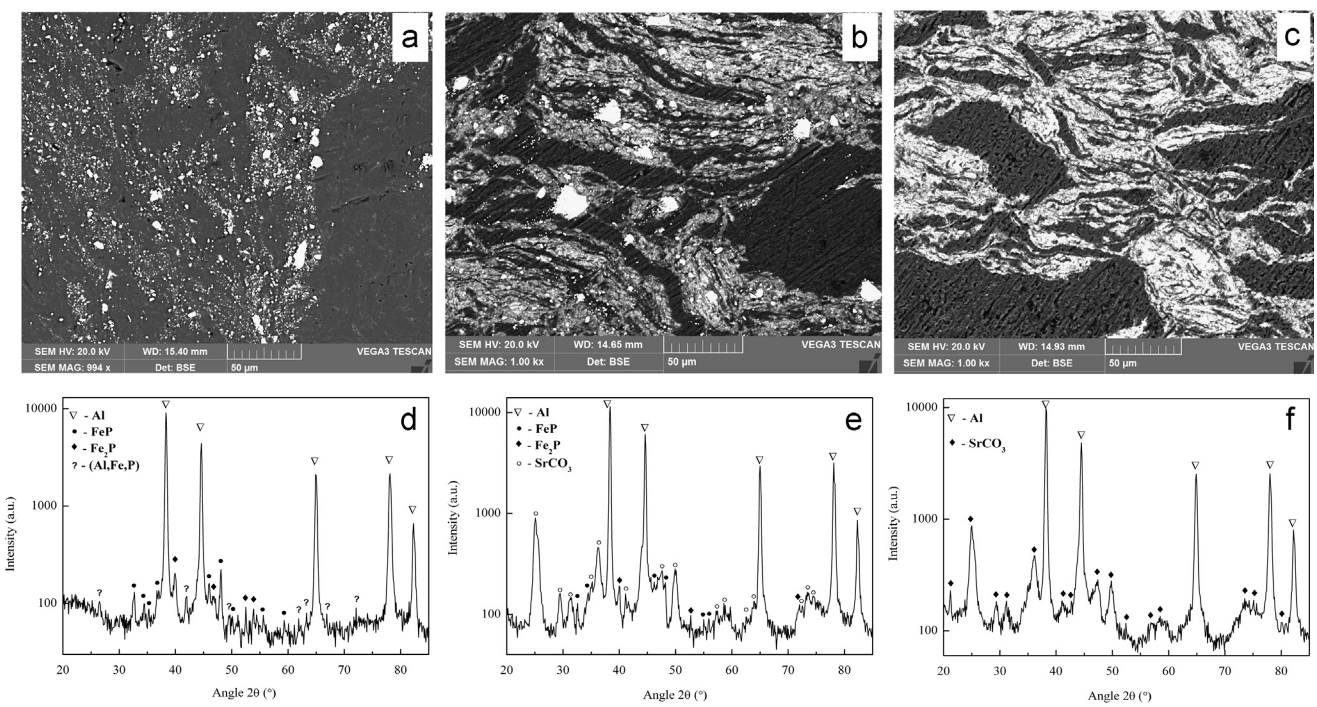

Fig. 1. Microstructure (a–c) and XRD (d–f) of master alloys: (a) Al–8Fe–2P, (b) Al–10Sr, and (c) Al–10Sr–1P (SEM).

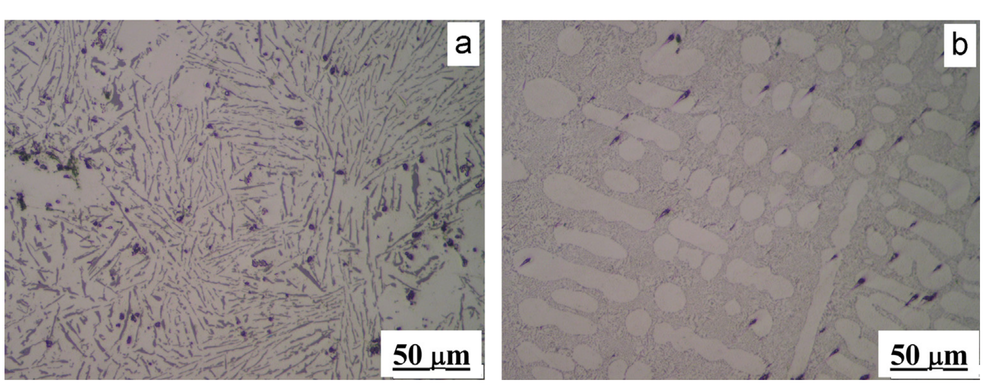

Fig. 2. Microstructure of the alloy Al–12Si unmodified (a) and modified with master alloy Al–10Sr (b) states (LM).