Journal of Alloys and Compounds 739 (2018) 30–34

Contents lists available at ScienceDirect

Journal of Alloys and Compounds

journal homepage: http://www.elsevier.com/locate/jalcom

Formation of Si nanoparticle in Al matrix for Al-7wt.%Si alloy during complex shear flow casting

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ARTICLE INFO

Article history:
Received 30 October 2017
Received in revised form
21 December 2017
Accepted 22 December 2017
Available online 26 December 2017

Keywords:
Al-Si alloy
Complex shear flow casting
Casting
Nanoparticle

ABSTRACT

Al-7wt.%Si alloy was produced by applying complex shearing to the liquid melt during graphite mould casting. Microstructure was analyzed with optical microscope (OM), scanning electron microscope (SEM) and high-resolution transmission electron microscope (HRTEM). Results show that Si nanoparticles, which can only be conventionally obtained by melt spinning technique for cast Al-Si alloy, are embedded in Al matrix. There is a cubic to cubic orientation relationship (OR) and a semi-coherent interface between Si nanoparticle and Al matrix. Formation mechanism of Si nanoparticles has been discussed.

1. Introduction

Al-Si alloys are widely used in many applications by means of their low-density, good castability, and excellent corrosion resistance [1]. Distributions, morphologies and sizes of Si phase are different by using various casting techniques and processes. For conventional casting method with low cooling rate less than 10 K/s (sand, graphite and permanent moulds) [2–5], lamellar Si with length of several tens of microns are distributed on the boundaries between α-Al grains, provided that no treatment is posed on the melt [3–5]. H.R. Kotadia and A. Das studied ultrasonicated Al-Si alloy, and found that Si phase which is distributed on the boundaries between α-Al grains shows compact globular morphology with size of 7–8 μm and long plate morphology with size of several tens of microns [3,4]. S. Nafisi et al. studied the electromagnetic stirring (EMS)-stirred Al-Si alloy, and found that EMS decreases average length and width of boundary-distributed Si flakes with several microns by comparing with no EMS-stirred samples [5]. Noticeably, by using melt spinning technique which has very high cooling rate, Si particles with size at nano or submicron scales can be obtained in the α-Al matrix as well as on the boundaries between α-Al grains [6–8], indicating that cooling rate has an effect on size and distribution of Si phase. C.R. Ho et al. [6] revealed that even for high purity melt-spun Al-3wt.%Si alloy, trace amount of phosphorus (<2 ppm) can forms AlP particles at the primary Al dendrite/eutectic liquid droplet interface, and on cooling further to below the equilibrium Al-Si eutectic temperature, AlP heterogeneously nucleate Si from the eutectic liquid droplets. In the present work, we report that by imposing complex shear flow on the Al-7wt.%Si alloy melt under graphite mould casting, Si nanoparticles in Al matrix can unprecedently form at relatively low cooling rate. Formation mechanism of Si particles in Al matrix is proposed which are expected to be significant for Al-Si alloy design.

2. Experimental procedure

Al-7wt.%Si alloy was prepared using an induction furnace and a complex shear flow casting (CSFC) treatment device. CSFC treatment device is composed of a centrifugal disk and a centrifugal mould, as schematically shown in Fig. 1. Liquid metal prepared by the induction furnace was poured into the centrifugal mould, then the centrifugal disk and centrifugal mould swirled until the melting metal is completely solidified. A detailed description of the CSFC can be found elsewhere [9]. Here, commercially pure Al and Al-20wt%Si alloy were first introduced into a graphite crucible

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https://doi.org/10.1016/j.jallcom.2017.12.243
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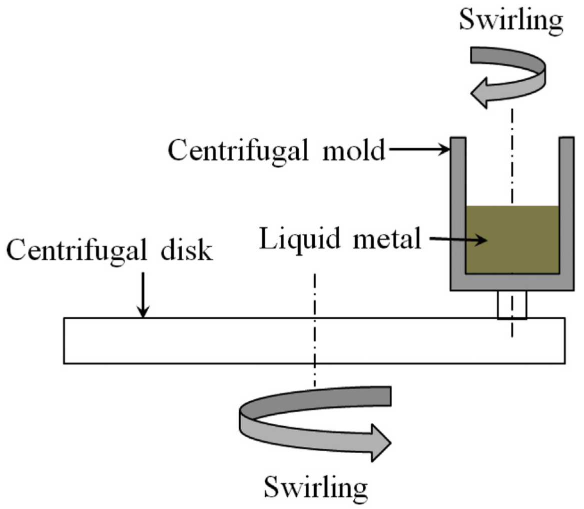

Fig. 1. Schematic illustration of CSFC treatment device.

located in the induction furnace then heated up to 750 °C to melt and superheat. After held for 30min and degassed, about 2 kg of the melt was poured at 750 °C into the centrifugal graphite mould. Mould with size of 70 mm × 80 mm × 110 mm and wall thickness of 10 mm was preheated to 250 °C. Then the melt was CSFC treated immediately after pouring. During CSFC process, centrifugal disk swirled at 1.25 Hz along clockwise direction, and the centrifugal mould which is on the edge of the centrifugal disk swirled at 7.5 Hz along counterclockwise direction. For comparison, conventionally solidified Al-7wt.%Si alloy, which had not been treated by CSFC, was fabricated using the same procedure.

Microstructure of Al-7wt.%Si alloy was analyzed by optical microscope (OM) and scanning electron microscope (SEM) equipped with energy dispersive X-ray spectrometer (EDS). Metallographic samples were polished using standard metallographic techniques. Microstructures were observed with electro-etched in 5 vol% perchloric acid and 95 vol% alcohol etching reagent at 30 V for 10 s. Samples used for observation of entrained Al-Si droplets were prepared by standard mechanical polishing and then etched with Keller solution (95 vol% H_2O, 2.5 vol% HNO_3, 1.5 vol% HCl and 1 vol% HF) for 10 s. Morphologies of nanoparticles and the interface between the nanoparticle and matrix were analyzed by transmission electron microscope (TEM) and high-resolution TEM (HRTEM) on a JEM-2010 TEM equipped with energy dispersive X-ray spectrometer (EDS) and operating at 200 kV. Thin foils for TEM were prepared as follows: 3 mm diameter discs were punched from the samples, mechanically ground down to 40 μm thicknesses and then thinned by precision ion polishing system.

3. Results and discussion

3.1. Characterization of nanoparticles in CSFC treated alloy

Typical OM microstructure of conventionally solidified and CSFC treated Al-7wt.%Si alloys is composed of α-Al phase and Si phase distributed on the boundary of α-Al grains, as shown in Fig. 2a and b. However, α-Al grains are dendrite structure for conventionally solidified Al-7wt.%Si alloy, and are small globular shape in CSFC treated Al-7wt.%Si alloy, respectively. Lamellar coarse Si grain in CSFC treated Al-7wt.%Si alloy is finer than that in conventionally solidified Al-7wt.%Si alloy. For CSFC treated Al-7wt.%Si alloy, bright field (BF)-TEM image shows that dispersed nanoparticles are embedded in primary α-Al (Fig. 2c). Corresponding dark field (DF)-TEM image shows that nanoparticles are homogeneously distributed in the Al matrix (Fig. 2d). Furthermore, some nanoparticles can be observed in eutectic Al (Fig. 2e). By magnifying view of white dashed box marked in Fig. 2e, distribution of nanoparticles in eutectic Al can be clearly seen (inset of Fig. 2e). In contrast, there is no strong evidence of the existence of nanoscaled particles in conventionally solidified Al-7wt.%Si alloy, only dislocations are observed (Fig. 2f).

Fig. 3a shows the enlarged TEM image of microstructure in CSFC treated Al-7wt.%Si alloy. Matrix is indexed to be Al from EDS result in the bottom inset of Fig. 3a. Nanoparticle is indexed to be Si from that in the upright inset of Fig. 3a. From HRTEM image shown in Fig. 3b, one Si particle can be seen. Diffraction pattern and indexing analysis in the upright inset of Fig. 3b indicate that Si particle maintains a perfect cubic to cubic orientation relationship (OR) with α-Al matrix, i.e., $\{111\}_{Si}\langle 011\rangle_{Si}//\{111\}_{\alpha\text{-}Al}\langle 011\rangle_{\alpha\text{-}Al}$. Another Si particle in 26 nm size is shown in Fig. 3c. By magnifying square part marked with D in Fig. 3c. As shown in Fig. 3d, it can be seen that dislocations emerge inside the particle, and the atomic ratio of Si to Al at Si/Al interface is 3:4, which is consistent with the atomic ratio of semicoherent interface [7].

3.2. Formation mechanism of Si nanoparticles in CSFC treated alloy

From equilibrium phase diagram, there is no solid-state Si phase in the melt during primary α-Al growth for hypoeutectic Al-7wt.%Si alloy. Singh et al. [10] studied structure of liquid hypoeutectic Al-Si alloys by using the Kumar-Samarin technique, and found that there are Al-Si clusters in hypoeutectic Al-Si alloy melt, which size are about a few nanometers. Gui et al. [11] studied influence of melt temperature on Al-Si atomic clusters for melt-spun Al-Si alloy. They revealed that homogeneous melt tends to become inhomogeneous with melt temperature decreasing, and Al-Si atomic clusters exist in the liquid hypoeutectic Al-Si alloy below 950 °C. Srirangam et al. [12] pointed out that the Si atomic clusters form as the hypoeutectic Al-Si alloy melt temperature decreases according to high-energy X-ray diffraction analysis. These suggest that Al-Si and Si atomic clusters may be the source of Si nanoparticles in the CSFC treated Al-7wt.%Si alloy.

Fig. 4a shows many large entrained Al-Si atomic clusters, namely Al-Si droplets, (marked with white arrows) inside α-Al for conventionally solidified Al-7wt.%Si alloy. By magnifying view of one droplet in Fig. 4a, Al-Si droplet is of spherical shape with size of about 15 μm, as shown in Fig. 4b. Numerous particles with size of hundreds of nanometers to several microns can be observed inside droplet. Elemental maps in Fig. 4c confirm that this droplet is composed of Al, Si and Fe. Particles inside droplet are all Si-rich. As is well known that Fe is a common element for commercially pure Al alloy, and iron-containing intermetallic phase can be considered as potential nucleation site for Si [12,13]. During solidification of conventionally solidified Al-7wt.%Si alloy, as schematically shown in Fig. 5a, b, c and d, large sized Al-Si droplets (clusters) are captured by growing α-Al (Fig. 5b). When melt temperature drops to eutectic point (Fig. 5c), Si cluster forms in the remained liquid and then changes into nucleus for solid Si. Solid Si has enough space to growth, and thus forms lamellar coarse Si distributed on the boundary of primary α-Al grain. Meanwhile, Al-Si droplet captured by α-Al has small volume but large contact area with the existing solid resulting in high cooling rate and limited growth space available for Si phase [3,4], leading to formation of numerous micron- or submicron-sized Si particles inside the droplet, as schematically shown in Fig. 5d. In contrast, for CSFC process, CSFC can create shear flow and asymmetry flow in alloy melt. It is reported that shear flow or asymmetry flow can breakup droplets from several tens of micrometers to submicron size [14,15].