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Full Length Article

Interfacial microstructure and nucleating mechanism of melt-spun CeB_6/Al composite inoculant

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ABSTRACT

The microstructure of the CeB_6/Al interface in an Al-Ce-B alloy prepared by melt-spinning was investigated by scanning electron microscope (SEM) and transmission electron microscopy (TEM). The *in situ* synthetic CeB_6 particle in Al melt generally shows cubic morphology in which the naked surface is (220) plane. After adding in Al melt during inoculation, a well-bonded interface was formed between the CeB_6 particle and the Al crystalline matrix. The experimental observation on the degree of mismatch between Al (220) plane and CeB_6 (220) plane was highly accorded with the theoretic calculated value. It was suggested that the surface of CeB_6 particle is the potential nucleant substrate of Al crystal. As a result, the grain size of aluminum was significantly decreased by addition of CeB_6/Al inoculant ribbons with a very small ratio of 0.3% in weight.

1. Introduction

With unique surface characteristics, refiner plays an important role of nucleant substrate for grain refinement of typical metals, which has become an essential strategy to promote columnar-to-equiaxed transition (CET) in the inoculated melt [1–3]. The microstructure of the interface between refiner particles and Al matrix has been the focus in the studies of inoculation mechanism [4,5]. For example, simulation studies have shown that the Al_3Ti film will firstly formed on the surface of TiB_2 and act as the neaclant substrate for the solidification of Al crystal [1]. However, this Al_3Ti film is very thin and cannot be experimentally observed yet. In addition, the detailed behavior of interfacial interaction of grain refiners and matrix during grain refining treatment has not been fully understood.

Cerium hexaboride (CeB_6) has high melting point, high hardness and good chemical stability, which has attracted widespread attentions in the field of materials engineering. For example, CeB_6 has been applied in scanning electron microscopy (SEM), transmission electron microscopy (TEM), various spectroscopic tools and other devices. Recently, LaB_6/CeB_6 particle has been used as inoculant to refine the grain size of aluminum alloys. It was found that CeB_6 has a good compatibility with pure aluminum, and the lattice constant of CeB_6 (0.4141 nm) is very close to that of Al (0.4049 nm) as well [6,7]. In other words, there is a small lattice misfit between them, making CeB_6 surface be potential nucleation substrate in Al melt. However, the interface microstructure of CeB_6/Al boundary lacks of experimental observation and the matching details of crystalline plane of CeB_6 and Al crystal should be carefully investigated.

In the present study, CeB_6 particles were prepared by fast solidification technique (namely melt-spinning) such that the CeB_6 phase can be *in situ* formed in the Al matrix, which is beneficial for observing the interface microstructure by TEM. The interfacial interaction between CeB_6 particles and Al matrix was also discussed.

2. Experimental procedures

Commercial pure Al ingot with purity of 99.99%, Al-20Ce (all compositions quoted in this work are in wt.% unless otherwise stated) and Al-3B alloy ingots were used as raw materials to prepare the Al-Ce-B inoculant. The mass ratio of Al-20Ce and Al-3B alloy is weighed as about 7:22 to achieve the atomic ratio of Ce:B = 1:6 for synthesis of CeB_6. The raw materials were firstly melted in a WK-II vacuum arc furnace under a Ti-gettered argon atmosphere

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to obtain the Al-4.8Ce-2.3B ($Al_{93}Ce_1B_6$ in at%) master alloy ingots. The Al-Ce-B ingots were then re-melted by arc-melting and blew on a high-speed rotary Mo roller with water-cooled under an argon atmosphere to produce CeB_6/Al ribbons. Phase identification of the specimens was carried out by Bruker D8 Discover X-ray diffraction (XRD) with Cu K_α radiation.

The interface microstructure of CeB_6/Al ribbon was examined by scanning electron microscopy (SEM, S4800) with energy dispersive spectrometer (EDS). To clearly know the crystal growth morphology of CeB_6 particle, the ribbons were etched in a 0.5% vol.% HF solution for macroscopic observation. Then the surface structure of CeB_6 particle was examined by SEM.

The melt-spinning ribbons were used as inoculants and added into aluminum melts at 720 °C, with different adding ratio of 0.1%, 0.2%, 0.3% and 0.4%, respectively. The treated melts were then poured into a steel mould with a cavity of 20 mm in diameter and 120 mm in height after smelting for 10 min. To investigate the microstructure, several specimens with 10 mm in height were cut from the middle part of the as-cast rod. Next, the specimens were polished and etched with Poulton's reagent (60%HCl + 30%HNO_3 + 5%HF + 5%H_2O) for microstructure examination by optical microscopy (OM, Olympus). The grain size analysis was carried out using the linear intercept method [8].

The interface of CeB_6 and Al formed in the inoculation processes was experimentally observed by high-resolution transmission electron microscopy (HRTEM, JEOL 2000FX). Fourier diffraction spectrum was obtained based on the HRTEM image of the nano-CeB_6 particle. The CeB_6/Al interface was also rebuilt by Materials Studio 6.0 for understanding the crystalline orientation relationship.

3. Results and discussion

In order to obtain the information of Al/CeB_6 interface, CeB_6 phase should directly joint Al crystal, without any other phases such as oxide, Al-B intermetallic, Al-Ce and Ce-B compound. In general, by in situ synthesis in liquid Al, the oxidation of CeB_6 and Al can be ruled out. Fig. 1 shows the typical XRD spectrum

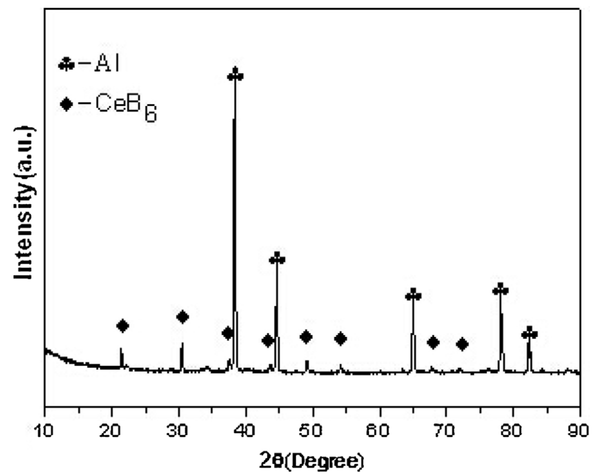

Fig. 1. (a) XRD spectrum of the melt spinning ribbons.

of the melt-spun ribbon sample, in which there are two groups of sharp diffraction peaks corresponding to Al phase and CeB_6 ceramic phase, respectively. It is suggested that the prepared ribbon actually is a dual-phase alloy as well as a CeB_6/Al composite. Except Al phase and CeB_6 ceramic phase, there is no other crystalline phases such as Al_2B or $Al_{11}Ce_3$ can be found. It is demonstrated that the obtained sample meets the requirement of the present study. Meanwhile, the intensity of Al peaks are much higher than that of CeB_6, indicating that the volume fraction of Al is larger than that of CeB_6. As it was known that CeB_6 phase is much brittle, such a phase composition that with small amount of CeB_6 phase is beneficial for the relative good toughness of the ribbon sample, which is beneficial for the preparation of TEM sample.

Fig. 2a shows the SEM image of the CeB_6/Al ribbons produced by melt spinning. It can be seen that there are many white pieces or particles distributing on the black matrix, homogeneously. According to the EDS results shown in Fig. 2b, the white phases can be speculated as cerium boride phase owing to a high Ce-B content. In combining consideration of the XRD result, the white phase

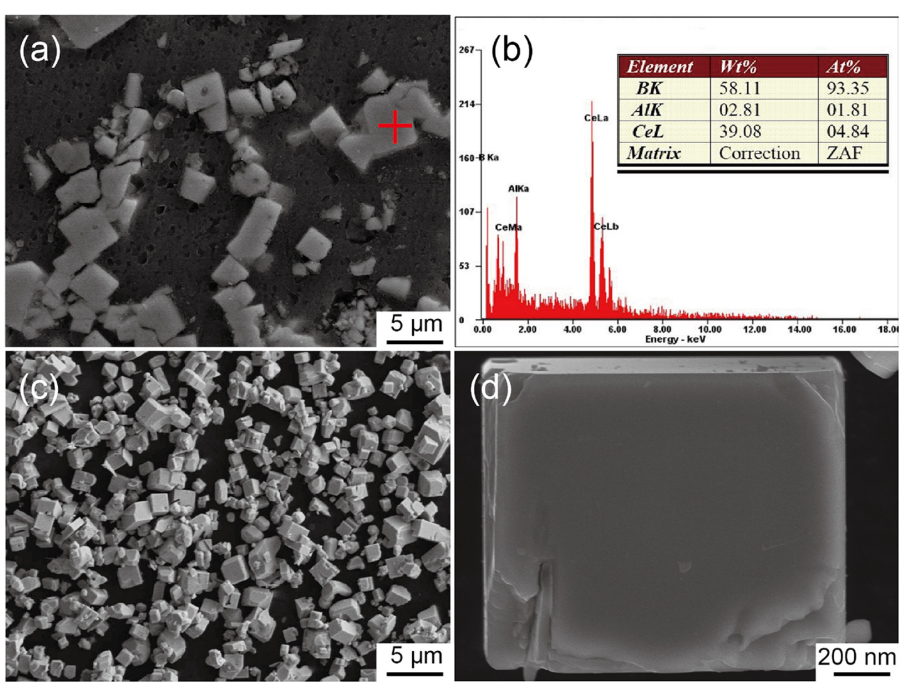

Fig. 2. (a) SEM image of CeB_6/Al ribbons, (b) the EDS spectrum of marked in (a), (c) and (d) are SEM image of the particles extracted from CeB_6/Al ribbons.

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