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In situ synthesis of FeSi–Al_2O_3 nanocomposite powder by mechanical alloying

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ABSTRACT

FeSi–Al_2O_3 nanocomposite powder was successfully synthesized via mechanical alloying of SiO_2 and Al powders in stainless steel cup and balls. Effects of the Al morphology, milling time and annealing temperature were investigated. Structural and morphological evolutions were monitored by X-ray diffraction (XRD) and scanning electron microscopy (SEM), respectively. Results show that the reduction of TiO_2 by spherical Al initiates after 30 h and completes after 45 h of milling. However, there is no reaction with flaky Al up to 45 h of milling. The mean grain size of 9 nm was obtained for FeSi at the end of milling. FeSi–Al_2O_3 nanocomposite powder was stable and maintained its nanocrystalline nature after annealing at 1000 °C. Combination of mechanical activation and heat treatment led to the formation of $Fe_{0.42}Si_{2.67}$ and mullite phases in the 10 h milled sample.

1. Introduction

Nanocrystalline materials are very attractive since the reduction of the grain size at the nanometric scale can improve their physical and mechanical properties. Due to their attractive high temperature properties, silicides of various metals have been the subject of numerous investigations .In particular, the nanocrystalline FeSi alloys have been the subject of various structural and magnetic studies from many researchers [1–3]. This silicide is not poisonous, highly resistant against oxidation and can be used in air without special precaution. Mechanical properties of this material can be improved by addition of a hard secondary phase such as Al_2O_3. Preparation in nanostructure is another mechanism to improve mechanical properties [4].

Two different methods have been developed in industry. The first method, based on the classical melting process, does not yield a homogeneous bulk product directly [5]. A very long period of annealing is required after the solidification. The second procedure uses powder metallurgical techniques to obtain the nanostructured powder of this material. There are several reports on the preparation of FeSi or $FeSi_2$ by powder metallurgical techniques [5–9], but there is no attempt to produce the FeSi–Al_2O_3 nanocomposite powder by this method.

FeSi–Al_2O_3 nanocomposite powder can be obtained easily by direct mixing of nano-alumina and iron silicide powders. But the resulting heterogeneous microstructure and high cost of the starting materials are two important set backs of this method. Alternatively, nano-metric FeSi–Al_2O_3 powders can be produced through high energy reactive milling of the mixtures of SiO_2, Al and Fe powders. Mechanical alloying (MA) is basically a dry and high energy ball milling process which has been used to synthesize alloys, oxide-dispersion-strengthened alloys, amorphous alloys and various intermetallics compounds [10,11].

The aim of this work is to synthesize FeSi–Al_2O_3 nanocomposite powder by ball milling of low cost starting materials. The effect of the milling time and annealing temperature were also investigated.

2. Experimental details

The MA experiments were performed in a planetary ball mill at nominal room temperature with a vial rotation speed (cup speed) of 500 rpm. Two kinds of Al, Flaky (MERK Co., 99.9 wt.%, <100 μm) and spherical (Fluka Co., 99.9 wt.%, <200 μm) were used (Fig. 1). Al and SiO_2 (Hamedan Pro., 99.9 wt.%, <200 μm) powders were mixed on the basis of following reaction:

$$3SiO_2 + 4Al \rightarrow 3Si + 2Al_2O_3 \quad (1)$$

The reduced Si will be reacted with the introduced Fe from the stainless steel cup and balls on the basis of following reaction:

$$Fe + Si \rightarrow FeSi \quad (2)$$

On the other hand the starting materials and iron impurity were mixed to give the FeSi–Al_2O_3 composite as follow [12]:

$$3SiO_2 + 4Al + 3Fe \rightarrow 3FeSi + 2Al_2O_3 \quad (\Delta H^{\circ}_{298} = -628.9\,kJ,\ \Delta G^{\circ}_{298} = -598.7\,kJ) \quad (3)$$

The ball to powder weight ratio (BPR) was 10:1. Seven balls with 20 mm and five balls with 10 mm diameter were used in the MA experiments. The mixture of the powders with the stainless steel balls was charged into a stainless steel cup (250 ml) under argon atmosphere. For preventing of excess agglomeration some process controlling agent (PCA) was used (1 wt.% stearic acid). Samples for analysis were removed by interrupting the mill at various intervals. Heat treatment of the milled powders was performed in a tube furnace in argon atmosphere (2 l min^{-1}). The heating rate was 10 °C/min and the holding time at the maximum temperature (600, 700 and 800 °C) was 2 h.

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