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Microstructure and magnetic properties of a two-phase alloy of α-Fe and metastable Fe_3B

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ABSTRACT

Applying the hypercooling technique, the metastable-phase Fe_3B, instead of the stable-phase Fe_2B, is formed directly in the bulk Fe–B eutectic alloy melt and can be further preserved at room temperature. Measurement of magnetic properties shows that, for the bulk Fe–B eutectic alloy with Fe_3B phase, the intrinsic coercivity and retentivity become smaller, and the saturation magnetization is larger, than the stable eutectic alloy (α-Fe/Fe_2B) and some Fe–B amorphous alloys.

1. Introduction

As one of the simplest amorphous metal–metalloid binary alloys and useful soft magnetic materials, Fe–B alloys have been investigated and applied widely. As compared to the stable α-Fe and Fe_2B soft magnetic phases, the metastable Fe_3B phase has been found to be a valuable soft magnetic phase, as a crystallized product of Fe–B binary and Fe–B-based amorphous alloys. Using a liquid quenching method, Khan [1], Antonione [2] and Coence [3] have reported Fe_3B as a crystallization product of Fe–B binary amorphous alloys in the composition range between 12 and 25 at% Palumbo and co-workers [4] also calculated the metastable-phase diagram of the Fe–B alloy system from a thermodynamic point of view and verified the results by thermal analysis experiments. The metastable-phase Fe_3B also occurs in the crystallization of $Fe_{60}Co_8Zr_{10}Mo_5W_2B_{15}$ amorphous alloy [5]. As a soft magnetic phase, Fe_3B combined with a hard magnetic phase such as $Nd_2Fe_{14}B$ or $(Pr,Tb)_2Fe_{14}Bn$ has been fabricated as exchange-coupled spring magnets due to the possibility of their application in magnetic field sensors and magnetic recording systems[6]. In addition, Fiorani et al. measured the magnetization versus magnetic field of an $Fe_{80}B_{20}$ amorphous alloy sample at 300 K and a sample annealed at 696 K. Annealing leads to an increase in the saturation magnetization (from 180.5 to 205 emu/g). They attribute this to the formation of the more magnetic-phase Fe_3B [7].

Until now, however, the metastable-phase Fe_3B has been investigated only as an intermediate product in amorphous alloys, and Fe–B alloys containing Fe_3B phase have been mainly limited to two-dimensional products. In practice, it is very hard to preserve as-prepared metastable Fe_3B at room temperature (RT) to study its potential applications. Applying the high undercooling technique, however, the metastable-phase Fe_3B can be obtained successfully and thus preserved to RT in bulk $Fe_{83}B_{17}$ eutectic alloy [8]. The influence of the metastable-phase Fe_3B on microstructure and magnetic properties of the Fe–B eutectic alloy will be discussed in the present paper.

2. Experimental

High-purity elemental Fe and B particles better than 99.8% purity were mixed to form approximately 5 g of $Fe_{83}B_{17}$ alloy. According to the equilibrium phase diagram [4], the composition $Fe_{83}B_{17}$ is the eutectic composition. Melting was carried out under

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argon gas in a high-frequency vacuum induction furnace. The sample was placed in a quartz crucible and covered with a molten B_2O_3 glass slag, which was melted with the help of a graphite heater. After evacuating to a pressure of 10^{-4} Pa the evacuated chamber was backfilled with high-purity Ar to nearly (−0.02 MPa) for the gas release in the alloy melt. Then the alloy was slowly heated up to a temperature of 100 K above its eutectic temperature and held for 3 min. Several heating and cooling cycles were necessary for each sample in order to homogenize the melt. The temperature was monitored by an infrared pyrometer with an absolute accuracy, relative accuracy, and response time of 10, 3 K, and 5 ms, respectively. The cooling curve was calibrated with a standard PtRh30-PtRh6 thermocouple, which was protected in a silica tube and then immersed into the melt under the identical condition. A more detailed experimental procedure is available in [8]. In this way, the bulk $Fe_{83}B_{17}$ alloy melts were undercooled and even hypercooled due to suppressing nucleation of alloy melts. As-prepared global samples of diameter 10 mm (which is also the inner diameter of quartz crucible) were observed and analyzed by selected area electron diffraction (SAED), X-ray diffraction (XRD) utilizing Cu-K_α radiation, and scanning electron microscopy (SEM). Chemical compositions have been checked by induction coupled plasma spectroscopy (ICP) with an accuracy of elemental composition determination less than 1% of the nominal content. Alloy specimens (with thickness of about 1.5 mm) for the magnetic property measurement were cut from the as-solidified bulk samples, and then were measured using a Lake Shore vibrating sample magnetometer (Model 7307-VSM), which can detect moments as small as 5×10^{-6} emu in magnetic fields ranging from −21 to +21 kOe.

3. Results and discussion

3.1. Analysis of as-solidified microstructure

A detailed description of the microstructure evolution of undercooled Fe–B eutectic alloy is available in Ref. [9]. Here we are solely concerned with the as-solidified microstructure for undercooling (ΔT) beyond hypercooling.[1] The hypercooling limit ($\Delta T_{\text{Hyper}} = \Delta H_f/C_p^l$, where ΔH_f and C_p^l are the latent heat and the specific heat of the undercooled liquid, respectively) is calculated to be about 300 K for the Fe–B eutectic alloy [11]. In the present experiments, the undercoolings (ΔT is a temperature difference between the equilibrium melting point and actual primary nucleating temperature) of 300–460 K were obtained successfully by observing the cooling curve of undercooled alloy melt, which means the hypercooling of alloy melts is attained. Herein, a control on the degree of hypercooling and an analysis of cooling curve are not presented as a content of paper, which have been discussed in detail in later paper. Fig. 1a and b, respectively, present the as-solidified morphologies subjected to hypercooling of 341 and 460 K. An irregular eutectic prevails independent of ΔT.

From the equilibrium Fe–B phase diagram, the eutectic structure is composed of α- Fe and Fe_2B phases. In the present experiments, however, the stable-phase Fe_2B was replaced by the metastable-phase Fe_3B in the RT structure if the alloy melt was undercooled more than 386 K. This has been confirmed by XRD analysis, as shown in Fig. 2. Further, SAED analyses [8] (Fig. 3) have identified that the lighter phases in Fig. 1a and b are Fe_2B and Fe_3B, respectively. The dark phase is α-Fe.

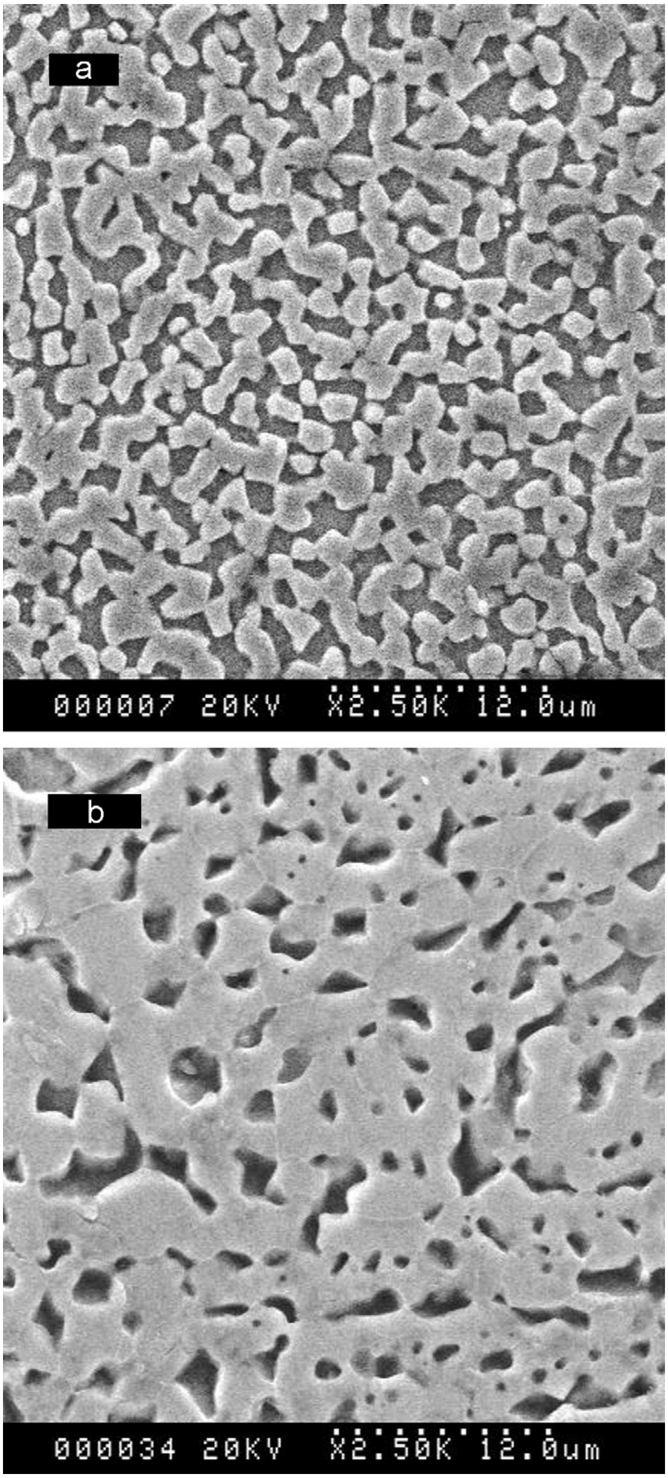

Fig. 1. Microstructures of the $Fe_{83}B_{17}$ eutectic alloy solidified under different hypercooling conditions: (a) $\Delta T = 341$ K, eutectic phase: α-Fe (dark)+Fe_2B (light); (b) $\Delta T = 460$ K, eutectic phase: α-Fe (dark)+Fe_3B (light).

At ΔT values of 341–460 K, the microstructures appear very similar, but the relative volume fraction and the grain size of the Fe_2B and Fe_3B phases change. The difference between the relative volume fractions is consistent with their compositional difference, and thus can be understood in terms of the lever principle, i.e. in the metastable and equilibrium Fe–B phase diagrams [4], the nominal composition of Fe_3B phase (25 at% B) is closer to the eutectic point (17 at% B) compared with that of Fe_2B phase (33.3 at% B). The difference between the grain sizes

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[1] Hypercooling is defined [10] as an isenthalpic solidification, i.e. the alloy melts finish rapidly mass nucleation (i.e. no liquid phase is left) and non-segregation solidification, which provides a unique opportunity to obtain the microstructural morphologies originally formed from the melts.