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Original Research Paper

Phase transformations and microstructural properties of nanocrystalline $Fe_{75}Si_{10}B_{10}Nb_5$ alloy synthesized by mechanical alloying

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ABSTRACT

Nanostructured $Fe_{75}Si_{10}B_{10}Nb_5$ powders were prepared by mechanical alloying of Fe, Si, B and Nb elements in a high-energy planetary ball mill. The structural properties and morphology changes were investigated as a function of milling time (in the 0–100 h range) by means of X-ray diffraction and scanning electron microscopy. At early stage, the dissolution of B and Si atoms into the Fe lattice leads to the formation of bcc Fe(Si,B) solid solution. After milling until 100 h, a mixture of Fe(Si,B) (~20 nm) and Nb(B) (~10 nm) is obtained. On the other hand, an interesting phenomenon called mechanical recrystallization, related to the formation of a crystalline phase of a partial amorphous powder (obtained at ~56 h), was investigated.

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1. Introduction

Nanocrystalline materials offer promising and exceptional physical, chemical, and mechanical properties, because of the important fraction of atoms residing in grain boundaries, which can be exploited for a variety of applications [1]. Among the techniques that are used to synthesize nanocrystalline materials, mechanical alloying (MA) has attracted considerable attention due to the favorable economy and flexibility of the process [2,3]. During the milling process, the powder particles are exposed to highly energetic compressive collision forces which lead to enhance the density of dislocations and the fraction of grain boundaries. The repeated fracturing and cold welding of the powder particles cause reactions between solid components of the initial mixture and lead to formation of nanocrystalline and/or amorphous structures [3].

Nanocrystalline Fe-based alloys combine the beneficial properties of different soft magnetic materials such as low coercive force, high saturation induction and high permeability [4,5]. The justification of this property combination is the correlation of the structural length which is much smaller than the ferromagnetic one [6]. That's why nanocrystalline Fe-based alloys have been studied and used for wide applications in technology such as power transformers, and inductors [7–9]. The addition of proper amounts of Si to Fe results in decreasing of magnetic anisotropy and coercive force and increasing of electrical resistivity which considerably reduces eddy current losses (lower hysteresis) [10]. For example, Fe–Si alloys can be used as magnetic refrigerant materials by varying the transition temperature with minor compositional changes [11]. The addition of Nb to Fe–Si alloys favors the thermal stability as does B in the generation of a strong magnetic coupling of the nanocrystalline structure. It also prevents grain growth because of its large atomic radius in comparison with that of Fe and subsequently its slow diffusivity [12,13]. The introduction of B in the Fe lattice favors the development of amorphous and/or nanocrystalline structures by being brought into solid solution into the bcc-Fe phase [14]. So, the formation of intermetallics and amorphous phases was noted in the binary Fe–B powder mixtures depending on the milling time. A maximum of about 22 at.% B was found to dissolve in Fe, and consequently FeB and Fe_3B intermetallics were formed in some of the powder mixture.

The present investigation is aimed to study the microstructure of the mechanically alloyed $Fe_{75}Si_{10}B_{10}Nb_5$ alloy by analyzing the whole X-ray diffraction patterns using the Rietveld method. Different structural properties such as the crystallite size, microstrains and lattice parameter are determined. The character of dislocation structure evolution is also studied in order to demonstrate that strain broadening caused by high energy mechanical alloying is consistent with the concept of dislocations.

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2. Materials and methods

The elemental powders of iron (99.7% purity, <10 μm), silicon (99.9% purity, <45 μm), niobium (99.85% purity, ~74 μm) and amorphous boron (>99% purity) were weighted and mixed in proportions corresponding to the nominal composition $Fe_{75}Si_{10}B_{10}Nb_5$ (at.%) and mechanically alloyed in a high-energy planetary ball-mill (Fritsch Pulverisette 7) using steel vials and balls with diameter of about 15 mm. The ball-to-powder weight ratio was 1:6 and milling was carried out at room temperature, in an argon atmosphere and at 600 rpm. Different milling times ranging from 1 to 100 h were used. To avoid the local temperature rise inside the vials during milling, each 10 min of milling was followed by a pause of 5 min.

Morphological changes of the powder particles during the milling process were followed by scanning electron microscopy (SEM) in DSM960A Zeiss equipment. The particle size distributions were made with image analyzer software (ImageJ, WRNIH, USA). The particles size distributions were obtained from dark field SEM images by measuring the mean size of more 50 diffracting particles. The structural changes of the milled samples were investigated by X-ray diffraction (XRD) by means of a Bruker D8 Advance diffractometer in a θ–2θ geometry using Cu Kα radiation ($\lambda_{\alpha Cu}$ = 0.15406 - nm). The microstructural parameters were taken out from the refinement of the XRD patterns by using the MAUD program [15] which is based on the Rietveld method. The procedure consists in modeling the diffraction profiles by analytical functions. The estimation of the average crystallite size D and the microstrains $\langle \varepsilon^2 \rangle^{1/2}$ is derived from the isotropic model [16,17] where the experimental profiles are fitted using a pseudo-Voigt (pV) analytical function [16–18]. The description of determination of these microstructural parameters has been well detailed by Laala-Bouali et al. [19].

3. Results and discussion

3.1. Evolution of particle morphology and size

Fig. 1 shows SEM-micrographs of $Fe_{75}Si_{10}B_{10}Nb_5$ powders obtained before and after milling for 2, 4, 10, 26 and 56 h. In the unmilled powders, Fe powder particles have roughly spherical shapes whereas the Si and Nb particles have thin plate-like morphology. After 2 h of milling, the particles fracturing and cold welding phenomena specific to MA are clearly notable. For extended milling time until 10 h, one can see a considerable change in the size, morphology and size distribution of powder particles. The average particle size enhances with increasing milling time and reaches a maximum value of about 12.7 μm after

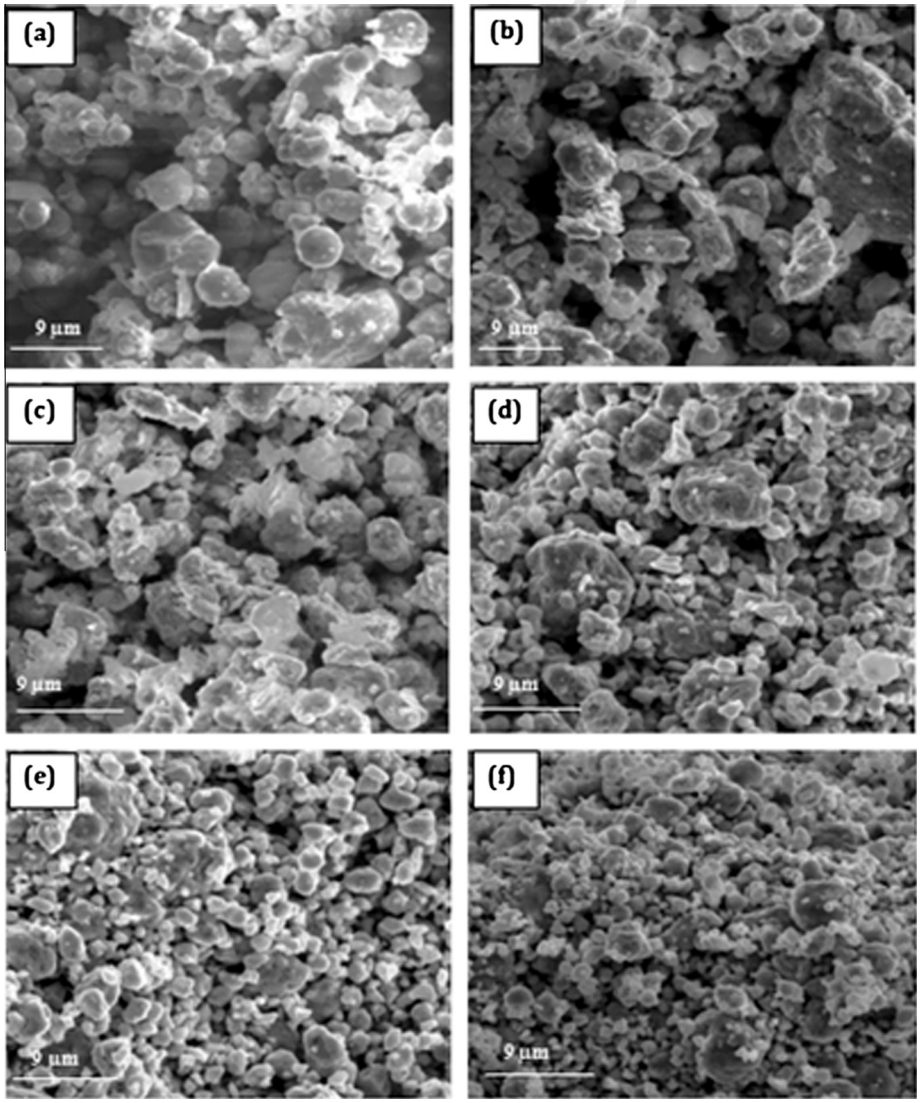

Fig. 1. SEM-morphologies of the $Fe_{75}Si_{10}B_{10}Nb_5$ powders milled for different times: (a) 0 h, (b) 2 h, (c) 4 h, (d) 10 h, (e) 26 h and (f) 56 h.

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