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Mechanical alloying of Fe–Ni based nanostructured magnetic materials

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Abstract

Alloys with the composition $Fe_{40}Ni_{38}B_{18}Mo_4$, $Fe_{49}Ni_{46}Mo_5$ and $Fe_{42}Ni_{40}B_{18}$ were processed from elemental powders by mechanical alloying and the microstructure was studied by differential scanning calorimetry (DSC), X-ray diffractometer (XRD), scanning electron microscopy (SEM) and transmission electron microscopy (TEM) techniques. Nanocrystalline fcc solid solution was achieved as a result of mechanical alloying in all three alloy compositions and the grain size reduced to nanoscale but lattice strain was introduced. Molybdenum was found to affect the products of mechanical alloying, specifically, the Fe_3B phase formed in the $Fe_{42}Ni_{40}B_{18}$ alloy while no boride phase formed in the $Fe_{40}Ni_{38}B_{18}Mo_4$ alloy. SEM studies indicated that the presence of boron was found to make the milling process easier. Elemental mapping by SEM as well as XRD results showed that molybdenum does not dissolve easily in the Fe–Ni solid solution produced by milling. The DSC results suggested that an amorphous structure together with nanocrystals was obtained in the $Fe_{40}Ni_{38}B_{18}Mo_4$ and $Fe_{42}Ni_{40}B_{18}$ alloys. A two-stage crystallization process was found in the $Fe_{40}Ni_{38}B_{18}Mo_4$ and $Fe_{42}Ni_{40}B_{18}$ alloys, the presence of boron was found to make amorphization easier. TEM investigations were consistent with these XRD and DSC results. Heat-treated samples of the $Fe_{40}Ni_{38}B_{18}Mo_4$ and $Fe_{42}Ni_{40}B_{18}$ alloys milled for 100 h showed that molybdenum inhibited the grain growth. The saturation magnetization of the heat-treated $Fe_{40}Ni_{38}B_{18}Mo_4$ alloy milled for 100 h was stable, coercivity was reduced; on the other hand, the M_s of heat-treated $Fe_{42}Ni_{40}B_{18}$ alloy milled for 100 h decreased and the H_c increased. This difference in magnetic behavior is due to the alloying addition of molybdenum which affected the microstructural evolution during heat treatment, specifically by inhibition of the increase in grain size.

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1. Introduction

Mechanical alloying (MA) is a useful powder processing technique that can produce a variety of equilibrium and non-equilibrium alloy phases [1,2]. The advantage of this process technology is that the powder can be produced in large quantities and the processing parameters can be easily controlled, thus it is a suitable method for commercial applications [3]. It can also be used to produce amorphous and nanocrystalline materials in commercially relevant amounts and is also amenable to the production of a variety of alloy compositions [4,5]. The Fe–Ni alloy system boasts excellent soft magnetic properties and in this work the mechanical alloying of Fe–Ni-based alloys from elemental powders was studied. Kuhrt [6,7] and Kaloshkin et al. [8–10] studied mechanically alloyed $Fe_{100-x}Ni_x$ alloy as a function of milling intensity, this work led to the availability of a non-equilibrium phase diagram, both bcc and fcc supersaturated solid solutions with high internal strain were reported. Pakala et al. [11,12] processed Fe–Ni alloys by low- and high-energy ball milling and reported that a bcc Fe–Ni solution was produced as a result of milling. The martensitic transformation in mechanically alloyed Fe–Ni alloy was studied by Zhu [13]. An iron–nickel-based melt spun $Fe_{40}Ni_{38}B_{18}Mo_4$ alloy was studied by other researchers [14,15] and it was reported that nanocrystallized fcc Fe–Ni solid solution formed from the amorphous precursor. Fe–Ni binary alloys have been studied earlier as discussed above; however, relatively little attention has been devoted to studying the effect of boron and molybdenum on the milling and subsequent crystallization behavior. Counterpart crystallization studies on melt spun Fe–Ni–B–Mo alloy with the same composition showed that molybdenum had a strong effect on the crystallization behavior and boron provides glass forming ability. Hence this study was performed to study the effect of molybdenum and boron alloying additions, both individually and together, on the milling behavior and subsequently on the magnetic properties. The synthesis of $Fe_{40}Ni_{38}B_{18}Mo_4$, $Fe_{49}Ni_{46}Mo_5$ and $Fe_{42}Ni_{40}B_{18}$ alloy compositions from elemental powders by mechanical alloying is reported in this paper. The aim of the present work is to study the process of amorphization in Fe–Ni-based soft magnetic materials, the effect of boron and molybdenum on the alloying process, the crystallization process and the magnetic properties of the mechanically alloyed powders. The experimental techniques used were scanning electron microscopy (SEM), X-ray diffractometer (XRD), differential scanning calorimetry (DSC), transmission electron microscopy (TEM) and vibration sample magnetometry (VSM).

2. Experimental procedure

The $Fe_{40}Ni_{38}B_{18}Mo_4$ composition produced by melt spinning is a commercial alloy and was chosen in counterpart melt spinning studies; in this paper the iron–nickel-based $Fe_{40}Ni_{38}B_{18}Mo_4$, $Fe_{49}Ni_{46}Mo_5$ and $Fe_{42}Ni_{40}B_{18}$ alloys were prepared by FRITSCH pulverisette 5 planetary ball milling under argon atmosphere from pure elemental powder (>99% purity), iron (22 mesh), nickel (170 mesh), boron (325 mesh) and molybdenum (3–5 μm). The $Fe_{49}Ni_{46}Mo_5$ and $Fe_{42}Ni_{40}B_{18}$ alloys were produced in order to study the individual effects of molybdenum and boron. The $Fe_{49}Ni_{46}Mo_5$ and $Fe_{42}Ni_{40}B_{18}$ compositions are chosen such that the omission of the molybdenum (or boron content) in the commercial alloy is compensated by a normalizing of the remaining components to 100%. For instance, in order to study the effect of boron, the molybdenum content was "removed" from the alloy composition $Fe_{40}Ni_{38}B_{18}Mo_4$ (thus yielding a composition of $Fe_{40}Ni_{38}B_{18}$), and the composition normalized to 100%, yielding $Fe_{42}Ni_{40}B_{18}$. A pair of tungsten carbon vials and balls were used as the milling medium. The ball to powder ratio (BPR) was 8:1. The powder was milled at a speed of 300 rpm for milling times ranging from 1 to 100 h.

The microstructure was studied by XRD (Shimadzu XRD-6000) and TEM (JEM 2010). TEM samples were prepared by the method introduced by Huang [16]. The powders were mixed into a small amount of epoxy resin then a piece of metal net was embedded into the mixture; finally, the sample was subjected to grinding, dimpling and