



Effect of Nb and Cr incorporation on the structural and magnetic properties of rapidly quenched FeCoSiB microwires

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ABSTRACT

Rapidly quenched microwires with a nominal composition of $\text{Fe}_{39}\text{Co}_{39}\text{Si}_8\text{B}_{14}$ ($\#A_0$), $\text{Fe}_{37}\text{Co}_{37}\text{Nb}_4\text{Si}_8\text{B}_{14}$ ($\#A_N$) and $\text{Fe}_{36}\text{Co}_{36}\text{Nb}_4\text{Cr}_2\text{Si}_8\text{B}_{14}$ ($\#A_{NC}$) have been investigated. Devitrification of as-quenched microwires showed that crystallization temperatures increased with simultaneous incorporation of Nb and Cr as in $\#A_{NC}$ alloy. Addition of these elements also contributed to an increase in activation energy in $\#A_N$ and $\#A_{NC}$ alloys. Nb addition reduced the particle size, which became much finer in the case of the Cr-containing alloy. Although Nb addition did not have much effect on lowering the Curie temperature T_C of the amorphous phase, Cr substitution lowered T_C to 698 K from high values of 785 K and 787 K observed in the $\#A_0$ and $\#A_{NC}$ alloys, respectively. However, the Cr addition revealed a better Giant magneto-impedance (GMI) response compared to the other alloys. Such improved GMI properties in the Cr-containing alloy are attributed to lower values of the coercivity and magnetostriction in the alloy containing both Nb and Cr.

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1. Introduction

The Fe-based amorphous and nanostructured systems developed in the form of ribbons through the melt spinning technique have been dominating the class of soft magnetic materials owing to their potential application as transformer cores, switch mode power supplies, magnetic delay lines etc. [1]. The incorporation of cobalt in Fe-based alloys had a subsequent demand in components like rotor assemblies of jet engines, auxiliary power systems, high frequency transformers etc. operating at elevated temperatures [2]. The ferromagnetic ordering temperature or the Curie temperature was elevated in these melt spun ribbons.

In recent years there was a paradigm shift in the application requirements with intensified demand for miniaturized sensors pertaining to surveillance, non-destructive testing, security and bio-medical [3]. The quest for development of micro-magnetic sensors led to the process of rapidly quenched wires through in-water quenching technique. The CoFe-based alloys prepared in the form of amorphous wires revealed not only high ferromagnetic ordering temperatures but also phenomenal property like “Giant magneto-impedance effect” [4]. The combinations of both of these properties are under intensive investigation for

development of GMI-based sensor materials in the form of wires with a fair thermal stability and sensitive response of impedance. The process of spinning amorphous wires involves rapid quenching of the alloy melt in a dynamical water flow within a rotating drum. Thus, the issues of alloy design pertaining to casting of amorphous wires are critically important. In addition to high magnetic moment and enhanced ferromagnetic stability requirements, metal-metalloid (Fe–B, Fe–Si, Fe–Si, B etc.) content, incorporation of thermally stable refractory elements (W, Ta, Nb etc.) and corrosion retarders like Cr are important factors determining the potentiality of GMI sensors. The stoichiometric ratio of Co:Fe::50:50 is expected to deliver high magnetic moment as per the Slater–Pauling curve [5]. The incorporation of element cobalt is not only expected to raise the ferromagnetic ordering temperature but also to play a dual role as a nucleating element in place of copper in FINEMET systems [6,7]. Moreover, $\text{Fe}_{50}\text{Co}_{50}$ -based alloy systems have been reported to reveal better GMI response [8].

Freezing of short range order in rapidly quenched materials demands a metalloid content (silicon + boron) in the range of 18–28% [9]. These elements play a pivotal role during nanocrystallization and thus modifying the magnetocrystalline anisotropy as well as magnetostriction of the system. Critical silicon content in phase with Co and Fe lowers the magnetostriction of the nanostructured system which is advantageous for achieving better GMI response. The larger element like niobium with atomic radius as large as 1.46 Å controls the reaction kinetics by retarding the growth of the nanophases and enhanced GMI [10,11]. The

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averaging out of the anisotropies in the nanophase lowers the overall anisotropy leading to enhanced soft magnetic properties. Thus, Nb incorporated Fe–Cu–Si–B system showed interesting GMI properties. Just as Nb has the role in attributing thermal stability, the addition of chromium is explored to minimize the oxidation effects during in-water quenching process and the consequent deleterious effects of corrosion on magnetic properties. In addition to corrosion inhibition, Cr addition in Fe-based system has been recently reported to enhance the GMI effect [11]. In view of the fact that such reports have been mostly focused on the effect of Cr addition in Fe-based alloys, the present investigation has been targeted to evaluate the influence of simultaneous incorporation of Nb and Cr in the Fe–Co based system.

The present investigation addresses three alloys of Fe–Co–Si–B-based rapidly quenched wires whereby the effect of Nb and Cr incorporation has been studied with respect to their thermal stability, ferromagnetic ordering temperature, phase evolutions and Giant magneto-impedance (GMI) effect.

2. Experimental procedure

Alloys with nominal composition of $\text{Fe}_{39}\text{Co}_{39}\text{Si}_8\text{B}_{14}$, $\text{Fe}_{37}\text{Co}_{37}\text{Nb}_4\text{Si}_8\text{B}_{14}$ and $\text{Fe}_{36}\text{Co}_{36}\text{Nb}_4\text{Cr}_2\text{Si}_8\text{B}_{14}$ represented by Nb/Cr free ($\#A_0$), Nb contained ($\#A_N$) and Nb/Cr contained ($\#A_{NC}$) alloys respectively were prepared by arc melting in an argon atmosphere using pure elements. For the preparation of the wires an in-rotating-water quenching system or the wire caster with induction melting facility was used. The alloy ingots taken in quartz crucible was induction melted and ejected out through a nozzle in the bottom of the crucible on a rotating drum containing water. Continuous wires of diameter 120 μm were obtained after optimizing different process parameters. The structures of as-cast and annealed wires were characterized by x-ray diffractometer (XRD) using $\text{CuK}\alpha$ radiation and particle size calculated through TOPAS software developed by M/S Bruker (Germany). Phase transformation was investigated using differential scanning calorimeter (Diamond DSC) and thermal electrical resistivity (Ulvac TER 2000) in argon atmosphere. About 9 cm long wire sample was placed along the axis of a Helmholtz coil for the GMI measurement. The impedance was measured by the four-probe technique where driving field was generated by passing ac current, I_{ac} , through the wire. A spectrum analyzer (Agilent-4401B) was used to measure the voltage across the voltage probe. A Helmholtz coil was used to apply a dc magnetic field parallel to the axis of the sample. The GMI ratio has been calculated from the first harmonic signal using Eq. (1):

$$\frac{\Delta Z}{Z} \% = \frac{[Z(H) - Z(H_{\max})]}{Z(H_{\max})} \times 100 \quad (1)$$

where $H_{\max}$ is the maximum dc applied field which is ± 7500 A/m in the present case. The GMI ratio at peak maxima ($\text{GMI}_{\max}$) was calculated from the plots. The microwires revealed their respective $\text{GMI}_{\max}$ outputs at optimized frequency and driving alternating current of 400 kHz and 10 mA. Magnetostriction of the wire samples was measured from a two terminal capacitance technique using a measurement system developed in the laboratory.

3. Results and discussion

3.1. Crystallization behavior

Devitrification of amorphous ferromagnetic materials is manifested through differential scanning calorimetry (DSC). Fig. 1

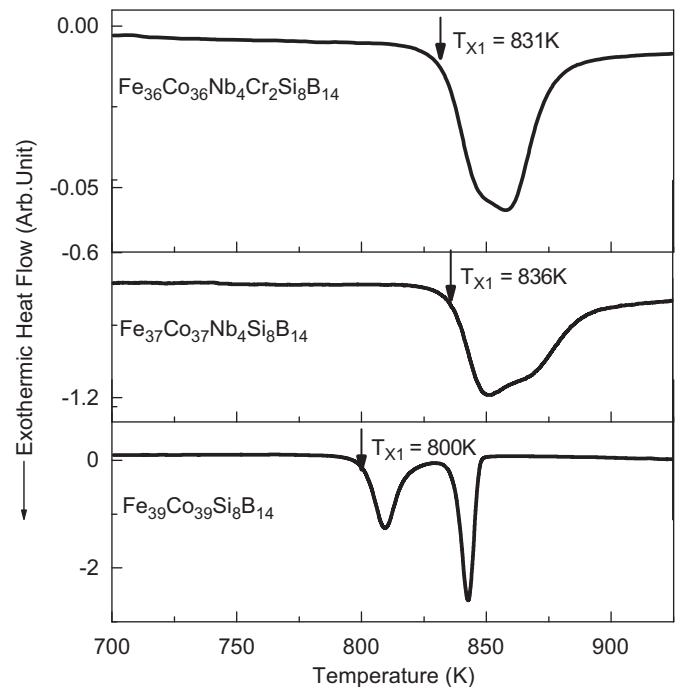


Fig. 1. DSC plot of the as-quenched microwires.

shows the DSC plots of the addressed alloys shown over the same span of temperature. All the alloys manifest exothermic transformation occurring at onset temperature T_{X1} . The base alloy $\#A_0$ revealed an onset T_{X1} at 800 K. Stoichiometric substitutions of Fe and Co by Nb increased the crystallization onset of the alloy $\#A_N$ from 800 K to 836 K. This behavior is attributed to the addition of large sized element Nb which retarded the kinetics of the crystallization process. The sluggish process was marked by a broader exothermic peak in $\#A_N$ compared to alloy $\#A_0$. With Cr addition, the onset of crystallization reduced from $T_{X1} = 836$ K in $\#A_N$ to 831 K in $\#A_{NC}$ alloy. In the later alloy, Cr addition also made the DSC peak sharper, suggesting an offset in the enhanced stability observed in $\#A_N$ alloy. The base alloy $\#A_0$ revealed a second crystallization onset at $T_{X2} = 837$ K while $\#A_N$ also had a shoulder between 850 K and 875 K which may be a consequence of an incipient second crystallization. The activation energy of primary crystallization was obtained from the modified Kissinger equation [12] given by

$$\ln \frac{h}{T_p^2} = -\frac{E_a}{RT_p} + \text{constant} \quad (2)$$

where R is a gas constant, T_p is an exothermic peak temperature and h is the heating rate. The Kissinger plots are shown in Fig. 2. The activation energies for the alloys $\#A_0$, $\#A_N$ and $\#A_{NC}$ were found to be 278 kJ/mol, 458 kJ/mol and 598 kJ/mol respectively. The higher activation energies in the later two alloys indicate the stability against devitrification with Nb and Cr incorporation. The high activation energies may also be attributed to enhanced retention of these elements in the amorphous matrix as compared to the ferrite phase [13].

3.2. Evolution of phases and electron transport property

To identify the phases, x-ray diffractograms were obtained for the amorphous wires in their as-prepared state and after heat treatment past their crystallization onsets T_{X1} and T_{X2} . It was observed (Fig. 3) that in the as-quenched condition all the present wires were in the amorphous state as revealed by broad halo in

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