Investigation of magnetic profile of $Fe_{77}Cr_2Si_5B_{16}$ metallic glass by using ferromagnetic resonance (FMR) and vibrating sample magnetometry (VSM) techniques

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The ferromagnetic resonance (FMR) technique was used to determine the magnetic anisotropies of the as-quenched and furnace annealed $Fe_{77}Cr_2Si_5B_{16}$ metallic glasses. The heat treatment of metallic glasses caused an increase in the effective magnetic anisotropy with the increase of annealing time due to the stress relaxation and than crystallization of the sample. Changing of the magnetic properties such as magnetic anisotropy, magnetic coercivity, susceptibility and magnetic hysteresis loops of the samples were observed for as-quenched and annealed samples. The magnetic hysteresis loops of as-quenched and annealed samples for 1, 2, 4, 6, 8, 10 minutes were measured by Vibrating Sample Magnetometer (VSM) at room temperature and stress relaxations were observed from M-H loops. The value of g-factor and effective magnetic anisotropy constants were calculated from the variation of resonance field with the rotation angle of the samples at out-of-plane geometry. As-quenched and treated samples' g-values were found as 2.19.

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1. Introduction

Fe-based metallic glasses are commercially important due to their enormous properties that make them indispensable for applications such as power transformer cores, magnetic sensors, security systems, etc. Metallic glasses are characterized by chemical and topologically disordered atomic arrangements.

Recent works on Fe-based alloys have been reported such as structural transformations from amorphous to nanostructure and crystallized structures [1,2]. A lot of studies on metallic glasses were carried out to improve and control their magnetic, mechanic and electrical properties [3-9]. Annealing methods are very important to control the magnetic, mechanic, electrical and structural properties of the metallic glasses. Jagielinski used flash annealing to improve magnetic properties of metallic glasses [10]. XRD and polarized neutron scattering techniques have also been used to examine the magnetic structures [11]. Magnetic properties of metallic glasses having different compositions were measured for isothermal condition and after pulse heating treatments [12]. Induced magnetic anisotropy changes in metallic glasses by current annealing were also investigated [13]. Previous studies in amorphous transition metal alloys pointed out that the magnetic and magnetotransport properties could be controlled precisely by furnace annealing [1,14-17].

2. Theoretical model for FMR spectra

In this study the magnetic properties and the magnetic anisotropies of as-quenched and furnace annealed samples were analyzed by using the Ferromagnetic Resonance (FMR) and Vibrating Sample Magnetometry (VSM) technique. The FMR spectra were analyzed by the following model. The free energy E for the magnetization is given by

$$\begin{aligned} &E = E_z + E_b \\ &E_z = -M_0 H(\sin\theta \sin\theta_H \cos(\phi_H - \phi) + \cos\theta \mathrm{os}\theta_H) \\ &E_b = K_{eff}\cos^2\theta,\ K_{eff} = (2\pi M_0 - K_\perp) \end{aligned} \qquad (1)$$

where E_z and E_b are the Zeeman and the bulk (overall) anisotropy energy terms, respectively. H is static magnetic field, M_o is the saturation magnetization, θ and ϕ are the spherical angles for magnetization, M, θ_H and ϕ_H are usual spherical polar angles for the applied field H as shown in Fig. 1. K_{eff} is the effective bulk anisotropy (shape-demagnetizing) constant and it is related with M_{eff} as $4\pi M_{eff} = \frac{2K_{eff}}{M_0}$. For the resonance condition the classical resonance equation given in many ferromagnetic resonance studies in literature was used [18, 19].

$$\frac{\omega_o}{\gamma} = \frac{1}{M_o \sin\theta}\left(E_{\theta\theta}E_{\varphi\varphi} - E_{\theta\varphi}^2\right)^{1/2} \qquad (2)$$

By using Eq. 2 we get following resonance

$$\left(\frac{w}{\gamma}\right)^2 = \left[H\cos(\theta_H - \theta) - 4\pi M_{eff}\cos^2\theta\right] \mathrm{x} \qquad (3)$$
$$\left[H\cos(\theta_H - \theta) - 4\pi M_{eff}\cos 2\theta\right]$$

The value of g-factor and effective magnetic anisotropy constants were calculated from the variation of resonance field with the rotation angle of sample at out-of-plane geometry for all samples.

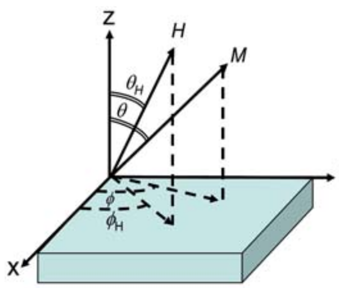

Fig. 1. The sample geometry and relative orientation of the sample with respect to the applied static magnetic field (H) and static magnetization (M).

3. Experimental procedures

3.1 Sample preparation

$Fe_{77}Cr_2Si_5B_{16}$ (METGLAS 2605 S3A) metallic glass used in this work was obtained from Honeywell Corporation. The structural and magnetic properties of as-received ribbon were characterized by X-ray diffraction XRD, and differential scanning calorimetric DSC. All the samples were cleaned with acetone and dried in desiccators before subjected to annealing treatments. A standard tube furnace was used to anneal the samples isothermally at fixed 723 K in air. Samples were placed in a preheated aluminum plate in the furnace, and carefully removed from the furnace at convenient intervals to measure coercivity. XRD patterns obtained from the wheel side and air side of the as–received ribbons revealed no Bragg peaks, just the amorphous halo. Curie temperature T_c, and crystallization temperatures $T_{x,}$ were found as 633 K and 803 K, respectively [20].

3.2 Ferromagnetic resonance (FMR) measurements

Rapidly quenched metallic glass $Fe_{77}Cr_2Si_5B_{16}$ was prepared by planar flow casting method. The field derivative FMR absorption spectra were recorded on a Bruker EMX type electron spin resonance (EPR) spectrometer at the X-band frequency (9.5-9.8 GHz). The angular dependence of the FMR spectra was investigated for parallel geometry that applied DC magnetic field is in the surface of the metallic glass and perpendicular geometry with the magnetic field perpendicular to the surface.

The ferromagnetic resonance (FMR) technique was used to determine the magnetic anisotropies of as-quenched and annealed $Fe_{77}Cr_2Si_5B_{16}$ bulk samples. The FMR measurements were carried out using commercial EMX X-Band (9.8 GHz) spectrometer equipped with pole-cap which provides a DC magnetic field up to the 22 kG magnetic field. A goniometer was used to rotate the sample holder which is parallel to the microwave magnetic field and perpendicular to the applied static magnetic field. The sample was replaced to sample holder at out-of-plane geometry where the sample was attached to the flat platform of sample holder. The magnetic field of microwave lie in film plane during measurement and static magnetic field is rotated from the sample plane to surface normal. The coordinate axes and relative orientation of applied static magnetic field and magnetization vector were illustrated in Fig. 1. The field derivative of microwave power absorption (dP/dH) was recorded as a function of the DC field. To obtain intensities of FMR signals, the double digital integration of the resonance curves were performed using Bruker WINEPR software package. The measured FMR spectra of as-quenched and thermally annealed samples for parallel and perpendicular geometry are shown in Fig. 1. The samples were cut into 1 mm x 1 mm dimensions and replaced in a furnace and annealed in atmospheric medium at fixed temperature (683 K) with different annealing time. After annealing treatment, the samples were carefully removed from the furnace to cool down to room temperature.

3.3 Vibrating sample magnetometer (VSM) measurements

Using a VSM, one can measure the DC magnetic moment, magnetic field. Some of the most common measurements are hysteresis loops, susceptibility and saturation magnetization as a function of angle and time. In this work, we measured magnetic anisotropy K_{eff}, coercivity H_c, and susceptibility χ, using VSM.

4. Results and discussion

Fig. 2 (a) shows the room-temperature FMR spectra recorded from as-received and stress relief metallic glass $Fe_{77}Cr_2Si_5B_{16}$ with individual layer having thickness 22 μm at fixed frequency of 9.91 GHz and fixed angle. In the measurements, the magnetic fields 0 to 20 kOe were applied. A single peak, a Lorentzian line shape, was observed in the sample for all orientations of external magnetic field. The sharp peak indicates the maximum resonance values of each step that makes the comparison of each step easier. It can be seen from the Fig. 2 that as-received sample has anisotropy and it starts to change after the annealing steps. Variations of magnetic anisotropy were recognized much easier from Fig. 2.(a) and (b). The resonance field of the peak has strong anisotropic behaviour with respect to the direction of the DC magnetic field for out-of-plane geometry (OPG) and in-plane geometry (IPG). OPG measurements do not show remarkable differences from IPG as shown in Fig. 2. (b). Similar anisotropic behaviour shapes or both geometries for the resonance fields were observed. The absorption intensity and line width characteristics of the peak show very drastic changes at fixed angle and temperature. There is one sharp peak with some width differences observed. FMR spectra of as-quenched and 1, 2, 4, 6, 8, 10 minutes treated samples were measured at 723 K as an indication of a relatively homogenous internal field induced parallel to the metallic glass, while the in–plane component of this field seems to be slightly inhomogeneous as shown in Fig. 2.(a) and (b), The broadening of the line for both OPG and IPG measurements were also measured to be same values.

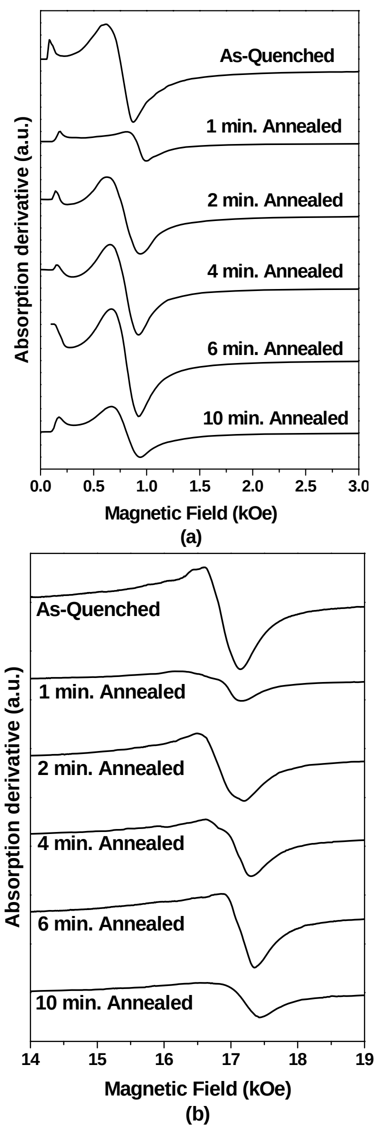

Fig. 2. (a) FMR spectra for parallel geometry of the quenched and thermally annealed samples at 723K (b) FMR spectra for perpendicular geometry of the quenched and thermally annealed samples at 723 K.

The calculated effective magnetic anisotropies of the samples for different annealing time were given in Fig. 3. The heat treatment of metallic glasses shows an increment of effective magnetic anisotropy with the annealing time due to the stress relaxations in the sample. At first two steps of the annealing treatments representing stress relaxation took place and for the rest steps the trapped stresses disappeared. Last part of the annealing treatments caused sharp increasing in the anisotropy that shows the start of nucleation and occurrence of the crystallization.

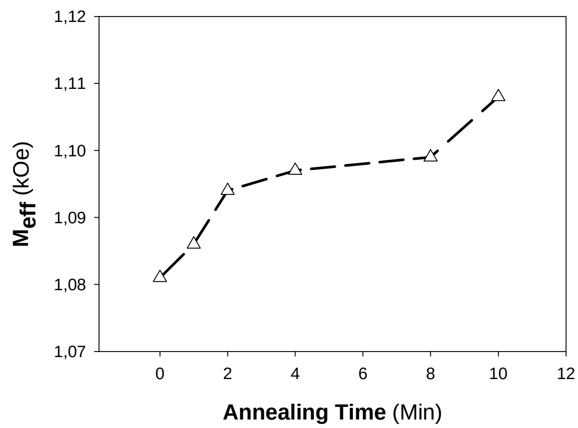

Fig. 3. The variation of effective magnetic anisotropies with annealing time.

The magnetic hysteresis loops of the samples were measured by Quantum design Vibrating Sample Magnetometer (VSM) at room temperature as seen in Fig. 4. The saturation magnetization didn't change significantly by annealing time. It can be seen from Fig. 4. that there are some differences in the shape and area of the loops observed. Stresses and strain were essential for sample preparation by rapid quenching method. Uniformly annealing helps the stress relief of the amorphous samples. As-cast and after treatments, hysteresis loops were taken at each step and the effects could be seen from hysteresis loops as shown in Fig. 4. It can be seen from the Fig. 4. that saturation magnetizations didn't change there, but onset of the saturation changed, however there were slight differences which were observed at the magnetic energy values. Loop area is getting rise because of nucleation and occurrence of crystallization. Strain annealing treatment causes to stress relaxation. As a metallic glass $Fe_{77}Cr_2Si_5B_{16}$ has very low coercive fields which are increasing with the annealing time as shown in Fig. 5.

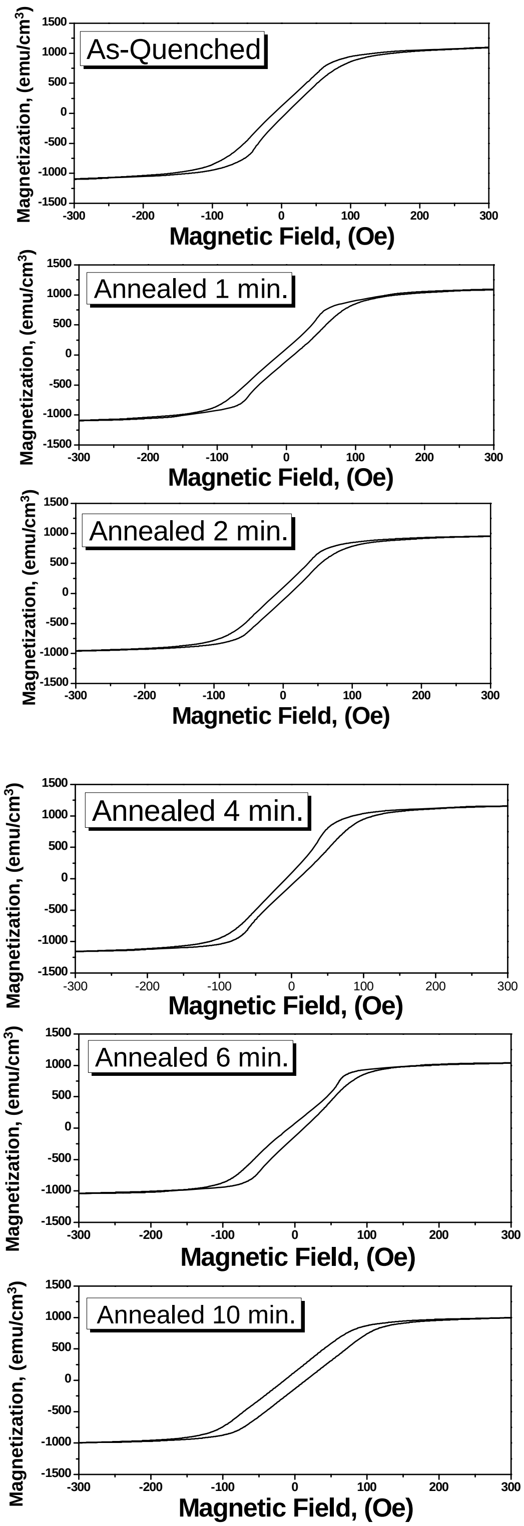

Fig. 4. Magnetization (M) as a function of magnetic field (H) at room temperature for the samples which were annealed at 723 K in different annealing time.

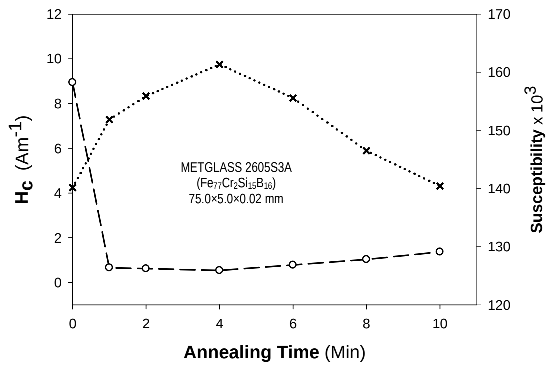

Fig. 5. The coercivity and susceptibility versus annealing time of the soft magnetic material, $Fe_{77}Cr_2Si_5B_{16}$.

5. Conclusions

As-cast metallic glass $Fe_{77}Cr_2Si_5B_{16}$ is very quality raw material and it needs treatments to improve their magnetic properties. In this study, the sample was treated with furnace annealing and after 4 minutes of annealing, it gave the best coercivity H_c value which reduced from 8.94 A/m to 0.54 A/m. The susceptibility χ, increased to pick point $161.3x10^3$ from $140.2x10^3$. Effective anisotropy K_{eff} also increased as expected and the variation of the anisotropy was found from 1.08 kOe to 1.11 kOe. All the changing of magnetic properties is related to the stress relaxation and some oxidations. Crystallization of the samples does not give advantages of the properties from application point of view, so, after ten minutes the annealing process was stopped. Further works will carry on with different annealing methods and samples and also the comparisons of them will be done.

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