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Study and Characterization of Soft Magnetic Properties of $Fe_{73.5}Cu_1Nb_3Si_{13.5}B_9$ Magnetic Ribbon Prepared by Rapid Quenching Method

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Abstract

Nanocrystalline Fe-based alloys offer a new opportunity for tailoring soft magnetic materials. Nanocrystalline alloy in the form of ribbon with the composition of $Fe_{73.5}Cu_1Nb_3Si_{13.5}B_9$ was prepared by rapid quenching method for soft magnetic properties analysis. The rapidly quenched alloy has been annealed in a controlled way in the temperature range 490˚C to 680˚C and annealing time 1 min to 60 min. The study of the structural parameters has been investigated by means of XRD analysis. Magnetic properties were analyzed by measuring B-H loop and frequency dependence of initial permeability. Enhanced value of initial permeability by two orders of magnitude and very low value of relative loss factor of the order of 10^{-3} has been observed with the variation of annealing temperature and time. The initial permeability for the optimum annealed sample has been found 23,064 as compared with 360 for its amorphous counterpart. The initial permeability spectra show dispersion around 100 kHz. Magnetic hysteresis has been investigated by measuring B-H loops at various magnetic fields for different annealing temperature and time. The coercivity and remanence has been found to decrease significantly for optimized annealed condition compared to as-cast state. The core loss of the samples decreases with the annealing time which indicates the good magnetic property of soft magnetic materials.

Keywords

Nanocrystalline Alloy, Magnetic Property, Hysteresis Loop, Permeability, Coercivity, Remanence

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1. Introduction

In 1988, a new class of superior soft magnetic iron based alloys was introduced by Yoshizawa, Oguma and Yamauchi [1]. Those alloys have low losses, high permeability and near zero magnetostriction which indicate the soft magnetic materials. Later soft magnetic properties have been successfully addressed by Herzer [2] with the help of random anisotropy model. Because of the ultrafine microstructure of bcc Fe-Si with grain sizes of 10 - 15 nm, the $Fe_{73.5}Cu_1Nb_3Si_{13.5}B_9$ alloys are known as nanocrystalline. The soft magnetic properties of $Fe_{73.5}Cu_1Nb_3Si_{13.5}B_9$ and related alloys were reported by many publications [3]-[8].

Based on random anisotropy model, by taking into account the nanocrystalline materials as a single phase magnetic system, Herzer explained the grain size dependence of the coercivity. By considering two phase materials, Hernando *et al.* [9] extended this model and were able to explain the thermal dependence of the coercivity [10]-[12]. Also based on the random anisotropy model, Herzer established the early explanation of the soft magnetic properties of these nanocrystalline alloys which were related to the ratio of the domain wall thickness to the mean crystallite size [5]. This model explained the main feature of the dependence of coercivity on the grain size [13] [14]. Some previous researches showed that at the very beginning of nanocrystallization, magnetic hardening took place [15]-[18]. Stoner and Wohlfarth [19] theoretically proposed single domain permanent magnets. From the single domain permanent magnets, high coercivity is obtained by mechanically locking the magnetized single domain particles. However, we can produce soft magnetic materials by reducing the size of the magnetic particle much below the critical size, which is normally of nanometer order.

If Fe-Si-B based amorphous ferromagnetic materials heated below their crystallization temperature, then good soft magnetic properties were observed. After partial crystallization of $Fe_{73.5}Cu_1Nb_3Si_{13.5}B_9$, subjected to heat treatment just above the crystallization temperature for a specific time, an extraordinary high permeability *i.e.* two order of magnitude was developed which was higher than their conventional Fe-Si-B based alloys. The annealed nanometric grain size of $\gg$ 4 - 30 nm is developed for the heat treatment of the alloy. If the grain size is $\gg$ 10 - 12 nm then good magnetic properties are obtained. If $D \ll Lex$, where Lex is the ferromagnetic exchange length [6] [8] and D is the average grain size, then best magnetic properties are obtained. According to calculation, for Fe(Si) alloy, Lex $\gg$ 35 nm [20].

The aim of the present research work is to synthesize Fe-based alloys of $Fe_{73.5}Cu_1Nb_3Si_{13.5}B_9$ composition in the amorphous state by using rapid solidification technique and study their magnetic properties with the evolution of different phases by varying annealing condition. Annealing of Fe-based soft nanocomposite magnetic materials has been performed in air. Then, the Curie temperature, initial permeability, hysteresisgraph (BH-loop), temperature dependence relative quality factor and loss factor of the composition $Fe_{73.5}Cu_1Nb_3Si_{13.5}B_9$ are determined. X-ray diffraction technique has been used for the characterization of nanostructured phases. The purpose is to correlate the evolution of nanograins, their volume fraction and size with the magnetic properties of the composition.

2. Experimental

The ribbons with composition $Fe_{73.5}Cu_1Nb_3Si_{13.5}B_9$ has been prepared by rapid solidification technique. The amorphous ribbons are prepared at a temperature 1500˚C in an argon atmosphere (0.2 to 0.3 atoms). The dimension of the ribbon was 10 mm width and 25 mm thickness. The purity of the material is Fe (99.98%), Cu (99%), Nb (99.8%), Si (99.9%) and B (99.5%) as obtained from Johnson Mathey (Alfa Aesar Inc.).

A PHILIPS PW3040 X' Pert PRO X-ray diffractometer was used to study the crystalline phases of the prepared samples in the Materials Science division, Atomic Energy Centre, Dhaka. The powder specimens were exposed to CuK_α radiation with a primary beam of 40 kV and 30 mA with a sampling pitch of 0.02˚ and time for each step data collection was 1.0 sec. A 2θ scan was taken from 10˚ to 90˚ to set possible fundamental peaks where Ni filter was used to reduce CuK_α radiation. All the data of the samples were analyzed using computer software "X'PERT HIGHSCORE". X-ray diffraction patterns were carried out to confirm the crystal structure. Instrumental broadening of the system was determined from θ - 2θ scan of standard Si. The average grain size of the crystallites α-Fe(Si) were determined by Scherrer's method [21] after correction for instrumental boarding.

As a function of frequency, dynamic magnetic properties of the nanocrystalline magnetic ribbon with composition $Fe_{73.5}Cu_1Nb_3Si_{13.5}B_9$ have been measured. By using a laboratory built furnace and Wayne Kerr 3255B impedance analyzer, temperature dependence of initial permeability of the as cast and annealed ribbons was measured. The Wayne Kerr 3255B impedance analyzer was operated with very low applied ac field of ~10^{-3} Oe from room temperature to above 600˚C with continuous heating rate of ~5˚C/min. With the equation of $\mu' = L/L_0$, the permeability μ' was calculated, where L is the measured sample inductance and L_0 is the inductance of the coil of same geometric shape of vacuum. With the equation of $\mu'' = \mu'$ D, the imaginary permeability μ'' was determined, where D = tanδ, the loss tangent of the sample. On piece of each ribbon has been measured at room temperature after subsequent annealing treatments in order to avoid the experimental error due to fluctuation in ribbon thickness and thermal treatments. From the temperature dependence of permeability measurements, the Curie temperature was determined. The permeability shows an abrupt drop to a very low value at Curie temperature. B-H loops were measured by using a B-H loop tracer.

3. Results

Figure 1 represents the X-ray diffraction spectra of quenched alloy and the alloy annealed at different temperatures for 30 min. In the figures, the parenthesis represents the indices of the reflecting planes of the phases. bcc α-Fe(Si) phases are found, identified by using standard software. From the X-ray fundamental line (110), the mean grain size of the nanograins was determined by using the Scherrer's formula.

In order to determine the Curie temperature of amorphous sample, the real part of the complex initial permeability μ' vs. temperature T curves has been presented in **Figure 2** for as-cast sample. The Curie temperature has been estimated to be 355˚C for this alloy composition in the as-cast condition. It has been found from **Figure 2** that μ' decreases gradually and then there is a sharp increase of μ' near the Curie temperature which is a characteristic feature of the Hopkinson effect.

In **Figure 4**, for as-cast and annealed samples, the real part of the complex initial permeability μ' up to f = 10,000 kHz has been measured and presented. The results refer to isothermal annealing time of 30 min. It can be noted that μ' increase with the increase of annealing temperature and it drops at critical frequencies rapidly.

In **Figure 6**, the annealing temperature dependence of relative quality factor μ'/D of the samples at different frequency and a fixed 30 minute annealing time are presented. The relative quality factor increases with increasing annealing temperature up to 560˚C when the applied frequency is 1 kHz, 2 kHz and 3 kHz. Then, a rapid decrease of relative quality factor with increasing annealing temperature is above 560˚C. In the similar manner, the relative quality factor increases with increasing annealing temperature up to 545˚C when the applied frequency was 5 kHz and 10 kHz. A rapid decrease of μ'/D with increasing annealing temperature above 545˚C was noticed when the applied frequency was 5 kHz and 10 kHz. For the sample annealed at 545˚C for 30 minute give the maximum quality factor if the applied frequency is lowers *i.e.* 10 kHz. This gives a choice of optimal annealing temperature for attaining best magnetic properties.

In **Figure 7**, the annealing temperature dependence of loss factor of the samples measured at different frequency have been presented. Loss factor rapidly decreases with increasing annealing temperature for all applied frequencies *i.e.* 2 kHz, 3 kHz, 4 kHz, 5 kHz and 50 kHz. Loss factor has high value at 50 KHz and decreases with the decrease of frequency. In **Figure 8**, the B-H loops at room temperature and at a constant frequency 1 kHz of the alloy were presented. **Figures 9(a)-(d)** represent the annealing time (ta) dependence of magnetic flux density (Bv), remanence (Br), coercivity (Hc) and loss (W/kG) of the sample annealed at temperature Ta = 545˚C respectively.

4. Discussion

Crystalline phases that developed through heat treatment have been studied by X-ray diffraction and presented in **Figure 1**. In the as cast condition, the sample is in the amorphous state having no sharp peaks of crystalline phase since no crystalline phase has formed due to rapid quenching. When the sample is annealed above the crystallization temperature, nanocrystalline grain of α-Fe(Si) was formed in the amorphous precursor. X-ray diffraction results indicate that no α-Fe phases are present in the alloys annealed below 510˚C. The appearance of broader diffused pattern is also the characteristic of amorphous material was observed bellow the annealing temperature 510˚C. Crystalline phase was developed on the amorphous ribbon, when the alloys were annealed at or above 525˚C. The grain size increases gradually and attains a limiting value of 7 to 14 nm until 560˚C, which is very suitable for the exchange coupling through residual amorphous matrix [23]. A sudden increase of grain size above 650˚C was observed, achieving a value of 18 and 23 nm at 600˚C and 680˚C, respectively [22]. Because annealing at higher temperature above 680˚C leads to the precipitation of Fe-borides [24]. The nanocrystalline material obtained in this way displays improved mechanical and magnetic properties in comparison to the

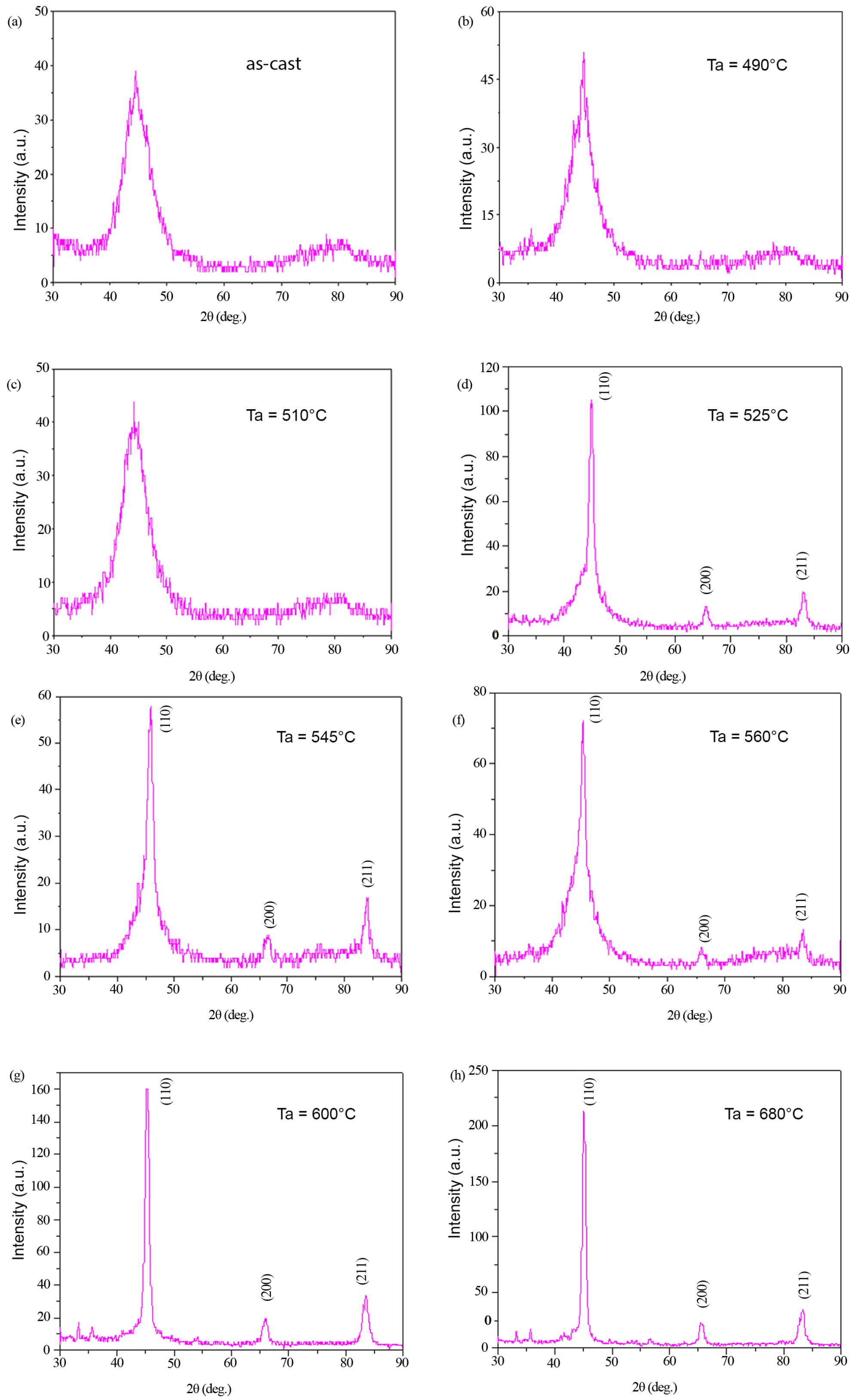

Figure 1. XRD patterns of $Fe_{73.5}Cu_1Nb_3Si_{13.5}B_9$ ribbon sample (a) as-cast and annealed samples at (b) 490˚C (c) 510˚C (d) 525˚C (e) 545˚C (f) 560˚C (g) 600˚C and (h) 680˚C for 30 min [22].

ones achieved by conventional annealing [2] [5] [25].

The temperature at which the ferromagnetic material becomes paramagnetic is known as Curie temperature. From the initial permeability μ' versus annealing temperature T curves, the Curie temperature (Tc) of the Nano-crystalline ribbons of the sample $Fe_{73.5}Cu_1Nb_3Si_{13.5}B_9$ has been determined. At fixed frequency (1 kHz), from the as-cast condition of $Fe_{73.5}Cu_1Nb_3Si_{13.5}B_9$ alloy, the temperature dependence of real part of the initial permeability has been presented in **Figure 2** in order to determine the Curie temperature of the sample. The ferromagnetic to paramagnetic transition of the amorphous phase can be characterized by Hopkinson effect and can be determined from the as-cast sample if the initial permeability passes through a maximum just before a sharp fall. Due to irreversible components of the structural relaxation like chemical short-range order, topological and long-range internal stress of the materials, it is very much difficult to determine the accurate Curie temperature, Tc of the amorphous materials. Curie temperature Tc may influence with this structural relaxation without destroying the amorphous state. So, the heating rate should be adjusted in such a way during the measurement of Tc that, no substantial relaxation takes place. **Figure 2** shows that, with increasing the annealing temperature the initial permeability of the nanocrystalline sample also increase. The maximum initial permeability μ' found at 350˚C and above the 350˚C annealing temperature, the permeability decrease rapidly. Thus Curie temperature has been estimated as 355˚C from the sharp fall of the initial permeability after 355˚C [26]. In the similar way, the various curie temperature of residual amorphous matrix are determined like 350˚C, 367˚C, 350˚C and 310˚C from the variation of μ' with temperature for the toroidal samples annealed at 400˚C, 450˚C, 490˚C and 525˚C respectively have been presented in **Figure 3**.

For as cast and annealed samples, real part of the complex initial permeability μ' has been presented in **Figure 4** up to f = 10,000 kHz. Magnetic initial permeability are measured in very low field of the toroidal shaped samples annealed at different temperatures, in order to correlate the soft magnetic properties of the alloys with microstructural features. From the domain wall mobility especially in the range of reversible magnetization, the magnetic properties of the soft magnetic materials are determined. **Figure 4** shows that the real part of initial permeability μ' decreases when the frequency increases and μ' becomes almost zero at very high frequency. This is because the very high frequency indicates the AC field and magnetization is opposed by AC field. As a result, at higher frequencies damping of the domain walls occurs and for this permeability drops to a very low value. This phenomenon defines the best frequency range at which a particular magnetic material can be used for practical application.

From the annealing temperature dependence of initial permeability and relative loss factor curve (**Figure 5**), the role of amorphous phase in intergrain coupling can be easily realize. The curve express itself strong depen-

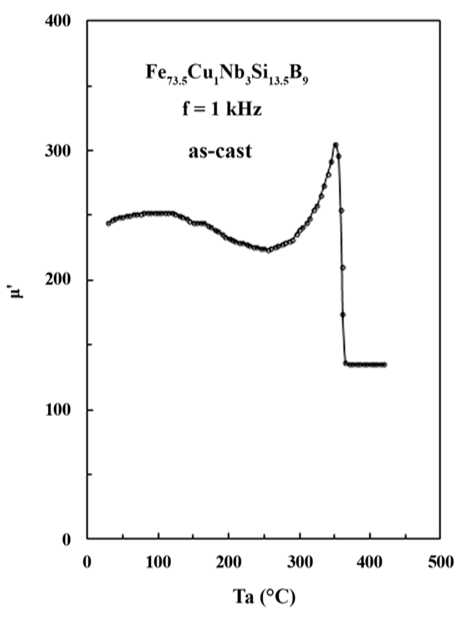

Figure 2. Variation of initial permeability μ' with temperature as-cast condition of $Fe_{73.5}Cu_1Nb_3Si_{13.5}B_9$ alloy at fixed frequency (1 kHz).

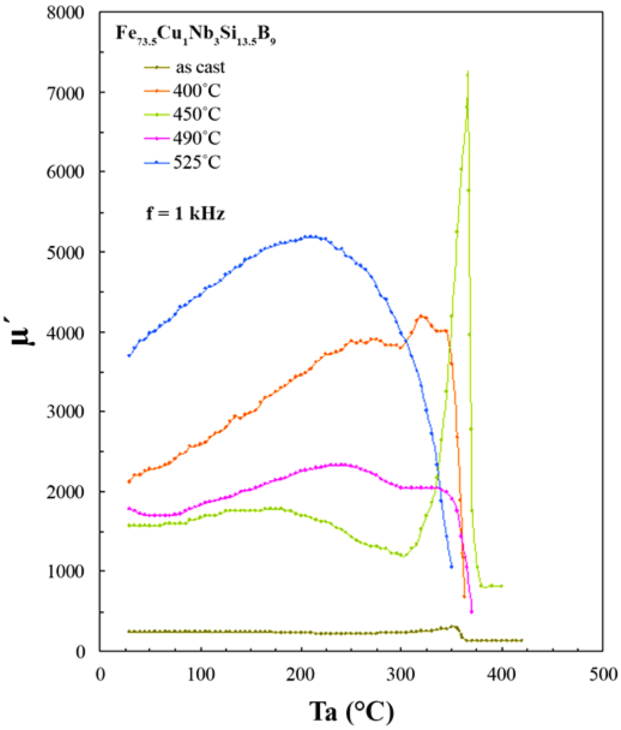

Figure 3. Variation of initial permeability μ' with samples annealed at different temperature for $Fe_{73.5}Cu_1Nb_3Si_{13.5}B_9$ alloy at fixed frequency (1 kHz).

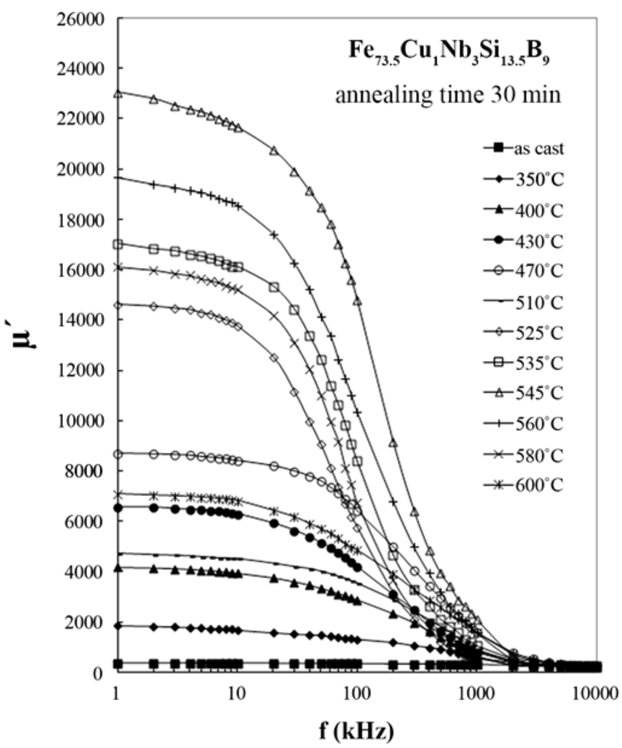

Figure 4. Frequency dependence of initial permeability μ' for $Fe_{73.5}Cu_1Nb_3Si_{13.5}B_9$ alloy at different annealing temperatures T (˚C) for 30 minute annealing time.

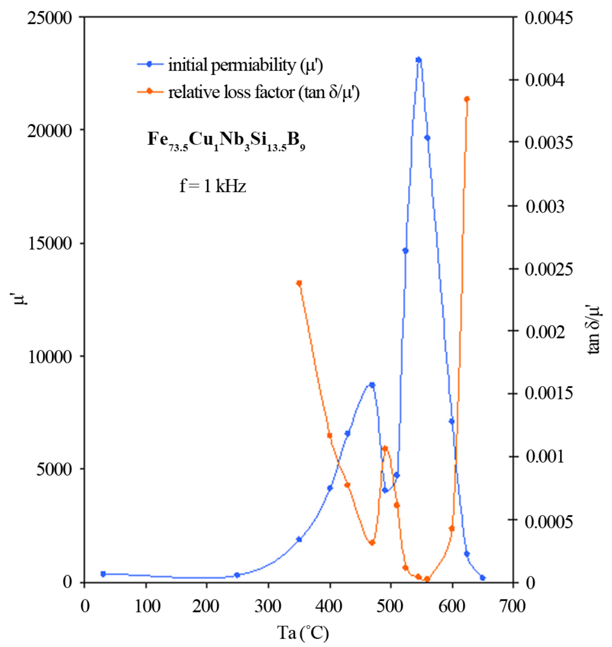

Figure 5. Variation of initial permeability μ' and of relative loss factor $\tan\delta/\mu'$ for $Fe_{73.5}Cu_1Nb_3Si_{13.5}B_9$ alloy at fixed frequency f = 1 kHz with various annealing temperature Ta.

dence of initial permeability on annealing temperature. Due to irreversible structural relaxation of the amorphous matrix, an increase of initial permeability with annealing temperature was observed when the sample annealed at temperature below the crystallization, *i.e.* up to 470˚C. Permeability decrease with the increase of annealing temperature up to 490˚C and beyond 490˚C annealing temperature *i.e.* from 510˚C to 545˚C a sharp increase of permeability was observed due to crystallization of α-Fe(Si) phase. For the annealing temperature of 545˚C, an enhancement of initial permeability by two orders of magnitude was observed (μ' = 23,065). From the other part of **Figure 5** it was observed that, the relative loss factor drops sharply as the annealing temperature increased and at annealing temperature 545˚C a minimum value relative loss factor (=4.002×10^{-5}) was found. Beyond this temperature μ' drops to lower value drastically and again increases very rapidly, as a result of magnetic hardening takes place [16].

At different frequency, the annealing temperature dependence of relative quality factor μ'/D and loss factor D of the samples are presented in **Figure 6** and **Figure 7** respectively. From the application point of view, for the soft magnetic materials both loss factor $\tan\delta$ or D and relative quality factors are the important parameters. Soft magnetic materials must have minimum loss factor and maximum relative quality factors. It is well-established that, minimum loss and very high relative quality factor with the order of magnitude 10^5 - 10^6 observed upon optimal annealing nanocrystalline alloys. By the successive annealing of the alloys from 350˚C to 600˚C, the optimal annealing temperature is determined. At high frequency the flux penetration becomes low, as a result the loss is mainly controlled by interaction between the grains of the alloy but at very low frequency the loss is controlled by hysteresis losses.

Measurement of a hysteresis loop is a very simple and fast method to characterize magnetic materials. The hysteresis curves and their properties such as the remanence (Br), coercivity (Hc), and hysteresis loss (W/kG) are very useful information from the application point of view. The B-H loops at room temperature of the investigated alloy $Fe_{73.5}Cu_1Nb_3Si_{13.5}B_9$ were measured at a constant frequency (f = 1 kHz), and the results are presented in **Figure 8**. From these loops, Br, W/kG, and Hc were determined. In **Figures 9(a)-(d)**, it can be observed that the magnetic flux density (Bv), remanence, coercivity and also hysteresis loss decrease with increase in annealing time, which implies good soft magnetic property of the alloy of $Fe_{73.5}Cu_1Nb_3Si_{13.5}B_9$ (**Table 1**).

5. Conclusion

Annealing at temperatures 545˚C for 30 min best soft magnetic properties has been achieved which corresponds

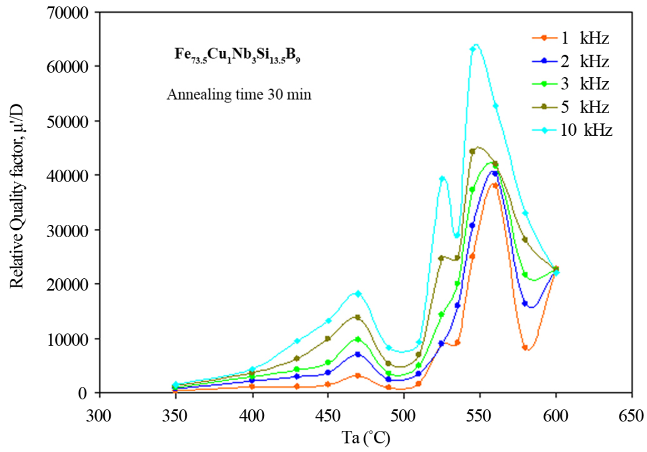

Figure 6. Temperature dependence of relative quality factor of $Fe_{73.5}Cu_1Nb_3Si_{13.5}B_9$ alloy with various frequencies.

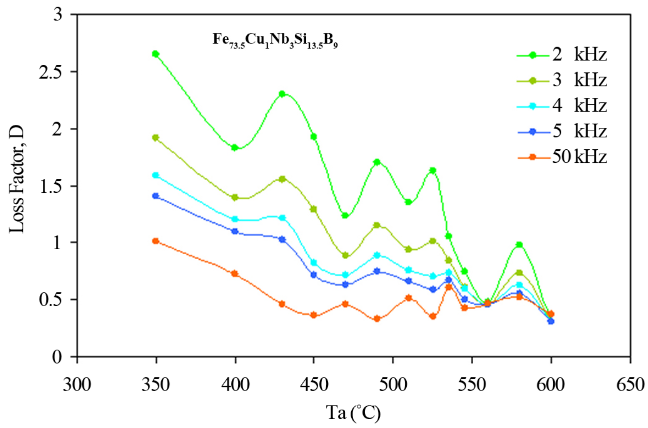

Figure 7. Temperature dependence of loss factor of $Fe_{73.5}Cu_1Nb_3Si_{13.5}B_9$ alloy with various frequencies.

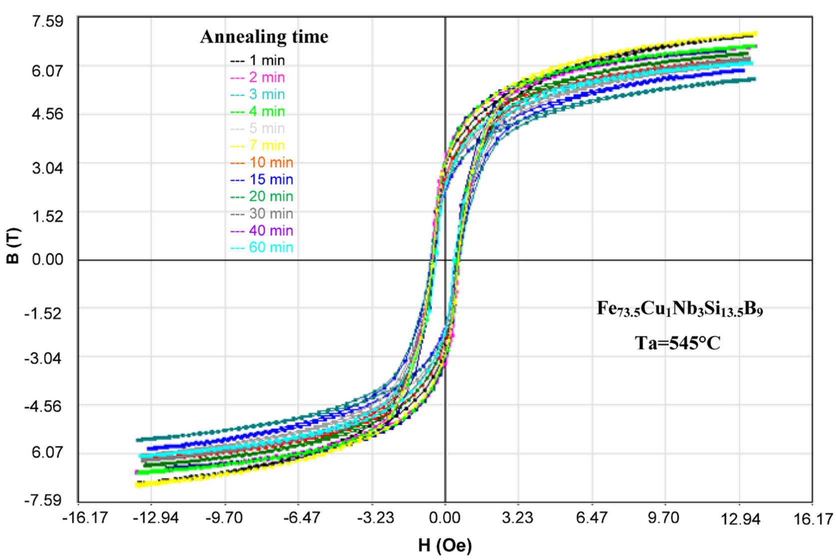

Figure 8. BH Loop of $Fe_{73.5}Cu_1Nb_3Si_{13.5}B_9$ alloy at constant B = 5 kG for the samples annealed at 545˚C for 1, 2, 3, 4,5, 7, 10, 15, 20, 30, 40, 60 minute.

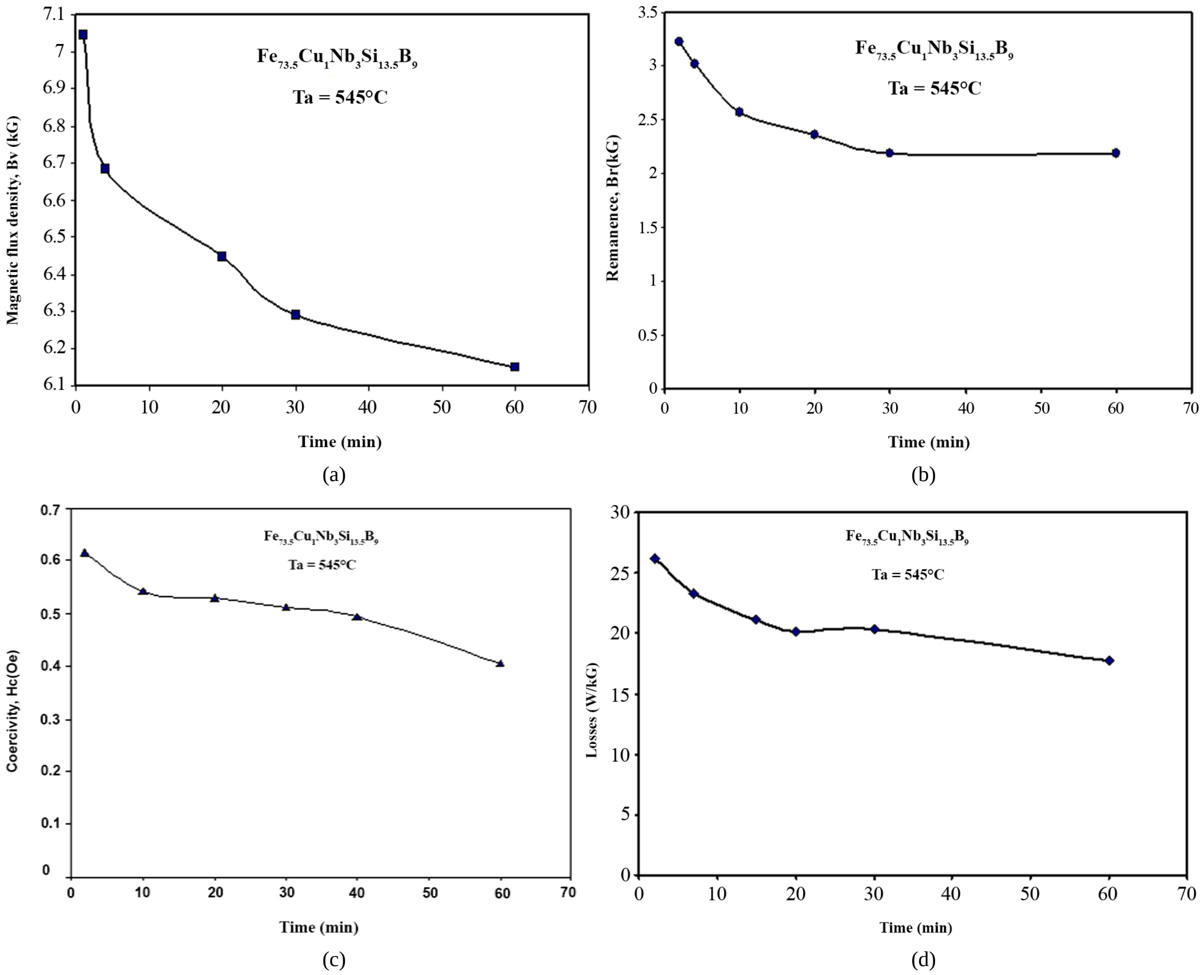

Figure 9. Annealing time (ta) dependence of (a) magnetic flux density (Bv) (b) remanence (Br) (c) coercivity (Hc) (d) loss (W/kG) of the sample annealed at temperature Ta = 545˚C.

Table 1. Annealing time dependence of magnetic flux density, remanence, coercivity and hysteresis loss for the samples annealed at temperature 545˚C at a fixed frequency 1 kHz.

Annealing Time (Ta)	Magnetic Flux Density (Bv)	Remanence (Br)	Coercivity (Hc)	Hysteresis Loss (W/kG)
2	6.674	3.224	0.616	26.234
4	6.683	3.021	0.509	22.832
30	6.291	2.184	0.513	20.378
60	6.150	2.183	0.406	17.752

to a maximum value of initial permeability (μ') which is 23,065 while the corresponding value of relative loss factor is 4.002×10^{-5} at a low frequency f = 1 kHz. At 545˚C, from magnetic hysterisisgraph we observe low core loss (26.234 - 17.752) W/kG, low remanence (3.224 - 2.183) kG and low coercivity (0.616 - 0.406).

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