MAGNETIC CHARACTERISATION OF AMORPHOUS AND NANOCRYSTALLINE MATERIALS

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ABSTRACT

Amorphous and nanocrystalline ferromagnetic materials have an important place among the metallic materials due to their unique and favourable association of mechanical, electrical and magnetic properties. The present paper deals with the magnetic properties like hysteresis, magnetostriction, domain wall movement and magnetoimpedance of these materials.

INTRODUCTION

The nanocrystalline and amorphous ferromagnetic alloys reveal much superior soft magnetic properties compared to their crystalline counterparts. The nanocrystalline ferromagnetic alloys introduced by Yoshizawa et.al [1] are the new generation of magnetic materials with excellent soft magnetic properties. Their high permeability and moderate saturation induction make them potential candidates for sensor application. They also exhibit ultra low coercivity (~mOe). However, evaluation of magnetic (coercivity, magnetostriction, anisotropy field and Barkhausen noise) needs special attention to establish their superiority to other materials.

PREPARATION OF MATERIALS

The material under study was Fe-Nb-Cu-Si-B ribbon prepared at the authors laboratory by melt spinning technique. Fig-1a shows the photograph of the melt-spinning unit. Melt spinning process parameters like speed of rotation, argon pressure, crucible dimensions were optimized to produce long uniform amorphous ribbons as shown in Fig.1b..

MAGNETIC CHARACTERISATION

Different techniques were used to characterise the various magnetic properties of amorphous and nanocrystalline soft magnetic materials. The characterisation techniques are discussed in the subsequent sections.

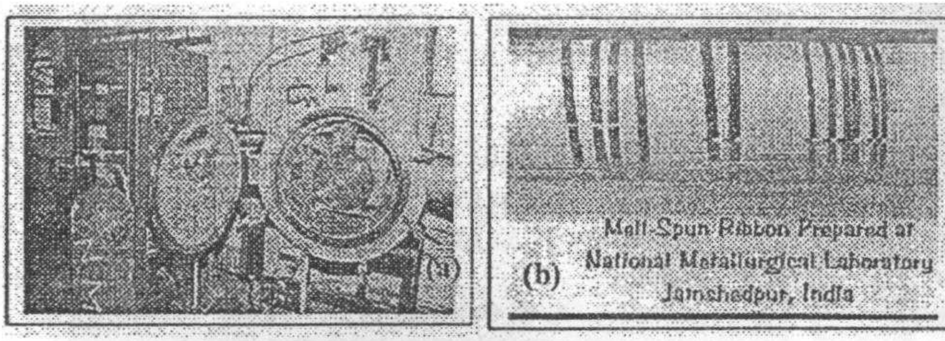


Fig. 1 : (a) The melt spinning apparatus for the preparation of amorphous ribbon. (b) The prepared ribbon at the laboratory

Magnetic hysteresis loop measurement

The conventional technique of hysteresis loop measurement involves magnetic testing in closed flux configuration using toroidal specimens. The ribbon shaped samples were ,therefore, tested by winding in the form of toroids. The primary and secondary windings were done for applying magnetic field and sensing the magnetic flux respectively. The tedious and long duration required for winding prolongs the characterisation time when the specimens are in large numbers. To get quicker results on hysteresis loop measurements, magnetic evaluation by fluxmetric method using Helmholtz Coils in open flux configuration was followed. Although this method of measurement is very convenient, error comes from the demagnetisation effect of the ribbon. The demagnetization field (H_d) caused by the free poles generated at the ends of the ribbon sample changes the applied magnetic field in open flux configuration of measurement of magnetic properties. Hence the properties depend on the dimension of the sample. Demagnetisation field effectively weakens the applied field (H_{app}) [2] and the effective field (H_{eff}) experienced by the sample becomes;

$$\begin{aligned} H_{eff} &= H_{app} - H_d \\ &= H_{app} - N_d M \end{aligned} \tag{1}$$

where N_d is the demagnetisation factor which depends on the sample dimension and M is magnetisation value measured at the applied field. The demagnetisation factor for ellipsoid has been extended for rod [3]. The demagnetisation factor of rods is given by

$$N_d = \frac{1}{K^2}[\log 2K - 1] \tag{2}$$

where K is the aspect ratio, that is the ratio of length to diameter.

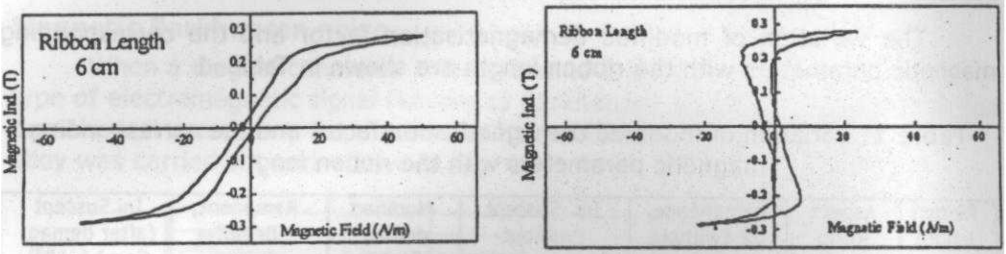
To calculate the demagnetisation factor for ribbon shaped sample, it can be considered to the first approximation as a rod with cross sectional area as that of ribbon and ribbon length equal to that of rod. With this assumption the aspect ratio becomes

$$K = \frac{L}{D} = \frac{L}{2\sqrt{A/\pi}} \qquad (3)$$

and,

$$N_d = \left[\frac{4A}{\pi L^2}\left[\log\frac{L}{\sqrt{A/\pi}} - 1\right]\right] \qquad (4)$$

The hysteresis loop of Fig. 2a of 6 cm long ribbon was corrected with the above N_d value (eq.4) and replotted in Fig. 2b.



(a) (b)

Fig. 2 : Hysteresis loop of 6 cm long ribbon (a) with demagnetisation correction and (b) after demagnetisation correction using eqn. 4

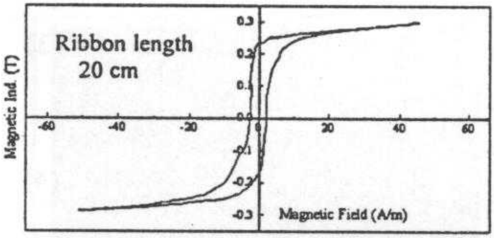
It was found that the calculated N_d using eqn.4 was too high for the hysteresis loop correction for the type of the samples under study. Hence, an empirical pre-factor (ϕ) which is less than one, has been multiplied with the calculated N_d of eq.4 to get modified demagnetisation factor (N_d^m).

With modified demagnetization factor, N_d^m, corrected hysteresis loop will be independent of ribbon dimension. Thus, eqn.1 becomes;

$$\begin{aligned} H_{eff} &= H_{app} - N_d^m M \\ &= H_{app} - \phi N_d M \end{aligned} \qquad (5)$$

To find the suitable empirical pre-factor, ϕ, hysteresis loop of a 20 cm long ribbon specimen was taken as a reference in the present study as its aspect ratio, L/D= 675.25 which was very high. The pre-factor ϕ was chosen in such a way that after demagnetisation correction the hysteresis loops for shorter ribbon of 6cm length (Fig-3b) would be similar to that of 20cm long ribbons (Fig-3a) and hence dimension independent. It was found that when ϕ = 0.65, the demagnetisation corrected hysteresis loop of 6cm long ribbon is similar to that of 20cm long ribbon. Similar corrections were valid for ribbons of different lengths. Thus, the modified demagnetisation factor becomes

$$N_d^m = 0.65 \times \left[\frac{4A}{\pi L^2}\left(\log\frac{L}{\sqrt{A/\pi}} - 1\right)\right] \qquad (6)$$



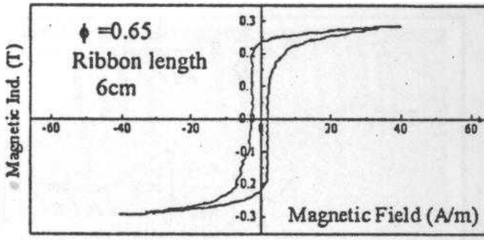


Fig. 3 : (a) Hysteresis loop of 20 cm long ribbon and (b) after demagnetisation correction using eqn. factor ϕ

The variation of modified demagnetisation factor and the corresponding magnetic parameters with the ribbon length are shown in Table -1

Table 1: Variation of modified demagnetisation factor and the corresponding magnetic parameters with the ribbon length

Ribbon length L (cm)	Aspect Ratio (L/D)	Remanence, Br (without demag. Corr.), T	Ini. Suscept (without Demag. Corr.) (×103)	Modified demag. Factor (0.65´Nd)	Remanenc, Br (after demag. Corr.) T	Ini.Suscept (after demag. Corr.) ($\times 10^3$)
6	202.58	0.03	4.29	7.9265	0.22	6.50
8	270.10	0.05	4.95	4.7149	0.21	6.45
10	337.63	0.07	5.33	3.1448	0.22	6.40
12	405.15	0.14	5.71	2.2561	0.22	6.55
16	540.20	0.17	5.94	1.3331	0.22	6.45
20	675.25	0.22	6.16	0.8850	0.22	6.52

The demagnetisation correction at an optimised empirical pre-factor of 0.65 gave consistent remanence and initial susceptibility values even for ribbons of different lengths. Using this prefactor magnetic coercivity of as-cast and heat-treated FeNbCuSiB alloy was determined from the demagnetisation corrected hysteresis loop. It was observed that optimum heat-treatment resulted in ultra low coercivity of 5mOe (0.32A/m) and very high susceptibility $\sim 10^5$

Magnetostriction coefficient

Another important magnetic property is the saturation magnetostriction constant (λ_s) which is the change in the length due to the presence of magnetic field. The conventional technique like strain gage, LVDT, three terminal capacitance probe are not suitable for the measurement of λ_s for the measured material, as it can not give sufficient force to activate sensors. The SAMR is determined by simultaneously applying a small a.c. transverse magnetic field (H_{ac}), saturating d.c. axial magnetic field (H_{dc}) and tensile stress to the samples [4]. The method is based on the detection of induced voltage V_{2f} (the second harmonic of the applied a.c. magnetic field) in a receiving coil set around the amorphous wire. When tensile stress s is applied on the wire (at fixed H_{ac} and H_{dc}), V_{2f} value increases or decreases as a consequence of the increment of magnetoelastic anisotropy having a transverse or an axial easy axis, respectively. The value of H_{dc} for which SAMR method is suitable, must be high enough so that $(V_{2f}H_{dc})^2$ constant and the magnetization saturation of the amorphous wire is achieved. The transverse magnetic field is obtained by passing an electric current through the wire. The current density was small enough to neglect the increase in temperature of the sample. The saturation magnetostriction constant was calculated from the following expression:

$\lambda_s = -(\mu_0\, Ms/3)(\Delta H_K / \Delta\sigma)$ for V_{2f} and H_{ac} = const.

Where Ms is the saturation magnetization of the sample.

Magnetic Barkhausen noise

When a magnetic material is subjected to a cyclic magnetic field, a burst type of electromagnetic signal (known as Barkhausen emissions) results from the domain wall movement. Barkhausen emissions of heat-treated Fe-Nb-Cu-Si-B alloy was carried out and shown in Fig. 4.

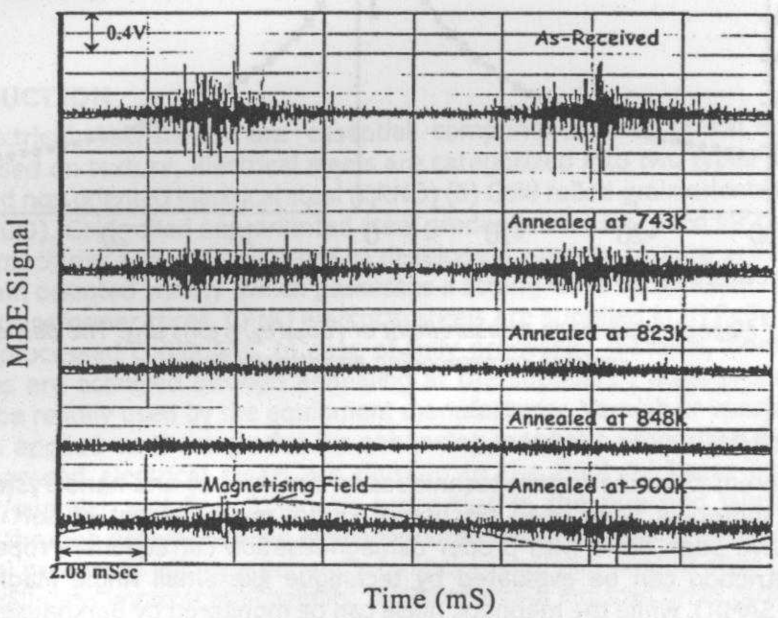


Fig. 4 : Barkhausen signal of as-received and heat treated material

It is interesting to note that the material showed minimum Barkhausen emissions at the nanocrystalline state where the soft magnetic properties of the materials are superior. The minimum barkhausen noise preferred for good magnetic sensor characteristics was obtained at suitable nanoparticle size obtained at optimum annealing temperature in the amorphous alloy.

Magneto-impedance

Now-a-days magnetoimpedance technique is also used to characterise amorphous and nanocrystalline magnetic materials which have very low anisotropy energy. It is a large variation of impedance of the material in presence of small DC magnetic field when high frequency alternating current passes through it [5]. The magnetoimpedance behaviour of the materials are used to evaluate the intrinsic magnetic properties like anisotropy field, permeability, susceptibility, etc. and are discussed in the paper. Fig-5 shows the estimation of circular anisotropy field from magnetoimpedance measurement for the $(Co_{94}Fe_6)_{72.5}Si_{12.5}B_{15}$. The DC axial field corresponding to the maximum MI ratio is attributed to the static circular anisotropy field H_k

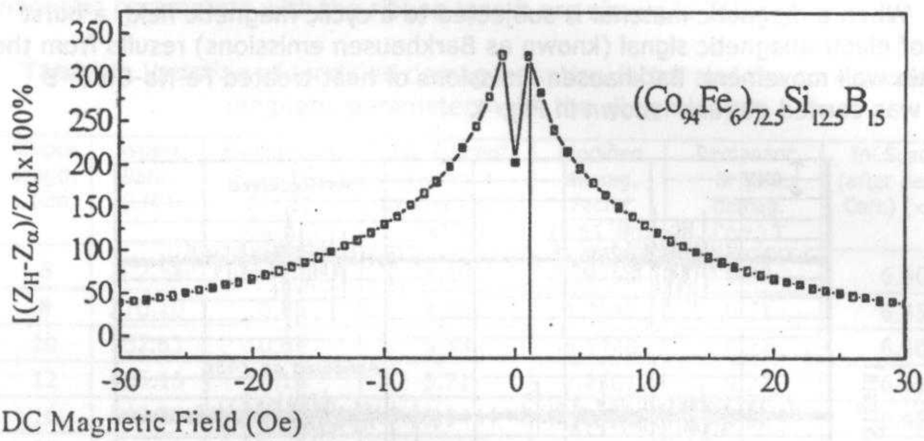


Fig. 5 : Magnetoimpedance measurement of $(Co_{94}Fe_6)_{72.5}Si_{12.5}B_{15}$. The dashed line indicate corresponding anisotrophy field H_k

CONCLUSION

Different characterisation techniques for amorphous and nanocrystalline soft magnetic materials have been discussed. Direct measurement of soft magnetic ribbons have been done with proper demagnetisation corrections. Properties like magnetostriction can be evaluated by technique like Small Angle Magnetisation Rotation (SAMR), while the magnetic noise can be monitored by Barkhausen signals. The intrinsic properties like anisotropy field, permeability etc can be inferred from the magneto-impedance behaviour of the materials.

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