

## Permeability and Hysteresis Loop Measurements of Amorphous and Annealed Samples of $\text{Fe}_{73.5-x}\text{Cr}_x\text{Cu}_1\text{Nb}_3\text{Si}_{13.5}\text{B}_9$ Alloys

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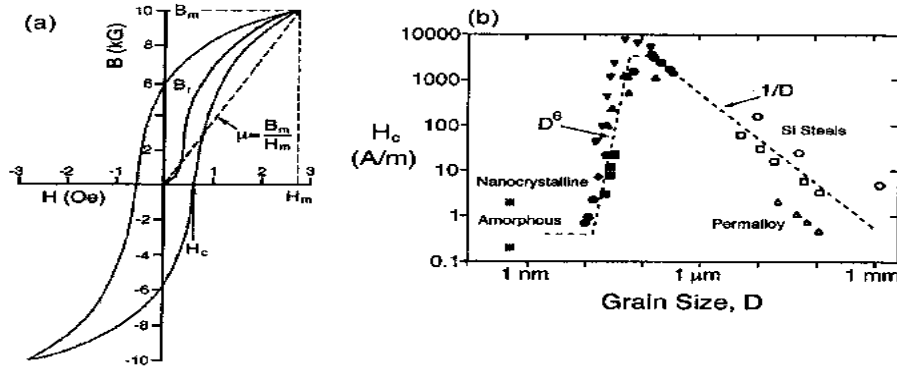
### Abstract

Permeability and coercivity are the most important parameters for the evaluation of soft magnetic materials. The criteria for the softest magnetic materials demand very high permeability and / or extremely low coercivity and these properties necessitate the anisotropy energy and the magnetoelastic energy tend towards zero. These unique demands are fulfilled when the FINEMET type of nanocrystalline materials are thermally treated around their primary crystallization temperature which facilitates the evolution of nanometric size of the Fe(Si) grains (10-12 nm) that are exchanged couple through the remaining thin residual amorphous interface. In order to correlate the microstructural features with soft magnetic properties of the alloys and initial permeability of the toroidal shaped samples annealed at different temperatures are measured at room temperature with an applied ac field of  $10^{-3}$  Oe. Magnetic hysteresis is a useful attribute of permanent magnetic material in which we wish to store a large metastable magnetization. On the other hand, a large class of applications requires small hysteresis losses per cycle. These include applications as inductors, low and high frequency transformers, alternating current machines, motors, generators and magnetic amplifiers. Present paper focuses on the measurement of permeability and hysteresis loops of samples both their amorphous and nanocrystalline states.

**Keywords:** Permeability, Coercivity, Magnetoelastic, Finemet, Nanocrystalline, Hysteresis, Etc.

## 1. Introduction

Magnetic hysteresis is a useful attribute of permanent magnet material in which we wish to store a large metastable magnetization. On the other hand, a large class of applications requires small hysteresis losses per cycle.



**Fig.1(a)** Hysteresis curve for a soft Fig.1 (b) Grain size (D) vs. coercivity ( $H_c$ ) curve  
Magnetic materials

These include applications as inductors, low and high frequency transformers, alternating current machines, motors, generators and magnetic amplifiers. The desired technical properties of interest for soft magnetic materials include [Fig.1 (a)].

- (i) **High permeability: Permeability:**  $\mu = \frac{B}{H} = (1 + \chi)$ , is the material's parameter which describes the flux density (B), produced by a given applied field (H). In high permeability materials, we can produce very large changes in magnetic flux density in very small fields.
- (ii) **Low hysteresis loss:** Hysteresis loss is the energy consumed in cycling a material between a field H and  $-H$  and then back again. The energy consumed in one cycle is  $W_h = \oint M dB$  or the area inside the hysteresis loop. The power loss of AC device includes a term equal to the frequency multiplied by the hysteretic loss per cycle. Also of concern at high frequencies are eddy current losses that are intimately related to the material's resistivity ( $\rho$ ).
- (iii) **Large saturation and remanent magnetizations:** A large saturation magnetization ( $M_s$ ), induction ( $B_s$ ) and small remanent magnetization ( $M_r$ ) are desirable in applications of soft magnetic materials.
- (iv) **High Curie temperature:** The ability to use soft magnetic materials at elevated temperatures is intimately dependent on the Curie temperature or magnetic ordering temperature of the material.

Conventional physical metallurgy approaches to improving soft ferromagnetic properties involve tailoring the chemistry and optimizing the microstructure. Significant in the optimizing of the microstructure is recognition of the fact that a measure of the magnetic hardness (coercivity,  $H_c$ ) is roughly inversely proportional to the grain size ( $D_g$ ) for grain sizes exceeding  $\sim 0.1-1 \mu\text{m}$  (where the grain size exceeds

the Bloch domain wall thickness ( $\delta_w$ ). In such cases grain boundaries act as impediments to domain wall motion, and thus fine-grained materials are usually magnetically harder than large grain materials. Significant recent developments in the understanding of magnetic coercivity mechanisms have lead to the realization that for very small grain sizes  $D < \sim 100$  nm [1, 2, 3-6],  $H_c$  decreases rapidly with decreasing grain size shown in Fig. 2.2 (b). This can be understood by the fact that the domain wall, whose thickness  $\delta_w$ , exceeds the grain size, now samples several (or many) grains so that fluctuations in magnetic anisotropy on the grain size length scale are irrelevant to domain wall pinning. This important concept suggests that nanocrystalline and amorphous alloys have significant potential as soft magnetic materials. Soft magnetic properties require that nanocrystalline grains be exchange coupled and therefore any of the processing routes yielding free standing nanoparticles must include a compaction method [9-11] in which the magnetic nanoparticles end up exchange coupled. Similar ideas have been stated for so called spring exchange hard magnetic materials [7-9].

Important microstructural features include grain size, shape and orientation, defect concentrations, compositional inhomogeneities, magnetic domains and domain walls. The interaction of magnetic domain walls with microstructural impediments to their motion is of particular importance to the understanding of soft magnetic properties. Extrinsic magnetic properties, which are important in soft magnetic materials, include the magnetic permeability and the coercivity, which typically have an inverse relationship. Remanant magnetization, squariness of the hysteresis loop and magnetic anisotropy (crystalline, shape or stress related) are also important in determining magnetic softness.

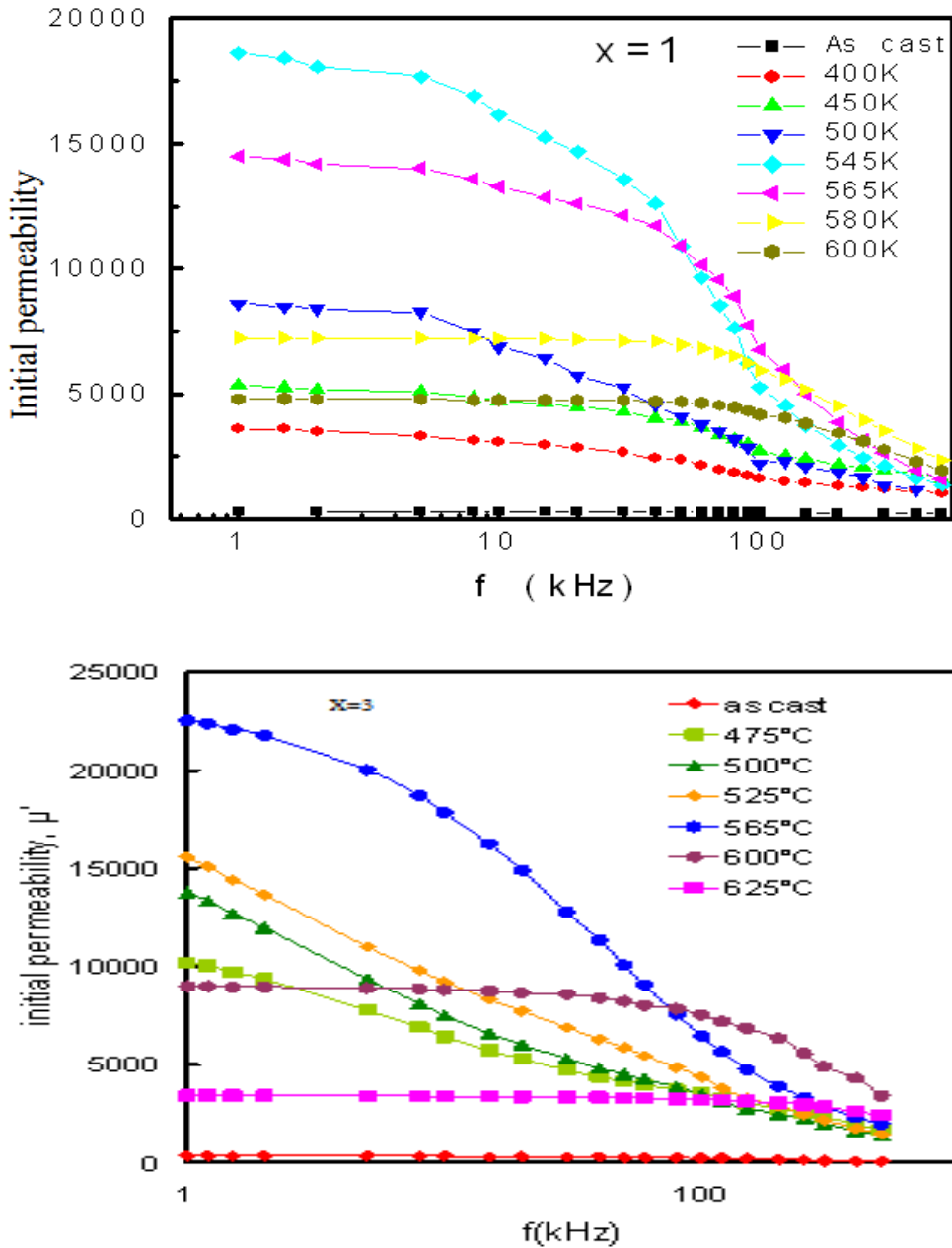
## **2. Experimental**

Ribbons of composition  $\text{Fe}_{73.5-x}\text{Cr}_x\text{Cu}_1\text{Nb}_3\text{Si}_{13.5}\text{B}_9$  alloys with  $x = 0, 1, 2, 3, 4, 5, 6, 7, 8, 9, 10, 12.5, 15, 17.5$  have been prepared by melt-spinning technique and heat-treated around the crystallization temperature. In the present paper complex permeability was measured by the LCR bridge method. The real ( $\mu'$ ) and imaginary ( $\mu''$ ) part of the complex permeability of the as-cast and annealed ribbons were measured as a function of frequency and temperature using Wayne Kerr 3255 B inductance meter and HP 4192 A impedance analyzer in conjunction with a laboratory built tubular furnace with continuous heating rate of  $\approx 5^\circ\text{C}/\text{min}$  with a very low ac driving field of  $\approx 10^{-3}$  Oe.

## **3. Results and Discussions**

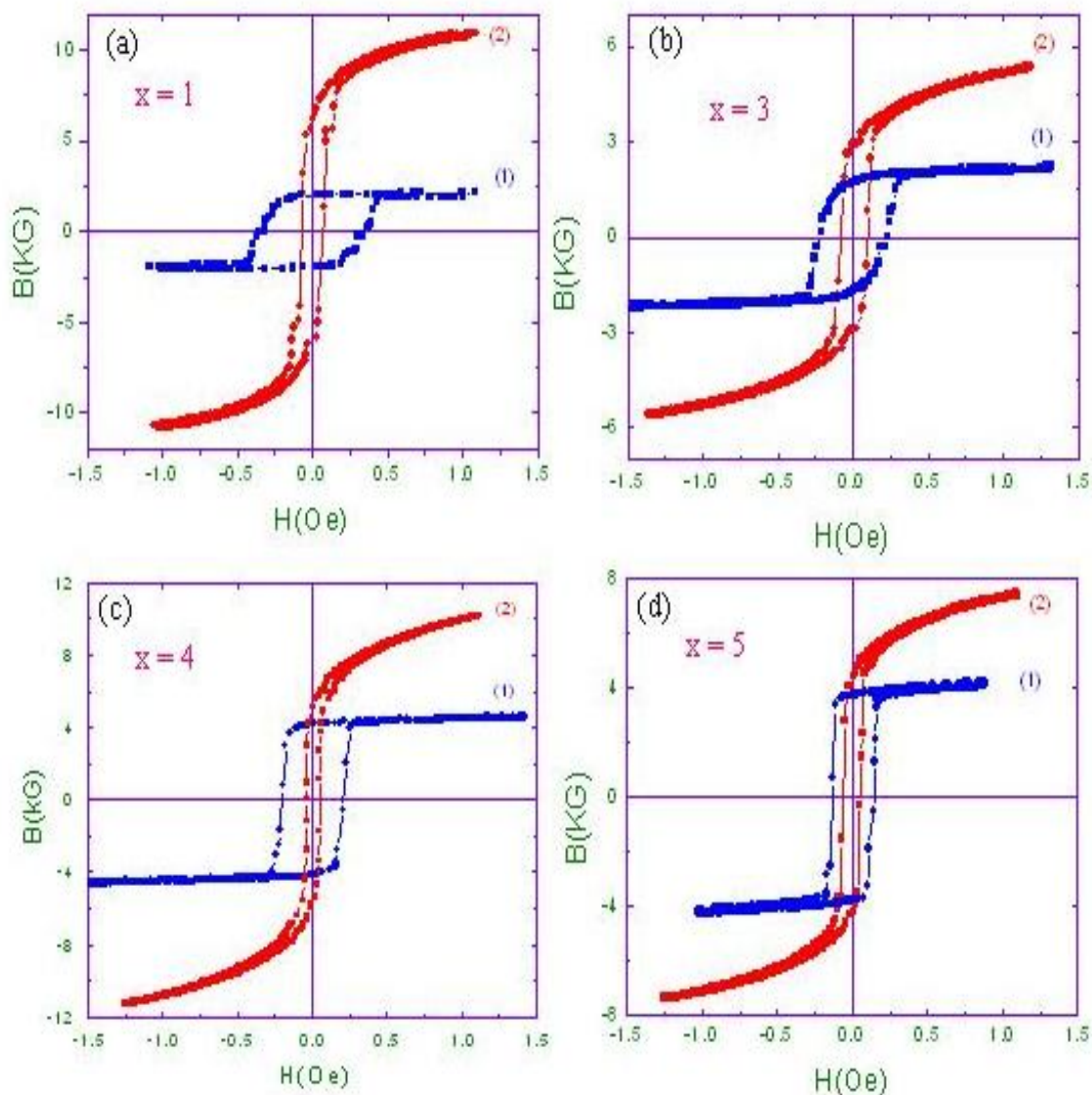
Figure 1 (a, b) shows the variation of initial permeability as a function of frequency from 1kHz-500kHz for the samples  $\text{Fe}_{73.5-x}\text{Cr}_x\text{Cu}_1\text{Nb}_3\text{Si}_{13.5}\text{B}_9$  with  $x = 1$  & 3 in the as cast and annealed at various temperatures up to  $625^\circ\text{C}$ . The magnetic properties of the soft magnetic materials are mainly determined by the domain wall mobility especially in the range of reversible magnetization. It is observed that  $\mu'$  increases with the

increase of annealing temperature  $T_a$  for both the sample  $x = 1$  & 3 attaining a maximum value of  $\mu'$  at  $T_a = 545$  and  $565^\circ\text{C}$  respectively, when  $T_a$  is increased beyond these temperatures,  $\mu'$  rapidly decrease to a very low value as indicated by both the samples. With the increase of annealing temperature,  $T_a$ ,  $\mu'$  increase rapidly in the low frequency region and also falls faster with simultaneous shift of the maxima of  $\mu''$  forwards lower frequency side.



**Fig. 1 (a, b).** Variation of initial permeability with frequency of the amorphous ribbon with composition  $\text{Fe}_{56}\text{Cr}_{17.5}\text{Cu}_1\text{Nb}_3\text{Si}_{13.5}\text{B}_9$  with  $x = 1$  and  $x = 3$  alloys

These effects are the typical features of domain wall relaxation dispersion. This signifies that the high permeability nanocrystalline materials are suitable for low frequency applications. All other samples show the similar frequency characteristics. The results show that the initial permeability of the amorphous sample has been enhanced by two orders of magnitude at low frequency when appropriately annealed.



**Fig. 2(a, b, c, d)** Hysteresis loops of amorphous ribbon with composition  $Fe_{73.5-x}Cr_xCu_1Nb_3Si_{13.5}B_9$  ribbons with  $x = 1, 3, 4$  &  $5$  in the as-cast (curve 1) and annealed (curve 2) at  $540^\circ C$  for 30 min.

The influence of partial substitution of Fe by Cr on the soft magnetic properties of  $Fe_{73.5-x}Cr_xCu_1Nb_3Si_{13.5}B_9$  ( $x = 1, 2, 3, 4$  &  $5$ ) have been studied through the measurement of magnetic hysteresis loops of amorphous and annealed samples at room temperature. Fig. 2 (a, b, c, d) shows low field ( $\approx 1.5$  Oe) hysteresis loop for the

samples  $x = 1, 3, 4, 5$ . It is clearly observed that the as cast amorphous samples have low magnetic induction  $B_{\max}$  value for all the samples which increases substantially on annealing at  $540^{\circ}\text{C}$  for 30 min. This annealing temperature is close to the onset of crystallization temperature.

Cr content x at. %	As Cast		Annealed at $540^{\circ}\text{C}$ for 30 min	
	$B_{\max}$ (KG)	$H_c$ (Oe)	$B_{\max}$ (KG)	$H_c$ (Oe)
1	2.0	0.35	10.8	0.069
3	2.3	0.23	5.8	0.080
4	4.5	0.20	10.2	0.054
5	4.2	0.14	7.7	0.057

Therefore an increase in magnetization  $M$  at room temperature is expected on annealed samples according to ref. [12, 13, 15]. With the increase of magnetic induction on annealing, a subsequent decrease of coercive force ( $H_c$ ) is noticed for all the samples implying magnetic softening of these alloys upon annealing. Amorphous alloys clearly show rectangular shaped hysteresis loops, the origin of which may be related to the magnetoelastic anisotropy contribution as a result of stress induced during rapid quenching process. But after annealing the shape of the hysteresis loops changes to normal form with the manifestation of enhanced soft magnetic properties i.e. high magnetic induction and low coercivity. Table-5.4 shows the value of  $B_{\max}$  and  $H_c$  for the samples annealed at  $540^{\circ}\text{C}$  for 30 min and as cast condition. The data shows that  $H_c$  decreases substantially on annealing and the maximum magnetic induction  $B_{\max}$ , at  $H \approx 1.5$  Oe also increases rapidly for all the annealed samples as compared with the amorphous precursor. Similar low field hysteresis behavior has been observed in Cr substituted FINEMET with Au instead of Cu [16]. The observed improvement of soft magnetic characteristic of the annealed samples at  $T_a = 540^{\circ}\text{C}$  for 30 min is likely due to the formation of  $\alpha\text{-Fe}(\text{Si})$  phase with optimum nanometric grains, their appropriate volume fraction and strong exchange coupling among them.

#### 4. Conclusion

An enhancement of initial permeability by two orders of magnitude and a subsequent decrease of relative loss factor have been observed for the optimum annealed samples. This outstanding soft magnetic properties of the studied samples have been achieved due to averaging out of the magnetic anisotropy energy by exchange interaction between the nanometric Fe(Si) grains with appropriate volume fraction transmitted through thin intergranular amorphous layer. Nanocrystalline FINEMET type of alloys offer the unique opportunity for tailoring ultrasoft magnetic properties that can be technologically exploited for the potential applications in many electronic and electrical devices. From the temperature dependence of permeability measurement, Curie temperature,  $T_c$  of the amorphous ribbons was determined. From the frequency dependence of complex permeability, evolution of permeability and magnetic loss component at different stages of nanocrystallization as affected by thermal treatment at different temperatures was determined using toroids prepared from the ribbons

wound with insulating Cu wire. The Wayne Kerr 3255 B inductance and HP 4192 A impedance analyzer directly measure the value of inductance, L and loss factor.

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