

## Field cooled and zero-field cooled magnetic effect on Cr-substituted Fe based amorphous $\text{Fe}_{73.5-x}\text{Cr}_x\text{Cu}_1\text{Nb}_3\text{Si}_{13.5}\text{B}_9$ alloys

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### Abstract

This paper focuses on the dependence of saturation magnetization and Curie temperatures as affected by Cr addition in the conventional FINEMET type of soft amorphous magnetic alloys with nominal composition  $\text{Fe}_{73.5-x}\text{Cr}_x\text{Cu}_1\text{Nb}_3\text{Si}_{13.5}\text{B}_9$  ( $0 \leq x \leq 17.5$ ). Cr substituted Fe based amorphous samples has been prepared by melt-spinning technique. The field cooled magnetization,  $M_{FC}$  and the zero-field cooled magnetization,  $M_{ZFC}$  was measured with an applied field of 1 Oe using SQUID magnetometer. The divergence between  $M_{FC}$  and  $M_{ZFC}$  were observed for the sample  $x = 10, 12.5, 15$  and  $17.5$ , which may be attributed to the magnetic hardening at low temperature. It has been observed that the saturation magnetization at 5 and 300K decrease linearly with Cr concentration for the entire composition range, which indicates that a simple dilution of the Fe moment takes place due to Cr substitution.

**Keywords:** Finemet, Magnetization, SQUID magnetometer, Field cool, Zero-field cool etc.

### INTRODUCTION

Magnetism is a discipline, which is stimulated by both basic and practical motivations for the study of different nanostructures. Those nanostructured materials are distinguished from conventional polycrystalline materials by the size of the crystallites that compose them. Defined broadly the term nanostructured is used to

describe materials characterized by structural features in the range of  $\approx 1-100$  nm corresponding typically to  $\approx 50-5 \times 10^7$  atoms respectively.

Nanoparticles are interesting from the fundamental point of view due to their extremely small size. The increasing surface to volume ratio with decreasing size results in an increased significance of the grain boundaries (i.e. surface energies) especially in the “real” nanometer range ( $\leq 10$  nm).

Magnetic nanoparticles show a variety of unusual magnetic behaviors when compared to the bulk materials mostly due to the surface / interface effects including symmetry breaking, electronic environment / charge transfer and magnetic interactions. Nanocomposite / nanocrystalline magnetic materials have been developed through appropriate heat treatment of the initial amorphous precursors of the Fe-Si-B and Co-Si-B based alloys for the ultra-soft magnetic properties with extraordinary high permeability and Fe-Nd-B based alloys for the spring exchange hard magnets with high energy product [1]. These technically important materials have enormous applications such as transformers, sensors for the soft and motors, actuators or generators for the hard nanocomposite magnetic materials have already been envisaged [2]

There are various methods of preparing nanoparticles and / or nanostructured materials which include: (i) plasma processing [3, 4] (ii) deposition technique [5, 6] and (iii) rapid quenching and subsequently crystallized to nanometric grains embedded in a still amorphous matrix [7].

In the present thesis work we are dealing with the third variant for the processing of nanocrystalline magnetic materials. In this method an amorphous precursor in the form of ribbons typically 20-30  $\mu\text{m}$  thick (metallic glass) is first obtained from the melt of the parent ingot with appropriate composition using a melt-spinning machine by rapid solidification technique. Since amorphous state is metastable, upon controlled crystallization above the crystallization temperature develops nanocrystalline grains dispersed in a residual amorphous matrix. Magnetic nanocomposites composed of nano-sized magnetic crystals of 10-15 nm embedded in an amorphous matrix have been shown to display excellent soft magnetic properties and found their applications in transformers inductive devices etc. [8, 9].

Amorphous materials are characterized by a lack of long-range atomic order, similar to that of liquid state. The lack of crystallinity causes amorphous materials to have a very low anisotropy based on random anisotropy model [11]. The lower anisotropy of the soft magnetic atoms allows that ferromagnetic exchange length to become larger. This enables a larger volume to be considered for randomizing anisotropy. In magnetic materials the ferromagnetic exchange length expresses the characteristic distance over which a magnetic atom influences its environment and the exchange energy starts to dominate over the anisotropy energy [12]. This characteristic distance is on the order of 100 nm. Thus if the magnetic materials have microstructure with grain diameters smaller than the ferromagnetic exchange length, it becomes possible to “average” the anisotropy of the grains to a very low bulk value by random walk consideration. Such a material then realizes the high saturation magnetization of the

crystalline state, low coercivity and high permeability due to randomized anisotropy. In the case of nanocrystalline soft magnetic materials with trade name FINEMET obtained from the careful control of crystallization of their amorphous precursor, it has been determined that an important averaging of the magnetocrystalline anisotropy over many grains with structural feature 10-15 nm coupled within an exchange length is the root of magnetic softening [8, 9, 10]. The fact that the ferromagnetic exchange length of this nanometric grain is typically  $\approx 35$  nm which illustrates the underlying importance of this length scale in this magnetic system.

## **EXPERIMENTAL**

Melt-spinning is a widely used production method for rapidly solidifying materials as well as preparing amorphous metallic ribbon [13, 14, 15]. In order to prepare amorphous of  $\text{Fe}_{73.5-x}\text{Cr}_x\text{Cu}_1\text{Nb}_3\text{Si}_{13.5}\text{B}_9$  alloys with  $x = 1, 2, 3, 4, 5, 6, 7, 8, 9, 10, 12.5, 15$  and  $17.5$ , a melt spinning facilities was used at the Centre for Materials Science, National University of Hanoi, Vietnam. The arc melted master alloy was crashed into small pieces and put inside the quartz tube crucible for re-melting by induction furnace using a medium frequency generator with a maximum power of 25 kW at a nominal frequency of 10 kHz.



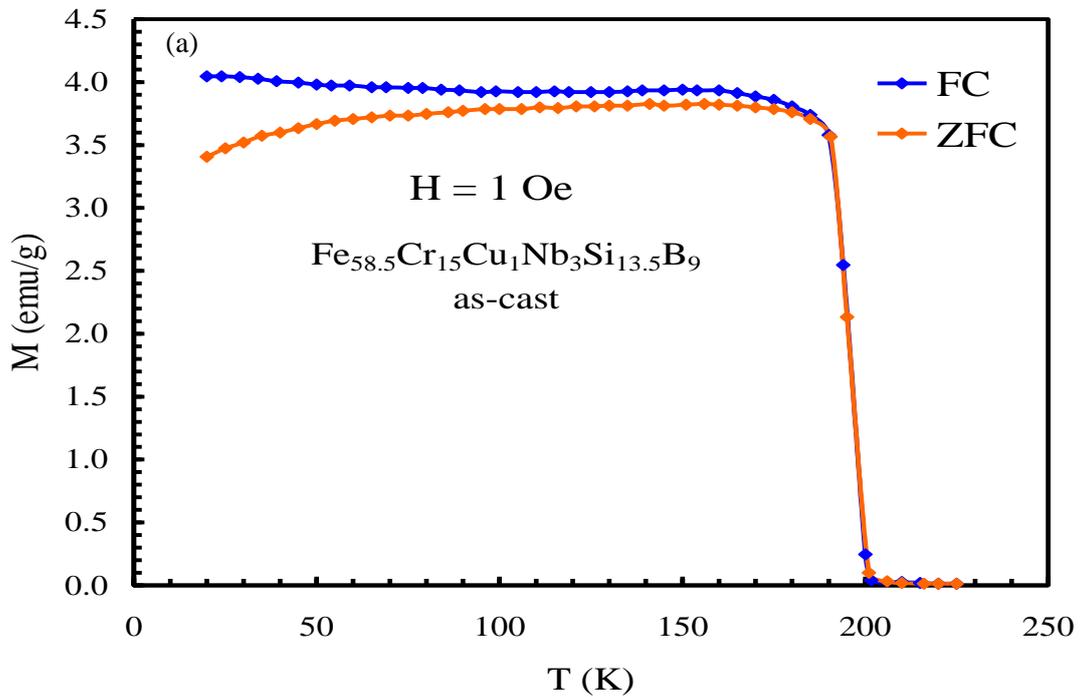
**Fig.1.** Melt-spinning Machine

Fig.1. shows the pictorial view of the melt-spinning machine. The quartz crucible has in its bottom part a rectangular nozzle tip of 8 mm length and 0.7 mm width. The position of the nozzle tip can be adjusted with respect to the copper wheel surface, so that the molten alloy was perpendicularly ejected onto the wheel surface from a distance of about 0.3 mm. The small pieces of the master alloy samples were inductively remelted inside the quartz tube crucible followed by ejecting the molten metal with an over pressure of 250 mbar of 99.9% pure Ar supplied from an external reservoir through a nozzle onto a rotating copper wheel with surface velocity of 30 m/sec. The temperature was monitored by an external pyrometer from the upper surface of the molten alloy through a quartz window. The metal alloys were ejected at a temperature of about 150-250 K above the melting point of the alloy. The resulting ribbon samples had thickness of about 20-25  $\mu\text{m}$  and width of  $\sim 6$  mm. Processing parameters such as the thermal conductivity of the rotating quench wheel, wheel speed, ejection pressure, thermal history of the melt before ejection, distance between nozzle of quartz tube and rotating wheel, as well as processing atmosphere have influenced on the microstructure and properties of melt-spun ribbons [16, 17, 18].

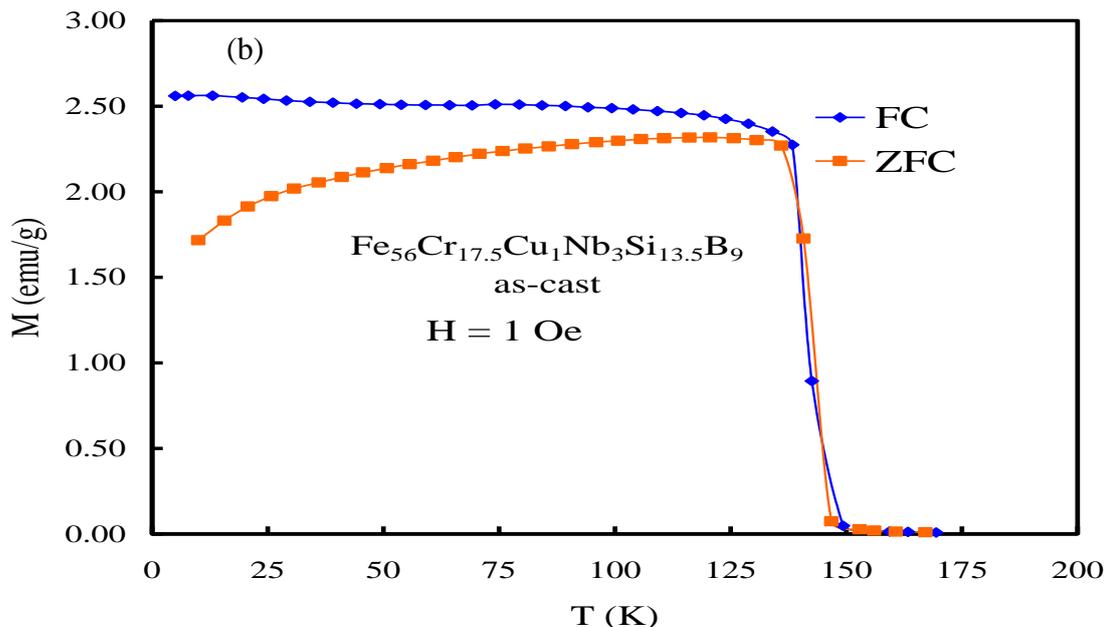
## RESULTS AND DISCUSSIONS

Effect of magnetic field on a ferromagnetic material is dependent on the state of ordering of magnetization. Zero-field-cooled magnetization curves as a function of field and temperature  $M_{ZFC}(H, T)$  of a magnetic material are the reflection of the domain structure of the individual material. When an ordered magnetic material is cooled in a zero external dc magnetic field from above its Curie temperature ( $T_c$ ) and magnetization is measured during heating the sample with the application of low dc magnetic field, zero-field-cooled magnetization ( $M_{ZFC}$ ) is obtained. In zero-field-cooled situation magnetic specimens are spontaneously magnetized under the influence of internal molecular field and associated competition between exchange energy, anisotropy energy etc. which determines the formation of domains. So,  $M_{ZFC}(T)$  gives the temperature dependence of virgin domain magnetization. In the field-cooled magnetization ( $M_{FC}$ ), the sample is cooled through the Curie temperature with the application of a magnetic field and the magnetization is measured during cooling it to the desired temperature keeping the magnetic field applied throughout the measurement. In the case of field-cooled magnetization ( $M_{FC}$ ) the external magnetic field greatly influences the domain formation since the magnetic moments are loosely coupled to each other above  $T_c$ . Therefore the virgin domain magnetization as effected by exchange and anisotropy energies is smeared out. It has been well demonstrated that  $M_{ZFC}$  curve below the Curie temperature is mainly determined by the magnetic anisotropy of the specific materials [19, 20] Therefore, it is expected that magnetically ordered materials will show divergence ( $M_{FC} \neq M_{ZFC}$ ) below the Curie temperature between FC and ZFC magnetization when measured with a magnetic field less than the anisotropy field ( $H_k$ ) associated with the specific material [21]. This is associated with a corresponding change in the temperature dependence of coercivity, which in turn is proportional to the temperature dependence of anisotropy field.

$M_{ZFC}$  and  $M_{FC}$  have been measured with an applied magnetic field of  $H = 1\text{Oe}$  for the samples  $x = 10, 12.5, 15$  &  $17.5$ . Fig. 2. (a) and 2. (b) shows the representative curves for  $x = 15$  &  $17.5$ . The divergence between the  $M_{ZFC}$  and  $M_{FC}$  just below the  $T_c$  is observed for all the studied alloys, which increases with the decrease of temperature and also with the increase of Cr content [22]. This phenomenon may be attributed to the increase of coercivity at low temperature, which is controlled by the magnetic anisotropy energy. It is noticed that the divergence between  $M_{FC}$  and  $M_{ZFC}$  at low enough temperature for  $x = 17.5$  is higher than that of  $x = 15$ . From these measurements, the sharp fall of magnetization at  $T = T_c$  is well demonstrated implying good homogeneity of the prepared samples. At the Curie temperature and above, the divergence between  $M_{FC}$  and  $M_{ZFC}$  is eliminated due to the vanishing of the magnetic anisotropy at  $T = T_c$  [23].



**Fig. 2. (a).** Field-cooled (FC) and Zero Field-cooled (ZFC) magnetization curves with an applied field of 1 Oe for the amorphous ribbon with composition  $\text{Fe}_{58.5}\text{Cr}_{15}\text{Cu}_1\text{Nb}_3\text{Si}_{13.5}\text{B}_9$



**Fig. 2. (b).** Field-cooled (FC) and Zero Field-cooled (ZFC) magnetization curves with an applied field of 1 Oe for the amorphous ribbon with composition  $\text{Fe}_{56}\text{Cr}_{17.5}\text{Cu}_1\text{Nb}_3\text{Si}_{13.5}\text{B}_9$

## CONCLUSION

The field-cooled ( $M_{\text{FC}}$ ) and zero-field-cooled ( $M_{\text{ZFC}}$ ) magnetization is a function of temperature for higher Cr content alloys, shows divergence below  $T_c$ . The divergence increases with the decrease of temperature and with the increase of Cr content in the amorphous alloys. This effect is associated with the increase of coercivity as the temperature is decreased and controlled by the temperature dependence of magnetic anisotropy energy.

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