Effect of annealing on specific magnetization of Fe-Cr-Nb-Cu-Si-B with the partial replacement of Fe by chromium

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Abstract

The samples Fe73.5-xCrxNb3Cu1Si13.5B9 [x = 7, 9, 10 and 12.5] are prepared in the amorphous state in the form of thin ribbons by rapid quenching technique at wheel speed of 25 m s⁻¹ in an Ar atmosphere. The composition was sintered at the temperature 450–8000 C for half an hour. The saturation magnetization (Ms) and Curie temperature (Tc) of these alloys decrease linearly with the increase of Cr content for the entire composition range due to dilution of Fe magnetic moment and weakening of exchange interaction between of magnetic atoms. The critical composition for disappearance of ferromagnetism fall of curve Ms with the replacement Fe by Cr, where the nearest neighbor coupling is longer dominant and intermediate range occur, giving rise to a significant portion of antiferromagnetic interaction. The Curie temperature decreases due the weaker interaction among the Fe magnetic moment. The structural relaxation is associated with the magnetization up to the annealed temperature 600 °C and the chemical disorderness arise with reference to enhancement of M of annealed samples. M versus H curves sharply rise which indicates the formation of crystallization and it seems to ferromagnetic and for x = 12.5 which is paramagnetic in the amorphous condition with Tc = 246 K. This increase of M for the four samples are due to the evolution of ferromagnetic α-Fe (Si) nanograin crystal.

Introduction

In 1988, Yoshizawa, Oguma and Yamauchi developed first FINEMET at Hitachi Metals Ltd from Fe-Si-B alloys where Cu and Nb were added [1]. Magnetically soft materials are needed for the applications of magnetic devices such as transformer, inductive devices, and biomedical applications space applications and many more other applications. Nowadays, researchers are thinking that the use of nanomaterials can give the hand of God to the man. By using the nanoscience and technology, it is possible to solve many unsolved real life problems such as the fatal diseases like cancer. Till date researchers are working on to synthesize the FINEMET matrix with the partial replacement of Fe by chromium. For enhancing the magnetic properties of the FINEMET, Ga is seen to add with FINEMET matrix [3]. By studying the surface morphology of the nanocrystalline precursors through melt-spinning process a substantial development of soft magnetic properties is developed [19, 20]. This is due to impart of the properties common to...
both amorphous and crystalline materials and the ability of these alloys to complete with their amorphous and crystalline counterparts. The benefits found in the nanocrystalline alloys stem from their chemical and structural variations on a nano properties.

At the present work the effect of sintering temperature on the magnetization process of Fe-Cr-Nb-Cu-Si-B amorphous precursors is studied thoroughly. The intrinsic magnetic properties including Curie temperature, saturation magnetization, and magnetostriction are influenced by the annealing effect. In the amorphous state of the FINEMET alloys, M_s and T_c increases at the early stage due to the annealing after that the M_s and T_c both decrease [21, 22]. This provides information about the nature of residual strain in as prepared melt-spun ribbons and their effect on domain wall pinning. This study also provides important technical information about the possibility of using ribbons at elevated annealing temperature and the optimum operating points of these ribbons; when they are used as soft magnetic materials under varying fields. The detailed quantitative analysis of the situation is, therefore, very complex and present understanding of the problem of magnetization process as affected by these defects is not yet clear. For this reason researchers got the opportunity to study the interaction of magnetization in broader range of temperature between the Curie temperature of the amorphous phase as well as nanocrystalline phase. In this article, the magneto crystalline higher content Cr substituted Fe-based metallic glasses under optimum heat treatment is the subject of intensive research not only for the promising technological applications but the coexistence of various magnetic phases at elevated temperature makes them attractive for studying basic magnetic phenomena.

**Materials and method**

The Fe_{73.5-x}Cr_xNb_3Cu_1Si_{13.5}B_9 [x = 7, 9, 10 and 12.5] amorphous precursors were prepared in a pure Argon atmosphere in an arc furnace on a water-cooled copper hearth. The materials used in the experiment were Fe (99.9%), Cr (99.9%), Nb (99.9%), Cu (99.9%), Si (99.9%) and B (99.9%). The purity level were justified from Johnson Mathey (Alfa Aesar Inc.). The mixing ratios were taken from flakes and measured sincerely with a sensitive digital balance and kept on the copper hearth inside the arc furnace. The furnace was evacuated and cleaned with Ar gas. It was repeated and lastly the Ar atmosphere was kept inside the chamber of the furnace.

**Melt spinning technique**

The Fe_{73.5-x}Cr_xNb_3Cu_1Si_{13.5}B_9 [x = 7, 9, 10 and 12.5] alloys were synthesized using Melt spinning technique [23, 24] at the Centre for Materials Science, National University of Hanoi, Vietnam. The lower part consists of quartz crucible.

The small piece of the master alloy samples were inductively remelted inside the quartz tube crucible. The melting metal were expelling using the over pressure 250 mbar at the pure Argon environment of purity level 99.9% from an external reservoir through a nozzle onto a rotating copper wheel with surface velocity of 30 m/s. The pyrometer is used externally to monitor the temperature through a quartz window. Above the melting point of the alloys the metal alloys were ejected and the temperature range was nearly about 150 to 250 K.

The thickness of the resulting samples was nearly 20–25 μm and width ~6 mm. The microstructure and properties of melt-spun ribbons has an effect on the thermal conductivity of the rotating quench wheel, wheel speed, ejection pressure, thermal history of the melt before ejection, distance between nozzles of quartz tube and rotating wheel, as well as processing atmosphere have been influenced. The lower pressure of 250 mbar as mentioned above stabilizes the turbulence between melt pull and rotating copper wheel enhancing the heat transfer resulting in a more uniform quenching. As a result, a more uniform ribbon microstructure can be obtained at relatively low wheel speed. With increasing wheel speeds for a given ejection rate, the increasing extraction rate results in thinner ribbons.

**Results and discussion**

**Specific magnetization measurement of amorphous precursors**

A vibrating sample magnetometer [25] was used to measure the magnetization of amorphous precursors Fe_{73.5-x}Cr_xNb_3Cu_1Si_{13.5}B_9 [x = 7, 9, 10 and 12.5] as a function of magnetic field. The magnetometer has no effect of the low susceptibility of the sample. The sample is vibrated up and down in an area surrounded by several pick up coils. The proportionality constant accounting for the particular coil geometry and susceptibility is obtained by calibration with a high purity circular disk shaped Ni-sample. The specimens were cut in to tiny shapes, weighed and glued to a standard sample holder.
Specific magnetization at room temperature

Specific magnetization of the specimen amorphous precursors \( \text{Fe}_{73.5-x}\text{Cr}_x\text{Nb}_3\text{Cu}_{13.5}\text{Si}_9 \) with \( x = 7, 9, 10 \) and 12.5 of quenched condition is measured, using a vibrating sample magnetometer. The magnetization process of the amorphous precursors with different field are shown in figure 1. With increasing of substitution Fe by Cr the specific magnetization decreases. On the contrary, an applied field of 8 kOe magnetization measurement are saturated at room temperature of the samples the sample with \( x = 7, 9 \) and 10 and for the value of \( x = 12.5 \) of Cr the ferromagnetic nature is almost absent instead of minor value of magnetization is observed linearly upto the applied field 10 KOe.

It is found that with increasing Cr the \( M_s \) decreases. For the higher percentage of Fe the Ms for these ribbons have higher values. This is quite understandable from the consideration of higher contribution of magnetic moments in Fe-rich ribbons. It is observed that while the ribbon with composition \( \text{Fe}_{66.5}\text{Cr}_7\text{Nb}_3\text{Cu}_{13.5}\text{Si}_9 \) reaches its saturation value around 5 kOe, \( \text{Fe}_{64.5}\text{Cr}_9\text{Nb}_3\text{Cu}_{13.5}\text{Si}_9 \) requires 7 kOe and \( \text{Fe}_{63.5}\text{Cr}_{10}\text{Nb}_3\text{Cu}_{13.5}\text{Si}_9 \) requires 4.5 kOe. Magnetization is also evaluated as a function of field to find the dependence of magnetization on the domain structure.

Due to the dilution of Fe by incorporation of Cr the magnetization, \( M \) decreases with increasing Cr-content as shown in figure 2. \( M_s = 160 \text{ emu g}^{-1} \) at room temperature for \( x = 0 \) which decreases monotonically with the successive replacement of Fe by Cr and reached a value of \( M_s = 55.41 \text{ emu g}^{-1} \) at room temperature for \( x = 10 \).

The saturation magnetization value \( M_s \) for the original FINEMET alloy i.e. \( x = 0 \) was found to be comparable within an experimental error of \( \pm 5\% \) by several investigators [26].

Several researchers showed that for the original FINEMET the saturation magnetization is comparable [26] within an experimental errors \( \pm 5\% \).

Figure 1. Field dependence of magnetization of amorphous precursors \( \text{Fe}_{73.5-x}\text{Cr}_x\text{Nb}_3\text{Cu}_{13.5}\text{Si}_9 \) with \( x = 7, 9, 10 \) and 12.5 at room temperature.

Figure 2. \( \frac{dM}{dT} \) versus temperature curve of amorphous precursors.
Magnetic saturation can be achieved only for Fe-based alloys. In this case, the total anisotropy is small and the next nearest neighbor exchange coupling leads to ferromagnetic order. The critical composition for the disappearance of ferromagnetism full of curve Ms with the replacement Fe by Cr, where the nearest neighbor coupling is no longer dominant and an intermediate range occur, giving rise to a significant portion of antiferromagnetic interaction.

When same composition of the different materials are produced using different quenching rate, magnetic properties, in particular the Tc can vary substantially which prevents a reliable comparison between results obtained different laboratories by the researchers. It is found that for the original FINEMET Tc is varied from 590 K to 630 K. Therefore the initial amorphous state should be taken into account non-magnetic Cr may be attributed to this simultaneous weakening of the strength of exchange interaction between the Fe magnetic moments. In spite of careful determination of Tc of the amorphous alloys by various methods ambiguity of Tc values still remains an open question especially for the amorphous glassy metal alloys are basically metastable materials. It has been demonstrated by numerous experimental evidences that when the materials with the same composition are produced with different quenching rate, magnetic properties, in particular the Tc can vary substantially. This fact prevents a reliable comparison between results obtained different laboratories [27]. The Tc of original FINEMET has been found to vary from 590 K to 630 K. Therefore the initial amorphous state should be taken into account.

From the above measurements it has been elucidated that the thermomagnetic measurement is a powerful technique to analyze the crystallization behavior of amorphous ferromagnetic materials provided the crystallization products are ferromagnetic and the Tc of amorphous alloys lie below the crystallization temperature.

**Variation of specific magnetization with isothermal annealing of higher Cr content amorphous precursors**

This is well known that amorphous state is metastable. Metastability of amorphous or glassy metal alloys offers the possibility of phase separation diffusion of various species and structural relaxation even through the alloys remains amorphous when they are annealed at temperature well below the crystallization temperature. The specific magnetization of the amorphous precursors Fe73.5-xCrxNb3Cu1Si13.5B9 [x = 7, 9, 10 and 12.5] alloys annealed for constant 30 min annealing time at varying temperature 500 °C to 600 °C have been measured as a function of magnetic field generated by an electromagnet using (VSM) Table 1.

We can distinguish reversible and irreversible types of relaxation due to annealing temperature. Irreversible types of relaxation are these which are connected with thermally initiated microscope jumps of defects or ordering atomic pairs which correspond to irreversible domain wall movement under external field [28, 29]. Thus reversibility of magnetization is not possible in these cases by reversing the external field. The reversible relaxation on the other hand means microstructural atomic rearrangement within the domain wall potential in a way that allows the reversal of the magnetic domain wall movements through retreating of the magnetic field. The present work will be confined to the later situation only. The criteria for the softest magnetic materials demand very high magnetization, high permeability and/or extremely low coercivity and these properties necessitate the anisotropy energy and the magnetoelastic energy and 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 (11–16 nm) that are exchanged couple through the remaining thin residual amorphous interface. The present results are interpreted in terms of conventional domain theory of ferromagnetization, where it is postulated that the effect of annealing temperature is to partially remove the pinning centers of the domain wall and thereby improving the magnetic softness of these ribbon.

**Effect of annealing temperature on specific magnetization at room temperature**

At the room temperature the FINEMET is ferromagnetic in nature. It has been observed in the table 2. Present investigations those amorphous precursors Fe73.5-xCrxNb3Cu1Si13.5B9 [x = 7, 9, 10 and 12.5] at room temperature show an increase of magnetization (M) at room temperature when annealed below the onset

| Cr-content, x at% | Saturation Magnetization Ms in emu/s at room temperature | Magnetization M in emu/s at 10 kOe | CurieTemp. °Tc in K | CurieTemp. °Tc in K |
|-------------------|----------------------------------------------------------|-------------------------------|-------------------|-------------------|
| x = 7             | 71.52                                                    | 71.52                         | 410               | 137               |
| x = 9             | 59.12                                                    | 59.12                         | 372               | 99                |
| x = 10            | 55.41                                                    | 55.41                         | 366               | 93                |
| x = 12.5          | —                                                        | 11.12                         | 246               | 28                |

**Table 1. Curie temperature and saturation magnetization of amorphous precursors of Fe73.5-xCrxNb3Cu1Si13.5B9.**
crystallization temperature. Ms at room temperature has been measured annealed samples. The annealing has been out for 30 min at Ta = 500 °C to 600 °C. Figures 3(a)–(d) shows the field dependence of M for nanocrystalline amorphous ribbon and thereby treated samples measured by VSM. From the curves it is clearly evidenced that the M is saturate for all samples in the applied field 10 kOe. Maximum Ms is reached at 600 °C for the all samples. It can be seen that clearly demonstrates an increase of magnetization upon Ta for all these four samples. Aranda et al [29] have studied the approach to saturation in nanocrystalline FINEMET materials. The magnetization prior to saturation is associated with reversible rotation and has been fitted to the:

\[ M(H) = M_s [1 - a_1/H - a_2/H^2] + bH^{0.5}, \]

where the term \( a_2/H^2 \) was described being a direct consequence of the random anisotropy model and attributable to Fe-Si grains.

The co-efficient \( a_2 \) reflects the Herzer’s predicted effective magnetic anisotropy of the nanocrystalline material, whereas in amorphous alloys it is postulated as being caused by local stress and magneto elastic coupling Ms. It is to be noted that an increase in Ms due to structural relaxation has also been detected in Fe based metallic glass [30], with reference to the enhancement of magnetization of annealed samples. DTA thermograms have been taken on samples annealed at Ta = 500 °C to 600 °C along with the as-cast samples. Ms increases substantially only up to 600 °C which is close to the onset of crystallization temperature (\( T_{x1} \)) for these alloy according to DTA data of table 2. The amorphous precursors of Fe_{73.5-x}Cr_xNb_3Cu_1Si_{13.5}B_9 [\( x = 7, 9, 10 \) and 12.5] has been paramagnetic at room temperature shown in figure 3(d). The area under the first crystallization event Fe(Si) phase slightly diminishes for \( x = 10 \) and 12.5 implying that initiation crystallization seems to take place and accordingly M versus H curves sharply rises and look like ferromagnetic for \( x = 12.5 \) which is paramagnetic in the amorphous condition with \( T_c = 246 \, K \). This increase of magnetization for the sample \( x = 12.5 \) is due to the evolution of ferromagnetic Fe(Si) crystallites.

| Cr–Content x at % | Ms at room temperature in emu/g | Ms at Ta = 500 °C in emu/g | Ms at Ta = 550 °C in emu/g | Ms at Ta = 600 °C in emu/g |
|-------------------|--------------------------------|-----------------------------|-----------------------------|-----------------------------|
| x = 7             | 71.52                          | 86.25                       | 96.62                       | 99.86                       |
| x = 9             | 59.20                          | 67.81                       | 73.38                       | 89.76                       |
| x = 10            | 55.41                          | 63.02                       | 68.91                       | 85.43                       |
| x = 12.5          | 11.12                          | 23.68                       | 33.72                       | 47.86                       |

Figure 3. (a) Field dependence of specific magnetization curves of amorphous precursors of Fe_{73.5-x}Cr_xNb_3Cu_1Si_{13.5}B_9 in the as-cast and different annealed samples. (b) Field dependence of specific magnetization curves of amorphous precursors of Fe_{73.5-x}Cr_xNb_3Cu_1Si_{13.5}B_9 in the as-cast and different annealed samples. (c) Field dependence of specific magnetization curves of amorphous precursors of Fe_{73.5-x}Cr_xNb_3Cu_1Si_{13.5}B_9 in the as-cast and different annealed samples. (d) Field dependence of specific magnetization curves of amorphous precursors of Fe_{73.5-x}Cr_xNb_3Cu_1Si_{13.5}B_9 in the as-cast and different annealed samples.
An increase of Ms for the annealed samples at 500 °C to 600 °C compared with amorphous state is due to the irreversible structural relaxation, changing the degree of chemical disorder of the amorphous state \([31]\) and enhanced volume fraction of Fe Si nanocrystals that are exchange coupled. The \(M_s\) are shown in table 2.

The magnetization of the specimen at \(T_a = 500 ^\circ C, 550 ^\circ C, 600 ^\circ C\) of the studied samples is nearly zero since they are paramagnetic in this temperature range shown in figures 3(a)–(d). At \(T_a = 410 ^\circ C\) (shown in table 2) above the crystallization behavior of the amorphous samples has been well demonstrated as the temperature continue to rise other. The sharp rise of magnetization of all the annealed amorphous nanocrystalline samples in their paramagnetic state is connected to the onset crystallization of \(\alpha\text{–Fe}(Si)\) ferromagnetic phase in the remaining amorphous matrix. The decrease of \(M\) after passing through the maximum is \(M_s\) connected to the Ta of magnetization of \(Fe\) (Si) crystallization phase which under goes a FM-PM phase at \(T_a = 600 ^\circ C\). It indicates that the crystallization \(Fe(Si)\) phase is completely suppressed for higher Cr content alloy which has also confirmed.

Conclusion

To find out the correlation between microstructural features and magnetic properties dependent on various stages of nanocrystalline during the isothermal annealing around the crystallization temperature of their amorphous precursors, FINEMET family of the specimen \(Fe_{73.5-x}Cr_xNb_3Cu_1Si_{13.5}B_9\) has been investigated here. After the systematic investigation on the crystallization, structural and magnetization of the present system few conclusions can be reached. With increasing the Cr at the room temperature the saturation magnetization of all the specimen decreases. For the Fe based alloys saturation magnetization is achievable. In this study, it is found that \(Fe_{61}Cr_{12.5}Nb_3Cu_1Si_{13.5}B_9\) is paramagnetic at room temperature. The Curie temperature of interfacial amorphous phase has been found to decreases as the Cr-content increasing implying the effect of Cr on the dilution of magnetic moment as well as the weakening of exchange interaction between Fe magnetic moment. Magnetization \((M)\) of the amorphous state increases with the increases of annealing temperature corresponding to the early stage of crystallization due to irreversible structural relaxation. At the room temperature, high Cr \((x = 12.5)\) content amorphous matrix is paramagnetic in nature are found to increase substantially when annealed below the crystallization temperature.

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