Thermal stability and magnetic properties of partially Co-substituted $(Fe_{71.2}B_{24}Y_{4.8})_{96}Nb_4$ bulk metallic glasses

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Abstract

The influence of partial replacement of Fe with Co in the quaternary $(Fe_{71.2}B_{24}Y_{4.8})_{96}Nb_4$ bulk metallic glasses on their structure, thermal stability and magnetic properties was studied. It was found that Co increases the thermal stability, as well as the Curie temperature, which monotonously increases as the Co content increases. The saturation magnetization shows a maximum of 1.01 μ_B per magnetic atom for $x = 0.1$, followed by a perfectly linear decrease for higher Co contents. The extension of the supercooled liquid region may reach even 98 K and the glass transition temperatures approach the theoretical value of 2/3 of the melting temperature. Using the mean filed theory for amorphous alloys allows to calculate the exchange stiffness constant and to correlate its variation with the variation of the magnetic saturation.

Keywords: Amorphous alloys, soft magnetic properties, exchange interactions, Curie temperature

1. Introduction

Metallic glasses have a unique combination of properties that makes them superior to their crystalline counterparts, such as high strength, high corrosion resistance and good soft magnetic properties.[1-3] Fe- and Co-based amorphous alloys have been the subject of considerable research interest and activities for the last two decades due to applications related to their outstanding soft magnetic properties.[3-7] During the past years, new bulk metallic glasses (BMGs) such as Ln-, Mg-, Zr-, Ti-, Fe- or Co-based glasses, have been developed (overviews are given in [8,9]). Most of these BMGs have a rather wide supercooled liquid region between the glass transition and the onset of crystallization, and a high resistance against crystallization, which enables the production of bulk glassy samples at low cooling rates.[9] Based on their attractive properties, such as good soft magnetism,[9,10] as well as mechanical behaviour (high yield strength, high hardness, good wear resistance)[9] or excellent corrosion resistance[9], many bulk metallic glasses have potential technological applications in various areas: machinery structural materials, die materials, tool materials, hydrogen storage materials, sporting goods materials, zero magnetostrictive materials, ornamental materials etc.[9] Fe-based BMGs typically exhibit a wide supercooled liquid region of almost 40 K, and good soft magnetic properties, such as 1.2 T for saturation magnetization and less than 2 A/m coercivity.[11,12]

In most of cases, Fe-based BMGs with very good glass-forming ability (GFA) contain many elements, as for example $(Fe_{44.3}Cr_5Co_5Mo_{12.8}Mn_{11.2}C_{15.8}B_{5.9})_{98.5}Y_{1.5}$ or $(Fe_{44.3}Cr_{10}Mo_{13.8}Mn_{11.2}C_{15.8}B_{5.9})_{98.5}Y_{1.5}$.[13] Few years ago, a new Fe-based BMG alloy system with high GFA but consisting of a small number of constituting elements was developed by Kim et al.[14] As a starting alloy composition, they selected $Fe_{71.2}B_{24}Y_{4.8}$ since high GFA (a maximum diameter for glass formation of 1 mm) has been reported by Zhang et al.[15]. Further, Nb was chosen as a candidate for the fourth alloying element based on the empirical rules for achieving high GFA.[9] The Goldschmidt atomic radius of Nb is 0.146 nm, which is significantly larger than those of Fe and B (0.126 and 0.098 nm, respectively), and smaller than that of Y (0.178 nm).[16] Nb exhibits a significant atomic size mismatch (>12%) with the main three constituent elements. The mixing enthalpy between the main constituents reaches large negative values (i.e., for Fe–Nb and B–Nb pairs −16 and −39 kJ/mol, respectively).[17] Furthermore, Nb has been reported to stabilize the liquid against primary crystallization of α-Fe[18-20] and within this class of BMGs, $(Fe_{71.2}B_{24}Y_{4.8})_{96}Nb_4$ shows the best GFA, while still keeping very good soft magnetic properties.[14]

In several cases it was observed that partial substitution of Fe with other magnetic elements (as Co or Co/Ni) may enhance the GFA.[21] At the same time, the soft magnetic properties are changed, basically due to the fact that such compositional variation may change the magnetostriction of the amorphous samples.[12] In the present work, the structural, thermal and magnetic property changes induced by partial substitution of Fe with Co in $[(Fe_{1-x}Co_x)_{71.2}B_{24}Y_{4.8}]_{96}Nb_4$ alloys (x = 0, 0.1, 0.3 and 0.5) are studied. It was found that upon substitution, the thermal stability becomes better, with glass transition temperatures exceeding 835 K and with a maximum extension of the supercooled liquid region (SCL) of almost 100 K for x = 0.3. This is unusual for Fe-based BMGs and indicates a very good GFA and high stability against crystallization

2. Experimental

The master alloys were prepared in several steps, using arc melting in a Ti-gettered high purity Ar atmosphere. First of all, an eutectic 25Fe75Nb (wt .%) prealloy was produced by melting pure Fe (99.9 mass %) and Nb (99.9 mass %) lumps. Further, proper quantities of FeNb prealloy, together with the rest of necessary Fe, Co lumps (99.9 mass %), crystalline B (99 mass %) and Y lumps (99 mass %) were melted together. The as-melted buttons were re-melted several times in order to assure a good homogeneity of the entire master alloy. Pieces of each master alloy were remelted in quartz tubes and then the melt injected into a water-cooled copper mould in a high-purity argon atmosphere to produce rod-shaped specimens with different diameters. In order to keep the repeatability and to have samples suitable for other measurements, we cast 2 mm diameter rods with a length of 60-70 mm. The structure of the as-cast rods was examined by x-ray diffraction (XRD) in transmission geometry at HASYLAB DESY (Hamburg, Germany), on the wiggler beamline BW5 at the DORIS III positron storage ring using monochromatic high energy high intensity synchrotron radiation of 100 keV (λ = 0.0124 nm).. The samples measured were exposed for 10 s to a well collimated incident beam with a cross section of 1 x 1 mm^2. Two-dimensional XRD patterns were collected using a MAR345 image plate detector (2300 x 2300 pixels, 150 x 150 μm^2 pixel size) carefully mounted orthogonal to the X-ray beam. The thermal stability and the melting behavior of the glassy samples were evaluated using a NETZSCH DSC 404 C differential scanning calorimeter (DSC) at heating and cooling rates of 20 K/min under a flow of high purity argon. For magnetic property investigation, *M-H* hysteresis loops were measured with a vibrating sample magnetometer (VSM) at ambient temperature. The Curie

temperature T_c of the samples were determined using an in-house developed Faraday magnetometer. Heating and cooling of the samples was performed at a constant rate of 20 K/min. In order to minimize the errors, the obtained data were analyzed using the method proposed by Herzer.[22] The coercivity H_c was measured using a Foerster Coercimat under an applied field high enough to saturate the samples. All magnetic properties were measured under DC magnetic field. Additionally, the density of the specimens was evaluated by the Archimedes's method, using a computer controlled microbalance and glycol as working liquid. The final values were obtained by averaging over 50 experimental values.

The accuracy of the experimental data lies within ± 2.5 K in the case of DSC measurements and thermo-magnetic curves, ± 0.1 A/m for coercivity, ± 80 A/m (~ 1 Oe) for VSM measurements and 0.5 % for density measurements.

3. Results and discussions

a). Structure and thermal stability

Fig. 1 shows the XRD patterns for all four as-cast 2 mm diameter rods. The patterns were obtained upon radial integration of the primary diffraction images as recorded by the 2D detector (image plate). The integration procedure is described in details elsewhere.[23] The patterns contain only broad maxima, without any additional sharp Bragg peaks, characteristic for fully amorphous samples. In order to ensure that the structure of the samples was homogeneous, each rod was scanned millimeter by millimeter along its axis. The diffraction images did not shown any traces of crystallinity.

Fig. 2 presents the DSC traces measured at a constant heating rate of 20 K/min. All samples show a glass transition, followed by an extended supercooled liquid region and crystallization. The crystallization proceeds in two steps, which are clearly observable for $x = 0$, 0.1 and 0.5, and less pronounced for $x = 0.3$. Melting occurs at relatively high temperatures, i.e. above 1350 K, and the features of the DSC signals reveal that all alloy compositions are close to an eutectic. The characteristic temperatures (glass transition T_g, crystallization T_x and melting, considered to be the liquidus temperature, T_{liq}), measured as the onset of the respective heat flow events, together with the extension of the supercooled liquid region $\Delta T_x = T_x - T_g$, the reduced glass transition temperature $T_{rg} = T_g / T_{liq}$ [24] and the γ parameter ($\gamma = T_x / (T_g + T_{liq})$ [25]) are summarized in Table 1. All data measured for $x = 0$, i.e. the starting alloy composition, as well as the DSC behavior, match perfectly with the data measured by Kim et $al.$[14] When

substituting Fe by Co, the characteristic temperatures slightly decrease, while the extension of the SCL remains very large. In fact, for $x = 0.3$, ΔT_x, T_{rg} and the γ parameter reach the highest values among all studied compositions: 98 K, 0.628 and 0.430, respectively. This suggests that $[(Fe_{0.7}Co_{0.3})_{71.2} B_{24}Y_{4.8}]_{96}Nb_4$ may have the best GFA, even better than for the starting Co-free composition. However, all four alloys show a large SCL, much larger that the interval of about 30-35 K found for Fe-based glasses.[9] Turnbull[24] has shown that the tendency for glass formation increases when the reduced glass transition temperature T_{rg} is as high as possible, but not beyond 2/3 (0.66). The present studied alloys almost approach this value (for example 0.628 for x = 0.3), thus underlining their very good GFA.

The crystallization and melting behavior also changes upon partial substitution of Fe by Co. The SCL shows also an interesting feature, a small exothermic event, more pronounced for x = 0 and 0.1. This could be done by a re-arrangement of the atoms or atom clusters, a change in the short-range order or a partial (nano)crystallization. There are several mechanisms possible, one of them being related to a possible annihilation of the free-volume generated by the rapid cooling upon casting.[26]

Other important feature which is indicated by the DSC experiments is that all compositions are very close to the eutectic (the melting takes place in a relatively narrow temperature interval). For $x = 0.1$, there we have an almost perfect eutectic alloy- the endothermic event is very sharp. As anticipated by Turnbul[24] and confirmed by Inoue,[9] the best glass-forming alloys should be in the vicinity of a deep eutectic. From the DSC traces (Fig. 2) one can clearly see that upon Fe replacements with Co, the melting temperatures decreases.

b). Magnetic properties

As expected, the fully amorphous samples display very good soft magnetic properties. Table 2 summarizes the measured values of coercivity H_c, saturation magnetization M_s (given in Am^2/kg and in Bohr magnetons μ_B per magnetic atom), saturation polarization J_s, and Curie temperature T_c. Additionally, the density (ρ) values are listed. All data were measured for as-cast samples. The coercivity values, which are almost around 1 A/m, are typical for soft magnetic amorphous alloys. Interesting is the compositional dependence of the saturation magnetization (and of the polarization as well), which reaches a maximum for $x = 0.1$. The saturation magnetization was derived from the hysteresis loops, presented in Fig. 3. The saturation polarization was calculated using the actual density values ($J_s = 4 \pi 10^{-7} \rho M_s$). Fig. 3 reveals that the shape of the loops is characteristic for very soft magnetic materials. The saturation increases for $x = 0.1$, followed by a decreasing trend, perfectly linear, for higher Co

contents. Fig. 4 shows in details the saturation polarization, together with the density values, as a function of Co content. The maximum magnetic saturation value corresponds to a minimum density. However, at the first glance the density variation is striking, because the density of Co (i.e. 8.92 g/cm^3) is higher than that of Fe (i.e. 7.86 g/cm^3). Moreover, the radii of the two atoms are very close (125 pm for Co and 126 pm for Fe) and one would expect a monotonous increase of the density as Fe is replaced by Co. This could be true in the case of crystalline materials, but it does not necessarily apply to amorphous structures. A metallic glass contains a certain amount of free volume as a consequence of the rapid cooling from the molten state.[26] Also, some cluster formation may occur during cooling. It seems that the presence of a small amount of Co could increases the amount of free volume. In fact, the real mechanism upon which the density takes a minimum value and the saturation magnetization a maximum value for $x = 0.1$ is not so simple. Most probably it has the root in the structure anomalies observed in the case of $3d$ transition metals.[27] The occupation numbers of the spin-up and the spin-down d band for Fe and Co are 4.4, 2.2 and 4.6, 2.9, respectively.[27] The energy difference between different phases for an almost filled d band is very small, thus the contribution of the spin-up electrons to the structural energy differences shall be neglected. The alloying of Co into Fe will first increase the spin-up and decrease the spin-down d occupations, and for a higher Co concentration the spin-up band is saturated and the filling of the spin-down band is increasing. This is a reflection of the fact that the magnetic moment is initially increasing with Co concentration and after reaching a maximum it decreases (Slater-Pauling curve[28]). In the case of crystalline ferromagnetic Fe$_{1-x}$Co$_x$, for small atomic fraction x the energetically favourable structure is the bcc. Theoretically, the energy difference between bcc and fcc structures should be zero when $x = 0.375$.[27] Of course, the BMGs do not show any crystalline network, but a short-range order, which may have the tendency to follow the atomic arrangement of the crystalline alloy, could be present. The packing coefficient of the bcc is lower than the one of fcc and this could explain the variation of the density for small x values.

However, the composition with $x = 0.1$ is the one which shows a perfect eutectic melting behavior in the DSC scan. The increase in free volume may also explain the slightly higher coercivity value, 1.7 A/m, found for this alloy compared to 1 A/m or 1.1 A/m measured for samples with $x = 0$ and $x = 0.3$, respectively. The values of H_c depend mostly on surface and volume pinning of magnetic domain walls. It has been reported[3] that due to surface irregularities H_c is proportional to the ratio of the surface roughness amplitude to the specimen thickness. This contribution to H_c should be rather low for bulk cast samples

because (i) the surface upon casting is very smooth and (ii) the thickness is significantly larger than that of rapidly quenched ribbons. The contribution of volume pinning to H_c results from the presence of internal stress. The stress sources are supposed to have their origin in the partial instability of the free volume below the melting point.[29] This contribution is proportional to the product of saturation magnetostriction and the amplitude of stress fluctuations.[30] This H_c contribution can be reduced by annealing, during which the internal stresses are released. But if the domain pinning is realized via some (nano)crystals, the coercivity will not decrease upon annealing.

The variation of saturation magnetization as a function of Co content should be explained in more detail. First of all, it does not follow the trend of the Curie temperature, which monotonously increases as the Co content increases. The thermomagnetic curves, measured at a constant heating and cooling rate of 20 K/min up to 900 K, are presented in Fig. 5. Apparently, crystallization did not start yet and the values correspond to the glassy phase. There are slightly differences between heating and cooling, due to the fact that upon cooling the samples are already relaxed. For temperatures close to T_c, the saturation magnetization can be described by:[22]

$$M_S(T) = M(0) \cdot \left(1 - \frac{T}{T_C}\right)^{\beta},$$
(1)

with the exponent $\beta = 0.36$. In order to minimize the errors, the experimental results were plotted as $(M_S)^{1/\beta}$ versus T (not presented here). The Curie temperature was considered the temperature were $(M_S)^{1/\beta}$ deviates from linearity and the corresponding values are summarized in Table 2. The increase of the Curie temperature with increasing Co content is rather obvious, because the Curie temperature of Co is much higher than that of Fe (1388 K versus 1044 K) and the Curie temperature is an intrinsic property of the material.

The ferromagnetism in amorphous materials is supposed to take place by exchange interactions between the spins of neighbouring unpaired electrons.[28] The interactions are stronger when the magnetic atoms are at short distance, while their separation, for example by metalloid atoms, makes the interactions weaker and, as a consequence, the magnetic saturation decreases. The exchange stiffness constant, A, can be obtained from the mean field model and from the Curie temperature.[31-33] If one assumes Hasegawa's model[31] to be valid, then one can derive the following relations:

$$A = \frac{C_{Fe} S_{Fe} k_B T_C}{4(S_{Fe} + 1) r_{Fe-Fe}} \qquad (2)$$

where C_{Fe} is the concentration of Fe in atomic percent, S_{Fe} is the spin moment of Fe, T_c is the Curie temperature, k_B the Boltzmann constant and r_{Fe-Fe} is the nearest-neighbor distance, which is considered to be double the Fe atomic radius. From the relation:

$$M(\mu_B) = 2 C_{Fe} S_{Fe} \mu_B \qquad (3)$$

in which $M(\mu_B)$ is the magnetic moment of the samples considered in Bohr magnetons, one can calculate S_{Fe}. However, the relations are only valid for the starting alloy sample ($x = 0$) or if one neglects Fe-Co and Co-Co interactions. Normally, when Fe is partially substituted by Co, the magnetic saturation should be considered as:

$$M(\mu_B) = w M_{Fe}(\mu_B) + (1 - w) M_{Co}(\mu_B) \qquad (4)$$

in which $M_{Fe}(\mu_B)$ and $M_{Co}(\mu_B)$ are the magnetic moment of Co-free and Fe-free (respectively) amorphous alloys and w is the atomic fraction Fe/(Fe+Co). In our present case, the total magnetism is given by three types of interactions: Fe-Fe, Co-Co and Fe-Co. In other words, there are two magnetic sublattices, which can interact. For a mean-field model,[30] the calculation of T_c for two sublattices reduces to:

$$3 k_B T_C = a_{11} + a_{22} + \left[(a_{22} - a_{11})^2 + 4 a_{12} a_{21} \right]^{1/2} \qquad (5)$$

in which

$$\begin{aligned}
a_{11} &= C_{Fe} Z_1 J_{11} S_{Fe}(S_{Fe} + 1) \\
a_{22} &= C_{Co} Z_2 J_{22} S_{Co}(S_{Co} + 1) \\
a_{12} a_{21} &= C_{Fe} C_{Co} Z_{Fe} Z_{Co} J_{12}^2 S_{Fe}(S_{Fe} + 1) S_{Co}(S_{Co} + 1)
\end{aligned} \qquad (6)$$

and where Z_i are the coordination numbers (the indicex 1 refers to Fe atoms and 2 to Co atoms), S_{Co} is the Co spin C_{Co} the Co concentration in atomic percents and J_{ij} the exchange constants.

Fig. 6 shows the variation of the exchange stiffness constant A as a function of the atomic fraction x, i.e. as a function of the Co content. The stiffness constant reaches a maximum

value for $x = 0.1$, and decreases monotonously and perfectly linear for higher Co contents. An increase of the stiffness constant usually indicates suppression of the magnetic anisotropy and an increase of exchange energy. As a consequence, the saturation magnetization may increase. The results are consistent with those presented in Fig. 4, i.e. the variation of the saturation magnetization as a function of x.

4. Conclusions

$[(Fe_{0.7}Co_{0.3})_{71.2} B_{24}Y_{4.8}]_{96}Nb_4$ BMGs with $x = 0$, 0.1, 0.3 and 0.5 were studied. Partial substitution of Fe with Co changes the thermal stability, melting behavior and magnetic properties. The thermal stability is very good, with glass transition temperatures exceeding 835 K and with a maximum extension of the SCL of almost 100 K for $x = 0.3$. This is unusual for Fe-based BMGs and indicates a very good GFA and high stability against crystallization. The magnetic properties are typical for soft magnetic materials. The Curie temperature increases upon Co addition and the saturation magnetization decreases, but first goes through a maximum for $x = 0.1$. Using the mean field theory allows to calculate the exchange stiffness constant and the data follow the trend of the experimental values, i.e. they also show a maximum for 10 at% of Co in the alloy.

5. Acknowledgements

6. References

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Table 1. Thermal stability data for $[(Fe_{1-x}Co_x)_{71.2} B_{24}Y_{4.8}]_{96}Nb_4$ glassy rods: glass transition temperature T_g, crystallization temperature T_x, extension of the supercooled liquid region ΔT_x, melting temperature T_{liq}, the reduced glass transition temperature T_{rg} and the γ parameter.

Atomic fraction x	T_g [K]	T_x [K]	ΔT_x [K]	T_{liq} [K]	T_{rg}	γ
0	862	955	93	1392	0.619	0.423
0.1	836	927	91	1374	0.608	0.419
0.3	855	953	98	1361	0.628	0.430
0.5	847	927	80	1366	0.620	0.418

Table 2. Magnetic data for $[(Fe_{1-x}Co_x)_{71.2} B_{24}Y_{4.8}]_{96}Nb_4$ glassy rods: coercivity H_c, magnetization at saturation M_s (given in Am^2/kg and in Bohr magnetons μ_B per magnetic atom), saturation polarization J_s, Curie temperature T_c. Additionally, the density (ρ) data are shown.

Atomic fraction x	H_c [A/m]	M_s [Am^2/kg]	M_s [μ_B]	J_s [T]	T_c [K]		ρ [g/cm^3]
					heating	cooling	
0	1	93.1	0.93	0.84	456	470	7.2207
0.1	1.7	100.7	1.01	0.90	509	519	7.1297
0.3	1.1	92.1	0.93	0.85	546	553	7.3606
0.5	1.5	85.5	0.87	0.80	579	595	7.5146

Figure captions

Fig. 1 XRD patterns, obtained by using synchrotron radiation in transmission configuration, recorded for $[(Fe_{1-x}Co_x)_{71.2}B_{24}Y_{4.8}]_{96}Nb_4$, $x = 0$, 0.1, 0.3 and 0.5 as-cast 2 mm diameter glassy samples.

Fig. 2 DSC traces measured at a constant heating rate of 20 K/min for $[(Fe_{1-x}Co_x)_{71.2}B_{24}Y_{4.8}]_{96}Nb_4$, $x = 0$, 0.1, 0.3 and 0.5 glassy samples.

Fig. 3 DC hysteresis loops for as-cast $[(Fe_{1-x}Co_x)_{71.2}B_{24}Y_{4.8}]_{96}Nb_4$, $x = 0$, 0.1, 0.3 and 0.5 glassy samples.

Fig. 4 Variation of density and saturation magnetization for $[(Fe_{1-x}Co_x)_{71.2}B_{24}Y_{4.8}]_{96}Nb_4$ as-cast amorphous samples as a function of the atomic fraction x

Fig. 5 Thermomagnetic curves for as-cast $[(Fe_{1-x}Co_x)_{71.2}B_{24}Y_{4.8}]_{96}Nb_4$, $x = 0$, 0.1, 0.3 and 0.5 glassy samples. The experiments were performed at a constant heating and cooling rate of 20 K/min.

Fig. 6 Variation of the exchange stiffness constant A as a function of the atomic fraction x.