Journal of Alloys and Compounds 509 (2011) 3843–3846

Contents lists available at ScienceDirect

Journal of Alloys and Compounds

journal homepage: www.elsevier.com/locate/jallcom

Synthesis, thermal stability and properties of $[(Fe_{1-x}Co_x)_{72}Mo_4B_{24}]_{94}Dy_6$ bulk metallic glasses

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ARTICLE INFO

Article history:
Received 24 August 2010
Received in revised form
12 December 2010
Accepted 15 December 2010
Available online 23 December 2010

Keywords:
Bulk metallic glass
Glass forming ability
Thermal stability
Soft magnetic property

ABSTRACT

A series of $[(Fe_{1-x}Co_x)_{72}Mo_4B_{24}]_{94}Dy_6$ (x = 0.1, 0.2, 0.3, 0.4 and 0.5 at.%) bulk metallic glasses (BMGs) in rod geometries with critical diameter up to 3 mm were fabricated by copper mold casting method. This alloy system exhibited good thermal stability with high glass transition temperature (T_g) 860 K and crystallization temperature (T_x) 945 K. The addition of Co was found to be effective in adjusting the alloy composition deeper to eutectic, leading to lower liquidus temperature (T_l). The $[(Fe_{0.8}Co_{0.2})_{72}Mo_4B_{24}]_{94}Dy_6$ alloy showed the largest supercooled liquid region ($\Delta T_x = T_x - T_g = 92$ K), reduced glass transition temperature ($T_{rg} = T_g/T_l = 0.622$) and gamma parameter ($\gamma = T_x/(T_g + T_l) = 0.424$) among the present system. Maximum compressive fracture strength of 3540 MPa and micro-Vickers hardness of 1185 kg/mm^2 was achieved, resulting from the strong bonding structure among the alloy constituents. The alloy system possessed soft magnetic properties with high saturation magnetization of 56.61–61.78 A m^2/kg and coercivity in the range of 222–264.2 A/m, which might be suitable for application in power electronics devices.

1. Introduction

Ferromagnetic Fe(Co)-based bulk metallic glasses (BMGs) have attracted considerable attention due to their unique magnetic properties for electronic devices and transformers applications [1,2]. Several Fe(Co)-based BMGs: (Fe,Co)-(Ga, Mo)-(P–C–B–Si) [3–6], Fe–Co–Zr–M–B (M = Zr, Mo, W) [7,8] and Fe–Co–Nb–Si–B [9–11] have been studied, which exhibit promising magnetic and functional properties. Among them, some alloy system [6,11] possessed good soft magnetic properties with saturation magnetization of 1.14–1.44 T and low coercive force of 1.5–20 A/M, which largely enhanced the possibility of their industrial applications. Some of Fe(Co)-based BMGs show poor glass forming ability (GFA) such as Fe–Co–Zr–Mo–W–B [8], Fe–Co–Ni–Zr–Mo–B [12], which have restricted their BMGs critical sizes up to 1.5 mm. Therefore, in order to expand their industrial applicability, search for advanced Fe(Co)-based BMGs possessing high GFA along with superior mechanical and magnetic properties is essential. Researchers have adopted two approaches to improve GFA along with properties in Fe(Co)-based alloys system either by composition modification or process optimization. The former approach was found to be more conventional and economical from manufacturing point of view than the later due to practical difficulties during synthesis process.

It has been found that the minor addition of rare-earth (RE) element has a beneficial effect on GFA, mechanical and magnetic properties of Fe(Co)-based BMGs [13–15]. Consequently, a number of Fe(Co)-based BMGs as Fe–Co–Pr–B and Fe–Co–Y–B alloys were developed [16,17]. Although, these alloys reflected improved GFA, but their critical sizes were still small as compared to Mg-[18], Zr-[19] and La-[20] based BMGs, which exhibits maximum sizes up to 32 mm. Recently, Li et al. reported that Dy element with relatively high bulk modulus (41 GPa) has positive effect in the development of BMGs [21]. Similarly, it was found that the addition of transition metal like Mo can simultaneously increase GFA, thermal stability and fracture strength in Fe-based BMGs [22,23]. Our previous studies showed that $(Fe_{72}Mo_4B_{24})_{94}Dy_6$ BMGs have good GFA, magnetic and mechanical properties [24].

With the aim of improving Fe(Co)-based BMGs with higher GFA, along with good mechanical and magnetic properties, a new series of alloy system $[(Fe_{1-x}Co_x)_{72}Mo_4B_{24}]_{94}Dy_6$ (x = 0.1, 0.2, 0.3, 0.4 and 0.5 at.%) was developed by copper mold casting and effects of Co content on GFA, magnetic and mechanical properties were discussed.

2. Experimental

Cast ingots with nominal compositions $[(Fe_{1-x}Co_x)_{72}Mo_4B_{24}]_{94}Dy_6$ (x = 0.1, 0.2, 0.3, 0.4 and 0.5 at.%) were prepared by arc melting the mixture of Fe (99.6 wt.%), Co (99.9 wt.%), Mo (99.8 wt.%), Dy (99.9 wt.%) metals and Fe–B alloy (79.58 wt.% Fe, 20.42 wt.% B) in an argon atmosphere. Each ingot was re-melted for five times to ensure the composition homogeneity. Alloy ingots were then crushed into small pieces in accord with the size of quartz crucible for copper mold casting. Rod shaped

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doi:10.1016/j.jallcom.2010.12.109

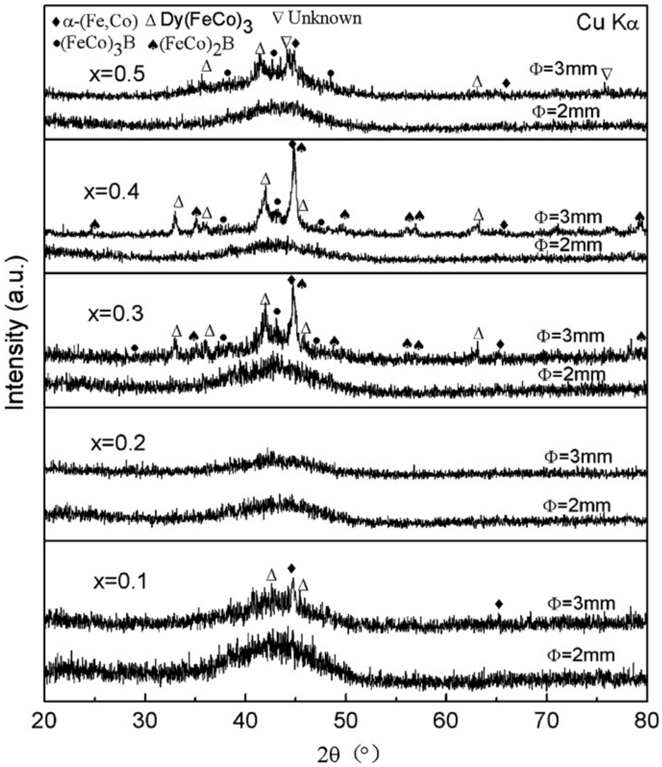

Fig. 1. XRD patterns of $[(Fe_{1-x}Co_x)_{72}Mo_4B_{24}]_{94}Dy_6$ (x = 0.1, 0.2, 0.3, 0.4 and 0.5 at.%) alloys with diameters of 2-3 mm.

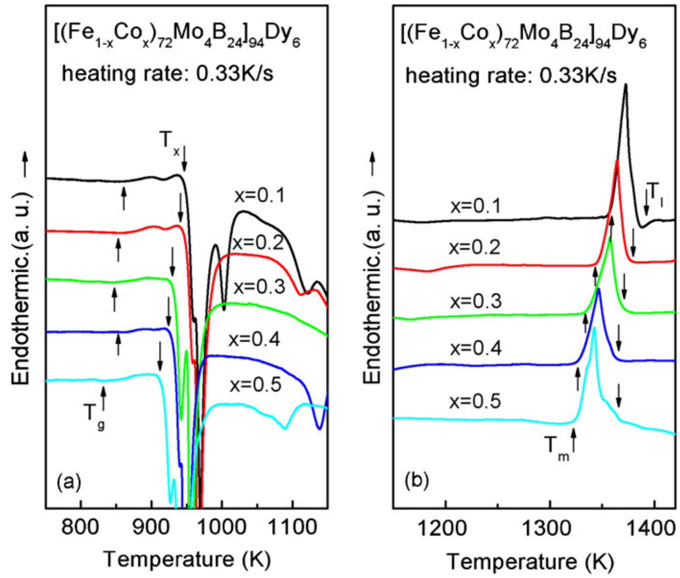

Fig. 2. DSC curves of $[(Fe_{1-x}Co_x)_{72}Mo_4B_{24}]_{94}Dy_6$ (x = 0.1, 0.2, 0.3, 0.4 and 0.5 at.%) alloy rods with 2 mm in diameter at low temperature range of 750–1150 K (a) and high temperature range of 1150–1450 K (b), respectively.

samples with diameters ranging from 1 to 3 mm and length up to 30 mm were obtained by copper mold casting.

Glassy structures of samples were identified by X-ray diffraction technique (XRD Thermo ARL X'Tra diffractometer) with Cu Kα radiation in a 2θ scan-range of 20–80°. The glass transition, crystallization and melting behaviors of the alloys were examined with differential scanning calorimetry (DSC, NETZSCH DSC 404 C) using a continuous argon flow at a heating rate of 0.33 K/s. The supercooled liquid region (ΔT_x) was defined by the temperature interval between the glass transition temperature (T_g) and the onset crystallization temperature (T_x). Curie temperature (T_c) of the glassy rods was measured by a magnetic thermal gravimetric analyzer (M-TGA, Delta Series TGA7) at a heating rate of 0.17 K/s. Magnetic properties such as saturation magnetization (M_s) and coercivity (H_c) were determined by the M–H hysteresis loops using a vibrating sample magnetometer (VSM, Lakeshore 7407). Uniaxial compressive tests on the glassy rods were performed with a universal testing machine (CMT5205 SANS, China) at a deformation rate of 5×10^{-4} s^{-1}. For compressive tests, specimens of 2 mm in diameter and 4 mm in length were obtained from the cast rods, and seven samples were subjected to compressive test to ensure reproducibility of the results. Vickers hardness (H_v) measurements were conducted using a micro-Vickers hardness tester with load and dwelling time of 200 g and 15 s, respectively. For hardness test, at least 12 measurements were performed for each sample.

3. Results and discussion

Fig. 1 shows the XRD patterns of $[(Fe_{1-x}Co_x)_{72}Mo_4B_{24}]_{94}Dy_6$ (x = 0.1, 0.2, 0.3, 0.4 and 0.5 at.%) samples of 2 and 3 mm in diameter. All the samples of 2 mm-diameter with different Co contents exhibit similar broad halo peaks, which represents an amorphous structure. It means that the present Fe–Co–Mo–B–Dy alloy system has a good GFA that enable us to cast amorphous rods at least 2 mm in diameter. The possibility of glassy rods with larger diameter from this alloy system was further examined. As shown in Fig. 1, the increase of diameter to 3 mm results in the appearance of crystalline phases and some sharp diffraction peaks correspond to α-(Fe, Co), Dy(Fe, Co)$_3$ and borides like (Fe, Co)$_3$B, (Fe, Co)$_2$B for the alloys with x = 0.1, x = 0.3, x = 0.4 and x = 0.5. The critical diameter where the rod is fully amorphous is 3 mm for x = 0.2 and 2 mm for the other alloys (x = 0.1, x = 0.3, x = 0.4 and x = 0.5), respectively. Therefore, the maximum rod diameter of keeping fully amorphous structure is 3 mm for the bulk $[(Fe_{0.8}Co_{0.2})_{72}Mo_4B_{24}]_{94}Dy_6$ alloy. This indicates that the $[(Fe_{0.8}Co_{0.2})_{72}Mo_4B_{24}]_{94}Dy_6$ alloy possesses the best tolerance in GFA among the present alloy system.

Fig. 2(a) and (b) illustrate DSC curves of $[(Fe_{1-x}Co_x)_{72}Mo_4B_{24}]_{94}Dy_6$ (x = 0.1, 0.2, 0.3, 0.4 and 0.5 at.%) glassy rods with diameters of 2 mm at the temperature range of 750–1150 K and 1150–1450 K, respectively. Each DSC trace exhibits distinct glass transition, followed by a wide supercooled liquid region. The glass transition temperature, T_g and the onset crystallization temperature, T_x (marked by arrows, respectively) determined from the temperature region of 750–1150 K are summarized in Table 1. In addition, the thermal parameters ΔT_x ($T_x - T_g$), T_{rg} (T_g/T_l) and γ ($T_x/(T_g + T_l)$) are also calculated and presented in Table 1. With the increase of Co content, T_g increases gradually from 854 K for x = 0 [24] to 860 K for x = 0.1, then decreases to 831K for x = 0.5. T_x decreases linearly from 945 to 912 K as a function of the Co content. The value ΔT_x shows weak dependence on the Co content and remains in a range of 80–92 K. Furthermore, the present BMGs also possess high T_{rg} (0.609–0.622) and γ (0.415–0.424) values. All the ΔT_x, T_{rg} and γ are higher than previously reported RE-free Fe(Co)-based BMGs [25–27], which is probably one of the main factors for obtaining high GFA in $[(Fe_{1-x}Co_x)_{72}Mo_4B_{24}]_{94}Dy_6$ alloys that enable us to manufacture BMG up to 3 mm in diameter. The onset melting temperature (T_m) and offset melting temperature (T_l) obtained from the temperature region of 1150–1450 K are also listed in Table 1. With the increase of Co content from x = 0 to 0.5, T_m and T_l monotonically decrease from 1377 to 1326 K and 1400 to 1365 K, respectively, which indicates that the addition of Co is effective in shifting the alloy close to a eutectic point. It is known that alloy compositions at or close to the eutectic point can favor the glass structure formation [28]. Thus, the addition of proper amount of Co in the present alloy system could stabilize the supercooled melt by lowering the liquidus temperature and consequently enhance the GFA.

According to the strong bonding nature theory suggested by Poon et al. [29], the Fe–Co–Mo–B–Dy system that contains mid-size Fe, Co atoms as the major alloy components, small size B atom as the next major component (20–30 at.%) and the large size Dy, Mo atoms as the minor component (∼10 at.%) falls into the 'majority

Table 1
The glass transition temperature (T_g), onset crystallization temperature (T_x), melting (T_m) and liquids temperature (T_l), thermal parameters (ΔT_x, T_{rg}, γ), Curie temperature (T_c), saturation magnetization (M_s), coercive field (H_c), compressive fracture strength (σ_f), and Vickers hardness (H_v) of $[(Fe_{1-x}Co_x)_{72}Mo_4B_{24}]_{94}Dy_6$ alloy rods with 2 mm in diameter.

Alloy (at. %)	T_g (K)	T_x (K)	ΔT_x (K)	T_m (K)	T_l (K)	T_{rg}	γ	T_c (K)	M_s (A m^2/kg)	H_c (A/m)	σ_f (MPa)	H_V (kg/mm^2)
$x = 0$ [24]	854	945	91	1377	1400	0.610	0.419	440	64.97	426.5	2986	1130
$x = 0.1$	860	945	85	1358	1385	0.621	0.421	461	60.36	222	3488	1165
$x = 0.2$	852	944	92	1347	1370	0.622	0.424	476	56.61	264.2	3529	1180
$x = 0.3$	845	929	84	1337	1367	0.618	0.420	503	59.88	241.1	3540	1185
$x = 0.4$	847	927	80	1328	1366	0.620	0.419	531	59.83	243.5	3535	1185
$x = 0.5$	831	912	81	1326	1365	0.609	0.415	582	61.78	249.9	3525	1178

atom-small atom-large atom' (MSL) alloy class. In the MSL alloys, L atoms (Dy, Mo) and S atom (B) may form a strong L–S percolating network or reinforced "backbone" in the amorphous structure which enhances GFA.

The good GFA and high thermal stability of Fe(Co)-based BMGs could be also understood by the empirical rules [30]. In the present system, the atomic radii of component atoms are Fe 0.124 nm, Co 0.125 nm, Mo 0.136 nm, B 0.09 nm, Dy 0.175 nm [31] and the ratio of atomic radii is 1.411 for $R_{Dy/Fe}$, 1.097 for $R_{Mo/Fe}$, 1.008 for $R_{Co/Fe}$ and 0.726 for $R_{B/Fe}$, respectively. This atomic mismatch enhances the local random packing density, which might result in the suppression of crystallization and thus lead to improve the stability of liquid phase. Furthermore, the enthalpies of mixing among the alloy constituents for Fe–B, Co–B and Dy–B atomic pairs are −27 kJ/mol, −9 kJ/mol and −51 kJ/mol, respectively [32]. These large negative values of heat of mixing enhance the cohesion and interactions among alloy constituents and promote chemical short range ordering in liquids, which, in turn, improve the local packing efficiency and restrain long range diffusion of atoms [33].

However, it is difficult to establish the relation between GFA and thermal stability because of the complex competition between thermodynamic driving forces, formation of inter-metallic phases and crystallization kinetics. Bhattacharya et al. [6] pointed out that high thermal stability in (Fe,Co)–Mo–B–C–P–Si metallic glasses can be obtained by replacing the boron with carbon atoms. Furthermore, high atomic packing density of the alloy components lead to a higher viscosity which in turn slow down the process of nucleation in the (Fe,Co)–Mo–B–C–P–Si alloys. Thus, high atomic packing density in the investigated $[(Fe_{1-x}Co_x)_{72}Mo_4B_{24}]_{94}Dy_6$ (x = 0.1, 0.2, 0.3, 0.4 and 0.5 at.%) alloys might reflect good thermal stability.

Fig. 3 shows the M–H hysteresis loops of amorphous $[(Fe_{1-x}Co_x)_{72}Mo_4B_{24}]_{94}Dy_6$ (x = 0.1, 0.2, 0.3, 0.4 and 0.5 at.%) rods with a diameter of 2 mm. The saturation magnetization (M_s) and coercivity (H_c) are presented in Table 1. M_s decreases initially from 64.97 to 60.36 A m^2/kg with Co content up to x = 0.2, then it increases as a function of Co content. This is in good agreement with the reported $Fe_{70-x}Co_xHf_5Mo_7B_{15}Y_3$ (x = 0–20 at.%) alloys [27]. The initial decrease in M_s is attributed to the reduction of magnetic moment caused by smaller Bohr magnetons of Co atoms as compared to Fe atoms [34]. While, the increase in M_s for $x > 0.2$ alloys could be presumed by the enhancement of chemical short range ordering in the amorphous state due to the excessive Co contents [35,36]. The H_c values in the present alloys are comparable to the $Fe_{70-x}Co_xHf_5Mo_7B_{15}Y_3$ (x = 0–20 at.%) alloys [27] but higher than the $[(Fe,Co,Ni)_{0.75}B_{0.2}Si_{0.05}]_{96}Nb_4$ [37] and $(Fe_{1-y}Co_y)_{78}Mo_1C_7B_3P_{10}Si_1$ (y = 0.15, 0.2, 0.3) alloys [6]. This discrepancy in coercivity is probably due to the difference in alloy composition, raw materials purity and processing conditions.

Curie temperature (T_c) values of the studied alloys are also listed in Table 1. The T_c of the BMG alloys increases monotonically with increasing Co content and reaches to a maximum 582 K for x = 0.5. It is well known that the Curie temperature is dependent mainly on the strength of the exchange interaction between magnetic atoms. This increase in T_c may result from the strong exchange interactions of Fe–Co and Co–Co atomic pairs.

Fig. 4 shows the compressive stress-strain curves of $[(Fe_{1-x}Co_x)_{72}Mo_4B_{24}]_{94}Dy_6$ (x = 0.1, 0.2, 0.3, 0.4 and 0.5 at.%) alloys at a deformation rate of 5×10^{-4} s^{-1}. The fracture strength (σ_f) and micro-Vickers hardness (H_V) are listed in Table 1. In comparison with Co free $(Fe_{72}Mo_4B_{24})_{94}Dy_6$ alloy [24], the present $[(Fe_{1-x}Co_x)_{72}Mo_4B_{24}]_{94}Dy_6$ alloys possess higher fracture strength up to 3540 MPa and micro-Vickers hardness up to 1185 kg/mm^2. The enhancement of fracture strength and hardness in Fe–Co–Mo–B–Dy alloys is ascribed to the strong interactions among its constituent elements due to large negative values of

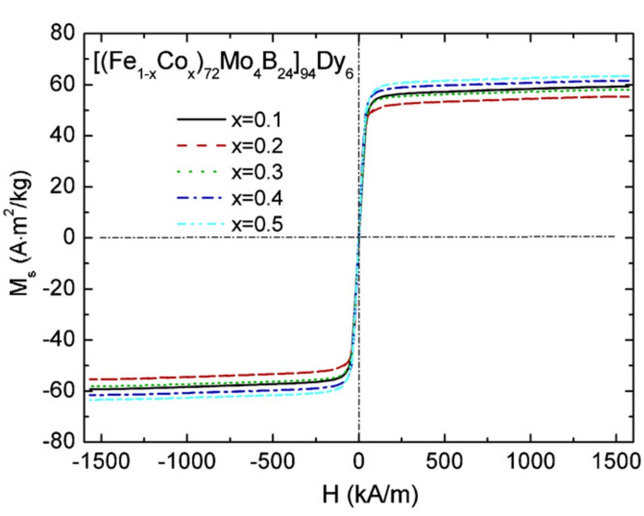

Fig. 3. Magnetization hysteresis loops of $[(Fe_{1-x}Co_x)_{72}Mo_4B_{24}]_{94}Dy_6$ (x = 0.1, 0.2, 0.3, 0.4 and 0.5 at.%) alloy rods with diameters of 2 mm.

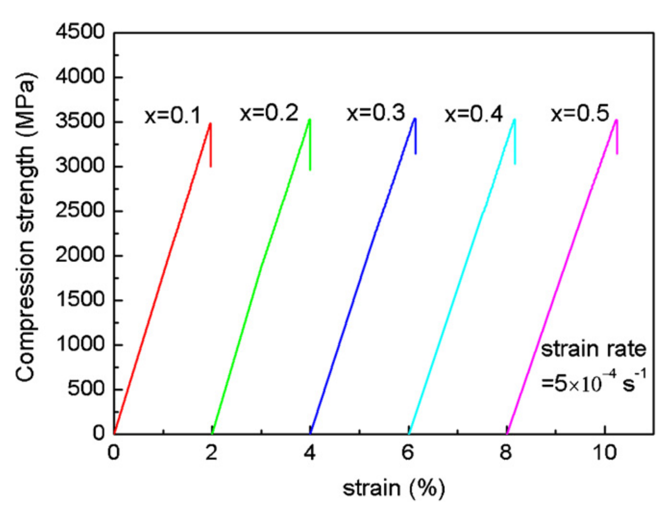

Fig. 4. Compressive stress-strain curves of $[(Fe_{1-x}Co_x)_{72}Mo_4B_{24}]_{94}Dy_6$ (x = 0.1, 0.2, 0.3, 0.4 and 0.5 at.%) alloy rods with diameters of 2 mm.

mixing enthalpies. Moreover, the atomic network structure formed by the large atoms and small atoms in the MSL alloy class may lead to enhance the fracture strength in the present alloy system.

4. Conclusions

The bulk glassy (Fe,Co)–Mo–B–Dy alloys were developed by copper mold casting method. Proper substitution of Co for Fe in (Fe,Co)–Mo–B–Dy alloy system lead to improvement of atomic packing density, stability of liquid phase and suppression of crystallization which in turn increase GFA, magnetic and mechanical properties. The $[(Fe_{0.8}Co_{0.2})_{72}Mo_4B_{24}]_{94}Dy_6$ alloy exhibits the largest ΔT_x (92 K), T_{rg} (0.622) and γ (0.424) values that enable us to manufacture bulk metallic glassy rods up to critical diameter of 3 mm. The high fracture strength and hardness in present alloys are attributed to the formation of network-like atomic structure and the strong bonding nature among the constituent's elements. The alloy system also possesses good soft magnetic properties with high saturation magnetization and modest coercivity. The magnetic properties of the present alloy system could be improved by taking the advantage of high purity raw materials, maintaining proper ratios of Fe and Co atoms and magnetic field annealing treatment, which could be suitable for future promising applications.

Acknowledgments

This work was supported by the National Natural Science Foundation of China (Grant No. 50971113) and 863 program of China (No. 2009AA03Z112).

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