Comparative Study of the Magnetic Properties and Glass-Forming Ability of Fe-Based Bulk Metallic Glass with Minor Mn, Co, Ni, and Cu Additions

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Abstract $\text{Fe}_{43}\text{M}_5\text{Cr}_{15}\text{Mo}_{14}\text{C}_{15}\text{B}_6\text{Y}_2$ ($M = \text{Mn}, \text{Co}, \text{Ni}, \text{and Cu}$ in at.%) bulk metallic glasses (BMGs) are synthesized using the suction casting technique, and the glass-forming ability (GFA), microstructure, and thermal and magnetic properties of these glasses are extensively examined using X-ray diffraction, differential scanning calorimeter, and vibrating sample magnetometer techniques. Among the four BMG alloys, $\text{Fe}_{43}\text{Ni}_5\text{Cr}_{15}\text{Mo}_{14}\text{C}_{15}\text{B}_6\text{Y}_2$ exhibits the lowest coercivity and the highest saturation magnetization, Curie temperature, effective magnetic moment, and GFA. By contrast, $\text{Fe}_{43}\text{Mn}_5\text{Cr}_{15}\text{Mo}_{14}\text{C}_{15}\text{B}_6\text{Y}_2$ presents the poorest magnetic properties, such as the highest coercivity and the lowest saturation magnetization, Curie temperature, and effective magnetic moment. $\text{Fe}_{43}\text{Cu}_5\text{Cr}_{15}\text{Mo}_{14}\text{C}_{15}\text{B}_6\text{Y}_2$ demonstrates the lowest thermal stability and GFA. The observed thermal, structural, and magnetic properties of these BMG alloys are discussed in terms of the kinetics of BMG synthesis and the formation of different ferromagnetic, ferrimagnetic, and antiferromagnetic phases.

KEY WORDS: Bulk metallic glasses; Saturation magnetization; Effective magnetic moment; Coercivity; Glass-forming ability

1 Introduction

Bulk metallic glasses (BMGs) have attracted considerable attention because of their unique disordered structure and exceptional physical and magnetic properties [1, 2]. Among various BMGs, Fe-based BMGs are the most attractive because of their potential structural and functional applications as well as their favorable magnetic properties, ultra-high strength, high strength-to-weight ratio, excellent corrosion resistance, and relatively low material cost [3–5].

BMG materials have numerous applications, such as in magnetic sensors, biomaterials, transformer cores, magnetic refrigerant materials, delay lines, motors, generators, and non-magnetic drill collar coating [6–8]. High and low [9–12] magnetized materials have various applications; therefore, the magnetic behaviors of BMG materials for such applications must be examined although some of these behaviors have not been extensively explored in the literature [13, 14].

Considerable efforts have been devoted to develop Fe-based BMGs with a high glass-forming ability (GFA) and favorable soft magnetic properties. The observed soft magnetism in several Fe-based BMGs is mostly attributed to the absence of less magnetocrystalline anisotropy; however, the rapid quenching during the fabrication process develops a local magnetoelastic anisotropy that may also be a source of magnetic hardening [15–17]. Incorporating B atoms may increase the packing density for enhancing GFA, but the excessive addition of these atoms

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may lead to magnetic hardening because of the formation of the FeB phase. The large grain size (>50 nm) of FeB increases coercivity (H_c) because FeB particles have a high magnetocrystalline anisotropy [18]. Therefore, the amount of element additions, such as Mo, C, and B, must be carefully determined.

Adding a small amount of Cu promotes the nucleation of a bcc ferromagnetic (FM) nanocrystalline phase that improves the beneficial properties of magnetic BMG materials. Given the insolubility of Cu in Fe, the addition of Cu can refine the primary particles and promote uniform grain dispersion [19, 20]. Ayers *et al.* [21] found that Cu functioned as a nucleation site for FM α -Fe particle formation and fluctuated the concentration of Fe between Cu-rich clusters, thereby generating additional nucleation sites and reducing grain size. Cu-containing nanocrystalline BMG alloys also demonstrated improved soft magnetic behaviors, such as high permeability, low H_c , and low core loss, than Cu-free nanocrystalline BMG alloys.

Adding a small amount of Ni or Co in Fe-based amorphous alloys can improve the magnetic properties of these alloys [18, 22]. According to the empirical rule of Inoue [23], at least three or more elements must be present to form a BMG material. However, the mechanism behind the formation of different BMG alloys remains unclear; in particular, researchers need to investigate why some alloys can be easily cast into bulk form, whereas others cannot be cast into this form [24, 25]. The addition of Co can also improve the magnetic properties of Fe-based BMGs [26]. Wang *et al.* [27] observed that the addition of Co decreased the onset crystallization temperature and increased the Curie temperature (T_C) of BMGs.

This study investigates the glass-forming ability, structure, magnetic properties, thermal stability of four kinds of BMGs ($\text{Fe}_{43}\text{Mn}_5\text{Cr}_{15}\text{Mo}_{14}\text{C}_{15}\text{B}_6\text{Y}_2$, $\text{Fe}_{43}\text{Co}_5\text{Cr}_{15}\text{Mo}_{14}\text{C}_{15}\text{B}_6\text{Y}_2$, $\text{Fe}_{43}\text{Ni}_5\text{Cr}_{15}\text{Mo}_{14}\text{C}_{15}\text{B}_6\text{Y}_2$, and $\text{Fe}_{43}\text{Cu}_5\text{Cr}_{15}\text{Mo}_{14}\text{C}_{15}\text{B}_6\text{Y}_2$) marked as Mn₅, Co₅, Ni₅, and Cu₅, respectively. Ni₅ has the lowest H_c and the highest GFA, saturation magnetization (M_s), and T_C .

2 Experimental

$\text{Fe}_{43}\text{M}_5\text{Cr}_{15}\text{Mo}_{14}\text{C}_{15}\text{B}_6\text{Y}_2$ ($M = \text{Mn, Cu, Co, and Ni}$ in at.%) BMGs with a length of 60 mm and a diameter of 3 mm were prepared via a two-step arc-melting and suction casting process. Ingots with nominal compositions of $\text{Fe}_{43}\text{M}_5\text{Cr}_{15}\text{Mo}_{14}\text{C}_{15}\text{B}_6\text{Y}_2$ ($M = \text{Mn, Cu, Co, and Ni}$ in at.%) were produced through arc-melting using a commercial-grade mixture of pure elements. All the elements had approximately 99.0 wt% purity, and melting was performed under a Ti-gettered Ar atmosphere. To ensure chemical homogeneity, each ingot was remelted at least

thrice. The rod-shaped BMG alloys were then fabricated with nominal dimensions of 3 mm $\times$ 3 mm $\times$ 60 mm by drop-casting the remelted alloys into the Cu mold. The rod was cut into disks with a thickness of ~ 2 mm using a low-speed diamond saw.

The thermal stability of the Fe-based BMG alloys was examined using a differential scanning calorimeter (DSC) (Q600, TG-DSC, TA-American) in an Ar atmosphere at a heating rate of 20 °C/min. The microstructure of the BMG alloys was characterized via X-ray diffraction (XRD) (D/max2550, Rigaku, Japan) with $\text{CuK}_{\alpha 1}$ radiation. The XRD device was operated at a voltage of 40 kV, a current of 100 mA, a step size of 0.02°, and a scanning rate of 5°/min. The M_s and H_c values were measured using a vibrating sample magnetometer (VSM) (Quantum Design, USA). The magnetization measurements were taken at two temperatures (20 and 300 K) under an applied magnetic field ranging from 0 to ± 50 kOe. During the temperature-dependent field-cooled (FC) magnetization test, temperature varied from 10 to 300 K under a magnetic field of 500 Oe. The VSM had a detection sensitivity of 10^{-6} emu.

3 Results and Discussion

Figure 1 shows the XRD patterns of the suction-cast Mn₅, Co₅, Ni₅, and Cu₅ samples with a nominal diameter of 3 mm. The XRD patterns show a major non-Bragg broad peak for all the samples with minor Bragg peaks, suggesting that the samples are mostly amorphous. Several minor peaks of α -Fe, FeCr, FeB, Co_2B , Co_5Y_8 , Mn_5O_8 , Fe_3C , MnO, CrMn, FeMn_3 , and $(\text{Cr, Fe})_{23}\text{C}_6$ are detected in the BMG alloys.

Figure 2 shows the DSC curves for the Mn₅, Co₅, Ni₅, and Cu₅ alloys. The curves indicate a distinct glass

Fig. 1 XRD patterns for suction-cast BMG alloys with 5 at.% Mn, Co, Ni, and Cu

Fig. 2 DSC plots for different BMG alloys with 5 at.% Mn, Co, Ni, and Cu

transition that is followed by a supercooled liquid region ($\Delta T_x = T_x - T_g$) before crystallization, which confirms that all the BMG alloys are essentially amorphous. The glass transition temperature (T_g) and crystallization temperature (T_x) are, respectively, defined as the onset of the endothermic DSC event and the onset temperature of the first exothermic event in the DSC curve. Table 1 lists the thermal properties of all the BMG samples, including T_g , T_x , and $\Delta T_x = T_x - T_g$, and the enthalpy of crystallization. The large ΔT_x widths of all the BMG alloys suggest that the glassy phase in the samples is highly stable and resistant to crystallization.

The GFA of BMG alloys is directly related to ΔT_x [28, 29], that is, a larger ΔT_x indicates better thermal stability of a BMG material. Table 1 shows that the Ni₅ alloy maintains the largest supercooled liquid region and the highest enthalpy of crystallization. The Fe–Cu, Fe–Co, Fe–Ni, and Fe–Mn pairs are mixed at +13, –1, –2, and 0 kJ/mol heat, respectively [30]. Therefore, the heat of mixing Fe with Ni, Cu, Mn, and Co is crucial in ensuring the thermal stability of the BMG materials. Given that Fe–Ni has the lowest mixing heat among the four pairs, Ni₅ has the highest GFA. By contrast, given that Fe–Cu has the highest mixing heat, Cu₅ exhibits the poorest thermal stability, smallest enthalpy of crystallization, and lowest GFA among the four alloys.

Given that Cu has a positive heat of mixing with Fe, these atoms tend to repel each other instead of forming a bond during the melting process. Therefore, the addition of Cu is detrimental to GFA because of the repelling effect between Cu and Fe. This repelling effect also effectively explains why the enthalpy of crystallization and GFA of the Cu₅ sample are significantly lower than those of the other samples.

Figure 3 shows the field-dependent magnetization for the suction-cast BMG alloys at room temperature (300 K). All the alloys have a relatively very low magnetization even after applying a magnetic field of 50 kOe. Ni₅ exhibits the highest magnetization, whereas Mn₅ presents the lowest magnetization. Although both Co and Ni are FM elements and despite the fact that the magnetic moment/atom of Co (1.76 μ_B) is significantly higher than that of Ni (0.606 μ_B), Ni₅ exhibits high magnetization after replacing the same amount of Fe with Co and Ni. By contrast, although both Cu and Mn are non-magnetic elements, Cu₅ achieves high magnetization after replacing the same atomic percentage of Fe with Mn and Cu.

Figure 4 shows the field-dependent magnetization for BMG alloys at 20 K. Although the magnetic properties and magnetization values of all the BMG materials under low temperature differ from those under room temperature, a similar magnetization trend is observed under both temperatures. In particular, Ni₅ obtains the highest M_s value, followed by Co₅, Cu₅, and Mn₅. The XRD measurement shows FM α -Fe, Fe₃C, FeB, FeCr, and ferrimagnetic (FIM) NiFe₂O₄ phases in Ni₅. By contrast, Co₅ contains neither the FM FeB phase nor the FIM NiFe₂O₄ phase within the XRD detection limit, but contains the antiferromagnetic (anti-FM) CoO phase. Co has a high affinity with oxygen, thereby readily forming oxides with ambient oxygen during the fabrication process. The aforementioned observation explains why Ni₅ has a higher M_s value than Co₅ although both alloys incorporate the same amount of Ni and Co atoms and the magnetic moment per Co atom is higher than that per Ni atom.

Although both Cu and Mn are non-magnetic elements, Fig. 5 shows that after replacing the same atomic percentage of Fe with Mn and Cu, Cu₅ obtains a higher M_s than Mn₅. Both Cu₅ and Mn₅ have FM α -Fe, FeCr, and

Table 1 Thermal properties of different BMG alloys with 5 at.% Mn, Co, Ni, and Cu

Alloy	T_g (°C)	T_x (°C)	ΔT_x (°C)	Enthalpy of crystallization (J/g)
Ni ₅	540.76	628.07	87.31	115.22 ± 5.75
Co ₅	541.59	614.89	73.30	92.67 ± 4.45
Cu ₅	580.0	647.83	67.84	88.0 ± 4.53
Mn ₅	542.69	616.81	74.12	111.62 ± 5.45

Fig. 3 Field-dependent magnetization for suction-cast BMGs with 5 at.% Mn, Co, Ni, and Cu at 300 K

Fig. 4 Field-dependent magnetization for suction-cast BMGs with 5 at.% Mn, Co, Ni, and Cu at 20 K

Fig. 5 Magnetization for the suction-cast BMGs as a function of 5 at.% Mn, Co, Ni, and Cu

Fe_3C phases, but the $\alpha\text{-Fe}$, FeCr , and Fe_3C phases have larger crystallite sizes in Cu_5 than in Mn_5 . Contrary to that of the Fe-Mn pair, the positive heat of mixing [30] of the

Fe-Cu pair induces a repelling effect between Fe and Cu, which, in turn, induces the segregation of different FM nanocrystals, thereby increasing the M_s value of Cu_5 . Anti-FM MnO and Mn_5O_8 phases also contribute to the reduced M_s value of Mn_5 .

As indicated in Table 2, Mn_5 shows the highest H_c value (63.52 ± 3.27), whereas Ni_5 exhibits the lowest H_c value (30.93 ± 1.85). H_c increases along with the mean grain size of different crystallites [31–33]. To observe the influence of this effect, we calculate the grain sizes of $\alpha\text{-Fe}$, Fe_3C , and FeCr in different alloys using the Scherrer equation, $\tau = \frac{K\lambda}{\beta \cos \theta}$, where τ is the grain size, K is a dimensionless shape factor, λ is the X-ray wavelength, β is the full width at half maximum of a Bragg peak, and θ is the Bragg angle [34].

The combined grain size of FM $\alpha\text{-Fe}$, Fe_3C , and FeCr phases in different BMG alloys determines the H_c values. In general, a larger combined grain size indicates a higher H_c value. Table 3 shows that the combined grain size of $\alpha\text{-Fe}$, Fe_3C , and FeCr phases in Mn_5 or Cu_5 is significantly larger than that in Ni_5 or Co_5 . Fe_3C has an extremely large magnetocrystalline anisotropy constant (430 kJ/m^3) [35]. Although Cu_5 has the largest Fe_3C and combined grain sizes, Mn_5 presents a higher H_c value. Therefore, Mn_5 has the highest H_c value among all the BMGs, which can be attributed to the large combined grain size of the three FM phases as well as to the formation of the intermetallic phases of FeMn_3 and FeMn_4 because such phases can function as a pinning center for the domain wall movement [36].

Figure 6 shows the temperature-dependent magnetization for suction-cast BMGs with 5 at.% Mn, Cu, Co, and Ni under an applied magnetic field of 500 Oe. For all the BMG samples, we calculate the T_C values from the temperature-dependent magnetization curves. We also calculate the effective magnetic moment (m_{eff}) and Curie–Weiss temperature (θ) by fitting the temperature-dependent magnetization curves into the Curie–Weiss law as follows:

$$\frac{M}{H} = \frac{C}{T - \theta},$$

Table 2 Magnetic properties of $\text{Fe}_{43}\text{M}_5\text{Cr}_{15}\text{Mo}_{14}\text{C}_{15}\text{B}_6\text{Y}_2$ ($M = \text{Mn}, \text{Cu}, \text{Co}, \text{and Ni at.}\%$) BMG alloys

Alloy	T_C (K)	H_c (Oe)	θ (K)	m_{eff} (μ_B)
Mn_5	28.13	63.52 ± 3.27	18.78 ± 0.27	2.82 ± 0.12
Cu_5	28.93	58.35 ± 2.80	45.56 ± 0.27	2.93 ± 0.12
Co_5	40.77	52.80 ± 2.46	80.25 ± 0.14	3.28 ± 0.13
Ni_5	58.21	30.93 ± 1.85	96.17 ± 0.23	3.58 ± 0.21

Table 3 Grain sizes of α -Fe, Fe_3C , and FeCr phases in $\text{Fe}_{43}\text{M}_5\text{Cr}_{15}\text{Mo}_{14}\text{C}_{15}\text{B}_6\text{Y}_2$ ($M = \text{Mn, Cu, Co, and Ni at.}\%$) BMG alloys

Alloy	Fe (nm)	Fe_3C (nm)	FeCr (nm)	Total (nm)
Mn_5	32.1	75.3	47.9	155.3
Cu_5	45.9	66.7	52.9	165
Co_5	75.1	22.2	33.1	130
Ni_5	37.6	46	39.7	123

Fig. 6 Temperature-dependent magnetization for suction-cast BMGs with 5 at.% Mn, Cu, Co, and Ni under an applied magnetic field of 500 Oe

where M is the magnetization, H is the applied magnetic field, C is the Curie constant, T is the temperature, and θ is the Curie–Weiss temperature.

As shown in Table 2, Ni_5 exhibits the largest T_C value, which is obviously attributed to its formation of the highest numbers of FM and FIM phases. Mn_5 presents the lowest T_C value despite the facts that both Cu and Mn are non-magnetic elements and FM α -Fe, FeCr, and Fe_3C phases are present in both Cu_5 and Mn_5 . In addition to FM phases, Mn_5 also contains anti-FM MnO and Mn_5O_8 phases, which explains why Mn_5 exhibits the lowest T_C value among all the alloys. Mn_5 also has an extremely low θ value compared with the other alloys because of the strong competition between FM and anti-FM interactions. Ni_5 has a considerably larger θ value than Mn_5 , which indicates a stronger positive exchange interaction in Ni_5 than in Mn_5 .

Ni_5 exhibits the highest m_{eff} value of Fe, whereas Mn_5 presents the lowest m_{eff} value. In general, Fe–Ni and Ni–Ni couplings are FM, and the Fe in Fe–Ni may have a larger magnetic moment than that in fcc [37]. By contrast, Fe–Mn [38, 39] and Mn–Mn couplings are anti-FM [40], which explains why Ni_5 has the largest m_{eff} value. Although both Cu and Mn are non-magnetic elements, Cu can couple with Fe ferromagnetically and antiferromagnetically depending

on the local environment [41], thereby explaining why the m_{eff} value of Cu_5 is slightly higher than that of Mn_5 .

4 Conclusion

The effects of 5 at.% Mn, Cu, Co, and Ni addition on the GFA, thermal, and structural and magnetic properties of $\text{Fe}_{43}\text{M}_5\text{Cr}_{15}\text{Mo}_{14}\text{C}_{15}\text{B}_6\text{Y}_2$ ($M = \text{Mn, Cu, Co, and Ni}$) BMG alloys were systematically studied.

Among the four BMG alloys, Ni_5 exhibits the lowest H_c value and the highest GFA, M_s , and T_C values. By contrast, Mn_5 demonstrates the poorest magnetic properties, whereas Cu_5 presents the lowest thermal stability and GFA. The heat of mixing of Fe–Mn, Fe–Co, Fe–Cu, and Fe–Ni pairs is important in ensuring the thermal stability of different BMG materials. Given that the Fe–Ni pair has the lowest heat of mixing value among the four pairs, Ni_5 exhibits the highest GFA among all the BMG alloys. By contrast, given that the Fe–Cu pair has the highest heat of mixing, Cu_5 demonstrates the poorest thermal stability, smallest enthalpy of crystallization, and lowest GFA among all the alloys. With regard to magnetic properties, Ni_5 achieves the highest M_s value, followed by Co_5 , Cu_5 , and Mn_5 . The formation of additional FM and FIM phases in Ni_5 explains why the M_s and T_C values in Ni_5 are higher than those in Co_5 although the magnetic moment per Co is higher than that per Ni. Although both Cu and Mn are non-magnetic elements, Cu_5 presents a higher M_s value than Mn_5 after replacing the same atomic percentage of Fe with Mn and Cu. The existence of numerous FM nanocrystals in Cu_5 , which can be attributed to the positive heat of mixing between Fe and Cu atoms, explains why Cu_5 has higher M_s and T_C values than Mn_5 . Few anti-FM phases are also detected in Mn_5 , thereby decreasing the M_s and T_C values of this alloy.

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