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Formability and magnetic properties of Dy-Co binary amorphous alloys

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In this study, binary Dy-Co ribbons were synthesized by a conventional melt-spinning approach and glassy ribbons were successfully obtained within the compositional range Dy$_{50}$Co$_{50}$ to Dy$_{68}$Co$_{32}$. The glass formability and magnetic properties of these amorphous alloys were examined. The compositional dependence of glass formability, Curie temperature and magneto-caloric response of the Dy$_x$Co$_{100-x}$ ($x=50, 55, 60, 65$ and $68$) amorphous alloys, as well as the mechanism involved, were determined.

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I. INTRODUCTION

Amorphous alloys have huge scientific and industrial significance because of their superior properties resulting from their unique structure induced by fabrication conditions far from the equilibrium state. Among these alloys, transition metal (TM) and rare earth (RE) based amorphous alloys have shown application potential because they exhibit pre-eminant magnetic properties. For instance, Nd(Pr)-TM-based bulk metallic glasses (BMGs) show anomalous large coercivity; (Tb, Dy)-TM-based amorphous alloys exhibit extraordinary magnetostriction; and Gd-Co-based metallic glasses exhibit an outstanding magnetocaloric effect (MCE).

Gd-TM-based glassy alloys possess a second order magnetic phase transition (MPT), which leads to a broadened magnetic entropy change ($\Delta S_m$) peak and also an ultra-high refrigeration capacity. However, the peak of $\Delta S_m$ ($\Delta S_m^{peak}$) of the Gd-TM-based glassy alloys is usually inferior to that of the intermetallic compounds undergoing a first order MPT. Therefore, it is essential to find ways to enhance the value of the $\Delta S_m^{peak}$ of Gd-TM-based amorphous alloys.

One of the valid methods to improve the MCE and formability of Gd-TM-based amorphous alloys is to replace the Gd elements by other RE metals, such as Tb, Dy, Ho, Er, Y and so on. However, the addition of Tb and Dy may induce spin-glass-like behavior and make the magnetic entropy change of the amorphous alloys irreversible. Therefore, systematic investigation of the formability and magnetic properties of Tb/Dy-TM binary amorphous alloys can be a simple but valid way to understand the influence of the addition of Tb or Dy on the formability and MCE of amorphous alloys in more detail.

In this investigation, we studied the glass formability and magnetic properties of a representative binary Dy-Co amorphous alloy system. The amorphous Dy-Co alloys were prepared as ribbons by the conventional melt-spinning method, and the best composition for glass formation in this alloy system was identified. The compositional dependence of the glass formability, Curie temperature ($T_c$) and $\Delta S_m$ of the binary glassy alloys were constructed, and the mechanisms were investigated.

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The findings are helpful for understanding the formability, $T_c$ and magnetocaloric responses of Tb/Dy-TM-based amorphous alloys.

II. EXPERIMENTAL PROCEDURES

Dy$_x$Co$_{100-x}$ (x=45, 50, 55, 60, 65, 68, 70 and 75) alloys were fabricated by arc-melting mixtures of high purity Dy (99.9 at. %) and Co (99.9 at. %) metals under a Ti-gettered argon atmosphere. Binary Dy$_x$Co$_{100-x}$ ribbons were fabricated by spinning the melting liquid on a copper wheel with a surface linear velocity of 30 m/s under a highly purified argon atmosphere. The approximate width of the as-prepared ribbon was 1 mm and the average thickness was about 40 $\mu$m. The structure state of the Dy$_x$Co$_{100-x}$ ribbons was confirmed using X-ray diffraction (XRD) analysis with Cu $K_\alpha$ radiation (Rigaku D\max-2550). High resolution electron microscopy (HREM) observations were performed using a JEOL JEM-2010F high resolution electron microscope to ascertain the disordered microstructure of the amorphous ribbons. Specimens for HREM observation were prepared by ion-polishing under a pure argon atmosphere using the GATAN 691 precision ion-polishing system. The thermal properties of the Dy$_x$Co$_{100-x}$ amorphous ribbons were measured using differential scanning calorimetry (DSC, Perkin-Elmer DIAMOND) under a highly purified argon atmosphere at a constant heating rate of 20 K/min. The magnetic properties of the Dy$_x$Co$_{100-x}$ ribbons were acquired using a Quantum Design Physical Properties Measurement System (PPMS 6000). The temperature dependence of the magnetization ($M$-$T$) curves were obtained under a magnetic field of 0.03 T in the heating process after cooling from room temperature to 10 K under a zero field (ZFC) or a magnetic field of 0.03 T (FC). The hysteresis loops and the isothermal magnetization ($M$-$H$) curves were obtained at different selected temperatures under a magnetic field of 5 T.

III. RESULTS AND DISCUSSION

Figure 1 displays the XRD patterns of the obtained Dy$_x$Co$_{100-x}$ (x=45, 50, 55, 60, 65, 68, 70 and 75) ribbons. The broadened diffraction maxima in the XRD patterns of the as-spun Dy$_{50}$Co$_{50}$, Dy$_{55}$Co$_{45}$, Dy$_{60}$Co$_{40}$, Dy$_{65}$Co$_{35}$ and Dy$_{68}$Co$_{32}$ ribbons reveal the typical amorphous features of these samples. In contrast, the Dy$_{45}$Co$_{55}$, Dy$_{70}$Co$_{30}$ and Dy$_{75}$Co$_{25}$ are partially or fully crystallized.

A. Glass forming ability of the Dy-Co binary alloys

The DSC curves for the Dy$_x$Co$_{100-x}$ (x=50, 55, 60 and 68) amorphous ribbons are shown in Fig. 2(a). The amorphous characteristics of these ribbons are further verified by the typical endothermic glass transition and exothermic crystallization behavior in the DSC curves. The onset temperature for glass transition ($T_g$) and crystallization ($T_x$) of each amorphous ribbon, as marked clearly in Fig. 2(a), are listed in Table I. Therefore, associated with the liquidus temperatures ($T_l$) of the alloys acquired from the binary phase diagram, we calculated the reduced glass transition temperature.
The continuous DSC curves of the Dy$_x$Co$_{100-x}$ (x=50, 55, 60, 65 and 68) ribbons obtained at a constant heating rate of 20 K/min, and the compositional dependence of $T_{rg}$ and $\gamma$ of the Dy$_x$Co$_{100-x}$ amorphous ribbons.

Fig. 2: (a) The continuous DSC curves of the Dy$_x$Co$_{100-x}$ (x=50, 55, 60, 65 and 68) ribbons obtained at a constant heating rate of 20 K/min, and (b) the compositional dependence of $T_{rg}$ and $\gamma$ of the Dy$_x$Co$_{100-x}$ amorphous ribbons.

The compositional dependence of $T_{rg}$ and $\gamma$ of the Dy$_x$Co$_{100-x}$ amorphous alloys. This enabled investigation of the formability of the amorphous alloys because the above two parameters are the most widely used gauges for evaluating the formability of metallic glasses. The compositional dependence of $T_{rg}$ and $\gamma$ of the Dy$_x$Co$_{100-x}$ amorphous ribbons is shown in Fig. 2(b). It is clear that the eutectic Dy$_{60}$Co$_{40}$ provides the best composition for glass forming in the Dy-Co alloy system, which is in accord with the deep eutectic rule for predicting the glass formability of the alloys.\textsuperscript{21,22}

B. Magnetic properties of the amorphous alloys

Figure 3(a) displays the ZFC and FC $M$-$T$ curves of the Dy$_{55}$Co$_{45}$ amorphous ribbon. The ZFC $M$-$T$ curve nearly overlaps with the FC $M$-$T$ curve within the temperature range 70 - 300 K and diverges from the FC $M$-$T$ curve at temperatures lower than 70 K. The difference between the ZFC and FC $M$-$T$ curves of the Dy$_{55}$Co$_{45}$ amorphous ribbon is analogous to those of spin glass systems.\textsuperscript{8,18,19} The spin-glass-like behavior is also illustrated by the $M$-$H$ curves of the Dy$_{55}$Co$_{45}$ amorphous ribbon, as demonstrated in the upper-right inset of Fig. 3(a). The decreasing magnetization under a very low magnetic field from 50K to 10 K indicates the typical spin glass behavior of the Dy$_{55}$Co$_{45}$ amorphous ribbon. However, this kind of ZFC-FC curve and $M$-$H$ curve can also be found in nano-structured amorphous composites as a result of their superparamagnetic behavior. Therefore, HREM observation of the Dy$_{55}$Co$_{45}$ ribbon was undertaken to ascertain the disordered microstructure of the as-spun ribbons. Figure 4 shows the HREM image of the as-spun ribbon. The Dy$_{55}$Co$_{45}$ as-spun ribbon is fully amorphous with only short ranged orders, and no obvious crystalline phases or small grains have been found in the amorphous matrix.

| Dy$_x$Co$_{100-x}$ ribbons | $T_g$ (K) | $T_{x onset}$ (K) | $T_f$ (K) | $T_{rg}$ (K) | $\gamma$ | $T_c$ (K) | $T_f$ (K) |
|-----------------------------|-----------|------------------|-----------|-------------|--------|-----------|-----------|
| x=50                        | 521       | 578              | 1253      | 0.416       | 0.326  | 106       | 80        |
| x=55                        | 514       | 620              | 1133      | 0.454       | 0.376  | 73        | 55        |
| x=60                        | 516       | 615              | 1018      | 0.507       | 0.401  | 54        | 45        |
| x=65                        | 518       | 610              | 1038      | 0.499       | 0.392  | 46        | 35        |
| x=68                        | 516       | 602              | 1028      | 0.502       | 0.390  | 43        | 33        |

| Dy$_x$Co$_{100-x}$ ribbons | $\Delta S_m^{peak}$ (J K$^{-1}$ kg$^{-1}$) |
|-----------------------------|-------------------------------------------|
| x=50                        | 1.8                                       |
| x=55                        | 2.5                                       |
| x=60                        | 3.19                                      |
| x=65                        | 3.65                                      |
| x=68                        | 3.81                                      |

| 1 T | 1.5 T | 2 T | 3 T | 5 T |
|-----|-------|-----|-----|-----|
| x=50| 1.8   | 2.55| 3.24| 4.53| 6.82|
| x=55| 2.5   | 3.5 | 4.41| 6.05| 8.75|
| x=60| 3.19  | 4.43| 5.53| 7.41| 10.39|
| x=65| 3.65  | 5   | 6.19| 8.2 | 11.3|
| x=68| 3.81  | 5.3 | 6.59| 8.86| 12.58|
FIG. 3. ZFC and FC $M-T$ curves, Curie temperatures ($T_C$) and spin freezing temperatures ($T_f$), $M-H$ curves (upper right inset) and hysteresis loops (lower right inset) of the (a) Dy$_{55}$Co$_{45}$, (b) Dy$_{60}$Co$_{40}$, (c) Dy$_{65}$Co$_{35}$ and (d) Dy$_{68}$Co$_{32}$ ribbons.

It is known that Dy(Tb)-based amorphous systems involve huge random magnetic anisotropy (RAM) due to the local random electrostatic field. According to the random anisotropy model, the RMA existing in amorphous alloys will break the rotational symmetry of the Hamiltonian and increase the hysteresis.$^{8,23}$ The hysteresis loops of the Dy$_{55}$Co$_{45}$ amorphous ribbon were measured under a magnetic field of 5 T at 10 K, 30 K, 45 K, 50 K, 60 K and 300 K, respectively, as displayed in the lower-right inset of Fig. 3(a). The ribbon is paramagnetic at ambient temperatures and is soft magnetic with nearly zero coercivity from 50 K to 60 K, but is hard magnetic at 10 K and 45 K. The coercivity is about 0.03 T at 45 K, about 0.09 T at 30 K and about 0.46 T at 10 K, all due to the strong RMA at lower temperatures. The decreased coercivity with increasing temperature is due to the unfreezing of the magnetic moment due to the thermal fluctuation. $T_C$ of the Dy$_{55}$Co$_{45}$ amorphous ribbon is about 73 K, and the spin freezing temperature ($T_f$) is about 55 K.

FIG. 4. HREM image of the Dy$_{55}$Co$_{45}$ as-spun ribbon.
The $M$-$T$ (including ZFC and FC) curves, $M$-$H$ curves and hysteresis loops of the other Dy$_x$Co$_{100-x}$ amorphous ribbons are shown in Fig. 3(b) for x=60, Fig. 3(c) for x=65 and Fig. 3(d) for x=68. Agreeing with our preliminary work on Dy$_{50}$Co$_{50}$ amorphous ribbons, all the Dy$_x$Co$_{100-x}$ amorphous ribbons show spin-glass-like behavior. The Curie temperatures and spin freezing temperatures for the Dy$_x$Co$_{100-x}$ (x=50, 55, 60, 65 and 68) amorphous ribbons are also listed in Table I.

One of the characteristics of amorphous alloys that is superior to intermetallic compounds is that $T_c$ of the amorphous alloys can be adjusted by customizing the composition of the alloys. In our previous work, we found a linear relationship between the Gd content and $T_c$ in Gd-Co binary amorphous alloys. The compositional dependence of $T_c$ of RE-Co-based amorphous alloys was investigated. Based on the model of Rudermann-Kittel-Kasuya-Yosida indirect interaction, a linear relationship between $T_c$ and the de Gennes factor (G) was found in various RE-Co-based amorphous alloys. For amorphous alloys containing only one RE element, the G factor is proportional to the molar fraction of the RE atoms. Therefore, from the viewpoint of $4f-4f$ indirect interaction, the compositional dependence of $T_c$ in Dy-Co amorphous alloys should be linear. However, in addition to the $4f-4f$ indirect interaction between the RE-RE atoms, there are two other kinds of interactions in RE-TM-based amorphous alloys: 3$d$-3$d$ direct interaction between the TM-TM atoms and the $3d$-$4f$ indirect interaction between the RE-TM atoms. The influence of 3$d$-3$d$ direct interaction on the Curie temperature in the RE-TM-based amorphous alloys is supposed to be similar to that of the $4f$-$4f$ indirect interaction. This is understandable because there is only (100%) 3$d$-3$d$ direct interaction in a pure TM metal and no (0%) 3$d$-3$d$ direct interaction in alloys free of TM elements. The contribution of the 3$d$-$3d$ direct interaction is proportional to the molar fraction of the Co element in Dy-Co amorphous alloys and the compositional dependence of $T_c$ in Dy-Co amorphous alloys is still linear from the viewpoint of 3$d$-3$d$ direct interaction. However, the effect of 3$d$-$4f$ indirect interaction on the Curie temperature in RE-TM-based amorphous alloys is more complicated. The 3$d$-$4f$ indirect interaction exists in RE-TM amorphous alloys but does not exist in either RE free or TM free alloys, which means that the compositional dependence of the 3$d$-$4f$ indirect interaction in Dy-Co amorphous alloys will not be linear. As a result, the compositional dependence of $T_c$, determined by the three kinds of interactions in RE-TM-based amorphous alloys, is not linear. Figure 5 displays the compositional dependence of $T_c$ for the Dy$_x$Co$_{100-x}$ glassy ribbons. The non-linear $T_c$-Dy (at. %) curve indicates that the non-linear compositional dependence of the 3$d$-$4f$ indirect interaction plays a significant role in dominating the Curie temperature of Dy-Co amorphous alloys. The nearly linear dependence of $T_c$ on the Gd content in Gd-Co amorphous alloys is likely due to the negligible 3$d$-$4f$ indirect interaction compared to the 3$d$-3$d$ direct interaction and $4f$-$4f$ indirect interaction in Gd-Co binary amorphous alloys.

![Figure 5](image-url)  
FIG. 5. The compositional dependence of $T_c$ for the Dy$_x$Co$_{100-x}$ (x=50, 55, 60, 65 and 68) ribbons.
C. Magneto-caloric response of the amorphous alloys

The spin freezing behavior is reported to make the magnetic entropy change irreversible at temperatures well below $T_f$ and reduce the magneto-caloric properties of Dy-based bulk metallic glasses.\(^{18,19}\) Therefore, we determined the temperature dependence of $-\Delta S_m$ from the $M$-$H$ curves of the Dy$_x$Co$_{100-x}$ amorphous ribbons above $T_f$, as shown in Fig. 6. The $-\Delta S_m^{\text{peak}}$ of the Dy$_x$Co$_{100-x}$ amorphous ribbons clearly increases with the decreasing Dy concentration. However, the MCE of the Dy-Co amorphous alloys is not so significant when compared to that of the Gd-Co-based amorphous alloys. This is possibly because that Gd has no orbital momentum with a relatively low magneto-crystalline anisotropy.\(^{10–18,28}\)

The dependence of $-\Delta S_m^{\text{peak}}$ on the $T_c$ of the amorphous alloys is usually described by the $-\Delta S_m^{\text{peak}} \propto T_c^{-2/3}$ relationship, as proposed by Belo et al., according to the mean field theory approach.\(^{26}\) According to the $-\Delta S_m^{\text{peak}}$ values for the Dy$_x$Co$_{100-x}$ amorphous ribbons under the various magnetic fields listed in Table I, we constructed $\ln(-\Delta S_m^{\text{peak}}) \ vs \ ln(T_c)$ plots under various fields for the amorphous alloys, as shown in Fig. 7(a). By linear fitting of the $\ln(-\Delta S_m^{\text{peak}}) \ vs \ ln(T_c)$ plots, we obtained the $-\Delta S_m^{\text{peak}} \propto T_c^{-m}$ relationship for amorphous alloys: where $m = -0.85$ under 1 T; $m = -0.82$ under 1.5 T; $m = -0.8$ under 2 T; $m = -0.74$ under 3 T and $m = -0.66$ under 5 T. Although the $m$ value approximates to $-2/3$ under a field of 5 T, it is much lower than $-2/3$ under magnetic fields lower than 5 T, indicating that the $-\Delta S_m^{\text{peak}}$-$T_c$ relationship of Dy$_x$Co$_{100-x}$ amorphous alloys is different from the mean field theory prediction.

FIG. 6. The $(-\Delta S_m)$-$T$ curves of the Dy$_x$Co$_{100-x}$ ($x=50, 55, 60, 65$ and 68) ribbons under the magnetic fields of 1.5 T and 5 T.

FIG. 7. (a) The linear fittings of the $\ln(T_c)$ vs $\ln(-\Delta S_m^{\text{peak}})$ plots under various fields, and (b) the linear fittings of $\ln(H)$ vs $\ln(-\Delta S_m^{\text{peak}})$ plots for the Dy$_x$Co$_{100-x}$ ($x=50, 55, 60, 65$ and 68) ribbons.
The deviation of the \(-\Delta S_m^{\text{peak}}-T_c\) relationship of the Dy$_x$Co$_{100-x}$ amorphous alloys from the mean field prediction can also be verified by the \(-\Delta S_m \propto H^n\) relationship of each amorphous ribbon. Figure 7(b) shows the linear fitting of \(\ln(H)\) vs \(\ln(-\Delta S_m^{\text{peak}})\) for the Dy$_x$Co$_{100-x}$ amorphous alloys. The \(n\) value near \(T_c\) is about 0.83 for \(x=50,\) 0.78 for \(x=55,\) 0.73 for \(x=60,\) 0.7 for \(x=65\) and 0.74 for \(x=68,\) all of which are larger than the value predicted from the mean field theory, but agree well with the results predicted by the Arrott-Noakes equation, according to the typical microstructure of amorphous alloys with numerous short range orders embedded in the disordered matrix.\(^{12,27}\)

IV. CONCLUSIONS

In summary, the glass formability and magnetic properties of the binary Dy-Co amorphous alloys were investigated in this work. Dy-Co ribbons were fabricated by the melt-spinning method at a wheel surface velocity of 30 m/s and Dy$_x$Co$_{100-x}$ \((x=50, 55, 60, 65\) and 68) amorphous ribbons were obtained. The glass formability of the Dy-Co binary alloys was examined, and the best composition for glass formation was found to be the Dy$_{60}$Co$_{40}$ alloy, which agrees well with the deep eutectic rule. The magnetic properties and the magnetocaloric response for the amorphous alloys were also investigated. It was found that the Curie temperature decreases with the augmenting Dy concentration. The compositional dependence of \(T_c\) shows a non-linear relationship, indicating that the non-linear compositional dependence of the 3d-4f indirect interaction plays a vital role in dominating the Curie temperature of Dy-Co binary amorphous alloys. All the Dy$_x$Co$_{100-x}$ amorphous ribbons show spin-glass-like behavior, with the spin freezing temperatures obtained. The \((\Delta S_m)^{-}T\) curves of the Dy$_x$Co$_{100-x}$ amorphous ribbons were constructed at temperatures above \(T_f\) to avoid the irreversible magnetic entropy change induced by the spin-glass-like behavior of the amorphous alloys. The dependence of \(-\Delta S_m^{\text{peak}}\) on the Curie temperature, as well as the magnetic field, was investigated. The deviation of the magneto-caloric behavior of the Dy$_x$Co$_{100-x}$ amorphous alloys from the mean field prediction was ascertained by both the \((\Delta S_m^{\text{peak}})^{-}T_c\) and the \(H-(\Delta S_m^{\text{peak}})\) relationships.

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