Thermal Stability and Soft Magnetic Properties of Co–Fe–M–B (M=Nb, Zr) Amorphous Alloys with Large Supercooled Liquid Region

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A ferromagnetic amorphous phase with a large supercooled liquid region prior to crystallization and good soft magnetic properties was formed in melt-spun Co$_{40}$Fe$_{22}$Nb$_{5-5x}$Zr$_x$B$_{30}$ ($x = 0, 2, 4, 6,$ and 8 at%) alloys. The glass transition temperature ($T_g$) increases monotonously with increasing Zr content. The crystallization temperature ($T_c$) also increases by the dissolution of Zr up to 2% and then decreases with further increasing Zr content. The increases are more significant for $T_c$, resulting in a maximum $AT_c$ ($=T_c - T_g$) of 98 K at 2% Zr. The Co-based amorphous alloys exhibit good soft magnetic properties, i.e., saturation magnetization of 0.41 T-0.42 T, low saturated magnetization of $2.4 \times 10^{-6}$-$2.9 \times 10^{-6}$, low coercivity of 1.2-2.0 A/m, and high permeability of 29000-33000 at 1 kHz over the whole Zr content range. In addition, the cylindrical Co$_{40}$Fe$_{22}$Nb$_{5-5x}$Zr$_x$B$_{30}$ glassy alloy with a diameter of 1.0 mm was prepared by the copper mold casting method. With increasing the cylindrical diameter up to 1.5 mm, the structure changes to coexistent amorphous, Co$_{21}$Nb$_2$B$_6$, Co$_3$ZrB$_2$ and Co$_2$B phases. It is noticed that the maximum thickness for glass formation is about 6 times larger than that for Co-based amorphous alloys reported up to date. The success of forming Co-based bulk amorphous alloys with high stability of supercooled liquid and good soft magnetic properties is encouraging further development of bulk amorphous soft magnetic materials.

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1. Introduction

Since the synthesis of a ferromagnetic Fe–P–C amorphous alloy in 1967, it has been found that some ferromagnetic Fe- and Co-based amorphous alloys exhibit good soft magnetic properties. However, the glass-forming ability of the Fe- and Co-based amorphous alloys is not always high and hence their formation has required higher cooling rates than about $10^3$ K/s. Therefore the resulting sample thickness by the ordinary melt spinning method has been limited to be less than about 50 µm for Fe–Si–B and Co–Si–B systems.

Recently, great effort has been devoted to the production of bulk amorphous alloys. As a result of great effort, Mg, Lanthanide (Ln), Zr, Pd–Cu, based amorphous alloys with a large supercooled liquid region above 60 K prior to crystallization have been found for the last decade. Among these amorphous alloys, the largest critical sample thickness reaches about 72 mm and the critical cooling rate is as low as 0.1 K/s. The search for an amorphous alloy with large supercooled liquid region prior to crystallization is important because the extension of the supercooled liquid region implies high resistance against crystallization. The use of such an alloy component enables us to produce a bulk amorphous alloy by the casting process with low cooling rates.

More recently, ferromagnetic Fe-based amorphous alloys with a large supercooled liquid region above 60 K before crystallization and good soft magnetic properties have been synthesized in Fe–(Al, Ga)–(P, C, B, Si, Ge) and Fe–(Co, Ni)–(Zr, Nb, Ta, Mo, W)–B systems. The critical thickness of Fe-based amorphous alloys reaches 3 mm for Fe–(Al, Ga)–(P, C, B, Si, Ge) and 5 mm for Fe–(Co, Ni)–(Zr, Nb, Ta, Mo, W)–B system by the copper mold casting method. Subsequently, ferromagnetic Co-based amorphous alloys with a large supercooled liquid region above 50 K were found in Co–Al–Ga–Fe–Cr–V)–P–B–C–Si and Co–Fe–(Zr, Nb, Ta, W)–B with 20 at% B systems. However, little is known about the production of Co-based bulk amorphous alloys and their magnetic properties. This paper is intended to present the thermal stability and magnetic properties of Co–Fe–M–B (M=Nb, Zr) amorphous alloys containing 30 at% B in melt-spinning and cast cylindrical forms.

2. Experimental Procedure

Multicomponent Co$_{40}$Fe$_{22}$Nb$_{5-5x}$Zr$_x$B$_{30}$ ($x = 0, 2, 4, 6$, and 8 at%) alloys ingots were prepared by arc melting pure Co, Fe, Nb and Zr metals and pure crystalline B in an argon atmosphere. From the master alloy ingots, ribbons with a cross section of about 1.0 x 0.02 mm$^2$ and cylinders with diameters up to 1.5 mm and a length of of 40 mm were prepared by single-roller melt-spinning and copper-mold casting methods, respectively, in an argon atmosphere. The amorphous structure was examined by X-ray diffractionmetry (XRD) with Cu-Kα radiation, optical microscopy (OM) and transmission electron microspectroscopy (TEM). The thermal stability associated with glass transition and crystallization was examined at a heating rate of 0.67 K/s by differential scanning calorimetry. Magnetization at room temperature was measured in a maximum applied field of 1500 kA/m with a vibrating sample magnetometer (VSM). Coercive force was measured with a I-H loop tracer. Permeability was evaluated at 1 kHz with an impedance analyzer. Saturated magnetostriiction was measured under a field of 240 kA/m by a three-terminal capacitance method.

3. Results

3.1 Thermal stability and crystallization behavior of melt-spun Co–Fe–Nb–Zr–B amorphous alloys

Figure 1 shows X-ray diffraction patterns of the melt-spun Co$_{40}$Fe$_{22}$Nb$_{5-5x}$Zr$_x$B$_{30}$ ($x = 0, 2, 4, 6$, and 8 at%) alloys.
Production of Co-based Amorphous Alloys with High-Glass Forming Ability

at room temperature. Broad diffraction peaks are seen at diffraction angles (2θ) of 44 and 78 degrees. No diffraction peak corresponding to a crystalline phase is seen for all the alloys. The X-ray diffraction data clearly indicate that these alloys are composed of an amorphous phase without any crystallinity. Figure 2 shows the DSC curves of the melt-spin Co_{40}Fe_{22}Nb_{x-y}Zr_{x}B_{30} (x = 0, 2, 4, 6, and 8 at%) alloys. The distinct glass transition, followed by a large supercooled liquid region is recognized in the temperature range prior to crystallization. Two exothermic peaks correspond to the crystallization have been observed for the 0% Zr alloy. It was reported that the glass transition temperature (T_g), the crystallization temperature (T_c), and the temperature interval of supercooled liquid region (ΔT_c = T_c - T_g) were plotted as a function of Zr content in Fig. 3. The T_g is 895 K for the 0% Zr alloy, increases by the dissolution of Zr, and shows a maximum value of 917 K for the 8% Zr alloy. The T_c also increases by the dissolution of Zr up to 2% and then decreases with further increasing Zr content. The increase is more significant for T_c than for T_g, leading to an increase in ΔT_c from 81 K for the 0% Zr alloy to 98 K for the 2% Zr alloy. The DSC data indicate that the substitution of Nb by 2% Zr in the Co_{40}Fe_{22}Nb_{x-y}B_{30} alloy is effective for the stabilization of the supercooled liquid.

In order to clarify the reason for the change in the crystallization behavior, the X-ray diffraction patterns of the Co_{40}Fe_{22}Nb_{x-y}Zr_{x}B_{30} (x = 0, 2, 4, 6, and 8 at%) alloys heated for 1.8 ks at the temperatures of T_g(T_g)+50 K are shown in Fig. 4. The X-ray diffraction pattern was identified as Co_{31}Nb_{2}B_{6} + Co_{3}B for the 0% Zr alloy and Co_{3}ZrB_{2} + Co_{2}Nb_{2}B_{6} + Co_{3}B phases for the 2% Zr and the 4% Zr alloys. No propriable diffraction peaks of Fe-containing compounds without Co are observed for the 0, 2 and 4% Zr alloys. It is therefore considered that the Fe element is dissolved into the Co sites in Co_{21}Nb_{2}B_{6}, Co_{3}B and Co_{3}ZrB_{2} phases for the 0 to 4% Zr alloys. With further increasing Zr content, α-Fe and FeB phases precipitated in the 6% Zr and the
8% Zr alloys, together with Co$_{21}$Nb$_2$B$_6$, Co$_2$ZrB$_2$ and Co$_2$B phases.

3.2 Magnetic properties of melt-spun Co–Fe–Nb–Zr–B amorphous alloys

Figure 5 shows the hysteresis I-H loops of the Co$_{40}$Fe$_{22}$Nb$_{5}$Zr$_{x}$B$_{30}$ ($x = 0, 2, 4, 6$, and $8$ at%) amorphous alloys subjected to annealing for 600 s at the temperature of $T_a - 50$ K. No distinct compositional dependence in $I_c$ and $H_c$ is seen. That is, the $I_c$ lies from 0.41 T at 0% Zr to 0.42 T at 8% Zr and the $H_c$ is in the range of 1.2 to 2.0 A/m. Figure 6 shows the changes in the saturated magnetostriiction ($\lambda_s$) and effective permeability ($\mu_e$) at 1 kHz with the Zr content for the Co$_{40}$Fe$_{22}$Nb$_{5}$Zr$_x$B$_{30}$ amorphous alloys, together with the data of $I_c$ and $H_c$. The $\lambda_s$ is in range of $2.4 \times 10^{-6}$–$2.9 \times 10^{-6}$ over the whole Zr content range. The small $\lambda_s$ values are nearly the same as those for the Co–Fe–Zr–M–20% B (M=Nb, Ta, W) amorphous alloys with $\Delta T_s$ above 40 K. The maximum $\mu_e$ has high values of 33000 at 8% Zr to 32000 at 2% Zr. Table 1 summarizes the thermal stability and magnetic properties of the Co$_{40}$Fe$_{22}$Nb$_{5}$B$_{30}$ and Co$_{40}$Fe$_{22}$Nb$_{5}$Zr$_x$B$_{30}$ amorphous alloys, together with the data of the other glassy alloys including commercial Co–Fe–Ni–Mo–Si–B amorphous alloy. The features of the present Co–Fe–Nb–Zr–30% B amorphous alloys are summarized as follows: (1) high thermal stability as is evident from the higher $T_a$ and the larger $\Delta T_s$ exceeding 80 K; (2) lower $H_c$ as compared with Co–Al–(Fe, Cr, V)–Ga–P–B–C–Si and Co–Fe–Zr–Ta–20% B amorphous alloys; (3) higher $\mu_e$ exceeding 3000 at 1 kHz as compared with the Co–Fe–Zr–Ta–20% B and METGLAS 2705M (Co–Fe–Mo–B–Si amorphous alloy); (4) low $\lambda_s$ comparable to the Co–Fe–Zr–Ta–20% B amorphous alloy.

It is thus characterized that the present alloys have higher
Table 1  Thermal stability and magnetic properties of the Co$_{40}$Fe$_{22}$Nb$_{5}$B$_{30}$ and Co$_{50}$Fe$_{22}$Nb$_{5}$Zr$_{2}$B$_{30}$ amorphous alloys. The data of the other Co-based amorphous alloys containing 20 at% B and commercial Co-based amorphous alloys are also shown for comparison.

<table>
<thead>
<tr>
<th></th>
<th>$T_g$ (K)</th>
<th>$T_x$ (K)</th>
<th>$\Delta T_x$ (K)</th>
<th>$I_e$ (T)</th>
<th>$H_c$ (Am$^{-1}$)</th>
<th>$\lambda_e$ (10$^{-6}$)</th>
<th>$\mu_e$ (at 1 kHz)</th>
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<td>Co$<em>{50}$Cr$</em>{1}$Al$<em>{1}$Ga$</em>{2}$P$<em>{1.3}$B$</em>{6}$C$_{1}$</td>
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<td>768</td>
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<td>Co$<em>{50}$V$</em>{1.3}$Al$<em>{1}$Ga$</em>{2}$P$<em>{1.3}$B$</em>{6}$C$_{1}$</td>
<td>722</td>
<td>769</td>
<td>47</td>
<td>0.29</td>
<td>5.8</td>
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<td>4600</td>
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<tr>
<td>Co$<em>{50}$Fe$</em>{16}$Zr$<em>{1}$B$</em>{20}$</td>
<td>840</td>
<td>879</td>
<td>39</td>
<td>0.77</td>
<td>8.0</td>
<td>8.0</td>
<td>17100</td>
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<tr>
<td>Co$<em>{50}$Fe$</em>{24}$Zr$<em>{1}$T$</em>{1}$B$_{20}$</td>
<td>858</td>
<td>895</td>
<td>37</td>
<td>0.54</td>
<td>1.7</td>
<td>3.4</td>
<td>23000</td>
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<tr>
<td>Co$<em>{40}$Fe$</em>{22}$Nb$<em>{5}$B$</em>{30}$</td>
<td>895</td>
<td>976</td>
<td>81</td>
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<td>2.4</td>
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<td>1001</td>
<td>98</td>
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<td>1.2</td>
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<td>Co-Fe-Ni-Mo-B-Si</td>
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</table>

METGLAS 2705M

thermal stability, higher $\mu_e$ and lower $\lambda_e$ as compared with the Co-based amorphous alloys with a large supercooled liquid region reported up to date.

3.3 Thermal stability and magnetic properties of Co-based bulk amorphous alloys

Figure 7 shows the outer morphology and surface appearance of the cast Co$_{40}$Fe$_{22}$Nb$_{5}$Zr$_{2}$B$_{30}$ cylinders with diameters of 1.0 and 1.5 mm. These samples have a smooth surface and metallic luster. To examine a cross sectional morphology and solidification structure of the cast cylinders, optical micrographs of both samples are shown in Figs. 8(a) to (d). As shown in Figs. 8(a) and (b), no appreciable contrast revealing the precipitation of a crystalline phases is observed in the cross sectional structure of the 1.0 mm sample. The featureless contrast indicates that the cast cylinder consists

![Fig. 7](image_url)

(a) $\phi$ 1.0 mm

(b) $\phi$ 1.0 mm

![Fig. 8](image_url)

Transverse cross sectional structures of the Co$_{40}$Fe$_{22}$Nb$_{5}$Zr$_{2}$B$_{30}$ cylinders with diameters of 1.0 mm and 1.5 mm. (a) central region, $\phi$1.0 mm, (b) new surface region, $\phi$1.0 mm, (c) central region, $\phi$1.5 mm, (d) new surface region, $\phi$1.5 mm.
of an amorphous phase. In the 1.5 mm sample, the featureless contrast due to the formation of an amorphous phase is observed in the outer region within 200 μm from the outer surface. However, columnar precipitates with a particle size of 5–10 μm are observed in the central cross section having a lower cooling rate in comparison with the outer surface region, as shown in Figs. 8(c) and (d). The formation of amorphous phase is also confirmed by means of X-ray diffraction. As shown in Fig. 9, no diffraction peak corresponding to a crystalline phase is seen for the 1 mm sample. On the other hand, as indexed in Fig. 9, the structure consists of coexistent amorphous, Co_{21}Nb_{2}B_{6}, Co_{3}ZrB_{2} and Co_{2}B phases for the φ1.5 mm sample.

Figure 10 shows the DSC curves of the cast amorphous Co_{40}Fe_{22}Nb_{5}Zr_{5}B_{30} cylinders with diameters of 1.0 and 1.5 mm. The data of the melt-spun amorphous ribbon with a thickness of 20 μm are also shown for comparison. As shown Fig. 10, the T_{g}, T_{x} and heat of crystallization (ΔH_{x}) are 903 K, 1001 K and 7.39 kJ/mol respectively, for the φ1.0 mm sample, which approximately agree with those for melt-spun amorphous ribbon. However, the T_{g} and T_{x} of the φ1.5 mm sample are 903 K and 995 K, respectively, indicating a slight decrease in T_{x}. Moreover, the ΔH_{x} of the φ1.5 mm sample is 2.46 kJ/mol which is about one third of that of melt-spun amorphous ribbon. The peak temperature and single-stage crystallization mode of the φ1.5 mm sample agree with those for the other samples. The slight decrease in T_{x} seems to result from an easy growth reaction of the crystalline phase at the interphase between the pre-existing crystalline and amorphous phases. Substantially, it is said that the cast sample has a large supercooled liquid region of 98 K, which is the same as that for the melt-spin ribbon amorphous alloy.

The hysteresis I-H loops of the bulk amorphous Co_{40}Fe_{22}Nb_{5}Zr_{5}B_{30} cylinders with diameters of 1.0 and 1.5 mm are shown in Fig. 11, where the data of the 1.0 mm sample crystallized by annealing at 1051 K for 1.8 ks and the as-spun amorphous ribbon are also presented for comparison. The I-H loop was measured by VSM because the conventional I-H loop tracer could not be used for the cylindrical bulk amorphous alloy. The cast sample has a saturation magnetization (I_{s}) of 0.41 T and a coercivity (H_{c}) of 78 A/m. One can't see any distinct difference in the hysteresis I-H loops.

![Figure 9](image9.png)  
Fig. 9 X-ray diffraction patterns taken from the transverse cross section of cast Co_{40}Fe_{22}Nb_{5}Zr_{5}B_{30} cylinders with diameters of 1.0 mm and 1.5 mm.

![Figure 10](image10.png)  
Fig. 10 DSC curves of cast Co_{40}Fe_{22}Nb_{5}Zr_{5}B_{30} cylinders with diameters of 1.0 mm and 1.5 mm. The data of the melt-spun amorphous ribbon are also shown for comparison.

![Figure 11](image11.png)  
Fig. 11 Hysteresis I-H loops of cast Co_{40}Fe_{22}Nb_{5}Zr_{5}B_{30} cylinders with diameters of 1.0 and 1.5 mm. The cast cylinder sample of 1.0 mm in diameters annealed for 1.8 ks at 1051 K and melt-spin amorphous ribbon are also shown for comparison.
between the φ1.0 mm sample and the melt-spun ribbon. The \( I_s \) and \( H_c \) of the crystallized φ1.0 mm sample are 0.66 T and 3.2 kA/m, respectively, which are higher than those of the as-cast amorphous sample, because of the precipitation of the ferromagnetic crystalline phases. The coercivity of the crystallized sample is about 40 times as large as that of the as-cast bulk amorphous sample. With increasing sample diameter up to 1.5 mm, \( I_s \) and \( H_c \) increase to 0.44 T and 170 A/m, respectively. This change is also due to the precipitation of the ferromagnetic phase.

From these results, it is concluded that the critical sample thickness for glass formation by copper mold casting lies in the range between 1.0 mm and 1.5 mm for the Co-40Fe-22Nb-3Zr-3B alloy. It is noticed that the critical sample thickness is about 6 times larger than that for Co-based amorphous alloys reported up to date. The Co-40Fe-22Nb-3Zr-3B amorphous alloy has the combination of high-glass forming ability and good soft magnetic properties.

4. Discussion

As is described above, the substitution of Nb by 2% Zr cause the significant extension of the supercooled liquid region before crystallization by an increase in \( T_x \) exceeding the degree of the increase in \( T_g \) (see Fig. 2). The largest \( \Delta T_x \) of 98 K at 2% Zr is larger by about 40 K than those for the Co-30Al-(Fe, Cr, V)-Ga-PB-C-Si and the Co-Fe-(Zr, Nb, Ta, W)-20% B glassy alloys. The significant increase of \( \Delta T_x \) allows us to expect the formation of bulk amorphous alloys with diameters of several millimeters by the copper mold casting process. Consequently, it is of interest to discuss the reason for the significant extension of supercooled liquid region prior to crystallization. It has recently been pointed out that all alloys having the high stability of supercooled liquid against crystallization satisfy the three empirical rules for achievement of large glass forming ability, i.e., (1) the multicomponent system consisting of more than three elements, (2) the different atomic size ratios more than 12% among the main constituent elements, and (3) the negative heats mixing among their elements. The atomic radius changes in the order of Zr > Nb > Co > Fe > B, and the predicted heat of mixing has negative values of 37-60 kJ/mol for equiatomic Co-Zr, Fe-Zr, 23-37 kJ/mol for Co-Nb, Fe-Nb pairs, 34-38 kJ/mol for Co-B, Fe-B pairs, and 79-102 kJ/mol for Zr-B, Nb-B pairs. The Co-Fe-Nb-Zr-B system also satisfies the three empirical rules. Moreover, it is noticed that the crystallized phases of the Co-40Fe-22Nb-3Zr-3B alloy and the Co-40Fe-22Nb-3Zr-3B alloys shown in Fig. 4 were including the Co-Zr-B and the Co-Nb-B ternary compounds, respectively. This result indicates a small amount of substitution by Nb or Zr in the Co-40Fe-22Nb-3B (M=Nb, Zr) amorphous alloys affects the stabilization of the supercooled liquid state. As a result, the significant increase in the supercooled liquid region was observed for the 2% Zr-containing alloy. With further increasing Zr content, the supercooled liquid region decreases from 98 K to 42 K. The precipitation of αFe and FeB phases implies that the crystallization becomes easy for the 6 and 8% Zr alloys, because their crystallization temperatures and \( \Delta T_x \) decrease rapidly.

Finally we discuss the glass forming ability of the Co-40Fe-22Nb-3Zr-3B alloy. A number of glassy alloys with a supercooled liquid region beyond 50 K have maximum thickness diameters of about 8-10 mm. Considering the empirical relation of \( \Delta T_x \), a more large scale bulk amorphous may be obtained for this alloy. There is clear tendency for glass forming ability which enhances with increasing the reduced glass transition temperature \( T_s/T_m \). Here, \( T_m \) represents the melting temperature. The \( T_m \) of the Co-40Fe-22Nb-3Zr-3B alloy is 1585 K, and the resulting \( T_s/T_m \) is estimated as 0.57. The \( T_s/T_m \) value is slightly lower than those (0.60 to 0.72) for the other glassy alloy. The \( T_m \) is also higher than those for the other glassy alloys. In the case of the copper mold casting method, it seems that the inconsistency for the maximum sample thickness with the empirical relation is due to the high \( T_m \) value as compared with the Mg-, La-, Zr-, and Pd-Cu-based amorphous alloys.

5. Summary

With the aim of developing a new ferromagnetic Co-based amorphous alloy, we investigated the compositional dependence of \( T_g \), \( T_x \), \( \Delta T_x \), crystallization behavior and soft magnetic properties for the Co-40Fe-22Nb-xZr-3B alloy (x = 0, 2, 4, 6, and 8 at%) amorphous alloys. Using a Co-40Fe-22Nb-3Zr-3B alloy with \( \Delta T_x \) of 98 K, we examined the formation of bulk glassy alloy in a cylindrical form by the copper mold casting method and the thermal stability and magnetic properties of the resulting bulk glassy alloys. The results obtained are summarized as follows;

(1) The glass transition and subsequent supercooled liquid region were observed in the temperature range before crystallization for all the alloys. The maximum \( \Delta T_x \) was 98 K for the 2% Zr alloy.

(2) The crystallization phases are CoB, Co3Nb2B5 and/or Co3ZrB2 phases for the alloys containing less than 4% Zr, and αFe, FeB, CoB, Co3NbB8, and/or Co3ZrB2 phases for the alloys containing more than 6% Zr.

(3) The \( I_s \) at \( H_c \) and \( \mu_0 \) at 1 kHz are 0.41 T-0.42 T, 2.4 x 10^{-6}-2.9 x 10^{-6}, 1.2-2.0 A/m, and 2900-33000 over the whole Zr content range for the melt-spun Co-40Fe-22Nb-xZr-3B amorphous alloys.

(4) The cast bulk glassy alloys with a length of 40 mm were produced in the diameter range up to 1.0 mm. The \( T_g \), \( T_x \), and \( \Delta H_c \) of the φ1.0 mm glassy alloy are 903 K, 1000 K and 7.39 kJ/mol, respectively, which are in agreement with those for melt-spun amorphous ribbon. The critical diameter is about 6 times larger than that for Co-based amorphous alloys reported up to date.

(5) The bulk glassy alloy of φ1 mm in diameter has good soft magnetic properties of 0.41 T for \( I_s \) and 78 A/m for \( H_c \). The magnetic properties are nearly the same as those for the melt-spun amorphous ribbon. The success of forming Co-based bulk amorphous alloys with high stability of supercooled liquid and good soft magnetic properties promotes further development of bulk amorphous soft magnetic materials.
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