Mechanical and Magnetic Properties of New (Fe,Co,Ni)–B–Si–Ta Bulk Glassy Alloys

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Abstract The alloying effects of the like-atom substitution of Ni and Co for Fe on the various properties of Fe₇₀B_{16.7}Si_{8.3}Ta₅ metallic glass are investigated in this present work. New Fe-based bulk glassy alloys, namely Fe_{60-x}Co_xNi₁₀B_{16.7}Si_{8.3}Ta₅ (at.%; $x = 10, 20$ and 30) with critical diameters up to 1.5 mm, were made by means of copper mold casting. A new glass-forming ability indicator, viz., the enthalpy of supercooled liquid, has been introduced for assessment of the glass-forming abilities (GFAs) of these Fe-based multi-component alloys. Nano-indentation results indicate that the calculated elastic modulus and hardness of the bulk glassy alloys are lower than those of the Fe₇₀B_{16.7}Si_{8.3}Ta₅ alloy. Among these bulk glassy alloys, Fe₇₀B_{16.7}Si_{8.3}Ta₅ exhibits the large elastic modulus and hardness with values of 178 ± 1 GPa and 12.9 ± 0.1 GPa, respectively. All the bulk glassy alloys exhibit good soft magnetic properties with high saturation magnetization $B_s \sim 0.75$ – 1.04 T but low coercive force $H_c \sim 0.2$ – 5.2 A/m.

KEY WORDS: (Fe, Co, Ni)–B–Si–Ta bulk glass alloys; Glass-forming ability; Mechanical property; Magnetic property

1 Introduction

Fe-based amorphous alloys are known to have attractive soft magnetic properties including high saturation magnetization (B_s), low coercive force (H_c), low core loss and

high magnetic permeability (μ) [1–3]. Indeed, this has led to their use in a wide variety of industrial applications especially in transformer cores and magnetic sensors [4]. However, their sizes are limited due to the rapid liquid cooling required for retention of amorphous structure, and this restricts their applications in some areas. There has been continued interest in the enhanced glass-forming ability (GFA) of Fe-based amorphous alloys to exploit excellent soft magnetic properties. Following the first discovery of Fe–(Al, Ga)–P–C–B bulk glassy alloys, various Fe-based bulk metallic glasses (BMGs) have been developed in many different multi-component alloy systems [5, 6]. Among them, the Fe–B–Si–ETM (Early Transition Metals, ETM = Nb, Zr, Hf and Ta) quaternary bulk glassy alloys exhibit large GFA (the critical diameter for glass formation $d_c \geq 2.5$ mm), high fracture strength ($\sigma_f \sim 4000$ MPa) and good soft magnetic properties ($B_s \sim 1.13$ – 1.51 T and $H_c \sim 1.5$ – 6.7 A/m) [7–11]. In particular, magnetic powder cores named “SENNTIX-I,” which are made of Fe–B–Si–Nb glassy alloys, have found

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to be practical use in the choke coils of AC-DC and DC-DC converters [6]. In the studies of Fe-based BMGs, like-atom-substitution has demonstrated an effective alloying strategy to reach large GFA and the improved mechanical and magnetic properties. For instance in the Fe–B–Si–Nb system, the replacement of Fe by Co has resulted in not only the improved GFA and mechanical properties [9, 12], but also the increases of B_s and the Curie temperature (T_c) [13, 14], and the former property improvements being mainly attributed to the greater negative mixing enthalpy of Co–Nb atomic pair than that of Fe–Nb, and the latter to the strong exchange coupling between Fe and Co.

The cluster-plus-glass-atom model has been successfully used for understanding and designing alloy compositions with large GFA in many systems [15]. In our previous work, a series of $[\text{Si-B}_2\text{Fe}_{8-x}\text{Ta}_x]\text{Fe}$ bulk glassy alloys with $d_c = 1.0$ mm were designed by using this model, and these BMGs exhibit good soft magnetic and mechanical properties, where in the cluster formula expression the center and the first neighbor atoms are separated by a hyphen, the cluster is enclosed in the square brackets and the glue atom locates outside the square brackets [10]. In the present work, the variations of the mechanical and soft magnetic properties of $\text{Fe}_{70}\text{B}_{16.7}\text{Si}_{8.3}\text{Ta}_5$ and $\text{Fe}_{60-x}\text{Co}_x\text{Ni}_{10}\text{B}_{16.7}\text{Si}_{8.3}\text{Ta}_5$ ($x = 0, 10, 20$ and 30) bulk glassy alloys have been investigated. In addition, a new GFA indicator, enthalpy of supercooled liquid (ΔH_{sup}), was proposed for these BMG-forming alloys.

2 Experimental

Alloy ingots with the desired compositions of $\text{Fe}_{70}\text{B}_{16.7}\text{Si}_{8.3}\text{Ta}_5$ and $\text{Fe}_{60-x}\text{Co}_x\text{Ni}_{10}\text{B}_{16.7}\text{Si}_{8.3}\text{Ta}_5$ (at.%; $x = 0, 10, 20$ and 30) were prepared by arc melting under argon atmosphere. The purities of raw materials are 99.999 mass% for Fe and Si, 99.95 mass% for Ta and 99.5 mass% for B, respectively. Alloy rods with different diameters were prepared by copper mold suction casting. For melt spinning, the surface speed of the copper wheel was set to 40 m/s. The dimensions of ribbon samples were approximately 1.5 mm in width and 20–30 μm in thickness. The structures of as-prepared ribbon and rod samples were examined by a Bruker D8 Focus X-ray diffractometer (XRD, Cu- K_α radiation, $\lambda = 0.15406$ nm). The thermal glass properties of the as-prepared alloys, such as the glass transition temperature (T_g), the crystallization temperature (T_x), the melting temperature (T_m), and the liquidus temperature (T_l), were measured by Differential Scanning Calorimetry (DSC) and Differential Thermal Analysis (DTA) at a heating rate of 0.67 K/s. The magnetic properties, such as B_s and H_c , were measured with a MATS-2010SD Hysteresis-graph. Nano-indentation experiments

on the 1.0 mm diameter glassy alloy rods were carried out by using a CSM nano-indenter at a constant strain rate of 0.05 s^{-1} and under a maximum load of 200 mN. The hardness and modulus of samples were calculated by using the method proposed by Oliver and Pharr [16]. The Vickers hardness (H_v) measurement was conducted on the 1.0 mm diameter rod samples under a load of 500 g. Five times for each of the specimens were carried out in mechanical properties testing. The morphology of the Vicker's hardness indentations was observed by optical microscopy (OM).

3 Results and Discussions

The X-ray diffraction patterns of $\text{Fe}_{70}\text{B}_{16.7}\text{Si}_{8.3}\text{Ta}_5$ and $\text{Fe}_{60-x}\text{Co}_x\text{Ni}_{10}\text{B}_{16.7}\text{Si}_{8.3}\text{Ta}_5$ ($x = 0, 10, 20$ and 30) melt-spun samples are presented in Fig. 1a. All the samples show a typical amorphous structure. The d_c values of different bulk samples are presented in Fig. 1b along with their diffraction patterns. Glassy alloy rods were produced at all compositions under investigation, viz., $d_c \sim 1.0$ mm for $\text{Fe}_{70}\text{B}_{16.7}\text{Si}_{8.3}\text{Ta}_5$ and $\text{Fe}_{60}\text{Ni}_{10}\text{B}_{16.7}\text{Si}_{8.3}\text{Ta}_5$ and $d_c \sim 1.5$ mm for $\text{Fe}_{50}\text{Co}_{10}\text{Ni}_{10}\text{B}_{16.7}\text{Si}_{8.3}\text{Ta}_5$, $\text{Fe}_{40}\text{Co}_{20}\text{Ni}_{10}\text{B}_{16.7}\text{Si}_{8.3}\text{Ta}_5$ and $\text{Fe}_{30}\text{Co}_{30}\text{Ni}_{10}\text{B}_{16.7}\text{Si}_{8.3}\text{Ta}_5$, respectively. The casting experiment indicates that substitution of Co for Fe results in the enhanced GFA in the present system. When the rod diameter of the casting samples exceeds the d_c , partially crystallization of the Fe_{23}B_6 , Fe_2B and bcc-Fe-type phases occurs (Fig. 1c). These results are similar to the previously reported in (Fe,Co,Ni)–B–Si–Nb alloys [17, 18].

Figure 2a shows the DSC curves of $\text{Fe}_{70}\text{B}_{16.7}\text{Si}_{8.3}\text{Ta}_5$ and $\text{Fe}_{60-x}\text{Co}_x\text{Ni}_{10}\text{B}_{16.7}\text{Si}_{8.3}\text{Ta}_5$ ($x = 0, 10, 20$ and 30) glassy alloys made by melt spinning. It is seen that glass transition becomes distinct in the samples with the addition of Ni and with increasing Co content. The thermal glass stability parameters of T_g , T_x , the supercooled liquid region spans of $\Delta T_x = T_x - T_g$, T_m and T_l are obtained from the DSC and DTA (Fig. 2b) curves, and these are summarized in Table 1. It was found that the T_g and T_x of glassy alloys decreased from 858 K and 889 K to 845 K and 881 K, respectively, with the addition of Ni, and then increased with increasing Co content. Subtle changes are found for the ΔT_x values of these BMG alloys, whereas T_m and T_l of them decrease rapidly due to the presences of Ni and Co. The reduced glass transition temperature, $T_{rg} = T_g/T_l$, was calculated to assess the GFAs of these alloys. The variation of T_{rg} against alloy composition is plotted in Fig. 3. It can be seen that T_{rg} increases with increasing Co content and the largest value of $T_{rg} = 0.618$ is reached at $\text{Fe}_{30}\text{Ni}_{10}\text{Co}_{30}\text{B}_{16.7}\text{Si}_{8.3}\text{Ta}_5$. The varied GFA as predicted by the T_{rg} values is consistent with the results of critical glass

Fig. 1 X-ray diffraction patterns of the melt-spun ribbons **a**, as-cast rods with the critical diameters **b** and the rod samples with a diameter larger than the critical value **c** of $\text{Fe}_{70}\text{B}_{16.7}\text{Si}_{8.3}\text{Ta}_5$ and $\text{Fe}_{60-x}\text{Co}_x\text{Ni}_{10}\text{B}_{16.7}\text{Si}_{8.3}\text{Ta}_5$ ($x = 0, 10, 20$ and 30) alloys

Fig. 2 DSC **a** and DTA **b** curves of $\text{Fe}_{70}\text{B}_{16.7}\text{Si}_{8.3}\text{Ta}_5$ and $\text{Fe}_{60-x}\text{Co}_x\text{Ni}_{10}\text{B}_{16.7}\text{Si}_{8.3}\text{Ta}_5$ ($x = 0, 10, 20$ and 30) glassy alloys

Table 1 Thermal, mechanical and soft magnetic properties of $\text{Fe}_{70}\text{B}_{16.7}\text{Si}_{8.3}\text{Ta}_5$ and $\text{Fe}_{60-x}\text{Co}_x\text{Ni}_{10}\text{B}_{16.7}\text{Si}_{8.3}\text{Ta}_5$ glassy alloys

Compositions	x	d_c (mm)	T_g (K)	T_x (K)	ΔT_x (K)	ΔH_{sup} (J/g)	T_l (K)	T_{rg}	H_v (GPa)	H_N (GPa)	E (GPa)	B_s (T)	H_c (A/m)
$\text{Fe}_{70}\text{Ta}_5\text{B}_{16.67}\text{Si}_{8.33}$	–	1.0	858	889	31	0.7	1479	0.580	11.1 ± 0.2	12.9 ± 0.1	178 ± 1	1.02	1.0
$\text{Fe}_{60}\text{Ni}_{10}\text{Ta}_5\text{B}_{16.67}\text{Si}_{8.33}$	0	1.0	845	881	36	2.6	1438	0.588	10.7 ± 0.1	11.5 ± 0.3	163 ± 1	1.04	0.2
$\text{Fe}_{50}\text{Co}_{10}\text{Ni}_{10}\text{Ta}_5\text{B}_{16.67}\text{Si}_{8.33}$	10	1.5	850	885	35	4.6	1434	0.593	10.8 ± 0.3	12.1 ± 0.1	158 ± 1	0.93	1.1
$\text{Fe}_{40}\text{Co}_{20}\text{Ni}_{10}\text{Ta}_5\text{B}_{16.67}\text{Si}_{8.33}$	20	1.5	845	885	40	7.1	1405	0.601	10.9 ± 0.1	11.3 ± 0.4	144 ± 3	0.83	2.3
$\text{Fe}_{30}\text{Co}_{30}\text{Ni}_{10}\text{Ta}_5\text{B}_{16.67}\text{Si}_{8.33}$	30	1.5	849	886	37	8.2	1373	0.618	11.0 ± 0.2	12.7 ± 0.3	159 ± 3	0.75	5.2

formation size. The area of endothermic peak, namely the enthalpy of supercooled liquid (ΔH_{sup}), has been considered to characterize the stability of supercooled glass-forming liquids instead of ΔT_x [19, 20]. The ΔH_{sup} exhibits the lowest value of 0.7 J/g in the Ni- and Co-free alloys but increases rapidly to a greater value of 8.2 J/g at the alloy composition with $x = 30$ (Table 1 and Fig. 3). The variation tendency of ΔH_{sup} is consistent with the d_c and T_{rg} assessments on GFA. Therewith, ΔH_{sup} rather than ΔT_x is

suitable for evaluating the GFA of (Fe,Ni,Co)–B–Si–Ta alloys. As shown in Fig. 3, this proposition finds further support in the (Co,Fe)–B–Si–Nb and other bulk glassy alloys derived from like-atom substitution alloying [19, 21–24].

The mechanical properties of $\text{Fe}_{70}\text{B}_{16.7}\text{Si}_{8.3}\text{Ta}_5$ and $\text{Fe}_{60-x}\text{Co}_x\text{Ni}_{10}\text{B}_{16.7}\text{Si}_{8.3}\text{Ta}_5$ bulk glassy alloys were measured by means of nano-indentation. The averaged load–displacement (P – h) curves of nano-indentations for these glassy alloys are

Fig. 3 Variations of d_c , T_{rg} , ΔH_{sup} and ΔT_x against x in $\text{Fe}_{60-x}\text{Co}_x\text{-Ni}_{10}\text{B}_{16.7}\text{Si}_{8.3}\text{Ta}_5$ **a** and $[(\text{Co}_{1-x}\text{Fe}_x)_{0.75}\text{B}_{0.2}\text{Si}_{0.05}]_{96}\text{Nb}_4$ **b** glassy alloys

presented in Fig. 4. Under the same load, the indentation depth of $\text{Fe}_{70}\text{B}_{16.7}\text{Si}_{8.3}\text{Ta}_5$ glassy alloys is smaller than those produced in other samples. The $\text{Fe}_{70}\text{B}_{16.7}\text{Si}_{8.3}\text{Ta}_5$ glassy alloy exhibits the highest nano-indentation hardness (H_N) [25, 26]. The calculated H_N and elastic modulus (E) are presented in Table 1. The values of H_N and E are decreased in the glassy alloys containing Ni and Co, in which the maxima of H_N and E (12.9 ± 0.1 GPa and 178 ± 1 GPa) are found in the $\text{Fe}_{70}\text{B}_{16.7}\text{Si}_{8.3}\text{Ta}_5$ bulk glassy alloy.

The compositional dependence of the Vicker's micro-hardness (H_v) of $\text{Fe}_{70}\text{B}_{16.7}\text{Si}_{8.3}\text{Ta}_5$ and $\text{Fe}_{60-x}\text{Co}_x\text{Ni}_{10}\text{B}_{16.7}\text{Si}_{8.3}\text{Ta}_5$ bulk glassy alloys exhibits variation tendency similar to that observed in the nano-indentation results (Table 1). The H_v values are smaller than those calculated from the nano-indentation tests. It should be noted that the hardness value is sensitive to the shape and size of indenters and the indentation depth, due to which the change in hardness obtained from different tests is partly attributed to the different indenter geometries, loading modes and indentation depths in the specific experiments [26]. Figure 5 displays the optical images of the Vickers hardness indentations of $\text{Fe}_{70}\text{B}_{16.7}\text{Si}_{8.3}\text{Ta}_5$ and $\text{Fe}_{60-x}\text{Co}_x\text{Ni}_{10}\text{B}_{16.7}\text{Si}_{8.3}\text{Ta}_5$ ($x = 0, 10$ and 30) bulk glassy alloys. Slip markings (arrows) are seen to form around the Vickers hardness indentation obtained with a rather high load of 500 g. The generation of a number of slip markings and the absence of appreciable cracking indicate that the Fe-based bulk glassy alloys are not extremely brittle [8].

The B_s and H_c of the $\text{Fe}_{70}\text{B}_{16.7}\text{Si}_{8.3}\text{Ta}_5$ and $\text{Fe}_{60-x}\text{Co}_x\text{Ni}_{10}\text{B}_{16.7}\text{Si}_{8.3}\text{Ta}_5$ ($x = 0, 10, 20$ and 30) glass ribbons were determined from their hysteresis loop curves (Fig. 6) and are summarized in Table 1. The Fe-based glassy alloys exhibit a combination of high B_s but low H_c , indicating of their good soft magnetic properties. Due to the alloying addition of Ni, the B_s of alloys is first increased from 1.02 to 1.04 T with

Fig. 4 Average load-displacement (P - h) curves of the $\text{Fe}_{70}\text{B}_{16.7}\text{Si}_{8.3}\text{Ta}_5$ and $\text{Fe}_{60-x}\text{Co}_x\text{Ni}_{10}\text{B}_{16.7}\text{Si}_{8.3}\text{Ta}_5$ ($x = 0, 10, 20$ and 30) bulk glassy alloys measured by nano-indentation

Fig. 5 Optical micrographs of the Vicker's hardness indentation of **a** $\text{Fe}_{70}\text{B}_{16.7}\text{Si}_{8.3}\text{Ta}_5$, **b** $\text{Fe}_{60}\text{Ni}_{10}\text{B}_{16.7}\text{Si}_{8.3}\text{Ta}_5$, **c** $\text{Fe}_{50}\text{Ni}_{10}\text{Co}_{10}\text{B}_{16.7}\text{Si}_{8.3}\text{Ta}_5$, **d** $\text{Fe}_{30}\text{Ni}_{10}\text{Co}_{30}\text{B}_{16.7}\text{Si}_{8.3}\text{Ta}_5$ bulk glassy alloys under a 500 g load

Fig. 6 Hysteresis B - H curves of the $\text{Fe}_{70}\text{B}_{16.7}\text{Si}_{8.3}\text{Ta}_5$ and $\text{Fe}_{60-x}\text{Co}_x\text{Ni}_{10}\text{B}_{16.7}\text{Si}_{8.3}\text{Ta}_5$ glassy ribbons

increasing Ni content, and then decreased to 0.75 T at the composition with 30 at.% Co. The $\text{Fe}_{70}\text{Ni}_{10}\text{B}_{16.7}\text{Si}_{8.3}\text{Ta}_5$ alloy shows the highest B_s but lowest H_c values in the series of (Fe,

Ni, Co)-B-Si-Ta alloys. In contrast to the evidently decreased B_s and drastically increased of H_c as brought about by the like atom of Ni, the Co element is found to be not effective in improving the soft magnetic properties of alloys. As for the B_s variation in the Ni and Co alloyed Fe-metalloid-type glassy alloys, the fact that the B_s first increases to a maximum and then decreases with increasing Ni and Co content would signal a transition from weak ferromagnetism, i.e., both spin bands are partially filled at the Fermi energy, to strong ferromagnetism, i.e., a completely filled majority-spin band, which is essentially the same as in the other (Fe,Co)-based glassy alloys [27, 28].

4 Conclusions

The glass-forming ability, mechanical and soft magnetic properties of the $\text{Fe}_{60-x}\text{Co}_x\text{Ni}_{10}\text{B}_{16.7}\text{Si}_{8.3}\text{Ta}_5$ ($x = 0, 10, 20$ and 30) glassy alloys with different Ni and Co contents are summarized as follows:

1. The alloying additions of Ni and Co in the Fe₇₀B_{16.67}-Si_{8.33}Ta₅ alloy result in improved glass-forming ability of the alloys. New bulk glassy alloys, namely Fe_{60-x}Co_xNi₁₀B_{16.7}Si_{8.3}Ta₅ ($x = 10, 20$ and 30) with a critical diameter of 1.5 mm were obtained. Large supercooled liquid spans of $\Delta T_x \sim 35\text{--}40$ K and a high $T_{\text{rg}} \sim 0.593\text{--}0.613$ are found in these bulk glassy alloys. The Young's modulus and the nano-hardness of this new bulk glassy alloy rods are $E \sim 144\text{--}178$ GPa and $H_N \sim 11.3\text{--}12.9$ GPa, respectively. All glassy ribbons exhibit high $B_s \sim 0.75\text{--}1.04$ T but low $H_c \sim 0.2\text{--}5.2$ A/m. An appropriate amount of Ni in these glassy alloys can effectively improve their soft magnetic properties. The Fe₆₀Ni₁₀B_{16.7}Si_{8.3}Ta₅ glassy ribbon exhibit the highest B_s (1.04 T) and lowest H_c (0.2 A/m) among these alloys.
2. Both the reduced glass transition temperature, T_{rg} , and the enthalpy of supercooled liquid, ΔH_{sup} , can be used to assess the glass-forming ability of the like-atom-type glassy alloys.
3. (Fe,Co,Ni)-B-Si-Ta ferromagnetic bulk glassy alloys are promising materials for both structural and functional applications due to the combination of high glass-forming ability, ultrahigh mechanical strength and good soft magnetic properties.

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