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Mechanical Testing of Iron based Bulk Metallic Glasses and Their Suitability for Force Sensors

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Abstract. Thermal, mechanical and magnetic properties of (Fe-Co)-(Zr/Si)-Nb-B alloys in the form of rapidly quenched rods of 1.2 mm in diameter were studied. The as-cast alloys with Zr were crystalline, and the alloys with Si were amorphous. Microhardness measured at 50 g load is from 500 to 2000 HV (the less cobalt, the higher), and the compressive strength reaches nearly 4000 MPa for Si doped alloys and 2000 MPa for Zr doped ones. This substantial difference may be attributed to partial crystallinity of the latter alloys. The magnetic hysteresis loops of fully amorphous rods measured under compression, exhibited a clear dependence of permeability vs. stress, proving that iron-based bulk metallic glasses may be promising materials for magnetoelastic force sensors.

1 Introduction

Iron-based bulk metallic glasses (BMGs) are a group of interesting materials for their magnetic softness (low coercivity, low hysteresis losses due to the lack of magnetocrystalline anisotropy), high magnetic induction and remarkably high strength [1]. The glass forming ability (GFA) may be improved if other metals, such as cobalt or nickel, substitute a part of iron. This substitution, however, increases the magnetostrictive strain and, in the presence of mechanical stress, increases the magnetoelastic anisotropy. As the result, higher coercivity and losses are observed. The stress may be either a result of rapid quenching (residual stress) or imposed by external forces. On the other hand, stress changes the shape of the magnetic hysteresis loop, e. g. permeability, and this may be used to detect and measure stress. This phenomenon has been observed in most of ferromagnetic materials, however, many of them exhibit poor mechanical strength. In this paper, bulk metallic glasses based on iron are proposed as the cores of magnetostrictive force sensors. Their mechanical properties (microhardness and compression strength) are measured, as well as sensitivity of magnetic properties is evaluated, both features are combined to verify if iron-based BMGs may be used in force sensors.

2 Experimental

Rods, with composition of $(Fe_{1-x}Co_x)_{70}Si_5Nb_5B_{20}$ and $(Fe_{1-x}Co_x)_{70}Zr_5Nb_5B_{20}$, where x = 0, 0.25 and 0.50, were obtained by injection casting into the copper mould with the diameter of 1.2 mm. Their amorphousness was investigated by x-ray diffraction (XRD) technique, where either fragments of rods were ground and exposed, or the cylindrical surfaces were exposed. Crystallisation temperature for each of the alloys was measured in a DTA Setaram Labsys calorimeter in the continuous heating mode with the heating rate of 40°C/min. Stress relieving of as cast rods was carried out in vacuum (samples were sealed in a quartz ampoule), at 400°C for 30 minutes. Hysteresis loops were recorded using the quasistatic hysteresis loop tracer with and without application of stress. The obtained loops were not corrected against demagnetising factor. The maximum magnetic field in the hysteresis loop was of 27.5 kA/m for the measurement of loops without stress, and 1000 A/m for the experiment with compression. Stress was applied with the special setup including pickup coil, compensation coil [2] and clamps, to which weights were attached using strings. Cylindrical samples for compression, with the height from 2.5 to 4.6 mm, were cut from the rods, the cylinders' bases were ground and polished. Microhardness was measured before compression test, on the bases of the cylinders, with the load of 50 grams, and the spacing between indentations was 50 μm.

3 Results

3.1. Structure and thermal properties

The as cast rods of the alloys with the composition of $(Fe_{0.5}Co_{0.5})_{70}Si_5Nb_5B_{20}$ and $(Fe_{0.75}Co_{0.25})_{70}Si_5Nb_5B_{20}$ were fully amorphous, which was indicated by XRD study (results not shown here). For the $(Fe_1Co_0)_{70}Si_5Nb_5B_{20}$ alloy, traces of bcc-Fe phase were revealed by the presence of weak diffraction lines. For the $(Fe_{1-x}Co_x)_{70}Zr_5Nb_5B_{20}$ alloys, all the rods contained mainly the bcc-Fe(Co) phase and some metastable phases, proving that these alloys are more difficult to amorphise. This observation was confirmed by the calorimetric study, which revealed two strong, separate exothermic effects in the case of the Si-containing alloys, and weak, or none, exothermic events for Zr-containing alloys (see figure 1). On the basis of these results it may be concluded that the addition of zirconium is less efficient in terms of GFA than the addition of silicon. Simultaneously, one can see that the partial replacement of iron with cobalt improves the glass forming ability of the investigated materials.

3.2. Magnetic properties

Magnetic hysteresis loops were recorded in order to verify their magnetic softness. As expected from structural investigations, the fully amorphous Si-doped samples showed narrow loops, typical of magnetically soft materials, while Zr-doped rods were wide. One can note that for $(Fe_1Co_0)_{70}Si_5Nb_5B_{20}$ alloy, although containing traces of crystallinity, coercive field reaches 460 A/m, as compared to around 20 A/m for the fully glassy rods. Stress relieving improved the magnetic softness of amorphous alloys: the values of coercive field (H_c) dropped by 20-40% after stresses were annihilated by heat treatment. This effect may be attributed to the reduction of magnetoelastic anisotropy of BMGs. The hysteresis loops of the rods, shown in figure 2, clearly show the difference in magnetic softness of the glassy and crystalline alloys: the alloys containing zirconium exhibit wider loops. The values of coercive field are given in Table 1. These results indicate that for applications, where magnetic softness is important, the Zr-doped alloys obtained in this work are useless due to their poor glass forming ability and, subsequently, partial or complete crystallinity. Furthermore, the DTA curves and hysteresis loops for the alloys with zirconium are well correlated: in the case of x = 0 and 0.25, where there is a significant volume of crystals (lack of crystallisation events in DTA), the values of H_c are large, whilst for x = 0.5, the coercive field is smaller, and an amorphous phase crystallises in DTA.

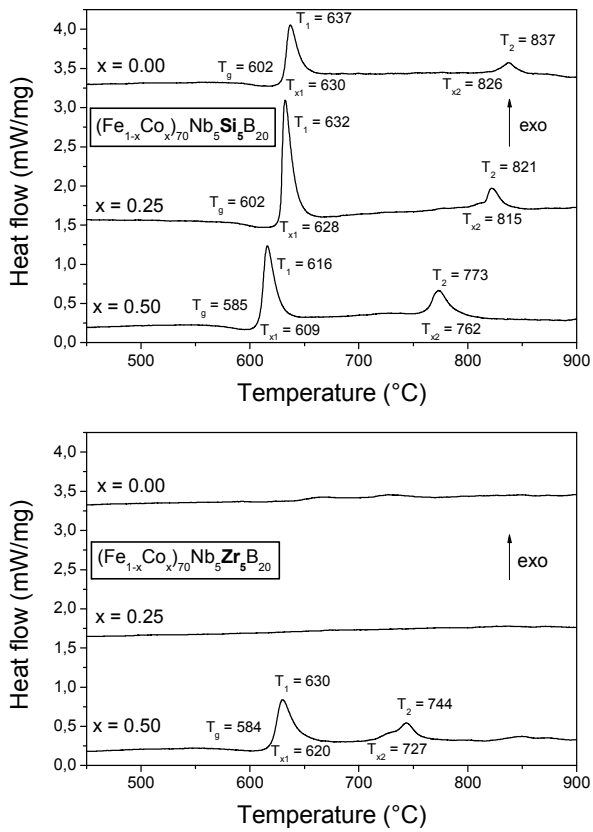

Fig. 1. DTA curves of $(Fe_{1-x}Co_x)_{70}Si_5Nb_5B_{20}$ (upper) and $(Fe_{1-x}Co_x)_{70}Zr_5Nb_5B_{20}$ (lower) alloys after casting.

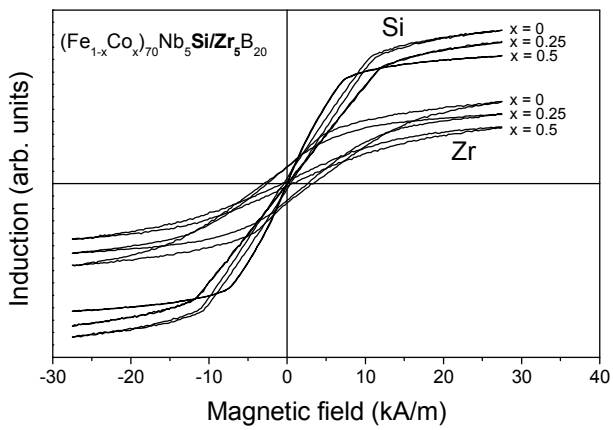

Fig. 2. Hysteresis loops of $(Fe_{1-x}Co_x)_{70}Si_5Nb_5B_{20}$ and $(Fe_{1-x}Co_x)_{70}Zr_5Nb_5B_{20}$ alloys. Plots intentionally distorted along induction scale to demonstrate the differences in coercive field..

Table 1. Coercive field (in A/m) of $(Fe_{1-x}Co_x)_{70}Si_5Nb_5B_{20}$ and $(Fe_{1-x}Co_x)_{70}Zr_5Nb_5B_{20}$ alloys after casting and stress relieving.

Alloying element	Co		
	x = 0	x = 0.25	x = 0.5
Si	460	23	19
Zr	3300	2700	700

3.3. Mechanical properties

The microhardness of samples of all the six alloys after casting was measured along the diameter. No significant differences in moicrohardness between the core and the shell of the samples were found. The statistic values are given in Table 2. The Zr-doped alloys exhibit larger values of microhardness than the Si-doped alloys, but the values are similarly scattered for both alloying elements. The consistent observation is that the microhardness is lower when cobalt is added, regardless of the alloying element and of crystallinity.

Table 2. Microhardness of $(Fe_{1-x}Co_x)_{70}Si_5Nb_5B_{20}$ and $(Fe_{1-x}Co_x)_{70}Zr_5Nb_5B_{20}$ alloys. Upper value is the average HV0.05, lower values is standard deviation in HV0.05 and in percent of the average.

Alloying element	Co		
	x = 0	x = 0.25	x = 0.5
Si	1513 120 / 7.9%	1019 208 / 20.4%	918 200 / 21.8%
Zr	1714 227 / 13.3%	1478 266 / 18.0%	912 232 / 25.4%

All the alloys were subjected to compression, and the stress-strain curves are shown in figure 3. The ultimate compression strength at the level of nearly 4000 MPa is very satisfactory, because it confirms that the results of the obtained bulk metallic glasses are comparable to the data from other laboratories [3, 4]. It should be noted that the elastic strain of the BMGs observed in the stress-strain curves reaches the level of 4%, which is also a good achievement, as usually 2% is reported. From the viewpoint of application of the BMGs as the members of force sensors, the available compression strength is much higher than any conventional material with ferromagnetic properties, which means that their application may result in either the increase of the maximum load of a sensor, or miniaturisation of a sensor.

Compared to the Si-doped alloys, the Zr-doped ones exhibit much lower compression strength (not exceeding 2000 MPa) and almost no plastic strain. Contrary to this, microhardness of the alloys with zirconium is higher. This difference may be explained by an easier crack formation and propagation in the partially crystalline alloys: the fully amorphous alloys are homogeneous in terms of properties, while in the amorphous-crystalline materials the stress on the phase interfaces and on the crystal boundaries may arise, leading to the earlier formation of cracks. It is therefore very important that the materials, which will be exploited due to their mechanical properties, must have a good glass forming ability in order to avoid any crystallinity after quenching.

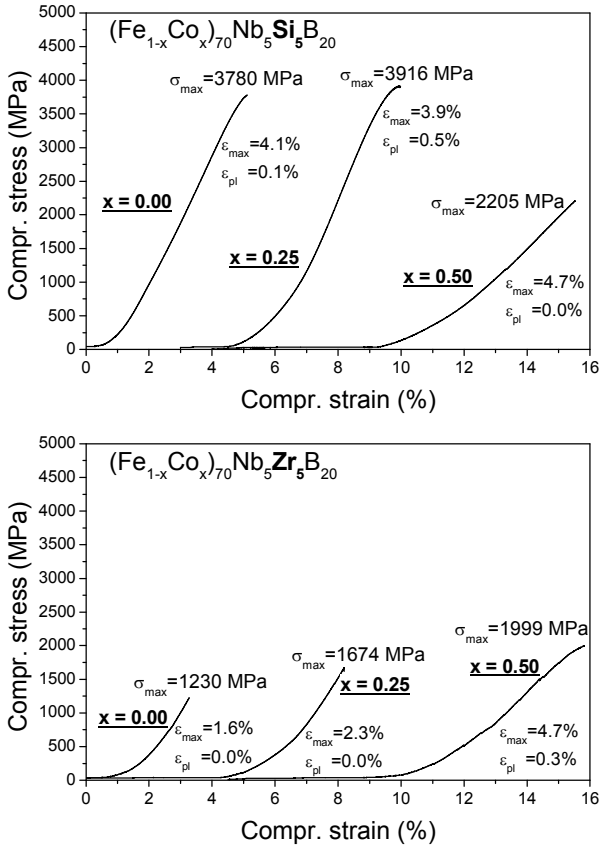

Fig. 3. Compressive stress – strain curves of $(Fe_{1-x}Co_x)_{70}Si_5Nb_5B_{20}$ (upper) and $(Fe_{1-x}Co_x)_{70}Zr_5Nb_5B_{20}$ (lower) alloys after casting and stress relieving.

Similarly to the microhardness results, for the Si-doped alloys it is observed that the addition of cobalt reduces the compression strength. However, the partial replacement of Fe by Co is important for the improvement of GFA, and also increases the magnetostriction coefficient of glassy alloys [5], which should result in the enhancing the sensitivity of the investigated BMGs to stress.

3.4. Sensitivity to stress

The $(Fe_{0.5}Co_{0.5})_{70}Si_5Nb_5B_{20}$ alloy was selected to verify its suitability as the core of the magnetoelastic force sensor. Hysteresis loops of the 30 mm rod were measured with and without stress, in quasistatic conditions, and are shown in Fig. 4. The application of compressive stress caused the flattening of loops, *i. e.* the reduction of permeability. This result indicates that the investigated BMG is a good material for a force sensor, because its magnetic properties are sensitive to the applied stress, and the compression strength is very high.

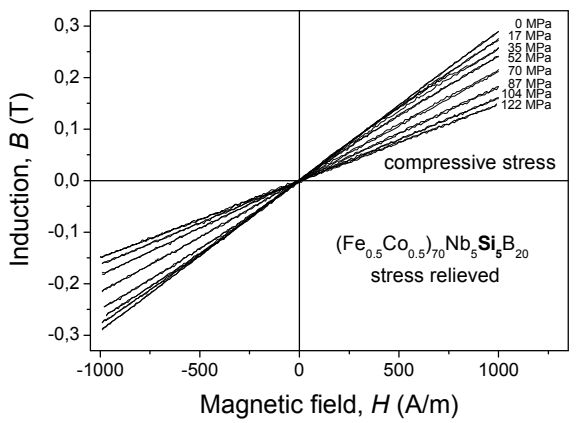

Fig. 4. Hysteresis loops of $(Fe_{1-x}Co_x)_{70}Si_5Nb_5B_{20}$ alloy under compression.

4 Conclusions

The $(Fe_{1-x}Co_x)_{70}Si_5Nb_5B_{20}$ and $(Fe_{1-x}Co_x)_{70}Zr_5Nb_5B_{20}$, where x = 0, 0.25 and 0.5 alloys were cast into 1.2 mm rods and their thermal, magnetic and mechanical properties were investigated. The alloys with Zr were not amorphous, which resulted in the loss of magnetic softness and poor strength as compared to amorphous alloys with Si addition. High strength, reaching 3900 MPa, and large elastic strain, up to 4%, were measured for the $(Fe_{0.75}Co_{0.25})_{70}Si_5Nb_5B_{20}$ alloy, which is comparable with other glassy, iron-based alloys. Addition of cobalt improves the glass forming ability, but reduces strength. Significant sensitivity of magnetic properties to stress of the Fe-based bulk metallic glass was observed, opening the new application field of these materials as cores of force sensors.

Acknowledgements

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