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Magnetic properties optimization of Zr/Fe dual amorphous phase bulk metallic glasses

Optimización de las propiedades magnéticas de vidrios metálicos masivos de Zr/Fe en fase amorfa dual

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Abstract

Dual amorphous phase bulk metallic glasses (DAPBMGs) contain two distinct amorphous alloys in order to combine the favorable properties of each phase. A viable method for obtaining dual bulk amorphous alloys is powder metallurgy. In this study, a Zr/Fe DAPBMG was successfully prepared by hot-pressing Zr-based and Fe-based glassy alloy powders in various volumetric proportions. The samples obtained were structurally analyzed by scanning electron microscopy and x-ray diffraction, and their magnetic properties were determined using an integrator fluxmeter-type hysteresis graph. It was found that increasing the volumetric ratio of the Fe-based alloy decreases coercivity and increases saturation magnetization.

Keywords: bulk metallic glasses; hot-pressing; magnetic properties.

Resumen

Los vidrios metálicos masivos de fase amorfa dual contienen dos tipos distintos de aleaciones amorfas con el fin de reunir todas las propiedades favorables de cada fase. Un método viable para obtener aleaciones masivas de fase amorfa dual es a través de pulvimetalurgia. Una fase amorfa dual de vidrio metálico masivo de Zr/Fe fue preparado prensando en caliente una aleación vidriosa en polvo basada en Fe y otra en Zr en diferentes proporciones volumétricas. Las muestras obtenidas fueron investigadas estructuralmente usando Microscopía de Electron-Scanning y difracción de rayos x. Las propiedades magnéticas fueron determinadas usando una curva de histéresis de integrador de tipo flujómetro. Se encontró que con un aumento de volumen en la tasa de aleación basada en Fe disminuía la coercitividad* y aumentaba la magnetización-saturación.

Palabras clave: vidrios metálicos a granel; prensado en caliente; propiedades magnéticas.

1. Introduction

Bulk metallic glasses (BMGs) are considered good candidates for functional and structural materials [1], [2] due to their high strength, corrosion resistance, polymer-like formability, and excellent magnetic properties.

Improving the magnetic properties of Zr/Fe dual amorphous phase bulk metallic glasses allows for their optimal utilization in microelectromechanical systems (MEMS), and microdevices [3].

Single-phase Zr-based bulk metallic glasses are known to exhibit very good glass-forming ability (GFA), outstanding mechanical properties with a high yield

strength close to the theoretical limit, and high hardness [4]. Fe-based bulk metallic glasses are brittle, but are characterized by higher saturation magnetization, lower coercive force, and lower core loss; attributes which fulfill modern requirements for high-performance soft magnetic materials [5].

Therefore, introducing Fe-based alloy to an Zr-based alloy yields a product with excellent magnetic properties and good elasticity, making it a useful electrotechnical material across various applications.

2. Experimental procedures

In this study, two different types of glassy alloy powder ($\text{Zr}_{48}\text{Cu}_{36}\text{Al}_8\text{Ag}_8$ and $\text{Fe}_{74}\text{Mo}_4\text{P}_{10}\text{C}_{7.5}\text{B}_{2.5}\text{Si}_2$) were used to prepare Zr/Fe DAPBMGs by the hot pressing method [6].

A $\text{Zr}_{48}\text{Cu}_{36}\text{Al}_8\text{Ag}_8$ glassy powder with an average diameter of 20 μm was produced using a high-pressure gas atomization process. Obtaining the $\text{Fe}_{74}\text{Mo}_4\text{P}_{10}\text{C}_{7.5}\text{B}_{2.5}\text{Si}_2$ glassy alloy powder involved two steps: (i) creating amorphous ribbons of approximately 30 μm thickness and 1.5 mm width using the melt-spinning method; (ii) milling the amorphous ribbons using a RETSCH PM400 planetary ball mill. This milling was performed at 150 rpm for 10 hours with a ball-to-powder mass ratio of 20:1. The size of the powders was determined by sieving, with only those particles smaller than 50 μm being retained.

The resulting Zr- and Fe-based glassy alloy powders were mixed in various volumetric ratios using a RETSCH PM400 planetary ball mill at 100 rpm for 20 hours with a ball-to-powder mass ratio of 10:1. Subsequently, the powders were compacted by hot pressing in the a WEBER-PRESSEN pressing device. The hot pressing was performed in the supercooled liquid region of the Zr-based glassy alloy, at 420°C, under an applied pressure of 50 kN, for 15 minutes.

The samples were formed with a diameter of 10 mm and a height of 5 mm.

The study analyzed three compositional variants, as follows:

- S1, samples containing 55 % Zr-based alloy powder and 45% Fe-based alloy powder;
- S2, samples containing 50 % Zr-based alloy powder and 50% Fe-based alloy powder;
- S3, samples containing 45 % Zr-based alloy powder and 55% Fe-based alloy powder.

The microscopic structure of the Zr/Fe DAPBMGs was examined using an FEI Inspect S scanning electron microscope. The structure of the alloy powders and DAPBMG was analyzed by x-ray diffraction with an X'Pert³ Powder diffraction system using radiation from a Cu anode at a wavelength $\lambda = 1.54 \text{ \AA}$.

The hot-pressed samples were subjected to magnetic analysis, with the $\text{Fe}_{74}\text{Mo}_4\text{P}_{10}\text{C}_{7.5}\text{B}_{2.5}\text{Si}_2$ glassy powder compared against a Ni crystalline powder (Sigma Aldrich, 3 μm medium size, 98% purity) considered to be a reference for good soft magnetic properties in the crystalline materials category. A fast but sufficiently accurate method for magnetic characterization of ferromagnetic materials is based on the use of integrative fluxmeter hysteresis; an instrument which displays or records the magnetic hysteresis loop.

3. Results and discussion

The SEM morphologies of the hot-pressed samples are shown in Figure 1. The dark-colored Fe-based amorphous phase can be observed to be uniformly distributed and embedded in the lighter-colored Zr-based amorphous phase. As the ratio of Fe-based glassy alloy powder increases, the Fe-based phase becomes more uniformly distributed in the Zr-based phase. This is explained by the fact that the Fe-based powder is harder and more fragile than the Zr-based powder. This property is advantageous when milling and mixing the powders in a planetary ball mill.

Both the Zr- and Fe-based glassy powders and the samples obtained by hot pressing were subjected to structural analysis by x-ray diffraction.

The XRD patterns (Figure 2) illustrate the amorphous structure of the Zr- and Fe-based powders. In the hot-pressed samples, the two amorphous phases are clearly distinguishable as a Zr-based amorphous phase and an Fe-based amorphous phase. The mixing time of the powders and the parameters of hot pressing can thus be seen to have preserved the amorphous structure. The x-ray diffraction analysis therefore indicates the formation of a dual bulk amorphous alloy, which combines two distinct metallic amorphous phases.

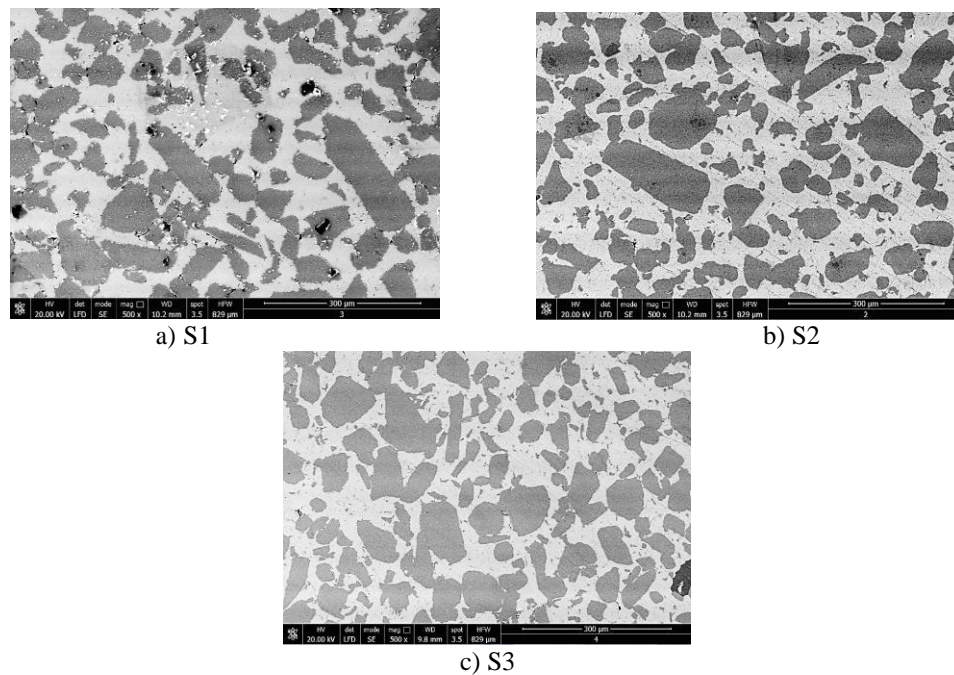


Figure 1. SEM morphologies of the hot-pressed samples

The magnetic properties of the hot-pressed samples, and of the Fe-based glassy and Ni powders were obtained using a fluxmeter-type installation and a conventional low-frequency induction method. The hysteresis loops thus obtained (Figure 3) allow for the estimation of the saturation magnetic induction, remanant magnetic induction, and of the coercive field. These results are summarized in Table 1.

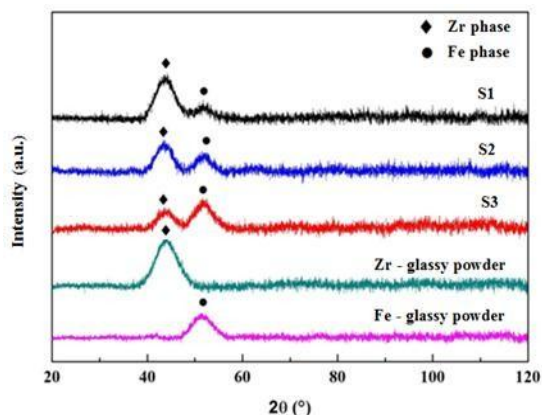


Figure 2. XRD patterns of the glassy powders and of the hot-pressed samples.

From the magnetization cycles in Figure 3, the very small coercive field values confirm the amorphous character of the

structure of the magnetic particles embedded in the zirconium matrix of the dual metallic material. The magnetic remanence (BR) values are also very small, this being partially attributable to a magnetostatic effect (the depolarization effect produced within the particles by the action of the demagnetizing field). This observation clearly indicates a superparamagnetic behavior. With respect to the saturation magnetic induction values (BS), the analyzed hot-pressed samples show a technically pure Ni approach (0.6 Tesla at room temperature).

Analysis of the values in Table 1 indicates that increasing the volumetric fraction of the Fe amorphous phase in Zr/Fe DAPBMGs raised the magnetic saturation induction values from 0.544 Tesla to 0.609 Tesla, and lowered the coercive field values from 1.458 kA/m to 1.262 kA/m. It can also be easily observed that, although the magnetic properties of the hot-pressed samples are slightly lower than the Fe-based amorphous powder, both the remanence and the coercive fields are an order of magnitude larger in the case of Ni powder compared to the bulk dual amorphous alloys. This characteristic of the analyzed Zr/Fe DAPBMGs, whose magnetic behavior is very close to superparamagnetism, affords them possible advantages in various applications such as electrotechnical materials (magnetic cores, magnetic screens, etc.).

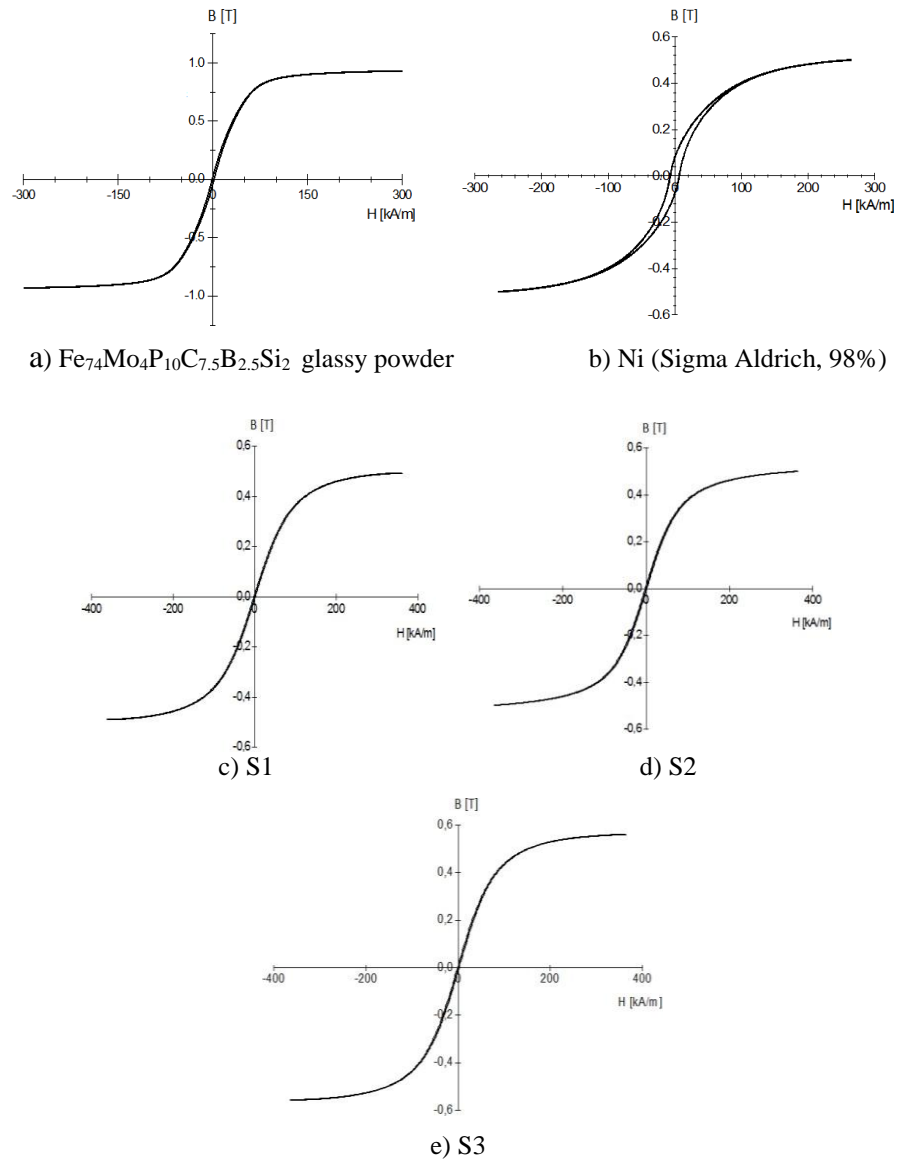


Figure 3. Hysteresis loops of the tested samples

Table 1. Magnetic properties of the tested samples

Sample	Saturation magnetic induction, B_s [T]	Remanent magnetic induction, B_r [T]	Coercivity, H [kA/m]
Fe₇₄Mo₄P₁₀C_{7.5}B_{2.5}Si₂ glassy powder	0.955	0.005	1.214
Ni crystalline powder	0.600	0.077	6.770
S1	0.544	0.008	1.458
S2	0.553	0.008	1.439
S3	0.609	0.007	1.262

4. Conclusions

Cylindrical Zr/Fe DAPBMGs with a diameter of 10 mm and height of 5 mm were successfully obtained by a hot-pressing method of Zr- and Fe-based glassy alloy powders.

X-ray diffraction analysis confirms the formation of a dual bulk amorphous alloy which combines two distinct metallic amorphous phases; a Zr-based amorphous phase and an Fe-based amorphous phase.

The magnetic properties of the hot-pressed samples are slightly lower than those of the Fe₇₄Mo₄P₁₀C_{7.5}B_{2.5}Si₂ glassy powder, but are superior to crystalline Ni powders. Increasing the volumetric proportion of the Fe-based amorphous phase leads to a decrease of the coercive field and an increase of the saturation magnetization. Both the remanent magnetization and the coercivity values of the analyzed Zr/Fe DAPBMGs indicate superparamagnetic behavior, making them useful in various applications as electrotechnical materials.

The compositional variant S3, containing 45% Zr-based alloy powder and 55% Fe-based alloy powder, yields magnetic properties closest to Fe₇₄Mo₄P₁₀C_{7.5}B_{2.5}Si₂, while exhibiting increased elasticity and ductility.

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