STUDY OF GLASS-METAL INTERFACE IN AMORPHOUS FERROMAGNETIC MICROWIRES

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Abstract: *In presented contribution two types of glass-metal bond are investigated by scanning electron microscope (SEM) equipped with EDS. Firstly, a mechanical bond, which usually provides weaker random joints. Secondly, chemical interaction, where the oxide layer on the metal surface forms a strong bond with the glass. Additionally magneto-impedance measurements are used to determine surface magnetic properties of microwires with and without glass cover. Considering giant magneto−impedance (GMI) effect, which is mainly a surface effect at higher frequencies, is very sensitive to the rotation of magnetization in the shell of a microwire. Thus GMI measurements are often used to determine magnetic anisotropy, hysteresis and residual magnetic domain structure formed around local defects (pits) on the surface of amorphous ferromagnetic microwires.*

Keywords: FERROMAGNETIC MICROWIRE, AMORPHOUS ALLOY, GLASS-METAL INTERFACE, MAGNETIC ANISOTROPY

1. Introduction

Glass-covered ferromagnetic amorphous thin wires (microwires) of a diameter about 10 μm are prepared by Taylor-Ulitovski technique [1, 2]. During the rapid quenching of microwires a glass-metal interface is formed. Because the coefficient of thermal expansion of metallic core is higher than that of the outer glass cover (Fig. 1), a rather complex inhomogeneous distribution of radial, axial and torsional mechanical stresses is induced in the metallic part of microwires [3]. Axial tensile stresses are dominant in inner part of metallic wire, and compressive stresses are dominant near the glass-metal interface. Magnetoelastic anisotropy of the investigated $Fe_{77.5}Si_{7.5}B_{15}$ microwire is given by axial tensile stresses and positive magnetostriction. Due to the minimization of magnetoelastic energy, the resulting magnetic domain structure of $Fe_{77.5}Si_{7.5}B_{15}$ microwire consists of a single axial domain in the center and radial multi domain structure near the glass-metal interface [3]. On the other hand the relatively small negative magnetostriction of amorphous glass-covered $Co_{70.5}Fe_{4.5}Si_{15}B_{10}$ microwire results in the creation of a wide almost circularly magnetized shell domain structure and a narrow axially magnetized core [4]. The preferential orientation of the spontaneous magnetization (magnetic anisotropy) in the microwire is given by magnetostriction and shape anisotropy. Different mechanical properties of the ferromagnetic metallic central part and of the glass cover of the microwire are responsible for deviation of spontaneous magnetization from circumferential (circular) direction in the shell of the microwire (helical magnetic anisotropy). Additional removing of the glass cover gives the possibility to decrease the helical anisotropy. Glass and metal can bond together by purely mechanical means, which usually gives weaker joints, or by chemical interaction, where the oxide layer on the metal surface forms a stronger bond with the glass. Therefore the detail understanding of mechanical and magnetic properties of the glass-metal interface is necessary for the possible applications of microwires in stress sensors, magnetometers and health monitoring [5].

2. Experimental methods

The scanning electron microscope (SEM) Hitachi TM3000 equipped with energy-dispersive X-ray spectrometer (EDS) Oxford Instruments Swift ED3000 has been used for experiment. We have prepared polished cross-section of $Fe_{77.5}Si_{7.5}B_{15}$ microwire. The atomic concentration of iron, silicon and oxygen in $Fe_{77.5}Si_{7.5}B_{15}$ microwire has been investigating by means of EDS line scan of cross-section. The gradient of oxygen and iron concentration has been detected.

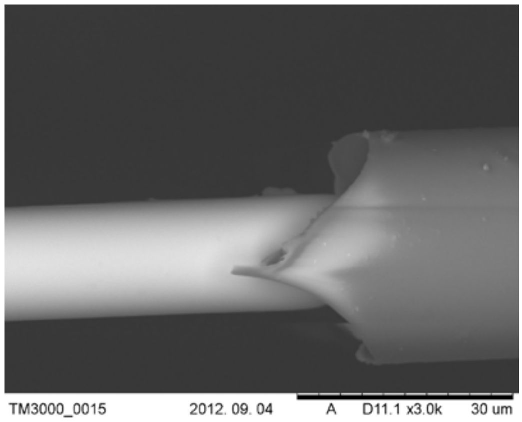

Fig. 1 *SEM image of glass-covered ferromagnetic amorphous $Fe_{77.5}Si_{7.5}B_{15}$ microwire.*

Mechanical glass-cover removing

The glass-covered microwire is usually fixed on a carbon tape during observation in SEM. After pulling down the microwire its glass-cover cracks and flakes away. Removed glass-cover remains glued to carbon tape (Fig. 2). Inner part of glass-cover interface can be visible in SEM and analyzed by EDS. Exposed surface of metallic part exhibits random distributed inclusions (pits) and small glass fragments (Fig. 3, 4).

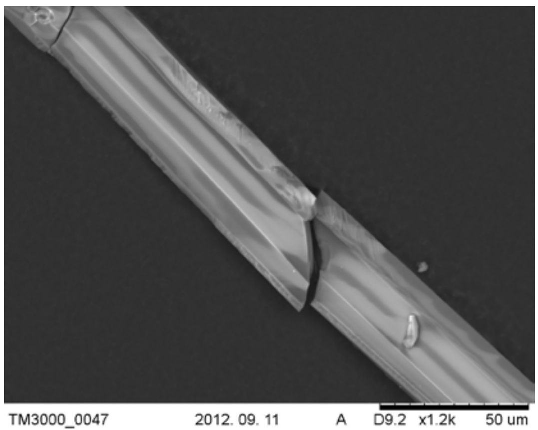

Fig. 2 *SEM image of mechanically removed glass cover from ferromagnetic amorphous $Fe_{77.5}Si_{7.5}B_{15}$ microwire.*

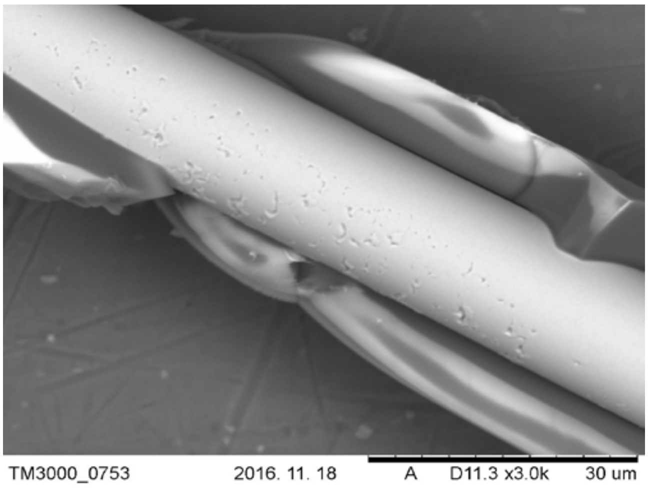

Fig. 3 *SEM image of ferromagnetic amorphous $Fe_{77.5}Si_{7.5}B_{15}$ microwire of a diameter d=17.8 μm with mechanically removed glass cover.*

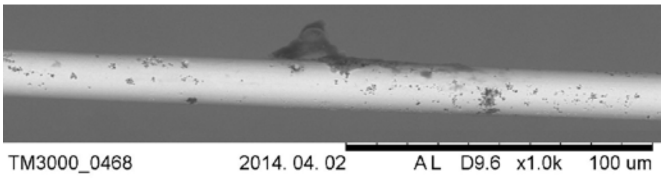

Fig. 4 *SEM image of ferromagnetic amorphous $Co_{70.5}Fe_{4.5}Si_{15}B_{10}$ microwire without glass cover of a diameter d=15.3 μm.*

Cross-section of glass-covered microwire

Cast-resin glass-covered microwire $Fe_{77.5}Si_{7.5}B_{15}$ has been polished and cleaned. SEM image of the microwire cross-section (Fig. 5) revealed the glass inclusions at the surface of inner metallic part. The glass anchored in the metallic part by means of inclusions produces strong axial and torsional mechanical stresses along the microwire (Fig. 6). On the other hand the radial tensile strength depends on the type of glass-metal bond: 1) mechanical bond, which usually provides weaker joints; 2) chemical interaction, where the oxide layer on the metal surface forms a strong bond with the glass. The character of the glass-metal interface can be investigated by EDS line scan of the microwire cross-section (Fig. 7).

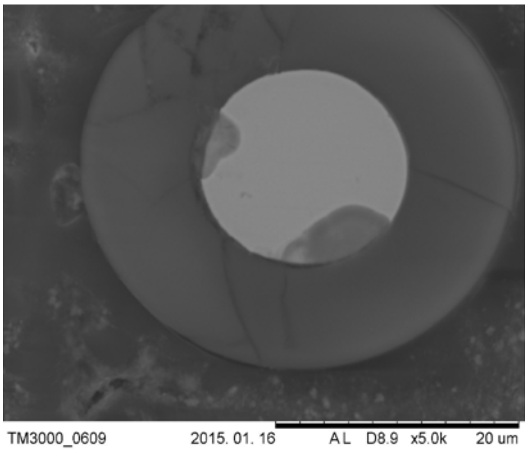

Fig. 5 *SEM image of the cross-section glass-covered ferromagnetic amorphous $Fe_{77.5}Si_{7.5}B_{15}$ microwire.*

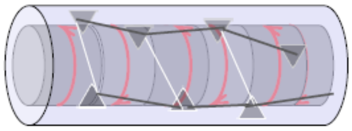

Fig. 6 *Scheme of random distribution of inclusion on the surface of the wire (black triangles). Axial and torsional mechanical stresses resulting from the coupling forces (black and white lines).*

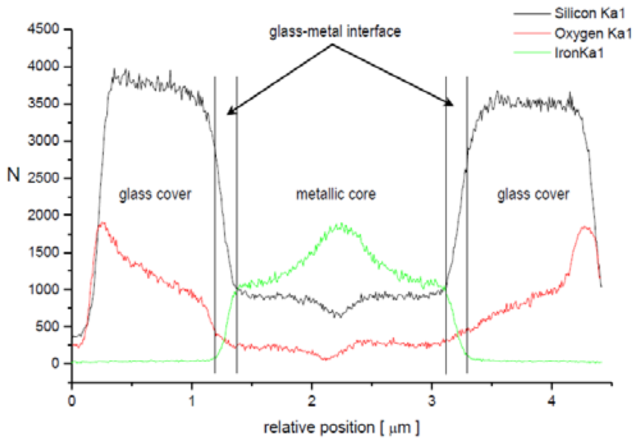

Fig. 7 *EDS line scan of the cross-section of glass-covered $Fe_{77.5}Si_{7.5}B_{15}$ microwire in Fig. 4 (green is iron, black is silicon and red is oxygen relative concentration).*

In Figure 7 the concentration gradient of oxygen (red curve) in glass-cover and the concentration gradient of iron (green curve) are displayed. This indicates that atomic diffusion processes occur during preparation of glass-covered microwire $Fe_{77.5}Si_{7.5}B_{15}$ consisting of rapid quenching and drawing of melted alloy in glass capillary. Glass and metal can bond together by purely mechanical means, which usually gives weaker joints, or by chemical interaction, where the metal oxide in glass-metal interface forms a stronger bond with the glass.

3. Results and discussion

Irreversible magnetization rotation

In case of a circular wire it is convenient to use cylindrical coordinates. Helical magnetic anisotropy is given by a preferential orientation (easy axis) of the spontaneous magnetization $\boldsymbol{M_0}$ of the microwire at zero external magnetic field what can be expressed as $\boldsymbol{M_0} = (0, M_\phi , M_z) = (0, M_s \cos\alpha, M_s \sin\alpha)$, where M_s is the saturation magnetization, α is the angle of deviation of the easy axis of magnetization from the circumferential direction of the microwire (spiral angle, $0<\alpha<90^o$). The angle α determines the shape of the hysteresis loop of the microwire (Fig. 8) during its magnetization along *z*-axis with irreversible magnetization rotation at the critical field [6]. The magnetization curve for $\alpha = 90°$ is without hysteresis (reversible magnetization rotation) and in this case the longitudinal wire *z*-axis represents a hard axis of magnetization.

Surface magnetic domain structure

Details of the metallic surface of the microwires, studied by means of SEM (Fig. 3, 4, 5), revealed surface defects (pits), where the glass cover is bonded to metal. The measured GMI dependences of as-cast $Co_{70.5}Fe_{4.5}Si_{15}B_{10}$ microwire with glass cover and after glass cover removing in Fig. 9 displays the double-peak behaviour [7, 8]. The theoretical explanation is that for very low amplitudes of circular field strength H_ϕ any reversible domain wall motion at higher frequencies (≥1 MHz) is negligible due to strong damping process and magnetization rotation takes place only in the shell of the microwire. The positions of the couple of sharp peaks ($H=\pm H_m$) are always symmetrical with respect to zero external magnetic fields strength $H=0$ and correspond to the critical field of irreversible magnetization rotation. The dispersion of the critical field altogether with local variation of the easy axis of magnetization affects the peaks shape.

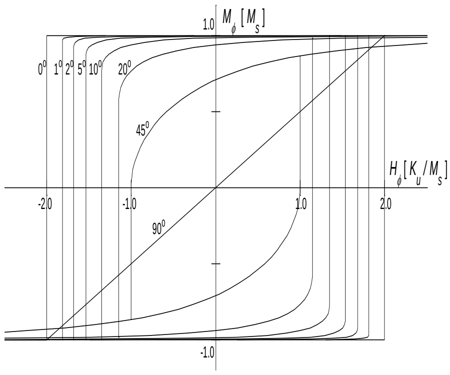

Fig. 8 *Calculated reduced magnetization curves M_ϕ (H_ϕ) in case of irreversible magnetization rotation and helical magnetic anisotropy for various value of α (in degrees).*

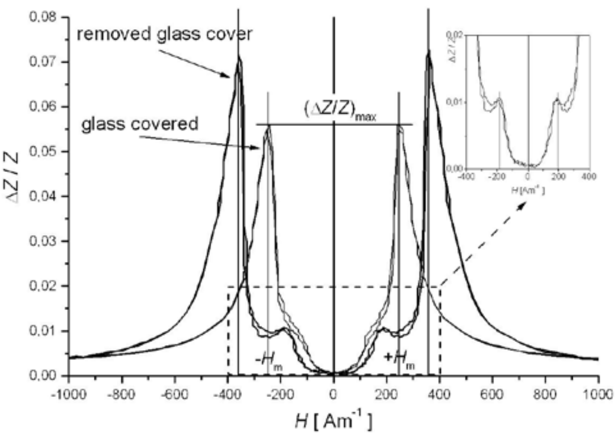

Fig. 9 *GMI dependence measured at the frequency of 1 MHz and at the amplitude i_{ac}=1 mA in as-cast $Co_{70.5}Fe_{4.5}Si_{15}B_{10}$ microwire of a diameter d = 8.1 μm with glass cover and after glass cover removing [7].*

The formation of a secondary small GMI peaks (inset in Fig. 9) has been observed after glass cover removing. The theoretical explanation is that the blade-shaped domains [6, 9], displayed in Fig. 10, are formed on both sides of surface defects (pits) to minimize magnetostatic energy. The blade-shaped domains are also responsible for hysteresis observed in GMI dependence.

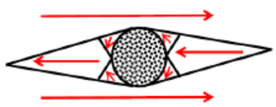

Fig. 10 *Scheme of the blade-shaped domains on the surface of the wire closing the defect (pit).*

4. Conclusion

The atomic diffusion processes occur during preparation of glass-covered microwire $Fe_{77.5}Si_{7.5}B_{15}$. The glass oxide bond is stronger than pure glass metal bond. The oxide forms a layer on the metal surface during preparation of glass-covered microwire consisting of rapid quenching and drawing of melted alloy in glass capillary. A too thick oxide layer tends to be porous on the surface and mechanically weak, compromising the bond strength and creating glass fragments along the metal oxide interface. Proper thermodynamic conditions are therefore critical for the homogeneous oxide layer creation. Another mechanism is the local glass growing into the metal and anchoring together the metal and glass, forming the inclusions and weaker mechanical bond.

The removing of the glass cover reduces tensile stresses in the microwire, changes the induced helical anisotropy and the angle α. This results in increasing of the critical field and the maximum value of GMI ratio $(\Delta Z/Z)_{max}$ in Fig. 9.

A residual domain structure formed around inclusions and local pinning centers of the glass fragments on the microwire surface manifests itself in formation of a secondary small GMI peaks (inset in Fig. 9).

Despite the fact that occurrence of the hysteresis in GMI dependences is a disadvantage from the application point of view, it introduces a valuable information about the magnetization dynamics and the magnetic behaviour of surface domain structure.

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References

[1] Larin, V. S., Torcunov, A. V., Zhukov, A., Gonzáles, J., Vázquez M., Panina, L.: Preparation and properties of glass-coated microwires. Journal of Magnetism and Magnetic Materials, Vol. 249, 2002, p. 39-45.

[2] Vázquez, M.: Soft magnetic wires. Physica B: Condensed Matter, Vol. 299, Issues 3-4, 2001, p. 302-313.

[3] Chiriac, H., Lostun, M., Óvári, T.-A.: Magnetization process and domain structure in the near-surface region of conventional amorphous wires. Journal of Applied Physics, Vol. 109, Issue 7, 2011, Article number 07B504.

[4] Chiriac, H., Óvári, T.-A.: Magnetic properties of amorphous glass-covered wires. Journal of Magnetism and Magnetic Materials, Vol. 249, 2002, p. 46-54.

[5] Praslička, D., Blažek, J., Šmelko, M., Hudák J., Čverha, A., Mikita, I., Varga R., Zhukov, A.: Possibilities of Measuring Stress and Health Monitoring in Materials Using Contact-Less Sensor Based on Magnetic Microwires. IEEE Transactions on Magnetics, Vol. 49, No. 1, 2013, p. 128-131.

[6] Chikazumi, S.: Physics of Magnetism, John Wiley &Sons, Inc., New York, 1964, p. 281.

[7] Kravčák J.: Hysteresis in Asymmetrical GMI Effect in Amorphous Microwires. Acta Physica Polonica A, Vol. 126, No. 1, 2014, p. 132-133.

[8] Kravčák, J.: Relation between helical anisotropy and asymmetric GMI effect. Acta Electrotechnica et Informatica, Vol. 13, No. 1, 2013, p. 53-55.

[9] Tebble, R. S., Craik, D. J.: Magnetic Materials, John Wiley&Sons, Inc., London, New York, 1969.