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Structural and Magnetic Properties of "Thick" Microwires Produced by the Ulitovsky-Taylor Method

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The structural and magnetic physical properties of initial and annealed Co-rich microwires in a glass shell with the diameter D = 70-125 μm and the diameter of the amorphous metallic core d = 70-95 μm, produced by the Ulitovsky–Taylor method, have been studied. The magnetic characteristics, in particular, the saturation field H_S and the coercive force H_C of the samples were found to depend on annealing temperature. The obtained experimental data was explained by the structural peculiarities of the microwires. The near-surface values of H_S and H_C were found to be larger than the bulk values by a factor of 5–10. These experimental data have been explained by the existence of structural and chemical ingomogeneities in the near-surface layer, which are inherent in amorphous materials.

Keywords: Magnetic properties, Magneto-optical effects, Microwires.

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1. INTRODUCTION

Although amorphous magnetic materials were obtained more 50 years ago, the interest of the investigation of their structural, magnetic and kinetic properties remains up to now. This fact is cased by the possibility of widely using amorphous materials in modern microelectronics (in particular, in high-sensitive sensors of magnetic fields, stresses, low pressures, strains, etc., and also new types of coding devices) with relatively low cost of their production [1-3].

One of the widely used methods of amorphous wire production is pulling from a melt with cooling in water, proposed by G.F. Taylor in 1924 [4] and later improved by A.V. Ulitovsky [5]. Amorphous ferromagnetic microwires, produced by the above-mentioned method, had the metallic core diameter no more than 40 μm and external glass layer about 0.5–15.0 μm. Recently, a new type of wires, namely, "thick" amorphous wires with a metallic core from 50 up to 120 μm in diameter were prepared using the modernized Ulitovsky–Taylor method [6]. The study of the physical properties of the new type of microwires deserves attention both scientific and practical stand points.

The aim of this work is the investigation of the structural, elastic, magnetic characteristics of the initial and annealed new thick Co-rich microwires and also the study of the influence of the glass cover on their magnetic characteristics.

2. PREPARATION OF SAMPLES AND EXPERIMENTAL TECHNIQUE

The $Co_{69}Fe_4Cr_4Si_{12}B_{11}$ microwires were produced by the modernized Ulitovsky–Taylor method by using alloys, prepared from components with purity higher than 99.8% [6]. The microwires with a glass shell had the diameter D = 125 μm and the diameter of the amorphous metallic core d = 90 μm. The microwires were annealed in a muffle furnace at temperatures T = 200-450°C for 10 min. The influence of the glass shell on the microwire properties was studied by its mechanical removal.

The fractograpfic study of the microwire was carried out by scanning electron microscopy. The amorphous state of the as-cast microwires was controlled by differential scanning calorimetry (DSC) using a Setaram Setsys Evolution microcalorimeter (the heating rate was20 K/min).

The plasticity level was estimated using a technological test of the microwire ability to knot formation. In this case, opposite ends of the microwire with a preliminary tied knot were stretched at a rate of 0.02 m/min. The character of the knot decrease was controlled by the optical method. The knot diameter, observed before the microwire fracture, was taken as the critical diameter $d$cr.

The magnetic characteristics of the ribbons were measured employing vibration and magneto-optical magnetometers. The simples under study had the length of 20 mm. The external remagnetizing magnetic field was applied parallel to the microwire length. The near-surface hysteresis loops were measured using the equatorial Kerr effect (EKE). The EKE magnitude δ is determined from relationship $\delta = (I - I_0)/I_0$, where I and I_0 are the light intensities reflected from the magnetized and nonmagnetized simples, respectively. Actually, the dependences $\delta(H)/\delta_S \sim M(H)/M_S$ were measured, where δ_S is the EKE magnitude at $M = M_S$, M_S is the saturation magnetization of the simple.

3. EXPERIMENTAL RESULTS AND DISCUSSION

Data of differential scanning calorimetry showed that the thick microwires under study retain amorphous structure. The pulling rate of the amorphous microwire was found to be almost two orders lower than the rates used in other methods of rapid quenching of melt when the microwires with similar cross sections are producing. Moreover, the glass shell in such wires is weakly adhered with the metallic core, and it can be removed easily. The wire core has the geometric parameters stable along its length, the smooth mirror surface almost without defects and very high, for amorphous alloys, plasticity level that is characterized by the ability of the wires to tighten into a knot without fracture (see Figure 1).

Fig. 1 – Image of the thick $Co_{69}Fe_4Cr_4Si_{12}B_{11}$ microwire, illustrating its ability to tighten into a knot without fracture

Figures 2 show typical near-surface hysteresis loops, observed for the initial and annealed $Co_{69}Fe_4Cr_4Si_{12}B_{11}$ samples. Analogous loops were obtained by using vibration magnetometer. These data allowed to find the dependences of the saturation field, H_S, and coercivity, H_C, on the annealing temperature (see Figure 3).

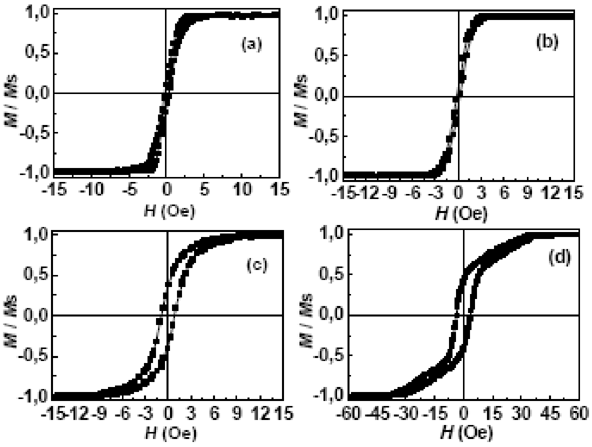

Fig. 2 – Hysteresis loops, observed for the initial $Co_{69}Fe_4Cr_4Si_{12}B_{11}$ microwire (a) and the microwires, annealed at T = 200 (b), 350 (c) and 430°C (d) by using the magneto-optical magnetometer

An analysis of the obtained experimental data showed the following. The marked increases of the near-surface values of the saturation field and the coercivity are observed at $T \geq 300$°C. At the same time, the values of H_C^{BULK} and H_S^{BULK} for the samples, annealed at $T < 400$°C, are almost unchanged. At $T > 400$°C, the near-surface and bulk values of the coercivity increase by almost one order of magnitude, and the shape of the near-surface hysteresis loop is changed (Fig. 2d).

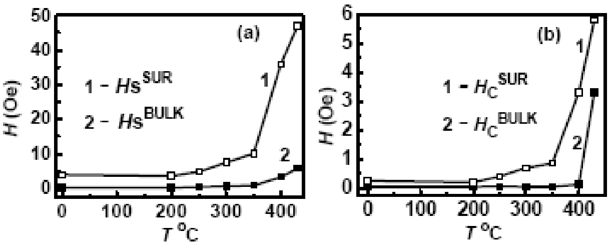

Fig. 3 – Dependences of the near-surface and bulk saturation fields (a) and the coercive forces (b) on the annealing temperature, obtained for the microwires

These experimental results can be explained by the structural peculiarities of the microwires. In particular, according to DSC data, obtained for the samples under study, temperatures of crystallization stages, the sequence of the exo effects, the total thermal effect of crystallization of the initial and the annealed up to 430°C of the thick wire samples are almost the same. These facts show that amorphous structure in the wire volume remains completely after annealing at temperatures up to 430°C. As a result, the bulk values of the saturation field and the coercivity depend weakly on temperature. At the same time, the magneto-optical studies showed that the near-surface magnetic characteristics of the microwire vary significantly sooner due to heat treatment, since the changes in the near-surface saturation field and coercive force as compared to bulk those are observed even at $T \geq 300$°C. It should be noted that this result agrees with our earlier magneto-optical studies of annealed amorphous ribbons [7], which showed that main variations of the magnetic properties of heat treated samples are observed in a near-surface layer about 1 μm in thickness. In particular, it was revealed in [7] that, after etching the near-surface layer up to the above noted thickness, the magnetic properties of the etched samples coincide with the characteristics of the initial ribbons within the limits of experimental error.

The hysteresis loop, measured by the magneto-optical method for the microwire, annealed at T = 430°C (Fig. 2d), has complex form. This fact shows that there is a two-phase magnetic structure on the metallic core surface. The specific shape of the hysteresis loop and also the sharp increase in H_C^{SUR} and H_S^{SUR} as compared to H_C^{BULK} and H_S^{BULK} permits us to make a justified assumption on the development of nanocrystallization in the near-surface layer, provided by thermal-activated diffusion redistribution of the components of the metal and glass. It is pertinent to note that similar effect of the surface nanocrystallization in amorphous Fe–Co microwires was also experimentally confirmed in [8, 9].

The difference between the bulk and near-surface values of H_S and H_C that increases at $T \geq 300$°C can be explained by the existence of chemical and microstructural heterogeneities on the microwire surface that are usually observed in amorphous materials [7].

To obtain complete information on the properties of the microwires under study, the magnetic characteristics were measured on the initial sample, in which glass shell was removed mechanically. It was found that after removal of the glass shell, the values of the near-surface and the bulk coercivity of the microwire are the same within the limits of experimental error. It should be noted that similar measurements were performed on the microwire of identical composition with the diameter of metallic core of 75 μm. In this case, the magnetic characteristics of the microwire after removing the glass shell were also unchanged (see Table 1).

Table 1

d / D, μm	H_C^{BULK}, Oe	H_C^{SUR}, Oe
90/125	0.05	0.2
90/ without glass	0.05	0.2
75/125	0.06	0.25
75/ without glass	0.06	0.25

This specific feature of the thick microwires can be explained by the fact that, as noted above, the Ulitovsky–Taylor method at producing the thick microwire, does not provide complete adhesion of the metallic core with glass. As a result, the stresses induced by the interaction of the magnetic core with a glass shell do not substantially influence the magnetic-field behavior of the samples.

4. CONCLUSION

The results of the investigation of the structural, thermo-physical and magnetic properties of the thick microwires allowed to make the following conclusions.

(1) The thick amorphous microwires have high soft magnetic properties in the initial rapidly quenched state.

(2) Heat treatment of the aforementioned samples at the annealing temperatures below 300°C does not substantially influence the hysteresis loop shape and also the values of the coercivity and the saturation field as compared to the initial microwire.

(3) Annealing at $T \geq 400$°C increases H_S and H_C by almost one order of magnitude, which is explained by nanocrystallization of the surface layer of the thick amorphous wire. At the same time, the main volume of the wire retains amorphous structure.

(4) The near-surface hysteresis loops, observed in the microwires, annealed at $T \geq 400$°C, have a more complex shape, which is indicative of structural changes of the near-surface layer.

As a whole, high technological properties of the thick amorphous microwires (in particular, the use of continuous method of production instead of the usual droplet method, low melting temperature, the absence of interaction between the melt and the glass, the possibility of performing mechanical removal of a glass shell) allow the production of long microwires without defects and with stable geometric parameters. The high strength of the thick amorphous microwire in combination with high plasticity and high soft magnetic characteristics makes allow to use such microwires as a force element with high sensor properties for monitoring of important constructions.

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