Structural and Magnetic Properties of Thick $Fe_{31}Co_{34}Ni_{10}(SiB)_{25}$ Microwires Produced by the Modernized Ulitovsky–Taylor Method

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Results on the study of the structural and magnetic properties of the as-cast $Fe_{31}Co_{34}Ni_{10}(SiB)_{25}$ thick microwires with glass shell, produced by the modernized Ulitovsky–Taylor method, are presented. The diameters of the amorphous metallic cores, *d*, of the microwires were equal to 55, 65, 95, 140, and 200 μm. The microwires had the stable geometric parameters along their lengths and the smooth (almost without defects) surface. The samples under study exhibited the high plasticity and high strength. The destruction of the microwires did not happen even after their full tightening into a knot. It was discovered that the glass shell of the microwires practically does not affect their magnetic characteristics. The saturation field H_S and coercive force H_C of the microwires raised with increasing the microwire diameter. The stable geometric parameters of the microwires along their lengths caused the slight dispersion of magnetic anisotropy in the near-surface layers. As a result, the $Fe_{31}Co_{34}Ni_{10}$ $(SiB)_{25}$ microwires exhibited the high homogeneity of the near-surface local magnetic characteristics. In particular, the changes of the local magnitudes of the saturation field were revealed to be $<5\%$.

Index Terms—**Amorphous magnetic materials, magnetic properties, material preparation, surface morphology.**

I. Introduction

Although amorphous magnetic materials were discovered more 60 years ago, the interest to studying their structural, magnetic, and kinetic properties remains up to now. This fact is cased by the possibility of wide use of amorphous materials in modern microelectronics and nanoelectronics with relatively low cost of their production. Amorphous magnetic materials have been used for producing magnetic heads; magnetic screens, transformer cores; magnetostriction vibrators; high sensitive sensors of magnetic fields, stresses, low pressures, strains, and also new types of coding devices, and so on [1]–[9]. The detailed review on applications of amorphous microwires is given in this paper [10].

At the present time, one of the widely used methods of producing of microwires without glass cover is the method of pulling of fusion from a quartz ampoule through the calibrated opening in cooling liquid. The metallic microwires with glass shell were produced by means of the method, proposed in [11]. Later, this method was improved in [12] and [13]. The amorphous microwires had the metallic core diameter no more than 40–45 μm and external glass layer ~0.5–15 μm. Recently, the Ulitovsky–Taylor method was modernized that allowed to receive new type of microwires from soft magnetic Fe–Co alloys, namely, thick amorphous wires with the metallic core up to 200 μm [14]. The study of the physical properties of the new type of microwires deserved attention as from the scientific and practical points of view.

In this paper, the results on the study of the mechanical, elastic, and magnetic characteristics of the as-cast thick $Fe_{31}Co_{34}Ni_{10}(SiB)_{25}$ microwires, produced by the modernized Ulitovsky–Taylor method, are presented.

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II. Samples and Methods of Investigations

The $Fe_{31}Co_{34}Ni_{10}(SiB)_{25}$ microwires with a glass shell were produced by the modernized Ulitovsky–Taylor method using alloys, prepared from components with purity higher than 99.8%. To increase the alloy purity during preparing the melt, the pumping, the refinement with use of helium-hydrogen mix and also homogenization of the fusion were performed. The ingot part was used for preparing rapidly quenched rods from which the microwires with glass shells were produced with the help of the modernized Ulitovsky–Taylor method. The method allowed to exercise continuous control of key parameters of process—temperature of fusion and hardening stream, speeds of the movement of the glass tube, and the precursor core, speeds of extract, and apportion of microwires, diameters of microwires.

The microwires had the diameters of the amorphous metallic cores d = 55, 65, 95, 140, and 200 μm. The influence of the glass shell on the microwire properties was studied by its mechanical removal.

The amorphous state of the as-cast microwires was checked with the help of X-ray diffraction (XRD). The plasticity level of the microwires was estimated by its ability to be tied into a knot without fracture. In this case, the opposite ends of a microwire, preliminary tied into a knot, were stretched with a speed of 0.02 m/min. The character of the knot decrease was controlled by the optical method. This method allowed to estimate the knot diameter, observed before the microwire fracture. Geometrical parameters of microwires, condition of a surface, and type of knot were investigated by methods of scanning electronic and the optical microscopy.

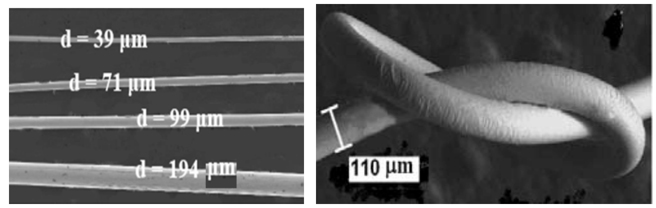
The bulk magnetic characteristics of the microwires were measured using the vibration magnetometer with the sensitivity 10^{-7} Gcm^3. The near-surface magnetic characteristics of the samples were measured employing the magneto-optical magnetometer, created on the basis of a high-resolution microscope [15]. The studied near-surface area of the sample was determined by the size of the slot, located in the image plane of the microscope before the light detector. The measurements of near-surface characteristics of the microwires were carried out by means of transverse Kerr effect (TKE). The TKE magnitude, δ, was determined from equation $\delta = (I - I_0)/I_0$, where I and I_0 are the light intensities, reflected from the magnetized and nonmagnetized sample, respectively. Actually, the dependences $\delta(H)/\delta_S \propto M(H)/M_S$ were measured, where δ_S is the TKE magnitude at $M = M_S$, M_S is the saturation magnetization of the sample, and H is the external remagnetizing magnetic field.

The magneto-optical Kerr effects are known to be sensitive to the magnetization of the near-surface layer of a certain thickness, corresponding to light penetration depth into medium, t_{pen}. The value of t_{pen} is determined from equation $t_{\text{pen}} = \lambda/4\pi k$, where λ is the wavelength of the incident light, and k is the absorption coefficient of the medium. According to the existing experimental data [16], the value of t_{pen} of magnetic materials in the energy range of incident light of 0.5–5.0 eV does not exceed 30–10 nm. In our case, the near-surface layer thickness under study was equal to 20 nm. The samples had 20 mm length. The external magnetic field was applied parallel to the microwire length, L. The near-surface hysteresis loops were measured at registration of magneto-optical signals from near-surface areas of 1×0.08 mm^2 (the large size is parallel to the microwire length, L). The measurements of the local magnetization curves for different near-surface areas and distributions of the magnetization components along the microwire length L, were carried out at registration of the magneto-optical signals from near-surface areas of 10×3 μm^2. These measurements allow to estimate degree of uniformity of the near-surface magnetic characteristics of the microwires under study. All the measurements were carried out on the central part of the samples to exclude the influence of end effects, in particular, to reduce the influence of variations of the local demagnetizing factors.

III. Results and Discussion

According to XRD data, the $Fe_{31}Co_{34}Ni_{10}(SiB)_{25}$ microwires are X-ray amorphous. The pulling rate of the microwires was found to be nearly two orders lower than the rate used in other methods of producing microwires with the same cross sections. The wire cores have the stable geometric parameters along their lengths; the smooth (almost without defects) mirror surface [Fig. 1(a)].

The morphology of the surface of the $Fe_{31}Co_{34}Ni_{10}(SiB)_{25}$ microwires was also studied by means of the atomic force microscope (AFM). The AFM data showed that the average value of the surface roughness of microwires does not exceed 20 nm that also confirms high quality of the surfaces of the microwires.



(a) (b)

Fig. 1. (a) Images of the amorphous microwires of various diameters, produced using the modernized Ulitovsky–Taylor method. (b) Image of the microwire, illustrating its ability to be tied into a knot without fracture.

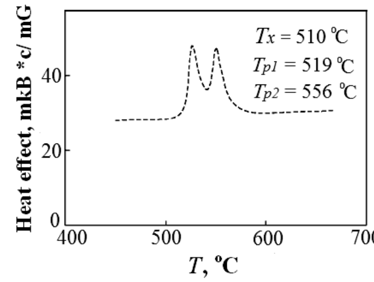


Fig. 2. DSK-thermograms of the $Fe_{31}Co_{34}Ni_{10}(SiB)_{25}$ microwires.

The glass shell in the microwires is weakly adhered with the metallic core and can be easily removed. This fact can be explained by the following. The main mechanism of adhesion in the studied microwires is mechanical adhesion, at which adhesive materials fill the voids or pores of the surfaces and hold surfaces together by interlocking. The modernized Ulitovsky–Taylor method in contrast of previously used drop method allows to obtain the microwires by continuous casting. The indicated specific of the method causes almost smooth surface of a metallic core of the microwires. As a result, the glass shell is weakly adhered with the metallic core.

The $Fe_{31}Co_{34}Ni_{10}(SiB)_{25}$ microwires were found to exhibit very high plasticity level, which is characterized by its ability to be tied into a knot [Fig. 1(b)]. It was found that the knot diameter, d_{cr}, before the fracture of the microwires under study did not exceed 0.2 mm.

The crystallization process of the samples under study was investigated by a method differential scanning calorimetry (DSK) at the continuous heating with the speed, equal to 20 °C/min, on the microcalorimeter Setaram Setsys evolution (Fig. 2).

The analysis of DSK data showed that the crystallization proceeds in two stages, in which the thermal emission size is practically identical. Temperature of the crystallization beginning of the alloy is rather high ($T_x = 510$ °C). Temperature interval between crystallization peaks $\Delta Tp = T_{P2} - T_{P1}$ is equal to 37 °C. As a whole, it was found that all the microwires are completely amorphous. The increase of the microwire diameter does not cause the noticeable decrease in the thermal stability of the alloy, the magnitudes of thermal effects, and the changes of temperature interval between peaks of crystallization.

The magnetic characteristics of the microwires were measured employing the magneto-optical and vibration magnetometers. Preliminary measurements showed that the distinction of magnetic characteristics of microwires with and

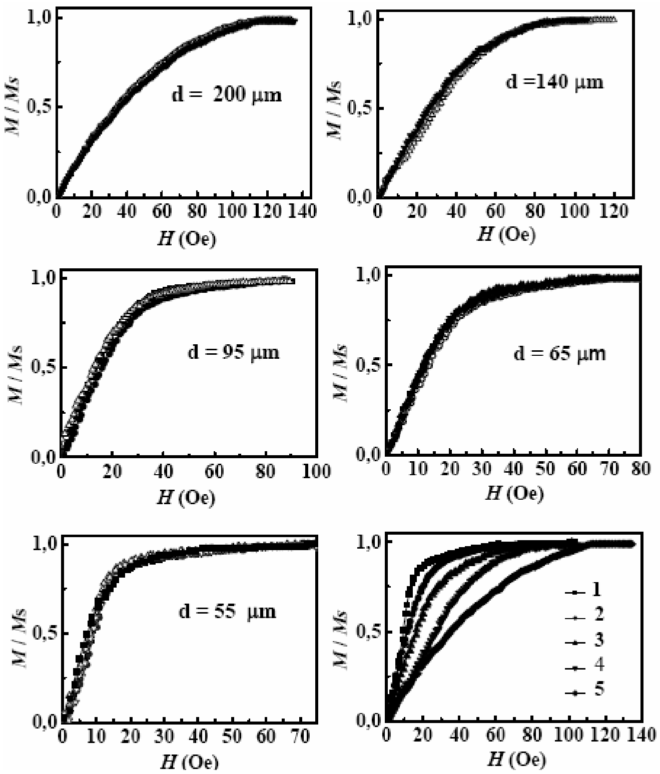


Fig. 3. Typical near-surface local curves of the magnetization, observed for the microwires under study using the magneto-optical magnetometer at registration of magneto-optical signals from surface area of 10×3 μm^2, and the magnetization curves 1, 2, 3, 4, and 5 of the microwires with $d = 55$, 65, 95, 140, and 200 μm.

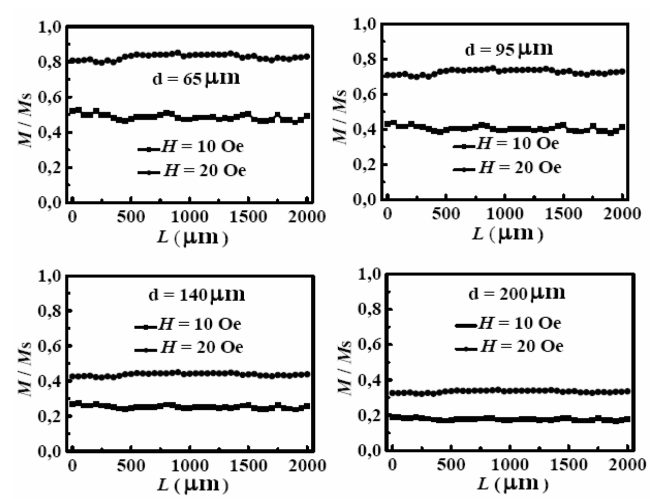


Fig. 4. Near-surface distributions of the magnetization components, parallel to L, observed for the $Fe_{31}Co_{34}Ni_{10}(SiB)_{25}$ microwires using the magneto-optical magnetometer.

without glass cover does not exceed 10% that is caused by weak adhesion between the glass shell and metallic core. All lower presented data were obtained for the microwires without glass cover. The typical local magnetization curves, distributions of magnetization components, parallel to L and hysteresis loops, received for the microwires using magneto-optical magnetometer, are shown in Figs. 3–5, respectively. Results of the bulk hysteresis loops are shown in Fig. 6.

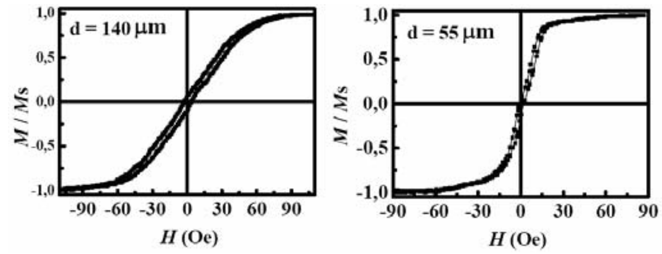


Fig. 5. Near-surface hysteresis loops, observed for the $Fe_{31}Co_{34}Ni_{10}(SiB)_{25}$ microwires using the magneto-optical magnetometer.

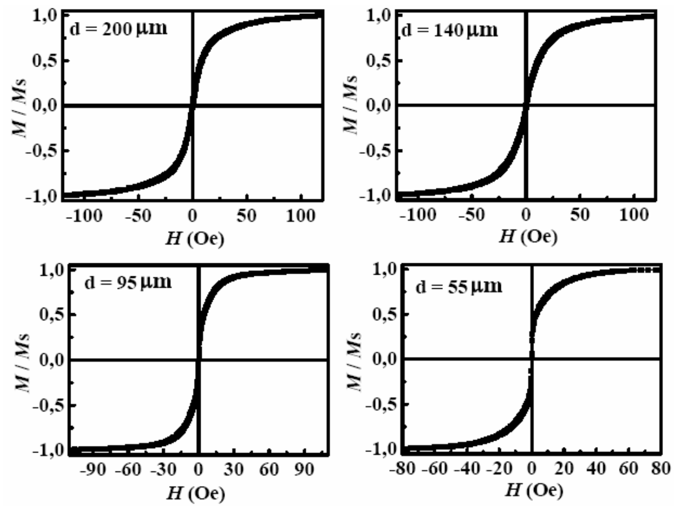


Fig. 6. Hysteresis loops, observed for the $Fe_{31}Co_{34}Ni_{10}(SiB)_{25}$ microwires using the vibration magnetometer.

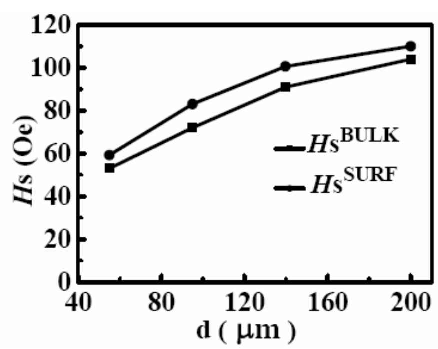


Fig. 7. Dependences of the bulk and near-surface values of the saturation field, H_S, on the diameter of the microwire core, d.

The data of Figs. 2 and 6 allowed to obtain the dependences of the bulk and near-surface magnitudes of the saturation fields, H_S, on the diameter of the microwire core d (Fig. 7).

Analysis of the above data showed the following. The variations of the near-surface magnetization curves and local values of the saturation field of the microwires do not exceed 5%. These results are evidence of the high homogeneity of the local magnetic characteristics of the samples under study, which can be ascribed to the slight dispersion of magnetic anisotropy, which is in turn caused by the stable geometric parameters of the microwires along their lengths and small roughness of the metallic core surfaces.

The linear dependence of the magnetization on the magnetic field, observed at small fields, show that in this case, the magnetic reversal of the samples realize in the main due to the rotation of magnetization vectors.

The magnitudes of $H_S{}^{\mathrm{SURF}}$ of the studied samples are higher than $H_S{}^{\mathrm{BULK}}$. However, this difference is much less in comparison with earlier observed by us for other amorphous microwires [17]. The observed insignificant distinction of $H_S{}^{\mathrm{SURF}}$ and $H_S{}^{\mathrm{BULK}}$ can be also explained by the stable geometric parameters of the microwires along their lengths and small roughness of their surfaces.

With growth of the microwire diameter, the values of the saturation field increase. This experimental fact can be explained by the influence of the demagnetization field on the magnetic characteristics of the studied samples. Expression for the macroscopic demagnetization field, H_N, of the microwires was received in the paper [18]

$$H_N = -4\pi \frac{\ln\left(2\frac{L}{D} - 1\right)}{\left(\frac{L}{D}\right)^2} M_S.$$

The calculation using this formula showed that the calculated curve of $H_S(d)$ qualitatively coincides with the experimental found dependence $H_S(d)$, but the experimental values of H_S are more the calculated ones. This quantitative difference can be caused by the following. The calculation of H_N was fulfilled without considering of the domain structure of the sample and, in particular, the domain structure of the near-surface layer of the microwires. However, the solution of this micromagnetic task is rather difficult and did not be found in literature.

IV. Conclusion

The results of the study of the structural and magnetic properties of the thick as-cast and annealed $Fe_{31}Co_{34}Ni_{10}(SiB)_{25}$ microwires allow to draw the following conclusions.

1) The microwire cores have the stable geometric parameters along their lengths and the smooth (almost without defects) surface.
2) The average value of the surface roughness of microwires does not exceed 20 nm that testify high quality of the surfaces of the microwires.
3) The amorphous microwires are characterized by the high plasticity and high strength. The destruction of the microwires does not happen even after their tightening into a knot. The knot diameter, d_{cr}, before the fracture of the microwires did not exceed 0.2 mm.
4) The crystallization of the microwires proceeds in two stages with close temperatures and thermal emission sizes.
5) The microwire glass shell practically does not influence on the magnetic properties of the samples due to the weak adhesion and can be easily removed.
6) With growth of the microwire diameter the values of the saturation field increase.
7) The stable geometric parameters of the $Fe_{31}Co_{34}Ni_{10}(SiB)_{25}$ microwires along their lengths cause the slight dispersion of magnetic anisotropy of the near-surface layers. As a result, the microwires are characterized the high homogeneity of the near-surface local magnetic properties.
8) The obtained properties of the $Fe_{31}Co_{34}Ni_{10}(SiB)_{25}$ amorphous microwires make these materials promising for practical applications. In particular, these microwires can find applications at manufacturing of constructional and functional materials, and also composites, containing microwires as power elements.

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