# Structural and magnetic properties of Fe<sub>73,5</sub>Si<sub>13,5</sub>B<sub>9</sub>Nb<sub>3</sub>Cu<sub>1</sub> microwires, produced by modernized Ulitovsky-Taylor method

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**Abstract.** Results of the investigation of the mechanical, elastic and magnetic characteristics of the "thick" Fe<sub>73,5</sub>Si<sub>13,5</sub>B<sub>9</sub>Nb<sub>3</sub>Cu<sub>1</sub> amorphous microwires, produced by the modernized Ulitovsky–Taylor method, are presented. The core of the Fe<sub>73,5</sub>Si<sub>13,5</sub>B<sub>9</sub>Nb<sub>3</sub>Cu<sub>1</sub> microwires were found to have the stable geometric parameters along their length and the smooth (almost without defects) surface. The studied samples are characterized by the slight dispersion of magnetic anisotropy of the near-surface layers and the high homogeneity of the near-surface local magnetic properties. Moreover, the microwires exhibit the high plasticity and the high strength. The strong influence of the stretching and torsion tensions on the remagnetization signal of the microwires was discovered.

### Introduction

Although amorphous magnetic materials were discovered more 60 years ago, the interest to studying of structural, magnetic and kinetic properties of ferromagnetic microwires remains up to now. This fact is cased by the possibility of wide use of amorphous materials in modern micro- and nanoelectronics with relatively low cost of their production, and also by the development of innovative materials and composites, derived on the base of the amorphous microwires. The detailed review on applications of amorphous microwires is given in the paper [1].

The metallic microwires with glass shell were produced by means of the method, proposed by G.F. Taylor in 1924 [2]. Later this method was improved by A.V. Ulitovsky [3]. The presence of a glass shell caused the strong inhomogeneity of magnetic properties along their length [4]. Recently, the Fe-rich amorphous microwires were prepared with the help of the modernized Ulitovsky–Taylor method [5]. This method in contrast of previously used drip method allowed to obtain the microwires by continuous casting and herewith to exercise continuous control of key parameters of process. The study of the physical properties of these microwires deserved attention as from the scientific and practical points of view. In this paper the results on the investigation of the structural, elastic and magnetic characteristics of the as-cast Fe<sub>73,5</sub>Si<sub>13,5</sub>B<sub>9</sub>Nb<sub>3</sub>Cu<sub>1</sub> microwires, produced by the modernized Ulitovsky–Taylor method, are presented.

## **Experimental**

The Fe<sub>73,5</sub>Si<sub>13,5</sub>B<sub>9</sub>Nb<sub>3</sub>Cu<sub>1</sub> microwires with a glass shell were produced by the modernized Ulitovsky–Taylor method by 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 shell were produced with the help of the modernized Ulitovsky–Taylor method. The pulling rate of the microwires was found to be nearly two orders lower than the rate used in other methods of producing microwires

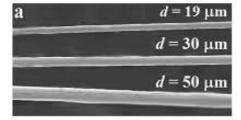
with the same cross sections. The studied microwires had the diameter of the amorphous metallic core d = 19, 30 and 50 microns.

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. The surface morphology of microwires was investigated by using methods of atomic force microscopy (AFM) and scanning electron microscopy (SEM).

The bulk and near-surface magnetic characteristics of the microwires were measured employing the vibration magnetometer with the sensitivity  $10^{-6}$  Gcm<sup>3</sup> and the magneto-optical magnetometer, described in [6]. The samples had the length of 20 mm. The external remagnetizing magnetic field was applied parallel to the microwire length, L. The near-surface hysteresis loops and the local magnetization curves were measured at the registration of magneto-optical signals from different near-surface areas of  $1 \times 0.08$  mm<sup>2</sup> and  $10 \times 3$   $\mu$ m<sup>2</sup>, respectively (the large size is parallel to the microwire length, L), located on the central part of the samples to exclude the influence of end effects, in particular, to reduce the influence of variations in the local demagnetizing factors. The influence of the glass shell on the magnetic characteristics of the microwire was studied by its mechanical removal.

#### **Experimental Results and discussion**

According to XRD data, the Fe<sub>73,5</sub>Si<sub>13,5</sub>B<sub>9</sub>Nb<sub>3</sub>Cu<sub>1</sub> microwires are amorphous. The glass shell in such wires is weakly adhered with the metallic core and can be easily removed. 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 weak adhesion of the glass shell with the metallic core can be the result of the smooth surface of the studied microwires. SEM data showed that the microwires core did have the stable geometric parameters along the length and the smooth (almost without defects) surface (Fig. 1 a and b). Moreover, according AFM data, the average value of the surface roughness of microwires doesn't exceed 20 nanometers that also confirms high quality of the microwires surface. The studied microwires were also found to exhibit very high plasticity level, which is characterized by its ability to be tied into a knot without fracture (Fig. 1 c).



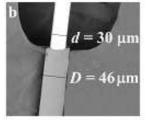




Fig. 1. Images of the microwires with different magnification (a) and (b), and the image, illustrating the microwire ability to be tied into a knot without fracture (c), obtained by using SEM.

The hysteresis loops for the studied samples were measured employing the magneto-optical and vibration magnetometers. Preliminary measurements showed that the distinction of the magnetic characteristics of the microwires without glass cover and in the presence of it doesn't exceed 10% that are also caused by the weak adhesion of the glass shell and metallic core. All presented data below were obtained for the samples without glass cover. The near-surface hysteresis loops and typical local magnetization curves, observed for the Fe<sub>73,5</sub>Si<sub>13,5</sub>B<sub>9</sub>Nb<sub>3</sub>Cu<sub>1</sub> microwires, are presented in Figs. 2 and 3, respectively. Analysis of the obtained data showed the following. The near-surface magnitudes of the coercivity, *H*c, and the saturation field, *H*s, increase with growth of the microwire diameter. This experimental fact can be explained by the influence of the demagnetization field on magnetic characteristics of the studied samples.

The microwires exhibit the negligible distinctions of the local near-surface magnetization curves. In particular, the variations of the near-surface local magnitudes of Hs and Hc of the microwires don't exceed 5%. The local hysteresis loops were revealed to have analogous behavior.

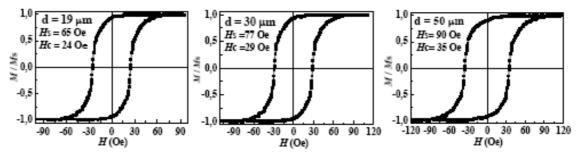


Fig. 2. Near-surface hysteresis loops, observed for the  $Fe_{73,5}Si_{13,5}B_9Nb_3Cu_1$  microwires with d=19, 30 and 50 microns employing the magneto-optical magnetometer.

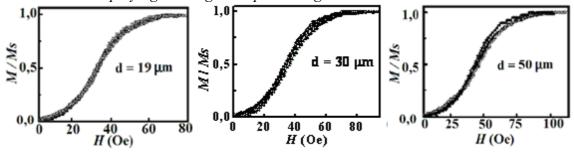


Fig. 3. Typical local magnetization curves, observed for the  $Fe_{73,5}Si_{13,5}B_9Nb_3Cu_1$  microwires employing the magneto-optical magnetometer.

The above results are evidence of the high homogeneity of the local magnetic properties of the microwires, 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.

The measurements employing the vibration magnetometer showed that the bulk values of Hs and Hc are less than the near-surface Hs and Hc. This difference doesn't exceed 13% and is much less in comparison with earlier observed by us for the microwires of the same composition. [4]. The magnetic-field behavior of the studied samples is different from the observed in [7-9]. In particular, the shape of the hysteresis loops is different from rectangular (the residual magnetization is about of 0,85 instead of 0,95), and the coercivity is almost an order of magnitude more than the found in [7-9]. This fact can be explained by the composition distinction of the samples, studied by us and authors of the papers [7-9].

The influence of the twisting tensions on magneto-field behavior of the microwires was investigated employing the magneto-optical method. In this case, one end of the microwires was rigidly fixed, and the second consistently twisted on angles, equal to 30°. The magneto-optical measurements of the hysteresis loops for such samples allowed to receive dependence of the magnetic characteristics on the torsion tensions. It was found that after the microwire twisting on 4 rad/cm, the magnitudes of *Hc* decrease at 5-7 times (see Fig. 4).

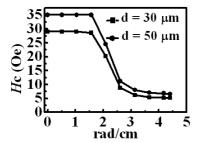


Fig. 4. The dependences of coercive force on the twisting angle, observed for microwires with d=30 and 50 microns.

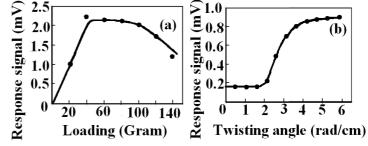


Fig. 5. The dependences of response signals of the microwire with d=30 microns on: (a) the stretching loading, (b) the twisting angle.

Moreover, the measurements of the Villari effect (response of magnetic materials, subjected to mechanical tensions, on an applied magnetic field) were carried out for the studied microwires. It

was found that the response signal (remagnetization signal) of the microwires linearly grows with increasing the loading up to 40 grams. The subsequent increase in the stretching tensions causes the signal decrease (Fig. 5 a). In case of the torsion tensions, the response signal increases beginning with the twisting angle, equal to 2 rad/cm. The increase of the twisting angle leads to the intensive growth of the signal (Fig. 5 b) that means the decrease of the microwire coercivity. These data are agreed with the results in Fig. 4. The measurements, executed in a cycling regime, showed that the signal changes are reversible. This fact means that the Fe<sub>73,5</sub>Si<sub>13,5</sub>B<sub>9</sub>Nb<sub>3</sub>Cu<sub>1</sub> microwires can be carried to group of alloys with rubber-like behavior, so-called "gum metals" [10].

### **Summary**

The investigation of the structural, elestic and magnetic properties of the Fe<sub>73,5</sub>Si<sub>13,5</sub>B<sub>9</sub>Nb<sub>3</sub>Cu<sub>1</sub> microwires allowed to draw the following conclusions. The microwire cores have the stable geometric parameters along their lengths and the smooth (almost without defects) surface. The average value of the surface roughness of the microwires does not exceed 20 nm that testify high quality of the surfaces of the microwires. As a result, the microwires exhibit the high homogeneity of the near-surface local magnetic characteristics. The studied samples 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 discovered properties of the microwires cause their wide practical application. In particular, they can be used in the products from polymers (pipes, panels, fitting), containing the microwires for the control of the tensile stress and twisting.

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#### **Achievements in Magnetism**

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