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Magnetic properties of iron nanowires in a porous aluminum oxide matrix

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Abstract. This paper is devoted to the study of magnetic properties of iron nanowire arrays synthesized using a porous Al_2O_3 template. Samples of porous aluminum oxide were obtained by double electrochemical anodization of the prepared foil in 0.5 M oxalic acid at a voltage of 60 V and a temperature of 25 °C and studied by scanning electron microscopy. The pore diameter is about 85 nm. Nanowires were electrodeposited in a three-electrode setup in the prepared matrices in a pulsed mode. The surfaces of porous membranes and the geometry of nanowires were studied using a scanning electron microscope. The magnetic properties of nanowire arrays were studied using a vibration magnetometer and micromagnetic modeling.

Keywords: anodizing, aluminum oxide matrices, nanowires, electrochemical deposition, vibrating magnetometry, micromagnetic modeling

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Магнитные свойства железных нанопроволок в пористой матрице оксида алюминия

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Аннотация. Настоящая работа посвящена исследованию магнитных свойств массивов железных нанопроволок, синтезированных с использованием пористого шаблона Al_2O_3. Образцы пористого оксида алюминия были получены путем двойного электрохимического анодирования подготовленной фольги в 0,5 М щавелевой кислоте при напряжении 60 В и температуре 25 °С и исследованы методом сканирующей электронной микроскопии.

Электроосаждение нанопроволок проводилось в трехэлектродной установке в подготовленные матрицы в импульсном режиме. Магнитные свойства массивов нанопроволок изучались с помощью вибрационного магнитометра и микромагнитного моделирования.

Ключевые слова: анодирование, матрицы оксида алюминия, нанопроволоки, электрохимическое осаждение, вибрационная магнитометрия, микромагнитное моделирование

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Introduction

One of the best and most extensive methods for ensuring repeatability and high quality of the resulting nanostructures is the use of porous anodic aluminium oxide (PAAO) templates [1–3], in particular arrays for the growth of controlled, self-organizing, as well as highly ordered structures, including nanorods, nanowires, nanotubes, nanocomposite materials with strict adherence to product dimensions at a packing density of 10^9–10^{11} units/cm^2 [4]. It is known that one of the most common method obtaining such nanowires (NW) is a matrix (template) synthesis – filling required material in narrow channels in a porous matrix. The unique properties of porous anodic aluminum oxide (PAAO) membranes make this material potentially the best template for electrochemical deposition of Fe nanowires [5]. Such nanowires are relevant as potential structures for 3D magnetic memory of the "racetrack memory" type [6].

Materials and Methods

To produce porous matrices of aluminum oxide, we used 2×2 cm Al (99, 9% purity) plates with a 0.5 mm thickness. To obtain a smooth surface, the samples were electropolished, resulting in a mirror-like surface. Anodizing was carried out using 0.5 M oxalic acid with the temperature about 25 °C in a potentiostatic mode at 60 V. The duration of the first anodization was $\tau = 30$ minutes, after which the oxide layer was removed in CrO_3 (1.8%) + H_3PO_4 (6%) at $T = 80$ °C for 10 minutes. Second anodization for the sample was carried out for 3 hours. Then, selective etching of aluminum was performed from the back side of the matrix in a solution of HCl + $CuCl_2$. After that, the barrier layer was removed in CrO_3 (1.8%) + H_3PO_4 (6%) at T = 80 °C for 2 minutes. To create a contact for further electrochemical anodizing, a Ti/Ag/Cu structure was created on the back side of the matrix by magnetron sputtering. The deposition of Fe into the pores was carried by the electrochemical method from $FeSO_4$ + K_2SO_4 + H_3BO_3 + ascorbic acid. The deposition was performed by the pulse method in a 3-electrode cell with an Ag/Cl reference electrode.

Results and Discussion

Fig. 1 shows an SEM image of the PAAO template before deposition, which shows that highly porous PAAO templates were successfully fabricated by two-step anodizing of the Al substrate. The SEM image of PAAO was further analyzed using ImageJ software, and pore diameter (D_p) = 85 ± 2.2 nm, pore distance (D_c) = 116 ± 5 nm.

Iron nanowires were formed by the electrochemical deposition method. Fig. 2, *a* shows a general plan of a sample of a porous matrix filled with nanowires. The length of the nanowires and the aspect ratio of the nanowires were 25 μm and 300, respectively. Fig. 2, *b* shows a SEM image of the sample on a smaller scale. Nanowires are continuous columns of a ferromagnet, the degree of filling is close to 100%.

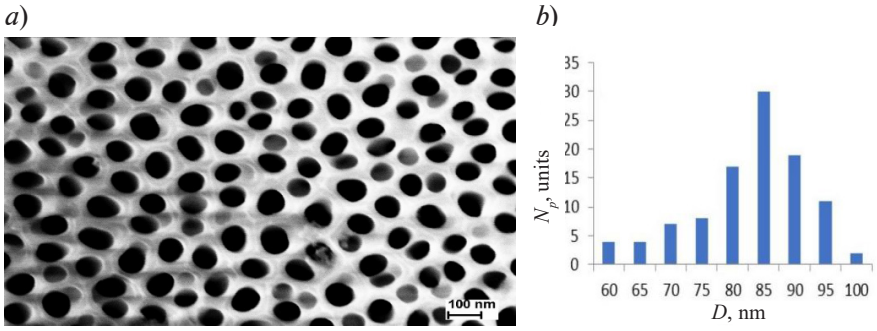

Fig. 1. SEM image of the PAAO template before deposition (*a*) and pores distribution (*b*)

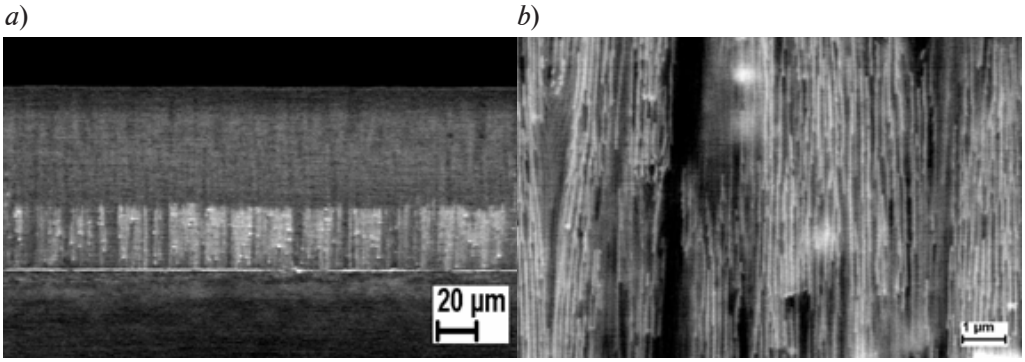

Fig. 2. Obtained Fe nanowires: chip profile of a PAO sample filled with iron nanowires (light tone of the sample below) (*a*), localized section of iron nanowires in an enlarged format (*b*)

Based on the obtained shape and ordering of the sample, micromagnetic modeling of the iron nanowire array was performed in the UberMag software package. The model is an array of 7 nanowires ordered in the form of a hexagon with one central thread. The distance between the centers, the length and the diameter of the nanowires were 120 nm, 500 nm and 85 nm, respectively. Each nanowire has the same parameters $A = 21 \cdot 10^{-12}$ J·m^{-1}, $M_s = 1.7 \cdot 10^6$ A·m^{-1}, $K_1 = 48 \cdot 10^3$ J/m^3. Fig. 3, *a* shows the resulting hysteresis loop for such an array. The coercive force was 340 Oe. The steps on the loop can be explained by the mechanism of magnetization reversal of such an array, where nanowires change the direction of magnetization alternately. The steps are formed due to the fact that the nanowires are remagnetized not in isolation, but in an ensemble. When one wire switches, it changes the local field acting on its neighbors. Micromagnetic modeling is based on the solution of the Landau-Lifshitz-Gilbert (LLG) equation on a discrete grid. Modeling records the energy of the system at discrete moments of time in the interval of specified discrete values of the field strength. A sharp change in m_z for one integration step or a step in the field strength appears as a step.

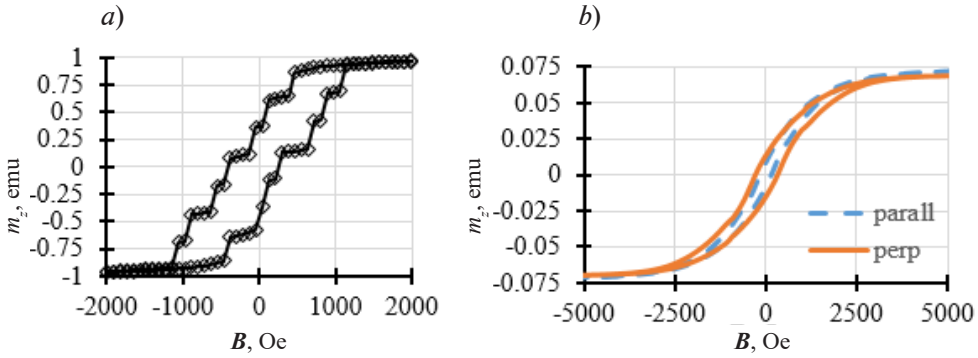

Fig. 3. Micromagnetic modeling of the hysteresis loop of the ensemble of iron nanowires (*a*), experimental loop for iron nanowires with parallel and perpendicular applied magnetic field (*b*)

Fig. 3, *b* shows the hysteresis loop of a real sample measured using a LakeShore 7407 vibration magnetometer. The measurements were carried out at room temperature from −16 kOe to 16 kOe with the magnetic field directed parallel and perpendicular to the sample surface. The dashed hysteresis loop is for the parallel orientation of the external magnetic field, and the solid one is for the perpendicular one. The coercivity was 310 Oe. Loops for the parallel and perpendicular directions of the external magnetic field are very similar, although with such sample parameters the loop for the parallel direction should have a larger slope and a smaller width. This fact can be explained by partial oxidation of iron, since it weakens the shape anisotropy by reducing the effective diameter of the ferromagnetic core. Greatly increases the coercivity in all directions due to pinning at the boundary with the oxide layer and possible exchange bias. As a result, even at high aspect ratios, demagnetizing fields can compensate for shape anisotropy, making hysteresis loops more similar.

However, comparing the simulation results, it can be seen that the coercive force for a perpendicular external magnetic field differs slightly and can be explained by the imperfect structure of the real sample. The absence of steps on Fig. 3, *b* is explained by the fact that in the real sample there are 10^9−10^{11} units/cm^2 and the steps are strongly smoothed.

Conclusion

In conclusion, can be noted that iron nanowires with an aspect ratio of 300 were obtained. A single iron nanowire in UberMag package was also simulated. The correspondence of the calculated and experimental data is confirmed. The coercive force for the model and experiment has a slight difference and is 340 Oe and 310 Oe, respectively. It can be concluded that shape anisotropy really exists for such samples, allowing them to be used as magnetic memory.

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