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INFLUENCE OF HIGH PRESSURE TORSION ON MAGNETIC PROPERTIES $Fe_{50}Ni_{50}$ AND $Fe_{49}Ni_{49}Al_2$ ALLOYS

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We report on magnetic properties of $Fe_{50}Ni_{50}$ and $Fe_{49}Ni_{49}Al_2$ alloys after severe plastic deformation (SPD) by high pressure torsion (HPT) technique. SPD considerably changes the magnetization behavior. The main magnetic characteristics of bulk and HPT materials were determined. Low temperature annealing was used to form the $L1_0$ structure. Low temperature annealing at $T = 300°C$ for $t = 1300$ hours of plastically deformed $Fe_{49}Ni_{49}Al_2$ alloy leads to an increase in saturation magnetization M_S by up to about 6%. However, it is premature to talk about the formation of even a small amount of phase $L1_0$. More research is required.

Keywords: *Hard and soft magnetic materials, high pressure torsion, severe plastic deformation, rare-earth free metals, permanent magnet, low temperature annealing, tetrataenite.*

Introduction

The China is a practically complete monopolist in the market of rare-earth elements. There are no alternatives to China in the supply of rare- earth elements. Growing in recent years, domestic demand for rare-earth elements in China has led to the restriction of their supplies to the international market, so there is an urgent need to develop alternative free rare-earth permanent magnets [1–6]. High performance permanent magnets have become indispensable materials in many industries, ranging from data storage to small motors and clean energy devices. Thus, the reduction of the content of critical elements in the production of permanent magnets is an adequate response to the crisis of the supply of rare-earth metals and their oxides and will make it possible to avoid the monopolistic dominance of China in the market of rare-earth elements.

Along with rare-earth systems, some Fe-based alloys are some of the most promising candidates for rare-earth compounds for the production of permanent magnets. Thus, the FeNi alloy with the $L1_0$ phase is a natural free rare-earth elements permanent magnet. Mineral tetrataenite has a high magnetization and energy density of magnetocrystalline anisotropy at room temperature in the range of 1.0-1.3 MJ/m^3 [7] and also large uniaxial magnetocrystalline anisotropy constant (K_u) of bulk $L1_0$-FeNi is

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as large as 1.3×10^7 erg/cc. [8; 9], which are comparable to the properties of the most modern permanent magnets such as Nd-Fe-B [10] and other $L1_0$-type alloys containing noble metals: CoPt [11], FePt [12], and FePd [13]. Phase is stable only at temperatures below 600 K, therefore diffusion of atoms is difficult, and for the formation of the $L1_0$ phase from the *A1* phase billions of years are needed [14; 15] without any external influences [16; 17], therefore an example of a magnetic material with the $L1_0$ phase is an iron or ironstone meteorite. The magnetic properties of tetrataenite are almost unexplored due to the difficulty of obtaining such a phase [18; 19].

Since the discovery of the hard magnetic phase in a meteorite, the search began in its receipt in laboratory conditions. Novadays there are several articles in the international press on attempts to synthesize the magnetic hard $L1_0$ phase. The presence of a magnetic hard phase was detected on FeNi samples which irradiated by intense neutron fluxes, expressed in an increase in the coercive force and a change in the saturation magnetization of the irradiated FeNi sample [20; 21]. The synthesis of the tetrataenite phase in thin films of the FeNi alloy was reported in [22–25] — in the work of K.B. Reuter [22] reported about using the method of irradiation with electrons with high energy, and in [23–25] reported about using the method of molecular epitaxy.

In addition to the experimental work, first-principle calculations were carried out using the electron density functional theory of phase formation processes in the FeNi system. For example, the results of modeling the spin structure, saturation magnetization, and others for the alloy with the composition $Fe_{2-x-y}Ni_xM_y$ (M = Al, P, S, Ti, V, Cr, Mn, Fe, Co) are given in [26]. Doping with different elements can stabilize the structure, increase the amount of $L1_0$ phase in the parent matrix of the FeNi alloy, and improve the magnetic properties. Some of these elements are included in the calculations purely from scientific curiosity, but most came from experimental work on this topic. For example, in [27; 28] it was suggested that sulfur has a positive effect on the formation of the $L1_0$ phase. The addition of phosphorus to calculate the possible properties of tetrataenite was due to the fact that meteorites may contain minor amounts of the phase $(Fe, Ni)_3P$, which, in turn, may influence the formation of the $L1_0$ phase.

In a previous work [29; 30], we reported the results of research in promising rare-earth free Fe-Ni-Ti alloys. The aim of this work is to study the magnetic properties of $Fe_{49}Ni_{49}Al_2$ alloy after severe plastic deformation (SPD) by high pressure torsion (HPT) technique. Low temperature annealing is planned to use for the formation of the $L1_0$ phase.

Sample preparation and measurements

The $Fe_{50}Ni_{50}$ and $Fe_{49}Ni_{49}Al_2$ alloy were prepared by argon-arc melting using a non-consumable tungsten electrode from the initial chemical elements Fe, Ni and Al with a purity of 99.99 at.%. The synthesized samples were inverted and remelted several times to achieve the best homogenization.

Our ingots were sliced and cut to disc about of 10 mm diameter and 1 mm thickness for HPT processing. The samples were placed between two anvils and was rotated under the applied pressure of 6 GPa at room temperature. SPD using HPT method were described in detail [31]. The total number of turns of the strikers for sample was $n = 5$. The diameter of the strikers was 10 mm.

X-ray diffraction (XRD) pattern were collected at room temperature by Brucker D8 Advance diffractometer using the monochromatic Cu Kα radiation and treated by modified Rietveld algorithm [32]. The elemental analysis of the synthesized materials were carried out using energy dispersive X-ray fluorescence spectroscopy (EDX). Magnetization curves were measured by a PPMS Quantum Design VersaLab at T = 50–350 K in magnetic fields up to 3 T. Low temperature annealing was carried out at $T = 300°$C for $t = 1300$ hours.

Result and discussion

The results X-ray diffraction analysis bulk and HPT of $Fe_{49}Ni_{49}Al_2$ samples are shown in Fig. 1. Severe plastic deformation by HPT method leads to the refinement of the structure of the material, which is reflected in the broadening of reflections on diffraction profiles. As the analysis shown, all samples are similar in phase composition and mainly consist of *fcc* (>99 wt%) and *bcc* (<1 wt%) phases. The lattice parameter a is equal (3.599 ± 0.001) Å for $Fe_{50}Ni_{50}$ bulk sample, is equal (3.600±0.001) Å for $Fe_{49}Ni_{49}Al_2$ bulk sample, is equal (3.578±0.001) Å for $Fe_{49}Ni_{49}Al_2$ HPT sample. As seen the lattice parameter a change insignificantly with the addition of aluminum for bulk samples and decrease after SPD.

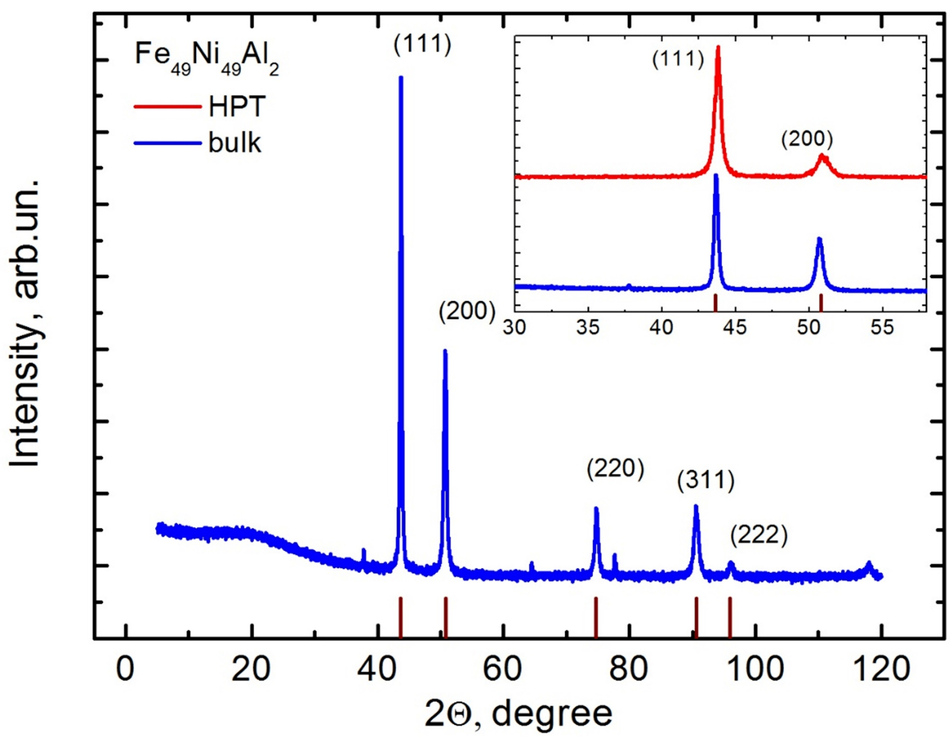

Fig. 1. X-ray diffraction pattern of bulk and HPT $Fe_{49}Ni_{49}Al_2$ samples

The results of magnetic field dependences of magnetization of $Fe_{50}Ni_{50}$ and $Fe_{49}Ni_{49}Al_2$ bulk samples at T = 300 K are shown in Fig. 2. The saturation magnetization M_S for $Fe_{50}Ni_{50}$ bulk samples is 154.6 Am^2kg^{-1} which is comparable, for example, with saturation magnetization of Morasko Meteorite-based $Fe_{51}Ni_{49}$ in as-quenched state (curve in Fig. 2 [33]). The coercive field is 67.6 Oe, and remanence is 18 Am^2kg^{-1}. The saturation magnetization decreased up to 10% with the addition of 2% Al and is to 147.8 Am^2kg^{-1}, coercive field is 66.2 Oe, and remanence is 5.3 Am^2kg^{-1} for $Fe_{49}Ni_{49}Al_2$ bulk sample. Almost the same values of the coercive force are also expected due to the fact that we study solid solution of aluminum in an iron-nickel alloy that are similar in composition.

As reported in [33], magnetization of the sample reaches its saturation M_S at about 2 T. We observed that magnetization of our $Fe_{50}Ni_{50}$ and $Fe_{49}Ni_{49}Al_2$ samples reaches its M_S at about 0.5 T. Also we observed that the samples after HPT technique reaches its saturation in significantly lower external magnetic field of about 0.2 T (Fig. 2). This behavior of magnetization is due to the microstructure and different shape anisotropy of bulk and HPT samples.

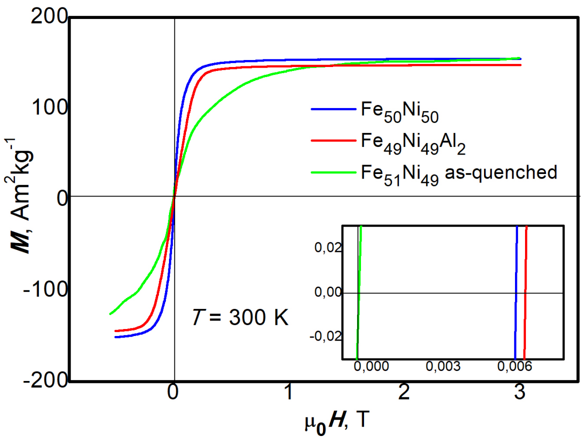

Fig. 2. Magnetic field dependences of magnetization of Fe-Ni bulk samples at $T = 300$ K. An enlarged low-field region of the hysteresis loop is shown in the inset. The result of magnetic field dependence of Morasko Meteorite-based $Fe_{51}Ni_{49}$ in as-quenched state is shown by a green curve [33]

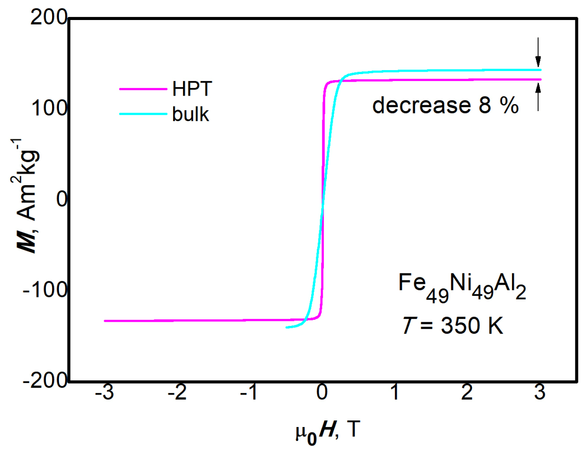

Fig. 3. Magnetic field dependences of magnetization of $Fe_{49}Ni_{49}Al_2$ bulk and HPT samples at $T = 350$ K

As a result of plastic deformation, the samples of $Fe_{50}Ni_{50}$ and $Fe_{49}Ni_{49}Al_2$ in the submicrocrystalline state were obtained with high concentration of defects and stressed state. As seen in Fig. 3, a decrease in saturation magnetization was found caused by an increase in the concentration of defects after plastic deformation. Value of M_S is about 8% smaller (145.2 Am^2kg^{-1} and 136.9 Am^2kg^{-1} for $Fe_{50}Ni_{50}$ and $Fe_{49}Ni_{49}Al_2$ alloys respectively) for HPT samples compared with the saturation magnetization of the bulk samples. Also a decrease in the coercive force of the plastically deformed material was found compared to the bulk samples. Decrease in the coercive force H_C is more than an order of magnitude. This behavior is quite different from most magnetic materials. One of the explanations may be the formation of a superparamagnetic state in which the coercive force is zero. The superparamagnetic state is interesting in that the loss of magnetization is practically zero due to the absence of magnetic hysteresis. The qualitative isothermal dependence of the coercive force H_C on the characteristic size of magnetic particles is shown in Fig. 4. One of the reasons for the increase in H_C with a decrease in the particle size is the following: with a decrease in the coercive force, more possibilities (mechanisms) appear for spin rotation in the direction opposite to the original. Rotation can be additionally associated with a displacement of the domain boundaries in multidomain particles. As the particle size decreases, the number of domains decreases, and the role of interdomain boundaries in magnetization reversal processes becomes less noticeable. Therefore, the coercive force increases with decreasing d up to the critical particle size d_{crit}. However, the role of thermal fluctuations increases with a further decrease in the particle size on going over to single-domain particles. This explains the decrease in H_C at $d < d_{\mathrm{crit}}$ [34].

According to [35], subsequent low-temperature annealing should accelerate the kinetics of phase formation and, possibly, lead to the appearance of nuclei of a stable chemically ordered tetrataenite phase. Our research (Fig. 5 and Fig. 6) shown that low-temperature annealing at $T = 300$ °C for $t = 1300$ hours of plastically deformed $Fe_{50}Ni_{50}$ and $Fe_{49}Ni_{49}Al_2$ samples leads to an increase in saturation magnetization M_S by up to

6% for different compositions. The value of magnetization also increases to 5% in an external magnetic field $\mu_0 H = 3$ T.

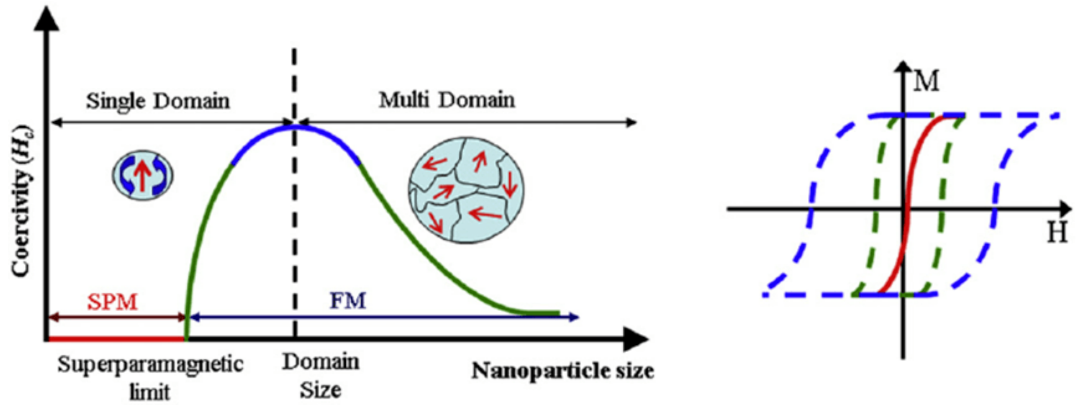

Fig. 4. Qualitative dependence of the coercive force H_C on the diameter particles [34]

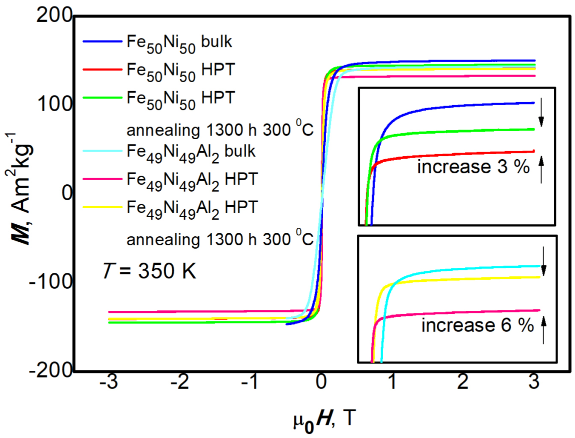

Fig. 5. Magnetic field dependences of magnetization before and after low temperature annealing at $T = 300°$C for $t = 1300$ hours of $Fe_{49}Ni_{49}Al_{49}$ and $Fe_{50}Ni_{50}$ HPT samples at $T = 350$ K

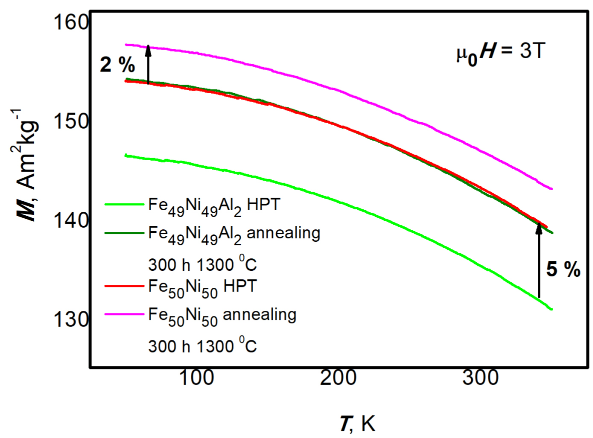

Fig. 6. Temperature dependences of magnetization before and after low temperature annealing at $T = 300°$C for $t = 1300$ hours of $Fe_{49}Ni_{49}Al_{49}$ and $Fe_{50}Ni_{50}$ HPT samples in magnetic field $\mu_0 H = 3$ T

Conclusion

Tetrataenite ($L1_0$-FeNi) is a promising candidate for use as a permanent magnet of rare-earth free elements because of its favorable properties such as high magnetic anisotropy and coercivity. Soft magnetic $Fe_{49}Ni_{49}Al_2$ and $Fe_{50}Ni_{50}$ metals were deformed by high pressure torsion and annealed at $T = 300°$C for $t = 1300$ hours for to form the $L1_0$ structure. When searching for phase we also used X-ray diffraction, energy dispersive spectroscopy, vibration sample magnetometry. However, our research shown that it is premature to talk about the formation of even a small amount of phase $L1_0$. More research is required.

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ВЛИЯНИЕ КРУЧЕНИЯ ПОД ВЫСОКИМ ДАВЛЕНИЕМ НА МАГНИТНЫЕ СВОЙСТВА $Fe_{50}Ni_{50}$ И $Fe_{49}Ni_{49}Al_2$ СПЛАВОВ

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Исследованы магнитные свойства сплавов $Fe_{50}Ni_{50}$ и $Fe_{49}Ni_{49}Al_2$, подвергнутых интенсивной пластической деформации (ИПД) методом кручения под высоким давлением. ИПД значительно изменяет поведение намагничивания. Определены основные магнитные характеристики литых и пластически деформированных материалов. Для формирования структуры $L1_0$ использовался низкотемпературный отжиг. Низкотемпературный отжиг при температуре $T = 300$ °C в течение $t = 1300$ часов пластически деформированного сплава $Fe_{49}Ni_{49}Al_2$ приводит к увеличению намагниченности насыщения M_S примерно до 6 %. Однако говорить об образовании даже небольшого количества фазы $L1_0$ преждевременно. Требуются дополнительные исследования.

Ключевые слова: *мягкие и жёсткие магнитные материалы, кручение под высоким давлением, интенсивная пластическая деформация, безредкоземельные металлы, постоянный магнит, низкотемпературный отжиг, тетратенит.*

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Сведения об авторах

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