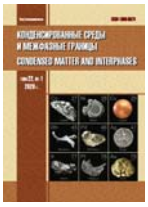

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X-ray Diffraction Analysis of Thin Metal Films with Magnetic Layers of Fe-Cr-Co Alloy

© 2020 V. S. Zayonchkovskiy✉[a], I. A. Antoshina[b], Aung Kyaw Kyaw[a], E. I. Isaev[b], I. M. Milyaev[c]

[a] *Kaluga branch of Bauman Moscow State Technical University (national research university), 2 Bazhenova str., Kaluga 248000, Russian Federation*

[b] *Obninsk Institute for Nuclear Power Engineering (OINPE) – a branch of National Research Nuclear University MEPhI, 1 Studgorodok, Obninsk 249040, Kaluga region, Russian Federation*

[c] *Baikov Institute of Metallurgy and Materials Science, RAS, 49 Leninsky prospekt, Moscow 119334, Russian Federation*

Abstract

The aim of this study was to determine the phase composition of the structures of permanent magnet films with layers of a Fe-Cr-Co alloy of micron range thickness, also known as the Kaneko alloy. The information about the phase composition is necessary for the development of physical and technical approaches for the production of optimal structures with permanent magnet films on single-crystal silicon wafers, the films being based on a dispersion-hardened alloy with the magnetization vector in the plane of the silicon substrate.

Three-layer metal films were obtained by magnetron sputtering on a silicon wafer: a dispersion-hardened alloy layer based on the Fe-Cr-Co system (3600 nm thick),), a compensating copper layer (3800 nm), and a vanadium adhesion barrier layer (110 nm). Multilayer films formed on a silicon wafer were subjected to one-minute of annealing in a high vacuum in the temperature range of 600–650 °C. A qualitative phase analysis of the structures obtained by magnetron sputtering and subjected to a single-stage thermal treatment was performed using X-ray diffraction.

It was determined that high-vacuum "rapid" one-minute of annealing of the Fe-Cr-Co dispersion-hardened alloy layer in the temperature range of 600–650 °C does not result in the formation of oxides of the main components or the σ-phase. At the temperature of 630 °C, the maximum intensity of the X-ray diffraction line (110) of the α-phase is observed, which indicates the formation of a predominantly α-solid solution and serves as a basis for the correct implementation of the subsequent annealing stages for the spinodal decomposition of this phase.

Keywords. magnetron sputtering, thin films, coercive force, vacuum annealing, phase composition.

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1. Introduction

Bulk permanent magnets are produced from various materials. However, permanent magnet films used in microelectronics can only be made from materials which are suitable for use with integrated circuit technology. Permanent magnet films are used to bias magnetically soft active layers of magnetoresistors of magnetoresistive integrated circuits (MIC) on silicon substrates [1–4]. Such MICs are used to produce contactless current sensors and read/write heads of disk drives. The in-plane bias of magnetoresistors is created in order to neutralise the Barkhausen effect. The high-quality magnet films described in [4] were based on precious metals, which increased the cost of such magnets significantly. An alternative to using precious metals is to create magnets using dispersion-hardened alloys (DHA) based on the Fe-Cr-Co system with micron range thickness, also known as the Kaneko alloy

✉ Vyacheslav S. Zayonchkovskiy,
e-mail: zajonc4340@gmail.com

[1, 5], and the Al-Ni-Co system [6]. The properties of these alloys depend on the properties of phases formed in them after spinodal decomposition, as well as the mode of thermal treatment. The formation of σ- and γ-paramagnetic phases has a destructive effect on magnetic properties of the materials in the two systems [4,5]. Bulk Fe-Cr-Co alloys have better mechanical properties than Al-Ni-Co alloys. Furthermore, the only way to produce Al-Ni-Co ingots used as targets for magnetron sputtering is by moulding. Fe-Cr-Co alloys can be mechanically shaped or cut. The production of Fe-Cr-Co based magnets involves thermal treatment in a magnetic field, during which crystalline anisotropy of the α_1-phase develops, which results in high coercivity [7]. The higher the magnetic field during the annealing in a magnetic field, the higher the coercivity during the subsequent stepped annealing [8]. In order to produce high-quality magnets from these materials, their phase states at various stages of thermal treatment should be carefully analysed.

[2, 3] describe the properties of Fe-Cr-Co alloy-based films formed on silicon wafers. Coercivity H_C is the most important property of hard magnetic materials. Its value in the submicron thick layers of DHA-based films is high enough for them to be used in magnetoresistive MICs. The H_C of the films with multi-micron thick layers of DHA is significantly lower [9]. For the H_C of the multi-micron thick layers to be optimal, it is necessary to control the spinodal decomposition of the body-centered α-phase of the uniform solid solution of chromium in iron in the absence of γ- and σ-phases. This can be done by analysing the phase composition of the layers of the above mentioned films.

The aim of this study was to determine the phase composition of the layers of DHA in order to develop physical and technical approaches for the production of an optimal heterostructure with a permanent magnet film based on dispersion-hardened alloy of Fe-Cr-Co of micron range thickness with a magnetization vector in the plane of the silicon substrate.

2. Experimental

2.1. The method of obtaining films

Thin metal films were produced using magnetron sputtering conducted in a modernized vacuum unit UVN-71P3, with three thermal evaporation sources having been replaced with three planar magnetrons. The fore-vacuum pressure was created using a 2NVR-5D rotary vacuum pump, and high vacuum conditions were created using an NBT-950 turbomolecular pump. In order to ensure the high quality of the residual gases, the lid of the vacuum chamber and the substrate holder were heated. The pressure of the residual gases in the vacuum chamber before the introduction of the plasma-forming gas (highly pure argon 5.5) did not exceed $5 \cdot 10^{-4}$ Pa. The substrate temperature prior to the deposition of the adhesion layer (vanadium) was 200 °C. The temperature was controlled by means of a temperature sensitive resistor with a sliding contact. The sputtering targets were vanadium, copper, and Fe-Cr-Co alloy disks with a diameter of 90 mm. The magnetrons were operating with the plasma current of up to 3 A. In order to reach a highly effective state, rapid thermal processing (rapid annealing) was performed in a vacuum furnace with vacuum created by an oil-diffusion pump. The residual gas pressure did not exceed 10^{-3} Pa. After the oil-diffusion pump was applied, the atmosphere in the furnace was reducing.

2.2. Measuring equipment

X-ray phase analysis was performed by a DRON-7M diffractometer using copper radiation with the wavelength $\lambda_{K\alpha m} = 0.154186$ nm [10].

The scanning was performed in step scan mode with the angular step size of 0.05°, the X-ray tube current of 30 kV, the anode current of 15 mA, and the exposure time of 5 sec. The angular scan range was 10–110°.

2.3. Film structures

The article presents the results of the study of structures obtained on single-crystal double side polished silicon wafers KDB-10 with (111) spatial orientation. For the X-ray analysis square samples were used with 15 mm sides. First, a 100 nm vanadium layer was deposited on the wafers. They were then covered with a 3800 nm copper layer followed by a 3600 nm Fe-Cr-Co layer. The thickness of the layers was measured by a confocal microscope NanoFocus,μ-Surf modification [11], based on the patterns obtained by means of photolithography with subsequent wet etching.

Concentrations of the main components of the target (Cr 25wt% and Co 12wt%) were determined by a X-ray fluorescence portable spectrometer MetExpert [12]. In our study, we used a target produced from Fe-Cr-Co alloy in order to obtain layers of DHA. The bulk alloy based on the Fe-Cr-Co system was subjected to basic thermal treatment, which, however, yielded comparatively low magnetic parameters.

It is possible to sputter a single layer DHA film on silicon within a wide thickness range (80–3.800 nm). However, with thicker DHA layers the bending strain of the round single-crystal silicon wafer becomes stronger. To reach high coercivity (HC) values annealing had to be performed, as it allowed for spinodal decomposition of the supersaturated solid solution of chromium in iron. The process was accompanied by partial decomposition of DHA layers, which became separated from the underlying layers.

In order to preserve the structure of the film, a compensating copper layer was introduced, which has a large deformation range due to the higher δ percent elongation ($\delta_{max\ Cu}$ = 45 %) [13]. However, this layer has low adhesion to single-crystal silicon, which is why we also added an adhesion vanadium layer. This layer provides poor compensation for the modulation of the crystal lattice of DHA layers ($\delta_{max\ V}$ = 17 %), [14], but it has high adhesion to single-crystal silicon [15] and is compatible with the copper layer [16]. This happens because vanadium does not form any intermetallic compounds with copper. The vanadium layer also serves as a diffusion barrier for copper, preventing direct contact between copper and silicon.

Most often, when manufacturing bulk magnets based on various DHA types by means of high-temperature homogenization and subsequent quenching, it is necessary to prevent the formation of undesirable γ- and σ-phases, which occur during the cooling of ingots in air at temperatures over 600 °C [17]. However, for the composition needed for our experiment (Fe – 25 wt % Cr –12 wt %Co) this requirement was optional [18]. The method we used allowed us to deposit the films by magnetron sputtering with the temperature of the wafers not exceeding 200 °C, which is enough for the melting of DHA layers.

3. Results and discussion

Fig. 1 shows an X-ray diffraction pattern of the metal film deposited on a (111)-oriented silicon wafer. Very intense (111) and (333) peaks of the silicon wafer are observed. The maximal peaks of the sputtered film layers are practically not visible at this resolution.

To analyse the three-layer metal film we used X-ray diffraction patterns recorded within an angle range of 40–94° in order to rule out highly intense peaks of the wafer (Fig. 2). The scanning step was 0.05°. In this range of angles 2θ a weak silicon peak (222) is observed. The presence of peaks of various intensity of reflection (111) complies with the International Centre for Diffraction Data PDF-2 (card No. 03-065-1060 ICDD PDF-2).

The diffraction pattern shows peaks corresponding to the α-phase of the solid solution FeCrCo (cards No. 03-065-4664 and 00-034-0396 ICDD PDF-2). Peak (110) (Fig. 2) was used to calculate the lattice constant of the α-phase a = 0.2858 nm, which corresponds to the lower bound of the range a = (0.286–0.289) nm, according to [21].

Copper peaks were indexed (Fig. 2). Copper has a face-centred cubic lattice with the lattice parameter a = 0.36148 nm [23]. According to [22], Fe-Cr-Co alloys may contain a γ-phase with a face-centred cubic lattice and the lattice constant a = (0.357–0.361) nm. The lattice parameters of copper and γ-phase are very similar. It was, therefore, impossible to separate the copper and γ-phase peaks within our study. The peaks may be separated, for example, by means of neutron diffraction.

High coercivity values for bulk samples were obtained only when the annealing temperature was within the range of 500–700 °C [17]. In order to determine the phase composition of the DHA layers after the thermal treatment, we used samples composed of a single silicon wafer with a metal film sputtered over it. The samples were annealed in a vacuum furnace for 1 minute at a temperature of 600–650 °C [17].

Before comparing the diffraction patterns of the annealed samples, we needed to make sure, that they had been recorded under identical conditions. In order to do this we needed to identify a diffraction peak that did not depend on the conditions of the annealing. This was

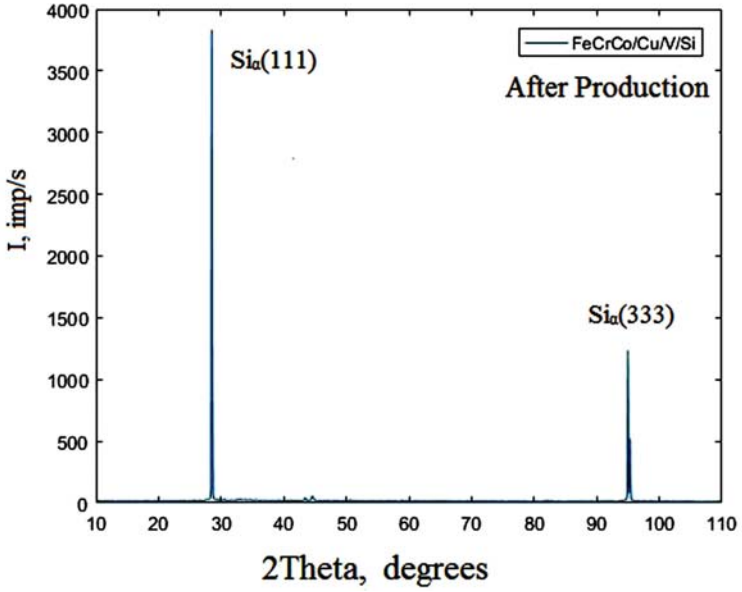

Fig. 1. Diffraction pattern of the three-layer metal film on a (111)-oriented single crystal silicon wafer. Scanning step 0.1°, recording interval (10–110)°

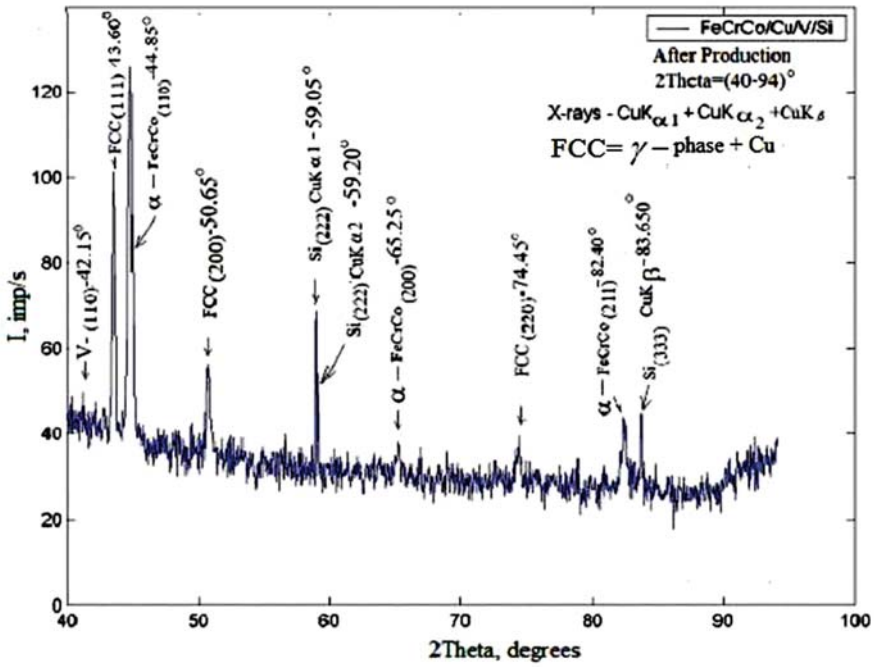

Fig. 2. Diffraction pattern of the three-layer metal film on a (111)-oriented single crystal silicon wafer. Scanning step 0.1°, recording interval (40–94)°

the (222) double peak, resulting from the X-rays going through the deposited thee-layer metal film and being reflected by the silicon wafer (Fig. 3). Figure 3 demonstrates the maximal peak (222) of the silicon wafer for the samples after the film sputtering and after the subsequent annealing at the temperatures of 600, 630, and 650 °C. The intensity of the peak stayed constant, which proves that the speed of photon counting during the scanning sessions was the same with a precision of up to 5 %.

Having established the stability of the recordings, we could analyse the alterations in the α-phase composition based on the difference in the intensity of the diffraction maximal peak (110). Figure 4 shows that line (110) has the

maximal integral intensity. This line is present in the sample annealed at 630 °C.

The profiles of the diffraction line (111) of the γ-phase + Cu in the samples after the film deposition and subsequent annealing at the temperatures of 600, 630, and 650 °C are presented in Fig. 5.

A characteristic feature of the conducted experiments was the dependence of the intensity of diffraction maximal peaks of line (110) of the α-phase and line (111) of the γ-phase + Cu on the annealing temperature (Fig. 6) The maximal intensity of line (110) of the α-phase is observed in the sample annealed at 630 °C. At the same time, line (111) of the γ-phase + Cu reaches its minimum. The thickness of the copper layer stays constant during the annealing within the chosen temperature range. Therefore, the

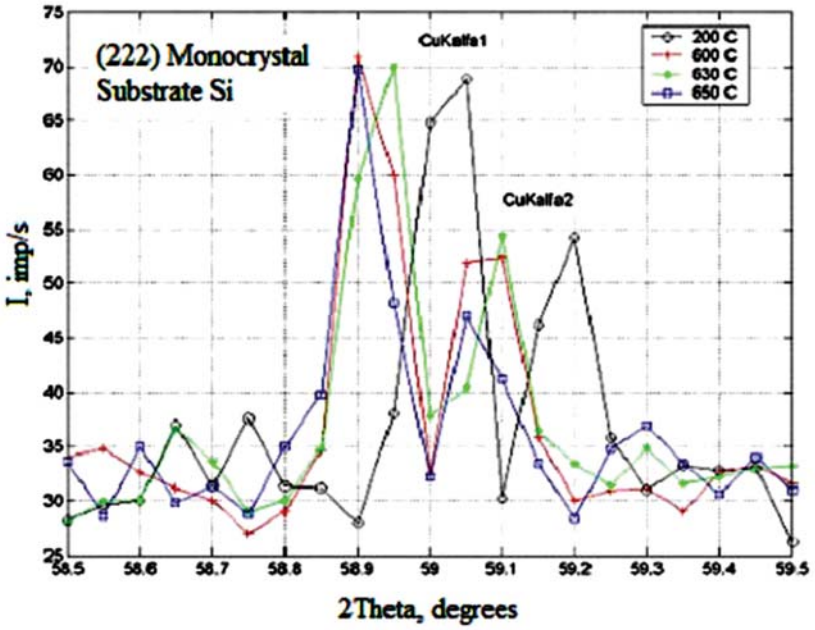

Fig. 3. The intensity of the X-ray diffraction peak (222) of silicon for the samples after sputtering and after one-minute of isochronous annealing at 600, 630, and 650 °C

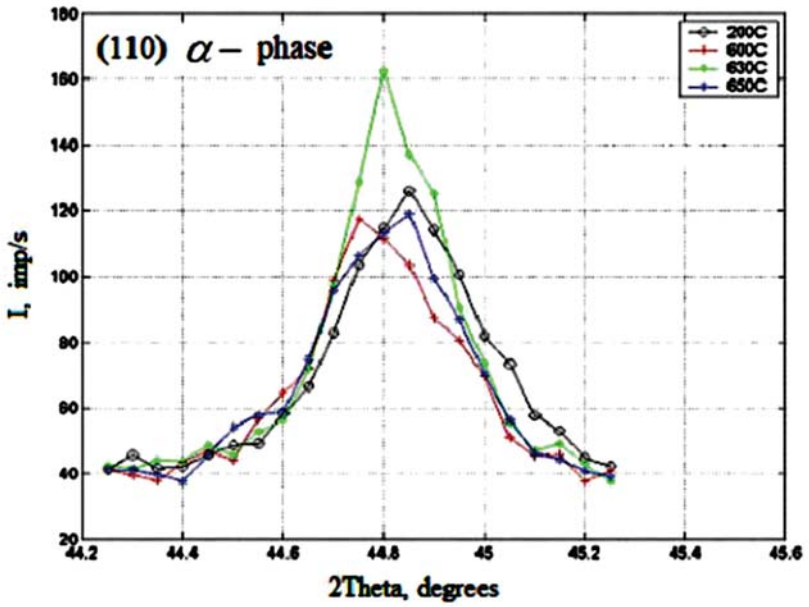

Fig. 4. The intensity of the X-ray diffraction peak (110) of the α-phase for the samples after sputtering and after one-minute of isochronous annealing at 600, 630 and 650 °C

alterations in line (111) of the γ-phase + Cu result from the difference in the concentration of the γ-phase. The intensity of this line is maximal for the sample annealed at 630 °C. This means that annealing at 630 °C yields the minimal concentration of the γ-phase and the maximal concentration of the α-phase. When the annealing time is long, the α-phase decomposes into phases α_1 and α_2, which results in higher coercivity values of the studied films [1].

The σ-phase is often present in bulk samples of Fe-Cr-Co alloys, which dramatically decreases the magnetic characteristics of these alloys. According to [21], this phase constitute up to 59% of the sample. For this phase, the most intense are the diffraction peaks (411) and (410) reflected from the σ-phase planes [24]. The diffraction

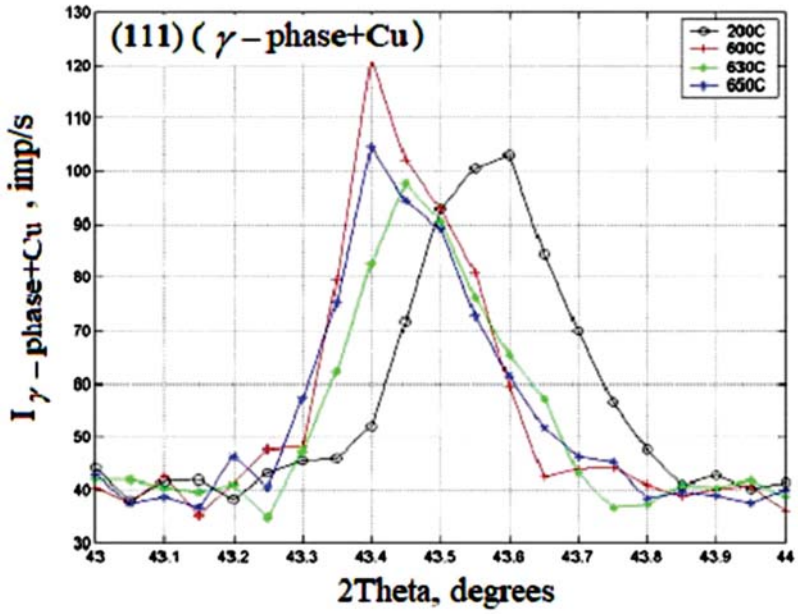

Fig. 5. The intensity of the X-ray diffraction peak (111) of the γ-phase + Cu for the samples after sputtering and after one-minute of isochronous annealing at 600, 630 and 650 °C

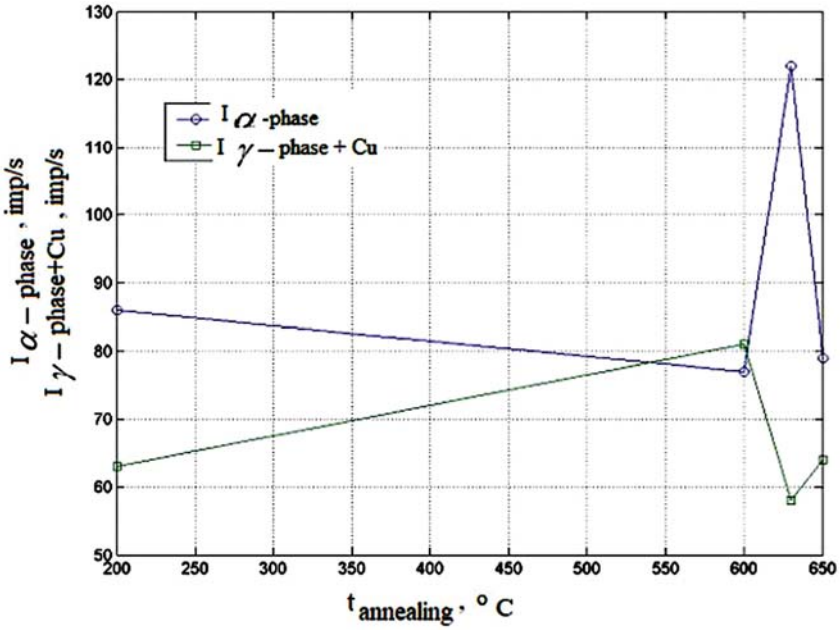

Fig. 6. Dependence of the intensity of the X-ray diffraction lines (110) of the α-phase, and (111) of the γ-phase + Cu on the annealing temperature, without taking into account the noise rate of 40 imp/s

patterns of all samples recorded before and after the annealing do not show any peaks of the σ-phase with a tetragonal β-uranium lattice. According to card No. 01-089-4790 ICDD PDF-2 [24], peaks (411) and (410) are the most intense for this structure. According to [21], the lattice parameters of the σ-phase are within the range: a = (0.8794–0.881) nm, c = (0.4552–0.458) nm. The calculations based on this data demonstrated that in CuK_α radiation the location of the maximal peaks (411) and (410) should correspond to the range of angles 2θ (46.91–47.05)° and (41.34–42.29)° respectively. Peaks of the σ-phase were observed in the same angular range in [5]. The diffraction patterns obtained in our experiment do not show these peaks, which allows us to state that the concentration of the σ-phase in the DHA layers is below the limit that can be detected using the method described in this article.

The absence of peaks at the angles 2θ = 49.5° and 2θ = 54.2° demonstrates the absence of the oxide of the main component of the films' surface layer – iron oxide Fe_2O_3.

4. Conclusions

1. Indexed diffraction patterns of the heterostructure of the permanent magnet film on a single-crystal silicon wafer with a magnetic layer of Fe-Cr-Co dispersion-hardened alloy demonstrated the presence of the α-phase whose concentration reaches its maximum during the one-minute of rapid annealing at 630 °C.

2. The study determined that the DHA layers of the composition Fe – 25wt % Cr –12 wt % Co obtained by magnetron sputtering do not contain the σ-phase. This phase is also not formed after one-minute of high vacuum annealing at 600–650 °C.

3. It was also determined that the heterostructure obtained by means of magnetron sputtering does not contain oxides of the main iron component either before, or after one-minute of high vacuum annealing at 600–650 °C.

Conflict of interests

The authors declare that they have no known competing financial interests or personal relationships that could have influenced the work reported in this paper.

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Information about the authors

Vyacheslav S. Zayonchkovskiy, PhD in Physics and Mathematics, Associate Professor, Department of Materials Science and Chemistry, Kaluga branch of Bauman Moscow State Technical University (national research university), Kaluga, Russian Federation; e-mail: zajonc4340@gmail.com. ORCID iD: https://orcid.org/0000-0002-6519-6003

Irina A. Antoshina, PhD in Physics and Mathematics, Associate Professor, Institute of Laser and Plasma Technologies, Obninsk Institute for Nuclear Power Engineering (OINPE) - a branch of the National Research Nuclear University MEPhI, Obninsk, the Kaluga region, Russian Federation; e-mail: antoshina_irina@mail.ru. ORCID iD: https://orcid.org/0000-0001-9143-2404.

Aung Kyaw Kyaw, postgraduate student, Kaluga branch of Bauman Moscow State Technical University (national research university), Kaluga, Russian Federation; ORCID iD: https://orcid.org/0000-0001-8427-3046.

Evgenij I. Isaev, PhD in Physics and Mathematics, Associate Professor, Institute of Laser and Plasma Technologies, Obninsk Institute for Nuclear Power Engineering (OINPE) - a branch of National Research Nuclear University MEPhI, Obninsk, the Kaluga region, Russian Federation; e-mail: e.isaev87@gmail.com. ORCID iD: https://orcid.org/0000-0002-1777-5342.

Igor' M. Milyaev, DSc in Physics and Mathematics, leading research fellow, Baikov Institute of Metallurgy and Materials Science, RAS, Moscow, Russian Federation.

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