



Letter to the Editor

Induced magnetic anisotropy of Co-P thin films obtained by electroless deposition

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ABSTRACT

Co_{1-x}P_x films ($x = 0.02\text{--}0.05$) deposited on cover glass by electroless plating from aqueous solutions in a continuous magnetic field with the strength of 2.5 kOe are presented. It has been found that in the *pH* range from 7.2 to 9.3, two special areas can be distinguished, they are characterized by the deposition of films with qualitatively different parameters in them. So, the change of the *pH* of working solutions from 7.2 to 8.7 causes the increase in the value of the induced magnetic anisotropy constant K_U from 2.5×10^5 erg/cm³ to 6×10^5 erg/cm³. If the value of the hydrogen index is still increasing, an uneven drop to 5×10^4 erg/cm³ is observed. In the same way, the coercivity H_C changes; at first it grows from 700 Oe to 1.5 kOe, and then it drops to Oe units. The concentration of phosphorus in the range of 7.2 to 8.7 increases linearly from 2 to 3 wt%, and in the range of 8.7 to 9.3, it increases from 3 to 5 wt%. The x-ray diffraction data show that the samples obtained at *pH* < 8.7 are characterized by the presence of the *hcp* phase of Co (α -Co). The observed reflections of films obtained at *pH* > 8.7 indicate the *fcc*-Co (β -Co) with the diffraction peak blurring: the greater the *pH* value of the samples obtained, the more the diffraction peak blurring. It is shown that the induced magnetic anisotropy in high-coercivity films relates to the modification of the Co lattice. The lattice passes from a distorted *fcc* to an *hcp* structure during the growth of crystallites in a magnetic field. The induced magnetic anisotropy appears in films that were obtained in the high *pH* field due to the ordering of magnetic ions pairs.

1. Introduction

Magnetic anisotropy refers to the dependence of the crystal energy on the direction of magnetization relative to the selected directions. It is one of the fundamental characteristics of magnetic materials and has a great influence on their properties. Its research is of both scientific and practical interest, which is related to the problems of obtaining magnetic materials with specified parameters [1–3]. In accordance with the magnitude of the coercive force, all magnetic materials can be divided into two groups: high-coercive and low-coercive with their corresponding areas of technical application [4]. At the same time, the high-coercive state of magnetic materials is mostly determined by their anisotropic parameters, while low-coercive materials have insignificant anisotropy and, as a result, high magnetic permeability.

Magnetic anisotropy can have different sources. In the case of crystallographic anisotropy, the energy dependence is determined with respect to the main crystallographic axes. Its primary reason is the spin–orbit interaction, which binds the electron spins of magnetic atoms to the crystal field. Induced anisotropy refers to the dependence of the anisotropy energy on a spontaneous magnetic moment. Its source is the ability of a substance to adapt in any way to the direction of the

magnetization vector of the external magnetic field. Induced anisotropy, in contrast to crystallographic one, can be created artificially by applying a magnetic field both in the process of obtaining samples and during additional thermal annealing. The physical nature of induced anisotropy is not always unambiguous, this is due to the possible influence of various causes of directional ordering of any properties that are not related to magnetization, for example, texture, or the presence of mechanical stresses [5]. Therefore, to find out the nature of anisotropy that occurs in a magnetic field, detailed studies of the features of the sample production technology are required.

This paper presents the results of a study of induced magnetic anisotropy of Co thin films with P additives obtained by electroless deposition in magnetic field. Phosphorus is introduced into cobalt films due to the peculiarities of the chemical deposition technique, namely, due to using Sodium hypophosphite as a reducing agent. Such films are widely used in a variety of microelectronic devices, which is largely due to the good magnetic characteristics of Co. Due to this, Co-P films have a wide range of practical uses for creating magnetic memory [6,7], as well as various control or reading devices [8,9]. The uniqueness of the electroless plating technology is that it allows to obtain Co-P films both highly anisotropic and low anisotropic, which significantly differ in the

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magnitude of the coercivity. This is achieved in a fairly simple way by introducing various additives into the solution that change the pH value [10].

The purpose of this work is to know the physical mechanisms that determine the anisotropic properties of Co-P films obtained by electroless deposition in magnetic field at different alkalinity of working solutions.

In the frames of this work a complex investigation of induced anisotropy in such films depending on the solution acidity has been carried out and physical mechanisms of its appearance at different values of the solution acidity have been determined. The induced anisotropy of the films obtained in the pH range from 7.1 to 8.7 has been shown to be related to the modification of the Co lattice from the distorted fcc into the hcp structure in the film growth process in the magnetic field. Induced anisotropy in the films obtained with the pH values above 8.7 appears due to the directed ordering of Co ion pairs with respect to the orientation of magnetization.

2. The technique

The study samples were obtained by the method of chemical reduction of Co from aqueous solutions of the composition of Cobalt (II) sulfate $\text{CoSO}_4 \cdot 7\text{H}_2\text{O}$ – 10 g/l, Sodium hypophosphite $\text{NaH}_2\text{PO}_2 \cdot \text{H}_2\text{O}$ – 15 g/l, Trisodium citrate $\text{Na}_3\text{C}_6\text{H}_5\text{O}_7$ – 20 g/l at different pH values at the range of 7.2 to 9.3 [11]. The pH value was set by adding 1 M of Sodium carbonate Na_2CO_3 solution and was determined at room temperature by means of a pH -150 MH device. Deposition was performed on a cover glass substrate at a temperature close to 100°C in a constant magnetic field $H = 2.5$ kOe applied in the film plane. Preparation of the substrates consisted of pre-annealing at 400°C , sensitization in SnCl_2 solution, and activation in PdCl_2 solution. The thickness of the films was ~ 30 nm. The composition and thickness of the films were determined by means of x-ray spectral analysis.

Diffractograms were taken by a Bruker DUO diffractometer (Mo $K\alpha$ radiation, graphite monochromator, 0.35 mm collimator, the CCD detector with 512×512 resolution) according to the method described in [12]. The polycrystalline Si – SRM640 sample was used as an external reference.

During the experiment, the detector was located at 40 mm from the sample and was rotated at an angle of $2\theta_D = 40^\circ$. The study sample was attached by a special adapter and centered by a standard video camera. The rotation of the sample relative to the primary beam was set by changing the angle φ , it was always equal to 30° . The change to the standard type of diffractogram (integration) was performed in the DIOPTAS program [13].

The structure and the local element composition of the obtained samples were studied with a JEOL JEM-2100 transmission electron microscope (TEM), equipped with an energy-dispersive spectrometer Oxford Inca x-sight, at the accelerating voltage of 200 kV.

The induced magnetic anisotropy constant K_U was determined by a conventional rotational anisometer [2] in a magnetic field $H = 10$ kOe, located in the plane of the film, at room temperature. The general layout of the measuring units and location of the sample are shown in Fig. 1.

3. The experiment

Fig. 2 shows the dependence of the uniaxial anisotropy constant K_U and the coercivity H_C on pH . The pH changes from 7.2 to ~ 8.7 lead to an increase in K_U from 2.5×10^5 erg/cm 3 to 4×10^5 erg/cm 3 with the maximum value $K_U = 6 \times 10^5$ erg/cm 3 at $pH = 8.5$; when films are deposited from solutions with $pH > 8.7$, the K_U value drops abruptly to 5×10^4 erg/cm 3 . Similar changes are observed in H_C value, it varies from 1.5 kOe to Oe units, it allows to break the pH scale into two areas, one of which ($pH < 8.7$) is characterized by the high-coercivity (high-anisotropy) samples deposition in it and the second area ($pH > 8.7$) has the low-coercivity (low-anisotropy) samples deposition.

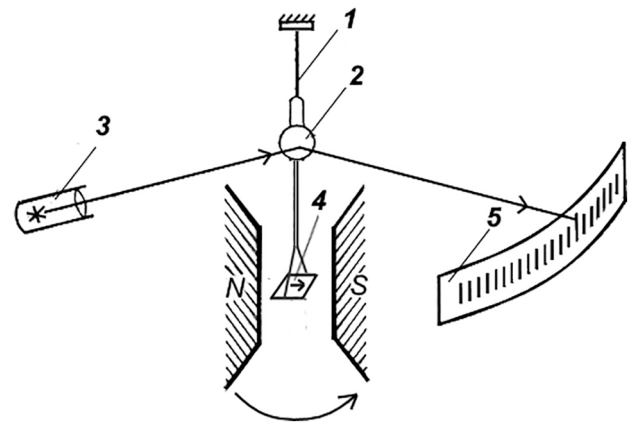


Fig. 1. Structural scheme of the rotational anisometer. 1 is the elastic suspension, 2 is the mirror, 3 is the light source, 4 is the sample under study, 5 is the measuring scale. The arrow shows the direction of rotation of the electromagnet.

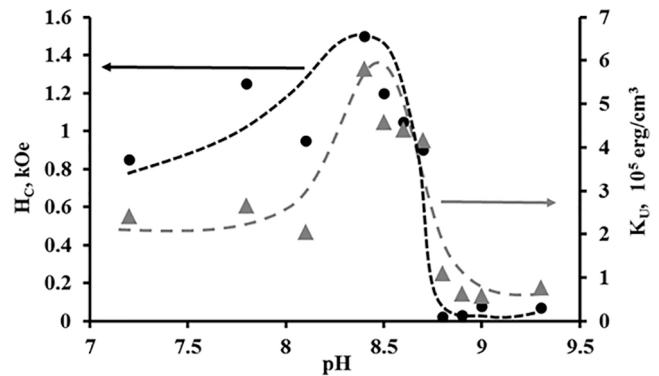


Fig. 2. The dependence of K_U and H_C on the alkalinity of the solutions.

The values K_U and H_C in the pH range of about 8,1 are below the curves, describing the tendency of increase in these values to $pH \sim 8.7$. A possible reason for the observed decrease of these values is the formation of the additional non-ferromagnetic compound CoP_3 . As it is shown below, the presence of this phase is determined with the help of the electron diffraction of the cross section. It is worth noting that the quantitative ratio between the observed Co phases requires further investigation.

In addition to the difference in absolute values, the difference between the anisotropy constants for films obtained at different pH is shown in their changes over time.

The K_U value of high-anisotropic films obtained at low pH does not change its value during a long annealing time in a magnetic field with the strength of 2 kOe which is orthogonal to the light axis at 300°C .

A completely different behavior is observed in the induced anisotropy of low-anisotropic films obtained at high pH . The typical H_K anisotropy field variation from the annealing time of such films is shown in Fig. 3. The magnetic field strength is 2 kOe is applied orthogonal to the easy magnetization axis created in the film cooling. The film was annealed at 100°C .

Over time the H_K value decreases up to zero ones, then it changes the sign and increases in absolute value during next annealing.

The sign change of H_K means that when annealing the film in the magnetic field, in the indicated configuration the initial direction of the easy magnetization becomes the direction of hard magnetization.

As it has been previously established [14], the Co-P films obtained at different alkalinity of working solutions sufficiently differ from the size of Co crystallites with a slight change in the concentration of

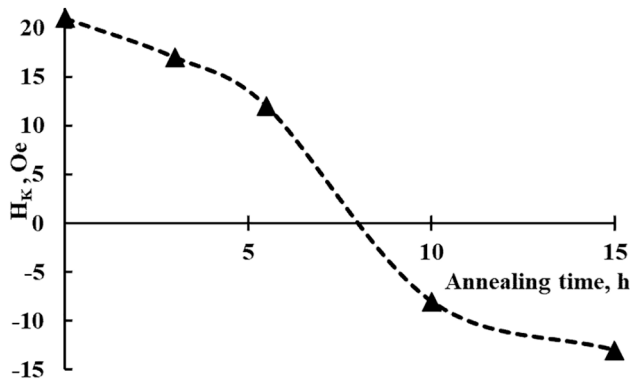


Fig. 3. The anisotropy field change of a low-anisotropic 30 nm thick $Co_{0.95}P_{0.05}$ film obtained at $pH = 9.3$ from the annealing time at $100^\circ C$.

phosphorus, the dependence of which on pH is shown in Fig. 4.

The average size of crystallites depends on the alkalinity of the solution and monotonously decreases with its growth. At $pH = 7.2$, the average size of the crystallites in the film plane is 50–70 nm and decreases to 5–10 nm at $pH > 8.7$.

According to the x-ray diffraction analysis data, the samples obtained at low $pH < 8.7$ are characterized by the *hcp* phase of Co. It is confirmed by x-ray diffraction data. A typical diffractogram of such samples is shown in Fig. 5, a. Fig. 5, b presents a low-anisotropic film diffractogram, which was obtained at $pH = 8.9$; the observed reflections demonstrate the *fcc* phase of Co.

4. The discussion of results

The differences in the values of the induced anisotropy constants in both types of films, as well as in the nature of their changes over time, indicate that the magnetic anisotropy in films obtained at different solutions alkalinity, does not have the same nature.

To determine the nature of induced anisotropy, it is necessary to consider all possible mechanisms of its occurrence. They can be divided into two groups for polycrystalline materials and magnetic films in particular. The first group includes mechanisms caused by the directed growth of crystallites in a magnetic field. It is well reflected in the morphology of the films surface. This mechanism occurs, for example, in Co films obtained by the method of angle evaporation [15,16], as well as during electrolytic deposition of Co-P films in a magnetic field [17]. In the latter case, the appearance of induced anisotropy is explained by Lorentz forces. They act on moving ions in the solution from the magnetic field, as a result, the directed growth of Co crystallites occurs.

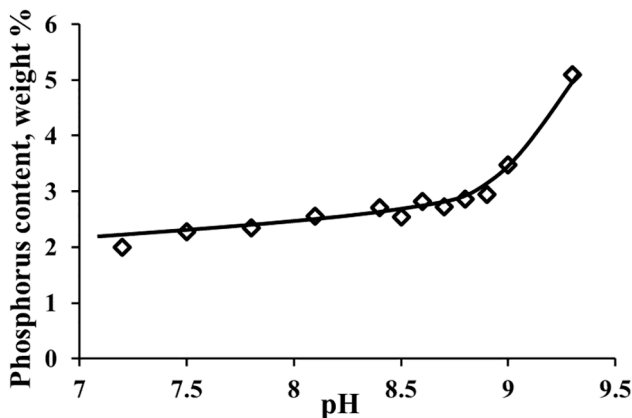


Fig. 4. Phosphorus concentration changes in films depending on the alkalinity of solutions.

As it has been shown before, anisotropy of crystallite growth in Co-P films obtained by electroless deposition in a magnetic field is absent. It can be seen from the analysis of electron microscopic images of the surface in the absence and with the superposition of a magnetic field; the superposition of a magnetic field does not change the size and shape of the crystallites [18].

The differences in the film growth in a magnetic field during electroless and electrolytic ions deposition on the substrate are related to the features inherent in both deposition methods. During electroless deposition, in contrast to electrolytic one, the ion current is only in the area of the solution-substrate contact, and its value is very small [19], and therefore the influence of the magnetic field is insufficient.

The second group of reasons that can cause induced anisotropy includes the mechanisms related to the immediate environment of the magnetic ion and their redistribution along the nodes of the crystal lattice in the magnetic field. At the same time, it is necessary to consider the mechanisms that are responsible for the environment to be rebuilt in the area of small and large pH values separately.

4.1. The nature of the anisotropy films obtained in the low pH area

The large value of induced anisotropy and its non-relaxation character in Co-P films obtained at low pH indicate that the most likely mechanism of its formation is the same as in the massive samples of polycrystalline Co. Induced anisotropy in a massive Co is observed after its heating in a magnetic field at the temperature above the phase change from α to β -phase ($420^\circ C$) and to the subsequent reverse change from β to α -phase during cooling [20]. The anisotropy is explained by the magnetic field that contributes to the predominant orientation of the Co *hcp* planes, which are formed from randomly oriented *fcc* planes of type (1 1 1) as a result of phase changes.

The energy of the induced anisotropy of the *hcp* Co, which occurs in this case, is determined by the expression [21]:

$$W_m = (0.434K_1 + 0.299K_2)\cos^2\theta + 0.293\cos^4\theta \quad (1)$$

where θ is the angle between the direction of the magnetic field and the light magnetization axis; K_1 and K_2 are the 1st and 2nd anisotropy constants of the hexagonal Co.

From Eq. (1), we can find that the value of K_U for polycrystalline *hcp* Co should be $2.82 \cdot 10^6$ erg/cm³, but we do not take into account the second term and consider K_1 and K_2 values at room temperature to be equal to 4.53×10^6 and 1.44×10^6 erg/cm³ respectively. As it has been noted above, in Co-P films, the K_U value increases from 2.5×10^5 erg/cm³ to 6×10^5 erg/cm³, or from 8.8 to 21% of the expected value, with pH growth from 7.2 to 8.5. K_U for polycrystalline *hcp* Co after annealing in a magnetic field is 2×10^5 erg/cm³ or 7% [20].

The magnetic anisotropy results in the fact that the sample in a magnetic field is influenced by a rotational moment, the magnitude and direction of which is determined by the ratio:

$$L(\theta) = -\frac{\partial W}{\partial \theta} \quad (2)$$

Or together with (1):

$$L(\theta) = (0.434K_1 + 0.592K_2)\sin 2\theta + 0.147\sin 4\theta \quad (3)$$

Fig. 6 shows the experimental dependence $L(\theta)$ of the $Co_{0.98}P_{0.02}$ film, as well as the theoretical curve which is determined by the formula (3) for the specified values of K_1 and K_2 . The experimental curve is in a satisfactory agreement with the theoretical one. Asymmetry of the angular dependence $L(\theta)$, according to (3), is caused by the contribution of the summand determined by the value of K_2 into the energy of the induced anisotropy of *hcp* Co.

In order to determine the reasons of phase changes of Co in the studied films, it is necessary to consider the peculiarities of their growth in the low pH field. As it has been shown before [22], the formation of

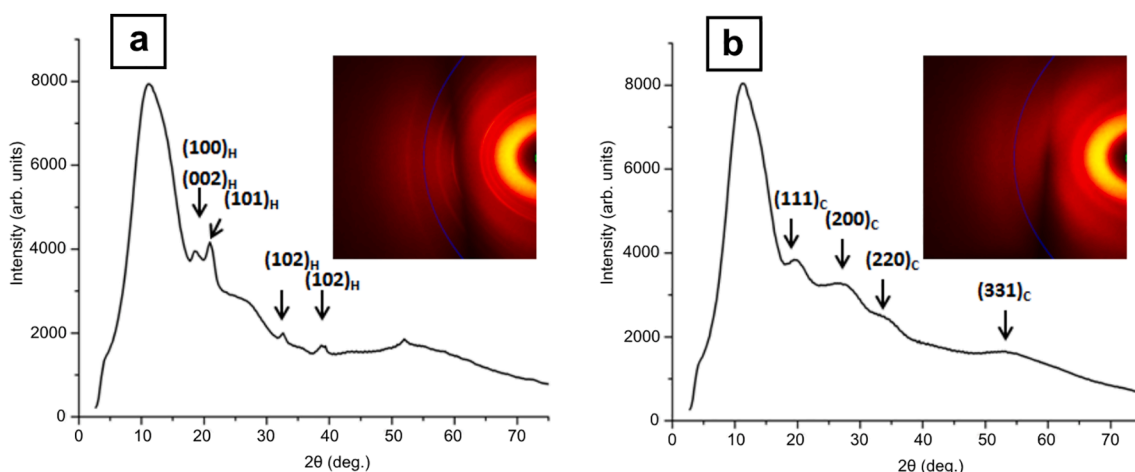


Fig. 5. a - The diffractogram of a high-anisotropic sample $\text{Co}_{0.973}\text{-P}_{0.027}$ (at $\text{pH} = 8.4$) with reflections from the crystallographic planes of the *hcp*-Co; b - the diffractogram of a low-anisotropic sample $\text{Co}_{0.971}\text{-P}_{0.029}$ (at $\text{pH} = 8.8$) with the reflections from the plane (111) of the *fcc*-Co.

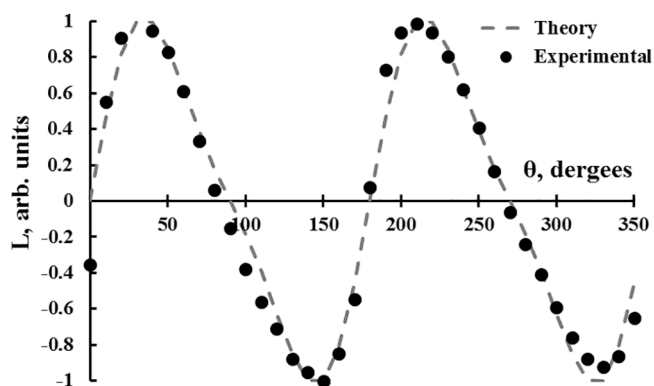


Fig. 6. The angular dependence of the rotational moment of the $\text{Co}_{0.98}\text{-P}_{0.02}$ film obtained at $\text{pH} = 7.2$.

the film during electroless deposition occurs according to the mechanism of nucleation with the initial formation of crystal phase islands and their subsequent growth. Thus, the structure of the film is varied from an island to a solid one, and due to the close coupling between the phase state of Co and the size of its particles [23,24], the crystal structure of the grains will change. In order to confirm the interconnection between the crystallite sizes and phase changes of Co in the films, Fig. 7a and 7b show the TEM image of cross-section of the $\text{Co}_{0.974}\text{-P}_{0.026}$ film and corresponding selected area electron diffraction (SAED) pattern, respectively. The film was deposited at $\text{pH} = 8.0$. The SAED pattern was obtained

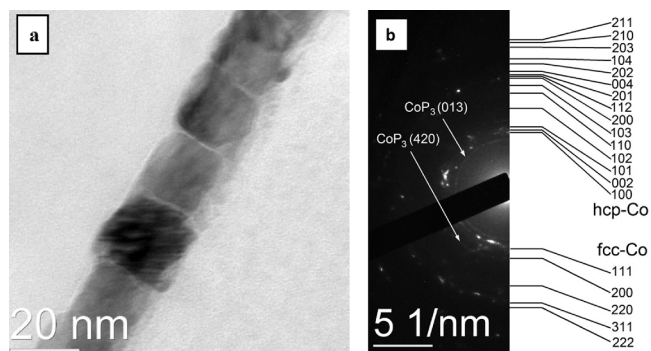


Fig. 7. The TEM image of the cross section of the $\text{Co}_{0.974}\text{-P}_{0.026}$ film ($\text{pH} = 8.0$) (a) and SAED pattern (b).

from the area with a diameter of 150 nm, i.e. the sample volume used to obtain the SAED pattern was 10^5 nm^3 . Interpretation of diffraction reflections shows that the film contains a mixture of Co phases: α -Co (*hcp*-Co, $\text{P6}_3/\text{mmc}$ space group, lattice constants: $a = 2.507 \text{ \AA}$, $c = 4.069 \text{ \AA}$, PDF4 + card # 04-001-3273) and β -Co (*fcc*-Co, Fm-3m space group, lattice constant: $a = 3.545 \text{ \AA}$, PDF4 + card # 00-015-0806). The film also contained some CoP_3 phase (Im-3 space group, lattice constant: $a = 7.707 \text{ \AA}$, PDF4 + card no. 04-004-4318). Point reflexes (see Fig. 7b) correspond to the α -Co and β -Co crystallites, and ring reflexes correspond to the CoP_3 phase. The evaluation of the crystallite sizes, which were assessed by means of the method presented in [25], shows that the size of the α -Co crystallites is in the range from 10 to 20 nm, β -Co - from 5 to 10 nm, and the CoP_3 size is 3–5 nm. It should be noted that reflections from CoP_3 crystallites were not detected by x-ray diffraction, due to their small size.

Thus, the specific growth of Co-P films obtained at low pH values determines their anisotropic properties. The formation of small Co particles on the substrate in the β -phase is typical at the initial stage. These particles are the nuclei of large crystallites, where the initial Co structure changes into the α -phase due to dimensional effects. As it is shown in papers [23,24], such changes are associated with the thermodynamic stability of the Co structure, since the β -phase is thermodynamically stable in the area of small sizes, and the α -phase is stable with increasing particle sizes.

The films are isotropic when there is no magnetic field, since they are formed by crystallites with a random direction of the crystallographic axes. The magnetic field distinguishes the directions that are closest to the orientation of the field in all possible directions of *hcp*-lattice growth. They are defined by the c axis. As a result, there is a uniaxial magnetic anisotropy.

4.2. The nature of the anisotropy of the films obtained in the high pH area

An increase in the solution alkalinity results in a size decrease of crystallites. It causes the changes in the physical properties of Co films, for example, its adhesion to the substrate [26]. As it follows from Fig. 3, when the Co film obtained in the area of high $\text{pH} > 8.9$ is annealed in a magnetic field applied perpendicular to the initial axis of easy magnetization, the K_U decreases to zero values and then the sign changes. It means that a new light axis is created, which is set along the direction of the magnetic field applied during the film annealing. The relaxation character in the anisotropy field change indicates that the cause of the induced anisotropy in the films is the directed ordering processes that occur in a diffusive manner. Temperature changes in the relaxation time of induced anisotropy K_U are subject to the Arrhenius activation law:

$$\tau = \tau_0 \cdot \exp(E_A/kT) \quad (4)$$

where E_A is the activation energy, k is the Boltzmann constant, and τ_0 is the constant.

In the $\text{Co}_{0.965}\text{-P}_{0.035}$ film (obtained at $pH = 9.0$) at room temperature, the value is $\tau \sim 200$ days, at $T = 100$ °C $\tau \sim 3$ h. Thus, it can be established that $E_A \sim 1$ eV, $\tau_0 \sim 10^{-9}$ s.

The found values of activation energy and τ_0 are typical for atom diffusion when there are some point defects in the lattice. They can be vacancies or impurities. The vacancies provide the redistribution of magnetic ions pairs. Their energy depends on their orientation relative to the direction of the spontaneous magnetic moment and is well explained by the Neel –Taniguchi pair ordering model [27,28].

5. Conclusion

The changes in the induced anisotropy in Co-P films obtained by electroless deposition in magnetic field are related to structural changes determined by the pH value of solutions. Abrupt differences between K_U values and its relaxation times indicate the difference in the physical mechanisms that determine the occurrence of induced anisotropy of Co-P films in the area of low and high pH values. The induced anisotropy in the films obtained at low pH is associated with the modification of the Co lattice. It transforms from a distorted *fcc* to the *hcp* structure when the Co crystallites grow during electroless film deposition in a magnetic field.

The behavior of induced anisotropy in films obtained at high pH indicates its diffusive nature, which is caused by the directional ordering of the Co ion pairs relative to the orientation of the magnetization.

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Declaration of Competing Interest

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

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