
ELECTRICAL
AND MAGNETIC PROPERTIES

Analysis of Phase Composition of Co–P Alloy Powders Using Magnetometric Data

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Abstract—A new method for the quantitative estimate of the phase composition of a multiphase ferromagnetic alloy is suggested that is based on measuring the temperature and field dependences of the magnetization. The phase composition of ultrafine Co–P powders is characterized. Phase diagrams of nonequilibrium Co–P solid solutions are constructed on particle size, i.e., the phosphorus concentration coordinates.

Keywords: phase analysis, multiphase alloys, characterization of materials, magnetic materials

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INTRODUCTION

The magnetic phase analysis is one of important methods used to analyze heterophase ferromagnetic alloys [1–6]. In this case, the phase composition is determined using the $M(T)$ dependence. The Curie temperature and saturation magnetization M_s of a phase are used as characteristics of the phase. The method is based on the following assumption. According to the additivity law, the high-temperature part of $M(T)$ can be separated into partial curves as follows:

$$M(T) = \sum p_i M_i(T), \quad (1)$$

where p_i and $M_i(T)$ is the volume fraction and magnetization of the i th phase, respectively.

In the case of powders of nonequilibrium alloys, the traditional phase analysis cannot be used, since nonreversible transformations start at temperatures substantially below the Curie temperature. In the present study, a modified magnetic phase analysis, which allows us to overcome the aforementioned difficulty, is demonstrated by an example of the investigation of powders of nonequilibrium amorphous and nanocrystalline Co–P alloys.

Bulk pure cobalt is known to have two modifications; these are α Co, which has a hexagonal close-packed (hcp) lattice, and β Co, which has a face-centered cubic (fcc) lattice, which are realized below and above 427°C, respectively. The state of equilibrium Co–P alloys with phosphorus contents of below 33 at % can be characterized as a mechanical mixture of Co and Co₂P phases [7]. It was shown [8–20] that, in

Co–P alloys prepared by either chemical or electrochemical deposition, phases, such as nonequilibrium solid solutions with the hcp and fcc structures and Co–P amorphous alloys, can be obtained at room temperature.

Studies of the atomic structure of Co–P alloys prepared in the form of coatings (30 μ m thick) by chemical and electrochemical deposition showed that, as the phosphorus concentration varies within 0–15 at %, two structural transformations occur. In particular, the hcp structure is realized at a phosphorus content of no less than 5 at % in the Co–P coatings; the fcc structure is obtained at a phosphorus content of 5–9 at %. The alloy with more than 9 at % phosphorus has an amorphous structure [9, 10].

The structure and magnetic properties of ultrafine Co–P powders prepared by chemical deposition were studied previously in [8, 11, 12]. X-ray diffraction data on the structure were found to be ambiguous. In particular, X-ray diffraction patterns of powders with phosphorus contents of less than 9 at % indicate reflections of the hcp phase; at higher phosphorus contents (more than 12 at %), only a halo typical of the amorphous phase is observed. X-ray diffraction patterns of alloys with 9–12 at % phosphorus exhibit highly broadened reflections of the hcp phase against the background of the amorphous-phase halo. This type of X-ray diffraction pattern results from the nanocrystalline state of the powders and do not allow us to judge their phase composition. The nuclear magnetic resonance spectra of the powders showed that powder particles also contain Co atoms, which have

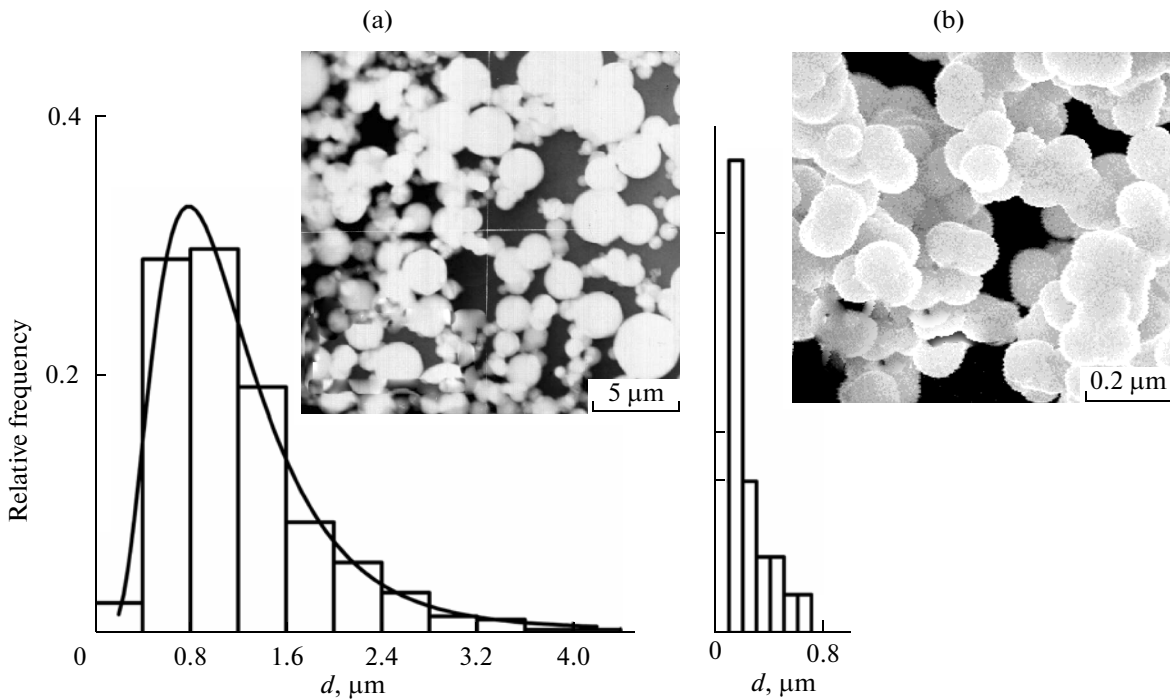


Fig. 1. Particle-size distribution histogram shown by an example of the $\text{Co}_{88}\text{P}_{12}$ powder: (a) initial and (b) fraction with a particle size of 0.1 μm .

the fcc nearest-neighbor environment [13, 21]. Thus, the phase composition of powders with 9–12 at % phosphorus can be qualitatively characterized as α Co–P (hcp) + β Co–P (fcc) + amorphous Co–P.

The aim of the present study is to use magnetic phase analysis methods to quantitatively estimate the volume fractions of phases that are present in ultrafine Co–P powders, in which the phosphorus content varies in the range of 0–18 at % and the particle size varies in the range of 0.1–3 μm .

EXPERIMENTAL

Co–P powders with a particles size of 0.1–3 μm were prepared by chemical deposition using aqueous solutions of Co salts and sodium hypophosphite (NaH_2PO_2) as the reducing agent. The phosphorus concentrations in the Co–P alloys were determined by chemical analysis. According to the average particle size, powders with each of the phosphorus concentrations were classified into six fractions (0.1, 0.4, 0.6, 0.8, 2, and 3 μm). The particle sizes were determined using electron-microscopic images taken with a JEM-100C electron microscope (the average particle size and its more probable magnitude and dispersion were determined using a plotted histogram). Magnetic measurements were performed at temperatures of 78–300 K in magnetic fields of 0–14 kOe using a vibrating-sample magnetometer.

The shape of powder particles is close to spherical. The particle-size distribution for the $\text{Co}_{88}\text{P}_{12}$ powder is shown in Fig. 1. The particle-size distribution obeys the lognormal law $f_X(x; \mu, \sigma) = \frac{1}{x\sigma\sqrt{2\pi}} \exp\left(-\frac{(\ln x - \ln x_0)^2}{2\sigma^2}\right)$ with a standard deviation $\sigma = 0.53$ and $x_0 = 1.04 \mu\text{m}$.

RESULTS AND DISCUSSION

The measured temperature dependences of magnetization showed that, at low temperatures, the saturation magnetization M_S of ultrafine Co–P powders obeys the Bloch's law, i.e., is proportional to $T^{3/2}$, which is typical of ferromagnets as follows (Fig. 2):

$$M(T) = M_0(1 - BT^{3/2}). \quad (2)$$

Thus, the magnetization measured at different temperatures for each of the fractions (particle size) and each phosphorus concentration in the powder under study allows us to determine the Bloch constant B (see table) as a function of the phosphorus concentration and particle size when using the approximation of experimental data by Eq. (2). The error of calculating the Bloch constant was 1%.

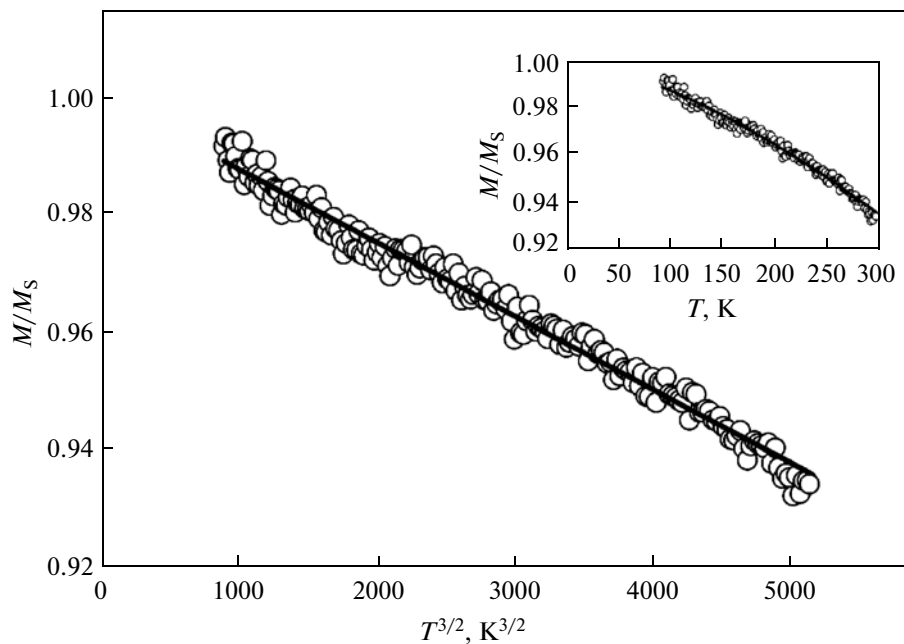


Fig. 2. Temperature dependence of saturation magnetization M_s of ultrafine Co–P powders.

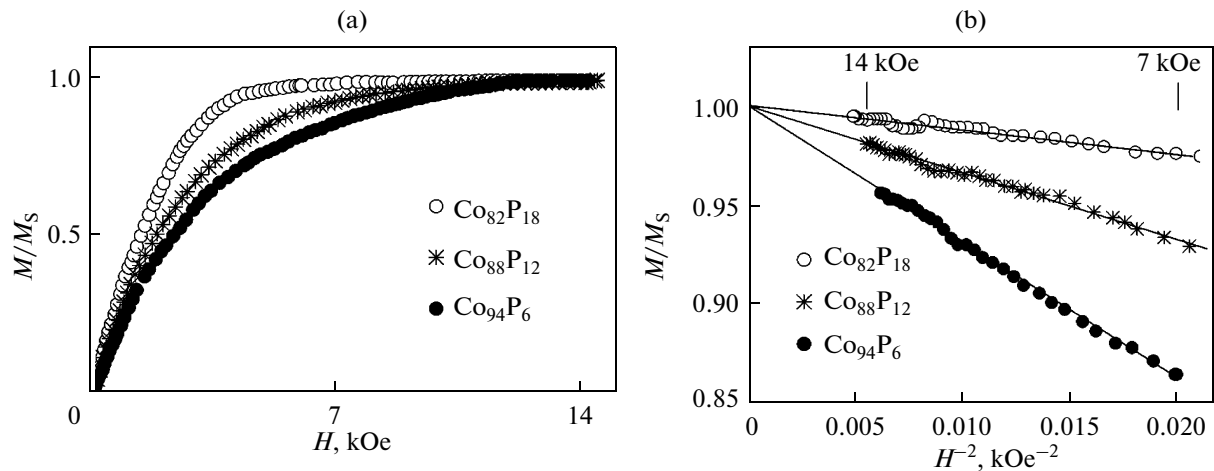


Fig. 3. (a, b) Magnetization going to saturation for ultrafine Co–P powders.

In approaching saturation, the magnetization of ultrafine Co–P powders obeys Akulov's law as follows (Fig. 3):

$$M(H) = M_s \left(1 - (aH_a/H)^2 \right). \quad (3)$$

This allowed us to calculate the saturation magnetization M_s and root-mean-square fluctuation of the local magnetic anisotropy field aH_a for ultrafine powders. Here, $H_a = 2K/M_s$; K is the local magnetic anisotropy energy of Co–P particles and a is the symmetry factor, which is equal to $(1/15)^{1/2}$ and $(2/105)^{1/2}$ for the uniaxial (hcp) and cubic (fcc) magnetic anisotropy, respectively [10]. The error of calculating aH_a is 1%.

The essence of the quantitative phase analysis is as follows. The magnetization of heterophase alloys, which is determined by magnetometry, is averaged additively for both the temperature and field dependences of saturation magnetization. This leads to the effective averaging of the measured B (in Eq. (2)) and aH_a (in Eq. (3)) magnitudes as follows:

$$\begin{aligned} M(T) &= M_1(T)x_{\text{hcp}} + M_2(T)x_{\text{fcc}} + M_3(T)x_A \\ &= M_{0,\text{eff}} \left(1 - B_{\text{eff}} T^{3/2} \right); \end{aligned} \quad (4)$$

$$\begin{aligned} M(H) &= M_1(H)x_{\text{hcp}} + M_2(H)x_{\text{fcc}} + M_3(H)x_A \\ &= M_{0,\text{eff}} \left(1 - (aH_{a,\text{eff}}/H)^2 \right), \end{aligned} \quad (5)$$

Bloch constant, rms fluctuation of local magnetic anisotropy field aH_a in Co–P alloy powders, and volume fractions x_{hcp} , x_{fcc} , and x_A calculated by system of equations (6)

Average powder-particle size d , μm	Phosphorus concentration, at %	Local anisotropy field aH_a , kOe	Bloch constant B , $\times 10^{-6} \text{ K}^{-3/2}$	x_{hcp}	x_{fcc}	x_A
3	6	2.6	5.4	0.48	0.32	0.20
	12	1.77	11.2	0.19	0.29	0.52
	15	1.25	13.2	0.06	0.30	0.64
	18	1.23	16	0.05	0.16	0.79
2	6	2.75	4.8	0.65	0.19	0.16
	10	3.01	6.9	0.17	0.53	0.30
	12	1.67	9.9	0.24	0.31	0.45
	15	1.95	15.5	0.06	0.18	0.76
0.8	6	3.25	2.28	0.77	0.21	0.02
	10	1.52	7.1	0.14	0.55	0.31
	12	1.84	7.2	0.22	0.47	0.31
	15	1.11	16	0.03	0.18	0.79
	18	1.12	18.3	0.02	0.07	0.91
0.6	6	3.46	2.04	0.87	0.12	0.01
	10	1.42	8.7	0.11	0.49	0.40
	12	1.92	5.4	0.25	0.54	0.21
	15	1.01	16.8	0.01	0.16	0.83
	18	1.06	18.4	0.01	0.07	0.92
0.4	6	2.3	2.3	0.94	0.04	0.02
	12	3.6	5.2	0.54	0.27	0.19
	15	2.77	17.6	0.05	0.08	0.87
	18	1.23	19	0.03	0.02	0.95
0.1	6	2.86	2.14	0.59	0.38	0.03
	10	1.55	8.4	0.14	0.48	0.38
	12	2.08	5.2	0.30	0.50	0.20
	15	1.2	18.5	0.04	0.04	0.92
	18	1.14	19	0.02	0.03	0.95

where x_i is the volume fractions of corresponding phases.

Hence (Eqs. (4) and (5)), the system of equations can be written as follows:

$$\begin{cases} x_{\text{hcp}} + x_{\text{fcc}} + x_A = 1 \\ B_1 x_{\text{hcp}} + B_2 x_{\text{fcc}} + B_3 x_A = B_{\text{eff}} \\ (a_1 H_{a1})^2 x_{\text{hcp}} + (a_2 H_{a2})^2 x_{\text{fcc}} \\ + (a_3 H_{a3})^2 x_A = (a H_{a,\text{eff}})^2 \end{cases} \quad (6)$$

Thus, the averaged magnitudes B_{eff} and $aH_{a,\text{eff}}$ determined from magnetometric measurements are found to be in right parts of the second and third equations of system (6). The solution of the system of equations for x_{hcp} , x_{fcc} , and x_A allows us to determine the volume fractions of the hcp, fcc, and amorphous phase in the Co–P powders under study. This estimation can be performed when the parameters of pure phases,

such as coefficients $a_1 H_{a1}$, $a_2 H_{a2}$, $a_3 H_{a3}$, B_1 , B_2 , and B_3 , are available.

The selection was performed as follows. Studies of Co–P coatings showed that single-phase Co–P solid solutions (hcp and fcc) are characterized by certain magnitudes of the Bloch constant B_i [9] and local anisotropy field $a_i H_{ai}$ [10], which are almost independent of the composition.

For calculations, the coefficients $a_1 H_{a1}$, $a_3 H_{a3}$, B_1 , and B_3 were taken to be equal to those for powders, which correspond to boundary phosphorus concentrations of the composition range under study (6 at % for $a_1 H_{a1}$ and B_1 and 18 at % for $a_3 H_{a3}$ and B_3). The causes for this choice are (1) the existence of clear diffraction pattern for samples with the 6% phosphorus concentration, which corresponds only to the hcp phase, and (2) the existence of only the amorphous-phase halo for the powders with a phosphorus concentration of 18 at %. The reference values of coefficients in Eq. (6), which were chosen in this manner, agree

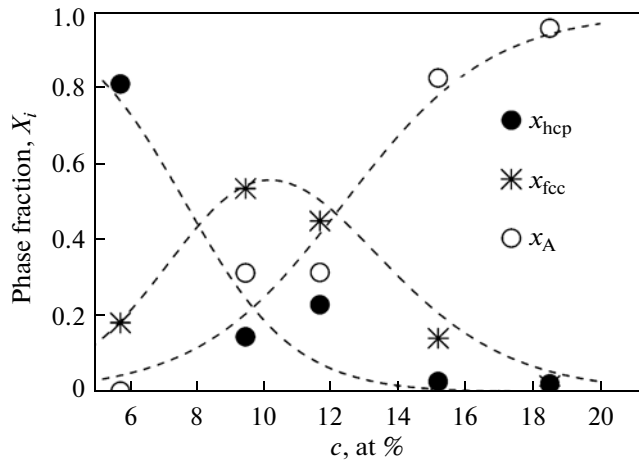


Fig. 4. Dependences of phase fractions on the phosphorus concentration $c(\text{P})$ (particles size is $0.8 \mu\text{m}$).

adequately with the analogous parameters aH_a and B for the hcp and amorphous phases, which were measured for Co–P coatings [9, 10]. The chosen a_2H_{a2} and B_2 magnitudes were proportional to the analogous parameters for single-phase Co–P coatings with the fcc structure [9, 10]. The magnitudes for the hcp phase of the powders (the phosphorus concentration is $c < 6$ at %) are $B_1 = 2 \times 10^{-6} \text{ K}^{-3/2}$, $a_1H_{a1} = 3.7 \text{ kOe}$; for powders with the fcc structure (the phosphorus concentration is $6 < c < 8$ at %), $B_2 = 1.17 \times 10^{-6} \text{ K}^{-3/2}$ and $a_2H_{a2} = 0.41 \text{ kOe}$. For amorphous powders (the phosphorus concentration is $c > 15$ at %), $B_3 = 20 \times 10^{-6} \text{ K}^{-3/2}$ and $a_3H_{a3} = 1 \text{ kOe}$.

The choice of the B_3 and a_3H_{a3} magnitudes for the amorphous phase, which are taken to be independent of the phosphorus concentration, is due to the following considerations. The Bloch constant for powders that correspond to the transition range of phosphorus concentrations of 15–18 at % is almost unchanged

(see table). This manifests itself clearly for powders with small particle sizes ($0.1\text{--}0.6 \mu\text{m}$), which are likely to be characterized by high compositional uniformity. The magnitude of the Bloch constant for ferromagnets is directly determined by the constant of exchange interactions, which is in turn related to the short-range order of atomic structure. Because of this, the independence of the Bloch constant for powders with a phosphorus concentration of 15–18 at % (transition range of concentrations) is explained by the formation of a two-phase mixture that consists of the Co–P amorphous solid solution of the unchanged composition and crystalline phosphide Co_2P [7].

Calculations of volume fractions x_{hcp} , x_{fcc} , and x_A by Eq. (6) showed that powders with the same particle size exhibit the qualitatively similar character of changes in their phase composition with changing phosphorus concentration. At a phosphorus content of less than 6%, the content of the hcp phase is close to 100%. The amorphous phase is the basic one in powders with phosphorus contents of more than 12 at %. At intermediate phosphorus contents in the powders, the heterophase state is realized, which is characterized by comparable fractions of the hcp, fcc, and amorphous phases (see table). The computational error of the fractions x_i of the phases is 10^{-2} to 10^{-3} (2–3%).

The dependences of volume fractions of hcp and fcc phases X_i on the phosphorus concentration c were approximated by the logistic function $X_i(c)$ as follows (dashed lines in Fig. 4):

$$X_i(c) = (1 + \exp(k_i(c - c_0)))^{-1}. \quad (7)$$

The obtained dependences $X(c)$ were used to plot the contour diagram on the c – d coordinates of the phosphorus content, where (c) is the fraction of i th phase (X_i) and (d) is the average particle size (Fig. 5). Figure 5 shows lines that correspond to identical phase compositions ($X(c) = \text{const}$), which were plotted for powders with different particle sizes on coordinates c – d .

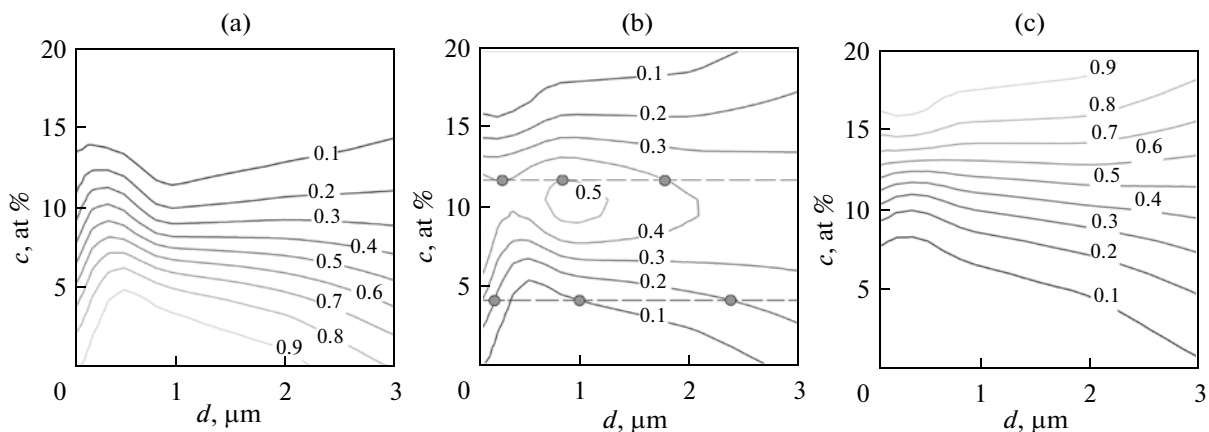


Fig. 5. Graphical presentation of volume fractions of (a) hcp, (b) fcc, and (c) amorphous phases by approximating functions $x(c)$. For amorphous and hcp phases, lines correspond to the same phase composition; volume fraction of the fcc phase was calculated by the expression $x_{\text{fcc}}(c) = 1 - x_{\text{hcp}}(c) - x_A(c)$.

An analysis of the obtained functions $X(c)$ showed that the characteristic phosphorus concentrations that correspond to a certain stage of phase transformations in the studied alloys depend on the particle size of the powder (d).

The phase diagrams given in Fig. 5 allow us to logically explain the experimental dependences of the local magnetic anisotropy field aH_a on the particle size (Fig. 6) obtained previously for amorphous and crystalline Co–P powders [8, 12]. The dependences $aH_a(d)$ for powders with phosphorus concentrations $c < 9$ at % are obtained in the form of convex curves with the maximum value aH_a corresponding to $d = 0.8 \mu\text{m}$; for powders with phosphorus concentrations $c > 12$ at %, a concave curve is observed, the minimum of which corresponds to $d = 0.8 \mu\text{m}$. This anomalous behavior of dependences $aH_a(d)$ can be explained based on changes in the phase composition of Co–P alloy powders (is shown in Fig. 5). The fact of the matter is that, among the three phases (hcp, fcc, and amorphous) that comprise the Co–P powder material, the fcc phase is the softest, i.e., it is characterized by the lowest values of H_a . At the same time, changes in aH_a in Fig. 6 can be compared to changes in the phase composition of a powder (Fig. 5) with a phosphorus concentration that remains unchanged with changing particle size d .

As an example, we consider the diagram shown in Fig. 5b. As d increases, the horizontal section corresponding to $c = 4$ at % cuts lines related to unchanged phase compositions of the fcc phases in sequence 0.2–0.1–0.2 (gray symbols in Fig. 5b). It is obvious that the achieved minimum content of the fcc phase for the region $d \sim 0.3\text{--}1 \mu\text{m}$ corresponds to the minimum average value aH_a (Fig. 6). As d increases, the horizontal section in Fig. 5b that corresponds to $c = 12$ at % cuts lines related to unchanged phase compositions of the fcc phases in sequence 0.4–0.5–0.4 (gray symbols in Fig. 5b). Here, the maximum content of the fcc phase is achieved near $d \sim 1 \mu\text{m}$ and leads to the minimum average value aH_a ; this also agrees with the data given in Fig. 6 (amorphous powders).

The cause for the dependence of the magnitude of the phosphorus concentrations, which correspond to the lines related to the unchanged phase composition in Fig. 5, on the particle size d can be the Laplace (bubble) pressure related to the surface curvature of particles. To analyze this assumption, we plot the phase diagram on coordinates $c\text{--}1/d$ (Fig. 7). The bottom and top lines in Fig. 7 correspond to fitting parameters c_0 for the hcp and amorphous phases, which were obtained when approximating experimental data using Eq. (7), respectively. Thus, in region I (Fig. 7), the hcp phase dominates (its content is more than 50%), while, in region III, the amorphous phase (its content is more than 50%) dominates. Region II corresponds to approximately equal fractions of coexisting hcp, fcc, and amorphous phases. This diagram also shows a point with coordinates $(0, 1/d^*)$, which

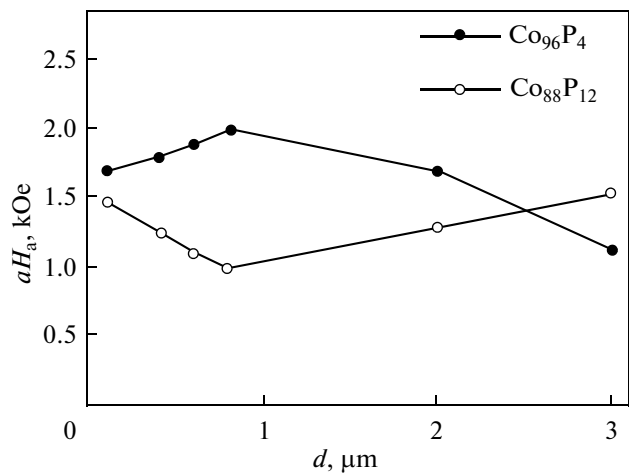


Fig. 6. Dependences of local anisotropy field on average diameter of Co–P powder particles [8].

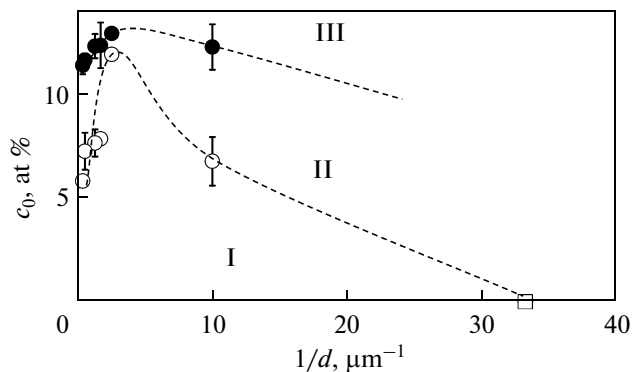


Fig. 7. Dependences of characteristic concentrations c_0 on average particle sizes for Co–P powders. Solid circles correspond to the parameters c_0 from Eq. (7) for the amorphous phase; open circles, for the hcp phase; and the open square, to the data from [22].

corresponds to the characteristic size for pure cobalt nanoparticles (d^*), below which the fcc structure becomes stable for Co particles at room temperature due to the bubble pressure [22, 23].

In our opinion, the bubble pressure related to the external surface of spherical particles, $\sim 1/d$, qualitatively explains the decrease in $c_0(1/d)$ for particles with $d < 0.4 \mu\text{m}$ since, in Fig. 7, the point corresponding to a powder with a particle size of $0.1 \mu\text{m}$ lies in the descending portion of curve that separates regions I and II, along with the point taken from [22]. For particles with $d > 0.4 \mu\text{m}$, the rise of curves that separate regions I, II, and III (Fig. 7) cannot be explained using the concepts on bubble pressure related to the external particle surface, since it is assumed that here we deal with the surface energy whose sign is opposite to that of the surface energy of the cobalt–air interface. We assume that the increase in $c_0(1/d)$ can be related to

the effect of internal pressure induced at the expense of excess energy of grain boundaries between crystallites, which comprise particles in powders under study.

It should be noted that the phase diagrams obtained for Co–P powders are the phase diagrams of metastable equilibrium, since they characterize the equilibrium state of metastable Co–P solid solutions.

CONCLUSIONS

The method used in the present study to quantitatively estimate the phase composition of a multiphase ferromagnetic alloy, which is based on measuring the temperature and field dependences of the magnetization, has a number of advantages as compared to the traditional magnetic phase analysis. First, the phase composition is estimated at low temperatures; this makes the method suitable for characterizing non-equilibrium alloys. The concept of achieving similar estimates was outlined in [24], which deals with the study of nanocrystalline ribbons, but was not carried out with a quantitative result. Second, when using the method, it is not necessary for the mass or volume of ferromagnetic material to be known. This makes the method useful in characterizing composites with a ferromagnetic component, the mass or volume of which is difficult to determine.

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