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Prediction of the concentration inhomogeneity of powder magnetic hard alloys based on the Fe–Cr–Co–Mo system and the effect of Sm additions on their magnetic properties

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Abstract. Hysteresis alloys based on Fe–Cr–Co system are extensively used in the instrument-making industry as a material for synchronous motors of navigation systems, in the electronic industry, and other mechanical engineering fields. The following requirements are imposed on Fe–Cr–Co alloys: temperature stability of magnetic characteristics over time, manufacturability, low porosity and concentration inhomogeneity, which allow to obtain high-quality magnetic and mechanical properties. Materials based on conventional alloying systems, such as Fe–Cr–Co, have outlived themselves. An urgent line of the development of new materials and improvement of the properties of existing ones is alloying with rare-earth metals. The effect produced by Sm addition on powder analogs of Fe–Cr–Co system remains unstudied. In this paper, 22Kh15K4MS powder magnetic hard alloy alloyed with samarium in an amount of 0.5 wt. % was studied. The billets were obtained by cold pressing at a pressure of 600 MPa followed by vacuum sintering. The concentration inhomogeneity of Cr, Co, Mo, Sm was determined after 12 different sintering modes. A model of diffusion homogenization of ridge alloys, which allows to numerically evaluate the effect of sintering modes on the concentration inhomogeneity, was plotted. The distributions of chromium, cobalt, and molybdenum correspond to the asymptotically logarithmically normal law. Samarium is unevenly distributed in the structure. The effect of samarium additions on the magnetic properties of the alloy has been demonstrated. The alloying of 22Kh15K4MS alloy with 0.5 wt. % of samarium allows to obtain powder hysteresis magnets with a coercive force in the range from 3.9 to 33.0 kA/m and a residual magnetic induction from 0.44 to 0.95 T.

Keywords: hard magnetic alloy, powder alloy, magnetic properties, concentration inhomogeneity, diffusion, concentration variation coefficient, Fe–Cr–Co–Mo

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Прогнозирование концентрационной неоднородности порошковых магнитотвердых сплавов на основе системы Fe–Cr–Co–Mo и влияние добавок Sm на их магнитные свойства

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Аннотация. Гистерезисные сплавы на основе системы Fe–Cr–Co широко применяют в приборостроении в качестве материала для синхронных двигателей навигационных систем, в электронной промышленности и других областях машиностроения. К сплавам Fe–Cr–Co предъявляется ряд требований: температурная стабильность магнитных характеристик во времени, технологичность изготовления, низкая пористость и концентрационная неоднородность, – позволяющих добиваться сочетания высоких магнитных и механических свойств. Материалы на основе традиционных систем легирования, такие как Fe–Cr–Co, исчерпывают себя. Актуальным направлением при разработке новых материалов и повышении свойств существующих является легирование редкоземельными металлами. Эффект, производимый добавкой Sm на порошковые аналоги системы Fe–Cr–Co, остается неизученным. В работе исследован магнитотвердый порошковый сплав 22Х15К4МС, легированный самарием в количестве 0,5 мас. %. Заготовки получены методом холодного прессования при давлении 600 МПа и последующим спеканием в вакууме. Определена концентрационная неоднородность Cr, Co, Mo, Sm после 12 различных режимов спекания. Построена модель диффузионной гомогенизации гребневых сплавов, позволяющая численно оценивать влияние режимов спекания на концентрационную неоднородность. Распределения хрома, кобальта и молибдена соответствуют асимптотически логарифмически нормальному закону, самарий распределен в структуре неравномерно. Показано влияние добавок самария на магнитные характеристики сплава. Легирование сплава 22Х15К4МС самарием в количестве 0,5 мас. % позволяет получать порошковые гистерезисные магниты с коэрцитивной силой 3,9–33,0 кА/м и остаточной магнитной индукцией 0,44–0,95 Тл.

Ключевые слова: магнитотвердый сплав, порошковый сплав, магнитные свойства, концентрационная неоднородность, диффузия, коэффициент вариации концентрации, Fe–Cr–Co–Mo

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Introduction

In order to obtain materials with specified characteristics, it is necessary to establish the relationship between structure and properties [1]. Porosity and concentration inhomogeneity have a considerable effect on the processes of structure formation and magnetic properties of alloys [2–4], reducing their mechanical, physical and performance characteristics [5]. The inhomogeneity of alloys is estimated using diffusion coefficients [6–8], however, their determination does not allow to predict the concentration inhomogeneity with reasonable accuracy, since it is associated not only with diffusion, but also depends on a number of other factors [9; 10]. The study of diffusion at the stage of sintering of powder materials is performed using diffusion couples, however, the application of this method within multicomponent systems results in considerable experimental difficulties [11; 12]. The numerical methods for solving homogenization equations [13–15] do not take into account the properties of powder particles, their shape and defectiveness, and models based on statistical processing of data on diffusion homogenization provide only qualitative information on the element distribution. One of the modern approaches to the study of diffusion is the experimental study of homogenization patterns [13].

In work [16], the non-monotonic effect of concentration inhomogeneity on the magnetic properties of a material was proved experimentally. Furthermore, there is an approach based on the assertion that the homogeniza-

tion process has a noticeable effect on the properties due to a change in the phase composition and distribution of phases, and a parallel transformation of the porous structure. In this paper, such a statistical value as concentration variation coefficient (V) was taken as a criterion for the inhomogeneity of the alloying element distribution. The technique allows to determine and predict the inhomogeneity and distribution of elements, provides a criterion for comparing the homogeneity of powder steels and alloys, allows to select the composition of materials and the technology for their production. The validity of this method for assessing inhomogeneity has been repeatedly confirmed in works [1; 11].

Recently, the alloys based on Fe–Cr–Co system with alloying additions, including rare-earth metals, have been extensively studied. The ferromagnetic materials based on SmCo_5 , $\text{Sm}_2\text{Co}_{17}$ compounds are widely used for the production of permanent magnets [17]. They exhibit a high Curie temperature, anisotropy field, and remanence, but exhibit high hardness and poor processibility. The alloys of Fe–Cr–Co system exhibit a high temperature stability, corrosion resistance, ductility and lower production costs compared to hard magnets made of rare-earth metals. In work [18], the alloying of a cast alloy of Fe–Cr–Co system with samarium up to 2 wt. % resulted in a significant increase in its magnetic properties: BH_{max} by 86 %, B_r by 47 %, and H_c by 28.7 %. The described approach to the alloying of chremel-copel alloys is of scientific and practical interest, however, the impact of concentration inhomogeneity on the effect of samarium additions has not been studied.

The study is aimed at the development of a homogenization model for predicting the modes of sintering of concentration-inhomogeneous powder hard magnetic alloys based on Fe–Cr–Co system with samarium.

Research methodology

This paper investigates the samarium-alloyed 22Kh15K4MS powder alloy, which exhibits the following chemical composition, wt. %:

Fe	Cr	Co	Mo	Si	Sm
57.0	22.5	15.0	4.0	1.0	0.5

The following powders of metals and alloys were used as initial components of the charge: chromium PKh-1S (TU 14-5-298-99, average particle size $r = 10 \mu\text{m}$, standard deviation $\sigma = 5 \mu\text{m}$); cobalt GP-OK (TU 1793-008-92, $r = 24 \mu\text{m}$, $\sigma = 13 \mu\text{m}$); iron OSCh 6-2 (TU 6-09-05808008-262-92, $r = 2 \mu\text{m}$, $\sigma = 2 \mu\text{m}$); ferrosilicon FS50 (GOST 1415-93-92, $r = 8 \mu\text{m}$, $\sigma = 4 \mu\text{m}$); molybdenum MPCh (TU 48-19-69-80, $r = 2 \mu\text{m}$, $\sigma = 1 \mu\text{m}$); samarium-cobalt (GOST 21559-76, $r = 39 \mu\text{m}$, $\sigma = 21 \mu\text{m}$).

The charge was sieved through a mesh with a cell size of $63 \mu\text{m}$ and averaged in a mixer with a shifted rotation axis for 8 h.

The sample billets were obtained by cold pressing at a pressure of 600 MPa, followed by vacuum sintering with a residual pressure of 10^{-2} Pa according to the modes specified in Table 1.

The sintering temperature range was selected on the basis of the fact that sintering at temperatures below 1200°C results in obtaining magnets with a non-magnetic γ -phase production. Increasing the sintering temperature up to 1400°C causes the evaporation of components from the surface of the billets. In both cases, these processes lead to the deterioration of the magnetic properties of the alloy [19].

All samples were quenched in 15 % aqueous NaCl solution at the temperature starting from 1250°C . The aging of the billets was performed sequentially at 9 stages,

Table 1. The sintering modes of 22Kh15K4MS alloy with 0.5 wt. % Sm

Таблица 1. Режимы спекания сплава 22X15K4MC с 0,5 мас. % Sm

Sample No.	Sintering temperature, $^\circ\text{C}$	Holding time, h
1	1250	1, 3, 8
2	1300	
3	1350	
4	1380	

and the processing indices are provided in Table 2. The main parameters and the shape of the magnetic hysteresis loop of the test samples were determined by means of “Permagraph L” hysteresis graph (Magnet-Physik, Germany) with PERMA software after the quenching and multistage aging.

To examine the patterns of changes in the concentration inhomogeneity of element distribution during sintering, the concentrations of Cr, Co, Mo, and Sm were measured at 100 points being equidistant from each other on the surface of the section. The concentrations were measured by the method of X-ray microanalysis, using “Tescan Mira 3” electron microscope (Tescan, Czech Republic). The measure of concentration inhomogeneity is the relative fluctuation of concentration, i.e. the concentration variation coefficient V , being equal to the ratio of the square root of the concentration dispersion to its average value:

$$V = \frac{\sqrt{D}}{C}, \quad (1)$$

where D is the element concentration dispersion, and C is the average concentration [16].

The assumption of the logarithmic normality of the distributions was tested using Pearson’s χ^2 test. The entire measurement range of the random variable was divided into disjoint intervals k . In accordance with the given division, the number of sample values that fell within the i -th interval (designated as n_i). The obtained histogram of the sampling distribution serves as the basis for selecting the distribution law [20]. The value characterizing the deviation of the sample distribution from the predicted one is determined by the following formula:

$$\chi^2 = \frac{(n_i - np_i)^2}{np_i}, \quad (2)$$

Table 2. Billet aging modes

Таблица 2. Режимы старения заготовок

Stage No.	Aging temperature, $^\circ\text{C}$	Holding time, min
1	670	30
2	640	40
3	600	40
4	575	40
5	555	30
6	535	30
7	525	30
8	500	30
9	480	30

where n is the sample volume; n_i is the number of sample elements, falling within the i -th interval; p_i is the probability that the random variable under study falls into the i -th interval, calculated in accordance with the hypothetical distribution law $F(x)$.

In order to predict the concentration inhomogeneity, the following homogenization equation was used:

$$V_j = \alpha_j \exp\left(\beta_j t c_j^{2/3} \exp\left[-\frac{Q_j}{RT}\right]\right), \quad (3)$$

where V_j and c_j are the variation coefficient and the average concentration of the j -th matrix component; t (h) and T (K) are the sintering time and temperature, respectively; R is the gas constant, J/(mol·K); Q_j is the effective activation energy of mutual diffusion in the system, J/(mol·K); α_j and β_j are the coefficients [16].

Research result and disussion

The structure of the alloys after quenching represented an α -solid solution of Cr, Co, Mo, and Si. Samarium was in the form of individual inclusions (Fig. 1) with very low solubility in the α -phase. The presence of dark areas in Fig. 1 testifies to the residual porosity of the material.

The first stage was to obtain the values of the concentration inhomogeneity for Cr, Co, Mo, Sm. The concentration inhomogeneity decreased with an increase in the sintering temperature and holding time (Table 3).

To determine the law of distribution of concentrations, Pearson's χ^2 criterion was calculated after each of 12 sintering modes. No distributions corresponding to the

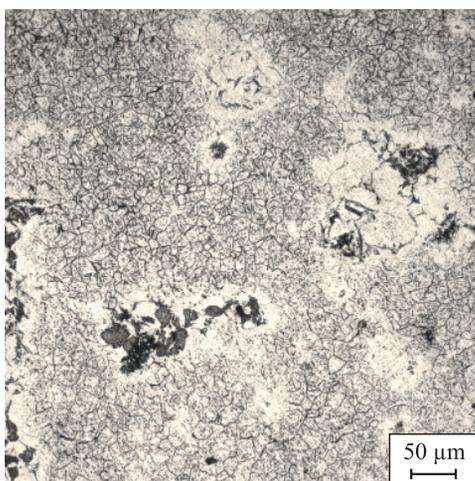


Fig. 1. The microstructure of 22Kh15K4MS alloy alloyed with 0.5 wt. % of Sm after the quenching

Рис. 1. Микроструктура сплава 22Х15К4МС, легированного 0,5 мас. % Sm, после закалки

normal law were identified for chromium. 7 distributions out of 12 distributions corresponded to the logarithmically normal law. A similar pattern was observed when calculating χ^2 for cobalt and molybdenum. Samarium is unevenly distributed in the structure, so it is impossible to establish the law of its distribution within the specified scope of the experiment.

Upon the construction of element distribution histograms, the entire range of data was divided into 8 intervals (Fig. 2). The distribution histograms of chromium, cobalt, and molybdenum exhibited a right-hand asymmetry, which increased with rising temperature and sintering time.

The approximation of experimental data (Table 3, numerator) allowed to determine α_j , β_j coefficients of the homogenization equation, activation energy Q (Table 4),

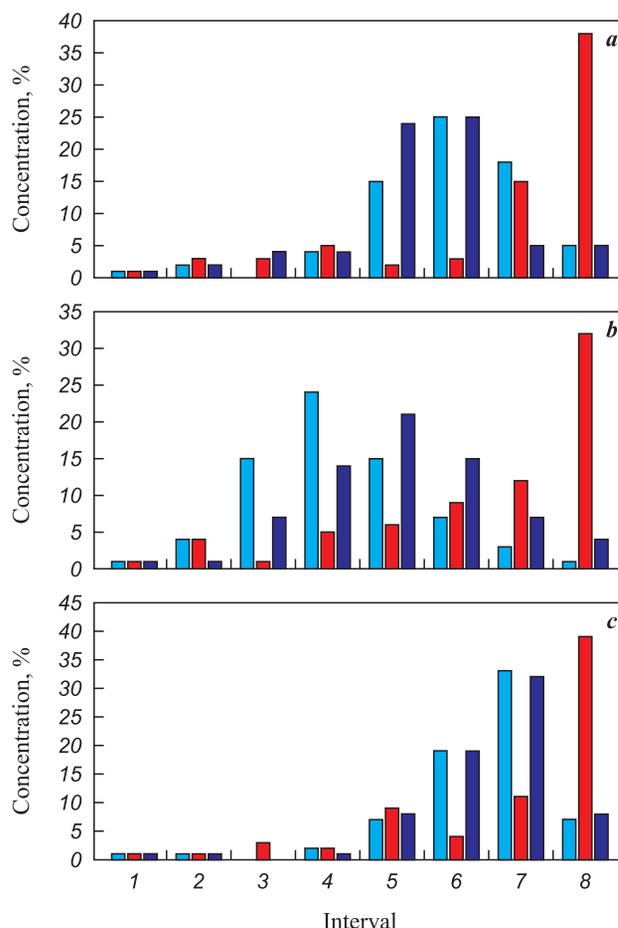


Fig. 2. Distribution histograms of cobalt concentrations (a), molybdenum (b) and chromium (c) after sintering at a temperature of 1250 °C, 3 h
 ■ – observed distribution, ■ – expected normal distribution, ■ – expected log-normal distribution

Рис. 2. Гистограммы распределения концентраций кобальта (а), молибдена (b) и хрома (с) после спекания при температуре 1250 °С в течение 3 ч
 ■ – наблюдаемое распределение, ■ – ожидаемое распределение по нормальному закону, ■ – ожидаемое распределение по логарифмически нормальному закону

Table 3. Experimental (numerator) and predicted (denominator) concentration variation coefficients

Таблица 3. Экспериментальные (числитель) и прогнозируемые (знаменатель) коэффициенты вариации концентрации

Sintering mode	Sintering temperature, °C	Holding time during sintering, ч	V			
			Cr	Co	Mo	Sm
1	1250	1	<u>0.16</u> 0.15	<u>0.30</u> 0.20	<u>0.52</u> 0.37	<u>0.87</u> –
2		3	<u>0.14</u> 0.15	<u>0.14</u> 0.18	<u>0.29</u> 0.29	<u>1.07</u> –
3		8	<u>0.12</u> 0.14	<u>0.13</u> 0.13	<u>0.21</u> 0.23	<u>0.88</u> –
4	1300	1	<u>0.20</u> 0.15	<u>0.19</u> 0.19	<u>0.34</u> 0.31	<u>0.70</u> –
5		3	<u>0.17</u> 0.15	<u>0.18</u> 0.17	<u>0.25</u> 0.29	<u>1.08</u> –
6		8	<u>0.09</u> 0.13	<u>0.11</u> 0.12	<u>0.29</u> 0.22	<u>1.03</u> –
7	1350	1	<u>0.11</u> 0.13	<u>0.10</u> 0.90	<u>0.33</u> 0.31	<u>1.23</u> –
8		3	<u>0.15</u> 0.15	<u>0.20</u> 0.14	<u>0.30</u> 0.27	<u>1.19</u> –
9		8	<u>0.10</u> 0.11	<u>0.90</u> 0.70	<u>0.20</u> 0.20	<u>1.15</u> –
10	1380	1	<u>0.15</u> 0.14	<u>0.16</u> 0.17	<u>0.34</u> 0.31	<u>0.91</u> –
11		3	<u>0.11</u> 0.15	<u>0.10</u> 0.12	<u>0.18</u> 0.27	<u>0.70</u> –
12		8	<u>0.09</u> 0.10	<u>0.06</u> 0.07	<u>0.16</u> 0.19	<u>1.66</u> –

as well as the predicted values V (Table 3, denominator). Due to the uneven distribution of samarium in the structure and high values of concentration inhomogeneity, the prediction of V_{Sm} was not performed.

The dependence of V_e ratio (the experimental coefficient of variation) to V_p ratio (predicted) on the sintering mode was plotted (Fig. 3). The closest V_e and V_p ratios were obtained upon modes 4, 6, 8, 9, 10, which correspond to sintering at temperatures of 1300 °C (1 and 8 h), 1350 °C (3 and 8 h), and 1380 °C (1 h).

Q_{Cr} values correspond to the activation energy of chromium in the α -Fe phase of the cast alloy obtained in work [21]. The activation energy amounted to

Table 4. Activation energies and equation coefficients

Таблица 4. Энергии активации и коэффициенты уравнений

Element	Q , kJ/(mol·K)	α	β
Cr	209.2	0.157	0.252
Co	288.0	0.207	5.134
Mo	282.0	0.340	1.190
Sm	40.3	0.940	0.003

197 ± 10 kJ/(mol·K) for the alloy with 25.8 wt. % of Cr, which is likely associated with the formation of low-soluble compounds during the heating. Q_{Co} value appeared to be higher than the one specified in the literary sources [22].

An increase in sintering temperature and sintering time resulted in an increase in homogeneity and a decrease in V coefficient. Activation energy Q_{Mo} appeared to be at the level of activation energy in binary alloys of iron with

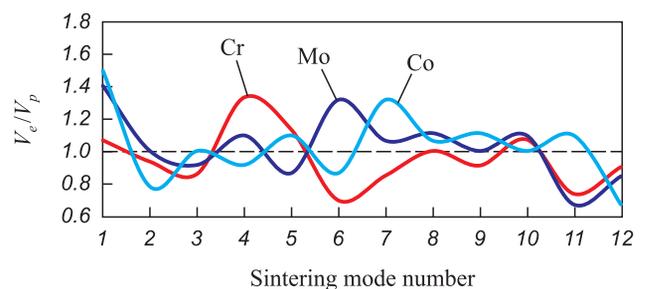


Fig. 3. The dependence of V_e ratio (experimental) to V_p ratio (predicted) on the sintering mode

Рис. 3. Зависимость отношения V_e (эксперимент) к V_p (прогноз) от режима спекания

Mo [20], which is associated with a high ratio of surfaces and the formation of low-soluble compounds during the heating. In Fe–6%Mo alloy, the activation energy is at the level of 262.5 J/(mol·K), and in powder alloy Q_{Mo} it accounts for 282 J/(mol·K) (Table 4).

Sintering at 1250 °C allows to obtain V at the level of 0.16–0.12 for Cr, 0.3–0.1 for Co, at the level of 0.5–0.2 for Mo, and at the level of 0.9–1.1 for Sm, depending on the sintering time. It is noted that the resulting concentration inhomogeneity is directly proportional to the atomic mass of the element. An increase in the sintering temperature up to 1300 °C results in V decrease only after 8 h of holding: 0.09 for Cr and 0.11 for Co, the inhomogeneity values remain the same for Mo. The lowest values of concentration inhomogeneity were obtained after sintering at 1380 °C for 8 h: 0.09 for Cr, 0.06 for Co, and 0.16 for Mo. A further increase in the sintering temperature requires more energy and may result in intense evaporation of chromium from the surface of the billets, which, in turn, will lead to inhomogeneity of the chemical composition between the surface layer and the volume of the sample and a decrease in magnetic properties [5; 23]. Thus, for powder magnets made of 22Kh15K4MS alloy, alloyed with Sm, the optimal sintering mode is the one at a temperature of 1350 °C for 1–3 h. The concentration inhomogeneity of Cr, Co, and Mo, depending on the holding time, varies within 0.01–0.06 (Table 3). The application of quenching after sintering allows to reduce the concentration inhomogeneity of chromium and cobalt to the level of $V = 0.06 \div 0.08$ [24].

The magnetic properties of the samples after sintering for 8 h, quenching, and multistage aging (see Table 2) at various sintering temperatures (Fig. 4) were determined. Upon an increase in sintering temperature from 1250

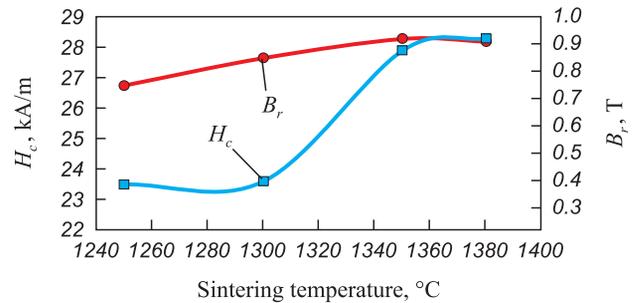


Fig. 4. The dependence of coercive force (H_c) and magnetic induction (B_r) on the sintering temperature

Рис. 4. Зависимость коэрцитивной силы (H_c) и магнитной индукции (B_r) от температуры спекания

to 1380 °C, an increase in the level of magnetic properties by 18–20 % was observed, which is associated with a decrease in the concentration inhomogeneity of the alloy (Table 3). The optimum magnetic properties were also obtained after sintering at 1350 °C.

For testing the homogenization model as per equation (3), the sintering time for each mode and element was calculated. Table 5 shows the calculated sintering time for the element with the longest homogenization time at a given temperature. According to the results obtained, the level of concentration inhomogeneity is achieved in a shorter time at temperatures of 1250 and 1300 °C (1, 3 and 8 h), 1350 °C (1–3 h), 1380 °C (1 h). At sintering temperatures of 1350 °C, 1380 °C and holding time of 3–8 h, the calculated sintering time significantly exceeds the actual time. This is associated with the fact that the activation energy may not match the value used in the calculation. The used effective activation energy is applicable precisely for this process at the boundary of the existing model.

Table 5. Calculated sintering times

Таблица 5. Расчетные значения времени спекания

Sintering temperature, °C	Element with the longest homogenization time	V	Holding time during sintering, h	
			Actual	Calculated
1250	Co	0.30	1	0.82
	Mo	0.29	3	1.10
	Mo	0.21	8	3.20
1300	Mo	0.34	1	0.61
	Mo	0.25	3	2.10
	Mo	0.29	8	2.90
1350	Mo	0.33	1	0.61
	Mo	0.30	3	0.83
	Cr	0.10	8	11.80
1380	Mo	0.34	1	0.61
	Co	0.10	3	6.90
	Co	0.06	8	13.50

To assess the effect of samarium addition on the magnetic properties of 22Kh15K4MS alloy, the multistage aging of samples without samarium as well as samples with 0.5 wt. % of Sm was performed. The fastest growth of the magnetic properties (B_r and H_c) of the alloy with 0.5 wt. % of Sm occurred at the first three stages of aging. In subsequent stages, only H_c growth continued. Upon that, the magnetic induction B_r reached its peak at the 4th stage of aging with further permanent decrease (Table 6).

The rectangularity coefficient of magnetic hysteresis loop (C_r) had the highest value after the 3rd stage of aging (Table 6). Compared to the initial alloy, C_r is 9–11 % higher for the alloyed alloy under this heat treatment mode. According to the X-ray diffraction analysis performed in work [25], the diffraction peaks corresponding to $\text{Sm}_2\text{Co}_{17}$ (64.9°, 74.68°) and SmCo_5 (50.798°) phases were identified in the samples after aging, except for the α -phase. This, in turn, testifies to the redistribution of samarium during aging and the production of a stoichiometric composition distinct from the initial component.

Conclusions

1. The distributions of chromium, cobalt, and molybdenum correspond to the asymptotically logarithmically normal law. Samarium is unevenly distributed in the structure, therefore, it is impossible to establish the law of its distribution within the specified scope of the experiment. The homogenization process did not effect the homogeneity of samarium distribution, which is apparently associated with the non-solubility of Sm in iron.

2. A complete statistical description of the distribution of Cr, Co, Mo of 22Kh15K4MS ridge alloy with

Table 6. Magnetic properties of the 22Kh15K4MS alloy with a additive of samarium after aging

Таблица 6. Магнитные свойства сплава 22X15K4MC с добавкой самария после старения

Aging temperature, °C:	22X15K4MC		22X15K4MC + 0,5 % Sm	
	H_c , kA/m	B_r , T	H_c , kA/m	B_r , T
670	2.7	0.40	3.9	0.44
640	5.1	0.77	6.5	0.82
600	8.5	0.97	10.7	0.93
575	16.8	0.99	19.5	0.95
555	24.7	0.99	26.4	0.92
535	28.4	0.98	29.7	0.90
525	28.4	0.93	29.4	0.86
500	31.3	0.92	32.9	0.86
480	32.1	0.92	33.0	0.85
BH_{\max} , kJ/m ³	12		11	
C_r , at 600 °C	0.66		0.73	

the addition of samarium in the amount of 0.5 wt. % is presented. Based on the experimental determination of V criterion, a homogenization equation, which allows to numerically evaluate the effect of sintering modes on the concentration inhomogeneity, was obtained.

3. The optimal sintering mode for powder magnets made of samarium-alloyed 22Kh15K4MS alloy was experimentally selected.

4. The alloying of 22Kh15K4MS alloy with samarium in an amount of 0.5 wt. % makes it possible to obtain powder hysteresis magnets with a coercive force of 3.9–33.0 kA/m and a residual magnetic induction of 0.44–0.95 T at C_r up to 0.73, which constitutes an actual index for precision tool-making.

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