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EFFECT OF PRASEODYMIUM ON MAGNETIC PROPERTIES AND PHASE COMPOSITION OF A MATERIAL OF THE Nd – Pr – Dy – Fe – Co – B SYSTEM

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Sintered magnetically hard materials with compositions $(Nd_{0.5-x}Pr_xDy_{0.5})_{18}(Fe_{0.7}Co_{0.3})_{rem}B_{5.5}$ ($x = 0.12 - 0.5$) and $(Nd_{0.6-x}Pr_xDy_{0.4})_{15}(Fe_{0.8}Co_{0.2})_{rem}B_6$ ($x = 0.03 - 0.4$) are studied. The role of praseodymium (at invariable content of dysprosium and cobalt) in the change of magnetic properties of the material (residual induction, coercive force, temperature stability) is determined. The phase composition of the material is identified. The role of praseodymium in the magnetic properties of the material is interpreted.

INTRODUCTION

The main disadvantage of magnetically hard materials of the Nd – Fe – B system is their low temperature stability characterized by the temperature coefficient of induction (TCI). The value of the TCI of these materials is about – 0.1 %/°C, which limits considerably the range of their application, especially in navigation devices. It is known that by alloying magnetically hard materials of the Nd – Fe – B system with heavy rare-earth metals (HREM) and cobalt their TCI can be changed in a wide range [1, 2]. The value of the TCI is determined by the nature of the temperature dependence of the saturation magnetization of the material (I_s) in the specified temperature range. The mechanism of the action of HREM and cobalt on the temperature dependence of I_s in the Nd – Fe – B system (and hence on the value of the TCI) can be explained qualitatively within the model of double-sublattice ferromagnetic with antiferromagnetic exchange interaction between the sublattices [3, 4]. In this case the effect of HREM and cobalt on the TCI is proportional to their concentration [1, 2, 4]. Unfortunately, alloying with HREM and cobalt lowers the residual induction (B_r) of the magnet and also in proportion to their concentration [1, 2]. Therefore, it is important in principle to choose such alloying elements that would raise B_r of the material without worsening its temperature stability. In addition, it is known that the characteristics of magnets are very sensitive to the structure and phase composition of the material [5]. Works devoted to the phase composition of magnetically hard materials of the Nd – Fe – B system alloyed with Co (with substitution of over 20 at.% Fe by Co) and HREM (with substitution of over 30 at.% Nd by HREM) are quite few. For example, a study of magnets with composition $Nd_{15}Fe_{67}Co_{10}B_8$ performed in [6] showed that in addition to the phases typical for the Nd – Fe – B system the metal contained a $Nd_3(Fe_{0.5}Co_{0.5})$ intermetallic compound and a Laves phase $Nd(Fe, Co)_2$. It is shown in [7] that magnetically hard materials with composition $Nd_{23-y}Fe_{77-2x}Co_{2x}B_y$ at $x > 7$ and $y = 6 - 9$ contain a Nd – Co – B boride phase of unidentified composition. The magnetic properties of alloys $(Nd_{1-x}Dy_x)_{15}Fe_{77}B_8$ are studied in [8] in the range of concentrations $x = 0 - 0.6$. The results of magnetic measurements of the authors of [8] explain the effect of the $DyFe_4B$ phase, which is doubtful, because a $DyFe_4B$ compound does not exist [9]. The $(Nd_{1-x}Dy_x)_{18.5}Fe_{75}B_{6.5}$ system with $x = 0 - 1$ is studied in [10], where an RFe_3 intermetallic compound (where R is one or several rare-earth metals) is detected in addition to the Laves phase. Magnets from a $(Nd_{1-x}Dy_x)_{15.5}(Fe_{77-y}Co_y)B_{7.5}$ material are studied in [11]. It is shown that at $x = 0.2$ and $y \leq 0.13$ the phases conventional for the Nd – Fe – B system are accompanied by $DyCo_2$; and $Nd(FeCo)_2$ and $(Nd, Dy)(Fe, Co)_{5.2}$ phases appear at $y \geq 0.18$ [11].

The aim of the present work consisted in a complex study of sintered magnetically hard materials $(Nd_{0.5-x}Pr_xDy_{0.5})_{18}(Fe_{0.7}Co_{0.3})_{rem}B_{5.5}$ ($x = 0.12 - 0.5$) and $(Nd_{0.6-x}Pr_xDy_{0.4})_{15}(Fe_{0.8}Co_{0.2})_{rem}B_6$ ($x = 0.03 - 0.4$).

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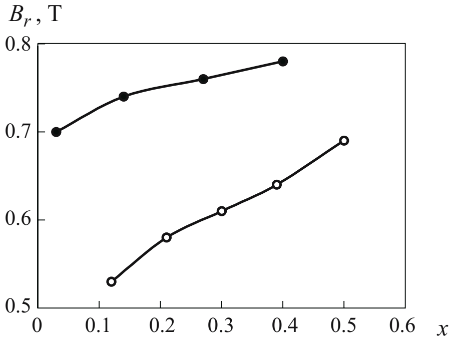

Fig. 1. Dependence of residual induction B_r on the relative concentration of neodymium x: ○) $(Nd_{0.5-x}Pr_xDy_{0.5})_{18}(Fe_{0.7}Co_{0.3})_{rem}B_{5.5}$ (group *1*); ●) $(Nd_{0.6-x}Pr_xDy_{0.4})_{15}(Fe_{0.8}Co_{0.2})_{rem}B_6$ (group *2*).

METHODS OF STUDY

We studied magnetically hard materials of the following groups (the composition is given in atomic fractions expressed in percent):

(*1*) $(Nd_{0.5-x}Pr_xDy_{0.5})_{18}(Fe_{0.7}Co_{0.3})_{rem}B_{5.5}$ ($x = 0.12 - 0.5$);

(*2*) $(Nd_{0.6-x}Pr_xDy_{0.4})_{15}(Fe_{0.8}Co_{0.2})_{rem}B_6$ ($x = 0.03 - 0.4$).

The ratio of the content of Dy to the sum of rare-earth metals (R) and the ratio Co/T, where T = Fe + Co, were kept constant within each group. For magnets of group *1* the mentioned ratios were Dy/R = 0.53 and Co/T = 0.31; for group *2* Dy/R = 0.44 and Co/T = 0.22. The values of Dy/R and Co/T were calculated from the mass fraction of the elements expressed in percent.

The alloys were melted in a vacuum induction furnace by a conventional method. The ingots were crushed into particles with a size below 630 μm in an inert atmosphere. Fine milling of the particles was performed in a centrifugal planetary mill in a trifluorotrichroroethane medium. Magnet preforms in the form of prisms were pressed in a transverse magnetic field of 800 kA/m by the method of "wet" pressing. The preforms were sintered in a vacuum furnace at 1100 – 1130°C for 1 h. The value of the TCI was measured in an open magnetic circuit in a temperature range of 20 – 100°C accurate to no worse than ± 0.005 %/°C.

We analyzed the local chemical composition of phases by the method of qualitative and quantitative microscopic x-ray spectral analysis (MXRSA) with the help of a "SUPERPROB-733" (JCMA-733, JEOL, Japan) apparatus. The area of analysis was 1 μm^2 and the depth was 1 μm. Photographs of the studied structures were obtained in the COMPO mode (the image was formed by reflected electrons) on unetched polished sections. The contrast of the images was determined by the mean atomic number of the phase. The higher the mean atomic number of the studied region (phase) the lighter the given region looks on the photograph. The exact chemical composition was determined by measuring the intensity of the lines of secondary x-ray radiation of the elements with subsequent automatic ZAF correction by a microcomputer. During the ZAF correction we introduced corrections allowing for the effects of the interaction between the electron beam and the specimen. This software allows for the reflectivity of the exciting (primary) electrons, for the absorptivity, and for the fluorescence of the secondary x-ray radiation. The random error introduced by the microcomputer was less than 1% on the average from element to element and from specimen to specimen. The presence of a systematic error was controlled from the deviation of the determined total content of elements from 100%. Such computation ensured determination of the concentrations with an accuracy no worse than ± 0.02 except for the cases of very high values of correction factors. The MXRSA technique was not used for determining the content of boron in the phases.

RESULTS

We established (Fig. 1) that with growth in the concentration of praseodymium the residual induction B_r increased monotonically, and this effect was more manifested in magnets of group *1*. The coercive force H_{cI} of magnets of group *1* increased monotonically from 1500 to 1800 kA/m with growth in the concentration of Pr; in magnets of group *2* it remained virtually unchanged, preserving the level of H_{cI} = 1700 kA/m. With growth in the amount of Pr the value of the TCI of magnets of group *2* changed from – 0.02 to – 0.01 %/°C; in magnets of group *1* it changed from 0 to + 0.01 %/C (in the range of 20 – 100°C).

The microstructure of magnetically hard alloys of group *1* is presented in Fig. 2, and the results of the MXRSA (for the main phases) are presented in Table 1. In addition to the phases given in Table 1 all the specimens contained insignificant amounts of intergrain phases enriched with rare-earth metals. In magnets of group *1* (x = 0.12 and 0.21) we also observed insignificant amounts of phases close to Laves ones in the composition, but we did not manage to identify them. The phases were identified in the following way. We considered all possible phases described in the literature [6 – 12] for the R – (Fe, Co) – B system including boride phases. Proceeding from the ratio R/T (Table 1) we chose the composition for presumed phases (Table 2) so that the ratio R/T and the composition of the phases expressed in wt.% were the closest to the experimental values.

DISCUSSION

We established that despite the growth in B_r, the TCI of the material of the magnets of group *2* decreased (in the absolute value). In magnets of group *1* even the sign of the TCI became positive with growth in the residual induction. We do not know of any work (at least for the Nd – Fe – B system) where the temperature stability of a magnetically hard material increases simultaneously with the growth in B_r. It is impossible to explain the effect of praseodymium on B_r and TCI without leaving the framework of the model of double-sublattice ferromagnetic (under the condition that Dy/R = const and Co/T = const). The point is that the magnetic moments of praseodymium ions, like those of all light rare-earth elements, are tied to the sublattice of transition metals (Fe, Co) by ferromagnetic ion interaction. At an invariable relative content of dysprosium the substitution of neodymium by praseodymium should not change the form of the temperature dependence of the magnetization and, hence, of the TCI (to say nothing of the change in the sign of this parameter, which is observed in magnets of group *1*) [3, 4]. It appears that growth in the concentration of praseodymium is accompanied by a change in the phase composition of the material and in the composition of individual phases.

A homologous series of compounds exists between RCo_5 and RCo_3B_2 [13]. This series is described by a common formula $R_{n+1}Co_{3n+5}B_{2n}$ (*n* may vary from zero to infinity). The RT_4B phase (*n* = 1) with a structure of type $CeCo_4B$ exists for all rare-earth metals except for Eu and Yb when T = Co. For T = Fe this phase has been detected only for the cases of R = Er, Tm, and Lu [9]. If we take an element that cannot form a RFe_4B phase for R, this phase can still exist in the case of partial substitution of Fe by Co. For example, the $RCo_{4-x}Fe_xB$ compounds, where R = Nd, Sm, Er, are studied in [14]. Thus, we can presume that a $R(Fe, Co)_4B$ phase can exist for any rare-earth metal or for a sum of rare-earth metals, of course, for specific values of the Fe/Co ratio. A phase of type RT_3 described in [10] exists for almost all R when T = Fe, Co, Ni [15].

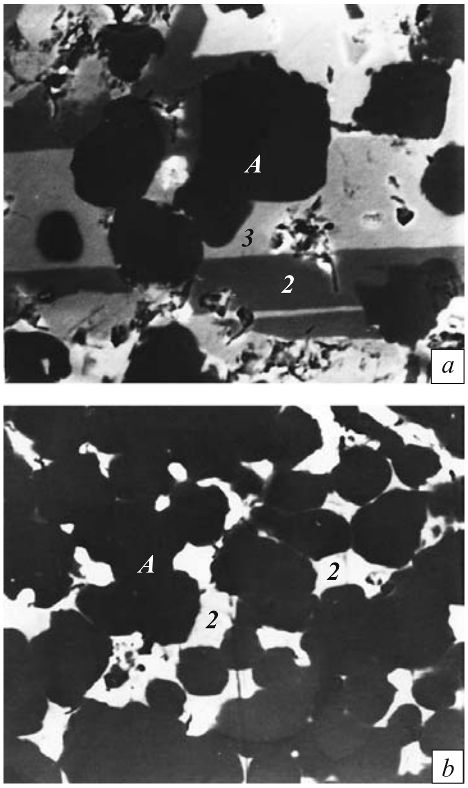

Fig. 2. Microstructure of magnetically hard materials of group *1* $(Nd_{0.5-x}Pr_xDy_{0.5})_{18}(Fe_{0.7}Co_{0.3})_{rem}B_{5.5}$ (× 1500): *a*) *x* = 0.12 [*A* is the main magnetic phase $R_2(Fe, Co)_{14}B$; *2*) a “gray” phase of type $R_3T_{11}B_4$; *3*) a “light-gray” phase of type RT_3B_2]; *b*) *x* = 0.5 [*A* is the main magnetic phase $R_2(Fe, Co)_{14}B$; *2*) a “light-gray” phase of type RT_3].

TABLE 1. Results of MXRSA of Main Phases in Studied Materials

Group of material	Phase	Content of elements, %					Dy/R	Co/T	R/T	Σ AE, %
		Nd	Pr	Dy	Fe	Co				
1 (*x* = 0.12)	*A*	6.5	3.3	20.3	54.2	15.5	0.67	0.22	0.43	99.8
	2	10.2	2.7	28.0	35.2	19.9	0.68	0.36	0.74	96.0
	3	11.2	3.2	30.7	32.2	18.2	0.68	0.36	0.89	95.5
1 (*x* = 0.2)	*A*	5.7	4.0	20.9	54.1	16.0	0.68	0.23	0.44	100.7
	2	7.9	5.1	28.0	34.0	20.1	0.68	0.37	0.76	95.1
	3	9.0	6.9	34.2	30.5	18.8	0.68	0.38	1.00	99.0
1 (*x* = 0.5)	*A*	–	9.3	27.0	45.5	18.0	0.74	0.28	0.57	99.8
	2	–	15.9	32.6	28.0	23.3	0.67	0.45	0.94	99.8
2 (*x* = 0.14)	*A*	10.7	2.6	20.2	55.4	10.7	0.60	0.16	0.51	99.6
	2	13.2	2.8	19.5	41.2	19.5	0.55	0.32	0.58	96.2
	3	32.4	6.7	15.7	26.0	17.7	0.29	0.41	1.25	98.5
2 (*x* = 0.4)	*A*	5.2	8.5	21.0	55.5	10.9	0.61	0.16	0.52	101.1
	2	7.0	11.0	27.7	34.3	19.4	0.61	0.36	0.85	99.4
	3	14.9	28.4	20.0	19.2	19.1	0.32	0.50	1.65	101.6

Notations: *A*) primary magnetic phase $R_2(Fe, Co)_{14}B$; *2*) gray or light-gray phase; *3*) light-gray phase (see Fig. 2); Dy/R, Co/T, R/T) ratios of the mass fractions (in %) of elements; R) total content of rare-earth metals; T) Fe + Co; Σ AE) total content of elements according to the data of MXRSA.

TABLE 2. Composition of Phases and Formulas of Assumed Compounds

Group of materials	Phase	Assumed compound	Composition of the phase	R/T
1 ($x = 0.12$)	*2*	$R_3T_{11}B_4$	$(Nd_{0.27}Pr_{0.07}Dy_{0.66})_{2.9}(Fe_{0,65}Co_{0.35})_{10.7}B_4$	0.74
	3	RT_3B_2	$(Nd_{0.27}Pr_{0.08}Dy_{0.65})_{1.0}(Fe_{0,65}Co_{0.35})_{3.1}B_{1.5}$	0.89
1 ($x = 0.2$)	*2*	$R_3T_{11}B_4$	$(Nd_{0.21}Pr_{0.14}Dy_{0.65})_{2,9}(Fe_{0.65}Co_{0.36})_{10.5}B_{4.6}$	0.75
	3	RT_3B_2	$(Nd_{0.17}Pr_{0.14}Dy_{0.68})_{1.0}(Fe_{0.62}Co_{0.39})_3B_2$	1.01
1 ($x = 0.5$)	*2*	RT_3	$(Pr_{0.36}Dy_{0.64})(Fe_{0.56}Co_{0.44})_{2.9}$	0.95
2 ($x = 0.14$)	*2*	RT_4B	$(Nd_{0.40}Pr_{0.09}Dy_{0.52})_{0.8}(Fe_{0.69}Co_{0.31})_{3.70}B_{1.2}$	0.59
	3	RT_2	$(Nd_{0.61}Pr_{0.13}Dy_{0.26})_{1.0}(Fe_{0.60}Co_{0.4})_2$	1.24
2 ($x = 0.4$)	*2*	RT_3	$(Nd_{0.17}Pr_{0.26}Dy_{0.57})_{1.0}(Fe_{0.65}Co_{0.35})_3$	0.86
	3	RT_2	$(Nd_{0.25}Pr_{0.48}Dy_{0.28})_{1.0}(Fe_{0.51}Co_{0.49})_{1.6}$	1.63

Note. The composition of the phases (see Table 1) is given in at.%. The ratio of the sum of rare-earth metals (R) to the sum (Fe + Co) (T) is calculated in wt.%.

The data of Table 2 show that phases *2* for magnets of group *1* ($x = 0.12$ and 0.2) are close to those of magnets of group *2* ($x = 0.14$) and are describable by formulas $R_{n+1}Co_{3n+5}B_{2n}$ at $n = 2$ ($R_3T_{11}B_4$) and $n = 1$ (RT_4B), respectively. Phase *3* in magnets of group *1* ($x = 0.12$ and 0.2) corresponds to an RT_3B_2 compound ($n = \infty$). Phase *2* in magnets of group *1* ($x = 0.5$) and of group *2* ($x = 0.4$) is identifiable as an RT_3 compound, and phase *3* in magnets of group *2* ($x = 0.14$ and 0.4) is identifiable as a Laves phase RT_2. It should be noted that the RT_4B_4 compound possible for the Nd – Fe – B system has the same R/T ratio as RT_4B. However, in this case we would obtain a too great difference in R + T with the total content of these elements determined by the method of MXRSA (see Table 1), because this compound has a higher relative content of boron.

The example of the studied magnets (Table 2) shows that with growth in the content of praseodymium the boride phases (except phase *A*) disappear, while the concentration of the primary magnetic phase in the material grows (Fig. 2).

It is known that the magnetization of a compound of type $R_{n+1}(Fe, Co)_{3n+5}B_{2n}$ is considerably lower than that of $R_2(Fe, Co)_{14}B$ [9, 13, 14]. It seems that praseodymium blocks the formation of secondary boride phases, and the entire boron is spent for the formation of a $R_2(Fe, Co)_{14}B$ phase, which is known to determine the value of B_r of a material. In addition, it can be seen from Table 1 that as the content of Pr increases (from $x = 0.12$ to $x = 0.5$) the ratio Dy/R in magnets of group *1* increases by 10% and Co/T increases by 33%. In magnets of group *2* the growth in x from 0.14 to 0.4 virtually does not affect the ratios Dy/R and Co/T (they remain approximately constant). The increase in the values of Dy/T and Co/T for magnets of group *1* with growth in the content of praseodymium in accordance with the model of double-sublattice ferromagnetic allows us to explain qualitatively the change in the TCI from 0 to + 0.01 %/°C [3, 4]. The magnetic properties of the $R_{n+1}(Fe, Co)_{3n+5}B_{2n}$, $R(Fe, Co)_2$, $R(Fe, Co)_3$ compounds are presented in the literature only for some R and in a limited range of concentrations of the component elements [9, 13 – 18]. For example, the anisotropy constant changes sign for the $Y(Co_{1-x}Fe_x)_4B$ and $Y(Co_{1-x}Fe_x)_3$ compounds at $x = 0.8$ and $x = 0.55$, respectively [16], i.e., is equal to zero at the mentioned concentrations. It can be seen from Table 2 that these values of x are very close to the content of iron in the $Y(Co_{1-x}Fe_x)_4B$ and $Y(Co_{1-x}Fe_x)_3$ phases.

Thus, due to the low anisotropy constant, inclusions of these phases can be centers of magnetization reversal and can reduce the coercive force of the material accordingly. In addition, the magnetization value in the considered compounds decreases with the content of dysprosium and can even be close to zero at room temperature (this is known as the compensation point) [15, 17]. In this case magnetization reversal is hindered, which promotes an increase in the value of the H_{cI} of the material. Since the disappearance of the $R_{n+1}(Fe, Co)_{3n+5}B_{2n}$ phases in magnets of group *1* ($x = 0.5$) causes growth in H_{cI}, the $R(Fe, Co)_3$ phase should have a lower negative action on the coercive force.

We have also shown for a $Pr_{n+1}Co_{3n+5}B_{2n}$ compound that the growth in n is accompanied by decrease in magnetization and in the Curie temperature [18]. This corresponds to elevation of the coercive force of the material bearing this phase. Indeed, we have established that the relative content of the RT_3B_2 phase in magnets of group *1* with $x = 0.2$ is higher than that of the $R_3T_{11}B_4$ phase as compared to magnets of group *1* with $x = 0.12$. Accordingly, the value of H_{cI} in magnets of group *1* ($x = 0.2$) is also higher. As for the $R(Fe, Co)_2$ phase, its negative influence on the value of H_{cI} in magnets is well known [4 – 7, 10]. It can be seen from Table 1 that as the content of praseodymium grows in magnets of group *2*, the ratio Co/T in the $R(Fe, Co)_2$ phase increases. It is known that in this case the Curie temperature and the magnetization of the $R(Fe, Co)_2$ compound decrease [15]. It is possible that this is connected with the rather high value of

H_{cI} (1700 kA/m) in this group of materials. It should be noted that the decrease in the absolute value of TCI in magnets of group *2* with growth in the content of praseodymium is not quite obvious from the data of Table 1 (Dy/R and Co/T in phase *A* are virtually identical at two concentrations of Pr). It can be assumed that in this case the contribution of phase *A* to the value of TCI is accompanied by a certain contribution of the other phases.

CONCLUSIONS

1. We have shown that alloying of magnetically hard materials of the Nd – Dy – Fe – Co – B system with praseodymium increases the residual induction of the material at simultaneous increase in the temperature stability of the magnets.

2. We have determined the phase composition of materials of the Nd – Dy – Fe – Co – B system and showed that it changes with growth in the concentration of praseodymium. The addition of praseodymium blocks the formation of boron-bearing phases of type $R_{n+1}(Fe, Co)_{3n+5}B_{2n}$ and makes it possible to increase the concentration of the primary magnetic phase *A* ($R_2(Fe, Co)_{14}B$).

3. The determined phase composition and its variation with growth in the concentration of praseodymium allow us to explain the results of magnetic measurements, i.e., the growth in the residual induction and coercive force and the increase in the temperature stability.

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