UDC 621.762.8:538.9:669.017.1

Gulyaeva T. V., PhD. Techn. Sciences

Zaporozhye National Technical University

# THE STUDY OF THE INFLUENCE OF EXTERNAL PRESSURE AND CARBON ON STRUCTURE AND PROPERTIES OF COMPACTS MADE ON THE BASIS OF RAPIDLY COOLED ALLOYS OF Nd-Fe-(B,C)-Cu-Ti

***Purpose.*** *Complex study of thermodynamic and physico-chemical conditions of phase formation in ready-made permanent magnets produced on the basis of systems Fe-Nd-B.*

***Methods of research:*** *metallographic, x-ray, x-ray spectral, magnetometer.*

***Results.*** *The influence of external pressure and insignificant amount of carbon (0,17...0.86 % at) on the structure and properties of permanent magnets made on the basis of Nd-Fe-B system doped with copper and titanium. For the manufacture of permanent magnets with high magnetic energy fast cooling products from the liquid state are used. To do this, the scales obtained by the LRS method were pressed in a mold and fused in a vacuum. The mold and bolts that hold them together are made of alloys posessing different coefficients of linear expansion. This method allows to achieve high pressure (≈ 1 GPa) during sintering. Sintering was performed in vacuum at a pressure of* $10^{-5}$ *mm of mercury and at a temperature of 1323 K, annealing occurred at a temperature of 823 K. Choice of these temperatures is due to the technological process of obtaining sintered magnets by the method of powder metallurgy.*

*As a result, sintered lump pacts with different degrees of hardness were obtained. The results of x-ray and x-ray spectral analyses showed that the main phases are* $Nd_2Fe_{14}B$, $Nd_{1.1}Fe_4B_4$, *Nd and* $NdCu_2$. *Metallographic studies have shown that phase* $Nd_2Fe_{14}B$ *under these conditions it does not have time to form into individual grains, and the size of the phase* $NdCu_2$ *is such that do not interfere with the movement of domain boundaries, in this case, the value of the coercive force of the sample remains almost constant and equals is 100...200 kA/m.*

*Statistical processing of the results of metallographic studies showed that the size of the paramagnetic phase* $NdCu_2$ *in the range of 0.3...2.2 μm. Comparison of mono-domain phase particle sizes* $Nd_2Fe_{14}B$ *(53.65 nm) with paramagnetic phase dimensions* $NdCu_2$ *showed that one particle accounts for 5 to 34 domains. Therefore, despite the fact that the structure of the compact paramagnetic phase* $NdCu_2$ *is present as well the areas of imperfections clusters (Suzuki-Kotrell cloud, dislocation nuclei, etc.), they are not an obstacle to the displacement of domain boundaries.*

***Scientific novelty.*** *It was found that the sintering of rapidly cooled alloy flakes* $Nd_{15,2}Fe_{75,5-x}C_xB_{6,6}Cu_{1,57}Ti_{1,38}$ *in conditions of high pressure of about 0.9 GPA at 1323 K promotes volume growth of phases* $Nd_2Fe_{14}B$, $NdCu_2$, *but it does not lead to their disintegration, which, in turn, negatively influence the physical properties of compacts (*$H_{Ci}$ *and Br).*

***Practical significance.*** *The obtained results are important for the further development of physical materials science of magnetically rigid materials and modern technology.*

***Key words:*** *sintering, heat pressure, annealing, the main hard magnetic phase, the phase with reduced metastability, coercive force.*

## Introduction

At present, the basis for the production of permanent magnets are transition metal alloys (T) with rare earth (R), or alloys in which the required level of magnetic properties is provided by the presence of intermetallic compounds such as $RT_5$, $R_2T_{17}$, $R_2T_{14}B$ [1]. Analysis of the literature suggests that the technology of production of compounds such as $R_2Fe_{14}B$ is constantly improving. Despite the fact that a sufficiently large number of magneto-porous compounds have been discovered, magnets based on the $Nd_2Fe_{14}B$ phase currently dominate.

One of the means of increasing the magnetic characteristics in materials based on alloys REM-PM is a comprehensive alloying and development of methods of primary processing of alloys to achieve high values of coercive force and residual induction in permanent magnets, which is an urgent problem of modern materials science. Today, the most promising system is the Nd-Fe-B. More common in use is the alloy “Neomax” based on the system Nd-Fe-B doped with cobalt, term, disposl, gadolinium and other heavy rare earth metals. Due to the high cost of the above-mentioned alloying elements, finished magnets have a high cost. When replacing these elements with cheaper ones such as carbon, titanium and copper, while maintaining and even increasing the magnetic characteristics, the cost of finished products is reduced several times. Therefore, the search for optimal concentrations of alloying elements and heat treatment

DOI 10.15588/1607-6885-2018-1-5

methods to obtain cheap high-energy magnets is currently relevant.

In the world and domestic literature, little attention is paid to the development of new methods for the manufacture of permanent magnets, namely, the use of one of the thermodynamic parameters of pressure to control the processes of crystallization and sintering of permanent magnets. Therefore, in this work, an integrated approach is applied during the study of the dependence of the coercive force on the doping and heat treatment of the nd-Fe-B magneto-porous alloy obtained by high-speed cooling, sintering into a compact at a pressure of up to 1 GPA. In this formulation of the problem, the problem of obtaining high-viscosity magnets is relevant and timely.

Thus, the aim of the work is to study the influence of the initial external pressure and alloying with carbon, copper and titanium alloy “Neomax” during sintering in the stressed state.

## Materials and methods studies

Quickly hardened tapes were used as initial materials for sintered compacts. Rapidly quenched ribbon was produced by the method spengemann melt in a vacuum plant rapid hardening “Tape-3” (NSC “KIPT”, Kharkiv). The chemical composition of the alloys obtained in the work are shown in table. 1.

**Table 1** – Chemical composition of the original alloy system Nd-Fe-(B,C)-Cu-Ti [2]

| № sample's | Chemical composition, % at. | | | | | |
|---|---|---|---|---|---|---|
| | Nd | Fe | C | B | Cu | Ti |
| 1 | 15,2 | 75,33 | 0,17 | 6,5 | 1,57 | 1,38 |
| 2 | 15,2 | 75,17 | 0,33 | 6,5 | 1,57 | 1,38 |
| 3 | 15,2 | 75,08 | 0,42 | 6,5 | 1,57 | 1,38 |
| 4 | 15,2 | 74,99 | 0,51 | 6,5 | 1,57 | 1,38 |
| 5 | 15,2 | 74,14 | 0,86 | 6,5 | 1,57 | 1,38 |

Sintering was carried out for the alloy $Nd_{15,2}Fe_{75,5\text{-}x}C_xB_{6,5}Cu_{1,5}Ti_{1,3}$ (x: 0.17...0.86 % at.) after compaction under mechanical pressure at different initial pressure $P_1$ = = 0.5 MPa, $P_2$ = 3 MPa, $P_3$ = 9.5 MPa (total pressure was 0.9 GPA + $P_i$ MPa) [3, 4]. The sintering took place under the process temperature to obtain permanent magnets based on Nd-Fe -B: $T$ = 1323 K (1050 °C) [5] for 1 h annealing at temperature of 823 K (550 °C) for 30 min.

The reliability of the obtained scientific results confirmed by the use of modern research equipment (optical microscope OLIMPYS IX-70, x-ray diffractometer Dron-3, a scanning electron microscope JEOL JSM-6360LA, vibration magnetometer, magnetometer vicious circle); the error in the reproduction of results is 3...10 %.

Thus, at this stage, a comprehensive study of the influence of external pressure and doping of fast cooling products on the structure and properties of finished compacts.

## Research result

The aim of this study is to study the influence of external pressure and during the sintering of rapidly cooled from the liquid state of amorphous and amorphous-crystalline flakes of alloys $Nd_{15,2}Fe_{75,5\text{-}x}C_xB_{6,6}Cu_{1,57}Ti_{1,38}$, on the structural-phase composition of compacts and their properties. The microstructure of the initial films is shown in Fig. 1.

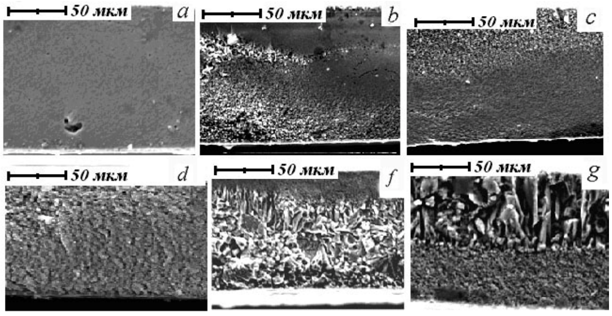

**Fig. 1.** The microstructure of the original alloy ribbons of the composition $Nd_{15,2}Fe_{75,5\text{-}x}C_xB_{6,6}Cu_{1,57}Ti_{1,38}$: *a* – 0.17% at. C; *b* – 0.33% at. C; *c* – 0.42% at. C; *d* – 0.51% at. C; *f* – 0.59% at. C; g – 0.86% at. C

The methods of metallographic and electron microscopic (Fig. 1), x-ray diffraction (Fig. 2) analyses of the investigated structure and phase composition of the films (flakes), which are obtained during spengemann.

It was found that the initial films (scales) have amorphous or amorphous crystal structure. The tendency to form an amorphous or micro crystalline state is manifested with an increase in the carbon content (see Fig. 2). Increase in titanium content from 0.15 % at. C. up to 1.38 % at. C. promotes dispersion of the structure of the original films. The main phase components of the original films in addition to the amorphous component is in phase $Nd_2Fe_{14}B$, $Nd_{1,1}Fe_4B_4$, Nd and x-phase, which is identified as phase $NdCu_2$, that exists in the system Nd-Cu, but at 66 % at. Cu, that is, for a given alloy phase $NdCu_2$ should be called by the classification of I. S. Miroshnichenko as a phase of limited metastability [6].

Obtained by spengemann film (flakes) was placed in a mold, compacted under pressure and spcala in a vacuum oven. The microstructure of the compacts obtained by sintering at a temperature $T$ = 1323 K (1050 °C) [5] and the initial pressure $P_1$ = 0.5 MPa, $P_2$ = 3 MPa, $P_3$ = 9.5 MPa (total pressure was 0.9 GPA + $P_i$ MPa), is highly heterogeneous and is represented in Fig. 3–7. It is seen that with increasing initial pressure density and homogeneity of compacts grow. Changing the carbon content helps to optimize the structure, it becomes more uniform. The grain of the main hard magnetic phase $Nd_2Fe_{14}B$become a crab-like shape (see Fig. 3).

Figures 4–7 show the microstructure of sintered compacts after annealing obtained with an electron microscope. As can be seen from the figure, there are white particles in the samples, their sizes range from 0.2 μm to 40 μm. The presence

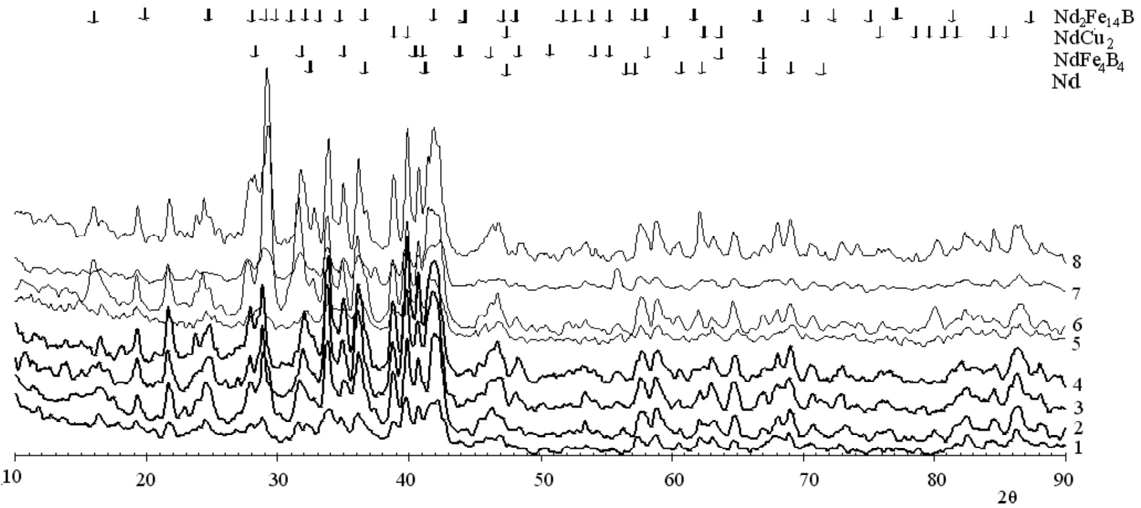

**Fig. 2.** A diffractogram obtained on a Dron-3-0 With K-α radiation from the scales of the alloys $Nd_{15,2}Fe_{75,5-x}C_xB_{6,5}Cu_{1,57}Ti_{1,38}$ (1, 5) and $Nd_{15,6}Fe_{76-x}C_xB_{6,8}Cu_{1,57}Ti_{0,15}$.(2–4, 6–8): 1–4 – contact surface; 5–8 – free surface; 1, 5 – 0.17% at. C; 2, 6 – 0.42% at. C; 3, 7 – 0,51% at. C; 4, 8 – 0.59 % at. C

of white particles is particularly evident in the compact, which was obtained at an initial pressure of 9.5 MPa (see Fig. 4 *c*, *g*). Scales were sintered with formation on borders of plates of particles of white color. Radiographically (see Fig. 9) and by means of micro-x-ray spectral analysis (JEOL JSM-6360 LA, see Fig. 5–7, table. 2–4) it was found that in addition to the stable phases that are formed in the alloy, there is also a phase that is identified as an intermetalid $NdCu_2$. In areas that had an amorphous structure before sintering (see Fig. 3 *c*, *g*), the average size of the inclusions of the $NdCu_2$ phase *is* ≈ 0.35 μm.

**Table 2 –** Phase composition of the compact № 4, which was baked at a pressure of 0.5 MPa

| № points | The content of elements, at. % | | | | | phase |
|---|---|---|---|---|---|---|
| | Nd | Fe | B | Cu | Ti | |
| 1 | 26,26 | 12 | 6,18 | 52,84 | 2,72 | $NdCu_2$ |
| 2 | 13,92 | 78,31 | 5,96 | 0,87 | 0,94 | $Nd_2Fe_{14}B$ |
| 3 | 14,56 | 76,86 | 5,85 | 1,24 | 1,51 | $Nd_2Fe_{14}B$ |
| 4 | 26,33 | 8,25 | 7,98 | 52,86 | 4,58 | $NdCu_2$ |

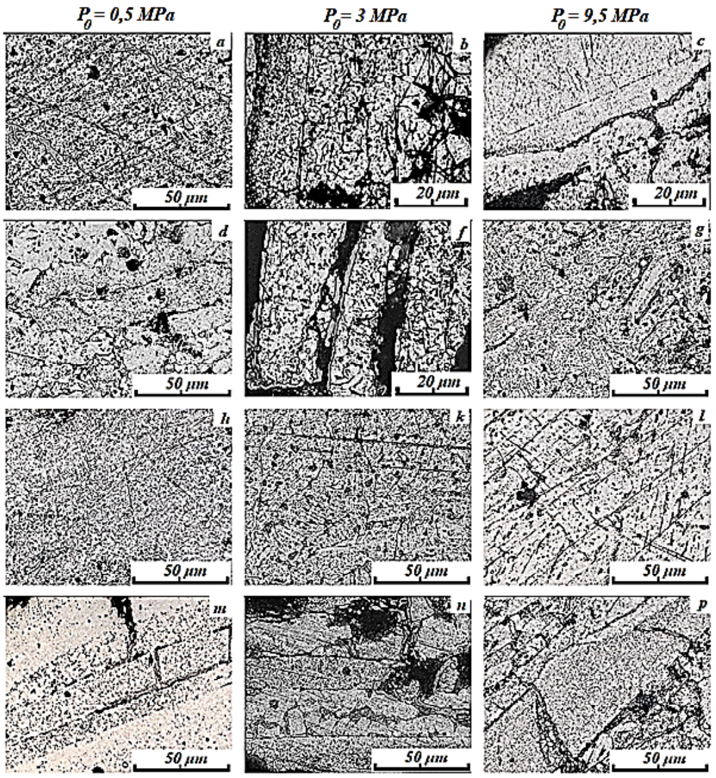

**Fig. 3.** The microstructure of sintered compacts of composition $Nd_{15,2}Fe_{75,5-x}C_xB_{6,5}Cu_{1,57}Ti_{1,38}$ after annealing for different initial pressure and content of carbon: *a*, *b*, *c* – № 1, *d*, *f*, *g* – № 2, *h*, *k*, *l* № 3, *m*, *n*, *p* – № 4

**Table 3** – The phase composition of the compact that was baked at an initial pressure of 3 MPa

| № points | The content of elements, at. % | | | | | phase |
|---|---|---|---|---|---|---|
| | Nd | Fe | B | Cu | Ti | |
| 1 | 24,87 | 6,68 | 17,02 | 42,58 | 8,85 | $NdCu_2$ |
| 2 | 0,8 | 97,19 | 0,89 | 0, | 0,92 | Fe |
| 3 | 21,49 | 4,84 | 1732 | 43,07 | 9,2 | $NdCu_2$ |
| 4 | 12,51 | 80,01 | 5,87 | 1,28 | 0,33 | $Nd_2Fe_{14}B$ |
| 5 | 13,81 | 80,33 | 5,86 | 0 | 0 | $Nd_2Fe_{14}B$ |

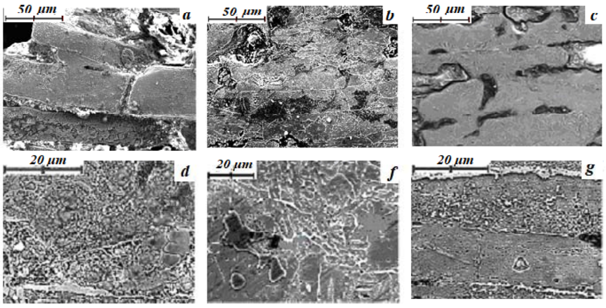

**Fig. 4.** The microstructure of the compacts of warehouse № 4, which was baked at different initial pressure and sintering temperature 1323 K, the annealing temperature 823 K: *a*, *d* – 0.5 MPa; *b*, *f* – 3 MPa; *c*, *g* – 9,5 MPa

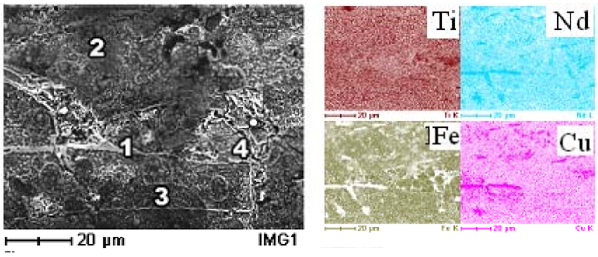

**Fig. 5.** The microstructure of the compact warehouse № 4, which was baked at $P_0$ = 0,5 MPa, *T* = 1323 K

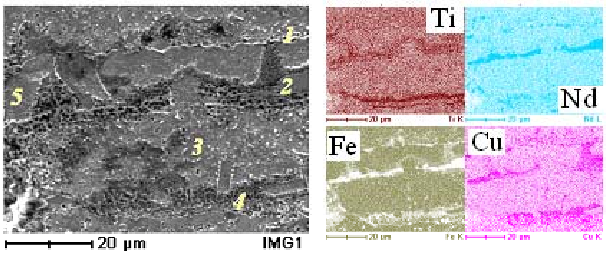

**Fig. 6.** The microstructure of the compact warehouse № 4, which was baked at $P_0$ = 3 MPa, *T* = 1323 K

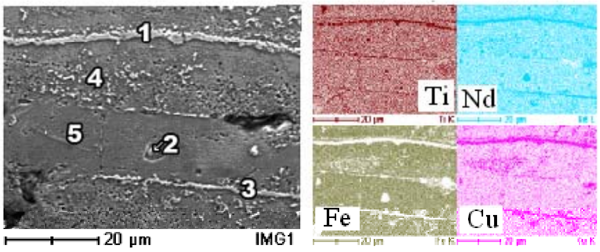

**Fig. 7.** The microstructure of the compact warehouse № 4, which was baked at $P_0$ =9.5 MPa, *T* = 1323 K

**Table 4** – the phase composition of the compact that was baked at an initial pressure of 9.5 MPa

| № points | The content of elements, at. % | | | | | phase |
|---|---|---|---|---|---|---|
| | Nd | Fe | B | Cu | Ti | |
| 1 | 30,68 | 0,06 | 10,74 | 49,88 | 8,64 | $NdCu_2$ |
| 2 | 89,18 | 4,21 | 5,17 | 1,25 | 0,19 | Nd |
| 3 | 28,38 | 0,28 | 9,92 | 55,27 | 6,15 | $NdCu_2$ |
| 4 | 13,81 | 77,66 | 5,86 | 1,37 | 1,3 | $Nd_2Fe_{14}B$ |
| 5 | 14,29 | 78,45 | 6,12 | 0 | 1,14 | $Nd_2Fe_{14}B$ |

To clarify the phase composition, a local chemical analysis of compacts using an electron microscope (JEOL JSM-6360 LA) was carried out, the distribution of elements (C, Ti, Cu, Fe, Nd) on the surface was studied (see Fig. 4–6, table. 2–4). Places with a high concentration of elements are characterized by the highest color saturation (dark). This was most clearly revealed in the samples obtained under a pressure of 9.5 MPa + 0.9 GPA (see Fig. 6). On microphotographs (see Fig. 6) it can be seen that in places of high concentration of copper there is also a high concentration of Nd and Te. This fact suggests that the phase of the white color ($NdCu_2$) is complex and contains Nd, Cu, Ti, and iron in its composition is almost absent (see Fig. 4–6).

From the above, it follows that with an increase in the primary pressure at which the compaction takes place in the mold with subsequent sintering (at a constant temperature and sintering time), firstly, the density of the primary compact increases, and, secondly, the particle size distribution of the white phase ($NdCu_2$) shifts towards smaller sizes, but the probability of the existence of particles with sizes more than 0.5 μm remains large, even in compacts with amorphous scales (see Fig. 3 *g*).

The dependence of the grain diameter of this phase on the pressure at which the compacts were joined is illustrated in Fig. 8. The figure shows that the average diameter of the granules depends on the pressure at which Spravce CD, law $D \sim P^{-1}$.

The results of x-ray spectral analysis were confirmed by x-ray structural analysis (see Fig. 9). From the

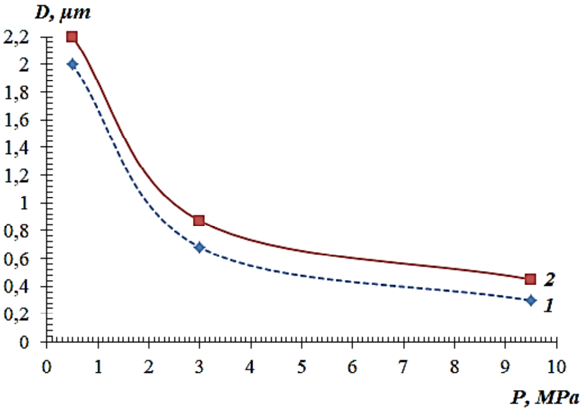

**Fig. 8.** The dependence of the average diameter of grains of the phase of $NdCu_2$ from the initial external pressure after sintering (1) and after annealing (2)

diffractograms it can be seen that when the external pressure changes, the maxima are redistributed in the direction of increasing the main magneto-rigid phase $Nd_2Fe_{14}B$ and paramagnetic phases$NdCu_2$.

The analysis of diffractograms showed that the main phase components of the compacts both after sintering and after annealing are: the main magneto-rigid phase $Nd_2Fe_{14}B$, paramagnetic phase $NdCu_2$, pure neodymium and phase. $Nd_{1,1}Fe_4B_4$. For paramagnet phase of $NdCu_2$, we calculated parameters a,b,c and the degree tetravalent *s*/*a*, *a*/*b*, *b*/*c*. The results are presented in Fig. 10, tabular data of these parameters – in table. 5.

As seen in Fig. 10 and table. 5, parameter a and $NdCu_2$ phase volume with increasing initial external pressure is almost unchanged and is approximately equal to the table value. Parameter with increasing initial pressure decreases and becomes less than the table value, and the parameter b increases and becomes greater than the table value. This can be explained by the fact that titanium, which enters the phases, with an increase in the initial seal and further heat treatment has the ability to change its position in the lattice phase $NdCu_2$ and thereby change the parameters *b*, *c*.

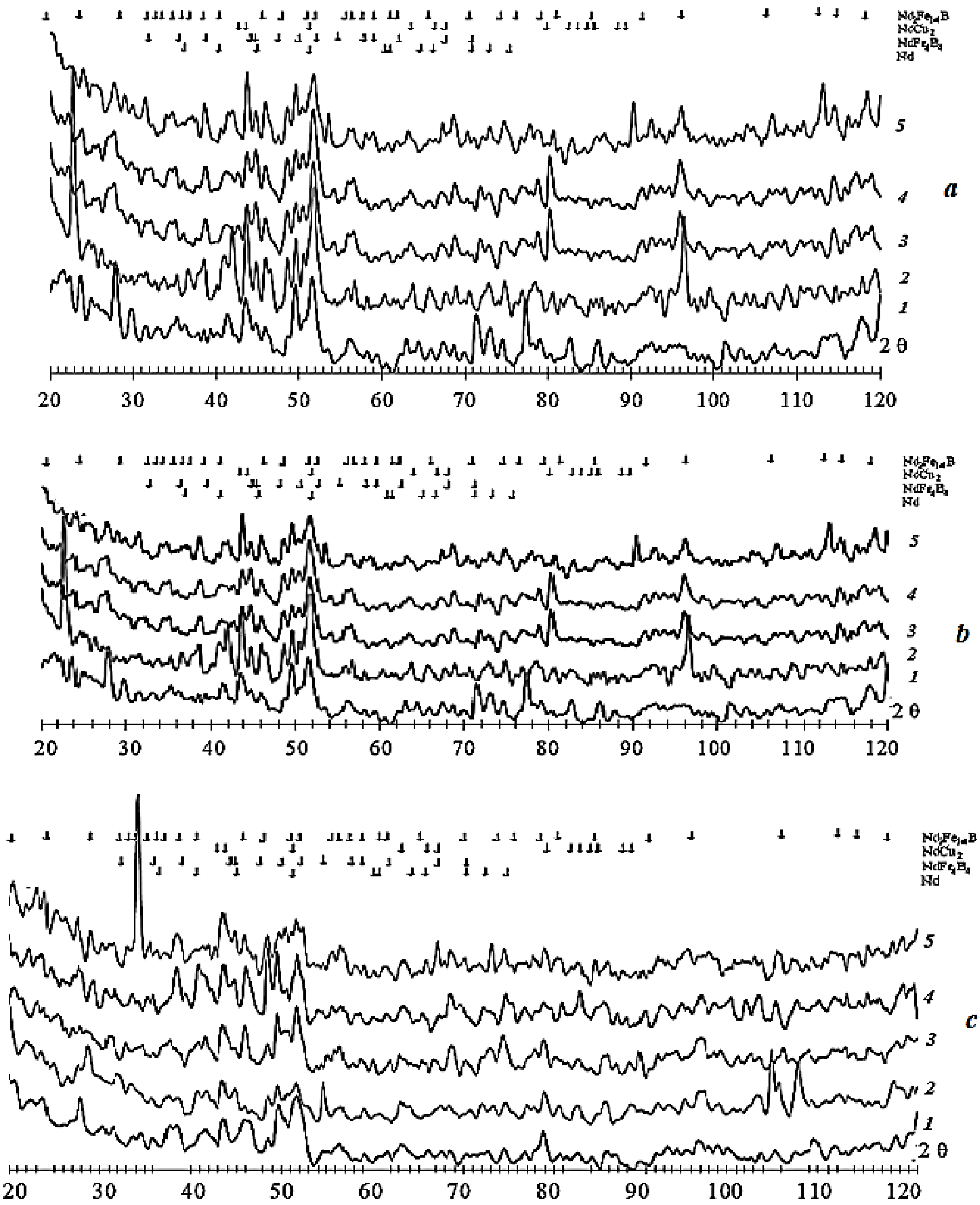

**Fig. 9.** Diffractograms obtained on the drone-3-0 in Co K-α radiation from sintered compacts $Nd_{15,2}Fe_{75,5\text{-}x}C_xB_{6,5}Cu_{1,57}Ti_{1,38}$ after annealing: *a* – 0.5 MPa, b –3MPa, *c*– 9.5 MPa; 1 – 0.17% at. C; 2 – 0.33% at. C; 3 – 0.42% at. C; 4 – 0.51% at. C; 5 – 0,86 % at. C

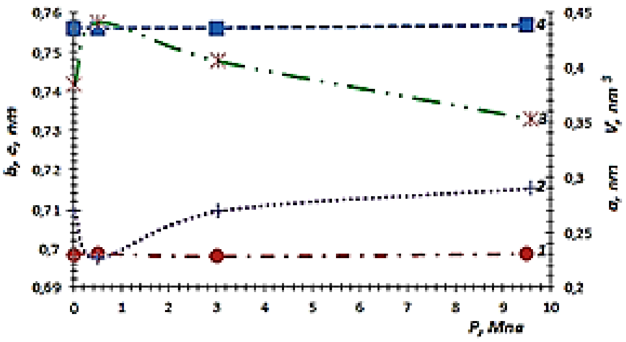

**Fig. 10.** Dependence: lattice parameters *a* (4), *b* (2), *c* (3), volume *V* (1) $NdCu_2$ phase on initial pressure for sintered compact № 3

**Table 5** – Table values of parameters *a, b, c* for $NdCu_2$ phase

| *a, nm* | *b, nm* | *c, nm* | *V, nm³* |
|---|---|---|---|
| 0,4387 | 0,71 | 0,74 | 0,23 |

In the future, the sintered compacts was investigated on a vibrating magnetometer for determining the values of the coercive force $H_C$ and the residual induction Br of the samples and was obtained the experimental curves of the hysteresis (Fig. 11). Calculations of working points showed that the values of the magnetic energy of the alloys $Nd_{15,2}Fe_{75,5-x}C_xB_{6,6}Cu_{1,57}Ti_{1,38}$, that sintered at a temperature of 1323 K and an initial pressure of 0.5...9.5 MPa, up to 2...10 kJ/m³, which is not significant.

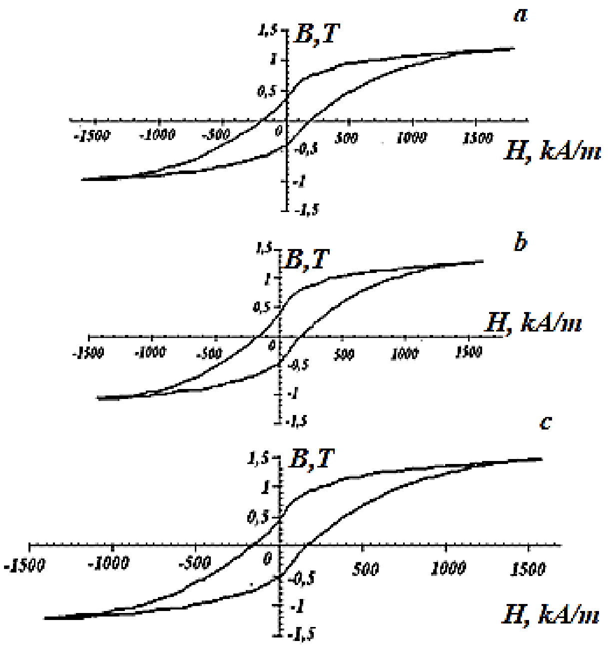

**Fig.11.** Loop gesturess CD, which was made from alloy № 4 at various initial pressures: $P_0$ = 0,5 MPa; *b* – $P_0$ = 3 MPa; *c* – $P_0$= 9.5 MPa

Studies have shown that the highest value of the coercive force before annealing is 200 kA / m at an external initial pressure of 3 MPa and a carbon content of 0.51% at., and with an increase in the initial pressure to 9.5 MPa, the value of the coercive force practically does not increase. After annealing, the coercive force values decrease slightly and range from 108 kA / m ($P_0$ = 0.5 MPa, C = 0.33 % at.) up to 180 kA/m ($P_0$ = 9.5 MPa, *C* = 0.51 % at.). The decrease in the value of the coercive force can be explained by the increase in size (see Fig. 8) paramagnet phase of $NdCu_2$. Also from Fig.12 it is seen that the maximum value of the coercive force obtained for 0.51% of the carbon stock.

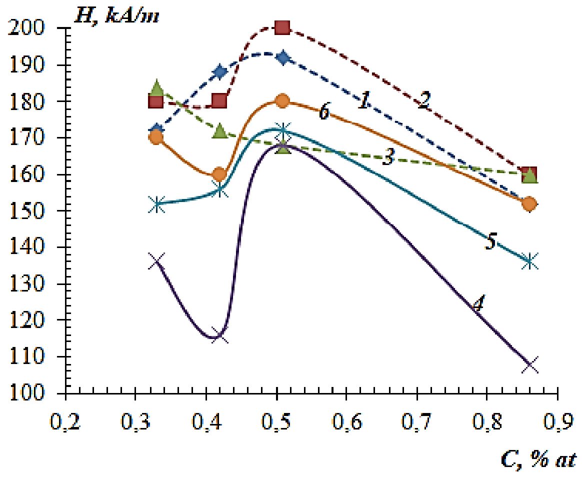

**Fig. 12.** Dependence of the coercive force of compacts on the carbon content before annealing (1–3) and after annealing (4–6) for different initial pressure: 1, 4 – $P_0$ = 0.5 MPa; 2, 5 – $P_0$ = 3 MPa; 3, 6 – $P_0$ = 9.5 MPa

## The discussion of research results

The analysis of experimental data allows us to state that the rapid cooling of the alloy $Nd_{15,2}Fe_{75,5-x}C_xB_{6,6}Cu_{1,57}Ti_{1,38}$ in the quenching products (flakes) are formed of amorphous and crystalline phases. It is established that the main crystalline phases are $Nd_2Fe_{14}B$, $Nd_{1,1}Fe_4B_4$, clean Nd phase, which is identified as intermetal $NdCu_2$.

It is known that $Nd_2Fe_{14}B$formed in the system Nd-Fe-B at protectionhow reaction. In case of rapid cooling from the liquid state, the phase $Nd_2Fe_{14}B$ crystallizes directly from the liquid, bypassing the equilibrium transformation. In addition to phase $Nd_2Fe_{14}B$ in this alloy, a phase of type NdCu2 is formed, which exists in the Nd-Cu system with an atomic composition of Cu + 33 at. % Nd [7], but in the investigated alloy copper content does not exceed 1.6 % at., and therefore the $NdCu_2$ phase, according to the equilibrium state diagram, cannot be formed in a given alloy. It is known that in the Fe-Cu system under certain conditions (the presence of carbon more than 0.3 at.% ), complete stratification in the fluid may occur [8]. Therefore, the probability of formation of micro domains, which are enriched in copper in the liquid alloy, a large because carbon stabilizes the region nesman of copper with the main alloy element, iron. Consequently, during fast cooling of the alloy from the liquid state intermetall $NdCu_2$ can be obtained as metastable phase on the basis of mccaughrean that is enriched with copper and other elements. For Miroshnichenko.C. [6], phases $Nd_2Fe_{14}B$, $NdCu_2$ you can call phases with limited metastablity.

The resulting flakes of the composition $Nd_{15,2}Fe_{75,5-x}C_xB_{6,6}Cu_{1,57}Ti_{1,38}$ placed in the mold and pressed using a mechanical press, then spcala in a vacuum oven at a temperature of 1323 K, the annealing was at a temperature of 823 K. These Temperatures were chosen according to the technological process of production of anisotropic permanent magnets. As noted earlier [9], the material of the clamp and mounting bolts was selected in such a way that it was possible to obtain an additional “thermal” pressure up to 1 GPa. Under high pressure conditions, stresses occur that accelerate diffusion processes in the sample [10, 11]. Thus, on the one hand the volume growth of phases is carried out $Nd_2Fe_{14}B$ and $NdCu_2$ and at the same time their resistance will be stable, since high pressure displaces the points of phase equilibrium in the region of high temperatures, in accordance with the law of Clapeyron-Clausius. As seen in Fig. 4, the size of the phase $NdCu_2$ with increasing primary pressure from 0.5 MPa to 9.5 MPa decreases in 6...7 times. Annealing promotes grain growth paramagnet phase of $NdCu_2$ in 1,3 times on average.

Since the phase $Nd_2Fe_{14}B$ under these conditions, it does not have time to form into individual grains, and The ndcu2 phase sizes are such that they do not interfere with the movement of domain boundaries, in this case, the value of the coercive force of the sample remains almost constant. To explain the fact, let’s calculate the size of the domain for the main hard magnetic phase $Nd_2Fe_{14}B$ for the case of absolute one-domain [12,13]. We assume that the domain has the shape of a sphere ($N_R = 4\pi/3$). Then:

$$R_0 = \frac{1}{I_s}\sqrt{\frac{5A}{2N_R}} = 53{,}65 \text{ нм},$$

where $I_s = 1.61 \cdot 10^4$ Gs – magnetic saturation,

$A = 1.25 \cdot 10^{-6}$ erg / cm-exchange interaction constant.

The width of the domain wall is $\delta = 5.24$ nm. That is, the total size of the domain $\approx$ 58.98 nm. In comparison with the size paramagnet phase $NdCu_2$ it can be seen that one particle accounts for 5 to 34 domains. Therefore, despite the fact that the structure of the CD is present in the paramagnetic phase of $NdCu_2$ and areas of clusters of imperfections (cloud Suzuki-Cottrell, the core of the dislocation, etc.), they are not an obstacle for the displacement of domain boundaries.

Thus, in order to optimize the structure of finished products and increase the magnetic characteristics, it is advisable to change the heat treatment modes in the future, namely, to reduce the temperature and increase the sintering time.

## Conclusion

1. Rapid cooling of alloys$Nd_{15,2}Fe_{75,5-x}C_xB_{6,6}Cu_{1,57}Ti_{1,38}$ leads to the formation of metastable phases$Nd_2Fe_{14}B$ i $NdCu_2$,bypassing the equilibrium of the reaction.

2. Sintering of quickly cooled flakes $Nd_{15,2}Fe_{75,5-x}C_xB_{6,6}Cu_{1,57}Ti_{1,38}$ in conditions of external pressure up to 1 HPa at a temperature of $t = 1323$ K promotes volumetric growth of the phase$NdCu_2$, but it does not lead to its disintegration, which in turn negatively affects the value of the coercive force and the residual induction of compacts.

3. In conditions of high external pressure, the maximum coercive force falls on the composition of alloys with carbon in the amount of 0.33...0.51 at. % , due to the maximum diffusion of C, Cu, Those in the field of imperfections of the structure and interference with the movement of domain boundaries.

## References

1. Сергеев В. В. Магнитотвердые материалы / В. В. Сергеев, Т. И. Булыгина. – М. : Энергия, 1980. – 185 с.
2. Гуляєва Т. В. Визначення раціонального легування та режимів спікання економно-легованих швидко охолоджених сплавів Nd-Fe-B-Ti-C-Cu для підвищення магнітних характеристик : автореф. дис. на здобуття наук. ступеня канд. тех. наук. : спец. 05.16.01 «Металознавство та термічна обробка металів» / Т. В. Гуляєва. – Запоріжжя, 2015. – 24 с.
3. Брехаря Г. П. Влияние меди на свойства постоянных магнитов, изготовленных на основе сплава $Fe_{76}Nd_{16}B_8$ / Г. П. Брехаря, Е. А. Харитонова // Металлофизика и новейшие технологии. – 2013. – 35, № 3. – С. 367–375.
4. Брехаря Г. П. Вплив стискаючих напружень, що прикладаються в процесі спікання аморфних лусочок складу $Nd_{33}Fe_{63,34}B_{1,1}Cu_{1,5}Ti_{1,0}C_{0,06}$, на структуру і магнітні властивості компактів / Г. П. Брехаря, Т. В. Гуляєва, Т. В. Калниш // Вісник дніпропетровського університету, сер. Фізика. Радіоелектроніка. – Дніпропетровськ : ДНУ. – Вип. 17. – Т. 18. – № 2. – 2010. – С. 92–97.
5. Buschow K. H. J. New permanent magnet materials / K. H. J. Buschow // Materials Science Reports. – 1986. – Vol. 1. – Issue 1. – P. 1–63.
6. Мирошниченко И. С. Закалка из жидкого состояния / И. С. Мирошниченко. – М. : Металлургия, 1982. – 168 с.
7. Лякишев Н. П. Диаграммы состояния двойных металлических систем : справочник в 3 т. / [Под общ. ред. Н. П. Лякишева]. – М. : Машиностроение, 1997. – Т. 2. – 1997. – 1024 с.
8. Хансен М. Структуры двойных сплавов: справочник : в 2-х т. / М. Хансен, К. Андерко [пер. П. К. Новик и др.]. – М. : Металлургиздат, 1962. – 1488 с.: ил.
9. Relevant issues of development and modernization of modern science: the experience of countries of Eastern Europe and prospects of Ukraine: monograph / edited by authors. – Riga, Latvia : Izdevnieciba “Baltija Publishing”, 2018. –P. 122-143. DOI: http://dx.doi.org/10.30525/978-9934-571-26-8_7
10. Шьюмон П. Дифузия в твердых телах / П. Шьюмон; [пер. с англ.]. – М.: Металлургия, 1966. – 195с.

11. Дудоров А. Е. Уравнения динамики и кинетики дислокаций при высоких скоростях пластической деформации / А. Е. Дуров, А. Е. Майер // Вестник Челябинского государственного университета, Физика. – 2011. – Вып. 12. – № 39 (254). – С. 48–56.
12. Вонсовский С. В. Магнетизм / С. В. Вонсовский. – М. : Наука, 1971. – 1032 с.
13. Кринчик Г. С. Физика магнитных явлений / Г. С. Кринчик. – М. : Изд-во Моск. ун-та, 1976. – 262 с.

*Одержано 05.09.2018*

### Гуляєва Т. В. Про дослідження впливу зовнішнього тиску та вуглецю на структуроутворення та властивості компактів, що спечені на основі швидко охолоджених сплавів Nd-Fe-(C,B)-Cu-Ti

***Мета роботи.*** *Комплексне дослідження термодинамічних та фізико-хімічних умов утворення фаз в готових постійних магнітах, що виготовляються на основі систем Fe-Nd-B.*

***Методи дослідження****: металографічний, рентгенографічний, рентгеноспектральний, магнітометричний.*

***Отримані результати.*** *Досліджується вплив зовнішнього тиску та незначної кількості вуглецю (0,17...0,86 % ат) на структуру та властивості постійних магнітів, які виготовлені на основі системи Nd-Fe-B, що леговані міддю та титаном. Для виготовлення постійних магнітів з високою магнітною енергією використано продукти швидкого охолодження з рідкого стану. Для цього лусочки, що отримані методом ЗРС, пресували у прес-формі та спікали у вакуумі. Прес-форма та болти, що їх скріплюють, виготовлені зі сплавів, у яких різні коефіцієнти лінійного розширення. Цей метод дає змогу досягти високого тиску (≈ 1 ГПа) під час спікання. Спікання виконували у вакуумі під тиском* $P = 10^{-5}$ *мм. рт. ст. та при температурі 1323 К, відпал відбувався при температурі 823 К. В результаті було отримано спечені компакти з різним ступенем ущільненості.*

*Результати рентгенографічного та рентгеноспектрального аналізів показали, що основними фазами є фази* $Nd_2Fe_{14}B$, $Nd_{1.1}Fe_4B_4$, *Nd та* $NdCu_2$. *Металографічні дослідження показали, що фаза* $Nd_2Fe_{14}B$ *в даних умовах не встигає сформуватися в окремі зерна, а розміри фази* $NdCu_2$ *такі, що не перешкоджають руху границь доменів, то в цьому випадку значення коерцитивної сили зразка залишається практично постійним і складає 100..200 кА/м.*

*Статистична обробка результатів металографічних досліджень показала, що розміри парамагнітної фази* $NdCu_2$ *коливаються у межах 0,3...2,2 мкм. Порівняння розмірів монодоменої частинки фази* $Nd_2Fe_{14}B$ *(53,65 нм) з розмірами парамагнітної фази* $NdCu_2$ *показали, що на одну частинку припадає від 5 до 34 доменів. Тому, не дивлячись на те, що у структурі компакту присутня парамагнітна фаза* $NdCu_2$ *та зони скупчень недосконалостей (хмара Сузукі-Котрелла, ядра дислокації та ін.), вони не є перешкодою для зміщення границь доменів.*

***Наукова новизна.*** *Встановлено, що спікання швидко охолоджених лусочок сплавів* $Nd_{15,2}Fe_{75,5-x}C_xB_{6,6}Cu_{1,57}Ti_{1,38}$ *в умовах високого тиску порядку 0,9 ГПа при температурі 1323 К сприяє об'ємному зростанню фаз* $Nd_2Fe_{14}B$, $NdCu_2$, *але не призводить до їх розпаду, що, в свою чергу, негативно впливає на фізичні властивості компактів (*$H_{Ci}$ *і Br).*

***Практична цінність.*** *Одержані в роботі результати мають важливе значення для подальшого розвитку фізичного матеріалознавства магнітожорстких матеріалів та сучасної техніки.*

***Ключові слова:*** *спікання, «термічний» тиск, відпал, основна магнітожорсткі фаза, фаза з обмеженою метастабільністю, коерцитивна сила.*

### Гуляева Т.В. Об исследовании влияния внешнего давления и углерода на структурообразование и свойства компактов, спеченных на основе быстро охлажденных сплавов Nd-Fe-(C, B)-Cu-Ti

***Цель работы.*** *Комплексное исследование термодинамических и физико-химических условий образования фаз в готовых постоянных магнитах, изготавливаемых на основе системы Fe-Nd-B.*

***Методы исследования:*** *металлографический, рентгенографический, рентгеноспектральный, магнитометрический.*

***Полученные результаты.*** *Исследуется влияние внешнего давления и незначительного количества углерода (0,17...0,86 % ат) на структуру и свойства постоянных магнитов, изготовленных на основе системы Nd-Fe-B, легированных медью и титаном. Для изготовления постоянных магнитов с высокой магнитной энергией использовано продукты быстрого охлаждения из жидкого состояния. Для этого чешуйки, полученные методом ЗЖС, прессовали в пресс-форме и спекали в вакууме. Пресс-форма и скрепляющие ее болты изготовливают из сплавов с различными коэффициентами линейного расширения. Этот метод позволяет достичь высокого давления (≈ 1 ГПа) при спекании. Спекание выполняли в вакууме под давлением* $10^{-5}$ *мм. рт. ст. и при температуре 1323 К, отжиг происходит при температуре 823 К. Выбор данных температур обусловлен технологическим процессом получения спеченных магнитов методом порошковой металлургии.*

*В результате было получено спеченные компакты с разной степенью уплотнённости. Результаты рентгенографического и рентгеноспектрального анализов показали, что основными фазами являются фазы*

*$Nd_2Fe_{14}B$, $Nd_{1.1}Fe_4B_4$, Nd и $NdCu_2$. Металлографические исследования показали, что фаза $Nd_2Fe_{14}B$ в данных условиях не успевает сформироваться в отдельные зерна, а размеры фазы $NdCu_2$ такие, которые не препятствуют движению границ доменов, то в этом случае значение коэрцитивной силы образца остается практически постоянным и составляет 100…200 кА/м.*

*Статистическая обработка результатов металлографических исследований показала, что размеры парамагнитной фазы $NdCu_2$ колеблются в пределах 0,3...2,2 мкм. Сравнение размеров монодоменои частицы фазы $Nd_2Fe_{14}B$ (53,65 нм) с размерами парамагнитной фазы $NdCu_2$ показали, что на одну частицу приходится от 5 до 34 доменов. Поэтому, несмотря на то, что в структуре компакта присутствует парамагнитная фаза $NdCu_2$ и зоны скоплений несовершенств (облако Сузуки-Котрелла, ядра дислокации и др.), они не являются препятствием для смещения границ доменов.*

***Научная новизна.** Установлено, что спекание быстро охлажденных чешуек сплавов $Nd_{15,2}Fe_{75,5-x}C_xB_{6,6}Cu_{1,57}Ti_{1,38}$ в условиях высокого давления порядке 0,9 ГПа при температуре 1323 К способствует объемному росту фаз $Nd_2Fe_{14}B$, $NdCu_2$, но не приводит к их распаду , что, в свою очередь, негативно влияет на физические свойства компактов ($H_{Ce}$ и $B_r$).*

***Практическая ценность.** Полученные в работе результаты имеют важное значение для дальнейшего развития физического материаловедения магнитожестких материалов и современной техники.*

***Ключевые слова:** спекание, «термическое» давление, отжиг, основная магнитожестких фаза, фаза с ограниченной метастабильнистю, коэрцитивная сила.*