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STRUCTURE, SOFT MAGNETIC PROPERTIES OF Fe-BASED MAGNETIC COMPOSITES AND ITS PRACTICAL APPLICATION

(Communicated by Corresponding Member Valery M. Fedosyuk)

Abstract. A multi-stage technique for applying insulating coatings to metal powder particles has been developed in order to create a new class of Fe-based soft magnetic materials with improved characteristics. The density value calculated from the data of X-ray diffraction analysis is approximately 3 % higher than the experimentally measured values, which are 7.4–7.45 g/cm^3. The low porosity of the composites is confirmed by the SEM and EDX results. The proposed method of encapsulation of iron powder with an oxide layer is a highly economical method for applying coatings of various chemical compositions to metal powders, and can be widely used in practice to obtain electrical materials. Comprehensive studies of the properties of the obtained samples of powder composite materials based on ABC100.30 iron, the particles of which are encapsulated with phosphorus oxide, have been carried out. It has been established that in a field of 1.5 T, the losses at a frequency of 1 kHz decrease 10 times with an increase in the thickness to 30 nm. The synthesized materials are recommended for use in the development of various types of high-frequency electric motors, generators, chokes, magnetic circuits and electrodes for high-frequency welding and other applications.

Keywords: Fe-based soft magnetic composites, insulating coatings, crystal structure, morphology, electromagnetic losts, magnetic permeability

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СТРУКТУРА, ЭЛЕКТРОМАГНИТНЫЕ СВОЙСТВА Fe-СОДЕРЖАЩИХ МАГНИТНЫХ КОМПОЗИТОВ И ИХ ПРАКТИЧЕСКОЕ ПРИМЕНЕНИЕ

(Представлено членом-корреспондентом В. М. Федосюком)

Аннотация. Разработана многостадийная методика нанесения изоляционных покрытий на частицы металлических порошков с целью создания нового класса магнитомягких материалов с улучшенными характеристиками. Величина плотности, рассчитанная из данных рентгеноструктурного анализа, примерно на 3 % больше, чем непосредственно измеренных значений, которые составляют 7,4–7,45 г/см^3. Низкая пористость композитов подтверждается результатами SEM и EDX. Предложенный метод капсулирования порошка железа оксидным слоем является высокоэкономичным методом для нанесения покрытий различного химического состава на металлические порошки и может быть широко использован в практике для получения электротехнических материалов. Проведены комплексные исследования свойств полученных образцов порошковых композиционных материалов на основе железа ABC100.30, частицы которого капсулированы оксидом фосфора. Установлено, что в поле 1,5 Тл потери при частоте 1 кГц снижаются в 10 раз. Синтезированные материалы рекомендуются для использования при разработке различного рода высокочастотных электродвигателей, генераторов, дросселей, магнитопроводов и электродов для ВЧ сварки и других применений.

Ключевые слова: Fe-содержащие магнитомягкие композиты, изолирующие покрытия, кристаллическая структура, морфология, электромагнитные потери, магнитная проницаемость

Для цитирования. Структура, электромагнитные свойства Fe-содержащих магнитных композитов и их практическое применение / О. Ф. Демиденко [и др.] // Докл. Нац. акад. наук Беларуси. – 2023. – Т. 67, № 6. – С. 508–516. https://doi.org/10.29235/1561-8323-2023-67-6-508-516

Introduction. Recently, world has been actively developing electric transport and related areas through the creation and production of materials with advanced characteristics for electric machines for various purposes, which is a consequence of the actualization of the climate agenda, in particular, the policy to reduce greenhouse gas emissions into the atmosphere. Economic stimulation of resource conservation, development and implementation of environmentally friendly technologies and equipment in industry, energy, agriculture and transport are priority areas for ensuring environmental security in many states [1; 2]. For this reason, the used electric machines are subject to special requirements, such as high efficiency, high torque and power density, a wide speed range, maintainability and reliability [3].

The desire to improve the energy efficiency and economy of materials constantly forces researchers to improve their properties. Soft magnetic materials are an important subclass of magnetic materials that are able to quickly switch their magnetic polarization in conditions of weak applied magnetic field and have low energy losses, which finds important application in the creation of electric motors, transformers, inductors or sensors [4; 5].

The growing demand for miniaturization of electronic components coupled with higher operating frequencies calls for new functional materials that combine reduced core EMI, higher power density and well-defined performance at elevated temperatures. Thus, advanced research and development continues to expand the range of new available soft magnetic materials with improved properties suitable for specific applications [6–8].

An information analysis of a large amount of data on the study of technological schemes for obtaining powder magnetically soft materials and the study of the magnetic and physico-mechanical properties of experimental samples shows that one of the promising directions for the further development of such materials is the creation of materials based on nanocrystalline powders [9]. A unique combination of magnetic properties is observed in nanocrystalline alloys based on the Fe-Si-B system of the “Finemet” type ($Fe_{73.5}Si_{13.5}B_9Nb_3Cu_1$) [10] with a mixed amorphous-crystalline structure and a grain size of ~10 nm, which makes it possible to obtain saturation induction not less than 1.0 T and high initial magnetic permeability. The absence of a domain structure ensures a low coercive force (5–10 A/m), a correspondingly small area of the hysteresis loop, and, as a consequence, low magnetization reversal losses in such materials.

Currently, soft magnetic composite materials are gradually replacing the widely used laminated electrical steel for the magnetic circuits of electrical devices manufacture. Soft magnetic composites are a special class of ferromagnetic materials for electromagnetic applications, consisting of ferromagnetic particles isolated from each other, which leads to a decrease in eddy currents and, consequently, a decrease in overall energy losses [11]. The eddy current that occurs in the core leads to losses in the form of heating. To reduce these eddy current losses, an insulating film must be formed on the surface of the soft magnetic material particles to prevent conduction between the particles. In the literature, there are similar studies of the formation of insulating coatings on the surface of particles based on resins, phosphates, oxides, and borates deposited by various methods, such as oxidation, vapor and liquid phase deposition, mechanical alloying, and others chemical methods[1] [12; 13]. An analysis of the available

[1] Ferromagnetic particle surface coating layers for obtaining soft magnetic composites (SMCs): patent no WO2018035595A1 / M. V. F. da Luz, R. Carlson, N. Sadowski, G. Hammes, V. Drago, G. Tontini, A. N. Klein, C. Binder, J. B. Rodrigues Neto, N. J. Batistela, M. T. Daros, A. I. Ramos Filho, C. Schmitz, R. D. A. Elias. 2018.

literature data on the methods for encapsulating a metal powder with a thin oxide layer showed that they do not provide a sufficiently high-quality coating and are ineffective. The main requirements for coatings can be formulated as follows: the coating layer must be continuous and uniform in thickness; the resistivity of the layer should be 10^2–10^6 Ohm cm.

Compared to electrical steel, which is a sheet material, the use of soft magnetic composites in the form of encapsulated powder results in virtually no material loss at the manufacture of electromagnetic components, compared with the standard technology, in which up to 40 % of electrical steel is remelted. One of the important advantages is that such materials make it possible to design and create a much greater variety of forms of components of electrical products, including the 3D printing method, while maintaining isotropic magnetic properties [14; 15].

An important factor is the increase in the efficiency of manufacturing products and the reduction of technological costs. Powder composite materials are characterized by thermal and magnetic isotropy. Many new electrical machines with 3D-flow control are only possible due to isotropic properties. In addition, the powder is pressed directly into the desired shape and then heat treated. This means that components with complex geometries can be manufactured easily and efficiently.

The purpose of this work was to study the effect of the thickness of the encapsulating insulating layer based on phosphorus oxide deposited on pure iron powder particles on the magnitude of the electromagnetic characteristics.

Materials and experimental methods. Taking into account the insulating coating requirements outlined above, a new method for applying oxide coatings to the surface of metallic iron particles has been developed by combining the encapsulation methods listed above, which makes it possible to obtain a given composition with given magnetic parameters and resistivity, which can be widely used in practice. The essence of the method consists in preliminary joint mixing of the powder with the addition of water-soluble salts and their thermal decomposition to oxides, followed by the formation of oxide coatings on the iron surface (Fig. 1).

In order to obtain encapsulated materials with good electrical characteristics, it is very important that the initial metal powders have a high chemical purity. These requirements are met by water-atomized iron powder ABC100.30, which contains a small amount of carbon 0.002 wt. % and oxygen 0.05 wt. %. Studies of the granulometric composition of ABC100.30 iron powders were carried out using a laser analyzer "Mastersizer-2000" from Malvern (Great Britain), and its results are presented in Table 1. The analysis of the given data shows that 83 % of the volume of the entire powder are particles with sizes from 20 to 178 microns.

T a b l e 1. **Granulometric composition of iron powders ABC100.30**

Particles size (mkm)	Volume (%)	Particles size (mkm)	Volume (%)
0.100		7.179	
0.291	0.02	20.895	2.73
0.847	1.25	60.822	27.40
2.466	2.23	177.040	56.08
7.179	2.19	515.325	8.11

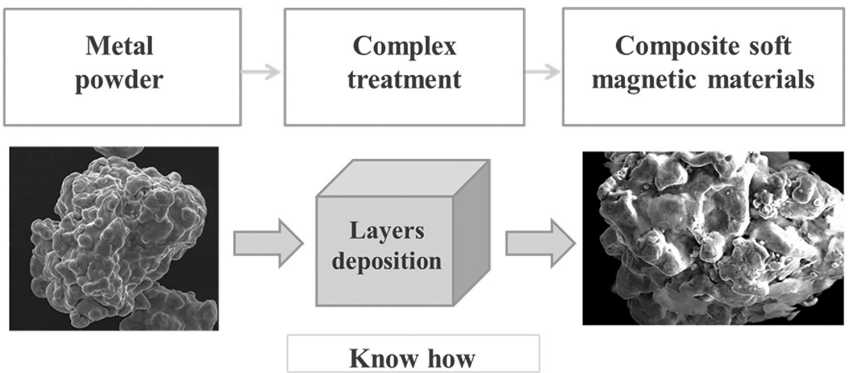

Fig. 1. Scheme for obtaining a magnetically soft composite material from metal powders

The method for manufacturing encapsulated powders and creating products from them is a multistage process that includes the following main operations. At the first stage, an operation is carried out for the reaction deposition of insulating coatings from the gas phase at a temperature of 150–200 °C[1]. Iron powder in a special reactor is transferred to a suspended state in a gaseous medium containing vapors of the deposited oxide material together with solvent vapors. Solvent vapors, which were used as ethanol, gasoline, acetone, isopropyl alcohol, and others, are gradually pumped out of the reactor. A phosphorus oxide insulating coating was applied to the surface of the iron particles. The insulating coating was formed in the form of one, two and three layers. The thickness of one layer of the insulating coating was determined based on the specific surface area of iron particles, the volume of the introduced oxidizer, and the deposition time, and was about ~10 nm.

The second stage is the operation of applying a wax-based lubricant to the surface of the isolated iron particles in an amount not exceeding 0.025 wt. % in the reactor at a temperature of 150–200 °C with constant vacuum pumping. Further, by hydrostatic pressing of encapsulated powders, products are manufactured in special molds under a pressure of 0.7–0.8 GPa under normal conditions. Pressed products are subjected to heat treatment to normalize physical parameters. Products are annealed at a temperature of 450 °C for 1.0–1.5 hours in special autoclaves in a nitrogen atmosphere or in air, depending on the requirements.

The developed application technology is very simple and easily scaled. The finished composite has a low cost compared to analogues.

The crystal structure of the composites was studied on a DRON-3 M diffractometer in CuKα radiation in $20° \leq 2\theta \leq 90°$ angle range at room temperature. The morphology and chemical composition of the powders were studied by scanning electron microscopy (SEM) using a Hitachi SEM, Zeiss microscope. Prior to the study, the surface of the pressed composite was preliminarily ground and polished to remove the surface oxide layer. To study the magnetic properties, samples of a composite magnetic material from an encapsulated powder were fabricated rings with dimensions of 24 × 3 × 8 mm and then subjected to annealing. The density of finished products ranged from 7.4 to 7.45 g/cm^3.

Measurements of magnetic properties were performed both on an developed in our laboratory express magnetometer and using a calibrated IFM06 Fluxmeter. The value of the electromagnetic parameters was calculated from the magnetization reversal curves of the samples.

Structure and morphology characterization. On Fig. 2 are shown X-ray diffraction patterns of ABC100.30+P_2O_5 composites. The results of the analysis of obtained by X-ray diffraction data showed that only one α-Fe phase of pure iron is present in the samples. Composites have a cubic unit cell with the space group Im3m. The parameter is $a \approx 0.2867 \pm 0.0003$ nm. In the small angles 25–35° range diffuse scattering is observed, which is inherent in the amorphous P_2O_5 phase. With an increase in the thickness of the insulating layer deposited on the iron particles, the scattering intensity increases. Based on the results of X-ray diffraction determination of the unit cell volume $V = a^3$, the theoretical density of composite materials was calculated. The calculated theoretical density value is approximately 3 % higher than the density values obtained by direct measurement by hydrostatic weighing (7.4–7.45 g/cm^3). This is consistent with the fact that the pressing fill is about 95 %. Such a difference in the density values is due to the low porosity of the synthesized composites.

SEM-images of the particles of the initial and phosphide encapsulated ABC100.30 iron powders and the surface of the pressed and sintered composite are shown in Fig. 3, *a–c*. Particles of initial water-atomized iron and after coatings application have a rather complex irregular morphology, which subsequently makes it possible to obtain dense composites with the necessary electrical characteristics. The formation of phosphide layer on the particles surface can be judged by the change in the contrast of the image compared to the original ABC100.30. The particles of the encapsulated powder acquire smoother shapes. The applied layer is distributed fairly evenly. In the image of the pressed composite in Fig. 3, *c* the grain boundaries of the composite consisting of encapsulated iron particles are clearly observed. The structure of the pressed sample is dense with small inclusions of pores, which confirms the results of density studies.

[1] Method for manufacturing a composite magnetically soft material: patent of Russian Federation No. 2465669 / G. A. Govor, V. I. Mitsyuk, A. V. Tamonov. Publ. 27.10.2012.

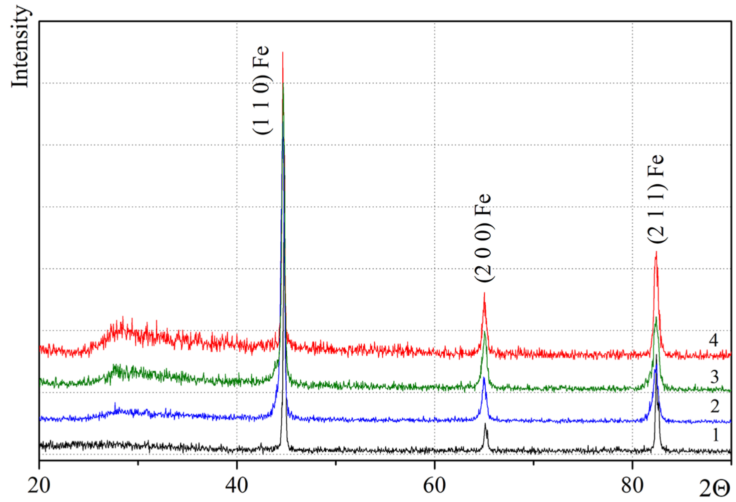

Fig. 2. Experimental X-ray diffraction patterns of composites based on phosphide-encapsulated iron powder ABC100.30: *1* – initial ABC100.30; *2* – encapsulation layer with thickness of 10 nm; *3* – 20 nm; *4* – 30 nm

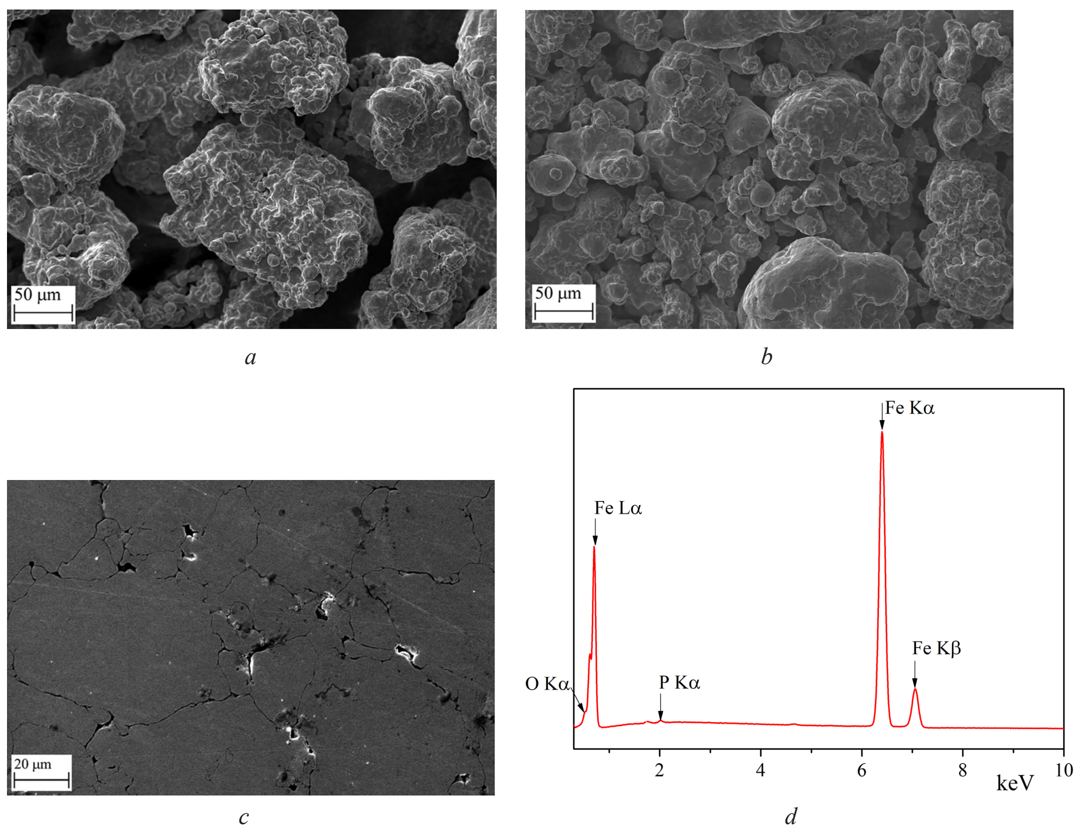

Fig. 3. *a* – SEM-image of the initial ABC100.30 iron powder; *b* – SEM-image of phosphide encapsulated iron powder ABC100.30 particles; *c* – SEM-image of the surface of a pressed composite based on phosphide encapsulated iron powder ABC100.30; *d* – spectrum of X-ray fluorescence analysis of the resulting composite

The results of chemical analysis by energy dispersive X-ray (EDS) spectroscopy are shown in Fig. 3, *d*, as well as in Table 2. The total EDS-spectrum of the composite surface showed a complex composition consisting of iron (F), oxygen (O), phosphorus (P). The map of the distribution of elements showed that oxygen and phosphorus are distributed unevenly throughout the study area, but only along the boundaries of iron particles, where their increased concentration is observed. This proves the formation of a phosphide coating at the grain boundaries and its retention after pressing and annealing.

T a b l e 2. **The content of chemical elements in the sample**

Chemical element	Weight, %	At., %
O	1.16	3.92
P	0.24	0.42
Fe	98.60	95.66
Total	100.00	100.00

Electromagnetic characterization. Studies of the main electromagnetic characteristics of all obtained composites have been carried out, which is very important for determining the practical potential of the developed materials.

Magnetic hysteresis loops have been obtained in a field of up to 20 kA/m at a frequency of 1 kHz for ABC100.30 powders, the particles of which are encapsulated by insulating coatings of various thicknesses. An analysis of the loops showed that with a change in the coating thickness, a change in the nature of the magnetization reversal is observed from hysteretic when encapsulated by a 10 nm coating to an almost linear hysteresis-free character of magnetization reversal when encapsulated by a 30 nm thick coating. From the obtained hysteresis loops, the losses due to reversal magnetization are calculated and their dependences on the magnetic induction are plotted. An analysis of the calculated data shows that a thin coating on the order of 10 nm does not provides high-quality isolation of particles, which leads to significant reversal magnetization losses. To ensure minimum of reversal magnetization losses, an insulating coating of particles of 20 nm or more is required. The frequency dependences of the magnetic permeability of a material based on ABC100.30 iron powder encapsulated by coatings of various thicknesses in the frequency range up to 1 MHz are plotted. From the analysis of the experimental results, we can conclude that for the case of encapsulation of iron particles by a coating 10 nm thick, the value of the magnetic permeability changes significantly with frequency. With an increase in the coating thickness, the value of the magnetic permeability at a frequency above 10 kHz increases to the values μ = 120–130. For a material with coated 30 nm thick particles, in a wide frequency range from 1 kHz to 1 MHz, the permeability remains practically unchanged, which is important from the point of view of the practical application of such a material.

Prospects for practical applications. Currently, in the production of electrical machines, known soft magnetic alloys are mainly used, 90 % of these are electrical steels of various types. Such materials have almost reached the limit of their physical, mechanical and operational properties, and in order to create a new generation of products, it is necessary to use a completely new class of soft magnetic materials with improved characteristics, which composite materials developed and studied in this work can be used.

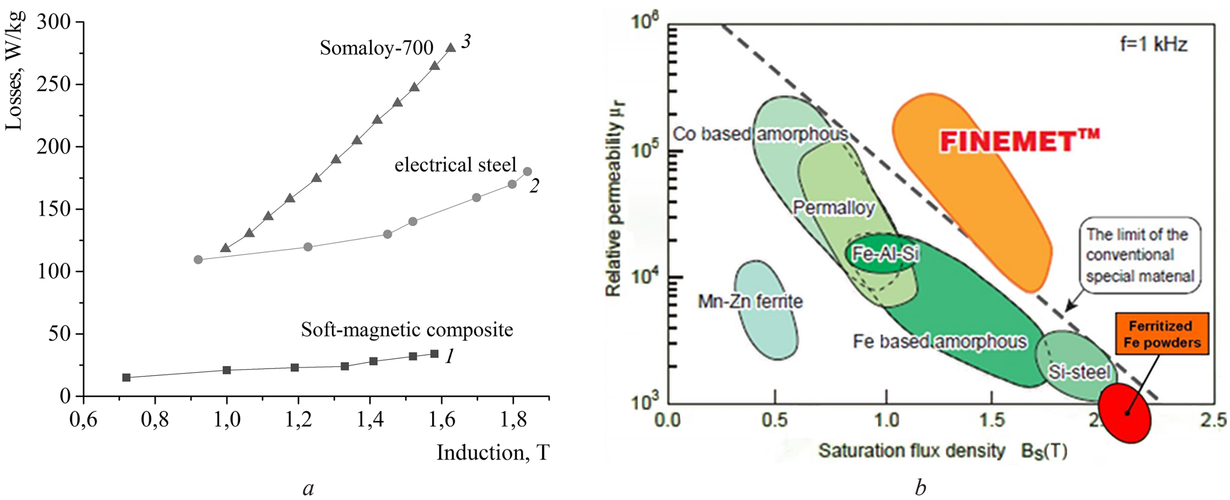

Fig. 4. *a* – Total electromagnetic losses for a soft magnetic composite based on capsulated iron powder (*1*), electrical steel 3412 (*2*) and Hoganes alloy Somaloy-700 (*3*); *b* – Relationship between permeability and saturation flux density in several soft magnetic alloys [14]

When comparing the total electromagnetic losses (Fig. 4, *a*) for the developped magnetically soft composite based on capsulated iron powder (*1*), electrical steel (*2*) and Somaloy-700 alloy (*3*), depending on the induction value, it is also seen that the losses for our material are lower. The advantages of a composite magnetic material over electrical steel and other soft magnetic alloys allow for their wider use in electrical machines in order to increase the power density at high rotation speeds with less losses values.

The diagram (Fig. 4, *b*) shows the relationship between permeability and saturation induction of a number of known soft magnetic alloys. It can be seen that composites based on iron powders with encapsulated particles have one of the highest saturation inductions, and surpass all other alloys in this parameter. In Table 3 for comparison it is shown the main parameters of electrical steel, Mn–Zn-ferrites and the investigated magnetic composite material based on iron powder. Our material has a higher value of saturation and working induction, the field dependence of the induction is close to linear, in addition, it is environmentally friendly, has a high electrical resistance, which means lower eddy current losses. And the manufacturability and simplicity of cores production by compacting exceeds the method of sheet assembly from electrical steel. There is practically no waste in the production of magnetic cores.

T a b l e 3. **Properties of electrical steel, Mn–Zn ferrite and Fe-based magnetic composite material**

Electromagnetic parameters	Electrical steel	Mn–Zn ferrite	Developped SMC
Induction saturation, T	1.8	0.45	2.1
Maximum magnetic permeability	4000–7000	500–4000	500–1000
Dependence of induction on the magnetic field	Nonlinear	Nonlinear	Close to linear
Ecology	Possible noise	Noiseless	Noiseless
Transformer assembling	Complicated	Simple	Simple
Electric resistance	10^{-6} Ohm m	10^{6}–10^{9} Ohm m	10^{6}–10^{9} Ohm m

The obtained scientific results indicate the possibility of creating components for various electrical devices based on new magnetically soft composite materials. Preliminary calculations have shown that such materials can be used to create high-frequency converters, highly efficient axial electric motors and generators, and magnetic components for a wide range of applications. Their advantage is that the dimensions of similar products are reduced by almost 2 times at the same power at frequencies up to 1 MHz.

Thus, the new composite magnetic material based on iron powder can successfully replace electrical steel in the manufacture of product components such as transformers, inductors, high-speed motors and generators.

Conclusion. New composite magnetosoft materials based on ABC100.30 iron powder encapsulated with phosphorus oxide have been obtained using the developed method. The results of experimental studies have shown that an increased concentration of oxygen and phosphorus is observed along the boundaries of iron particles; an increased concentration is observed at the boundaries of iron powder grains, which proves the formation of a phosphide coating. For obtained composites the regularities of changes in the electromagnetic properties depending on the thickness of the insulating coating have been studied. It has been found that for materials coated with particles 10 nm thick, the magnetization process has a hysteresis character, while for composites with particles coated with a thickness of 30 nm, an almost linear magnetization pattern is observed. With an increase in the coating thickness, the value of the magnetic permeability at a frequency above 10 kHz increases to the values of μ = 120–130. For a material with coated 30 nm thick particles, in a wide frequency range from 1 kHz to 1 MHz, the permeability remains practically unchanged, which is important from the point of view of the practical application of such a material. In comparison with electrical steel and widely used ferrites main parameters the investigated magnetic composite material based on iron powder has a higher values of induction, has a high electrical resistance, which means lower eddy current losses. The developed materials with magnetic permeability μ = 100–150 and induction up to 1.8 T are promising for use in the frequency range up to 1 MHz in the manufacture of various types of electrical devices.

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