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RESEARCH ARTICLE OPEN ACCESS

Investigation of soft magnetic composite materials in the frequency range up to 100 kHz

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ABSTRACT

In a wide range of temperatures and magnetic fields, a study of soft magnetic composite (SMC) materials based on ABC100.30 iron powder with an insulating coating on phosphorus oxides in a frequency range of up to 100 kHz was carried out. It is shown that, under normal conditions, the composite material, due to the presence of interparticle conduction channels, forms the electron density on the Fermi surface, which is characteristic of the metallic state. In this case, the specific resistivity of the SMC material is orders of magnitude higher than for the metallic state.

With a decrease in temperature, it occurs as a result of thermal compression of metal particles with an exponential decrease in the number of interparticle conduction channels. As a result, the magnetic state can be characterized as superparamagnetic, in which only interparticle magnetic interaction is retained.

The study of magnetization reversal losses in the frequency range up to 100 kHz has shown that they can be used in various energy conversion devices due to the high saturation induction and low losses.

Keywords: soft magnetic composites, magnetic properties, low core loss, high magnetic induction, insulating layers.

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I. INTRODUCTION

Soft magnetic composites (SMC) based on iron powder have increasingly received intensive research interests because of the potential using for the SMC within many engineering fields covering electrical equipment, high-power transformers and modern communication. Compared with traditional silicon steel material, the SMC have remarkable advantages such as high magnetic inductions, high frequency properties, decreased core losses, three-dimensional isotropic magnetic properties [1–4], low magnetocrystalline anisotropy constant and flexible design [5]. With the development of miniaturization and integration for electronic devices, the SMC require to have high magnetic flux density, low loss and low production cost.

Magnetic properties and performances of the SMC are determined by their compositions and microstructures. It was reported that iron and its alloys can be a promising candidate for SMC due to their high magnetic flux density and low cost [6]. However, the large eddy current losses in pure iron and its alloys is not desirable for practical applications. A feasible way to reduce the eddy current losses was used coating an insulating layer on the materials based on iron [7,8].

The coating materials can be mainly divided into organic (such as epoxy resin or organic-silicon [9]) and inorganic materials such as insulating metal oxides [10], phosphates, ferrites [11] and some metal or alloy layers. In general, various organic insulating layers can greatly increase the electrical resistivity of metal-based SMCs, however, low density and poor thermodynamic stability of these organic layers impede the extensive practical applications of these organic-coating SMC [12]. Inorganic materials such as MgO, Al_2O_3, SiO_2 and phosphates as the coating layers for metal-based SMC can significantly improve their thermal stability and compaction density [13,14]. However, magnetic dilution of all these non-magnetic organic and inorganic coating layers in traditional metal-based SMC would inevitably decrease their saturation magnetization and lead to their low magnetic induction [15]. Recently, some ferrites such as $(NiZn)Fe_2O_4$, Fe_3O_4, $CoFe_2O_4$ have been used as the coating layers to fabricate metal-based SMC with high magnetic induction due to their ferromagnetism [16]. However, it is difficult to obtain nanoscale ferrite layers with homogenous structure in these ferrite-coated SMC and thus comparatively high core loss often occurs in the composites [12,17]. Moreover, the ferrites as a typical type of oxide ceramics exhibit high brittleness and weak adhesion, which would lead to low compaction density and low mechanical strength of metal-ferrite structured SMC [14]. Thus, it is necessary to explore suitable coating layers and feasible preparation methods for fabricating high-performance SMC with the ideal composite structures to thoroughly improve their overall performance.

In this work, we investigated the structural and magnetic properties of SMC materials based on high-purity iron powders ABC100.30 with nanoscale insulating coatings based on phosphorus oxide. In [18], the static properties of composite soft magnetic materials were considered in comparison with the characteristics of sheet steel. In [19] the possibility of using composite materials for the construction of electric motors and other products for industrial frequencies of 50 - 60 Hz is studied. The most demanded and often used in the practice of magnetic materials for building various kinds of power supplies, inverter converters for various purposes, induction heating panels and a number of other applications is the frequency range of oscillations up to 100 kHz. It is of interest to study the behavior of composite soft magnetic materials in this frequency range to determine their applicability in comparison with other magnetic materials.

In this regard, the purpose of this work is to study the properties of composite magnetically soft materials, primarily the frequency dependences of magnetization reversal losses in the oscillation range up to 100 kHz.

II. EXPERIMENTAL TECHNIQUE

The technology for the manufacture of isolated powders of soft magnetic materials and the manufacture of products from them is a multistage process that includes the following basic operations.

1. Operation on the reactive deposition of insulating coatings from the gas phase at a temperature of 150 - 200 °C [21]. During this operation, the iron powder is suspended in a gaseous medium containing the vapor of the deposited oxide material together with the vapor of the solvent. Solvent vapors, which were used as ethanol, gasoline, acetone, isopropyl alcohol and others, are gradually removed from the reactor. In this work, we investigated composite materials based on high-purity ABC100.30 iron powders on the surface of which insulating coatings were applied using phosphorus oxide. The chemical composition of the main impurities of ABC100.30 iron powders is shown in Table 1.

Table 1. Chemical composition, weight. %

	C	O
ABC100.30	0,002	0,05

2. Operation on the manufacture of products by hydrostatic pressing of isolated powders in special molds, on the working surfaces of which a lubricant was applied, under a pressure of 0.7 - 0.8 GPa under normal conditions. The pressed products were heat treated to normalize the physical parameters. The samples are annealed at a temperature of 600-650 °C in vacuum or in air.

One of the main advantages of the developed technology, even taking into account the fact that the technology is being finalized, is the possibility of uniform application of protective and insulating coatings of various compositions in a wide range from nanometers to micrometers. In addition, products made of a composite material according to the developed technology retain their dimensions after pressing and subsequent annealing.

To study the magnetic properties, samples of a composite magnetic material were made in the form of rings with dimensions of 24x13x8 mm. The density of the finished products was in the range from 7.4 to 7.75 g/cm^3 in dependences of the isolation layer thickness.

The measurements of the magnetic properties were carried out both on an express magnetometer, where the losses and other magnetic parameters were determined from the magnetization reversal curves of the samples, and additionally by a direct method by measuring the heating rate of the core during operation in the adiabatic mode. Both of these methods showed good agreement between the measurement results.

III. RESEARCH RESULTS

Figure 1 shows the results of an electron microscopic study of the surface of isolated iron particles - microstructure and chemical composition. It can be seen that the iron particle size is generally less than 100 μm. In terms of the composition of surface coatings of iron powder, in addition to phosphorus oxide, the content of which is close to 3.3 wt.%, silicon oxides are also present, about 2 wt.% is a powder lubricant. The X-ray diffraction pattern of the SMC-treated material shows the presence of pure iron only (Figure 2). Some rise in the diffuse background is associated with the presence of impurities of less than 1%.

Figure 3 shows the temperature dependence of the resistivity of the composite material under study, which has an exponential decrease in its value as the temperature rises from 100 K to room temperature from 10^4 Ohm·m to 30 Ohm·m.

The SMC magnetization curve in a magnetic field of 800 kA/m and a wider temperature range is shown in Figure 4 from which it follows that the Curie temperature of the material is Tc = 1000 K.

Figure 5 shows the hysteresis loop when the SMC material is magnetized at a frequency of 1 kHz. It can be seen that the change in the magnetic induction from the magnitude of the magnetic field strength is almost linear with the magnitude of the coercive force of the order of 10-20 A / m

Figure 6 shows the curves of magnetization and losses at a frequency of 1 kHz of the material SMC based on ABC100.30 in magnetic fields up to 10 kA/m Figure 7 shows the magnetization curve of the composite SMC material in strong magnetic fields, which is characterized by linear changes magnetic induction in the range of magnetic fields up to 300 kA/m.

Losses of SMC material based on ABC100.30 phosphorus oxide insulation depending on frequency at values of induction 0.2 and 0.35T are shown in Figure 8.

IV. DISCUSSION OF RESEARCH RESULTS

Unlike magnetodielectrics, where each metal particle is completely isolated and conductivity between the particles is excluded, for a composite magnetically soft material, under normal conditions, adjacent metal particles are connected by conduction channels to form a common conduction band. In this case, the isolation of the metal particles is local, allowing the mutual exchange of conduction electrons between the particles. As a result of the mutual overflow of electrons, a conduction band is formed, in which the electron density of the Fermi surface is determined by the degree of overlapping of metal particles.

Based on the theory of direct exchange interaction, the electron densities on the Fermi surface should be close for both the metallic ferromagnetic and the composite materials (Figure 9) In this case, the magnetic properties of the metallic ferromagnetic and the composite material will be identical. This condition can be fulfilled in the case of composite magnetic materials if the grain insulation is local and has the minimum possible thickness. As shown by the present studies, the average calculated thickness of the insulating layer should be nanometer in size.

At low temperatures, the metal particles become practically insulated as a result of thermal contraction. This magnetic state is close to the so-called superparamagnetic state, which is characterized by a decrease in magnetization with decreasing temperature in low magnetic fields. In this case, the population of the Fermi level changes from a maximum value for a state close to metallic $E_f = E_{f2}$ to a minimum value for a superparamagnetic state $E_f = E_{f1}$ to (Figure 8). The superparamagnetic state of a composite material is characterized by a weak dipole-dipole magnetic interaction between metal particles, for which saturation is achieved in strong magnetic fields.

The equilibrium magnetization of an ensemble of such superparamagnetic particles can be described, as for a classical paramagnet, by the Langevin function

$$I=nm(cth(mH/kT) – kT/mH) \qquad (1)$$

where I — magnetization; m — magnetic moment of the granule; n — number of granules per unit volume; H — strength of the external magnetic field.

Figure 10 shows the calculated change in the magnetic induction as a function of temperature for an ideal Langevin paramagnet (curve 1) and the magnetic induction of an SMC material based on ABC100.30 in a magnetic field with a strength of 800A/m (curve 2). The graph shows a significant difference in the behavior of the temperature dependences of the magnetic induction, which is associated with a change in the population of the Fermi level in the latter case.

The studies carried out have shown that further progress in improving the magnetic properties of composite materials and, first of all, achieving the minimum values of losses, is associated with an improvement in the properties of the iron powder itself. Defectiveness of grains of iron powder, determined by the content of carbon forming iron carbides, is one of the factors in the growth of coercive force and losses due to magnetization reversal.

V. CONCLUSION

In a wide range of temperatures and magnetic fields, a study of soft magnetic composite (SMC) materials based on ABC100.30 iron powder with an insulating coating on phosphorus oxides in a frequency range of up to 100 kHz was carried out. It is shown that, under normal conditions, the composite material, due to the presence of interparticle conduction channels, forms the electron density on the Fermi surface, which is characteristic of the metallic state. In this case, the specific resistivity of the SMC material is orders of magnitude higher than for the metallic state.

With a decrease in temperature, it occurs as a result of thermal compression of metal particles with an exponential decrease in the number of interparticle conduction channels. As a result, the magnetic state can be characterized as superparamagnetic, in which only interparticle magnetic interaction is retained.

It is shown that under normal conditions, the developed SMC composite materials have minimal losses when using high-purity iron powders with minimum carbon content as a base, for example, ABC100.30. At the same time, the optimal calculated thickness of the insulation coating based on phosphorus oxide is about 10-20 nm

The study of magnetization reversal losses in the frequency range up to 100 kHz has shown that they can be used in various energy conversion devices due to the high saturation induction and low losses.

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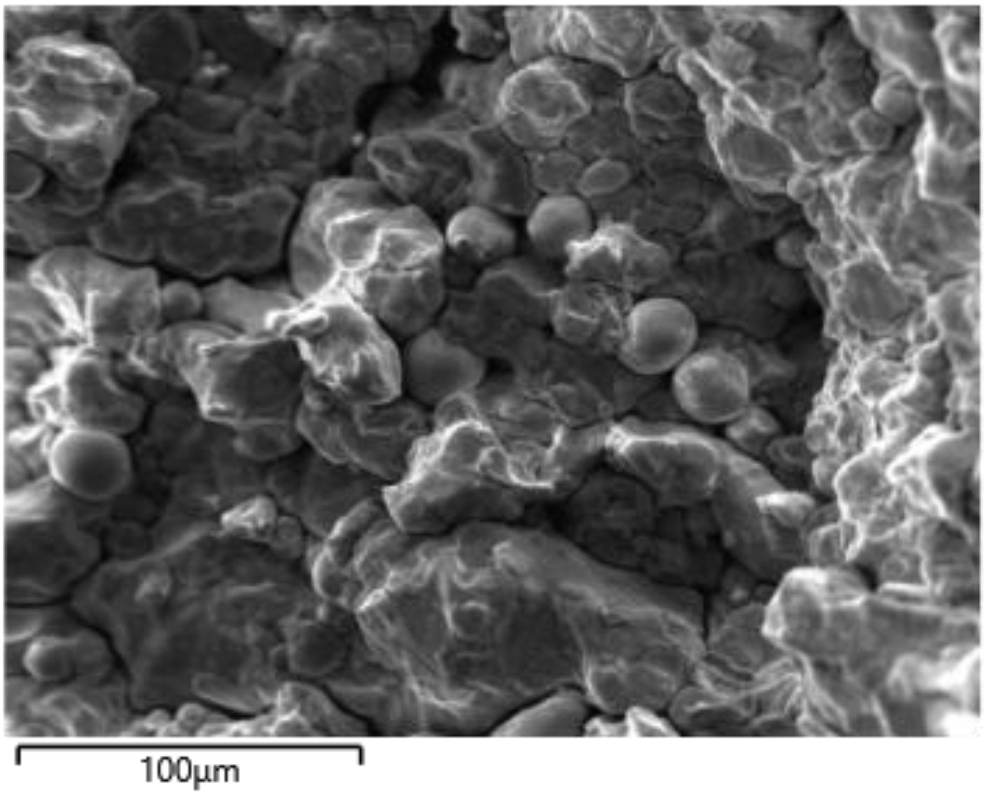

Element	Line type	Conditional concentration	Weight, %	Sigma weight, %	Atom., %
C	K-series	2.48	7.74	0.06	21.48
O	K-series	31.47	14.64	0.04	30.50
P	K-series	6.67	3.55	0.01	3.82
Fe	K-series	113.98	74.07	0.06	44.21
Total:			100.00		100.00

Fig. 1. Results of electron microscopic examination of the surface of isolated iron particles **-** microstructure and chemical composition.

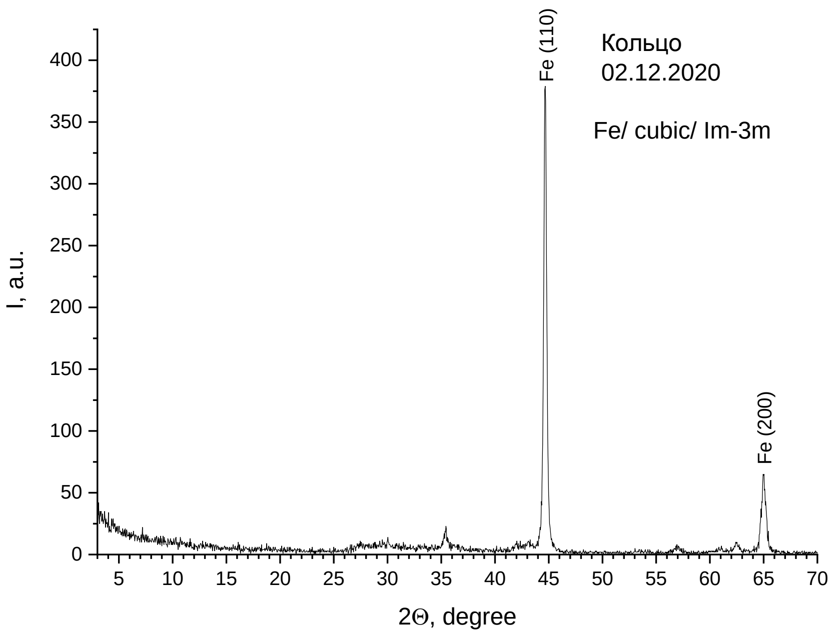

Fig. 2. X-ray diffraction pattern of SMC powder based on ABC100.30 - Fe reflection lines and a diffuse peak are observed, most likely associated with a disordered solution of Fe + P_2O_5.

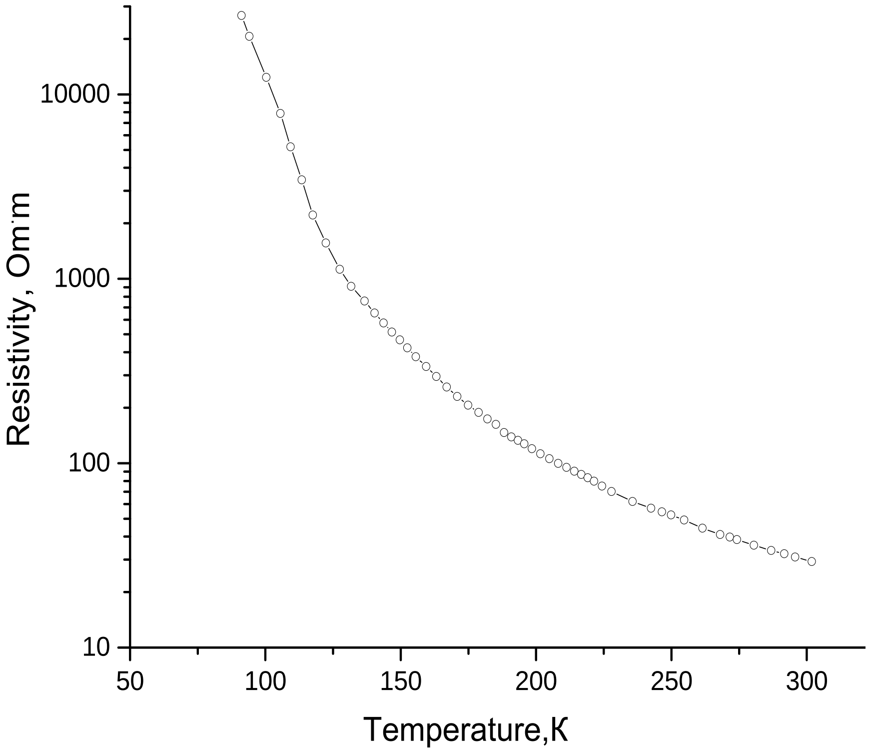

Fig. 3. Temperature dependence of the resistivity of SMC material based on ABC100.30.

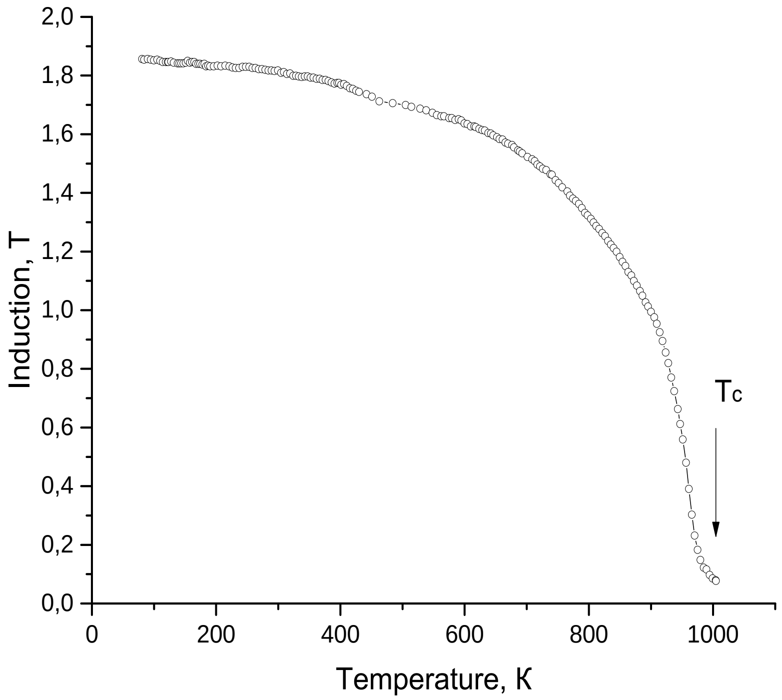

Fig. 4. Temperature dependence of the magnetic induction of an SMC material based on ABC100.30 in a field of 800 kA/m.

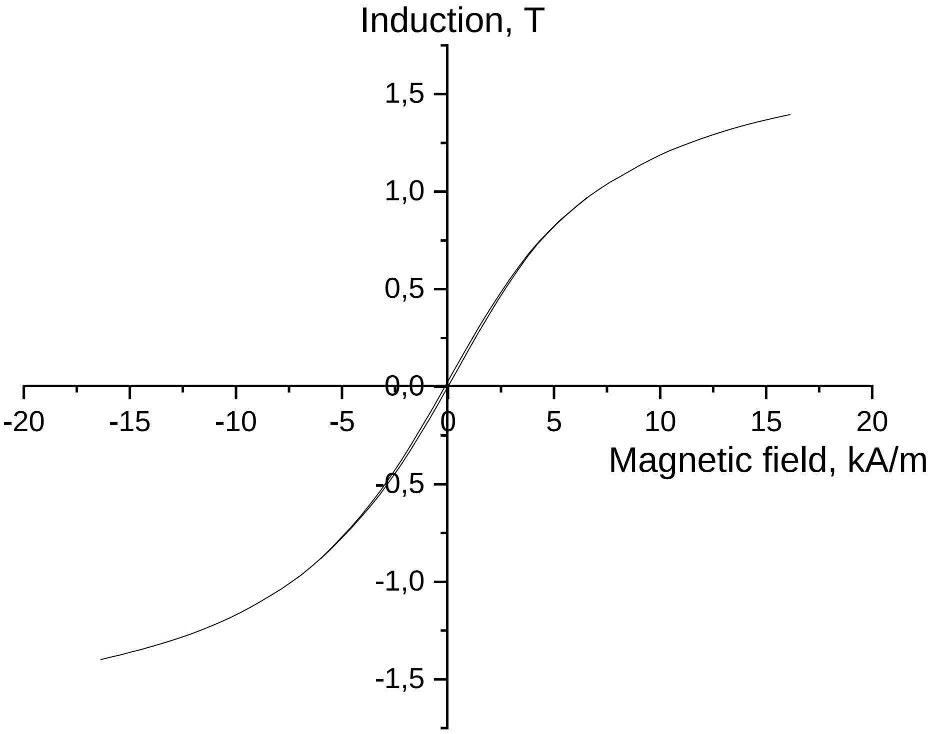

Fig. 5. SMC material remagnetization loop at a frequency of 1 kHz

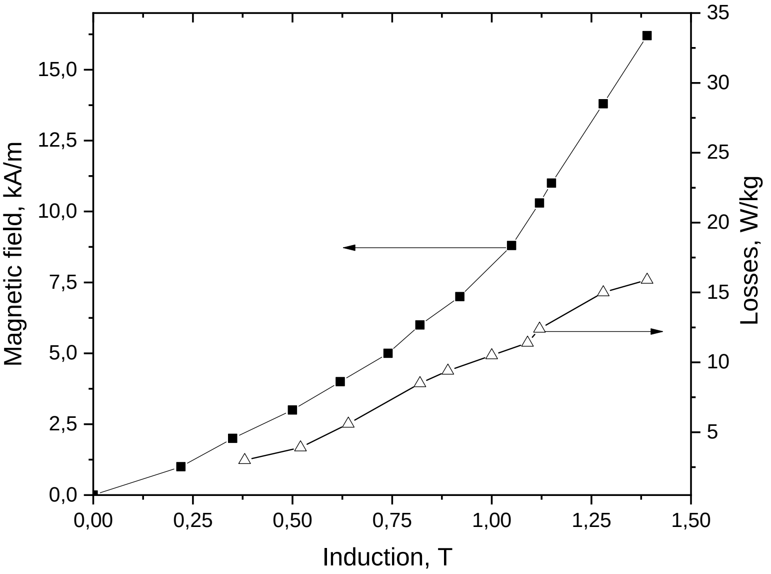

Fig. 6. Magnetization and loss curves at a frequency of 1 kHz SMC based on ABC100.30 in magnetic fields up to 15 kA/m.

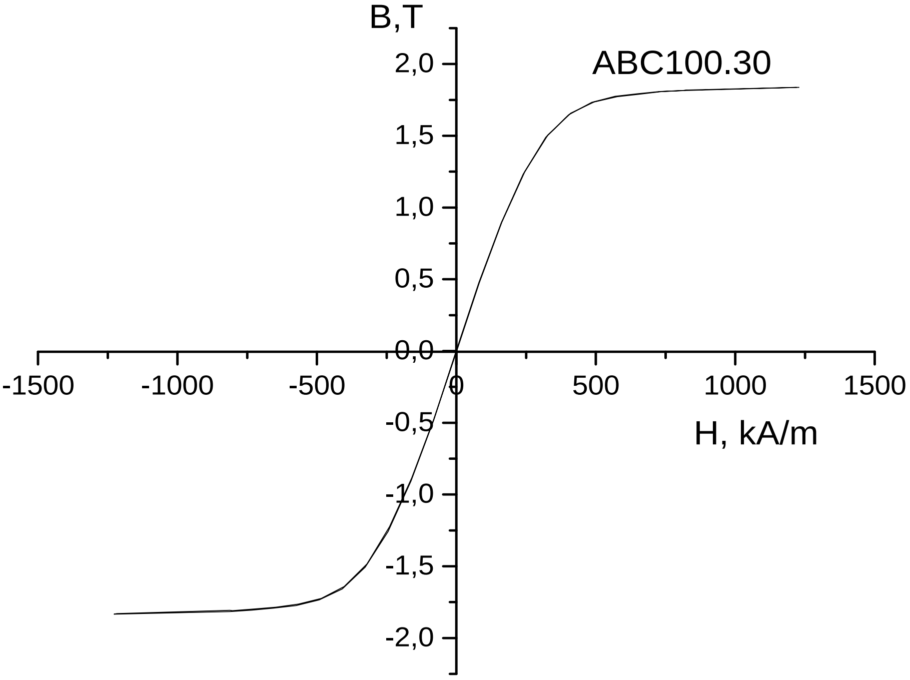

Fig. 7. Magnetization curves of SMC material based on ABC100.30 iron powder.

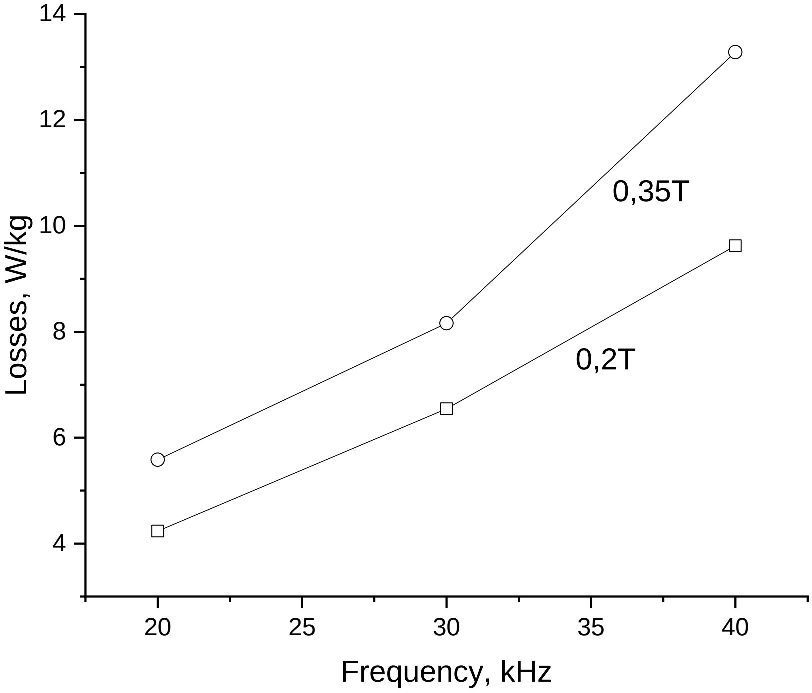

Fig. 8. Losses of SMC based on ABC100.30 with phosphide oxide insulation depending on frequency at values of induction 0.2 and 0.35 Tesla.

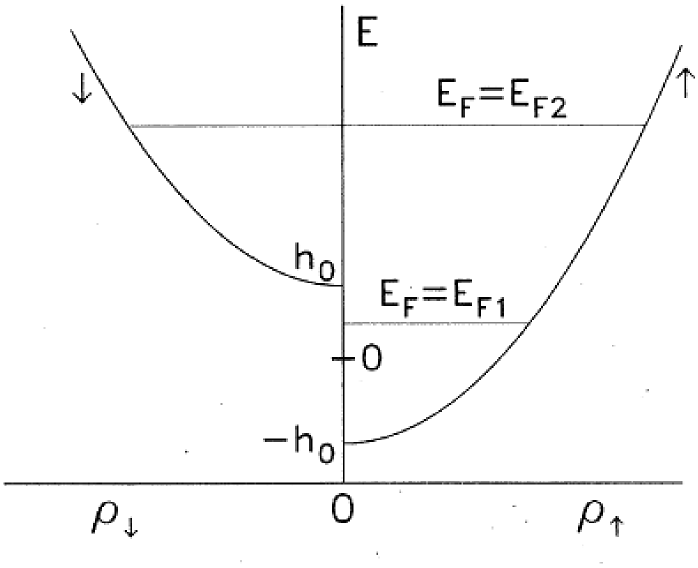

Fig. 9. Fermi level change from the minimum for the low-temperature region $E_f = E_{f1}$ to the maximum high-temperature value $E_f = E_{f2}$.

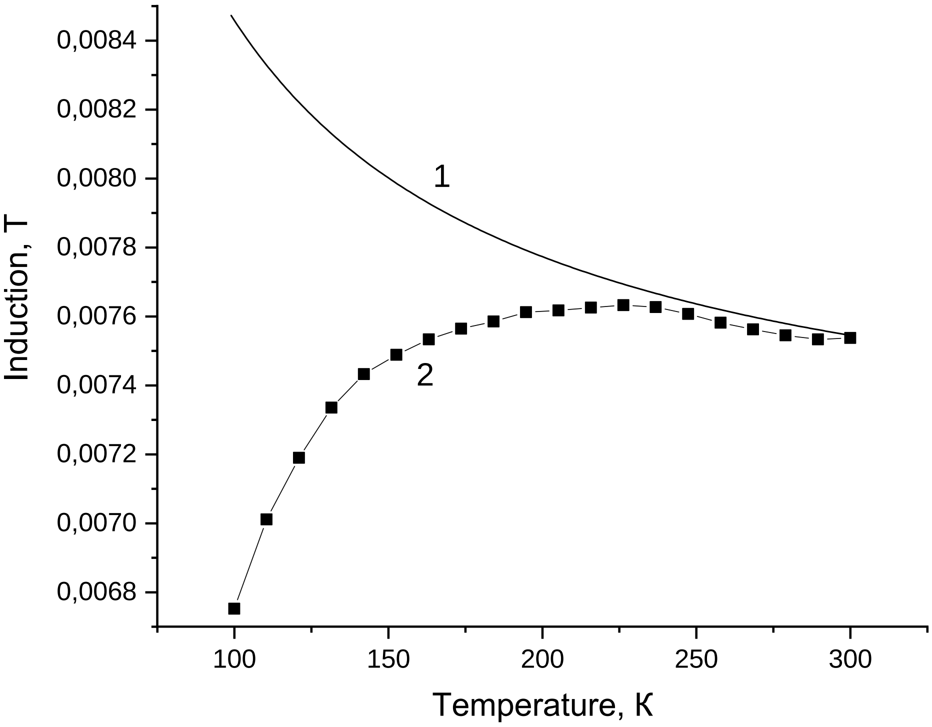

Fig. 10. The calculated change in the magnetic induction as a function of temperature for an ideal Langevin paramagnet (curve 1) and temperature dependence of the magnetic induction of the SMC material based on ABC100.30 in a magnetic field of 800 A/m (curve 2).

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