Optimization of the method of oxide coating of metallic iron powder particles

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Abstract. This article considers the issue of wide implementation in various branches of the national economy caused the intensive growth of electric machines production. Due to the high specific consumption of magnetic materials in the production of electric machines, a very promising direction is the development of waste-free technology of manufacture of magnetic cores and cores by methods of powder metallurgy. The use of powder metallurgy allows reducing losses of electrical steel and eliminating many labor-intensive operations, using powder metallurgy for electrical production, it is possible to obtain sufficiently high efficiency values andη reduce hysteresis losses.

1 Introduction

Composite materials with the necessary set of performance characteristics are widely used in nodes of various mechanisms. Any technical applications use those or other properties of solids: electrical, magnetic, optical, thermal, mechanical, corrosion-resistant, etc. Production of magnetic materials with low energy loss during remagnetization is one of the current problems of industry. Despite the fact that research and development of such materials have been conducted since the beginning of the last century, studying the mechanism of remagnetization and improving the quality of these materials is relevant even now. This is due to the fact that magnetic materials are widely used in various electrical devices (generators, electric motors, measuring equipment, inductance coils, etc.) [1-3].

Magnetically soft materials are a very wide range of materials, both in their composition and properties, and in their purpose. Such materials should:

- easily magnetized and demagnetized without loss;

-have a high saturation induction Bs, i.e. to ensure the passage of maximum magnetic flux through a single cross section of the magnetic core, which reduces its size and weight;

- provide low losses when operating in alternating fields, which reduces the temperature of the product heating, size and weight, increases the efficiency and operating induction;

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-satisfy additional requirements related to mechanical properties, stability over time and at different temperatures, low cost.

The main share of production of magnetically soft materials are electrical steels of different types (up to 90%), which are used in the manufacture of electrical machines [4,5]. Parameters of electrical sheet steel mainly meet the requirements for soft magnetic materials for the manufacture of new electrical devices, but they have very high electromagnetic losses, due to low efficiency, especially when working in alternating fields at high frequencies. In addition, during the manufacture of magnetic components of products made of sheet electrical steels, a lot of waste remains [4, 6].

Traditional materials (electrical steel, nickel-iron alloys, Sendast, etc.) and products based on them have now almost reached the limit of their physical-mechanical and operational properties. To develop and create a new generation of high-performance electrical products it is necessary to use an entirely new class of soft magnetic materials with improved characteristics. Such materials must meet a whole set of requirements: high values of saturation induction, low electromagnetic losses, required mechanical strength, thermal stability and low cost. To reduce the mass-size characteristics the devices should be able to operate at high frequencies [4,5,7].

Such requirements are satisfied by composite materials based on metal powders, the particles of which are covered with a very thin layer with electrical insulation. The application of insulating layers provides reduction of electromagnetic losses and increases the quality of composites. The final properties of materials and articles made of them depend on the types of initial powders and methods of their processing. Such materials with the necessary properties are actively developed and researched in the leading scientific centers of the world. A rational choice of techniques for obtaining composites opens up additional possibilities for their practical application [8,9]. For this purpose, it is important to ensure the controlled chemical composition and structure of the components during synthesis, which, in turn, guarantees the required physical and functional properties.

2 Methods

As follows from the above consideration, the known methods of encapsulating iron powder with a thin oxide layer, namely by mechanical deposition of the oxide layer, formation of the oxide layer as a result of decomposition of sulfates and metal nitrides and formation of the oxide layer from the gas oxide layer, create poor quality coating and are ineffective [7, 10-12].

In this regard, a combined method of obtaining oxide coatings on the surface of iron particles was proposed [2,4,5,8,10]. The proposed new method is based on the combined use of the above methods.

The method of applying insulating coatings based on phosphorus oxide has been developed on the basis of a method of making composite magnetically soft material [2,13], in which the actual method of adding to the initial metal powder in a rotating vacuum drum at a pressure of 0.15 - 1.5 Pa, heated to a temperature of 150-200º C and processed to obtain a uniform distribution of lubricant in the material within 15-30 minutes, with the lubricant content in the composite material is 0.01 to 0.1%.

The essence of the method is explained in the Fig.1, which shows the installation for the preparation of soft magnetic material, including the vacuum pump electric drive (1), connection sleeve (2), vacuum pump (3), movable vacuum connector (4), drum with composite soft magnetic material placed in it (5), temperature sensor (6), heater (7) and electric drive of reactor (8).

In the first step, the lubricant is introduced by mixing it with insulated metal powder in ball mills or other mixers. The composite material prepared with the lubricant is placed inside the drum. After that the vacuum pump is switched on, reducing the air pressure inside the drum with the material to 0,15 - 1,5 Pa. After achieving the required vacuum the drum is heated to a temperature of 150 - 200ºC. The process of powder processing continues for 15 to 30 minutes Cooling of the soft magnetic material takes place after switching off the heating while the vacuum is maintained.

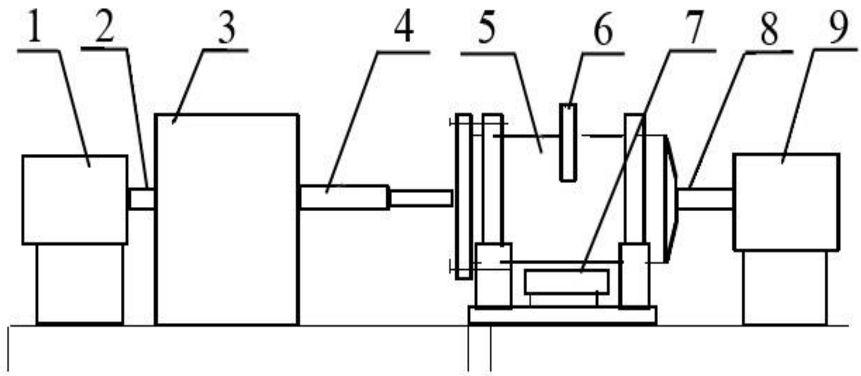

Fig. 1. Reactor for insulating coatings on the surface of powders.

The advantage of this method in comparison with the known ones is the reduction of the lubricant content to 0.01-0.1%, which allows to obtain in the future a quality pressed product of magnetically soft composite material with a density of 7.5 - 7.65 g/sm^3 and, as a consequence, having high magnetic characteristics [9,11].

In order to apply phosphorus oxide coatings on the surface of metal powder particles, the proposed methodology was modified. The deposition technique included the stage of preliminary mixing of initial metal powders with a given amount of reagent, which included an alcoholic solution of orthophosphoric acid in the ratio of - 40% H_3PO_4+60% ethanol [7,14,15].

In the next step the prepared powder was placed in the insulating coating reactor (Fig.1). The powders with the reaction additive were treated in a reaction drum at a pressure of up to 10^5-10 ^{6}Pa heated to a temperature of 150-200ºC for 15-30ºC. As a result, a complex coating of ferrite and phosphate compounds was formed on the surface of iron particles. Investigation of elemental composition was carried out with the help of X-ray spectral analyzer by "Oxford Instruments" company (England) [4,9], minimal limit of element detection was 0.5%, error of method was 3-5 relative percents. The chemical composition of the oxide coatings is a complex system of iron oxides FeO, Fe_2O_3 and phosphorus oxide P_2O_5. Small amounts of carbon and silicon are also present. The thickness of the insulating layer on the iron particle depends on the processing time of the powder and the concentration of the alcoholic solution of orthophosphoric acid. Subsequently, the coating process was repeated 2, 3 and 4 times to obtain different thicknesses of coatings on metal powders.

3 Results and discussion

The proposed method of encapsulating iron powder with an oxide layer is a highly economical method that practically does not change the cost of the latter and, from the standpoint of obtaining a given composition with specified magnetic parameters and specific electrical resistance, can be widely used in practice for obtaining MDM-alloys with special magnetic and electrical properties.

Based on the requirements to the initial powders and taking into account the same price category, two types of powders were chosen as the main ones on which oxide layers were applied according to the developed method: water-atomized powder Hoganes ASC100.29 (Sweden) and LiaoNing (China). The purity by impurity content of the ASC100.29 powder was 99.9% and LiaoNing 98.69%. [5,14]. The chemical composition of the powders is presented in Table 1. In terms of other certification parameters the powders are identical. As a comparison in fulfillment of international contracts, electromagnetic characteristics were investigated on cores obtained by pressing from ready-made industrial iron powders of well-known firms Atomet1001HP (Canada) with 99.4% purity and Micrometals (USA) with 99% purity, on particles of which the manufacturer itself has already applied dielectric layers [3, 10, 16].

Table 1. Composition of air atomized powder LiaoNing (China) and water atomized powder ASC100.29 (Sweden) [3].

Typeofironpowder	**Fe**	**Mn**	**Si**	**C**	**P**
ASC100.29 (Sweden)	99.5	0.08	0.04	0.08	0.01
LiaoNing (China)	98.69	0.35	0.1	0.022	0.028

From the analysis of the above research results it follows that the powder ASC100.29, the particles of which are covered with oxide layers, and composites based on it have a number of advantages. Compared to the Atomet1001HP powder, the total electromagnetic losses in the frequency range of 250-2000 Hz are 2 times less. The maximum induction of the composite magnetically soft material based on Micrometalspowders with dielectric insulation in the fields up to 50 kA/m is 60% lower than that of the composites based on ASC100.29. As for LiaoNing powders, it also loses to ASC100.29 powders in all characteristics. Therefore, ASC100.29 iron micropowders (Sweden) were chosen as the object for further studies [11, 13].

An important factor is to increase the efficiency of manufacturing products and to reduce the technological costs. To this end, we studied the dependence of electromagnetic parameters on the thickness of the oxide layer to be applied.

The figure shows a 2histogram comparing the maximum magnetic permeability for the composite based on ASC100.29 with coatings of 10, 20 and 30 nm for frequencies of 50, 200, 1000, 2000 Hz [15,17]. The analysis suggests that as the thickness increases from 10 to 20 nm, the value of the maximum magnetic permeability increases over the entire frequency range from 50 to 2000 Hz. With further increase in thickness up to 30 nm decreasesμ_{max} by 15-20%.

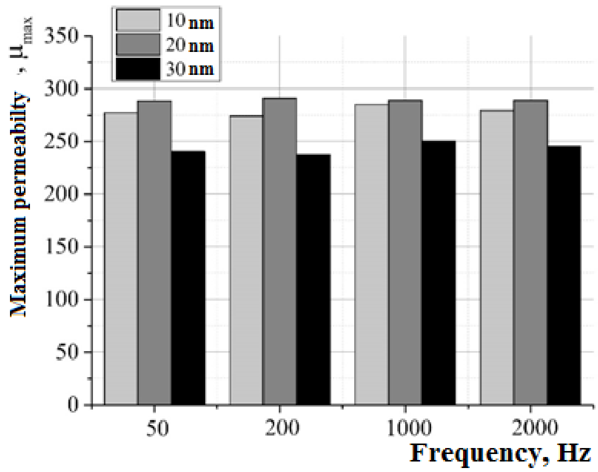

Fig. 2. Comparison of maximum magnetic permeability for composite based on ASC100.29 for frequencies 50, 200,1000, 2000 Hz.

Fig. 3. shows the results of the study of the dependence of the magnetic induction value for the composites based on the ASC100.29 powder with the coating thickness of particles of 10, 20 and 30 nm. For all samples, the induction increases with increasing magnetic field and reaches the maximum value of 1.3 T in the field of 10 kA/m for the sample with the coating thickness of 20 nm. Further, as well as for the dependence of magnetic permeability on the thickness of the material particles coating, the induction begins to decrease with increasing thickness.

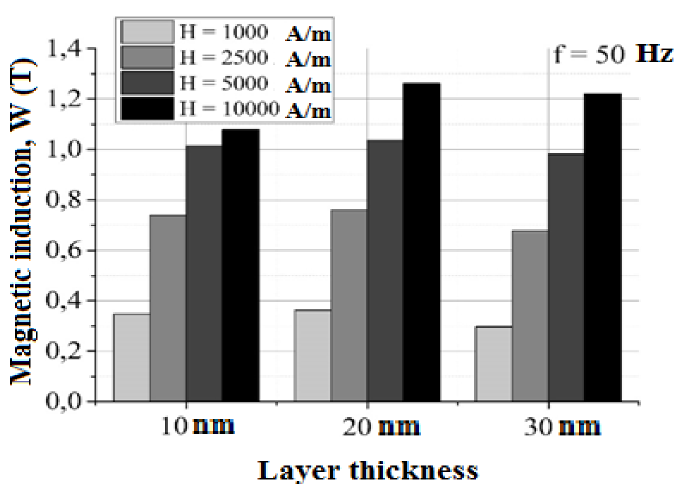

Fig. 3. Comparison of magnetic induction values (at H = 1000, 2500, 5000, 10000 A/m) of composites based on ASC powder100.29.

The analysis of the dependences of figures and2 allows us 3to conclude that the electromagnetic characteristics of composite materials based on powders with particles coated with oxide layers from 1 to 20 nm differ by no more than 10-15%, and with further increase in coating thickness begin to decrease. From the point of view of processability and efficiency of powder processing it is reasonable to apply oxide coatings with thickness not more than 20 nm. Further in this paper, the description of the methods and the results of studies of the structure and physical characteristics of Hoganes ASC100.29 iron powders, on which oxide layers with a thickness of ~ 1 - 3 microns are applied on the particles.

4 Conclusion

The carried out theoretical calculations showed that the magnetic parameters of the alloy will be close to the parameters of magnetically soft steels when the thickness of the oxide layer in such an alloy is within =δ 0.001 - 0.2 μm. A method of applying oxide layers to the surface of metallic iron particles by combining encapsulation methods has been developed, which makes it possible to obtain a given composition with specified magnetic parameters and specific electrical resistance, and can be widely used in practice [16, 18].

The electromagnetic characteristics of composite materials based on powders with particles coated with oxide layers from 1 to 20 nm differ from each other by no more than 10 - 15%, and with further increase in coating thickness begin to decrease. From the point of view of processability and efficiency of powder processing it is reasonable to apply oxide coatings with thickness not more than 20 nm [17, 19].

This difference can be explained by the fact that a material with a thin layer has a lower electrical resistivity, which means that the total electromagnetic losses will also be higher due to an increase in the contribution of losses to eddy currents. At the same time, the magnetic permeability at a frequency of 1 kHz differs by 30 units, and with an increase in frequency to 1 MHz, it decreases by 10-15% for both materials [7, 10, 18]. This makes it possible to use iron-based soft magnetic materials for electrical engineering.

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