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Article in Eastern-European Journal of Enterprise Technologies · April 2019

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Проведено дослідження по впливу хімічного осадження міді на зміну фізичних, хімічних та технологічних властивостей розпиленого порошку на основі залізу. Вивчено кінетику процесу осадження міді із розчину гліцерату міді та встановлено технологічні параметри процесу осадження, що дозволяють отримати мідні покриття з регульованою товщиною. Дослідження фізичних властивостей композиційних порошків показало, що плакування змінює форму, розмір та морфологію поверхні частинок залізного порошку. Завдяки цьому відбувається зміна технологічних характеристик залізних порошків, зокрема збільшується їх текучість та насипна щільність. Вивчення ущільнення композиційних порошків показало відмінність пористості при низьких тисках після пресування, порівняно із вихідним залізним порошком, що обумовлено кращою переукладкою частинок на стадії структурної деформації. Це дозволяє проводити процес пресування при більш низьких тисках з отриманням задовільної пористості. Допресовка брикетів на основі композиційних порошків приводить до зменшення загальної пористості матеріалу на 6–7 %, що відбувається за рахунок впливу пластичної міді, що легше деформується при тиску допресовки 800 МПа. Дослідження фізико-механічних властивостей матеріалів на основі композиційних порошків показало, що плакування міддю дозволяє підвищити міцність на згин та ударну в'язкість з твердістю. Зростання властивостей пов'язано із розчиненням міді у α-Fe. Крім цього введення міді методом хімічного осадження дозволяє отримати матеріал із рівномірним розподілом легуючого компоненту по об'єму заліза, що підтверджується результатами металографічного аналізу і вимірювання питомого електричного опору.

Є підстави стверджувати про можливість регулювання хімічного осадження, що дозволяє отримати порошковий матеріал із високими експлуатаційними характеристиками

Ключові слова: плакування, хімічне осадження, композиційний порошок, формуємість, компактування, ущільнювальність, ударна в'язкість, морфологія, електричний опір

UDC 621.762.4.04

DOI: 10.15587/1729-4061.2019.164017

A STUDY OF THE STRUCTURE AND PROPERTIES OF MATERIAL BASED ON AN IRON – COPPER COMPOSITE POWDER

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1. Introduction

The materials obtained by the powder metallurgy have unique properties that cannot be attained by other methods of metallurgical production [1]. In this case, the quality of powder products significantly depends on the technological regimes that provide high functional properties. A characteristic disadvantage of parts obtained by the powder metallurgy is the residual porosity [2], which is due to the feature of compacting powder particles at the pressing stage, during which a porous workpiece is formed, which then undergoes heat treatment – sintering. In fact, sintering should ensure the formation of a dense material due to the diffusion processes occurring between the particles of the powder [3]. Due to various objective and subjective factors (quality of the initial powder, deviation from the technological regimes, and the use of non-matching forma-

tion technologies), a certain percentage of pores remains in the powder materials, which reduces the performance of the parts. Taking into account the advantages of the powder metallurgy method such as a high coefficient of material use and the provision of precise chemical composition, the development of methods for obtaining high-density products with high functional properties is an urgent task of research.

2. Literature review and problem statement

There are two basic approaches to the quality of powder-including parts based on iron. The first is the improvement of the characteristics of the initial powders and their mixtures; the second is the application of special technologies that provide a high density of the product. Papers [4, 5]

show traditional technologies that allow for high-density powder products, including hot stamping and hot pressing. The disadvantage of these technologies is the need to manufacture a special press tool, which is expensive and has a small work life (for example, graphite moulds). There is a technology for rolling metal powders, which produces dense billets [6], but the disadvantage of this technology is the impossibility of manufacturing products of complex shape. It is known from [7] that porous frames can be saturated with low-melting metals. However, this technology is sufficiently energy intensive and requires special equipment, which also limits its use in the manufacturing practice of powder metallurgy. That is why the most common way of forming iron powders is static pressing in hard press moulds, followed by sintering of the received presses, which is confirmed by the results of work [8]. It was shown in [9] that the density of the powder during the formation of the billets depends on its characteristics, primarily on the size, shape, granulometric composition, and particle surface morphology.

Today, most metal powders, including iron, are produced by atomizing melts, which determines the shape and size of the particles obtained [10]. Typically, atomized powders have a spherical shape of particles of 50 μm or more, which results in good sealing and high bulk density and fluidity. However, the lack of spherical powders is poorly formed due to the low surface area of the contact surface, which, in turn, complicates the processes of sintering and requires the use of higher temperatures and a long time of isothermal exposure to the presses during sintering. To eliminate these defects, it is necessary to conduct preparatory operations with spherical powders, which help increase the physical and technological properties of iron powder. It is these approaches that were used in [11, 12] to improve the characteristics of the powders by rolling, regenerating annealing, treatment with aqueous solutions of acids, and plating of particles. Each of these methods changes certain characteristics of the powder. Thus, the recovery of oxide films leads to increased plasticity and, consequently, density of the powder. Acid treatment provides high roughness of the surface of the particles and better formability. Rolling and plating change the shape and increase the fluidity, bulk density, and compaction.

However, some methods have limitations in production due to the dangers such as the use of concentrated acids or explosive remnants. Therefore, one of the most promising ways to change the physical, chemical and technological properties of powders is to apply plating on their surface by chemical precipitation from solutions of metal salts. This method is relatively inexpensive as does not require special equipment and scarcity materials [13]. The advantage of the method of chemical precipitation of metals from salt solutions is also the possibility of applying various materials with their even distribution on the surface of particles. One of the most common alloying elements for structural powder materials on the iron base is copper [14], which improves mechanical properties and regulates shrinkage during sintering of products. In this connection, the most expedient direction is to study the features of the process of chemical precipitation of copper on iron powder. However, despite the advantages of the method of chemical deposition, there are no systematic data and clear formulations on the plating of iron powders of copper, which necessitates such studies.

3. The aim and objectives of the study

The aim of the work is to study the influence of copper plating modes on the change of chemical, physical and technological properties of iron powder particles and to obtain a compact material based on such composite powders with high physical and mechanical properties.

To achieve the aim, the following objectives were set and done:

- to investigate the influence of the time of chemical precipitation of copper on the change in the composition, morphology, shape and size of particles of iron powder;
- to research the influence of copper plating on the technological properties of iron powder: fluidity, bulk density, and compaction;
- to study the structure, chemical composition and physical and mechanical properties of the material on the basis of the iron-copper composite powder.

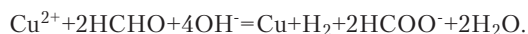
4. Methods of studying the influence of time of chemical deposition on the physical and technological properties of powders

4. 1. Methods and materials for chemical precipitation of copper on particles of iron powder

Water-atomized iron powder of grade PZhRV 200.28 was used, with a mean particle size of 100 μm ; the copper source was copper sulphate $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$; the complexing agent was glycerol; the alkali was caustic sodium; and the reducing agent was a solution of formaldehyde (30–40 mg/dm^3). It was found that the main factors influencing the reaction and deposition of the plating on particles of iron powder are the composition and pH of the solution [15, 16]. The main advantage of chemically deposited copper plating is that the process is carried out at room temperature. In this case, the chemically deposited copper is more elastic in comparison with other chemical platings and can serve as an independent type of plating and underlayer for other platings. The deposition process is based on the recovery of copper from its complex salt formalin in alkaline medium. Two containers were used to prepare the solution. In one vessel in one volume of distilled water, copper sulphate was dissolved, and in the other – in the proportion of distilled water – the caustic sodium and glycerol were dissolved. Then the first solution was poured into the second, stirring thoroughly. When adding a solution of alkali and glycerol to a copper sulphate solution, a copper hydroxide precipitate first appears, which then reacts with glycerol to form a dark blue solution of copper glycerate.

The solution thus prepared can be stored for a long time. Formalin is poured directly before the beginning of copperization. Besides, an additional stabilizing additive was introduced into the solution, which prevented copper recovery in the solution volume and contributed to its maximum deposition on powder particles. As a stabilizer, thiourea was used in an amount of 0.1 mol/dm^3 , which was injected into the bath every 30 minutes. Before the plating process, the iron powder was degreased in gasoline, dried in air, and loaded in the copper bath. In this case, the bath was equipped with a stirrer that rotated with a frequency of 30 rpm, which allowed the powder to be mixed to ensure the full contact of the particles with the solution and achieve an even layer of plating.

Copper recovery occurs in the following reaction:



It was established that the recovery of copper depends on the concentration of copper salt in the solution and the content of the complexing agent as well as increases with an increase in $\text{pH} > 10$. The deposition time was 30 to 180 minutes. Particles of iron powder were obtained covered with a layer of copper plating.

4.2. Methods of studying the properties of plated powders

The chemical analysis of the source and plated iron powders was carried out using an X-ray fluorometre EXPERT 3L (Ukraine).

Metallographic analyses conducted on a microscope NEOPHOT 21 (Germany) at increments from 100 to 1000. Morphology studies were performed on a scanning electron microscope SELM 106 (Ukraine). The granulometric composition of the powders was determined using the Malvern Mastersizer 2000 (United Kingdom) laser diffraction analyser, which was controlled using standardized measurement protocols, or standard operating procedures (SOPs).

The flux density and bulk density of the powders were determined according to standard methods, in compliance with the requirements of ISO 4490 and ISO 3923 for metal powders. Static compression of the powders was carried out in a steel collapsible mould with a diameter of a matrix of 10.0 mm on a hydraulic press PG-10 in the pressure range of 300–800 MPa. Pressed briquettes weighed 8.0 grams, which ensured the ratio of the height of the presses to their diameter of about 1.5. After pressing, the resulting briquettes were measured by the micrometre and weighed on analytical weights according to ISO 3927–77. The densities and porosity of the presses were calculated according to DSTU 18898–89, while the theoretical density of the iron was calculated to be 7.81 g/cm^3 , and the theoretical density of the composite powder when converted to copper content was 7.85 g/cm^3 .

To determine the physical and mechanical properties of the powder materials on the basis of iron, standard techniques were used with all the necessary conditions. Determination of HRB hardness was carried out on the instrument TK 2 by the Rockwell method of pushing a ball onto the surface of the specimen, following the requirements of ISO 4498. Testing of the samples for stretching was carried out by ISO 2740 on the test machine PM 102M. The impact test was carried out on the pendulum impact testing machine KM 5, which meets the requirements of ISO 148 according to DSTU 3669-97 on rectangular specimens of 55 mm in length.

The electrical resistance of the samples was measured using a single-double DC bridge RZOO9 with elemental verification and adjustment, designed for measuring in a range of 10^{-8} to $1.1111 \cdot 10^8 \Omega$ at a DC current of TE 3.454.019.

5. Results of studying the structure and properties of materials on the basis of composite powders

The results of chemical analysis in Table 1 show that copper plating has led to a change in the chemical composition of iron powder. Composite powder contains about 4.5–5.0 %

copper after precipitation for 120 minutes. It was found that in the investigated time interval the amount of copper was from 2.5 to 7.0 %. This amount corresponds to the content of total dissolution of copper in iron at temperatures of sintering of powder materials on the basis of iron powder 950–1300 °C, according to the diagram of equilibrium state of the dual system Fe–Cu [17].

Table 1

The chemical composition of the initial iron powder and plated with copper 120 minutes after deposition

Element	Mass fraction, %	
	Fe	Fe–Cu
Fe	99.482 ± 0.057	94.538 ± 0.027
Cu	0.043 ± 0.016	4.721 ± 0.117
S	0.174 ± 0.055	0.325 ± 0.041
Others	0.301 ± 0.053	0.416 ± 0.098

Thus, the concentration of the solution for deposition provides the required amount of copper in the iron and does not exceed the limit of solubility. Such concentration of the solution in the future should ensure the formation of solid solutions of copper in α -Fe after sintering without intermetallic phases and improve the mechanical properties of the material.

It was found that particles of iron powder were plated with layers of copper of different thickness, depending on the time of deposition (Fig. 1).

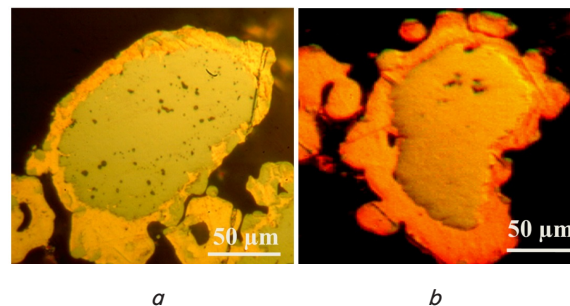


Fig. 1. The structure of particles of copper-plated iron powder after different deposition time:
a – 60 min, b – 120 min

As can be seen from the metallographic analysis, the placement of particles of iron powder leads to a change in their shape. The shape of the particles becomes closer to spherical. In this case, the thickness of the plating on different particles differs even at the same time of depositing, which is due to different surface area and the form of the initial iron particles. Thus, on particles with a more developed surface, a thicker plating layer is observed. Moreover, there is a change in the surface relief of the particles: before plating, the iron powder particles have a more developed non-uniform surface due to the peculiarities of the crystallization process of the droplets of the melt during atomization.

Plating changes the morphology of the surface of iron particles by reducing their roughness and smoothing the relief (Fig. 2).

The study of the kinetics of depositing has shown that after 60 minutes the thickness of the plating is 12–15 μm ; increasing the time to 120 minutes leads to an increase in the plating layer to 18–22 μm (Fig. 3). A further increase in time

does not lead to a significant change in the thickness of the plating, indicating the completion of the reduction process.

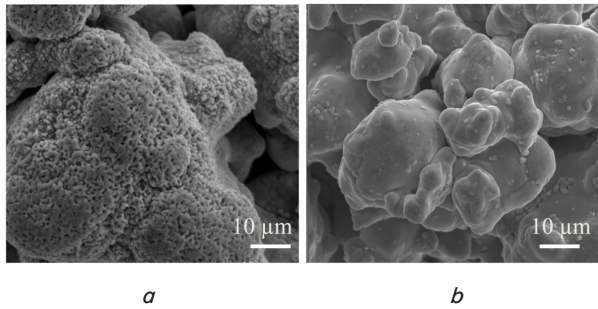


Fig. 2. The morphology of the surface of the particles:
a – iron powder; *b* – copper-plated iron powder

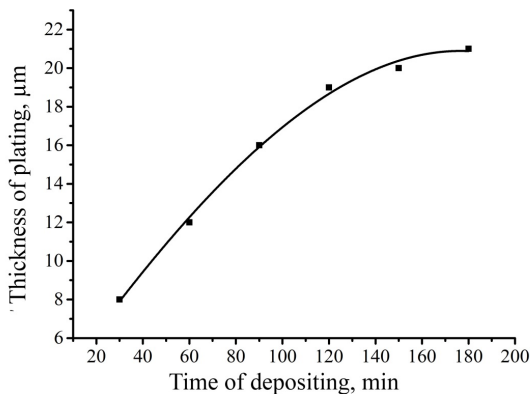


Fig. 3. The dependence of the thickness of the plating on the time of the process of depositing copper on particles of iron powder

In addition to changing the shape of the particles of iron powder, plating also changes the size in the granulometric composition (Fig. 4). Fig. 4 shows that the content of the main fraction of the original iron powder is about 8–9 % with the particle size of 60–100 μm. Pouring iron powder results in an increase in the content of the main fraction to 10–11 % when the particle size is 80–110 μm.

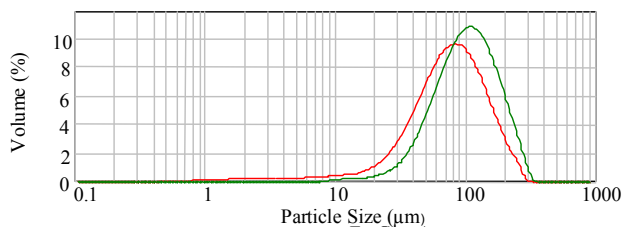


Fig. 4. The distribution of the particle size of the input iron powder and the copper clad powder as to the granulometric composition: — Fe — Average; — Fe-Cu — Average

The shape and size of the powder particles determine its technological properties such as flow and bulk density. The results of the measurements of the technological characteristics of the powders showed that plating of iron leads to an improvement in the yield from 33–34 s/50 g to 28–30 s/50 g, as well as to the growth of the bulk density of the powder from 2.85–2.9 g/cm³ to 3.12–3.15 g/cm³.

An important technological characteristic of metal powders is its sealing in the process of static pressing, which de-

termines the possibility of creating compact presses with low porosity and, accordingly, high technological strength. This allows working with raw presses without any risk of damage to sintering. Fig. 5, *a* shows the dependence of porosity of powdered briquettes on the pressure of pressing.

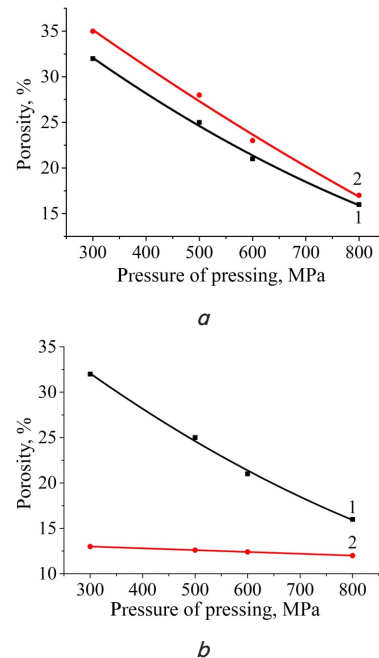


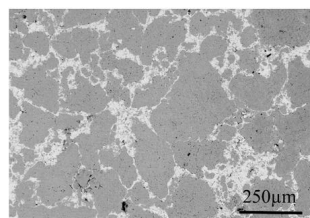
Fig. 5. The dependence of porosity of the powder briquettes on the pressure of pressing and recompacting:
a – 1 – copper-clad iron powder; 2 – initial iron powder;
b – 1 – iron powder clad with copper after pressing;
2 – iron powder clad with copper after recompacting at 800 MPa

As can be seen from Fig. 5, *a*, the plating of particles of iron powder reduces porosity of briquettes by several per cent. At low pressures, the difference in the values of porosity of the presses reaches 6–7 %. With increasing pressure of pressing from 600 MPa, the difference in porosity decreases to 1–3 %. The reason for this difference in porosity is due to the difference in the mechanisms of sealing of plastic metal powders at different pressures. At low pressures, compression happens due to the structural deformation of the particles, which is characterized by the destruction of bridges and the regrouping of particles. Accordingly, an increase in the particle size leads to a decrease in the specific surface, which facilitates a better slip of the powder particles relative to one another. At high pressing pressures, the main factor affecting the density of powders is the plasticity of the material. Iron powder plating with copper can increase the plasticity of the material and improve its deformation.

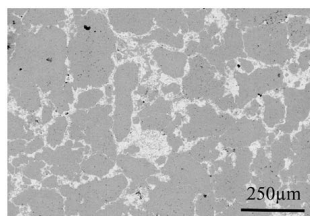
To determine the effect of copper plating on the density of iron, a re-deformation of powdered briquettes was carried out by statically recompacting them in a collapsible mould at a single pressure of 800 MPa (Fig. 5, *b*). It is shown that recompacting of briquettes leads to a decrease in the total porosity of the material to 11–13 %. In this case, recompacting of briquettes at one pressure results in the unification of their porosity, regardless of the porosity of the briquettes after the primary pressing. This is due to the fact that the recompacting eliminates the zone of non-prescription of the central part of the briquettes, which is typical for the condi-

tions of static pressing of the powder in strong press moulds and unifies the density of briquettes in height. A relatively small increase in the density of briquettes after second pressing (5–6 %) is due to the fact that recompacting was carried out without prior annealing, which could have increased the plasticity of the briquettes.

The analysis of the structure of the samples obtained from the composite powder showed that after compression, there is a consolidation of particles of iron powder, surrounded by a layer of copper plating (Fig. 6). In this case, neither pressing nor recompacting leads to the destruction of copper plating, which retains a solid layer around the iron particles.



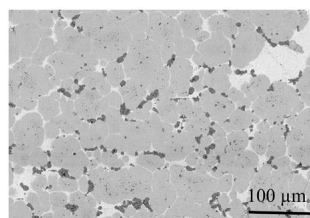
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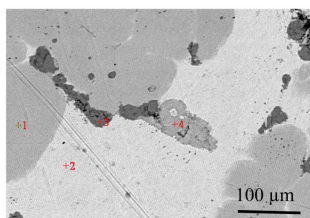
b

Fig. 6. The microstructure of the samples on the basis of iron powder clad with copper: *a* – after pressing at 800 MPa; *b* – after recompacting at 800 MPa

Sintering of the samples at a temperature of 1150 °C for 1 hour in vacuum results in the material compaction to porosity of about 4–5 %, which occurs as a consequence of liquid-phase sintering. At the same time, there is a thinning of the plating layers due to the diffusion of copper in the iron (Fig. 7).



a



b

Fig. 7. The samples on the basis of composite powder after sintering at 1150 °C: *a* – the microstructure of the material; *b* – the renegenspectral analysis

As shown by the X-ray spectral analysis of the material (Table 2), after the sintering, there was a partial dissolution of copper in the iron with the formation of the solid solution and the preservation of thin copper layers in the structure of the material. In this case, the boundaries of the interaction of iron grains and copper are observed in layers saturated with sulphur, which is due to the modes of chemical precipitation of copper from sulphuric salt.

Table 2

The chemical composition of the material based on composite powder after sintering at 1150 °C

Element	Content, %			
	1	2	3	4
Fe	95.29	5.5	99.03	18.24
Cu	4.71	94.5	0.97	59.13
S	–	–	–	22.63

Formation of solid solutions of copper in iron strengthens the material, expanding areas of using the powder products. At the same time, the necessary condition that ensures high properties of the material based on iron is to limit the amount of copper to 8 % in accordance with the diagram of the state of the system of iron and copper. The employed method of chemical deposition enables clear adjustment of the amount of copper that gets deposited on the iron powder.

The results of the tests of the physical and mechanical properties are shown in Table 3.

Table 3

The mechanical properties of powder materials on the basis of iron powder sintered at a temperature of 1150 °C for 1 hour, in vacuum

No.	Material	σ_0 , MPa	σ_i , MPa	KCV, kJ/cm ²	HRB
1	on the basis of the original iron powder	360	620	46	62
2	on the basis of iron powder clad with copper	320	680	61	79

The results of the tests of the mechanical characteristics have shown that the materials of clad powders have greater bending and impact strength values of 680 MPa and 61 kJ/cm², respectively. Although slightly inferior to tensile strength, this is due to reduced plasticity and increased hardness. Determination of the hardness of the obtained materials showed that the materials based on composite powders have a post-sintering hardness of 78–80 HRB. This is 20 units more than the iron powder sintered in the same modes (60–62 HRB).

The specific electrical resistance of the sintered material from the composite powder is 3.5–4.5 μΩ·cm, which is almost 3 times less than the specific electrical resistance of the sintered iron (11–12 μΩ·cm). This distinction is also explained, in addition to the smaller number of pores act-

ing as dielectrics, by the formation of a continuous chain of copper layers in the structure of a material of composite powder, which provides better electrical conductivity of the material.

6. Discussion of the results of studying the influence of plating on the characteristics of composite powders and materials

The results of the tests have shown the possibility of forming a solid layer of copper plating on particles of iron powder, which changes the shape, size and morphology of the surface of the particles (Fig. 1, 2). It should be noted that the thickness of the plating depends, first of all, on the kinetics of the precipitation process (Fig. 3). By changing the deposition time, the thickness and size of the deposited particles can be adjusted (Fig. 4).

Changing the physical properties of the powder leads in turn to changes in the technological characteristics of the iron powder. The growth of technological characteristics is due to several reasons: firstly, changing the shape and increasing the size of the particles leads to a decrease in their specific surface, and secondly, plating leads to the smoothing of the surface, which reduces the roughness of the surface of the particles. These circumstances collectively affect the reduction of friction between the particles of powder, which contributes to their better repositioning and increases bulk density.

The study of the compression process of composite powders has shown a difference in the densification of the plated powders from the input at the first stage of structural deformation (Fig. 5, *a*). This is explained by the fact that the mechanism of the first stage of powder compacting is based on the processes of repositioning the particles, and the main factors influencing this process are the shape, size and morphology of the surface of the particles. The second pressing, or recompacting, of the briquettes from composite powders can reduce the total porosity to 11–13 %, which is due to the high plasticity of clad iron powders (Fig. 5, *b*).

The study of the structure of pressed and sintered materials has shown that the chemical deposition of iron powder with copper allows for uniform distribution of copper over the entire volume of the material (Fig. 6, 7). The uniform distribution of copper in the structure of the samples provided an increase in

the physical and mechanical properties of the material based on iron (Table 3) and the reduction of its electrical resistance.

The tests have shown that iron powder plating provides an improvement in its properties at the level of powders that are annealed in a reducing medium. However, the advantage of plating is the simultaneous change in the characteristics of the powder with its alloying, with a significant reduction in the cost of the process. Therefore, chemical deposition can be recommended as a preparatory operation, which effectively helps change the technological properties of the iron powder obtained by atomization.

Of course, it should be taken into account that the chemical precipitation process is optimal for an iron powder with a particle size of at least 50–100 μm , since for particles of smaller fractions, the process of applying copper is complicated due to their large specific surface.

In the future, it would be essential to compare the physical and mechanical properties of powder materials, depending on the method of introducing copper: chemical precipitation and mechanical mixing. Such an integrated approach would clearly identify the advantages and disadvantages of composite powders.

7. Conclusion

1. The kinetics of the process of chemical precipitation of copper on a powder of iron was investigated. It has been established that after 60 minutes of depositing the thickness of the plating is 12–15 μm ; increasing the time to 120 minutes leads to an increase in the plating layer to 18–22 μm .

2. It has been shown that iron powder plating with copper changes its physical properties, which leads to an increase in the technological characteristics: the yield to 28–30 c/50 g and the bulk density of the powder to 3.12–3.15 g/cm³. The compaction of composite powders at low pressures increases by 5–6 %, and adding composite powder helps obtain material with a total porosity not exceeding 11–13 % without the use of the sintering operation.

3. Plating of iron powder with copper allows obtaining a material with an even distribution of the alloying element throughout the volume, with a high bending strength of 680 MPa, a toughness of 61 kJ/cm², and a hardness of 78–80 HRB. It can be used in the design of economically alloyed powder materials for structural and electrical purposes.

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