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Low temperature gas-phase technology cladding powdered silicon carbide

A technology for applying a metal coating to the surface of powdered silicon carbide has been developed. For this purpose, a film of copper phosphide is initially applied to the surface of the powder. The process consists in wetting the powdered carbide with a solution of copper sulfate and subsequent treatment with gaseous phosphine. The resulting film of metal-like copper phosphide has sufficient electrical conductivity, and is also a catalyst for the process of chemical nickel plating. This makes it possible to use chemical or electroplating methods for plating the surface of powders with the necessary metal. Testing this technology in the laboratory allowed obtaining a nickel coating that has the necessary adhesion to silicon carbide particles.All samples of powders at various stages of the process were examined using a scanning electron microscope ISM-6490-LV (JEOL, Japan), which allows to obtain electronic images (photos) of individual areas (spectra) of the surface at specified magnifications and chemical composition. The results of the scanning electron microscope study showed that the nickel coating contains 7 % phosphorus and covers about 90 % of the powder surface, and the estimated coating thickness is 0.8–1.0 microns. This makes it possible to use such silicon carbide powders in the preparation of composite products or coatings. If necessary, the degree of closure of the powder surface can be increased by re-performing the operation specified in this article. The article is intended for scientists and researchers, who are also interested in the problems of obtaining composite coatings.

Keywords: silicon carbide, copper sulfate, phosphine, copper phosphide film, chemical nickel plating, composite material.

Introduction

Modern production requires the creation of new engineering materials that can be used as special materials in fillers for composites. Such materials can be metalized dielectric powders that have thin-film metal coatings on the surface. Combining the useful properties of dielectrics, such materials have unique properties: high strength, thermal and heat resistance, electrical conductivity and can be used as dual-use materials [1–5].

The main purpose of coating is to improve the retention of grain in the tool matrix, increase the thermal conductivity of the working layer, which allows intensifying cutting modes and thereby increasing the efficiency of the tool. This is especially important in the production of abrasive powders that can be used in the manufacture of various tools, as well as an independent product.

In addition, the metallization of particles can significantly improve the process of feeding the powder into the plasma jet, which has a positive effect on the uniformity of the formed coating, reduce the oxidation rate of sprayed powders (for example, carbides), reduce the porosity of coatings, increase the adhesion strength of coatings to the substrate and increase their wear resistance [6–8].

One used for this purpose is silicon carbide. Because of its increased hardness, silicon carbide is widely used in engineering. On the basis of silicon carbide, lining wear-resistant plates, o-rings and bushings, reaction-bonded ceramics are produced. Rings and bushings made of wear-resistant silicon carbide material are designed for sealing shafts and sliding bearings in pumps that pump oil, petroleum products, liquefied hydrocarbon gases, organic solvents, neutral liquids, as well as acids (except fluoric acid) and salt solutions at temperatures from –100 °C to +500 °C. The products are suitable for use in any designs of friction pairs with the presence of lubrication or self-lubrication [9–11].

The literature describes many methods for applying metal films to dielectric materials that can be used for applying films to silicon carbide. The most commonly used of these are so-called physical and chemical methods. In the physical method, pre-molten metal particles are sprayed onto the coated surface with compressed air or gas. Liquid metal particles on the way to the surface are partially cooled and only melt the surface, sticking to it. A variation of this method is vacuum metallization [12].

For chemical deposition of metal coatings, reducing agents in the gas phase or compounds dissolved in an electrolyte solution are used [13–15].

When using hydrogen gas as a reducing agent, the reduction reaction is carried out at a high temperature of several hundred degrees. In this way, it is possible to obtain coatings from a fairly large number of metals, but the need to have special equipment for heating and dosing gas mixtures limits the wide application of the method.

Thermal decomposition reactions are often used for metallization in the gas phase. The most suitable compounds for this purpose are metal carbonyls. During the reaction, under certain conditions, they decompose, leaving a metal on the coated surface and releasing carbon monoxide, which can again be used to produce metal carbonyl. That is, CO plays the role of a metal carrier reagent. This is not only convenient in terms of production, but also minimizes the unproductive costs of auxiliary reagents, and eliminates environmental pollution. The relative high temperature of the process and the high cost of metal carbonyls prevent the widespread use of this method [9].

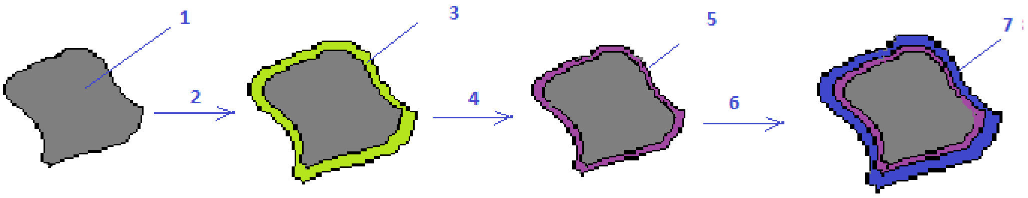
In chemical-electrolytic metallization [13–15], to create an electrically conductive layer, the dielectric surface is activated by creating catalytic centers, which are then coated chemically.

Chemical-electrolytic technology has limitations associated with the use of expensive substances, the use of high temperatures, and the inability to apply coatings on highly porous materials.

The above analysis shows that the task of developing effective methods for applying metal coatings to powdered silicon carbide is relevant.

Experimental

In this paper, we consider the technology of metallization of silicon carbide powders by preliminary application of metal-like copper phosphide (Fig. 1). At the same time, the process of obtaining a copper-phosphorus film is based on the reaction of reducing divalent copper with gaseous phosphine. To do this, first a film of copper salt solution is obtained on the surface of the sample, and then this surface layer is treated with phosphine. In this case, the film of the copper salt solution is converted to copper phosphide. Sulfates, nitrates, and copper chlorides were investigated as copper salts. However, in most experiments we used the available and cheaper copper sulfate.



1 — separate particle of silicon carbide; *2* — wetting in a solution of copper sulfate;
3 — sorption layer of copper sulfate; *4* — treatment with phosphine; *5* — a film of copper phosphide;
6 — chemical nickel plating; *7* — nickel coating

Figure 1. Scheme of metallization of silicon carbide particles

The initial powders are degreased in a 15–20 % solution of hydroxide or sodium carbonate at a temperature of 40–500 °C until the surface of the particles is completely wetted, washed in hot running water. Then a layer of metal-like copper phosphide is applied to the surface of the powder particles. To do this, the powder is immersed in a solution containing 100–200 g/l $CuSO_4 \cdot 5H_2O$. The ratio of the volume of the powder to the volume of the solution is 1:1.2–1.5 at concentrations of copper sulfate less than 100 g/l, the resulting copper phosphide film will have: a thickness insufficient to build up the metal with a chemical or galvanic metal. At concentrations of copper sulfate more than 200 g/l due to the formation of large crystals of copper sulfate in the subsequent drying of the powder increases the heterogeneity of the thickness of the resulting films. The remnants of the solution are not adsorbed by the surface of the powder is separated by filtration. After that, a sorption layer of the solution with a thickness of 50–70 microns is formed on the surface of the powder particles. The powder is dried for 40–50 minutes at a temperature of 18–25 °C to create gas permeability between the individual particles. The dried powder is placed in a sealed chamber and treated with phosphine gas. Phosphine for research was obtained by acid decomposition of powdered technical zinc phosphide. This phosphide contains about 22 mass % of phosphorus and therefore it can be considered that the main component is zinc phosphide with a small amount of free zinc.

When treated with phosphine in the metallization chamber a reaction occurs between phosphine and the adsorption film of the copper sulfate solution leading to the formation of copper phosphide by reaction:

$$6CuSO_4 + 3PH_3 + 3H_2O \rightarrow 2Cu_3P + 6H_2SO_4 + H_3PO_3 \quad (1)$$

A small amount of phosphine remains in the gas environment after its reuse, so this gas must be purified. For this purpose, the residual gas was passed sequentially through a layer of copper carbonate and an oxidizing solution (500 g/l of potassium permanganate). After this treatment, phosphine is not detected in the waste gas.

During the experiments, 10 g of skimmed silicon carbide powder was filled with 10 ml of $CuSO_4 \cdot 5H_2O$ (200 g/l). After stirring for 5 minutes, the excess solution was filtered out. The powder was transferred to a Petri dish and dried at room temperature until moist state (40–50 minutes).

A Petri dish with dried powder was placed in a sealed chamber. The air was forced out of the free space of the chamber with nitrogen. Then phosphine was fed into this chamber by forcing it out of the bottle with water. At the same time, phosphine was absorbed by the surface film of copper sulfate.

The phosphine treatment process took place at room temperature for several minutes.

Further build-up of the metal film was carried out by chemical Nickel plating in a solution of the following composition: $NiSO_4 \cdot 7H_2O$ — 30 g/l; NaH_2PO_2 — 10 g/l; CH_3COONa — 10 g/l, at an electrolyte temperature of 90 °C for 30 minutes.

Samples of powders at various stages of the process were examined using a raster electron microscope ISM-6490-LV (JEOL, Japan), which allows obtaining electronic images (photos) of individual sections (spectra) of the surface at specified magnifications. The microscope made it possible to simultaneously obtain the elemental compositions of the spectra in the form of a table.

Results and Discussion

Figure 2 shows an electronic image of carborundum powder after applying a film of copper phosphide. The spectral analysis in Table 1 shows that the content of this phosphide (Cu+P) is about 44 %. However, the entire surface of the powder has a light gray tone characteristic of metal-like copper phosphide and the presence of silicon is due to the fact that due to the small thickness of the copper phosphide film in some areas, the scanning probe partially captures the signals of the lower layer of silicon carbide. If this is the case, then after the deposition of a sufficiently thick layer of chemical nickel, the signals of the lower layer should disappear. In addition, the phosphide film has good adhesion to the carbide particles. This is due to the fact that the copper salt solution penetrates all the pores of silicon carbide, which become gas-permeable as the solution dries. Therefore, the course of the reduction reaction in the pores also provides good adhesion of the film with silicon carbide.

Table 1

Elemental composition of carborundum powder

Element	Weight, %
C	12.68
O	13.79
Al	0.92
Si	25.76
P	7.69
Cu	36.75

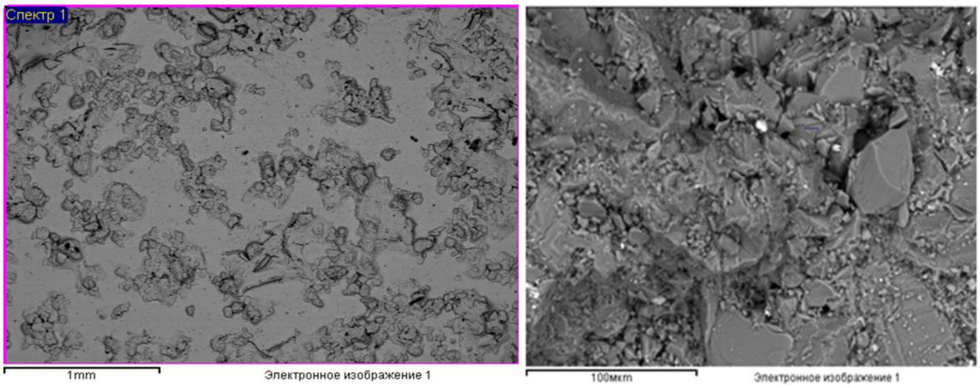


Figure 2. An electronic image of the silicon carbide powder after application of a film of copper phosphide

Copper phosphide is a catalyst for the chemical Nickel plating process, so nickel deposition occurs on the surface of silicon carbide particles covered with a phosphide film, including in the pores. This should also provide the necessary adhesion of the coating to the base. In addition, the process of chemical nickel plating on the surface of copper phosphide is characterized by increased gas release [16, 17]. This leads to some fluctuations of the powder particles and contributes to the formation of a nickel coating over the entire surface of the powder particles. Therefore, such a coating will provide the necessary adhesion between the carborundum particles and the matrix in the preparation of the composite product or coatings.

The reaction occurring in this case is described by the following chemical equations:

$$NaH_2PO_2 + H_2O \rightarrow NaH_2PO_3 + 2H^+ + 2e^- \qquad (2)$$

$$NaH_2PO_2 + 2H_2O \rightarrow NaH_2PO_4 + H_2 + 2H^+ + 4e^- \qquad (3)$$

$$3NaH_2PO_2 - 2e^- \rightarrow Na_3PO_3 + 2P + 3H_2O \qquad (4)$$

$$5NaH_2PO_2 - 4e^- \rightarrow Na_3PO_4 + 4P + 2NaOH + 4H_2O \qquad (5)$$

$$Ni^{2+} + 2e^- \rightarrow Ni \qquad (6)$$

$$2H^+ + 2e^- \rightarrow H_2 \qquad (7)$$

As can be seen from these equations, the process of chemical nickel plating is accompanied by the release of hydrogen. In addition, elemental phosphorus is also released, which gives the coating additional hardness.

Figure 3 shows the electronic image and the elemental composition (Table 2) of the powder after applying a layer of chemical nickel. The estimated coating thickness is 0.8–1.0 microns.

This data shows that the nickel coating contains 7 % phosphorus and covers about 90 % of the powder surface. This makes it possible to use such silicon carbide powders in the preparation of composite products or coatings. If necessary, the degree of closure of the powder surface can be increased by repeated conduct of the above operations.

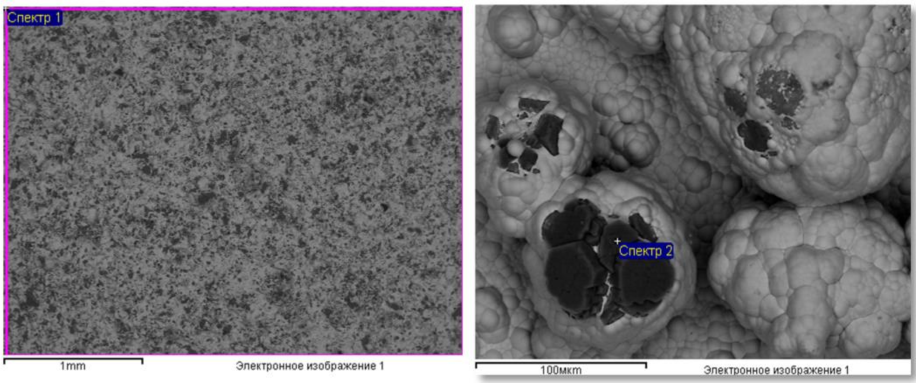


Figure 3. An electronic image of silicon carbide powder after application of chemical nickel

Table 2

Elemental composition of silicon carbide powder after application of chemical nickel

Element	Weight, %
C	7.18
O	2.38
Al	0.39
Si	13.22
P	5.48
Ni	71.35

In a similar work [18], a nickel coating was obtained by electrodeposition. This method is very well known, but has its drawbacks. The main disadvantage is the reduced hardness and complexity of production.

Conclusions

Phosphine gas is used for applying metal coatings to powdered materials. Thus, for metallization of silicon carbide powder, an adsorption layer of copper sulfate solution is first created by wetting on its surface, which is then transformed by phosphine treatment into a metal-like copper phosphide. Copper phosphide is a catalyst for the chemical nickel plating process. This allows you to apply a layer of chemical nickel. Although the thickness of the copper phosphide film is insignificant, it catalyzes the chemical nickel plating process well. This allows you to apply a coating of the desired thickness to the surface of the silicon carbide powder.

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Ұнтақ тәрізді кремний карбидін қаптаудың газофазды төментемпературалы технологиясы

Ұнтақ тәрізді кремний карбидінің бетіне металл қаптамасын төсеудің технологиясы әзірленген. Бұл мақсат үшін ұнтақ бетіне бастапқыда мыс фосфидінің қаптамасын қолданады. Үрдіс ұнтақ тәрізді карбидті мыс сульфаты ерітіндісімен сулап, газ тәрізді фосфинмен өңдеуден тұрады. Бұл ретте түзілетін металл тәрізді мыс фосфидінің қаптамасы жеткілікті электр өткізгішіне ие, сондай-ақ химиялық никельдеу процесінің катализаторы болып табылады. Бұл ұнтақтың бетін қажетті металмен қаптауға арналған химиялық немесе гальваникалық әдістерді пайдалануға мүмкіндік береді. Осы технологияны зертханалық жағдайда тексеру кремний карбидінің бөлшектерімен қажетті ұстасуы бар никель қаптамасын алуға мүмкіндік берді. Процестің әртүрлі сатыларында ұнтақтардың барлық үлгілері ISM-6490-LV (JEOL, Жапония) растрлық электрондық микроскоп көмегімен зерттелген. Растрлық электрондық микроскоп әдісімен жүргізілген зерттеу нәтижелері никельді қаптаманың құрамында 7 % фосфор бар және ұнтақ бетінің шамамен 90 % жабатынын көрсетті, ал қаптаманың қалыңдығы 0,8–1,0 мкм құрайды. Бұл композициялық бұйымдарды немесе қаптамаларды алу кезінде кремний карбидінің осындай ұнтақтарын қолдануға мүмкіндік береді. Қажет болған жағдайда ұнтақ бетін қаптау дәрежесін осы мақалада көрсетілген операцияны қайта жүргізу арқылы іске асыруға болады. Мақала ғалымдар мен зерттеушілерге, сондай-ақ композициялық қаптамаларды алу мәселелеріне қызығушылық танытатын тұлғаларға арналған.

Кілт сөздер: кремний карбиді, мыс сульфаты, фосфин, мыс фосфидінің қаптамасы, химиялық никельдеу, композициялық материал.

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Низкотемпературная газофазная технология плакирования порошкообразного карбида кремния

Разработана технология нанесения металлического покрытия на поверхность порошкообразного карбида кремния. Для этой цели на поверхность порошка первоначально наносят пленку фосфида меди. Процесс заключается в смачивании порошкообразного карбида раствором сульфата меди и последующей обработке газообразным фосфином. Образующаяся при этом пленка металлообразного фосфида меди обладает достаточной электропроводностью, а также является катализатором процесса химического никелирования. Это дает возможность использования химических или гальванических методов для плакирования поверхности порошков необходимым металлом. Проверка данной технологии в лабораторных условиях позволила получить никелевое покрытие, имеющее необходимое сцепление с частицами карбида кремния. Все образцы порошков на различных стадиях процесса исследовались при помощи растрового электронного микроскопа ISM-6490-LV (JEOL, Япония), позволяющего получать электронные изображения (фотографии) отдельных участков (спектров) поверхности при задаваемых увеличениях и химический состав. Результаты исследования методом растрового электронного микроскопа показали, что никелевое покрытие содержит 7 % фосфора и закрывает около 90 % поверхности порошка, а расчетная толщина покрытия составляет 0,8–1,0 мкм. Это дает возможность применения таких порошков карбида кремния при получении композиционных изделий или покрытий. При необходимости степень закрытия поверхности порошка можно увеличить повторным проведением операции, указанной в данной статье. Статья предназначена ученым и исследователям, а также лицам, интересующимся проблемами получения композиционных покрытий.

Ключевые слова: карбид кремния, сульфат меди, фосфин, пленка фосфида меди, химическое никелирование, композиционный материал.

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