# A galvanic-chemical method for preparing diamond containing coatings 

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## HIGHLIGHTS

- A new method to prepare diamond containing coating is presented.
- The material is dipped in $\mathrm{CuSO}_{4}$ and diamond particles are sprayed on the surface.
- Phosphine is used to convert $\mathrm{CuSO}_{4}$ into copper phosphide.
- The diamonds are finally enclosed in a copper matrix through electroplating.


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GRAPHICALABSTRACT



#### Abstract

The formation of a diamonds containing coating on the surface of tools is a way of increasing hardness and wearability of the working surfaces. We have developed a new process to prepare these composite coating. The surface is initially wetted with copper sulphate solution and then diamonds powder is sprayed onto the surface. Upon subsequent exposure to gaseous phosphine, a surface layer of solid copper phosphide, firmly holding the diamond particles, is formed. Copper phosphide has sufficient electrical conductivity, allowing conventional metal electro-plating. The effect of process parameters such as duration of the exposure to phosphine gas, concentration of the $\mathrm{CuSO}_{4}$ solution and thickness of the electro-deposited copper layer have been investigated and the surface roughness of the prepared samples increased about 50 BHN (Brinell hardness number).


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## 1. Introduction

The formation of a composite coating layer containing diamond on the surface of materials is a method to prepare surfaces with higher hardness and wearability. Methods of producing abrasive surfaces, grinding belts and other metal-based tools by introducing

[^0]diamond into a metal surface through a gas stream or plasma are known [1-5]. When using a gas-thermal or plasma-chemical methods to produce diamond-containing composite coatings, pre-clad powders of synthetic or natural diamond are used [5-7]. Metallisation of diamonds grains induces a significant improvement of their performance when in the coating layer, especially at elevated processing conditions. Micro-cracks present in diamond grains are filled and the grains are encased in a metal shell, such coatings increase the strength of diamond powder, which leads to enhanced productivity of the tools $[8,9]$.

An alternative method to prepare composite coatings containing diamonds is electroplating. In such process the non-metallic

Table 1
Element analysis of diamond powder ACM brand 5.

| Element | Content (wt.\%) |
| :--- | :--- |
| C | 99.6 |
| Si | 0.25 |
| Ca | 0.20 |
| Fe | 0.04 |

phase (diamond), in the form of dispersed particles, is distributed in an electrolyte solution, and, when it reaches the surface of the cathode, it undergoes metallisation. In this way, diamonds containing copper, nickel and chrome have been produced [10-13]. The disadvantages of the electrochemical process for producing composite coatings include the need for intensive mixing of the electrolyte, which complicates the design of the plating bath; difficulty in obtaining coatings with high content of non-metallic phase and the need for filtration of the electrolyte.

One of the important properties of diamond is its high biocompatibility compared to the other substances. This has led to the use of different kinds of natural and synthetic diamond in medicine [14-16]. For these purposes, diamond-like coating (DLC), obtained by high-temperature decomposition of hydrocarbons, has found widespread use. These carbon-based materials are not pure diamond, but they have the structure and physicochemical properties similar to them $[17,18]$. Surfaces of prosthetic devices and implants having a diamond or diamond-like coatings are chemically inert, non-toxic for cells and do not cause inflammation or pathogenic processes [19].

In this work, we present an inventive method to introduce diamonds in a composite coating, based on the preliminary fixing of the diamonds on the surface of the product and a subsequent overgrowth by metal, adapting previous approaches based on the generation of a sorption layer of copper sulphate on the surface of metallic or non-metallic products, subsequently this layer is treated with gaseous phosphine resulting in the formation of a metal-like copper phosphide layer on the product surface [20,22].

## 2. Materials and methods

### 2.1. Chemicals and substrates

To study the process of obtaining diamond-containing composite coatings flat copper samples with a width of 2 cm , length of 6 cm and a thickness of 0.5 cm were used; when immersed in the respective solutions the working height was of 5 cm .

Synthetic diamond powder ACM grade 5 (Russia), which is produced by sympathetic detonation were used as the non-metallic phase of the composite coating; the composition of the diamond crystals is presented in Table 1. From Fig. 1 it is clear that this powder comprises prismatic particles with an average size of $0.5-1.0 \mu \mathrm{~m}$. Table 1 shows the results of spectral analysis of diamond crystals, which exhibit a relatively high purity diamond powder.

### 2.2. Diamond containing coating formation

The process of obtaining composite coatings is schematised in Fig. 2 and consisted of the following consecutive steps:
(i) preconditioning of samples;
(ii) creation of a surface layer of copper sulphate solution;
(iii) deposition of diamond powder onto the sample surface;
(iv) consolidation of diamond particles; and
(v) application of a layer of a metal.


Fig. 1. SEM of diamond powder ACM brand 5.

For the preconditioning of the samples degreasing and etching operations were performed as in other electroplating methods [20]. A surface layer of $\mathrm{CuSO}_{4}$ solution was achieved through dipping the samples for $3-5 \mathrm{~s}$ in a $\mathrm{CuSO}_{4}$ solution ( $200 \mathrm{~g} / \mathrm{l}$ ) followed by shaking to remove liquid excess.

Spraying of diamond powder onto the surface sample was conducted with the device shown in Fig. 3. To prevent any premature drying of the surface film of $\mathrm{CuSO}_{4}$ solution, the spraying time did not exceed $20-30 \mathrm{~s}$. The mass of the diamond powder sprayed was $1.6-1.9 \mathrm{mg} / \mathrm{cm}^{2}$.

In order to fix the particles of diamond powder onto the sample surface was treated by phosphine-containing gas by the technique described in our previous work [22]. This briefly requires the placing of the sample in a hermetically sealed chamber that is filled with phosphine-containing gas produced by acid decomposition of zinc phosphide
$\mathrm{Zn}_{3} \mathrm{P}_{2}+6 \mathrm{HCl} \rightarrow 3 \mathrm{ZnCl}_{2}+2 \mathrm{PH}_{3} \uparrow$
During this step, gaseous phosphine (at room temperature) reacts with copper sulphate in the liquid phase generating copper phosphide according to the following reaction:
$6 \mathrm{CuSO}_{4}+3 \mathrm{PH}_{3}+3 \mathrm{H}_{2} \mathrm{O} \rightarrow 2 \mathrm{Cu}_{3} \mathrm{P}+6 \mathrm{H}_{2} \mathrm{SO}_{4}+\mathrm{H}_{3} \mathrm{PO}_{3}$
Usually, the process required $7-10 \mathrm{~min}$ and the remaining unreacted phosphine-containing gas was neutralised by blowing through a solution of potassium dichromate.

The final copper plating was carried out using a solution of $\mathrm{CuSO}_{4} \cdot 5 \mathrm{H}_{2} \mathrm{O} \mathrm{g} / \mathrm{l}$ and $\mathrm{H}_{2} \mathrm{SO}_{4} \mathrm{~g} / \mathrm{l}$ as electrolyte: the current density was $100 \mathrm{~A} / \mathrm{m}^{2}$ and temperature of $20^{\circ} \mathrm{C}$.

### 2.3. Kinetics of conversion of copper phosphate with phosphine and copper phosphine film thickness determination

The reaction rate of transformation of copper sulphate into copper phosphide was determined using the apparatus schematically shown in Fig. 4. Its working principle is that the reduction of the water level in the surge vessel 7, over a certain period of time, is linked to the amount of phosphine consumed; this is also dependent on the amount of copper phosphide formed in reaction (2). According to this reaction, the formation of 1 g of copper phosphide requires $150 \mathrm{~cm}^{3}$ (in normal conditions) of phosphine.

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