JOÃO DALLAMUTA
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(ORGANIZADORES)

AMPLIAÇÃO E APROFUNDAMENTO DE CONHECIMENTOS NAS ÁREAS DAS ENGENHARIAS 3

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Ampliação e aprofundamento de conhecimentos nas áreas das engenharias 3

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APRESENTAÇÃO

Caro(a) leitor(a)

Como definir a engenharia? Por uma ótica puramente etimológica, ela é derivada do latim *ingenium*, cujo significado é “inteligência” e *ingeniare*, que significa “inventar, conceber”.

A inteligência de conceber define o engenheiro. Fácil perceber que aqueles cujo oficio está associado a inteligência de conceber, dependem umbilicalmente da tecnologia e a multidisciplinaridade.

Nela reunimos várias contribuições de trabalhos em áreas variadas da engenharia e tecnologia. Ligados sobretudo a indústria petroquímica com potencial de impacto nas engenharias. Aos autores dos diversos trabalhos que compõe esta obra, expressamos o nosso agradecimento pela submissão de suas pesquisas junto a Atena Editora. Aos leitores, desejamos que esta obra possa colaborar no constante aprendizado que a profissão nos impõe.

Boa leitura!

João Dallamuta

Henrique Ajuz Holzmann

Rennan Otavio Kanashiro

SUMÁRIO

CAPÍTULO 5

DIAMOND INTEGRATED COATING BY ELECTROPLATING PROCESS - AN OVERVIEW

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ABSTRACT: The electroplating process is a method widely used industrially to the metal coating on a solid substrate. In recent years, this technique has also been applied to the manufacturing of diamond tools. Such tools play an important role in the most diverse industrial sectors, from the drilling of water and oil wells to high precision machining. Thus, this work aims to bring a review of the main information of the electroplating process for the manufacture of diamond tools. This review paper will talk about the main advances already achieved for this type of technology, as well as the main parameters and conditions used in the industries. The leading applications, challenges, and prospects for this market will also be discussed.

KEYWORDS: Electroplating, diamond tools, diamond deposition, materials engineering.

REVESTIMENTOS DIAMANTADOS OBTIDOS PELO PROCESSO DE ELETROPLATING - UMA VISÃO GERAL

RESUMO: O processo *electroplating* ou *eletrodeposição* é um método muito utilizado industrialmente para revestir substratos sólidos com metais. Nos últimos anos, essa técnica tem sido bastante aplicada para fabricação de ferramentas diamantadas. Tais ferramentas desempenham um importante papel nas mais diversas esferas industriais, desde a perfuração de poços de água e petróleo até usinagem de altíssima precisão. Assim, este trabalho tem como objetivo trazer um levantamento sobre as principais informações acerca do processo de electroplating para a fabricação de ferramentas diamantadas. Esta revisão abordará os principais avanços já alcançados para esse tipo de tecnologia, bem como os mais importantes parâmetros e condições utilizados nas indústrias. As principais aplicações, desafios e perspectivas para esse mercado também serão discutidas.

PALAVRAS-CHAVE: Eletrodeposição, ferramentas diamantadas, deposição de diamante, Engenharia de Materiais

1 I INTRODUCTION

Materials science and engineering have development, creation, and improvement of materials in several fields as the main objective. Such study is based on the relationship between structure, manufacturing process, properties, and application. From then on, it is possible to choose the best material, or set of materials, needed for certain purposes. The innovations within this comprehensive science offer novel technologies, more efficient processes, and better products. (DOBRZAŃSKI, 2006)

Within this context, advances in industrial sectors require more innovations. For this reason, the field dedicated to cutting and wear tools, especially diamond tools, is quite emerging. These tools are mainly coated with polycrystalline diamond (PCD), which consists of a composite of diamond particles sintered from a metal binder. Currently, PCD is one of the hardest and most abrasion-resistant materials in the world. It also shows properties such as chemical resistance and high thermal conductivity. (BARRETO *et al.*, 2020; GURGEL *et al.*, 2020; NEVES, 2017)

Electroplating is one of the most important industrial techniques for applying particulate coating. In this process, a plating bath with suspended particles is used to put on coating on a prepared substrate. In most cases, a complementary metal, usually nickel or cobalt, is used to aid in the deposition and fixation of the hard layer on the substrate. (HE et al., 2012; SUNG J.; SUNG M., 2009)

Based on this principle, scientists applied the electroplating process for the deposition of diamond particles on special substrates, since there is a difficulty in depositing diamond by other traditional methods. Besides, it avoids wasting diamond particles. (TSUBOTA *et al.*, 2005; WU *et al.*, 2019)

This technique is one of the suitable methods due to its simplicity, low cost, low process temperature, easy access to the nanoscale structure as well as the production of high-density and porosity-free coatings. The electroplating is also used as an economical and high productivity method given the fact most time requires just one step, without any secondary operation. (DEZFULI; SABZI, 2019; KANDA *et al.*, 1995)

Besides, compared to other methods, it allows a low cost of the total process, low initial investment value, high productivity, equipment mobility, and less required industrial space. (LI *et al.*, 2007)

Thus, currently, electroplating is highly used to prepare diamond tools. The main manufactured products include cutting and grinding discs, drills, grinding rollers, and shank or thread tools. Other applications consist of tools for high-speed cutting, ultra-precision machining, and ultrasonic vibration techniques, for example. (CUI *et al.*, 2016)

To achieve good properties of the final products, it is very important to understand the function of the electroplating process, as well as the main variables and parameters involved.

2 | THE ELECTROPLATING PROCESS

The electroplating is based on the electrolysis process. This technique consists of immersing a cathode-anode pair in an electrolytic solution composed of the element to be used in the coating. The electrical current must be associated with the system, allowing the metal ions to transfer from the anode to the cathode surface, where the film will grow. (LEE; CHOI, 2001; SUZUKI; KONNO, 2014)

For the manufacture of diamond discs, a steel substrate (cathode), a nickel bar (anode), and diamond particles suspended in an electrolyte with nickel salts are generally used. The diamond particles will be dragged by the movement of the anode particles, depositing together on the substrate. Thus, the deposition of hard particles on the substrate is relatively simple. (REID *et al.*, 2004; SIKDER *et al.*, 1999)

The use of nickel in the electrodeposition process began in 1843, by R. Bottger. He provided a formulation using an aqueous solution of nickel and ammonium sulfates. Over the years, the process was improved by Isaac Adams Jr, who established the use of neutral pH in the solution to obtain better quality control of the deposited nickel. Subsequently, in 1916, Oliver P. Watts developed an electrolyte called a Watts bath. This bath was composed of boric acid, nickel sulfate, and nickel chloride. Due to its high effectiveness, this composition triggered the elimination of nickel sulfates and ammonia, and its formula is still used today. (SCHLESINGER; PAUNOVIC, 2011)

In system assembly, the cathode is the element that will receive deposition. Steel is the most used material, but there are also records of using some types of ceramic materials, such as tungsten carbide or silicon nitride. The anode is the material used for the coating, commonly nickel. To avoid contamination with other ions, the electrolyte must consist of a solution of some anode salt, as nickel sulfate, for example. (CHIBA *et al.*, 2003; ZHOU *et al.*, 2009). Figure 1 shows how the apparatus is usually assembled.

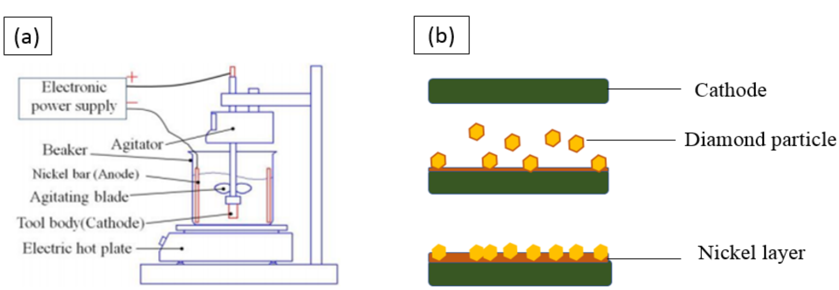

Figure 1: (a) The common configuration of the bath used for electrodeposition. (b) The schematic representation of the coating formation process. Adapted from Zhang *et al.* (2014), and Zhang *et al.* (2016).

At the electrodeposition bath, there is also the presence of diamond particles suspended in the electrolyte. During the current application, these particles will be dragged and deposited into the cathode. The particle characteristics and the size distribution range are defined by the desired application. One the other hand, the thickness of the diamond layer is related to the process parameters, such as bath agitation speed, employed current, temperature, and diamond solution concentration. (PUSHPAVANAM *et al*., 2007; ZHANG *et al*., 2016)

Figure 2 shows the surface schematization of the diamond discs after the electroplating process.

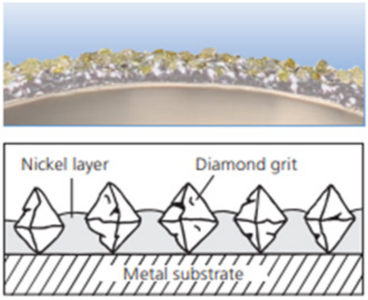

Figure 2: The surface schematization after the electrodeposition of the Ni-diamond coating. Source: PFERD (2020).

3 I ADDITIONAL METHODS FOR IMPROVING DEPOSITION

Over the years, several authors have worked on improving the diamond deposition process. Fletcher (1979) proposed the use of etching to create small grooves in the substrate before the deposition. Such a process creates small cavities in the workpiece surface, thus providing a stronger mechanical connection between the diamond and the workpiece. Figure 3 illustrates the product resulting from the process.

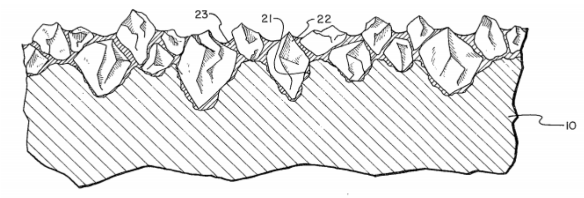

Figure 3: Electrodeposited piece proposed by Fletcher (1979). (10) substrate; (21) cavities made before the electroplating process. (22) diamond particles; (23) metal used to drag diamond particles. Source: Fletcher (1979).

Another option for improving adhesion between the diamond and substrate is the metallization of the diamond particles. Du *et al*. (2020) explain that the diamond bonded on the substrate by a covalent bond has high interfacial energy. Besides, the interfacial bonding force is poor, resulting in a small bond holding force on diamond/substrate. Thus, diamond is easy to fall off prematurely, which reduces the service life and processing efficiency of diamond tools.

Methods such as electroless plating, magnetron sputtering, CVD, and even electroplating itself, can be used to perform metallization and thus reduce the interfacial energy. The metal film enhances the chemical affinity of diamond with metals and their alloys, and that way, improve the interaction with the substrate during the electroplating process. (DU *et al*., 2020; WERNER, 2003)

4 I SUBSTRATE PREPARATION

To obtain a high-quality deposition, a prior cleaning of the involved materials is necessary. Cleaning prevents the presence of oils, greases, and surface impurities, which can hinder adsorption reactions. Several authors have been proposing different methodologies for carrying out this process.

Onikura *et al*. (2003), deposited a diamond layer on a cemented carbide substrate. They performed the traditional ultrasound cleaning with acetone and immersion in sulfuric acid, as well as electrolytic degreasing as an additional step. Degreasing consisted of immersing the substrate in a solution of NaOH, Na_2CO_3, and $Na_3PO_4.12H_2O$, applying a current density of 8.44 and 12.2 A/dm^2, for 3 and 5 minutes. Soon after, the substrate was soaked in sulfuric acid diluted 20 times. As a result, the authors verified that samples without electrolytic degreasing had the diamond layer peeling very quickly, with a useful life quite limited. However, those that underwent this process showed flaking only in small regions. Also, the samples treated for 5 minutes had a better result than the one treated for 3 minutes.

Zhang *et al*. (2016) also indicated a chemical cleaning carried out on carbon steel substrates before depositing Ni/W-diamond. The substrates were washed successively with soap, rinsed with NaOH, HCl, and distilled water; and activated in 14% HCl, to improve the adhesion of the layer.

Zhou *et al*. (2019) studied the cleaning of a NiTi alloy substrate for Ni-diamond deposition, for dental applications. The cleaning consisted of immersing the samples in acetone for 10 min in the ultrasound, with subsequent washing in distilled water. Then, it was immersed in alkaline liquid (unspecified), washed in distilled water, and dipped in a mixed solution of H_2SO_4 and HCl. Finally, it was taken to the electroplating process.

The patent CN1544706A also suggests carrying out a treatment on the diamond particles before deposition. As described in the invention, the particles must be immersed in a 10% NaOH solution for 30 minutes and washed with distilled water. Then, it must be immersed in a 10% HCl solution for another 30 min and washed with water again. In the end, activation with hydrochloric acid is indicated.

5 I PROCESS PARAMETERS

The parameters used in the electroplating process directly affect the quality of the final products. Among them, the main ones are the type of bath, the agitation speed, current density, the type of diamond used, pH, and bath temperature.

5.1 Type of bath

There are two different bath compositions to be used to obtain coating via electrodeposition: Watt Bath and Sulfamate Bath. Both types are similar in their purpose, differing according to the used reagents. It is also known that the Watt bath is more commercially developed compared to the sulfamate one (HE *et al.*, 2012; TSUBOTA *et al.*, 2005).

Table 1 shows the reagents and their average concentrations used for each type of bath.

Watt Bath	Sulfamate Bath
$NiSO_4$ - 200 to 350 g/L	$Ni(NH_2SO_3)_2$ - 300 to 500 g/L
$NiCl_2$ - 20 to 45 g/L	$NiCl_2$ - 5 to 30 g/L
H_3BO_3 - 20 to 35 g/L	H_3BO_3 - 30 to 45 g/L

Table 1: Composition of the baths for the electroplating process.

5.2 Agitation speed

Agitation in the process is essential for electrodeposition to occur, once the diamond particles in suspension must not settle at the container bottom. Rotational movement is also necessary to promote homogeneous particle dispersion, in addition to preventing sites without diamond coating on the substrate. Previous studies indicate the ideal stirring speed is in the range of 300 and 500 rpm. If the speed is too high, the suspended diamond particles can come in contact with the layer already deposited, wearing it away. On the other hand, if the speed is too low, the particles can settle to the bottom of the container, instead of being attracted to the substrate. (LEE; CHOI, 2001; ZHOU *et al.*, 2015)

5.3 Current density

The current density influences the metal deposition speed. If this parameter is extremely high, internal stresses can occur in the coating, leading to cracking and flaking in the final product. Furthermore, as the anode undergoes reduction, if the current density is very high, there will be much more anode deposited on the cathode than the diamond particles themselves. On the contrary, if the current density is low, the metal particles may not have enough speed to drag the diamond particles. As reported in previous works, the conventional current density ranges between 2 and 8 A/dm^2. (LIU *et al.*, 2020; WANG *et al.*, 2014)

5.4 Diamond particles characteristics

The diamond particles used at the electroplating process usually have an average particle diameter below 30 μm. In general, as lower the particle size, better the deposition is. This happens because smaller particles have a higher surface area, optimizing the adhesion of Ni^{2+} ions and diamond particles with the substrate. However, if the particles were too small, agglomeration may occur, and some anti-agglomeration agent should be used. Some studies reported the use of nickel-plated diamonds to facilitate the transport of these particles to the substrate (cathode). Furthermore, diamond concentrations in the bath ranged from 1 to 50 g/L. (MEDELIENE *et al.*, 2003; YUSHCHENKO *et al.*, 1997; ZHOU *et al.*, 2009)

5.5 pH and temperature of the bath

Most electroplating processes occur at acidic pH, usually varying between 3 and 4 by treatment with sulfuric acid (H_2SO_4) or sodium hydroxide (NaOH). Temperatures are controlled throughout the process and generally ranged between 40 and 60 °C. (QIN *et al.*, 2014; ZHOU *et al.*, 2015)

6 I HEAT TREATMENT AFTER THE ELECTROPLATING

Some authors have been reported improvements in adherence and mechanical properties of coatings through heat treatments after the deposition.

Wang *et al.* (2014) studied the heat treatment of Ni-W/nanocrystalline diamond deposited at stainless steel. The process was carried out for 1 hour in a nitrogen atmosphere (120ml/min and heating rate of 10 ºC/min), varying the temperature between 400 and 700 ºC. For samples treated from 500 ºC, oxidation occurred with the formation of the $NiWO_x$ phases. However, there was also an increase in hardness up to 600 ºC, and the wear rate of the samples decreased as the treatment temperature increased. This could indicate an improvement in the materials' useful life.

Huang *et al.* (2019), exposed steel samples coated with Ni-diamond and Ni-B-diamond to annealing at temperatures between 100 and 500 ºC for 30 minutes. They observed an increase in the hardness of the treated samples up to 300 ºC. Above 300 ºC, the hardness decrease. Even so, there was an increase in the useful life of all coatings, and no cracks were found in any of the annealed parts, only those that did not undergo annealing. Besides, the treated samples had better performance in machining tests.

Finally, Ogihara *et al*. (2012) studied the deposition of Ni-B-diamond on a copper substrate, with treatment at 300 ºC in an air atmosphere (without time specifications). As a result, an increase in hardness between 300 and 600 HV was observed.

7 I CONCLUSION

In conclusion, electroplating is a simple and low-cost process for diamond deposition on tools and, therefore has great importance for diverse sectors of industrial manufacturing. Although already well consolidated, with the advancement of current technologies and industrial needs, this process still can receive more improvements for the coming years. It is notorious the great efforts from researchers and scientists to develop novel materials as anode and cathode, new processes for surface preparation of substrates, and treatments to improve adhesion between the layers. Thus, for the next few years, greater optimizations and cost reductions of this process, as well as improvements in the performance and useful life of the electroplated tools are expected.

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