The Influence of Nanosized Materials on Microhardness of Iron-Based Electroplating

Valentin Vladimirovich Safonov, Sergey Aleksandrovich Shishurin, Pavel Aleksandrovich Gorbushin, Svetlana Valentinovna Chumakova, Vasiliy Vasilievich Chekmarev

Abstract*: *The influence of nanosized particles on microhardness of composite platings has been experimentally detected. Using the proposed procedure, test specimens of iron-based composite electroplating have been obtained containing nanosized particles of silicon carbide, aluminum oxide, aluminum nitride, titanium carbide, and tungsten carbide. Optimum material and concentration of nanodispersed phase have been determined: aluminum nitride as the most efficient substance increases the microhardness of iron-based electroplating by 1.5 times.

Index Terms*: *Composite plating; electroplating; iron plating; nanosized particles; microhardness.

I. INTRODUCTION

Nowadays electroplatings are widely used in various industries. Among them, iron-based electroplatings produced of hot chlorine electrolytes are especially important. Their advantages are as follows: high efficiency of electroplating procedure, its cost efficiency, no thermal impact on deposited surface, and significant layer thickness (up to 1.5 mm) [1, 2]. Nevertheless, the iron plating is characterized by certain disadvantages constraining its application. One of them is moderate plating microhardness.

Iron plating microhardness can be improved by its modification with nanosized particles. This procedure is comprised of addition of nanosized particles of carbides, borides, oxides, sulfides of various metals, polymer powders, etc., to regular electrolyte of iron plating. The nanosized materials improve physicomechanical properties of composite platings and increase their microhardness [1, 3-10].

In order to obtain composite plating, the initial electrolyte is admixed with nanosized particles in amount from 0.1 to 10% [4, 6, 11] which modify its physicomechanical properties. As a consequence, the current efficiency of the metal and the throwing capacity are increased, which can be attributed to variations in its polarization and electric conductivity [11, 12].

Kinetic stability of suspended electrolytes depends on numerous factors, such as their density and temperature, sizes and properties of nanoparticles, their concentration [4, 13]. With the decrease in particle sizes, the interface surface area increases, the system achieves the highest kinetic stability. Such state is characteristic for colloidal suspended electrolytes. Many researchers [3-5, 6, 11, 14-16] believe that in order to maintain stability of suspension and to include particles into plating, their optimum sizes should be from 10 to 100 nm.

It is known [12, 13, 17] that physicomechanical properties of the obtained electroplatings depend on their structure and composition, but mainly they are determined by microhardness. In this regard scientific and practical concern is attracted to the influence of nanosized particles on microhardness of the considered platings.

II. METHODS

In order to determine the most efficient nanosized material and its concentration in electrolyte aiming at improvement of microhardness, the laboratory experiments were carried out.

The following requirements were applied to selection of material: the nanosized particles should be characterized by high hardness; the particles should be chemically stable in iron plating electrolyte and be wettable.

Analysis of existing production technologies of nanosized materials made it possible to use plasma recondensation which was applied for production of powders of various metals with the particle sizes from 10 to 100 nm. The method is based on evaporation of coarse powder (raw stuff) in plasma flow at 4500…6000°C and vapor condensation in the form of particles of the required size [18-20].

Taking into account the presented requirements as well as on the basis of performed studies, the following group of nanosized powders was proposed: tungsten carbide (WC), aluminum oxide (Al2O3), titanium carbide (TiC), silicon carbide (SiC), and aluminum nitride (AlN) [14, 15].

The experiments were performed with hot chlorine electrolyte for iron plating of medium concentration, g/l [2, 20, 21]:

– ion chloride (FeCl2·4H2O): 200…300;
– hydrochloric acid (HCl): 1…1,5;
– manganese chloride (MnCl2): 5…10.

The electrolysis modes were as follows:

– electrolyte temperature: 80°C;
– current: 20 A/dm2;
– duration of plating: 30 min under straight polarity current.

The iron-based suspended electrolyte was prepared in several stages. In order to disaggregate particles and to

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Valentin Vladimirovich Safonov, Saratov State Agrarian University, Saratov, Russia.
Sergey Aleksandrovich Shishurin, Saratov State Agrarian University, Saratov, Russia.
Pavel Aleksandrovich Gorbushin, Saratov State Agrarian University, Saratov, Russia.
Svetlana Valentinovna Chumakova, Saratov State Agrarian University, Saratov, Russia.
Vasiliy Vasilievich Chekmarev, Saratov State Agrarian University, Saratov, Russia.

improve sedimentation stability of electrolyte, the highly concentrated suspension was prepared. Predefined amount of nanosized particles was filled with electrolyte, then by attrition in 10 min the suspension was conditioned to paste-like state. Then the electrolyte was added to the mix, the highly concentrated suspension was obtained which was processed by ultrasonic generator at 22 kHz in 10 min. Then the obtained suspension was added to iron plating bath upon intensive agitation of the electrolyte [20].

Composite electroplatings were applied in several main steps:

– preparation of workpiece surface;
– application of plating onto specimen;
– neutralization of the obtained plating;
– plating quality control.

Preparation of workpiece surface for application of plating was comprised of degreasing and etching. It was degreased in 10% solution of sodium hydroxide (NaOH). Distilled water was used.

The processing modes were as follows:

– solution temperature: 50…70°C;
– current: 20 A/dm2;
– degreasing duration: 1 min under reverse polarity current, 5 min under straight polarity current.

Then the specimens were washed in hot water at 70…80°C in 1–2 min and then in circulating water at ambient temperature in 1–2 min.

In order to remove grease films, scaling, and corrosion products from the workpiece surface, it was etched in 30% solution of sulfuric acid (H2SO4).

The processing modes were as follows:

– solution temperature: 30…50°C;
– current: 10 A/dm2;
– duration of etching: 1 min under straight polarity current.

After etching the workpiece was washed in circulating water at ambient temperature in 1–2 min.

The plating was applied onto specimens using a laboratory ultrasonic electroplating facility (Fig. 1). Schematic view of the facility is illustrated in Fig. 2.

Fig. 1. Laboratory ultrasonic electroplating facility.

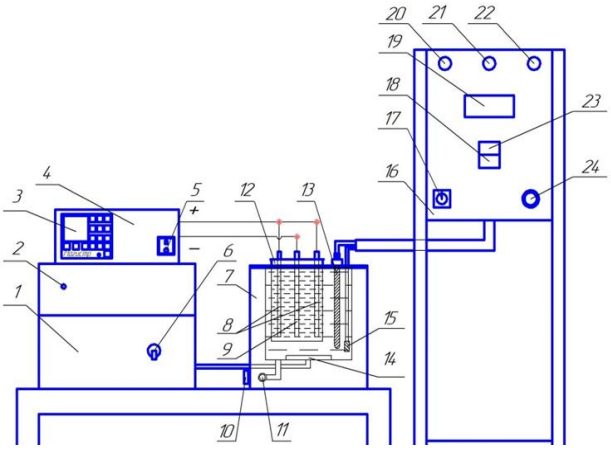

Fig. 2. Schematic view of laboratory ultrasonic electroplating facility

*1 – ultrasonic generator (USG); 2 –USG operation indicator; 3 – data display and control panel of current source; 4 –GITP500-30(5)×12R-220-P2/-V2 power source; 5 – ON/OFF button of power source; 6 –toggle switch of USG; 7 – ultrasonic bath; 8 – anodes; 9 – cathode (workpiece); 10 – drain valve; 11 – drain channel; 12 – tank for electrolyte; 13 – heating element; 14 – ultrasonic element; 15 – temperature sensor; 16 – cabinet of automatic temperature control; 17 – temperature control cabinet toggle switch; 18 – stop button of automatic temperature control; 19 – data display and control panel of cabinet; 20 – power indicator; 21 – heating indicator; 22 – fluid level indicator; 23 – start button of automatic temperature control; 24 –cabinet emergency shutdown.

The facility is comprised of the ultrasonic bath 7, equipped with the three elements 14, connected to the ultrasonic generator 1. Under the impact of ultrasound, electrolyte is agitated and nanosized particles are supplied to the cathode for better laminating by electroplating. Temperature is maintained by the cabinet 16 of automatic temperature control, the cabinet is connected to the heating elements 13 and the temperature sensor 15. Preset current is maintained by the power source 4.

The obtained plating was neutralized in 30% solution of sodium hydroxide (NaOH).

The processing modes were as follows:

– solution temperature: 50…70°C;
– processing time: 10 min.

In order to maintain the preset concentration of constituents, the electrolyte was adjusted by addition of new components according to the known procedure [2, 21].

The platings were applied on rod specimens made of medium carbon steel, grade 25 with the length of 150 and the diameter of 7 mm. Concentration of nanosized particles added to the electrolyte was selected experimentally in the range from 1 to 5 g/l.

The efficiency of the considered nanosized material was estimated by microhardness of the obtained plating. Microhardness was determined using a PMT-3 device with indentation of diamond pyramid on metallographic specimens. The force onto the indenter was 0.981 N, the impact duration of indenter onto specimen was 10 s.

Indentation for one plating specimen was repeated three times.

In order to obtain distinct boundary between metallic layers, the polished section was etched in 2–3s in the following solution:

– nitric acid (HNO3): 5 cm3;

– isopropyl alcohol (C3H8O): 95 cm3.

III. RESULTS AND DISCUSSION

The polished cross sections after tests are illustrated in Fig. 3. The measurements of microhardness are summarized in Table 1. The maximum microhardness is demonstrated by platings with the content of nanosized particles of aluminum nitride equaling to 3 g/l.

High microhardness of platings with nanodispersed aluminum nitride in comparison with other considered nanosized particles could be attributed not only to high hardness of aluminum nitride but probably to chemical properties of nitrides which evolve nitrogen upon interaction with acids, its penetration into electroplating additionally improves its physicomechanical properties [3, 4].

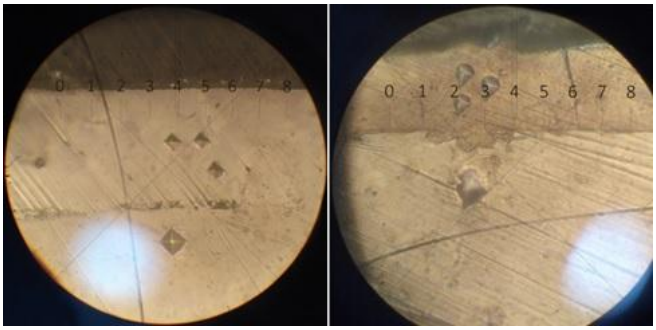

Fig. 3. Polished cross sections of specimens with nanocomposite plating after measurements of microhardness.

Table 1. Microhardness of composite electroplatings as a function of material and content of nanosized particles in electrolyte

Nanosized particles	Content of nanosized particles in electrolyte, g/l	Plating microhardness, MPa
SiC	1	5,976
	3	6,680
	5	6,224
Al_2O_3	1	6,417
	3	6,760
	5	6,570
AlN	1	6,352
	3	6,820
	5	6,780
TiC	1	6,255
	3	6,520
	5	6,364
WC	1	6,340
	3	6,540
	5	6,436
Coating w/o nanosized particles		4,520

The studies of plating microhardness determined the most promising material and concentration of nanosized particles which provided the highest plating microhardness in comparison with other materials.

IV. CONCLUSION

Specimens of composite iron-based electroplatings have been obtained containing nanosized particles of SiC; Al2O3; AlN; TiC; WC. The studies of influence of material and content of nanosized particles revealed that the highest microhardness had been demonstrated for platings modified by AlN nanosized particles with the concentration of 3 g/l and microhardness of 6,820 MPa, whereas the microhardness of basic plating was 4,520 MPa, i.e. by 1.51 times lower.

The experimental results demonstrated that development of composite platings with preset properties could be an efficient tool upon solution of existing technological problems.

On the basis of analysis of the obtained results, it is possible to believe that further investigations into the main operation properties of composite platings would be reasonable.

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