

DEVELOPMENT OF OPTIMAL TECHNOLOGICAL PARAMETERS FOR PLASMA COATING DEPOSITION

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This paper considers using ion-plasma spraying of mold surfaces. This makes it possible to replace scarce and expensive tungsten-containing steels with other materials. 4X5MΦC and 5XHM steel grades were chosen as materials for copper alloy die-casting molds in this work. The choice of these steel grades is due to the fact that these steels do not contain tungsten due to its sharply increased scarcity and limited molybdenum content, and they also meet the requirements for the substrate material on which the titanium nitride coating is applied. Coatings were applied to samples for laboratory tests and die-casting mold parts by the condensation method with ion bombardment. Titanium nitride is applied at different partial nitrogen pressures – from $3 \cdot 10^{-3}$ to 1 Pa to determine the required nitrogen pressure, which ensures that the working surfaces of mold parts receive coatings with the best performance characteristics. The coatings obtained at different nitrogen pressures differ in the amount and size of the droplet phase. The largest amount of the droplet phase containing α -Ti is observed in coatings obtained at nitrogen pressures of $3 \cdot 10^{-3}$, $3 \cdot 10^{-2}$ Pa. An increase in nitrogen pressure ($4 \cdot 10^{-1}$, 1 Pa) significantly reduces the level of microdistortions of the crystal lattice in the coating, and its plasticity increases. In this regard, the coating's brittleness is reduced at a sufficiently high hardness. The titanium nitride coating obtained at a nitrogen pressure of 1 Pa is the most effective in protecting the working surfaces of mold parts from destruction. Laboratory tests have shown that the titanium nitride coating applied under optimal process parameters increases the corrosion resistance of mold parts to which it is applied by 3 times and the scale resistance by 2–4 times.

INTRODUCTION

Various methods of material processing [1–3] are used to obtain a given structure and properties.

One of the most progressive ways to produce castings is through die-casting. This method makes it possible to produce high-precision castings, virtually eliminates their subsequent machining, and ensures high labor productivity.

At the same time, the widespread introduction of die-casting into production is constrained by the low stability of molds and their relatively high cost.

At present, about 30% of the cost of castings from alloys including copper produced by this casting method is reimbursement for tooling wear [4].

It is known that during die casting of copper-based alloys, thermal fatigue is the main cause of failure of dies and liners, the main molding parts [5]. The working surfaces of molds undergo volumetric changes at each heating and cooling, which cause and amplify stresses at a sharp change in temperature. The maximum stress values are reached in a layer with a thickness of 100...200 Å [6].

Firing cracks are formed under the influence of alternating temperature stress. During operation, the metal being poured flows into the cracks and accelerates the destruction of the mold. Thus, the main responsibility for the destruction of the molding surface of mold parts lies with the thin surface layer. It has the highest values of temperatures and stresses, and is also exposed to the aggressive effects of liquid metal [10]. The inner layers are exposed to a temperature difference of only 20...30 °C per cycle, and they are protected

from the melt by the working surface, operating under less aggressive conditions [6].

Therefore, the solution to the problem of increasing the stability of molds can be achieved by affecting only a thin surface layer in order to increase its resistance to cyclic temperature stresses and aggressive environments. The presence of such layer will significantly reduce the requirements for the base metal from which the molds are made. This makes it possible to replace scarce and expensive tungsten-containing steels with other materials. Chemical, chemical-thermal, and surface alloying methods were used to affect the working surfaces of molds [6, 7].

These treatment methods allowed solving the following problems: weakening the physical and chemical interaction between the mold and the casting, increasing wear resistance, and some others [7], which only partially increases the mold resistance (by 20...30%) [6].

PROBLEM STATEMENT

At the same time, new steels such as 4X5B2C, 4X2B2MΦC, and 4X4BMΦC have been developed and are being introduced to reduce the consumption of acutely scarce high-tungsten steel 3X2B8Φ, which is considered the main material for die-casting molds. However, the durability of parts made from these materials is low [6], and they also contain tungsten.

All of the above determines the importance of work aimed at creating a surface layer that must have: low thermal conductivity to reduce the temperature gradient and increase heat resistance, good adhesive interaction

with the base metal from which the molds are made, which ensures the strength of its connection and prevents delamination during operation, low adhesive interaction with the casting metal, which significantly reduces melt adhesion to the working surfaces, and guarantees high wear and corrosion resistance.

The use of plasma coatings made of wear-resistant materials opens up great opportunities. Currently, there are various methods of applying such coatings, including chemical deposition from a gas environment, condensation with ion bombardment (CIB) [7–9, 12–14]. Among these methods, preference should be given to the CIB method, which allows for adjusting the process temperature ($\sim 300\text{--}800\text{ }^\circ\text{C}$), and, therefore, applying coatings to parts made of both high-speed steels and hard alloys, making it a universal and leading hardening technology.

The technology of hardening by deposition of the coating material from the gas phase due to high temperatures ($\geq 1.000\text{ }^\circ\text{C}$) is successfully used to increase the operational stability of carbide tools.

Currently, the CIP method has found application in the automotive, aviation, and electrical industries to protect machine parts and assemblies from abrasive, erosive, corrosive, and other types of environmental influences [7].

As for the use of this method for applying plasma coatings to mold parts, this issue has not been studied. At the same time, the CIB method is of considerable interest for such purposes, since it makes it possible to adjust the composition of the coating within a wide range, which allows obtaining coatings with low

thermal conductivity, reducing the temperature gradient, creating compressive stresses on the surface, providing high strength characteristics, corrosion resistance and scale resistance in this layer, and reliable adhesion of the coating to the substrate.

In connection with the above, a scientifically sound choice of mold material for subsequent CIB processing, optimal coating composition and technical modes of their application, which ensure increased mold reliability and economic feasibility of the proposed development, is an urgent problem.

MATERIALS AND METHODS OF THE STUDY

Since the surface layers of mold parts are exposed to a cyclically varying temperature load, the level of which is reduced (to $t \sim 650\text{ }^\circ\text{C}$) due to the protective effect of the plasma coating, they are in the same temperature conditions as when die-casting of low-melting point alloys (aluminum, zinc). The material was chosen from among semi-heat-resistant steels used in die-casting of low-melting point alloys (aluminum, zinc).

From these steels, we selected those that do not contain tungsten, given its sharply increased scarcity and limited molybdenum content, and that also meet the requirements for the substrate material to be coated with titanium nitride.

In this work, 4X5MΦC and 5XHM steels were chosen as materials for copper alloy die-casting molds (Table 1).

Table 1

Chemical composition of steels used to produce die-casting molds

Steel	Content of elements, %				
	C	Cr	Mo	V	Ni
5XHM	0.50...0.60	0.60...0.80	0.15...0.30	–	1.40...1.80
4X5MΦC	0.37...0.44	4.50...5.50	0.80...1.10	0.80...1.20	–

Coatings were applied to samples for laboratory research and die-casting mold parts using the method of substance CIB. The coatings obtained by this method are characterized by high stability of properties, low porosity, and good adhesion to the substrate.

The coatings were applied using a “Bulat-3T” unit (Fig. 1). The CIB method includes two main stages:

1. Cleaning, heating, and activation of the substrate surface by ion bombardment of the material to be deposited, accelerated to the required energy.

2. Deposition of the coating during continuous ion bombardment of condensate in a mode that ensures the formation of a coating with the required service properties [10].

Coating time is also a technological parameter to be optimized. The speed of TiN coating application at the “Bulat-3T” unit is $10\text{ }\mu\text{m/h}$ [9].

Given that titanium nitride coatings with a thickness of 2 to 5 μm have the highest adhesive activity to the substrate and the greatest impact on increasing the thermal endurance of 4X5MΦC and 5XHM steels, the coating time ranged from 12 to 30 min.

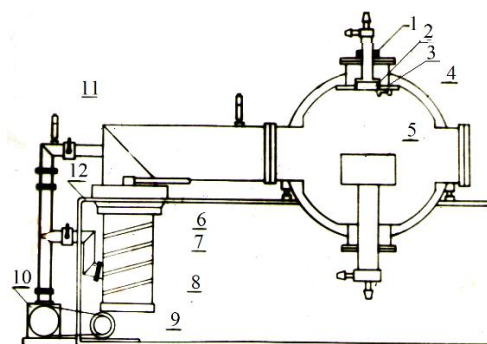


Fig. 1. General arrangement drawing of “Bulat-3T”:
 1 – focusing coil; 2 – cathode; 3 – ignition electrode;
 4 – anode chamber; 5 – substrate; 6 – nitrogen trap;
 7 – water trap; 8 – high-vacuum unit; 9 – heater;
 10 – backing vacuum pump; 11 – gauge lamp;
 12 – water cooling system

The selected steel grades were quenched at temperatures that dissolve most of the carbides and produce high-alloy martensite (Table 2). At the same time, the steels retain fine grain and sufficient toughness. After quenching, the steels are subjected to

high-temperature tempering to obtain high values of impact strength and endurance. This tempering contributes to additional hardening due to dispersion

hardening [11], which is accompanied by the release of fine carbides and a hardness of 42...45 HRC.

Table 2

Heat treatment modes of 5XHM and 4X5MΦC steels

Steel grades	Quenching					Tempering		
	loading temperature	heating temperature, °C	holding time during heating	cooling medium	hardness, HRC _e	temperature, °C	holding time, hour, including heating of the batch of parts	hardness, HRC _e
5XHM	700...750	830...860	1-2 min per 1 mm per course	cooling in oil	47...50	500...520	4...5	42...45
4X5MΦC		1050...1070			55...61	560...580		42...45

Note. Before being loaded into the heat treatment furnace, the manufactured parts are wrapped in asbestos paper and covered with cast iron shavings or used carburizing material. The layer thickness is 35 mm. Quenching and tempering are performed with a minimum time gap.

The reaction gas pressure in the unit chamber, the heating temperature of the substrate to be coated, and the deposition time were selected as the optimal coating parameters.

In accordance with the developed thermal endurance test methodology, the criterion for evaluating the optimal coating parameters is the number of thermal cycles that a sample with a deposited coating can withstand before it breaks.

It is known that the most important parameter of the condensation with ion bombardment is the reaction gas pressure [12].

It should be noted that the optimal pressure of the reaction gas in the vacuum chamber should be determined in each case and depends on the

composition of the cathode, unit model in which the coating is applied, and the operating conditions of the coated part [9, 13, 14].

Titanium nitride is applied at different partial nitrogen pressures, ranging from $3 \cdot 10^{-3}$ to 1 Pa (Table 3) to determine the required nitrogen pressure, which ensures that the working surfaces of mold parts receive coatings with the best performance characteristics. This range of nitrogen pressures was chosen based on literature data on the performance of plasma-coated parts and tools [13, 15].

After coating, the surface microstructure of the obtained coatings and their microhardness were studied (Table 4).

Table 3

Influence of nitrogen partial pressure on coating characteristics

Nitrogen pressure, Pa	$3 \cdot 10^{-3}$	$3 \cdot 10^{-2}$	$4 \cdot 10^{-1}$	1
Microhardness, GPa	22	26.0	23.0	18.0
Color of the coating	gray	yellowish-gray	yellow	brownish-golden

Table 4

Influence of nitrogen partial pressure on the thermal endurance of a coating

Steel grade	Pressure				
	uncoated	$3 \cdot 10^{-3}$	$3 \cdot 10^{-2}$	$4 \cdot 10^{-1}$	1
thermal endurance					
4X5MΦC	2,000	3,000	5,000	9,500	12,000
5XHM	2,000	3,500	5,000	9,000	11,500

Note. 5 μm thick titanium nitride coating applied at substrate temperature 500°C.

RESULTS AND DISCUSSION

The coatings obtained at different nitrogen pressures differ in the amount and size of the droplet phase. The largest amount of the droplet phase containing α -Ti is observed in coatings obtained at nitrogen pressures of

$3 \cdot 10^{-3}$, $3 \cdot 10^{-2}$ Pa (Fig. 2,a,b). They are gray and yellowish-gray in color. With an increase in nitrogen pressure to 1 Pa, the coatings acquire a brownish-golden color; the amount of the droplet phase on the surface decreases markedly (see Fig. 2,d).

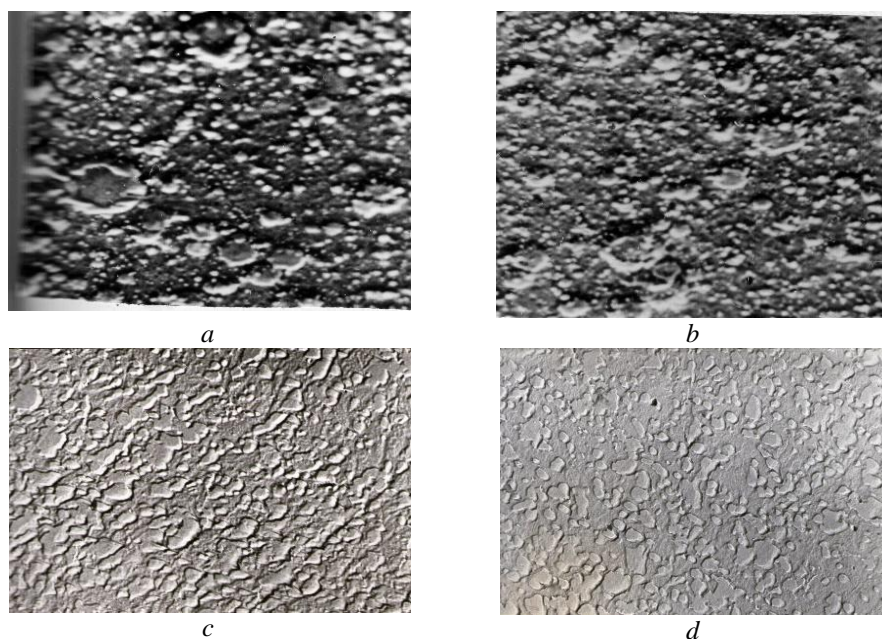


Fig. 2. Surface microstructure of titanium nitride coatings obtained at different reaction gas pressures: $3 \cdot 10^{-3}$ Pa (a); $3 \cdot 10^{-2}$ Pa (b); $4 \cdot 10^{-1}$ Pa (c); 1 Pa (d)

The highest microhardness is obtained at nitrogen pressures of $3 \cdot 10^{-3}$, $3 \cdot 10^{-2}$ Pa (up to 26 hPa). With increasing pressure, the microhardness decreases and at a pressure of 1 Pa it is 18 hPa. Laboratory tests on the thermal endurance of samples with coatings applied at different nitrogen pressures showed that the samples with titanium nitride coating obtained at a pressure of 1 Pa can withstand the greatest number of cycles. It should be noted that the samples coated at nitrogen pressures of $3 \cdot 10^{-3}$ and $3 \cdot 10^{-2}$ Pa show significant melt adhesion, which decreases markedly with increasing pressure and is practically absent starting from 1 Pa. At pressures of $3 \cdot 10^{-3}$, $3 \cdot 10^{-2}$ Pa, along with the highest microhardness, an increase in the width of diffraction maxima (333) is also observed near the coatings, which characterizes the microdistortion of the crystal lattice of the chemical compounds that make up the coatings.

This is probably due to the introduction of nitrogen atoms whose diameter is larger than the diameter of the sphere inscribed in the titanium lattice [16]. High hardness and a high level of microdistortions lead to an increase in coating brittleness, which causes rapid destruction under the influence of alternating stresses and technical shocks.

The presence of a significant amount of the droplet phase on the surface intensifies the fracture process.

Increasing the nitrogen pressure ($4 \cdot 10^{-1}$, 1 Pa) significantly reduces the level of microdistortions of the crystal lattice in the coating, and increases its plasticity [8]. In this regard, the brittleness of the coating decreases at a sufficiently high hardness.

The titanium nitride coating obtained at a nitrogen pressure of 1 Pa most effectively protects the working surfaces of mold parts from destruction (see Table 4).

Another important parameter that significantly affects the stability of plasma-coated parts is the temperature of the working surface during ion bombardment, since the energy of ions during bombardment is much higher than the energy of ions during coating condensation [9].

According to the technical data, the temperature of the substrate surface in the chamber of the "Bulat-3T" unit during ion bombardment varied from 300 to 800 °C.

The temperature was monitored using a witness sample with a chromium-aluminum thermocouple built into it, which measures the temperature of the deposition surface.

Ion bombardment has the most favorable effect on the substrate made of 4X5MΦC and 5XHM steels at a temperature of 500 °C, which corresponds to the best adhesion of the coating to the substrate and, accordingly, the highest durability of parts with such a coating during operation.

At lower temperatures, there is a tendency to decrease the adhesion of the coating, which is accompanied by peeling of the coating during operation and a significant decrease in durability.

This phenomenon is especially noticeable when the heating temperature of the surface to which the coating is applied exceeds the tempering temperature of the steel due to the hardening of the latter.

Coatings applied to the working surfaces of mold parts to increase their durability must reliably protect

mold parts from external factors, i.e., have low adhesion activity to the casting material, high scale and corrosion resistance, and at the same time provide the greatest strength of the coating-substrate bond.

To determine the adhesion activity of the coating to the casting material, the interaction between the studied inserts made of 4X5MΦC and 5XHМ steels with a 4-μm titanium nitride coating and copper, lead, and zinc melts was determined by the droplet method, since these metals are brasses.

The obtained values of the wetting edge angles (Table 5) indicate a slight adhesive interaction of the titanium nitride coating applied at the optimal mode with copper, lead, and zinc.

Thus, the coating significantly reduces the adhesive interaction of steels used as mold material with brass melt.

Table 5

Wetting the titanium nitride coating with melt

Melt	Atmosphere	0°
Cy	vacuum	134
Pb	vacuum	138
Zn	vacuum	130

As the coating thickness increases, the substrate should be more reliably protected from external factors due to increased hardness, temperature resistance against oxidation and corrosion. But at the same time, the probability of the appearance of such dangerous defects as macro- and micropores, microcracks, which can lead to the destruction of the coating, increases in the volume of the coating. Therefore, the coating thickness is a parameter to be optimized in each specific case [9, 17–19].

To determine the optimal thickness of the coating, which ensures its strong adhesion to the working surface of parts made of steel grades 4X5MΦC and 5XHМ, the dependence of the strength of the connection of the coating with the substrate on the thickness of the coating was investigated (Fig. 3). Coatings with a thickness of 2...5 μm have the best adhesion. When the coating is more than 5 μm thick, its scaly peeling occurs (Fig. 4). In the process of operating parts of molds with a coating thickness of 1 μm, its destruction occurs within several hours of operation.

Corrosion resistance tests were performed on samples made of 4X5MΦC and 5XHМ steels with and without titanium nitride coating. The tests were carried out in water at a temperature of 20 °C for $\tau = 1,000$ h. The corrosion rate at the beginning of interaction with the aqueous medium (first 24 h) is high for both studied materials, although it is noticeable that the corrosion rate of coated samples increases more slowly. This dependence occurs in the first 24 h. Then a significant decrease in the corrosion rate is observed. Subsequently, the value of the corrosion rate stabilizes and subsequently slowly decreases (Figs. 5, 6).

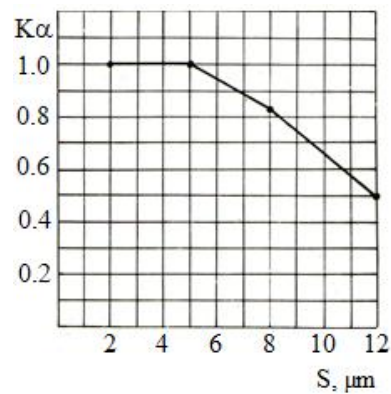


Fig. 3. Effect of coating thickness S on adhesion $K\alpha$

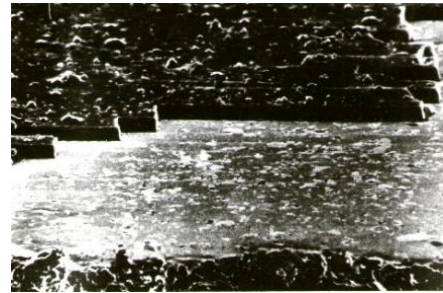


Fig. 4. The initial stage of peeling of an 8 μm thick coating, $\times 1,000$

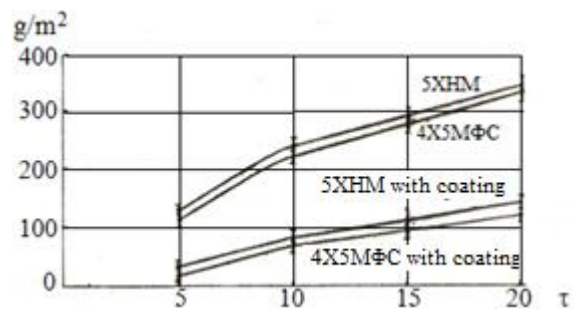


Fig. 5. Ocular resistance of steels as a function of time τ (h) 4X5MΦC, 5XHМ

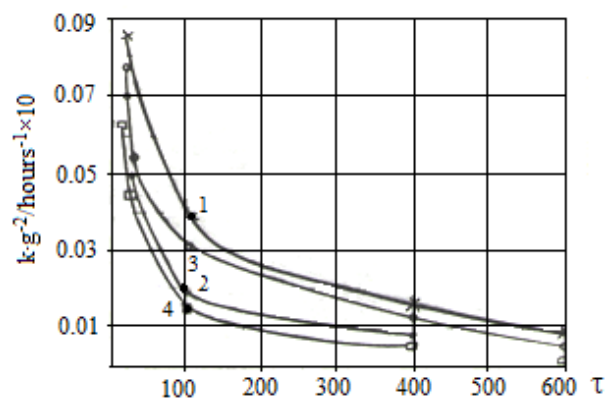


Fig. 6. Dependence of the corrosion rate Z of 4X5MΦC and 5XHМ steels on the test time, h: 1 – 5XHМ steel; 2 – 5XHМ steel with titanium nitride coating; 3 – 4X5MΦC steel; 4 – 4X5MΦC steel with titanium nitride coating

The highest rate of corrosion destruction occurs in the first 12 h, and at this time the titanium nitride coating has its protective effect against corrosion. These results apply to samples with a coating thickness of more than 3 μm . In samples with a coating thickness of 1...2 μm , coating delamination and rapid corrosion development were observed 30 h after the start of the test.

When determining the scale resistance of coated materials, its protective properties can be judged by weight.

We determined the scale resistance of 4X5M Φ C and 5XHM steels after uncoated heat treatment and after titanium nitride coating. The results shown in Fig. 6

indicate a strong effect of titanium nitride coating on the oxidation resistance of 4X5M Φ C and 5XHM steels: after 10 h of operation, the weight gain of coated samples is three times less than that of uncoated samples.

The effect of coating thickness on the oxidizability of 4X5M Φ C and 5XHM steels was also studied (Table 6). It can be seen that at a coating thickness of 3 μm , the oxidation resistance of the selected steel grades increases significantly, and a continuous increase in this effect is observed up to a thickness of 6 μm . Further increase of the coating thickness leads to a decrease in the oxidation resistance of the steels.

Table 6

Influence of titanium nitride coating thickness on the oxidation of samples from 4X5M Φ C and 5XHM steels ($t = 850\text{ }^\circ\text{C}$, $\tau = 20\text{ h}$, continuous heating in the air)

Material	Average weight gain of the sample, g/m^2 , at the coating thickness, μm					
	1	2	3	4	5	6
4X5M Φ C	160	140	110	120	140	220
5XHM	190	150	135	140	160	250

The research has shown that mold parts with plasma coating applied to their working surfaces have maximum resistance when the coating is deposited at a substrate temperature of 500 $^\circ\text{C}$ and a partial nitrogen pressure of 1 Pa.

Laboratory tests have shown that the titanium nitride coating applied under optimal technological parameters provides a 3-fold increase in the corrosion resistance of mold parts on which it is applied, and a 2–4-fold increase in scale resistance.

CONCLUSIONS

Summarizing the results obtained, it can be concluded that the maximum durability of mold parts with plasma coatings is achieved when the working surface is heated during ion bombardment to high temperatures, but not higher than the tempering temperature of the mold material to ensure good adhesion of the coating to the substrate and with subsequent condensation on this heated surface of the coating with a minimum content of the droplet phase to prevent the coating from sticking to the casting material.

In the case of titanium nitride coating on the “Bulat-3T” unit on the working surfaces of mold parts made of economically alloyed steels, the best adhesion of the coating to the substrate, its minimal interaction with the brass melt, and a significant increase in the thermal endurance of the substrate material are achieved at a temperature of 500 $^\circ\text{C}$ and condensation of the coating at a partial pressure of nitrogen of 1 Pa.

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РОЗРОБКА ОПТИМАЛЬНИХ ТЕХНОЛОГІЧНИХ ПАРАМЕТРІВ ОСАДЖЕННЯ ПЛАЗМОВИХ ПОКРИТТІВ

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Розглянута можливість застосування іонно-плазмового напилення поверхні прес-форм. Це уможливило заміну дефіцитних дорогих вольфрамівмісних сталей іншими матеріалами. Як матеріали для прес-форм лиття під тиском мідних сплавів обрані у цій роботі сталі 4Х5МФС та 5ХНМ. Вибір даних марок сталей обумовлений тим, що ці сталі не містять вольфраму, зважаючи на його різко зросло дефіцитність, і зміст молібдену обмежений, а також задовольняють вимогам до матеріалу підкладки, на яку наноситься покриття нітриду титану. Нанесення покриттів на зразки для лабораторних досліджень та деталі прес-форм лиття під тиском здійснювалося методом конденсації речовини в умовах іонного бомбардування. Для визначення необхідного тиску азоту, що забезпечує отримання робочих поверхонь деталей прес-форм покриттів з найкращими експлуатаційними характеристиками, нітрид титану наноситься при різних парціальних тисках азоту – від $3 \cdot 10^{-3}$ до 1 Па. Покриття, отримані при різних тисках азоту, відрізняються кількістю та розміром краплинної фази. Найбільша кількість крапельної фази, що містить α -Ті, спостерігається у покриттів, отриманих при тисках азоту $3 \cdot 10^{-3}$, $3 \cdot 10^{-2}$ Па. Збільшення тиску азоту ($4 \cdot 10^{-1}$, 1 Па) значно зменшує рівень мікроспотворень кристалічних ґрат у покритті, зростає його пластичність. У зв'язку з цим знижується крихкість покриття при досить високій його твердості. Отримане при тиску азоту 1 Па покриття нітриду титану найбільше ефективно захищає робочі поверхні деталей прес-форм від руйнування. Як показали лабораторні випробування, нанесене при оптимальних технологічних параметрах покриття нітриду титану забезпечує підвищення корозійної стійкості деталей прес-форм у 3 рази, окислюваність – у 2–4 рази.