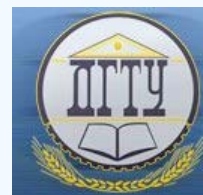


## MACHINE BUILDING AND MACHINE SCIENCE



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## Selection of technologies for metal film application using physical deposition techniques

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**Introduction.** Obtaining high-quality thin metal films is important for advances in the technologies of applying antifriction and wear-resistant coatings on cutting tools or parts of friction pairs. Various techniques of physical film deposition are applied using technologies of cathode (ion), magnetron and ion beam assisted sputtering. The work objective is to analyze, compare and determine the feasibility of techniques for the physical deposition of thin metal films when applying antifriction and wear-resistant coatings on cutting tools or parts of friction pairs.

**Materials and Methods.** Technologies of cathode (ionic), magnetron and ion-beam sputtering are considered. Schematic diagrams, conditions and parameters of the considered processes are presented.

**Results.** An advanced technology for the deposition of thin films, alloying and hardening of the surfaces of metal parts is magnetron sputtering. Continuous wave (cw) magnetrons are used to apply coatings of complex composition or multilayer coatings on flat substrates. Ion beam sputtering is considered as slow sputtering of the target surface by bombardment with a high-energy ion beam and deposition on the substrate surface. Under the ion implantation, the surface of metals is doped with recoil atoms, which receive high energy from accelerated ions and move a few nanometers deeper. This enables to obtain ultra-thin doped layers. Low temperature of ion implantation, the possibility of sufficiently accurate control of the depth and the impurity distribution profile create the prerequisites for the process automation. Wear tracks are more acidified under the same wear conditions on implanted steel compared to non-implanted steel. The nonequilibrium process under ion implantation causes the formation of such alloys in the surface layers that cannot be obtained under normal conditions due to diffusion of components or limited solubility. Ion implantation makes it possible to obtain alloys of a certain composition in the surface layer. Surface properties can be optimized without reference to the bulk properties of the material. Implantation is possible at low temperatures without a noticeable change in the size of the product.

**Discussion and Conclusion.** Cathode (ion), magnetron and ion-beam sputtering have common advantages: due to the relatively low temperature, the substrate does not overheat; it is possible to obtain uniform coatings; the chemical composition of the deposited coatings is accurately reproduced. The rest of the advantages and disadvantages of the considered methods are individual. The results can be used to create thin films through alternating magnetron and then ion-beam deposition processes, which enables to obtain films uniformly modified in depth. This is important in the production of parts of friction pairs and cutting tools to improve their quality.

**Keywords:** metal film, physical deposition, anti-friction coating, wear-resistant coating, ion sputtering, magnetron sputtering, ion-beam sputtering.

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**Introduction.** Obtaining high-quality thin metal films is important for advances in the technologies of applying antifriction and wear-resistant coatings on cutting tools or components of friction pairs [1–6].

The thermal vacuum method, which was used earlier, did not provide the reproducibility of the properties of the films, especially during the deposition of materials of complex composition. The transition to continuous processing contributed to the development of ion-plasma deposition of thin films.

Currently, various methods of physical film deposition are utilized using cathode (ion) sputtering, magnetron sputtering, and assisted ion-beam sputtering. This creates thin films that cannot be obtained by other techniques (for example, films of refractory or multicomponent materials). The work objective is to analyze and compare the feasibility of methods for the physical deposition of thin metal films when applying antifriction and wear-resistant coatings on cutting tools or parts of friction pairs.

The use of cathode (ion), magnetron and ion-beam sputtering provides fully automation of the production of film coatings in the continuous technological installations.

**Materials and Methods.** In cathode (ion) sputtering, ions of the discharged gas bombard and destroy the cathode material [1]. Its atoms evaporate and condense on the substrate.

A schematic diagram of the thin films deposition through cathode sputtering is shown in Fig. 1.

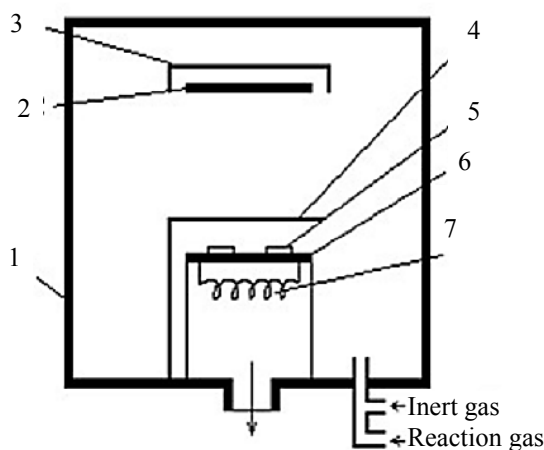


Fig. 1. Schematic diagram of the process of applying thin films by cathode sputtering: 1 — chamber, 2 — cathode, 3 — ground shield, 4 — shutter, 5 — substrate, 6 — ground anode, 7 — substrate resistive heater

Under this spraying, air is pumped out from the chamber in which the anode and cathode are installed to a vacuum state with pressure of  $10^{-4}$  Pa. Then, an inert gas (e.g., argon) with pressure of 1–10 Pa is fed into the chamber [1]. When constant high potential difference of 1–5 kV is applied to the cathode and anode, glow discharge arises between them, in which positively charged ions of the inert gas are formed. Ions are accelerated by an electric field and bombard the cathode causing evaporation and sputtering of material atoms, which are deposited on the substrate in the form of a thin film [1, 3].

The initial ideas on the cathode sputtering were based on the predominant role of chemical processes. It was believed that the transfer of the cathode material to the substrate to be coated consists of:

- a) the formation of fragile compounds with the cathode material on the substrate surface,
- b) the evaporation of compounds of the substrate and cathode materials,
- c) the partial decay of these compounds.

Inert gases in an ionized state (as opposed to a normal state) can exhibit some chemical activity.

However, it is not possible to explain all cases of such sputtering through chemical processes since there is cathode sputtering of weakly volatile compounds (for example,  $\text{Al}_2\text{O}_3$ ), which practically cannot evaporate at cathode heating temperatures.

Therefore, not a chemical but a physical mechanism of cathode sputtering was proposed. According to this approach, material particles leave the cathode surface because the cathode material atoms receive energy directly from the bombarding atoms or molecules. There are two ideas on such energy transfer.

1. Impacts of bombarding atoms cause a strong local increase in the temperature of microscopic areas of the cathode material surface resulting in its evaporation.

2. The bombarding atom transfers kinetic energy to the atom on the cathode surface. As a result, the bonds of the cathode atom with neighboring atoms are destroyed, and it escapes from the cathode surface.

Such a mechanism better represents physical sputtering, especially in accordance with studies on ionic and neutral emission (i.e., emission of neutral atoms) of solids under ion bombardment.

**Research Results.** When considering cathodic sputtering in general, both physical and chemical mechanisms should be kept in mind. One of them may prevail depending on the actual process conditions.

The advantages of the cathode sputtering method are listed below.

1. The process runs at a relatively low temperature, the substrate does not overheat.
2. Uniform in thickness coatings can be obtained.
3. The chemical composition of the deposited coatings is accurately reproduced.
4. The areas of the coatings are quite large since the material is applied to the substrate not from a point source.
5. The resulting coatings have high adhesion to the substrate material due to high energy of the condensing atoms.
6. It is possible to achieve a high utilization rate of the coating material.

Let us list the disadvantages of the cathode sputtering method.

1. Films are characterized by a high level of mechanical stress.
2. The deposition rate is low (0.3–2 nm/s).
3. In some cases, working pressure in the chamber is 1–10 Pa; therefore, the films are contaminated by the working gas.

Magnetron sputtering is an advanced technology for the deposition of thin films, alloying and hardening of the surfaces of metal parts. Its advantages:

- high speed of obtaining film coatings;
- low level of contamination by foreign inclusions counting gases;
- low heating temperature of the substrate material;
- availability of spraying conducting and dielectric materials;
- availability of ultrathin films (<20 nm) with small defects;
- laglessness of the process

Magnetron sputtering is used to deposit ultra-thin films of chromium, aluminum (and its alloys) and various refractory metals.

The pattern of the sprayed particles motion in the magnetron sputtering system (MSD) is shown in Fig. 2.

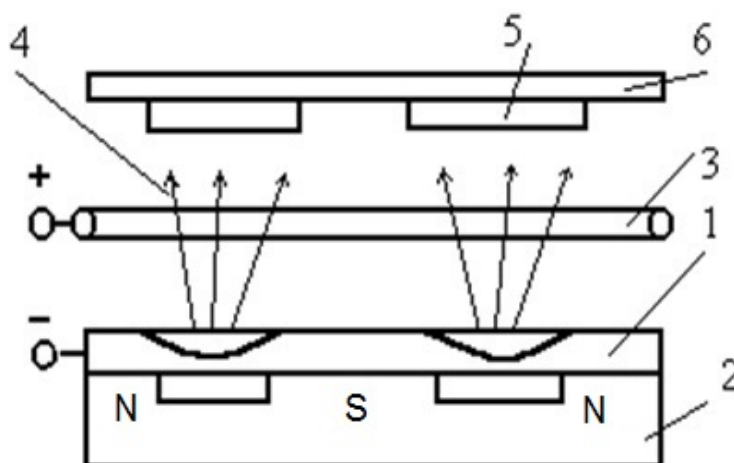


Fig. 2. Motion pattern of sputtered particles in MSD:  
1 — target cathode, 2 — permanent magnet, 3 — anode, 4 — flux of sputtered particles, 5 — substrate, 6 — substrate holder

The MSD is mounted in a vacuum spray chamber from which air is evacuated. Argon or a mixture of inert gases is used as a working gas. The total pressure is kept constant with an accuracy of  $\pm 5\%$ . Through selecting the partial pressure of the gas mixture components, it is possible to change the coating characteristics in a wide range, including electrical.

The working environment in the chamber changes through varying the amount of oxygen, nitrogen, carbon dioxide, sulfurous gas compounds. This provides the creation of thin films of oxides, nitrides, carbides, sulfites of various materials, which cannot, for example, be obtained by thermal evaporation.

The composition of compounds in film coatings containing oxides, carbides, nitrides directly depends on the purity of materials and gases. Therefore, chemically pure materials should be used for spraying. To control the partial pressure, the vacuum units should be equipped with pumps that provide a constant pumping speed in the required pressure range.

MSDs use direct current sources. A constant electric field above the cathode target forms a glow discharge, creates plasma, and causes ion bombardment of the target (cathode). The MSD closed magnetic field concentrates plasma at the surface of the target cathode.

Under the action of ion bombardment, electrons are emitted from the target cathode. They are collected and held by the magnetic field. The electrons perform a complex cyclic motion over the surface of the target cathode. Before reaching the anode, the electrons collide with the atoms of the working gas (argon) and spend a significant part of their energy under the collision on ionizing the working gas (argon). This increases the number of ions at the surface of the target cathode, enhances bombardment, and increases the rate of material sputtering, deposition and film formation.

In the MSD, electric and magnetic fields intersect, which creates a magnetic trap at the sputtered target surface and increases the sputtering rate. The magnetic trap captures high-energy secondary electrons. They do not participate in the bombardment of the substrate, and this explains its insignificant heating.

The deposition rate of the coating material under magnetron sputtering depends on the pressure of the working gas in the installation chamber, the current strength, and the discharge power, which sets strict requirements for energy sources. To provide the process stability, it is required to maintain the discharge current with an accuracy of  $\pm 2\%$ . If the process is stabilized by the discharge power, it should be maintained with an accuracy of  $\pm 20$  W in the control range of 0–10 kW.

The average deposition rate of molybdenum is 12–37 nm/s under the following setup parameters:

— cobalt target in the form of a flat disk 150 mm in diameter,

- 4 kW power supply,
- molybdenum substrate is located at a distance of 60 mm from the power source [7].

In this case, the temperature of heating the substrate is approximately equal to the temperature of thermal evaporation in vacuum of low-melting metals, but much lower than the temperature of evaporation of refractory metals. This provides applying thin films to materials with low heat resistance (e.g., plastics).

Stability of film deposition on a substrate is provided by correctly selected parameters of the MSD:

- supply voltage of electrodes,
- discharge current,
- current density on the target,
- specific power,
- magnetic field induction,
- working gas pressure in the chamber.

The cathode — anode potential difference does not exceed 1000 V. The current of the electric discharge is established empirically. The current density on the target is about 200 mA/cm<sup>2</sup>, but it can be higher in the central spray zone. The specific power of the electric discharge is from 40 to 100 W/cm<sup>2</sup>. It is specified by the thermal conductivity of the deposited material and the cooling conditions.

The current-voltage characteristic of the electric discharge between the anode and cathode depends on the working gas pressure and the magnetic induction. With a decrease in the pressure and the magnetic field induction in the chamber, the current-voltage characteristics of the discharge shift to the region of high operating voltages.

The deposition rate of the film material is almost linearly dependent on the discharge power. With an increase in the discharge power, the deposition rate increases.

The discharge power reaches its maximum with an increase in the magnetic field induction to 0.08–0.1 T and a low working gas pressure (from 1 to 10 Pa). If the working gas pressure is high, the maximum discharge power is reached at a magnetic field induction of 0.04–0.06 T.

It should be noted that despite these advantages, as well as direct deposition of dielectrics with a high-frequency magnetron, this method is characterized by a low rate of film deposition, i.e., low productivity. There are also difficulties in matching the power supply of the magnetron sputtering set with the load when operating at high frequencies. In addition, the power source should have an arc discharge quenching system, as arc discharges cause instability of the parameters of the MSD.

In the batch-type MSDs, spraying means are located along the axis of the cylindrical chamber or along its generatrix. In the first case, cylindrical sputters are used, in the second, planar ones. Substrates move through the plasma region.

According to the operation principle, magnetron vacuum installations with ion-plasma sputtering sources are divided into two types — batch-type and continuous.

Batch-type magnetrons are used for coating dielectrics. In this case, to lower the substrate temperature, one should:

- improve the thermal contact of the substrate with the cooling system;
- make parts of the cooling system from materials with high thermal conductivity; or to increase the thermal conductivity of the gas layer between the cooling system and the film;
- cool the units of the cooling system to temperature of 243–253 °K;
- expand the coverage area and increase the size of the cooling system.

High rate of release of substrate gases liberation, as well as the possible interaction of ionized gases with the deposited material, causes the use of condensation vacuum traps.

Continuous magnetrons are used to apply coatings of complex composition or multilayer coatings on flat substrates [8]. Typically, continuous installations consist of a chain of flat rectangular chambers separated by locks and gates. Continuous installations use either top-down spraying or vertical movement of substrates and lateral placement of sputters.

When operating a magnetron sputtering system, as a rule, one parameter is controlled. The rest ones are fixed at the optimal value through adjusting the thickness of the films, changing the deposition time, etc.

The advantages of magnetron sputtering are listed below.

1. The process runs at a relatively low temperature, the substrate does not overheat.
2. Uniform in thickness coatings can be obtained.
3. The chemical composition of the deposited coatings is accurately reproduced.
4. The process is fast. High sputtering speed of materials at low working voltages (600–800 V) and low pressure of the working gas ( $5 \cdot 10^{-1} - 10$  Pa) are marked.

The disadvantages of magnetron sputtering are as follows:

1. High requirements for the purity and dryness of the protective gas (argon).
2. Requirement for locks in front of the discharge chamber for protection against oxidation. The chamber should be supplied with shielding gas.
3. Probability of breakdowns between anode and cathode.
4. High requirements for the accuracy of positioning the substrates relative to the evaporators to provide uniformity of the films in thickness and in composition.

Ion-beam sputtering is used for deposition of thin films in vacuum, as well as for modification and doping of surface layers of metals through implantation of ions from separated beams.

Fig. 3 shows a diagram of an ion-beam sputtering tool.

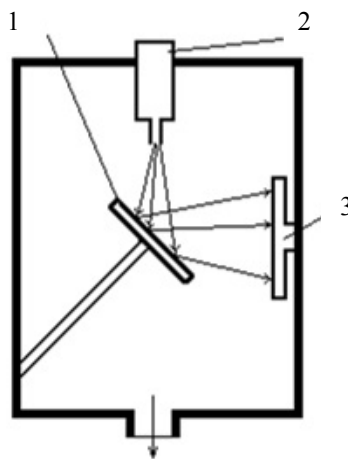


Fig. 3. Ion-beam sputtering tool diagram: 1 — target, 2 — ion source, 3 — substrate

Ion beam sputtering is considered a slow sputtering of a target surface under bombardment with a high energy ion beam and deposition on a substrate surface. This process is physical, not chemical in nature. An impulse is transmitted to the surface atoms from the incident ion:

- directed away from the surface,
- strong enough for the atoms to leave the surface.

Under ion implantation, the surface of metals is doped with recoil atoms, which receive high energy from accelerated ions and move deeper by several nanometers. This provides obtaining ultra-thin doped layers. Low tempera-

ture of ion implantation, the possibility of sufficiently accurate control of the depth and profile of impurity distribution create the prerequisites for the process automation.

Ion implantation is also used to modify the surface properties of metals: increase in hardness, increase in wear resistance, corrosion and radiation resistance, increase in resistance to fatigue failure, and reduction of the friction factor. Ion implantation is used to obtain anti-friction wear-resistant surfaces. For machine parts, the wear resistance of a material is, as a rule, a more important characteristic than its hardness or friction factor [4–8].

It has been noted that, e.g., a large dose of implanted nitrogen can significantly reduce the wear rate [9]. In addition, it was found that the implantation of ions of inert gases (e.g., neon, argon), which creates compressive stresses in the surface layer, does not cause a decrease in the wear rate. However, the implantation of interstitial atoms (boron, carbon, and nitrogen) at ion irradiation doses of  $10^{17} \text{ cm}^{-2}$  ions was very effective, and this effect exceeded considerably the penetration depth of the implanted ions in thickness of the wear layer.

The implanted interstitial atoms segregate even at normal temperatures under ion-beam sputtering (for example, nitrogen, carbon or boron). This blocks their motion in the deposited material, hardens the surface layer, and increases wear resistance.

During friction wear, two processes occur, whose action explains the effect of ion implantation on a layer deeper than the depth of ion penetration.

The first is the nucleation and development of new dislocations under the action of high local loads on the contact spots of the surface microroughness. Impurity atoms diffuse deep into the solution under the action of a stress field arising around a pileup of dislocations.

The second is local heating of the surface at the contact points. For example, when testing materials for wear resistance, the temperature at microroughnesses reaches 600–700° C. The motion of impurities occurs under the influence of large temperature gradients. Diffusion along dislocation lines is the most probable transport mechanism.

The friction factor in steels is also reduced under ion implantation due to two effects.

1. Welding bridges at the contact of two surfaces become brittle due to hindered dislocation motion.
2. The oxide film is more stable under these conditions, and its presence reduces adhesion.

The analysis confirmed that the wear marks are more oxidized under the same wear conditions on the implanted steel compared to the non-implanted steel.

When considering a composite material (for example, tungsten carbide on a cobalt bond), a more complex situation occurs. At high temperatures, wear is accompanied by diffusion, cobalt is carried to the surface, and substances such as iron (from the metal being processed) diffuse into the bulk, causing the carbide grain breakdown. At low temperatures, adhesion and abrasion of cobalt are more likely, and the process is intensified if the shear forces reach values sufficient to squeeze out the cobalt soft bond between the carbide grains.

Implanted nitrogen or carbon ions can be displaced to dislocations in cobalt, as in iron alloys. Cobalt, unlike iron, does not form stable nitrides or carbides, so the implanted atoms remain in the hard alloy. Using an electron microscope, martensitic transformations were detected in the cobalt bond of the implanted hard alloy based on tungsten carbide, which indicates a distortion of the crystal lattice and possible hardening upon dissolution of nitrogen in the hard alloy.

In addition, nitrogen can segregate to the interfaces between carbide grains and cobalt binder enhancing chemical bonds on these surfaces and hardening the composite.

Under ion implantation in tungsten hard alloys (tungsten carbide on a cobalt bond), the implanted nitrogen or carbon migrates along the insertion sites of the cobalt bond. It is accelerated by large thermal gradients under the surface microroughnesses. For this reason, if the wear conditions are relatively mild and the cooling is intense, the implanted atoms are inactive, and the process should be less intense.

The advantages of ion implantation are listed below.

1. The process runs at a relatively low temperature, the substrate does not overheat.
2. Uniform in thickness coatings can be obtained.
3. The chemical composition of the deposited coatings is accurately reproduced.
4. The process runs fast.
5. The process is recommended for doping with impurities with low solubility in the solid phase or with low diffusion coefficients.



The nonequilibrium process during ion implantation causes the formation of such alloys in the surface layers that cannot be obtained under normal conditions due to diffusion of components or limited solubility.

Ion implantation provides obtaining alloys of a certain composition in the surface layer. Surface properties can be optimized without reference to the bulk properties of the material. Implantation is possible at low temperatures without a noticeable change in the size of the product.

The disadvantages of ion implantation should be noted.

1. Implantation is a surface treatment process only in the area of direct action of the ion beam due to defocusing of the beam at large deviations. Therefore, it cannot be used to process substrates with complex surface geometry.

2. The shallow depth of penetration of the ion beam does not allow the deposition of coatings of sufficient thickness ( $> 1 \mu\text{m}$ ) on the parts of friction pairs and the cutting tool.

3. Rather sophisticated equipment is used.

**Discussion and Conclusions.** A comparative analysis of the methods of physical deposition of films has shown their pros and cons.

Cathode (ion), magnetron, and ion-beam sputtering have common advantages.

1. The processes run at a relatively low temperature, the substrate does not overheat.

2. It is possible to obtain coatings uniform in thickness.

3. The chemical composition of the deposited coatings is accurately reproduced.

The rest of the advantages and disadvantages of the considered methods are individual.

The process of cathode (ion) sputtering allows for the deposition of films on sufficiently large areas with high utilization rate of the sprayed material, but it has the lowest deposition rate (0.3–2 nm/s). Films have high adhesion to the substrate, but are characterized by a high level of mechanical stress. In addition, films are contaminated by the working gas since the working pressure in the chamber can be 1–10 Pa. Nevertheless, cathode (ion) sputtering provides using refractory materials as targets and synthesizing multicomponent compounds.

Versatility is the major advantage of magnetron sputtering systems. They can use DC sputtering, RF sputtering and reactive ion plasma film deposition.

Advantages of magnetron sputtering systems are as follows.

— high deposition rate of film coatings (several microns/min) and its adjustability in a fairly wide range;

— high chemical purity of film coatings;

— low thermal effect on the substrate and the deposited film coating;

— possibility of applying films uniform in thickness to fixed substrates.

Ion sputtering has the following advantages over magnetron:

— low working gas pressure ( $10^{-3} - 10^{-2}$  Pa);

— integrity of the chemical composition of the target material (cathode);

— energy-handling of the ions bombarding the target;

— increase in the rate of sputtering the target through ion bombardment at an angle to its surface (impossible under magnetron sputtering).

For the deposition of thin films of dielectric and composite materials under ion-beam sputtering, cold cathode ion sources are proposed, which create radially directed converging or radially diverging ion beams [3, 8].

Ion sources for ion-beam sputtering with a cold cathode have the following advantages:

— promote the formation of ion beams of both inert and chemically active gases (e.g.,  $\text{O}_2$ );

— have long life of the cold cathode;

— provide uniformity of the chemical composition of film coatings with large areas on stationary substrates;

— allow for automating the deposition of films of dielectric and composite materials with specified properties.

To obtain thin films, using the technique of alternating magnetron and then ion-beam sputtering processes is effective. This is how film coatings uniformly modified in depth are obtained [10]. This is important in the production of parts of friction pairs [5–7] and cutting tools to improve their quality [11]

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*The author has read and approved the final manuscript.*