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Equipment and processes of vacuum electron-ion plasma surface engineering

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ABSTRACT

The paper presents a COMPLEX laboratory setup for surface engineering, describes its components and principle of operation, and provides experimental data on its use for treatment of commercially pure VT1-0 titanium. The setup comprises gas and metal plasma generators based on a low-pressure arc discharge and a pulsed plasma-cathode electron source, allowing complex treatment of materials through nitriding in low-pressure arc plasma and subsequent TiN coating deposition in a single vacuum cycle. The experimental data demonstrate that such complex surface modification decreases the friction coefficient of VT1-0 titanium 1.3 times and increases its wear resistance more than 30 times.

1. Introduction

Surface modification by electron-ion plasma technologies can provide functional layers and coatings that greatly increase the physical and mechanical properties of materials (tools, parts, mechanisms, etc.), their performance, and service life under extreme operating conditions, thus providing higher energy efficiency and resource saving [1–31].

Such treatment creates nonequilibrium structural-phase states in surface layers of metals and alloys [8,14,15,24], forming a multiscale structure which comprises nano-, meso-, and macroscale elements and is capable of providing efficient elastic stress relaxation along with high physical and strength properties.

The technologies of electron-ion plasma treatment use concentrated energy fluxes of electron, ion, plasma, and laser beams [1,2,9,12,13,15,17]. The major advantage of such modification is in reactive interactions of dispersed inclusions through which nanosized metastable phases with unique physicochemical properties arise in material surface layers and provide their high service characteristics. New opportunities for surface modification are offered by hybrid methods combining different types of concentrated energy fluxes. The now available methods of complex electron-ion plasma treatment use several special setups, each serving for one or another individual process to provide a desired sequence of modification. Their disadvantages are obvious: they are expensive because it is expensive to maintain several setups, each with backup units (vacuum system, working chamber, power supplies, control units); they take a rather long time to realize complex treatment because each individual process is realized on a separate setup; and for the same reason, they fail to provide high surface purity as treated materials are to be transferred from setup to setup and their surface can thus be contaminated, e.g., by atmospheric gases.

For efficient electron-ion plasma treatment, it is reasonable to combine its constituent processes in a single vacuum cycle [2,15,18,19,23,24,26,28,29,31], including (1) surface saturation with nitrogen, carbon, oxygen, etc. to form a gradient multiphase surface layer, (2) synthesis of thin metal films or superhard nanostructured coatings based on nitrides, carbides, borides, etc., and (3) surface alloying through mixing of film–substrate systems to form layers with predictable functional properties or alloying of coatings into substrates for their better adhesion.

Here we present unique equipment and technology for complex electron-ion plasma treatment in a single vacuum cycle which can provide radical surface modification of the structure and properties of metals and alloys used to manufacture friction pairs, metal- and wood-working tools, high-duty parts in oil-extracting industry, gas industry, processing industry, etc. rated to operate under severe

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conditions (high loads, temperature gradients, corrosive environment, etc.).

2. COMPLEX laboratory setup for surface modification in a single vacuum cycle

The COMPLEX setup allows successive surface engineering of metal and ceramic materials in a single vacuum cycle through ion plasma nitriding, plasma-assisted coating deposition, and electron beam treatment with surface polishing and mixing to attain desired surface properties, including those radically different from substrates. Thus, in a single vacuum cycle, one can form thin surface layers of required composition, melt and mix them with substrates by an electron beam, and obtain a structure with properties gradually varying from substrate to surface due to diffusion of their interface by electron beam mixing which, in addition, resolves the problem of film to substrate adhesion. What is also noteworthy is that the setup provides for any sequence of nitriding, coating deposition, and electron beam treatment. The proposed method of complex modification opens the way to impart unique surface properties to mechanical parts and components initially made of inexpensive and accessible materials.

The COMPLEX laboratory setup for electron-ion plasma modification of metals and alloys in a single vacuum cycle consists of a base, a vacuum system, a cooling system, an automated control system, a chamber of bulk ion plasma treatment, a chamber of electron beam treatment, a three-dimensional object manipulator, and power supplies of the plasma and electron sources. Fig. 1 shows a simplified 3D schematic of the setup.

The manipulator provides for arranging specimens either on a stainless steel plate fixed in the horizontal plane at one of the manipulator satellites or directly in the satellite mount or in a special holder. Once the specimens are arranged, the setup realizes a vacuum pumping cycle, and on approaching an ultimate pressure below 6.6×10^{-3} Pa, their ion plasma treatment begins. The manipulator moves into the chamber of bulk ion plasma treatment, the gate between two chambers is closed, and the specimens are exposed to ion plasma cleaning and heating with the PINK plasma generator in an argon atmosphere at a pressure of 0.1–0.5 Pa, discharge current of 10–80 A, and negative bias voltage gradually varied up to 1000 V. After Ar ion bombardment and cleaning, coatings are deposited on the specimens according to a process chart for which one of the two arc evaporators with a discharge current of 150–150 A is turned on at a typical PINK generator current of 10–30 A, negative bias voltage of 30–50 V for deposition of metal coatings and 150–250 V for ceramic ones, and gas pressure of 0.05–0.3 Pa depending on the coating type. The gas atmosphere is argon for deposition of pure metals (Ti, Al, Zr, Cu, etc.) and commercially pure nitrogen for deposition of nitride coatings. After deposition, the film–substrate systems are exposed to surface melting and mixing by an electron beam for which the gate between the chambers is lifted, the manipulator moves into the chamber of electron beam treatment, taking the position under the beam, and then displaces according to a specified treatment program. Before irradiation, argon is pumped to the chamber to a pressure of up to 3.5×10^{-2} Pa controlled with a gas leak, and then, the specimens are irradiated at a pulse energy density of 10–30 J/cm^2, average electron energy of 15–20 keV, pulse duration of 50–200 μs, pulse repetition frequency of 0.3–1 Hz, and number of pulses $N = 3$–30. After irradiation, the manipulator either moves into the chamber of bulk ion plasma treatment for further deposition or nitriding or remains in position for cooling and removal of the specimens. Typically, ion nitriding is realized in nitrogen at a pressure of 0.5–1 Pa, PINK generator current of up to 100 A, and negative bias voltage controlled in the range 300–900 V to maintain a desired temperature.

3. Design and principle of operation of the COMPLEX setup

3.1. PINK hot-cathode plasma generator

Fig. 2 shows a schematic of the PINK hot-cathode plasma generator used for ion cleaning, surface activation, and nitriding.

The PINK generator comprises cylindrical hollow stainless steel cathode *7* (inner diameter 80 mm, length 350 mm) located on its water-cooled flange, W-shaped hot cathode *3* made of four parallel-connected tungsten wires (each of diameter 0.8 mm and length 180 mm) located on two water-cooled copper current leads *5* inside the hollow cathode, cathode unit *6* fastened to water-cooled case *1* via an insulating spacer, and magnetic coil *2* which produces a discharge-stabilizing longitudinal magnetic field of 0.1–3 mT. The hollow cathode is pumped with working gas to increase the ionization efficiency. The operating pressure in the region of treatment (working chamber) is about 0.1–2 Pa. The PINK generator case is connected to the vacuum chamber whose inner walls serve as the arc discharge hollow anode.

The PINK plasma generator based a non-self-sustained discharge provides a wide range of operating parameters for which it is equipped with trigger electrode *4* inserted in hollow cathode *7* and connected to the main anode (vacuum chamber walls), thus stabilizing the discharge initiating voltage, increasing the efficiency of gas ionization and plasma formation, and decreasing the minimum pressure for stable discharge ignition and operation. For better operation stability of the PINK plasma generator, the trigger electrode has a fixed contact with the main discharge anode via ballast resistance R_b, as shown in a block diagram of the generator and its power supply in Fig. 3.

The basic idea of using trigger electrode *5* positioned near hot cathode *8* (Fig. 3) is that the trigger electrode serves as an auxiliary discharge anode. When a discharge is ignited between hot cathode *8* and trigger electrode *5*, hollow cathode *4* is filled with plasma which, due to its density gradient, then propagates into main hollow anode *1* (chamber); and because of ballast resistance *2* in the trigger electrode circuit, the discharge switches to the main anode, and the trigger electrode current becomes low (up to an ampere). This stabilizes the operation of the main discharge, prevents its extinction in response to pressure variation in the chamber, and provides its re-ignition on operation of the microarc switching system.

The PINK plasma generator is powered by a special system comprising a discharge power supply, a filament power supply, and a magnetic coil power supply.

3.2. Arc evaporator with magnetic cathode spot stabilization

The arc evaporator of the setup operates at a discharge current of up to 150 A, provides magnetic cathode spot stabilization in the operating cathode zone, and allows efficient deposition of functional coatings. Its schematic is shown in Fig. 4.

The maximum evaporated cathode diameter is 78 mm, allowing one to have a compact arc evaporator and sufficient cathode material for long-term operation. The cathode is shaped as a truncated cone with a half-circle groove of radius 2 mm near its larger diameter for mounting the cathode in its holder. The cathode shape provides the ignition of a cathode spot at the lateral cathode surface and its motion to and stable operation in the effective cathode area. Fig. 5 shows a block diagram of the arc evaporator with its power supply.

The arc evaporator power supply consists of a discharge power supply, two magnetic coil power supplies, and an arc trigger unit.

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