

Analysis of work and efficiency increase of of the ILUR-03 installation magnetron system for tubular specimens outer surface modification

B A Kalin¹, N V Volkov¹, R A Valikov¹, A S Yashin¹, V P Krivobokov², S N Yanin² and Yu N Yuriev²

¹ National Research Nuclear University MEPHI (Moscow Engineering Physics Institute), 115409, Russia, Moscow, Kashirskoe highway, 31

² National Research Tomsk Polytechnic University, 634050, Russia, Tomsk, Lenin Avenue, 30

E-mail: nvvolkov@mail.ru, bakalin@mephi.ru, yashin_itf@mail.ru

Abstract. The method of material near-surface layers doping by mixing of alloying elements films with ion beam is widely used in science and technology. Three magnetrons with independent power systems, integrated in installation for ion-beam treatment of long-range products ILUR-03, were used as deposition systems. Targets for magnetrons were in the form of disks 60 mm diameter and 5 mm thickness and consisted of the following elements: Al, Fe, Mo, Zr, Cr of purity better than 99.99 at.%. Deposition was performed in argon atmosphere at 1-5 Pa pressure and room temperature in stable current mode at 30-100 mA. Analysis of the obtained films on the surface of cylindrical specimens from zirconium alloys with the outer diameter of 9.15 mm showed high uniformity of coating on length of 300 mm, good adhesion and absence of discontinuities in the films body.

1. Introduction

The method of magnetron coating is the deposition of the target atoms, which are sprayed in the magnetron plasma, on the substrate. This method allows us to put films with accuracy up to tens of nanometers on a wide class of substrates, and therefore it is widely used in various fields of science and technology. For example, the effectiveness of the surface modification of metal materials by mixing of the surface layer by ion beam depends on the quality and uniformity of previously deposited films of alloying elements. The magnetron sputtering method enables multi-component substrate doping as film deposition is not a thermally-activated process [1].

2. Materials and tools

Installation ILUR-03, which is a system for the complex ion-beam processing of long cylindrical products, is used in this paper [2]. As can be seen from figure 1, the installation vacuum system consists of a preliminary evacuation chamber, an ion source chamber and a chamber for deposition, in which the three independent magnetrons are installed. The specimens are delivered through the gateway system using a manipulator operating in automatic mode.



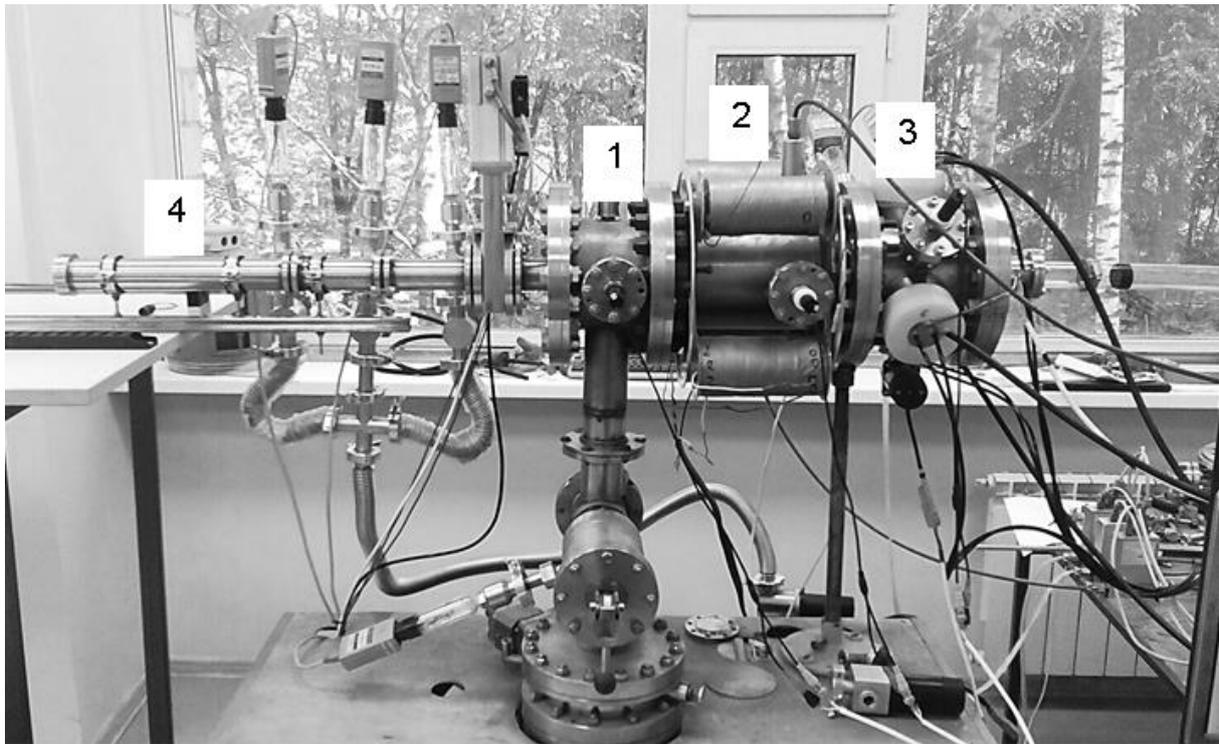


Figure 1. Installation ILUR-03: preliminary evacuation chamber (1), ion source chamber (2), chamber for deposition (3) and the gateway system (4).

Voltage is applied to the magnetron through specialized power supplies that can operate in a mode both DC and impulse voltages up to 800 V. The targets are in the form of disks 60 mm in diameter and 5 mm thick. Metal plates from Al, Fe, Mo, Zr, Mg, Cr with purity better than 99.99 at.% were used as materials for sputtering. The substrates were pieces of cladding tubes from E110 alloy (Zr-1%Nb) with the outer diameter of 9.15 mm and a length of 300 mm. The uniformity of coating was achieved by the continuous rotation of the tube and its movement in the longitudinal direction. Rotation and movement speed was between 1-10 rpm and 5-50 mm per min respectively.

The processing modes were selected during the preliminary experiments. Deposition was performed in an argon atmosphere at a pressure of 1-5 Pa in the discharge current stabilization mode at 30-150 mA. Residual gas pressure was maintained at 10^{-3} Pa. The appearance of the magnetron discharge at different current values is shown in figure 2.

To improve the adhesion surface the substrates were pre-treated by a radial beam of Ar^+ ions with the mean energy of 3-4 keV and total dose irradiation 10^{17} - 10^{18} ion cm^{-2} . As a result of this treatment the contaminants and particles adsorbed with the surface of the tubes are removed, and the relief becomes smoother [3]. After the coating doping of the substrate surface layer was carried out by ion mixing of material by Ar^+ beam with the total radiation dose of 10^{18} - 10^{19} ion cm^{-2} .

The current-voltage characteristics of the discharge, taken in the operating pressure range for a variety of targets are shown in figure 3. As can be seen on the example of Al, Mo and Cr, functions are close to linear in this voltage range, and the voltage discharge initiation is about 250-350 V.

These samples were examined using optical and scanning electron microscopes. The thickness of the films was determined by the gravimetric method on the GH-252 A & D analytical balances with a sensitivity of 0.01 mg. The elemental composition of the surface layer of the samples was monitored by X-ray microanalysis.

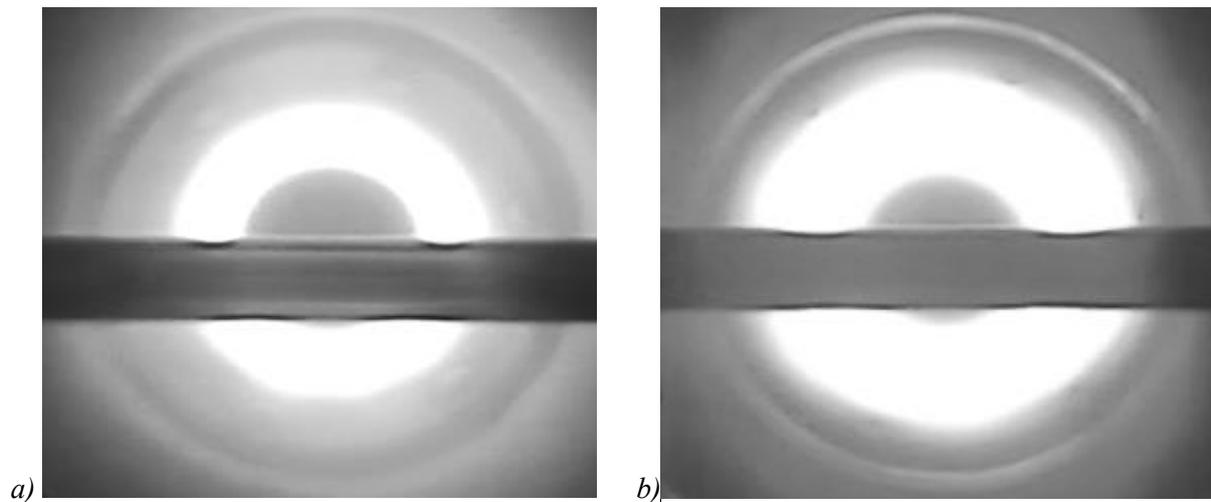


Figure 2. Appearance of magnetron discharge on a target of Al at a current of 50 mA (a) and 100 mA (b), a dark line in the foreground - a cylindrical substrate.

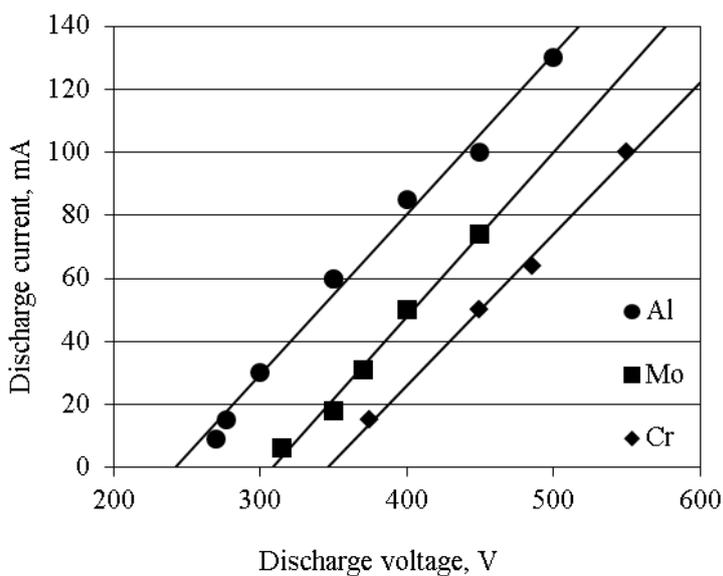


Figure 3. The current-voltage characteristics of the magnetron discharges on targets of Al, Mo and Cr, solid lines indicate a linear approximation.

3. Results and discussion

Figure 4 presents images of the sample with the iron sprayed layer obtained in the mode of the secondary and back-scattered electrons as an example. No significant irregularities of the deposited film are visually observed, and the film itself follows the surface topography of the original sample. Thus, the technological scratches formed during the finish machining of the tube and the inclusions of less than 1 μm size are clearly distinguished.

Table 1 summarizes the results of the elemental composition of the surface layer of doped samples obtained by X-ray microanalysis. As it can be seen, the atomic percentage of material in element films whose thickness was from 60 to 150 nm after the doping ranges from 0.3 to 3.5.

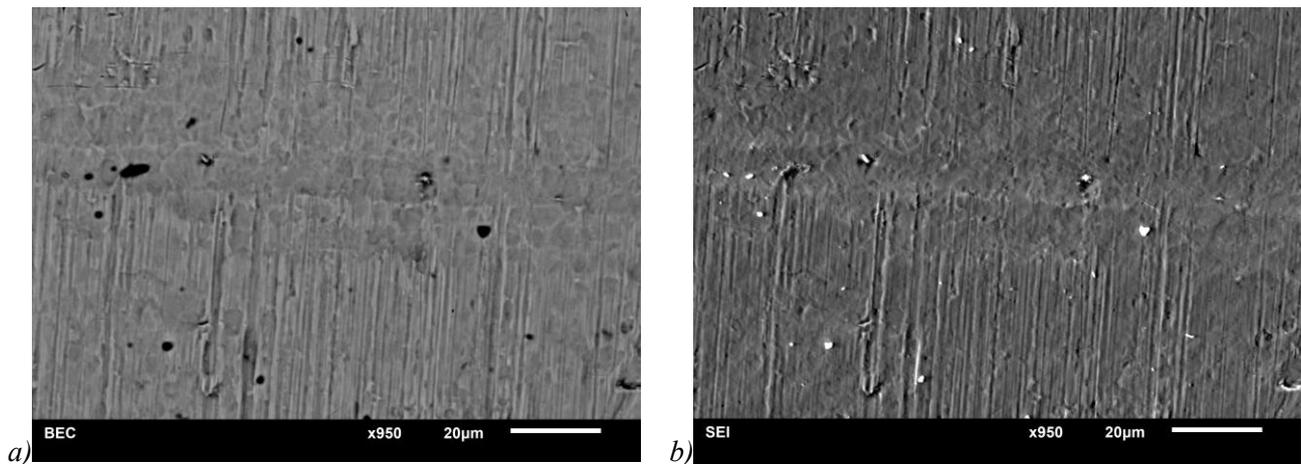


Figure 4. Images of the sample surface with iron sprayed layer obtained in the mode of secondary (a) and back-scattered electrons (b).

Table 1. The results of X-ray microanalysis of the sample surface after doping by ion mixing of deposited films.

| | №1 | №2 | №3 | №4 | №5 |
|---------------------|-------------|-------------|-------------|-------------|-------------|
| | Al-alloying | Mg-alloying | Fe-alloying | Cr-alloying | Mo-alloying |
| Zr (at %) | 98.4 | 98.7 | 94.9 | 97.9 | 98.3 |
| Nb (at %) | 0.5 | 0.8 | 0.9 | 0.9 | 0.8 |
| Fe (at %) | - | - | 3.5 | - | - |
| Cr (at %) | - | - | - | 0.6 | - |
| Mo (at %) | - | - | - | - | 0.5 |
| Al (at %) | 0.8 | - | - | - | - |
| Mg (at %) | - | 0.3 | - | - | - |
| Film thickness (nm) | 80 | 90 | 150 | 60 | 70 |

4. Conclusion

The operation of the magnetron system of the ILUR-03 installation for ion beam processing of long cylindrical parts is analyzed. The range of operating parameters to ensure the effectiveness deposition of the films of several alloying elements Al, Fe, Mo, Mg, Cr on samples for subsequent surface doping in ion mixing mode is determined.

References

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