DEPOSITION OF REFRACTORY METAL FILMS WITH PLANAR PLASMA ECR SOURCE

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The condition of the deposition of refractory metal and alloy films such as W, Mo, Ti, Ta, Cr, Ni, $Fe_{80}B_{20}$ with using ECR plasma source at the different magnitudes of bias voltage on targets and substrates were investigated. It was revealed that the maximal rate of the films growth amounted to 0,052 µm/min for Cr. The thicknesses of the films were obtained of 16-20 µm in the current experiments.

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INTRODUCTION

Advanced development of modern engineering is connected with the using of the high technology directed to ensuring of competitive ability, the prime cost reduction, the renewal period of the machines and assembly units operation. Improvement of operating characteristics of constructional materials is achieved by utilization of modification work surfaces technology, deposition of protective and functional coatings. For example, the deposition of solid corrosion-resistant covering on permanent magnets has been made by using of rare earths elements (Fe,Nd,B; Sm,Co and others) will allow not only to increase the service life of the manufactures but also to extend notably the field of their application. The methods of construction materials protection by the deposition of the unbroken corrosion and heat-resistant coating are also important for structure units of the atomic reactor heat zone and their functional elements. Among numerous modes of depositions the ion-plasma methods that allow to deposit multilayer and combined films of refractory materials on practically any substrates occupy an important place [1-5].

In this paper the conditions of the deposition of thick (up to 20 µm) refractory metal and alloy films such as W, Mo, Ti, Ta, Cr, Ni, Fe_{80},B_{20} on polished copper substrates and commercial permanent magnets (Fe_{14},Nd_2,B) by ECR plasma enhanced physical sputtering method were investigated and optimized in dependence on the magnitude of applied bias voltage to the targets and substrates.

EXPERIMENTAL SETUP

The experiments were performed in the water-cooled vacuum chamber of 200 mm in diameter and 250 mm in length on one end which was installed planar rectangular ECR plasma source with multipolar magnetic field and double-slotted antenna generating microwave radiation (2,45 GHz) in magnetic structure. This plasma source was described in detail at [6]. The plasma source housing was galvanic connected with grounded vacuum chamber. Resonance zone was created at the distance of 12 mm from the upper copper screen protecting permanent magnets from plasma stream. The scheme of the experiment is showed in Fig.1. The flat sputtered target of rectangular form (50 × 50 mm^2 in dimension) was arranged at the distance of 50 mm from resonance zone and was oriented at the angle of 45° to the plasma source plane. The position of the target was chosen from consideration of the maximum ion current on it.

The sample-substrates from polished copper or hard magnetic material were disposed at the distance of 10 mm from lower edge of the target. In order to prevent the direct impact of rapid technological ions with type-high substrate surface the substrate was installed practically parallel to the ion stream impacting on the target. The movable holders of the target and the sample were derived from vacuum chamber by the vacuum sealing and had not galvanic contact with grounded elements of the chamber. The targets and the sample were not forcibly cooled.

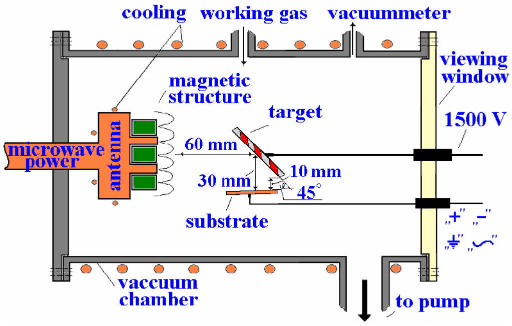


Fig.1. Experimental set-up

The ultimate pressure in vacuum chamber was about 5 $\times 10^{-6}$ Torr. The process pressure (10^{-3} Torr) was attained by variation of process gas (Ar) flow and pumping speed. At the gas flow of 1 sccm and the microwave power up to 300 W the plasma parameters in the range of target disposition and sample amounted: electron density up to 10^{10} cm^{-3}, electron temperature equaled of 12 eV, current density of ion flow to grounded substrate was equaled ~ 3,5 mA/cm^2.

In this experiment the currents to target and sample were measured. The thickness of deposed films was evaluated by profilometer and then compared with the indication of the optical microinterferometer MII-4. For this measurement the surface sample part was closed by thin screen to keep the reference area.

EXPERIMENTAL STUDIES AND RESULTS

During the process of pretreating the samples for deposition films ultrasonic washing, degreasing was used. The final preparation was realized by ion etching. The regime of the ion-plasma etching was carried out at voltage bias on the substrate about -300 V and the ion current density of 5.5 mA/cm^2 within 10 min. During this time the depth of etching attained to 0.43 μm and the sample temperature increased up to 400°C that facilitated to complimentary degassing of the type-high surface.

The experience was executed at the different magnitudes of microwave power, the voltage on the target and the voltage bias on the substrate. The gas flow and the working pressure were kept permanent. The primary factor determining the work regime choice was the thickness of deposited coating. For comparison deposed on copper substrate films were analyzed at the dc and the pulsating voltages with the frequency of 100 Hz on the tungsten target. The voltage amplitudes on the target amounted to -1500 V and the substrate was grounded. The temperature of the target totaled to 600°C.

From the visual analysis of the deposed films it was followed that the thickness of the coating at the pulsating bias on the target was about 20 % higher than at the permanent voltage on the target and microdrops were practically missed on the film. It seems such difference is due to the vacuum pumping system. When used the diffusion pump it was real possibility to form on the hot target surface the non-conducting film fragments from products of the diffusion oil dissociation, which could accumulated the positive charge and thereby the ion flow on this areas was limited. Furthermore the positive charge on the parasitic film fragment could promote the development of the unipolar microarcs that cause of appearance of the drop fraction on the deposed coating. In the case of the pulsating voltage the accumulated charge was limited and the isolated areas were easily etched.

The measurement of ion current to the titanic target the magnitude which determines the sputtering rate and consequently the coating thickness on the magnitude of the bias on the target and the supply power showed that the ion current ran in the target circuit at the zero potential on the target equal 30 mA. The existence of its current was maintained by the ion flow with the energy of 20 eV in the diffusing expansible plasma from resonance zone of plasma source [6]. At supply power of 150 W with growth of target potential up to -200 V ion current fast increased and then starting at 400 - 500 V reached the saturation. With growth of supply power up to 300 W the plasma density amplified too and the saturation threshold amounted up to -1500 - 1600 V at the ion current of 160 mA. The similar measurement was carried out for W, Mo, Ta, $Fe_{80}B_{20}$ targets.

The basic experiments were implemented under the maximum voltage bias at the target of -1500 V and supply power of 300 W. The current density amounted to 6,7 mA/cm^2 at the full current 160 mA.

The following experiment series was dedicated to research of influence of the direct positive, negative and alternating substrate bias on growth of deposed film. The temperature of the substrate reached up to 600°C in all experiments. It is shown that the maximum film thickness was obtained at positive substrate voltage of +60 V. The increasing or the substantial reducing of the bias value was led to diminish of coating thickness. At the negative substrate bias of -60 V the growth rate of the film was by a factor of two lower then under the positive voltage. Such result can be connected with bombarding of the type-high surface by the process ions and sputtering of growing film despite to the fact that substrate position excluded the direct impact of Ar ions from ECR zone. At the same time the background plasma density in the area of the substrate disposition was large sufficiently (10^{10} cm^{-3}) to provide with the ion current on the substrate ~ 40-50 mA that exerted appreciable influence on growth film rate. Unfortunately we could not move away the target and the substrate on larger distance from resonance zone. The analogous results were received for the targets from W, Ti, Mo.

The result with alternating (frequency of 50 Hz) bias on the substrate was somewhat unexrected. At the bias amplitude of ± 42 V the rate of the film growth slightly differed from maximum that was obtained under the positive voltage on the substrate. Furthermore in sputtering the target from $Fe_{80}B_{20}$ alloy and in deposing of the film on the substrate from Nd_2,Fe_{14},B magnetic material the thickness of the formed film turned out more thick namely under the alternating substrate bias in comparison with the direct bias. Perhaps as in case of the target the process of the film growth was accompanied by the precipitation and pulling down of the extrinsic chemical compound. As a whole this effect remains incomprehensible and requires complementary studies.

At the alternating substrate bias the film growth rate were determined while sputtering the W, Mo, Cr targets. The thicknesses of the coatings depending on the exposition time up to 8 hours are showed in Fig.2. One can see that the trend of curves conform to the coefficient of sputtering the targets material [7].

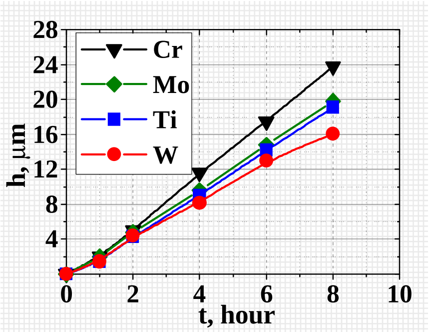
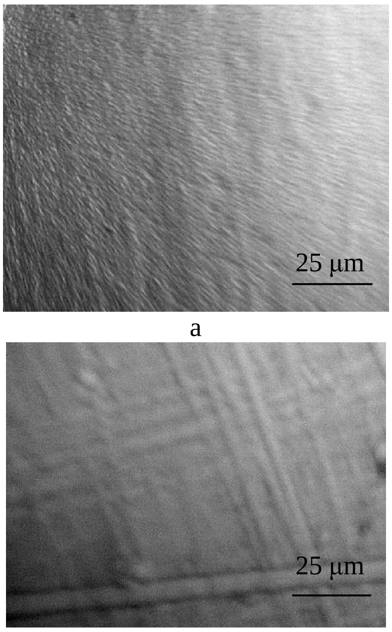


Fig. 2. The films thickness as a function of deposition time

The linear dependence of the film thickness remained constant during all deposition time. The peak coating thickness up to 24 μm was received for chromium film at the growth rate of 0,052 μm/min.

In this paper only visual analysis of the received films was realized. As an example the photograph of the Ti coating on the copper substrates and on the Fe_{14},Nd_2,B alloy are shown in Fig.3 a,b.

The streaks on the Ti films (Fig.3,b) is the track of the diamond saw cut in the process of the sample preparation. As state above the additional treatment of the samples from magnetic material were not carried out. The analysis of the film on the copper substrates are displayed that the thin films up to 5 - 7 μm entirely replicates the structure of the etched copper surface.



a

b

Fig. 3. Mmicroscopic photograph of Ti film :
a) on the Cu substrate, the film thickness is 14.4 µm;
b) on the Fe_{14},Nd_2,B alloy

With growth of the film thickness the grains boundaries was smoothed and the film surface became unbroken and homogeneous. The crystal structure of deposed coating began manifest on some films with the thickness more than 16 µm. Thus the thick films deposed by ECR plasma can close a defect of the substrate surface (pores, crevices) that it is especially important for a corrosion - resistant coating of magnetic materials. In this time the Ti and $Fe_{80}B_{20}$ coatings deposed on the Fe_{14},Nd_2,B magnetic samples are examining on the corrosion stability.

CONCLUSIONS

The presented experimental results indicate that the thick films from refractory materials such as W, Mo, Ti, Cr can be deposited on different materials by using of ECR plasma. It was investigated the influence of the magnitude and form voltage bias to different targets and substrates on the rate of the film growth. At the microwave power about 300 W the optimal film characteristics were received under the pulsating voltage of - 1500 V with the frequency of 100 Hz on the target and the alternating (frequency of 50 Hz) substrate bias of 42 V.

It was demonstrated that the coating thickness linearly increased while raising the exposition time. The maximum rate of the film thickness growth up to 0.052 µm/min was obtained for chrome.

The condition of the deposition of the unbroken corrosion-resistant coating on the samples of the commercial permanent magnets (Fe_{14},Nd_2,B) were executed.

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НАНЕСЕНИЕ ПОКРЫТИЙ ТУГОПЛАВКИХ МЕТАЛЛОВ С ИСПОЛЬЗОВАНИЕМ ПЛАНАРНОГО ЭЦР ПЛАЗМЕННОГО ИСТОЧНИКА

В.Д. Федорченко, В.В. Чеботарев, А.В. Медведев, В.И. Терешин

Исследованы условия нанесения покрытий тугоплавких материалов, таких как W, Mo, Ti, Ta, Cr, Ni, а также сплава $Fe_{80}B_{20}$ с использованием ЭЦР плазменного источника при различных величинах смещения напряжения на мишени и подложке. Показано, что максимальная скорость роста толщины покрытия составляла 0,052 мкм/мин для Cr. В данных экспериментах были получены покрытия толщиной порядка 16 - 20 мкм.

НАНЕСЕННЯ ПОКРИТІЙ ТУГОПЛАВКИХ МЕТАЛІВ З ВИКОРИСТАННЯМ ПЛАНАРНОГО ЕЦР ПЛАЗМОВОГО ДЖЕРЕЛА

В.Д. Федорченко, В.В. Чеботарьов, О.В. Медведєв, В.І. Терешин

Досліджено умови нанесення покритій тугоплавких металів, таких як W, Mo, Ti, Ta, Cr, Ni, а також сплава $Fe_{80}B_{20}$ з використанням ЕЦР плазмового джерела при різних величинах напруги на мішені та підкладці. Показано, що максимальна швидкість росту товщини покриття складала 0,052 мкм/хв для Cr. В даних експериментах були отримані покриття товщиною порядку 16 - 20 мкм.