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DEPOSITION OF COATINGS OF TUNGSTEN AND TWO-LAYER COPPER-TUNGSTEN COMPOSITION

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In the work a technology of tungsten films and two-layer copper-tungsten coatings deposition on stainless steel substrates was developed. The coatings were deposited by magnetron and arc sputtering of materials with condensation of them on the testing substrates. The deposition objects were probe of the ICRF antenna on fusion devices and reference samples, on which the properties of obtained coatings were studied. The possibility of tungsten coatings forming on long-length elements of functional blocs was considered.
KEY WORDS: sputtering systems, multilayer coatings, tungsten films, copper films, adhesion

НАНЕСЕНИЕ ПОКРЫТИЙ ВОЛЬФРАМА И ДВУХСЛОЙНОЙ КОМПОЗИЦИИ МЕДЬ-ВОЛЬФРАМ
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В данной работе разработан способ нанесения пленок вольфрама и двухслойных покрытий медь-вольфрам на подложки из нержавеющей стали. Покрытия наносились методами магнетронного и дугового распыления материалов с конденсацией их на тестируемые подложки. В качестве объектов для нанесения выступали пробники ICRF антенны высокочастотного нагрева плазмы в установках управляемого термоядерного синтеза и образцы-свидетели, на которых изучались свойства полученных покрытий. Рассмотрена возможность формирования вольфрамового покрытия на длинномерные элементы функциональных узлов.
КЛЮЧЕВЫЕ СЛОВА: распылительные системы, многослойные покрытия, вольфрамовые пленки, медные пленки, адгезия

НАНЕСЕННЯ ПОКРИТТІВ ВОЛЬФРАМУ І ДВОШАРОВОЇ КОМПОЗИЦІЇ МІДЬ-ВОЛЬФРАМ
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У даній роботі розроблено спосіб нанесення плівок вольфраму і двошарових покриттів мідь-вольфрам на підкладки з нержавіючої сталі. Покриття наносилися методами магнетронного і дугового розпилення матеріалів з конденсацією їх на тестовані підкладки. В якості об'єктів для нанесення виступали пробники ICRF антени високочастотного нагріву плазми в установках керованого термоядерного синтезу та зразки-свідки, на яких вивчалися властивості отриманих покриттів. Розглянуто можливість формування вольфрамового покриття на довгомірні елементи функціональних вузлів.
КЛЮЧОВІ СЛОВА: розпилювальні системи, багатошарові покриття, вольфрамові плівки, мідні плівки, адгезія

The problem of the first wall is the most relevant at the present stage of creating of installations of controlled thermonuclear fusion [1]. In the systems with magnetic plasma confinement surfaces of structural materials facing the plasma will undergo intensive thermal load. According to the experts estimation heat flux on the first wall will be 1 MVt·m^{-2}. It could reach 20 MVt·m^{-2} in the divertor area and limiters. As energetically loaded elements antennas of RF heating of the plasma also may be considered. The first wall materials should withstand such heat flux without degradation. Besides of the high thermal resistance of structural materials, in fusion power devices the requirements to the influx of impurities from the wall into the plasma should be kept. The most important demand is the low level of impurities atoms as they may lose electrons in the plasma filament not in full; that resulting in radiation cooling of plasma [2]. For reducing energy losses due to radiation effects elements with low atomic number Z should be preferably used as the materials of the first wall. Atoms of these elements must be fully ionized in fusion devices plasma and radiation losses by impurity atoms will be minimal. The next important parameter to determine the applicability of a particular material is the retention of the hydrogen isotopes, especially tritium, in a material [3].

As main candidates for construction elements facing plasma carbon composite materials, beryllium and tungsten are consider. Beryllium has the smallest Z among them so it is the most do for the first wall material. At the same time its insufficient thermal resistance does not permit use it for elements [exposed with under powerful thermal fluxes (limiters and divertor plates). Carbon and carbon composite materials seem more attractive as compared with beryllium because after their thermophysical properties they can stand up to powerful thermal load [4]. At this the impurity atoms going into the plasma have acceptable atomic number and do not resulted in intense plasma cooling. But it should be

noted that at all positive properties carbon-base materials suffer from grave shortcomings. Carbon easy forms chemical compounds with hydrogen isotopes, including tritium. In the conditions existing in fusion devices chemical degradation of carbon-base structural materials may be rather high. Besides, carbon-hydrogen compounds spread to vacuum chamber and form films of complicated chemical compositions on construction elements. Such surface contaminations include chemically bounded tritium. That required additional radiation safety precautions for personnel leading in turn to deterioration of operating characteristics of fusion devices. MAGATE recommendation limites tritium amount accumulated in different constructional elements. Therefore the ways to remove carbon-hydrogen contaminations from the surfaces of structural elements of fusion devices are searched.

In spite of high atomic number tungsten has a number of incontestable advantages. These include: high thermal resistivity (the melting temperature is 3380 °C), low sputtering coefficient for hydrogen ions and isotopes, weak hydrogen isotopes retention. High atomic number is a considerable limitation for the material of the fusion devices first wall. For this allowable concentration of tungsten atoms incoming in the plasma must be several orders lower than one of low Z element. In spite of the severe restrictions imposed on the flow of impurity atoms from the wall into the plasma tungsten is considered as a promising material for facing plasma functional assemblies which are exposed to powerful thermal loads in ITER [5]. The studies [6, 7] show validity of this approach.

Tungsten having a number of positive properties that determined its applicability in the fusion devices is a hard and brittle material. Production of irregular shape objects from pure tungsten is technologically complex and expensive task. It is possible to reduce the costs of production of future reactors assemblies, using cheaper and more technologically promising structural materials, protecting the plasma facing surfaces with tungsten-based coatings. In this regard, the development of technology of tungsten and tungsten-based composite coatings on structural elements of experimental facilities of controlled thermonuclear fusion is an important task.

Functional elements of RF heating antennas facing the plasma filament work in hard conditions. Between the antenna and the plasma filament there are powerful high-amplitude high-frequency fields. Functional coatings for antenna elements working in such conditions must have properties characteristic of tungsten. In addition they must be mechanically strong, have good adhesion strength to the substrate, to withstand without breaking repeated cyclic thermal loads. Also, some specific requirements to these coatings exist: they must withstand repeated microbreakdowns without significant coating crippling, have low capacity for arcing. Microbreakdown may be caused on the surface of the coating by local impurities having low electron work function, and by point micro irregularities. Therefore, reducing the surface roughness is an additional technological problem in coatings creating.

In this work the method of deposition of tungsten films and two-layer copper-tungsten coatings on stainless steel objects of irregular shapes was developed. A modifying object was the probe of the ICRF antenna [8]. The aim of this paper is to get the tungsten films of 0.5 μm and 2.2 μm thicknesses on stainless steel (stl.st) and on stl.st with copper sublayer of the thickness at least 3 μm. It was assumed that the copper sublayer reduced the local heat load at the microbreakdown through the redistribution of heat in the volume of material with high thermal conductivity [9]. This resulted in reducing the local temperature and, consequently, to reduce the possibility of arcing. Copper sublayer could also be a damper redistributing the stresses at the locations of microbreakdowns, increasing the mechanical resistance of tungsten coating.

MATERIALS AND METHODS

The list of ways to get the tungsten coating is limited. Metal films can be obtained by electron-beam evaporation, plasma-arc deposition and magnetron sputtering. We chose the latter, as it compares favorably with the previous two. In electron-beam evaporator the source of the coating material is a small region of the melt. In the magnetron one the source of coating material is extended zone of erosion, which is preferable at the coating of objects with irregular shapes. In plasma-arc method material comes on the surface of the modified objects both as an atomic vapor and as clusters. At this there are droplets of molten material of the cathode, and in the case of tungsten even some particulates of the material. These particulates are pulled out from the surface of the cathode by the stresses that arise as a result of strong local overheating of the applying material in the area of the cathode spot. The presence of droplets and particles worse the quality of the surfaces of the coatings; magnetron deposition method is practically free from this imperfection.

The coating deposition was made on the device VUP-5 with the planar magnetron sputtering system of direct target cooling [10] placed on the top of working chamber. The target diameter was 180 mm. It permitted to realize deposition of uniform coating on bulky objects. The targets from pure tungsten, copper and titanium were used.

In addition to carried researches on the creation of multi-layer coatings and check-out of the effectiveness of their use in controlled thermonuclear fusion devices trial experiments on deposition of these coatings on long-length functional units were done. Despite named shortcomings of arc method of application, this method is the most suitable for the modification of large-size products. To realize such a task the coatings deposition on the extended arc sputtering device was tested.

For the coatings deposition by arc discharge in the work modernized in accordance with our requirements extended tube-type arc vapor source of continuous operation was used (Fig. 1) [11-13].

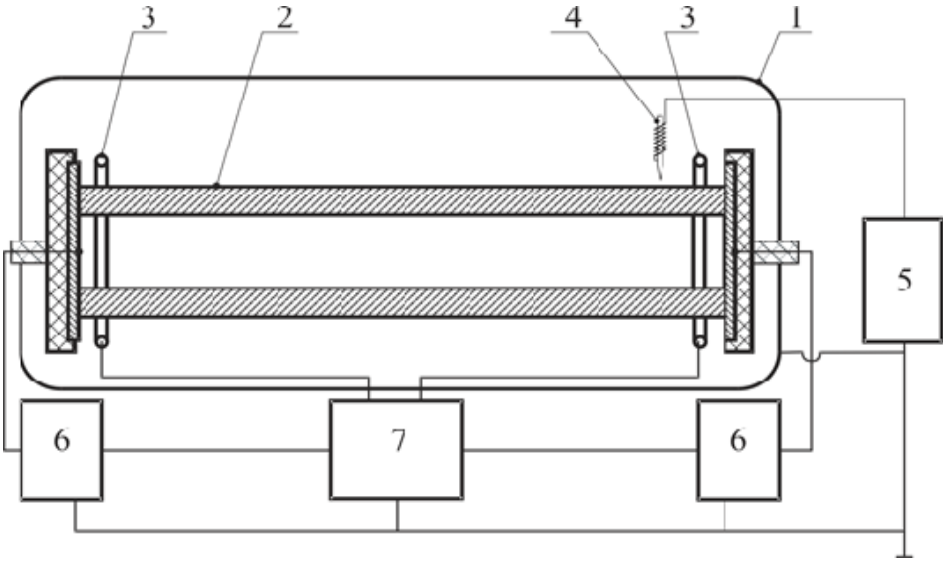

Fig. 1. Extended tube-type arc vapor source
1 – chamber; 2 – tube-type cathode; 3 – arc positional-sensing detector; 4 – ignition unit; 5 – circuit of ignition unit control; 6 – arc power supply unit; 7 – commutation circuit.

Continuous operation mode was provided with two identical power supply units (0-100 V, 0-300 V) [14]. Each modified unit (Fig. 2) was a three-phase thyristor rectifier used three-phase power transformer with a falling current-voltage characteristic and high leakage inductance. Powers from these units were connected to the opposite ends of a tube-type cathode.

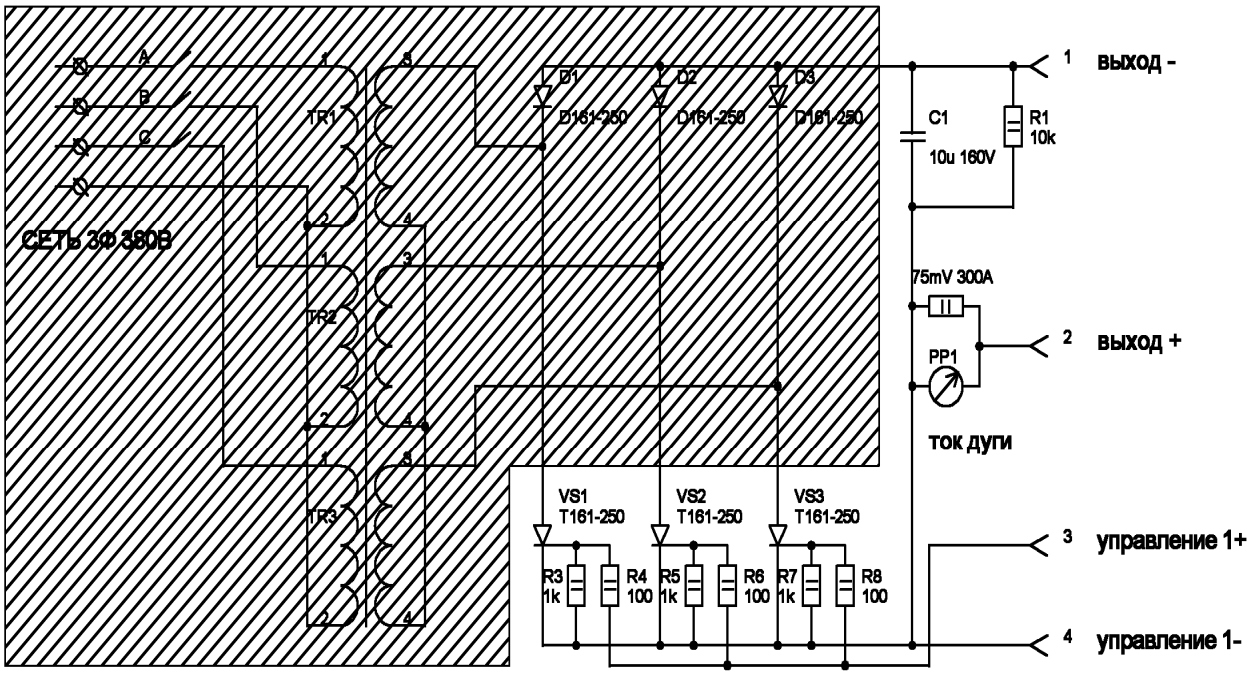

Fig. 2. Arc power supply unit.

Voltage was commuted with a control unit by a signal of arc positional-sensing detector [15]. The modernized circuit (see Fig. 3) was a pulse former converted a signal from the arc positional-sensing detector into an actuating signal for arc power supply thyristors. It permitted to prevent arc extinction in a reconnection moment as the control unit provided short-time simultaneous work of the power supply units before one of them switching-off. Besides, such option of the arc power supply enabled to avoid the commutation of high-current (up to 250 A) circuits.

Arc ignition was done by an electromechanical device powered directly from the cathode. The simplicity of design and small number of operations at continuous duty of arcing provided high reliability of the device and eliminated the relatively complex and often quite uncertain circuits of the high-voltage ignition.

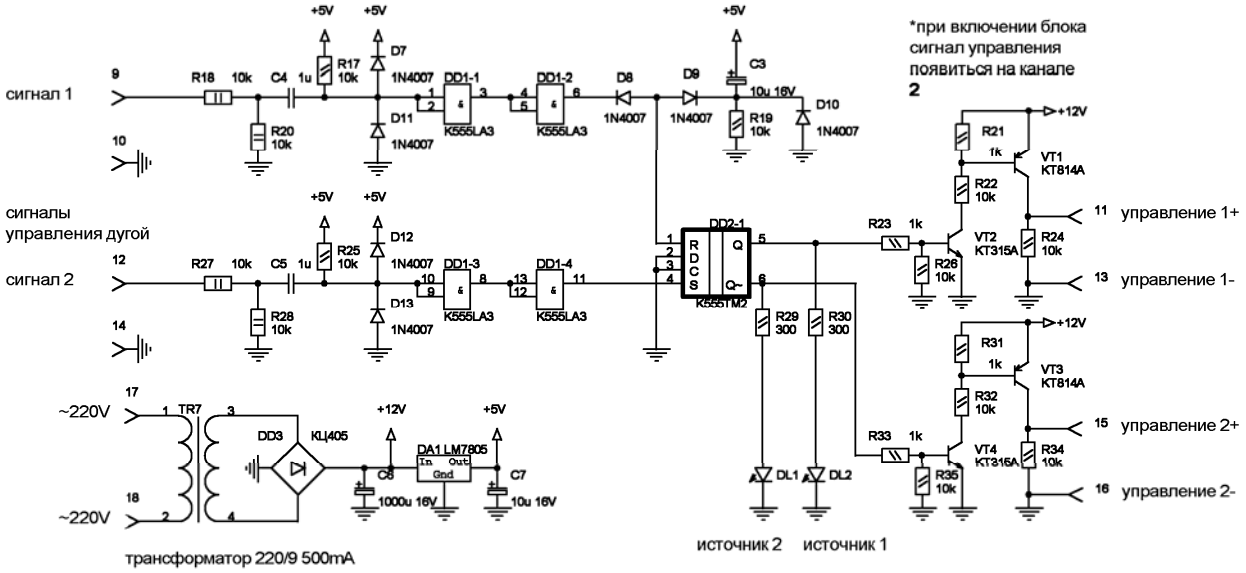

Fig. 3. Diagram of commutation of arc power supply unit

RESULTS AND DISCUSSION

The experiments showed that a tungsten film deposited on a cold substrate had high tensions. They appeared in the process of a tungsten coating forming. Internal stresses were so strong that caused film destruction. In some cases, the destruction of coating as well as the surface layer of the reference samples from a silicon single crystal was observed. We achieved satisfactory results depositing tungsten on the substrate heating up in the temperature range 330-350 °C. The choice of this temperature range was due to the optimum level of adhesion and was consistent with the regime of tungsten-based coatings deposition described in [16].

As our experiments shown coating strength and its adhesion parameters were influenced not only by strains arisen in the film itself but also by intrinsic strains in the substrate and in intermediate layers. The objects for the coating deposition were made from the stainless steel of the next composition: Cr 20 at.%, Fe 70 at.%, Ni 8.5 at.%, Ti 1.5 at.%. Under the requirements to the surface roughness the objects were preliminary polished. Samples were annealed in high vacuum at 800 °C and the residual pressure in the chamber $1 \cdot 10^{-5}$ Torr during 30 min to remove intrinsic strains. Then the samples were cooled in high vacuum down to the room temperature. The surfaces of samples became lustreless after annealing, defects that appeared in the result of preliminary mechanical treatment revealed. The surfaces of annealed in vacuum samples were polished again using a diamond paste of a small grain-size. As pastes were additional contaminant sources the sample surfaces were thoroughly rinsed and degreased in an ultrasonic bath.

In the coatings deposition chamber reference samples were placed on a heated table. RF probe was placed on the specially made heated holder that permitted to rotate the simulator angularly to the flux of the deposited material. The position and rotation velocity of the holder were chosen so as the thickness variations of the coatings did not exceed 15%. The table surface was copper-made to ensure the uniform temperature distribution. The samples temperature was controlled with a thermocouple attached to the copper table. The distance between the table and the target was 150 mm; that ensured the uniform thicknesses of coatings on the samples.

The time of deposition was chosen so as the film thicknesses on the RF probe and on the planar objects were within defined parameters. The chamber was evacuated to a high vacuum by a diffusion pump with a nitrogen trap. The target and substrate were preliminary cleaned in glow discharge. The samples were heated in vacuum up to 330-350 °C. Than the titanium sublayer was deposited on the heated samples with the thickness up to 100 Å. The discharge parameters were the next: discharge current was 2.5 A, discharge voltage was 450 V, the chamber pressure was $1 \cdot 10^{-3}$ Torr, the working gas was argon, the deposition time was 30 s. Titanium covered samples cooled in a high vacuum down to the room temperature. After that nitrogen was inlet in the chamber up to air pressure. This prevented the oxidization of the surface. The copper target was mounted in the magnetron sputtering system. The copper deposition was done at the substrate temperature 300-350 °C. The samples were kept in a high vacuum at this temperature during 15-20 min and after that the discharge in the magnetron sputtering system was light up.

In the first 5÷10 min on all the stage of deposition there was a baffler between the sputtered target and work-in-process items. During this time the target surface was cleaned. Oxides and other contaminations sputtered from the target surface settled on the baffler and did not fall on the object surfaces. That heightened a purity of obtained coatings. Copper was deposited at the next discharge parameters: discharge current was 2.5 A, discharge voltage was 560 V, the chamber pressure was $1 \cdot 10^{-3}$ Torr. The copper film was deposited on reference samples for 30 min, the first 3 minutes of which deposition was done on hot objects, and then the heating was switched off. The reason of the heating switching

was a substantial sample temperature rising due to heat transmission from the magnetron sputtering system and deterioration of coating adhesion and structure in the result. When depositing the copper in the operation condition the objects temperature decreased from 330-350 °C to 180 °C. The obtained in such conditions copper film had tolerable surface roughness. Copper layer was deposited to a thickness of 3 ÷ 4 μm. Samples after the copper deposition cooled down in vacuum to the room temperature.

Fig. 4 shows an electron microscope image of surface of the copper film deposited on the object with the temperature during deposition higher 350 °C. Pores are clearly seen.

The important stage of copper coating deposition was also transient-state conditions from the beginning of the film formation at indirect heating to further the film building-up without this heating. In the experimental conditions of films under study formation if the time of deposition on samples with indirect heating was less than 3 minutes, the substrate cooled down under 150 °C. At this the film was overstressed and had more porous structure than at the deposition on the overheating substrate but consisting from separate columns of significantly smaller cross-section.

At forming the film with transient-state regime at more than 3 minutes there appeared some relief on it and it became lustreless. This is due to the increasing size of the columns that formed up the film.

Studying the structure of the coating in a scanning electron microscope revealed that the film has a densely packed fibrous structure. Fig. 5 shows an electron microscopy image of this copper. Between arrows there is a fracture of the object with a copper coating. It is seen that the lateral dimensions of columns are less than a micron. This is consistent with the results presented in [17]. After that titanium sub-layer was deposited on the copper coating on the technology described above, but without pre-heating of objects.

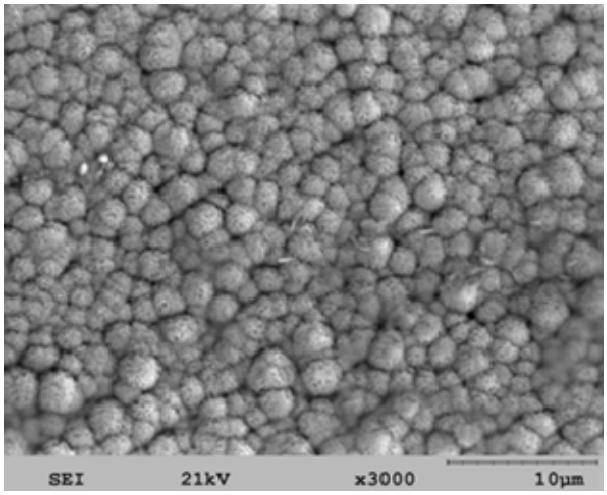

Fig. 4. Copper film on the overheating object.

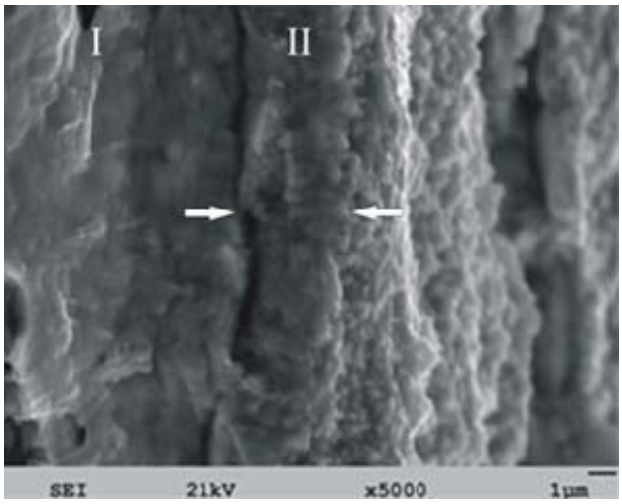

Fig. 5. Fracture of the object with copper coating deposited at the working operating mode

Tungsten was deposited on the hot samples. At that the temperature range 330-350 °C was strictly kept as only at such temperatures good adhesion of tungsten films to the surface of copper or stainless steel was achieved. The discharge parameters at the tungsten deposition were: discharge current was 2.5 A, discharge voltage was 520-560 V and the chamber pressure was $6\text{-}8\cdot10^{-3}$ Torr. Tungsten films of 2.2 μm thickness were created during 1 hour, and of 0.5 μm thickness – in 15 minutes. Samples cooled in a vacuum to the room temperature. The surface morphology of composite structures (stl.st + W) and (stl.st + Cu + W) is shown in Fig. 3. Roughness of the two-layer copper-tungsten coatings apparently was due to the restructuring of the copper sub-layer in the result of copper heating at the deposition of subsequent coating layers.

Along with the deposition of coatings on RF antenna probe for the study of the capture and retention of helium and hydrogen isotopes reference samples of 2 × 10 mm were prepared using the same technology. They were also used for electron microscopic studies. The substrate was stainless steel of 0.5 mm thickness. The samples of two types were created: 1 - stainless steel with Ti sub-layer of thickness up to 10 nm and 0,5 μm or 2.2 μm W layers (stl.st + W (nm, μm)) 2 - stainless steel with Ti sub-layer of thickness up to 10 nm, Cu layer of 3 μm thickness, Ti sub-layer of less than 10 nm thickness, the W layer of 0.5 μm or 2.2 μm thickness (stl.st + Cu + W (nm, μm)).

Tungsten coatings in composite structures (stl.st + Cu + W) had polycrystalline bcc structure with the average grain size near 20 nm in (stl.st + Cu + W (nm)) and 60 nm in (stl.st + Cu + W (μm)) (Fig. 6,7 and Fig. 8,9 respectively).

The tungsten coating was texturing in a little degree ($\Delta\psi > 30^{\circ}$), a texture axis was [110]. The texture axis was inclined towards the normal to the tungsten film to the angle $\Delta\rho \approx 4\text{–}14^{\circ}$. Tungsten coatings had a little tensile macrostress $\sigma = +0.64$ GPa in the composite structure (stl.st + Cu + W (2.2 μm)) and compressive macrostress $\sigma = -8.0$ GPa in the composite structure (stl.st + W (2.2 μm)).

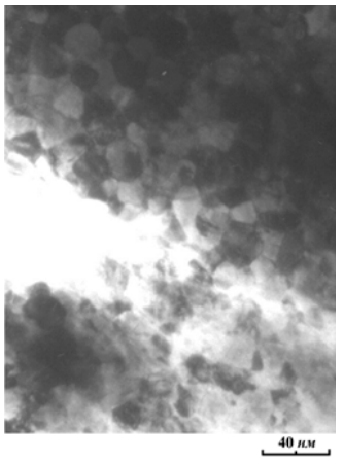

Fig. 6. Electron microscope image of W coating (stl.st + Cu + W (nm)) composition.

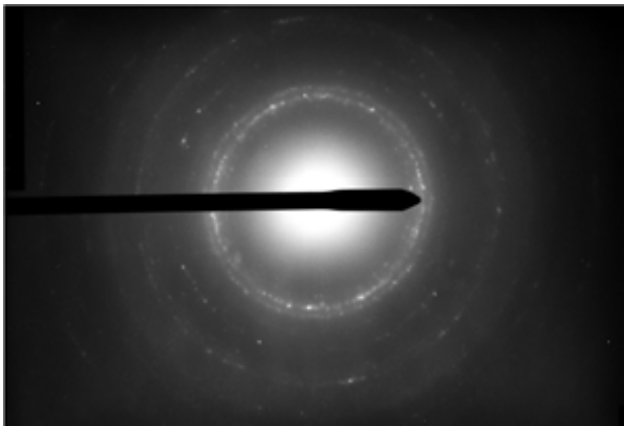

Fig. 7. Microdiffraction pattern of W coating (stl.st + Cu + W (nm)) composition.

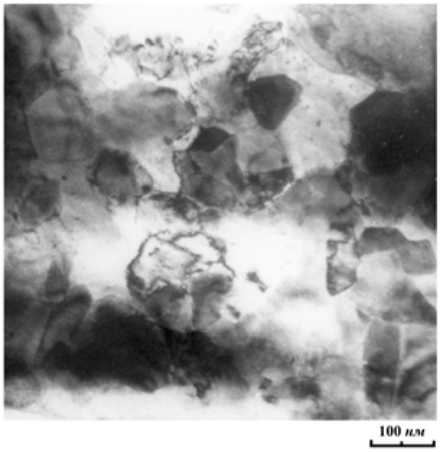

Fig. 8. Electron microscope image of W coating (stl.st + Cu + W (μm)).

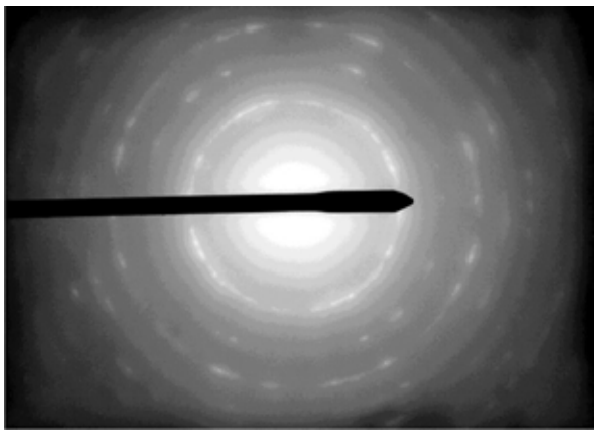

Fig. 9. Microdiffraction pattern of W coating (stl.st + Cu + W (μm)) composition.

The studies of tungsten coatings obtained with the magnetron sputtering system showed that in films there were residual stresses (see the Table). Internal stresses could be both compressive and tensile depending on the thickness of the tungsten film and elemental individuality of the substrate. In any case the presence of the copper sub-layer reduced internal compressive stress

Table.

Values and the type of internal stresses in the coatings

Cu layer thickness, μм	W layer thickness, μм	Internal stresses σ, GPa	The type of internal stresses
-	0.5	- 10.0	compressive
-	2.2	- 8.0	compressive
3	0.5	- 4.5	compressive
3	2.2	+ 0.6	tensile

The coatings in the form of copper sub-layer and working tungsten film coated by the arc sputtering device considerably differed from those formed with magnetron method. After deposition of the copper sub-layer with the thickness of several μm on stainless steel substrate by this technology the strong surface relief turned out so before deposition of the final tungsten layer tumbling of the object surface was needed. After tumbling cold deformation of the copper sub-layer was done. As a result at the following tungsten deposition there appeared high internal stresses that prevented forming final continuous coating. Annealing the object with the deposited copper layer in vacuum at 450°C, the following polish using a diamond paste of a small grain-size and thoroughly surface cleaning in solvents were the necessary operation to get good adhesion of tungsten films to the copper sub-layer.

CONCLUSION

The method of deposition of tungsten and two-layer copper-tungsten coatings on the objects of irregular shapes from stainless steel by magnetron sputtering was developed in the carried studies. It was established that the substrate temperature should be held in the range 330-350 °C to get optimal adhesion and allowable object surface finish class. To get the necessary properties the initial item should be expose to normalizing anneal in vacuum.

It was established that increasing of the coatings thickness leads to the decrease of internal stresses and, if the copper sub-layer is present, to changing of the stress type. The obtained coatings were polycrystalline but with the dominating W [110] plate outcome. Produced with the mentioned above technology coatings on RF probe agreed the requirements to mechanical strength, adhesion and surface roughness. On objects produced by this technology, studies of coatings resistance to the influence of the particle fluxes, the capture and retention of hydrogen isotopes were carried down.

Multilayer copper- and tungsten-based coatings were got with the arc sputtering system. At this the surface roughness was higher than at the magnetron sputtering system using. Functionality of such coatings needed further investigations.

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