APPLICATIONS AND TECHNOLOGIES

SOURCE OF DROPS-FREE PLASMA FLOWS OF MONOCRISTALINE ZIRCONIUM

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In experiments, the characteristics of a non-self-sustained arc discharge in pure vapors of monocrystalline zirconium were investigated. The minimum ignition power of the discharge in zirconium vapor was determined for experimental conditions. Also the main characteristics of the generated plasma streams were measured. The growth rates of zirconium films have been determined for various modes of the discharge operation. It is proved that the plasma stream generated by the arc discharge in zirconium vapor has a compensated volume charge and can be used to created metal films and coatings on the substrates of different materials. The coefficients of plasma streams ionization are determined and the possibility of their controlled change is shown. This capability can be used for the targeted control of the properties of the films and coatings.

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INTRODUCTION

Zirconium is of interest not only as constructional material used in nuclear power engineering, but also as a material with high acid-resistant and biocompatible properties. In atomic energetics, it is used to create fuel claddings and as a protective coating for nuclear fuel [1]. In materials science, this material serves as the basis for the creation of multi-component coatings and alloys. Zirconium is also widely used in the chemical industry, including the creation of protective coatings for parts and constructions working in hydrogen-containing environments. Therefore, the development of sources of drop-free and highly ionized plasma flows of zirconium vapor is an important and urgent task.

EXPERIMENTAL SETUP

The basic scheme of the experimental device is shown in Fig. 1. The non-self-sustained arc discharge was ignited between the anode 3, that is water cooled, and the grounded cathode 1 in the vapors of the working material 2. The working material in the experiments described was zirconium. It was placed in the crucible 8 that was located directly on the upper surface of the anode 3. The cathode of discharge was made of tungsten wire with a diameter of 1 mm and had the form of a two-circle ring with 1.2 cm in diameter. The distance between the discharge cathode and the working material located in the crucible on the upper surface of the discharge anode was 3…5 mm. The heating current of the cathode was 60 A. Magnetic field was created in the discharge gap by means of the magnetic coil 5 and was used to facilitate the conditions of the discharge ignition and its burning. In the experiments carried out, the induction of the magnetic field, B, in the zone of the discharge gap was $B = 80\times10^{-4}$ T and corresponded to the condition of the maximum current of the ions on the electrode 7, at the constant value of discharge current. The electrode 7 was used not only for measuring the ion current in the plasma stream, but also for placing substrates on it. Usually its potential was negative and was -200 V. Metal cylinder 4 excluded the possibility of a plasma flow directly on the magnetic field coil 5 and in most experiments had the potential of a cathode. It was also used as an anode for additional discharge in crossed magnetic and electric fields created by coil 5 and cylinder 4, and in these modes it had a positive potential with respect to the potential of the discharge cathode. The additional discharge was formed between the anode, electrode 4, and the cathode of discharge 1. The discharge could burn in the gas, in pure zirconium vapor or in zirconium vapor with a gas.

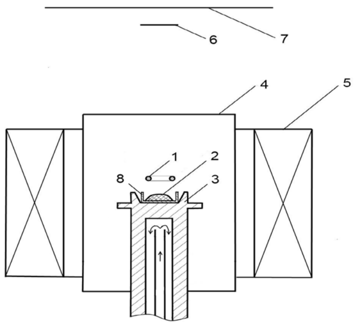

Fig. 1. Scheme of experimental device:
1 – heated cathode; 2 – working material; 3 – cooled anode; 4 – cylindrical electrode; 5 – magnetic coil; 6 – flat Langmuir probe; 7 – ion collector – substrate holder; 8 – crucible

Flat electrical probe 6 was used to measure the parameters of the plasma flow. The probe was located on the axis of the system at a distance of 0.17…0.19 m from the anode, and it usually had a negative potential relative to the discharge cathode, U6 = -200 V. The boundary pressure in the vacuum chamber was $p = (1\ldots2)\times10^{-3}$ Pa. In the mode of deposition of films, the pressure in the vacuum chamber was close to the boundary pressure and practically did not exceed $p = 2\times10^{-3}$ Pa.

In experiments, the current of an electron emission from a heated cathode exceeded the total current of the main discharge in metal vapor. The direct placement of zirconium on the surface of the cooled anode 3 required powerful power supplies. This caused by the thermophysical characteristics of this material. Therefore, zirconium was placed on the anode in a crucible.

The method of "gas ignition" of non-self-sustained arc discharge in vapors of anode material was proposed earlier [2, 3]. It was used in our experiments to ignite a vacuum arc discharge in zirconium vapor. This method involves the use of an additional gas discharge in crossed electric and magnetic fields. The ignition of vacuum arc discharge in metal vapors occurred in a few stage. In the first stage after inlet of the argon into the vacuum chamber the gas discharge, or additional discharge in the gas, was ignited between the electrode 4 (additional anode) and the heated cathode. Then a direct ignition of the main discharge between the anode 3 and the cathode 1 was carried out. The voltage applied between then was resulted in the occurrence of a current between these electrodes, the heating of the working material 2 and to the appearance in the gap between the cathode 1 and the anode 3 of the vapor of the zirconium. The increase in voltage was accompanied by an increase in the discharge current and the increase in the vapor pressure of zirconium in the discharge gap and to the ignition of the arc discharge in the mixture of metal vapors and working gas. Subsequently, the discharge in the mixture of metal vapors and gas was transferred to the arc discharge mode in vapors of pure metal under pressure in a vacuum chamber p = 2×10^{-3} Pa.

EXPERIMENTAL RESULTS

A typical volt-ampere characteristic of a non-self-sustained arc discharge in pure zirconium vapors is given in Fig. 2. It is obtained with a pressure in the vacuum chamber p = 1.4×10^{-3} Pa.

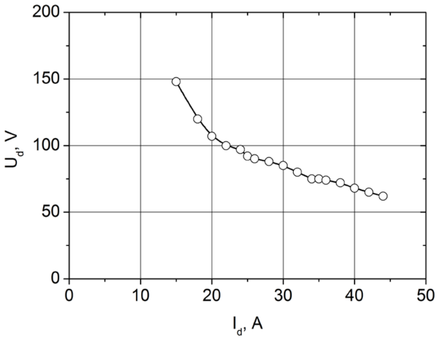

Fig. 2. Volt-ampere characteristics of non-self-sustained arc discharge in vapors of zirconium

The voltage between the anode and the cathode of the main discharge in zirconium vapors decreases with increasing discharge current. At the discharge current $I_d = 15$A the discharge voltage in zirconium vapors is about $U_d = 150$ V, and at discharge currents $I_d = 20$ A and $I_d = 40$ A the discharge voltage is respectively 100 V and 70 V. When the discharge current is lower than $I_d \leq 14…15$ A the discharge does not exist. That is, at such discharge currents, the vapors pressure of zirconium in the discharge gap is insufficient for ignition and stable burning of the discharge. In these conditions, the reduction of vapor pressure of zirconium leads to a decrease in the discharge current and the disappearance of the discharge.

The dependence of the discharge power on the discharge current is shown in Fig. 3.

This data show that in the described experiments, the minimum burning power of discharge in zirconium vapor was approximately $W_d \approx 2.2$ kW. Evaporation of zirconium, sufficient for the realization of a stable discharge, has a place when the discharge power $W_d \approx 2.2…3$ kW at currents $I_d = 20…45$ A.

The given data show that when the discharge current is changed from 15 to 40 A, the value of the ion current increases by 5 times, changing from 20 to 100 mA. Linear growth of the ion current occurs at a significant interval of change in discharge current. However, with currents of more than 35 A, the tendency of nonlinear dependence of the ion current on the discharge current takes place.

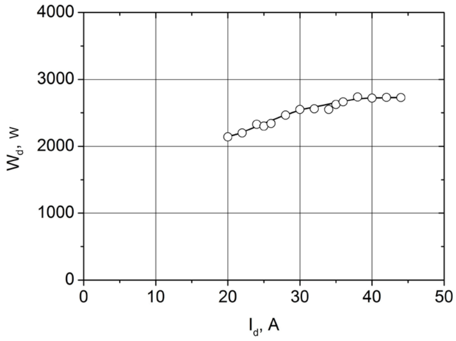

Fig. 3. Dependence of discharge power $W_d = I_d \cdot U_d$, on the discharge current I_d

The ion current in the plasma flow at the various current of discharge is an important characteristic of it. In Fig. 4 shows the dependence of the ion current on the electrode 7 from the value of the discharge current. Usually, the electrode 7 in experiments had a size of 0.08×0.08 m and a negative potential of -200 V relative to the grounded cathode of discharge.

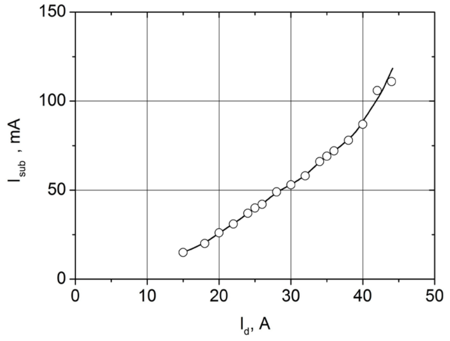

Fig. 4. Dependence of ion current on electrode 7, I_{sub}, on the current of discharge, I_d, in vapor of zirconium

In Fig. 5 shows the results of investigations of the values of the potential of the isolated electric probe 6.

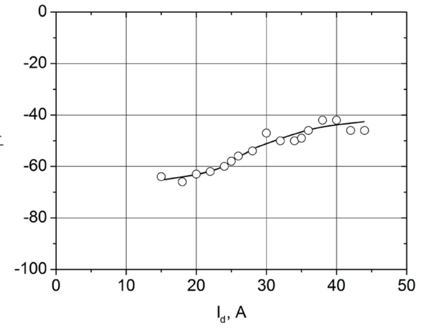

Fig. 5. Dependence of the floating potential of the isolated probe, V_f, on the discharge current in zirconium vapor

The probe was located at the center of the plasma flow at a distance of 0.17…0.19 m from the discharge anode. The probe is directly connected to a divider with a total resistance of 300 MΩ. The measurement of the potential was carried out from a part of the divider with a resistance of 1 MΩ using a digital voltmeter B7-35. Static voltmeter C 50 was used for monitoring.

The obtained data indicate that the potential of an isolated electric probe, placed in the center of the plasma flow non-self-sustained arc discharge in the zirconium vapor, is negative and makes $V_f = -(60…40)$ V. Potential of the probe varies from $V_f \approx -60$ V to $V_f \approx -40$ V at a change in discharge current from $I_d = 20$ to $I_d = 40$ A. These data indicate that the surface of an isolated substrate of any material will have a negative potential relative to the potential of the discharge cathode when substrate is placed in such a plasma flow. It is this potential that, together with the potential of the plasma of the main discharge, will determine the energy of the ions arriving at the deposition surface. The ions with the energy of more than 100 eV can create undesirable defects, and ions with energies up to 100 eV can be used to form the necessary properties of the created coatings. The dependence of the growth rate of a zirconium film, q, on the discharge current, I_d, is shown in Fig. 6.

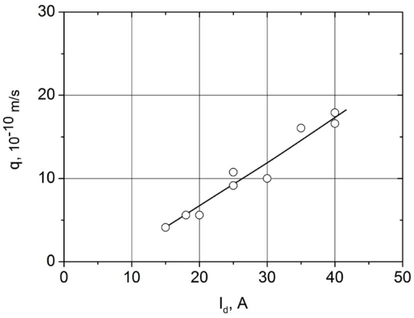

Fig. 6. Rate of films deposition, q, on the discharge current, I_d, for zirconium as a working material

The main characteristics of plasma sources are the rate of growth of deposition films and their adhesion, which depends on the ion energy in the plasma stream used. In our experiments the film was deposited on a dielectric substrate in a vacuum $p = (1.2…2.0)\times10^{-3}$ Pa. The data show that in the range of main discharge currents up to 40 A film deposition rate q has a linear dependence on the discharge current and is $q = (5…15)\times10^{-10}$ m/s or q = 1.8…6.5 mkm/h.

In Fig. 7 data on the dependence of the ionization coefficient of the plasma flow from the discharge current in zirconium vapor are given.

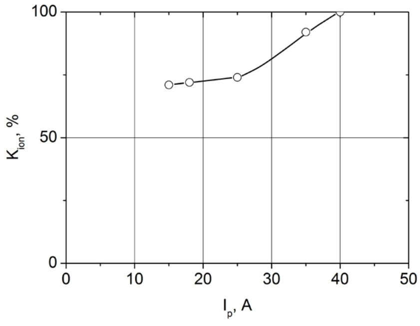

Fig. 7. Ionization coefficient of plasma flow, K_{ion}, on the current of main discharge, I_d

The coefficients of ionization of the plasma flow were calculated taking into account the process of self-sputtering of the deposited film [4]. It is evident that the non-self-sustained arc discharge in the anode material vapors really creates a highly ionized plasma flow. In this case, the coefficients of ionization of created streams were fixed in experiments more than 70%.

As noted earlier, an additional discharge between the cylinder 4 and the cathode of discharge 1 in zirconium vapor is able to change the parameters of the created plasma flow. In particular, it leads to additional ionization of the plasma flow. The effect of the additional discharge current in zirconium vapor on the ionization coefficient of the plasma flow is shown in Fig. 8. According these data additional discharge with currents up to 5 A, at main discharge current $I_d = 25$ A is capable of increasing the ionization coefficient of the plasma flows by a factor of 1.4.

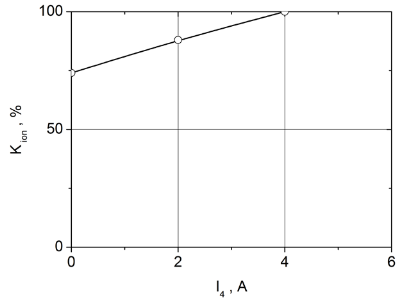

Fig. 8. Ionization coefficient of plasma flow, K_{ion}, and rate of Zr film deposition, q, vs current of additional discharge on electrode 4, I_4

At the same time, an additional discharge practically does not change the intensity of the plasma flow, or the amount of mass transfer stream. This data show that the additional discharge can be used to significantly increase K_{ion} and consequently it can be used for targeted influence on the formation of the structure and other properties of deposited films.

CONCLUSIONS

The presented results show that the source of the plasma streams of monocristallike-zirconium vapor on a basis of the non-self-sustaining arc discharge in the anode material vapors allows creating plasma flows with an ionization coefficient of more than 70%. The generated plasma flows allow obtaining films and coatings of metallic zirconium on various materials with growth rates of up to 6 μm per hour.

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ИСТОЧНИК БЕЗКАПЕЛЬНЫХ ПОТОКОВ ПЛАЗМЫ МОНОКРИСТАЛЛИЧЕСКОГО ЦИРКОНИЯ

А.Г. Борисенко, Е.Г. Костин, О.А. Рокицкий, О.А. Федорович

В экспериментах были исследованы характеристики несамостоятельного дугового разряда в чистых парах монокристаллического циркония. Определена минимальная мощность зажигания разряда в парах циркония. Измерены основные характеристики генерируемых плазменных потоков. Определены скорости роста осаждаемых пленок циркония в различных рабочих режимах разряда. Доказано, что создаваемый дуговым разрядом в парах циркония плазменный поток имеет компенсированный объемный заряд и может быть использован для нанесения металлических пленок и покрытий из циркония на подложке из разных материалов. Определены коэффициенты ионизации плазменных потоков и показана возможность их управляемого изменения. Эта возможность может быть использована для целенаправленного управления свойствами осаждаемых пленок и покрытий.

ДЖЕРЕЛО БЕЗКРАПЕЛЬНИХ ПОТОКІВ ПЛАЗМИ МОНОКРИСТАЛІЧНОГО ЦИРКОНІЮ

А.Г. Борисенко, Є.Г. Костін, О.А. Рокицький, О.А. Федорович

Експериментально були досліджені характеристики несамостійного дугового розряду в чистих парах монокристалічного цирконію. Визначена мінімальна потужність запалювання розряду в парах цирконію. Досліджені основні характеристики генерованих плазмових потоків. Визначені швидкості росту осаджуваних плівок цирконію в різних робочих режимах розряду. Доведено, що створюваний дуговим розрядом у парах цирконію плазмовий потік має компенсований об'ємний заряд і може бути використаний для нанесення металевих плівок і покриттів з цирконію на підкладинки з різних матеріалів. Визначено коефіцієнти іонізації плазмових потоків і показана можливість їх керованої зміни. Ця можливість може бути використана для цілеспрямованого управління властивостями осаджуваних плівок і покриттів.