OPTOELECTRONICS AND ADVANCED MATERIALS – RAPID COMMUNICATIONS Vol. 3, No. 11, November 2009, p. 1231 - 1234

Arc plasma tailoring for the synthesis of compact Tungsten films

M. BADULESCU, I. GRUIA[a*], C. SURDU-BOB, C. IACOB
National Institute for Laser, Plasma and Radiation Physics, Romania
[a]*Faculty of Physics, University of Bucharest, Romania*

In this paper, a study for the correlation of electrical parameters of the Thermo ionic Vacuum Arc (TVA) ignited in Tungsten vapours with ion energy distributions is presented. The plasma source used in this study is a PVD technique used for thin film deposition that works under high vacuum (10^{-6}torr), high voltage (500÷2500 V) and currents of 1÷3 A. This plasma source is confined into a small volume, it does not fill the vacuum chamber. The samples to be deposited are placed a few tens of centimetres away from the core of the plasma. The main assets of the source are reflected in the properties of the films that can be deposited: high compactness, adherence, smoothness and purity. Moreover, the fact that thin films of refractory metals can also be deposited, makes TVA a promising deposition tool for industry, including the high temperature applications. In order to come closer to applications and technological control, the type, density and energy of the impinging particles at the substrate need to be analyzed. An in-house, computer-controlled RFA analyzer was used for determination of ion energy distributions at 35 cm away from the TVA plasma ignited in Tungsten vapours. Compositional and topographical analyses of the tungsten films were also undertaken. EDX analyses have shown high purity of the W films. Two Tungsten oxides, WO_2 and WO_3 were found by XPS. No macro particles are formed during deposition, as observed in the SEM images.

(Received October 19, 2009; accepted October 29, 2009)

Keywords: TVA plasma, Arc plasma, W, WO_2, WO_3 films

1. Introduction

Tungsten is a refractory metal with the highest melting point of all metals, 3422 °C. This fact makes W suitable for aerospace, turbine blades and high temperature uses like fusion reactors. In the diverter region of a fusion reactor, high particle fluxes of low energies would severely degrade the lifetime of plasma facing components made of low-Z materials. Thus, high-Z materials, in particular tungsten, are considered as a favourable option, primarily due to their low sputtering yield and their high sputtering threshold energy.

Plasma-enhanced chemical vapour deposition (PECVD) using WF_6 as supply gas as well as physical vapour deposition methods like vacuum arcs are being used to coat with Tungsten the plasma facing components. The main disadvantage of the PECVD systems is the inclusion of process gas molecules/atoms in the W film causing internal stress and delamination. A variety of sputtering techniques (magnetron, unbalanced magnetron, ionized sputtering, pulsed sputtering, ion beam sputtering, etc.) have also been developed for the deposition of metallic films. These deposition methods provide low energy ions of only a few eV and therefore high film adherence is obtained if additional substrate heating or bias is used. Other methods like vacuum arcs provide a straightforward method for the efficient production of dense and pure metal plasma. The drawback in such systems is that the Cathodic arc processes also produce unwanted droplets and debris particles in the mm and sub-mm range on the film, commonly referred to as macro particles. For some applications, such as reactive deposition of decorative and protective coatings, macro particles can be tolerated. For high-tech applications, macro particles must be removed. While filtering is needed in Cathodic arcs [1], the original anodic arc developed at the Low Temperature Plasma Laboratory of the National Institute for Lasers, Plasma and Radiation Physics – Bucharest provides high quality smooth films without filtering due to the low arc currents used (0.5 to 2A). The original method named Thermo ionic Vacuum Arc (TVA) is an anodic arc with an external electron source which allows gentle evaporation of the anode material.

The current paper presents a study for the correlation of electrical parameters of the Tungsten TVA plasma with ion energy distributions.

The results show that the energy and density of the impinging ions at the substrate can be easily tailored via operating parameters, this giving the possibility for tuning film properties like stress, adhesion, surface roughness, hardness, elastic modulus etc.

2. Experimental setup

The experiments were carried out in a stainless steel vacuum chamber. The Thermo ionic Vacuum Arc (TVA) ensemble consists of a Tungsten anode and a grounded electron source – the cathode [2]. The electron source is

made of a loop filament of W wire externally heated with electrical currents of 40 to 60 A (see Fig. 1).

For metals with low vapour pressure (less than 10^{-1}torr) at their melting temperature, ignition of the TVA plasma can only be obtained if the metal is melted in a crucible. Details on the TVA plasma are given in ref [2]. For the other metals with high melting points like Tungsten, the anode is a rod which is evaporated from the top, like a candle; no crucible is needed. Tungsten rods are only available as sintered material. As such materials contain high amounts of impurities with different vapour pressures at a certain temperature, unwanted sparks appear and ignition of the TVA plasma is not possible using such an anode. A specially designed anode allowed ignition of very stable plasma in W vapours. The W anode consisted of a bunch of thin W wire (0.2 mm) which allowed thorough melting of the material upon electron heating, as can be seen in Fig. 1.

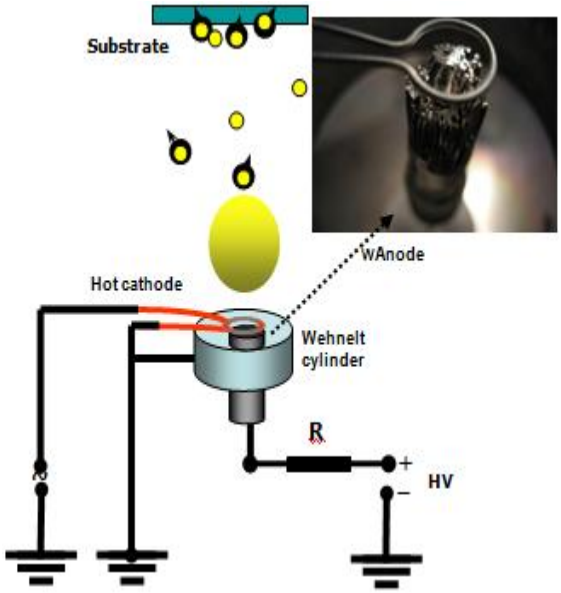

Fig. 1. Experimental setup of the Thermo ionic Vacuum Arc plasma. Inser t image: anode made of Tungsten wires.

As the TVA plasma is localized at the anode and does not fill the vacuum chamber, ions and neutrals escape from the plasma and travel without collisions (negligible phenomenon) towards the chamber walls. The ions escape from the plasma due to the potential difference between the plasma and the grounded chamber walls. Thus, the RFA is not immersed into the core of the TVA plasma and charged particles travel in straight lines.

The retarding field analyzer (RFA) is an electrical probe capable of providing ion energy distributions in plasmas. The RFA used in this study had a typical construction containing a collector and a single grid encapsulated in a stainless steel cylinder provided with a 3 mm diameter entrance slit. As the TVA plasma does not fill the chamber (it is localized) and the RFA is placed outside the plasma volume, a simpler construction with only one grid for repelling the electrons was used. The grid was negatively biased with constant voltages up to 450 V. The bias at the collector was swiped between -1000 and +1000 V. The electrical circuit is presented in Fig. 2. The RFA was placed 35 cm distance from the anode. I-V characteristics of the RFA collector and also of the W arc plasma were acquired automatically using an in-house computer-controlled system. Stainless steel and PK-7 glass substrates were placed at 35 cm from the anode, also. Compositional and topographical analyses on the W films deposited on these substrates were undertaken.

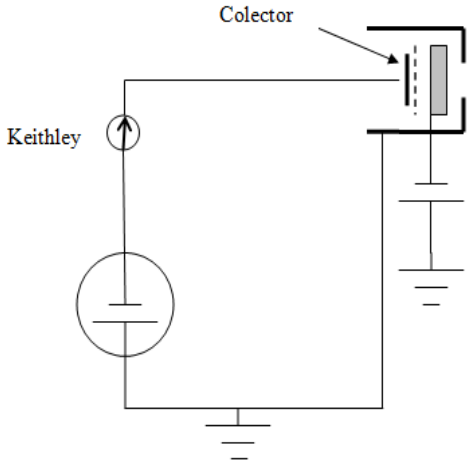

Fig. 2. RFA electron/ion energy and density analyzer – schematic representation.

3. Results and discussion

The main operating parameters of the TVA plasma are: filament current (I_f), interelectrodic distance (d_i) and applied voltage.

Parametric studies revealed that with increasing interelectrodic distance, a higher applied voltage is necessary for plasma ignition, as can be seen in Fig. 3.

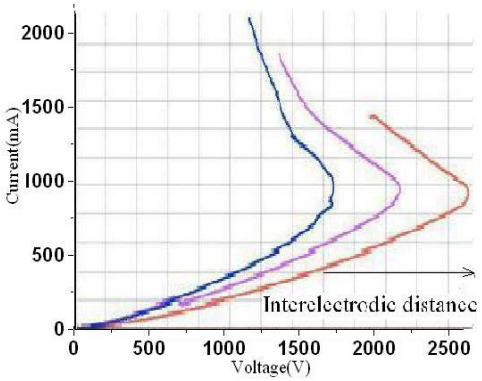

Fig. 3. I-V characteristics for different interelectrodic distances, If = 60 A.

Also, ignition of the plasma using higher filament currents resulted in a displacement of the I-V characteristics to higher values of the arc current (see Fig. 4).

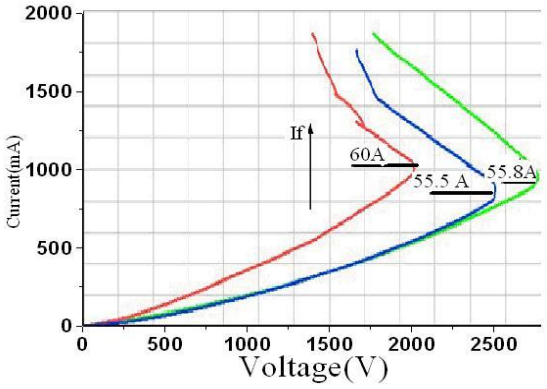

Fig. 4. I-V characteristics of the arc for different filament currents.

The deposition rate was measured in-situ using a Cressington gage and values between 0.8 to 2.3 Angstroms per second were found.

A typical current–voltage characteristic at the collector is presented in Fig. 5. The negative current at the collector suggests a strong presence of electrons. The electronic contribution to the collector current revealed a strong correlation of the electron energy and density distribution with plasma operating parameters. It was found that, while the maximum of the electronic distribution moved towards higher energy with increasing anode voltage, the peak height decreased (see Fig. 6).

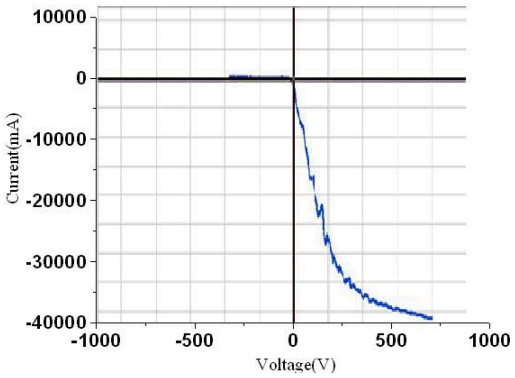

Fig. 5. Typical I-V characteristic at the RFA collector. A high electron current is observed.

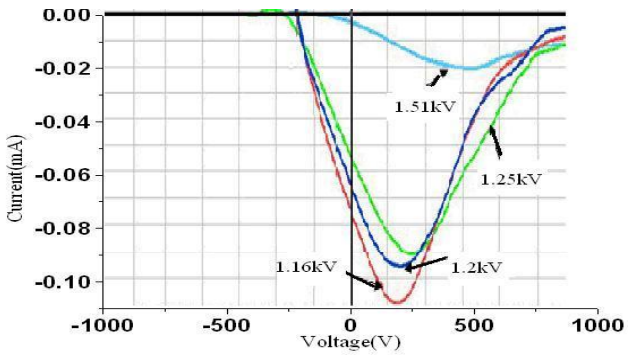

Fig. 6. First derivative of the I-V characteristic at the RFA collector for different plasmas (different voltages applied on the anode) at I_f = 60 A.

This result is in perfect agreement with the I-V characteristic of the TVA plasma (the higher current stage) where a higher number of electrons are formed in the plasma with increasing applied voltage. Thus, the plasma becomes more and more conductive and therefore the arc voltage and plasma potential decreases. As the ions escaping the plasma travel in vacuum, they only ”see” the potential difference between the plasma potential and the substrate potential (which is usually grounded) the ion energy is given by the plasma potential. This correlation between plasma potential and electron current at the RFA collector is important, as plasma tailoring can be obtained by parametric control.

Compositional analysis of the films revealed high purity and also formation of two Tungsten oxides, WO_2 (32.4, 34.4) and WO_3 (36.1, 38.1), as can be observed in Fig. 7. These oxides form a very thin layer of native oxides which appeared due to exposure to the ambient air.

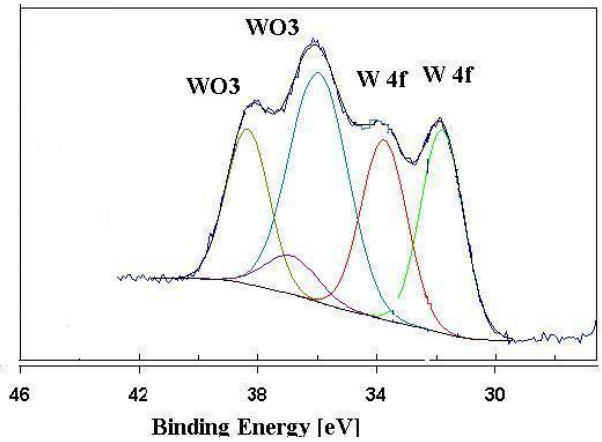

Fig. 7. XPS spectrum of a typical W film deposited by TVA.

EDX analysis showed that perfectly pure W coatings can be obtained using the TVA method, in contrast with other known techniques where nickel and iron impurities coming from the steel used in the deposition equipment were found [4]. A typical EDX result is presented in Fig. 8.

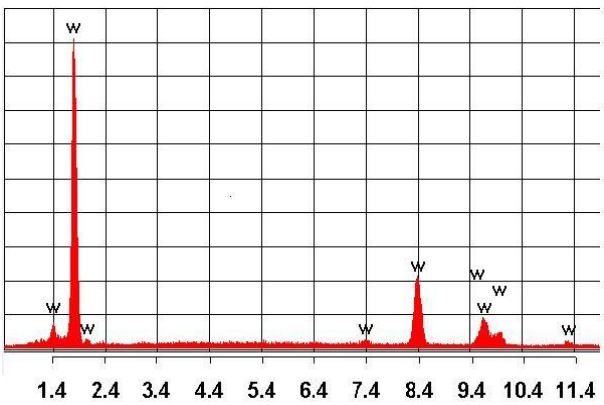

Fig. 8. EDX analysis of a typical W film deposited by TVA.

SEM analysis showed that no macro particles are formed on the films deposited by TVA, in contrast to the classical arc techniques (see Fig. 9).

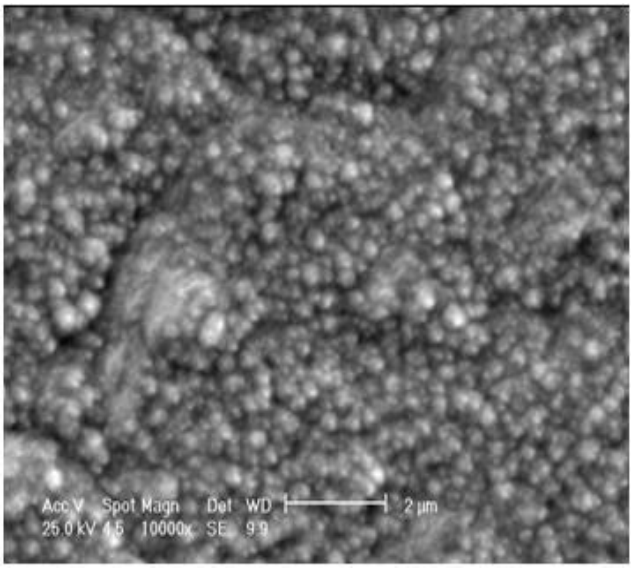

Fig. 9. SEM image of a typical W film deposited by TVA.

4. Conclusions

It was shown here that the TVA method is a reliable original method that provides pure and dense W films.

The tailored plasma parameters of the TVA allow selectivity in the surface process through the control of the electron/ion energy and flux. The strong correlation of the ion energy with plasma operating parameters via applied voltage can be used for plasma tailoring.

Acknowledgements

The authors wish to thank the Ministry of Education, Research and Youth (MECT) – Bucharest Romania for financial support.

References

[1] A. Anders, Vacuum **67**, 673 (2002).
[2] C. Surdu-Bob, I. Mustata, C. Iacob, J. Optoelectron. Adv. Mater. **9(**9), 2932 (2007).
[3] C. Bohm, J. Perrin, Rev. Sci. Instrum. **64**(1), 1993.
[4] S. Lehto et al., Fusion Engineering and Design **66-68** 241 (2003)

*Corresponding author: gruia_ion@yahoo.com