JOURNAL OF OPTOELECTRONICS AND ADVANCED MATERIALS Vol. 8, No. 1, February 2006, p. 71 - 73

Thermoionic vacuum arc (TVA) deposited tungsten thin film characterization

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In this paper, we present the characterization of the tungsten thin films deposited by Thermo-ionic Vacuum Arc (TVA) method. Characterization of the obtained tungsten thin films has been made by Transmission Electron Microscope (TEM) with a magnification of 1.4 M and a resolution of 1.4 Å. Other techniques were used as Grain Size Distribution, Selected Area Diffraction (SAED), Fast Fourier Transmission (FFT). The obtained films were characterized by nano-indentation and atomic force microscopy (AFM). The AFM measurements have proved the smoothness of the deposited films (however with some droplets) with peak to valley roughness in the range of 20-30 nm. As regards tribological results, the hardness of deposited films was measured by a Karl Zeiss microhardner tester and the coefficient of friction was measured with an Amsler tribometer. The samples (graphite substrates 30 mm x 30 mm x 8 mm coated with W) were tested using depth sensing indentation tester Fischerscope H100 Xyp. We can report that the tungsten film had significantly higher resistance against indentation.

(Received October 14, 2005; accepted January 26, 2006)

Keywords: Tungsten thin films, Thermo-ionic vacuum arc, AFM, HRTEM, Nano-intendation

1. Introduction

Tungsten, with the highest melting point and lowest vapor pressure of all metals, has – at temperature over 1923 K - the highest tensile strength. It has excellent corrosion resistance and is attacked only by most mineral acids. Despite of the highest melting point and lowest vapor pressure of all metals, tungsten thin film can be obtained with the Thermo-ionic Vacuum Arc (TVA) method. This type of arc ignites in high vacuum conditions in the vapors of the anode material, continuously generated by the electron bombardment of the anode [1-4]]. The electrons, emitted from a heated tungsten cathode, are accelerated towards the anode, by a d.c. high voltage applied across the electrodes.

2. Experimental set-up

The TVA method [1-8] is characterized by producing plasma in the pure vapors of the metal to be deposited (W) without using any buffer gas. The evaporation of the metal takes place in high vacuum conditions (10-3 Pa and less). An external heated cathode (W + 0.2%Th filament) produces thermally emitted electrons of about 100 mA. These electrons are accelerated and focused by a Whenelt cylinder to the anode which is biased to high voltage (1 – 6 kV). The electron bombardment creates space tungsten atoms above the anode at a local pressure of about133 Pa.

The thermo-electrons produced by the heated cathode are able to build up plasma by electron-tungsten atoms collisions. The new electrons generated in the plasma together with the original ones emitted by cathode enhance once more the anode evaporation and produce high quantity of ions. Usually the cathode potential fall is in the range of 200 - 300 V and, therefore, the plasma potential in comparison with ground ensure generation of the high energy ions which collide the substrate.

Because this system can heat any material at elevated temperature, it is one of the most adequate technologies of high melting point materials. In the case of such materials, instead of a crucible containing the material to be evaporated, a rod of refractory metals or carbon is used directly as anode. Moreover, the discharge can be ignited in high vacuum condition, ensuring high purity of the deposition.

In Fig. 1 is shown the experimental setup used for the tungsten deposition. Such an arrangement has been used for the deposition of Re or Nb [3].

The anode, a W rod of 8 - 10 mm in diameter and 60 mm in length was sustained by a Mo support, which could be rotated during deposition by an electrical motor.

The ignited thermo-ionic vacuum arc parameters were: cathode filament current - 150 A, arc current - 2 A, the arc voltage drop on the arc - 1000 V d.c.

The AFM measurements have proved the smoothness of the deposition, which have a roughness in the range of 5 nm.

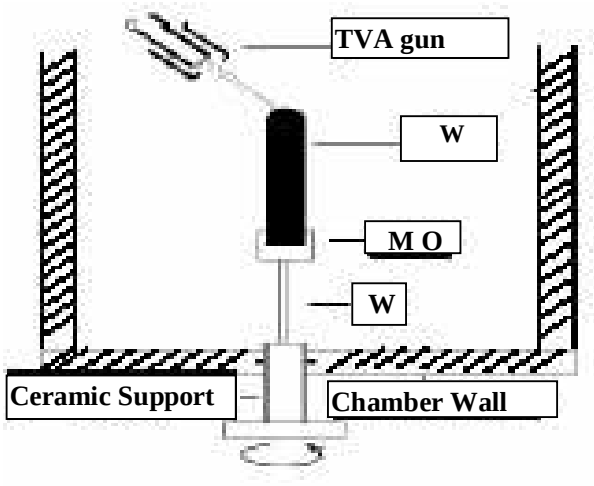

Fig. 1. Experimental set-up for tungsten deposition.

3. Results and discussion

The samples (graphite substrates 30 mm × 30 mm × 8 mm coated with W) were tested using depth sensing indentation tester Fischerscope H100 Xyp. We can notice from the Fig. 2 that the tungsten film had significantly higher resistance against indentation.

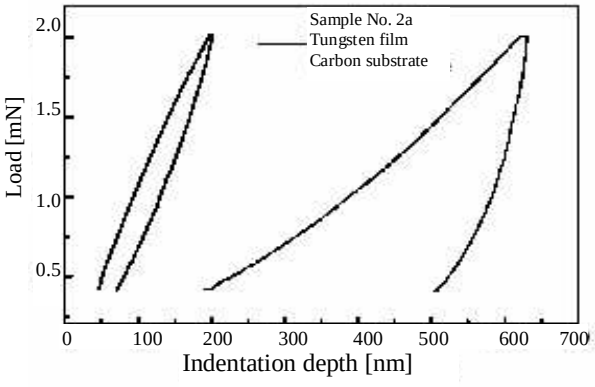

Fig. 2. Comparison of loading/unloading curves obtained for sample 2a and the carbon substrate for maximum intendation load of 2 mN.

The material parameters obtained on the graphite substrate and the tungsten films are listed below in Table 1.

Table 1. The material parameters got on the graphite substrate.

Sample	HU[N/mm^2]	W_e/W_{tot}[%]	HU_{pl}[N/mm^2]	H_{max}[μm]	T[Gpa]
substrate	158	24,81	205	0.692	6,0
2a	30	0.23	5700	0.250	80,0

where: - HU – universal hardness (resistance against elastic and plastic deformation)

- W_e/W_{tot} – ratio of the elastic indentation work to the total indentation work

- HU_{pl} – plastic hardness (resistance against plastic deformation – equivalent of the so called Vickers hardness

- h_{max} – maximum depth at given maximum load

- $Y = E/(1-v^2)$, where E is the Young's modulus and v is the Poisson's ration

The AFM measurements have proved the smoothness of the deposited films (however with some droplets as can be seen in Fig. 3) with peak to valley roughness in the range of 20-30 nm.

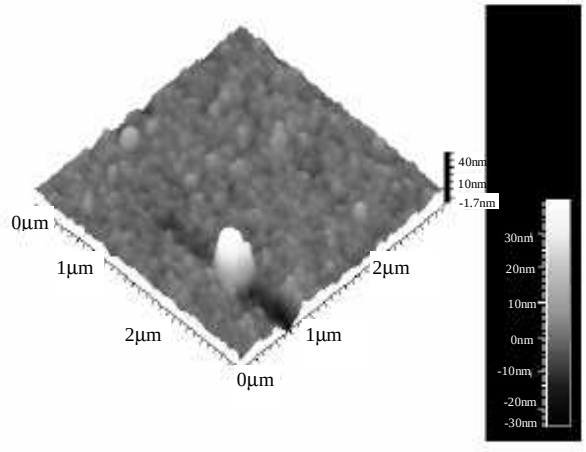

Fig. 3. The AFM image of the tungsten film deposited by TVA deposition.

The structure of the deposited W films were studied using TEM electronic microscopy with a magnification of 1.4 M and a resolution of 1.4 Å. The samples of tungsten films deposited on small size NaCl or KCl single crystals have been submitted to TEM examination (after solving the single crystal supports in water). TEM analysis of thin layers (10-20 nm thickness) revealed the nanostructured tungsten film with grain size in the range of 10 nm (Fig. 4 and Fig. 5).

Also in Fig. 5 one can see clustered tungsten nanoparticles with mean diameters bellow 10 nm.

Fig. 6 shows HRTEM image of W nanoparticles that exhibits (110) planes. Left inset shows FFT (Fast Fourier Transmission) representation of selected zone. SAED (Selected Area Electron Diffraction) image confirms the cubic structure of W. (SG: Im3m, a = 3.158 nm).

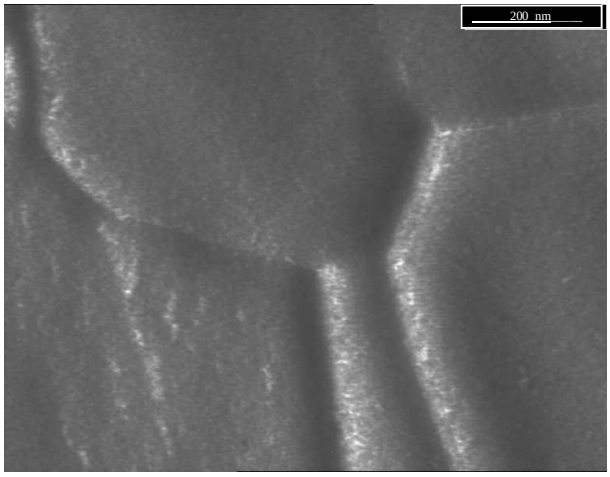

Fig. 4. BF-TEM image of W film.

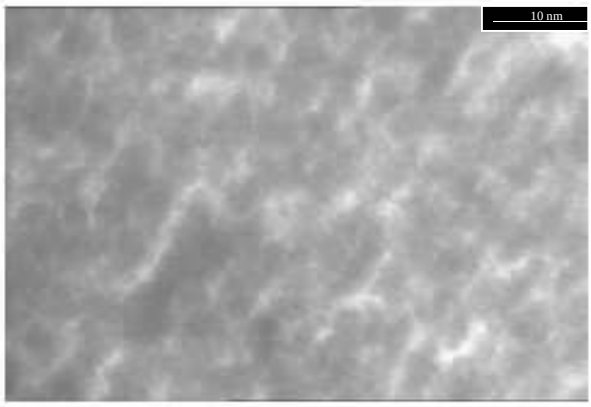

Fig. 5. Detail image of W film at 10 nm scale.

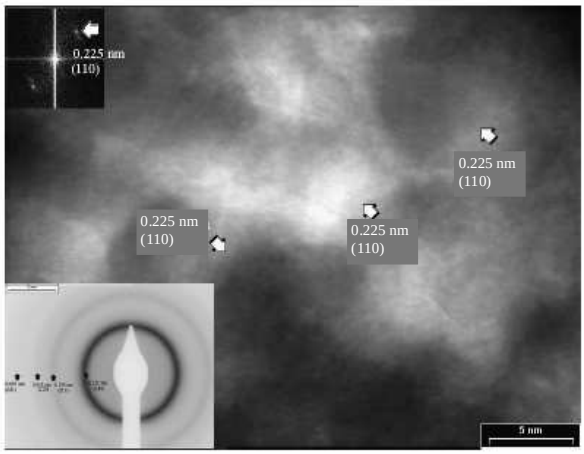

Fig. 6. HRTEM image of the deposited thin tungsten film with FFT (left up inset image) and SAED (left inset down image).

4. Conclusion

The obtained results prove the possibility to use TVA for high quality, pure tungsten film deposition with nanohardness in the range of 5700 N/mm^2 and peak to valley roughness in the range of 20-30 nm. Tungsten film deposition is now considered in fusion programs to be used to cover the wall of the divertors and also as thermal barrier coating on the Nb based superalloys for gas turbine blades.

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