

TRIBOLOGICAL PROPERTIES OF TUNGSTEN THIN FILMS DEPOSITED BY TVA ON SILICON SAMPLES AND ON GLASS SUBSTRATES

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Abstract:

This paper is focused on the investigation of mechanical properties of tungsten thin film produced by Thermionic Vacuum Arc. The indentation tests were performed using the Fischerscope H100 DSI tester equipped with Vickers indenter. This tester enables to register the indentation depth as a function of the applied load during both the loading and unloading part of the indentation test. The applied load L ranges from 0.4mN to 1N and the accuracy of the depth measurement is of about ± 1 nm. The elastic modulus obtained on glass samples using depth sensing indentation test was lower, than that of for bulk tungsten. The obtained values are significantly influenced with the substrate hardness. Tungsten film W was deposited on silicon substrate also. This film did not delaminate. The hardness obtained on this film at low indentation depth was a little bit lower, than in case of films on glass substrate because of lower intrinsic stress. On the other hand the elastic modulus was higher, because higher elastic modulus of the silicon substrate compared to the glass substrate.

Keywords: Tungsten, Thermionic Vacuum Arc, Tribological properties.

1. Introduction

Tungsten has – at temperature over 1923K – 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 vapour pressure of all metals, tungsten thin film can be obtained with the Thermionic Vacuum Arc (TVA) method. This type of arc ignites in high vacuum conditions in the vapors of the anode material, continuously generated by the electronic bombardment of the anode [1]. 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 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 about 133 Pa. The thermoelectrons 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. The ignited thermionic 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 following testing conditions were used to study the mechanical properties of the given samples: The loading period of 20s was followed by a hold time of 5s, an unloading period of 20s and finished after holding the minimum load for 5s. Several tests were made at different maximum indentation loads (i.e. several different indentation depths) in order to study the load (depth) dependence of the mechanical properties. At least five different maximum loads were chosen in the interval from 2 to 15 mN.

3. Results and discussion

The samples were tested using depth sensing indentation tester Fischerscope H100 Xyp. We can notice from the Fig.1 that the tungsten film had significantly higher resistance against indentation.

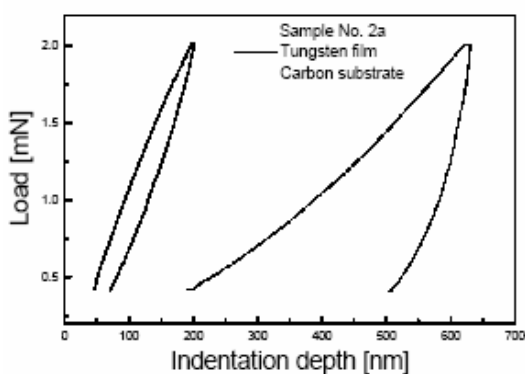


Fig.1 Comparison of loading / unloading curves obtained for sample 2a and the carbon substrate for maximum indentation load of 2 mN

On the basis of this method material parameters such as universal hardness HU (DIN50359-1), elastic part and irreversibly dissipated part of the indentation work (W_e and W_{irr}), plastic hardness HU_{pl} , effective elastic modulus Y_{HU} etc. may be determined. The material parameters obtained on the graphite substrate and the tungsten films are listed below in Table 1.

Sample	HU [N/mm ²]	W _e / W _{tot} [%]	HU [N/mm ²]	hmax [μm]	Y [Gpa]
substrate	158	24,81	205	0,692	6,0
2a	30	0,23	5700	0,250	80,0

The W films deposited on glass substrate showed high hardness, more than two times exceeded the hardness of the glass substrate (8 GPa). The observed hardness is higher than the tabulated hardness of bulk tungsten. One reason could be, that the films exhibited high intrinsic compressive stress. The high stress caused delamination of the films from the glass substrate at the margins of the film.

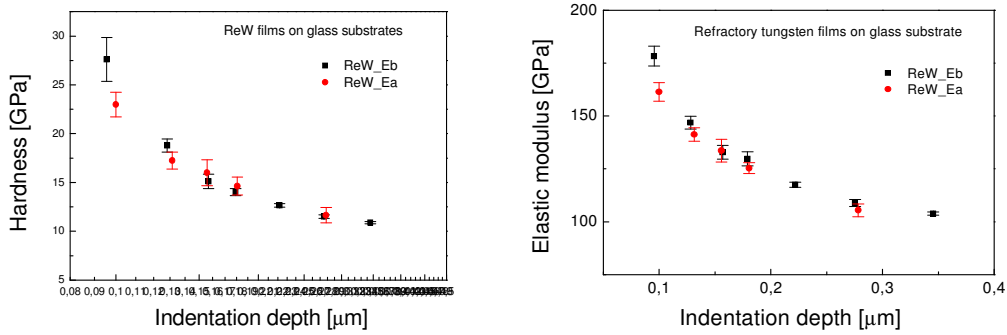


Fig.2 (a) The dependence of the hardness on the indentation depth obtained on thin ReW films deposited on glass substrates; **(b)** Dependence of the elastic modulus of the ReW films deposited on glass.

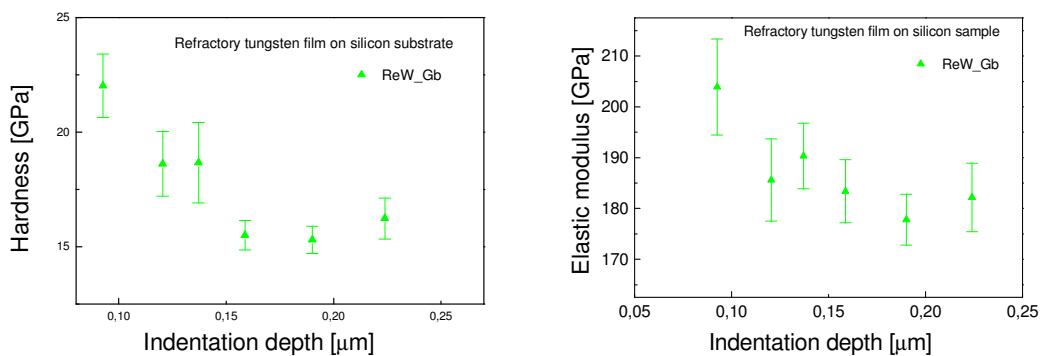


Fig.3 (a) The dependence of the hardness on the indentation depth obtained on thin ReW film deposited on silicon substrate. **(b)** The dependence of the elastic modulus of the ReW film deposited on silicon substrate on the indentation depth.

The resistance of the film against indentation is influenced also by film stress, mainly at low indentation depths. The hardness values obtained on samples at low indentation depths differ, what was probably caused with the higher compressive stress in sample. The difference in compressive stress may be the reason for creation of different delamination patterns at the film boundary. The AFM measurements have proved the smoothness of the deposited films (with some droplets as can be seen in Fig.4) with peak to valley roughness in the range of 20 – 30 nm.

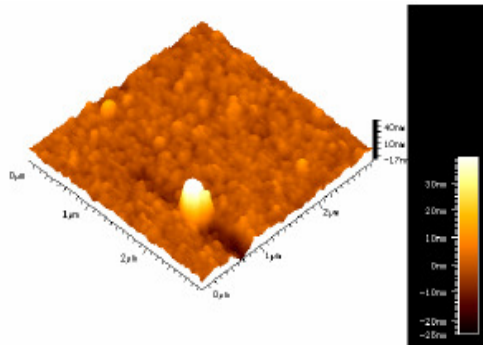


Fig.4 The AFM image of the film

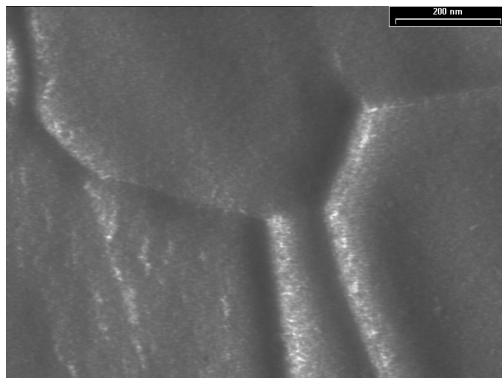


Fig.5 BF-TEM image of W film.

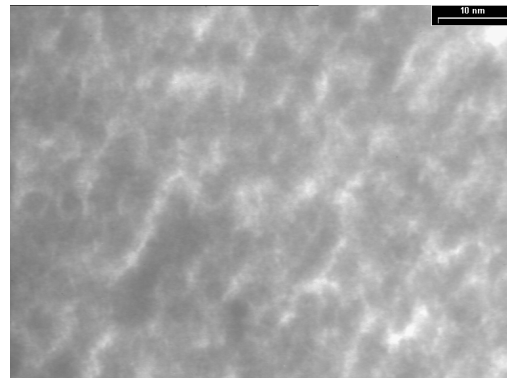


Fig.6 Detail image of W film that shows clustered nanoparticles with mean diameters below 10 nm

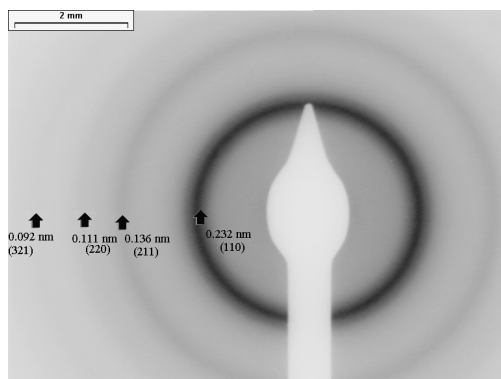


Fig.7 SAED image that confirm cubic structure of W (SG: $Im\bar{3}m$, $a = 3.158$ nm)

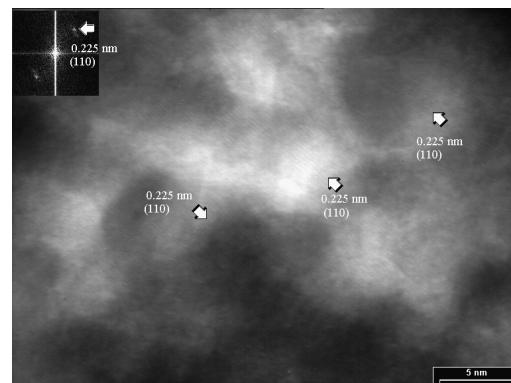


Fig.8 HRTEM image of W nanoparticles that exhibits (110) planes. Left inset show FFT representation of image

TEM analysis of thin layers (10 – 20 nm thickness) revealed the nanostructured tungsten film with grain size in the range of 10 nm (Fig.5 and Fig.6). Fig.7 and Fig.8 show HRTEM image of W nanoparticles that exhibits (110) planes.

Conclusions

The obtained results prove the possibility to use TVA for high quality, pure tungsten film deposition with nanohardness in the range of 5700 N/mm² 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.

References

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