JOURNAL OF OPTOELECTRONICS AND ADVANCED MATERIALS Vol. 8, No. 2, April 2006, p. 617 - 620

# Characteristics of boron thin films obtained by TVA technology

G. MUSA[a,b] , R. VLADOIU[a*], V. CIUPINA[a], C. P. LUNGU[b], I. MUSTATA[b], S. PAT[c], T. AKAN[c], N. EKEM[c]
[a]Department of Physics, Ovidius University,Mamaia 124,900527, Constanta, Romania
[B]National Institute for Laser, Plasma and Radiation Physics POBox MG-36, Magurele, Bucharest, Ramania
[c]Osmangazi Üniversitesi Fen Edebiyat Fakültesi Fizik Bölümü, Eskişehir, Türkiye

In this paper we report the results on the characterization of boron thin film deposited by TVA method. After finishing the successive depositions, the probes covered with boron thin films were analyzed using various techniques: 1) AFM analysis - we obtained a low roughness of the deposited film (less than 10 nm). 2) SEM images analysis - SEM with magnification of 5000 or 10 000 shows again the smoothness of the films as well as the lack of droplets which appears for example in cathodic arc plasma. 3) HRTEM images indicate the d-spacing corresponding to a rhomboedral lattice space group R-3m with a=10.92 Å and c = 23.81Å parameters, according with the calculated data from the electron diffraction pattern. The obtained thin films were of high quality and the evaporation process quite stabile in spite of the used high voltages and arc currents to support the TVA discharge. Diffusion and boron carbide producing at carbon crucible interaction with evaporated boron have been observed.

(Received January 18, 2006; accepted March 23, 2006)

*Keywords:* Boron film deposition, TVA technology, Characterization

## 1. Introduction

Boron is one of the materials of interest for the coming years. Used up to now as basical element in a large number of chemicals or drugs, a growing interest for boron in order to be used as an element (atom) or simple diatomic compound (like boron carbide) can be observed. For example, recently discovered superconductivity of magnesium diboride ($MgB_2$) has generated an increased interest for boron and magnesium processing [1]. From the fast growing papers on the magnesium diboride preparation technologies, co-evaporation method has been also mentioned[2].

If we consider boron from its melting point of view (2300 $^oC$), we can observe that boron is close to the refractory metals.To process such materials for thin film deposition is hard to find convenient crucible materials because of necessary high temperature for crucibles. At such temperatures, compound like boron carbide, tungsten carbide, etc. are easily formed with subsequent failure of the crucible or of the heated cathode filaments.

One of the main advantages of the Thermionic Vacuum Arc (TVA) technology [3-5] is the bombardment of the growing thin film just by the ions of the depositing film. Moreover, the energy of ions can be controlled by TVA voltage drop and changed at will even during deposition, achieving more than 400 eV. This potential is roughly equal to 40 % from the potential drop. Specially designed device with retarding potential for ions has been used for energy measurements. Due to the energetic incident ions, the obtained thin film is compact, with roughness under a few nanometers and nanostructured [6-12].

Few other technologies can compete TVA thin film deposition of materials like C, W, Mo, Nb, Ta, Re, etc. In the present paper we are considering boron thin film deposition using TVA technology and the main characteristics of the obtained boron thin films.

## 2. Experimental

Because of low thermal conductivity of the refractory metals in the case of the use of TVA technology for refractory metal processing, we can replace the anode- which usually is a crucible containing the material to be evaporated- with a rod of the chosen high melting point material. In this way we avoid the use of the crucible. In this case, the accelerated electron beam is concentrated on the top of the rod (usually in vertical position) and a melting and also boiling anode material generates the needed metal vapors to ignite and sustain the thermionic vacuum arc (TVA). Because of the fast decrease of the temperature along the rod type anode, the melted side of the anode material remains only on the top side of the rod. Of course to achieve such and equilibrium, a correct size of the rod must be selected experimentally. Also, anode can be rotated or displaced along the rod axis.

One of the most important advantages of the use of rod type anode is the high purity of the deposited thin film because no crucible is necessary. In Fig. 1 it is schematically shown the TVA electrodes arrangement and electrical supply.

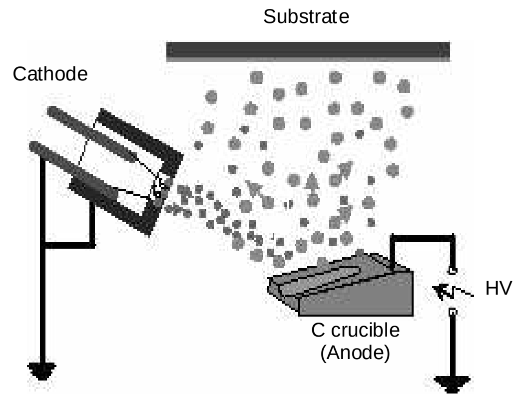
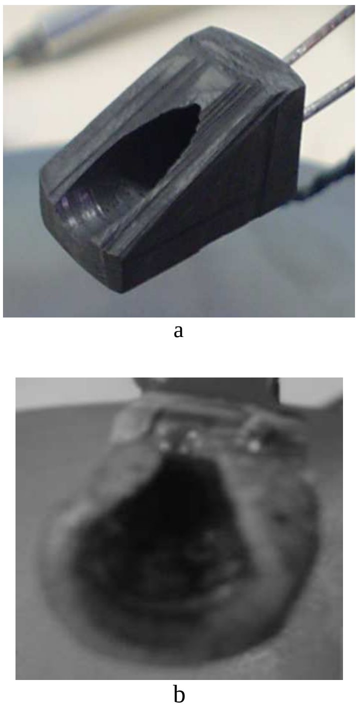



*Fig. 1. Schematically arrangement of the TVA electrodes.*

Other shape of carbon anode is shown in Fig. 3 before and after a number of boron evaporation.

In fact the anode, initially made from pure carbon was covered after successive deposition with a thick but non-uniform layer of boron. However, we still observed a content of tungsten in the deposited boron thin film (around 10 % carbon in boron thin film).



a

b

*Fig. 2. The anode-carbon crucible before(a) and after (b) using it for boron deposition.*

As cathode is used a simple electron beam gun, which consist from a tungsten filament mounted inside of a Wehnelt cylinder. The cathode made of tungsten wire of 0.5 mm diameter was three times wounded over a 1 mm diameter using a special piece only to "shape" the filament. The cathode can be mounted in various positions against the anode. These positions are defined by the angle $\phi$ and the distance between electrodes. The anode is a carbon crucible which is filled up with particles of the material to be evaporated- in the present experiment boron. The electrodes assembly is mounted in a vacuum vessel.

The high vacuum vessel having a diameter of 450 mm and a length of 450 mm is provided with a pumping down system coupled with a mechanical and diffusion pump, ensuring an end pressure in the vessel close to $10^{-6}$ Torr.

For a convenient applied D.C. high voltage over the cathode and anode space, a melted spot appears on the anode surface and a continuous evaporation of the anode material from this melted spot is established due to the accelerated electrons emitted from the cathode. If we use as anode material, boron, a steady state density of the boron atoms is established in the inter-electrodic space.

At further increase of the applied high voltage, suddenly a bright discharge appears in the inter-electrodic gap, with a simultaneous decrease of the voltage drop over the discharge and a significant increase of the arc current.

### 3. Results and discussion

The thin boron films were obtained using Thermionic Vacuum Arc ignited and maintained in the vapors of boron generated at the anode. Besides the difficulties related to the high percentage of carbon atoms in the deposited boron thin film, the hot cathode filament can interact with boron vapors. At high TVA currents, as a result of boron vapor interaction with the tungsten cathode, tungsten carbide is produced, followed by the destruction of the cathode's filament.

However, an acceptable duration of the boron vapors arc discharge is obtained (up to 45 s.). The thickness of the film was measured during the deposition using Cressington thickness monitor MTM [13]. The rate of boron film deposition on the level of probes was up to 200 nm/min. The probes were mounted at a vertical distance of 100 mm from TVA discharge.

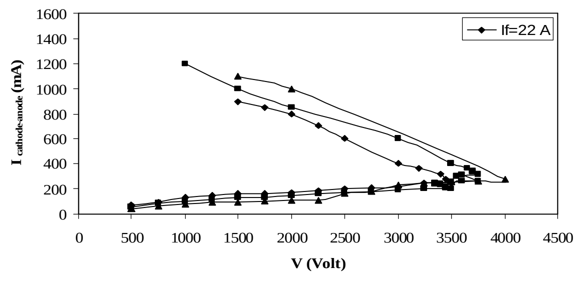



*Fig. 4. The volt-ampere characteristics of boron TVA discharges.*

In Fig. 4 are shown the volt-ampere characteristics of TVA using boron as anode material. Voltages up to 4000 V are used to ignite and to maintain the TVA. The negative slope of the volt-ampere characteristics corresponds to TVA discharge. For comparison, in Fig. 6 are given the volt-ampere characteristics in the case of some metals with melting temperatures between 232 - 1660 $^oC$.

Following supports were prepared for the deposition of boron thin films; glass plate, stainless steel, copper and Mg deposited glass plates. After finishing the successive depositions, the probes covered with boron thin films were analyzed using various techniques: AFM, SEM images analysis, SEM-EDX analysis HRTEM images.

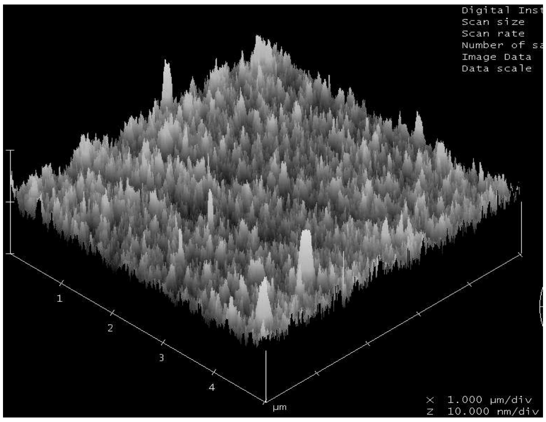



*Fig. 5. The AFM image of the boron thin film deposited using TVA.*

The obtained surfaces were smooth. The images taken at SEM microscope with a magnification of 10000 X, surface appeared completely smooth.

Additional data on the quality of the deposited boron thin film has been obtained using AFM images. In Fig. 5 is shown the AFM images of the deposited boron thin film. The measured values of the roughness of the deposited boron thin film were under 10 nm.

High resolution transmission electron microscope (HRTEM) was performed on a Phylips CM 120 ST (120 kV) with a point resolution of 1.4 Å. In Fig. 6 is represented the structure of the boron thin film deposited by TVA technology on KCl substrates. The samples were floated in water before TEM examination.

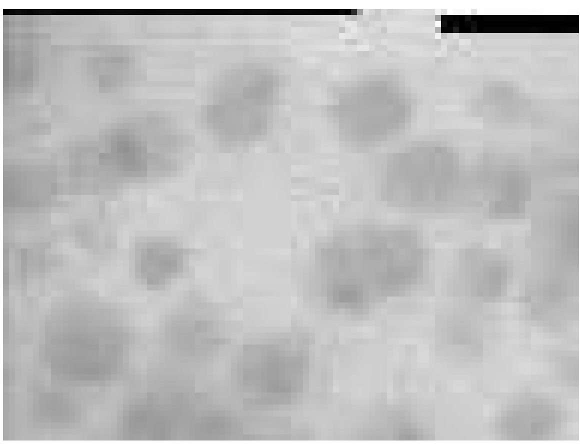


*Fig. 6. HRTEM image of Boron thin film.*

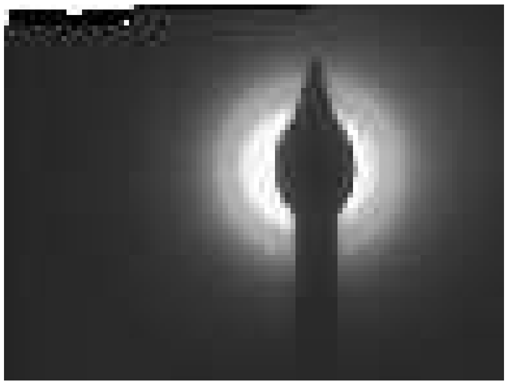


*Fig. 7. Electron diffraction pattern.*

Electron diffraction performed on the boron nanostructures indicated the presence of the well-defined rings (Fig. 7), with d-spacing corresponding to a rhomboedral lattice space group R-3m with a = 10.92 Å and c = 23.81 Å parameters.

## 4. Conclusions

The obtained thin films were of high quality and the evaporation process quite stable in spite of the used high voltages and arc currents to support the TVA discharge.

Boron thin films were analyzed using various techniques:1) AFM analysis - we obtained a low roughness of the deposited film (less than 10 nm). 2) SEM images analysis - SEM with magnification of 5000 or 10 000 shows again the smoothness of the films as well as the lack of droplets which appears for example in cathodic arc plasma. 3) HRTEM images indicate the d-spacing corresponding to a rhomboedral lattice space group R-3m with a=10.92 Å and c = 23.81 Å parameters, according with the calculated data from the electron diffraction pattern.

Diffusion and boron carbide producing at carbon crucible interaction with evaporating boron has been observed. The observed difficulties in the development of a new technology based on TVA for boron processing can be avoided. Already an auxiliary heated cathode has been realized in order to extend significantly the running time of TVA.

## References

[1] J. Nagamatsu, N. Nakagawa, T. Muronaka, Y. Zentani, J. Akimitsu, Nature **410**, 63 (2001).
[2] H. Shimakage, A. Saito, A. Kawakami, Z. Wang, Physica C **392**, 1291 (2003).
[3] G. Musa, I. Mustata, V. Ciupina, R. Vladoiu, G. Prodan, C. P. Lungu, H. Ehrich, J. Optoelectron. Adv. Mater. **7**(5), 2485 (2005).
[4] T. Akan, N. Ekem, S. Pat, R. Vladoiu, G. Musa, J. Optoelectron. Adv. Mater. **7**(5), 2489 (2005).

[5] S. Pat, N. Ekem, T. Akan, Ö. Küsmüs, S. Demirkol, R. Vladoiu, C. P. Lungu, G. Musa, J. Optoelectron. Adv. Mater. **7**(5), 2495 (2005).
[6] G. Musa, Roumanian Report in Physics **37**, 917 (1992).
[7] G. Musa, H. Ehrich, M. Mausbach, Journal of Vacuum Science and Technology A **12**, 2887 (1994).
[8] G. Musa, H. Ehrich, J. Schuhmann, IEEE. Transactions of Plasma Science **25**, 386 (1997).
[9] G. Musa, H. Ehrich, A. Popescu, I. Mustata, A. Salabas, M. Cretu, Procedd. XII. Int. Conf. on Gas Discharges and their applications, Geifswald, (1997).
[10] C. Biloiu, H. Ehrich, G. Musa, J. Vac. Science and Technology A **19**, 757 (2001).
[11] G. Musa, I. Mustata, V. Ciupina, R. Vladoiu, G. Prodan, E. Vasile, H. Ehrich, Diamond and Related Materials **13**, 1398 (2004).
[12] G. Musa, C. P. Lungu, I. Mustata, A. M. Lungu, V. Zarosky, T. Iwanaga, R. Tanaka, Y. Matsumoto, H. Tanaka, T. Oi, K. Fujita, K. Iwasaki, Proceed, 17 ESCAMPIG Int. Conference, 1-5 September 2004, Constanta, Romania.
[13] H. Shimakage, A. Saito, A. Kawakami, Z. Wang, Physica C **392**, 1291 (2003).

*Corresponding author: rodica_vladoiu@yahoo.com