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Boron evaporation and related difficulties

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Boron is one of the materials of interest for the coming years. Used up to now as compounds 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. If we consider boron from its melting point of view, we can observe that boron is close to the refractory metals. Indeed, boron has a melting point of 2300°C, other materials with higher melting points being Niobium (2468°C), Molybdenum (2617°C), Tantalum (2996°C), Rhenium (3180°C), Tungsten (3407°C) and Carbon (3550°C). Boron coating is one of the technologies recently considered to be of special interest due to the qualities of boron. The aim of this paper is to present the related difficulties of the evaporation of boron using Thermionic Vacuum Arc (TVA) Technology.

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1. Introduction

In a number of papers we presented Thermionic Vacuum Arc (TVA) as a new technology for thin film deposition [1-4]. Recently discovered superconductivity of magnesium diboride (MgB_2) has generated an increased interest for magnesium and boron processing [3]. From the fast growing papers on the magnesium diboride preparation technologies, co-evaporation method has been also mentioned [2]. Pure magnesium and boron were used as evaporation sources. Magnesium was evaporated using a thermal heater and simultaneously, boron was evaporated using a deflected electron beam. The main advantage of this method is the fact that no post processing such as high-temperature annealing is necessary [2]. Co-deposition of magnesium and boron using TVA technology instead of that one described in ref [2] might improve the quality of the superconductive magnesium diboride. Our expectation is based on the nanometric structure of the condensing films from TVA. So a better mixing co-processed B and Mg is possible.

As a first step in using TVA technology for superconductors, in this paper we present preliminary results on boron thin film deposition.

2. Experimental arrangement

TVA can be ignited in vacuum (or UHV) between a heated cathode surrounded by an electron focusing Wehnelt cylinder and an anode (crucible) containing the material to be evaporated and deposited Due to the electron bombardment of the anode by the accelerated thermo-electrons from the grounded cathode towards the anode (which is at a high voltage), anode material first melts and afterwards starts to evaporate ensuring a steady state concentration of the evaporated atoms in the cathode – anode space [6,7].

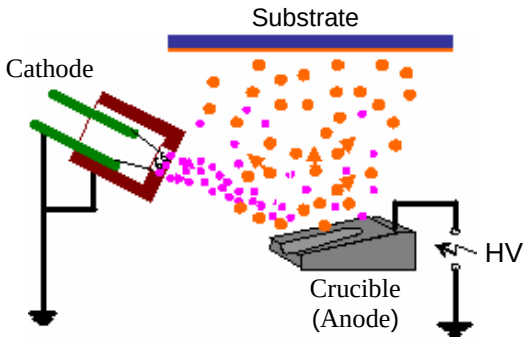

Fig. 1. Schematically the TVA electrodes arrangement.

At further increase of the applied high voltage, a bright discharge is established inside of the vacummated vessel in the vapors of the anode material. The produced metal vapor plasma is localized around the electrodes, the plasma density quickly decreasing radially. In Fig. 1 it is schematically shown the TVA electrodes arrangement and electrical supply.

As cathode is used a simple electron beam gun, which consist from a tungsten filament mounted inside of a Wehnelt cylinder. The cathode can be mounted in various positions against the anode. These positions are defined by the angle ϕ 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.

Boron coating is one of the technologies recently considered to be of special interest due to the qualities of boron. 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. In this experimental research, different crucibles were used for boron evaporations like as wolfram, crucibles wolfram wires, different shape of carbon crucibles, glassy carbon, molybdenum, etc. Different used crucibles for deposition of boron are shown in Fig. 2.

Fig. 2. Different crucibles for deposition of boron.

2.1. Using Wolfram Crucible

Firstly, we tried wolfram crucible for boron experiments for TVA discharge. During the experiments, wolfram crucible and boron material alloyed as wolfram boride, etc. These crucibles were deformed during the depositions. The photo images of crucible are shown in Fig. 3.

Fig. 3. Wolfram crucibles for deposition of boron. The last photo-image show damaged crucible from tungsten.

2.2. Using Molybdenum Crucible

Fig. 4. Molybdenum crucible for deposition of boron.

At using molybdenum crucible, molybdenum and boron material alloyed like in the case of wolfram-boron alloys. Corresponding photo images of crucibles are shown in Fig. 4.

2.3. Using Wolfram Wires Crucible

Fig. 5. Wolfram Wires Crucible for deposition of boron.

In this step home made, wolfram wires crucible was used by. Boron was run away from the heated side of crucible toriad colder point of crucible.

2.4. Using Carbon Crucible

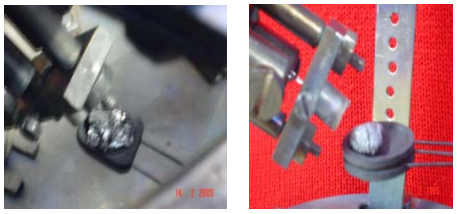

Fig. 6. Carbon Crucible for deposition of boron.

At using carbon crucible, during the discharge, boron run away from the point of impact the electron beam on crucible. The melted boron was displaced towards sides of

carbon crucible. Different geometrical carbon crucibles are shown in Fig. 6.

2.5. Using Boron Rod Crucible

Fig. 7. Boron Rod Crucible for deposition of boron.

We directly used a boron rod as crucible as shown Fig. 7. During the discharge, boron was run away from the point of impact of electron beam on boron crucible.

3. Experimental results

The obtained boron thin film surfaces were smooth. From the images taken at SEM microscope with a magnification of 10000x, the surface appeared completely smooth. Additional data on the quality of the deposited boron thin film has been obtained using AFM images. In Fig. 8 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.

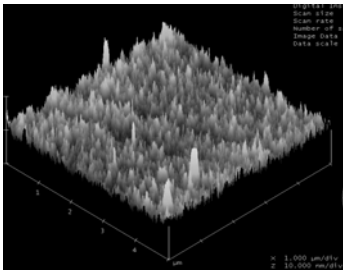

Fig. 8. The AFM image of the boron thin film deposited using TVA[5].

High resolution transmission electron microscopy (HRTEM) was performed on a Philips CM 120 ST (120 kV) TEM having a point resolution of 0.14 nm and a magnification of 1.2 million. HRTEM images and diffraction patterns have been obtained in the films grown on the KCl substrates after they were floated in water. The structure of the deposited film using TVA is shown in Fig. 9.

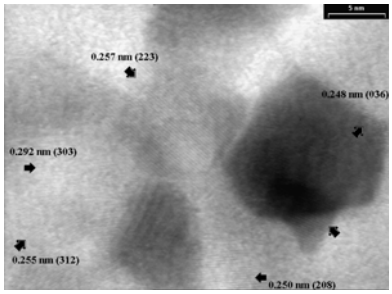

Fig. 9. HRTEM image of boron thin film [5].

In this figure, the arrows indicate the d-spacing corresponding to a rhombohedral 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 (Fig. 10).

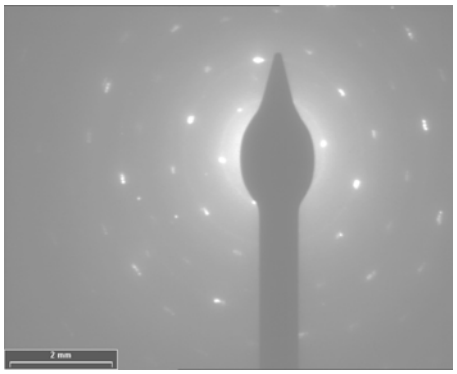

Fig.10. Electron diffraction pattern [5].

Electron diffraction performed on the boron nanostructures indicated the presence of the well- defined rings, (the diameters are showed in the Table I), and the d-spacing have confirmed the rhombohedral structure.

Table 1. Calculated data from the HRTEM images and from the diffraction pattern [5].

Circle-Area	Circle-Perim.	Diameter	dhkl	hkl	Boron 80-0324
mm¸	mm	mm	nm		
5.17403	8.06342	2.56667	0.34675	122	0.34248
6.97075	9.35933	2.97917	0.29874	303	0.29308
8.43461	10.29526	3.27708	0.27158	220	0.27312
11.36603	11.95114	3.80417	0.23395	042	0.23537
12.92228	12.74309	4.05625	0.21941	404	0.21981
18.53807	15.26290	4.85833	0.18319	146	0.18315
21.44186	16.41482	5.22500	0.17033	213	0.17080

4. Conclusions

At the evaporation of boron, difficulties related to crucible and to the alloying of cathodes filament occurred. Also, alloying crucibles (Wolfram, Molybdenum etc.) and boron material clearly appeared. In addition crucibles were generally damaged. Wolfram wires crucible and glassy carbon crucible were very proper for evaporation of boron. The other difficulty was tungsten filament's life time. Tungsten filament's life times were very short (a few minutes) because of filament-boron interaction. The obtained thin films were of high quality and the evaporation process was quite stable in spite of the high voltages used and relatively high arc currents to support the TVA discharge in boron vapors.

The structure of the films have been characterized by AFM images and HRTEM performed on a Phylips CM 120 ST (120 kV) TEM with a resolution point of 1.4 Å and a magnification of 1.2 million times.

The surface appeared completely smooth, the roughness of the boron thin film were under 10 nm, and the measured interatomic distances reveal a nanostructure boron film with a d-spacing corresponding to a rhombohedral structure space group R-3m with a=10.92 Å and c=23.81Å.

The evaporation rate was relatively small (under 10 nm/s) and must be increased. One of the main difficulties is the reduced life time of the tungsten filament. Both of these drawbacks mentioned above can be avoided by using an indirectly heated cathode, first used by us for carbon film deposition using TVA technology.

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