DEPOSITION OF TITANIUM-ZIRCONIUM-VANADIUM FILMS BY DC MAGNETRON SPUTTERING

Yong Wang*, Bo Zhang, Jianping Wang, Wei Wei, Weimin Li, Yaofeng Zhang, Le Fan
National Synchrotron Radiation Laboratory, USTC, Hefei, Anhui 230029 China

Abstract

Titanium-Zirconium-Vanadium (TiZrV) nonevaporable getter (NEG), which can be fully activated after 4 hours heating at 200°C, has been applied in many accelerators owing to the outstanding vacuum performance. In our experiments, TiZrV films have been deposited onto the inner face of stainless steel pipes via DC sputtering using argon gas as the sputtering gas. Samples have been investigated by Scanning Electron Microscopy (SEM), Energy Dispersive X-ray spectroscopy (EDX) and X-ray Photoelectron Spectroscopy (XPS) to determine film composition and thickness, and by X-ray diffraction (XRD) to determine film structure and morphology. Second Electron Yields (SEY) of the TiZrV film have also been measured.

INTRODUCTION

Non-evaporable getter (NEG) coating, which can chemically pump gases such as H_2, CO, CO_2 and N_2 after activation, provides a means to achieve extreme high vacuum, especially for conductance limited vacuum systems in particle accelerators. Besides the outstanding pumping performance, NEGs display lower second electron yield (SEY), photon stimulated desorption (PSD) yield and electron stimulated desorption (ESD) yield than those of uncoated surfaces. Consequently, NEGs have been applied in some high energy machines and synchrotron radiation facilities, such as LHC, ESRF, and SOLEIL [1-3].

The NEGs with the lowest activation temperature are TiZrV alloys, which can be fully activated after heating at 180°C for 24 hours [4-6]. The pumping capacity of a 2 μm thick porous TiZrV coating can be more than that of a 100 μm thick available commercial NEGs [7].

Magnetron sputtering is an appropriate method to coat TiZrV films. The aim of this research is to gather engineering experience in magnetron sputtering process and investigate the relation between the TiZrV film properties and the deposition parameters, so that the TiZrV films with appropriate thickness, composition and good pumping performance can be obtained inside vacuum pipes with various sizes by adjusting the deposition parameters, such as discharge current and cathode voltage. Preliminary experiment result is shown as follows.

TiZrV FILM DEPOSITION

A deposition system was designed to coat TiZrV film onto the inner face of a SST pipe via DC sputtering method using argon as the sputtering gas. The Schematic diagram is shown in Fig. 1. The chamber to be coated, 86 mm in diameter and 500 mm in length, is connected to two auxiliary chambers by Con-Flat flanges. The system is pumped by a turbo-molecular pump which is connected to one auxiliary chamber. The high purity argon intake is located on the other auxiliary chamber and flow rate can be adjusted by a mass flow controlling system. The cathode is obtained by twisting together Ti, Zr and V wires (Φ2mm). A ceramic plate is fixed at the end of the cathode to keep it insulated with the interior wall. Silicon substrates are mounted inside the chamber for evaluation of film thickness, morphology, and composition. The magnetic field is generated by a coaxial solenoid coil, which can generate fields up to 500 Gauss. A 3 KW DC power supply together with the coil are used to ignite and sustain the discharge.

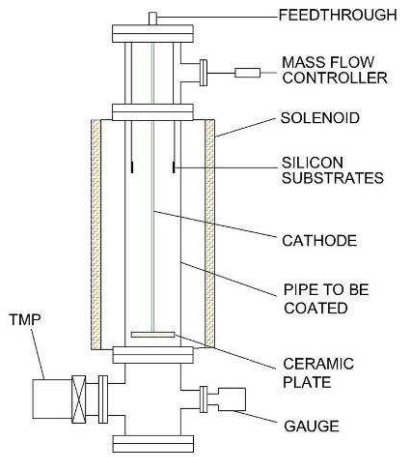

Figure 1: Schematic diagram of NEG deposition system.

Both straight DC sputtering and DC magnetron sputtering were carried out. The deposition rate of straight DC sputtering was much lower than that of DC magnetron sputtering, so DC magnetron sputtering was adopted.

Before deposition, Si substrates were ultrasonically degreased and cleaned in acetone and ethyl alcohol. Then they were dipped into the dilute HF solution, washed with deionized water and dried by purging with nitrogen gas. After deposition, the substrates were analyzed with XPS, EDX, SEM and XRD (Cu K_α radiation).

The optimal substrate temperature during deposition is 250°C, which can increase the roughness and porosity of TiZrV coatings [5]. But a rougher film surface could

*Corresponding author: ywang@ustc.edu.cn

absorb more oxygen after air venting-activation cycles and thus results in an accelerated ageing [8]. Consequently, the substrates were not intentionally heated during deposition and the temperatures varied from 70°C to 180°C with different sputtering current.

The temperature of the TiZrV target, made of twisted wires, may be higher than their melting points due to the bombardment of Ar+ ions, so the discharge current should be low enough to prevent thermal evaporation of the target. Fig. 2a, b shows cross-sectional micrographs of TiZrV film deposited with a discharge current of 0.3A and 0.2A. In Fig. 2a, the micrograph can be divided into two parts: the upper one and the lower one. The lower part exhibits a columnar structure while the upper part doesn't. XPS analysis showed that the composition of the surface was Ti 25 at.%, Zr 48 at.%, and V 27 at.%, different from other films deposited with lower discharge current, which will be shown below. The large increase of Zr and decrease of V content in the film remains unknown. It may result from the deposition of thermal evaporated atoms from the target. In Fig. 2b, the micrograph doesn't show a double-layer structure, so the discharge currents in our experiments were lower than 0.2A. The typical deposition parameters are: 2 Pa Ar pressure, 150-G solenoid field, 500-V cathode voltage, 100-mA discharge current.

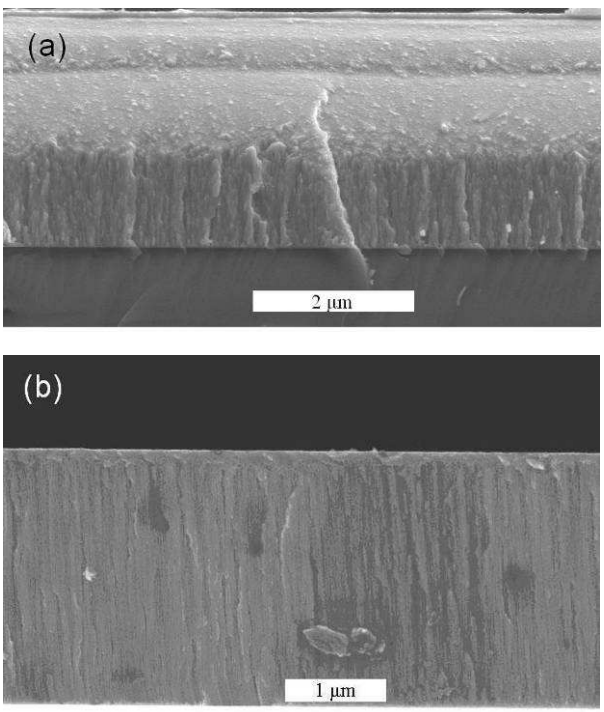

Figure 2: SEM picture of the cross-section of a TiZrV film deposited on Si substrate with a discharge current of (a) 0.3A and (b) 0.2A.

ANALYSIS OF FILM PROPERTY

Film Composition

A typical XPS and EDX spectrum is shown in Fig. 3 and Fig. 4. XPS and EDX measurement, consistent with each other, showed that the average composition of TiZrV samples deposited under various conditions was Ti 29 at.%, Zr 30 at.%, and V 41 at.% . The higher content of V in the film than that in the target results from its higher sputtering yield than that of Ti and Zr [9]. A great amount of oxygen and carbon contamination on the TiZrV surface appeared in XPS spectrum, while only little oxygen and no carbon appeared in EDX spectrum. It results from the difference of sampling depth for XPS and EDX, which is 1-3 nm for XPS and 1000 nm for EDX. It implies that there is almost no carbon and much less oxygen in the getter bulk than in the top surface layer. The oxygen in the bulk comes from the diffusion of surface adsorbed atoms.

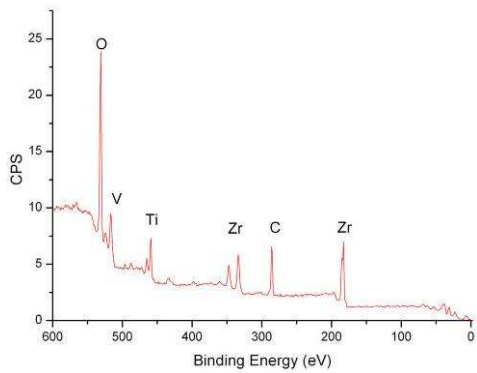

Figure 3: Wide scan XPS spectrum of an as received TiZrV film deposited by magnetron sputtering, showing oxygen and carbon contamination on the surface.

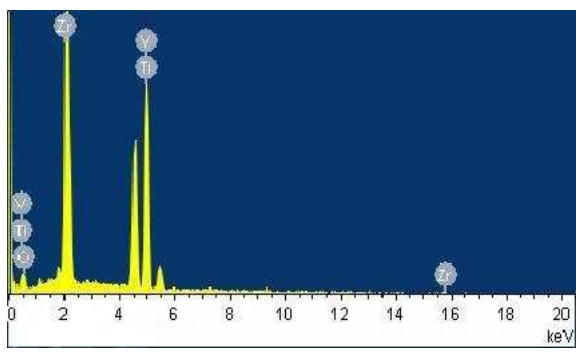

Figure 4: EDX spectrum of an as received TiZrV film deposited by magnetron sputtering, showing no carbon contamination.

XRD Measurement

Fig. 5 shows an XRD pattern of the film. The only diffraction peak occurs at $2\theta \approx 38°$ and is very broad. The crystalline size is less than 5 nm by analyzing the peak according to Scherrer equation. It can be concluded that

the film with this composition and crystalline size should have the lowest activation temperature of 180°C with a heating time of 24 h [6]. Further studies on the activation behaviour of the TiZrV coating will be carried out and verify this conclusion.

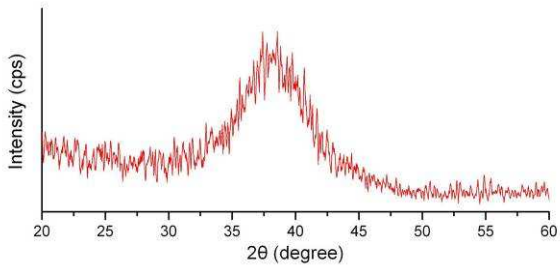

Figure 5: XRD patterns of an as received TiZrV film on Si substrate deposited by magnetron sputtering.

Second Electron Yield (SEY)

TiZrV film can be used as a remedy against electron cloud. The SEY of TiZrV film was measured, as shown in Fig. 6. Primary electrons with energy between 50 and 2900eV bombard the surface at normal incidence angle. The maximal SEY of the TiZrV sample as received was 2.03 and decreased to 1.55 after an in situ thermal treatment at 200°C for 2 h, which is higher than that in Ref. [10]. It may be due to the recontamination of the gas desorbed from the sample and the SST sample holder during heating and electron conditioning.

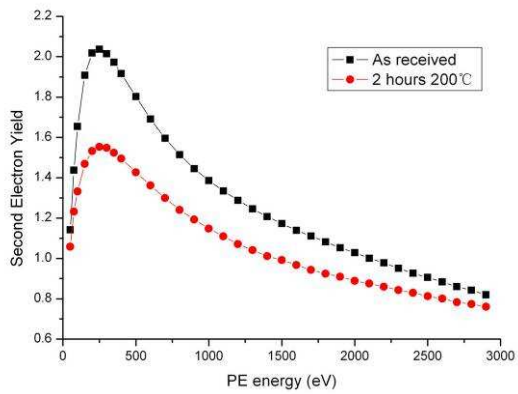

Figure 6: SEY as a function of incidence electron energy for sputtering deposited TiZrV film as received and after 2 h heating at 200°C.

SUMMARY

TiZrV NEG thin films were coated onto SST pipe via DC magnetron sputtering. Deposition parameters were adjusted to obtain NEG films that have appropriate thickness, composition, low SEY and the feature of low activation temperature. Much effort will be taken to optimise parameters to enhance the porosity of the TiZrV film. Pumping characteristic of porous TiZrV film will be measured. A new facility is being assembled to coat a chamber (160mm in diameter, 3000mm in length) and its performance under real accelerator circumstance will be tested.

ACKNOWLEDGMENT

Financial supports for this work were provided by the National Nature Science Foundation of China under Grant Nos.10675119.

REFERENCES

[1] G. Bregliozzi, et al., Proceedings of EPAC 2008, Genoa, Italy, p. 3685-3687.
[2] R. Kersevan, Proceedings of EPAC 2002, Paris, France, p. 2565-2567.
[3] C. Herbeaux, et al., Proceedings of EPAC 2008, Genoa, Italy, p. 3696-3698.
[4] C. Benvenuti, et al., Vacuum 60 (2001), p. 57-65.
[5] C. Benvenuti, et al., Vacuum 71 (2003) p. 307-315.
[6] A. Prodromides, et al., Vacuum 60 (2001) p. 35-41.
[7] A. Prodromides, Ph.D thesis No. 2652 (2002).
[8] P. Chiggiato and P. C. Pinto, Thin Solid Films 515 (2006) p. 382-388.
[9] H.H. Anderson, H.L. Bay, in: R. Behrisch (Ed.), Sputtering by Particle Bombardment I, Springer, Berlin, 1981, chapter 4.
[10] B. Henrist, et al., Applied Surface Science 172 (2001) p. 95-102.