ISBN: 978-3-95450-208-0 doi:10.18429/JACoW-IPAC2019-TUPMP021

COMPARISON OF TIZRV NON-EVAPORABLE GETTER FILMS DEPOSITED BY DC MAGNETRON SPUTTERING OR QUANTITATIVE DEPOSITION

Xiaoqin Ge, Jieqiong Shao, Yigang Wang, Wei Wei, Bo Zhang*, Sihui Wang, Yuxin Zhang, Weimin Li, Yong Wang†
National Synchrotron Radiation Laboratory, USTC, Hefei 230029, China

Abstract

Ti-Zr-V non-evaporable getter (NEG) films have been widely used in vacuum chambers of various accelerators since their discovery. Recently, we have used a new method called "quantitative deposition" to deposit Ti-Zr-V NEG films on nichrome substrates. The surface morphology and surface chemical bonding information were collected by scanning electron microscopy. Although the film deposited by DC magnetron sputtering has more uniform grain growth, smoother grain boundaries and higher porosity, the two films all have porous network structure and can be used as getter films.

INTRODUCTION

Non-evaporable getters (NEG) are now widely used in large particle accelerators, colliders and small precision ultra-high vacuum devices to create ultra-high vacuum environments. A NEG film is applied to the inner wall of the vacuum chamber to change the wall from a venting source to a ventable surface. It will directly improve the vacuum degree of particle accelerators, which is quite negative for long-time operation.

After decades of development, NEG has been produced in many different ways. Vacuum coating is a commonly used coating method, which is a method of applying a special performance coating on a solid surface by physical or chemical means in a vacuum environment [1]. Vacuum coating can be divided into Physical Vapor Deposition (PVD) and Chemical Vapor Deposition (CVD) [2]. PVD is a process that uses physical methods to achieve controlled transfer of material atoms from source to film. Commonly used PVD methods are evaporation coating, sputter coating, and ion plating. CVD is a technique in which a chemical substance in a gaseous or a vapor state forms a solid deposit at a gas phase or a gas-solid interface by a chemical reaction using heating, plasma excitation, and light irradiation. Different methods can be used to obtain films with different structures and properties [3]. Non-evaporable thin film getters are usually prepared by vapor deposition, screen printing, coating, etc., among which magnetron sputtering is most commonly used. We deposited a 720 nm thick film on a Si (111) substrate using DC magnetron sputtering. In addition, we have devised a new method called quantitative deposition. In this method, Ti, Zr and Hf powders were mixed in a ratio of 1:1:1 and uniformly placed on a nichrome substrate, then sintered in a vacuum tube furnace to form a film with a thickness of about 30 μm.

Herein, scanning electron microscopy (SEM) was used to detect the films produced by the two methods and we found that the two films have similar properties. This work provides a very convenient new coating method, quantitative deposition method, and confirms the feasibility of this method.

EXPERIMENTAL PROCEDURES

Using DC Magnetron Sputtering

A layer of Ti-Zr-V film was deposited on the Si substrate using DC magnetron sputtering. The specific experimental procedure has been discussed in our previous article [4]. The substrate was ultrasonically cleaned, then immersed in a dilute HF solution, finally rinsed in deionized water and dried with nitrogen gas. The cathode target was made of three twisted metal wires (Ti, Zr, and V) with a purity of 99.9%, 99.5% and 99.5%, respectively. The discharge gas is Kr gas which is more stable than Ar gas. The main coating parameters are coating time (8 h), discharge current (0.2 A), magnetic field strength (200 G), working pressure (2 P) and cathode voltage (-500 V). The DC magnetron sputtering coating system is showed in Fig. 1 with a stainless steel pipe to be plated.

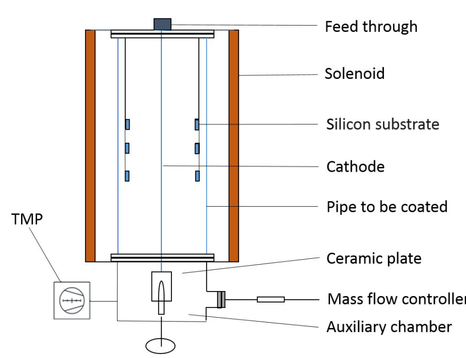

Figure 1: DC magnetron sputtering coating system.

Using Quantitative Deposition

The apparatus for quantitative deposition consists of a vacuum tube furnace, a turbo molecular pump and a vacuum gauge as shown in Fig. 2. The prepared nichrome substrate is first sanded to increase its roughness and enhance the

* zhbo@ustc.edu.cn
† ywang@ustc.edu.cn

ISBN: 978-3-95450-208-0 doi:10.18429/JACoW-IPAC2019-TUPMP021

adhesion of the film. The cleaning process of the nichrome substrate is the same as that of the Si substrate. Then, take a certain amount of Ti, Zr and Hf powders of about 300 mesh in a ratio of 1:1:1, use alcohol as a binder, stir them evenly on the substrate and wait for the alcohol to evaporate. The substrate is shown in Fig. 3. Finally, the vacuum degree of the vacuum tube furnace was pumped to 10^{-3} Pa using a turbo molecular pump, and the dried substrate was placed in the tube and sintered to 1000 °C. The heating rate was about 5 °C/min and held at 1000 °C for 150 min [5].

Figure 2: Quantitative deposition coating system.

Figure 3: The Si substrate used in quantitative deposition method.

RESULT AND DISCUSSION

Figures 4 and 5 show films deposited on a Si substrate using DC magnetron sputtering and quantitative deposition, respectively. The film deposited by DC magnetron sputtering has a stronger metallic color, and the film deposited by the quantitative deposition method is relatively inferior in compactness and adhesion.

Figures 6 and 7 show the surface morphology information of the films deposited by DC magnetron sputtering and quantitative deposition, respectively, obtained by SEM [6]. It can be seen from the figures that both films have porous network structures. The surface is composed of many fine particles, and the particles are bonded to each other to ensure porosity and mechanical strength. The pores between the particles can be regarded as a container for storing gas. They are likely to be adsorbed on the surface after the reactive gas molecules enter these holes.

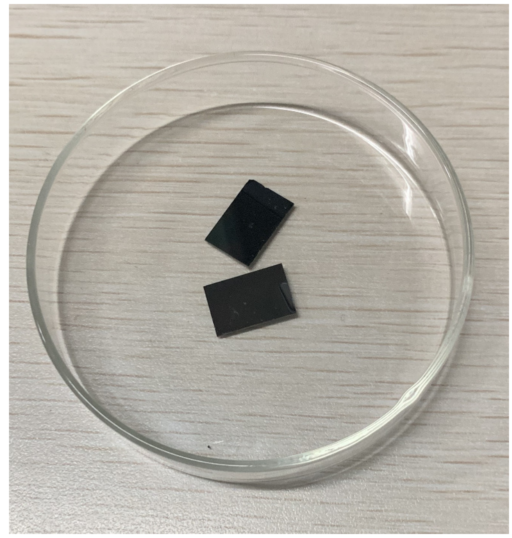

Figure 4: The film deposited by DC magnetron sputtering.

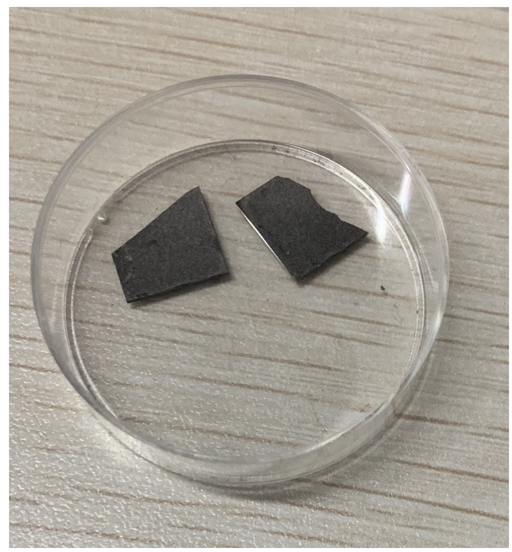

Figure 5: The film deposited by quantitative deposition.

In the case of DC magnetron sputtering, the properties of the sputtering target directly determine the stability of the sputtering and the properties of the film. The utilization of the target is directly related to the sputtering rate and the sputtering cost. In the case of quantitative deposition, the film needs to be sintered using a vacuum tube furnace. Sintering is divided into contact, formation of sintered neck, densification, grain growth and other stages. As the sintering temperature continues to increase, the sintered neck grows rapidly. The interface between particles quickly closes and forms a closed hole between the interfaces, affecting the

ISBN: 978-3-95450-208-0 doi:10.18429/JACoW-IPAC2019-TUPMP021

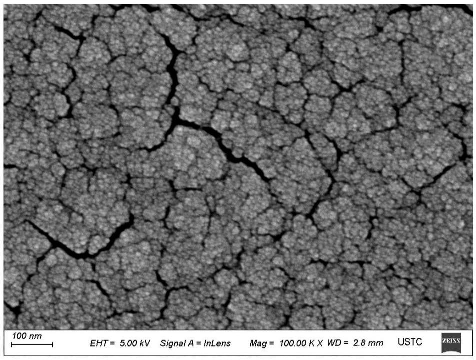

Figure 6: Surface Observation of the TiZrV film obtained by SEM using DC magnetron sputtering.

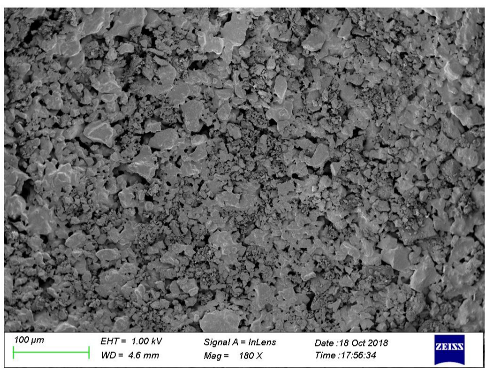

Figure 7: Surface Observation of the TiZrV film obtained by SEM using quantitative deposition.

uniformity of the target. At the same time, when the grain boundary moves faster than the velocity of the hole at high temperature, the grain boundary and the hole are unhooked, and closed pores are formed in the grain. These closed holes are difficult to shrink and disappear under the action of diffusion, which affects the density of the target [7]. It can be seen from Fig. 7 that the surface of the film is relatively uneven, and its porosity is also slightly worse than that of the film prepared by DC magnetron method (lower porosity results in a large weight and a faster decay of the gettering rate). But the overall structure of the two films is similar, so we can think of them as having similar gettering properties.

CONCLUSION

In this work, Ti-Zr-V NEG films were successfully deposited on Si substrates by DC magnetron sputtering and quantitative deposition. The surface properties of the two films were examined by SEM. Although the porosity and density of the two films differed somewhat, they all have very similar porous network structures. This work not only provides a new alternative strategy for the deposition of NEG films, but also confirms the feasibility of this method.

ACKNOWLEDGEMENTS

This work was supported by the National Natural Science Foundation of China (11475166, 11575214).

REFERENCES

[1] Calatroni, S *et al.*, "NEG thin film coating development for the MAX IV vacuum system", in *Proc. 4th Int. Particle Accelerator Conf. (IPAC'13)*, Shanghai, China, May 2013, paper THPFI044, pp. 3385–3387.

[2] Konyashin, I Yu *et al.*, "PVD/CVD technology for coating cemented carbides", in *Surface and Coatings Technology 71 (1995) 277–283*. doi:10.1016/0257-8972(94)02325-K

[3] Kıvak, Turgay *et al.*, "Optimization of surface roughness and flank wear using the Taguchi method in milling of Hadfield steel with PVD and CVD coated inserts", in *Measurement 50 (2014) 19–28*. doi:10.1016/j.measurement.2013.12.017

[4] Ge, Xiaoqin and He, Tianlong and Pei, Xiangtao and Wang, Yigang and Wang, Yong and Wei, Wei and Zhang, Bo and Zhang, Yuxin, "Deposition and characterization of TiZrHfV films by DC magnetron sputtering", in *Proc. 9th Int. Particle Accelerator Conf. (IPAC'18)*, Vancouver, Canada, Apr.-May 2018, pp. 4983–4985. doi:10.18429/JACoW-IPAC2018-THPML129

[5] Yue, S and Pilliar, RM and Weatherly, GC, "The fatigue strength of porous-coated Ti–6% Al–4% V implant alloy", in *Journal of biomedical materials research 18 (1984) 1043–1058*. doi:10.1002/jbm.820180908

[6] Wang, Jie and Wang, Yong and Xu, Yanhui and Zhang, Bo and Wei, Wei, "Research on the secondary electron yield of TiZrV-Pd thin film coatings", in *Vacuum 131 (2016) 81–88*. doi:10.1016/j.vacuum.2016.05.001

[7] Jie, Wang and Bo, Zhang and Yan-Hui, Xu and Wei, Wei and Le, Fan and Xiang-Tao, Pei and Yuan-Zhi, Hong and Yong, Wang, "Deposition and characterization of TiZrV-Pd thin films by dc magnetron sputtering", in *Chinese Physics C 39 (2015) 127007*. doi:10.1088/1674-1137/39/12/127007