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DEVELOPMENT OF NON-EVAPORABLE GETTER(NEG) COATINGS ON SMALL DIAMETER VACUUM CHAMBERS FOR DIFFRACTION-LIMITED STORAGE RING*

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Abstract

Design of the fourth generation Diffraction-Limited Storage Ring reduces aperture of vacuum chambers to a few centimeters. To satisfy the small aperture, the intense photon bombardment and the requirement of low pressure, most of the beam pipes need to be deposited with Ti-Zr-V non-evaporable getter (NEG) thin films. NEG can provide distributed pumping and low gas desorption and allow to achieve low pressure in narrow and conductance limited chambers. In this paper, Ti-Zr-V thin film was deposited by DC magnetron sputtering using Ti-Zr-V alloy target. The morphology and thickness of Ti-Zr-V are characterized by Scanning Electron Microscopy (SEM). The average grain size is evaluated using X-ray diffraction (XRD). The composition and the corresponding chemical bonding of the thin film are analyzed by X-ray Photoelectron Spectroscopy (XPS). Finally, the adhesion between the film and substrate and the vacuum performance are evaluated.

INTRODUCTION

With the development of Multiple Bend Achromat (MBA) lattice design, construction of new generation synchrotron light source having low horizontal emittance to gain high brilliance becomes a global interest [1]. In order to reach a small emittance electron beam, the aperture of magnet units is much smaller than that of the traditional storage ring [1]. This induces a challenge for the vacuum system design as traditional vacuum pumps are unable to achieve an ultra-high vacuum. NEG is widely used to capture molecules impinging on their active surfaces in order to achieve ultra-high vacuum in vacuum systems [2]. It not only provides in-situ conductance-free pumping speed but also reduces outgassing from the walls [2]. These features are ideal for conductance limited narrow vacuum chambers. Besides these benefits, gas desorption under photon, ion and electron bombardment, as well as secondary electron yield (SEY) of NEG coatings also have been reported to be lower than uncoated surfaces [2].

In this paper, alloy target will be used to deposit Ti-Zr-V as it can provide homogeneous composition of the thin film and result in a lower NEG activation temperature [3]. Surface characterization, such as the morphology, grain size, composition and so on, will be analysed. Finally, the adhesion will be measured by tensile experiment and the correlation between the vacuum property and deposition parameters will be evaluated.

THIN FILM DEPOSITION

The DC magnetron deposition was carried out in a new, dedicated system, a schematic of which is shown in Fig. 1. The 100 mm external deposition chamber allows different sizes of narrow tubes to be coated in the deposition system. A 6 mm diameter Ti-Zr-V alloy target with equal atomic percentage of Ti, V, and Zr was used. Two types of samples were deposited. One type was deposited on small pieces of silicon and OFC copper substrate that were placed 12 mm away from the target. Another type was deposited on the inner wall of a stainless steel tube with a inner diameter of 35 mm and a length of 550 mm.

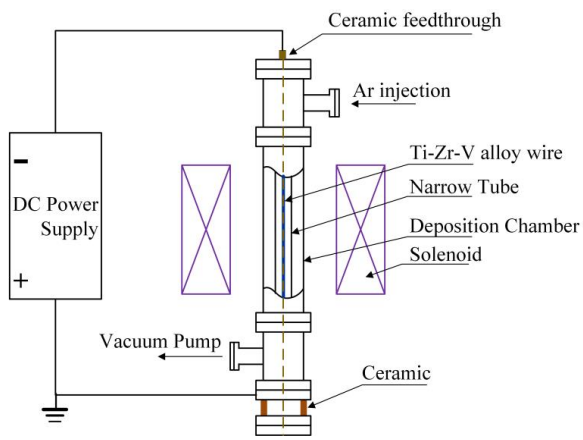

Figure 1: Layout for DC deposition system.

Pre-cleaning of small test samples and narrow stainless steel tube involved degreasing and etching in order to ensure adequate adhesion of the film to the substrate. Ar was used as sputtering gas. The base pressure before deposition was 8.0×10^{-5} Pa. A power of 60 W and a magnetron field of 160 G was used during the deposition.

SURFACE CHARACTERIZATION

Morphology and structure of thin films were determined by Sirion200 schottky field emission scanning electron microscope and grazing angle x-ray diffraction, respectively. The grazing angle for XRD was 1° and 2θ data were collected between 20° and 80° using a step size of 0.02°. The

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x-ray anode target was Cu Kα. This geometry allows to minimize the diffraction information from the substrate, thus to enhance that from the thin film. The surface chemical bonding and composition was determined by XPS. The XPS analysis was carried out using an Thermo ESCALAB 250Xi photoelectron spectrometer. XPS data was measured with monochromatic Al Kα ($h\upsilon$=1486.7 eV) operated at 150 W, with a beam spot size of 500 μm.

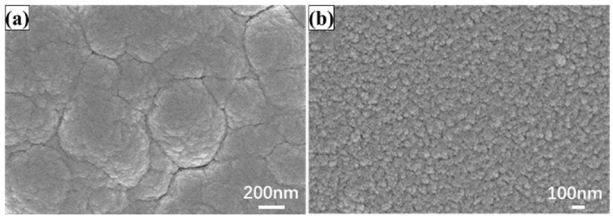

Figure 2: The surface morphology of Ti-Zr-V films on a) OFC copper, b) silicon.

Figure 2 displays the surface morphology of Ti-Zr-V films deposited on OFC copper and Si (100) by SEM. In both cases, the films have nanometer structures and a large number of nucleations can be observed. Compared with OFC copper substrate, the surface of Ti-Zr-V film on Si is more uniform. This is due to the specific orientation of the single cystal Si substrate and the roughness of substrate surface.

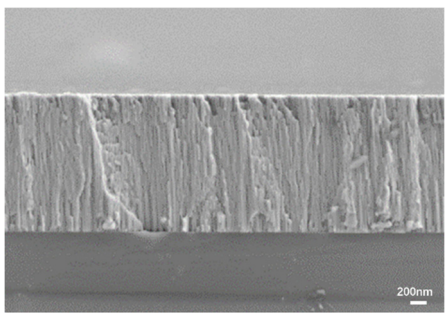

Figure 3: The cross-sectional structure of Ti-Zr-V film on silicon.

Figure 3 shows cross-section SEM micrographs for Ti-Zr-V film on Si (100). The grains are columnar. The estimated thickness of Ti-Zr-V film is 1.8 μm.

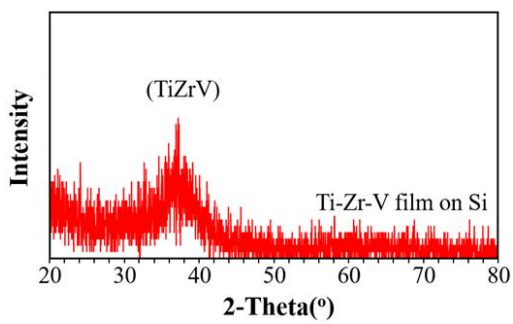

Figure 4:XRD result for Ti-Zr-V film on Silicon.

In Fig. 4, XRD result for Ti-Zr-V film deposited on Si (100) substrate is exhibited. The peak at 2θ=37.6° associated with the film presents a rather broad width. Grain size is calculated by Scherrer's equation :

$$D = \frac{K\lambda}{B\cos\theta}$$

where D is the grain size; K is Scherrer's constant; λ is the wavelength of Cu Kα; B is the full width at half maximum (FWHM); θ is the diffraction peak angle. The average grain size calculated from this diffraction peak is 2.6 nm.

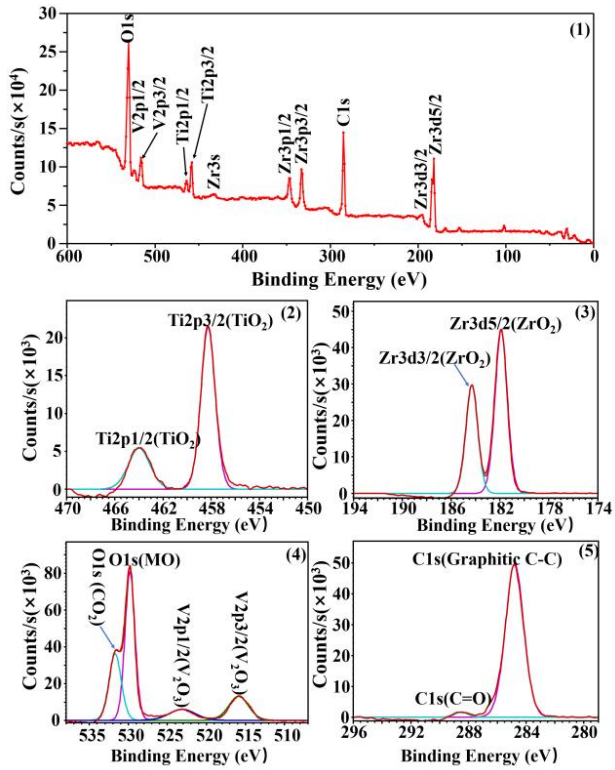

Figure 5 : XPS spectra of Ti-Zr-V film on OFC copper (1) wide scan, (2) region scan of core level of Ti, (3) region scan of core level of Zr, (4) region scan of core level of V and O, (5) region scan of core level of C.

In order to confirm the chemical state of the film, as-recieved Ti-Zr-V on OFC copper was analyzed by XPS， as shown in Fig. 5. Figure 5 (1) represents a wide scan XPS spectrum. It can be seen that Ti, Zr, V, C and O peaks are present on the top surface of the film, and share a concentration of 4.6 %, 5.6 %, 3.1%, 47.8% and 38.9%, respectively, shown in Table 1. The C and O peaks are dominated due to formation of passivation layer on the top of the film during exposure in the air.

Table 1: Surface Composition Measured by XPS for Ti-Zr-V on OFC Copper

Element	O 1s	C 1s	Ti 2p	Zr 3d	V 2p
Atomic [%]	38.9	47.8	4.6	5.6	3.1

Figure 5 (2)-(5) show the region scan of core levels of Ti, Zr, V, O and C of as-recieved Ti-Zr-V depostied on OFC Cu, seperately. The backgrounds of XPS region scans were corrected by the shirley method and fitted into several primary peaks. In Fig. 5 (2), Ti 2p peak is a doublet due to spin-orbit coupling. The binding energies of Ti 2p3/2 and Ti 2p1/2 peaks are 458.3 and 464.0 eV, respectively, which are associated with TiO_2. Similarly with Ti 2p peak, Zr 3d

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and V 2p peaks also exhibit two characteristic peaks due to spin-orbit coupling, as shown in Fig. 5 (3) and Fig. 5 (4). The peaks of Zr 3d5/2 and Zr 3d3/2 at the binding energy of 181.9 and 184.3 eV correspond to ZrO_2. V 2p3/2 peak at 515.9 eV and V 2p5/2 peak at 523.0 eV are related to V_2O_3. The dominant peak of O1s at 529.8 eV is associated with metal oxide. There is a small shoulder at the higher energy of 531.6 eV. The C 1s peak of as-recieved sample can be decomposed into two peaks, as displayed in Fig. 5 (5). The major peak at 284.8 eV is attributed to graphitic C-C binding and the minor peak at 288.8 eV is related to C=O binding.

ADHESION MEASUREMENT

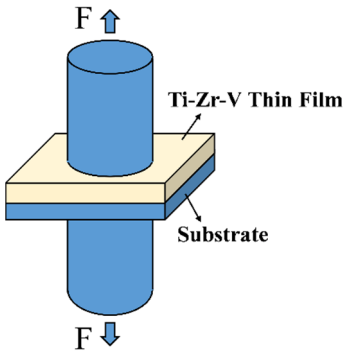

Figure 6 : Schematic layout of tensile experiment for Ti-Zr-V film on OFC copper.

Figure 6 displays the schematic layout of tensile experiment for the measurement of adhesion strength of Ti-Zr-V film on OFC copper. In this test, Ti-Zr-V film and OFC copper substrate were glued onto two cylindrical blocks with diameter of 4 mm. Two equal vertical forces are applied using an universal tensile machine. The results of the stress evolution during tension test is shown in Fig. 7. The film delaminated at the stress of 4.2 MPa. It should be noted that the crosshead displacement mainly origins from the sliding of the cylindrical blocks within the grippers rather than from the film/substrate adhesion. The adhesion between film and substrate depends on the surface state of substrate, such as roughness, cleaning proceduces and so on.

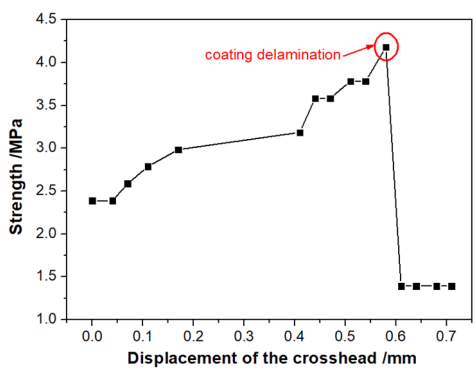

Figure 7 : The adhesion strength of Ti-Zr-V film on OFC copper.

PUMPING PROPERTY

The initial pumping property of Ti-Zr-V film deposited on the inner wall of the stainless steel tube, with an inner diameter of 35 mm and a length of 550 mm, was measured using a simple test setup, as shown in Fig. 8. Before activating the Ti-Zr-V film, a base pressure of 2.5×10^{-4} Pa was achieved by the turbo pump. After activating the Ti-Zr-V film at 200 °C for 24 hours, the valve was closed and the pressure dropped dramatically to 1.4×10^{-7} Pa. This illustrates that Ti-Zr-V film with the current composition and deposition parameters has promising pumping ability.

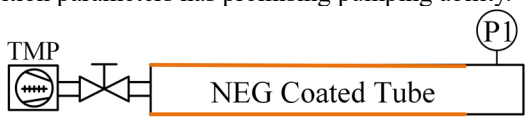

Figure 8 : Layout of pumping property test.

CONCLUSION AND FUTURE WORKS

Ti-Zr-V films were deposited by sputtering an alloy target (equal Ti, V, and Zr atomic percentage) on silicon, OFC copper and stainless steel tube. Ti-Zr-V films on both Si and OFC Cu shows a columnar nanostructure, however, the film on Si is more uniform due to the specific orientation of the single cystal and flat substrate surface. The thickness of Ti-Zr-V film on Si is 1.8 μm and the average grain size is about 2.6 nm. Due to the air contamination, the surface of Ti-Zr-V film on OFC Cu formed a passivation layer. Its adhesion strengh is 4.2 MPa. Initial pumping property of Ti-Zr-V film on stainless steel tube was evaluated. It shows that the current Ti-Zr-V film provides promising pumping ability.

These are the initial studies of NEG deposited by alloy target. To meet the demand of vacuum in small diameter vacuum chamber, the properties of NEG films need a further optimization by varying deposition parameters and enhancing surface treatments. The ongoing work will also study the activation temperature and pumping speed of the deposited films.

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