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PREPARATION AND CHARACTERIZATION OF NON-EVAPORABLE Ti–Zr–V GETTER FILMS FOR HEPS*

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Abstract

For the low activation temperature and high pumping speed, surface pumping capacity, the TiZrV coatings were chosen to high energy photo source (HEPS). Films of TiZrV alloy have been deposited on 1.5 meter long, cylindrical vacuum chambers of 22mm diameter copper substrates in krypton ambient using DC magnetron sputtering system.

Film composition, the activation temperature and pumping properties have been investigated in order to optimize the deposition parameters for vacuum applications. The films were also studied using the X-ray photo-emission electron spectroscopy (XPS) after annealing them at different temperatures ranging from 180℃ to 300℃ for half hour in ultra-high vacuum environment.

Pumping speed and surface pumping capacity testing facilities were also being constructed to investigate the characterization of TiZrV.

INTRODUCTION

The present work is being undertaken in the frame of Beijing Advanced Light Source project at Institute of High Energy Physics (IHEP). To further develop the technologies necessary for diffraction-limited storage rings based light source, it involves five areas: vacuum system/non-evaporative getter (NEG) coating of small chambers, injection/pulsed magnets, RF systems/bunch lengthening, magnets/radiation production with advanced radiation devices, and beam physics design optimization. By focusing the current beam to develop high brightness x-ray synchrotron, it requires beam lines in centimetre or even millimetre range in diameter in order to gain good control of beam position and shape. When the vacuum conductance is much reduced in such narrow chambers, they are difficult to reach ultrahigh vacuum (UHV) that is necessary for accelerators. Getter films deposited on the inner surface of the chamber would transform the vacuum chamber from an outgassing source into a pump.

The extensive use of NEG based pumping systems for large UHV systems, such as particle accelerators and Tokamak reactors, was pioneered by the European Organization for Nuclear Research (CERN) at the time of the design phase of the Large Electron Positron (LEP) collider [1 - 3]. The NEG strip covered about 23 of the 27 km of the LEP machine, providing vacuum in the range of 10e-10 Pa. Since then, SAES-Getters have made the NEG strip commercially available. NEG pumps contribute to the achievement of UHV in storage rings for particle physics research and synchrotron radiation production, and are widely accepted by the accelerator community. In recent years [4 - 6], different getter materials have been investigated, Innovative vacuum pumps, based on the combination of sputter-ion pumps (SIPs) with NEG technology have been invented [7].

In this context, our work will focus on the progress of the deposition of NEG coatings in very narrow chambers, as well as engineering and physics challenges they face today.

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PREPARATION OF THE GETTER FILMS

A schematic diagram of the experimental setup for NEG deposition is shown in Fig. 1. The sample is a 1.5 m long, 22 mm in inner diameter copper cylindrical tube. The cathode was made by twisting three wires of high-purity (99.95%) titanium, vanadium and zirconium, each of 1 mm diameter. TiZrV type was chosen because of its lowest activation temperature among ternary getters.

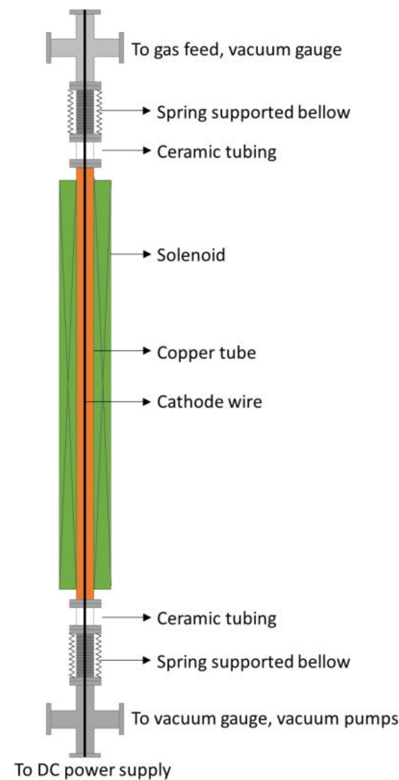

Figure 1: Schematic diagram of the experimental system for NEG deposition.

The tube sample is electrically isolated mounted to the chamber in the direction of gravity. The cathode wire runs through the tube sample and is positioned approximately

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along the axis of the tube. The bottom end of the cathode wire was connected to a high voltage feedthrough, and the top end was mounted to a viewpoint. Gas inlet was mount on the top of the system, and the vacuum pumps are mounted in the bottom. A solenoid was mounted outside and coaxial to the sample tube, providing a magnetic field parallel to the electric field.

The system was evacuated with a 350 l/s turbo molecular pump backed by an oil-free mechanical pump and baked for 2 days at 200℃ to a base pressure in the 1.0e-9 mbar range. High purity krypton was injected using a mass flow controller. The flow rate could be adjusted up to 10 sccm to obtain the desired operational pressure, which was typically in 1.5×10^{-2} mbar. The solenoid is powered by a DC power supply, providing a desired magnetic field about 200 ~1000G. The discharge was powered by a DC power supply, capable of delivering up to 1000V and 1A. During deposition, the power was set to be approximately 80 W. The deposition time was 12 h. As shown in Fig. 2, the NEG coating was uniformly deposited on the inner surface of the entire copper tube.

Figure 2: A photo of the NEG coating deposited on the inner surface of a 1.5 m long, 20 mm inner diameter copper tube. The tube was sliced open manually.

CHARACTERIZATION OF THE GETTER FILMS

The average composition of the TiZrV films, measured by Rutherford backscattering (RBS) analysis, is Ti 21at.%, Zr 29 at.%, and V 50 at.% with the assumption that the sum of the atomic percentage of Ti, Zr and V is 100%. XRD analysis showed that the size of nanocrystalline is lower than 5nm.

The Activation Process

The activation process of TiZrV films was characterized by the XPS in an ultra-high-vacuum (UHV) chamber. The thermal activation process was performed by heating at 80℃ for 8h to degas, and annealing at intervals throughout 180, 250 and 300 °C for 30 min and then were cooled down to room temperature. Then the XPS spectra were recorded. Figure 3 shows the Ti2p, V2p, Zr3d XPS spectra of TiZrV film. The binding energy (BE) of the Ti 2p spectrum peak of as-deposited (air-exposed) film is 458.3 eV (doublet separation of Ti 2p, Δ=5.7 eV), whose value corresponds to that of titanium oxide TiO_2, as shown in Fig. 3A. A mixture of metallic state Ti_0 at BE=454.1 eV and low valence Ti_{n+} (n<4) states is observed after activation at temperature of 180 °C or higher. The variations for V 2p spectrum peaks on the activation temperature are similar to those of Ti 2p, as shown in Fig. 3B. For the air-exposed TiZrV film, BE of the $V2p_{3/2}$ is 515.5 eV, whose value corresponds to oxidized V (V_{3+}). The vanadium oxide of the surface layer is markedly reduced to the metallic state V_0 after activation at temperature of 180 °C or higher. The series of Zr 3d spectra of TiZrV film are shown in Fig. 3C. For the air-exposed TiZrV film, the binding energy of the Zr $3d_{5/2}$ is 182.5 eV, whose value corresponds to closely oxidized Zr (Zr_{4+}). The Zr $3d_{5/2}$ peak also shifts to lower binding energy of lowly oxidized state Zr_{n+} (n<4) or metallic Zr state (Zr_0) after the activation from 180 °C to 300 °C. These mean that the highly oxidized Zr (Zr_{4+}) gradually changes to sub-oxidized Zr (Zr_{n+}, n<4) and metallic Zr_0 with the increase of activation temperature. [8]

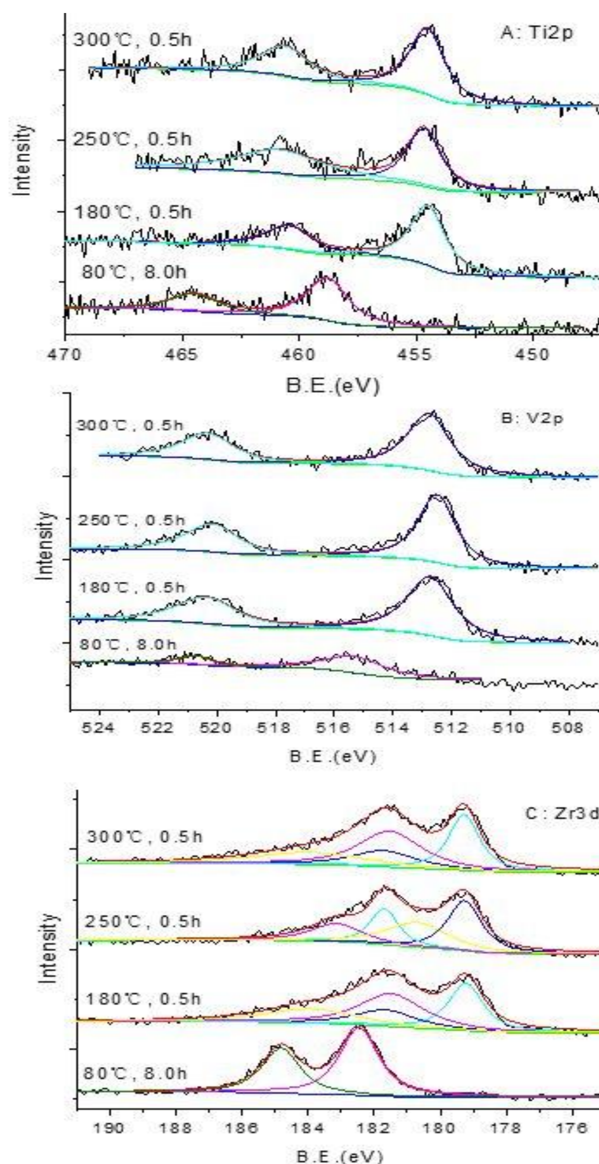

Figure 3: The series of Ti 2p, V 2p and Zr3d XPS spectra of TiZrV film at various heating temperatures.

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Pumping Properties

The pumping speed per unit area is defined as:

$$S = \alpha \cdot C_{gas}\ [l \cdot s^{-1} \cdot \mathrm{cm}^{-2}] \quad (1)$$

where α is the sticking probability ($0 \leqslant \alpha \leqslant 1$), which could be calculated by Monte Carlo simulation through simulation, and C_{gas} is the conductance, expressed in $[l \cdot s^{-1} \cdot \mathrm{cm}^{-2}]$. In the molecular flow regime, C_{gas} does not depend on the pressure. It may be expressed as:

$$C_{gas} = 3.64\sqrt{\frac{T}{M}}\ [l \cdot s^{-1} \cdot \mathrm{cm}^{-2}] \quad (2)$$

The total gas absorbed by NEG film is define as:

$$Q_0 = \int_0^t C_{or} \cdot (P3 - P2)dt\ [\mathrm{mbar} \cdot l \cdot s^{-1}] \quad (3)$$

The schematic of pumping properties test facility of NEG pipe is show in Fig. 4. After 48h, 250°C degassing of vacuum chamber, and 24h, 180°C or higher activation process. The sticking factor of CO, H_2 were tested. From the test we would get the P_1/P_2 ratio corresponding to CO or H_2, and capacity of TiZrV NEG film calculated by equation (3). The conductance of orifice for CO is 3.89 l/s.

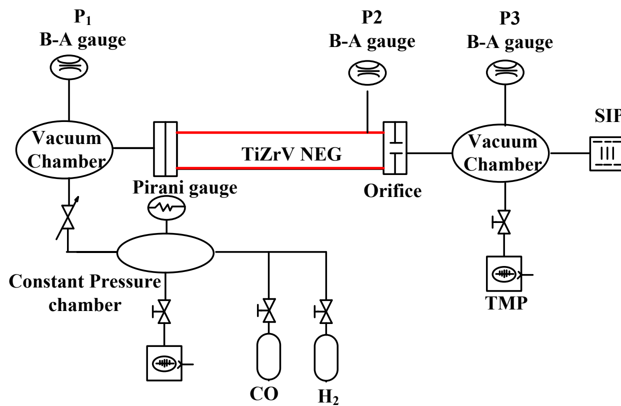

Figure 4: The schematic of pumping properties test facility of NEG pipe.

When CO inlet, P_1 is about 1.0×10^{8}mbar, P_2 is about 1.0×10^{5}mbar, P_3 is about 3.0×10^{5}mbar. The pumping test result is show in Table 1. Maybe because the P_1 is measured by total pressure, so the P_2/P_1 ratio is very low and correspond sticking factor is lower than 0.01 which is about 1/80 of literature [9].

Table 1: Result of Pumping Test of NEG Film at Different Temperature

No. run	Test gas	capacity	Vacuum Ratio
		mbar.l/cm^2	P_2/P_1
NO30-4-300	CO	8.32E-05	717.9
NO30-5-180		7.36E-05	1454
NO30-6-180		6.67E-05	1200
NO30-7-250		7.78E-05	1333.3
NO30-9-250	H2	2.75E-03	220

CONLUTION

Facility of TiZrV NEG film coating system and pumping test system were built. 1.5 meter long and 22mm diameter cylindrical copper substrates vacuum chambers were coated. XPS analysis show that Ti2p, Zr3d, V2p spectra shift to low binding energy when activation temperature increase to 180°C. But may due to the measured error, the pressure ratio between two sides of NEG pipe is low. Consequently, the sticking factor α to CO and H_2 is low.

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