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Investigation of vacuum performances of TiZrV coated pipe

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Abstract Some metal compounds called as Non-Evaporable-Getter have been widely used to improve vacuum system performance of accelerator facility. In this paper, a TiZrV film on the surface of stainless steel vacuum pipe is made by direct current magnetron sputtering, and its vacuum performance is experimentally studied. Our results show that the TiZrV film is partly activated at 160°C, and its pressure performance is similar with one at higher temperature. The coating reduces the ultimate pressure and prominently shortens the pressure-down time in a sputter ion pump system, thus creating evenly distributed pressure profile in a coating pipe. The adsorption rate is steady, and adsorption amount increases linearly. Such TiZrV-coated pipe behaves like pump other than gas source in vacuum system.

Key words TiZrV, Activation, Adsorption, Pressure, Direct current magnetron sputtering

1 Introduction

Titanium, zirconium, hafnium, and vanadium can react with gas molecules, such as H_2, CO, CO_2 and O_2, at room temperature[1]. Placing them in vacuum pipes improves the vacuum, with the merits of clean, no vibration, and low ultimate pressure. As a non-evaporable-getter (NEG), these materials are used in vacuum systems. At first, NEG was used in accelerator vacuum systems as lumpy pump. Since 1995, when the Large Hadron Collider (LHC) at CERN achieved good vacuum with vacuum pipes of getter-coated inner surface[2], NEG coating has been widely adopted in large scale accelerator projects[3–8].

At Shanghai Synchrotron Radiation Facility (SSRF), many insertion devices will be installed for producing high quality synchrotron radiations for the users. To obtain high magnetic field of an insertion device, its magnet gap is designed as small as possible, and so is the vacuum pipe. This lathy structure would cause such a serious problem of gas conductance that it is virtually impractical to acquire preferable vacuum performance, even with large speed pumps. Therefore, inner getter coating of the vacuum pipe is the best solution.

NEG coatings produce less photon-induced desorption than stainless steel (SS) or oxygen free copper[9,10]. It is reported that the H_2 pressure rose to 7×10^{-9} and 3×10^{-7} Pa from NEG and SS under synchrotron radiation bombardment, respectively[9]. NEG only works after activation. The preferable activation temperature for TiZrV coating is usually 180°C[2]. A combination of sputter ion pump with Ti sublimation pump are often used in ultimate pressure test to acquire ultra-high vacuum[2], but Ti sublimation pump is not ideal in an NEG-combined vacuum system because of its strong thermal radiation during activation. Sputter ion pump is commonly adopted as the main pump in an NEG-combined vacuum system, to check vacuum performance of NEG coating.

In this paper, a vacuum pipe is coated with TiZrV. Its vacuum performance at lower activation temperature is investigated in a sputter ion pump system. The TiZrV coating is partly activated at 160°C. The ultimate pressure is evidently reduced and attained in short time.

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2 Materials and Method

2.1 Preparation of NEG coating

The NEG materials are Ti, Zr and V, because their compound enhances gas dissolution and diffusion[1]. The direct current magnetron sputtering technique was used to coat the NEG onto a 304 SS pipe of Φ100 mm ×1200 mm (Fig.1a). The cathode was a twisted thread of three Φ2 mm wires, placed along the pipe axis, applied with a negative voltage, while the pipe was grounded, having been baked to eliminate water and oil on the surface, the pipe was evacuated by a turbo molecular pump (TURBOVAC 151). Ar gas (99.999%) was injected into it, and discharged in the electric field. The magnetic field produced by the solenoid coil prolonged the electron flight path, and increased the ionization efficiency. The pressure was measured by a BPG400 gauge. By adjusting the Ar gas flow carefully, the inner surface was NEG-coated by sputtering when pipe was at an ultimate pressure. The glow discharge occurred at 5×10^{-1} Pa, with a cathode voltage of 600 V; magnetic field of 120–180 G; and discharge current of 0.7–1.0 A.

2.2 The vacuum performance tests

Three tests were conducted to investigate the TiZrV coating, with an experimental set-up of the TiZrV-coated pipe. As shown schematically in Fig.1(b), two auxiliary chambers (A and B) could be connected to the pipe. The heat jacket was used to bake the pipe. The system has a turbo molecular pump and a 100 L/*s* sputter ion pump.

Test I was to determine the NEG activation condition. The coated pipe was evacuated to ultimate pressure by the turbo molecular pump, filled with dry N_2, and baked for 24 h at 160°C or 180°C. The temperature was measured by thermal couple.

Chambers A and B were pumped by the sputter ion pump alone in Test II(a), and in Test II(b) it was pumped by the ion pump and the TiZrV-coated pipe, having been baked at 180°C for 24 h. The gas adsorption of TiZrV coating was evaluated by pressure comparison of different systems.

In Test III, the NEG-coated pipe baked at 180°C for 24 h, and a nude pipe of the same dimension, were used. And pressures near the pumps (Gauge 1, P_{near}) and far from the pumps (Gauge 2, P_{far}) were measured, so as to compare the pressure ratio of the two systems.

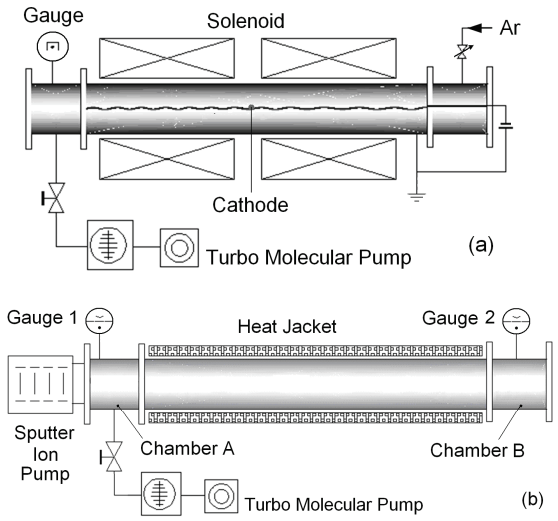

Fig.1 Schematics for coating a pipe with NEG (a) and experimental setup with the NEG-coated pipe (b).

3 Results and Discussion

3.1 Coating ingredient

The coatings were examined by scanning electron microscope (LEO 1530 VT). Fig.2(a) shows a fine topography of the TiZrV coating. Fig.2(b) is EDX (energy dispersive X-ray) spectrum of the film, which indicate that the film contains 36.10% Ti, 31.27% Zr and 32.64%V.

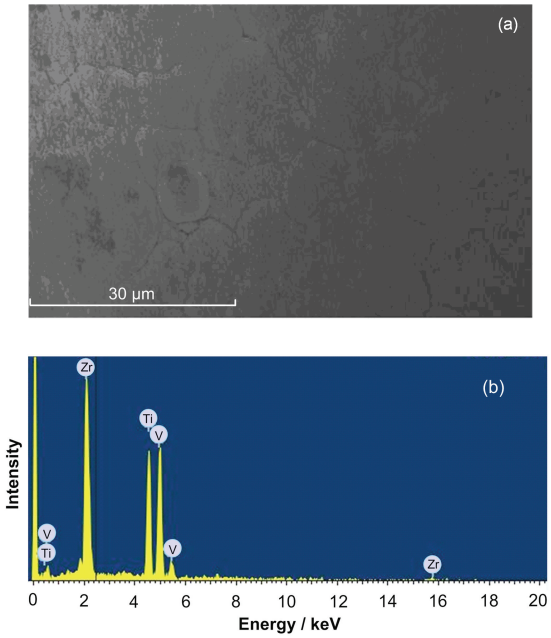

Fig.2 SEM image and EDX spectrum of the TiZrV coating.

3.2 Activation temperature

Figure 3 shows the pressure performances at 180°C and 160°C. The pressure decline rates were almost the same during the initial 3–4 h, but the ultimate pressures were 9×10^{-9} Pa and 2.2×10^{-8} Pa for 180°C and 160°C baking, respectively, indicating that the TiZrV coating was partly activated at 160°C.

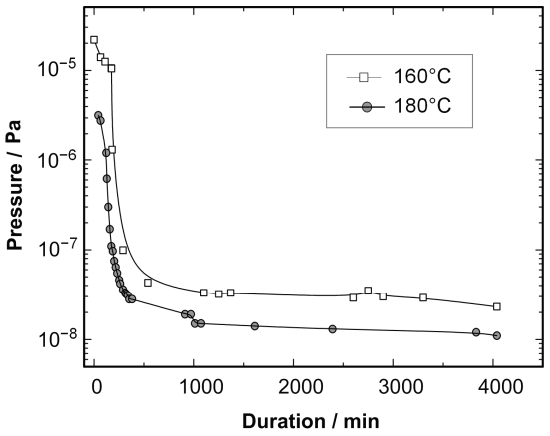

Fig.3 Vacuum performance of the pipe coated with TiZrV activated at 160°C and 180°C.

3.3 Pressure performance

As shown in Fig.4, the ultimate pressures were 9×10^{-9} Pa and 6×10^{-8} Pa, pumped by the sputter ion pump plus the TiZyV coating and by the sputter ion pump alone, respectively. It took about 12 h for the system with just the sputter ion pump to go from 3×10^{-6} Pa to 1×10^{-7} Pa, while it took 3–4 h for the system helped by the residual gas-adsorbing TiZrV coating to go from 3×10^{-6} Pa to 2×10^{-8} Pa.

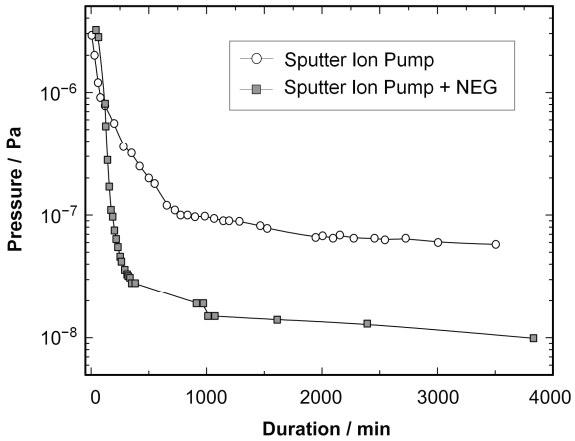

Fig.4 Vacuum performance of the system pumped by the sputter ion pump with or without the NEG help.

In Test III, pressures of Chamber A and B (P_{near} and P_{far}) were measured. They can be approximately expressed by Eqs.(1) and (2) according to gas flow principle.

$$(P_{far} - P_{near})C = P_{far}S_e \quad (1)$$

$$P_{far}/P_{near} = (S_e/C)+1 \quad (2)$$

where, S_e is effective pumping speed, and C is pipe conductance. As shown in Fig.5, the pressure ratio of nude pipe was P_{far}/P_{near} = 1.2–1.3, which is in accordance with theoretical value; while P_{far}/P_{near} of the TiZrV-coated pipe was closed to 1.0. The TiZrV coating rendered the system a uniform pumping behavior, which is of significance for pumping a vacuum system of lathy structures.

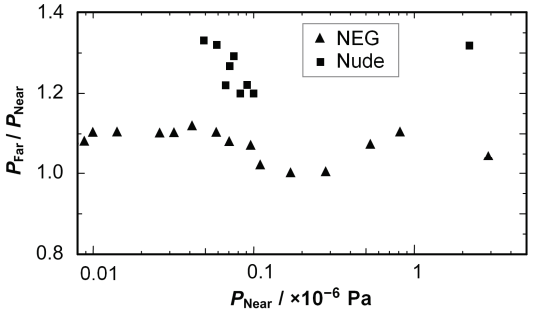

Fig.5 Ratio of pressures at two ends of the pipe with or without the NEG coating.

3.4 Adsorption capacity

When pressure in the vacuum system declines from P_i to P_{i+1} at an interval of t_i, the gas adsorption rate in the NEG-coated pipe is $Q_i=(P_i-P_{i+1})V/t_i$, where V is the pipe volume. Assumed Q_i and Q_s as pressure changes for the vacuum systems pumped with and without the help of TiZrV coating, respectively, the gas adsorption amount of TiZrV coating is $Q_N=Q_i-Q_s$. The gas-adsorption rate of TiZrV coating was 3×10^{-9} – 5×10^{-9} Pa·L·s^{-1} (Fig 6a), and the adsorption amount is 10^{-8}–10^{-7} Pa (Fig.6b).

Adsorption capacity of the TiZrV coating is affected by activation-air venting cycles. A TiZrV coating may keep steady pumping performance under 10^{-8} Pa for years. When it is activated, however, its oxygen content may increase because of oxygen diffusion, resulting in decreased pumping speed and increased activation temperature. About 25 cycles is allowable to keep activation temperature at 350°C[11].

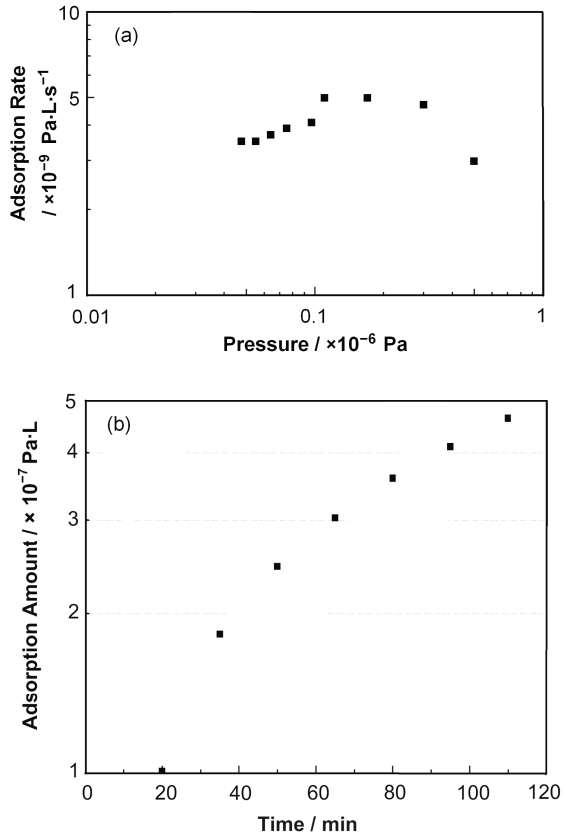

Fig.6 Adsorption rate (a) of TiZrV coating, and amount of the adsorption (b).

4 Conclusions

Homogeneous TiZrV coating is deposited on inner surface of a stainless steel vacuum pipe using direct current magnetron sputtering. Activated at 180°C, the coating adsorbs gas effectively and uniformly, which is of great meaning for a vacuum system with lathy pipes, such as the insertion devices at SSRF.

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