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NEG FILM DEVELOPMENT AND MASSIVE COATING PRODUCTION FOR HEPS*

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Abstract

Massive production facilities of NEG coated vacuum chambers have been developed for HEPS in Huairou, Beijing, which based on the NEG coating prototypes of HEPS-TF. The facilities can achieve simultaneous coating of 16~20 vacuum chambers of HEPS including irregular shaped vacuum chambers. The pumping performance of the NEG coated vacuum chambers has been measured by test facilities. After heating at 200 °C for 24 hours, the highest pumping speed of H_2 is about 0.65 l/s·cm^2, and the highest capacity of CO is about 1.89×10^{-5} mbar·L/cm^2. The lifetime is more than 20 cycles of air exposure and re-activation. The pumping performance meets the design requirements of HEPS. Currently the NEG coated vacuum chambers are applied to the storage ring of HEPS.

Introduction

HEPS (High Energy Photon Source) was designed to be a fourth-generation synchrotron radiation light sources with the lowest emissivity and highest brightness in the world. One crucial technology is to coat non-evaporable getter (NEG) films on the inner wall of vacuum chambers of small aperture in order to meet ultra-high vacuum requirements.

The NEG coating is a deposition of a titanium, zirconium, vanadium alloy on the inner surface of the chamber, typically achieved through DC magnetron sputtering. The utilization of NEG coatings has been widespread in the fourth generation of light sources to meet the stringent vacuum requirements, primarily due to the low conductance of the vacuum chambers, like MAX-IV [1] and Sirius [2]. NEG coating have been massively empoyed in the straight sections of the LHC [3], approximately 6 km of vacuum chambers were coated with NEG film.

NEG Coating Development

A DC magnetron sputtering facility has been established at IHEP for the investigation of NEG coating since 2016, as depicted in Fig. 1 [4].

For achieving a uniform thickness distribution, the NEG coating chamber is equipped with a cathode made of twisted wires of high-purity (99.95 %) titanium (Ti), zirconium (Zr), and vanadium (V), each having a diameter of 1 mm. To maintain the proximity of the cathode wires to the chamber's axis, several ceramic spacers are strategically placed along the chamber's length, along with two adapters at the ends.

To create the necessary magnetic field, a solenoid with dimensions of 1500 mm in length and 280 mm in diameter is externally mounted on DT4. The ion pump is utilized to attain ultra-high vacuum (UHV), and the NEG bulk pump is employed to evacuate residual gases such as CO and H_2O to achieve UHV conditions.

The chambers are initially evacuated using a turbomolecular pump group, reaching a range of 10^{-9} mbar. They are then subjected to a 48-hour bake-out process at a temperature of 200°C, followed by a helium leak test to ensure their integrity before the coating process. A Residual Gas Analyzer (RGA) is utilized for monitoring residual gases during the coating process.

Prior to the coating process, thorough cleaning of the NEG-coated chamber with etching 50 μm and passivion is conducted to prevent any significant contamination or surface defects that may adversely affect the quality of the film.

During the coating process, krypton gas of high-purity (99.999 %) is used as the working gas, set at approximately 0.01 mbar. The chamber temperature is maintained at around 120 °C to facilitate the sputtering process.

Figure 1: Prototype of NEG coating facility.

Massive Coating Production for HEPS

Previously, high-quality NEG coating has been achieved by NEG coating prototypes. However, the circumference of the HEPS storage ring is approximately 1360.4 m, including about 1000 vacuum chambers to be coated. Therefore, massive production facilities has been developed to meet engineering requirements (see Fig. 2).

Except for the dipole-magnet vacuum chambers, which were made of 316L stainless steel, the other vacuum chambers in the storage ring were made of Cr-Zr-Cu alloy copper (C18150). To reduce the impedance of the stainless steel vacuum chambers, a 20 μm copper film has been coated on the inside. All of the Cr-Zr-Cu vacuum chambers, including those with a diameter of 22 mm, ante-chamber, and racetrack shape, with NEG coating is ongoing.

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Figure 2: (a) Massive produce facilities of NEG coating, (b) Vacuum chambers were installed in parallel and series to massive coating.

The coating device A: Vacuum chambers are connected in parallel to 6 groups, each group of vacuum chambers length should be lower than 3.5 m, outer diameter is about 0.47 m.

The coating device B: Antechamber are connected in parallel to 4 groups, each group of vacuum chambers length should be lower than 1.5 m, due to its discharge difficulty.

Two setups of NEG coating have been built for vacuum chambers of HEPS at IHEP Lab. And a lot of test vacuum chambers have been coated, which shows that NEG film has good adhesion and thickness distribution.

Pumping Properties Evaluation

Given that the residual gases in the accelerator primarily consist of H_2 and CO (constituting approximately 99 %) and a smaller portion of Ar and CH_4, the pumping properties of H_2 and CO are of utmost importance when considering the NEG coating. To evaluate the performance of the NEG coating for accelerator applications, it is crucial to characterize its pumping speed and absorbing capacity.

The measurement of pumping speed and absorbing capacity is carried out using the transmission factor method, which was first introduced in NEG coating vacuum chambers by C. Benvenuti in 1999 [5]. The schematic diagram of the pumping speed measurement setup is depicted in Fig. 3. Following each activation cycle of the NEG coating, test gases are injected into the vacuum chamber using variable leak valves. The gases then pass through an orifice into the NEG coated pipe, where a significant portion of the gases is absorbed by the NEG coating. Two Residual Gas Analyzers (RGAs) are positioned at the ends of the pipe to monitor the pressure levels. By measuring the pressures P1 and P2 and calculating the ratio P2/P1, the sticking factor or pumping speed can be determined using molflow simulation.

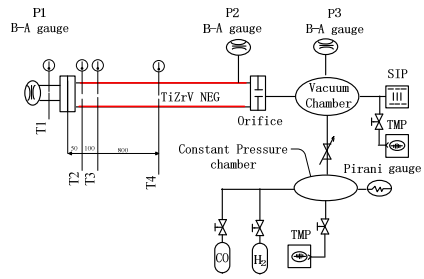

Figure 3: Schematic diagram of the pumping performance test facility.

The pumping speed of CO by the NEG coating is approximately 6-8 times higher than that of H_2, while the capacity of CO is much smaller than that of H_2. The pumping speed of H_2 and the capacity of CO are therefore considered to be more critical factors. Test results for the pumping speed of H_2 at different activation temperatures are presented in Fig. 4, and capacity of CO are presented in Fig. 5.The NEG coatings of TiZrV and TiZrVHf can be effectively activated at 160 °C. The pumping speed of H_2 increases as the activation temperature rises from 160 °C to 250 °C but decreases when the activation temperature exceeds 250 °C.

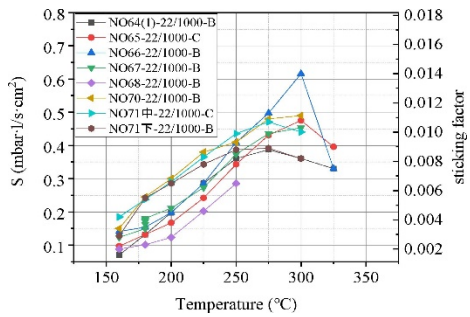

Figure 4: Pumping speed of NEG coating under different activation temperature.

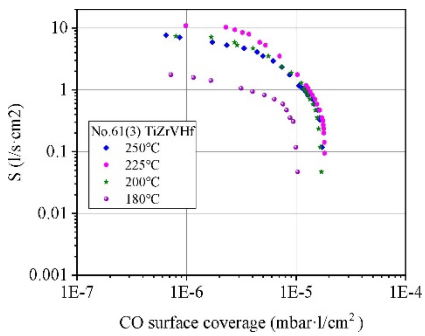

Figure 5: CO capacity of NEG coating under different activation temperature.

When all the absorption sites of the NEG coating are filled, its ability to pump gases diminishes. Molecules that contain oxygen atoms, such as H_2O, CO, O_2, etc., chemically adhere to the surface of the NEG coating, resulting in the formation of metallic oxides (M_xO_y). During the activation process, the metal oxides gradually decrease, and oxygen atoms diffuse into the bulk of the coating due to concentration gradients. This activation mechanism imposes limitations on the number of reactivation cycles that can be performed. Figure 6 presents the results regarding

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the lifetime of a 1 μm thick NEG coating, highlighting its finite lifespan.

Massive produce of NEG coating presents a significant challenge, due the complex process and large number of vacuum chambers. Figure 2 shows the massive production facilities for HEPS, which is in Huairou, Beijing.

Due to impedance limitations of HEPS, it is crucial to adhere to a average thickness of 1um for the TiZrV NEG coating. However, this constraint imposes a limitation on the lifetime of the coating, allowing for fewer than 20 re-activation cycles.

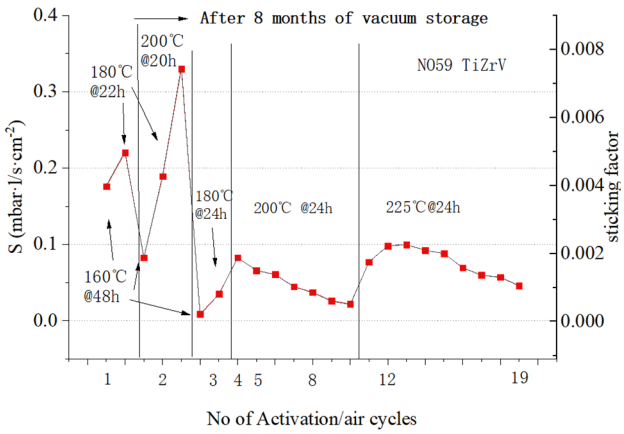

Figure 6: The lifetime of NEG coating of 1 μm thickness.

Thickness Distribution

To ensure a uniform distribution of thickness, one cathode is applied for round vacuum chambers with a 22 mm diameter. However, for ante-chambers and racetrack shape vacuum chambers, two cathodes are used as presented in Fig. 7.

Figure 7: Prototype of racetrack shape vacuum chamber, two cathodes were mounted along the axis direction.

The thickness distribution of the NEG coating along the axis of the vacuum chamber depends on factors such as the uniformity of plasma discharge pressure, magnetic field, and cathode position. Extensive experiments conducted in HEPS have demonstrated that achieving a distribution within ±30 % error is relatively straightforward.

CONCLUSION

As a fourth-generation synchrotron radiation light source, vacuum chambers with small apertures were employed for HEPS, making the performance of NEG coating is very crucial for its vacuum system. After years of development, the highly stability of the NEG coating has been achieved. Massive production of NEG coating is currently underway, and all vacuum chambers are expected to be completed within six months.

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