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SPUTTERING CHARACTERISTICS OF A COMPACT NEG-COATING DEVICE AND PERFORMANCE EVALUATION OF THE TiZrV THIN FILMS

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Abstract

Non-evaporable Getter (NEG) coating is a breakthrough technology wherein the inner walls of a vacuum chamber are coated with a material that functions as a vacuum pump. This technology is expected to gain widespread adoption across various fields in the future. However, the current coating method, originally developed for long beam ducts, is not adaptable to a wide range of vacuum chamber designs. Therefore, we have developed a compact NEG-coating device that can be adapted to chambers of various geometries. The primary advantage of this device is its ability to coat complex-shaped chambers, which was difficult with conventional methods. Additionally, by reducing the uncoated surfaces as much as possible, it significantly improves pumping performance in terms of pumping speed and reducing Photon Stimulated Desorption (PSD) yields.

We explore the optimal sputtering conditions for depositing high-performance NEG thin films with the device, and have performance evaluations of the NEG films, with observing the morphologies and XPS measurement.

INTRODUCTION

NEG-coating technology has been widely used in modern vacuum systems, such as particle accelerators, where ultra-high vacuum (UHV) conditions are required within increasingly compact and complex geometries. NEG-coating works by transforming the inner surface of a vacuum chamber into a vacuum pump through the deposition of a TiZrV alloy film. This film efficiently removes residual gas molecules via chemisorption, providing high effective pumping speed and significantly lowering the Photon Stimulated Desorption (PSD) yield. Unlike conventional pumping methods that rely on discrete exhaust ports, NEG-coating contributes to more uniform and efficient vacuum performance [1].

To fully exploit these advantages, it is essential to coat not only straight ducts but also intricate and irregularly shaped vacuum chambers. However, with conventional deposition methods, it is difficult to deposit films in a variety of containers. In response to this limitation, we have developed a Compact NEG-coating device that can be directly attached to various vacuum components.

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A COMPACT NEG-COATING DEVICE

A Compact NEG-coating Device is a flange-mountable vacuum apparatus, as shown in Figure 1. This device employs magnetron sputtering but introduces two major innovations. First, it uses a cylindrical TiZrV alloy pipe as the sputtering target instead of the conventional trifilar wire. Second, it generates the necessary magnetic confinement using a periodic array of Sm-Co permanent magnets embedded inside the target pipe, replacing the external solenoid magnets used in standard setups. These modifications significantly miniaturize the system while preserving plasma confinement efficiency, enabling the device to be directly mounted onto vacuum chambers of various shapes and orientations.

This configuration not only facilitates versatile application but also allows for greater control over film composition by adjusting the alloy ratios in the target material. As a result, high performance NEG films with optimized properties can be reproducibly formed. This compact and adaptable approach significantly broadens the practical usability of NEG technology in achieving ultra-high vacuum environments for next generation accelerator and advanced vacuum technologies.

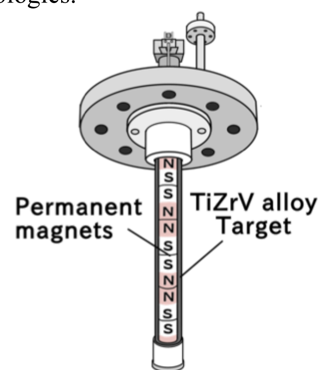

Figure 1: Compact NEG-coating device.

EXPERIMENT

Equipment

A schematic diagram of the equipment for experiment shows in Figure 2. We use DN63 SUS304 cross tube, shown in Figure 3, for a coating duct and mounted the compact device at the top of the tube. The ultimate pressure of the NEG-coated duct is measured by triaxial cold-cathode gauge (CCG) with an accuracy of up to 10^{-9} Pa order. For

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the film evaluation, we introduced two SUS304 sample ribbons inside of the tube, one is located in the vertical upper side, named "TOP sample", and another one is in the horizontal right side (from the direction of Figure 3), named "SIDE sample". In the introduction section of Kr gas, which is the sputter gas, the inflow gas volume is controlled by a piezo valve, and the exhaust volume of the Kr gas was fine-tuned by a manual valve and pumped into a turbo molecular pump.

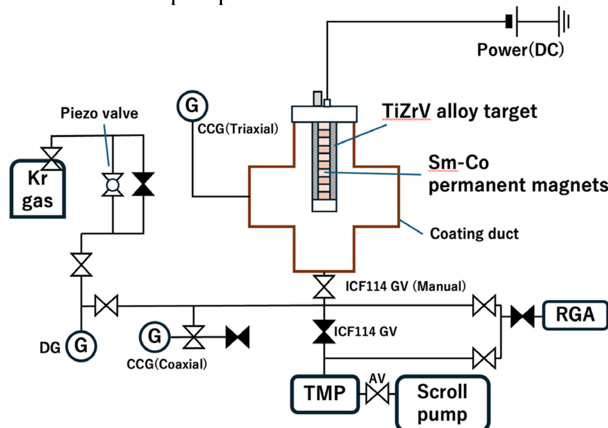

Figure 2: Experiment equipment.

Figure 3: DN63 coating duct.

Sputtering

To reproducibly deposit high performance NEG films, precise control of the plasma state is critical. Since sputtering parameters directly affect plasma stability [2], we systematically explored conditions that yield stable and uniform discharges. Using our compact NEG-coating device, we optimized these parameters to form TiZrV films on the inner surface of an DN63 cross tube. The selected condition served as the tentative optimal settings for achieving consistent NEG film performance. The cross tube was baked at 150 °C for 18 hours prior to sputtering, resulting in a system pressure of 8.5×10^{-7} Pa. The optimal sputtering conditions we obtained are; 100 ℃ for the substrate temperature, CV mode for the operation, 2.0 Pa for the sputtering pressure, 19 ~ 25 W for the power, 225 V for the voltage, and 84 ~ 109 mA for the current, and the deposition time was 2 hours 30 minutes (Figure 4).

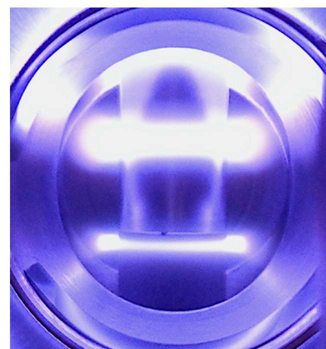

Figure 4: Plasma during sputtering.

Activation

Activation is a necessary step to make the NEG film function as a vacuum pump. Right after depositing TiZrV alloy film, NEG film has an oxide layer on its surface, so it is needed to diffuse such element into the film. We activated the NEG by heating to 200 °C for 18 hours. The ultimate pressure we measured in the duct reached to 1.7×10^{-9} Pa.

RESULT

We evaluated the NEG films deposited on TOP and SIDE samples with Scanning Electron Microscope (SEM), Energy Dispersive X-ray Spectroscopy (EDX), and X-ray Photoelectron Spectroscopy (XPS).

SEM, EDX

A high-performance NEG film is generally characterized by fine crystalline grains (typically up to 20 ~ 30 nm) and a columnar structure in the cross section. Figure 5 (a) shows the surface morphology of the TOP sample after NEG-coating. Grain size analysis based on SEM images revealed grain diameters ranging from approximately 20 to 50 nm. The elemental composition obtained by EDS was $\mathrm{Ti:Zr:V} = 33:33:35$, which is relatively close to the ideal 1:1:1 ratio often targeted for optimal NEG performance. Figure 5 (b) presents the cross-sectional SEM image of the TOP sample. Although the image quality is not sufficient to clearly identify a columnar structure or measure the film thickness precisely, we estimate the thickness to be approximately 530 to 700 nm, corresponding to a deposition rate of about 200 to 300 nm/h. Figure 5 (c) shows the surface morphology of the SIDE sample. SEM-based grain analysis indicated grain sizes of about 15 to 45 nm, again within the desired range for high-performance NEG films. The EDS analysis yielded a composition of $\mathrm{Ti:Zr:V} = 33:33:34$, which is also close to the ideal 1:1:1 ratio. Figure 5 (d) illustrates the cross section of the SIDE sample. The film thickness is estimated at approximately 630 nm, corresponding to a deposition rate of around 200 to 300 nm/h. However, cross-sectional observation was limited due to damage during sample preparation. Cutting the long ribbon substrate with metal scissors caused partial delamination of the film. To improve this, we plan to use properly sized silicon substrates for clearer imaging and analysis.

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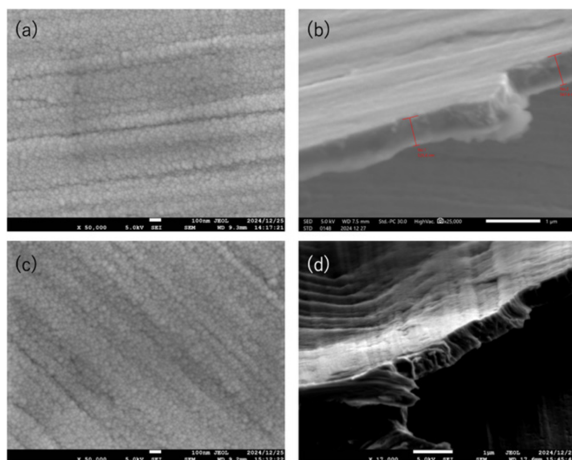

Figure 5: Fine grain on the surface of TOP sample (a), cross section of TOP sample (b), fine grain on the surface of SIDE sample (c), and cross section of SIDE sample (d).

XPS

XPS measurements were conducted on the TOP sample to evaluate activation behaviour at room temperature, 160 °C, 200 °C, and 250 °C. S XPS results indicated that the NEG-coating began to show signs of activation at temperatures as low as 160 °C. Figure 6 (a) to (c) show the spectral changes for Ti, Zr, V and O elements, respectively. Figure 6 (d) presents the temperature-dependent reduction of the O 1s peak area, indicating oxygen diffusion into the film. From room temperature to 250 °C, the O 1s signal decreased by approximately 55%, suggesting progressive activation. The TiZrV alloy target tends to produce an amorphous film structure, which contributes to achieving a relatively low activation temperature.

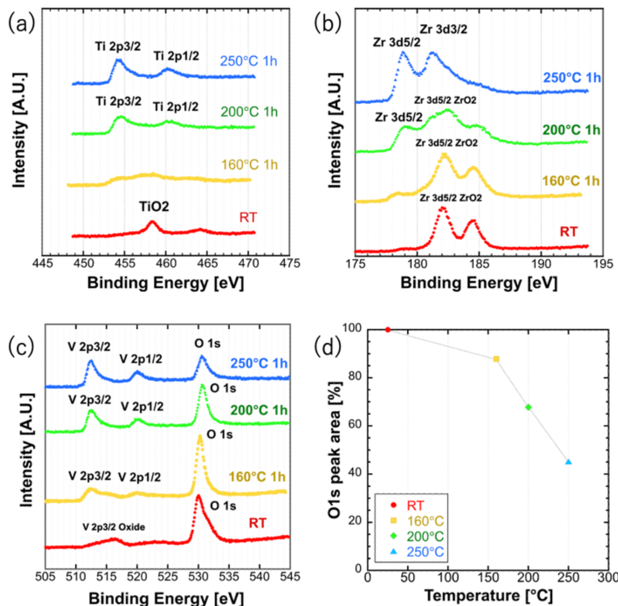

Figure 6: XPS results of Ti (a), Zr (b), V and O (c), and reduction in O1s peak area on TiZrV film as a function of activation temperature.

MOLFLOW+ SIMULATION

We modelled the experimental vacuum setup using Molflow+ to better understand the influence of gas flow on plasma stability. Based on experimental observations, we hypothesized that adjusting the conductance to the turbo molecular pump could help stabilize the plasma. To test this, we simulated the effect of introducing an orifice gasket at the pumping port. The simulation indicated that an orifice diameter of 2 ~ 3 mm would be effective for achieving stable plasma and high-quality NEG deposition in this equipment (Figure 7).

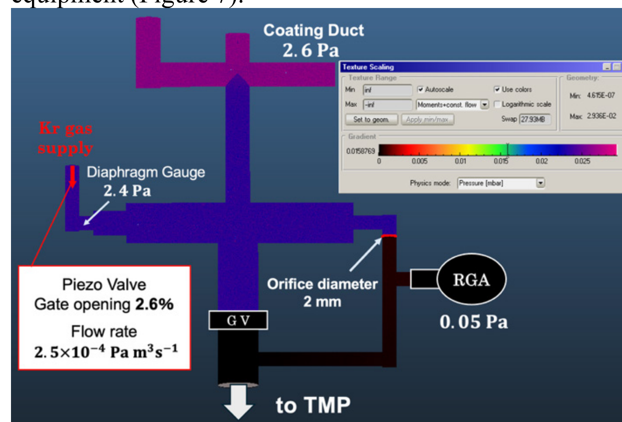

Figure 7: Sputtering gas flow by Molflow+ simulation.

CONCLUSION

Using the compact NEG-coating device, we identified deposition conditions that yield high-performance NEG films. Under the tentative optimal settings, crystal structures typical of high-performance NEG were partially observed, and the ultimate pressure reached 1.7×10^{-9} Pa in the coating duct, promising for accelerator applications. Further optimization of sputtering conditions will enable reproducible deposition of such films through stable plasma control.

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