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PROGRESS OF WALS NEG COATING EQUIPMENT AND TECHNOLOGY*

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Abstract

The objective of WALS (Wuhan Advanced Light Source) is to establish a world-class radiating light source. For the entire storage ring vacuum vessels, chromium-zirconium-copper has been selected as the primary material. Additionally, the magnetron sputtering (PVD) process has been employed to apply NEG (Non-Evaporable Getter) coatings to the inner surfaces of the copper vacuum chambers. This coating process enhances the vacuum performance.

Currently, the coating laboratory has taken shape and includes various components such as a standard cleaning platform, a coating platform, an ultimate vacuum test platform, an extraction rate test platform, and a coating microstructure test process. In terms of coating equipment, a bias power supply and customized ceramic components have been integrated to provide additional functionality. Multiple electrode control is utilized to manage different target materials, and experiments are conducted to deter-mine the composition of multilayers for various deposition ratios. Furthermore, sample tube bias control access is maintained during the coating process, and diverse combinations of target materials and bias parameters have been thoroughly investigated. Coating is presently in progress, and specific test results are underway.

INTRODUCTION

Process Difficulty Introduction

The 1.5 GeV storage ring vacuum system designed by WALS has a circumference of 180 m. According to the characteristics of physical design, the storage ring vacuum system was divided into 8~cells (standard segment) and 8 straight segments of 6.8 m. Chromium-zirconium-copper was chosen as the primary material for the entire ring vacuum vessel [1, 2]. At the same time, 316L was selected as the material for the pumping, bellows unit, and BPM mechanical shell.

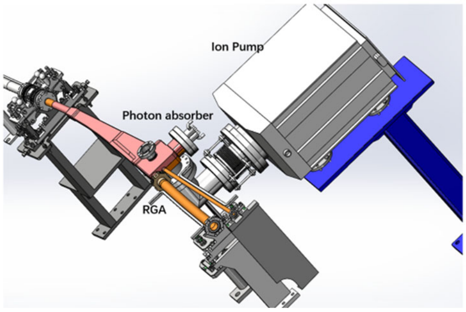

Figure 1: Model of vacuum chamber (Super Bend composite magnet).

One of the major challenges of WALS vacuum system design is magnet clearance small aperture (14 mm). The vacuum chamber (Super Bend composite magnet) has an oval profile with an inner diameter of 30 × 12 mm, a wall thickness of 1 mm, and a front chamber structure, which is a vacuum chamber about 1.8 m long, where the bending angle at the bipolar magnet is 10.8°, and the reverse bending is 1°, as shown in Fig. 1. The corresponding sample tube with the NEG coating [3] deposited on the inner wall is shown in the *Technical Status* section.

COATING MACHINE EQUIPMENT

Currently, the coating laboratory has taken shape and includes various components such as a standard cleaning platform, a coating platform, an ultimate vacuum test platform, an extraction rate test platform, and a coating microstructure test process, as shown in Fig. 2.

Figure 2: The coating laboratory.

Figure 3 shows the schematic diagram of the vacuum coating system [4]. The copper alloy pipe to be plated reaches vacuum through the flange and the auxiliary vacuum box at the lower end, and an appropriate amount of high-purity krypton gas is injected into the pipe as the discharge gas. The sputtering cathode target is made of a wire wound with a diameter of 1 mm. The end of the sputtering cathode target is fitted with a ceramic sheet to ensure insulation from the inner wall of the pipe. The magnetic field is provided by an external solenoid coil, which generates an adjustable magnetic field of 0.03 – 0.08 T at the central axis.

Three feasible research directions for NEG coating have been pursued:

1. Different target materials were controlled by means of multi-electrode control, while experiments were performed on deposited compositions of different ratios of multilayers,

2. Sample tube bias control access during the coating process, additional control parameters, and different crystal types of multilayers have been completed,
3. Multiple combinations of target materials and bias parameters have been investigated for this technique.. Research on the different components of the multilayer NEG coatings is currently underway, and specific test results are in progress.

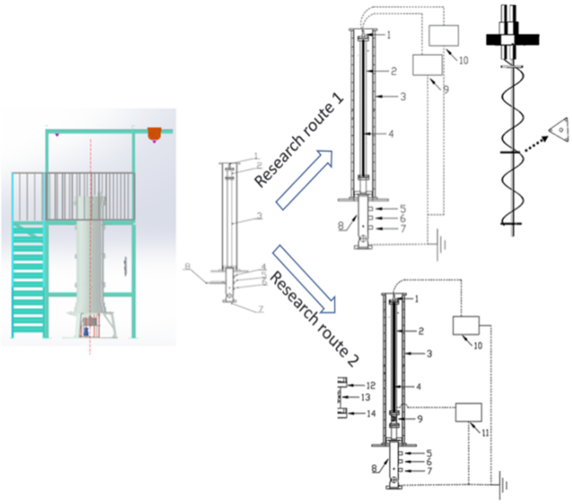

Figure 3: Te schematic diagram of the vacuum coating system.

TECHNICAL STATUS

Generally, the thickness of the plated pipe coating was controlled within 800~1500 nm, and the ratio of Ti:Zr:V atoms was maintained at 1:1:1.

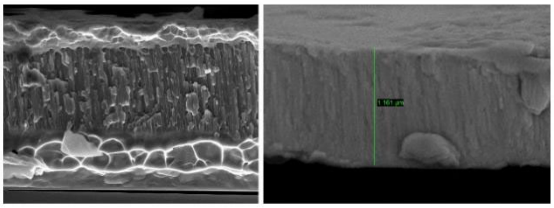

Figure 4: The section structure of NEG coating.

At an early stage, experimental work was carried out for the study of pipeline coating technology. By controlling relevant coating parameters, NEG coating [5] with a stable structure and good extraction performance was obtained. The morphology and mechanical characteristics of NEG coatings were tested using utilizing scanning electron microscope and nano-indentation. As can be seen from Fig. 4, the surface of the NEG coating obtained is smooth, the lattice distribution is uniform, and the thickness is about 1.1 µm.

Figure 5: Measured number of limiting vacuum.

The NEG coating has a smooth surface, a fairly uniform lattice distribution, and a thickness of around 1 µm. Nano-indentation test coating adhesion was 17 N. With 180 °C high temperature roasting activation, in the case of no external pump maintained for half a year, NEG ultimate vacuum coating of pipe was 7.43 x10^{-9} Pa, as shown in Fig. 5.

Table 1: Current NEG Coating Pumping Rate

	$\alpha(H_2)$	α (CO)	Q (CO)
180 °C 24 h	0.0041	0.1125	0.0201 mL

A 32 mm straight round tube with an inner diameter of 700 mm (**C** in Fig. 6) was baked and activated by 24 h at 180 ℃ standard vacuum. The viscosity coefficient of the NEG coatings after the first activation are shown in Table 1.

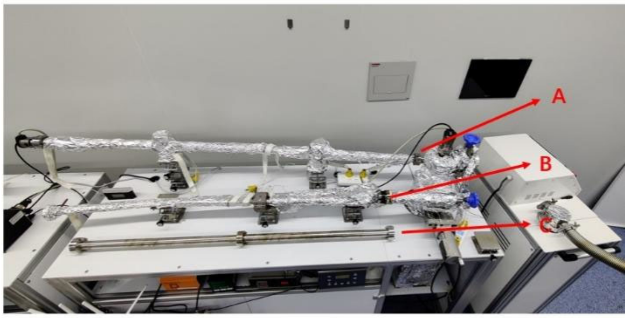

Figure 6: Typical sample tube.

Custom ceramics were used to ensure shielding and securing of the target wires in the chamber of the Super Bend composite magnet, and similar sample coatings were carried out, as shown in **B** in Fig. 6, **A** is a 1.9 m long circular tube vacuum chamber with a bending angle of about 6° and an inner diameter of 32 mm, which is characterized by two small pump ports with grilles and a 1° reverse twist.

Currently, the three typical vacuum chamber structures in Fig. 6 have been coated.

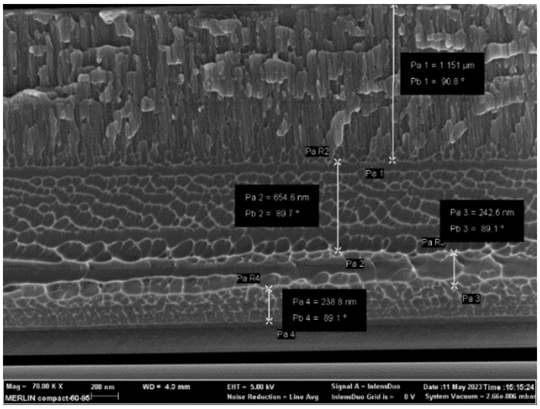

Figure 7: The section structure of NEG coating.

In the course of investigating the NEG coating process, WALS introduced a minor modification to the coating equipment. This entailed the addition of a small ceramic component to insulate the sample tube, isolating it from both the bottom transition vacuum chamber and the target

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point. This adjustment was made to create a suitable vacuum environment for the sample tube. Concurrently, an additional bias power supply was integrated to establish an electrical connection with the sample tube.

During the coating process, an additional bias control was introduced for the sample tube, supplementing the conventional coating power supply that provides the target wire. As depicted in Fig. 7, the SEM (Scanning Electron Microscope) image of the NEG coating section on the silicon wafer can be broadly categorized into three layers: the binding layer, the dense structure layer, and the columnar structure layer.

A noteworthy observation is that, under the same coating DC power supply parameters applied to the compact layer, elevating the bias voltage of the sample tube by 101V induces a notable transition from a dense structure to a distinct columnar structure. This phenomenon can be attributed to the additional bias effects, where electron bombardment of the inner wall leads to heating of the sample tube. Simultaneously, positive ions in the filtered plasma bombard the inner wall, reducing the energy of the metal particles within the coating, thus resulting in the transformation into a columnar structure.

CONCLUSION

WALS has established an experimental platform for NEG coatings, successfully conducting experiments on sample tubes of different sizes within the storage ring.

These experiments have resulted in improved NEG coating properties, with viscosity coefficients of H_2 0.0041 and CO 0.1125. A single activation process yields a CO volume of 0.02016 mL. Notably, the coating machine has been enhanced to introduce additional bias control, enabling the creation of a unique columnar structure.

REFERENCES

[1] G. Cattenoz *et al.*, "Vacuum Acceptance Tests for the UHV Room Temperature Vacuum System of the LHC during LS1", in *Proc. IPAC'14*, Dresden, Germany, Jun. 2014, pp. 2357-2359. `doi:10.18429/JACoW-IPAC2014-WEPME041`

[2] O. B. Malyshev and R. Valizadeh, "Further Optimisation of NEG Coatings for Accelerator Beam Chamber", in *Proc. IPAC'14*, Dresden, Germany, Jun. 2014, pp. 2399-2402. `doi:10.18429/JACoW-IPAC2014-WEPME056`

[3] P. H. Nallin *et al.*, "The Sirius Heating System for the In-situ NEG Activation", in *Proc. IPAC'19*, Melbourne, Australia, May 2019, pp. 4109-4111. `doi:10.18429/JACoW-IPAC2019-THPTS004`.

[4] M. M. Dehler et al., "Characterization of NEG Coatings for SLS 2.0", in *Proc. IPAC'19*, Melbourne, Australia, May 2019, pp.1662-1665. `doi:10.18429/JACoW-IPAC2019-TUPGW108`

[5] R. Sirvinskaite, M. D. Cropper, A.N. Hannah, O.B. Malyshev, R. Valizadeh, and S. Wang, "Electron Stimulated Desorption from Cryogenic NEG-Coated Surfaces", in *Proc. IPAC'19*, Melbourne, Australia, May 2019, pp. 2193-2196. `doi:10.18429/JACoW-IPAC2019-TUPTS114`