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PUMPING PROPERTIES OF SINGLE METAL ZIRCONIUM NON-EVAPORABLE GETTER COATING

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Abstract

Non-evaporable getter (NEG) coating has been used for years in many particle accelerator facilities due to its evenly distributed pumping speed, low thermal outgassing, and low photon and electron stimulated desorption yields. We have previously demonstrated that quaternary Ti-Zr-Hf-V coating deposited from an alloy wire has the lowest desorption yields, the highest sticking probability and sorption capacity. In this work, we explore the single element targets which are widely available and can be produced in a form of a wire that is easy to apply for a uniform coating of various shapes of vacuum chamber. Single metal Zr coatings have been tested to find a more efficient and cheaper way of producing the NEG-coated vacuum chambers. Two samples coated with Zr of dense and columnar structure were analysed and results of the pumping properties are reported. The results show that pure Zr coating could be an economic solution, despite not being as effective as can be achieved with quaternary NEG film. It shows that columnar Zr coating can be activated and reaches full pumping capacity at 160°C. This is close to the activation temperature of Ti-Zr-Hf-V and lower than that for the widely used ternary Ti-Zr-V alloy.

INTRODUCTION

The non-evaporable getter (NEG) coating was invented at CERN [1-5] and has been shown to have characteristics that enable this type of film to be used for distributed pumping along extended and narrow accelerator chambers, reducing the number of pumps needed to reach UHV pressures. However, one of the biggest disadvantages of NEG coating is that it needs to be activated at temperatures greater than 150 °C, making it difficult to use in beamlines that must be operated at cryogenic temperatures or have parts sensitive to high temperatures. Another downside is the complicated deposition of the coating. TiZrHfV has the lowest activation temperature for any known NEG coating, must be deposited from an alloy wire or a twisted wire. As this process is costly and time consuming, new coatings have been tested to minimise the activation temperature, along with creating a film that is easier to be deposited. Zr is known to have high oxygen solubility and has been used for compound NEG, either ternary TiZrV or quaternary TiZrHfV. In this paper single metal Zr coating was tested to find its activation temperature and pumping capacity.

DEPOSITION

Two samples were deposited from a pure Zr target. The substrates were 316LN stainless steel tubular samples with a length of 50 cm and an inner diameter of 3.8 cm equipped with CF40 flanges on both sides. The deposition facility is described in ref. [6]. A pure Zr target in a form of a 1-mm diameter wire was used for the deposition. After installing the sample substrate, the deposition facility was baked at 150 °C for four days. After cooling down, the pressure in the system was approximately 2×10^{-9} mbar. The deposition parameters are described in Table 1. The first sample was coated with dense Zr coating; the second had a columnar structure.

Table 1: Deposition Parameters

Parameter	Dense	Columnar
Gas	Kr	Kr
Pressure	10^{-3} mbar	0.1 mbar
Power supply	Pulsed DC	DC
Temperature	90-110 °C	90-110 °C

EXPERIMENTAL FACILITY

The substrates were transferred to the NEG-characterisation facility in ASTeC, STFC Daresbury laboratory [6-8] and experiments performed. To find the sticking probability of the NEG material, various gases, e.g. H_2, CO and CO_2 were injected in the system through a gas injection line at the bottom of the NEG tube. Pressures were measured at the top and bottom of the tube by using residual gas analysers (RGAs). The ratios of partial gas pressures were calculated and used for the sticking probability and pumping capacity measurements. During the gas injection, the valves to the other pumps were closed, therefore we observed the pumping effect of the NEG film only.

Experimental Procedure

The following experimental procedure for the NEG activation was applied:

- System bake-out to 200 °C for 24 hours, keeping the NEG-coated sample at approximately 80 °C. This is necessary to outgas the non-coated chamber parts;
- System cool-down to the room temperature;

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- Heating the NEG-coated tube to the temperatures in the range from 140 to 300 °C for 24 hours. Pumping sites become available during the activation as NEG film is desorbing;
- System cool-down to room temperature and left overnight;
- Short (3-5 min) H_2 injection (to minimise NEG saturation) recording partial pressures with two RGAs;
- Injection of a mixture of CO (90%) and CO_2 (10%) until full saturation, recording partial pressures with two RGAs;
- NEG reactivation to the next temperature.

MOLFLOW SIMULATIONS

To obtain NEG coating sticking probabilities from the pressure measurements, test particle Monte Carlo modelling of the experimental facility was performed using Molflow software [9]. It was critical to have the correct geometry for the simulations since for high sticking probabilities the number of particles that made it through the tube was low – therefore the effect of modelling the top RGA incorrectly was very noticeable in the preliminary results before building the RGA precisely. The molecular flow along the NEG coated tube was determined for a discrete number of sticking probabilities ranging from 10^{-4} to 1. Figure 1 shows simulation results for the pressure ratio of the top and bottom RGA partial pressure measurements as a function of sticking probability. These results were used for converting the partial pressure measurements to sticking probabilities.

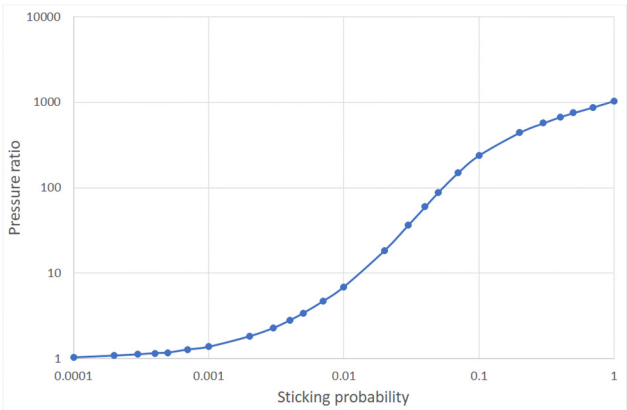

Figure 1: Molflow simulations of the pressure ratio between the top and bottom RGAs as a function of sticking probability for NEG-coated tube with diameter of 3.8 cm and length of 50 cm.

EXPERIMENTAL RESULTS

The sticking probabilities were found for both dense and columnar NEG coating activated at temperatures ranging from 140 to 300 °C and shown in Fig. 2. The sticking probability reaches its maximum value when the columnar coating is activated at 160 °C, while the dense coating does not reach the same value even when heated to 250 °C.

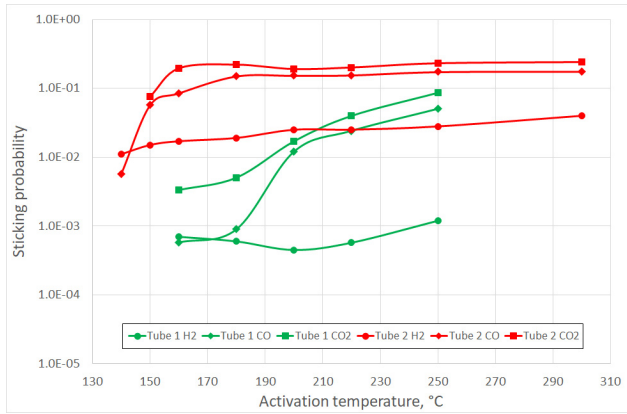

Figure 2: Sticking probabilities for the dense (green) and columnar (red) NEG coating as a function of activation temperature.

The CO pumping capacity was also calculated for both coatings. Figure 3 shows that full capacity for the CO gas is reached for the columnar NEG coating activated at 160°C. The dense coating does not reach the same capacity – there are not as many pumping sites available in the dense structure.

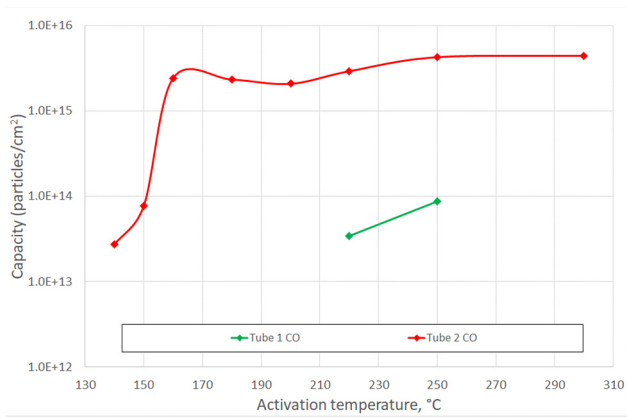

Figure 3: CO pumping capacity of dense (green) and columnar (red) Zr coating as a function of activation temperature.

DISCUSSION

The pumping properties of dense Zr are lower than columnar Zr over the whole range of activation temperatures measured. Fig. 2 shows that there was no pumping observed after the activations to 140 and 150 °C. After the activation to 160 °C, the sticking probability of H_2 for the dense Zr is approximately two orders of magnitude lower than for the columnar Zr film and remains approximately the same for higher activation temperatures. The sticking probability of CO and CO_2 for dense Zr increases with temperature but does not reach the value for the columnar Zr film at temperatures up to 250 °C. The CO pumping capacity of the dense coating is negligible after heating to 140 – 200 °C. After activation to higher temperatures (220 – 250 °C), the CO pumping capacity increases but does not reach 1 monolayer sorption capacity.

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The performance of the columnar Zr is much better than that of the dense coating. Partial activation and some pumping can be observed after heating to 140 °C. Sticking probabilities and CO sorption capacity reach the maximum values after the activation to 160 °C and do not change for higher activation temperatures. The CO pumping capacity reaches ~3 monolayers after the activation to 160 °C and remains 3 – 5 for higher activation temperatures.

Figure 2 shows that the increase of H_2 sticking probability with activation temperature is not as noticeable as the increase in CO sticking probability. The significant difference in behaviour of dense and columnar NEG films can be explained by morphological and structural differences in both films. The pumping properties of the dense Zr film likely represent the intrinsic properties of Zr on a geometrically flat surface. We can assume that the intrinsic sticking coefficient is the same for both structures. However, the columnar structure consists of long pumping channels (similar to Faraday cups), where the intrinsic sticking coefficient of Zr wall is combined with the high aspect ratio of the length of the pumping channel to its cross-section. This explains high sticking probability and high pumping capacity of the columnar structure, defined by greater physical surface area available for pumping.

Comparing the measurements presented in this paper to those previously reported, the pumping properties of columnar Zr film are quite similar to those of quaternary TiZrHfV alloy [10]. The sticking probability of H_2 is twice that of quaternary alloy. The CO sticking probability is at least two times lower than for the TiZrHfV. Therefore, it is a cheaper alternative to the widely used TiZrV and advanced TiZrHfV.

The single metal target enables deposition of a vacuum chamber with a small inner aperture. Single wire targets have a diameter of 1 mm or less. It can be stretched easier and guarantee no electrical contact with the vacuum chamber walls. In comparison to this, twisted wire targets take up more space and cannot provide a uniform coating. The uniformity of the coating is not an issue for the alloy targets, but they are usually as wide as the twisted wires, 3 mm in diameter.

CONCLUSIONS

Pumping properties of dense and columnar single metal Zr coatings have been measured. The columnar Zr NEG coating has demonstrated good pumping properties with an activation temperature as low as 160 °C and CO capacity of a few monolayers. This is comparable to the results achieved with quaternary TiZrHfV coating and better than the properties of the ternary Ti-Zr-V film. While the sticking probability and CO pumping capacity of the dense film are considerably lower than that of columnar film, single metal columnar Zr coating is a good candidate for the coating of accelerator vacuum chambers due to the wider availability of a single metal target and a lower cost of Zr, compared to Hf and V or alloys. Furthermore, the deposition process is simplified when single a metal is used.

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