MEASUREMENT OF THE SORPTION CHARACTERISTICS OF NEG COATED PIPES: THE TRANSMISSION FACTOR METHOD

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Abstract

ZrTiV Non Evaporable Getter (NEG) coatings of vacuum chambers have found application in particle accelerators to lower the gas pressure, during the operative conditions. For that, the characterization of the actual pumping speed of the NEG coating is a key issue. It is carried out by means of the dynamic sorption method according to ASTM F798-82 standard, conducted "offline" on a sample (coupon), suitably positioned inside the chamber to be coated and recovered after the process. To evaluate in-situ the sorption characteristics of getter coated chambers, a different measurement technique (Trasmission Factor Method) is here described. It is based on the measurement of pressures ratio at the inlet and the outlet of a coated pipe, under a flow of test gas. A calibration curve permits to evaluate sticking probability of the coated surface from the pressure ratio. The use of reference samples to calibrate the method is quite difficult. A better approach is a modellistic one, finding the dependency of pressure ratio on the average sticking probability, the pipe length, the section geometry and dimensions. Preliminary experimental results will be shown.

INTRODUCTION

In the last few years the interest of the particle accelerators community towards ZrTiV non evaporable getter (NEG) coatings of vacuum chambers has been continuously increasing. The final achievable pressure is basically limited by hydrogen outgassing from the internal walls as for any vacuum system. In the case of long pipes and vacuum chambers like those of particle accelerators, this kind of problem is particularly important. The use of a new distributed pumping concept in pipes started in 1995 at CERN for the LHC project [1]. CERN developed and patented the deposition technique and now it's commercially available under the brand name IntegraTorr® through the SAES Getters Group. Even if the basic properties of the sputtered getter films were extensively studied [2], less papers [3,4] described the pumping behaviour of real pipes or tubulation coated with the TiZrV getter film, where strongly anisotropic conditions are imposed. In fact, the measure of the actual pumping speed of the NEG coating is typically carried out by means of the dynamic sorption method according to ASTM F798-82 standard. This approach can be followed in case of favourable chamber geometry, but it is not applicable directly on NEG coated vacuum pipes. Normally the test is conducted "offline" on a discrete sample (coupon), suitably positioned inside the chamber to be representative of the coating process.

The transmission factor method has been described in previous works [5]. This is an experimental technique to evaluate in-situ the sorption characteristics of getter coatings. The method is based on the measurement of pressures ratio at the inlet and the outlet of a coated pipe, under a flow of test gas. A calibration curve permits to evaluate sticking probability of the coated surface from the pressure ratio. In this paper, the results relative to two different pipes will be presented, obtained by using the same experimental set up. Main issues related to the measurements are discussed.

EXPERIMENTAL SET UP

Fig.1 shows the proposed system layout. The test gas is admitted at one end of the coated pipe through two valves connected to CO and H2 cylinders, measuring the pressure p_2 at the inlet by the pressure gauge. The gas passes through the coated pipe and is absorbed by the wall. The pressure p_1 is measured at the outlet by another pressure (extractor) gauge.

The activation of the coated pipe is performed by increasing the wall temperature to 200 ˚C for 24-60 hours. After that, a sample gas is introduced by the injection valve. The p_2/p_1 ratio is related to the average sticking probability of the pipe walls, so providing a rough indication of the sorption performances of the getter film. The actual value of the pumping speed per unit surface can be determined by a calibration curve, calculated by a suitable model.

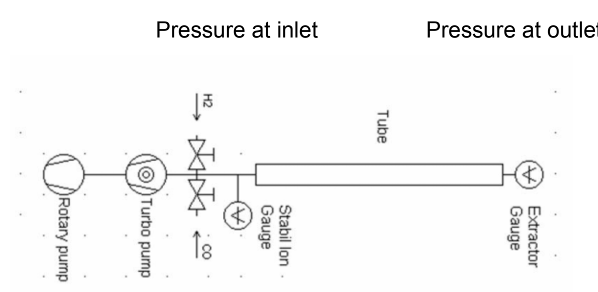

Fig.1 Experimental layout

In order to obtain meaningful results, the residual pressure due to the outgassing of components of the experimental set up must be limited. In particular, at the outlet, a background pressure not related to gases that are transmitted by the coated tube from the inlet must be taken into account. Furthermore, it is known that filament of the pressure gauges can catalyse H_2 in CH_4 that is not gettered by the TiVZr as observed in [6]. A QMS has been installed near the inlet only for testing the effect of the pressure gauges on the residual pressure and on the

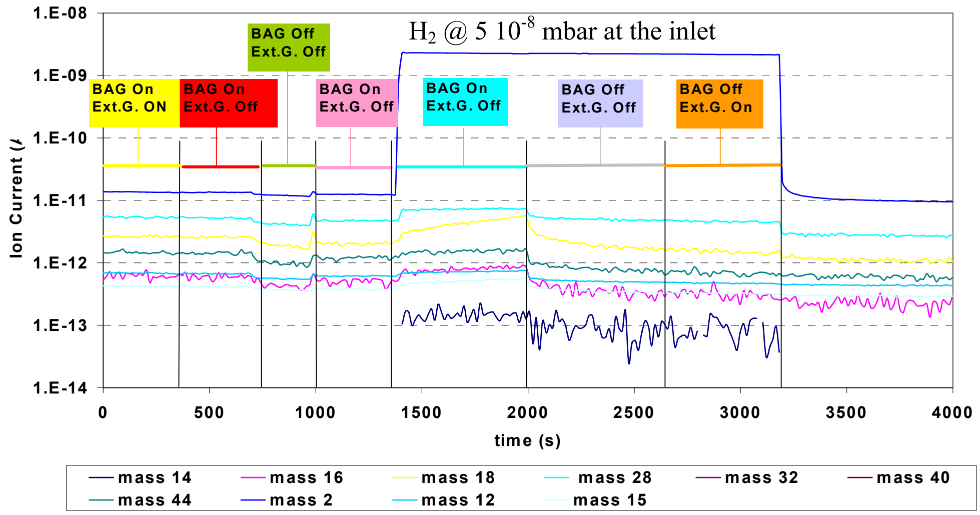

Fig.2 QMS test results. The gauges Bayard-Alpert (BAG) and extractor gauge (Ext.G.) have been alternatively switched on, with and without H_2 flux in.

CH_4 formation. Before the test the getter has been activated.
The variation of the signal related to the mass seems to be not too high to damage the validity of the test, as shown in Fig.2.

CALIBRATION CURVE

A relationship between the pressure ratio and the sticking probability of the coated pump is needed. A possible way to go would be to take measurements on calibrated samples of getter coatings with known pumping speed, varying in a given range, but this methodology is not easy.

A better approach is a modellistic one, applicable to a wider spectrum of situations, finding the dependency of pressure ratio on the average sticking probability, the pipe length and the section geometry and dimensions.

In a previous work [7], it has been shown that the one-dimensional approach, based on continuous differential equations, is not suitable to this need, in particular when the sticking coefficient is high and anisotropy of incident flux into the pipe start to be a dominant phenomenon. A three-dimensional approach is needed.

The method of angular coefficients [8] is based on a mass flux balances system of equations. The total surface is divided in n elementary surfaces, where the density of the total emitted molecular flux ν is assumed to be constant. For every element, labelled by the subscript i, we can write:

$$\nu_i = \nu_{0i} + (1-\varepsilon_i)\nu_{inc\,i} \qquad (1)$$

where $\mathbf{\nu_{0i}}$ is the molecular flux density generated by the i-th surface (e.g. the gas desorption flux), $\mathbf{\nu_{inc\,i}}$ is the flux density incident on the elementary area dF_i and ε_i is the adsorbed fraction of incident flux.

We can write $\mathbf{\nu_{inc\,i}}$ as:

$$\nu_{inc\,i}\, dF_i = \sum_{j=1}^{n} \varphi_{j->i}\, \nu_j\, dF_j \qquad (2)$$

where $\varphi_{j->i}$ is the mean angular coefficient from the j-th surface to the i-th surface and n is the number of elements.

Joining equation (1) and (2), we obtain a algebraic system of equation in the unknown $\mathbf{\nu_i}$ is obtained.

EXPERIMENTAL RESULTS

Two coated pipes with a length of 1.5 m and a diameter of 35 mm have been characterized. The thickness of the coating is 2 µm. Fig.3 shows the calibration curve for the specific geometry. H_2 and CO sticking coefficient has been investigated.

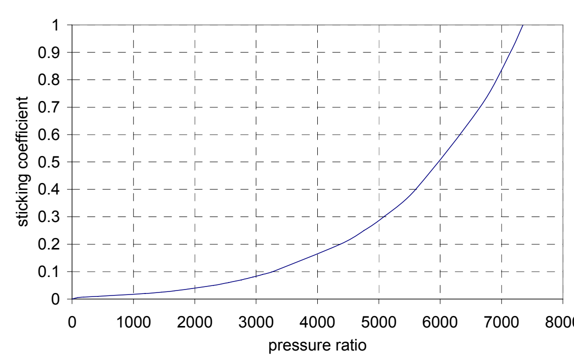

Fig.3 Calibration curve for 1 1.5 m long pipe with a diameter of 35 mm

Table 1 shows the tests carried out on the two pipes. The indication AISI 304 is related to a non coated pipe, the indication NEG to a coated one. The main parameters, controlled during the experiment, have been the activation temperature and time. Trend of performances for following activation at 200 °C for 24 hours has been the main aim.

Table1 Test conditions

Index	surface	Activation Temperature (ºC)	Activation Time (h)
ss.1	AISI 304	330	15
PIPE1.1	NEG	200	24
PIPE1.2	NEG	200	24
PIPE1.3	NEG	200	60
PIPE2.1	NEG	200	24
PIPE2.2	NEG	200	24
PIPE2.3	NEG	200	24
PIPE2.4	NEG	200	24

Another parameter controlled during the test is the throughput of the H_2 or CO introduced into the pipe. If the getter is far from saturation the sticking coefficient and then the measured pressure ratio must be not dependent on the throughput. This is observed in Fig.4.

Changing the flux, the inlet and outlet pressure changes, but not the pressure ratio determined by the sticking coefficient.

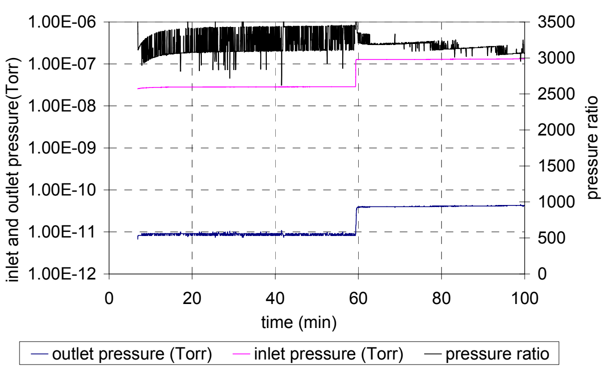

Fig.4 Trend of CO pressure at the inlet at the outlet and of the pressure ratio, by changing the throughput

Table 2 and Table 3 show the initial sticking coefficient measured for H_2 and CO in the subsequent tests.

Table 2 measured H_2 sticking coefficient

Index	initial H2 pressure ratio	H2 sticking coefficient
PIPE1.1	144	0.006
PIPE1.2	260	0.008
PIPE1.3	385	0.01
PIPE2.1	50	0.0028
PIPE2.2	95	0.0051
PIPE2.3	100	0.0052
PIPE2.4	100	0.0052

Table 3 Measured CO sticking coefficient

Index	initial CO pressure ratio	CO initial sticking coefficient
ss.1	10	5.70E-04
PIPE1.1	2800÷3000	0.07÷0.08
PIPE1.2	3300÷3400	0.11÷0.12
PIPE1.3	3600÷3700	0.13÷0.14
PIPE2.1	2800÷3000	0.07÷0.08
PIPE2.2	4000÷4210	0.161÷0.183
PIPE2.3	3000÷3100	0.08÷0.09
PIPE2.4	2800÷3000	0.07÷0.08

CONCLUSIONS

The transmission factor method is a very useful tool for characterization of the SNEG coated pipes. Measured pumping speed for H_2 agrees with previous work [2].

The initial CO sticking coefficient agrees with SAES values obtained with a roughly similar layout [6]. This data seems however lower than measurements obtained by CERN [2].

The calibration curve has been calculated using a suitable three-dimensional model. Furthermore, this can be applied to geometry more complex than cylinder with a circular section.

Repeatability and reproducibility of the test must be investigated, but the first two results for different pipes seem to give signals of good robustness of the tests.

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