# *In situ* characterization of a Ti-Zr-V non-evaporable getter film inside a 3 m long pipe using the Transmission Factor Method

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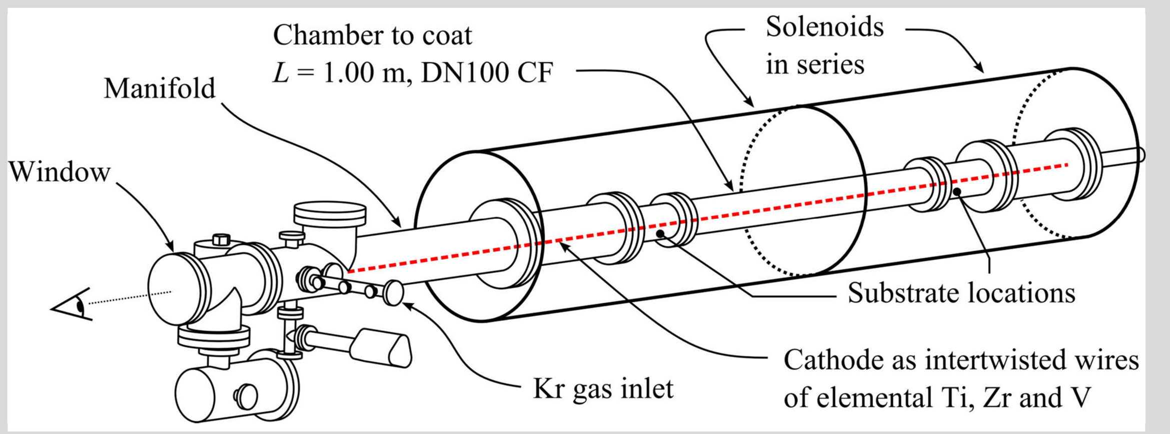
## Introduction

In the frame of the FAIR project, a new accelerator facility is being built at the site of the GSI Helmholtz Centre for Heavy Ion Research in Darmstadt, Germany. The current GSI facility will act as a heavy-ion injector to the new facility. A recent upgrade of the SIS 18 heavy-ion storage ring included the use of Ti-Zr-V non-evaporable getter (NEG) coatings, which have been developed by CERN and licensed by GSI [1]. Performance tests showed improvement in terms of beam intensity and beam lifetime due to contribution of the NEG [2]. This work has been conducted in context of the FAIR project to study the characteristics and performance of Ti-Zr-V NEG coatings for the future use.

## Experimental Setup

Three chambers with 1 m long each were coated successively with Ti-Zr-V using magnetron sputtering (Fig. 1). During the sputtering process, two 14 mm × 14 mm stainless steel substrates were placed inside the chamber close to the flanges. The coated substrates were then analyzed using scanning electron microscopy (SEM), energy-dispersive x-ray spectroscopy (EDX) and x-ray photoelectron spectroscopy (XPS). The results of the surface analysis are shown in Fig. 2 to Fig. 5.



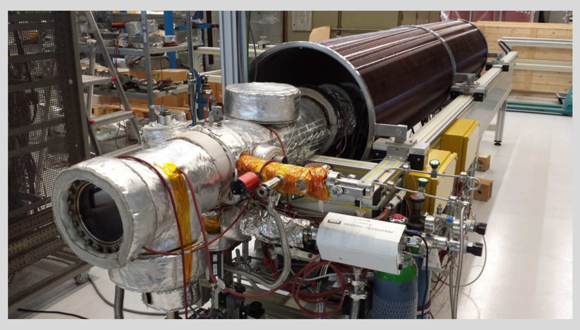



**Fig. 1:** Schematic drawing (left) and a photograph of the GSI magnetron sputtering facility (bottom).

### NEG coating parameters

- Substrate temperature 100 °C
- Discharge voltage -500 V
- Discharge current 0.12 A/m
- Mean Krypton pressure of $4 \cdot 10^{-3}$ mbar
- Mean sputter rate 0.156 µm/h
- Solenoid diameter 60 cm, length 166 cm, 332 loops
- Magnetic flux density ~180 G

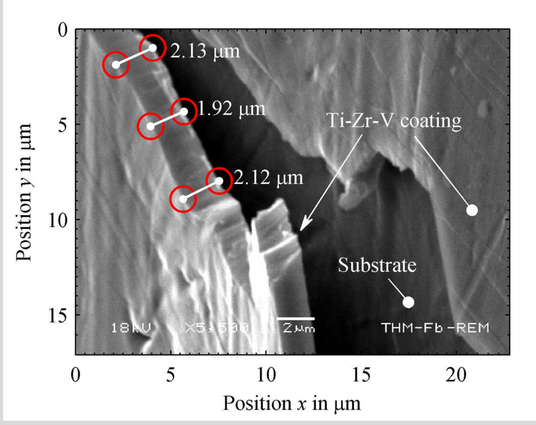


**Fig. 2:** SEM photograph showing an average of NEG coating thickness of about 2 µm. The sputter duration was 13.17 h.

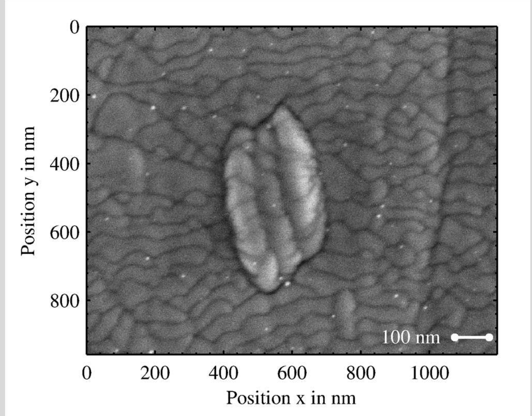


**Fig. 3:** The film surface morphology showing nanocrystalline structures. The size of the crystallites varies from 30 nm to >300 nm.

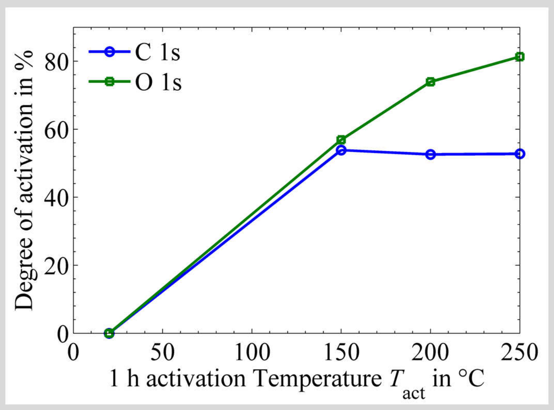


**Fig 4:** NEG activation results determined by XPS (left). The degree of activation is measured as the decrease of O 1s and C 1s peaks relative to the peak area at room temperature (RT).

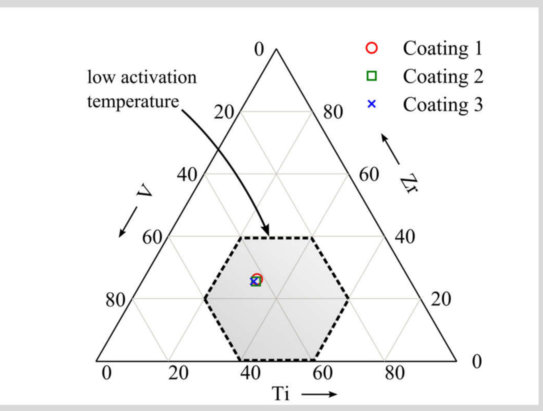


**Fig. 5:** The results of EDX analysis in a ternary diagram (right). The mean NEG composition is Ti 31 – Zr 26 – V 43 (at. %), which is in the range of compositions with good getter properties [3].

## Determination of Sticking Coefficient

The Transmission Factor Method will be used to evaluate *in situ* sorption properties of NEG coatings. A Monte Carlo simulation using MolFlow+ [4] was employed to obtain a correlation between the pressure ratios $p_{\mathrm{inj}}/p_{\mathrm{RGA}}$ and sticking coefficient $\alpha$. The sticking coefficient of the NEG coating was then determined by extrapolating the measured pressure ratios $p_{\mathrm{inj}}/p_{\mathrm{RGA}}$ to the simulation results (Fig. 9). The pressure ratios were measured for each gas ($H_2$, CO and $N_2$) independently at room temperature after activation. The activation was done by heating the NEG coating stepwise from 200 °C to 300 °C over a duration of 24 h (Fig. 8).

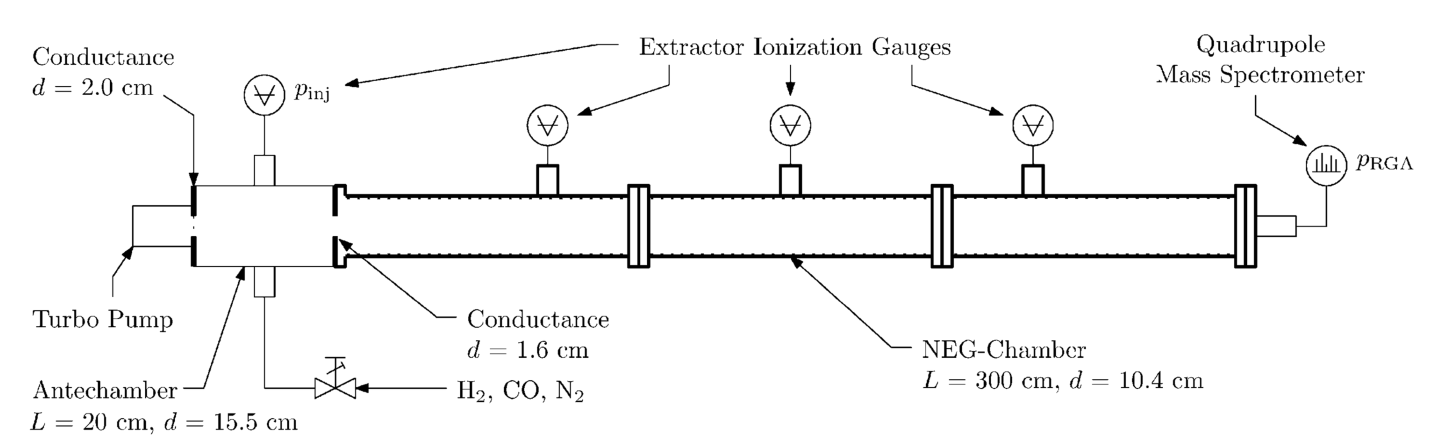



**Fig 6:** The experimental setup used for transmission and saturation measurements. $L$ is the length and $d$ is the inner diameter.

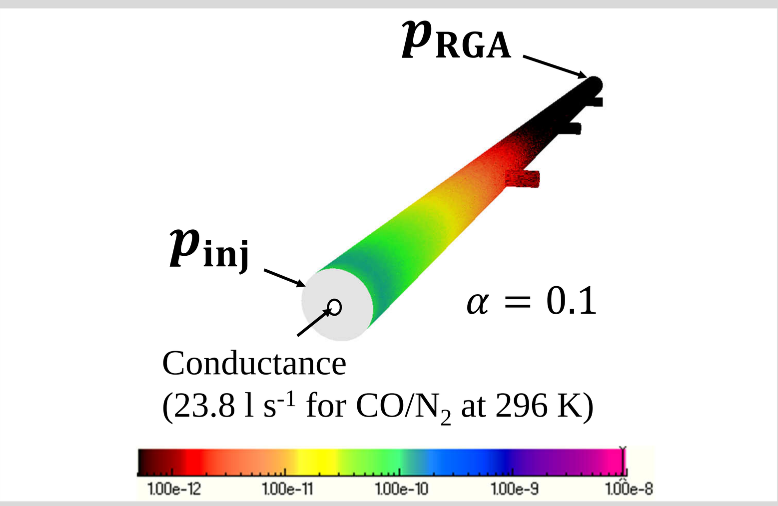


**Fig 7:** 3D model of the NEG chamber used in MolFlow+. The colors indicate the pressure inside the chamber.

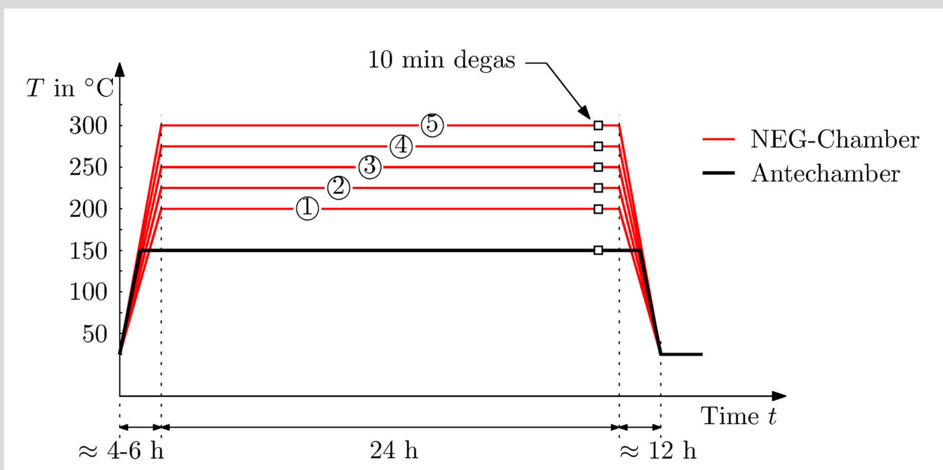


**Fig. 8:** Schematic representation of the NEG activation cycles. The activation procedure is adopted from the SIS 18 storage ring.

## Transmission measurement results

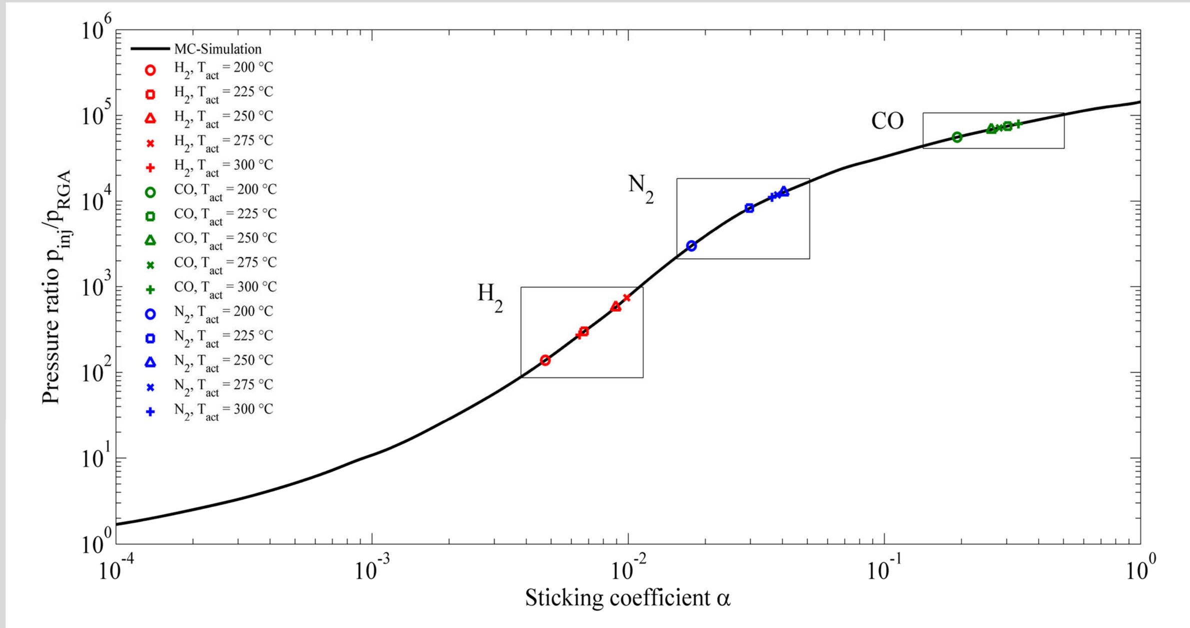


**Fig. 9:** Measured pressure ratios $p_{\mathrm{inj}}/p_{\mathrm{RGA}}$ extrapolated to the simulated curve to obtain the corresponding sticking coefficients $\alpha$.

The maximum sticking coefficients for $H_2$ ($9.9 \cdot 10^{-3}$) and $N_2$ ($3.8 \cdot 10^{-2}$) obtained by this method are close to those reported in [5], namely $8 \cdot 10^{-3}$ and $1.5 \cdot 10^{-2}$, respectively. Surface roughness and initial surface contamination may cause spreads between reported values. The maximum sticking coefficient of CO (0.33) is lower than 0.7 as reported in Ref. [5].

The activation temperature dependence for different gas species is shown separately in Figs. 10 (a) to (c).

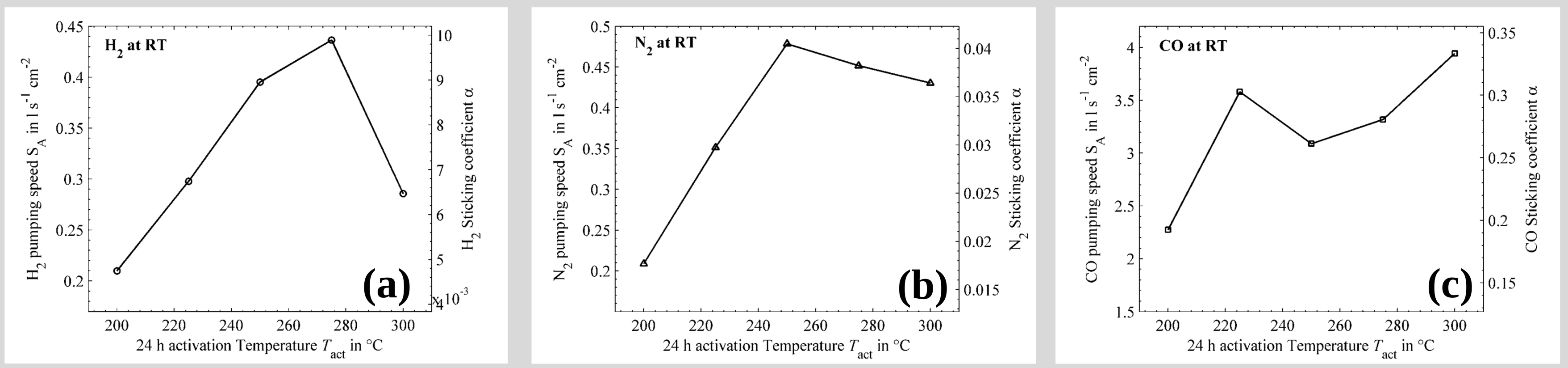


**Fig. 10:** The relationship between the 24 h activation temperature and the pumping speeds/sticking coefficients of different gas species at RT (a) $H_2$, (b) $N_2$, (c) CO. The pumping speeds are calculated from the relation $S_A = \alpha C$, where $C$ is the conductance of the aperture ($\ell\ \mathrm{s}^{-1}\ \mathrm{cm}^{-2}$). The pumping speeds of $H_2$ and $N_2$ in Fig. 10 improve with the increase of activation temperature, but decrease above 275 °C and 250 °C, respectively. The pumping speed of CO decreases from 225 °C and regenerates as the activation temperature rises. Similar behavior has been observed in [3].

## NEG film saturation experiment results

In this experiment, CO has been injected through the antechamber in order to saturate the NEG surface. During the injection, a pressure front moved slowly in axial direction of the NEG chamber with a mean speed of $v \approx 0.35$ m/h as illustrated in Fig. 11. The saturation measurement result is shown in Fig. 12. The actual data plots of Fig. 11 are shown in Figs. 13 and 14.

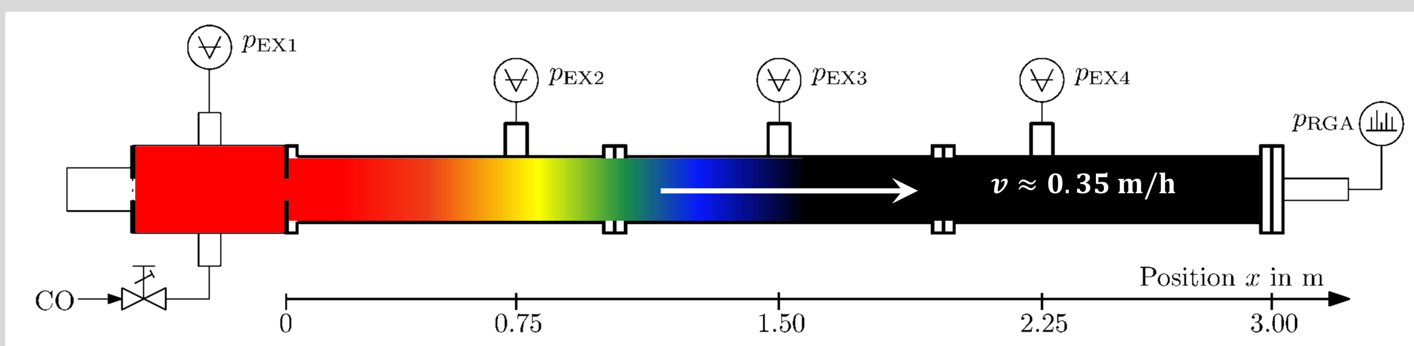



**Fig. 11:** Pressure gradient in the chamber after CO injection. The colors indicate the pressure gradient schematically.

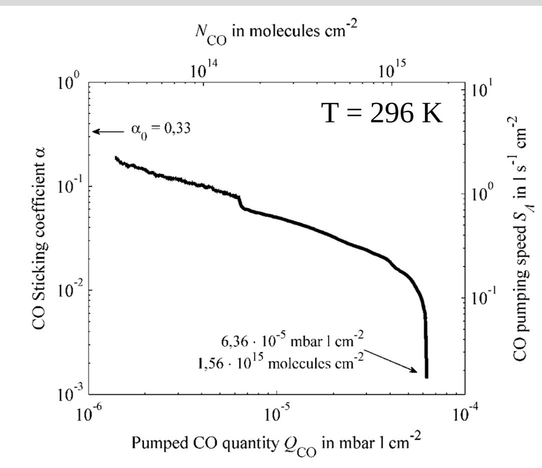


**Fig. 12:** Surface capacity of NEG coating as a function of the sticking coefficient. The surface capacity of the Ti-Zr-V NEG reported in [5] is $8 \cdot 10^{14}$ molecules/cm² compared to approx. $1.6 \cdot 10^{15}$ molecules/cm², respectively.

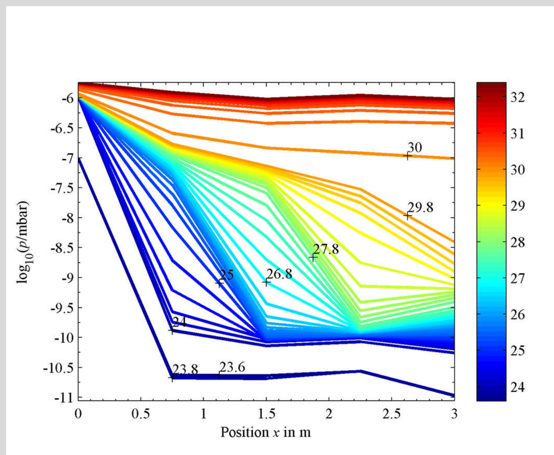


**Fig. 13:** Axial pressure profile during the saturation. The color bar indicates the time in h. The density of contour lines indicate the pressure front.

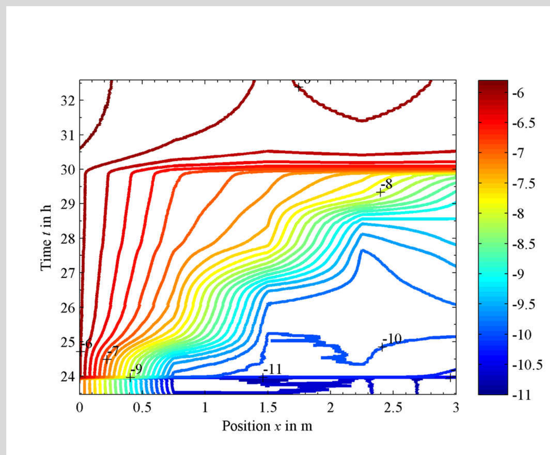


**Fig. 14:** Time dependent pressure evolution along the NEG chamber. The color bar shows $\log_{10}(p/\mathrm{mbar})$ $N_2$ equivalent. The wavy contour lines indicate the pressure front.

## Conclusions

- Surface analysis (SEM, EDX, XPS) of magnetron sputtered Ti-Zr-V NEG coatings have been conducted. The results show good properties in terms of elemental composition, surface morphology and activation behavior.
- Sticking coefficients for $H_2$ and $N_2$ determined by transmission measurements are comparable to those obtained in literature. In case of CO, there is a discrepancy which has to be investigated.
- The 24 h activation temperature and gas dependent gettering behavior is shown.
- The CO surface capacity from saturation experiments is in good agreement with the values reported in the literature.
- The pressure distribution inside the NEG chamber during the CO saturation is not uniform. A slow movement of saturated pressure front in axial direction has been observed.

## Acknowledgements

The author would like to thank to Dr. M. C. Bellachioma, J. Kurdal, Prof. Dr. H.-J. Eifert, Dr. P. M. Suherman and the rest of the GSI Vacuum Group who made this work possible. The SEM/EDX measurements were performed at the University of Applied Sciences Mittelhessen and GSI with kind help from Dr. F. Sakhibov and Dr. I. Alber, respectively. The XPS measurements were conducted by B. Garke of University Magdeburg.

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