NEG THIN FILM COATING DEVELOPMENT FOR THE MAX IV VACUUM SYSTEM

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Abstract

The new synchrotron radiation facility of the MAX IV Laboratory is under construction and expected to deliver the first light beam in 2016. To cope with the small aperture, the intense photon bombardment and the low-pressure requirement, most of the beam pipes for the 3-GeV ring are going to be coated with Ti-Zr-V non-evaporable getter (NEG) thin films. To take advantage from the experience acquired during the construction of the Large Hadron Collider (LHC), a collaboration between CERN and MAX IV Laboratory has been set up. The choice of the extruded Cu tubes, the preliminary surface treatments, the coating configuration and the performance validation of the small-diameter vacuum chambers have been addressed. In parallel, an intense development has been tackled at CERN for the coating of vacuum chambers where photon and electron beams circulate in separate pipes. The most important results of the collaboration are presented and future perspectives pointed out.

CHAMBER DESIGN

In order to meet specific requirements, most of the vacuum chambers for the MAX IV 3 GeV storage ring are small aperture tubes manufactured from Oxygen Free copper with addition of Silver (OFS). The tubes were extruded with two different internal diameters: 22 and 27 mm with 1 mm wall thickness. After the extrusion process they were cut in 3 m and 4 m long pieces respectively. Most of the vacuum chambers are bent by 1.5 or 3 degrees with 19 m bending radius whereas a few vessels employ an antechamber of small vertical aperture to allow for extraction of the synchrotron radiation to the beamlines [1]. All the chambers are foreseen to be NEG-coated. Due to their novel geometry, this is a challenging process that required validation. All needed trials and measurements were performed at CERN.

PREPARATION OF THE CHAMBERS FOR NEG-COATING

In order to ensure the NEG film adhesion to the OFS substrate and relying on the experience from the production of Long Straight Section (LSS) copper chambers now installed in the LHC [2], all the extruded tubes for MAX IV were subjected to similar surface treatments prior to the NEG-coating.

The vacuum chamber design of the 3 GeV storage ring involves complex bellows assemblies (that also include Radio Frequency (RF) fingers) welded at the extremities of almost each vessel. In such assemblies there is high risk of trapping cleaning agents. Therefore, the surface treatment was done as the first step of the manufacturing i.e. directly after the tube extrusion process.

Surface Treatments

All the copper tubes used for the production were subjected to three step surface treatment at CERN: degreasing utilizing standard CERN procedure used for Ultra High Vacuum (UHV) compatible pieces with additional steps of ethanol soaked rag cleaning and high pressure rinsing, etching with ammonium persulphate solution to remove up to 50 μm of material and passivation with chromic acid solution.

Figure 1: Series etching of the extruded copper tubes.

Before and after each step of the cleaning process all the tubes were visually inspected for any sign of contamination. In total about 8% of the extruded tubes were excluded due to strong contamination difficult to remove. After the treatments, all the tubes were individually packed and delivered to manufacturing.

NEG-COATING ADHESION TESTS

The final design of the vacuum chambers employs different copper machining techniques. The OFS copper tubes were extruded as described. However, short parts such as tapers and absorbers were made by Electrical Discharge Machining (EDM). Both types of surfaces had to be verified for the NEG-coating adhesion.

OFS Extruded Tubes

To verify the NEG film adhesion on the OFS extruded copper substrate, used for manufacturing of the vessels, two 3 m long vacuum chambers were prepared at CERN from OFS extruded tubes with the surface treatment described above. Both chambers were NEG-coated, thermally cycled and validated for any sign of the film

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non-uniformities or peel-offs. After 10 activation and 8 venting cycles, the chambers were disassembled from the test system and visually inspected. No sign of bad film adhesion was found.

EDM Elements

Several tests were done to justify that the EDM copper surfaces are compatible with the Ultra High Vacuum (UHV) environment and that they will provide good coating adhesion for the NEG film. A few small EDM samples were prepared, subjected to surface treatments, NEG-coated and analysed by X-ray Photoelectron Spectroscopy (XPS) for surface composition and activation behaviour. The results of the XPS showed the expected composition and good activation behaviour of the coating. Furthermore, one vacuum chamber was manufactured by EDM and its inner surface was characterized before and after the etching with pump down and outgassing. The results of the measurements indicate that the analysed EDM surface is UHV compatible and has low outgassing rates. The vacuum chamber was NEG-coated and awaits sticking factor measurements and thermal cycling tests.

COATING OF SMALL APERTURE VACUUM CHAMBERS

In order to validate the NEG-coating quality and its deposition method on small aperture vacuum chambers, the sticking factor for hydrogen of the film was evaluated. For this purpose, two 1 m long, 21 mm inside diameter stainless steel tubes were prepared and NEG-coated. The sticking probability of the film activated at 200^{0}C for 24 hours was assessed by the transmission method. The ratio of the pressures at injection and at the end of the tube (P_{inj}, P_{end} respectively) was measured and compared with a test-particle Monte Carlo simulation of the system. Simulation results relating the sticking coefficient to the pressure ratio for the studied tube are plotted in the Fig. 2.

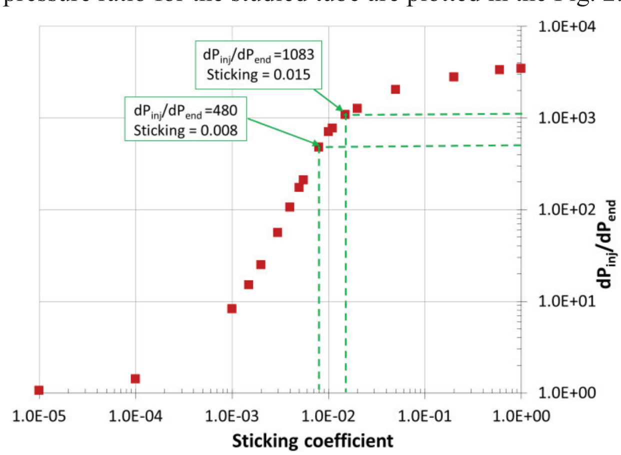

Figure 2: Monte Carlo simulations results for a 1 m long NEG-coated tube of 21 mm inside diameter.

The highest sticking factor obtained for hydrogen was in the range of 1.5×10^{-2}, which corresponds to the pressure ratio P_{inj}/P_{end}=1083 as highlighted in Fig. 2. The second point highlighted on the graph for which the sticking factor is 8×10^{-3}, was obtained for a pressure ratio P_{inj}/P_{end}=480 corresponding to the NEG-film after two additional venting and activation cycles (cycles of exposure to air at atmospheric pressure and activation at 200^{0}C for 24 hours). The values of both sticking coefficients are in accordance with published data [2].

COATING OF BENT VESSELS

In each of the 20 achromats of the 3 GeV ring, 7 out of 10 vacuum sections are of similar, bent geometry. Therefore, an important step in the development was to evaluate the feasibility of the NEG-coating by magnetron sputtering in small aperture, curved vacuum chambers. For this purpose, two stainless steel chambers of 21 mm inside diameter, total length 2.3 m, bending angle 3 degrees and bending radius 19 m were manufactured and NEG-coated at CERN. For the coating process, ceramic spacers were used to ensure that the cathode is centred. A simplified sketch of the chamber is presented in Fig. 3.

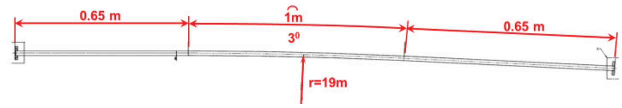

Figure 3: Bent chamber layout.

The estimated target film thickness of the first chamber was 1.3 µm. The coating thickness of two samples placed at the top and bottom of the chamber was measured by Scanning Electron Microscopy (SEM) and was equal to 0.7 µm and 1.2 µm respectively as shown in Fig. 4.

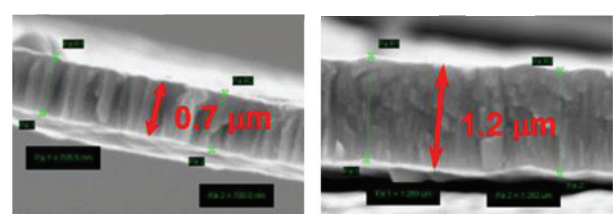

Figure 4: NEG film thickness measurement by SEM.

In order to verify the thickness distribution of the NEG film along the coated chamber length and perimeter, the tube was sliced every 10 cm and each ring was cut radially in four pieces. The samples were analysed by X-ray fluorescence (XRF) for thickness and composition of the film. The result of the XRF thickness measurement is presented in Fig. 5.

VESSEL WITH ANTECHAMBER

The most complicated vacuum chamber to be NEG-coated was the vessel with antechamber through which the synchrotron radiation travels out of the storage ring to the beamlines. This vacuum chamber is placed downstream of the first dipole magnet of each achromat. The chamber is 73.5 cm long and its shape, three main cross-sections and dimensions are presented in Fig. 6. Furthermore, the electron and photon beams are marked on the Fig. 6 with red and blue colours respectively.

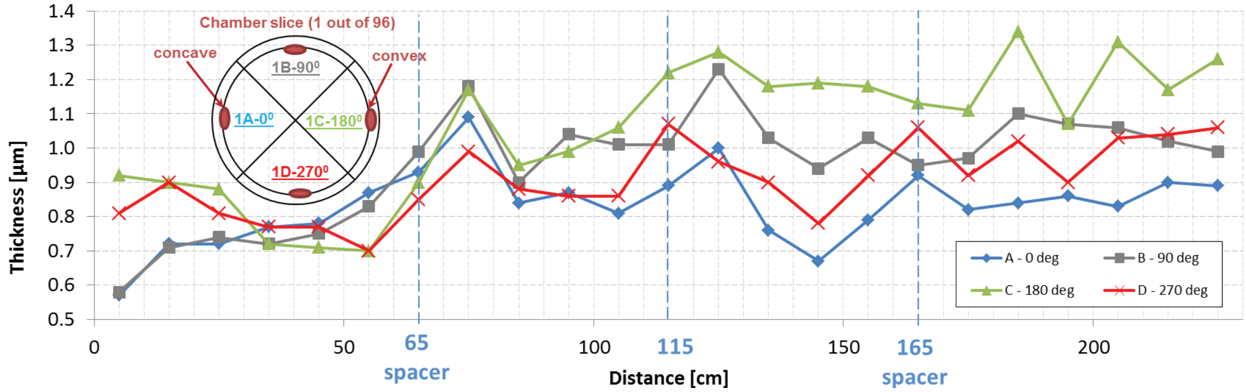

Figure 5: XRF NEG-coating thickness distribution measurement results of the 3 degrees bent vacuum chamber.

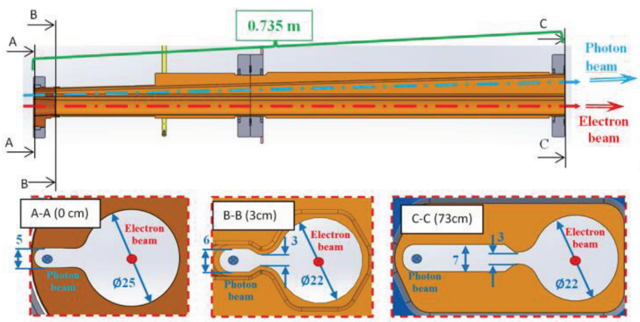

Figure 6: Vessel with antechamber.

The antechamber of the vessel with vertical opening varying from 5 mm at the start to 7 mm at the end was the most demanding part to be NEG-coated due to limited space for the plasma to develop uniformly. The goal was to coat with a NEG film of good activation behaviour 100% of the antechamber inner surface. To accomplish this goal, a prototype of the chamber was prepared and coating trials were performed at CERN. The prototype was made in such a way that it was possible to open it in the mid-plane and visually inspect the film coverage.

For the trials, different number and design of the cathodes were used: from 1 to 3 cathodes intertwisted from 0.5 mm diameter Ti, Zr, V wires. The cathode voltage, process gas pressure and magnetic field were varied to develop uniform plasma all along the antechamber length. In order to monitor the plasma behaviour, a custom coating system was constructed which enables to observe the glow discharge during the coating process through a viewport. The sample results of two coating trials are presented in Fig. 7.

Figure 7: Examples of two antechamber coating trials.

The first presented run was performed for the full chamber i.e. also including the 22 m channel. The antechamber was coated using three cathodes. The second coating run was done only for the antechamber part with two cathodes. In both cases, the NEG coating coverage is uniform. However, in the second case, the film analysed by XPS did not show good activation behaviour. This is due to the excess of vanadium in the film composition, as a consequence of the high power used for the deposition process to force the plasma to be distributed along the full length of the targets.

FUTURE PERSPECTIVES

To meet the objective to coat the entire antechamber surface with a functional NEG film the coating parameters and cathode configurations need a further optimization. Furthermore, thermal cycling of non-etched and etched EDM surfaces will be made to verify the NEG film adhesion on such surfaces.

CONCLUSIONS

During the collaboration many aspects of the NEG-coating by magnetron sputtering of small aperture, novel vacuum chamber geometries and materials were addressed, tested and validated. The preliminary surface treatment of the OFS copper substrate was performed. Adhesion of the NEG film on the OFS substrate was tested and confirmed. The coating feasibility of small diameter, bent vacuum vessels was studied and the coating procedure established. The NEG film properties inside small aperture chambers were verified.

ACKNOWLEDGMENT

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