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DEVELOPMENT AND PRODUCTION OF NON EVAPORABLE GETTER COATINGS FOR THE VACUUM CHAMBERS OF THE 3 GeV STORAGE RING OF MAX IV

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Abstract

MAX IV is presently under construction at Lund, Sweden, and the first beam for the production of synchrotron radiation is expected to circulate in 2016. The whole set of 3 GeV ring beam pipes is coated with Ti-Zr-V Non Evaporable Getter (NEG) thin film in order to fulfil the average pressure requirement of 1×10−9 mbar, despite the compact magnet layout and the large aspect ratio of the vacuum chambers. In this work, we present the optimizations of the coating process performed at CERN to coat different geometries and mechanical assembling used for the MAX IV vacuum chambers; the morphology of the thin films is analysed by Scanning Electron Microscopy; the composition and thickness is measured by Energy Dispersive X-ray analysis; the activation of the NEG thin film is monitored by X-ray Photoemission Spectroscopy; the vacuum performance of the coated beam pipes is evaluated by the measurement of hydrogen sticking coefficient. The results of the coating production characterization for the 84 units coated at CERN are presented.

INTRODUCTION

The 3 GeV storage ring of the MAX IV photon facility has a circumference of 528 m and includes 20 achromats with 19 straight sections devoted to insertion devices. The ring is characterized by ultra-low emittance obtained by applying a multi-bend achromat (MBA) magnet concept. Compact magnet design, (bore of 25 mm), and limited space between consecutive magnets poses drastic limitations on the vacuum system. To integrate the vacuum system with the magnets, the majority of the vacuum chambers were designed as 22 mm inner diameter, 1 mm thick copper tube. In order to ensure ultra-high vacuum conditions in the ring, the conductance limited chambers are coated with Non-Evaporable Getter (NEG) by DC magnetron sputtering [1]. To adapt the existing technology to coat the specific beam pipes of the 3 GeV ring, CERN and MAX IV Laboratory have set a collaboration. Currently, the majority of the chambers are manufactured and are being coated at three different facilities, including CERN where chambers with uncommon geometries are treated.

COATING PRODUCTION

All chambers were fabricated by FMB-Berlin. VC01, VC02A and VC02B are made of OFS copper while the VC02L is in 316LN stainless steel. The surface treatment was done at the fabricant following CERN standards [2], with the only exception of the copper tubes used in the construction of the VC01 type. For this case, the tubes were previously etched at CERN in a solution of ammonium persulphate in order to remove the outmost 80 μm of the surface (damaged skin) and passivated with a sulpho-chromic acid bath.

The VC01 is composed of three bent sections for a total deviation of 1.5°, a total length of 2.55 m and a circular aperture of 22 mm (Figure 1 a). In the bent sections, alumina spacers were used to ensure the centring of the cathode, which is made of 3 intertwisted elemental wires, each one of 1 mm diameter, of Ti, Zr and V). Each coating run lasted four days and two chambers were coated per run.

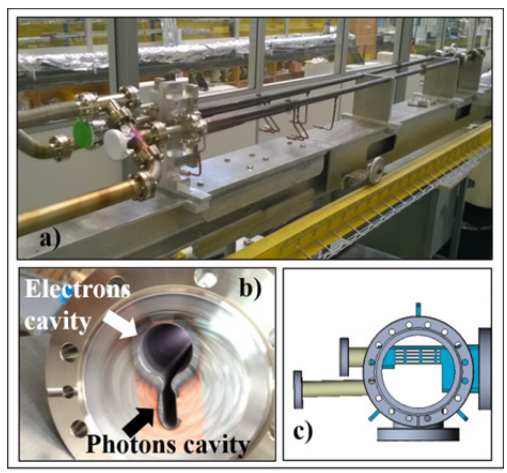

Figure 1: a) VC01 chambers on the coating support; b) photon and electron cavities for VC02B chamber (similar to VC02A); c) top view of the VC02L, with the inner grid.

The VC02A and VC02B are composed of two cavities, for the electron and extracted photon beams, respectively (Figure 1 b). They have a total length of 300 mm and 435 mm, respectively. The aperture of the electron cavity is the same for both, i.e. 22 mm, and the apertures of the photon cavities are tapered from 5 mm × 11 mm to 6 mm × 22 mm for VC02A and from 6 mm × 22 mm to 7 mm × 34 mm for VC02B. Electron cavities are coated using a cathode made of 1 mm elemental wires and presented no particular difficulty. Coating the photon cavities was the main challenge for this type of chambers. The cathodes were made from 3 intertwisted elemental wires with a

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diameter of 0.5 mm each. For the VC02A, a single cathode was sufficient, while for the VC02B two adjacent cathodes were necessary to assure acceptable thickness uniformity. Furthermore, one of the cathodes was tilted in order to follow the change in the aperture. The main difficulty was to obtain uniform plasma inside the photon cavities. This was achieved only in a narrow range of parameters, (pressure, power density, magnetic field). The power density must be high enough to allow the plasma to spread along the whole cathode, but cathode overheating must be avoided. Each coating run, (4 chambers each), lasted 1.5 weeks. For the VC02A and B chambers, the deposition sequence started with the burn-in of all the cathodes to remove the native oxide layer. Then, the photon cavity is coated and, finally, the electron cavity.

Table 1: Coating Parameters for the Different Chambers

Chamber	Photon	Electron	VC02L	VC1
Power density	25 [W/m]	25 [W/m]	80 [W/m]	25 [W/m]
Pressure mbar	6.6×10^{-1}	1.1×10^{-2}	1.4×10^{-2}	1.1×10^{-1}
Magnetic field G	500	185	185	185
Coating duration	8h	10h	13h	10h

The VC02L (Fig. 1 c) is the last element of the beam extraction line. It is composed of a main DN100 cylindrical vessel with an inner grid around the path of the electron beam to reduce beam impedance and several ports. A central cathode, made of 3 mm elemental wires, is placed in the DN100 tube. At the level of the inner grid, the cathode is off-centred in order to avoid an over thickness onto the grid. Each coating run lasted three days and three chambers were coated per run.

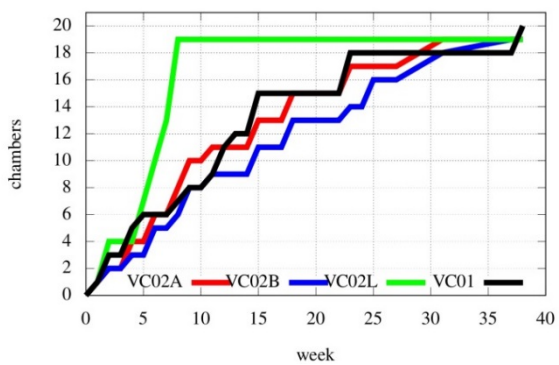

Figure 2: Dashboard of the coating production.

For all chambers, the coating duration is calculated to achieve an average thickness of about 1.2 µm. Krypton was used as discharge gas and the coating parameters for all chambers are listed in Table 1. The production campaign started in July 2014 and, by March 2015, more than 95% of the chambers were coated (see Fig. 2).

RESULTS AND DISCUSSION

The quality control of the NEG thin film was performed on samples installed at the extremities of the chambers. For the VC01, VC02A and VC02B, the samples were made of OFE copper with the surface machined by electrical discharge, as for the real chamber's surface. For the VC02L, 316 LN stainless steel ribbons were used. All the samples had the same surface finishing as the respective real chambers.

Activation and Surface Composition

Surface composition and activation performance were measured by XPS. The activation performance is evaluated by the reduction of the area of the oxygen O1s peak during a cycle of 2 hours at 250°C (higher the O1s reduction, better the activation). Figure 3 shows the reduction of the oxygen peak as a function of the surface concentration of vanadium. It can be seen that the coatings on the electron cavities perform better than the coatings on the photon cavities. This difference cannot be explained neither by the surface composition measured by XPS, (usually an excess of vanadium above 50% is known to be detrimental), nor by the crystallographic structure. A few samples measured by X-Ray Diffraction present grain sizes below 10 nm, compatible with what has been considered a necessary condition for low temperature activation [3]. As the photons cavity is the first to be deposited, at the end it is covered by a thin layer sputtered from the cathode on the electron chamber. The XPS measurement reflects the behaviour and composition of this topmost layer. To further investigate the reason of the poor activation behaviour of the coatings on the photon cavities, the bulk composition was measured by EDX.

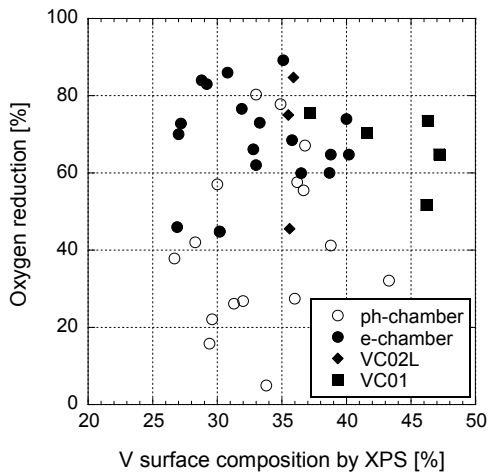

Figure 3: Activation performance: reduction of the area of the O1s peak from the XPS spectrum during activation.

Bulk Composition and Thickness

To investigate a possible variation of the stoichiometry in depth, a preliminary EDX analysis was performed on a coating lamella prepared by Fast Ion Beam (FIB) technique. Figure 4 shows the cross section of a coating on the photons cavity. Three different layers are

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identified: (3) corresponds to the burn-in of the cathodes (~ 100 nm); (2) to the coating from the cathode on the photon cavity, (~ 500 nm); (1) to the layer coming from the coating on the electron cavity, (~ 180 nm). The angle of the columnar growth confirms that this last layer comes from the electron cavity; porosity can be observed in layers (1) and (2). The vanadium content measured in EDX on the three points of Fig. 4 is: 42.5 at% for (1); 47.5 at% for (2) and 52.9 at% for (3). The origin of the 52.9 at% in (3) can be explained by the relatively high power density used during the burn-in of the cathodes to force the plasma to spread along the cathode. However, the 47.5 at% in (2) cannot be easily explained by power density arguments; such a content of vanadium approaches the limit that can be at the origin of the poor activation behaviour. In fact, once exposed to air, is the topmost layer (1) that is oxidized and the activation depends on its capability to diffuse and dissolve the oxygen in the bulk. With a content of 42.7 at% of vanadium, the layer (1) still has a good composition to activate correctly, provided it is thick enough to accommodate all the oxygen. If the layer (1) is too thin and the layer (2) is not able to diffuse the oxygen in the oxide layer, the activation starts but cannot be completed. This is compatible with the observed evolution of the XPS spectra during activation. This hypothesis could also explain why some samples from the photon chamber have good activation performance: the topmost layer (1) was thicker than for the poorly activating ones. Further studies and comparison with a similar FIB analysis on the electron chamber are necessary to confirm this hypothesis.

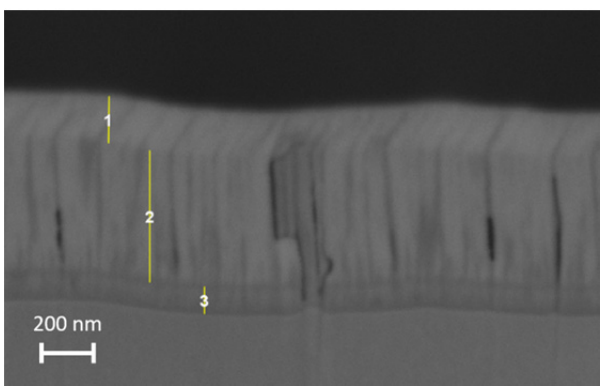

Figure 4: Cross section of a coating on a photon cavity with the three growing regions.

Sticking Coefficient

The sticking coefficient of hydrogen, *s*, was measured for a VC01 chamber by the transmission method. After an activation cycle at 180°C for 24 hours, hydrogen was injected at one extremity and the total pressure measured at both ends using calibrated Bayard-Alpert ionization gauges. The pressure ratio across the full length of the chamber, P_{inj}/P_{end}, is correlated with *s* via a Monte Carlo simulation of the system [4].

A sticking coefficient of $s = 3.0\text{x}10^{-3}$ was found. Although this is below the expected value for an activated NEG surface, i.e. $s \sim 10^{-2}$, it is an underestimate of the real value for the sticking coefficient. This discrepancy is due to the high ratio between the length and the diameter of the chamber, $L/D \sim 116$, that results in a very low transmission probability for the hydrogen molecules (the simulated P_{inj}/P_{end} varies from ~ $5\text{x}10^{2}$ for $s = 10^{-3}$ up to ~ $2\text{x}10^{6}$ for $s = 10^{-2}$). This means hydrogen should be injected at very high pressures, (above 10^{-6} mbar), to obtain a practically measurable pressure increase at the end of the tube (~ 10^{-11} mbar). However, at the high pressure injection side, a small fraction of the hydrogen molecules are dissociated in the hot filament of the vacuum gauge and produces methane. Since this gas is not pumped by the NEG it will reach the end of the chamber, contributing to the increasing of P_{end}, and so masking the real hydrogen pressure.

CONCLUSIONS

By the end of March 2015, more that 95% of the CERN coating production for MAX IV was finished. NEG coating in VC02L and in the electron cavities (VC01, VC02A and VC02B) performed as expected while in the photon cavities, some coatings presented reduction slower activation process. The multilayer structure and the high vanadium content as found in the FIB lamella sample analysed by EDX may be at the origin of this behaviour. Further studies are required to confirm this hypothesis Even a partial activation of the NEG provides a reduction of the photon stimulated desorption and the delayed activation of the photon chamber is not crucial for the performance of the machine [5].

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