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SAES EXPERIENCE IN NEG COATING OF CHALLENGING VACUUM CHAMBERS

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Abstract

In recent years, SAES has deepened its knowledge in the NEG coating field, aiming at uniformly coating vacuum chambers with challenging geometries and fine-tuning the film characteristics, according to the needs and requirements of the final users.

To achieve these goals, several complex vacuum chambers have been coated and studied, both at SAES and in collaboration with various research institutes around the globe. Tests made on NEG-coated samples include pumping speed and sorption capacity measurements, extensive film characterisations by XRF, SEM-EDS and XRD analyses, vacuum and plasma simulations, and photon-stimulated desorption yield measurements.

At the same time, SAES has been committed to the NEG coating of hundreds of vacuum chambers and of several prototypes for ongoing and upcoming machine upgrades, respectively.

An overview of the most significant achievements and results is presented, not only focusing on the technical challenges and the optimisation of few prototypes, but also giving an industrial perspective in terms of reliability, when large batches of tens or hundreds of vacuum chambers should be deposited.

INTRODUCTION

Since the early 2000s, non-evaporable getter (NEG) coatings have become increasingly popular in the particle accelerator and vacuum technology community, thanks to their ability to provide a distributed pumping effect and to keep the required ultra-high vacuum (UHV) pressure, both in static and dynamic conditions. The use of NEG coating results in a significant reduction of the thermal outgassing along the beam pipes, as well as in very low secondary electron (SEY), photon stimulated desorption (PSD) and electron stimulated desorption (ESD) yields [1-4].

Nowadays, NEG coating is even more essential to meet the strict vacuum requirements of the next generation of ultra-low emittance light sources, where the size of beam pipes is greatly reduced to allow the use of compact and powerful magnet lattices. Small-aperture vacuum chambers and, in general, a lack of space for big, conventional UHV pumps make NEG coating an enabling technology to ensure the target beam commissioning and long-term operation conditions of these new machines.

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The NEG coating deposition process and the characteristics of the deposited film have been extensively studied over the years [5-7]. However, the current need to deposit NEG coating in chambers with complex shapes and high aspect ratios push the technology to its limits and poses a number of issues in terms of process limitations and performance assessment [8, 9].

NEG FILM PROPERTIES FINE TUNING

In most cases, a NEG coating consists of a thin film (~1 μm) deposited by magnetron sputtering, using a cathode made of three intertwisted wires of Ti, Zr, and V.

The thickness, elemental composition and microstructure of the deposited film are key characteristics, which are strictly dependent on the sputtering configuration and parameters (pressure, applied voltage, magnetic field, substrate temperature, sputtering time). These three characteristics determine the performance of a NEG coating in terms of low activation temperature (160–180 °C), high pumping speed and sorption capacity, low impedance, and low SEY, ESD and PSD.

At the same time, the choice of the best combination of sputtering parameters should also come to terms with the need to fully coat complex geometries, in which plasma ignition and stability may be compromised or even inhibited without an adjustment of the sputtering parameters themselves [10].

Thickness Control and Uniformity

One of the main requirements of the new light sources is to keep the NEG coating thickness below a given threshold (typically in the range of 0.3–1.0 μm) and to carefully control its uniformity, in order to mitigate the beam impedance of the deposited film. Surface roughness and film growth (dense or columnar) are also very important from this point of view and, in addition, they have a strong impact on the PSD and ESD yields of the NEG coating [11]. Finally, the film growth and thickness also influence the sticking coefficient of the NEG coating and its sorption capacity [12].

Standard sets of sputtering parameters do not allow to ensure the required thickness uniformity in small-aperture and high-aspect ratio chambers. To address this issue, an extensive experimental campaign carried out at SAES has investigated the influence of sputtering parameters on NEG coating thickness uniformity. Several copper chambers with a high aspect ratio (ID 12–22 mm, length 1 m) have been coated—using different sets of sputtering parameters—and then cut into several pieces, to get a

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detailed mapping of the thickness profile along each vacuum chamber. Figure 1 compares the thickness profile obtained using a standard set of sputtering parameters with the profile given by a process optimised for high-aspect ratio chambers. This striking comparison shows how it is possible to fine tune the plasma sputtering process and obtain a strictly-controlled thickness profile, even when the high aspect ratio makes plasma ignition and stability quite challenging. The thickness analyses have been made by x-ray fluorescence spectroscopy (μ-XRF) and about 300 data points have been collected on each vacuum chamber.

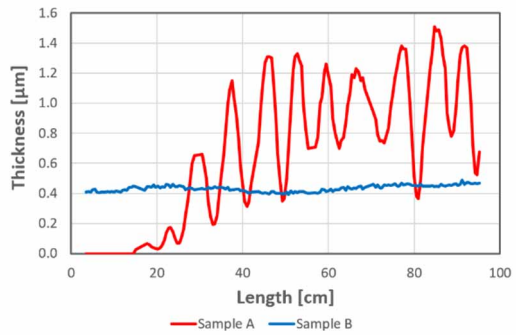

Figure 1: Non-optimised (Sample A) and optimised (Sample B) NEG coating thickness profiles along high-aspect ratio copper chambers (ID 12 mm, length 1 m).

Elemental Composition and Microstructure

Along with the coating thickness, it is also very important to monitor its elemental composition and crystal structure, because they define the minimum activation temperature of the NEG coating; more specifically, a nominal composition close to Ti_{30}-Zr_{30}-V_{40} (at.%) and a nanocrystalline structure (grain size 1–5 nm) are envisaged [5-7].

μ-XRF has been used also to map the elemental composition along the high-aspect ratio vacuum chambers under investigation. Figure 2 reports the elemental distribution along the same optimised "Sample B" chamber shown in Fig. 1: the results prove that, alongside thickness, elemental composition can also be carefully controlled and kept as close as possible to the desired nominal values.

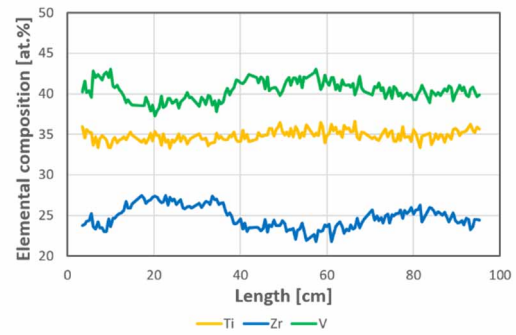

Figure 2: Elemental composition profile along a high-aspect ratio copper chamber (ID 12 mm, length 1 m).

In parallel, x-ray diffraction spectroscopy (XRD) allows to monitor the crystalline structure and grain size of the deposited NEG films. Figure 3 shows the typical spectrum of a nanocrystalline NEG coating, deposited on a high-aspect ratio chamber (ID 22 mm, length 1 m): according to the Scherrer equation, the broad peak at 38° is the footprint of a nanocrystalline structure—with a grain size of 2–3 nm—which guarantees that the coating has the desired low activation temperature (160–180 °C).

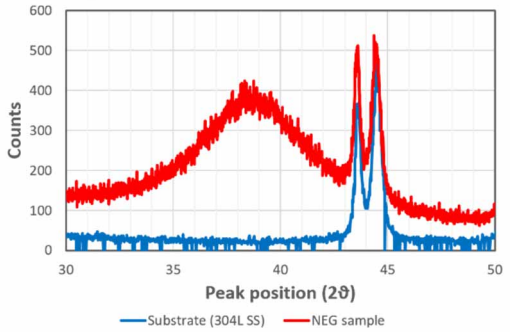

Figure 3: Typical XRD spectrum of a nanocrystalline NEG coating (the spectrum of the stainless steel substrate is shown as a reference).

Further evidence of this comes from a recent study made at KEK [13]: an undulator chamber with a high aspect ratio (90x15 mm elliptical cross-section, length 4.1 m), NEG coated by SAES, was installed in the Photon Factory storage ring (PF-ring) and activated at 160 °C for 48 h, with highly-satisfying results in terms of ultimate pressure, residual gas composition, and beam conditioning behaviour. The analyses performed on a NEG-coated silicon coupon evidenced the desired nanocrystalline structure, elemental composition and film thickness.

PSD Yield Measurements

In the past couple of years, SAES has provided a series of NEG-coated chambers (ID 10–63 mm, length 1 m) to SOLEIL, in order to ascertain whether NEG-coating properties remain unchanged when small-gap vacuum chambers—like those envisaged for the future SOLEIL Upgrade —are used.

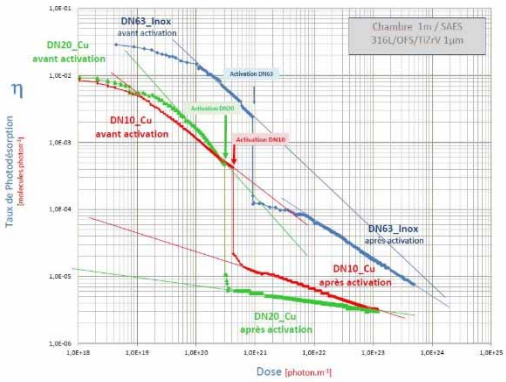

Figure 4: PSD yield of NEG-coated chambers, before and after activation, as a function of the photon dose (courtesy of Vincent Le Roux, Synchrotron SOLEIL).

Figure 4 shows the PSD yield as a function of the photon dose, before and after NEG coating activation, for three different 1-metre long chambers (ID 10, 20, and 63 mm,

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respectively): no significant difference is observed, indicating that no downscaling issues occur if the NEG coating process for high-aspect ratio chambers is properly tuned.

Pumping Performance Assessment

The pumping performance of a NEG-coated chamber is usually described in terms of sorption capacity (expressed in mbar·l·cm^{-2}) and sticking coefficient α, a dimensionless quantity which is deduced from experimental measurements of the chamber's transmission factor (*i.e.*, the pressure ratio at its ends during gas injections) and Monte-Carlo simulations [14]. Measuring the sticking coefficient of high-aspect ratio chambers is quite challenging and the risk to incur measurement artefacts is high [15]. Figure 5 shows a CO sorption test carried out on a 0.2-metre long, ID 10 mm chamber, whose total sorbed quantity and initial transmission factor (corresponding to α>0.1) are in line with literature data [6].

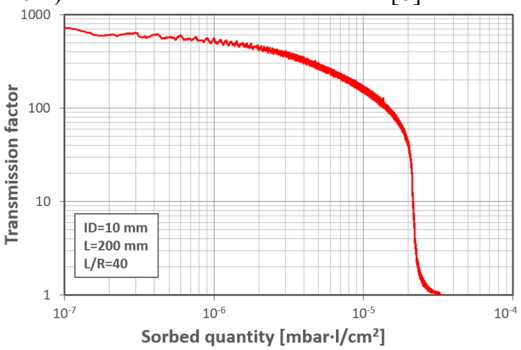

Figure 5: Transmission factor vs. CO sorbed quantity of a small-aperture NEG-coated chamber (ID 10 mm, length 0.2 m).

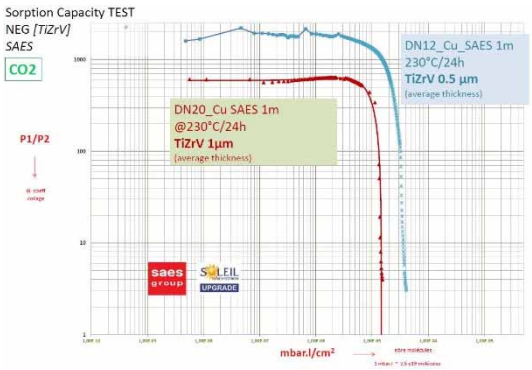

Figure 6: Transmission factor vs. CO_2 sorbed quantity of small-aperture NEG-coated chambers with different film thicknesses: ID 12 mm, length 1 m, 0.5 µm vs. ID 20 mm, length 1 m, 1.0 µm (courtesy of Vincent Le Roux, Synchrotron SOLEIL).

Sorption tests carried out at SOLEIL on NEG-coated chambers provided by SAES were also good (cf. Fig. 6), despite their aspect ratio being quite high (ID 12–20 mm, length 1 m). Tests made on an ID 12 mm, 1-metre long chamber with a 0.5 µm deposited NEG film, in particular, confirmed the effectiveness of NEG coating even when thickness is limited to avoid beam impedance issues.

FROM R&D TO SERIES PRODUCTION

In light of all the previous considerations, it is easy to understand how prototypes are important to identify and overcome all the issues given by the need to coat chambers with complex shapes and high aspect ratios, such as UHV cleaning, cathode insertion and positioning, and presence of delicate components (beam position monitors, bellows, beam screens, RF fingers, …). Once the prototyping phase is completed, the NEG coating process is optimised and ready to be transported to a production phase, where several identical chambers should be coated and time and cost effectiveness become of primary importance. Strict quality control and quality assurance procedures are implemented at SAES, to ensure that the machine requirements in terms of NEG film characteristics are always fulfilled.

Over the past two years, SAES has coated 528 Inconel and aluminium ID 22 mm vacuum chambers for the APS-U project at Argonne National Lab. An example of the NEG process controls in place is given in Fig. 7, where the results of 906 µ-XRF analyses carried out during the production phase show that the elemental composition of the NEG coating is strictly under control and inside the literature acceptance range [7].

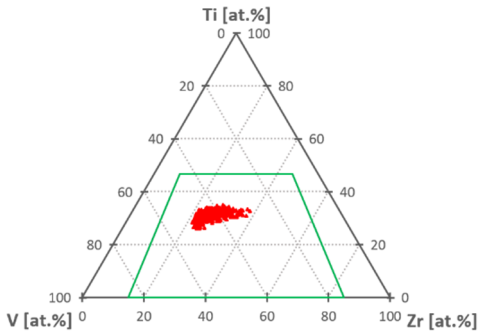

Figure 7: Results of 906 µ-XRF analyses carried out for the APS-U project (the green trapeze is the acceptance range).

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