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DETERMINATION OF PUMPING AND DYNAMIC VACUUM PROPERTIES OF CONDUCTIVE QUATERNARY ALLOY OF TiZrVAg NON-EVAPORABLE GETTER

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Abstract

Non Evaporable Getter (NEG) coating has been employed extensively in the particle accelerator especial-ly where the vacuum conductance of the vessel is se-verely restricted and ultra-high vacuum condition is required. NEG coating will significantly reduce the outgassing rate and provides active pumping surface for H_2, CO and CO_2. In addition, it has been proven that NEG coated surfaces have a very low secondary electron yield, as well as low photon and electron stimulated desorption yields. However, the existing NEG film increases the RF surface resistance of the beam pipe.

In order to increase NEG coating conductivity, at ASTeC, in the past several years, the alternative NEG composition have been studied by adding more con-ductive element such as Cu, Au, Al and Ag. In this study, we report on the photon stimulated desorption, activation temperature and surface resistance from room temperature to cryogenic temperature for a new NEG quaternary alloy of TiVZrAg as function of the film composition.

INTRODUCTION

Non-evaporable getter (NEG) coating, originally invented at CERN, is already used in many accelerators [1-5] due to three main properties:

1. In a dense structure, it acts as barrier between a vacuum chamber material and an inner vacuum hence even in non-activated state it provides lower residual gas pressure.
2. In a columnar structure, it has a large surface area, hence, a fully coated vacuum chamber has large distributed pumping speed, the benefit of this is essential for the narrow vessels with a limited vacuum conductance.
3. In activated state, the NEG coating has low secondary electron (SEY) yield that helps to supress electron multipacting and electron cloud in high intensity accelerators.

Over recent years, ASTeC Vacuum Solutions Group made a good progress in further development of NEG coatings. New quaternary alloy coating which, can be activated at 140 °C, it is 40 °C lower than for an original invention [6, 7]. A good progress was achieved in reducing photon and electron induced desorption [8, 9]. There are two main challenges in further NEG coating optimisation: (1) the beam impedance in the NEG coated chamber, which related to beam parameters and NEG coating RF surface resistance, this is a problem for short-bunch machines [10, 11]; (2) coating with NEG on narrow tubes with a diameter less than 10 mm, this is a problem for small aperture machines like a future UK-XFEL.

In this report we will concentrate mostly on solution of decreasing NEG surface RF resistance while keeping the integrity of its main three properties.

SAMPLE PREPARATION

Two type of samples was used in this set of experiment: tubular sample that, is the best geometry for evaluation of the pumping properties and flat samples for surface analysis and surface resistance. For the tubular sample a 316 LN stainless steel tube of inner diameter of 38 mm and the length of 0.5 m equipped with two CF40 flanges at either end was deposited with TiZrVAg. The film was deposited using a coaxial cylindrical magnetron configuration with an external magnetic field provided by a solenoid. The deposition method and apparatus is described in our previous publications [6]. The target was a single 3-mm diameter NEG alloy wire of TiZrV specially made for ASTeC, with 1-mm diameter Ag wire raped around the NEG wire. The concentration of Ag is controlled by the number of turn used within the 0.5 m length of the tube. The deposition was done at 10^{-2} mbar and with DC sputtering mode. The flat samples were produced by placing different size of Ag wire in a 3" planar magnetron target. The deposition condition was kept the same as one chosen for tubular deposition.

PSD MEASUREMENT

The PSD was carried out at Taiwan National Synchrotron Radiation Research centre, beam line TLS-BL19B of 1.5 GeV beamline. The PSD experimental system contains a turbo-molecular pump (TMP), a conductance-limited long aluminium pipe, RGA, and two gauges, that connected the NEG-Tube for carrying out the measurement. The whole experimental system with the NEG-coated tube was performed the vacuum baking and the NEG-activation and then achieve the ultimate pressure of 2×10^{-10} mbar.

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Then open the gate valve to link the beamline system, isolate the TMP, and reach to the static vacuum pressure of 2.4×10^{-10} mbar that was ready for measuring the yield of the photon stimulated desorption (PSD) via the throughput method.

The outgassing during the baking (150 °C, 24 h) contained typically the H_2O, H_2, CO_2 and CO. While the Kr (from coated film) was observed as well. The outgassing during the NEG activation (180 °C, 24 h) contains majorly the H_2 and hydrocarbons when arising the temperature (T) over 150 °C, and the burst of CH_4 and Kr at T over 180 °C, and, finally, the residual gases of H_2, H_2O, CO, CO_2 and Kr, precede the cool down.

RF SURFACE RESISTANCE MEASUREMENT METHOD

The measurement method selected for these experiments is based on a Hakki-Coleman dielectric resonator (DR) [12]. Our design has been fitted to perform measurements at low temperature under DC magnetic fields and adapted to the size of the samples available. It consists of a cylindrical metallic cavity (radius 5.5 mm, height 3 mm) loaded with a c-oriented, high-permittivity (ε_r(50K) ≈ 110), and low-loss (tan(δ)(50K)<10^{-5}) rutile (TiO_2, radius 2 mm), shielded axially by one pair of samples to be analysed. The structure sustains a TE_{011} mode [13, 14] in which induced RF currents on the samples are azimuthal. The absence of radial currents makes the resonator parameters sensitive to the properties of the materials being tested but insensitive to the electrical contact between the samples under test and the lateral walls. This technique has proven capable of measuring surface resistances (R_s) over four orders of magnitude with high reproducibility [15].

RESULTS & DISCUSSION

Figure 1 depict surface chemical state of each elements of a TiZrVAg with 5 atomic % of Ag. For as air exposed sample all active elements of Ti, Zr and V are of their oxide state of TiO_2 , ZrO_2 and V_2O_5. There is significant amount of C at the surface up to 54 at% at and O up to 30 at%. The activation cycle was set at two cycle of 20 hours. The first was at 140 °C and the next was at 180 °C. XPS analysis carried out after each activation cycle. An interesting point is that Ag is in metallic state the spin orbital doublet at 374 and 368 eV for j = 3/2 and j =3/5 respectively. Ag stays the same throughout activation temperature range employed in this study. After 140 °C activation, all active elements are in mixed metallic and oxide state. V is almost fully activated while Ti (Ti^0, Ti^{1+}, Ti^{2+}, Ti^{3+}) and Zr (Zr^0, Zr^{1+}, Zr^{2+}, Zr^{3+}) are mainly in their sub oxide states. After activation at 180 °C the NEG is fully activated with all the metallic elements being at metallic state and carbide state. The metallic carbide state is evidence by the C1s peak is split at two binding energy of 185 eV corresponding to amorphous carbon and 182 eV corresponding metallic carbide as shown in Fig. 1e. The SEM image of NEG sample is shown in Fig. 2. It can be seen that film is grown in dense structure.

During the PSD-measurement, the SR light of 2.53 mrad horizontal-span from the BL19B beamline was introduced to the NEG- tube at 3° of incidence angle that spread the 18.6 W power of SR to the inner side of tube. The TLS is operated at 1.5 GeV, 362 mA top-up mode, and the critical photon energy of 2.14 keV. The NEG-coated tube was cooled by wrapping the Cu-jacket brazed with water cooling tubes that maintained the tube at 25.1±0.2°C of temperature. The increase of temperature at the NEG-coated tube during the SR exposure is 0.3 °C on the upstream and 0.1 °C on the downstream, respectively. The variation of the ambient temperature is about 0.5 °C, in the experimental hall due to the climate change of day/night, through the overall experiments.

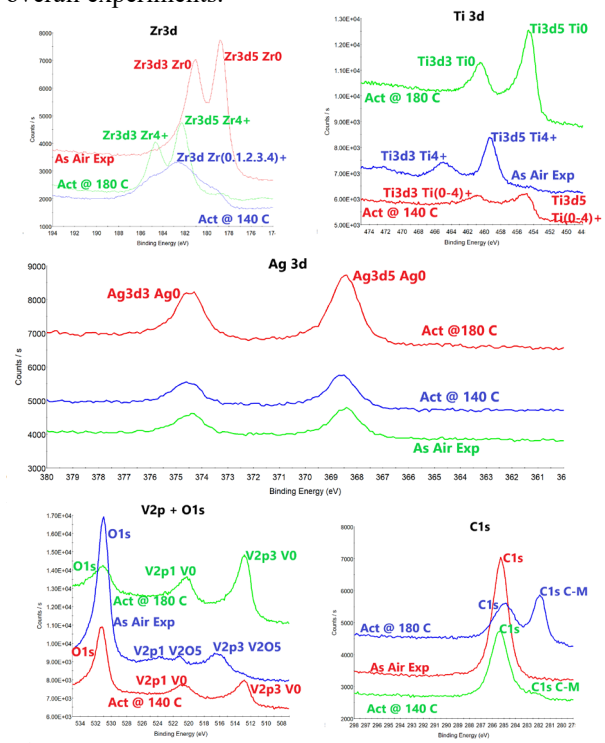

Figure 1: XPS region spectra of main elements of TiZrVAg: a) Zr3d, b) Ti3d, c) Aged, d) V2p and O1s and e) C1s.

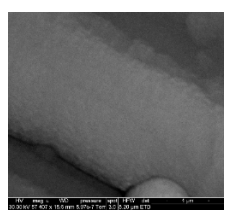

Figure 2: A planar SEM image of TiZrVAg deposited on copper.

The PSD yield for the NEG-coated tube was measured about 2×10^{-5} molecules/photon at 0.01 Ah of accumulated Beam Dose at preliminary SR-exposure stage, then reduced to 1.2×10^{-5} molecules/photon at 0.2 Ah in 30 minutes, and continuously reduced to 3.4×10^{-7} molecules/photon at 160 Ah along a simulated linear curve with a slope of -0.556. The total exposure time taken for achieving the accumulated beam dose of 160 Ah, equal to

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1.27×10^{22} photons/m, is equivalent to 18.5 full-days continuous- exposure. The PSD-yield of NEG-tube is about 1–2 order of magnitude lower than those of the aluminum (AL) or SS tubes with various surface cleaning or treatments. These can be seen in Fig. 3.

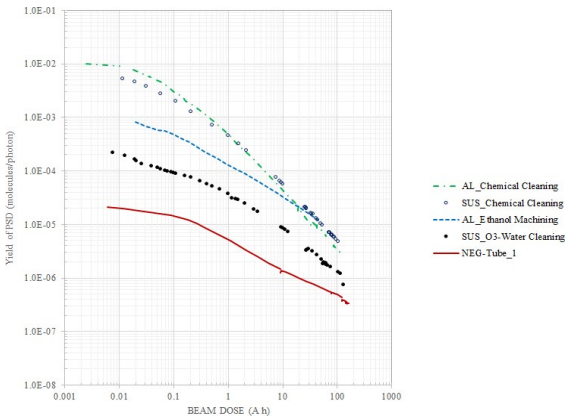

Figure 3: The PSD results after activation at 180 °C.

As can be seen in Fig. 4, the PSD from the NEG-coated tube contains H_2, C_xH_y, CO, CO_2, H_2O and Kr, that decreased continuously through the photon exposure. It reveals the effect of beam conditioning that depicts the fact of the PSD molecules are generated from the NEG coating on the tube.

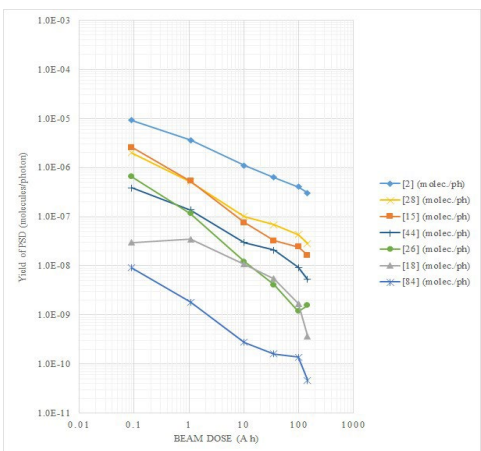

Figure 4: PSD yields as function of beam dose.

The desorption of hydrocarbons, C_xH_y, must be generated by the recombination of hydrogen and carbon atoms resided in the NEG film during the SR irradiation since the hydrocarbons should not be originally pump-absorbed on the NEG film. The desorption of the Kr is definitely from the NEG film (as Kr was a discharge gas during the coating process). The pumping of NEG for the H_2, CO, and CO_2 is promising since the rise of desorbed quantities of these gases are not significant in comparison with that of hydrocarbons and Kr. The evidence of lower PSD yield is consistent as expected. The measurement of the surface resistance performed as function of temperature in the 8 GHz dielectric resonator, for three different Ag atomic ratio ranging from 2 at% to 6 at%.

The results is shown in Fig. 5. In here Sample 6Ag is the same sample as the XPS results is shown above. It shows a much higher quality factor at low temperatures as compared to the rest of the samples. It should be noted that in this set of measurement film associated with Sample 2Ag was delaminated and very patchy.

Figure 5: Unloaded quality factor Q_0 as a function of temperature.

Table 1: Surface Resistance of Various Ag Concentration in TiZrVAg Film Deposited on Copper

Samples	R_s (mOhm) at 300 K
No Ag	41.7
2 Ag	49.0
4 Ag	40.1
6 Ag	30.6
OFCH Cu *	25.7

Table 1 shows the surface resistance which of the measured samples at 300 K. The value for OFCH copper is added for comparison. As it can be seen a 5 at% Ag can considerably reduce the NEG resistance. The surface resistance of a copper coated sample with conventional NEG increases from 25.7 mΩ for a bare copper to 41.7 mΩ, an increase of 62%. By adding 5 at% Ag the increase in surface resistance is reduced to 18%. This is considerable reduction. This is firstly due to the added conductive element, in this case Ag, stays in metallic state. Furthermore, the SEY should be the same or at least better for non-activated or as received NEG. The activation temperature of 140 °C is comparable to our previous quaternary TiZrVHf and conductive TiZrVCu reported in ecould18 and EVC15.

CONCLUSION

We optimised the effect of additional conductive metal to conventional NEG in order to compensate the increase of NEG surface resistance comparing to bare copper used as vacuum chamber. We have successfully showed that despite fact that the added material will not act as a getter material, nevertheless has barely any consequence on other essential property of NEG such as PSD and activation temperature.

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