Characterisation of technical surfaces at cryogenic temperature under electron bombardment.

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Abstract

The vacuum chambers of the LHC's arcs operate in a temperature range between 1.9 K, *i.e.* the temperature of the superconducting magnets, and 20 K. At such low temperatures, most of the residual gas species are efficiently adsorbed on the cold surface.

LHC's proton beam emits synchrotron radiation inside its bending magnets and, consequently, electrons are extracted from the surrounding walls by the photoelectric effect. The successive proton bunches accelerates the photoelectrons, building-up an "electron cloud" which generates gas desorption from the vacuum chamber and heat load for the cryogenic system. This phenomenon might become a limiting factor for the operation of the High Luminosity LHC upgrade, where more intense proton bunches will circulate.

In order to study the electron interaction with gas adsorbed at cryogenic temperature, a new facility has been designed and built at CERN. It reproduces in the laboratory the typical conditions of a cryogenic ultra-high vacuum surface present in the accelerator.

In this paper, the first results obtained with selected accelerator materials at different surface gas coverages are presented.

EXPERIMENTAL

A sample representing the inner surface of the accelerator is mounted on a 4-axis manipulator able to regulate the temperature between 10 K and 250 K. Known quantities of gas can be adsorbed on the sample surface that can be bombarded by an electron beam at different energies.

The experimental vacuum system is composed of three parts shown in Fig. 1: the main chamber made of mumetal to shield against earth magnetic field, the storage chamber to keep the samples under ultra-high vacuum and the load-lock to insert new samples.

Three linear feedthroughs and two gate valves, allows to transfer the sample, in less than half an hour, from the atmosphere to the ultra-high vacuum around 2.10^{-10} mbar, passing through the sample storage rack kept under 10^{-9} mbar.

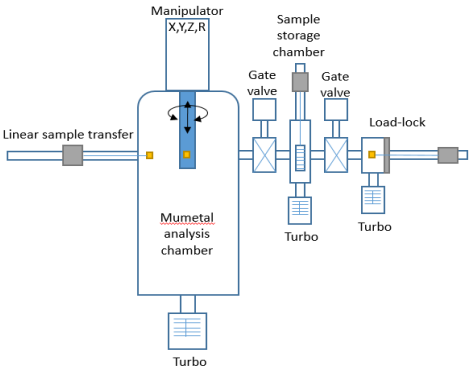

Fig. 1: Experimental setup.

The manipulator, shown in Fig. 2, controls the sample position with the help of four motorized axis. It has one thermo-regulated sample holder able to set a temperature between 10 K and 500 K using liquid helium and a thermo-coax heater. A front cover, closed, while using a wobble stick, minimise the ambient radiation towards the sample.

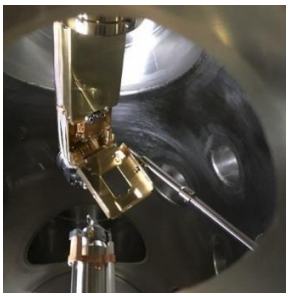

Fig. 2: Sample on the manipulator, thermal shield opened using the wobble stick and electron gun.

A second sample holder is installed just under the main one. It is not thermo-regulated, but can be used for example to hold a phosphor target to monitor the electron beam shape. The beam size is typically smaller than 2 mm and can be measured with a digital microscope installed in front of a viewport. The microscope also provides a mean to insure the reproducibility of the sample position.

Finally, on the back of the manipulator is also placed a Faraday cup to measure the intensity of the electron beam.

Pure gas can be injected inside the vessel through a diaphragm of known conductance C. According to equation (1), the injected flux, Q_{inj}, can be determined from the pressure difference across either side of the conductance, Δp_{cond}.

$$Q_{inj} = C \cdot \Delta p_{cond} \quad (1)$$

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Knowing the injected flux, the pumping speed of the system, S, is computed with equation (2). This value depends on the nature of the gas, the temperature of the sample and must be known to estimate the molecular desorption rate.

$$S = \frac{Q_{inj}}{p} \quad (2)$$

The gas can also be injected into the vessel using a calibrated volume, V. Injecting via this volume allows to compute the number of injected molecules, n, by recording the variation of the pressure in the volume, Δp_V, and using equation (3), where k is the Boltzmann constant and T the temperature.

$$n = \frac{\Delta p_V \cdot k \cdot T}{V} \quad (3)$$

The injection from the calibrated volume to the sample surface is performed through a retractable injector, as shown in Fig. 3. Doing so, the ice layer thickness can be controlled.

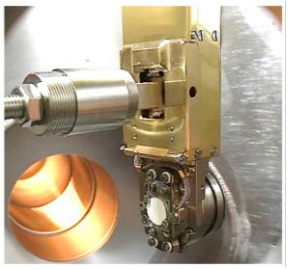

Fig. 3: Retractable gas injection device.

SEY MEASUREMENT

The secondary electron yield (SEY) of a material is defined by the ratio of the number of emitted secondary electrons to the number of incoming electrons. Thus, to quantify the SEY, the electron beam current and the secondary electron current must be measured.

Three different methods can be used to measure the electron beam current:

1. A Faraday cup set in front of the electron gun as shown on the right side of Fig. 4 could measure the current exiting the gun.
2. A Faraday cup placed at the back of the manipulator as shown on the left side of Fig. 4 is used to check the beam profile.
3. The use of a positive bias on the sample as shown on the right side of Fig. 5. This method is used to perform electron-conditioning measurement.

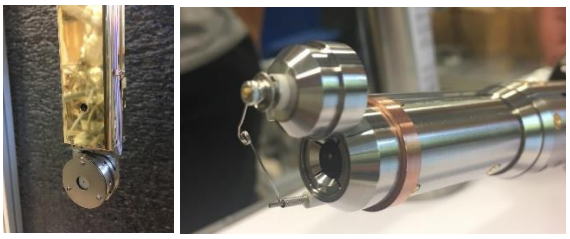

Fig. 4: Faraday cups on the manipulator and on the electron gun.

On the left side of Fig. 5, a negative voltage is applied to the sample to repel the secondary emitted electrons (I_{out}). This mode is called "SEY mode" and the measured current I_s is the difference between I_{out} and I_{beam}.

On the right side of Fig. 5, the bias is positive to prevent the escape of the secondaries and in this configuration the measured current I_s is equal to the beam current I_{beam}. It is called "Beam Mode".

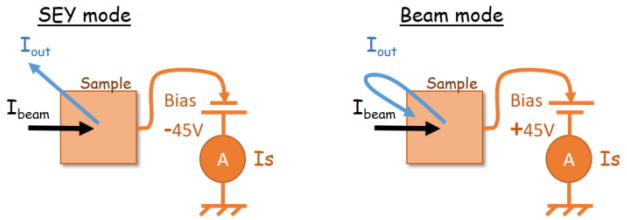

Fig. 5: SEY measurement mode (left) and Beam measurement mode (right)

After the determination of the beam current done in "Beam mode", the value of the SEY, δ, can be deduced using the "SEY mode" and the following formula (4).

$$\delta = \frac{emitted\ current}{incident\ current} = \frac{I_{out}}{I_b} = 1 - \frac{I_s}{I_b} \quad (4)$$

ICE LAYER PREPARATION

As described before, a known quantity of gas can be injected on the cold surface using the injector to condense the desired number of monolayer (ML). For a metallic technical surface like a copper sheet, the layers are supposed to be stacked homogeneously and the molecular surface density is assumed to be 1 ML=8.10^{14} molecules/cm^2. This assumption could be wrong for rough or porous material.

ELECTRON CONDITIONING

Electron bombardment is a well-known method to reduce the SEY of a surface [1, 8]. For example, this method is routinely used for RF conditioning and for beam scrubbing in the LHC ring.

In this paper, the electron conditioning and the SEY measurement are done at fix energy of 300 eV with an electron beam impinging at normal incidence on unbaked samples held at either room temperature (RT) either at 10 K.

The measurement consists to determine the beam current, I_b, using the "Beam mode" and to switch in "SEY mode" monitoring the current, I_s, to compute δ using equation (4). Some hours are necessary to reach a total electron dose of about 10^{-2} C/mm^2. The electron beam current is measured every hour to guarantee its stability during the process.

The measurements were done on Oxygen Free Electronic (OFE) copper, Deoxidized High Phosphorus (DHP) copper, laser treated DHP copper and amorphous carbon coated (a-C) copper.

OFE Copper

OFE copper, also called C10100, is a 99.99% pure copper with an amount of 0.0005% oxygen. It is a commonly used material in vacuum systems. It minimises the presence of oxygen, which deteriorate the thermal and electric properties of the copper and may cause cracks at welds.

Fig. 6 shows the conditioning curves at 300 eV of OFE Cu at RT and at 10 K.

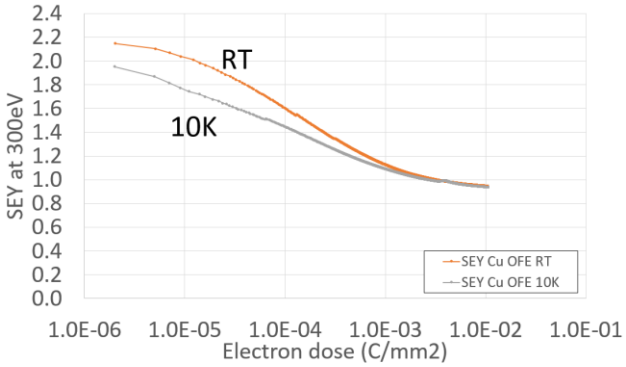

Fig. 6: Conditioning curves of OFE copper with 300 eV electrons at RT and at 10 K.

At RT, the initial SEY at 300 eV is 2.2 and 2 at 10 K. The difference may be attributed to the physisorption at 10 K of some molecules. The conditioning with 300 eV electrons leads, in both cases, to the same final SEY value of 0.9 at a dose of 10^{-2} C/mm^2. These results are in agreement with published data [1, 2, 3].

DHP Copper

DHP copper, also called C12200, is a 99.9% pure copper deoxidized by addition of phosphorus (0.015% to 0.040% P). It is an alternative to the OFE copper used in other technologies than vacuum.

Fig. 7 compares the conditioning curves at RT of DHP and OFE Cu under 300 eV electron bombardment.

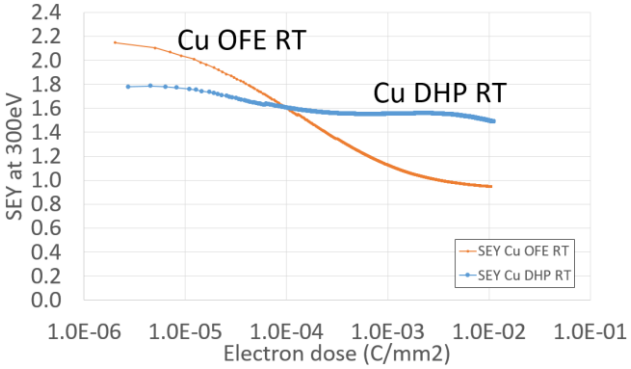

Fig. 7: Conditioning of DHP copper with 300 eV electrons at room temperature compared with OFE Cu.

The result obtained with DHP copper is different from the OFE copper. Although the initial SEY is lower for DHP (1.8) than OFE (2.2), the 300 eV conditioning rate of DHP is smaller. Thus, a final SEY of 1.5 is reached after a dose of 10^{-2} C/mm^2, suggesting a different surface state of DHP from OFE Cu. Preliminary XPS analyses have revealed some silicon traces which origin and impact on the SEY are not clear today [4].

a-C coating on DHP Copper

a-C coating is proposed for the upgrade of the LHC (*i.e.* the High Luminosity LHC) to reduce the SEY of a surface. Since carbon has a low SEY, and since the coating morphology is rough, the SEY of the surface is reduced as compared to metallic samples.

The a-C coating was carried out at CERN by dc pulsed Magnetron Sputtering at 10 kHz under Ar atmosphrere on a DHP copper with a sublayer of 500 nm of titanium, to provide adherence, and a top layer of 50 nm of carbon.

Fig. 8 shows the conditioning curves at 300 eV a-C coating at RT and 10 K.

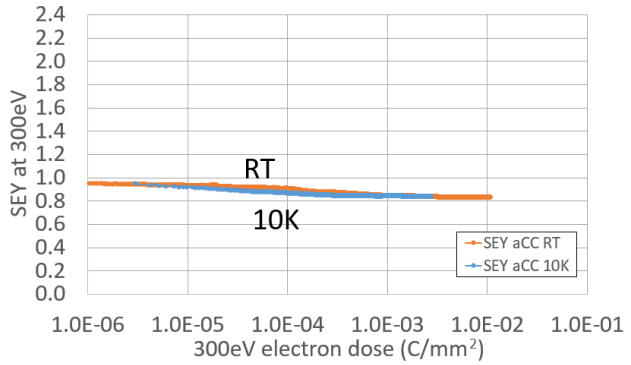

Fig. 8: Conditioning curves of a-C coated copper with 300 eV electrons at room temperature and at 10 K.

Although the Cu bulk is of different nature than stainless steel, since at 300 eV, the electron penetration depth is less than 10 nm; the initial SEY value equals 0.9, in agreement with previous data [5]. The SEY decreases to 0.8 after a 300 eV electron dose of 10^{-2} C/mm^2. The temperature of the sample has no effect on the conditioning level and rate.

Laser treated DHP Copper

Laser treatment is a recent technology that modifies the geometry and the surface state of the material by ablation of matter [6]. Beside the surface modification, it increases the roughness allowing trapping the outgoing electrons to reduce the SEY. This treatment was applied on a DHP copper sample by the University of Dundee using laser parameters similar to the COLDEX samples [7].

The laser surface structuring was performed using a linearly polarized pulsed (10 ps) laser system operating at a wavelength of 532 nm and at a repetition rate of 200 kHz. The diameter of the focused spot was ~13 µm.

The treatment was performed with N_2 flowing at the laser focus point. The structures were obtained by a raster scanning speed of 10 mm/s and 240 pulses per spot using Line Hatch (LH) pattern. The distance between consecutive spots was kept at ~24 µm. The treatment was performed at average laser pulse energy of 5 µJ (laser beam intensity of ~0.4 TW·cm^{-2}).

Fig. 9 shows the conditioning curves at 300 eV of the laser treated DHP Cu at RT and at 10 K.

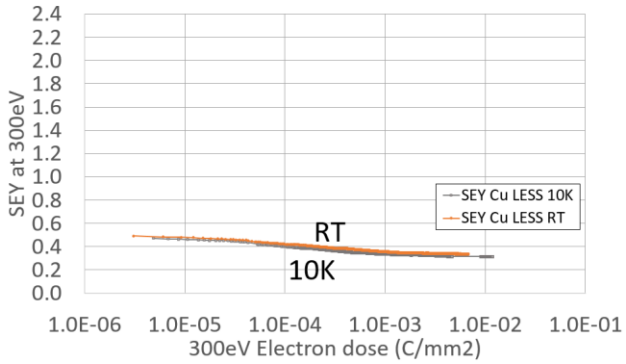

Fig. 9: Conditioning curves of laser treated DHP copper (COLDEX like) with 300 eV electrons at RT and 10 K.

For both temperature, the SEY starts at 0.5. This value is smaller than the one obtained with OFE copper [6]. Beside different laser parameters, a possible origin of this lower value is the use of a DHP copper bulk instead of OFE. Indeed, as shown in Fig. 7, DHP has an initial SEY lower than OFE Cu.

The conditioning rate is weak with a decrease from 0.5 to 0.3 at 10^{-2} C/mm^2. The temperature has no effect on the conditioning rate.

Gas coverage

In order to study the impact of a large air leak, 500 ML of nitrogen was condensed on an OFE Cu sample held at 10 K. It was observed that the conditioning behaviour of this surface is strongly different from the uncovered metallic surfaces.

As shown in Fig. 10, image 1, when a 300 eV electron beam impinges on the ice layer, the solid nitrogen phosphorescence is observed as a green spot. During the irradiation, a "dark stain" appears (image 2). Further bombardment is enlarging the diameter of the "dark stain" as shown in images 3 and 4. The "dark stain" is attributed to the removal of the N_2 layer by the continuous electron bombardment

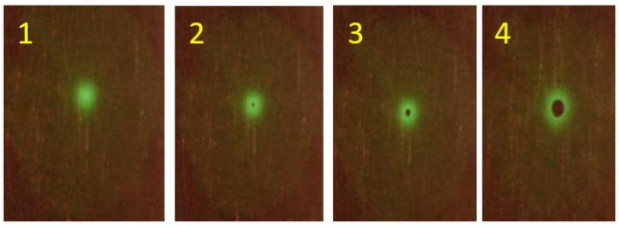

Fig. 10: Evolution under electron bombardment of the green phosphorescent spot due to 300 eV electrons irradiating 500 ML of condensed N_2.

The increase of the N_2 partial pressure due to the impact of electrons was recorded with a calibrated residual gas analyser. The molecular desorption rate was deduced from this measurement. At 10 K for 300 eV electrons, the molecular desorption yield equals 1.8 N_2/e^-.

Fig. 11 shows the time evolution of a typical N_2 residual gas analyser signals for masses 14 and 28 superposed with the measured SEY of the surface. The apparent SEY equals 1 (label 1). Since the surface is an insulator, no electron can reach the surface to neutralise the charge during the bombardment. This leads to a zero current measured on the sample. Thus, according to equation (4), when I_s=0, δ=1.

When the "dark stain" appears (image 2, Fig. 10), label 2, the amount of desorbed gas decreases and in the same time, the measured SEY reach a maximum of about 1.4. After this time, the beam can influence directly the substrate, the conditioning is effective and the SEY decrease again towards 1.

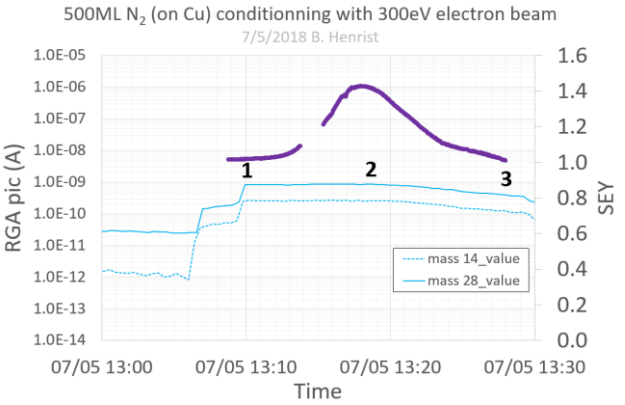

Fig. 11: Conditioning and desorption of 500 layers of N_2 ice on copper with 300 eV electrons at 10 K.

CONCLUSION

A new experimental set-up to measure ESD and SEY of samples held at cryogenic temperature was successfully commissioned at CERN. Electron conditioning studies were performed at a fix energy of 300 eV for which SEY and removal coefficient of N_2 were measured.

At 10 K, the initial SEY of OFE Cu is smaller than at RT. However, after an electron dose of a few 10^{-3} C/mm^2, the difference disappears and both SEY at 300 eV reaches 1. Irrespectively of the sample temperature, the as received SEY of a-C coated and laser treated Cu is below 1. Electron beam conditioning does not trigger temperature difference either. DHP copper has a much different as received SEY and conditioning behaviour than OFE copper, underlying the importance of the material and surface specificities.

A thick layer (500 ML) of condensed N_2 at 10 K is phosphorescent when exited by 300 eV electron. This ice layer is charging like an insulator with a molecular removal coefficient of 1.8 N_2/e^-.

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