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### THE SECONDARY ELECTRON YIELD OF TECHNICAL MATERIALS AND ITS VARIATION WITH SURFACE TREATMENTS

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#### Abstract

Secondary electron emission of surfaces exposed to oscillating electromagnetic field is at the origin of the multipacting effect that could severely perturb the operation of particle accelerators. This contribution tries to illustrate by measurement results, the origin of the secondary electron emission as well as the main reasons for the discrepancies between technical materials and pure metals. The variation of the secondary electron yield with the incident electron energy will be discussed for various types of technical surfaces. The influence of a gas condensation on these surfaces will also be addressed in the context of the LHC accelerator. Various treatments aiming at a permanent reduction of the secondary electron yield will be presented. A special attention will be paid to the decrease of the secondary electron yield under electron or photon impact and to its possible beneficial consequences for the processing of devices prone to multipacting.

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## THE SECONDARY ELECTRON YIELD OF TECHNICAL MATERIALS AND ITS VARIATION WITH SURFACE TREATMENTS

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#### Abstract

Secondary electron emission of surfaces exposed to oscillating electromagnetic field is at the origin of the multipacting effect that could severely perturb the operation of particle accelerators. This contribution tries to illustrate by measurement results, the origin of the secondary electron emission as well as the main reasons for the discrepancies between technical materials and pure metals. The variation of the secondary electron yield with the incident electron energy will be discussed for various types of technical surfaces. The influence of a gas condensation on these surfaces will also be addressed in the context of the LHC accelerator. Various treatments aiming at a permanent reduction of the secondary electron yield will be presented. A special attention will be paid to the decrease of the secondary electron yield under electron or photon impact and to its possible beneficial consequences for the processing of devices prone to multipacting

#### **1 INTRODUCTION**

The electron multiplication on surfaces exposed to an oscillating electromagnetic field causes the phenomenon of multipacting, which can degrade significantly the performance of particle accelerators. Such phenomena have been described in many accelerators, especially in accelerating cavities and more recently in the SPS [1] where measurable pressure increases have been observed during the circulation of LHC type proton beams. In this case the electric field generated by the passing bunches can accelerate electrons which, at their impact with the vacuum chamber create secondary electrons and stimulate neutral molecular desorption. Among other parameters, the multiplication of the incident electrons depend on the secondary electron yield (S.E.Y.) of the bombarded surface which has been measured for most pure metals before 1940 [2], [3], [4], [5], [6]. However these data are not applicable to the real environment of accelerators built from technical materials e.g. stainless steel, aluminum alloys or copper covered with their natural oxide and contaminants. In this contribution we will present the experimental set up and the procedure used to study the S.E.Y of technical materials then show some results concerning these. Lastly, possible methods used to limit the secondary electron yields will be addressed and their

effectiveness discussed in the context of a possible use in an accelerator.

# 2 EXPERIMENTAL SET UP AND PROCEDURE



Figure 1: The experimental set-up

The measurement principle consists in recording simultaneously the current from a sample and at a secondary collector called cage while bombarding the sample by primary electrons with a variable energy between 60 and 3000 eV. A scheme of the set up is shown on Figure 1. The measurement assembly is mounted in a bakeable, all metal U.H.V. system pumped by a 260 l/s turbomolecular pump and equipped with calibrated Bayard Alpert gauge and residual gas analyser. A leak valve is used to feed various gases in the system in



Figure 2: The S.E.Y. of copper for various surface treatments

order to modify the studied surface using ion bombardment. The samples (maximum 13) are mounted on a rotatable sample holder.

A very important feature for the measurement of the S.E.Y. is to control carefully the electron dose needed for

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a measurement. It will be shown later in this paper that the S.E.Y. is strongly dependent on the dose of primary electrons. For this reason the measurements are made using short pulses (typically 30 ms) of low primary electron current (some nano amperes). Under these conditions, the total dose of electrons for a measurement between 60 and 3000 eV is less than 10 nC/mm2.

#### 3 THE DIFFERENCE BETWEEN PURE METALS AND TECHNICAL SURFACES

The difference between pure metals and technical surfaces is illustrated in the Figure 2 which shows the variation of the S.E.Y. measured for a copper sample in the as



received state and after two different *in-situ* treatments: a 300 °C bake-out and an argon glow discharge. In this latter case the S.E.Y. of the pure material is obtained: maximum yield 1.3 at 600 eV primary energy. The highest yield is obtained for the as received sample (greater than 2). A 300 °C bake out decreases this value to



Figure 4: The S.E.Y. of aluminum alloy 6061 as a function of the argon ion dose

1.8. The S.E.Y. of various technical materials is shown on Figure 3 in the as received state: Aluminum alloys have the largest S.E.Y. (greater than 3). For primary energies lower than 300 eV, copper, stainless steel and titanium are equivalent.

The origin of the S.E.Y. difference between pure materials and technical materials is due to the presence of a surface layer (oxide and contaminants) which can be removed by an argon ion bombardment. Furthermore, the S.E.Y. of as received samples is significantly reduced by baking under vacuum (e.g. to 300°C), a process leading mainly to the removal of the water vapor adsorbed on their surface.

The two following experiments illustrate the effect of the oxide layer and of the adsorbed water on the S.E.Y.:

The influence of the natural oxide layer is shown on the Figure 4, displaying the evolution of the S.E.Y. of an aluminum alloy (6061) as a function of the dose of argon



with condensed water

ions impinging on its surface. Starting at an initial value of 3, at 300 eV, the S.E.Y. decreases with increasing bombardment close to the value obtained for pure aluminum: 0.95. The dose of argon ions  $(3.5 \ 10^{17} \text{ ions/cm}^2)$  needed to reach this value is equivalent to the sputtering of a 30 nm thick oxide layer, a value close to that obtained for a similar layer by Auger analysis [7]. If the sample is re-exposed to air, the S.E.Y. is increased to a value higher than 2.5 which is subsequently decreased to the bulk value after sputtering the equivalent of a 6 nm thick aluminum oxide layer.

The S.E.Y. of water condensed at liquid nitrogen temperature on a baked copper surface has been measured for various water thicknesses. In Figure 5 the variation of the S.E.Y. with the primary electron energy is plotted for a clean sample and for coverages corresponding to 45, 85 and 200 monolayers of condensed water.

#### 4 METHODS TO DECREASE THE SECONDARY ELECTRON YIELD

The preceding measurements have shown the strong influence of an air exposure on the S.E.Y. of technical materials. For example the argon ion glow discharge treatment can reduce drastically the S.E.Y. of aluminum but the effect is almost completely lost after an air exposure. In the case of accelerator components or vacuum chambers, it is almost excluded to treat the surfaces *in-situ* with the possible exception of bake- out. Hence the efficiency of all attempts to decrease the S.E.Y. by modifying the surface composition is limited by the unavoidable subsequent exposure to air and to water vapor during installation. Another possible way to lower the S.E.Y. is to change the surface roughness. This causes indirectly a reduction of the emissivity of the surface since the solid angle for electrons to escape without further

interaction with the vacuum chamber can be significantly decreased. A third type of process will be described later

### 4.1 Changing the surface composition

The argon ion glow discharge treatment is a powerful way to modify surfaces by ion bombardment that has been applied previously to the 2 km vacuum system of the former ISR at CERN [8]. By changing the gas used during this treatment, it is possible to produce various surface layers lowering permanently the S.E.Y. even after



Figure 6: Maximum S.E.Y. for niobium after various surface treatments

an exposure to air. This is illustrated in Figure 6 showing in the case of niobium the maximum S.E.Y. after various glow discharge treatments using argon (A.G.D.), argon + 10% oxygen (AO<sub>2</sub>GD), nitrogen (N<sub>2</sub>GD). If most of the beneficial effect of the discharge is lost after anair exposure following an AGD., a significant improvement subsists after a 24 hours exposure to air following the AO<sub>2</sub>GD. or an N<sub>2</sub>GD. In both these cases, the final yield after exposure to air is close to 1.4, a value that can only



Figure 7: S.E.Y. of copper after various surface treatments

be obtained after an in-situ bake-out (B.O.) to 300 °C.

In the case of copper, the curves giving the evolution of the S.E.Y. following various treatments are given in Figure 7. The permanent reduction of the S.E.Y. obtained after an N2GD is also visible. A mild *in-situ* bake out to 100°C restores most of the effect of the treatment when the system has been exposed for 24 hours to air.



Figure 8: S.E.Y. of a TiZrV getter as a function of the activation temperature

Titanium nitride is known to produce a reduction of the S.E.Y. [9] that depends largely on the deposition condition of the films. TiN coatings from various sources show a large scatter in the maximum yields (between 2.5 and 1.5). The best layers can have a maximum yield significantly lower than the S.E.Y. of any known metal in the as received state. After a subsequent 150°C bake out, the maximum yield is close to 1.4, baking to 300 °C lowers the yield to 1.2.

The activation of a getter layer [10] is another elegant way to eliminate the oxide layer. In figure 8 the variation of the S.E.Y. of a Ti Zr getter layer is plotted as a function of the baking temperature. A marked decrease of the S.E.Y. is visible when the layer is heated above its activation temperature. After a 300 °C bake out, the highest yield is less than 1.2.



Figure 9 : SEY of copper after a 24 hours 350°C bakeout compared to the SEY of copper after 5 minutes air exposure at 350°C and 6 hours bakeout at 350°C under vacuum

#### 4.2 Changing the surface roughness

As explained above, the creation of a surface layer with increased roughness can permanently decrease the S.E.Y. This modification can be produced either by a chemical reaction with the substrate or by the deposition of a strongly dendritic layer. The first approach was applied to the case of copper [11] which was oxidized at elevated temperature in air.



The stresses produced during the oxide growth often lead to cracks in the oxide film resulting in an increased surface roughness. It is also important that the formed oxide has a low S.E.Y. as it is typically the case for semi-conducting oxides like Cu<sub>2</sub>O. An *in-situ* bake out to  $350^{\circ}$ C at atmospheric pressure for 5 minutes followed by a 6 hours vacuum bake-out creates a surface with a low S.E.Y. (1.05) as shown on the Figure 9. That treatment causes an increase of surface roughness measured by xenon adsorption at 77K (B.E.T. method) [12] from 1.4 (as received state) to 6.5 (after air bake).

The creation of a strongly dendritic surface on a small copper sample, as shown on the electron microscope picture in figure 10 produces a surface which has a S.E.Y lower than 1, even after an air exposure as it is shown on the figure 11. Similar results have been published by A.N.Curren et al [13].



Figure 11: Secondary electron yield of a textured copper surface

These methods seem to be able to produce surfaces with very low secondary electron yields (lower than 1) but are difficult to apply on a large scale as in an accelerator.

#### 4.3 The "dose" effect

When a surface is exposed to the impact of an electron beam, its S.E.Y. decreases [14] . In Figure 12 the



Figure 12: Variation of the S.E.Y. of copper with the incident electron dose

variation of the S.E.Y. for an unbaked copper sample is plotted as a function of the electron dose. The S.E.Y. decreases for doses larger than  $10^{-6}$  C/mm<sup>2</sup> and its maximum stabilizes for doses greater than  $1 \times 10^{-3}$  C/mm<sup>2</sup> at a value close to 1.2. This effect is permanent when the surface is kept under vacuum (pressures <  $1 \times 10^{-5}$  Pa). The dose effect decreases the S.E.Y. for all primary electron energies as can be seen in figure 13 but the effect is more pronounced for the lower primary electron energies. XPS analysis have shown that after irradiation, the carbon



Figure 13: Decrease of the secondary electron yield with the electron dose normalised to 1 before bombardment

content on the sample surface is increased from 40% to 60% exclusively in the zone of the electron impact. The origin of the effect might be linked with the formation, stimulated by the electron bombardment, of a carbon rich surface layer. The origin of the carbon could be the residual gas (e.g. CO, CO<sub>2</sub>, hydrocarbons) or molecules desorbing from the sample during the electron irradiation (CO, CO<sub>2</sub>) [15]

Although not completely understood and difficult to investigate, as the electron doses involved are too small

for the usual analysis techniques, this effect has been used since very long time in accelerators and is called: processing!

As this effect could be a remedy for the electron cloud effect in the LHC, it has been studied in EPA ( a low energy electron accumulator) at CERN, using a



Figure 14: Variation of the S.E.Y. of with the photon dose

remotely controlable system to measure the secondary electron yield of a copper sample. The synchrotron light radiated by the EPA electron beam (194 eV critical energy) travels along a tangential photon beam line. The sample is mounted at 90 ° from the plane of the synchrotron light and thus is not exposed to direct photons. Three dose experiments have been performed using different bias applied to the sample: -45 V ( i.e. the



Figure 15: The variation of the copper maximum secondary electron yield as a function of the photon dose in EPA

sample was only conditioned by diffused photons), +100 V, +350 V and 820 V (i.e. the sample was bombarded by photoelectrons with approximately 100 eV and 350 eV energy and 820 eV). The variation of the S.E.Y. measured at 820 eV is given in Figure 14 as a function of the primary photon dose. When the sample is positively biased, the curves are very similar to those obtained in the laboratory and saturate at a value close to 1.2. When the sample is only bombarded by diffused photons, the effect is much less pronounced and seems to saturate at a S.E.Y. of 1.6. The maximum secondary electron yields measured under these various conditions are displayed in figure 15 which shows also that the decrease is faster for higher bias voltages (i.e. higher incident electron energies).

#### **5** CONCLUSIONS

The secondary electron emission is a surface dependent phenomenon, more influenced, for technical metals, by the surface preparation than by the material itself. Any insulating layers (e.g. oxides as well as adsorbed water) significantly enhance the emissivity of surfaces. Various surface treatments involving a modification of the surface (e.g. ion bombardment) or coatings (e.g. titanium nitride) can reduce the S.E.Y. although a subsequent ambient air exposure reduces significantly their efficiency. The dose effect (processing) is a well-established and very powerful method to circumvent the problems related with electron multiplication. Nevertheless, this method can only be applied under two conditions:

-A significant electron flux should be available to reach a dose close to  $10^{-3}$  C/mm<sup>2</sup> within a reasonable time and the surfaces involved must be able to withstand the electron bombardment.

-The electron multiplication must be controlled in order to avoid a runaway and possible damage.

Further studies to better understand the physical mechanisms at the origin of the dose effect could result in an improved efficiency of the conditioning procedures in accelerators.

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