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ULTRAHIGH VACUUM SURFACE ANALYSIS USING ELECTRON STIMULATED DESORPTION

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ABSTRACT

The static and dynamical pressures of storage rings vacuum chambers are intrinsic dependent of its cleaning quality. Seeking the improvement of the LNLS vacuum chambers cleaning quality, it was developed a study to identify a cleaning agent which best fits in this application. The qualification process was based on the surface analysis technique known as electron stimulated desorption. As result, it was qualified the detergent IC-115 as the new cleaning agent of the standard cleaning procedure used for LNLS Vacuum Group.

1. INTRODUCTION

The Brazilian Synchrotron Light Source (LNLS) is based on a 1.37 GeV electron storage ring with 93.2 m of circumference, which operates with a beam current of 250 mA. Its vacuum system is mainly constituted of sputter ion pumps and titanium sublimation pumps. The average operating pressure is low 10^{-10} mbar. However, there is a pressure incremental during operation, which is strongly dependent of gas desorption due to synchrotron radiation impinging the chamber walls.
For a good beam lifetime, one electron storage ring has to operate in vacuum pressures at low 10^{-9} mbar [1]. As described above, the pressure of these machines is dependent of the gas desorption during operation and the rate of the gases with high atomic mass has to be low [2]. The gas desorption of species with low atomic mass is essentially dependent of the cleaning quality of the chamber exposed to synchrotron radiation. The cleaning of vacuum chambers for storage ring has been done with the use of organic solvents and in fewer amounts with detergents. But a few years ago, the majority of the organic solvents used to do this job was prohibited and increased the necessity to find out new agents to substitute them. The most provable substitute is the alkaline detergents, as suggested in Ref. [3].
The LNLS does not use organic solvents to clean vacuum chambers since about 1996, but to improve the vacuum system of its vacuum chamber is essential to study the quality of cleaning. In special, find a cleaning agent that best fits the synchrotron machines necessities, a technique called electron stimulated desorption (ESD) is applied. To use this technique to qualify the agents it is necessary make the assumption that a surface tested for ESD gives the same results than photon stimulated desorption (PSD). The PSD is the main phenomenon that desorbs gases in storage rings. In other words, it is necessary to assumptive that ESD is analogous of PSD [4].

2. EXPERIMENTAL METHOD

2.1 ESD EXPERIMENTS

The experiments of ESD were developed in an ultrahigh vacuum chamber (Figure 1). This chamber can be divided in three parts: measurement system, sample chamber and pumping system. The measurement system consists of a residual gas analyser (RGA) QMG 420. The samples were hold to be irradiated with electrons by applying a potential difference between a hot filament (Rhenium) and the sample. The pumping system consists of a 400 *l*/s sputter ion pump and a 950 *l*/s (N_2 equivalent) titanium sublimation pump, this configuration allows the main chamber attain pressures within the range of 10^{-10} mbar.
The execution of the experiments began submitting the samples to the cleaning procedure. The samples were square of 30x30x2 mm obtained from cutting one sheet of stainless steel AISI 316L. Each sample was submitted to the cleaning procedure right before the experiments. The cleaning procedure used was the standard cleaning procedure used for LNLS Vacuum Group and this procedure can be described as:

1. Place the sample in an ultrasonic system during 10 minutes. The solution used was the *detergent* to qualify, diluted in demineralised water, the dilution was specified by the fabricant;
2. Rinse the sample in soften water during 3 minutes. This water is basically a type of water with absence of ions of calcium and magnesium. It is used to take off the contaminants that were attained by the detergent;
3. Rinse the sample in demineralised and deionised water during 3 minutes. It is used to take off the residuals of detergent on the surface and;
4. Dry the sample with hot air during 1 minute.

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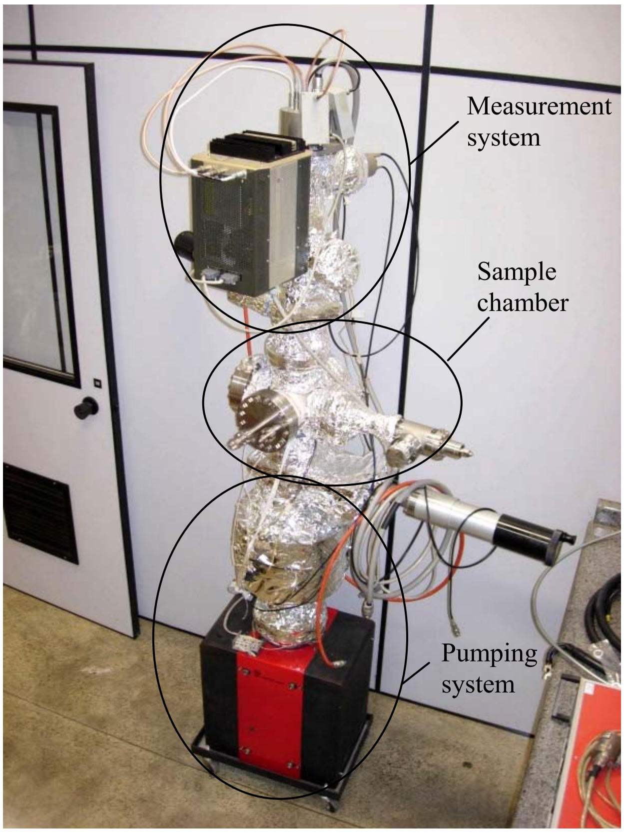

Figure 1 – Experimental chamber.

After cleaning process, the sample was assembled in the sample holder (Figure 2) and the sample chamber was closed to start the pump down, during one hour and half, until the sample chamber pressure reaches low 10^{-6} mbar. During this time the measurement system and the pumping system were isolated from the sample chamber by all-metal gate valves. After the pressure reaches low 10^{-6} mbar, it was started the pump down opening the all-metal gate valve that separates the pumping system from the sample chamber (see Figure 1). The experimental chamber was pumped during 72 hours, until the chamber pressure enter in the 10^{-9} mbar range. From this pressure it was possible to start the bombardment with electrons, but before it was done the outgassing of the electron gun filament during 30 minutes. The samples were bombarded during 5 minutes and the residual atmosphere was monitored with the RGA.

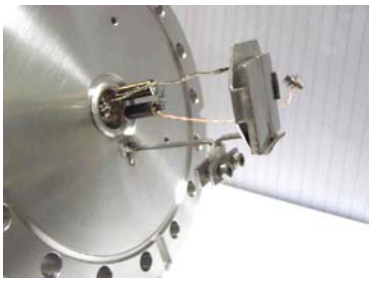

Figure 2 – Sample assembled on the sample holder.

2.2 PUMP DOWN TEST

To complete the experimental test it was submitted a sample using the detergent that presented good results on ESD experiments to simulates a common vacuum use. This test consisted of cleaning a tube with 1.5 m long and 0.0351 m internal diameter. The outgassing rate was compared with a previous calculus presented on ref. [5], which shows an estimate of the outgassing rate of a chamber cleaned with detergent #1. Figure 3 shows a schematic drawing of the system used to evaluate the pump down test.

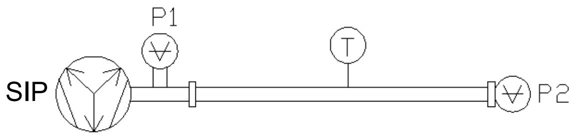

Figure 3 – Schematic drawing of the test system for the pump down test. Sputter ion pump (SIP) 20 *l*/s; P1: cold cathode gauge one; T: thermocouple type "k" and P2: cold cathode gauge two.

It can be seen in this figure that it was used two gauges (cold cathode) to accompany the evaluation of the pump down and a thermocouple type "K" to accompany the temperature of the system. The tube was submitted to a baking process in the range from 140 to 160 °C for a period of approximately 50 hours. The pressure difference between the gauges P1 and P2 can describe efficiently the influence of the detergent in the cleaning quality. The gauge P2 measures directly the quantity of gas resulting from the tube walls with low influence of the pump speed.

3. RESULTS

In Table 1 it can be seen the detergents studied. These detergents are not really specified for vacuum use. The field area of the detergents are following: #1 food industry, #2 laboratory thin film deposition, #3 food industry, #4 molecular biologic area and #5 food industry.

The Figure 4 shows a typical spectrum of residual gas obtained before the bombardment with electrons. While Figure 5 shows a typical spectrum of residual gas obtained during the electron bombardment. In both figures can be seen that the main residual gas are H_2, CH_4, CO and CO_2. The amount of water (m/e=18) is due to not bake out the chamber before the experiments.

Table 1 –Detergents studied.

Designation	Detergent	pH
#1	Break-Up	13
#2	Extran MA02	7.5
#3	IC-115	12.5 – 13.5
#4	Hellmanex II	11.7
#5	Gold Matrix Alu	> 12.0

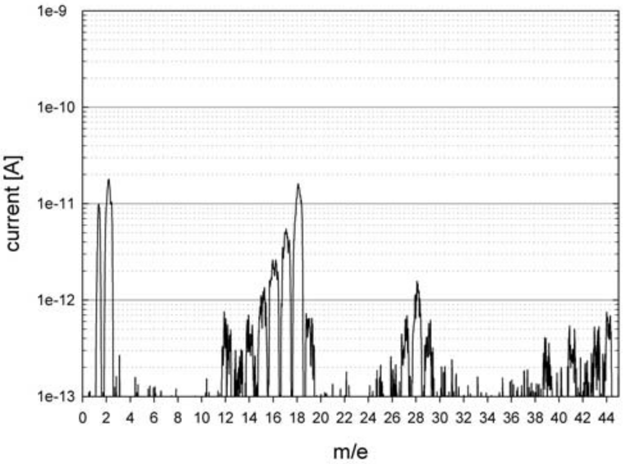

Figure 4 – Typical spectrum before bombardment with electrons.

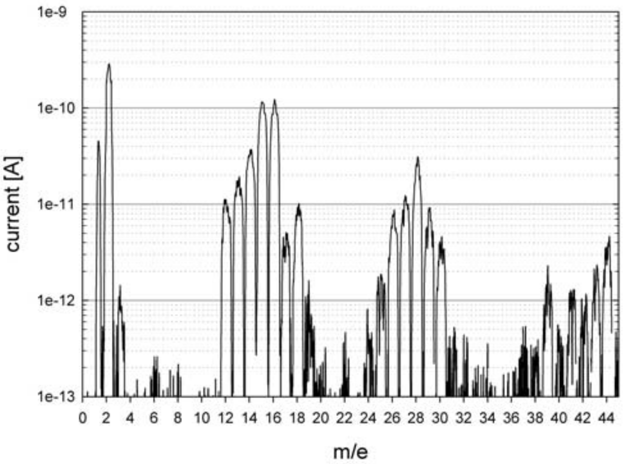

Figure 5 – Typical spectrum during electron bombardment.

To compute the total gas desorption due to electron bombardment, it was done direct the subtraction of the spectrum before bombardment. This was done for all samples studied and the comparison between them. Figure 6 shows the comparison of the gas desorption results from all detergents, which was attained only in the main or more intense gases: H_2, CH_4, CO and CO_2.

The main interest factor for the application in question is desorption of gases with high atomic mass. Thus, it is important the results for CO_2 desorption. It can be seen that the detergents #1, #3 and #4 presented the same results, considering the uncertainties of the experiments execution. The detergent #2 presented the best results for CO_2, while the detergent #5 presented the worst results for CO_2 and for the other gases. Because of the good result presented for all gases presented by the detergent #3 it was selected for the pump down test. Hence, its result can be compared with the outgassing rate of a chamber cleaned with detergent #1 as presented on ref. [5]. Detergent #1 is the actual agent of the standard cleaning procedure of the LNLS Vacuum Group.

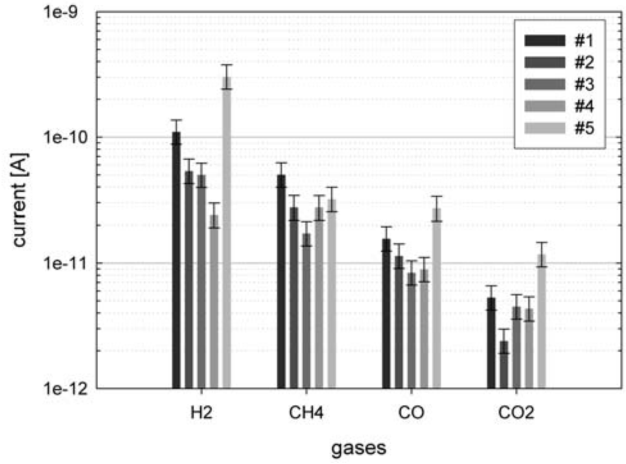

Figure 6 – Results to make the comparison between the studied detergents.

Therefore, the pump down test was accomplished measuring the behavior of the pressures (P1 and P2) during a baking process of a tube cleaned with detergent #3. The Figure 7 shows the evolution of the pressures during the pump down.

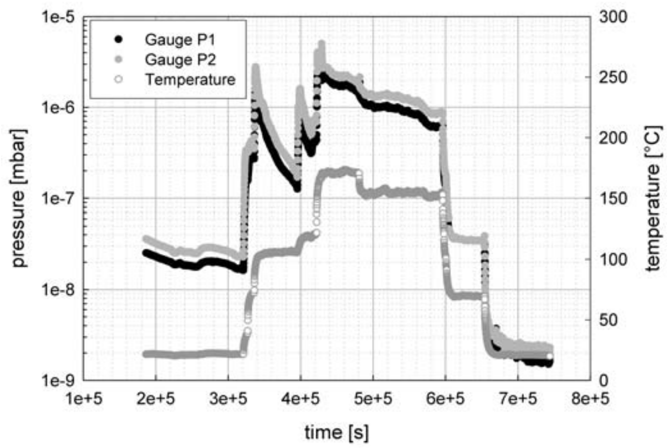

Figure 7 – Pump down during the baking process of the tube 2.

In graphic above it can be seen that the baking process was done in three steps of heating and one step of cooling. The more important thing in this graphic is the pressure difference between P1 and P2 after the baking process, which was very small. This little difference shows low outgassing rate and it can be attributed to a good efficiency of the cleaning process provided by the detergent #3, acquiring with the results obtained on ESD experiments. This difference of pressure is in good agreement with theoretical calculus presented on ref. [6]. Table 2 presents the comparison of the values between the calculated pressure [6] and the pressure obtained on the pump down test, measured approximately 3 days after the baking process. It can be seen a good agreement between the calculated and measured pressures.

Table 2 – Comparison between measured pressure and calculated pressure ref. [6].

	P1 [mbar]	P2 [mbar]
Measured	1.0×10^{-9}	1.3×10^{-9}
Calculated ref. [6]	1.0×10^{-9}	1.3×10^{-9}

Based on the good agreement obtained between the measured and calculated pressures, it can be assumed the calculated outgassing rate [6] as being the outgassing rate obtained on the pump down test. Hence, this value can be compared with the outgassing rate presented on ref. [5], which was calculated for a chamber cleaned with detergent #1. Table 3 shows the values of outgassing rate as consequence of a chamber cleaned with detergents #1 and #3.

Table 2 – Comparison between outgassing rate obtained for a chamber cleaned with detergents #1 and #3.

	Q [mbar·*l*/s·cm^2]
Detergent #1 ref. [5]	1.75×10^{-11}
Detergent #3 ref. [6]	3.0×10^{-12}

In this table, it can be seen a reasonable difference of outgassing rate, showing that the detergent #3 presented a lower outgassing rate than detergent #1, again acquiring with the results obtained on ESD experiments. This result helped to emphasize the better efficiency showed by detergent #3 on cleaning vacuum chambers that will work in ultrahigh vacuum.

4. CONCLUSIONS

It was possible to qualify the detergents for vacuum use with synchrotron radiation. However, it was necessary to make the assumption that the techniques ESD and PSD are analogous. These studies provided important information about the use of the detergents in cleaning chamber for vacuum use with synchrotron radiation. In special, it provided information about the detergent #1, which was used in the standard cleaning procedure of the LNLS Vacuum Group until now. The detergents #4, #3, #2 and #1 showed an adequate behavior for the application required. However, the detergent #3 was qualified to integrate the standard procedure of the LNLS Vacuum Group, because of its adequate behavior and its low cost if compared to the other ones.

When the detergent #3 was used in a common vacuum use, it showed good result. The ultimate pressure obtained on pump down test was in good agreement with a theoretical calculus presented in ref. [6]. In addition, the outgassing rate for detergent #3 was lower than for detergent #1. Hence, these results confirm the results obtained in ESD experiments and show that the detergent #3 fits better in the LNLS Vacuum Group standard vacuum cleaning procedure.

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