

Particle generation of a CapaciTorr[®] D400-2

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Non Evaporable Getter pumps have been used since four decades in various scientific and industrial Ultra High and Extremely Ultra High Vacuum applications. For the majority of applications properties like high pumping speed vs. small size, powerless operation and hydrocarbon cleanliness are main aspects for the usage. In addition to this a growing number of applications nowadays also require particle free systems. In this paper we report on investigations on in-vacuum particle creation during the conditioning and activation process of a CapaciTorr[®] D400-2.

I. INTRODUCTION

Non Evaporable Getter (NEG) pumps are sorption pumps which capture gases by a chemical reaction on their active surface¹. They find application in a variety of Ultra High and Extremely High Vacuum (UHV-XHV) systems encompassing scanning/transmission electron microscopes², photo cathode guns, load-lock and transportation systems³⁻⁵ and portable mass spectrometers⁶. NEG pumps are also used in large research facilities such as particle accelerators and synchrotrons, Fusion machines or large physics experiments⁷⁻¹⁰. Most of these applications are highly demanding not only in terms of the residual gas composition and pump down times but also with respect to particles which might be emitted by the pumping system into the vacuum envelope during their operation. The presence of particles can be highly detrimental to the successful operation of most of the vacuum systems mentioned above. In the case of accelerators, for example, particles can create a variety of issues. They cause beam instabilities and lifetime disruption or serious malfunctioning of superconducting cavities and other high RF-power component because of field emission. To prevent a degradation due to particle contaminations, particle free sections of accelerators at DESY are specified to be ISO 5¹¹ compatible or even ISO 4¹¹ compatible for the superconducting accelerator modules.

Therefore great efforts are spent to reduce as much as possible the generation of particles in advanced accelerator vacuum systems nowadays. This is mainly accomplished through the accurate selection, cleaning and conditioning of the materials and components used in the machine, including UHV pumps. Also, assembling and handling procedures are carried out in suitable certified clean room environment according to high level of standards¹²⁻¹⁴. In spite of this, however, the amount of scientific work so far dedicated to the measurement and quantification of the particle contamination generated by the different types of UHV pumps is quite limited. NEG pumps are very compact devices with large pumping speed, so that they can be mounted very close

to the gas source (e.g. nearby a radio frequency cavity or inside an in-vacuum undulator) effectively reducing the pressure level. To fully exploit this unique feature it is therefore particularly important to measure the level of particles which are released by the NEG pumps during the operation and assess that this level is compatible with the application.

The first generation of NEG pumps were developed in the 70s (SORB AC[®]) and used a cartridge obtained from a coated strip of getter material. The getter powder was cold laminated onto a constantan substrate metal strip which was folded in a concertina shape cartridge^{10,15}. Starting from the 90s a new generation of pumps based on sintered getter disks (CapaciTorr[®]) has been made available¹⁶. The disks, which are vacuum sintered at high temperature, are stacked in a highly efficient and compact pumping structure⁵. The sintering process imparts to the getter disks great mechanical stability while retaining high porosity and surface area, as required to ensure large pumping performance. Thanks to the sintering process the risk for emission of particle from the getter cartridge during the pump operation is greatly reduced. For this reason, even though both families of NEG pumps are currently used in industrial and scientific applications, the CapaciTorr[®] pumps are generally preferred when it comes to particle sensitive applications.

In the present paper the emission of particles generated during the use of a CapaciTorr[®] D 400-2 NEG pump is measured by means of a sophisticated laser particle counter. The NEG pump was subjected to particle count as such, after being cleaned with a nitrogen blow and after a moderate conditioning at 200 °C under vacuum. The effect of a full activation at 450 °C for 1 hour on particle emission was also measured following the conditioning. Finally, the pump was subjected to several conditioning and activation cycles with intermediate nitrogen venting to verify if the particle emission was a continuous process or not.

II. PREPARATION

Before starting the measurements on particle creation during activation it was of interest whether the pump

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generally can be prepared for usage in particle free areas. Therefore the pump was cleaned by blowing with dry ionized nitrogen of 3 bar pressure inside a clean room of class ISO 5¹¹. The particles before and after the cleaning have been measured by means of an MetONE 2400-6¹⁹ particle counter. This version of the MetONE 2400 series detects particle ranges of 0.3, 0.5, 1.0, 3.0, 5.0 and 10.0 μm and operates with an air flow of 1 cfm. The results of the cleaning process are presented in figure 1 where the counted particles per minute and ft^{-3} are shown as function of time. The presented particle sizes

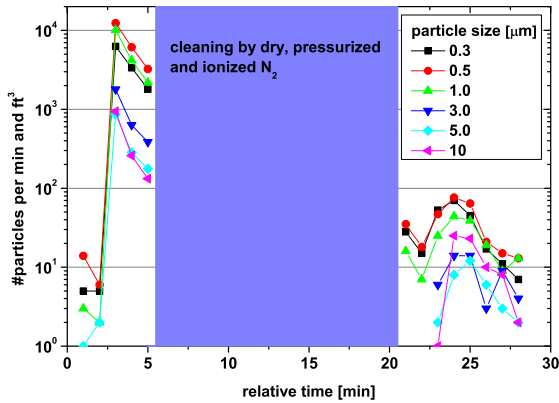


FIG. 1. Particles measured as function of time before and after the cleaning by dry ionized nitrogen CapaciTorr[®] D400-2.

in figure 1 represent the differential measuring results. For instance the black curve labeled with 0.3 represents all particles with sizes between 0.3 and 0.49 μm .

After about 15 minutes of cleaning the number of emitted particles is strongly reduced and compatible to clean rooms of ISO 4¹¹. Based on this result the CapaciTorr[®] D400-2 is basically compatible with the requirements for particle free systems of the DESY accelerators and could be used even close to the superconducting accelerator modules.

III. EXPERIMENTAL SET-UP

To allow for measurements of the particle creation during activation of the CapaciTorr[®] D400-2 an in-vacuum particle counter was used. The particle counter used was a HYT 70XE¹⁸. This sensor is operable between $1.33 \cdot 10^{-11}$ to ≈ 7 bar. The detectable particle size ranges are 0.17, 0.25, 0.3, 0.5 and 1 μm . A drawback of this type of detector is that the effective sensitive volume of the detector is quite small in comparison to its vacuum tube. Therefore the measured number of particles does not represent the absolute number but clearly shows the trends.

After the cleaning procedure described in section II the pump was mounted inside the clean room into a

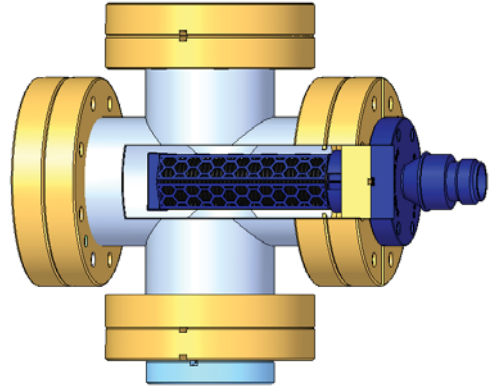


FIG. 2. Experimental set-up: The D400-2 (dark blue) is mounted horizontally into an DN63CF cross. The in vacuum particle counter is connected to the DN40 port on the bottom of the cross.

DN63CF cross. The orientation was chosen to be horizontal while the particle counter was mounted vertically below the D400-2. Evacuation of the set-up was performed by a scroll vacuum pump and a turbo-molecular pump which was connected by means of a bellow to the particle counter. The set-up is schematically shown in figure 2.

IV. RESULTS

To display its sorption performances, the getter material must be first activated under vacuum at 400-500 $^{\circ}\text{C}$ for about 1 hour. This process is required to dissolve the oxide and carbide compounds which are present on the surface of the getter material and which act as a passivation layer. Such a layer (≈ 1 nm thickness) is generated by the exposure of the getter material to air during pump manufacturing and handling and prevent the gas absorption process. During the activation, oxygen and carbon diffuse from the surface into the sub-surface and finally in the bulk, leaving a metallic and reactive surface ready to chemisorb impinging molecules¹⁷. Before carrying out the activation process it is generally advisable to heat the getter at a lower temperature (150-250 $^{\circ}\text{C}$) for some hours to "degass" it, thus allowing physisorbed species, mainly water and CO/CO_2 to be released under vacuum. This process, called conditioning, can be done as a specific step or it can be just carried out during the bake out of the whole vacuum system with the getter pump already installed.

To investigate the particle emission during the whole procedure the particle measurement was started before the conditioning and kept running until the activation was finished. For all the measurements the pressure at the D400-2 and the particle counter was below E-5 mbar and so counted particles are only related to the activation cycles and not to transport from other components²⁰.

Figure 3 shows the emitted particles as function of time for the first activation at all of the D400-2. It is clearly

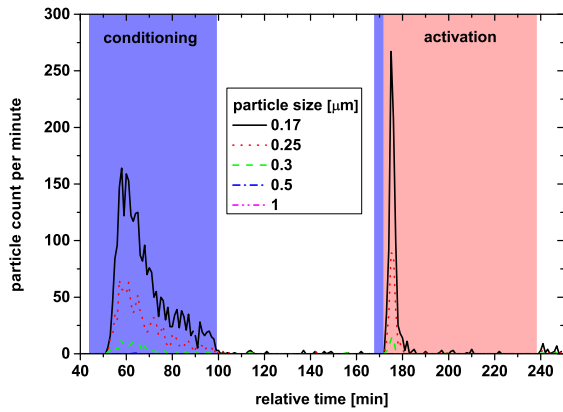


FIG. 3. Measured particles in vacuum as function of time for the first conditioning and activation cycle.

visible that during both processes particles are emitted but different in peak intensity and time dependence. For the conditioning most particles are emitted in the measured size range of $0.17 \mu\text{m}$ with a maximum of about 160. For the activation process in the same particle size range up to 270 particles per minute have been measured. But in contrary to the higher maximum the emission of particles drops down rather fast to background level in case of the activation, during the conditioning process particles are emitted until the end of the process. This results in sum in a higher particle count during conditioning than during activation, which is shown in the end by figure 5.

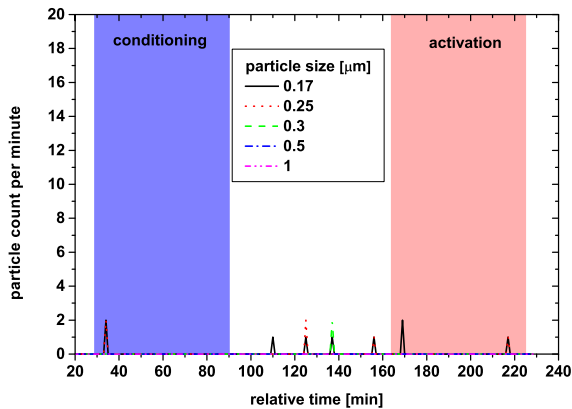


FIG. 4. Measured particles in vacuum as function of time for the fourth conditioning and activation cycle.

To investigate the behaviour of particle emission of the D400-2 in terms of conditioning, the described activation procedure has been repeated a couple of times. In-between each cycle the set-up has been vented with dry nitrogen. The observed particles during the fourth cycle are presented in figure 4. The figure clearly shows a

strongly reduced number of particles counted in comparison to the first activation cycle (figure 3). The number of counted particles during the fourth cycle are so low that they can be attributed to the noise of the detector.

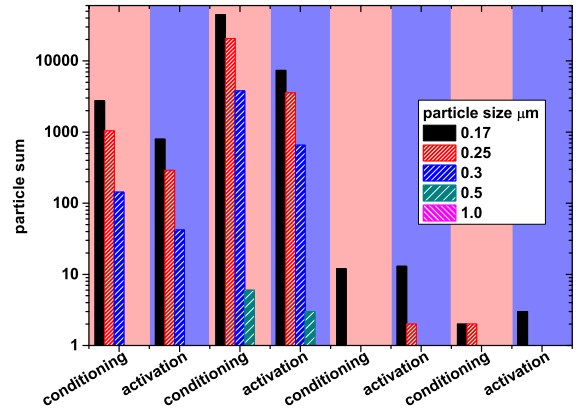


FIG. 5. Summary of the sum of particles measured in vacuum for the four conditioning and activation cycles.

Figure 5 shows the summary of all performed activation cycles. For better comparison the time dependence of particle emission is not shown but only the sum for all particle sizes.

For the time being it is not understood why for the second cycle the counted particles are about one order of magnitude higher than for the first. In addition during the second conditioning and activation particles bigger than $0.5 \mu\text{m}$ have been observed.

After the observed increase of particle emission for the second cycle already the third cycle shows a strongly reduced number of particles, while for the fourth the measured numbers are in the order of the noise of the detector.

From the observed behaviour we conclude that the CapaciTorr[®] D400-2 can be conditioned towards usability in particle free areas.

V. SUMMARY

We have investigated the particle emission caused by a CapaciTorr[®] D400-2 in vacuum during the activating of the pump. The first two activations showed huge numbers of particles emitted from the pump. But already during the fourth activation no particles could be detected. The results show that after proper preparation the pump can be used in particle free vacuum systems of DESY.

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