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COMPATIBILITY OF NON-EVAPORABLE ZAO®-BASED GETTER PUMPS WITH PARTICLE-SENSITIVE VACUUM APPLICATIONS

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Abstract

Many vacuum applications, such as accelerators, optical chambers, superconducting cavities, scanning electron microscopes (SEM), transmission electron microscopes (TEM), are particularly sensitive to dust and require an ultra-clean working environment. Non evaporable getter (NEG) pumps with porous sintered elements are already extensively used in ultra-high vacuum (UHV) and extremely high vacuum (XHV) particle-sensitive systems as well as in industrial applications, laboratories and large R&D facilities. In order to assess the compatibility of NEG pumps with ultra-sensitive devices in vacuum applications, in terms of particle release, SAES research team has been focusing on the development of optimized methods which allow to check, in a controlled and repeatable way, the deep level of cleanliness of a getter pump. In particular, the development of a robust method for particle counting is presented: the main challenges are given by the minimization of background effects and the detection of extremely low levels of counts.

INTRODUCTION

Particle release constitutes a crucial issue in many vacuum applications [1]. Indeed, the presence of dust might affect technological devices creating severe mechanical, electromagnetic and optical concerns: particles can generate electric shorts, sparks and act as field emitter tips under high electric fields; on silicon wafers, they can change properties of the deposited layers, create opens, breaks, morphological defects; they can contaminate masks and optical systems, reducing resolution and introducing optical artefacts; moreover, abrasion and loss of tightness might occur in load lock systems. For these reasons, many industrial and scientific systems are particle-sensitive: from inspection tools, such as SEMs or TEMs to large R&D facilities, i.e. particle accelerators, synchrotrons and superconducting cavities [2, 3]. SAES ZAO® UHV and HV sintered NEG pumps are already widely used in dust-sensitive vacuum systems [4, 5, 6], since they provide high pumping speed and trapping efficiency for H_2, extreme compactness, negligible magnetic interference, they don't vibrate nor need power supply in working conditions and don't require maintenance [7].

Getter alloys developed by SAES from the 70's to the last decades (Fig. 1) have been evolving towards, among the others, the goal of minimizing particle emission from NEG pumps. Indeed, sintered alloys are much less prone to particle release with respect to the compressed ones: the high-temperature sintering process make getter powders be tightly bounded in a single body, with a dramatic reduction of dust emission, still guaranteeing high internal porosity and large surface area. In particular, the most recently developed ZAO® alloy, besides providing larger sorption capacity and pumping speed, lower H_2 equilibrium pressure and better mechanical properties, is characterised by negligible particle emission, making ZAO® NEG pumps products of choice for pumping dust-sensitive vacuum systems.

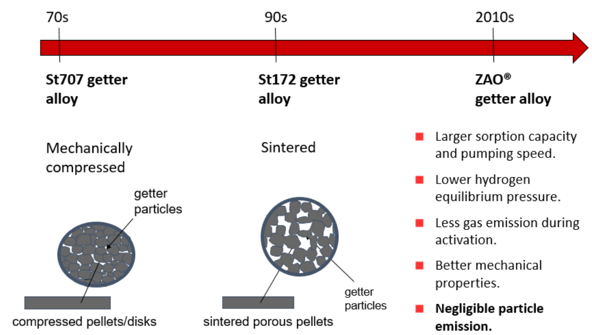

Figure 1: SAES getter alloys evolution during the last decades.

PARTICLE DETECTION SYSTEM

Past Methods for Particle Counting

SAES High Vacuum Division (HVD) across the years has been testing different methods for particle detection, from dipping NEG material in ultrasonic bath to blowing it under filtered and ionized nitrogen flow installing the sample in a closed system [8]. Both methods have shown that compressed disks release more particles than sintered ones, as pointed out in Figure 2 : as an example, a comparison is made for weight lost after ultrasonic bath cycles by getter samples made of sintered (St172, ZAO®) and compressed (St707) alloys. However, the above-mentioned methods have some limitations: dipping tests might force the release of getter material and are invasive, since pumping performance might be affected by the solvent without a controlled drying procedure. As regards blowing the sample in a closed system, it might results in turbulent motions of air within the chamber and the possibility to have trapped particles which are not detected by the collector: these effects generate a high S/N ratio, giving results which might not be reliable for high levels of background.

A New Optimized Particle Detection System

The lack of a general consensus in the vacuum community on the most adequate particle-counting procedure and

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the need to investigate over cleaning treatments for making getter pumps particle-free have lead SAES HVD research team to start a new experimental campaign, with the aim of setting a new procedure for particle counting. The focus has been put on ZAO® UHV disks and tests have been carried out in an ISO-6 cleanroom (24 m^2) at SAES R&D laboratories: a robust particle detection system has been developed, it can give reliable and repeatable results, overcoming the main critical issues of past detection configurations and procedures.

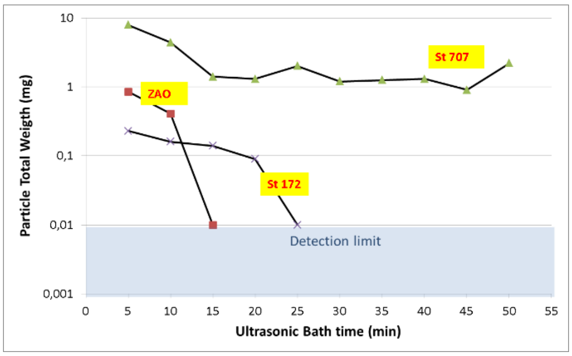

Figure 2: Results of weighting compressed (St707) and sintered (St172, ZAO®) samples after ultrasonic baths.

Limitations of blowing a sample installed in a closed system have been already mentioned: the preferred configuration consists in an open system, where the sample is installed on a rotating bar between the N_2 blowing gun and the collector. The first developed setup is provided with a wheel for manual discretized rotation of the sample, then, in the final setup, with an automatic continuously rotating system (Figure 3). The ionized nitrogen flow passes through a zero-particle filter (< 30 nm) and particles emitted by the sample are detected by a portable counter (TSI AeroTrak® 9510). Counts are reported in particles/ft^3 and are differential: for example, 0.3 micron refers to all particles of dimension included between 0.3 and 0.5 micron. This choice is due to the will of discriminating particles of different dimensions coming from background or from the NEG sample.

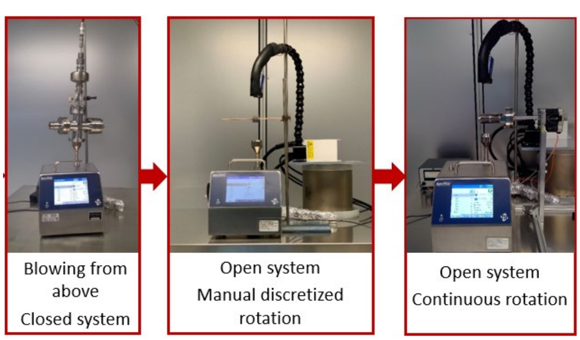

Figure 3: Evolution of particle detection system implemented in an ISO-6 cleanroom in SAES HVD labs.

PROCEDURES AND RESULTS

Minimization of Background Effects

The firstly investigated aspect regards the minimization of background, which could be affected both by N_2 blowing pressure and by the presence of the operator in proximity of the counter. The lowest impacting pressure resulted to be 0.5 bar, which has been selected for sample processing. Moreover, a 30 s countdown time between the trigger given by the operator on the counter and the effective starting of the measurement has been set, in order to let the operator be far enough from the system when the counter is running, avoiding perturbation of the detected signal.

Experimental Setup and Procedure

The optimized experimental configuration is shown in Figure 4 : the sample stack is placed between the blowing gun and the collector, continuously rotating during particle measurement.

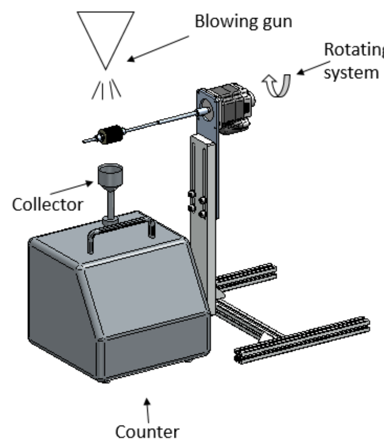

Figure 4: Configuration of the new optimized particle detection system.

The experimental procedure is the following:

- Background acquisition for 5 minutes with nitrogen flowing at 0.5 bar and active rotating system.
- The sample stack is placed on a rotating bar right below the ionized and filtered nitrogen gun, above the counter.
- The stack is made continuously rotate below the jet of nitrogen at 0.5 bar for 5 minutes.
- 30 s of countdown time is set before the 5 minutes measurement starts.

As already mentioned, the detection system and procedure described above have been used for making robust and repeatable measurements on NEG samples, in order to assess the compatibility of NEG pumps with dust sensitive vacuum systems. Moreover, different cleaning (i.e. particle removing) treatments have been investigated and their effectiveness has been checked with the optimized particle counting system.

In Figure 5 background measurements (left) and counts (right) coming from a ZAO® UHV 11-disks untreated stack are shown.

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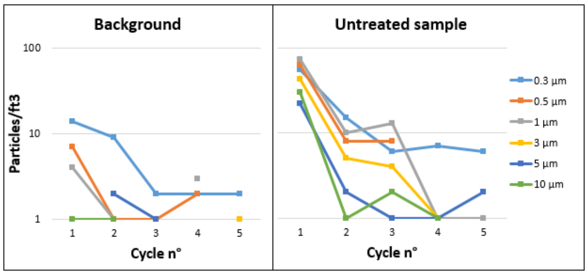

Figure 5: Background counts detected for 5 minutes before sample installation (left); counts detected from an untreated stack of 11 ZAO® UHV disks (right).

At the first minute of counting, detected particles of all dimensions rise up with respect to the background. Masses of high dimensions are few units in background, but few tens are detected when the stack is installed: they are most probably due to getter powder losses.

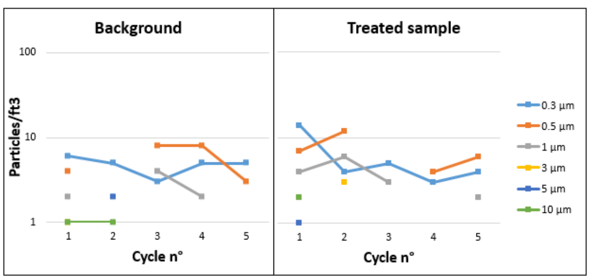

Figure 6: Background counts detected for 5 minutes before sample installation (left); counts detected from a treated stack of 11 ZAO® UHV disks (right).

In Figure 6 it's shown an example of counts coming from a treated ZAO® UHV 11-disks stack: there is no rising up of counts at the first minute. The levels of counts for particles of all dimensions are comparable to those of background; high masses are almost absent: the treatment is effective in removing the initial excess of getter dust after stack assembly.

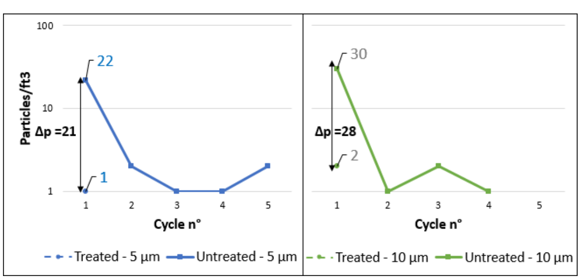

Figure 7: Counts detected from untreated and treated stacks of 11 ZAO® UHV disks for masses ranging between 5 and 10 microns (left) and above 10 microns (right).

A more useful comparison between the two samples can be made by particulate dimension. Figure 7 shows counts detected from the two samples for the mass range between 5 and 10 microns (left) and above 10 microns (right): the effectiveness of treatment becomes evident. High masses are almost absent in background, thus counts recorded for the untreated stack at the first minute are unequivocally coming from getter particulate excess after assembly of the stack, while they are not detected for the treated one. Notice also that the level of cleansing of the treated stack is deep: the untreated sample has some residual counts along the whole duration of the measurement, while the treated one doesn't induce any count after the first minute.

CONCLUSIONS

A robust particle-detection system has been implemented in SAES R&D labs, in order to collect reliable data on particle release, minimizing background effects. The results of counts made in an ISO-6 cleanroom coming from ZAO® UHV getter alloy samples of 11-disks stacks show the repeatability of the procedure and the effectiveness of the investigated cleaning treatments. Further steps include the introduction of translational movement of the sample below the blowing gun and an increased statistics.

The study also shows that ZAO® UHV sintered getters are extremely clean in terms of particle generation and compatible with very demanding dust-sensitive environments. More validation tests will be done also on ZAO® HV samples, which have already shown a good mechanical stability and low particle emission in past dust-detection tests [4].

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