PARTICLE CONTAMINATION IN VACUUM SYSTEMS

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Abstract: Many vacuum devices, like RF cavities, are sensitive to particle contamination. This fact has motivated a considerable effort of cleanliness from the SRF community. The present paper reports the first results of a general study trying to identify the most contaminating steps during assembly and vacuum operation of the cavity. The steps investigated here are gasket assembly, evacuation and venting of the vacuum system, and operation of sputter ion pumps.

1- Introduction

It is well know that dust particles are terrible enemies of Superconducting Cavities [ref 1]. Recently, efforts have been made to improve the cavity cleaning techniques. Generalised use of automatized chemical treatment [ref 2] and high pressure rising facilities [ref 3] have improved considerably the cavity performance level. But this effort towards cleanliness can be spoiled if the next steps in the cavity life (assembly, and operation under vacuum) recontaminate its surface. It has been shown on a statistical basis that cavities having had trouble during assembly steps or vacuum operation, have a significantly lower field emission threshold and overall performance level [ref 4].

The risk of contamination during the cavity assembly and vacuum operation must be measured, and minimized. The present paper reports the first results of a general study intended to identify the most dangerous steps and components:

1. gasket assembly
2. valve operation
3. pre-pumping and venting
4. steady state pumping by ion-pumps and getters
5. particle liberation by walls under the influence of shocks or vibrations

The present paper will describe only results on items 1, 3 and 4. The main tools for this study are particle counters operating in air or under vacuum. The counters are placed close to the component or to the location of the suspected contamination during the abovementioned operations.

2- Particle counters description

Met-One 205 Model
This counter detects the number of particles contained in 28.3 liters air volume. This volume is pumped through a conic head sensor and then the particles are detected with a laser diode. The detected particle sizes are: 0.16, 0.2, 0.3, 0.5 , 1 and 5 μm.

HYT PM 250 Model
The HYT PM250 sensor can operate in air or in vacuum. The particles fall through a window crossing in its center a laser beam of about 1 mm diameter. Photodetectors receive the light scattered by the particles. The particle size is proportional to the intensity peak. The detected particle sizes are: 0.19, 0.27, 0.3, 0.4 and 0.5 μm.

Counter calibration
The two counters were used in the same gas flow in order to compare their countings. The counting ratio K between both counters was not exactly the same for all particle sizes:

- 0.16 μm < X < 0.29 μm  ⇒ K = 7
- 0.3 μm < X < 0.49 μm  ⇒ K = 5.4
- All particle sizes  ⇒ K = 6.2

From this last value, the sensitive area of detection of the PM250 counter was determinated: $S_{eff} \equiv \frac{S_{\text{window}}}{6.2} = 0.4 \text{ cm}^2$

In the rest of this paper, the particle countings by the PM250 sensor can be transformed into particle fluxes by division by the effective area of detection: $S_{eff}$.
3- Particle contamination by gaskets setting

The particle contamination by the gasket setting was evaluated for conflat (CF35) and helicoflex gaskets. This kind of measurement does not require a counter able to work under vacuum, so the Met-One sensor was used for convenience. The sensor was installed directly downstream the tested gasket (fig: 3.1). Due to the flow aspiration, it can reasonably be assumed that all generated particles are detected. The experiment was conducted in a clean room class 100, by trained operators. All components were washed with 18 MΩ deionised water prior to assembly.

For the tests we used a particular process which is as follow:
1. Installation of a cleaned joint
2. Installation of a cleaned flange
3. Installation of the cleaned screws on the top of the flange
4. Installation of the cleaned nuts and washers
5. Tightening without moving the screws

Experimental device:

Figure 3.1: Experimental set-up used to measure the particle contamination during CF or Helicoflex setting.

Results:

Diagram 1: Setting of a CF35 flange - Average on 10 gaskets - Pipe of 120 mm. long.

Diagram 2: Setting of the CF35 flanges Average on 10 gaskets - Pipe of 500 mm. long.

Diagram 3: Setting of the flanges with an Helicoflex gasket (no:15040) - Average on 8 gaskets - Pipe of 365 mm. long.

The standard deviation S results, on 10 measurements for all the processes are as follow:
Diagram 1: S = 180 for m* = 480
Diagram 2: S = 16 for m* = 19
Diagram 3: S = 18 for m* = 42
(*: m = average)

Conclusion

- Gasket assembly is contaminating;
- There is no clear influence of the nature of the gasket;
- No particles are liberated between assembly steps, this particle generation is due to the operator. Some of the particles are of human origin (this contribution is smaller if the pipe is higher); some particles may also be liberated by shocks or vibrations during the assembly.
4- Particle contamination by Sputter-Ion pump

The sputter-ion pump tested was a Varian Vacion Plus 75 StarCell. The PM250 sensor was placed just below the pump for a better sensitivity (fig 4.1).

As can be seen in fig 4.2, the pump in normal operation (\( p < 10^{-4} \) Pa) does not generate particles since the measured contamination level (1.42 particles/min) is almost the same as the detector background noise (1.25 particles/min). The only particle generation observed occurs during startup (\( p \approx 10^{-3} \) Pa). Some particles bursts (about 10 particles) are also observed during arcing. Of course, the ion-pump can also librate particles because of shocks or vibrations, like any vacuum chamber wall. These contributions can be minimized if the pump is operated in vertical position, with the flange on the top.

**Experimental device:**

![Experimental device](image)

*Figure 4.1: Experimental device for the measurement of the dust contamination by the sputter-ion pump.*

**Results:**

![Particles counting](image)

*Figure 4.2: Particles counting in UHV system after a 300°C baking of the sputter-ion pump*

Generally, the particle sizes distribution is as follows:

- \( \approx 50\% [0.19 \, \mu m; 0.27 \, \mu m] \)
- \( \approx 10\% [0.27 \, \mu m; 0.3 \, \mu m] \)
- \( \approx 15\% [0.3 \, \mu m; 0.4 \, \mu m] \)
- \( \approx 10\% [0.4 \, \mu m; 0.5 \, \mu m] \)
- \( \approx 15\% \geq 0.5 \, \mu m \)

5- Contamination during pre-pumping and venting operations

Evacuation or venting of the cavity are also potentially contaminating steps because turbulent gas flow can release and transport particles. To evaluate this contamination we used an experimental device shown in fig 5.1. For pre-pumping, the regulation valve \( V_1 \) was closed; for venting \( V_1 \) was opened.

This experiment showed that the particle generation is observed mainly at the beginning of the evacuation process, and practically stops when the vacuum level becomes lower than \( 5.10^4 \) Pa. A smaller number of particles is generated if the evacuation is slower.

Particle generation during venting is also very significant, specially at the beginning of the process. Here, the opening of valve \( V_1 \) determines the speed of the venting. The influence of this speed on particle generation has not yet been examined in detail.

As will be justified in the next paragraph, particles are likely to be generated at the level of the valve \( V_1 \) itself, where the air speed and turbulence is largest. Reduction of the opening of \( V_1 \) reduces the air flow, but not the air speed at
V₁, and this might explain why particles are always generated during venting. The only plausible remedy we propose against this contamination is a filter located between V₁ and the vacuum vessel.

We have noticed also, when we pumped quickly, a formation of water droplets which can be eliminated with a 60°C baking of the vacuum vessel (fig 5.2). The difference between the percentage of the 0.5 μm particles for the experiment at 20 °C and for the experiment at 60 °C proves that the biggest particles are water droplets.

**Experimental device:**

![Diagram 5.1: Dust particle transport device](image1)

**Figure 5.1**: Dust particle transport device

**Figure 5.2**: Particle sizes distribution function of the operating temperature. Regulation valve is closed, V₂ is opened suddenly.

6- Contamination induced by the flow speed

In order to investigate this contamination, clean air coming from a class 100 laminar flow was pumped through a 1.20 m. long DN 40 pipe, with an adjustable flow rate (fig 6.1). The flow rate was controlled by a diaphragm at the upper end of the pipe, and by a regulation valve located close to the pump.

The source of the observed particles is probably the diaphragm, where the air speed is highest. As can be seen from fig 6.2 and 6.3, the relevant variable is not the flow rate, but the flow speed, which should be kept below 2 m.s⁻¹ at the diaphragm location for a particle-free operation. Note that the threshold between laminar and turbulent flow in the vicinity of the diaphragm occurs for flow speeds of about 2 m.s⁻¹. It is then tempting to correlate the onset of particle generation with the onset of turbulence. Further experiments will be undertaken to confirm this hypothesis, but we can already say that evacuation and venting of the cavity or vacuum vessel are contaminating steps, unless the flow is kept slow and / or laminar during these operations. This constraint applies not only at the cavity level, but also everywhere in the duct, including the narrowest sections, since the particles generated there can be transported on long distances (1.2 meters in our experiment).

**Experimental device:**

![Diagram 6.1: Experimental set-up to measure the particle contamination induced by the flow speed](image2)

**Figure 6.1**: Experimental set-up to measure the particle contamination induced by the flow speed.
Results:

Figure 6.2: Number of the detected particles (0.19µm < size < 0.5µm) as a function of the pumping speed.

Figure 6.3: Number of the detected particle (0.19µm < size < 0.5µm) as a function of the flow speed.

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References

[ref 3]: HP rinsing
  D. BLOESS, Private communication;
  P. KNEISEL et al
  6th Workshop on RF superconductivity, CEBAF (1993), p. 628
[ref 4]: "A statistical analysis of the risk of dust contamination during assembly of RF superconducting cavities" C. Antoine, 6th Workshop on RF superconductivity, CEBAF (1993), p. 1047