COMPARATIVE STUDIES OF THREE METHODS TO AVOID PARTICULATE CONTAMINATION IN LARGE UHV DEVICES

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Abstract :

With the aim to arrive at procedures allowing the replacement of superconducting cavity main couplers in-situ, three methods to avoid particulate contamination have been tested : a localised and movable clean room named "Lola", a class 100 clean room and a dynamical confinement method. The results show that contamination by particles normally present in ambient air can be completely avoided by these three methods. As to contamination by particles generated in-situ as a result of human and mechanical interventions, we observed that only dynamical confinement can reduce the level of contamination. Clean room and "Lola" local clean room have not a strong efficiency on this kind of contamination. We observed that the efficiency of the dynamical confinement method is correlated with the speed of the flow used for the confinement and in our case, a speed of around 1,5 m/s is required to minimise the contamination instead of 0,3 m/s recommended by semiconductor manufacturers. In fact, the particles generated in-situ in large UHV device have higher size and density compared to particles identified in semiconductor clean rooms.

1 - Experimental set-up

Three methods - Class 100 clean room, Local clean room "Lola", dynamical confinement method - to reduce particulate contamination during the replacement of the main couplers in-situ have been checked in different conditions to estimate their efficiency.

In order to have some results close to the real case of the superconducting cavities, we used for our measurements a "dummy cavity" with the same flanges as a cavity.

The number of particles is measured with a HIAC ROYCO particle counter. This counter uses a laser diode to detect the number of particles contained in a 28,3 l air volume flowing through the counter per minute. The particle counter had been tested with the suction cone placed in different positions with respect to the flow of contamination and the measurements show that the position of the suction cone had not a strong influence on the number of particles detected on a contaminated air flow with a speed lower than 20 m/s.

1.1 - Local clean room named "Lola"

"Lola" is a flexible and transportable local clean room. The Lola local clean room is used to reduce the level of contamination when we want to work outside the clean room (see figure 1) [1].

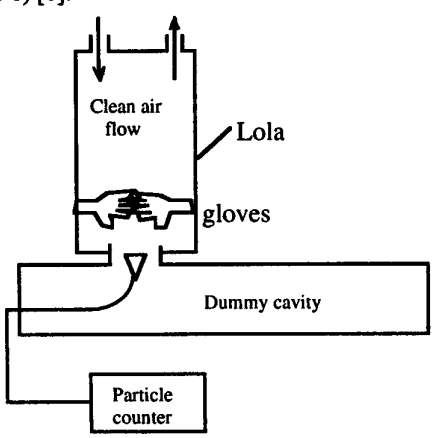

Figure 1 : *Standard set-up of the Lola.*

1.2 - Dynamical confinement method

The dynamical confinement method consists of the injection of a clean air flow in the device to be protected - in our case through the cavity - to repulse the particles outside the critical area (see figure 2). The speed of the flow is chosen to compensate the speed of sedimentation (figure 3). This speed (in m/s) is given by the equation :

$$V_s = \frac{D_P^2\,\rho\,g}{18\,\mu}\,C \quad \text{(Eq. 1)}$$

D_P: particle diameter (m)
ρ: volumic mass (kg / m^3)
g: 9,81 m / s^2
μ: air viscosity NTP = 1,8.10^{-5} Pa.s
C: CUNNINGHAM factor

The Cunningham factor is given by the relation :

$$C = 1 + 1{,}246\,K_n + 0{,}42\,K_n\,e^{\frac{0.87}{K_n}} \quad \text{with} \quad K_n = \frac{2 \times I}{D_p} \quad [2][3]$$

where I is the mean free path of the gas

in the case of NTP air, $I = 6{,}52.10^{-8}\,m$

$$so : C = 1 + \frac{1{,}625.10^{-7}}{D_p} + \frac{5{,}477.10^{-8}}{D_p}\,e^{-6.672.10^6\,D_p}$$

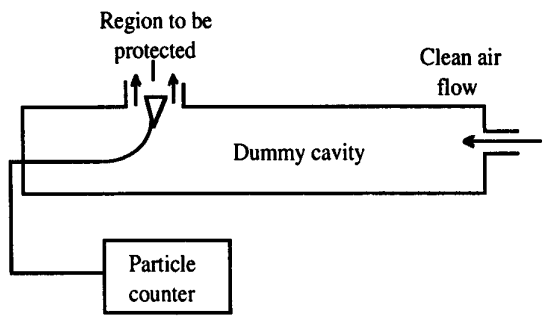

Figure 2 : *Method of dynamical confinement to prevent contamination by particles.*

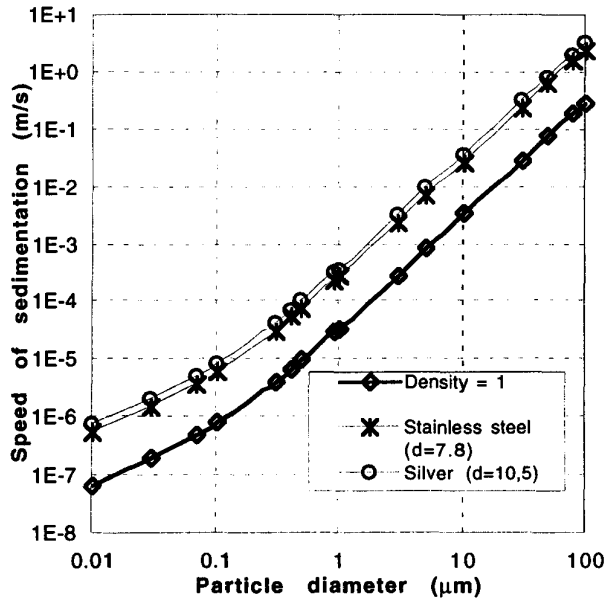

Figure 3: Dependence between the speed of sedimentation and the density of the particles (calculated with Eq. 1).

For our experiments, the clean air flow is provided by a fan (Isoconcept type CVP) and an absolute filter (Millipore 0,5 micron). The speed of the flow can be regulated step-by-step and Table 1 gives the equivalence between the level indicated on the fan and the flow speed measured in the region to be protected. To reduce the contamination around the region to be protected, we associated the localised clean room Lola and the dynamical confinement method.

Level	Flow speed (m/s)
10	1.6
5	1.4
3	0.7

Table 1 : Equivalence between the level indicated on the fan and the flow speed measured in the region to be protected.

2 - Natural particulate contamination

Definition : We call "natural particulate contamination", the contamination contained in the ambient air of the room where the counter is installed. Semiconductor manufacturers always work in clean rooms and all their devices and tools are manufactured in "clean conditions" to minimise contamination. These conditions cannot be applied easily to large UHV devices so "our" natural particulate contamination is different from that observed in semiconductor plants.

2.1 - Orders of magnitude

The reproducibility of the number of particles detected is quite good. The use of a roof to confine a room in the building reduces the natural contamination by a factor of 2 from 30 000 to 15 000 particles/minute detected in a volume of 28,3 l. The slope of the curve :

$$Log(\text{Nbr of parts}) = f\left[Log(\phi\ \text{parts})\right]$$

is close to -2 which is the value predicted by the theoretical distribution of natural particulate contamination in a standard aerosol [4].

2.2 - Efficiency of the three methods

To compare these methods, we counted the number of particles detected during one hour without any intervention. Four measurements have been done for each value and to maximise the natural contamination in the case of the dynamical confinement method, we made our measurements outside the clean room (30 000 parts/min in 28,3 l). The results show that the level of contamination obtained by the three methods is very low, less than 15 particles per hour, so 5 orders of magnitude lower as compared with the natural particulate contamination. All these methods are very efficient to prevent natural particulate contamination and there is no significant difference between the efficiency of these three methods.

3 - Contamination generated in situ

Definition : We include in this sort of contamination, all contamination resulting from a human or mechanical intervention except the natural contamination which has been quantified before.

3.1 - Studies of some sources of particles contamination

To test the efficiency of the method proposed to reduce particulate contamination, we need a reproducible source of contamination. The natural particulate contamination has a very low density of particles bigger than 10 microns in diameter and the experiments using an aerosol generator were unsuccessful. In fact, this device first mixes the particles in a liquid before vaporisation. But, the bigger the particle is, the higher is the sedimentation speed and thus, it is very difficult to obtain an aerosol with reproducible characteristics. To obtain some in-situ generated particles, we decided to study some suspected sources of particles such as : hands movements (applauses), silver plated screws, flanges... The reproducibility of these sources of contamination is quite good. This contamination is generated at the axis of the main coupler port, at one centimetre above the flange. The main coupler port is in a vertical position.

3.2 - Effect of the flow used for the dynamical confinement on the number of particles detected

We observed that after 15 seconds injecting a cleaned air flow (1,5 m/s), the number of particles detected during 5 seconds decrease from around 1200 to 1 particle.

3.3 - Efficiency of the three methods

The clean room and the "Lola" device seem to have no effect on the number of particles detected after an in-situ contamination. Only the dynamical confinement method reduces the level of contamination : a few particles detected after several applauses above the dummy cavity flange (figure 4) after scratching some silver plated screws (figure 5) and after mounting and dismounting the flange (figure 6).

To reproduce a scratch with the same force, we used a spring diapason. Each count corresponds to the average of 5 tests in which always a new screw (M8-55) is scratched 25 times in a minute.

3.4 - Particles released from surfaces

Surfaces are always covered by a large number of particles of contamination. In fact, on niobium and after a chemical etching of 60 microns and a water rinsing in a clean room, the number of particles is about 100 per square centimetre

[5]. This gives, "in clean conditions", more than 170 000 particles on the surface of a main coupler extension. As it is very difficult to manufacture large UHV devices in clean conditions, it is more realistic to expect a contamination one order of magnitude higher. Theses particles, attached on the surface by Van der Waals or electrostatic forces, can be released by mechanical vibrations.

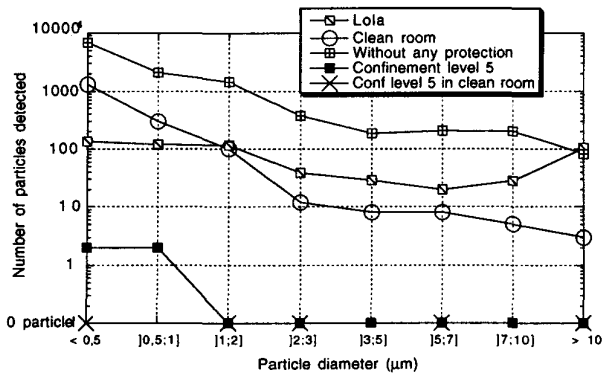

Figure 4 : *Contamination due to ten applauses in front of the particle counter. Counting duration : 1 minute.*

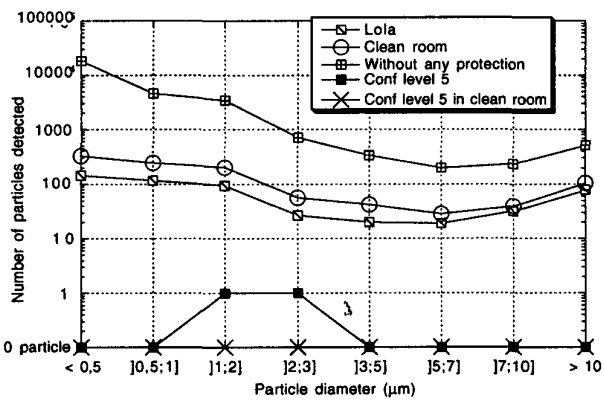

Figure 5 : *Contamination generated by scratching a silver plated screw. Counting duration : 1 minute.*

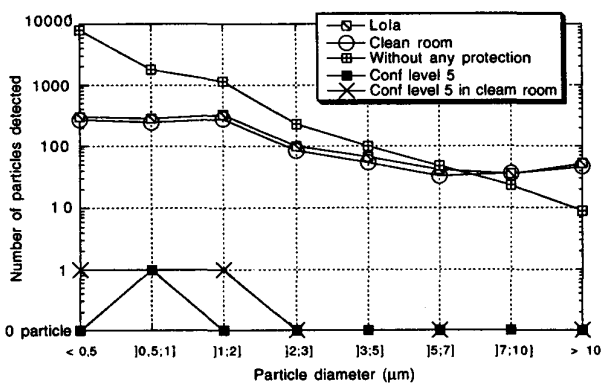

Figure 6 : *Contamination generated when mounting and dismounting the flanges. Counting duration : 1 minute.*

In this part, we will try to estimate the level of contamination generated by this in-situ source of contamination. Two sorts of experiments were carried out : mounting and dismounting a flange with all the screws (16) and shocks on the wall of a main coupler extension (Ø103-530 mm). All the measurements were done in the clean room to avoid natural particulate contamination.

Table 2 collects all the results obtained. The series marked by an asterix(*) correspond to a "smooth" mounting or dismounting of a flange. We just try to reduce the vibrations generated when tightening the 16 screws. We observed that the number of particles detected is much lower.

	]0,5;1]	]1;5]	]5;30]
mounting	1549	286	173
dismounting	300	164	29
mounting*	325	168	34
dismounting*	18	10	5
Shocks in clean room	1477	749	141
Shocks + conf in clean room	86	90	8

Table 2 : Contamination measured when mounting a flange and squeezing the 16 screws and by shocking a main coupler extension.

The "shocking test" consist in five strong shocks for each test on the wall of the tube. The results (average of 4 tests) show that only a confinement method can reduce the level of contamination generated by vibrations.

4 - Consequences of the injection of a clean air flow on the contamination of the cavities

The clean air flow is injected across the valve and we measured the number of particles detected without any operation. Three series of measurements (5 counts for each series) have been done in the clean room using a flow of level 0, 5, 10. The results obtained (counting duration 1 hour) show no particle detected.

To achieve a dynamical confinement near the flanges of the main couplers in the cavity, the valve must be opened and closed to inject the clean air flow. With these measurements, we wanted to estimate the number of particles generated and transported by the clean air flow injected across the valve into the cavity.

Two sorts of valves from VAT had been tested : "a cavity pre-vacuum valve" which is an All-Metal Angle Valve (series 57) and an RF All-Metal Gate Valve (series 47).

The results obtained for a pre-vacuum valve proved that the number of particles generated by one opening and one closing of a non-cleaned valve is, in the worst case lower than 60 particles.

The number of particles generated by an opening or a closing of an RF gate valve is quite reproducible (see figure 7) and much higher than the one obtained with the previous kind of valve. Moreover, the number of particles is higher when opening the sector valve[6]. This can be easily explained by the mechanical vibrations generated during the closing or the opening of the valve. These vibrations produce a strong release of particles from the surfaces inside the valve.

5 - Effect of the flow of the clean room

In the previous measurements, we observed that the air flow of the clean room can upset a dynamical confinement method. In this section, we wanted to see if the use of clean rooms for assembly of large UHV devices always protect these devices from contamination. Two series of tests were done using in-situ generated particles obtained by scratching silver-plated screws and by opening and closing a flange. All the counts were done in the clean room.

5.1 - Measured contamination using scratched silver-plated screws

Our measurements show that a circulating flow generated by the flow of the clean room inside the dummy cavity increases

the level of contamination by a factor of 5 (figure 8). This contamination may be reduce by closing properly the dummy cavity.

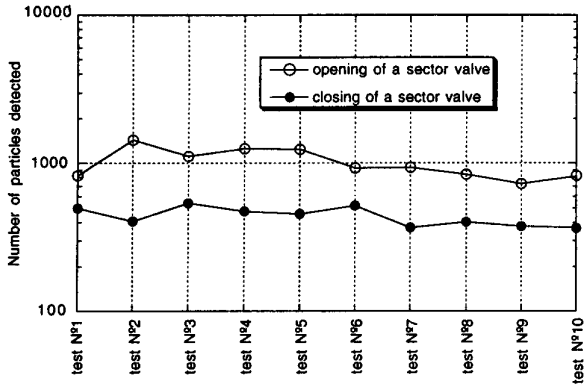

Figure 7 : *Number of particles generated by an opening or a closing of an RF gate valve (named sector valve at CERN).*

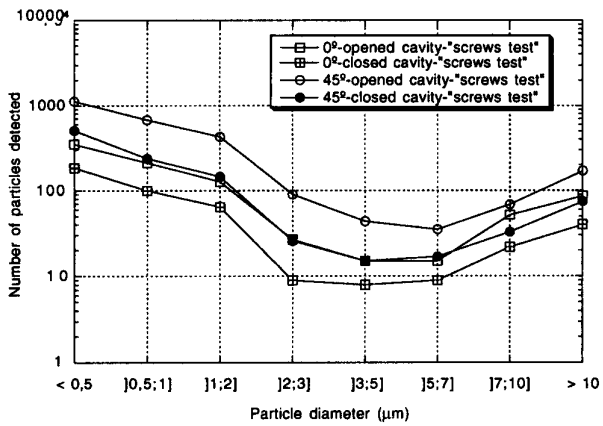

Figure 8 : *Effect of a circulating flow inside the dummy cavity on the number of particles detected after scratching some silver plated screws. Counting duration : one minute.*

Figure 9 confirms that the best results are obtained using the dynamical confinement method with a speed of around 1,5 m/s (level 10). We also observed that the direction between the flow of the clean room and the axis of the main coupler tube has a strong influence on the number of particles injected in the dummy cavity. An angle of 45° gives the worst results. This result can be explained by two factors. Firstly, the oblique direction of the flow of the clean room induces turbulences near the flange and these turbulences can cause the injection of particles into the dummy cavity. Secondly, the gravity force tends to introduce the particles into the cavity. In fact, an angle of 0° gives the best results compared to 45° or 90°. The same remarks can be applied to the results obtained using the dynamical confinement method.

The number of particles detected with an angle of 90° is higher for heavier particles and this observation confirms the influence of the mass of the particles generated in-situ on the number of particles detected in the cavity.

Similar results were obtained with in-situ generated contamination by opening and closing a flange.

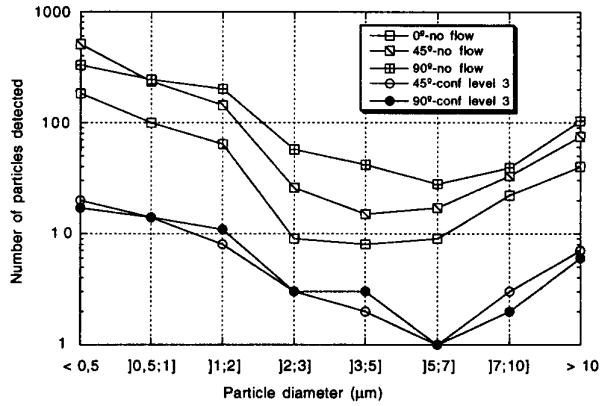

Figure 9 : *Influence of the direction of the flow of the clean room on the number of particles detected in the dummy cavity after scratching a silver-plated screw in the top of the flange. Counting duration : one minute.*

Conclusion

Our measurements show that the three methods tested are efficient to prevent natural particulate contamination. The results obtained for all these methods are similar and the contamination level without any human or mechanical operation is improved by 5 orders of magnitude with respect to unprotected rooms.

The results proved that the clean room and the Lola protection have the same low efficiency to avoid in-situ generated particles. To avoid these particles, the dynamical confinement gives the best results, close to zero particles are detected with a clean air flow injection speed around 1,5 m/s, 5 times more than what is recommended by semiconductor manufacturers.

The dynamical confinement method is also the only method which can reduce the contamination due to the particles released by the surfaces during mechanical vibrations.

Injecting the clean air flow across the valve does not increase significantly the contamination. Most of our measurements give zero particle detected, in the worst case, less than 60 particles with a diameter lower than 1 micron.

References :

[1] A. GRILLOT, CERN Technical note AT-VA 92-03.

[2] M. KNUDSEN, WEBER S., *Ann. Physik*, **36**, 982 (1911)

[3] E. CUNNINGHAM, *Proc. Roy. Soc.*, **83 A**, 357 (1910)

[4] N.A. FUCHS, R.E. DAISLEY, M. FUCHS, "The Mechanics of Aerosols", Pergamon Press (1964)

[5] J.M. JIMENEZ, PhD Thesis, CEA-Saclay/DSM/Service d'Etude des Accélérateur (1994)

[6] J.M. JIMENEZ, N. HILLERET, CERN LEP200 Note (1995).