DUST CONTAMINATION DURING CHEMICAL TREATMENT OF RF CAVITIES : SYMPTOMS AND CURES.

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ABSTRACT

The accelerating gradient in RF superconducting cavities is presently limited by surface defects, either emissive or dissipative. Dust contamination is known to be a significant source of such defects. Measurements of dust density were made at SACLAY on well characterized surfaces during various steps of a standard cavity preparation process. The significant dust contamination observed triggered us to develop a dedicated apparatus for the cavity chemical treatment, featuring etching with filtered, recirculating acids ; spray rinsing and drying under hot nitrogen flow. There is no dismounting between steps, and the process is fully automatized, with no human intervention. This device should yield a reduced dust contamination and improved reproducibility in cavity performances.

I) INTRODUCTION

The accelerating gradient in RF superconducting cavities is presently limited by surface defects, either emissive or dissipating. Dust contamination is known to be a significant source of such defects [ref. 1].

Usually only the end of the cavity preparation process occurs in dust-free conditions : last rinsings, drying and assembly in configuration to be RF tested. Most of the time, the first steps : degreasing, buffered chemical polishing (BCP) and several steps of rinsing-drying are carried out in normal atmosphere since it would otherwise require wide and expensive clean room installations, and it is hoped that very careful and long rinsings in filtered conditions is enough to get rid of the main contamination.

In order to check this statement we have tried to measure the dust contamination brought by the different steps of our standard cavity preparation at SACLAY, as it is hereafter described :

Step A : Ultrasonic degreasing of the cavity in hot alkaline detergent. Rinsing with DI-water. The cavity is then allowed to dry, usually one night long in the normal laboratory atmosphere.

Step B : BCP in 1HF-1HNO3-2H3PO4 in volume (usual time : 30-60 mn).

Step C : First rinsing : the cavity is dipped into DI-water, then the rinsing goes further with aspersion for 20 mn.

Step D : Second rinsing : the cavity is dipped into DI-water with an ultrasonic generator for 10 mn.

Step E : aspersion with DI-water for 10 mn.

Step F : Transport to and going through the clean-room sieve (2 mn, class 10000 atmosphere).

Step G : Third rinsing : the cavity is rinsed with forced circulation of filtered DI-water for 30 mn.

Step H : Drying under class 100 laminar flow.

In the case of contamination measurements on wafers, we shall add one step more :

Step I : Dust-free transportation from and back to the analyze set-up.

II) CHARACTERIZATION OF THE DUST CONTAMINATION

This kind of measurements is already done routinely in microelectronic industry in order to control the quality of silicon wafers. Optical reflection is used to count particles : a well focused scanning laser beam is sent to the surface, and the light scattered at 90% by an eventual dust particle is recorded by mean of a PM. This method enables us to measure the density of particles, and even gives information on their optical size. But this technique cannot be directly applied to niobium cavities which are not flat and optically polished.

We therefore mimicked all the steps of our cavity preparation process using polished silicon wafers supplied by the IBM-Society . Measurements of dust density were made at the IBM-Clean Laboratory at Corbeil-Essones (France). These wafers were dust-free, and were transported in dust free conditions to our clean-room. All test ended in clean-room, and the wafers were transported back in the same dust-free conditions to be measured. Results are in the form of the distribution of particles with optical size from 0.46 μm^2 to 5 μm^2 and up. For simplicity, only the global counts will be exposed hereafter. One should note that if the results could be measured on niobium instead on silicon, they should be more important because the niobium surface is rougher, and this point promotes adherence of dust particles.

As the different steps cannot be tested separately, only differential measurements can be made. For example, let us suppose that a treatment occurs in four steps :

1- chemical polishing
2- rinsing
3- clean-room rinsing
4- clean-room drying

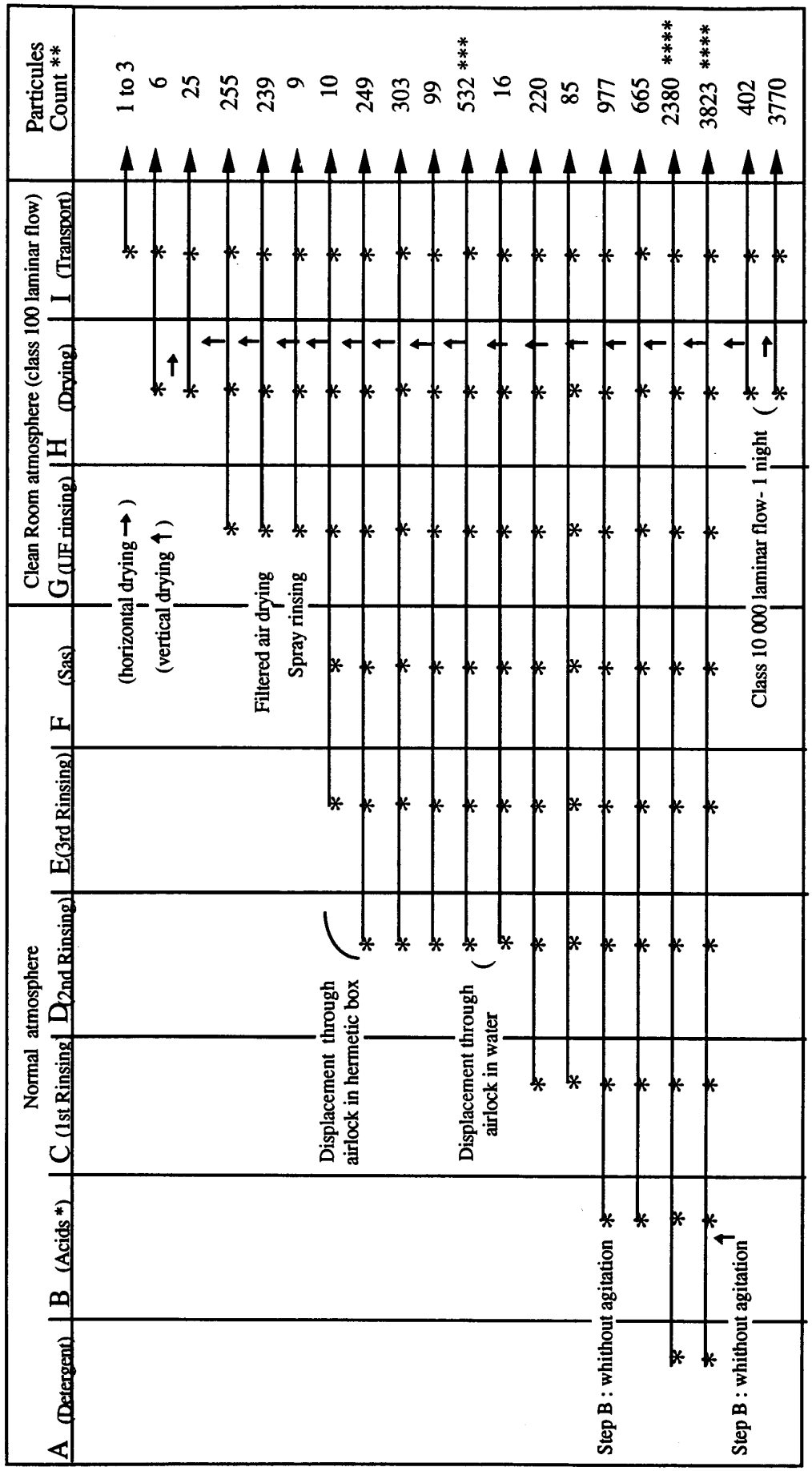
Four measurements can be made to test these four steps :

A = 1+2+3+4
B = 2+3+4
C = 3+4
D = 4 only

Step 1 of course could not be done separately just even for safety reasons but by comparing process A and B we can give an account about the contamination brought by this step 1, and so on... Moreover, as all these processes end in clean room, we are sure that no further contamination will appear between the treatment and the measurements (of course, the inocuity of this transportation step has been verified).

Table I) gathers the global particles counts measured on wafers (effective observed surface : 95 cm2). The letters A, B, C...to I refer to the standard procedure at SACLAY, as described in the introduction, * indicate which steps were applied to the measured wafer. As explained, we started each test at some step of the procedure, and then followed the rest of the procedure until its end in clean-room.

Table 1



* without HF, which attacks Si

** If not otherwise precised, most of the contamination is found near the bottom os the wafers, i. e. were the contact with fluids lasted the longer

*** 99% concentrated in one spot Ø 1cmn (i.e. accidental)

**** top of wafers, which have not been treated with acid are much more contaminated

III) CONCLUSION OF DUST MEASUREMENTS

First observations that arise from these results are the following :

1- Work in cleanroom does not bring contamination.

2- Rinsings conducted either in normal atmosphere or in cleanroom also do not bring noticeable contamination, but the long, low pressure rinsing used at Saclay is inefficient for particles removal.

3- Other processes conducted in normal atmosphere like degreasing, BCP, leave large amounts of particles on the surface, which cannot be eliminated by further dust-free treatments.

4- Some particles seem to be soluble in acids, but a significant amount of it is not removed by a BCP.

5- Exposed humid surfaces are very sensitive to contamination : exposure of wet surfaces should be reduced as possible, even under laminar flow.

IV) CURES

Following IBM-specialists recommendations we should establish a new cleaning procedure which takes into account the following rules :

- All fluids should be filtered.

- Polishing should produce surfaces as smooth as possible (choice of the polishing mixture).

- Displacements and exposure of wet cavities should be avoided.

- Rinsing should be turbulent and if possible, carried out with hot water for a rapid elimination of the acid layer (diffuse layer).

- Drying should be as fast as possible because of the high sticking coefficient of humid surfaces.

- Human intervention should be as reduced as possible (source of irreproducibility).

One solution which does not need any further clean-room installation is to work all the needed operation in a closed circuit : indeed it is much easier to get very low dust classes in a small closed space where all fluids will be filtered, than in a large room with human activity.

We developed in collaboration with a small industrial society[1], specialized in building facilities for wet treatment for microelectronic industry a dedicated apparatus, trying to take into account all the recommendations cited above and the constraints due to the particular geometry of the cavities. Figure 1 shows a scheme of its running. All of it is automatized, and checked by an Automat, which also surveys the security parameters (times, temperatures, and pressure controls for instance).

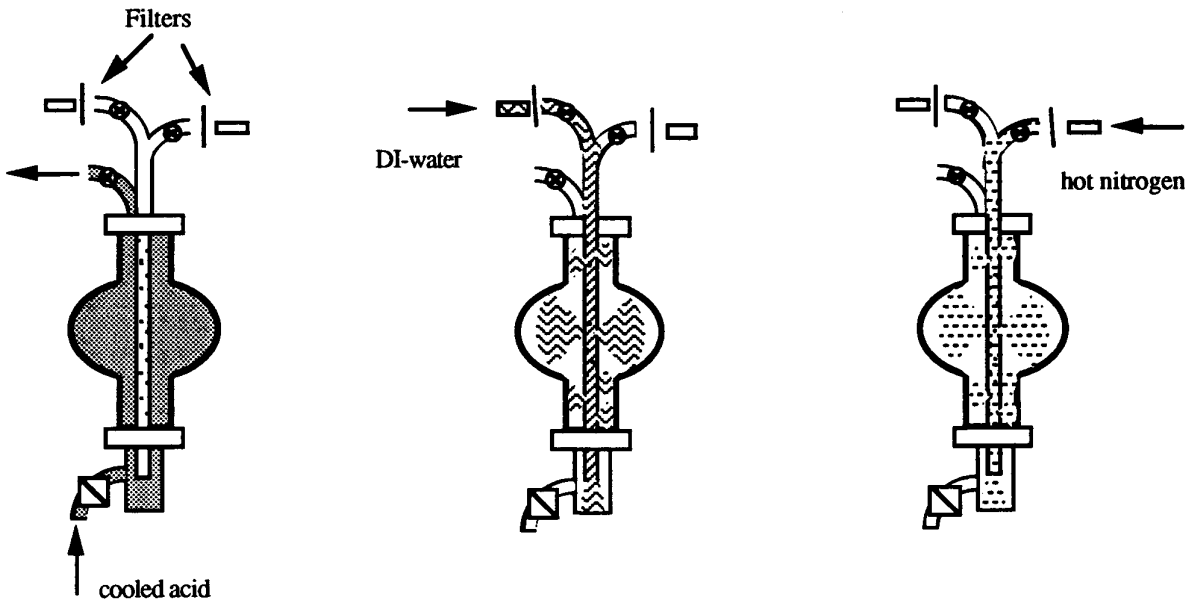


Figure 1 : running scheme

- Cavity is hermetically connected to the system.

- BCP is processed with filtered (0.2μm) cooled acids. There is a continuous recirculation, filtration and thermal control.

- Rinsing is made via a central spray with filtered DI water. At the beginning the DI water is heated (60°C) in order to get a better effectiveness in the dissolution of the acid

[1] SAPI-Equipement. 109, Av. des Chutes-Lavie- 13013 Marseilles-FRANCE

layer ; then cold water is used (because of its better resistivity).

- Cavity is dried with hot filtered nitrogen, which allows to reduce a lot the drying time.

- It is then hermetically closed and dismounted from the system without exposure to the external atmosphere.

- Opening occurs in clean room, under a class 100 laminar flow .

The clean, dried cavity is now ready to be installed directly on its RF test facility, having only spent a very short time exposed to the clean room atmosphere. Human intervention is minimized, and does absolutely not interfere during the critical parts of the treatment : BCP and rinsings.

With this apparatus, we hope not only to reduce a lot field emission in cavities, but also increase the reproducibility of the chemical treatments.

REFERENCES

1) See e. g. 4th Workshop on RF superconductivity, Aug 14-18 1989, KEK, TSUKUBA, Japan, Y. Kojima Ed.