

## Avoiding dust contamination during chemical treatment of RF cavities ?

C. Z. ANTOINE, B. BONIN, J.M. CAVEDON, C. CHIANELLI,  
J.M. HISLEUR, B. MAHUT, J.P. POUPEAU

C. E. SACLAY, FRANCE

### 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 dismantling between steps, and the process is fully automatized, with no human intervention. The first cavity treated with this facility already gave excellent results with respect to Q0 value and maximum accelerating gradient.

### 1. INTRODUCTION

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 [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.

### 2. 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 photomultiplier. This method allows the measurement of 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-Company. Measurements of dust density were made at the IBM-Clean Laboratory at Corbeil-Essone (France). These wafers were dust-free, and were transported in dust free

conditions to our clean-room. All tests ended in clean-room, and the wafers were transported back in the same dust-free conditions to be measured. Detailed results are exposed in reference 2. For simplicity, only four significant results will be exposed hereafter. (One should note that if the results could be measured on niobium instead of 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- ultrasonic degreasing in alkaline detergent
- 2- chemical "polishing" in  $1[\text{HNO}_3]-2[\text{H}_3\text{PO}_4]$  in volume<sup>1</sup>
- 3- rinsing + clean-room rinsing
- 4- clean-room drying

Four measurements can be made to test these four steps :

procedure	particles counts ( $\varnothing > 0.2 \text{ mm}$ )
A = 1+2+3+4	3800
B = 2+3+4	900
C = 3+4	100
D = 4 only	<20

(effective observed surface : ~95 cm<sup>2</sup>)

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.

### 3. CONCLUSION OF DUST MEASUREMENTS

First observations that arise from the results are the following [Reference 2]:

- 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

<sup>1</sup> HF should be avoided to preserve the optical polish of the wafers.

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 proportion 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.

4. CURES

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

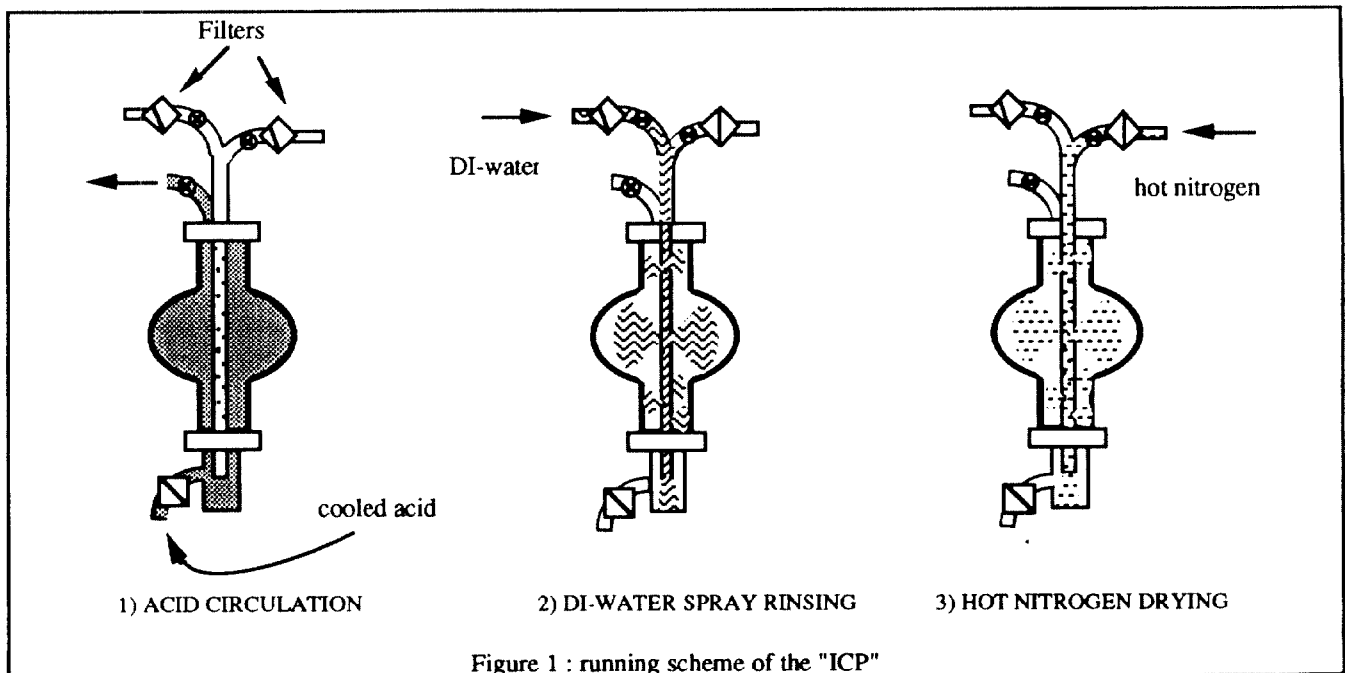
- All fluids should be filtered.
- Polishing should produce surfaces as smooth as possible (choice of the polishing mixture).
- Displacements and exposure of wet cavities should be avoided.
- Rinsing should be turbulent and if possible, carried out with hot water for a rapid elimination of the acid layer (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 company, specialized in building facilities for wet treatment for microelectronic industry a dedicated apparatus, that we shall mention further as "Integrated Chemical Polishing" (ICP), 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, with a full control of the security parameters (timings, temperatures, and pressures).

- Cavity is hermetically connected to the system.
- BCP is processed with filtered (0.2 μm) cooled acids. There is a continuous recirculation, filtration and thermal control. The temperature of the acid mixture can be stabilized (better than ± 2° C) within the range 0° C to 30° C.
- 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 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 (t < 1h for one-cell cavity).
- The cavity is then hermetically closed and dismantled 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 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.

## 5. SOME RESULTS

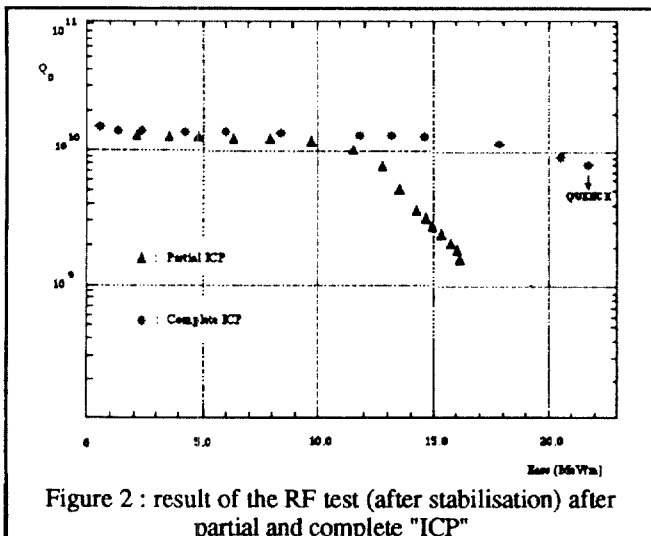
Two procedures were tested :

(a) a cavity was first treated by acids (1[HF]-1[HNO<sub>3</sub>]-2[H<sub>3</sub>PO<sub>4</sub>]) followed by a short rinsing in ICP, it was then dismantled and submitted to the end of our former procedure, i. e. forced UF DI water rinsing in clean room and subsequent drying under laminar flow. Figure 2 displays the results of the RF test that followed this treatment (triangles).

(b) The same cavity was then totally treated in ICP (acids, rinsing and drying) and the results of the RF test that followed this second treatment are showed in figure 2 (stars).

One can see that these tests show rather good cavity performances : in both cases,  $Q_0$  is above  $10^{10}$  ; for (a) , electrons appeared around 15 MeV/m, but after a quench, a degradation appeared and the emission threshold stabilized near 12 MeV/m. On the contrary, in test (b) , electrons appeared near 13 MeV/m, but disappeared after very short RF processing. The performance of the cavity was then only limited by a quench around 22 MeV/m.

Of course these very preliminary results, although promising, are not sufficient to fully qualify ICP, and we are now undertaking systematic tests in order to compare the statistical appearance of electrons



## 6. REFERENCES

- (1) See e. g. Proceedings of the 4th Workshop on RF superconductivity, Aug 14-18 1989, KEK, TSUKUBA, Japan, Y. KOJIMA Ed.
- (2) "Dust contamination during chemical treatment of RF

cavities : Symptoms and cures."C. Z. ANTOINE, B. BONIN, J.M. CAVEDON, C. CHIANELLI, J.M. HISLEUR, B. MAHUT, J.P. POUPEAU. Proceedings of the 5th Workshop on RF superconductivity, Aug 19-23 1991 at DESY, HAMBOURG, Germany, to be published

(3) SAPI-Equipement. 109, Av. des Chutes-Lavie- 13013 Marseilles-FRANCE