

# Low, Medium, High Field Q-Slopes Change with Surface Treatments

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**Abstract.** Hydrofluoridric chemistry, hot chemistries, plasma discharge, different kinds of baking, etc... several surface treatments modify the  $Q_0(E_{acc})$  profile of bulk niobium cavities. The analysis of these modifications allows a critical comparison with theoretical predictions. Furthermore, given the high field Q-slope improvement by baking and its insensitivity to an additional hydrofluoridric chemistry, the diffusion process cannot be only considered as a consequence of the thermal process. In this perspective some additional experiments are undertaken at Saclay where an “integrated baking system” will soon be built.

## INTRODUCTION

To improve the cavity performances and reach higher intrinsic quality factor  $Q_0$ , higher accelerating field  $E_{acc}$  or higher quench field, it is necessary to understand the  $Q_0$  vs.  $E_{acc}$  curve (Fig.1). For that purpose, several paths have to be explored:

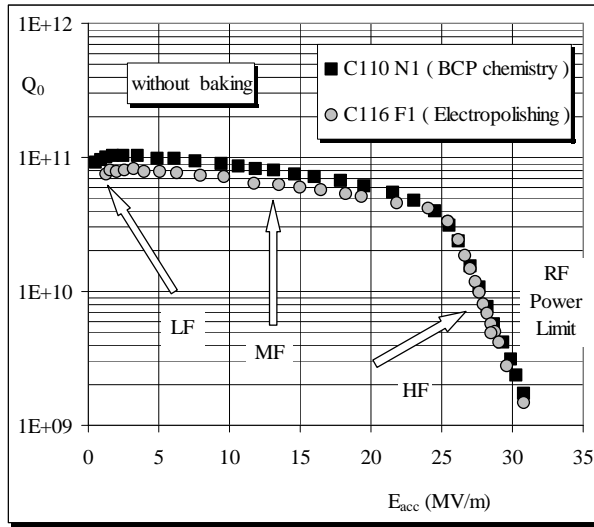
- Macroscopic experiments developed on niobium cavity which give a direct access to the  $Q_0(E_{acc})$  profile. Advances on this way can be made independently of theory and surface analysis through more or less empirical observations and arguments.
- Microscopic surface analyses on niobium samples are useful for punctual verification, to sustain a theoretical argument or to suggest new macroscopic experiments. Unfortunately by this way, it is difficult to have a direct link with macroscopic observations (Q-slope, for example). For that reason, surface analyses remain strongly connected to theories and to experiments on cavity.
- Theory that can ensure physical explanation if it is in agreement with observations from macroscopic experiments.

The subject of this paper is to describe macroscopic experiments carried out at Saclay and based on surface treatments that modify the  $Q_0(E_{acc})$  profile. These results will be confronted to theories developed to explain the different parts of  $Q_0(E_{acc})$  characteristics.

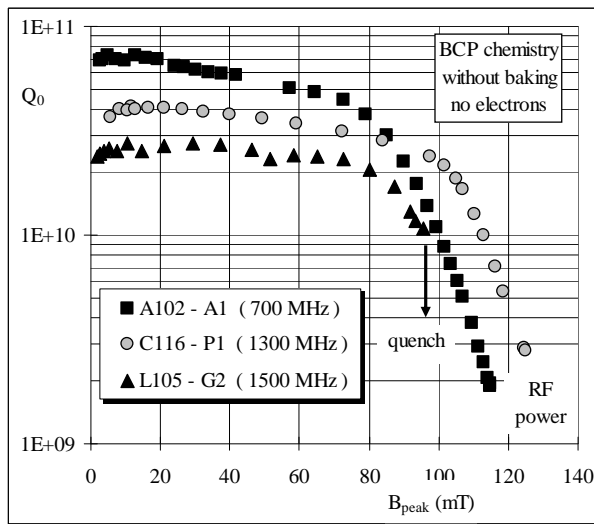
## Q-SLOPES

On Fig.1, the curve  $Q_0(E_{acc})$  shows clearly three parts with different slopes at low, medium and high fields (LF, MF and HF Q-Slopes). These three parts are visible on bulk niobium cavities regardless of their resonant frequency (Fig. 2). Theories that can be proposed to explain the Q-slopes origin are:

- at low field, the  $NbO_x$  clusters theory [1]
- at medium field, several theories based on thermal dissipation [2]
- at high field, among a lot of theories [3], the Interface Tunnel Exchange [1] or the Magnetic Field Enhancement [4] can be mentioned, although that last one cannot explain the high field Q-slope similarity observed on chemically (BCP) and electro-polished (EP) cavities (see Fig.1).



**FIGURE 1.**  $Q_0$  vs.  $E_{acc}$  curve showing the different Q-slopes for cavities (1.3 GHz) treated with standard chemistry (BCP) and electropolishing (EP). We can note Q-slope similarity (especially at high field) for cavities with BCP or EP chemistry.



**FIGURE 2.** Q-slopes of monocell niobium cavities, with BCP chemistry, designed at different resonant frequencies (700, 1300 and 1500 MHz).

## BAKING AT LOW TEMPERATURE

### Description

Baking is the first known treatment able to modify the three Q-slopes. It was first discovered on a BCP cavity after it underwent a soft heat treatment (110 °C for 48 hours), the inner volume being pumped in ultra high vacuum (UHV) [5]. The consequences for its performances were (Fig.3):

- the  $R_{BCS}$  decrease about 50% at 4.2K,
- the residual resistance  $R_{res}$  increase,
- the Q-slope enhancement at low field,
- the slight flattening of the medium field Q-slope,

- the strong Q-slope improvement at high field.

These observations are the mark of the “baking effect” and it can be demonstrated that it also takes place in baking under air at the atmospheric pressure [6] (Fig.4).

Furthermore, without such treatment, cavities (even electropolished) do not reach 40 MV/m (Fig.5).

The high field Q-slope improvement after baking is quasi-definitive: almost four years after baking, storage in open-air on shelves, the cavity shows an unchanged profile (Fig.10).

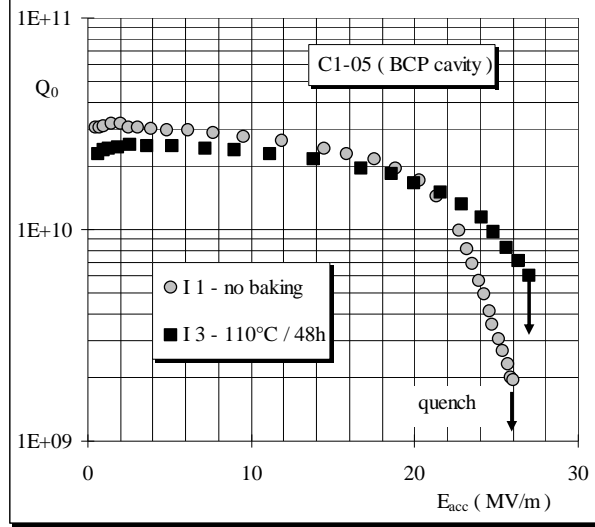


FIGURE 3. Q-slopes change after UHV baking on a BCP cavity.

### Baking Consequence for the Medium Field Q- Slope

The baking effect on the medium field Q-slope reported in [7] from RF tests on CEBAF and TESLA multi-cell cavities show an increase by a factor of 3 or 4 and a change from a quadratic to a linear dependence.

According to the experimental results obtained at Saclay the analysis is different. Saclay data are fitted in using the formula from the Halbritter’s model [1]:

$$(R_s - R_0)/R_0 = 1 + \gamma B_p^2 / B_C^2 \quad (1)$$

where  $R_0 = R_s(B_p = 13 \text{ mT})$ ,  $B_C = 200 \text{ mT}$

and  $B_p/E_{acc} = 4.221 \text{ mT}/(\text{MV}/\text{m})$  or  $4.156 \text{ mT}/(\text{MV}/\text{m})$  for D122 cavity.

Data are analysed between 3 and 18 MV/m to prevent the influence of the low and high field Q-slopes. Results are compiled from twelve RF tests made on eight one-cell cavities, two of them being electropolished. A quadratic variation is found before and after baking, with a slight flattening ( $\Delta\gamma/\gamma \sim 5\%$ ) after baking (Fig.6-7).

The very different results between these experiments and those reported in [7] are probably to be found in the difference of the conditions in which the baking was performed in each laboratory.

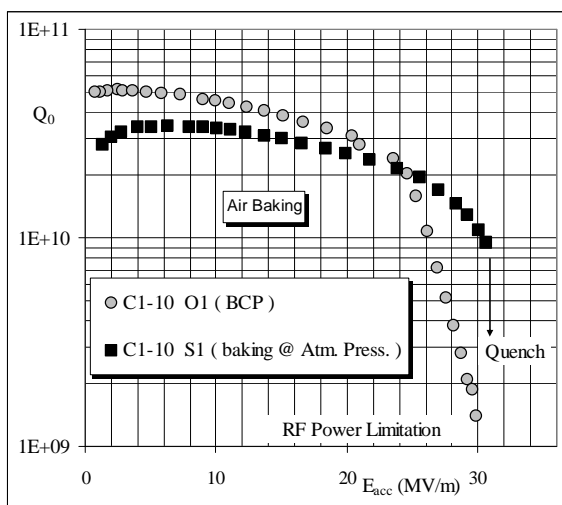


FIGURE 4. Air baking at the atmospheric pressure of a BCP cavity.

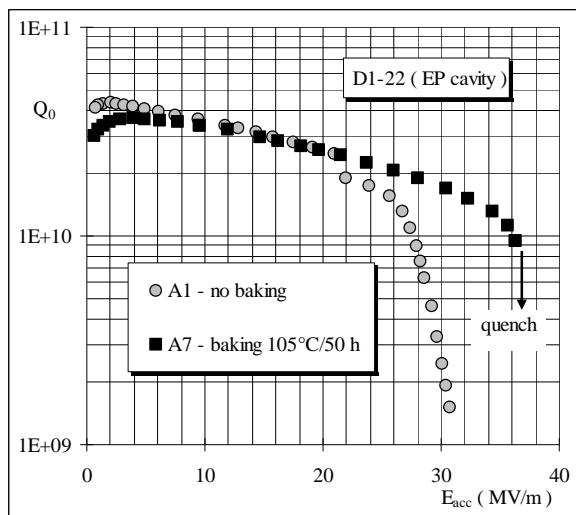
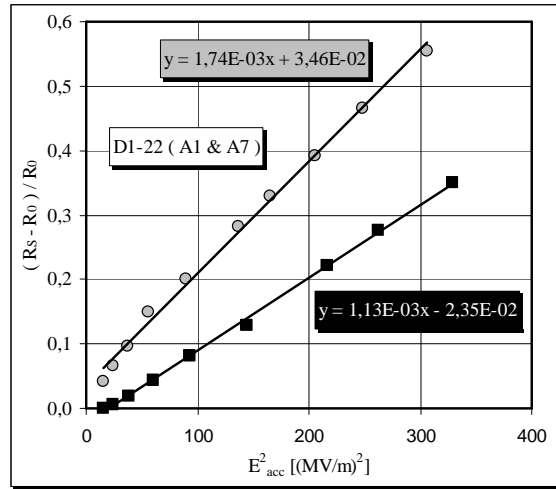
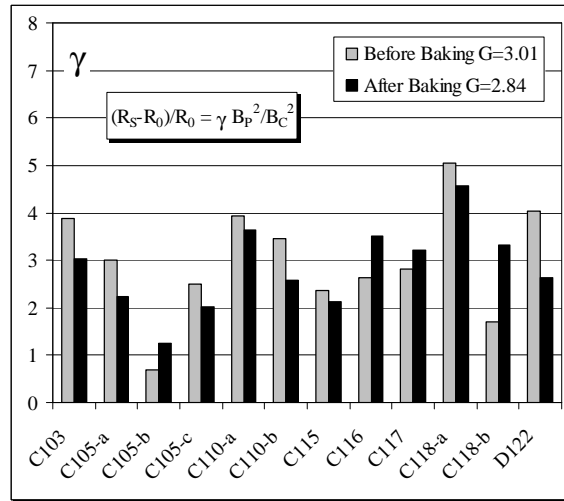


FIGURE 5. UHV baking on an electropolished cavity.



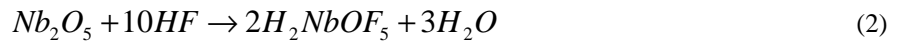
**FIGURE 6.** Quadratic behaviour of the medium field Q-slope before and after baking ( $B_p/E_{acc} = 4.156 \text{ mT/MV/m}$ ).



**FIGURE 7.** Gamma factor of the medium field Q-slope before and after UHV baking on BCP cavities, except for C103 and D122 (electropolishing) and for C110-b (air baking).

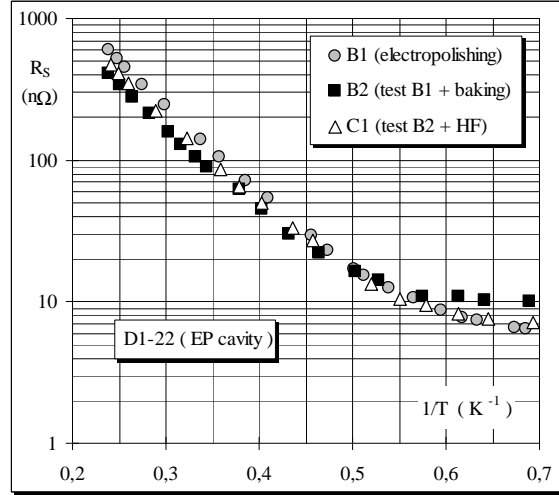
## SURFACE TREATMENT BY HYDROFLUORIDRIC ACID

Surface treatment by hydrofluoridric acid is commonly and successfully used at Saclay to suppress field emission from the cavity surface (Fig. 9 C1, Fig. 10 I1), especially when emitters can not be removed by an additional high pressure rinsing (HPR). This result can be explained by the Nb surface renovation. During hydrofluoridric chemistry (HF), Nb pentoxide surface layer is dissolved according to the chemical reaction (Equation 2):



$\text{Nb}_2\text{O}_5$ -Nb interface is also affected by this chemistry on a sizeable depth. Although this statement should be verified by surface analysis on samples, a change in the surface resistance  $R_S$  (Equation 3) is clearly shown on baked cavities after hydrofluoridric chemistry (Fig.8 and Table 1). The consequence of such a treatment is a trend to restore the  $R_{res}$  value before baking and in a smaller extent the  $R_{BCS}$  one. The energy gap  $\Delta$  seems unchanged before and after HF chemistry. Of course, the  $R_S$  change after HF chemistry has consequences on the Q-slope profile.

$$R_S = R_{BCS} + R_{res} = \frac{A\omega^2}{T} e^{-\Delta/kT} + R_{res} \quad (3)$$



**FIGURE 8.** Surface resistance change after baking and HF chemistry.

**TABLE 1.** Change of surface resistances after baking treatment, followed by HF chemistry.

Cavity	Test		$R_S$ @ 1.5 K nΩ	$A\omega^2$ $10^5$ nΩ.K	$R_{res}$ nΩ
	N°	Chemistry			
C103	E1	EP	6,0	2,3	4,6
	E4	baking	9,1	1,3	8,3
	G1	HF	6,3	1,5	5,5
C105	J1	BCP	1,8	2,4	1,2
	K1	baking	3,2	1,5	2,7
	L1	HF	2,2	2,0	1,7
C116	P1	BCP	7,1	2,1	6,2
	P2	baking	8,3	1,3	7,4
	Q1	HF	6,1	1,3	5,5
D122	B1	EP	6,5	2,1	6,0
	B2	baking	10,0	1,4	9,7
	C1	HF	7,1	1,7	6,7

### Low Field Q-Slope Analysis

Due to  $R_S$  modifications, we can see on Figures 9 and 10 that the low field Q-slope, enhanced by baking (Fig.9 B2, Fig. 10 E5-F1), is removed by HF treatment (Fig. 9 C1, Fig. 10 I1) and that the slope before baking (Fig. 9 B1) is roughly recovered. Moreover, an additional baking restores the Q-slope enhancement (Fig. 9 C2). Surface treatments by baking and HF chemistry have an opposite effect on Q-slope at low field.

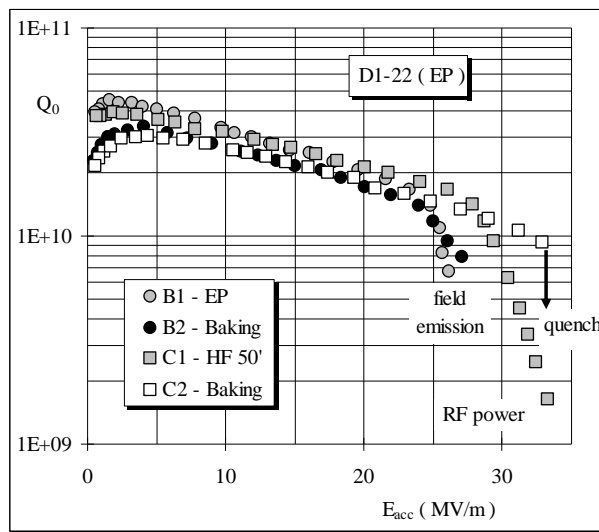
Consequently, the origin of the Q-slope at low field ( $H < 10$  mT) is probably localised at the interface niobium-oxide or very close to the niobium surface, in the small layer removed by HF chemistry. These observations anyway, are not incompatible with the  $NbO_x$  clusters theory [1] that gives an explanation for the low field Q-slope.

## High Field Q-Slope Analysis

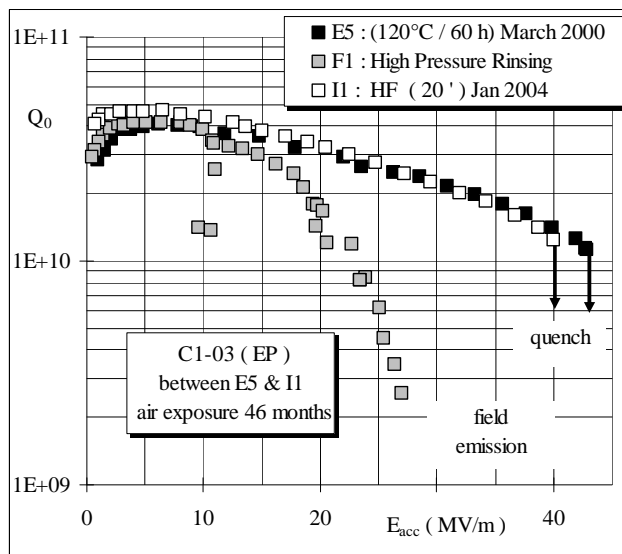
Modifications by HF chemistry are only visible at low field. Medium and high field Q-slopes are unaffected. Especially, the high field Q-slope before baking is not restored and the baking benefit is maintained (Fig. 10-I1). This means that the changes induced by baking to remove the high field Q-slope, are deep enough to be unaffected by HF chemistry (renovation of Nb<sub>2</sub>O<sub>5</sub> layer and Nb-oxide interface). This observation refutes particularly the “Interface Tunnel Exchange” (ITE) theory [1] which argues that the decrease of Nb<sub>2</sub>O<sub>5</sub> thickness during baking explains the Q-slope removal.

After HF chemistry the metal-oxide interface is rebuilt with interstitial oxygen forming a bad superconducting layer (Nb<sub>4</sub>O, Nb<sub>6</sub>O), true origin of the high field Q-slope according to the theory [8]. In this theory, Q-slope removal after baking is explained by the oxygen dilution in the bulk. However, HF treatment applied on baked cavity does not restore the initial Q-slope (before baking), refuting consequently this theory.

As we have already noted, the baking treatment was made four years before: the baking effect and the cavity change can be considered as definitive.



**FIGURE 9.** HF chemistry suppresses field emission and modifies Q-slope at low field.



**FIGURE 10.** HF treatment and its consequences on a baked EP cavity (field emission and low field Q-slope).

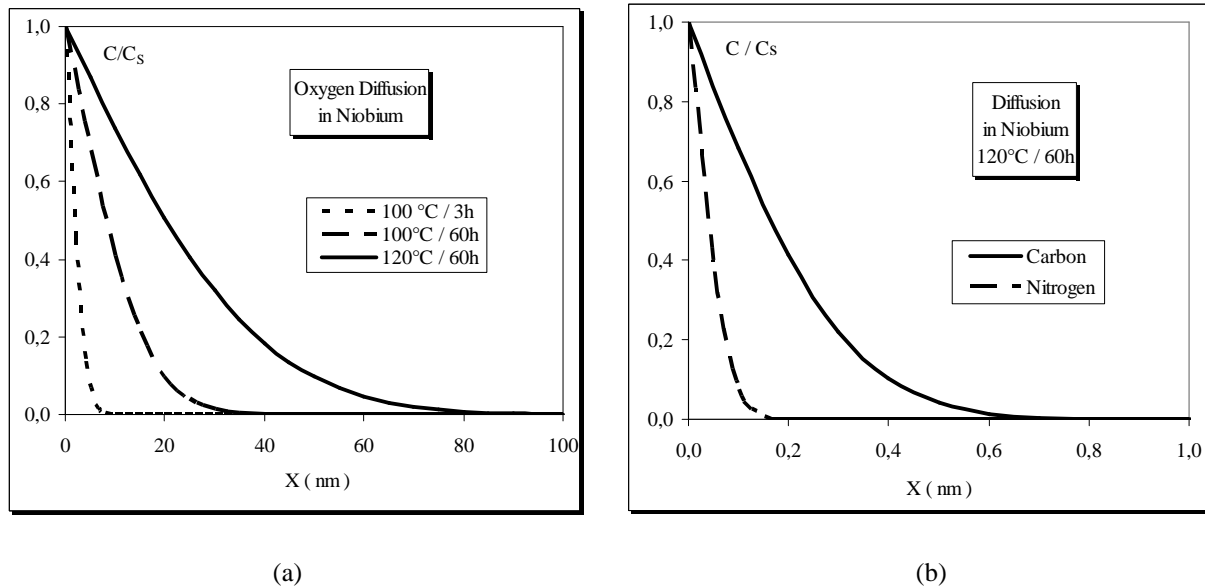
## DIFFUSION PROCESS

Before the baking effect discovery, the oxygen diffusion, concomitant to any heat treatment, has been widely considered. This is because a lot of oxygen is found at the niobium surface as niobium oxide, sub-oxides or as interstitials and because it easily diffuses in the bulk material, even at low temperature [9].

To evaluate this process, we can use a simple model based on the second Fick's law giving an analytic solution (Equation 4) for the concentration of a diffusing element in a semi-infinite medium:

$$C(x,t)/C_s = \operatorname{erfc} \frac{x}{2\sqrt{D(T)t}} \quad (4)$$

where  $D(T)$  is the diffusion coefficient of the considered element [10], with initial condition in the bulk  $C(x,0) \equiv 0$  and the boundary condition on the surface  $C(0,t) = C_s$ . According to this model, we can see in Fig.11 for the usual baking parameters ( $T=120^\circ\text{C} / t = 60$  hours) that oxygen penetration by diffusion is similar to the RF penetration depth at 2 K ( $\sim 50$  nm) and that other elements likely to be present at the surface (nitrogen or carbon) diffuse 100 times less than oxygen.



**FIGURE 11.** a) Oxygen diffusion profiles in Niobium for different temperatures and baking times.  
b) Nitrogen and Carbon diffusion profiles at  $120^\circ\text{C}$  for 60 hours.

Some observations of the baking effect can be directly attributed to the oxygen diffusion like the decrease of the  $R_{\text{BCS}}$  resistance through its dependence on the electron mean free path [11]. The high field Q-slope can also be linked to the diffusion process because a strong correlation has been observed at Saclay between the  $E_{\text{acc}}$  value of the high field Q-slope onset and the baking time (Fig.12).

The right parameters to improve the performances of the cavity by baking seem to be  $100^\circ\text{C}$  for 60 hours (Fig.13) with a "moderate" oxygen concentration in the RF superconducting layer to dope niobium. At higher temperatures (up to  $250^\circ\text{C}$ ), decreases of  $Q_0$ , quench field and critical temperature  $T_C$  have been observed [12-13]: this corresponds to niobium pollution with probably too much oxygen under the surface. Above  $250^\circ\text{C}$ ,  $\text{Nb}_2\text{O}_5$  is totally dissolved and the trend is reversed with the increase of these parameters, corresponding to oxygen depletion on the cavity surface.

All these observations lead to think that the oxygen diffusion process plays a crucial role in the cavity performances at high field.



To continue with the diffusion hypothesis, we studied the effect of temperature and time changes. Except for the oxygen amount present at the Nb surface, “fast baking” (145°C / 3 hours) should be roughly equivalent in terms of diffusion with the usual baking (110°C / 60 hours) and lead to a similar behaviour for the high field Q-slope.

The other advantage of the “fast baking” combined with “air baking” is to be easier to implement than the usual UHV baking, not to mention the avoided risk of helium leaks. Such baking could be directly realized in clean room under laminar flow. This procedure will be used instead of the usual air-drying, between high pressure rinsing and cavity assembly, using IR heaters and remote thermal sensor (Fig.14) to regulate the temperature for 3 hours.

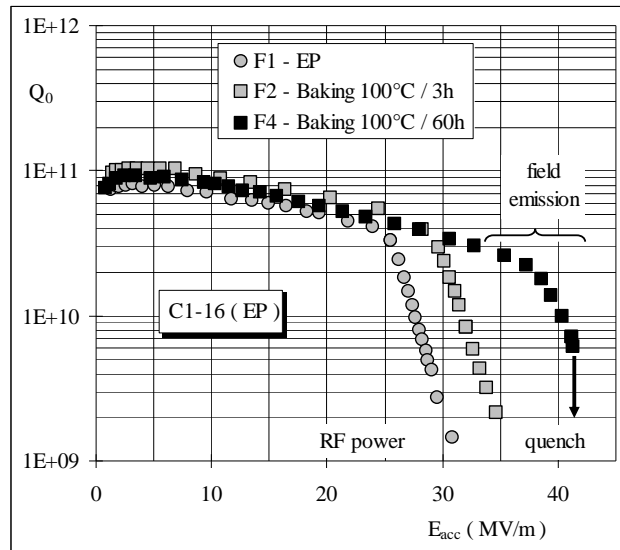


FIGURE 12. The  $E_{acc}$  value of the Q-slope onset is increasing with the baking time.

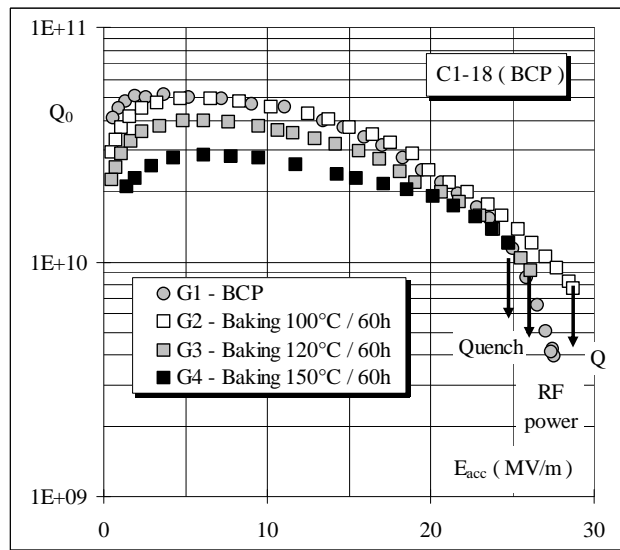


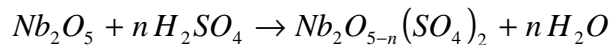
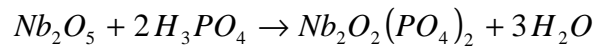
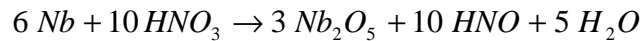
FIGURE 13. Deterioration of  $Q_0$  and quench position with the increase of UHV baking temperature.



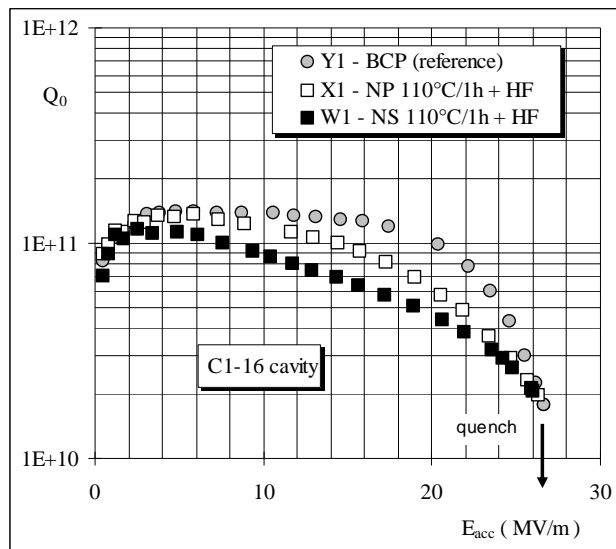
**FIGURE 14.** 1.3 GHz cavity, IR heaters and thermal sensor for “fast / air baking” in clean room.

### SURFACE TREATMENTS BY HOT CHEMISTRY

These surface treatments are characterized by an acid attack of the niobium pentoxide by baths of nitric-phosphoric or nitric-sulphuric acids at 110°C during one hour.



As we can see on Fig. 15, such treatments cause a general deterioration of Q-slopes at medium field with a less marked transition between medium and high field Q-slopes. These results are only incidentally reported here, because of the general and disappointing deterioration of the  $Q_0(E_{acc})$  curve. Causes of this deterioration are not well understood at this moment but they are certainly due to a surface pollution correlated with a thermal diffusion of some elements.



**FIGURE 15.** Treatments by hot chemistries lead to the medium field Q-slope worsening.

## CONCLUSION

Modifications of Q-slopes, induced by some specific treatments of the cavity surface, can help us to understand the  $Q_0(E_{acc})$  curve:

- Hydrofluoridric chemistry improves the low field Q-slope, suggesting its origin in the Nb<sub>2</sub>O<sub>5</sub> -Nb interface. This is compatible with the cluster theory,
- HF treatment is very useful to suppress the field emission. Applied on a baked cavity, this treatment does not affect the high field section of the curve. High field Q-slope and its cure by baking find their origin in the niobium metal and not at the interface. This result refutes the “Interface Tunnel Exchange” and the “Bad Superconducting Layer” models as theoretical explanations,
- Baking modifies low, medium and high field parts of the curve with an increase of the slope at low field, its suppression at high field and a trend toward flattening at medium field.
- Strong correlation between high field Q-slope and oxygen diffusion leads not only to consider this diffusion as a consequence of the cavity baking but as the probable real cause of the high field Q-slope improvement.

Future experiments on “fast baking” should learn more about this hypothesis and open the possibility to make the baking in the clean room.

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