

FIRST RESULTS ON FAST BAKING

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OXYGEN DIFFUSION

Abstract

High gradient performances of bulk niobium cavities go through a low-temperature baking during one or two days, the temperature parameter is adjusted in a narrow tuning range around 110 or 120 °C. With such treatment, the intrinsic quality factor Q_0 is improved at high fields.

Assuming the oxygen diffusion is involved in this phenomenon, we have developed the “Fast-Baking” (145 °C / 3 hours) as an alternative method. Similar results have been achieved with this method compared to standard baking. Consequently, for the first time, a link between oxygen diffusion and high field Q-slope has been proved.

Furthermore this method open the way to a simpler and better baking procedure for the large-scale cavity production due to:

- § the baking time reduction and
- § the possibility to combine baking and drying during cavity preparation.

INTRODUCTION

At the origin, the «baking-effect» was observed on an electron-beam welded cavity, built from bulk multi grain niobium and chemically polished by a standard BCP chemistry (1:1:2 in volume mixture of HF:HNO₃:H₃PO₄ acids) [1]. The inner part of the cavity was pumped out under ultra high vacuum conditions (UHV).

Since then and complementarily, similar observations have been made on several kinds of cavities:

- § hydroformed without welds,
- § manufactured from clad niobium-copper or single crystal niobium sheets [2-3],
- § electropolished or chemically treated by BCP 1:1:1,
- § with or without prior thermal treatment at 800 or 1300 °C.

Baking appears thus like a general recipe to increase high field performances of any niobium cavity. Moreover this treatment is permanent while, four years after, a baked cavity keeps its improved performances, even after air opening and surface chemistry by hydrofluoric acid [4].

Q_0 is not the only one physical quantity modified after cavity baking: BCS resistance decreases by a factor two, presuming change in superconducting characteristics of niobium.

A consequence of baking is the interstitial oxygen diffusion from the oxidized niobium surface to the bulk material. Following experiments performed by Palmer and Kneisel [5-6], R_{BCS} change can be explained by the electron mean free path decrease due to oxygen diffusion.

Concerning Q-slope improvement, consensus is more difficult to reach while a lot of theories attempt to explain the experiments with mixed success as reported in [7]. However, experimental results [4] lead to consider again oxygen diffusion as the probable real cause of the Q-slope improvement.

OXYGEN PENETRATION

Such consideration can be easily verified by changing parameters of baking, time and temperature, keeping unchanged oxygen penetration profile in niobium.

For a conservative system, the second Fick's law describes the spatiotemporal evolution of the diffusing atom concentration

$$\frac{\partial C}{\partial t} = D(T) \frac{\partial^2 C}{\partial x^2}$$

where D is the diffusion coefficient of the considered atom in material.

Valid in very restrictive conditions, this equation can not be solved analytically, except for few simple cases with particular initial and limit conditions [8]. Two cases can closely describe our situation (oxygen diffusion from the surface of a semi infinite niobium material):

§ the diffusion from a thin layer at the surface, with Q as the total quantity of diffusing atoms

$$C(x, t) = \frac{Q}{\sqrt{\pi D t}} \exp\left(-\frac{x^2}{4 D t}\right)$$

§ the diffusion with a constant concentration C_s at the surface

$$C(x, t) = C_s \operatorname{erfc} \frac{x}{2\sqrt{Dt}}$$

These formulas allow to simulate oxygen concentration profiles in both cases (blue curves in Fig.1) with $D = 5.6 \cdot 10^{-3} \exp(-109600 / RT)$ cm²/s [9], $T = 110$ °C and $t = 60$ h.

For a reduced baking time, 3 hours for example, an equivalent oxygen distribution is observed with 145 °C (red curves in Fig.1).

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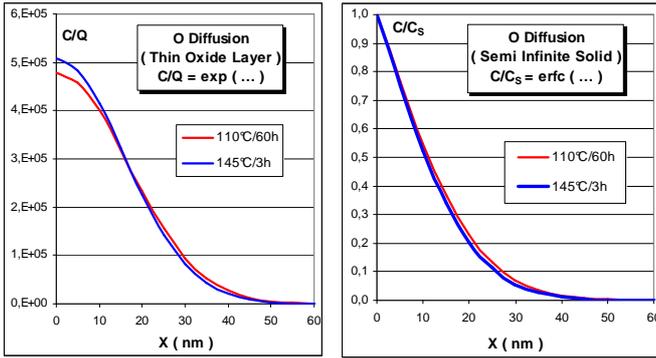


Figure 1: Simulations of the penetration profile of O in Nb using two different analytic solutions.

FAST UHV-BAKING

These new parameters (145 °C / 3 h) define a modified baking process in terms of time and temperature. The other experimental conditions are similar to the standard baking:

§ as mentioned above, the oxygen penetration in niobium is the same,

§ the inner part of the cavity is still pumped out with a turbo-molecular pump, justifying the term “fast UHV-baking”.

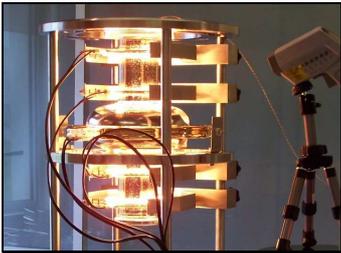


Figure 2: Experimental set-up for “fast baking”.

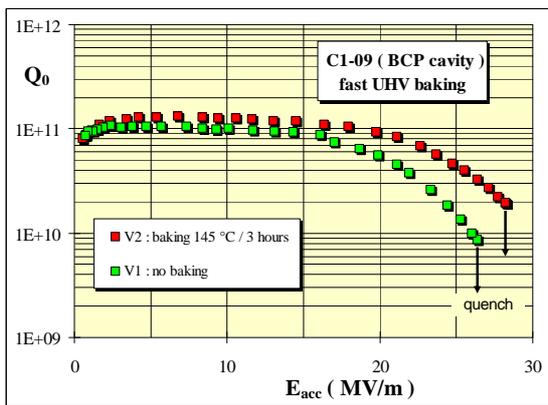


Figure 3: High field Q-slope improvement induced by “fast UHV-baking”.

Nevertheless, in order to decrease the temperature rise time, the implementation of the “fast baking” process needs a different approach in comparison with the «in-situ» baking. The cavity is surrounded with five

infrared emitters (short wavelength: 1.3 μm, 2 kW) manufactured by Heraeus Noblelight. The temperature regulation is monitored by an IR thermal sensor Rayteck® (Fig. 2).

RF measurements results carried out on cavity C1-09, before and after “fast UHV-baking”, are shown in Fig.3. They are very similar to the standard baking results with:

§ R_{BCS} decrease and

§ particularly the same slope improvement, proving by this way the implication of the oxygen diffusion in the Q_0 change at high fields.

FAST AIR-BAKING

Cavity baking at room atmosphere has been already demonstrated with success [10]. This possibility leading to a possible simplification in the baking process, we attempted to treat by “fast baking” the same cavity open-ended in the clean room atmosphere. Unfortunately the curve $Q_0(E_{acc})$ is highly deteriorated in Q_0 and quench field (Fig.4).

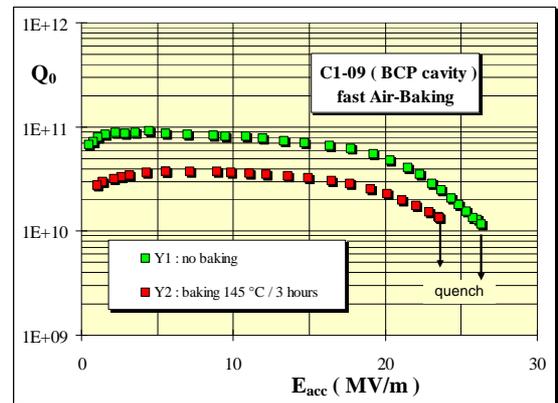


Figure 4: Degradation of Q_0 vs. accelerator field curve after fast baking at the room atmosphere.

When temperature is too high, standard “UHV-baking” (150 °C / 60 h) shows similar results [4]. The reason of this degradation is the excess of oxygen and the modification of niobium thermal conductivity.

In “fast air-baking” the reason is probably the same: the oxygen excess coming from water adsorbed on the niobium surface as consequence of the clean room hygrometry (60 %). This analysis can actually explain (see Fig.5):

§ difference between “fast baking” results (air and UHV) by the surface contribution in additional oxygen,

§ difference between air-baking results (fast and standard) by easier oxygen diffusion at 145°C through the niobium oxide.

In consequence to carry out with success “fast air-baking” 3 hours is a too long period. It is necessary to adjust more precisely the oxygen diffusion: SIMS analysis on samples could help us to find the right time.

		O % from NbO _x , Nb ₂ O ₅ →			
		110 °C 3 h	110 °C / 60 h standard	145 °C / 3 h fast	145 °C 60 h
O % from H ₂ O on surface ↓	UHV	=	+	+	-
	Air		+	-	

Figure 5: Overview on the different baking experiments with improvement (+), degradation (-) or without modification (=) of the Q₀ (E_{acc}) curve.

Moreover, as “fast air-baking” is well adapted to the cavity mass production, this method could be used for its practical aspects [12]. In fact, such process allows to save time and to simplify the standard cavity preparation: baking in clean room, directly after the high pressure rinsing, could avoid the air-drying step.

CONCLUSIONS

From these experimental results we can affirm that the link existing between cavity baking and Q-slope improvement is due to oxygen diffusion in niobium. The real mechanism is not understood but oxygen is necessary to improve the niobium RF performances.

This result, combined with experimental observations made after hydrofluoric acid treatments [4], suggests definitively that modifications of R_{BCS} and high field Q-slope involve deep material and not restrictively the Nb₂O₅/Nb interface. On the contrary R_{res} and low field Q-slope are linked with niobium sub-oxide (Fig.6).

The effective improvement at high field needs a very narrow tuning range of the oxygen concentration in niobium, probably caused by two antagonistic effects:

§ interstitial diffusion leading to the electron mean free path decrease with, as consequences, R_{BCS} and thermal conductivity modifications,

§ “doping” effect which nature needs to be identified.

Niobium vacancies are known, among others, to pin flux lines dissipating RF power. Filling vacancies with oxygen (Frank Turnbull mechanism) could be an explanation to this “doping effect”...

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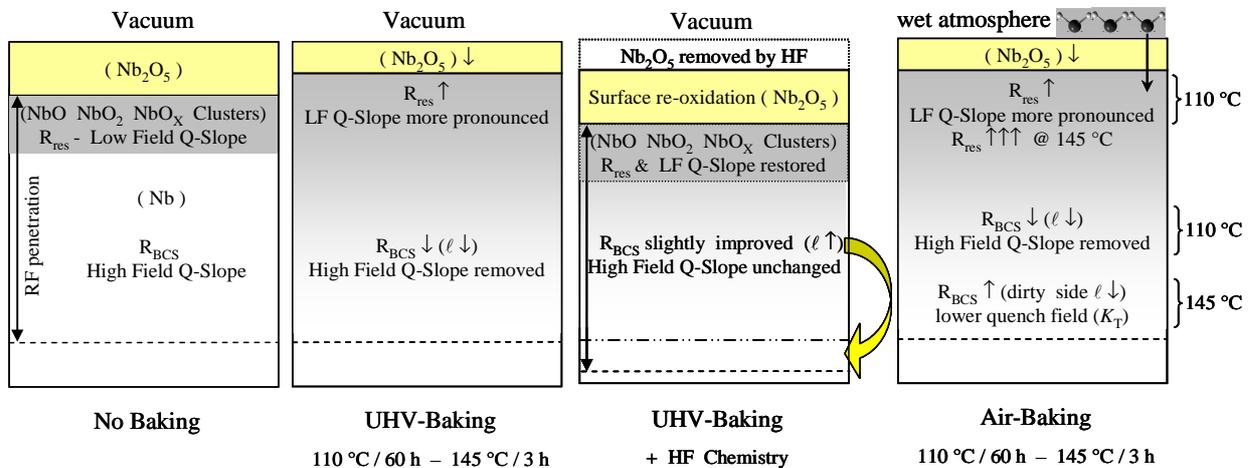


Figure 6: Sketches of Nb surface (layer scale is not respected) summarising experimental changes of R_{BCS}, R_{res}, high and low field Q-slopes after baking and HF cavity treatments.