« FAST ARGON-BAKING » PROCESS FOR MASS PRODUCTION OF NIOBIUM SUPERCONDUCTING RF CAVITIES

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Abstract

In this alternative process superconducting RF cavities are quickly baked, open-ended in argon atmosphere. «Fast Argon-Baking» is especially well adapted for cavity mass production because it combines short baking time (only three hours) and simplicity since it does not require maintaining the cavity under vacuum during treatment. Oxygen-free atmosphere or vacuum is a necessary condition in "Fast Baking" techniques.

For better understanding of baking experiments, Secondary Ion Mass Spectroscopy analyses have been performed on Nb samples baked in similar conditions to cavities. While interstitial oxygen diffusion is assumed playing a role in Q-slope improvement, results show an insignificant change of oxygen concentration profiles.

INTRODUCTION

Baking is a necessary final stage in cavity preparation to reach high gradients with niobium superconducting cavities. To improve its performances at high accelerator field, cavity is commonly pumped out in Ultra High Vacuum conditions and baked around 110 °C for 2 days: this is the standard "In-situ UHV Baking"[1].

Nevertheless this technique is too long and too restrictive for the large-scale cavity preparation because of UHV requirements.

First improvement has been introduced in this process to avoid vacuum pumping during baking and its inherent risks of leak: "Air-Baking" at 110 °C for 2 days was successfully tested in 2003 [2].

With the same simplification concern and to drastically decrease the baking duration, "Fast UHV Baking" was developed last year [3] on the basis of a possible oxygen diffusion: this technique lasts only 3 hours at 145°C.

Obviously for the next step, these two last advantages should be combined into only one process, the "Fast Air or Argon-Baking".

FAST AIR/ARGON-BAKING

As reported in [3], fast air-baking was experimented on a 1300 MHz cavity, open-ended in clean room atmosphere and baked at 145 °C for 3 hours using infra-red emitters. Preliminary result was very disappointing. The quality factor vs. accelerator field Q_0 (E_{acc}) curve was deteriorated after "Fast Air-Baking" without high field Q-slope improvement. To explain such degradation in Q_0 value and quench field, an inadequate choice of time-temperature parameter was argued, considering an uncontrolled oxygen concentration and a possible diffusion from the surface.

So, complementary investigations have been carried out on the same C1-09 cavity, air-baked at 145°C for different durations. Before each baking, 20 µm of Nb are systematically removed from the inner surface of the cavity by buffered chemical polishing (BCP chemistry). RF measurements ensure that cavity performance is roughly restored (Fig.1).

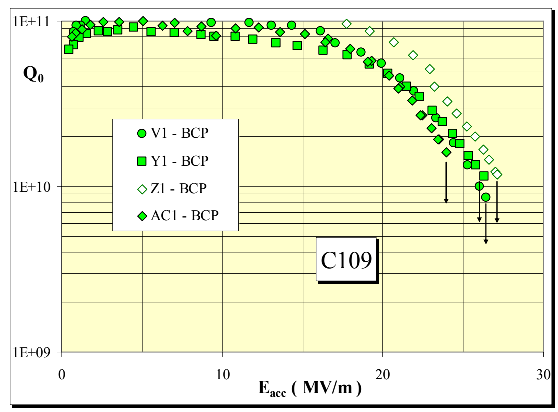

Figure 1: RF tests of C1-09 cavity before baking. Presence of downward arrow indicates thermal breakdown (quench).

As we see in Fig. 2, results show performance degradation in Q_0 and quench field value when the cavity is baked at 145°C in clean room air, whatever the baking duration.

In spite of these poor results and persisting to combine fast baking without UHV requirements, we decided to bake the open ended cavity in a stove, filled with argon. The result is shown in Fig.3 where we can see the attempted effect with the high field Q-slope improvement after baking (curves AE1 and AE2). Degradation is one more time confirmed when the cavity is baked with air instead of argon atmosphere (AF1).

Q-slope improvement, visible on Fig.3-AE2 could be more noteworthy using a new cavity because thermal conductivity of C1-09 is modified after successive baking treatments and quench is therefore reached at lower accelerator fields.

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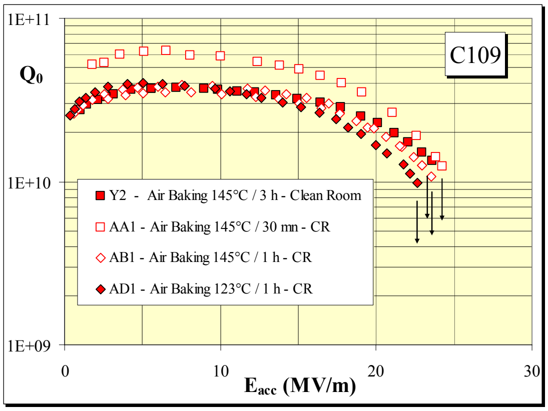

Figure 2: Degradation of cavity performances observed after "Fast Air-Baking" performed in different conditions.

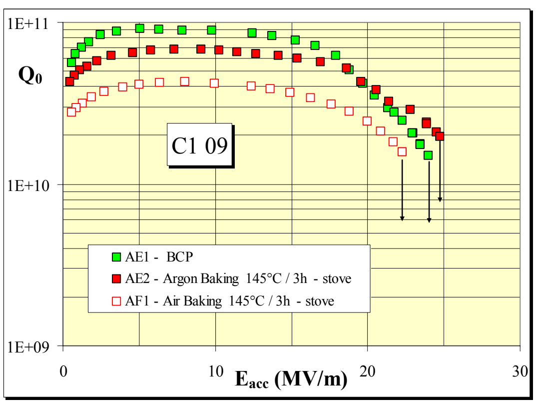

Figure 3: Comparison of "Fast Baking" effect in stove filled with air or argon.

TOF-SIMS ANALYSES

For a better understanding of baking phenomenon, Secondary Ion Mass Spectroscopy (SIMS) analyses were performed on Niobium samples to analyse the oxygen concentration profiles for different baking conditions.

Four niobium samples (14 x 14 x 2 mm) have first undergone a heat treatment at 1150°C during 9 hours in UHV furnace (4.10^{-7} mbar). Large grains are visible at the sample surface. The reference (sample A) is not baked; the other ones are put inside a pill-box cavity and baked according to:

- a standard UHV baking in a vertical cryostat at 110 °C for 60 hours (sample 8),
- a fast UHV baking in clean room at 145 °C for 3 hours (sample 3),
- a fast air baking in clean room at 145°C for 2, 1 and 1/2 hours respectively for samples 5, 6 and 7.

"Time of Flight SIMS" analyses were carried out on these samples at BIOPHY Research with a TOF-SIMS IV apparatus. Secondary Ions, components of the analysed surface (50 μm^2), are sputtered by a low intensity primary ion beam (Ga^+ - 0.5 pA). They are separated according to their charge on mass ratio value by a time of flight mass analyzer, reflectron type. The analysis area is chosen in the middle of a flat grain near the sample centre.

The analysis in depth (100 nm) has been carried out by the alternate use of a second high intensity ion beam (Ar^+ - 50 nA) to sputter a large surface (300 μm^2).

During the analysis, ion beam interacts with surface by sputtering and ionisation: molecules can be fragmented and / or associated with atoms in cluster forms. These phenomena produce a very complex ionic spectrum to be analysed. Moreover, due to differences in sputtering rate and ionisation cross section, the different TOF-SIMS intensities of ions can not be compared between them. It is just possible to have a qualitative comparison, from one to the other sample, for the same element.

TOF-SIMS signal is also dependant on the niobium lattice structure (lattice effect). For example, we can see on Fig. 4 signal change at the Nb_2O_5 – Nb transition. This is the way the niobium pentoxide depth might be estimated around 4 nm. Deeper, in bulk area, smaller change with grain structure can also be observed on niobium signals. This effect is clearly shown on sample 5 where two different grains are analysed in curves 5(1) and 5(2).

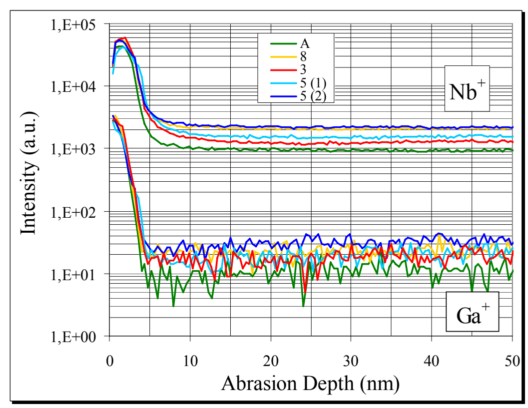

Figure 4: TOF-SIMS profiles of Nb and implanted Gallium ions (coming from the analysis beam).

Interstitial oxygen appears under cluster forms and similar profiles are observed on NbO, Nb_2O, Nb_3O and Nb_4O positive ion signals. To get rid of the 'lattice effect" Nb_XO/Nb_X signals are recorded (Fig.5). The analysis of Fig.5 shows deep oxygen diffusion into bulk niobium after air baking at 145°C for 30 mn,1 and 2 hours. That could explain the poor performances, observed in Fig.2, of the cavity treated with "Fast Air Baking".

On the contrary, samples 8 and 3, baked using "Standard" or "Fast UHV" processes, do not show significant difference with the reference profile (A). That is a very surprising result because oxygen diffusion was until now alleged playing a role in the baking effect. In the light of these experiments, it is necessary to reconsider the role of the oxygen coming from material surface or oxide-metal interface. Oxygen diffusion appears like a physical process to be avoided. The optimum time-temperature parameter, leading to Q-slope improvement (110 °C / 60 h, 145 °C / 3 h), appears then like the upper limit before noticeable oxygen diffusion in superconducting material begins.

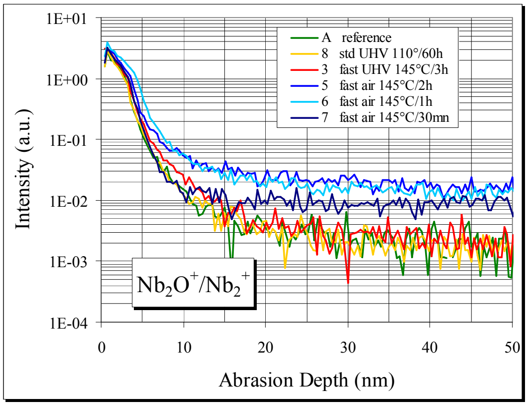

Figure 5: TOF-SIMS profiles of Nb_2O/ Nb_2

« BAKING - RESISTANT » CAVITIES

Since 1998, baking effect has been widely demonstrated on various cavities at Saclay. Performances of these cavities, manufactured by CERCA with Nb material provided by different suppliers (cross-rolled sheets with RRR > 260), are similar:

- Without any thermal treatment on cavities, the accelerator field was limited by quench around 15 MV/m,
- After annealing at 1350°C with titanium, the quench field was improved up to 25-30 MV/m,
- The high field Q-slope can be removed after baking treatment.

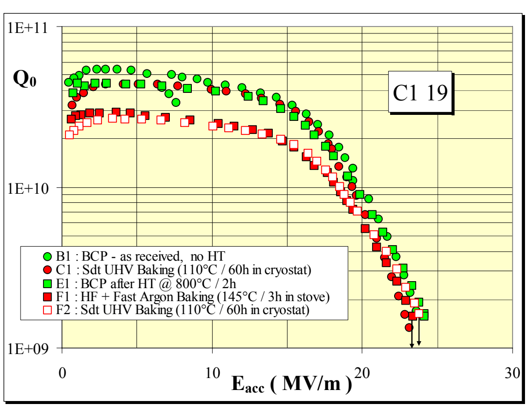

Figure 6: Q-slope unchanged after baking on C119.

Nevertheless, new cavities, C1-19 (Tokyo-Denkai niobium) and AC1-01 (Wah Chang Nb), respectively manufactured by CERCA and ACCEL, revealed an unusual behaviour. These cavities, thermally untreated, show very good performances, compared to the previous ones, with Q-slope limited by RF power supply around 23 MV/m. Their distinguishing mark is their insensitivity to baking treatment (Q-slopes unchanged in Fig.6 & 7), before or after heat treatments at 800 °C (C1-19) and 1350 °C with titanium (AC1-01). In particular the "Fast Air-Baking", in Fig.7 - A4, which usually causes heavy degradations in cavity performance, has no effect on AC1-01.

There is every indication that in some cases high field Q-slope could result from a different origin and so could not be solved by cavity baking. As there is yet no alternative solution to remove Q-slope for these atypical cavities, a serious problem to reach high gradients is raised.

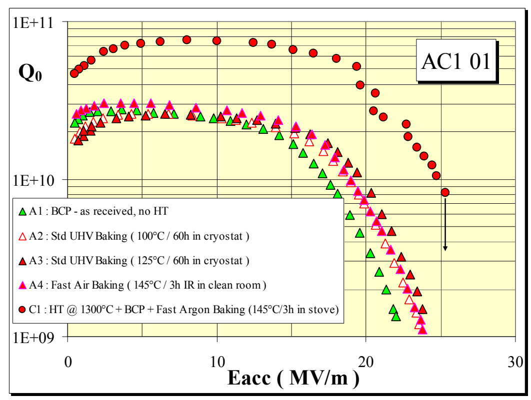

Figure 7: No Baking effect on Q-slopes for AC1-01.

CONCLUSION

«Fast Argon Baking» simplifies the treatment by baking of high gradient superconducting cavities through duration shortening and avoiding inner pumping of the cavity. This technique should find its application in cavity mass production.

In few cases, cavities are insensitive to baking treatment without any apparent reason. This phenomenon has to be understood and solved because it put out of reach the high gradients for such cavities.

TOF SIMS analyses on baked Nb samples show that interstitial oxygen diffusion is linked to degradation of cavity performances. On the contrary, O concentration profile is unchanged after baking linked to the high field Q-slope improvement.

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