

## Change In High Field Q-slope By Baking And Anodizing

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### Abstract

Low temperature RF performance of two niobium cavities that underwent different chemical treatments was measured after they were heat treated at 100<sup>o</sup>C for 48 hours. After heat treatment cavities were anodized in ammonia hydroxide solution for sequentially increasing voltage until baking effect was gone. The thickness of niobium finally consumed is estimated to be 20 nm. The results are discussed in view of one of the current models for the baking effect on the high field Q-slope.

### INTRODUCTION

The high field Q-slope is a well-known effect of degradation of the quality factor of niobium cavities at high RF fields without X-rays. A low temperature baking reduces the high field Q-slope by modifying the niobium layer corresponding to the RF penetration depth in a manner that is not yet understood.

One crucial point in the understanding of the phenomenon is a depth scale of the baking effect. From our studies we know that a chemical etching for as few as several micrometers is enough to remove the baking improvement. Assuming a finite length of the baking effect we called a layer of niobium, adjacent to the oxide, a "baking modified layer". In our attempts to find a thickness of the baking modified layer we performed a series of experiments after repeated anodizing[1] of a baked cavity. Anodizing allows a controllable growth of the niobium oxide on the niobium surface. By anodizing we were sequentially growing thicker oxide layer in place of the "baking modified layer". RF currents were sequentially shifted further into the bulk, because the grown oxide is an insulator. After growing oxide layer of less the one micrometer thickness we expected to reach a niobium layer that didn't possess the properties of the baking modified layer. As a result the improvement in the cavity performance due to mild temperature baking should vanish.

### EXPERIMENTS

The summary on the cavities' properties is presented below:

	<i>CavityNo.1</i>	<i>CavityNo.2</i>
Frequency	1500 GHz	1500 GHz
Shape	Elliptical MARK-III[2]	Elliptical MARK-III
RRR	500	300
Grain size	1 mm	1 mm
Thickness	3 mm	3 mm
Initial Treatment	BCP(1:1:2)	Vertical EP[3]

Before each test cavities were rinsed for two hours in the clean room with high purity water under 1000 psi high pressure. Assemblings and disassemblings from the test stand were done in the clean room class 100. The description of test stand, cryogenic and RF setup can be found in [4]. Temperature mapping system[2] was used to see heating distribution on the cavity surface. Baking was done by hot air flow, where heater was controlled by computer. The temperature distribution around the cavity was within 5<sup>o</sup>C.

### Experiments with cavity No.1

- The cavity was baked at 100<sup>o</sup>C "in situ" for 48 hours.
- The cavity was tested at the helium bath temperature of 1.7<sup>o</sup>K.
- The cavity was anodized for 5 volts in 15% ammonia hydroxide solution.
- The cavity was tested at the helium bath temperature of 1.5<sup>o</sup>K.
- The top-half of the cavity was anodized for 60 volts, the bottom-half of the cavity was anodized for 30 volts in 15% ammonia hydroxide solution. The difference in RF properties between top and bottom half-cell was determined by thermometry.
- The cavity was tested at the helium bath temperature of 1.5<sup>o</sup>K.
- The cavity was baked at 100<sup>o</sup>C "in situ" for 48 hours.
- The cavity was tested at the helium bath temperature of 1.5<sup>o</sup>K.

### Experiments with cavity No.2

- The cavity was baked at 100<sup>o</sup>C "in situ" for 48 hours.
- The cavity was tested at the helium bath temperature of 1.6<sup>o</sup>K.
- The cavity was anodized for 10 volts in 15% ammonia hydroxide solution.
- The cavity was tested at the helium bath temperature of 1.6<sup>o</sup>K.
- The cavity was anodized for 20 volts in 15% ammonia hydroxide solution.
- The cavity was tested at the helium bath temperature of 1.6<sup>o</sup>K.
- The cavity was baked at 100<sup>o</sup>C "in situ" for 48 hours.
- The cavity was tested at the helium bath temperature of 1.6<sup>o</sup>K.

## RESULTS

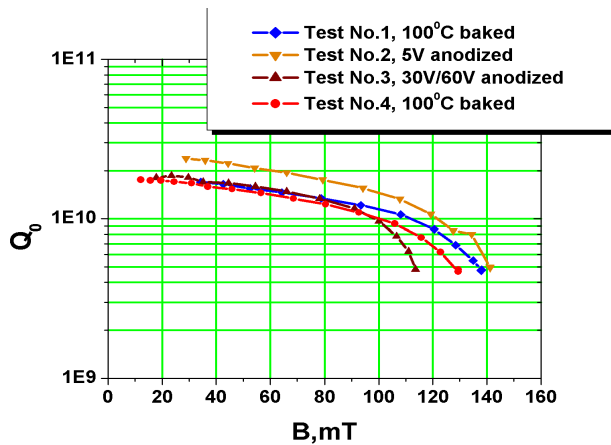


Figure 1: Results for anodizing tests of BCP cavity. Typical experimental error is 10% in quality factor and 5% in field.

Results for cavity No.1 are presented in Fig.1 (in all figures quality factor is plotted versus peak surface magnetic field). After 100°C "in situ" baking for 48 hours the cavity's highest peak magnetic field was 140 mT with quality factor of  $5 \cdot 10^9$  (Fig.1, blue curve). X-rays were not detected.

The 5 volts anodizing slightly improved low field quality factor, but high field and Q-slope at high fields remained unchanged (Fig.1, yellow curve). X-rays were not detected in this test.

After 30/60 volts anodizing the performance of the cavity became as it is before baking [5] (Fig.1, green curve). X-rays were not detected. The temperature maps at high field showed that top half and bottom half of the cavity were equally lossy.

After another 100°C "in situ" baking for 48 hours the cavity performance improved (Fig.1, red curve). X-rays were detected.

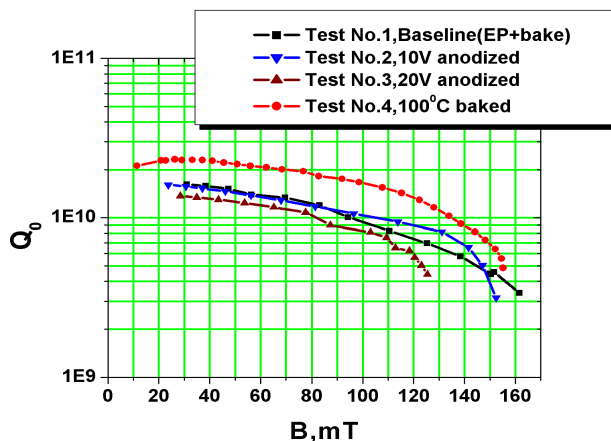


Figure 2: The test results of electropolished cavity.

Results for cavity No.2 are presented in Fig.2. The first RF measurements with electropolished cavity were carried after baking at 100°C "in situ" for 48 hours. The cavity reached 150 mT with quality factor about  $5 \cdot 10^9$ . X-rays were detected. Temperature map showed that the cavity's performance was suppressed by field emitter.

After anodizing for 10 volts the low field performance was the same, and the highest field was about 150 mT with  $5 \cdot 10^9$ . But the slope at high fields is stronger and temperature map suggests that cavity performance wasn't limited by field emission this time. The temperature map shows that the heating was distributed around the cavity surface.

After anodizing for 20 volts the low field quality factor was slightly lower than in previous tests. The cavity had a strong high field Q-slope, the highest field we reached was 125 mT with quality factor about  $5 \cdot 10^9$ . X-rays were detected. Temperature map suggests that field emission was not a dominant loss mechanism.

After the cavity was baked again 100°C "in situ" for 48 hours, the low field quality factor was more than  $2 \cdot 10^{10}$ . The highest field was more than 150 mT with quality factor  $5 \cdot 10^9$ . X-rays were detected. The temperature map showed that field emission was dominant loss mechanism in this test.

Results (Fig.1., Fig.2.) suggest that 30 volts of anodizing is sufficient to completely remove the benefit of 100°C baking for 48 hours. In [6] 20 Angstrom per volt is used as the oxide growth rate for almost the same electrolytic solution. Thus anodizing for 30 volts corresponds to oxide thickness of 60 nm. In [7] three different values for niobium pentoxide density quoted depending on the structure: amorphous,  $4.36 \text{ g/cm}^3$ ;  $\gamma$ ,  $5.17 \text{ g/cm}^3$ ; and  $\alpha$ ,  $4.55 \text{ g/cm}^3$ . We took the density to be  $4.47 \text{ g/cm}^3$  and atomic mass of two atoms of niobium and five atoms of oxygen is  $4.42 \cdot 10^{-22} \text{ g}$ . The density of niobium is  $8.58 \text{ g/cm}^3$  and atomic mass of two niobium atoms is  $2 \cdot 1.54 \cdot 10^{-22} \text{ g}$ . Thus volume per an atom of niobium in niobium pentoxide is 2.74 times more than that in bulk niobium. The oxide grew only in one dimension, so we estimate the amount of niobium consumed, in order to make the oxide of 60 nm, to be around 22 nm. This is the depth of baking benefit for 100°C baking for 48 hours.

## DISCUSSION

Several models have been purposed to explain baking effect and high field Q-slope (a short review can be found in [8]). The most attention receives an oxygen pollution model (e.g [9]) and modified oxygen pollution layer [10]. This model suggest degradation of quality factor of niobium cavities due to high concentration of interstitial oxygen in niobium near oxide. In the framework of this model the fact that 30/60 volts anodizing removes baking effect means that in course of anodizing niobium RF layer was polluted by oxygen to the same concentration as before baking. But then during 5 volts anodizing the niobium layer is also polluted by oxygen and this treatment should

have removed baking improvement too, while the baking improvement after this treatment stayed.

The modification of niobium oxide and the diffusion of interstitial oxygen in niobium during mild temperature baking is verified both by surface measurements[11,12] and measurements done with cavities at low fields[13,14]. But the fact that the first several nanometers of niobium can be oxidized with little or no change in high field Q-slope is surprising in the view of any oxygen based model for the high field Q-slope.

Other light impurities usually are out of the consideration, because they don't seem to be mobile enough[15] to move from the RF penetration depth into the bulk. But first we want to point out that there could be a concentration gradient between the bulk concentration of an impurity and the concentration of the impurity in niobium pentoxide, which grows to 3-5 nm thickness during high pressure rinsing with ultra high purity water. Secondly in [16] we found different diffusion coefficients cited for carbon in the temperature range of interest. Both [15] and [14] used the same method in the determining of the diffusion rates, so it is not clear, where the difference in the results came from.

To illustrate what was said above we considered a simple model problem: we assumed an initial step-function in the carbon concentration between the niobium pentoxide and the bulk niobium; we assumed the same constant diffusion coefficients both for the oxide and for the bulk niobium; and we assumed that carbon can freely move through the niobium-niobium oxide interface. For this model problem the diffusion equation is:

$$\frac{\partial C(x, t)}{\partial t} = D_0(T) \frac{\partial^2 C(x, t)}{\partial x^2}$$

$$C(x, 0)|_{0 < x < \delta} = C_0; C(x, 0)|_{\delta < x} = C_1;$$

$$C(x, 0)|_{x=\delta} = \frac{C_0 + C_1}{2};$$

$$C(x, t)|_{x=+\infty} = C_1; \frac{\partial C(x, t)}{\partial x}|_{x=0} = 0;$$

, where  $C(x, t)$  is concentration of interest in arbitrary units,  $D_0(T) = 1.5 \cdot 10^{-6} \cdot \exp\left(\frac{-13600}{T(0K)}\right) \text{m}^2 \text{s}^{-1}$ , where  $T$  is temperature in Kelvin, is diffusion coefficient from [16],  $\delta$  is oxide thickness,  $C_1$  is concentration of impurity in the bulk and  $C_0$  is concentration of impurity in the oxide. The solution to the model problem is:

$$C(x, t) = C_1 + \frac{C_0 - C_1}{\sqrt{\pi}} \int_{\frac{x-\delta}{2\sqrt{D_0(T)t}}}^{\frac{x+\delta}{2\sqrt{D_0(T)t}}} \exp(-z^2) dz$$

Now we assume that the concentration of the carbon in the niobium pentoxide is 2.74 times less in the bulk niobium and consider a redistribution of the carbon under different baking conditions (Fig.3). The solution of the model problem shows that baking at 100°C for 48 hours lowers the concentration of the impurity in the first 30 nm of bulk niobium in this model. But baking at 150°C for 48 hours has a minor effect on the impurity concentration in first 50 nm

of bulk niobium. The baking at 150°C for 2 hours also reduces the amount of the impurity in the RF layer.

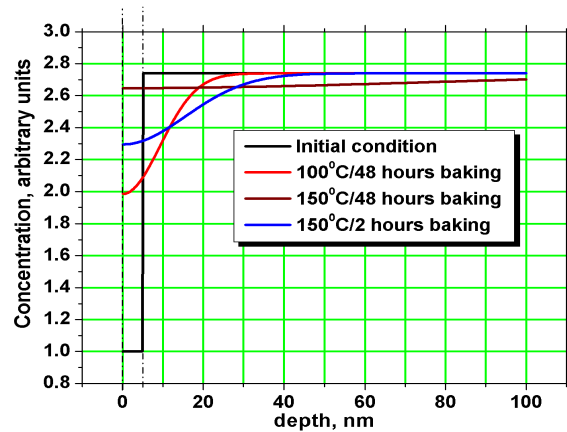


Figure 3: Solution of model problem

## CONCLUSION

The baking effect takes place in a first few tens nanometers of niobium adjacent to the niobium oxide. For 100°C for 48 hours baking less than 30 volts anodizing is required to remove baking effect. These anodizing experiments present difficulties for the pollution model to explain the Q-slope, and the dilution during baking to explain the improvement of the Q-slope. Other impurities may be involved in the baking effect. A careful surface analysis is required to answer this question.

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