Temperature Map Studies on Nearly Oxide-Free, Thin-Oxide and Standard-Oxide Cavities.*

G. Eremeev†, H. Padamsee,
CLASSE, Cornell University, Ithaca, NY, 14853, USA

Abstract

A few nanometers of niobium oxide cover niobium in niobium cavities, prepared by standard treatments. Since the RF penetration depth is a few tens of nanometers, the niobium oxide and the metal-oxide interface may play role in RF losses of superconducting niobium. In order to understand the cause of phenomena such as the high field Q-slope, medium field Q-slope, and residual resistance, it is important to distinguish the contributions of the niobium oxide and its interface to losses at medium and high fields. XPS and Auger studies have shown that it is possible to reduce significantly the thickness of the oxide layer by heating to 300^0C - 400^0C for a few hours in vacuum. Leaving the surface in the vacuum does not re-grow the oxide layer. Applying such treatment to a cavity one can reduce the niobium oxide and measure the superconducting RF properties of a nearly oxide-free cavity. Then via controllable air exposure one can re-grow oxide and investigate the change in properties as a function of exposure. We performed these experiments and report results of nearly oxide-free, thin-oxide and standard-oxide cavities.

EXPERIMENTS

The cavity was a 1.5 GHz elliptical cavity, made of 300 RRR niobium sheet of 3 mm thickness with 1 mm grain size. The half-cells, shaped by deep drawing, were postpurified at 1300^0C for 2 hours and then at 1200^0C for 4 hours. The half-cells were vertically electropolished [1] before welding. After welding the cavity was vertically electropolished for about 100 μm. After electropolishing the cavity was chemically treated with BCP (HF:HNO_3:H_3PO_4 - 1:1:2) for 10 sec (flash BCP). The temperature of the solution was kept below 15^0C. After the chemical treatment the cavity was rinsed with ultra pure water and transferred to the clean room of class 100. In the clean room the cavity was rinsed for two hours with high purity water under 1000 psi pressure. Then the cavity was assembled on the 1500 MHz test stand in the clean room. Each test was carried out at the bath temperature of 1.5^0K at low fields and increased to 1.7^0K at high fields.

For the heat treatment a steel box was put around the cavity and pressurized with argon, preheated to 250°C, to avoid oxidation of the outer surface of the cavity during the heat treatment. The inside of the cavity was kept under UHV. The heating elements were two 1.5 kW band heaters placed on copper rings, which were shaped to follow the shape of the cavity. Copper foils were used to improve the temperature distribution. Only the cell itself was heat treated, the rest of the vacuum system was kept at a room temperature by water-cooling clamps on beam pipes. The pressure during the heat treatment was $4.5 \cdot 10^{-7}$ Torr. The main constituents were water($2 \cdot 10^{-7}$ Torr) and CO($3 \cdot 10^{-8}$ Torr) as was indicated by residual gas analyzer. The oxygen partial pressure was $8 \cdot 10^{-11}$ Torr. The mean delay between heat treatment and RF cryogenic test was two days.

For air exposure VOC Zero Grade Air from Airgas was used. After air exposures the cavity was pumped down to 10^{-7} Torr.

The sequence of all tests is presented below:

- The cavity was tested.
- The cavity was baked.
- RF test.
- The cavity was exposed to $\approx$ 150 Torr·sec of dry air.
- RF test.
- The cavity was baked.
- RF test.
- The cavity was exposed to $\approx$ 150 Torr·sec of dry air.
- RF test.
- The cavity was exposed to $\approx$ 10^7 Torr·sec of dry air(1 atm for 12 hours).
- RF test.
- The cavity was baked.
- RF test.
- The cavity was exposed to $\approx$ 300 Torr·sec of dry air.
- RF test.
- The cavity was exposed to $\approx$ 10^7 Torr·sec of dry air(1 atm for 24 hours).
- RF test.

RESULTS

First set of experiments

In order to compare the performance of a nearly-oxide-free, thin-oxide and standard-oxide niobium cavities, it was necessary to establish a baseline for the high field performance. Thus the first step was to test after chemistry. After the cavity was chemically treated, assembled in the clean room, the thermometry system [1] was assembled and

* Work supported by NSF
† gve2@cornell.edu

the cavity was tested in the cryostat. The low-field quality factor of the cavity was about $3{\cdot}10^{10}$, Fig. 1[squares]. Above B_{peak} = 110 mT the high field Q-slope was observed, which is typical for this type of cavities. At B_{peak} = 123 mT we were limited by available RF power(30 W), the quality factor was about $3{\cdot}10^{9}$. The temperature map taken above 110 mT shows three hottest regions on the bottom half-cell, Fig. 2. All temperature sensors, both in these hot regions and in other high-magnetic-field regions equally showed a non-quadratic increase in heating, the high field Q-slope, above 100 mT, Fig. 4[squares]. No x-rays were peresent during the test. Thus in this test the baseline for high field Q-slope's studies was established.

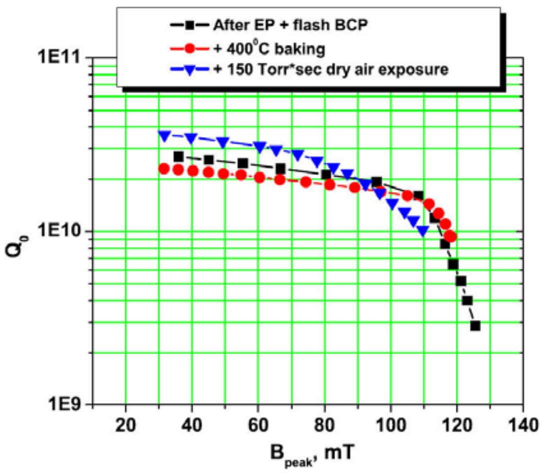

Figure 1: Quality factor versus peak magnetic field for the first three tests: after chemical treatment the excitation curve has a typical medium- and high-field Q-slope, after the cavity was baked at 400°C, after the cavity was exposed to ≈ 150 Torr·sec of dry air.

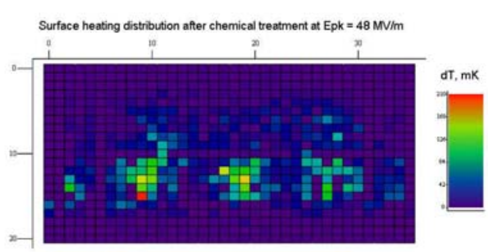

Figure 2: Temperature map at B_{peak}=117 mT after the chemistry show three broad hot-spots.

Having established the baseline for the study of the high field Q-slope, we proceeded to 400°C baking. The cavity was taken out of the cryostat, the baking setup was put onto the cavity and the cavity was heated to 400°C for two hours under UHV condition. Then the baking setup was removed, the thermometry system was put onto the cavity, the cavity was lowered into the cryostat and tested. The excitation curve for this test is presented in Fig. 1[circles]. The excitation curve for this experiment differs little from that in the baseline test. The highest B_{peak}, which was reached, was 117 mT in this test, where the cavity was limited by a thermal breakdown. The temperature maps in the high field Q-slope's region shows four broad hot regions, Fig. 3, which typical for the high field Q-slope, but the analysis of the reading of individual thermometers shows that three of these regions were also hot at low fields, because they have higher surface resistance after baking, Fig. 5[circles]. The resistance in these regions was order of magnitude higher than in other regions. Except for the thermometers in these three hot regions, which cover about 15 percent of the cavity surface, all thermometers show readings typical for the high field Q-slope, Fig. 4[circles]. So we concluded that the cavity still exhibited the high field Q-slope after the 400°C baking.

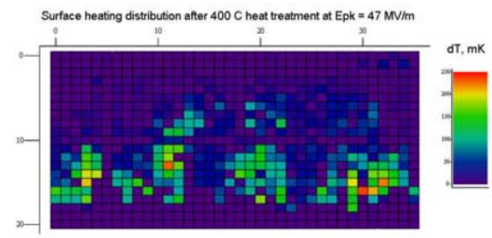

Figure 3: Temperature map at B_{peak}=117 mT after the cavity was baked at 400°C.

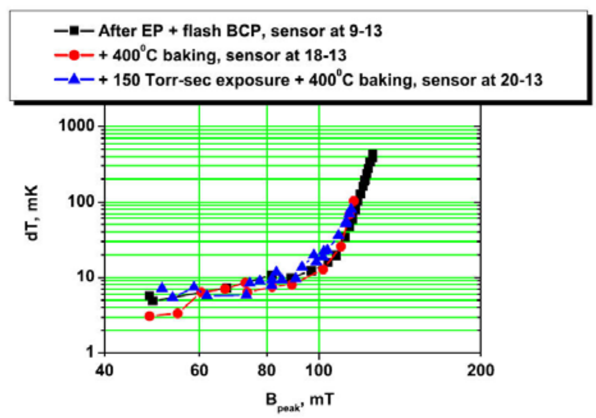

Figure 4: Thermometry data from the regions that showed high field Q-slope: after chemistry, after first 400°C baking, after second 400°C baking.

Since 400°C baking dissolved the oxide, we speculated that lossy suboxides can be created by a controllable exposure to dry air. Typically a delay between the 400°C baking and RF test was two days, therefore the niobium surface is inevitably exposed to about .1 Torr-sec of water vapors, which put the lower limit on the exposure. The upper limit is set by exposure to one atmoshpere of air. First it was decided to expose the cavity to about 100 Torr-sec. The cavity was warmed up and then, while still inside the cryostat, was pressurized to 2.4 Torr for a few minutes. After exposure it was evacuated and an RF test was performed. In this test the low-field quality factor improved, Fig. 1[triangles], but the cavity exhibited a strong medium-field Q-slope. The cavity was limited by a thermal breakdown at B_{peak} = 114

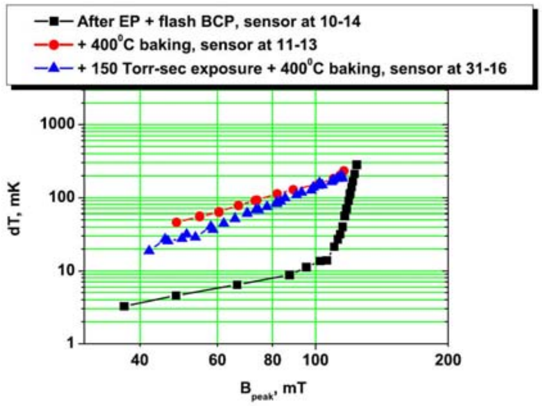

Figure 5: Thermometry data from the regions that showed high surface resistance: after chemistry, after first 400°C baking, after second 400°C baking.

mT. The field at which the cavity was limited in this test is lower than that at which the high field Q-slope typically starts. The temperature map shows that, following the short exposure, a hot spot appeared on the top half-cell, Fig. 7. This hot spot was a major source of losses. The readings of the thermometers from this region show non-quadratic increase with field, Fig. 8[circles]. The three regions with a high surface resistance from the previous test still exhibit losses approximately an order of magnitude higher than typical. However these high-surface-loss regions were improved after the short exposure, Fig. 6[circles].

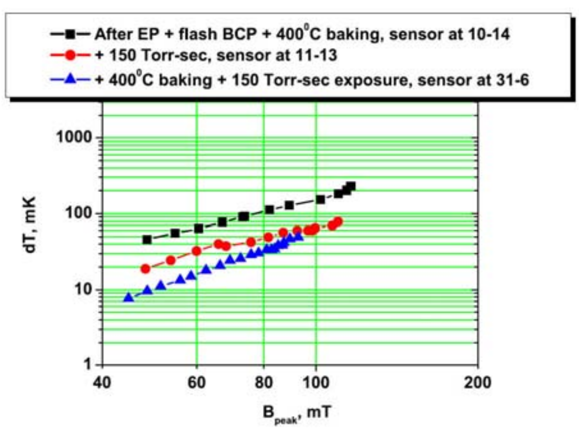

Figure 6: Thermometry data from the regions with high surface resistance after 400°C baking, that shows improvement of this regions by short exposure: after first 400°C baking, after first 150 Torr·sec air exposure, after second 150 Torr·sec air exposure.

Second set of experiments

In order to confirm results of the first 400°C baking and air exposure, the experiments were repeated. Since 400°C baking remove the oxides from niobium surface, it was suggested that the strong medium field Q-slope will be removed by the 400°C baking and the cavity performance

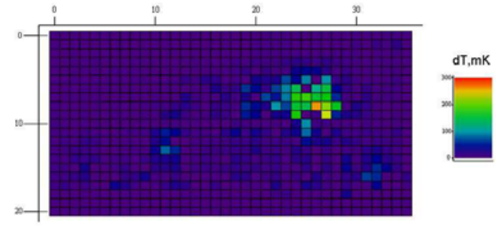

Figure 7: Temperature map at B_{peak}=110 mT after the cavity was exposed to ≈ 150 Torr·sec.

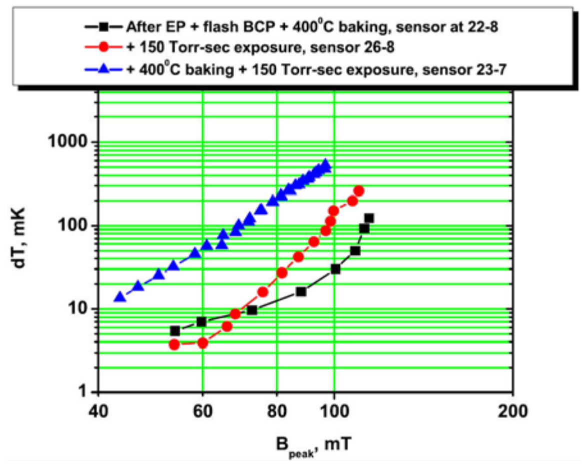

Figure 8: Thermometry data from the regions that showed anomalous losses after short exposures: after first 400°C baking, after the cavity was exposed to ≈ 150 Torr·sec, after the cavity was exposed to ≈ 150 Torr·sec following a second 400°C baking.

will be as it was after the first 400°C baking. Thus after the short exposure the cavity was taken out of the cryostat, the thermometry system was removed, the baking setup was arranged and the cavity was baked at 400°C for about two hours. After the baking the cavity was lowered in the cryostat and RF test was performed. In this test the excitation curve was similar to the one after the first 400°C baking, Fig. 9[squares]. The cavity had a medium-field Q-slope similar to that after chemistry and after 400°C baking. The limitation was a thermal breakdown at B_{peak} = 115 mT. Temperature maps at high fields show that the region with anomalous losses, which appeared after the short exposure, was still present, but was substantially improved. The quench site was in the one of the three high-surface-resistance regions, which appeared after the first 400°C baking. Except thermometers from these four regions, all thermometers show field dependence typical for the high field behavior, Fig. 4[triangles].

In order to confirm the appearance of the strong medium field Q-slope and accordingly high-loss anomalous regions, another short exposure, 150 Torr·sec, was done. After the second short exposure the low-field quality factor decreased by a factor of two and was about 10^{10}, Fig. 9[circles]. As it was expected the excitation curve exhibits a strong medium field Q-slope. The highest B_{peak},

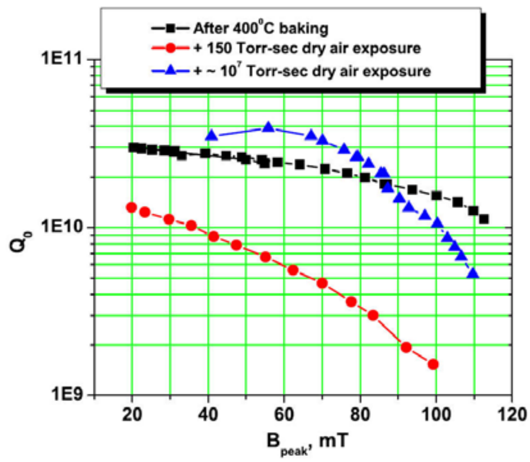

Figure 9: Quality factor versus peak magnetic field for the second group of three tests: after the cavity was baked at 400°C, after the cavity was exposed to ≈ 150 Torr·sec of dry air, after the cavity was exposed to ≈ 10^7 Torr·sec

achieved, was 100 mT due to lack of power. The quality factor of the cavity at B_{peak} = 100 mT was about 10^9. The temperature map shows that again a high-loss region was formed in the process of the short exposure. The temperature sensors again shows non-quadratic dependence, Fig. 8[triangles].

Because it was suspected that the high-loss regions formed after the short exposure are caused by formation of suboxides, the cavity was warmed up and then exposed to 1 atm of dry air for about 12 hours, ≈ 10^7 Torr·sec, in attempt to convert possible metallic suboxide to dielectric pentoxide. Indeed the low-field quality factor was recovered by the long exposure. The excitation curve in this test has a unusually pronounced low field Q-slope, which was present up to B_{peak} = 60 mT. After the summit at B_{peak} = 60 mT the quality factor started to decline similarly to the previous experiment. At B_{peak} = 110 mT the experiment was stopped due to lack of power. The temperature maps at high fields show that the anomalous losses, though reduced after long exposure, were still present, Fig. 10.

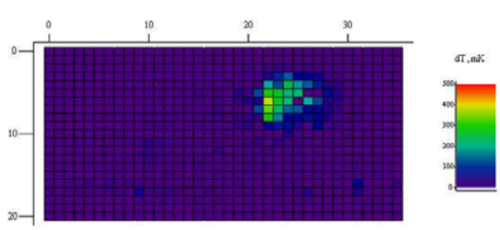

Figure 10: Temperature map at B_{peak}=115 mT after the cavity was exposed to ≈ 10^7 Torr·sec.

Third set of experiments

Finally third set experiments was carried out to confirm findings of the previous experiments. The cavity was taken out of the cryostat, the thermometry system was removed and the cavity was baked at 400°C for an hour, then the temperature was raised to 500^0C and the cavity was kept at this temperature for about half an hour. Then the baking setup was removed, the thermometry system was assembled, the cavity was lowered in the cryostat and tested. In this test there was no strong medium field Q-slope, the Q-slope was similar to those after previous bakes. The low-field quality factor was about $4{\cdot}10^{10}$. The cavity was limited by a thermal breakdown at B_{peak} = 117 mT. The analysis of the thermometry data show that all high-loss regions, which appeared after the short and long air exposures, were removed by the 400°C baking.

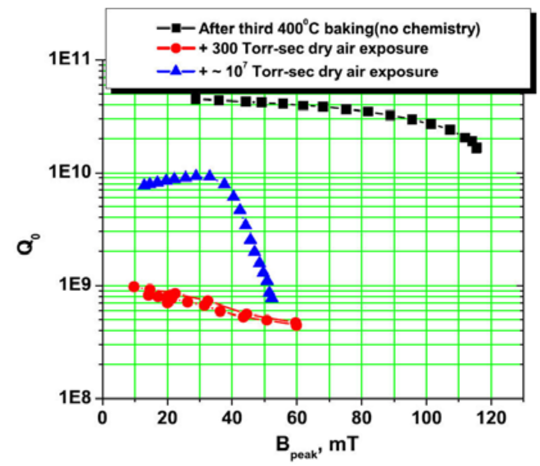

Figure 11: Quality factor versus peak magnetic field for the third group of three tests: after the cavity was baked at 400°C, after the cavity was exposed to ≈ 300 Torr·sec of dry air, after the cavity was exposed to ≈ 10^7 Torr·sec

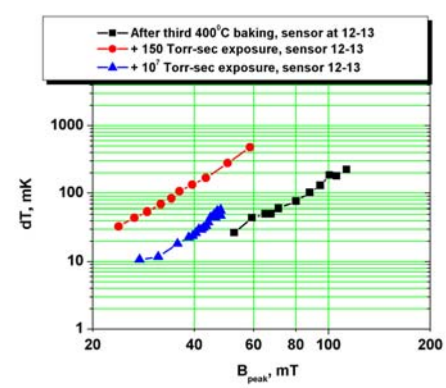

Figure 12: Thermometry data for the third set of experiments: after third 400°C baking, after the cavity was exposed to ≈ 150 Torr·sec, after the cavity was exposed to ≈ 10^7 Torr·sec

After the 400°C baking the cavity was warmed up and exposed to about 300 Torr·sec of dry air, then the baking setup was removed, the thermometry system was assembled, the cavity was lowered in cryostat and tested. The low-field quality factor was 10^9, more than an order of magnitude lower that in previous test. The exposure formed lossy bands on top and bottom half-cell of the cavity as it is shown on the temperature map, Fig. 13. These bands did not correspond to the highest-magnetic-field regions, but roughly corresponds to the places, where the heating bands were located during the 400°C baking. The thermometry data shows that both surface resistance and slope were higher in this regions compared to previous test, Fig. 12[circles].

To convert suboxides, possibly formed by the short exposure, the cavity was warmed up and exposed to a 1 atm of dry air for 24 hours, $\approx 10^7$ exposure. After the long exposure the low-field quality factor improved. The low-field quality factor was about 10^{10}. An interesting feature in this experiment is a pronounced low field Q-slope up to B_{peak}=30 mT. After the summit B_{peak}=30 mT the excitation curve has a strong medium field Q-slope. The measurements were limited by available power at B_{peak} = 50 mT with the quality factor of $8{\cdot}10^8$.

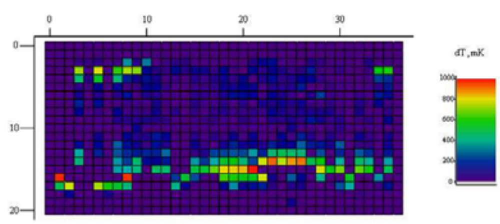

Figure 13: Temperature map at B_{peak}=50 mT after the cavity was exposed to $\approx$ 150 Torr·sec

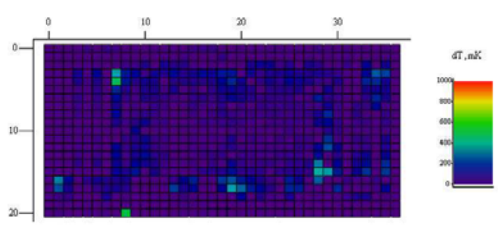

Figure 14: Temperature map at B_{peak}=48 mT after $\approx 10^7$ Torr·sec

DISCUSSION

Our study was aimed at the high field Q-slope. The 100°C baking is a common procedure used world-wide to improve a high-field performance of niobium cavities. To explain the high field Q-slope and the baking effect several models were suggested. Our studies have addressed two of them: the interface tunnel exchange model, ITE model [6], and the oxygen pollution model [3].

The former model attributes the high field Q-slope to losses caused by the natural oxide at high fields. In the ITE model the modification in the niobium pentoxide during the mild baking explains the improvement in the high-field performance. However the 400°C baking, which was done, removes the natural oxide [4], [5] and therefore the high field Q-slope should be removed according to ITE model. This is contrary to what was observed in the experiment.

The latter model assumes an oxygen pollution layer underneath the oxide. The oxygen-polluted layer has superconducting properties that are worse than those of a pure niobium. Baking of a cavity at 100°C for 48 hours, a typical baking procedure, dissolves the oxygen pollution layer into the bulk. The model however failed to explain the results after baking at higher temperatures, i.e. 150-180°C for 48 hours, in which the high field Q-slope remains unchanged or even degrades. In order to accommodate these results, the oxygen pollution model was modified by G. Ciovati [7]. He suggests that during baking the oxide layer dissolves and oxygen atoms from the oxide contribute to the oxygen pollution layer. Thus due to this enrichment process the oxygen pollution layer is not removed by baking at temperatures higher that 100-120°C. In order to see the predictions of this model for our experiment, we have calculated oxygen depth profile within the model after 400°C baking. From the calculation it follows that the oxygen concentration in the penetration depth is reduced by 400°C baking, Fig. 15. So we expected that the high-field performance will be improved by such baking, still we observed the high field Q-slope in our experiments and it was the same as before baking. So this model is also contradicted by these experiments.

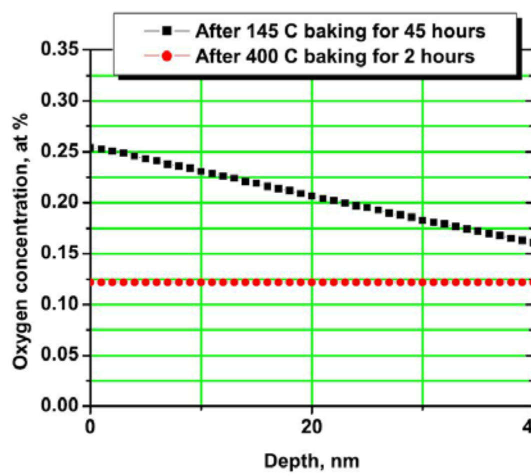

Figure 15: Diffusion calculation based on the modified oxygen pollution model proposed by G. Ciovati.

The presence of the high field Q-slope after 400°C baking can be explained with the model that was suggested earlier [8]. This model does not explain the high field Q-slope, but addresses only the baking effect. In this model a natural oxide is attributed a role of purifier of niobium during the mild baking. During the standard 100°C baking for 48 hours, impurities diffuse from niobium into adjacent oxide, thereby improving the superconducting properties of niobium. The higher temperature baking is less efficient because of a finite capacity of the oxide. Within this model no improvement can be expected after a 400°C baking, because the oxide layer is removed, and therefore the niobium purity is not improved. However recent surface studies suggest that carbon, which was initially proposed as a diffusing impurity in this model, is not responsible for the high field Q-slope. Surface studies have shown that during 300°C baking for 1 hour carbon segregates to the surface [9]. Thus if the carbon were responsible for the high field Q-slope, a degradation in the high field performance would be expected after the 400°C baking due to segregation of the carbon to the surface. But the high field Q-slope did not degrade, so even if the model holds true, carbon is not involved in high field Q-slope phenomenon.

An interesting result in these studies was a strong medium field Q-slope and high surface resistance after short exposures. The strong medium field Q-slope is sometimes observed when the niobium is enriched with hydrogen during chemistry. The excitation curve then has a strong medium field Q-slope, because niobium hydrides are formed during the cooldown if the hydrogen concentration is ≥ 6 at. percent [10]. We however suggest that hydrogen contamination is not the cause of the strong medium field Q-slope and a high surface resistance in our experiments, firstly, because the quality factor was improved by longer exposure, which does not help in the case of the hydrogen Q-disease. Secondly, because the medium field Q-slope was removed by 400°C baking, whereas in order to remove the typical Q-disease, cavities are treated at 800°C for several hours. We suggest that the reason behind a strong medium field Q-slope and high surface resistance is a formation of niobium suboxide. The idea supported by the fact that performance was improved by longer exposure, which presumably caused conversion of the suboxides to pentoxide, and then was completely restored by 400°C, which dissolved the pent- and suboxides. Thus these experiments open an interesting question about contribution of the niobium suboxides to the medium field Q-slope and to a low-field quality factor. To study these questions however the current design of the baking setup is to be improved in order to provide a more uniform temperature distribution during 400°C baking and surface studies on samples should be carried out in order to find out the surface composition after baking and exposures.

CONCLUSION

The performance of the nearly-oxide-free, thin-oxide and standard-oxide cavities was studied via 400°C baking. The performance of the niobium cavity after 400°C baking, i.e. the nearly-oxide-free cavity, was the same as before treatment except the performance was limited by thermal breakdown. The high field Q-slope was not removed by such treatment which contradicts the interface tunnel exchange model and the modified oxygen pollution model. The performance of the niobium cavity after short exposure, i.e. thin-oxide cavity, degraded both in quality factor and in field behavior probably due to formation of suboxides. The long exposure after short exposure improved the quality factor and field performance.

REFERENCES

[1] R.L.Geng et al., 12th Workshop on RF Superconductivity, 2005.

[2] J.Knobloch, PhD Thesis, Cornell University, 1997.

[3] Q. Ma et al., JAP, Volume 96, Issue 12, pp. 7675-7680, 2004.

[4] R. Kirby et al., SLAC-TN-05-049, 2005.

[5] J. Halbritter, 10th Workshop on RF Superconductivity, 2001.

[6] H. Safa, 9th Workshop on RF Superconductivity, 1999.

[7] G. Ciovati, Applied Physics Letters 89,022507, 2006.

[8] G.Eremeev et al., 12th Workshop on RF Superconductivity, 2005.

[9] J.Kaufman, personal communications.

[10] T.Schober and H. Wenzl, Hydrogen in Metals II, Topics in Applied Physics, Vol.29, p.32, 1978.