

## Q Degradations in Superconducting Niobium Cavities

Kenji Saito and Peter Kneisel

Continuous Electron Beam Accelerator Facility  
12000 Jefferson Avenue  
Newport News, VA 23606

### Abstract

In the past year, several laboratories around the world have observed degradations of the Q value of superconducting niobium cavities made from high thermal conductivity niobium under certain cooldown conditions. Especially under slow cooldown or warmup to temperatures <200 K of larger systems severe degradations have been reported.

A systematic study of the influence of the cooldown speed, warmup conditions, multiple cooldowns and chemical surface treatment on cavity performance of cavities manufactured from niobium of different purity has been conducted.

Possible cures such as anodic oxidation are being explored and results of these investigations are reported.

### INTRODUCTION

In 1989, R. Röth[1] observed a significant degradation of the Q value of a 1500 MHz cavity made from high thermal conductivity niobium after it had been kept at nitrogen temperature for several days after the initial test in superfluid helium. A temperature map indicated that these unexpected losses were distributed uniformly over the whole cavity surface.

Other laboratories[2][3] observed similar degradation of the Q value when the cavities were kept for longer periods of time at temperatures between 200 K and 50 K. These observations triggered a series of investigations into this phenomenon in several laboratories working with niobium cavities[4][5][6], and soon the initial hypothesis expressed in Ref. [1] of precipitation of the  $\epsilon$ -Nb-hydride phase was supported by additional experimental data provided by other laboratories. The detrimental effects of hydrogen precipitation in niobium has been known for many years[7].

It was also experimentally verified that a hydrogen degassing of the niobium at temperatures above 850° C eliminated the Q degradation phenomenon[3][4][6]. A procedure like this was used a long time ago for niobium cavities which had performed badly after long periods of electropolishing and associated hydrogen pick-up[8].

At CEBAF a phenomenon as described above had never been observed in the vertical testing of cavities and cavity pairs for--with our present knowledge--the following reasons: the chemical treatment of CEBAF's cavities was always done in a chemical solution producing very little hydrogen and the cooldown of the cavities from room temperature to 4.2 K always took place in a short period. In cavity tests in the horizontal cryostat, some low Q values were observed and the connection to possible degradations caused by slower cooldown rates[9] was made later.

This paper describes a series of experiments in which the Q degradation was first verified under certain experimental conditions and secondly it was attempted to find a possible cure for the increased losses in the cavity walls by anodic oxidation. A procedure like this, if successful, could be easily applied to complex cavity systems, which could not be subjected to heat treatment because of mechanical restrictions.

### CAVITY SURFACE PREPARATIONS

For the investigations reported here, single and 5-cell 1500 MHz cavities with elliptical cross sections[10] were used. One cavity was manufactured from reactor grade niobium with a RRR value of  $\sim 40$ ; all other cavities used were fabricated from high purity niobium with RRR values  $\geq 250$ .

All cavities were chemically treated in a standard way prior to testing:

#### A. Buffered Chemical Polishing

After initial degreasing in a caustic solution under ultrasonic agitation, the cavities were chemically polished in a buffered 1:1:1 solution of hydrofluoric (49%), nitric (65%), and phosphoric (85%) acids at room temperature for several minutes resulting in the removal of  $\sim 60 \mu\text{m}$  of niobium from the surface.

After thorough rinsing in ultrapure water and ultrasonic agitation for up to 1 hour with several exchanges of the rinse water, the cavities were rinsed with reagent grade methanol and subsequently a variable RF input coupling system and a transmission RF probe were assembled to the cavities in a Class 100 cleanroom. The cavities were attached to the cryogenic test stand using extreme caution to avoid contamination of the cavity surfaces.

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## B. Anodizing

Under the hypothesis that the Q degradations are caused by the precipitation of the  $\epsilon$ -phase of niobium hydride, and with the knowledge that such severe degradations have not been observed in the early days of the reactor grade niobium, it seemed to be worthwhile to investigate whether or not additional oxygen introduced into the surface layer of the niobium by anodizing would act as a "trap" for the hydrogen by forming OH bonds and, in this way, reduce the amount of precipitation, especially since the hydrogen in niobium seemed to be concentrated in a thin surface layer.

The cavity surfaces to be anodized were renewed by removing  $\sim 3 - 4 \mu\text{m}$  of niobium by chemical polishing as described above, rinsed in ultrapure water and subsequently subjected to a 15% solution of Ammonium hydroxide at a maximum voltage of 100 V and a current density of  $\sim 0.8 \text{ mA/cm}^2$ , which decayed in  $\sim 10$  min to 1/10 of its value. The resulting amorphous  $\text{Nb}_2\text{O}_5$  layer is purple in color and has a thickness of  $\sim 2000 \text{ \AA}$  ( $20 \text{ \AA/V}$ ). In one case, the oxide layer was only grown to a thickness of  $1300 \text{ \AA}$ .

Rinsing in ultrapure water and methanol as described above followed prior to the assembly in the cleanroom.

## CAVITY TESTING AND TEST RESULTS

Prior to cooling down the cavities to helium temperature, they were evacuated to a vacuum of  $< 8 \times 10^{-7}$  Torr. The standard cooldown from room temperature to 4.2 K took less than 1 hour. The external magnetic field during cooldown was shielded to  $< 10$  mG by two layers of  $\mu$ -metal wrapped around the cryostat and an active compensation coil system.

The cavity tests consisted of measuring a) the temperature dependence of the surface resistance at low RF field of 1 - 2 MV/m in the cavity between 4.2 K and 1.7 K; the temperature of the cavity was measure by two Si diodes attached to the cavity and b) the dependence of the Q value on the peak fields in the cavity.

For the experiments, during which the cavities were held at intermediate temperatures, the cavity temperature was monitored through the two Si diodes attached to the outside of the cavities. The natural warmup from 50 K to 100 K after a cold test took place in our cryostat in  $\sim 20$  hrs.

For the analysis of the temperature dependence of the surface resistance, we compared the experimental data to the BCS theory as supplied by Halbritter's program[11]. With the experimentally obtained value for the gap parameter, the measured data were fitted to theoretical calculations with the

mean free path in the penetration depth as a fit parameter. Other material parameters such as critical temperature, London penetration depth and coherence length were not varied and we used the values of 9.25 K,  $360 \text{ \AA}$  and  $640 \text{ \AA}$ , respectively [12].

Fig. 1 shows an example of the temperature dependence of  $R(T) - R_{\text{res}}$  ( $R_{\text{res}}$  = residual surface resistance) for a "bare" and an anodized niobium surface after warmup to an intermediate temperature of 100 K for  $\sim 20$  hrs.

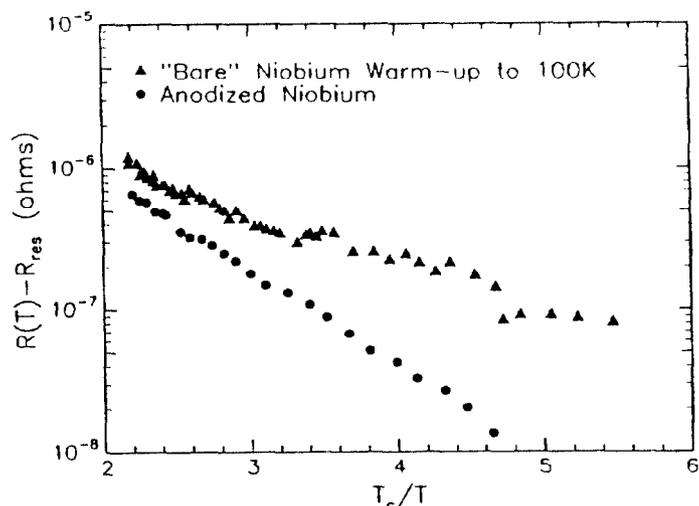


Fig. 1:  $R(T) - R_{\text{res}}$  for "bare" and anodized cavity after warmup to 100 K for  $> 16$  hrs

Fig. 2 and Fig. 3 are examples of the dependence of the Q value at 1.7 K on the RF field in the cavity for a "bare" and an anodized cavity before and after warmup to an intermediate temperature of 100 K for  $\sim 20$  hrs.

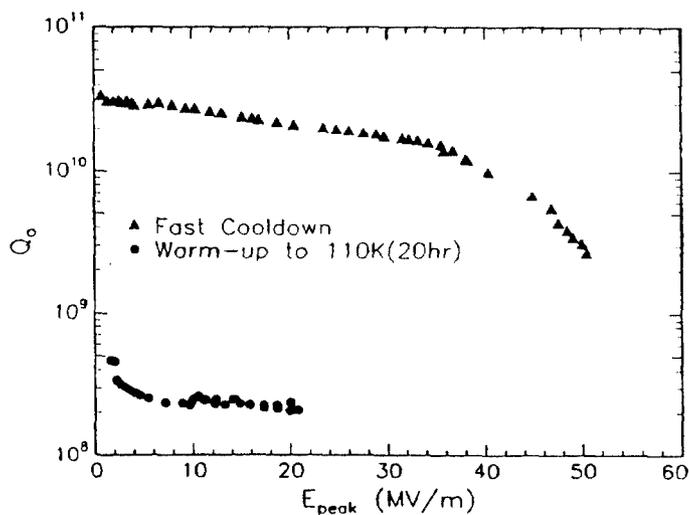


Fig. 2:  $Q_0$  vs  $E_{\text{peak}}$  for "bare" niobium cavity

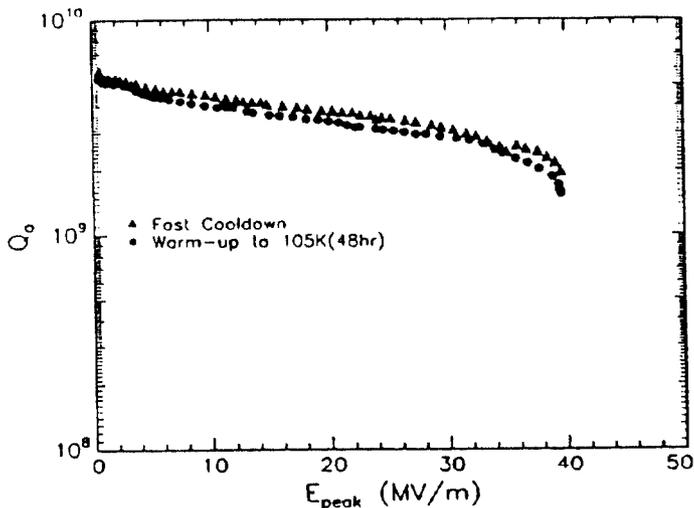


Fig 3:  $Q_0$  vs  $E_{\text{peak}}$  for anodized niobium cavity

### DISCUSSION

The results of our investigations can be summarized as follows.

- A). A cavity fabricated from reactor grade niobium did not show any degradation in  $Q$  value due to warming up to intermediate temperatures  $\leq 100$  K.
- B). Cavities made from high purity niobium showed severe  $Q$  deterioration when warmed up to  $\leq 100$  K and kept for  $> 16$  hrs. between 50 K and 100 K. A warmup to 170 K and maintaining this temperature for 16.5 hrs. did not result in  $Q$  degradation.
- C). A  $Q$  degradation can be fully recovered by warmup to room temperature.
- D). Anodic oxide layers reduce the amount of  $Q$  degradations and seem to be more effective at a thickness of 2000 Å than at 1300 Å. Whereas for "bare" surfaces, a degradation  $> 10$  times was observed, anodized surfaces showed degradations  $< 50\%$ .
- E). The temperature dependence of the surface resistance of the degraded surfaces cannot be expressed by a single superconductor in the measured temperature range between 4.2 K and 1.9 K. Multiple phases are generated within the penetration depth by the slow warmup. In the temperature range  $2.9 \text{ K} < T < 2.5 \text{ K}$ , the deviation from the BCS behavior is most pronounced. A similar behavior has been reported in Ref. [5]. Between 4.2 K and 2.9 K, the experimental data can be fitted only with a reduced critical temperature. The mean free path is also reduced in comparison to a "virgin" surface.

F). Anodized surfaces, which have been subjected to the same warmup procedures, do not show the multiphase behavior in the temperature dependence of the surface resistance. The mean free path around the interface between anodized film and the niobium is increased by about a factor of 2 in comparison to a "virgin" surface. This fact suggests that the additional oxygen introduced by anodizing acts as a "trap" for the hydrogen and prevents the hydrogen from precipitating into niobium hydride at a slow cooldown rate or warmup to intermediate temperatures.

### CONCLUSION

Our experiments provide some evidence that anodic oxide layers on niobium surfaces of high purity niobium are capable of significantly reducing  $Q$  degradations due to precipitation of the niobium hydride phase and thicker  $\text{Nb}_2\text{O}_5$  layers seem to be more effective. This observation, coupled with the fact that hydrogen preferentially is located in a thin surface layer[13], suggests that by diffusing oxygen into the surface by moderate "in situ" heating of an anodized cavity might further reduce the danger of  $Q$  degradations.

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