# Enhanced susceptibility of Nb cavity equator welds to the hydrogen related $Q$-virus*

J. Knobloch and H. Padamsee
Floyd R. Newman Laboratory of Nuclear Studies
Cornell University, Ithaca, New York 14853

**Abstract**

Thermometric studies of chemically treated niobium cavities have demonstrated that preferential niobium-hydride precipitation takes place along the electron-beam-welded equator ("$Q$-virus"). High residual losses are recorded as a result of the hydride in this region. Such precipitation can take place even when the cavity is rapidly cooled from 300 K to 4.2 K, a procedure that is known to be effective at preventing the $Q$-virus in bulk niobium.

## 1 Introduction

Niobium cavities are subjected to extensive cleaning procedures prior to assembly in the cryostat to avoid, among others, field emission and thermal breakdown. Included in these steps is chemical polishing with a mixture of nitric, phosphoric, and hydrofluoric acid (BCP). [1] Other laboratories use electropolishing as an alternative.

Studies [2–4] have shown that extensive chemical and electro-chemical polishing at temperatures exceeding 15 °C can result in a drastic reduction of the cavity quality ($Q_0$) at low field and a further drop as $E_{\mathrm{pk}}$ is raised. Due to historical reasons, this situation has been termed the "$Q$-virus" or the "$Q$-disease." The $Q_0$ drop is exacerbated if the cavity is "parked" during cooldown in a temperature range between 60 and 150 K for a period of one hour or more. $Q_0$ reductions by more than two orders of magnitude have been observed. High purity niobium (with a RRR[1] exceeding 100) is known to be especially sensitive to these effects.

The accepted mechanism for the $Q$-degradation is hydride formation. Depth profiles of chemically etched niobium have shown that up to 5 at. % (0.054 wt. %) of hydrogen is stored in the niobium bulk near the surface during the etch. [3] The longer the etch and the warmer the acid, the more hydrogen is absorbed. In addition, typically 1 wt. ppm of hydrogen is already present in delivered commercial niobium. A study of niobium's phase diagram [5] reveals that niobium hydride precipitates and undergoes several phase transitions between 220 and 130 K. It is believed that these hydride phases have a lower critical temperature and critical field and a higher surface resistance ($R_{\mathrm{s}}$) than niobium. Significant quantities of hydride are formed if the original hydrogen concentration exceeds 2 wt. ppm. However, between 100 K and 200 K the formation time for large areas of hydride phase is long due to the slow diffusion of hydrogen, so that a rapid cooldown through this temperature range prevents the manifestation of the $Q$-disease.

Correspondingly, one frequently (but not always) finds that the original $Q_0$ of an afflicted cavity can be recovered by thermally cycling it to room temperature and cooling down rapidly to 4.2 K. The danger zone between 150 K and 60 K should be crossed in less than about $1/2 - 1$ hour to avoid renewed $Q$-degradation.

The $Q$-disease can be avoided altogether by vacuum baking the cavity at temperatures exceeding 900 °C for several hours. The high temperature drives out most of the hydrogen from the niobium bulk. Following the furnace treatment, an etch is required to clean the surface once again, but provided it is short and the acid temperature is kept below 15 °C the $Q$-disease does not re-establish itself.

Similarly, it is believed that low RRR cavities are not afflicted by the $Q$-disease because defects and impurities trap the hydrogen [3, 6] and prevent hydride precipitation.

However, contrary to previous experience, we have now found during the course of thermometric studies of 1.5-GHz (L-band) cavities [7] that the $Q$-disease may manifest itself to some extent even if all etching is

*Supported by the National Science Foundation with supplementary support under the US–Japan Agreement.
[1]RRR = residual resistance ratio.

performed at acid temperatures below the established 15 °C cutoff. Our results suggest that the electron-beam weld along the equator is especially susceptible to hydride precipitation. Enhanced losses in this region, we will show, cannot always be eliminated completely by a rapid cooldown.

## 2 Experimental setup

The experimental setup to study 1.5-GHz niobium cavities has been described in a number of papers [7–9] and will not be repeated here in detail. The main diagnostic tool is a fixed-array thermometry system comprising 756 thermometers attached to the cavity exterior. The array is capable of mapping the cavity temperature distribution in superfluid helium at 1.6 K. Particularly important to the study of residual loss mechanisms in cavities, such as the $Q$-virus, is the system's high resolution (as good as 30 $\mu$K), allowing us to resolve a surface resistance down to the nanohm scale. An automated $Q_0$ versus ${E_{\mathrm{pk}}}^2$ measurement system [7] operates in conjunction with the thermometry system, to provide information on the integrated cavity losses.

Single-cell cavities of the CEBAF shape [10] were made with RRR = 250 niobium using the deep-drawing process to make two half cells. These were joined with an electron beam weld along the equator. [11] Another set of welds was used to attach beam tubes to the cavity irises.

The cavity preparation prior to testing consisted of a standard chemical treatment. [1, 7] First the cavity is immersed in nitric acid to remove any indium remaining from the previous test. The cavity is then etched with BCP 1:1:2[3], for about five minutes.[4] Care is taken that the acid temperature never exceeds 15 °C, to minimize the hydrogen uptake by the niobium.[5] A rinse with deionized water for one hour followed the chemical etch, before drying the cavity with hot, filtered nitrogen gas and mounting the cavity on the test stand.

Precooling of the cryostat with liquid nitrogen was *not* performed, to minimize the danger of hydride precipitation. Instead the cavities were cooled very rapidly with liquid helium, with average cooldown rates from 300 K to 4.2 K as high as 5 – 10 K/min. Past experience has demonstrated that such cooldown rates prevent hydride precipitation.

## 3 Hydride precipitation at cavity the equator

### 3.1 Mild case

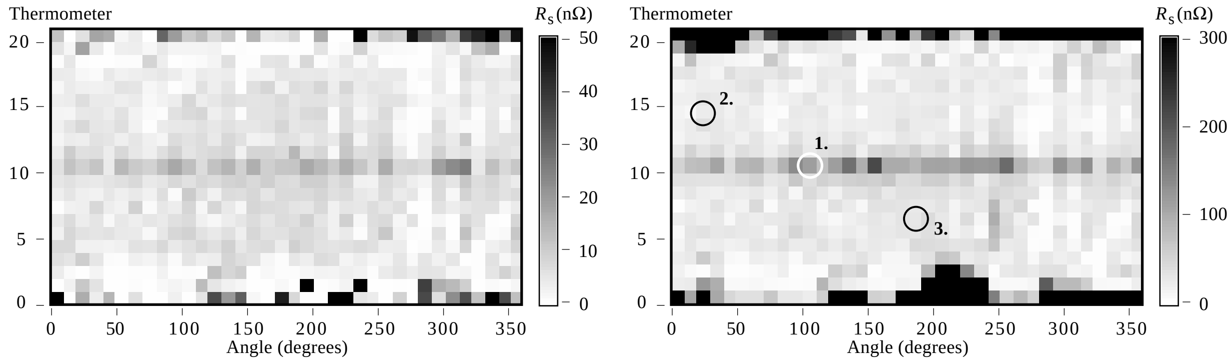
Altogether, 11 different cavities were tested with the thermometry system. All these cavities had $Q_0$'s in excess of $10^{10}$ and were essentially free of the severe $Q$-virus that is sometimes reported in the literature. However, in four cases we noticed a band of slightly elevated losses along the cavity equator. At low fields this effect was generally not very pronounced, but the equator losses increased more rapidly with $E_{\mathrm{pk}}$ than the surface resistance elsewhere, resulting in a very visible lossy band at high fields. An example of these observations is shown in Figure 1. Figure 2 illustrates the rapid increase of the equator surface resistance by taking the ratio of the site marked "1" in Figure 1(b) to that marked "3", a random place in the cavity. For comparison, the same ratio is shown for the site marked "2." In both cases the ratios are normalized to one at low field. Ratios are plotted, rather than true $R_{\mathrm{s}}$ values, so that global, non-quadratic losses due to, for example, x-ray bombardment are canceled out. The rapid increase of $R_{\mathrm{s}}$ along the equator is very apparent. At 30 MV/m, the mean surface resistance of the equator is 99 n$\Omega$, almost 5 times the resistance measured away from the equator (20 n$\Omega$). The increased losses are responsible for a 20 % drop in the $Q_0$.

Another cavity (LE1-21) had received a 900 °C heat treatment to remove all hydrogen, and it did *not* display anomalous equator losses (Figure 3). Hence the results in Figure 1 are unlikely to be due to a measurement artifact. Instead, we suspect that the equator is susceptible to hydride precipitation, despite the fact that we always cool cavities rapidly at rates equal to or greater than 5 – 10 K/min.

### 3.2 Severe case

We obtained confirmation of our hypothesis when we tested cavity LE1-34. In total, this cavity had received a 100 $\mu$m etch (inside and outside) with BCP 1:1:2. At no time did the acid temperature exceed 14.5 °C. During the very first test, the cavity did *not* have a resistive band along the equator, even at the highest fields (Figure 4(a)). The cavity was then permitted to warm to 77 K $< T <$ 273 K without the cryostat being moved.

[2] $E_{\mathrm{pk}}$ is the peak surface electric field in the cavity.
[3] One part nitric acid, one part hydrofluoric acid, and two parts phosphoric acid.
[4] Newly manufactured cavities are etched for about 60 minutes to remove nearly 100 $\mu$m of niobium.
[5] In many cases the temperature was maintained close to 10 °C.




(a) (b)

Figure 1: Surface resistance of cavity LE1-20 at (a) $E_{\rm pk} = 15$ MV/m and (b) 30 MV/m. The losses along the equator (thermometer 10) increase faster with $E_{\rm pk}$ than elsewhere, typical of hydrogen related losses. Dark regions along the irises (thermometers 1 and 19) are either field emission related, or due to noisy signals because the magnetic field is too low for reliable $R_{\rm s}$ extraction from the temperature data.

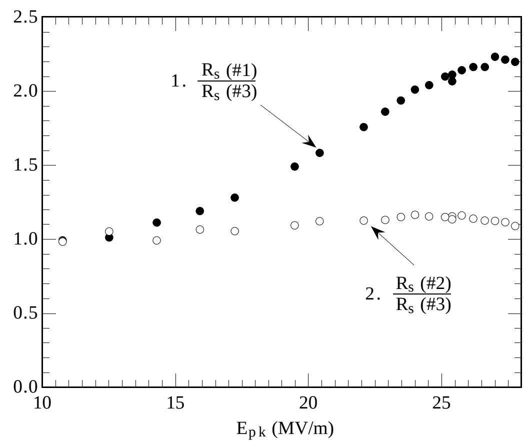
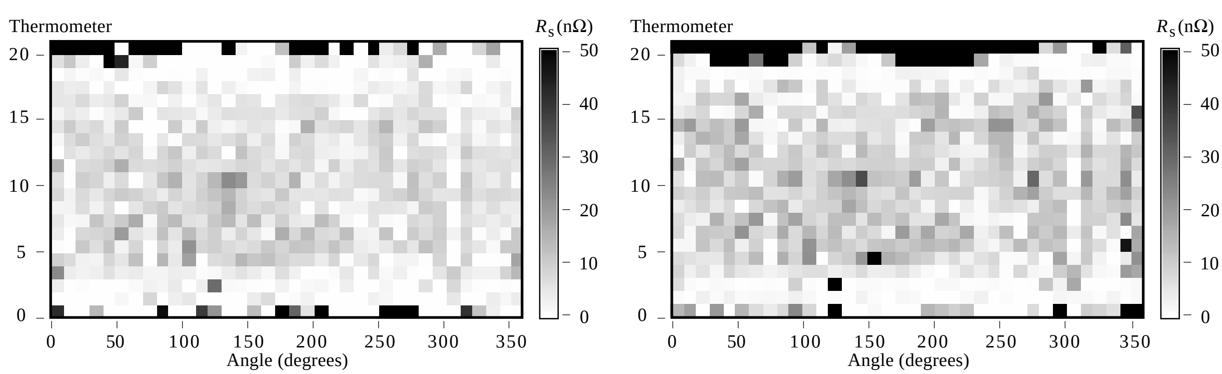
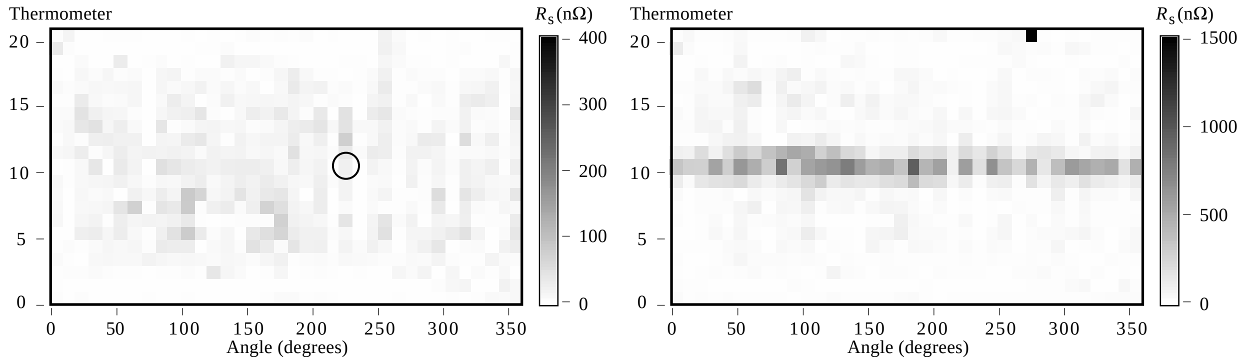



Figure 2: Ratio of the surface resistance measured by (a) thermometer "1" in Figure 1 to that measured by thermometer "3", and (b) thermometer "2" to that measured by thermometer "3". Both curves are normalized to one at low field.



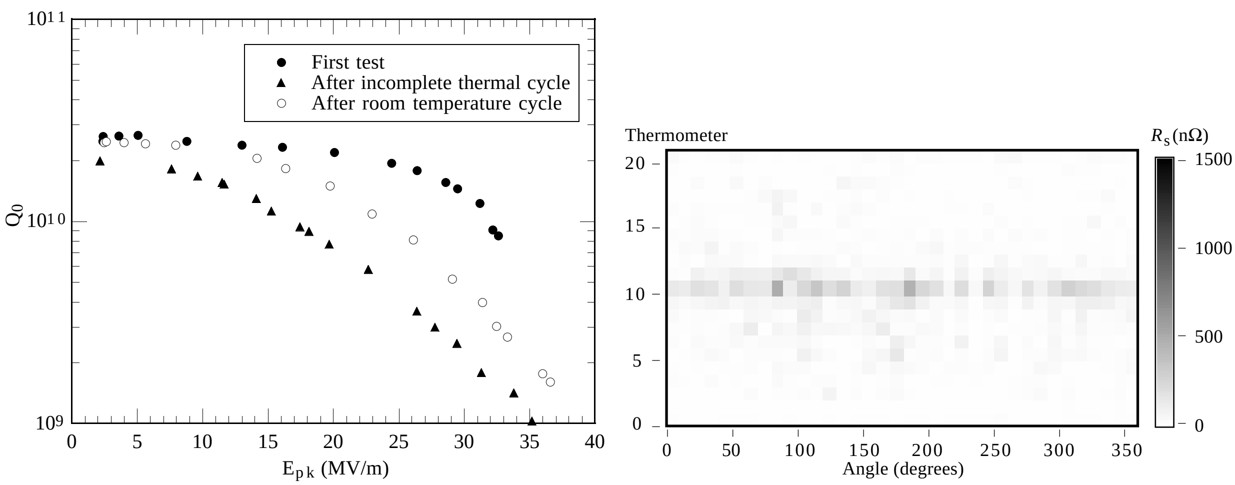

(a) (b)

Figure 3: Surface resistance of cavity LE1-21 following a 900 °C heat treatment to remove all hydrogen. (a) At $E_{\rm pk} = 10$ MV/m, and (b) at 27 MV/m. No enhanced losses were observed along the equator. Dark regions along the irises are either field emission related, or due to noisy signals because the magnetic field is too low for reliable $R_{\rm s}$ extraction.




(a) (b)

Figure 4: Surface resistance of cavity LE1-34 at 30 MV/m. (a) First test, (b) second test following a temperature cycle to 77 K $< T <$ 273 K. The losses recorded at the circled site are summarized in Figure 6.




(a) (b)

Figure 5: (a) $Q_0$ results obtained with cavity LE1-34. In all cases, x rays were only detected at $E_{\rm pk} = 20$ MV/m or higher. (b) Surface resistance of cavity LE1-34 at 30 MV/m, following an incomplete thermal cycle and then a complete cycle to room temperature.

The exact temperature is not known, but we suspect it was much lower than 273 K because there was significant ice build-up on the top of the cryostat. At that point, liquid helium was retransferred for another test of the cavity.

A band of very lossy material then appeared along the equator, both at low fields and even more so at high fields (see Figure 4(b)). Correspondingly, the cavity $Q_0$ had degraded by 11 % at the lowest field ($E_{\rm pk} =$ 2.3 MV/m) and by a factor of 1/2 at 15.3 MV/m. As the fields were increased the $Q_0$ dropped very rapidly despite the fact that no new field emitters were active when compared to the previous test. The first x rays were not detected until 20 MV/m by which time the $Q_0$ was only $7 \times 10^9$. At 30 MV/m the $Q_0$ was reduced by over a factor of six with respect to the previous test (Figure 5(a)).

To study the new losses, we warmed the cavity once more, this time all the way to room temperature. Helium was then retransferred to the cavity as quickly as possible (it took only 25 minutes to cool the cavity to 4.2 K). The low field $Q_0$ recovered completely. At high fields, though, a $Q_0$ reduction was still observed, albeit to a lesser extent than after the incomplete thermal cycle (Figure 5(a)). Not surprisingly we found that the losses along the equator, although significant, were not as severe as before (see Figure 5(b)).

The losses observed by the thermometer circled in Figure 4(a) are summarized in Figure 6. Note the semi-logarithmic scale. The dramatic increase in losses following the incomplete thermal cycle is very apparent. The mean $R_s$ recorded by all the equator thermometers during the first test was 28 nΩ at 30 MV/m, which

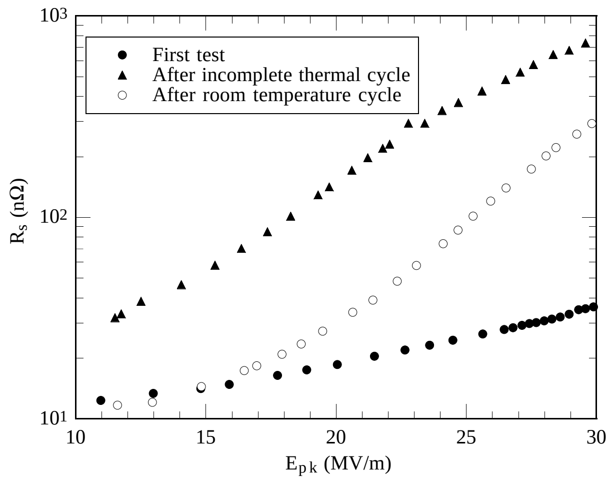



Figure 6: Surface resistance recorded at the circled site in Figure 4(a) during three different tests. The slight increase in $R_\mathrm{s}$ with $E_\mathrm{pk}$ during the first test was observed throughout the entire cavity and is probably not related to hydrogen contamination.

is consistent with a measured cavity $Q_0$ of over $10^{10}$. Following the first temperature cycle, the mean equator resistance increased to a staggering 672 nΩ! If the entire cavity had increased its losses to this value, a $Q_0$ of only $4 \times 10^8$ could be expected. However, since only the equator was afflicted a $Q_0$ of $2 \times 10^9$ was recorded. A significant improvement following the complete temperature cycle and subsequent fast cooldown was registered. At low field, the original $R_\mathrm{s}$ was recovered but the losses increased with $E_\mathrm{pk}$, reaching a mean value of 278 nΩ at 30 MV/m. This value is still a factor of 10 times worse than the original losses, and the $Q_0$ at this point was down by more than a factor of two from the first test.

# 4 Discussion

The results obtained with cavity LE1-34 point to an affliction by the $Q$-disease. The evidence is, that the equator is more susceptible to hydrogen precipitation than the remainder of the cavity. Since the material properties of the weld are different than those of the rest of the cavity, preferential hydride precipitation along the equator is not necessarily surprising. The grain sizes of the weld are quite large (several millimeters), and the impurity concentrations may also differ from the bulk. Past experience has shown that niobium with fewer interstitial impurities (i.e., a higher RRR) is far more susceptible to the $Q$-virus than low RRR material. [4] The interstitial impurities serve as trapping centers for hydrogen, thereby preventing hydride precipitation. Similarly, vacancies and grain boundaries are also very effective at trapping hydrogen by forming Cottrell clouds. [6, 12–14] It is possible that the welds, because of their large grains, are more susceptible to the $Q$-virus due to the lack of trapping centers that otherwise prevent hydride precipitation.[6] For confirmation of this assumption, though, a detailed analysis of the weld's material properties is required.

Although a large fraction of cavity LE1-34 was not seriously affected by the hydride precipitation, the losses along the equator were so severe that they lowered the $Q_0$ substantially. Even a very rapid cooldown, faster than 10 K/min, did not "cure" the cavity completely. In an accelerator, cooldown rates of 10 K/min are not permitted due to the dangers of stress induced component failures. A "cure" of $Q$-virus afflicted cavities thus requires a costly disassembly, heat treatment, and reassembly.

Cavity LE1-34 was an extreme case. Nevertheless, augmented losses along the equator were observed in other cavities as well. In cavity LE1-20 they accounted for a 20 % drop in $Q_0$. For the highest $Q_0$ applications it would be advantageous, if welds could be eliminated in high magnetic field regions, to avoid the danger of hydride enhanced losses. Techniques such as hydroforming for cavity manufacturing rather than deep-drawing should be considered.

Our results also point to the dangers of repeated temperature cycles of cavities. Hydride precipitation may not occur after the first cooldown, but a second cooldown can cause problems. This effect was also observed with cavities at DESY. [15]

[6]Note that the iris welds do not cause operational problems, since they are in a low magnetic field region, where the $Q$-virus is not apparent.

# References


[1] P. Kneisel, Surface preparation of niobium, in *Proceedings of the Workshop on RF Superconductivity*, edited by M. Kuntze, pages 27–40, Karlsruhe, 1980, Proceedings also published as internal Kernforschungszentrum Kalsruhe report KFK-3019.

[2] B. Aune et al., Degradation of niobium superconducting rf cavities during cooling times, in *Proceedings of the 1990 Linac Conference*, pages 253–255, Albuquerque, NM, 1990.

[3] C. Z. Antoine et al., The role of atomic hydrogen in $Q$-degradation of niobium superconducting rf cavities: analytical point of view, in *Proceedings of the 5$^{th}$ Workshop on RF Superconductivity*, edited by D. Proch, pages 616–634, Hamburg, Germany, 1993, Proceedings also published as internal DESY report DESY M-92-01.

[4] B. Bonin and R. W. Röth, $Q$ degradation of niobium cavities due to hydrogen contamination, in *Proceedings of the 5$^{th}$ Workshop on RF Superconductivity*, edited by D. Proch, pages 210–244, Hamburg, Germany, 1991, Proceedings also published as internal DESY report DESY M-92-01.

[5] J. F. Smith, *Bulletin of Alloy Phase Diagrams* **4**, 39 (1983).

[6] J. K. Nørskov and F. Besenbacher, *Journal of the Less-Common Metals* **130**, 475 (1987).

[7] J. Knobloch, *Advanced Thermometry Studies of Superconducting RF Cavities*, PhD thesis, Cornell University, 1997, Laboratory of Nuclear Studies thesis CLNS 97-3.

[8] J. Knobloch, H. Muller, and H. Padamsee, *Review of Scientific Instruments* **65**, 3521 (1994).

[9] J. Knobloch and H. Padamsee, *Particle Accelerators* **53**, 53 (1996), Also published in the Proceedings of the 7$^{\text{th}}$ Workshop on RF Superconductivity, Gif-sur-Yvette, France, pp. 95–103 (1995).

[10] J. Knobloch and H. Padamsee, Multipacting in 1.5-GHz superconducting niobium cavities of the CEBAF shape, in *Proceedings of the 8$^{th}$ Workshop on RF Superconductivity*, Padua, Italy, 1997.

[11] H. Padamsee, J. Knobloch, and T. Hays, *RF Superconductivity for Accelerators*, Wiley and Sons, New York, 1998, To be published.

[12] J. A. Rodriguez, *Scripta Metallurgica* **17**, 159 (1983).

[13] G. V. Khaldeev and V. K. Gogel, *Russian Chemical Reviews* **56**, 1057 (1987).

[14] K. W. Jacobsen, *Comments on Condensed Matter Physics* **15**, 129 (1988).

[15] G. Enderlein, W. Körber, A. Matheisen, and D. Proch, Investigations on hydrogen contamination of superconducting cavities, in *Proceedings of the 1991 IEEE Particle Accelerator Conference*, pages 2432–2434, San Francisco, CA, 1991.