

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

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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_{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 RRR¹ 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_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

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¹RRR = residual resistance ratio.

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 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 μ K), allowing us to resolve a surface resistance down to the nanohm scale. An automated Q_0 versus E_{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 1:1:2³, 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

² E_{pk} is the peak surface electric field in the cavity.

³One part nitric acid, one part hydrofluoric acid, and two parts phosphoric acid.

⁴Newly manufactured cavities are etched for about 60 minutes to remove nearly 100 μ m of niobium.

⁵In many cases the temperature was maintained close to 10 °C.

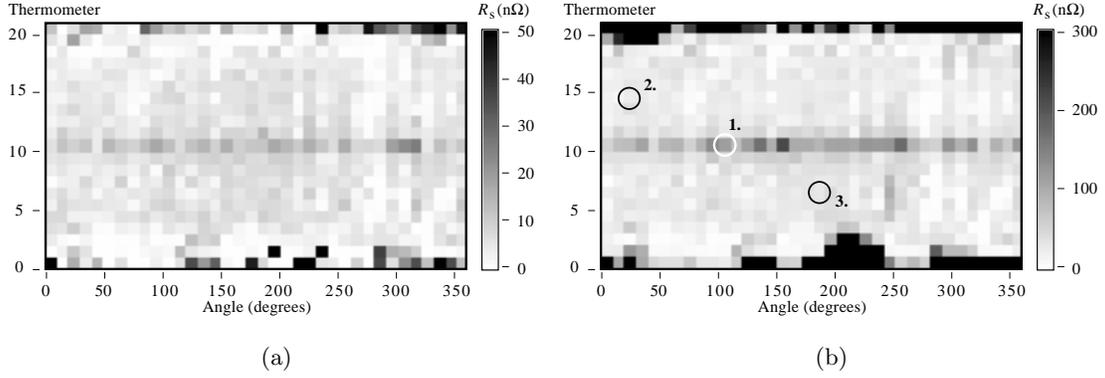


Figure 1: Surface resistance of cavity LE1-20 at (a) $E_{pk} = 15$ MV/m and (b) 30 MV/m. The losses along the equator (thermometer 10) increase faster with E_{pk} than elsewhere, typical of hydrogen related losses. Dark regions along the irises (thermometers 1 and 19) are either field emission related,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

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_{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_s values, so that global, non-quadratic losses due to, for example, x-ray bombardment are canceled out. The rapid increase of R_s along the equator is very apparent. At 30 MV/m, the mean surface resistance of the equator is 99 nΩ, almost 5 times the resistance measured away from the equator (20 nΩ). 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.

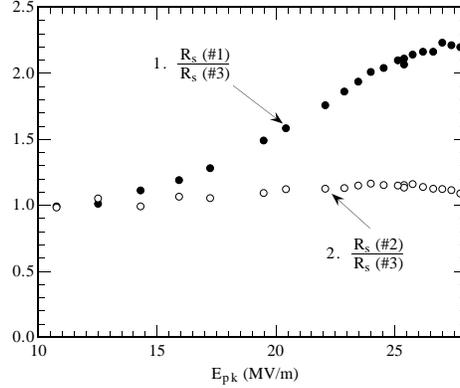


Figure 2: Ratio of the surface resistance measured by (a) thermometer “1” in Figure 1 to that measured by thermometer “3”, and (b) thermometer “2” to that measured by thermometer “3”. Both curves are normalized to one at low field.

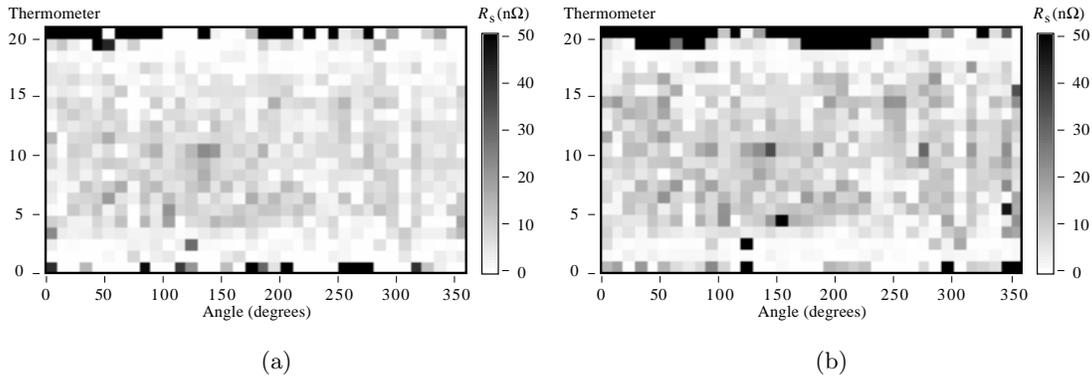


Figure 3: Surface resistance of cavity LE1-21 following a 900 °C heat treatment to remove all hydrogen. (a) At $E_{pk} = 10$ MV/m, and (b) at 27 MV/m. No enhanced losses were observed along the equator. Dark regions along the irises are either field emission related, or due to noisy signals because the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

3.2 Severe case

We obtained confirmation of our hypothesis when we tested cavity LE1-34. In total, this cavity had received a 100 μm 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 $77\text{ K} < T < 273\text{ K}$ without the cryostat being moved. 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 ($E_{pk} = 2.3$ 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

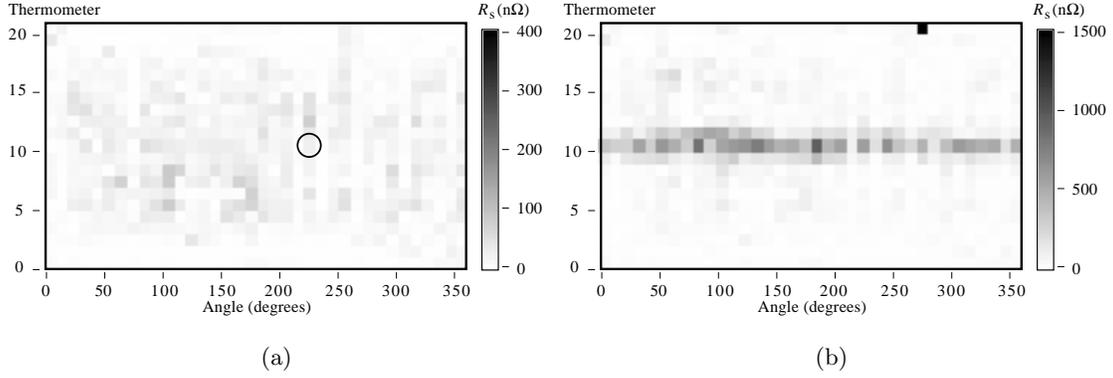


Figure 4: Surface resistance of cavity LE1-34 at 30 MV/m. (a) First test, (b) second test following a temperature cycle to $77\text{ K} < T < 273\text{ K}$. The losses recorded at the circled site are summarized in Figure 6.

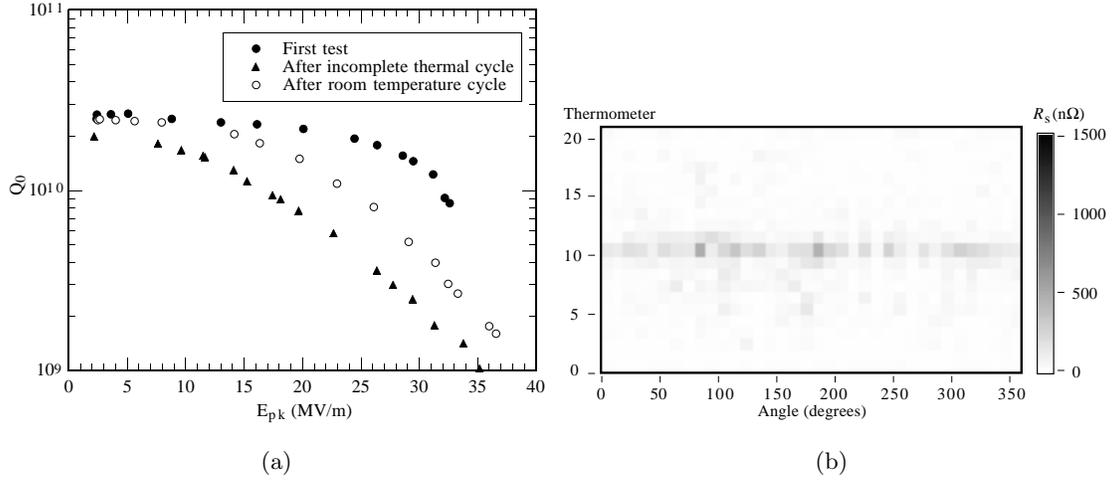


Figure 5: (a) Q_0 results obtained with cavity LE1-34. In all cases, x rays were only detected at $E_{pk} = 20\text{ MV/m}$ or higher. (b) Surface resistance of cavity LE1-34 at 30 MV/m, following an incomplete thermal cycle and then a complete cycle to room temperature.

until 20 MV/m by which time the Q_0 was only 7×10^9 . 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 n Ω at 30 MV/m, which is consistent with a measured cavity Q_0 of over 10^{10} . Following the first temperature cycle, the mean equator resistance

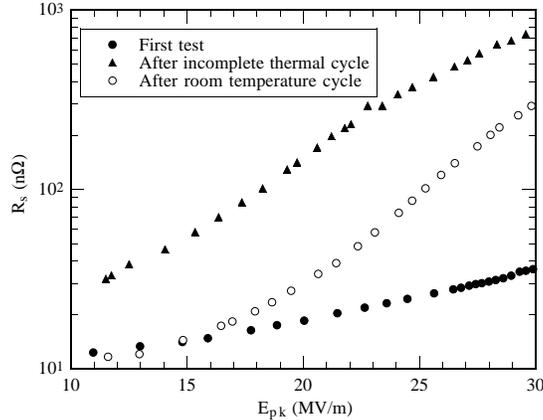


Figure 6: Surface resistance recorded at the circled site in Figure 4(a) during three different tests. The slight increase in R_s with E_{pk} during the first test was observed throughout the entire cavity and is probably not related to hydrogen contamination.

increased to a staggering 672 nΩ! If the entire cavity had increased its losses to this value, a Q_0 of only 4×10^8 could be expected. However, since only the equator was afflicted a Q_0 of 2×10^9 was recorded. A significant improvement following the complete temperature cycle and subsequent fast cooldown was registered. At low field, the original R_s was recovered but the losses increased with E_{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

⁶Note 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.

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]

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