HIGH TEMPERATURE HEAT TREATMENT OF 3GHz NIOBIUM CAVITIES*

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Abstract

We applied high temperature heat treatment to 3GHz cavities to determine the preparation of niobium cavities using the titanium solid-state gettering process. Some preliminary results showed peak surface electric fields of up to 65MV/m at a Q-factor of $2x10^{10}$. We evaluated improvement in the purity of the niobium by measuring the residual resistance ratio (RRR) at 10K. The purest niobium we prepared reached a RRR of 670. To study the potential application to large-scale accelerator structures at lower frequencies, we are testing the possibility of heat treating the cavity half-cells before welding them. Therefore, we investigated the influence of electron beam welding on the RRR of high-purity niobium. In this paper we present the results of the investigations.

Introduction

Single cell, 3GHz cavities are being used to investigate various techniques for preparing superconducting niobium accelerator structures. The performance of niobium cavities is mainly limited by field emission [1]. As the final step prior to cavity testing, two techniques are used in all laboratories: 1) A preparation by a heat treatment above T=1200°C or, 2) a chemical preparation with a final ultrapure water rinse.

It has been found that the high temperature heat treatment reduces the field emission and improves the performance of the cavities [2]. Heat treatment of niobium above T=1200°C must be done by using the titanium solid-state gettering [3] in order to avoid the contamination of niobium by the residual gases of the furnace. This process is also used for the post-purification of niobium structures. A reduction of the interstitial impurities results in a higher thermal conductivity of the material which reduces the probability of cavity quenches.

At Los Alamos the chemical preparation technique was successfully modified. In addition to the normal polishing by BCP(1:1:1) the cavities are pretreated for fifteen minutes in nitric acid [4].

This paper compares the heat treatment with the improved chemical preparation technique. We also prepared samples to evaluate the performance of titanium heat treatment in our furnace. A first result of a cavity built out of heat treated half-cells is presented in the last section of this paper.

Experimental Techniques

All cavities and samples were machined out of Wah Chang™ niobium with a RRR of 230±20.

All heat treatments were done at T=1375°C. We chose this temperature based on the mean diffusion length of the interstitial impurities in niobium, the vapor pressure of titanium, and the maximum obtainable temperature of our furnace. At T=1375°C the pressure in the cold zone was approximately $1*10^{-6}$ Torr. Heater elements and heat shields are built out of molybdenum sheet. The furnace is sealed with elastomer and pumped with a cryo pump.

To do the single-sided titanium heat treatment (SST) [5] the entire cavity was placed vertically in a niobium box; the inner surface of the box was covered with titanium sheet. Niobium caps were placed over the flanges of the cavity to avoid titanium contamination of the inner cavity surface. Before the SST the cavity was chemically prepared with the standard procedure (15min HNO_3 soak and 2min BCP(1:1:1) polishing). After the heat treatment the cavity was first rinsed with clean methanol before it was assembled in the clean room (class 10).

The double-sided titanium heat treatment (DST) of half-cells was done by placing the half-cells with some additional titanium sheets in the Nb-box so that both sides were coated with titanium during the heat treatment.

We heat treated niobium samples in the same way as the cavities so that we could evaluate the process of niobium purification in our furnace. Simulation of DST was done by placing a sample, surrounded by a titanium strip, in the niobium box. To simulate single-sided titanium heat treatment (SST) we placed the samples in the furnace so that only one side was coated with titanium. The other side was either exposed to oven vacuum (SSTov) or to the inside of the cavity (SSTcv).

The RRR is defined as the ratio between the normal conducting resistivity at T=300K and at T=4.2K. We measured the resistance of the samples just above the superconducting transition temperature, eliminates the need for a magnet to quench the superconductivity. The measurement was done in a Helium-Flow-Cryostat. The value for the RRR was extrapolated using the measurement and fit from Webb on a niobium sample of RRR=16500 [6]. The error of the RRR measurement was ±5%.

For the RRR measurement, we cut a piece (about 0.03in. x 0.063in. x 0.85in.) from the center of the original sample. The electrical connection was done by spot welding small platinum wires on the sample.

Results

Heat Treatment of Niobium Samples

We were unsuccessful in our first attempt to heat treat the niobium samples with titanium. The RRR of the samples were reduced from 230 to 190 for the DST and from 230 to 95 for the SST (other side exposed to the oven vacuum). A

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chemical analysis[1] of the C, N and O content of these samples showed an increase in the C content from 7±3 to 16±3 wt.-ppm because of the SST. There was no change in the O and N content. A residual gas analysis of the furnace vacuum indicated a raised peak at mass 28 caused by carbon monoxid and nitrogen. The mean diffusion length of carbon in niobium for our parameters (8 hours at T=1375°C) is 0.055in. (1.4mm) [7]. The presence of carbon monoxid can explain the decrease of the RRR. The carbon monoxid in the furnace may have been caused by the degasing of the molybdenum shields and heater elements [8] and the decomposition of the Buna O-rings[9], in use at this time.

For the next sample treatment some changes were made: Increased pumping was achieved by covering the entire inner surface of the Nb-box with a titanium sheet. Titanium has a good gettering ability for CO_2, CO, O and N at these temperatures [10]. We also replaced the Buna O-rings by Viton O-rings. These changes resulted in a reduction of the peak at mass 28 by more than a factor of two.

After these modifications we improved the RRR of the niobium. In addition to investigating the performance of SST and DST we also investigated the influence of the sample thickness and the duration of the DST. The results of these investigations are shown in Table 1.

Tabel 1:
RRR Results of heat treated samples

sample #	thickness [in.(mm)]	heat treatment	RRR
Ia	0.03 (0.8)	6 h DST	473
Ib	0.03 (0.8)	12 h DST	504
Ic	0.03 (0.8)	18 h DST	510
IIa	0.063 (1.6)	6 h DST	450
IIb	0.063 (1.6)	12 h DST	501
IIc	0.063 (1.6)	18 h DST	497
IIIa	0.125 (3.2)	6 h DST	264
IIIb	0.125 (3.2)	12 h DST	292
IIIc	0.125 (3.2)	18 h DST	346
A	0.02 (0.5)	8h DST	671
B	0.063 (1.6)	8h SST ov	182
C	0.063 (1.6)	8h SST ov	232
D	0.063 (1.6)	8h SST cv	276
E	0.063 (1.6)	8h SST cv	274

All results were obtained on different samples. With the samples of size I (0.03in.) and II (0.063in.) no further improvement was noted after 12 hours DST. The size III sample showed almost a linear increase in the RRR up to 18 hours. Considering the mean diffusion length of O (0.026in. (6.5mm) for 18h at T=1375°C) [7], the RRR improvement from 230 to 346 was surprisingly low. Further tests in order to investigate possible saturation effects are necessary. Sample #A does not agree with sample #Ia-Ib. The small difference in thickness should not affect the RRR improvement. A possible explanation could be that the impurities were not uniformly distributed.

The SSTov did not improve the RRR but compared with the results of the initial test, sample #B and #C demonstrate the improvement of the vacuum. On sample #C and #D (SSTov) the increase of the RRR from 230 to 275 indicates that the vacuum in the cavity was better than the furnace vacuum.

From these results we see that only the DST purifies the niobium. With SST the RRR can only be improved slightly. A further test of the time dependence of the SSTcv would be interesting.

Welding Samples

Investigations of the RRR of the weld were made on four samples (#W1-W4). For samples #W3 and #W4 we changed the parameters of the electron beam welding:

	#W1, #W2	#W3, #W4
vacuum :	$\leq 1*10^{-5}$ torr	$\leq 5*10^{-6}$ torr
cool down time before let up :	7 min	15 min
let up with	air	argon

Sample pair #W4 was DST prior to the welding which improved the RRR from 230 to 386. The RRR of these samples which were cut out of the center of the weld is:

#W1: RRR = 136
#W2: RRR = 142
#W3: RRR = 153
#W4: RRR = 140

In a chemical analysis of the welding samples, we could not detect any difference in the carbon and nitrogen content. For the oxygen content we measured:

untreated sample: 18±3 wt.-ppm
after DST: 12±3 wt.-ppm
after welding: 23±3 wt.-ppm

These results demonstrate that oxygen is the relevant impurity in our case. As expected we are able to remove some oxygen during the purification process. But during the electron beam welding even more oxygen diffuses in the weld region.

The decrease of the RRR in the welded region indicates that the heat-treated half-cells are not suitable for building accelerator structures. Further investigations to improve the welding process are required. Therefore a residual gas analysis of the weld chamber and the use of another pumping system (at the moment: oil diffusion pump) would be the first items to investigate.

Cavity Results

The first heat treatment on cavities was done with the same setup used for the first sample preparation, namely, two titanium strips inside the niobium box. Based on the sample measurements we know that the RRR decreased. Figure.1 shows the Q vs. E_{peak} curves for the first two cavities which were prepared by SST. These two cavities were picked out of ten cavities which had been chemically treated and previously tested. The ten cavities produced the distribution in Figure 2.

[1] The chemical analysis was done by LECO Corporation with a combustion method for C and an inert gas fusion for O and N.

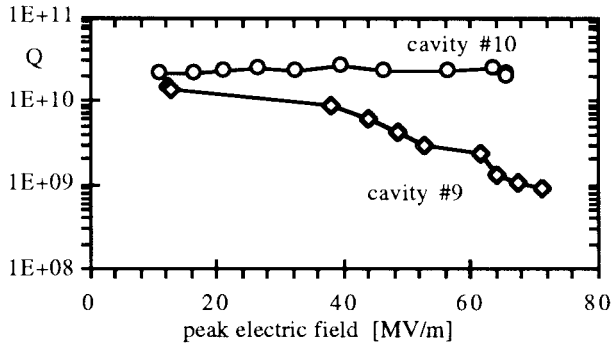

Figure 1: $Q(E_{peak})$ curve of cavity #9 and #10

After SST, cavity #10 did not show any field emission at all. The Q vs. E_{peak} curve showed a constant Q of $2*10^{10}$ up to E_{peak}=65MV/m (H_{max}=76mT). We could reproduce this behavior after an additional SST using only a nitric acid soak (no BCP) prior to repeating the SST. In this second test, the cavity had a lower Q ($Q=5*10^9$) because of titanium contamination in the cavity resulting from difficulties while assembling the cavity in the furnace. With cavity #9 we still had field emission after the SST which limited the field at E_{peak}=71MV/m. With all three heat treated cavities we reached high fields, at the upper part of the distribution for the chemically treated cavities (see fig 2).

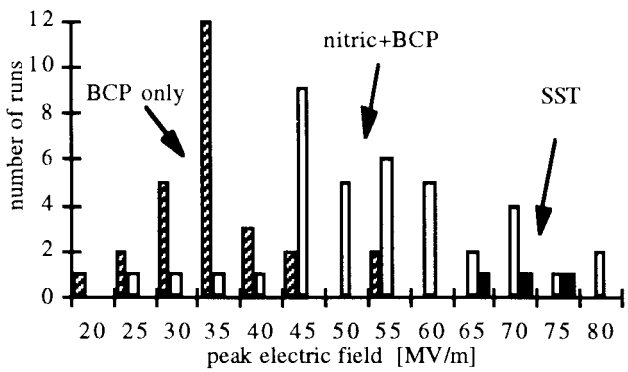

Figure 2: Cavity Result Distribution

It seems that the improvement achieved by the changing the chemical polishing procedure can be further improved by applying of SST, even if the RRR of the niobium was not improved in the tested cavities. More tests are necessary in order to obtain a distribution for the heat-treated cavities which can be compared in detail with the distribution of the chemically-treated cavities.

The first two cavities with heat treated half cells have been fabricated. After DST of the half-cells, the step joints for the welding were machined and the cavities were welded under the same conditions as welding samples #W3 and #W4. The first cavity was tested after a removal of 140µm by chemical polishing (BCP(1:1:1)). Figure 3 shows the obtained Q vs. E_{peak} curve.

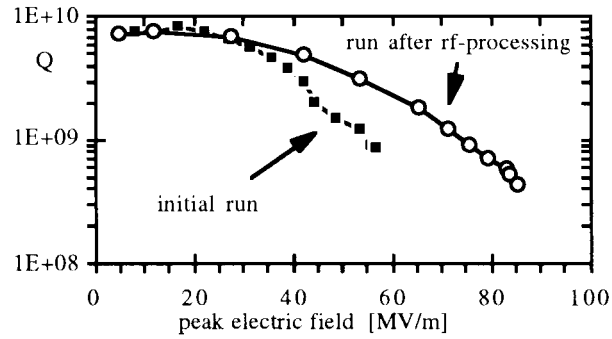

Figure 3: $Q(E_{peak})$ curve for cavity with DST on half cells

In the first power run the cavity went up to E_{peak}=57MV/m. After rf processing, in which we put about 70W in the cavity for ten minutes, the next power run reached E_{peak}=85MV/m.

As a result of this encouraging measurement we will try to investigate the efficiency of rf processing on cavities with a higher thermal conductivity.

Summary

Heat treated niobium samples show, that with ~10^{-6} Torr vacuum and a high partial pressure of CO and N in our furnace, we can purify niobium only by a double-sided titanium heat treatment. Results from single-sided titanium heat treated cavities achieved high gradients even after a reduction of the RRR during the process. This indicates that further improvements by heat treating the cavities after the chemical treatment can be obtained compared to the improved chemical preparation. The first cavity, where only the half-cells were heat treated, achieved an electrical peak field of 85MV/m.

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