INFLUENCE OF TA CONTENT IN HIGH PURITY NIOBIUM ON CAVITY* PERFORMANCE: PRELIMINARY RESULTS

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Abstract

In a previous paper [1] a program for reducing the costs of high purity niobium was outlined. This program was based on the fact that niobium prices could be reduced, if a higher content of Ta, which does not significantly affect the RRR–value, could be tolerated for high performance cavities. This contribution reports on the execution of this program and its present status.

Four ingots with different Ta contents have been melted and transformed into sheets. In each manufacturing step material quality has been monitored, using chemical analysis, thermal conductivity measurements and evaluation of mechanical properties. The niobium sheets have been scanned for defects by an eddy current device.

Two single cell cavities (CEBAF geometry) have been fabricated from each of three ingots, with Ta concentrations of 150, 600 and 1300 wtppm. A series of tests have been performed on each cavity with increasing amount of material removal.

This contribution reports on the test results and gives an analysis of the data.

INTRODUCTION

High purity niobium with RRR–values > 250 is exclusively used for the fabrication of high performance cavities for SRF accelerator projects such as SNS, TESLA, RIA or CEBAF Upgrade. Material cost is a significant fraction of total cavity cost, and one reason for the high costs is related to the specifications for tantalum content, which is in some cases required to be < 500 wtppm. Naturally occurring niobium–containing deposits such as columbite/tantalite or pyrochlore typically have high Ta content. Low Ta content in high purity niobium can only be obtained from niobium oxide, which appears as a byproduct of Ta production. Limited supply may be a big barrier to price stability and reliability for large projects such as TESLA.

The RRR–value (and therefore the thermal conductivity) of high purity niobium is mainly determined by interstitial impurities such as nitrogen, oxygen, hydrogen and carbon [2]. The contribution of Ta to the residual resistivity is significantly lower. It seems therefore quite reasonable to investigate the influence of Ta content in high purity niobium. However, one has also to make sure that the Ta is uniformly dispersed throughout the material, avoiding Ta clusters, which have in some cases been identified as causes of premature quenches in cavities. This is done by careful eddy current scanning of the sheets used for cavity fabrication [3].

In 2001 a project was launched to pursue this study with the following major objectives:

- Manufacturing of four niobium ingots with different Ta contents (< 150 wtppm,~ 600 wtppm, ~ 1200 wtppm and ~ 1300 wtppm)
- In these ingots only the Ta content was to be varied, content of other interstitial impurities was kept the same and verified by chemical analysis
- QA steps such as thermal conductivity measurements of ingots and rolled sheets and evaluation of mechanical properties of the rolled sheets accompanied production
- From the niobium sheets two single cell cavities each of the CEBAF variety were fabricated for Ta contents of < 150 ppm, ~ 600 ppm and 1300 ppm
- The rf performance of these cavities was measured at several stages during material removal from the surfaces

In the following the execution of this plan and the results from a total of 21 cavity tests are reported.

NIOBIUM SHEET MANUFACTURE

Ingot Production

The starting material for ingot production was pyrochlore as found at CBMM's deposit in Araxa, Brazil. Standard processing procedures [4] converted the ore to niobium ingots after triple electron beam melting of aluminothermically reduced niobium oxide. Addition of different amounts of Ta produced the four different ingots as mentioned above.

Material samples were taken from each ingot and the RRR-values were measured; the results are listed in Table 1.

Sheet Production

The ingots were converted into 2.8 mm thick sheets at the Tokyo Denkai facility in Japan in accordance with their standard processing procedures [5]. The RRR-values

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of sheets with different Ta content were measured on samples before and after a post-purification vacuum heat treatment at 1400 C for 3 hrs (Table 1).

Table 1: Residual Resistivity Ratios for different Ta contents for ingot and sheet

Ta content Sheet #	RRR Ingot	RRR sheet	RRR sheet heated
150 ppm 1161	180	178	323
~600 ppm 1162	170	231	345
~1200 ppm 1163	150	177	266
1300 ppm 1164	150	168	240

As expected, the post – purification heat treatment increased RRR, however, because of the relatively low interstitial impurity content, the improvement remained modest.

Mechanical Properties

Stress-strain curves for the different materials are shown in Figure 1. The strain rate was chosen as $2.2x10^{-5}$ /sec, which gives good resolution in the "Hooke's Law" region.

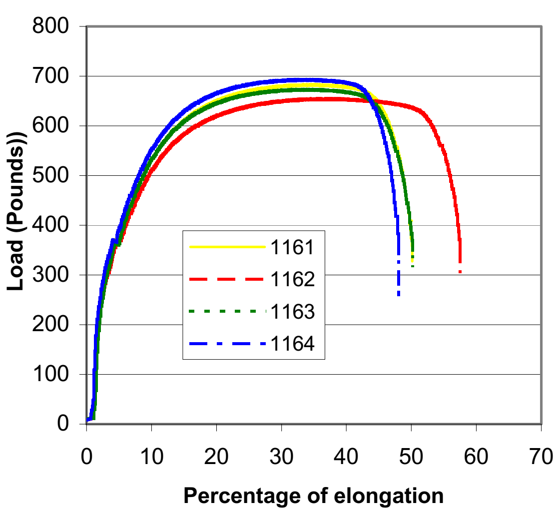

Figure 1: Stress-strain curves for the different materials.

Impurities

As mentioned above, impurity levels during ingot production were carefully controlled and kept at the same level for the materials with different Ta contents. Analyses were performed by 5 different laboratories with the average values for interstitial impurities as follows (in wtppm): oxygen: 7; hydrogen: 5; nitrogen: 4; carbon: 3.

CAVITY FABRICATION AND TESTING

Cavity Fabrication

Standard fabrication techniques were applied: half cells were deep drawn, machined to dimension with a self centering welding recess and a beam pipe/flange assembly was electron beam welded (EBW) to the half cells. After mechanical polishing to remove any surface imperfections and a removal of app. 30 µm from the surface by Buffered Chemical Polishing, (BCP) the two half cells were joined at the equator by EBW.

Surface Treatment

All six cavities were subjected to the following – equal – surface treatments:

- Degreasing in a soap/water solution with ultrasonic agitation for 30 min.
- (BCP), followed by thorough rinsing with ultra pure water and 30 min of high pressure rinsing (HPR) with a fan generating spray nozzle. Removal of 100 µm, 200 µm and 300 µm with rf measurements in-between.
- After HPR each cavity was dried in a class 10 clean room for 2 hrs prior to assembly of the rf input – and output coupling probes and evacuation on the test stand.
- After 12 hrs on the test stand the cavity vacuum had improved to the low 10^{-8} mbar range, and the cavity was cooled down to liquid helium temperature.

RF Tests

The subsequent rf tests consisted of measuring the surface resistance between 4.2K and 2K and the dependence of the Q-value on accelerating gradient (Q vs. E_{acc}) at 2K. The following observations were made:

- All cavities "quenched" at the field levels indicated in Table 2; no field emission was observed. No improvement in performance with additional material removal was observed.
- All cavities showed a strong Q-drop at gradients $E_{acc} > \sim 20$ MV/m as shown in Figure 2
- Residual resistances ranged between 12 nΩ and 3 nΩ; no systematic trend with material removal was observed
- Energy gap values $\Delta / k\, T_c$ ranged between 1.72 and 1.82; no systematic trend with material removal was observed.

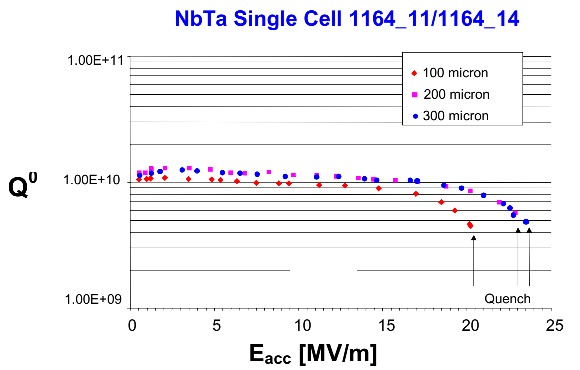

Figure 2: Typical cavity performance after removal of 100, 200 and 300 micron of material from the surface.

A summary of the tests is shown in Table 2

Heat Treatment

As a first step in assessing different heat treatments, one cavity of each Ta content was subsequently heat treated in vacuum at 800 C for 6 hrs in a Ti box. No improvement in quench fields was observed after removal of an additional 100 µm of material. Post–purification heat treatment to improve the thermal conductivity will be the next step.

SUMMARY

A systematic investigation of the effect of Ta contents in high purity Nb on cavity performance was conducted: no significant differences in performances for 150 wtppm < Ta < 1300 wtppm was found. Additional heat treatment at 800 C did not improve cavity performance. No improvement of quench fields was observed with increased material removal as has been seen in previous investigations [6]. This might suggest that no "defect gradient" exists in this material. The presence of a Q-drop in each of the tests around 20 MV/m (~ 100 mT) is not too surprising since it has been observed in cavities made from low Ta content niobium with RRR – values up to 300.

The achieved accelerating gradients of ~23 MV/m are not yet acceptable for an accelerator such as TESLA; however less demanding requirements for e.g. RIA, SNS, CEBAF Upgrade or the X-ray FEL can be accommodated. Further post purification heat treatments are needed to determine the limiting capability of the materials. It is also planned to fabricate a multi-cell cavity from the high Ta content material for further qualification.

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REFERENCES

[1] T. Carneiro et al; http://conference.kek.jp/SRF2001/
[2] K.K. Schulze, "Preparation and Characterization of Ultra-High-Purity Niobium", Journal of metals, Vol. 33 (5), May 1981, p. 33-41
[3] W. Singer et al.; "Quality requirements and Control of High Purity Niobium for SC RF cavities", Physica C386 (2003), 379-384
[4] H.R. Salles Moura, "Melting and Purification of Niobium", Proc. Intern. Symposium Niobium 2001, Dec. 2-5, 2001, Orlando, FL, p. 147
[5] H. Umezawa, "Impurity Analysis of High Purity Niobium in Industrial Production", Matereaux & Techniques, Vol. 7-9, p.33 (2003)
[6] E. Mahner et al.; "Effect of Chemical Polishing on the Electron Field Emission of Nb Samples and Cavities", Proc. 6th Workshop on RF Superconductivity, Newport News (1993),1085

Table 2: Summary of cavity tests

Material # Sheet #	Ta content [wtppm]	Test #1 100 micron E_{acc}[MV/m]	Test #2 200 micron E_{acc}[MV/m]	Test #3 300 micron E_{acc}[MV/m]	Average E_{acc}[MV/m] H_p [mT]
1164_12_12	1300	**18.1**	**15.7**	**20.6**	**18.1 / 83**
1164_11_14	1300	**20.2**	**22.9**	**23.6**	**22.2 / 102**
1161_31_34	~150	**22.3**	**22.2**	**21.2**	**21.9 / 100**
1161_32_33	~150	**23.6**	**22.5**	**23.7**	**23.2/ 106**
1162_33_34	~600	**23.3**	**22.6**	**23.5**	**23.1 / 106**
1162_32_35	~600	**20.4**	**23.4**	**22.8**	**22.2 /102**