HIGH-TEMPERATURE ANNEALING OF SUPERCONDUCTING CAVITIES FABRICATED FROM HIGH PURITY NIOBIUM*

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Abstract

Surface cleaning of superconducting cavities by high-temperature annealing (HTA) under UHV conditions is considered to be an effective means to achieve both high Q_o values and high accelerating fields, which are essential for linear collider applications as well as for upgrading of electron accelerators for nuclear physics research. Suppression of defect induced quenching by the use of high purity niobium has brought surface magnetic fields beyond 100 mT within reach. However, field emission loading has become a significant field limitation. Recent dc field emission studies have shown that the number of field emitters can be reduced drastically by HTA above 1100°C. We have investigated the influence of HTA on the performance of superconducting S-band single-cell cavities fabricated from niobium of high thermal conductivity, which has been postpurified by solid state gettering. Peak surface magnetic and electric fields up to 97 mT and 59 MV/m respectively, have been achieved.

Introduction

Present plans for the large scale application of superconducting (sc) rf cavities in high energy electron storage rings and in recirculating electron linacs are based on minimum design values for the accelerating gradient of E_a = 5 MV/m and for the quality factor of $Q_o = 2 \cdot 10^9$.[1] Theoretically sc niobium cavities could provide about one order of magnitude higher performance values. Practically local thermal breakdown (quenching) induced by defects or by impinging electrons and non-resonant electron loading (NREL) due to field emission are the present field limitations. Both anomalous energy loss mechanisms reduce in addition the Q_o values at high field levels.

The thermal stability of the cavity wall can be improved by the use of high purity Nb. Model calculations have shown that the threshold field for quenching caused by large (> 100 μm) normal conducting defects scales with the square root of the residual resistivity ratio (RRR).[2] This prediction has been confirmed statistically by experimental results on single cell cavities [3,4] and guarantees to exceed a design gradient of 5 MV/m with multicell cavities built from high purity Nb.

For accelerating field levels above 5 MV/m often strong NREL of sc cavities is observed as the dominant field limitation. At the University of Geneva dc field emission studies on Nb samples are performed to clear up the physical nature of local field emitting sites.[5] In most cases the enhanced field emission is originated by micron-size particles as for example dust. Systematic investigations on the effect of in situ high temperature annealing (HTA) have shown that the density of field emitters can be reduced significantly by HTA above 1100°C, while for 30 min HTA above 1400°C emission free surfaces of cm^2 size at dc field levels of 100 MV/m have been obtained repeatedly.

During the seventies HTA up to 2000°C has been considered to be a useful surface cleaning method for the preparation of reactor grade Nb cavities.[6] Unfortunately, the residual gas pressures of existing UHV furnaces for HTA of Nb cavities are high enough to degrade the purity level of the presently used Nb (RRR > 100) at high temperatures. Therefore, a careful increase of moderate temperatures controlled by thermal conductivity measurements has been pursued for this investigation on HTA of high purity Nb single-cell cavities.

Preparation of the cavities

In this paper the results of a test series on two spherically shaped 3 GHz cavities built from medium high purity Nb (W.C.Heraeus, RRR = 90 and 135) are summarized. The cavity with the lower RRR value has been postpurified at Cornell after four tests to study the influence of an increased thermal conductivity on the performance directly. Using yttrium as a getter material for the interstitially dissolved oxygen content [7], the cavity was baked out at 1250°C and 10^{-5} torr for 4 hours in a package of Y and Nb foils. Thermal conductivity and RRR measurements on comparable Nb samples have shown a resulting purity level up to about RRR = 400 (see Fig. 1).

For the HTA of the cavities at Wuppertal a resistive heating furnace with a double vacuum system has

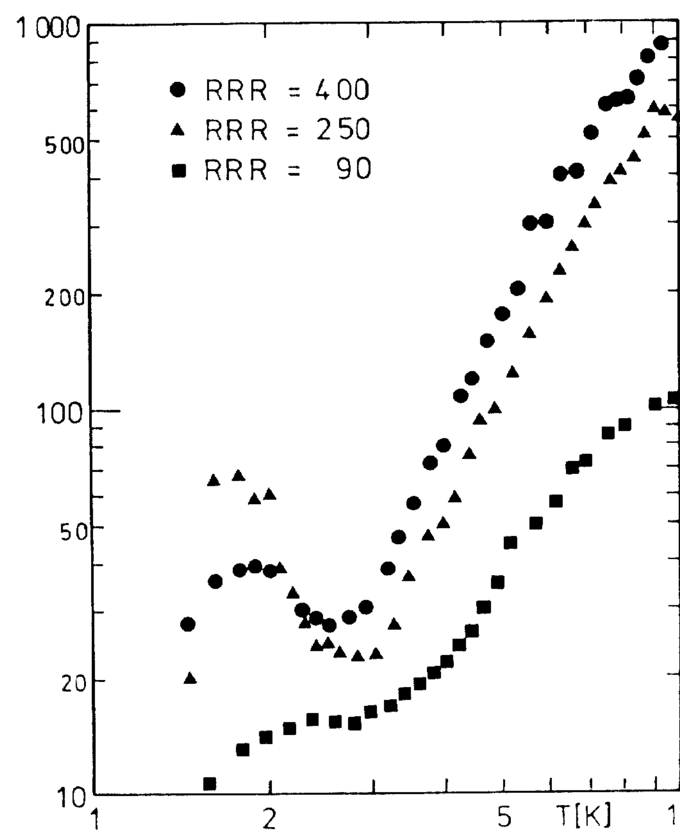

Fig. 1: Change of the thermal conductivity λ(T) of medium high purity niobium (RRR = 90) by yttrium treatment (→RRR = 400) and HTA (6h 1400°C → RRR = 250)

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Tab. 1: Summary of cavity tests

Cavity/ Test No.	treatment	RRR	Q_0(4.2K)/ 10^8	Q_{res} /10^9	onset E_p(MV/m) of NREL initial	rf-proc.	He-Proc.	max. surface fields E_p(MV/m)	H_p(mT)	E_{acc}* (MV/m)	limitation
1/3	HCl,CP15,H_2O,Meth 1h 850°C	135	1.05	10.0	5.0	17.5 (10min)	25.0 (60min)	41.1	67.4	16.1	NREL
1/4	HCL,CP15,H_2O,Meth 1h 850°C,DP	"	1.30	6.3	40.0	40.0 (10min)	-	47.7	78.2	18.7	Quenching
1/5	CP20,H_2O,DD	"	1.0	5.0	7.5	18.0 (30min)	23.0 (20min)	40.3	66.1	15.8	NREL
1/6	CP15,H_2O,DD	"	1.05	4.5	21.0	28.0 (60min)	35.0 (60min)	46.4	76.1	18.2	NREL e^-induced Q
1/7	HCl,CP15,H_2O,Meth 1h 1100°C, DP	≤ 135	1.45	13.1	6.5	35.0 (20min)	-	44.6	73.2	17.5	Quenching
2/2	HCl,CP15,H_2O,Meth	90	1.25	4.0	12.8	20.0 (20min)	no(>30) (1min)	30.6	50.2	12.0	Quenching
2/3	HCl,CP15,H_2O,Meth 1h 850°C	"	1.3	12.0	19.0	19.0 (20min)	16.0 (15min)	30.1	49.4	11.8	Quenching
2/4	HCl,CP15,(H_2O),Meth 1h 850°C	"	1.2	3.9	1.0	15.0 (20min)	17.5 (5min)	26.3	43.1	10.3	Quenching
2/5	Yttrified,HNO_3 HCl,CP25,H_2O 1h 850°C, DP	≤ 400	0.6	8.0	12.0	25.0 (30min)	42.5 (45min)	58.9	96.7	23.1	NREL
2/6	HCl,CP10,H_2O,Meth 1h 850°C, DP	"	0.6	5.7	1.2	32.5 (30min)	32.5 (20min)	55.8	91.6	21.9	NREL
2/7	CP10,H_2O,Meth 1.5h 1100°C	"	1.4	2.0	1.0	3.7 (90min)	-	16.3	26.8	6.4	NREL
2/8	CP15,H_2O,CP10,H_2O	"	0.8	21.0	15.0	37.5 (120min)	38.5 (20min)	52.5	86.2	20.6	NREL
2/9	CP5,H_2O, 4h 1200°C, DP	≤ 200	0.95	11.4	42.0	20.0 (30min)	37.5 (20min)	47.1	77.4	18.5	Quenching

Abbreviations: CP15: 15 µm chemical polishing in a 1:1:1 (or:2) solution of $HF:HNO_3:H_3PO_4$; H_2O: rinsing in demineralized dust free (> 0.2 µm) water; (H_2O): rinsing in tap water; Meth: rinsing in dust free methanol; HCl,HNO_3: dissolution of contaminants over typ. 1 day; DD: dust free drying; DP: dust free precautions during assembly in furnace; 1h 850°C: 1h bakeout at 850°C in UHV furnace; NREL: non-resonant electron loading
* E_{acc} is defined as the computed voltage gain of a relativistic particle (v = c) passing a single cell cavity with cutoff tubes devided by the length of the cell (L = λ/2)

been used, the hot zone of which is separated from the heating and shielding parts by a Nb vessel. Depending on the residual gas pressure which is typically in the order of 10^{-9}torr at room but only 10^{-7}torr at the operating temperature (measured in the cold zone), sample measurements have shown a degradation of the RRR-value for HTA above 1000°C. With this furnace typically a 10% decrease of RRR for HTA of some hours at 1100°C and about a factor of two at 1400°C (see Fig. 1) has been obtained. A recently built, similar UHV furnace at Cornell which is provided for HTA of L-band cavities has given a change of RRR from 380 to 290 for 1h at 1200°C and from 360 to 60 for 1.5h at 1400°C.

For most of the tests special precautions to avoid dust contamination have been applied, as for instance covering of the vertically mounted cavity inside the furnace with a hood and slow initial pumping and venting of the furnace and cavity vacuum. The assembly of the cavities was performed always under laminar air flow conditions.

Discussion of the test results

For a better comparison of different preparation techniques the results given in Tab. 1 include some older tests of these cavities. [3] The measured Q_0 values at 4.2 K reflect approximately the purity level as expected from the BCS theory, which predicts a higher surface resistance R_{BCS} for increasing RRR. [8] The discrepancy of the highest Q_0 (4.2 K) after HTA at 1100°C is a strong hint for a surface recontamination with interstitial impurities, since in test 2/8 after a CP of 15 µm the low value recovers. Because of this dependence on the surface preparation there is no strong correlation between R_{BCS} and the bulk RRR.

The residual Q_{res} measured at low field levels scatter within the same range for wet and dry surface preparations, but values above 10^{10} have been obtained only after careful rinsing procedures with dust free water or methanol even in case of a subsequent HTA. Since much higher values have been obtained after HTA at 1850°C [9] the surface cleaning effect of HTA seems to be reduced for moderate temperatures.

The achieved maximum surface fields follow on average the predicted $H_p \sim \sqrt{RRR}$ law in case of quenching, which has been localized by temperature mapping[10] always in the near of the cavity equator. Moreover, with the postpurified cavity accelerating gradients of more than 20 MV/m have been achieved repeatedly without quenching, which reappears after the final HTA straightforwardly. These results clearly demonstrate the major role of the Nb purity for the thermal stabilization of still present defects.

In about half of the tests NREL has become the dominant field limitation. The expected benefit of HTA compared to a final wet treatment can be discussed best by the observed onset field levels (probe current > 1nA).

While in test 1/5 and 1/6 NREL has not been overcome, the same cavity has been driven easily into its quench limitation after HTA (test 1/4 and 1/7). Nevertheless, it should be mentioned that HTA without dust free precautions (test 1/3 and 2/7) has led to strong NREL too. For very low initial onset levels much improvement is achieved usually by rf processing, while subsequent He-ion sputtering performed at He pressures just below the discharge level mostly leads to a steady but slow increase of E_p. In both cavities the highest initial onset field levels of $E_p \geq 40$ MV/m occurred after HTA.

Obviously, for a deeper understanding of NREL the spatial distribution of field emitters must be investigated with improved diagnostic techniques. We have started systematic measurements of the X-ray intensity

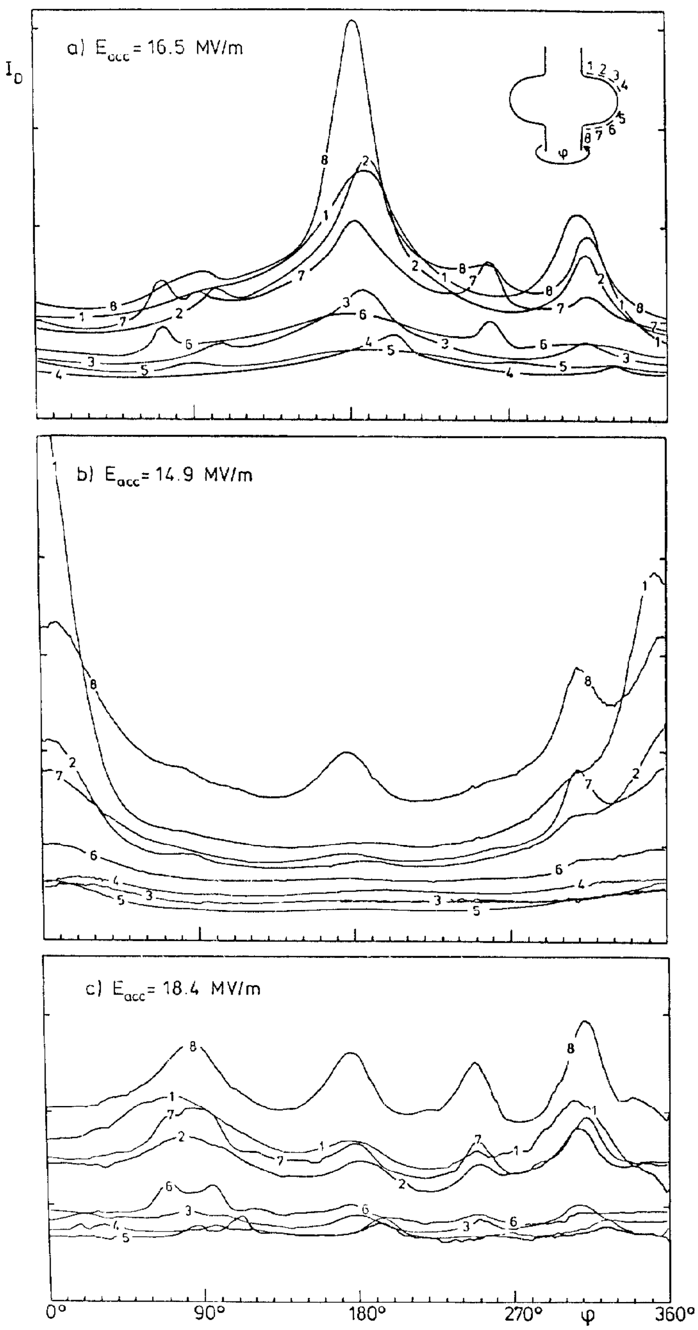

Fig. 2: NREL in test 2/9 spatially resolved with a rotating frame of 8 radiation detectors shortly after the initial onset (a) and final status after rf processing (b) and He-ion sputtering (c). The photoelectric currents I_D are plotted against the azimuthal location of the detectors.

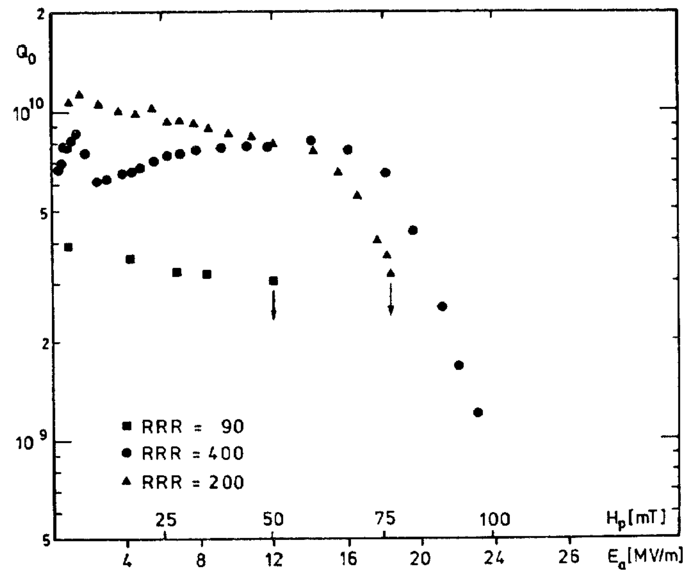

Fig. 3: $Q_0(E_a)$ dependence measured at 1.6 K on the same 3 GHz cavity for different purity levels.

around sc cavities with high-sensitive (25 mm^2) photodiodes. In Fig. 2 several peaks at different angles rise from a uniform background which can provide useful information about the number and the field enhancement factor β of emitting sites and the change of NREL during processing. For the dominant emitters at E_p>50 MV/m β values of about 100 have been found from probe current and X-ray measurements consistently. Since standard thermometry in subcooled Helium [10] cannot be applied to sc Nb cavities at field levels $E_a \geq 5$ MV/m·3 GHz/f, [11] thermometry in superfluid He is under development. [12]

In Fig. 3 the competition between quenching and NREL as present field limitation is well demonstrated. A further improvement of field gradients needs purity levels of at least RRR = 300 for thermal stabilization as well as an effective suppression of NREL. HTA of Nb cavities at moderate temperatures offers a possible, but riskful way towards the fundamental field limit.

Conclusions

This test series on two 3 GHz single-cell cavities has confirmed the square root correlation between the maximum achievable magnetic surface field and the purity level of the Nb for field limitation by quenching. For the postpurified cavity (RRR = 400) NREL has become the dominant field limitation. HTA up to 1200°C seems to be suited for the suppression of NREL, but leads already to a reduced RRR at the Nb surface and therefore to the reappearance of quenching. Lower residual gas pressures of UHV furnaces and improved diagnostic techniques are requested to overcome NREL.

References

1 H.Padamsee, this conference
2 H.Padamsee, IEEE Trans.MAG-19,1322(1983)
3 H.Lengeler et al., IEEE Trans.MAG-21,1014(1985)
4 H.Padamsee, IEEE Trans.MAG-21,149(1985)
5 Ph.Niedermann et al., J.Appl.Phys.59,892(1986)
6 P.Kneisel, Proc. of the Workshop on RF Superconductivity, Karlsruhe,ed.by M.Kuntze,KfK 3019(1980)p.27
7 H.Padamsee, IEEE Trans.MAG-21,1007(1985)
8 J.Halbritter, Z.Physik 238,466(1970)
9 A.Matheisen, University of Wuppertal,Diplomarbeit(1984)
10 H.Piel, ibid Ref. 6, p.85(1980)
11 G.Müller, Proc. of the Workshop on RF Superconductivity, CERN, ed. by H.Lengeler (1984)p.377
12 G.Müller and P.Kneisel, Cornell Univ.,SRF851201EX(1985)