

## SUPPRESSION OF FIELD EMISSION IN SUPERCONDUCTING S-BAND ACCELERATION STRUCTURES

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**Abstract:** Since quenching caused by local defects has been overcome as the main field limitation in superconducting Nb cavities by the use of high purity bulk material ( $RRR > 250$ ), field emission loading has become the dominant barrier. We have shown that high temperature annealing above  $1300^\circ\text{C}$  for at least 18 h combined with the evaporation of a Ti-layer on the outer cavity surface strongly suppresses the field emission without lowering the bulk purity. Moreover, multicell accelerator structures built from reactor grade Nb are thereby postpurified. This technique does not change the cavity resonance frequency and allows a completely dry surface cleaning and dustfree mounting. With single cell 3-GHz cavities we obtained accelerating gradients of up to  $27\text{ MV/m}$  at  $Q_0 = 2 \cdot 10^9$ , quench limited without any field emission loading. A 5-cell structure improved from  $12.6\text{ MV/m}$  to  $22\text{ MV/m}$  at  $2 \cdot 10^9$ . First application to 20-cell structures shows very promising results. The new method is now applied for the upgrade of the accelerator structures of the superconducting Darmstadt recycolotron. A very strong but partially reversible  $Q_0$  degradation dependent on cryogenic cycling observed with these and other cavities of high RRR will be discussed.

### Introduction

The benefit of superconducting cavities for electron storage rings, recycolotrons and linear colliders depends on the field levels and quality factors which can be achieved reliably in multicell structures. Since the thermal conductivity of the niobium sheet at  $4.2\text{K}$  could be pushed above  $50\text{ W/m}\cdot\text{K}$  ( $RRR > 200$ ), the threshold for defect-induced quenching in single cell S- and L-band cavities increased to values well above rf magnetic surface fields of  $50\text{ mT}$  and field emission loading became the dominant limit for high acceleration gradients [1,2].

High temperature firing above  $1200^\circ\text{C}$  in a UHV furnace is known to reduce the number and size of field emitters, increasing the onset field strength for dc [3] and rf [4] field emission. In addition, the reduction of the residual surface resistance due to homogenisation and effective surface cleaning is well established. To prevent the pickup of oxygen from the residual gas in the furnace, which would reduce the RRR again, the in-situ solid state gettering by evaporated titanium on the inner and outer cavity surface has been developed and applied successfully to single cell cavities [1,5,6]. Resonators built from reactor grade niobium are even postpurified to  $RRR > 200$  by this technique. Field emission free single cell cavities with  $E_p$  up to  $70\text{ MV/m}$  have been achieved [7].

The main drawback of this treatment is that the normal conducting titanium on the inner cell surface has to be removed finally by an etching of at least  $50\mu\text{m}$  followed by an extensive rinsing with  $\text{H}_2\text{O}$  and/or methanol. The risk to insert emitting dust particles or drying residues again is especially high for large multicell cavities which are difficult to rinse and to handle. Moreover the question if and how the acids or the water may produce intrinsically new emission sites still needs an answer [6]. In addition the frequency shift connected with the etching forbids the repeated application of double-sided titanisation to multicell cavities.

During our development of  $\text{Nb}_3\text{Sn}$  coated Nb resonators we succeeded to keep the RRR of the cavity high by titanisation of its outside alone while depositing Sn vapor from inside at  $1150^\circ\text{C}$  [8]. The application of this method to pure Nb multicell structures is very promising and will be discussed in the first part of this paper.

### Single-sided postpurification with titanium (SST)

Our UHV furnace for high temperature annealing (HTA) of 3-GHz structures with up to 20 cells is shown in Fig.1a. While single cell resonators can be heated up to  $1900^\circ\text{C}$ , the allowed temperature range for longer structures is limited by their mechanical instability. Because of a new supporting frame consisting of six Nb rods ( $\varnothing 10\text{mm}$ ) and some fixtures which support every 7-th iris and the lower beam tube, 5- and 20-cell structures can now be treated up to  $1360^\circ\text{C}$  without a change of their field profile. To protect the cavities from the residual gas in the furnace, the cavity

is surrounded sectionally by cylinders made from  $0.5\text{mm}$  titanium (grade 2) sheet. The furnace is shielded against these Ti cylinders by a box made from  $0.2\text{mm}$  Nb foil. The cutoff tubes are only partially covered. At the operation temperature of about  $1350^\circ\text{C}$ , the titanium vapor pressure of  $5 \cdot 10^{-5}\text{ mbar}$  ensures a complete coating of the structure from outside with a titanium layer of about  $12\mu\text{m}$  thickness in 10h.

To protect the inner surface of the cavities from residual traces of titanium vapor as well as from dust particles falling inside during handling, the beam tubes are closed with niobium hats. The structures are evacuated through a chicanery in these hats. At  $1350^\circ\text{C}$  the pressure in the cold zone of the furnace stays below  $10^{-6}\text{ mbar}$ . For a HTA in an UHV the structures can be installed into a niobium hot pot which can be separately pumped to below  $10^{-9}\text{ mbar}$ . The cooling period of the furnace takes typically 12 hours.

After all heat treatments the furnace is slowly floated within 3h with dustfree nitrogen gas. The dry cavities are then dismounted from the furnace insert in front of a laminar-air-flow box. Closed with clean PTFE caps they are transported into our clean room (class 10) for the final assembly of the rf couplers and the beam tube valves. The duration of exposure to air is usually less than 3h.

### Cryogenic and microwave testing

In our cryogenic inserts all cavities are evacuated from the bottom by means of an ionisation pump. Due to the elbows, valves, bellows, and the up to  $2.6\text{m}$  long stainless steel pumping tube the pumping speed at the cavity port is limited to  $0.5\text{ l/s}$  at  $300\text{K}$ . After typically one day, a pressure of less than  $5 \cdot 10^{-8}\text{ mbar}$  is reached even with the wet treated resonators that are dried by pumping. The cooling rates from  $300\text{K}$  to  $4.2\text{K}$  range from  $5\text{K/min}$  to  $0.4\text{K/min}$  for single and multicell structures. Especially the larger systems are often precooled for about 60h to  $77\text{K}$ .

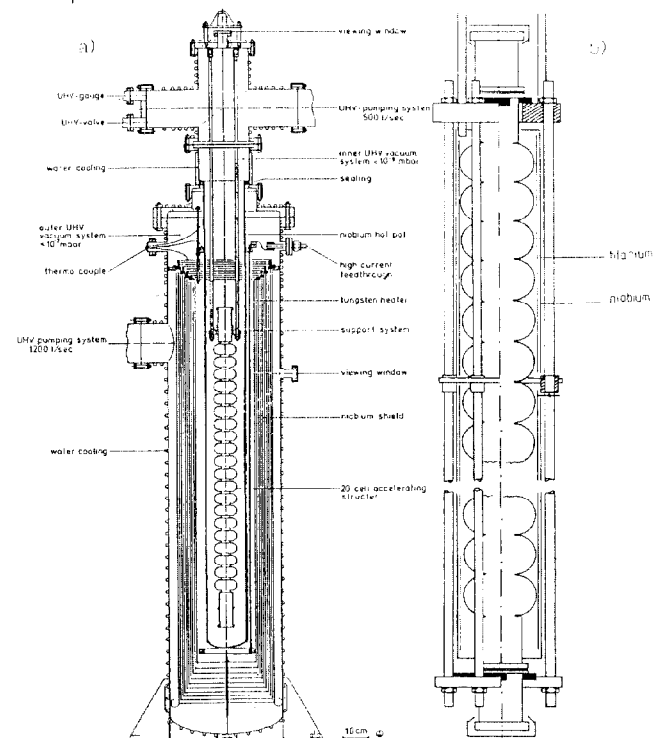


Fig.1: UHV furnace for high temperature treatment up to  $1900^\circ\text{C}$ :

- Cross section of the furnace with hot pot installed.
- Support system for single-sided titanisation.

The cryostat inserts for single cell experiments are equipped with adjustable input and fixed output couplers. Diagnostic systems for high resolution x-ray and temperature mapping in superfluid helium allow a detailed study of the effect of different surface treatments on the residual losses and rf field emission. A 500W cw klystron provides sufficient power for high field experiments with the multicell structures.

**Results and discussion**

Our latest results are summarized in in Table 1 and Fig.2. They confirm that the quench field strength of high purity niobium cavities (RRR > 200) is increased by a factor of 2-4 compared to resonators built from reactor grade material. Wet treated single cell cavities often yield very high rf surface field strengths without any field emission loading (e.g. S3-8). But the procedure of dustfree rinsing and drying still has to be optimized and, especially for multicell structures, heat treatment above 1200°C is more reliable to obtain defect- and dustfree surfaces. For two-sided titanisation the necessity of a subsequent etching, however, sacrifices the benefits of such high temperature treatment (B20-a, E5-a). In this case, a final firing at 850°C increases the performance again (B20-b, C20).

A clear improvement was made with single-sided titanisation (SST). We could show that the RRR can be kept or even increased to values above RRR 200 with a treatment of more than 18h at 1300-1360°C. In the experiments S3-13 and E5-b, for example, the quench limit was pushed to 113mT and 89 mT and no field emission was observed up to nearly 70 MV/m even in the five cell cavity. With the 20-cell structures built in 1984 from reactor grade niobium for the Darmstadt recyctron, acceleration fields well above the design value of 5 MV/m can now be reached reproducibly.

HTA in UHV is known to produce very low a residual resistance due to bulk homogenization and effective surface cleaning (e.g.  $R_{res} = 3.6n\Omega$  in S3-5). In comparison, exceptional high losses are observed after SST (e.g. B5-a, I20). Possible explanations are a surface contamination with evaporated Ti or too high a pressure inside the cavity during SST due to the protection Nb caps. A third possibility -the pickup of  $H_2^-$ - is discussed below. A short BCP (5-7µm) and water rinsing in combination with a subsequent HTA at 1000°C (with the titanium box) successfully yielded much higher quality factors (B5b, B5c). The often observed increase of  $R_{res}$  with the square of the rf surface field (Fig.2a) needs further investigation. Temperature maps in superfluid helium of field emission free cavities proof that even at field levels above  $H_p = 110mT$  the cavity  $Q_0$  is mainly limited by some local defects.

Meanwhile two postpurified structures (G20, D20) have been installed and operated in the Darmstadt recyctron. While G20 showed up with the same performance in the accelerator as in the vertical test, D20 (RRR 250 after SST) had a surprisingly low  $Q_0$  of  $2 \cdot 10^7$  only. Analysing the quality factors of all 20 passband modes established high residual losses in all cells. Another cavity, built from high purity Nb (RRR 300), which yielded originally  $E_{acc} = 6.6MV/m$  and a  $Q_0 > 10^9$ , showed the same effect after a thermal cycle to

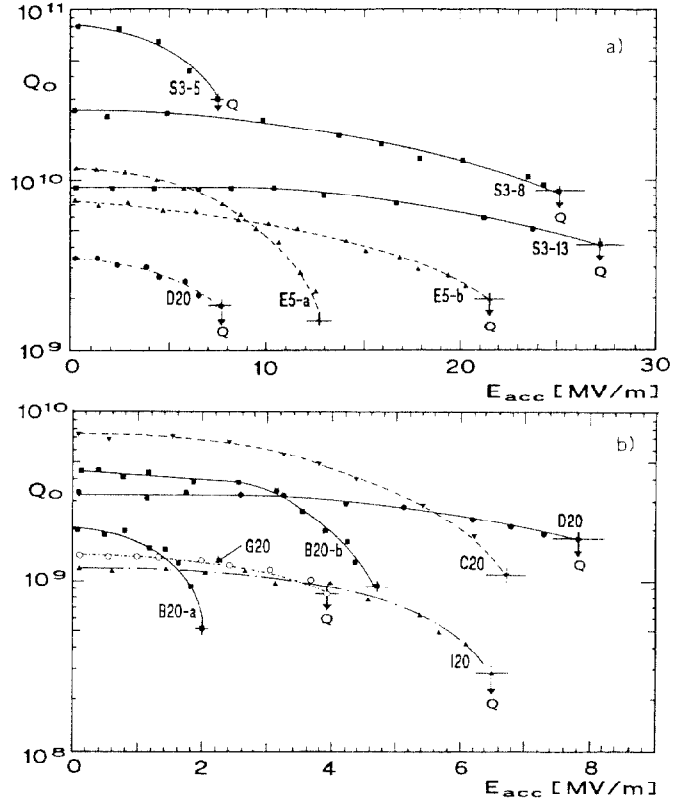


Fig.2:  $Q_0(E_{acc})$  dependence, measured at 1.4K (a) for our best single- and multicell- and (b) for the postpurified 20-cell S-band structures. The arrows mark field limitation by quenching.

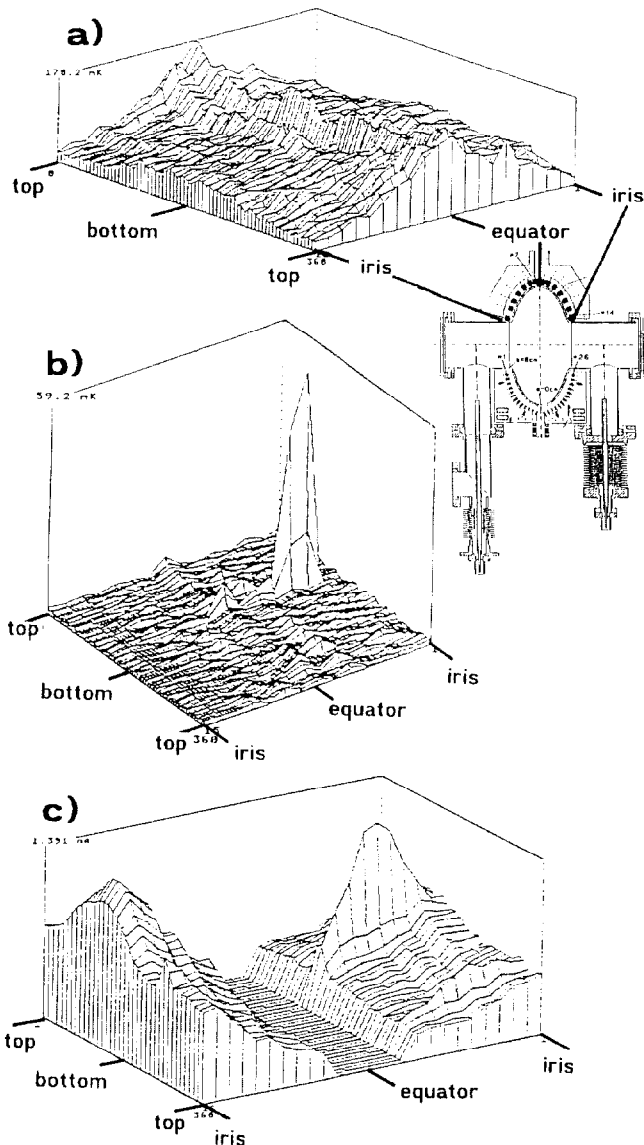
Table 1: Summary of 3-GHz-cavity treatments and vertical test results measured at T=1.4 K.

prot.: protection against gas; HP: UHV hot pot; Ti: titanisation from both sides (i,o) or from outside alone (o); BCP: buffered chemical polishing (1:1:1); H<sub>2</sub>O: rinsing with 18 MΩ·cm water; onset: start of detectable field emission (FE) loading; Q: quench; power: available RF power; meth.: final rinsing with dustfree methanol; dry/wet: surface of cavity as mounted to pump.

Cavity Test	# of cells	final surface cleaning:					$Q_0^{max}$ [10 <sup>9</sup> ]	$E_{acc}^{onset}$ [MV/m]	$Q_0(E_{acc}^{onset})$ [10 <sup>9</sup> ]	$E_{peak}$ [MV/m]	$H_{peak}$ [mT]	$E_{acc}^{max}$ [MV/m]	$Q_0(E_{acc}^{max})$ [10 <sup>9</sup> ]	limitation (comments)
		T [°C]	t [h]	prot.	CP [µm]	H <sub>2</sub> O [l]								
S3-5	1	1850	4	HP	--	--	80	--	--	18.6	30.3	7.3	30	Q, low RRR dry
S3-8	1	1310	10	Ti (i,o)	100	150	26	--	--	64.2	104.7	25.2	8.5	Q wet
S3-12	1	--	--	--	15	300	3	10	3	33.9	55.3	13.3	2.5	Q wet
S3-13	1	1350	18	Ti (o)	--	--	10	--	--	69.4	113.0	27.2	4.3	Q, meth. wet
B5-a	5	1330	17	Ti (o)	--	--	0.66	--	--	43.4	58.2	14.0	0.35	power dry
B5-b	5	--	--	--	7	300	9.5	9	2	31.0	41.5	10.0	1.2	Q, meth. wet
B5-c	5	1000	8	Ti (o)	--	--	10	11	2	43.4	58.2	14.2	0.83	Q (FE) dry
E5-a	5	1300	8.5	Ti (i,o)	70	350	12	10	5	39.1	52.3	12.6	1.5	FE, power wet
E5-b	5	1360	18	Ti (o)	--	--	8.5	--	--	66.7	89.3	22.0	2.0	Q dry
B20-a	20	1300	11	Ti (i,o)	75	200	2	1	2	6.2	8.3	2.0	0.5	FE, power wet
B20-b	20	820	3.5	HP	--	--	4.5	3	3	14.6	19.5	4.7	1.0	FE, power dry
C20	20	1300	11.5	Ti (i,o)	75	200	--	--	--	--	--	--	--	not measured before HTA
		835	3	HP	--	--	7.6	5	3	20.8	27.8	6.7	1.1	FE, power dry
D20	20	1350	21	Ti (o)	--	--	3.5	5	2	24.2	32.4	7.8	1.9	Q dry
G20	20	1350	20	Ti (o)	--	--	1.5	--	--	12.1	16.2	3.9	0.9	Q dry
I20	20	1350	20	Ti (o)	--	--	1.1	4	0.9	20.2	27.0	6.5	0.3	Q dry

about 190K, necessary for the repair of a roots pump. After 4 days at 300K the  $Q_0$  of this cavity recovered by a factor of 10 to  $2 \cdot 10^8$  in the next accelerator run.

Effects of this kind have been observed previously at Wuppertal with different cavities of high purity ( $RRR > 150$ ). For example the residual  $Q_0$  of a 1.5 GHz single cell resonator dropped from  $2 \cdot 10^{10}$  to  $2 \cdot 10^7$  ( $R_{res} = 13 \mu\Omega$ ) after a slow warmup to 180K. A subcooled temperature map at 3.8K and  $E_{acc} = 0.8 \text{ MV/m}$  displays mostly homogeneous losses with a small local maximum opposite to the pumping port (Fig.3a). After a thermal cycle to room temperature (3 days at 300K), the residual  $Q_0$  recovered to  $10^{10}$ . Superfluid thermometry at 1.4K (Fig. 3b) and x-radiation mapping (Fig. 3c) have shown no correlation of the residual losses or the field emission to



**Fig. 3:** Diagnostics of anomalous losses in an 1.5-GHz single-cell cavity: (a) Temperature map in subcooled helium at 3.8 K, and  $E_{acc} = 0.8 \text{ MV/m}$ , and  $Q_0 = 2 \cdot 10^7$ , showing high losses due to adsorbed gases. (b) Temperature map in suprafluid helium at 1.4 K, and  $E_{acc} = 12.5 \text{ MV/m}$ , and  $Q_0 = 10^9$  showing mainly losses due to field emission loading. (c) x-ray-map, corresponding to the temperature map shown in (b).

the previously observed surface problems and have reproduced the maps after the very first cooldown.

A possible explanation to these effects may be the solution of hydrogen gas in the surface of the Nb wall. Experiments with Nb cavities, which gettered large amounts of H during a chemical surface treatment, gave evidence for residual resistances of up to  $0.25 \text{ m}\Omega$  [9]. At temperatures below 200K hydrogen soluted in Nb is known to precipitate into the normalconducting  $\epsilon$ -phase ( $\text{Nb}_4\text{H}_3$ ,  $T_c = 1.2 \text{ K}$ ), the clusters having a typical diameter of  $0.25 \mu\text{m}$  [10]. From the observed  $R_{res} = 13 \mu\Omega$  a hydrogen concentration in the Nb surface of about  $7 \cdot 10^{-3} \text{ at}\%$  can be estimated. At  $T > 200 \text{ K}$  such low H concentrations are soluted randomly in the Nb lattice ( $\alpha$ -phase) and don't disturb the cavity  $Q_0$  any longer. Because the formation of the  $\epsilon$ -phase hydrid needs thermal equilibrium and sufficiently high a diffusion speed or time, the cooling velocity of sc cavities may be an important parameter which might even explain the often observed  $Q_0$  reduction from vertical experiments to horizontal beam test.

The strong  $Q_0$  reduction has been observed with cavities of high purity after a second cooldown from below 200K. During the first cooldown the residual gas in the vacuum system may be efficiently cryopumped at parts of small heat capacity (bellows etc.) before the cavity itself hits the critical temperature region. With a slow warmup the situation is just opposite and the whole surface of the cold high purity cavity adsorbs and getters hydrogen before it can be pumped externally. At temperatures above 200K the precipitation becomes instable and most of the residual resistance disappears again.

#### Conclusions

Combining the benefits of HTA and postpurification with the possibility of a dry and dustfree structure assembly, single-sided titanisation for 18-21 h at  $1350^\circ\text{C}$  has proven to be a reliable process to reach higher acceleration gradients even in multicell structures. Moreover, due to the suppression of field emission,  $Q_0$  stays high up to the field limits. In 5-cell 3-GHz structures  $E_{acc} = 22 \text{ MV/m}$  at  $Q_0 = 2 \cdot 10^9$  has already been achieved, an important result for the development of future sc linear colliders. The use of zirconium as solid state getter material during single-sided postpurification at even higher operation temperatures will be tried in the future.

For a better understanding of the residual resistance of Nb cavities, a further investigation of the adsorption and diffusion of residual gases (e.g. hydrogen) in the cavity's UHV system below 200K is necessary.

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#### References

- [1] G. Müller et al., Proc. of the European Particle Accelerator Conference, Rome (1988), 1289, editor, F. Tazzari
- [2] H. Padamsee et al., Proc. 3<sup>rd</sup> Workshop on RF Superconductivity, 13, Argonne, 1982 ANL-PHY-82-1, editor, K.W. Shepard
- [3] P. Niedermann et al., J. Appl. Phys. **59**, 892 (1986)
- [4] G. Müller et al., IEEE trans. Electr. Ins., EI-24, 1013, 1989
- [5] P. Kneisel, J. Less Com. Met., **139**, 1988
- [6] Q.S. Shu et al., Proc. 4<sup>th</sup> Workshop on RF Superconductivity, 539, KEK, Tsukuba, KEK Report 89-21, editor, Y. Kojima
- [7] R.W. Röth et al., *ibid.* ref. 6, 437
- [8] D. Dasbach et al., IEEE Trans. on Magn., Mag.-25, 1862, 1989
- [9] M. Peiniger, *ibid.* ref.1, 1309, and private communication
- [10] T. Schober et al. in G. Alefeld, J. Völkl (eds.), Hydrogen in Metals I, Topics in Appl. Phys. **28**, 11-71 (1978) Springer Verlag Berlin