An Improved Surface Treatment for Superconducting Niobium Cavities

D.Reschke, W.Görlich, G.Müller, H.Piel, J.Pouryamout, R.Röth
Fachbereich Physik, Bergische Universität Wuppertal
42097 Wuppertal, Germany

Abstract

A high performance of superconducting (s.c.) cavities - i.e. high surface fields at a low surface resistance - requires the cleanliness of the inner surface as well as a high thermal conductivity of the niobium (Nb). Firing under vacuum at T>1200°C combined with a solid state gettering process is proven to achieve both. Usually, cavity and getter material are placed in the same vacuum system, causing the danger of a contamination by the evaporation of the getter material and during re-venting the furnace. The implementation of a separate vacuum system for the inside of the cavity resulted in accelerating gradients E_{acc} above 30MV/m (E_{peak}>75MV/m) on 3GHz single cell cavities. The results are compared to a series of 12 experiments using standard preparation techniques. Gradients of E_{acc} above 20MV/m and quality factors Q_0 above 10^{10} are achieved with a good reliability. In a series of tests with exposure of the inner cavity surface to clean gases, we found no deterioration of the $Q_0(E_{acc})$-performance.

1. INTRODUCTION

For a superconducting linear accelerator in the TeV-regime (e.g. TESLA) [1][2] acceleration gradients E_{acc} above 20MV/m at quality factors of $>5\cdot10^9$ are required. Despite the progress on suppressing the two limitating phenomena of s.c. accelerator cavities - field emission and local thermal breakdown - such performance is achieved today in laboratory experiments, only.

To prevent thermal breakdown at local defects, high purity Nb material (RRR>300) is used for today's cavity fabrication. In addition, the postpurification of the cavity by solid state gettering with titanium at 1300°C-1500°C allows a further increase of the thermal conductivity [3][4][5]. The getter material prevents the pick-up of residual gases from the furnace vacuum and reduces the interstitial contaminations (O,N). Best results of purification are obtained with the double sided titanisation (DST). Starting with initial RRR=270 and RRR=500, DST results in values of RRR=760 and RRR=1500 [6][7], respectively. The drawback of the DST is the necessity of a chemical etching of the inner cavity surface to remove the normal conducting titanium. The effect of the reduction of field emission by firing above 1200°C [8][9] often gets lost with the standard open chemical treatment, which, in addition, shifts the resonance frequency of the cavity. One promising way out may be the use of new 'closed' chemical facilities eliminating the risk to insert new emitters (dust particles, drying residues, etc.) [10]. A proven alternative is given by the single-sided titanisation (SST) [11][12] coating the cavity with a titanium layer from the outside, only. To prevent dust particels falling inside the cavity as well as a titanium vapor diffusion, the cavity openings are protected by niobium hats. An RRR-value of 300 is kept constant or slightly increased [13] depending on the used furnace. Though SST is well-tried in a large number of successful experiments, a significantly increased residual resistance is often observed compared to preparation procedures without titanium. The most probable explanation is given by a small amount of titanium vapor diffusing into the cavity despite the niobium hats. This negative aspect is overcome by the implementation of a separate vacuum system for the cavity in the furnace.

2. SST WITH SEPARATE CAVITY VACUUM

Our modified UHV furnace is shown in Fig.1. A clean niobium tube is flanged to the cavity from the top, while the bottom end is closed with a niobium plate. To avoid damage of the cavity flanges, tantalum foil is used at both flanges as a 'sealing'. The niobium tube contains heat shields and ends with an adapter to a standard CF 35 UHV-system at the (cold) top plate of the furnace. This vacuum system is separately pumped with a 180 l/s turbo pump and controlled with a quadrupole mass spectrometer.

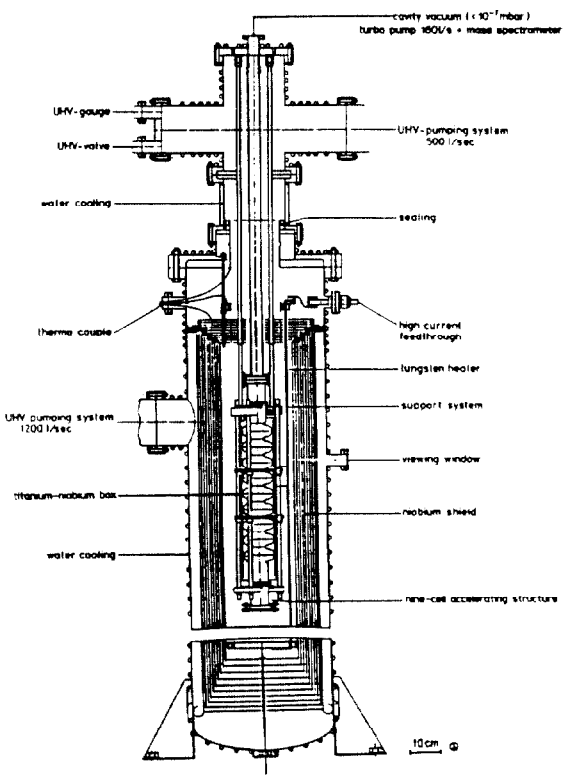

Fig.1: Cross section of the UHV-furnace with the separate vacuum system for single-sided titanisation

The typical duration of the treatment is 6h at T=1350°C. Before heating as well as at maximum temperature the pressure stays below $5 \cdot 10^{-7}$mbar and 10^{-7}mbar in the outer and inner vacuum system, respectively. The maximum pressure is kept below 10^{-6}mbar between 700°C and 900°C, where the hydrogen degases. Though the flanging of the cavity to the niobium tube is not UHV leak tight, we have no hint for a contamination of the cavity from the furnace vacuum. The inner vacuum can be floated in a controlled way with pure (>99,999%), filtered (0,2µm) gases. For the first experiments we used synthetic air up to 10^{-1}mbar and nitrogen up to normal pressure. The outer vacuum is slowly floated over several hours with filtered (0,2µm) nitrogen.

3.PREPARATION AND TESTING

The cavity is dismounted from the furnace insert and closed with clean PTFE caps in front of a laminar flow box. After the transport into our clean room (class 10), the cavity is rinsed for several hours with ultrapure water (2l/min, <10part. of >0,6µm/liter) under ultrasonic agitation. After drying with filtered (0,02µm) nitrogen (>99,999%) the rf couplers and the UHV valve are assembled.

Five 3 GHz single-cell cavities (E_{peak}/E_{acc}=2,55) with RRR=250-1000 were used for the 16 experiments. Before the improved SST the cavities were chemically etched and rinsed with ultrapure water. The preparation of all experiments are described in Table1.

RF- and He-processing has been done up to 100W in cw and pulsed (>10ms) operation. Diagnostic systems x-ray and temperature mapping in superfluid helium allowed a detection of the local Bremsstrahlung and distribution of the RF-losses. A quadrupole mass spectrometer was used to observe the residual gas composition of the cavity vacuum.
Fig.3 $Q_0(E_{acc})$-dependence after 3 various surface treatments

4.RF RESULTS

The rf-results are summarized in Table 1. In the 4 rf-tests carried out after the improved SST (included SW4-24 after additional ultrapure water rinsing) we achieved Q_0-values of $(2\text{-}4) \cdot 10^{10}$ (Fig.3) instead of Q_0-values of $5 \cdot 10^9$ to 10^{10}, typical for the "old" SST [12][14]. This demonstrates impressively, that the problem of increased residual resistance after SST is cured.

In Fig.2 the electric peak surface fields after the improved SST and the previous 12 standard preparations are compared. Three tests resulted in E_{peak}>60MV/m (E_{acc}>24MV/m). One test had to be stopped at low fields due to a leak. In test SW5-12 an accelerating gradient of E_{acc}=30,5MV/m (H_{peak}=127mT) was obtained (Fig.3) without any thermal breakdown. This is up to now the best result in Wuppertal. Without any processing two tests showed no field emission up to E_{peak}>62MV/m. This corresponds to gradients E_{acc} above 25MV/m at Q_0-values of 10^{10}. A comparable performance was measured only once after a standard preparation (Fig.3).

Analysing all 16 experiments, several general results can be derived:

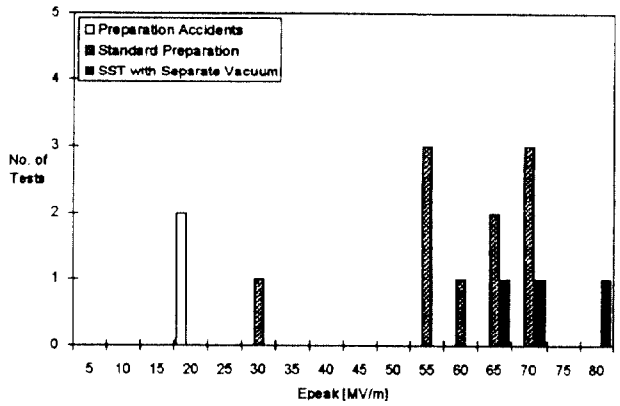

Fig.2 Maximum electric peak fields of all experiments.

- The applied surface treatments (Tab.l) result with a high reproducibility in E_{peak}>50MV/m and Q_0 above 10^{10}.
- Field emission started (excl. accidents) beyond >30MV/m.
- As expected for the high RRR niobium, cavities SW1-SW5 sustained magnetic surface fields above 100mT without a thermal breakdown.
- The best measured value of $Q_0=7 \cdot 10^{10}$ (Fig.3) corresponds to a residual surface resistance of R_s=4,1nΩ. This is in good agreement to the sum of the BCS resistance $R_{s,BCS} \approx$ 2nΩ at 1,4K and the residual resistance $R_{s,fir} \leq 5$nΩ due to frozen-in-flux expected for $H_{res} \leq 0{,}4$µT in our cryostat
- A preparation consisting of a HNO_3 soak + ultrapure water rinsing was used after SW2-18 to preserve the successfully tested surface before a new assembly. Contrary to our expectation, both tests (SW2-19,SW2-20) resulted in a poor Q_0 of $(4\text{-}6) \cdot 10^9$. Even if this degradation was not caused by the first treatment, the HNO_3-treatment was not sufficient to remove the responsible contaminations.
- Beside the usual slight improvements of the $Q_0(E_{acc})$-performance during low power processing, in a few cases drastic Q_0-degradations occured due to strong field emission events.
- The cavity performance usually does not change after a room temperature cycle, even if the test insert is moved by

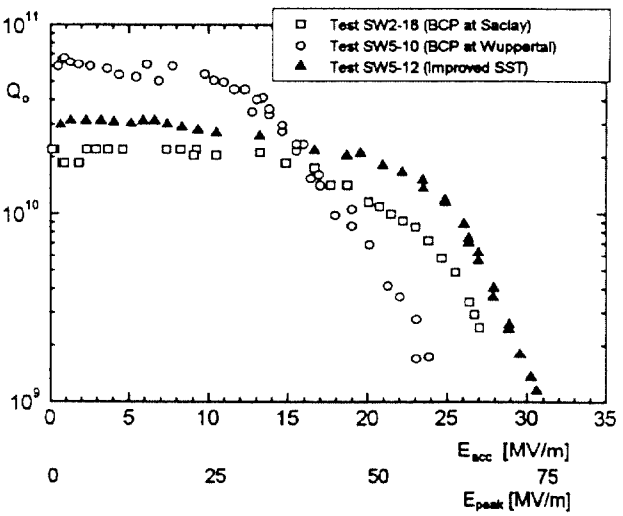

Table 1: Details of preparation and RF-results

Test	Preparation	Q_0^{max} [10^9]	E_p^{max} [MV/m]	Q_0 (E_p^{max}) [10^9]	E_p^{onset} [MV/m]	Comments (limitation)
SW1-19	BCP35,HTA	7,0	26,5	3,5	12,8	FE
SW1-20	BCP15	35	>45,4	8,0	38,3	
SW1-20-1	RT-cycle	26	61,2	1,4	39,8	FE,power
SW1-20-2	RT-cycle,He-gas	18	66,3	2,0	43,9	FE,power,He-proc.
SW1-20-3	RT-cycle, N_2-gas	24	64,0	2,0	39,5	FE,power,He-proc.
SW1-20-4	RT-cycle,syn.Air	19	>56,1	7,0	43,4	
SW1-20-5	RT-cycle	14	60,4	3,7	36,0	FE,power
SW1-20-6	RT-cycle,CO_2	15	55,9		34,4	FE,power
SW2-16	BCP33,HTA	30±20	62,0	1,0	40,8	FE,power,β>>1
SW2-17	Re-assembling	30±20	56,1	1,0	38,3	FE,power,β>>1
SW2-18	BCP9 (Saclay)	25±15	68,9	2,5	66,3	Q
SW2-18-1	RT-cycle	9,5	67,6	2,3	>67,6	Q
SW2-19	HNO_3,N_2-drying	4,2	54,3	1,2	34,4	FE,power,He-proc.
SW2-20	HNO_3	5,0	53,6	1,3	34,4	FE,power,He-proc.
SW2-20-1	RT-cycle	6,0	55,3	1,4	31,9	FE,power
SW2-21	BCP10,N_2-drying (Saclay)	35±15	67,1	0,8	41,6	FE,power,β>>1
SW2-21-1	RT-cycle	25±5	69,1	0,9	44,4	FE,power,β>>1
SW2-22	BCP20,ISST, water,N_2-drying	35			33,2	Test stopped due to leak
SW4-23	BCP8,ISST, water,N_2-drying	12	55,6	0,6	37,5	FE,power, He-proc.
SW4-23-1	RT-cycle	16	63,0	0,3	39,3	FE,Q,He-proc
SW4-24	water,N_2-drying	25	67,6	3,7	>67,6	Q
SW5-9	BCP30,HTA	0,6	17,6	0,4	15,3	vac. accident
SW5-10	BCP15,N_2-drying	70	(61,0)	1,7	39,5	decrease due
		33	50,0	0,6	31,9	to FE
SW5-10-1	RT-cycle	34	53,8	0,6	34,4	FE,power
SW5-10-2	RT-cycle	22	63,8	0,7	32,6	FE,power, He-proc.
SW5-11	BCP10,N_2-drying	41	(31,1)	19	25,5	decrease due
	(Saclay)	6	53,6	0,6	~33	to FE
SW5-12	BCP10,ISST, water,N_2-drying	31	78,0	1,1	62,5	FE
SW5-12-1	RT-cycle	18	63,2		>51,0	FE ind. Q
SW6-2	BCP25	4,0	15,8	1,8	>15,8	Q

BCP10: 10μm etching with final ultrapure water rinsing ; HTA: High temperature annealing at T≤1100°C without titanium; Q: Quench (local thermal breakdown) ; FE: Field emission ; RT: Room temperature ; N_2-drying: Drying with pure, dustfree nitrogen ; ISST: Single-sided titanisation with separate cavity vacuum

crane. This indicates, that there are no loose particles at the top flanges and beam tubes "snowing" into the cavity.

- After 2 preparations at CEN Saclay the cavity was closed with teflon caps and transported to Wuppertal, where the final assembly was performed in the class 10 cleanroom. With E_{peak}>65MV/m both tests showed the best results of the standard preparations. This demonstrates clearly, that a careful transport does not lead to a degradation of the cavity performance for gradients up to E_{acc}≈25MV/m.
- The diagnostic systems identified clearly two accidents during fabrication (SW6-2: bad welding) and preparation (SW5-9: contaminated vacuum system).

During the experiment SW1-20-i we studied the influence of gases like air, N_2, He, CO_2 with respect to residual resistance and field emission. Each gas exposure took place at room temperature with a few millibar pressure. Over the 7 tests the Q_0-value degraded from $3,5\cdot10^{10}$ to $1,4\cdot10^{10}$, but - as mentioned above - mainly due to field emission events. The field emission activity itself was not found to be correlated to the gas exposures. The onset field varied between E_p^{onset}=34-44 MV/m caused by 2-3 alternately active field emitters identified on the x-ray maps. In summary, a careful and clean exposure to pure gases gives no degradation even of a high cavity performance.

5. CONCLUSIONS

The improved single-sided titanisation procedure using a separate vacuum system for the inside of the cavity combines the benefits of HTA and postpurification - i.e. high electric and magnetic surface fields - with low surface resistances. In a test series on 3GHz single-cell cavities, low field Q_0-values of $(2\text{-}4)\cdot10^{10}$ and surface fields E_{peak}>60MV/m (best: E_{peak}=78MV/m) have been obtained in nearly all tests. The effective suppression of field emission allows gradients E_{acc} of 25MV/m at Q_0-values of 10^{10}. In comparison, standard preparation techniques resulted reproducibly in E_{peak}>50MV/m and $Q_0>10^{10}$, but field emission started typically at E_{peak}=30-40MV/m corresponding to gradients E_{acc}>15-20MV/m. Clean gas exposure, reassembly and transport of a cavity affects neither the residual resistance nor the threshold of field emission.

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