CHANGE OF RF SUPERCONDUCTIVITY PARAMETERS INDUCED BY HEAT TREATMENT ON NIOBIUM CAVITIES.

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Abstract

We have investigated the thermal quench evolution of a Niobium cavity, chemically polished, during ultra high vacuum annealings from 100 to 800°C.

Ten cavities have been tested before heat treatment (HT) and the results show a good agreement with the BCS theory for a diffuse reflection of the conduction electrons on the metal surface.

All along the HT we have clearly identified at first the oxygen diffusion from the surface, followed by a cleaning phase of the RF surface and finally, above 800°C, the appearance of a new contamination probably due to the furnace residual gases. All this events involve consequences for the quench position through the thermal conductivity.

1. INTRODUCTION

The quest for high gradients in accelerating structures is a important economical challenge for the collider project TESLA. The "Q_0 slope" vanishing, observed after the cavity in-situ baking [1,2,3,4] and due to the reduction of residual losses by Interface Tunnel Exchange [5], allows to push away the maximum of accelerator field achieved in Niobium cavities. So the ultimate performances of the cavity is now due to the "thermal breakdown limitation" rather than to the "RF power limitation".

Several experimental observations [2] seem to indicate that this limit is not intrinsic to the cavity:

- the electropolishing (EP) treatment, applied to clean the cavity surface, gives a better result (maximum accelerator field value E_{quench}) than the buffer chemical polishing (BCP) with roughly 40 MV/m for a single-cell cavity (10 MV/m more than BCP).
- performances with BCP are increased by using a fresh chemical bath.
- the in-situ baking at 150°C reduces E_{quench}.

These observations let think of a possible surface pollution by chemical wastes.

At this time, the EP process is better than the BCP one. Nevertheless it requires a more advanced equipment and the resulting surface is more sensitive in particular when the cavity is subject to the action of processed emitters.

For all these reasons and also to avoid a too early rejection of the BCP process, it is important to understand the reason of this low thermal breakdown value for a BCP cavity.

2. THERMAL BREAKDOWN

A surface defect in a RF cavity generates losses and consequently the temperature increases locally. If it exceeds the critical temperature T_C (9.2K), Nb around the defect becomes normal conducting and a thermal instability appears: this is the breakdown, also named quench.

A simple model [6-7] allows to express the maximum of the accelerator field versus the defect radius r, the normal conducting resistance of the material R_{nc} , the thermal conductivity κ and temperature T_{bath} to cool the cavity (in our case 1.6K):

$$E_{quench} \propto \sqrt{\frac{4\,\kappa\,(T_c - T_{bath})}{r\,R_{nc}}}$$

According to this formula the E_{quench} fluctuations in our observations could be due to a modification of the defect size and, or to a change in the Nb thermal conductivity near the surface, all around the defect. In other words if we want to improve the quench limit of the cavity it is necessary to reduce r and increase κ. A possible way, through a diffusion process, is a furnace treatment after the chemical etching of the cavity.

3. PARAMETERS MEASUREMENT

In order to understand what happens in the cavity when it is heated, we have systematically measured different RF superconductivity parameters (ℓ, T_C , R_{BCS} , Δ/k, R_{res}, E_{quench}). they are determined through the surface resistance R_S by measuring the intrinsic quality factor Q_0 of the cavity: $R_S=G/Q_0$ where the constant G is the geometrical factor. These measurements are made during the cooling down of the cavity from 10K to 1.6K. Above 4.2K, the measurement of the loaded quality factor Q_L is enough to determine Q_0 .

3.1 Electron Mean Free Path ℓ in Normal Conducting State.

In the normal conducting state (T>9.2K), the RF fields penetrate a depth δ (ℓ), from 300 to 1200 nm, and a simple relationship exists between R_S and ℓ, if the condition $\ell<\delta$ is satisfied (normal skin effect):

$$R_S = \rho / \delta \propto 1 / \sqrt{\ell}$$

where ρ is the electrical resistivity.

In the "anomalous skin effect" ($\ell>\delta$) an empirical formula [7-8] allows to calculate ℓ :

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$$R_S(\ell) = R_S(\infty)\left(1 + 1.157\,\alpha_S^{-0.2757}\right)$$

where $\alpha_S = 3/4\,\mu_0\,\omega\,(1/\rho\,\ell)\,\ell^3$.

This experimental value corresponds to a measurement on a depth δ, so we will write it ℓ_δ.

3.2 Surface Resistance in Superconducting State

Around 9.2K Niobium becomes superconducting and the sudden R_S drop down allows to measure T_C.

When $T < T_C/2$ the surface resistance becomes the sum of two terms. The first term R_{BCS} decreases exponentially with the temperature and is well describe by the BCS theory, the second term R_{res} is independent of the temperature.

$$R_S(l,T) = A\left(\lambda_L, \xi_F, \ell\right)\frac{\omega^2}{T}\,e^{-\Delta/kT} + R_{res}$$

where λ_L, ξ_F and Δ are respectively the London penetration depth, the coherence length and half the energy of the Cooper's electrons pair.

In the superconducting case the magnetic field penetration depth $\lambda(\ell,T)$ is lower than δ, few tens of nanometers. To avoid confusion and by analogy with the normal conduction we will write ℓ_λ the electron mean free path which appears in the formula above (ℓ_λ and ℓ_δ could be different if the material purity is or becomes non uniform in depth).

Experimentally, R_S has the same behaviour described by the theoretical formula, so it is easy to determine $R_{BCS}(\ell,T)$, R_{res} and the slope of the exponential decrease Δ/k.

3.3 Maximum Accelerator Field at 1.6K

When the cavity is at 1.6K we increase the injected RF power until E_{quench} is found.

4. $R_{BCS}(l)$ BEHAVIOUR

The experimental plot, R_{BCS}(T=4.2K) vs ℓ_δ (Fig.1), can be compared to the calculated one resulting from the BCS theory using Halbritter's code [9] where we use the Nb material data λ_L=31nm, ξ_F=62nm and the Δ value that we have measured (1.46 meV).

Among the ten 1.3 GHz cavities that we have tested at Saclay, five have been purified at 1300°C with titanium and three others at only 800°C (without Ti) to remove hydrogen. Before the test, a clean RF surface is achieved by either a BCP or an EP process, followed by a high pressure water rinsing (HPR). At last cavities are assembled in a class 100 clean room.

The Fig.1 plot calls for some comments:

- A good agreement appears between experimental points $R_{BCS}(\ell_\delta)$ and the theoretical curve $R_{BCS}(\ell_\lambda)$ for a diffuse reflection of the conduction electrons on the metal surface (continuous line) rather than a specular reflection (dotted line). This demonstrates the homogeneity of the niobium impurities in the cavity wall since $\ell_\delta \approx \ell_\lambda$ ($\approx \ell_{bulk}$ for the C114, see table 1).
- As expected, Ti treatment improves the RRR of the cavity. On the contrary, the strong degradation of cavities treated at 800°C, is amazing: it probably results from a contamination by the residual gas in the furnace.

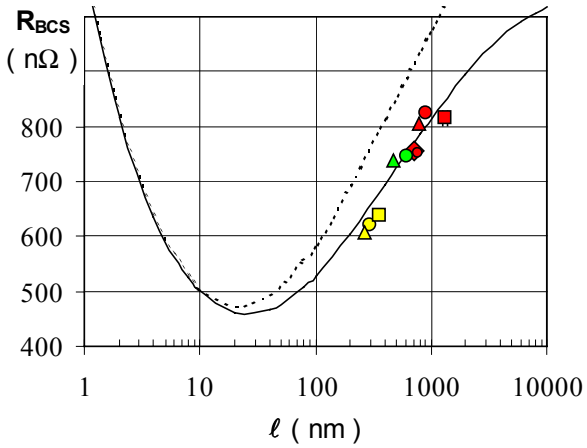

Figure 1: BCS resistance at T=4.2K vs electron mean free path. Theoretical curves and experimental points. Color refers to Table 1.

Table 1: Characteristics of experimental data.

Cavity	Nb manufacturer RRR (initial - final)	Purifying °C	ℓ_{bulk} nm	Chemical Etching	R_{BCS} nΩ	ℓ_δ (nm) ab/normal skin effect
D101	Wah Ch. (300 - ?)	1400 + Ti	?	BCP	815	1320
C105	Heraeus (280 - ?)	1200 + Ti	?	BCP	825	875
C118	Tokyo D. (200 - ?)	1300 + Ti	?	BCP	805	795
C114	Tokyo D. (190 - 350)	1300 + Ti	800	EP KEK	755	750
C117	Tokyo D. (200 - ?)	1300 + Ti	?	BCP	755	710
C103	Heraeus (280)	no	640	EP KEK	750	605
P101	Tokyo D. (250)	no	570	BCP	740	475
D123	Wah Ch. (300)	800 DESY	690	EP CERN	640	360
C116	Wah Ch. (280)	800	640	EP CERN	620	290
D121	Wah Ch. (300)	800	690	EP CERN	605	265

5. HEAT TREATMENT OF THE CAVITY

Until 150°C, the C118 cavity is baked in-situ on the test bench [1] during 60 hours to ensure a stabilisation of the diffusion process [3]. For the other temperatures, a furnace HT is achieved each time after a BCP (20 µm) and before a HPR. In these conditions we start with the same initial layer on the surface. The furnace, where a turbomolecular pump (1000 l/s) provides a clean vacuum ($p=1.10^{-7}$ mbar at 1000°C) is refilled, after the cavity heating, to the atmospheric pressure by introduction of Ar, O_2 or N_2 gas.

The experimental results, shown on Table 2, require some comments:

Table2: RF superconductivity parameters change versus the heating temperature of BCP cavities.

Cavity	Atm. Refill.	T °C	T_C K	R_{BCS} nΩ	ℓ_δ (nm) ab/normal skin effect	E_{quench} MV/m	R_{res} nΩ
C118	-	30	9,23	805	**795**	power limit	4,2
	vacuum	100	-	600	**695**	29	6,2
	vacuum	120	-	465	**675**	26	9,4
	vacuum	150	9,23	410	**600**	25	12,1
	Ar	190	9,11	460	**130**	22	8
	Ar	250	8,95	455	**45**	electrons	47,8
	N_2	320	9,00	460	**90**	electrons	9,4
	O_2	400	-	525	**120**	electrons	53,1
	N_2	490	9,04	470	**145**	18	2,4
	N_2	650	9,08	455	**280**	electrons	14,5
	N_2	800	9,12	415	**255**	21	2,6
	-	30	9,08	630	**605**	21	1,6
C117	-	30	-	735	**575**	24	1,2
	Ar	1000	-	420	**135**	22	3,8

- At low temperature (T≤150°C), as expected, R_{BCS} decreases and suggests, through the ℓ_λ decrease, a diffusion process in the superconducting layer [1-3]. On the contrary, the normal conducting layer is still not affected (ℓ_δ almost unchanged). Between 150 and 250°C, the diffusion coefficient increases, the diffusion depth grows and ℓ_δ decreases.
- The critical temperature T_C has the same evolution than ℓ_δ. This implies that the oxygen is the diffusing element in niobium [10]. The oxygen, at the beginning, comes from NbO_x , the metallic interface oxyde and then from Nb_2O_5 , the dielectric oxyde of the surface which is totally decomposed at 250°C [11].
- Above 250°C the avalaible oxygen quantity decreases and the diffusion is so deep that the ℓ_δ trend is inverted.
- After the heat treatment of the cavity, the furnace is refilled with gas to the atmospheric pressure. If the injected gas is not reactive with Nb (e.g. Ar or N_2), it acts as an impurity for RF superconducting layer so ℓ_λ decreases and R_{BCS} becomes anomalously low. Only refilling with O_2 gives a R_{BCS} value such as the data point $R_{BCS}(\ell_\delta)$ is restored on the theoretical curve of Fig.1.
- Around 800°C a new diffusion appears which induces a new decrease of ℓ_δ . The HT at 1000°C on C117 cavity confirms this fact. It is also the same phenomenum involved in the three cavities, treated at 800°C to remove H_2 , and that we have mentionned earlier in the Fig.1 comments. This new contamination is probably due to the residual gas of the furnace. The residual gas analysis shows CO as the main component.
- The last test on C118, without heating, shows a bulk pollution since we do not find the initial results. However, the initial characteristics have been restored on C118 and C117 cavities by a HT at 1300°C with Titanium as a getter.
- About E_{quench} , as for T_C , a correlation with ℓ_δ is found. This can be explained by the dependence between ℓ and the thermal conductivity κ. During the experiment we have been confronted with some electron emission, perhaps due to the activation of electron emitters with the temperature as mentionned in [12].
- All along the cavity HT, the Δ/k value (16.96 K^{-1}) which is the exponential slope of $R_S(1/T)$ did not change.

6. CONCLUSION

These preliminary results concerning the heat treatment of BCP cavity demonstrate:

- The good agreement between experimental data and BCS theory,
- The clear identification of oxygen diffusion in the superconducting layer even at low temperature,
- The importance for R_{BCS} of the gas choice to the furnace refilling,
- The cavities pollution for a HT from 800°C.

Unfortunately the residual gas contamination screens the E_{quench} evolution above 800°C and a possible improvement beyond the starting value. So, in the next future, we plan to continue the HT experiment at 800 and 1000°C by using a titanium box to protect the cavity from the furnace vacuum.

7. ACKNOWLEDGEMENT

We would like to thank our colleagues A.Aspart, B.Coadou, Y. Gasser, M. Jablonka, G. Monnereau, and J.P Poupeau for their contribution to this work.

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