COMPARATIVE MEASUREMENTS ON NIOBIUM SHEET AND SPUTTER COATED CAVITIES

G. Arnolds-Mayer and W. Weingarten
CERN, Geneva, SwitzerlandAbstract

Comparative measurements of sheet metal and sputter coated Nb cavities were performed at 500 MHz. The sputter coated ones had a higher low field Q, a stronger decrease of Q with the accelerating field, and showed no static magnetic field dependence of the Q value. Throughout the layer, larger amounts of impurities were found, which may explain the experimental results.

Introduction

At CERN presently two alternative superconducting cavity fabrication methods are being studied. One is based on commercially available niobium sheet, with a sufficiently high thermal conductivity, the other on OFHC copper sheet, onto which a thin niobium layer is sputter deposited.

Each method has its advantages and drawbacks. Sheet metal niobium cavities have already shown good results [1], but they suffer occasionally from fast thermal breakdowns ("quench"). Sputter deposited cavities are virtually thermally stable against local heat sources (e.g. normalconducting defects, impinging electrons etc.) and have less defects [2]. However, the reason for a degradation of Q with the accelerating field E_a is not yet well understood.

The Q value decreases either stepwise or gradually. The cause of the stepwise drop of Q ("Q switch") are small niobium surface regions becoming normal due to an insufficient thermal contact with the copper (e.g. blisters [2]). The gradual decrease of Q is not understood. In this paper we will investigate variables determining the Q value and give possible causes for the Q degradation. We include also results on sheet metal cavities for comparison.

Experimental ResultsAccelerating fields and Q values

Figure 1. Typical $Q(E_a)$ curves of sheet metal (line) and sputter coated cavities (hatched).

The hatched region of fig. 1 represents typical $Q(E_a)$ curves for sputter coated cavities at 4.2 K without "Q switches", the line for a sheet metal cavity. The sputter coated cavities show higher low field Q values, but with a larger scatter and a bigger slope.

Spatial distribution of r.f. losses

(a) Calibration of resistor sensitivity:

A copper cavity without niobium coating was cooled down and powered by r.f. in the same way as a superconducting cavity. Since the r.f. losses can be supposed to be strictly proportional to the square of the magnetic field, variations in the temperature response of the individual resistors express their different sensitivity. At the same occasion we also measured the residual resistivity ratio RRR (86) and the Q values (47×10^3 at 300K and 2.7×10^5 at 4.2K). The information turned out to be useful for the determination of the RRR value of the niobium coating in situ.

(b) Overall r.f. losses:

After this individual calibration of the carbon resistors (sensitivities varied by up to a factor of 3) we measured the average loss distribution along a meridian (fig. 2) of a sputter coated cavity. The result shows

Figure 2. Average loss distribution in sputter coated cavity along meridian.

that the losses are created by the r.f. magnetic field. Within the precision of the measurement ($\pm 15\%$) their relative amplitude (normalized to the maximum amplitude) is independent of the r.f. field.

(c) Local r.f. losses:

In about one third of all measured Nb coatings, film peel off and Q switches occurred. In taking temperature maps we were able to find the corresponding "hot spots" which show up at the same field at which the Q switch is observed, and can be located on the cavity surface with

a precision of some mm. A subsequent optical inspection and careful local removal of the film revealed in nearly all cases foreign material at the copper niobium interface (dust particles with Fe and Al elemental composition, drying residuals). This foreign material prevents good adhesion between Cu and Nb resulting in small (<2mm²) or larger (some cm²) blisters. Very small blisters, which did not show up as a Q switch but only as a "hot spot", could also be located by temperature mapping. One such blister covered a surface of 0.03mm² and was located in a region of high r.f. magnetic field. Another one was observed near the iris (.4mm² area).

However, in a cavity without such defects, the adhesion of the Nb layer was good. We proved this by repeatedly plunging this cavity into liquid nitrogen, a treatment which did not change the performance.

Variables determining the surface resistance

The $Q(E_a)$ curve can often be parameterized by

$$Q^{-1}(E_a) = Q_0^{-1} \exp(\alpha E_a) = Q_0^{-1} (1 + \alpha E_a + \dots)$$

Then the surface resistance R_s can be written ($G=270 \Omega$)

$$R_s = R_{s0} + (dR_s/dE_a) E_a + \dots = (G/Q_0) + (\alpha G/Q_0) E_a + \dots$$

 The R_{s0} term is the field independent contribution to R_s . The field dependent terms may be approximated by $R_s' = dR_s/dE_a = \alpha G/Q_0$ as long as the error introduced is lower than the experimental one. This is fulfilled if the change in Q with E_a does not exceed 2.

(a) Heat transfer from the niobium layer to the helium:

Heat transfer conditions of the cavity wall, the niobium copper interface, and the copper helium interface may affect the Q value for increasing field. We therefore measured directly the temperature increase at the inside of a cavity fed by r.f.. We used the cavity as a gas thermometer with a helium gas pressure of $\sim 10^{-5}$ mbar, just below the limit of a gas discharge. In a first step we calibrated the pressure versus the temperature of the bath, the r.f. power being switched off. Then with an r.f. power of 13W applied, no pressure increase was noticed. Thus we concluded, that the average temperature increase at the inner wall was less than 50mK. If the Q decrease corresponding to this r.f. power (from 3×10^9 to 6×10^8) were due to a homogeneous heating of the inner surface, we should have seen a temperature increase of 3.3K. We also figured out that for niobium sheet material (RRR=40 and thickness=2 mm) in boiling helium we would expect $\Delta T = 25$ mK, which corresponds to $R_s' = .3 \text{ n}\Omega/(\text{MV/m})$, much smaller than the observed values.

(b) Bath temperature:

For $T < T_c/2$ the BCS surface resistance R_{s0}^{BCS} follows the relation $R_{s0}^{\text{BCS}} = (A/T) \exp(-\Delta' T_c/T)$ with T_c the critical temperature, Δ' a fit parameter related to the superconducting energy gap, and A another fit parameter. There was no difference in Δ' between sputter coated and sheet metal cavities.

Table 1

Correlation of RRR and surface resistance

Cavity	RRR	$Q^{\text{BCS}}(4.2\text{K})$ A		Δ'	R_{res} [nΩ]
		[10 ⁹]	[10 ⁻⁵ ΩK]		
Nb/Cu ^(a)	7±3	5.2±.6	2±1	2.0±.2	17±11
Nb/Cu	25±5	7±2	1.8±.4	2.1±.1	5.6±.4
Nb	40±5	3.7±.4	2.4±.2	2.0±.2	17±2
Nb	110±10	3.1±.6	6±1	2.2±.2	14±1

(a) Average from several measurements

Figure 3. Bath temperature dependence of R_s' .

The BCS surface resistance at 4.2K showed a difference: $49 \pm 8 \text{ n}\Omega$ for sputter coated and $87 \pm 14 \text{ n}\Omega$ for sheet metal cavities.

We found for sputter coated cavities a bath temperature dependence of the field dependent term as $R_s' [\text{n}\Omega/(\text{MV/m})] = 3.74/(1 - (T/T_c^*)^2)$ with $T_c^* = 5.1 \pm .2 \text{ K}$ (fig. 3).

(c) R.f. magnetic field:

The energy gap Δ decreases for large r.f. fields [3], which for low fields gives rise to $R_s' < .01 \text{ n}\Omega/(\text{MV/m})$, also much smaller than observed.

(d) External static magnetic field:

We cooled down a vertically mounted sheet metal cavity in an arrangement of two coils, by which the earth magnetic field could be compensated to zero. At Geneva this field amounts to $H = 15 \mu\text{T}$ horizontally and $V = 24 \mu\text{T}$ vertically. The horizontal field component generated by the coils was adjusted to compensating, anticompensating and zero action. The vertical compensation remained always constant. The average residual surface resistance $\langle R_{\text{res}} \rangle$ follows a linear relation with H_{ext} (fig.4): $\langle R_{\text{res}}(H_{\text{ext}}) \rangle (\text{n}\Omega) = 1.5 \pm .3 H_{\text{ext}} (\mu\text{T})$. By

Figure 4. Static magnetic field dependence of the residual surface resistance.

taking into account the inhomogeneous distribution of magnetically induced losses we found for the local surface resistance $R_{\text{res}}(H_{\text{ext}}) (\text{n}\Omega) = 5 \pm .1 H_{\text{ext}} (\mu\text{T})$. We also monitored the magnetic flux at the equator perpendicular to the surface by a Hall probe and found that 75% was frozen in.

We performed similar experiments on several sputter coated cavities and found a surprising result. We never

observed a variation of the residual surface resistance with the external static magnetic field, also in those cases, when we had verified from the signal of the rotatable Hall probe that the magnetic flux remained constant at $T_C = 9.2$ K. In addition, we supplied a local field of ~ 100 μ T at the equator. Neither for that experiment nor for all others, when we had deliberately changed the static external magnetic field, did we find the corresponding signal on the temperature map, indicating magnetic losses. At temperatures below T_C the Hall probe signal showed fluctuations, at times, with temperature.

We also studied the dependence of R_S' on H_{ext} (fig. 5). For sheet metal cavities we found for the

Figure 5. Static magnetic field dependence of R_S' .

average $\langle R_S'(H_{ext}) \rangle (n\Omega/(MV/m)) = .23 H_{ext}(\mu T)$. We found a correlation of R_S' to the frequency f , the residual resistivity ratio RRR, and the bath temperature T for different cavities measured in the same external magnetic field $H_{ext}=10\mu T$ (table 2). Again, there was no dependence of R_S' on H_{ext} for all sputter coated cavities.

Table 2

R_S' for sheet metal cavities cooled down in $H_{ext}=10\mu T$

Cavity	T	R_S'
RRR f [MHz]	[K]	[$n\Omega/(MV/m)$]
40 500	2.5	5.9 ± 2.5
	4.2	11.1 ± 1.5
110 500	2.5	$3.2 \pm .9$
	4.2	$3.8 \pm .5$
40 350	2.5	$2.8 \pm .4$
	4.2	$5.2 \pm .3$
110 350	2.5	$1.1 \pm .3$
	4.2	$2.2 \pm .3$

(e) Residual resistivity ratio (RRR):

R_{S0}^{BCS} depends on the RRR via the mean free path of the electrons [4]. We measured the RRR of the sputter coated cavities either from samples prepared similarly as the cavity or from the Q value just above T_C . Since the copper properties are known (see before) the normal-conducting properties of a Nb layer of known thickness can be deduced from the r.f. losses per unit area, i.e. a Q measurement. The RRR of the sheet metal cavities is known from the measurement of the thermal conductivity λ at 4.2K: $RRR=4*\lambda/(W/(mK))$. The BCS surface resistance R_{S0}^{BCS} was determined by means of the MINUIT data reduction routine [5]. Results are listed in table 1 and plotted versus RRR (fig. 6). A general tendency to higher R_{S0}^{BCS} for increasing RRR is visible.

We also investigated the correlation of R_S' and RRR for some sputter coated and sheet metal cavities

Figure 6. RRR dependence of BCS surface resistance at 4.2K and 500MHz.

Figure 7. Histogram of R_S' versus RRR for sputter coated and sheet metal cavities.

(RRR = 110). The results for the latter were corrected by the contribution to R_S' due to the external magnetic field present during cool down. There is an indication of a correlation between R_S' and RRR, too (fig. 7).

Measurements on sputter coated samples

(a) Critical temperature T_C :

T_C was measured by an inductive method. It turned out, that the sputtered samples had a broader transition width than the sheet metal samples. For a diode sputtered sample from the iris region of the cavity the shielding due to supercurrents is increasing even down to 5K (fig. 8). The transition width is narrower for the diode sputtered sample from the equator.

Figure 8. Measurement of T_C for sputter coated Nb/Cu sample.

(b) RRR and conductivity measurements:

RRR values of diode sputtered samples were on the average 9 at the iris and 14 at the equator, the conductivity at room temperature was $2.9 \times 10^6 (\Omega \text{m})^{-1}$ at room temperature, which is comparable to sheet metal. (RRR=6), which is half that of sheet metal.

For the magnetron sputtered sample RRR varied between 3 and 17 depending on sputter parameters. The one with RRR=17 had a conductivity of $6.5 \times 10^6 (\Omega \text{m})^{-1}$ at room temperature, which is comparable to sheet metal. The RRR values determined from samples corresponded to those from the r.f. measurement.

(c) SNMS and XPS-measurements:

To get an idea of the depth profile of impurities Sputtered Neutrals Mass Spectrometry (SNMS) was performed on Nb sheet metal (RRR=40), two diode sputtered samples (D), and on a magnetron sputtered sample (M) (table 3). For an anodized sample we found the expected Nb/O atomic ratio of 2/5 near the surface of the sample. In bulk niobium the C and O impurity content was 10^{-5} and 10^{-4} respectively, as expected. The most prominent impuri-

Table 3

SNMS intensity of prominent impurities

Sample	RRR	Impurity content		
		Nb C	O	Cu
Nb ₂ O ₅	-	10^{-1}	1	-
Nb	40	10^{-5}	10^{-4}	-
Nb/Cu (D)	9	10^{-2}	$10^{-1}-10^{-2}$	$10^{-2}-10^{-4}$
Nb/Cu (D)	14	10^{-2}	10^{-3}	$10^{-1}-10^{-2}$
Nb/Cu (M)	17	$10^{-2}-10^{-3}$	$10^{-2}-10^{-3}$	$10^{-3}-10^{-3}$

ties in the sputtered samples were O, C, and Cu. The relative impurity content ranged from 10^{-1} to 10^{-3} (O), 10^{-2} to 10^{-3} (NbC), and 10^{-1} to 10^{-4} (Cu) in intensity, between the surface and the Nb/Cu interface. The XPS analysis showed, that the oxygen is nearly exclusively bound in the oxide Nb₂O₅, but a small fraction (1%) possibly also in the oxide NbO.

Discussion

The surface resistance of a superconducting cavity depends on several variables and consists of several contributions which we have split into two parts:

$$R_s(T, H_{\text{ext}}, \text{RRR}, f, E_a, \dots) = \sum_i R_{s0}^i(T, H_{\text{ext}}, \text{RRR}, f, \dots) + (\sum_i R_s^i(T, H_{\text{ext}}, \text{RRR}, f, \dots)) E_a + \dots$$

The first part describes the field independent contributions, the second one the field dependent ones.

Losses independent of the r.f. field amplitude

(a) The dependence of R_{s0}^{BCS} on RRR:

R_{s0}^{BCS} is a well known function of T, RRR and f. All our results of R_{s0}^{BCS} (RRR) at 4.2 K and 500 MHz follow the slope of the extrapolated calculations from [6], but differ in the absolute value. This might be explained by more recent extensions of these calculations, which take into account the anisotropic energy gap [7].

(b) External static magnetic field:

The field independent surface resistance R_{s0} increases in a static external magnetic field. As we verified, this can be understood in terms of "frozen in" magnetic flux [8]:

$$\langle R_{s0}^m \rangle = (R_s^n / \gamma) (1/H_{c2}(T)) H_{\text{ext}}$$

with

$$H_{c2}(T) = H_{c20} (1 - (T/T_c)^2).$$

(R_s^n =surface resistance of normalconducting niobium at low temperatures, γ =phenomenological fit parameter close to 1, H_{c2} =upper critical magnetic field).

The striking observation that R_{s0} and R_s' in sputter coated cavities do not depend on H_{ext} may be understood as follows. Uniformly distributed within the sputtered niobium layer exist many small spotlike areas of normalconducting or insulating material, the magnetic flux being concentrated within these "pinholes". However, the fact that the magnetic field inside cannot surpass the critical magnetic field imposes a lower bound on the total area of these spots, which turns out to be big enough to influence the residual surface resistance, if they are normalconducting. This was not observed, an indication for their non conducting character.

R.f. field dependent losses

There are three parameters H_{ext} , RRR, and T, for which we have experimental evidence that they influence the r.f. field dependent term R_s' . Among these three H_{ext} was only relevant for sheet metal cavities, T for sputter coated cavities, and a correlation of R_s' and RRR existed between both species.

(a) Sheet metal niobium cavities:

With the notion of fluxoids carrying a constant quantum of magnetic flux the proportionality of R_s' with H_{ext} can be understood. The number of flux lines increases linearly with H_{ext} . Then, the dependencies of R_s' on T, RRR and f are features of a single flux line. As there is an indication of a f^2 dependence of R_s' , we tentatively propose a heating of the superconducting environment with increasing r.f. field. The reduction of R_s' with lower temperatures and higher RRR and hence thermal conductivity λ can then be understood in terms of reduced local heating.

(b) Sputter coated cavities:

It is surprising that the relation between R_s' and T resembles the one of R_{s0}^m with H_{ext} in sheet metal cavities (with a different fit parameter T_c). This leads us to propose that they are virtually the same, only differing by how the fluxoids are generated. We hypothesize that they are produced by the r.f. field in localized surface regions where the reduced critical fields and temperatures are surpassed ("weak superconducting spots").

Impurity content of sputter coated samples

The O and C impurity content is by one or two orders of magnitude higher than in sheet metal (table 3). NbC is known to be a superconductor with $T_c=6\text{K}$ and a broad transition width of 3K [9]. Dissolved O decreases T_c and H_c of Nb [10] by 1K per atomic % O. If the O were uniformly distributed, T_c would be lower than observed. Thus, it must be concentrated in clusters.

From XPS measurements we know that they contain mainly Nb_2O_5 (and probably also lower oxides and dissolved O). This would explain:

- the absence of a dependence of R_{s0} and R_s' on H_{ext} (resulting from pin holes filled with Nb_2O_5);
- the bath temperature dependence of R_s' indicating a critical temperature $T_c = 5.1$ K due to weak superconducting spots; if they were made up of dissolved oxygen this would correspond to 5 atomic percent (extrapolated from [10]);
- the increase of superconductive shielding for temperatures from T_c down to ~ 5 K;
- the increase of R_s' with RRR, which is proportional to the amount of dissolved interstitial impurities, the most effective being O [10].

For bulk niobium cavities similar weak superconducting spots have been postulated [11].

Conclusion

We performed comparative measurements on sheet metal and sputter coated cavities. With regard to sheet metal cavities sputter coated ones show:

- a higher Q_0 at low field;
- a stronger decrease of Q with field;
- comparable maximum accelerating fields and residual surface resistances;
- a larger scatter in Q_0 and maximum attainable field;
- the absence of a static magnetic field dependence of $Q(E_a)$;

and deduced from sample measurements:

- a broader transition width at T_c ;
- larger impurity contents (O, C, Cu).

We gave a hypothesis to explain these features by "weak superconducting spots" containing impurities (probably O), and also by "pinholes" filled with Nb_2O_5 . The origin of the impurities is not yet understood.

Acknowledgements

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References

- [1] Ph. Bernard et al., LEP note 551, CERN (1986).
- [2] C. Benvenuti et al., IEEE Trans. Magn. MAG-21 (1985) 153.
- [3] H.A. Schwettman et al., IEEE Trans. Nucl. Sci. NS-14 (1967) 336.
- [4] D.C. Mattis and J. Bardeen, Phys. Rev. 111 (1958) 412;
A.A. Abrikosov et al., Sov. Phys. JETP (1959) 182.
- [5] F. James and M. Roos, MINUIT, CERN computer centre program library.
- [6] J. Halbritter, Zeitschr. Phys. 238 (1970) 466;
J. Halbritter, Zeitschr. Phys. 266 (1974) 209.
- [7] R. Blaschke and G. Mueller, private communication.
- [8] B. Piosczyk et al., IEEE Trans. Nucl. Sci. NS-20 (1973) 108.
- [9] G.F. Hardy and J.K. Hulm, Phys. Rev. 93 (1954) 1004.
- [10] W. DeSorbo, Phys. Rev. 132 (1963) 107.
- [11] W. Schwarz and J. Halbritter, Journ. Appl. Phys. 48 (1977) 4618.

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We cooled down a vertically mounted sheet metal cavity in an arrangement of two coils, by which the earth magnetic field could be compensated to zero. At Geneva this field amounts to $H = 15 \mu\text{T}$ horizontally and $V = 24 \mu\text{T}$ vertically. The horizontal field component generated by the coils was adjusted to compensating, anticomensating and zero action. The vertical compensation remained always constant. The average residual surface resistance $\langle R_{\text{res}} \rangle$ follows a linear relation with H_{ext} (fig.4): $\langle R_{\text{res}}(H_{\text{ext}}) \rangle (\text{n}\Omega) = 1.5 \pm .3 H_{\text{ext}} (\mu\text{T})$. By

Figure 4. Static magnetic field dependence of the residual surface resistance.

taking into account the inhomogeneous distribution [5] of magnetically induced losses we found for the local surface resistance $R_{\text{res}}(H_{\text{ext}}) (\text{n}\Omega) = 5 \pm .1 H_{\text{ext}} (\mu\text{T})$. We also monitored the magnetic flux at the equator perpendicular to the surface by a Hall probe and found that 75% was frozen in.

We performed similar experiments on several sputter-coated cavities and found a surprising result. We cooled

down the cavity exposed to a non-zero static magnetic field, as described before, and monitored the magnetic flux at the equator. We found that at $T_c=9.2K$ all flux was frozen in. After warming up above T_c and cooling down again in a different static magnetic field the residual surface resistance R_{res} did not change.

Moreover, we also applied a static magnetic field of $\sim 200\mu T$ at a distinct position at the equator. Again, we did not observe an additional loss distribution on the temperature maps. Thus, we never found a variation of the residual surface resistance with the external static magnetic field (up to $200\mu T$).

We also studied the dependence of R_s' on H_{ext} (fig. 5). For sheet metal cavities we found for the

Figure 5. Static magnetic field dependence of R_s' .

average $\langle R_s'(H_{ext}) \rangle = (n\Omega/(MV/m)) = .23 H_{ext}(\mu T)$. We found a correlation of R_s' to the frequency f , the residual resistivity ratio RRR, and the bath temperature T for different cavities measured in the same external magnetic field $H_{ext}=10\mu T$ (table 2). Again, there was no dependence of R_s' on H_{ext} for all sputter coated cavities.

Table 2

R_s' for sheet metal cavities cooled down in $H_{ext}=10\mu T$

Cavity	T	R_s'
RRR f [MHz]	[K]	[$n\Omega/(MV/m)$]
40 500	2.5	5.9 ± 2.5
	4.2	11.1 ± 1.5
110 500	2.5	$3.2 \pm .9$
	4.2	$3.8 \pm .5$
40 350	2.5	$2.8 \pm .4$
	4.2	$5.2 \pm .3$
110 350	2.5	$1.1 \pm .3$
	4.2	$2.2 \pm .3$

(e) Residual resistivity ratio (RRR):

R_{SO}^{BCS} depends on the RRR via the mean free path of the electrons [6]. We measured the RRR of the sputter coated cavities either from samples prepared similarly as the cavity or from the Q value just above T_c . Since the copper properties are known (see before) the normal-conducting properties of a Nb layer of known thickness can be deduced from the r.f. losses per unit area, i.e. a Q measurement. The RRR of the sheet metal cavities is known from the measurement of the thermal conductivity λ at 4.2K: $RRR=4*\lambda(W/(mK))$. The BCS surface resistance R_{SO}^{BCS} was determined by means of the MINUIT data reduction routine [7]. Results are listed in table 1 and plotted versus RRR (fig. 6). A general tendency to higher R_{SO}^{BCS} for increasing RRR is visible.

We also investigated the correlation of R_s' and RRR for some sputter coated and sheet metal cavities

Figure 6. RRR dependence of BCS surface resistance at 4.2K and 500MHz.

Figure 7. Histogram of R_s' versus RRR for sputter coated and sheet metal cavities.

(RRR = 110). The results for the latter were corrected by the contribution to R_s' due to the external magnetic field present during cool down. There is an indication of a correlation between R_s' and RRR, too (fig. 7).

Measurements on sputter coated samples

(a) Critical temperature T_c :

T_c was measured by an inductive method. It turned out, that the sputtered samples had a broader transition width than the sheet metal samples. For a diode sputtered sample from the iris region of the cavity the shielding due to supercurrents is increasing even down to 5K (fig. 8). The transition width is narrower for the diode sputtered sample from the equator.

Figure 8. Measurement of T_c for sputter coated Nb/Cu sample.

(b) RRR and conductivity measurements:

RRR values of diode sputtered samples were on the average 9 at the iris and 14 at the equator, the conductivity at room temperature was $2.9 \times 10^6 (\Omega \text{m})^{-1}$ (RRR=6), which is half that of sheet metal.

For the magnetron sputtered sample RRR varied between 3 and 17 depending on sputter parameters. The one with RRR=17 had a conductivity of $6.5 \times 10^6 (\Omega \text{m})^{-1}$ at room temperature, which is comparable to sheet metal. The RRR values determined from samples corresponded to those from the r.f. measurement.

(c) SNMS and XPS-measurements:

To get an idea of the depth profile of impurities Sputtered Neutrals Mass Spectrometry (SNMS) was performed on Nb sheet metal (RRR=40), two diode sputtered samples (D), and on a magnetron sputtered sample (M) (table 3). For an anodized sample we found the expected Nb/O atomic ratio of 2/5 near the surface of the sample. In bulk niobium the C and O impurity content was 10^{-5} and 10^{-4} respectively, as expected.

Table 3

SNMS intensity of prominent impurities

Sample	RRR	Nb C	Impurity content O	Cu
Nb ₂ O ₅	-	10^{-1}	1	-
Nb	40	10^{-5}	10^{-4}	
Nb/Cu (D)	9	10^{-2}	10^{-1} - 10^{-2}	10^{-2} - 10^{-4}
Nb/Cu (D)	14	10^{-2}	10^{-3}	10^{-1} - 10^{-2}
Nb/Cu (M)	17	10^{-2} - 10^{-3}	10^{-2} - 10^{-3}	10^{-3} - 10^{-3}

The most prominent impurities in the sputtered samples were O, C, and Cu. The relative impurity content ranged from 10^{-1} to 10^{-3} (O), 10^{-2} to 10^{-3} (NbC), and 10^{-1} to 10^{-4} (Cu) in intensity, between the surface and the Nb/Cu interface. The XPS analysis showed, that the oxygen is nearly exclusively bound in the oxide Nb₂O₅, but a small fraction (1%) possibly also in the oxide NbO.

Discussion

The surface resistance of a superconducting cavity depends on several variables and consists of several contributions which we have split into two parts:

$$R_s(T, H_{\text{ext}}, \text{RRR}, f, E_a, \dots) = \sum_i R_{s0}^i(T, H_{\text{ext}}, \text{RRR}, f, \dots) + (\sum_i R_s^i(T, H_{\text{ext}}, \text{RRR}, f, \dots)) E_a + \dots$$

The first part describes the field independent contributions, the second one the field dependent ones.

Losses independent of the r.f. field amplitude

(a) The dependence of R_{s0}^{BCS} on RRR:

R_{s0}^{BCS} is a well known function of T, RRR and f. All our results of R_{s0}^{BCS} (RRR) at 4.2 K and 500 MHz follow the slope of the extrapolated calculations from [8], but differ in the absolute value. This might be explained by more recent extensions of these calculations, which take into account the anisotropic energy gap [9].

(b) External static magnetic field:

The field independent surface resistance R_{s0} increases in a static external magnetic field. As we verified, this can be understood in terms of "frozen in" magnetic flux [10]:

$$\langle R_{s0}^m \rangle = (R_s^n / \gamma) (1/H_{c2}(T)) H_{\text{ext}}$$

with

$$H_{c2}(T) = H_{c20} (1 - (T/T_c)^2).$$

(R_s^n =surface resistance of normalconducting niobium at low temperatures, γ =phenomenological fit parameter close to 1, H_{c2} =upper critical magnetic field).

The striking observation that R_{s0} and R_s' in sputter coated cavities do not depend on H_{ext} may be understood as follows. Uniformly distributed within the sputtered niobium layer exist many small spotlike areas of normalconducting or insulating material, the magnetic flux being concentrated within these "pinholes". However, the fact that the magnetic field inside cannot surpass the critical magnetic field imposes a lower bound on the total area of these spots, which turns out to be big enough to influence the residual surface resistance, if they are normalconducting. This was not observed, an indication for their non conducting character.

R.f. field dependent losses

There are three parameters H_{ext} , RRR, and T, for which we have experimental evidence that they influence the r.f. field dependent term R_s' . Among these three H_{ext} was only relevant for sheet metal cavities, T for sputter coated cavities, and a correlation of R_s' and RRR existed between both species.

(a) Sheet metal niobium cavities:

With the notion of fluxoids carrying a constant quantum of magnetic flux the proportionality of R_s' with H_{ext} can be understood. The number of flux lines increases linearly with H_{ext} . Then, the dependencies of R_s' on T, RRR and f are features of a single flux line. As there is an indication of a f^2 dependence of R_s' , we tentatively propose a heating of the superconducting environment with increasing r.f. field. The reduction of R_s' with lower temperatures and higher RRR and hence thermal conductivity λ can then be understood in terms of reduced local heating.

(b) Sputter coated cavities:

It is surprising that the relation between R_s' and T resembles the one of R_{s0}^{BCS} with H_{ext} in sheet metal cavities (with a different fit parameter T_c). This leads us to propose that they are virtually the same, only differing by how the fluxoids are generated. We hypothesize that they are produced by the r.f. field in localized surface regions where the reduced critical fields and temperatures are surpassed ("weak superconducting spots").

Impurity content of sputter coated samples

The O and C impurity content is by one or two orders of magnitude higher than in sheet metal (table 3). NbC is known to be a superconductor with $T_c=6\text{K}$ and a broad transition width of 3K [11]. Dissolved O decreases T_c and H_c of Nb [12] by 1K per atomic % O. If the O were uniformly distributed, T_c would be lower than observed. Thus, it must be concentrated in clusters.

From XPS measurements we know that they contain mainly Nb_2O_5 (and probably also lower oxides and dissolved O). This would explain:

- the absence of a dependence of R_{s0} and R_s' on H_{ext} (resulting from pin holes filled with Nb_2O_5);
- the bath temperature dependence of R_s' indicating a critical temperature $T_c = 5.1$ K due to weak superconducting spots; if they were made up of dissolved oxygen this would correspond to 5 atomic percent (extrapolated from [10]);
- the increase of superconductive shielding for temperatures from T_c down to ~ 5 K;
- the increase of R_s' with RRR, which is proportional to the amount of dissolved interstitial impurities, the most effective being O [12].

For bulk niobium cavities similar weak superconducting spots have been postulated [13].

Conclusion

We performed comparative measurements on sheet metal and sputter coated cavities. With regard to sheet metal cavities sputter coated ones show:

- a higher Q_0 at low field;
- a stronger decrease of Q with field;
- comparable maximum accelerating fields and residual surface resistances;
- a larger scatter in Q_0 and maximum attainable field;
- the absence of a static magnetic field dependence of $Q(E_a)$;

and deduced from sample measurements:

- a broader transition width at T_c ;
- larger impurity contents (O, C, Cu).

We gave a hypothesis to explain these features by "weak superconducting spots" containing impurities (probably O), and also by "pinholes" filled with Nb_2O_5 . The origin of the impurities is not yet understood.

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References

- [1] Ph. Bernard et al., LEP note 551, CERN (1986).
- [2] C. Benvenuti et al., IEEE Trans. Magn. MAG-21 (1985) 153.
- [3] Ph. Bernard et al., Nucl. Instr. Meth. 190 (1981) 257.
- [4] H.A. Schwettman et al., IEEE Trans. Nucl. Sci. NS-14 (1967) 336.
- [5] Ph. Bernard et al., Nucl. Instr. Meth. 206 (1983) 47.
- [6] D.C. Mattis and J. Bardeen, Phys. Rev. 111 (1958) 412;
- [7] A.A. Abrikosov et al., Sov. Phys. JETP (1959) 182.
- [8] F. James and M. Roos, MINUIT, CERN computer centre program library.
- [9] J. Halbritter, Zeitschr. Phys. 238 (1970) 466;
- [10] J. Halbritter, Zeitschr. Phys. 266 (1974) 209.
- [11] R. Blaschke and G. Mueller, private communication.
- [12] B. Piosczyk et al., IEEE Trans. Nucl. Sci. NS-20 (1973) 108.
- [13] G.F. Hardy and J.K. Hulm, Phys. Rev. 93 (1954) 1004.
- [14] W. DeSorbo, Phys. Rev. 132 (1963) 107.
- [15] W. Schwarz and J. Halbritter, Journ. Appl. Phys. 48 (1977) 4618.