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CERN STUDIES ON NIOBIUM-COATED 1.5 GHZ COPPER CAVITIES

C. Benvenuti, S. Calatroni [#], M. Hakovirta, H. Neupert, M. Prada and A.-M. Valente,
CERN, Geneva, Switzerland

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Studies at CERN on niobium-coated 1.5 GHz superconducting cavities are aimed at understanding and possibly curing the causes of the residual resistance increase observed when increasing the accelerating gradient above 15 MV/m. Amongst the possible causes, the surface defects and roughness, the grain size, the hydrogen content and the thermal effects are currently being investigated. The present status of understanding, together with some recent results on high field operation, are presented and discussed.

1 INTRODUCTION

CERN studies of the surface resistance of sputtered niobium films at 1.5 GHz have shed a new light on the various mechanisms responsible for their radiofrequency losses [1]. The films, 1.5 μm thick, have been produced with a variety of different coating parameters and procedures, which are here briefly summarised. The copper surface prior to coating may be prepared either by chemical polishing (CP) or by electropolishing (EP). The coating is then performed either on the passivated copper surface (standard coating), or after the elimination of the passivation layer (oxide-free coating). The latter procedure may also be carried out in two ways, either by sputter etching or by coating a pure copper underlayer, both resulting in identical niobium coatings in all respects.

The choice of EP and standard coatings, using krypton as sputter gas, has been made to optimise the fluxon losses and the residual surface resistance up to 10 MV/m [2]. However, recent experiments at high gradient [3] have put in evidence a steep increase of the residual resistance for accelerating fields larger than 15 MV/m (Fig. 1). Therefore, the adopted set of optimised parameters has to be questioned.

2 RESIDUAL RESISTANCE

The origin of the residual resistance is a long-standing problem, both for bulk niobium and for niobium-coated cavities, which is still far from being fully understood. No single dominant source of residual resistance is expected, thus several hypotheses are being specifically addressed to identify its causes in film cavities, and possibly reduce its extent. To exclude that the measured results are in reality only an instrumental effect, some tests have been carried out to verify that the heating of the small (60 litres) liquid helium bath used for this studies is not a
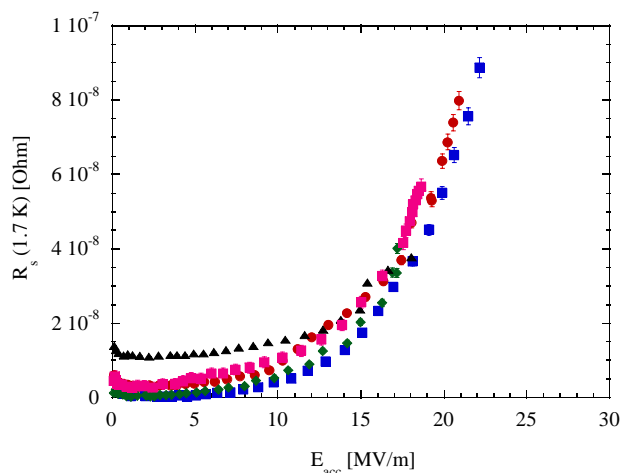

Figure 1: Surface resistance at 1.7 K of several Nb-coated cavities.

relevant issue. Fig. 2 illustrates a measurement carried out on a cavity with the usual RF test bench, and in one of the LEP cryostats. Here the large helium volume (>1 m³) guarantees perfect temperature stability for several minutes, even when operating at 250 W of RF power, compared to the 100 W of the usual test bench. The behaviour of the cavity is linearly extended to higher field (28 MV/m in this case), confirming the absence of instrumental effects and also that the maximum gradient of film cavities can be increased by adequate surface cleanliness, as much as it happens for bulk niobium.
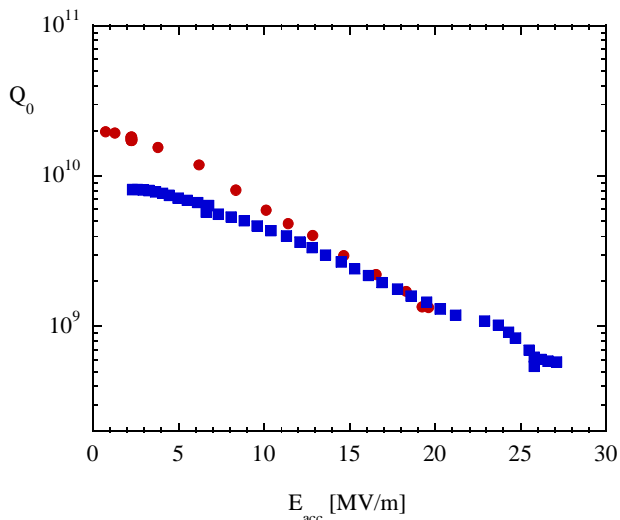

Figure 2: Q_0 measured for the same cavity with the standard RF apparatus (at 1.7 K, dots) and in a large LEP cryostat (at 2.0 K, squares).
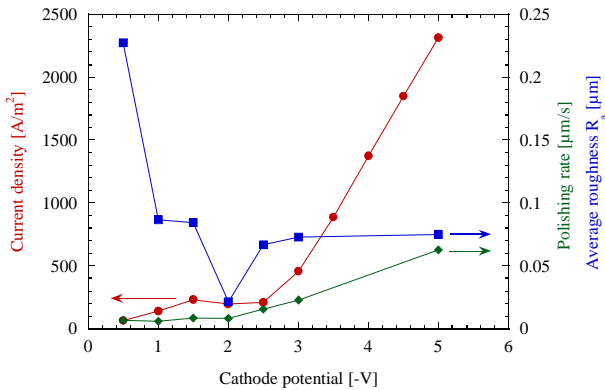
[#] sergio.calatroni@cern.ch

Figure 3: Current density (dots/left axis), average roughness (squares/right axis) and polishing rate (diamonds/right axis) for the standard EP bath, obtained on laboratory samples.

2.1 Surface Defects and Roughness

The residual resistance is usually described by the quantities R_{res}^0 and R_{res}^1, which refer to a linear fit of the low-field residual resistance, being the former the zero-field value and the second the slope. It has already been shown [4, 5] that niobium coatings on electropolished copper cavities have considerably lower both R_{res}^0 and R_{res}^1 compared to standard CP. EP provides a consistent reduction in the density of film defects, which have typically a diameter of 0.3 μm. Although clearly visible on SEM micrographs, this effect has been quantified by measuring the conductance for helium gas of bare films, obtained by chemically dissolving the substrate, within an accuracy of ±30%. The fraction of film surface permeable to helium gas is 4.4 ppm for a chemically polished substrate, and decreases to 0.2 ppm for an electropolished substrate, when the coating is performed in class 100 dust-free conditions. Simple calculations comparing the surface resistance of niobium with that of the copper area that might be exposed through the holes suggests that this effect should give only a very minor contribution to the residual resistance.

EP results also in much-improved average roughness of the copper surface, which decreases down to 0.02 μm from 0.2 μm for CP. These values, achieved on small laboratory samples, are probably the best attainable
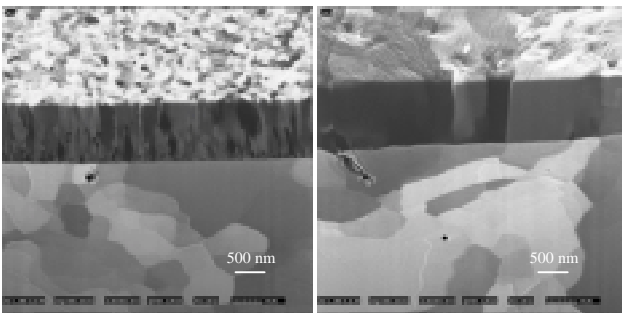

Figure 4: Cross sectional FIB images of niobium films on oxidised (left) and oxide-free (right) copper substrates

within the limits of the chosen techniques. The optimum operating window for the EP parameters is rather narrow, as illustrated in Fig. 3, and is very sensitive to the geometry of the polishing apparatus. In the low-voltage region before the plateau, it has been identified that ~1 nm of $Cu(OH)_2$ is produced on the copper surface after a 20 μm polishing, from which hydrogen may migrate into the niobium film. This hydrogen may have a deleterious effect on the RF performance, as illustrated in Section 2.4. It has been verified on test samples mounted on a dummy cavity that large regions of the cavity are not polished in the best conditions, when using a simple quasi-homothetic cathode. Although EP cavities perform already better than CP cavities, there is room for improving the EP uniformity, which might result in improved performance and reduced hydrogen contamination. Numerical simulations are being carried out to optimise the cathode shape, and in parallel the EP bath is being investigated for stability and composition tolerances.

The film roughness can also be influenced by the coating process itself. In the standard coating process, there is a wide variation of the incidence angle of the niobium atoms from equator to iris. This, possibly coupled with a certain roughness of the copper substrate itself, results in a slightly rougher topography at the iris, as seen by SEM imaging. A wide study has been undertaken showing that the standard coating parameters, optimised in the past for obtaining the optimum RRR combined with the lowest fluxon losses, guarantee also a low roughness and good film uniformity. The sputter gas pressure, the sputtering potential, the coating temperature have been widely scanned. The outcome is, as expected, that higher temperature or higher potential favour larger grains, which have a rougher topography. An increase of pressure reduces their size and can reduce the differences between equator and iris, although this happens only at pressures for which the grains start to be non-connected, and the topography rougher.

2.2 Grain Size

The grain size of standard niobium coatings on an oxidised copper substrate is on average (100±10) nm, while the grain size of coatings on oxide-free copper is of the order of several μm, similar to the copper grain size. Fig. 4 is a Focussed Ion Beam image of a cut through the surface of two reference films, illustrating the dramatic difference in grain size, and the replication of the copper grains by the niobium grains in the oxide-free case. This size replication is the result of a heteroepitaxial growth mechanism, which favours the growth of some niobium planes parallel to particular copper planes for which there is good lattice match. These families of planes have been measured with TEM and EBSD mappings and are summarised in Table 1, confirming earlier results obtained with the X-ray diffraction technique. [6, 7].

The larger grain size of oxide-free films is reflected by the increase of RRR from 11.5 to 29, verified on samples

cut from Ar-sputtered cavities. Numerical simulations based on the model [8] and on known film data [9] confirm that a ~1 μm grain size, together with the known reduction in trapped gas [1], can explain the RRR increase, keeping all the other film characteristics constant.

Table 1: Nb/Cu orientations

Lattice planes	Azimuthal orientation
Nb(110) // Cu(010)	Nb[111] // Cu[011]
Nb(110) // Cu(111)	Nb[111] // Cu[110]
Nb(100) // Cu(110)	Nb[001] // Cu[001]

The grain size of oxide-free films does not positively influence the RF performance. The low-field average residual resistance of films deposited with Ar or Kr as sputter gas on oxide-free EP substrates is compared to films deposited on oxidised substrates in Fig. 5, together with the same data for CP substrates. The R_{res}^0 and the R_{res}^1 term are consistently larger for oxide-free films than for standard ones. The performance is thus clearly not dominated by a simple grain-boundary Josephson-dissipation mechanism [10], which would require the opposite. The above average values are however made for films measured up to about 10 MV/m. No information is yet available on the high-field behaviour of oxide-free films, to be compared for example with those of Fig. 1. It cannot be excluded at present that at larger field the grain-boundary dissipation may play a role, thus favouring a larger grain size.

A direct consequence of the difference in grain size and in purity of the two films is a large difference in the upper critical field $H_{c2}(0)$ [11]. These values measured with a SQUID magnetometer are (1.15±0.1) T for argon-coated standard films and (0.73±0.05) T for oxide-free films. The ratio of the two values is in excellent agreement with the theoretical prediction based on the resistivity-dependent Ginzburg-Landau parameter κ. H_{c1} values, typically more difficult to obtain with good precision, have been measured with the trapped flux method. The results, (85±5) mT and (31±3) mT respectively at 1.8 K are of difficult interpretation, since they are opposite to what could be expected from the H_{c2} values.

The residual resistance has often been correlated with H_{c1}, in particular for higher-T_c superconductors [12]. Actually, an H_{c1} value of 85 mT for standard Nb films corresponds to 18.7 MV/m of accelerating field in a cavity, close to the inflexion value shown in Fig. 1. No reliable high-field measurements are yet available to verify if a similar correlation holds true also for oxide-free films.

Another quantity that one may expect to be influenced by the critical magnetic field is the RF-field dependence of the BCS surface resistance, which is usually well described by a term $(1+\alpha E^2)$. The α coefficient is 8.4×10^{-3} within experimental error for both types of films, at least when argon-sputtered films measured up to 10 MV/m are considered, for which enough statistics exists. This coefficient is similar also in bulk niobium [1], suggesting that the critical magnetic field does not play a role in the above dependence.

2.3 Hydrogen content: studies

The higher residual resistance of films having a larger grain size and better purity may be due to the effect of hydrogen. In bulk niobium [13, 14] hydrogen is known to be a potentially important cause of residual resistance. It is the result of the formation of ε–hydride precipitates having typical dimensions ranging between a few tens of nm and a few μm [15], occurring in the range of temperatures around 100 K. Such a formation can be prevented by simply crossing the temperature for the ε–phase formation fast enough to make sure that the dissolved hydrogen atoms have no time to diffuse to the niobium surface, before their mobility is reduced to negligibly small values. It can also be prevented by using niobium of low RRR, in which case the hydrogen atoms are trapped by defects and impurities before having a chance to migrate. The net contribution to the residual resistance, often referred to as the "hydrogen disease", is therefore the result of many factors and sometimes difficult to predict.

In the case of films, additional complications may be expected, such as effects related to the presence of the copper substrate. In order to study the effect of hydrogen on the surface resistance, a set of experiments was performed on cavities prepared under different condition Some of the results have already been reported in [4] and [16], and are here briefly summarised.

The measurement of the equilibrium pressure of hydrogen as a function of temperature (isosteric curve) has been used to deduce its binding energy and concentration in the niobium lattice. The Sievert's equation $p_{H_2} \propto (c_H)^2 exp(-2E_H/kT)$, where E_H is the activation energy of single hydrogen atoms and c_H their concentration, ideally applies to reversible desorption from non-oxidised surfaces. The experiment has been
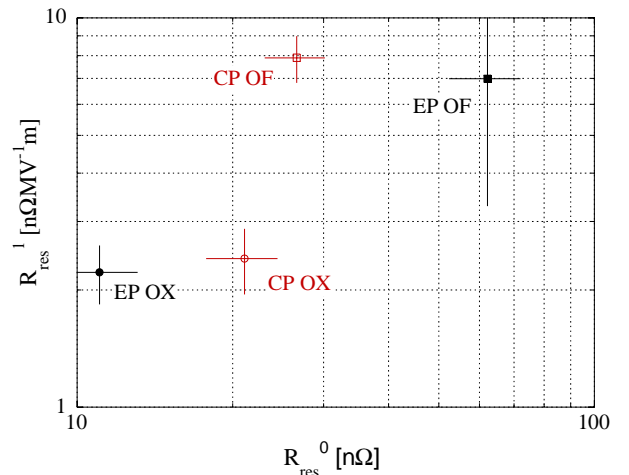

Figure 5: Average low-field residual resistance and its slope for films on oxidised (OX) and oxide-free (OF) substrates. The averages for OF films are carried out only on a very limited number of coatings.

performed on the double-cathode coating system [1], where the possibility of retracting the sputtering cathode from the coating position into a separate chamber without breaking the vacuum allows for the correct execution of these measurements. The cavities have been heated at a rate of 50 ºC/h up to 350 ºC and cooled down to room temperature at the same rate, and the hydrogen pressure has been monitored with a set of calibrated gauges. Care was taken in keeping the remaining of the vacuum system at room temperature to prevent an increase of the background signal. Since the measurement was performed in static conditions a liquid nitrogen trap has been used to reduce the partial pressure of the methane produced by the filament of the ionisation gauges. Subsequent injections of known quantities of H_2 into the coating system, which are completely absorbed by the film, and the measurement of the relative isostere, allow for the extrapolation of the original H_2 content of the film before loading. The results are shown in Table 2, together with the values of activation energy. The latter should be compared with a reported literature value of -0.4 eV/at for ultra-pure niobium [17]. At the first heating up, the binding energy is however much larger, namely -0.65±0.02 eV/at for standard films and -0.60±0.02 eV/at for oxide-free films [4]. Subsequent heating cycles reproduce then exactly the downward part of the first, indicating that hydrogen-trapping sites have been annealed out. It is not excluded that sites with higher binding energy may exist, which cannot be eliminated by heating. In this case, some of the hydrogen trapped in the film may not be accounted for in these measurements.

Impurities such as oxygen are possible traps for hydrogen, and its presence at the niobium/copper interface could contribute to larger trapping in the standard films, and reduced hydrogen release. It has been verified by GDOES profiling that the oxygen at the niobium/copper interface vanishes below sensitivity level after a 350 ºC heating, diffusing into the film and possibly providing additional trapping.

Table 2: Hydrogen content measured with different techniques

Coating	H content (isosteres) [at%]	Binding energy [eV/at]	H content (thermal outgassing) [at%]	Peak temperature (thermal outgassing) [ºC]	H content (thermal outgassing – passivated Nb) [at%]	Peak temperature (thermal outgassing - passivated Nb) [ºC]
Argon, standard	0.04±0.02	-0.34±0.01			0.22±0.02	296±3 ºC
Argon oxide-free	0.1±0.02	-0.32±0.01				
Krypton, standard			0.08±0.02	295±3 ºC	0.22±0.02 (0.14±0.02 2nd cycle)	314±3 ºC (337±3 ºC)
Krypton, oxide-free			0.098±0.02	265±3 ºC	0.20±0.02	312±3 ºC

An independent approach for measuring the hydrogen content is thermal outgassing. Different cavities were outgassed at 350 ºC for several hours, while connected to a pumping system of calibrated throughput. Typical measurements are reported in Fig. 6. After a first peak, taking place typically between 290 ºC and 340 ºC, the hydrogen pressure reaches a constant equilibrium value, which is only function of temperature. The hydrogen content has been calculated by integrating the area below the peak, and the results for different types of cavities are given in Table 2. When performing this for hydrogen loaded films, there is reasonable agreement between the known quantity injected and the one measured by outgassing, when the hydrogen content of the non-loaded film is taken into account.

The same measurement has also been performed for some cavities immediately after coating, before breaking the vacuum to passivate the surface with the usual dry air injection. The peak temperature is in this case shifted to lower temperatures, as reported in Table 2, probably because of the absence of the oxide barrier at the surface, and is about 30 ºC lower for oxide-free films, in agreement with a possible less-efficient trapping. The hydrogen left after the outgassing has been measured in an ad-hoc experiment with an isosteric cycle, resulting in less than 0.01 % for both types of films.

It is reasonable to conclude, by comparing the data of Table 2, that hydrogen is for about one half dissolved in the films during deposition, either from the coating process itself or from the copper, and for another half introduced when exposing the surface to air. In this respect, the usual 24-hours dry air passivation is not sufficient to prevent the formation of hydroxides. Independent measurements by XPS [18] and ERDA [19] suggest that this quantity lies between 0.3 and 1.3 monolayer to be compared with the 2.5 monolayer that can be extrapolated from our experiments. The hydrogen in the bulk of the film is however below the sensitivity threshold for these techniques.

It is noteworthy that a cavity outgassed a second time after air exposure, produced at the second cycle a quantity of hydrogen that is roughly half of that produced at the first cycle, and peaking at a higher temperature. This suggests that both the bulk and the surface hydrogen of the film are emptied while heating, but the latter builds up again after air exposure, and is energetically more trapped
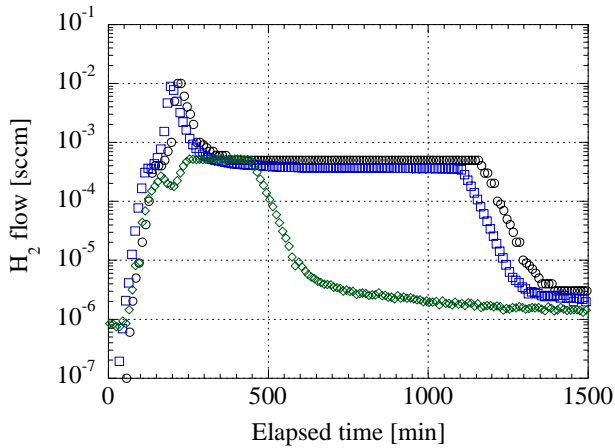
Figure 6: Hydrogen flow released during the outgassing of two cavities, standard (black) and oxide-free (blue), together with the background flow released by a bare copper cavity (green). The plateau at 350 ºC was of shorter duration for the latter cavity, and results in an earlier drop of the hydrogen flow.

because probably of a larger oxygen content in the film. The latter hypothesis is confirmed by T_c measurements on cavities and on ad-hoc samples, showing a reduction of ~0.3 K after first heating which is in qualitative agreement with the diffusion of a few ‰ of oxygen inside the film.

Bare copper cavities were also outgassed to identify whether the copper substrate could be the source of the hydrogen contained in the films, and in particular to identify the origin of the high-temperature plateaux. In Fig. 6 is also reported a thermal cycle performed on a spun copper cavity. The absence of a peak indicates that the film is indeed responsible for its appearance. However, the equilibrium value at constant temperature is the same as the one obtained for coated cavities, suggesting that the origin of this hydrogen is the copper itself. An experiment performed keeping the outside of a bare copper cavity also under vacuum suggests that the
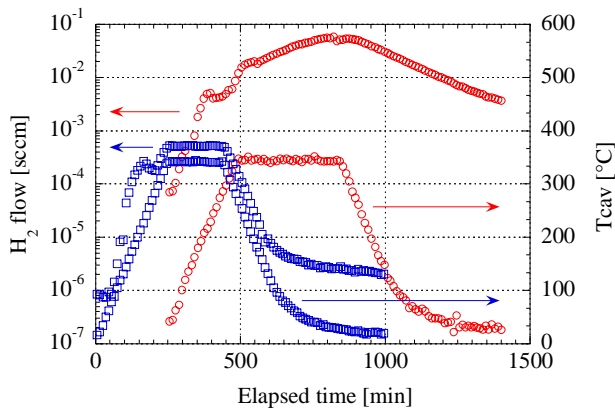

Figure 7: Hydrogen flow (left) released during the outgassing of two bare copper cavities, spun (squares) and hydroformed (circles), together with the relative temperature cycle (right)

hydrogen is not coming from the outside ambient air. Hydroformed copper cavities, which were used in an earlier part of this study instead of the spun type in use now, have been found having a hydrogen content some hundred times higher, as illustrated in Fig. 7.

2.4 Hydrogen: effects on RF performance

It was already discussed in [16] that loading 1.4% at. hydrogen in oxide-free films has a disastrous effect on their residual resistance, while this is not so dramatic in the case of standard films. Considering the results mentioned in the previous paragraph, one could conclude that hydrogen is more likely to form hydrides in oxide-free films, due to the lower density of trapping sites. It is probably not such a far-fetched assumption to believe that this can also justify the larger residual resistance measured for conventional oxide-free films, when compared with standard films (see Fig. 4). The same conclusion may also justify the very large residual resistance measured for oxide-free films deposited on hydroformed cavities, compared to spun ones [16].

It is then obvious to look for ways of reducing the hydrogen content of the niobium coatings. The first straightforward solution of outgassing coated cavities at 350 ºC before venting to air resulted in residual resistances not lower than usual values. Milder outgassings at 150 ºC, the coating temperature, did not prove beneficial either. Another approach, i.e. to use a gettering underlayer, namely titanium, did not prove effective, and the niobium films behaved in all respects like films coated on passivated copper [11].

Along this same line, all possible sources of hydrogen during coating have been reduced. The largest source is probably the sputtering cathode itself, which has thus been outgassed at 600 ºC for several hours in-situ prior to coating. This temperature is sufficient to release at least 90% of the hydrogen that might be present in the niobium [20]. The hydrogen released at first outgassing, if entirely absorbed by a film, would amount to about 1 % atomic concentration. Subsequent outgassings release progressively lower amounts, at least by one order of magnitude at the second cycle and more for further cycles, indicating that the cathode does not reabsorb hydrogen over time. Several cavities of both types have been coated after cathode outgassing, but no dramatic improvement has been measured in terms of RF performance. In practice, the reproducibility of the cavity coating process is within some 10 nΩ for R_{res}^0 and 1 nΩ MV^{-1}m^{-1} for R_{res}^1, and the effect of cathode outgassing and of the other procedures mentioned above is smaller than this spread. However, most of the best results have been obtained by following this procedure, which has then become a standard whenever employing a new cathode.

2.5 Thermal Effects

A long-debated issue in superconducting cavities [21] is the possibility that the overall thermal impedance of the

cavity wall leads to a temperature increase of the inner surface exposed to the RF, resulting in an increased surface resistance. This issue is further complicated in thin film cavities, since the thermal impedance across the film/substrate interface is unknown and adds to the Kapitza impedance at the copper/helium boundary and the thermal conductivity of the copper wall (neglecting the thermal impedance of the thin niobium film itself). The Kapitza impedance at the Cu/He interface has been measured by several authors [22], and at 1.8 K ranges between 4700 and 7000 W/Km^2. This is a value similar to the value of 4000÷6500 W/Km^2 that has been measured for bulk niobium [23]. Even the purest bulk niobium used for cavity manufacturing suffers however from a large thermal impedance across the cavity wall, which has the same order of magnitude as the Kapitza impedance and is about a factor 10 larger than for OFE copper.

The film/substrate thermal barrier is however more difficult to evaluate. Some first-principle considerations, taking into account the speed of sound in both materials, give the quite large critical angle for acoustic phonons of 77°. The mismatch of acoustic impedance, calculated with the product of the material density times the speed of sound assures an energy transmission probability larger than 98% within the critical angle. The contribution of the electrons to the thermal exchange is probably negligible, as it is with any superconductor, but the overall thermal transmission should be better than for bulk niobium cavities.

A direct measurement of the temperature of the film would in principle be possible by adsorbing on its surface some helium gas (possibly less than 1 monolayer) and measuring its equilibrium pressure during RF operation, which is then related to the surface temperature. This measurement has been carried out with a Residual Gas Analyser, having a background He pressure in the range of 10^{-11} mbar and by injecting gas quantities resulting in pressures 10 to 100 times larger. No increase of pressure has been recorded during operation with RF field. The measurement was however strongly hampered by the long times required for reaching an equilibrium, and the comparatively short times for the heating of the helium bath, or for loosing its level, both resulting in huge pressure rises.

To overcome this difficulty, a different approach has been attempted. The surface resistance has been transformed into surface temperature by numerical calculations, under the assumption that its increase is fully justified by the BCS mechanism. The temperature increase has then been correlated with the dissipated RF power. The result, for the same cavity of Fig. 1, is reported in Fig. 8. If the cavity dissipation were dominated by a Kapitza-like effect, a linear dependence would be expected, i.e. very different from the square-root-like behaviour shown in the figure. For comparison, the temperature increase at high-power is roughly a factor 10 larger than what could be estimated for bulk niobium.

Along a further line of experiments, two 1.3 GHz bulk niobium cavities which had reached a 1.7 K quench field
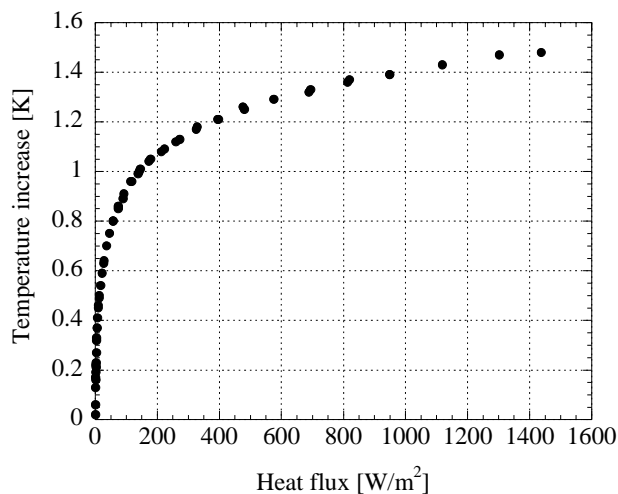

Figure 8: Calculated temperature increase as a function of the power dissipation for a given film cavity.

above 30 MV/m have been coated with a niobium film. Although having normal film R_{res}^0 and R_{res}^1 values at low field, the quench limit has been reduced to about 10 MV/m. The results are still preliminary and need further confirmation, but they suggest that a strong dissipation may happen in some part of the film, which due to the poor thermal conductivity of the niobium substrate induces a quench. The higher thermal conductivity of the copper substrate may prevent quenching, but not the enhanced losses, which probably result in the observed Q degradation at high fields.

3 CONCLUSION

Several causes for the sudden increase of residual resistance above 15 MV/m have been investigated. Although clear conclusions have not yet been reached, surface defects seem to play a dominant role. Investigations are under way to address part of the problem by optimising the EP process. It is hoped that this will extend to high fields the excellent results already obtained at lower field: this may be of great interest for several applications.

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6 REFERENCES

[1] C. Benvenuti, S. Calatroni, I.E Campisi, P. Darriulat, M.A. Peck, R. Russo, A.-M. Valente, Physica C 316 (1999) 153-188

[2] V. Arbet-Engels, C. Benvenuti, S. Calatroni, P. Darriulat, M.A. Peck, A.-M. Valente, C.A. Van't Hof, NIM A 463 (2001) 1-8

[3] S. Calatroni in: Proc. of the 9th Workshop on RF Superconductivity, B. Rusnack ed. (DOE, Los Alamos, 2000) 6-9

[4] C. Benvenuti, S. Calatroni, P. Darriulat, M.A. Peck, A.-M. Valente, C.A. Van't Hof, Physica C 351 (2001) 412-428

[5] A.-M. Valente in: Proc. of the 9th Workshop on RF Superconductivity, B. Rusnack ed. (DOE, Los Alamos, 2000) 550-554

[6] M.A. Peck, PhD. Thesis, Vienna Technical University, 2000

[7] C. Benvenuti, S. Calatroni, P. Darriulat, M.A. Peck, A.-M. Valente, "Influence of the nature of the substrate on the growth of superconducting niobium films", CERN Internal Report EST-2000-005-SM, submitted for publication to Thin Solid Films

[8] G. Reiss, J. Vancea, H. Hoffmann, J. Phys C 18 (1985) L657

[9] C. Benvenuti, S. Calatroni, G. Orlandi, Physica B 197 (1992) 72-83

[10] C. Attanasio, L. Maritato, R. Vaglio, Phys. Rev B 43 (1991) 6128

[11] C. Benvenuti, S. Calatroni, P. Darriulat, M.A. Peck, A.-M. Valente, Physica C 351 (2001) 429-437

[12] T. Kaiser, W. Diete, M. Getta, M.A. Hein, G. Müller, M. Perpeet, H. Piel in: Proc. of the 8th Workshop on RF Superconductivity, V. Palmieri and A. Lombardi Eds. (INFN, Padova, 1998) 925

[13] B. Bonin and W. Roeth, Proc. 5th Workshop on RF Supercond., ed. D. Proch, (DESY-M-92-01) (DESY, Hamburg, 1991) p. 210, and references therein.

[14] S, Isagawa, J. Appl. Phys. 51 (1980) 4460 and 6010

[15] T. Schober, Scripta Metallurgica 7 (1973) 1119

[16] C. Benvenuti, S. Calatroni, I.E Campisi, P. Darriulat, M.A. Peck, R. Russo, A.-M. Valente, IEEE Trans. Appl. Supercond. 9 (1999) 900

[17] G. Hörz, H. Speck, E. Fromm, H. Jehn, Gases and Carbon in Metals, Pt. VII (2) (Fachinformations-zentrum, Eggenstein-Leopoldshafen, 1981) pp. 4-34

[18] Adriana Daccà, PhD Thesis, Genova University, 2000: 0.3 ML of water (hydrogen containing compounds, Nb-OH)

[19] Sergei Mikhailov – CAFI Neuchatel (ERDA, Elastic Recoil Detection Analysis, measurement report 11/1998): H <0.6% in bulk, 2% in the first 20 nm

[20] M. Hakovirta, Proceedings of this conference

[21] P. Boccard, P. Kneisel, G. Müller, J. Pouryamout, H. Piel in: Proc, of the 8th Workshop on RF Superconductivity, V. Palmieri and A. Lombardi Eds. (INFN, Padova, 1998) 1094

[22] D. Camacho, S. Chevassus, C. Policella, J.M. Rieubland, G. Vandoni, R. van Weelderen, CERN LHC Project Report 232

[23] J. Amrit, M.X. Francois. J. Low Temp. Phys. 119 (2000) 27-40