

RF-LOSSES OF SUPERCONDUCTING NIOBIUM-CAVITIES COATED WITH Nb<sub>2</sub>O<sub>5</sub>

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Abstract

Measurements on anodized niobium-cavities have been carried out in the GHz-region. Residual surface resistances of 10<sup>-8</sup> Ohm at S-band frequencies and magnetic peak fields of 80 mT at X-band frequencies were achieved without firing the cavities in an UHV-furnace. Besides, the dense coating of niobium with Nb<sub>2</sub>O<sub>5</sub> seems to prevent ageing of the superconducting surface.

I. Introduction

High Q superconducting cavities show ageing effects and electron emission at high RF-field strengths, effects, which limit the practical application of superconducting cavities. The coating of the superconducting surface with a dense dielectric not only could protect the surface but also could reduce the electric field strength at the metal surface, possibly lowering the number of the emitted electrons.

These ideas have been confirmed by anodizing massive niobium, which results in a dense layer of amorphous Nb<sub>2</sub>O<sub>5</sub>. The surprising result that anodizing gave improvements<sup>1), 2)</sup> as well in residual losses as in breakdown fields seemed to be in contrast to the opinion that one must always avoid oxidation of the metal surface and showed that the knowledge about surfaces in relation to RF-residual losses<sup>3)</sup> and limits at high field levels<sup>4)</sup> is not very good. To clear up the questions mentioned above, we have started an experimental program of additional measurements on anodized niobium cavities, in S-band cavities at Karlsruhe and in X-band cavities at Siemens-Research Laboratories, Erlangen. In part II of this paper the preparation of the cavities and the experimental results are given, which will be discussed in part IV based on a consideration of the oxidation of niobium in part III.

II. Experimental Results

In the following we have omitted bad results which were believed to be due to failure in preparation. The experimental set-ups are described elsewhere<sup>1), 2)</sup>. The measurements at Karlsruhe are carried out with a solid niobium cavity, the same one as described in<sup>2)</sup>, but electron-beam welded at the joint. Before welding the cavity was electropolished<sup>6)</sup> with a removal of about 100 μ of niobium from the surface and hydro-

gen-degassed. Further preparation after electron-beam welding was very weak chemical polishing in a solution of 60 % HNO<sub>3</sub>/40 % HF by volume<sup>5)</sup> at about -15°C for 1 minute, followed by rinsing in distilled water and anodizing through the coupling holes in a solution of 12.5 % NH<sub>3</sub> at a starting current density of 0.5 mA/cm<sup>2</sup> for about 20 minutes. Subsequently the cavity was rinsed in distilled water and methanol before assembly. Results are shown in Table I.

Mode	test 1 <sup>1)</sup>	test 2 <sup>2)</sup>	test 3 <sup>1)</sup>	test 4 <sup>3)</sup>
TM <sub>010</sub> 2.16 GHz 302 Ohm	0.97 × 10 <sup>10</sup> 21 mT	0.44 × 10 <sup>10</sup> 17 mT	1.4 × 10 <sup>10</sup> 6 mT	0.56 × 10 <sup>10</sup> 7.5 mT
TE <sub>111</sub> 2.19 GHz 322 Ohm	2.73 × 10 <sup>10</sup> 26 mT	1.17 × 10 <sup>10</sup> 25 mT	2.85 × 10 <sup>10</sup> 13 mT	0.91 × 10 <sup>10</sup> 10 mT
TM <sub>011</sub> 2.60 GHz 273 Ohm	0.75 × 10 <sup>10</sup>	0.28 × 10 <sup>10</sup> 15 mT	1.07 × 10 <sup>10</sup> 8 mT	0.23 × 10 <sup>10</sup> 7 mT
TM <sub>110</sub> 3.44 GHz 482 Ohm	1.2 × 10 <sup>10</sup>	0.48 × 10 <sup>10</sup>	3.17 × 10 <sup>10</sup> 15 mT	0.61 × 10 <sup>10</sup> 14 mT
TM <sub>012</sub> 3.61 GHz 373 Ohm	1.0 × 10 <sup>10</sup>	0.37 × 10 <sup>10</sup>	1.65 × 10 <sup>10</sup> 21 mT	0.2 × 10 <sup>10</sup>
TE <sub>011</sub> 3.73 GHz 780 Ohm	5.9 × 10 <sup>10</sup> 33 mT	1.95 × 10 <sup>10</sup> 24 mT	7.15 × 10 <sup>10</sup> 9 mT	1.56 × 10 <sup>10</sup> 8 mT
TM <sub>111</sub> 3.74 GHz 390 Ohm	0.9 × 10 <sup>10</sup>	0.18 × 10 <sup>10</sup>	0.95 × 10 <sup>10</sup> 21 mT	0.16 × 10 <sup>10</sup> 12 mT

Table I: Measurements on TM-cavity at S-band  
 1) 400 Å Nb<sub>2</sub>O<sub>5</sub>, T = 1.3 K  
 2) 800 Å Nb<sub>2</sub>O<sub>5</sub>, T = 1.5 K  
 3) no oxide, T = 1.3 K

As to be seen in Fig. 1 the surface resistance depends strongly on field strength, especially at low RF-field levels, which makes it very difficult to define the residual RF-losses. With increasing field one reaches a peak in R (H) at about 1 mT; towards higher field-levels the surface resistance is always decreasing, reaching its lowest value near the breakdown field. The TM<sub>011</sub>- and TM<sub>012</sub>-mode show one side multipactoring<sup>4)</sup> at about 2 mT differing from run to run, which is accompanied with large frequency-shifts towards higher frequencies but could be overcome by processing. In every case the final limitation was magnetic breakdown, indicated by a large Q<sub>0</sub>-degradation of about 3 orders of magnitude.

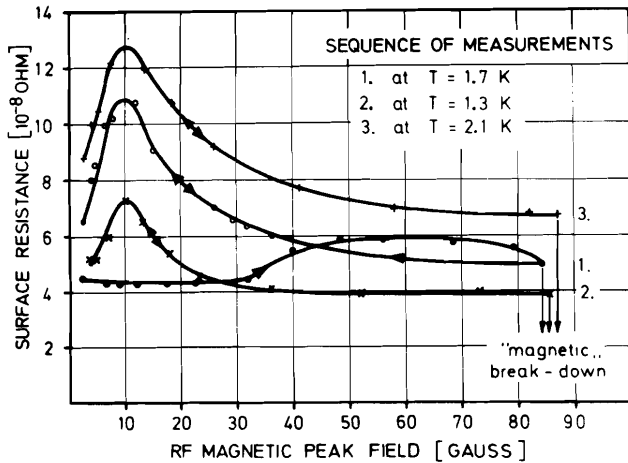


Fig. 1: Surface resistance as a function of magnetic field for the  $TM_{010}$ -mode.

Further observations achieved during the measurements can be summarized as follows: the breakdown fields seem to decrease with the number of treatments which the resonator went through; measured  $\Delta/kT_c$ -values of 1.3 to 1.4 are smaller than the values of clean niobium.

The measurements at Siemens, Erlangen were carried out with top-shaped, transmission-type X-band cavities operating in the  $TE_{011}$ -mode at 9.5 GHz. The cavities were machined from large grain solid niobium (Kawecki-Berylco) and then treated as described in Table II.

surface preparation		$Q_0$	$H_{crit} [mT]$
cavity Nr. 1	chemically polished	$0.6 \times 10^{10}$	22
	+ anodized	$1.1 \times 10^{10}$	33
cavity Nr. 2	electropolished	$0.3 \times 10^{10}$	46
	+ anodized	$2.7 \times 10^{10}$	77
	+ two days later not warmed up	$2.8 \times 10^{10}$	76
	+ warming up, 15 minutes at 80 K, recooling	$2.8 \times 10^{10}$	77
vacuum was not interrupted	+ warming up, 4 days at 300 K, recooling	$2.7 \times 10^{10}$	79
	+ dissolving the oxide layer	$0.8 \times 10^{10}$	44

Table II: Q-values and critical magnetic fields of Nb- $TE_{011}$ -cavities after different surface treatments.  $T=1.4$  K,  $f=9.5$  GHz,  $G=780$  Ohm.  $Q_0$ -values always measured just below  $H_{crit}$ .

The cavities were not hydrogen-degassed after electropolishing<sup>6)</sup>. In chemical polishing the same solution as described in<sup>5)</sup> was used. Anodizing was performed in a solution of 25 %  $NH_3$  at room temperature<sup>1)</sup>, the resulting thickness of the oxide layer was

0.28  $\mu$ . During the measurement the cavities were not sealed off.

### III. Oxidation of Niobium

Under normal conditions a niobium surface is always covered with an oxide layer of about 60 Å thickness<sup>7)</sup>. The state of this oxide is not well defined, because the oxidation is influenced by lattice distortions and surface irregularities. This state of the surface may contribute to high residual RF-losses<sup>3)</sup>. In contrast externally controlled chemical reactions lead to a well-defined growth of the oxide. An example for such a controlled reaction is anodizing, where an externally applied voltage instead of local intrinsic fields defines the growth of the oxide. In this process, in the case of amorphous  $Nb_2O_5$  ions move at the very high field of more than  $10^8$  V/m<sup>8)</sup>,<sup>9)</sup>. Therefore it is possible that the not well-defined oxides on the metal surface and the surface irregularities like protrusions will be due to their electric field enhancement-preferentially converted into the amorphous  $Nb_2O_5$ . That is, the metal surface will be smoothed and at the same time shifted into purer regions of the metal. On the other hand oxide on a niobium surface prefers to get dissolved. Therefore, anodized  $Nb_2O_5$  on a niobium-surface is not stoichiometric, but contains oxygen vacancies<sup>9)</sup>,<sup>10)</sup> the concentration of which is the larger the faster the anodization-process has taken place. The number of O-vacancies is continuously increasing towards the niobium interface<sup>9)</sup>,<sup>10)</sup>. During anodizing the percentage of oxygen, i.e. the density of the layer, increases with time, while the current density is already decreasing. This all means that one can produce  $Nb_2O_5$ -coatings of different behaviour<sup>11)</sup>, which can be changed further by filling up the electron-traps in the coating oxide to a different amount by e.g. electron impact.

### IV. Discussion

At first one should state that the oxide coating seems to be a fairly good protection against oxidation<sup>9)</sup> of niobium, dust and other surface contaminations. As to be seen from Table II no ageing of the X-band cavities takes place under vacuum conditions. This result is in contrast to ref.<sup>11)</sup>. Possible explanations for these discrepancies are time differences of exposure to room-temperature, differences in the structure of the  $Nb_2O_5$ -coating and the niobium used.

In the following we split the discussion in residual losses and high field effects.

#### a) Residual losses

As mentioned above it is very difficult to give a definite value of  $R_{res}$  because  $R(H)$  can be irreversible (see Fig. 1) and

depends strongly on H. The residual losses observed (see tables) show a clear improvement of anodized over chemically or electrochemically treated surfaces giving results comparable to an UHV-treatment<sup>11),12),13)</sup>. The reasons for the reduction of residual losses by anodizing are consistent with the hypothesis<sup>3)</sup> that the residual losses are mainly due to microscopic inhomogeneities of the surface, because anodizing eliminates these partly by shifting the metal-oxide interface into deeper purer regions of the niobium metal. The observations (Table I) show that dielectric losses in the amorphous Nb<sub>2</sub>O<sub>5</sub>-coating are not the dominant part in the residual absorption, because otherwise the resistances of TE- and TM-modes should be markedly different.

#### b) High field effects

The improvement in (magnetic) breakdown field by anodizing can be sometimes as high as 2 compared to surfaces, where chemical reactions could take place. Like in the case of residual losses, anodizing and outgassing gives comparable results. The enhancement of the peak-fields is consistent with the picture that by removing surface inhomogeneities and lattice defects the accompanied magnetic field enhancement<sup>16)</sup> will be reduced and hence the peak-fields increased.

As to be seen from Table I there seems to be a general decrease of critical fields in time. This could be due to additional solution of oxygen in the niobium surface, which will shorten the mean free path and hence makes nucleation of the normal state simpler. A further indication for oxygen solution are the small measured  $\Delta/kT_c$ -values. The other limiting effect observed is accompanied with electron emission, resulting in one side multipactoring at about 2 mT, which can be overcome by processing. The processing-time is larger in the case of anodized surfaces in comparison to the uncoated surface and increases with increasing oxide-thickness. This may be due to a higher secondary electron emission yield of the oxide.

Like outgassed cavities with less oxygen<sup>13),14)</sup> anodized cavities show a peak in R(H). But anodizing seems to develop special features to be seen in Fig. 1. After treating the cavity at higher power levels ( $\approx 2$  MV/m) resonance absorption can occur at lower power levels, which can be explained by field induced surface states<sup>15)</sup> indicating a change in reflection of conduction electrons at the interface. An explanation of this enhancement of specular reflection can be a charging up of the oxide by electron impact.

### V. Conclusion

The above results have shown that anodizing of niobium together with electrochemical polishing is a method to reduce the residual losses and to increase magnetic breakdown fields. At the same time the oxide coating is able to prevent ageing of the superconducting surface under vacuum conditions. An effect, which could limit the practical application of oxide coated cavities in accelerators or separators could be field-emission which was not observed in our cavities because of early magnetic breakdown. In this case electrons with high impact energies would hit the surface and probably destroy it.

On the other hand, high applied fields could, in spite of the dielectric shielding of the oxide, cause the motion of ions in the oxide due to the reduced, but still present field enhancement at protrusions. This migration of ions will probably influence the features of the oxide.

We hope to clear up these questions in the near future.

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