THE INFLUENCE OF SPECIALLY CREATED OXIDE FILMS ON THE EMISSION PROPERTIES OF NIOBIUM

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Abstract

One of the ways of solving the problem of increasing the accelerating fields in the superconducting cavities is to suppress the field emission effects. This work is devoted to the study the influence of oxide films on the niobium surface, specially created to decrease the field emission, and also to study their influence on the electrophysical parameters of superconducting cavities. The theoretical modelling of oxide cover influence on the factor Q of TESLA-shape superconducting cavities at frequency 3 GHz of the tgδ value (angle of dielectric losses) and the cover thickness at the accelerating fields of 10 MV/m and 20 MV/m well correlates with experimental dependence of dark currents from the thickness of dielectric cover with minimum losses. The optimal thickness, which provides the minimum dark current during the measuring of DC field emission, is found.

1. INTRODUCTION

The practical application of RF Superconductivity in accelerator technology requires SC activities to be developed with the large Q-value and capable to operate at a high level of microwave power.

The application of very pure SC materials and special treatment techniques helps to solve the problem of higher accelerating fields in SC cavities [1-4].

In previous articles we have considered various techniques of electropolishing [5-7] and feasibilities of anode oxipolishing and anodising methods in the SC cavities technology. [8-9].

As is known, the considerable reduction of field emission can be reached by covering a metallic surface with dielectric film, e.g. the Nb_2O_5 film to be laid on Nb surface. In this case anode oxidising is preferable. Such covering can reduce the field emission current by four orders but it causes additional dielectric losses. One should also keep in mind that with a dielectric placed in the accelerating cavity a configuration of electric and magnetic fields changes which causes a shift of a resonance frequency.

Investigation of Nb - Nb_2O_5 in the wide temperature range is of certain interest because 300 K provides a normal metal - semiconductor system, 70 K provides normal metal-dielectric, and 4.2 K a superconductor - dielectric.

The goal of this report is to present some calculated and experimental results of the influence of thin oxide films on

- the Nb field emission properties (dark currents);
- electrophysical parameters of accelerating cavities.

2. THE TECHNIQUE OF ANODE OXIDATION

One should keep in mind that obtaining the reproducible parameters of the oxide can be faced with technological difficulties [5]. The point is that anode oxidation is a very complicated process and it depends on many technological factors such as the film formation voltage V_f, anode oxidation current density j_a, concentration C, electrolyte temperature T and exposition time τ at V= const. In additional, the quality of the forming film is affected by structure properties of the working metal surface (its texture, roughness, graininess) as well as the technological conditions of oxidation process (preliminary electrolyte outgassing, gas environment above the electrolyte [8].

The specific features of anode oxidation impose certain requirements on technological equipment used in this process:

1. The electrolyte bath should be placed into a thermostat that could maintain the electrolyte temperature in the range of 4÷20 °C to an accuracy of ±0.01 °C;

2. The setup for oxidation should be furnished with [8]:

- system of preliminary electrolyte outgassing and inert gas bleeding-in;
- system of cavity or cathode rotation during anode oxidation;
- special cavity holder ensuring the required reliable and movable electric contact and geometric cathode and anode similarity.

Fig. 1 shows the equipment for anode oxidation of CM-band cavities and different samples.

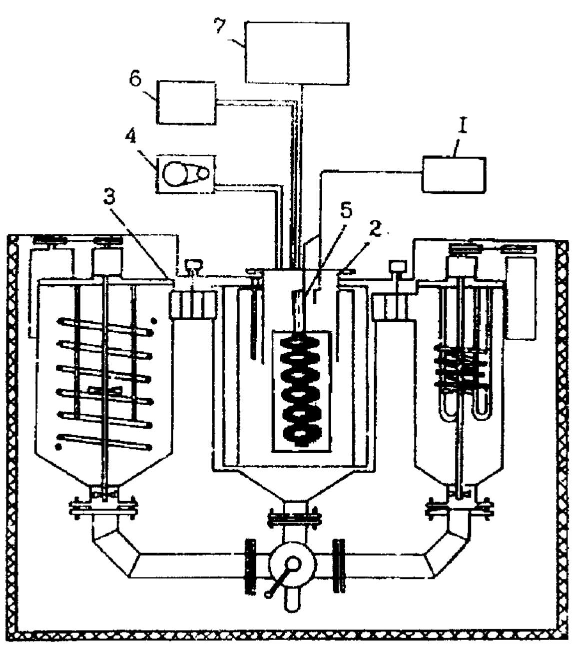

Fig. 1. The setup view for anode oxidation of CM-band of the SC cavities: 1 - block monitoring anode oxidation (ramp rate, voltage at which the oxide film is formed, stabilisation of anode current, etc.), 2 - electrolyte bath, 3 - ultra-thermostat, 4 - vacuum system for electrolyte outgassing, 5 - SC cavity, 6 - inert gas bleeding-in system, 7 - thermostat control block.

The setup consists of electronics to control the anode oxidation process, electrolyte bath placed into a temperature-controlled volume of ultra-thermostat.

In all cases the best results were obtained when anode oxidation was carried out in a mixed regime [8]: the first regime is galvanostatic one, which lasts until the specified voltage V_f of the film formation will be achieved and the second regime is potential-static one when the voltage V_f is constant [9].

It should be kept in mind that different purposes of oxide covering application dictate a choice of different very pure type of electrolytes based on H_2SO_4, H_3PO_4 or NH_4OH which have various additives.

The combination of the technological parameters of anode oxidation is different in each application case and was determined as a result of multi-factor optimisation. In order to determine the structure of the forming oxide and the condition for higher valence oxides to form we have measured the volt-ampere characteristics [8].

The dependence analysis shows that the film produced in the j_a = const mode is a multi layer one. For all j_a under study there were three noticeable sections on the volt-ampere curve though with no pronounced boundaries between them.

The section of noticeable rise corresponds to the initial time period when Nb oxide is formed, the second section (the bend of the curve) corresponds to formation of oxide NbO, and the third section is characterised by formation of oxide Nb_2O_5 [8].

With the time of film formation increased (growth of V), a fraction of the higher-valence oxide increases and if V = const the film consists only of the higher-valence oxide.

3. OPTIMIZATION OF Nb ANODE OXIDATION AIMED AT OBTAINING THE MINIMUM FIELD EMISSION CURRENTS

Some articles and books contain various controversial data on the mode of anode oxidation of Nb and the required thickness of oxide coatings. Moreover, this process was studied only in terms of some single factors with the other ones fixed.

Therefore it was necessary to construct a mathematical model of the process of anode oxidation of Nb. The problem of optimising this process was reduced to finding such a technological mode with parameters X_1, X_2, X_3, X_4, X_5, which could make it possible to obtain the minimum value of tgδ and Q. As a result of optimisation the following formula was obtained:

$$\begin{aligned}10^3 J(X) = {} & 0.01X_1^2 + 31X_1X_2 + 1400X_1X_3 + 1.5X_1X_4 + 50X_1X_5 - 215X_1 + 23X_2^2 - 222X_2X_3 \\ & + 25X_2X_4 + 184X_2X_5 - 130X_2 - 412X_3^2 - 128X_3X_4 - 688X_3X_5 - 129X_3 - 5.5X_4^2 \\ & + 130X_4X_5 - 66X_4 + 440X_5^2 - 623X_5 + 282\end{aligned}$$

We have received an optimal block of parameters [8]:

U = 30 V; j_a = 1.59 mA/cm ; C = 12 NH_2SO_4 ; τ = 2.3 ;
t = 10.2°C [10,11].

Experiments show that the minimal field emission properties can only be obtained if there are:

- minimum content of lower-valence oxides;
- minimum content of admixtures from the electrolyte;
- maximum possible density of oxide film.

These conditions are given in detail in [8].

4. THE STAND FOR FIELD EMISSION MEASUREMENTS

The schematic view of a stand for electron field emission measurements is demonstrated on Fig. 2.

The high-voltage leak-in (1) allows one to apply from the high-voltage power supply (2) a working voltage of 120 kV onto the electrode system into the adjustable vacuum gap.

The vacuum chamber (3) and the device (4) handling the samples to be measured make it possible to study the field emission properties of 30 samples within one pumping-down cycle.

The stand is equipped with the system (5) cooling the samples down to nitrogen temperatures.

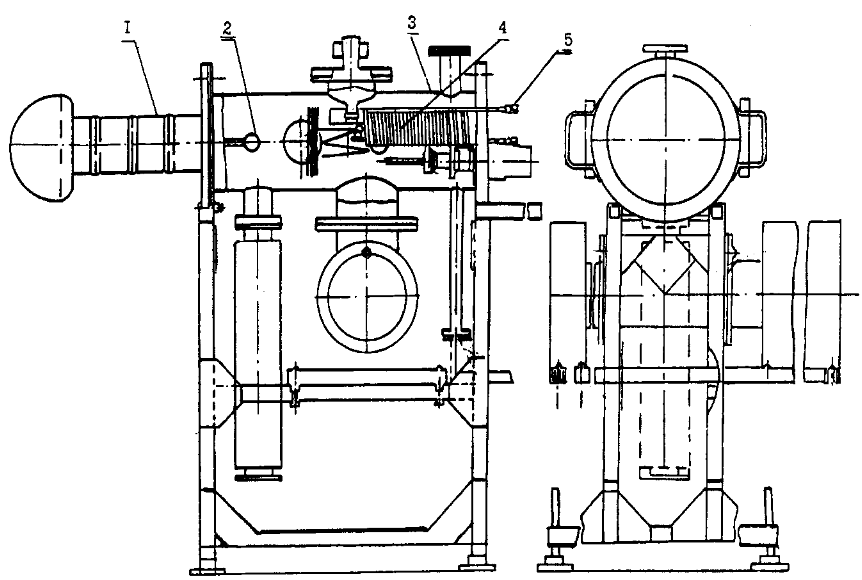

Fig. 2. The outward appearance of a stand for electron field emission measurements [8,12-14].

The high voltage lead-in construction enables the working voltage to be applied by value to 120 kV to adjustable vacuum gap. A vacuum chamber construction and design for transmission of specimens for measurement enable one to study the electron field emission properties of 30 specimens and to investigate the electrodes of different configuration. The main stand parameters [8]: working chamber diameter - 246 mm; working chamber length - 760 mm; the number of samples which may be investigated for one pumping cycle - 30; the analysed samples diameter - 30 mm; working vacuum - 10^{-7} Pa; supplied power - 7 kWt.

5. EXPERIMENTAL AND CALCULATION RESULTS

5.1 THE FIELD EMISSION DARK CURRENTS

It is important to know the emission properties of Nb with thin oxide films. Fig. 3 contains data on influence of different conditions on the emission dark currents of Nb with thin oxide films of 200 Å to 1000 Å thickness.

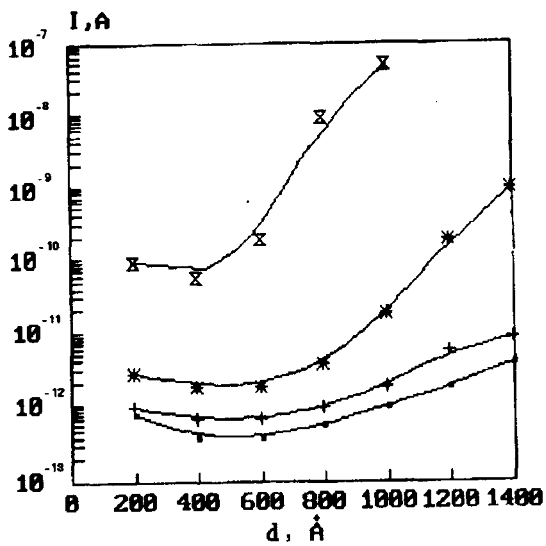

Fig. 3. The dependence of field emission dark currents on the Nb_2O_5 film thickness onto the Nb surface:

1 - high pressure pure water (10 MΩ); 2 - high pressure pure water with UV; 3 - high pure water without UV; 4 - have used water rinsing with R = 1 MΩ.

As one can see from these figures, the minimum emission dark current exists at the thickness (d) of 480÷520 Å.

5.2 THE LOSSES THROUGH DIELECTRIC ON THE SC SURFACE

Losses through dielectric on the superconducting surfaces can be determined as [16-19]

$$P_d = \tfrac{1}{2}\omega\varepsilon_d\varepsilon_0 tg\delta \int_V E^2 dV,$$

where ε_d and ε_0 are the dielectric penetrability of Nb_2O_5 film and vacuum respectively.

The mean diffusion power through emission may be determined as [15]

$$P_e = \frac{k\beta^{\frac{5}{2}} E_{acc}^{\frac{7}{2}} d}{2\pi c} e^{-\frac{c}{\beta E_0}},$$

where E_{acc} - a macroscopic accelerating field; d - the distance of emitted electrons before a collision; K, C - constant values; $\beta = - E_{loc}/E_{mean}$ (where E_{loc} is a local field at the emission point).

In order to calculate the losses due to dielectric films it is necessary to receive the dependence of the ε-factor from the oxide film thickness.

5.3 DEPENDENCE OF THE ε-FACTOR FROM THE OXIDE FILM THINKNESS

It is worth to show the dependence of factor ε on the oxide film thickness which can be seen on Fig. 4. for superconducting cavities (3 GHz). Some of the calculated problems in more details are given in the report [16].

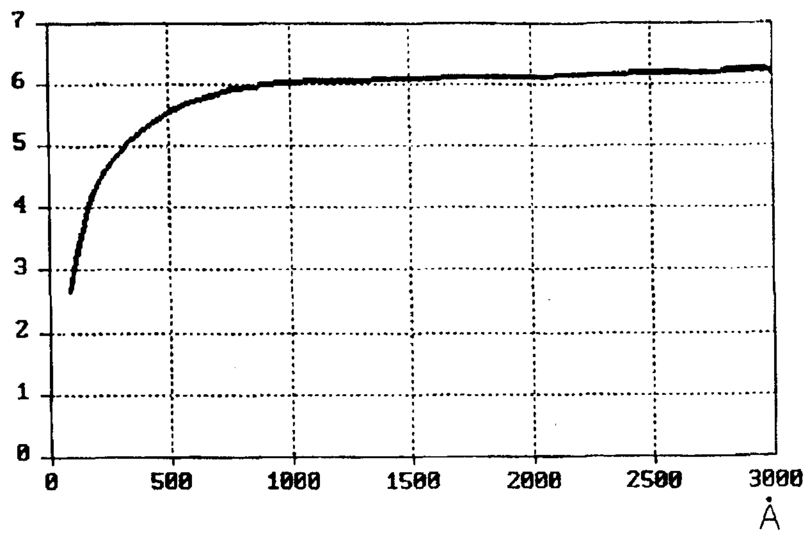

Fig.4. The dependence of ε from the oxide film thickness.

In the given calculations the program SCAT written in MATHCAD PLUS 6.0 (Professional Edition of MATHSOFT with using the program library AEDP v.2.0) and self-developed subprograms on C++ compiled in BC++ v.4.0 have been used [16,19].

5.4 THE INFLUENCE OF DIELECTRIC FILM ON Q-FACTOR ON THE LOW LEVEL OF RF ENERGY

To estimate an influence of oxide covering Nb_2O_5 and emission load with this film on the factor Q of TESLA-shape SC cavity working at the frequency of 3 GHz, may be of interest.

On the Fig. 5 the influence of oxide films thickness (Nb_2O_5) on the Q-factor without taking into account the emission effects, i.e. at low RF-power level, is given.

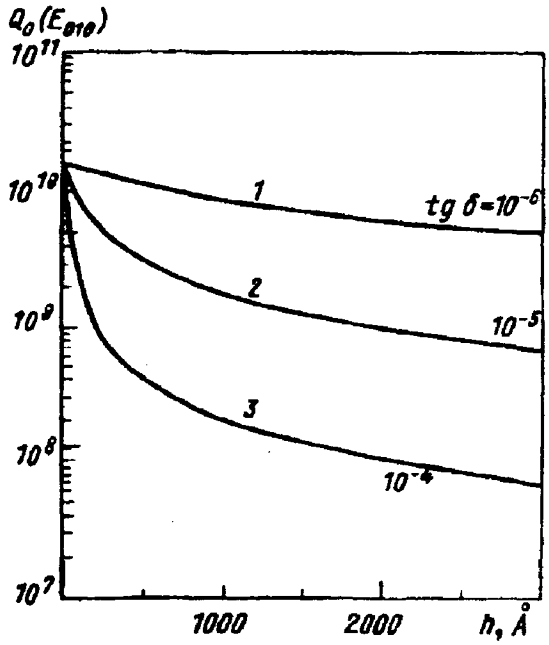

Fig. 5. The calculated dependence of the Q-factor from tgδ and oxide films thickness at 4.2 K.

From this picture one can see that Q-factor depends especially from the tgδ of the oxide film. On one side it is necessary to use the more thin film in order to decrease the losses through the SC cavity, on the other side the coating should provide the decreasing of field emission.

5.5 CALCULATING THE INFLUENCE OF ε AND tg δ ON THE ELECTROPHYSICAL PARAMETERS

Calculation program of influence the Nb_2O_5 dielectric covering on the factor Q of SC cavity has been made as follows:

1. The RF tunnel current calculation through the boundary phase /Nb - Nb_2O_5 - vacuum/ [16-19].
2. Emission load calculation [18, 19].
3. Losses calculated in the dielectric film volume [5,23,26].
4. The energy dissipation in the cavity walls [5, 23, 25].
5. Calculation of factor Q of the cavity without dielectric layer but with emission load [5, 23,25].

Calculation results of the TESLA-shape cavity factor Q properly [1-4] with the dielectric layer depending on the oxide film thickness d taking into account the emission load at accelerating field E_{acc}= 10 MV/m and E_{acc} = 20 MV/m are given in Fig. 6 and Fig. 7.

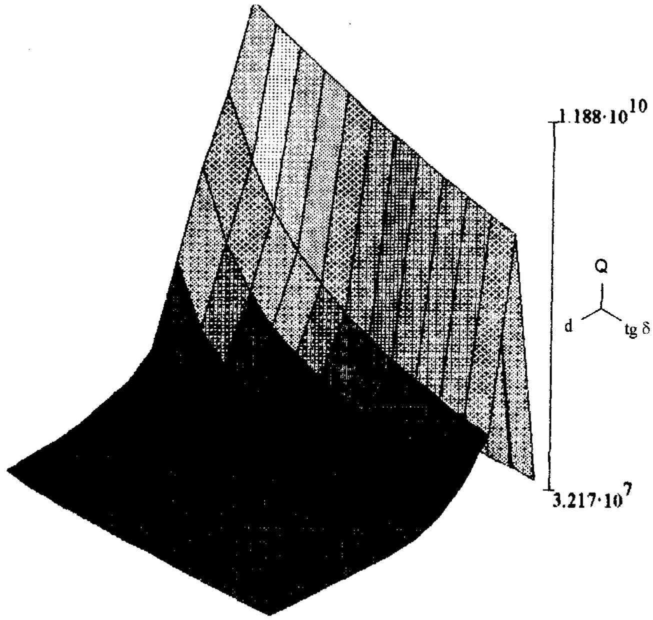

Fig. 6. The factor of the superconducting cavity with Nb_2O_5 thin film versus the thickness and tgδ at E_{acc} = 10 MV/m.

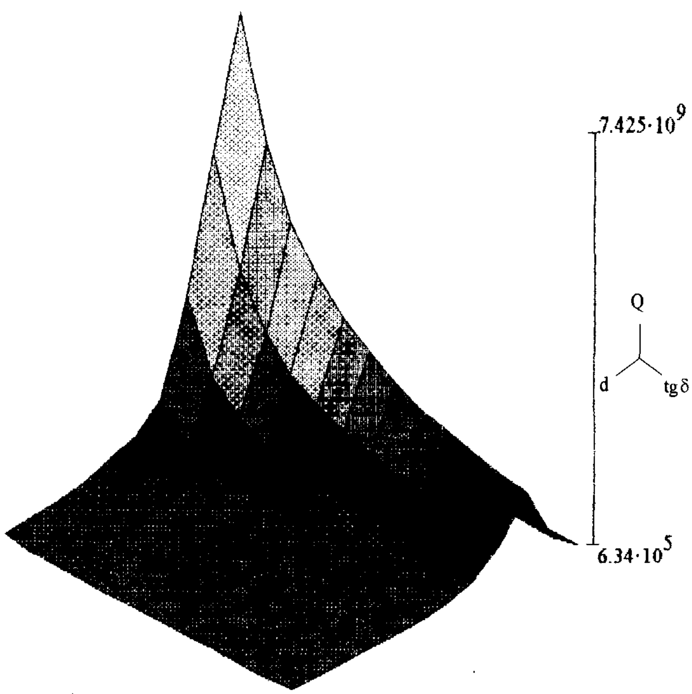

Fig. 7. The Q-factor of the superconducting cavity with Nb_2O_5 thin film versus the thickness and tg δ at E_{acc} = 20 MV/m.

In all cases the best results have been obtained when thickness range was 480÷520 Å (Figs. 3, 6, 7) if the specific conditions of oxidation are provided.

All results presented here show the existence of suppression effect for field emission when we have used Nb with thin Nb_2O_5 films.

The calculation was made on the base of the data and formulas from [22-33].

6. CONCLUSION AND FUTURE

The study of anode oxidation of Nb has not yet been finished. Surface oxidation influence onto the different types of emission (field emission, acoustic, secondary and exoelectron) is being planned to study. It would be very interesting to study the influence of these kinds of emission on the electrophysical characteristics for 3 GHz SC cavities.

We have obtained the interesting results for the SC cavities with oxide films coated on the Niobium surface with different RRR. This problem requires are more accurate study.

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