EUROPEAN ORGANIZATION FOR NUCLEAR RESEARCH
European Laboratory for Particle Physics

CERN EST/97-05 (SM)

STUDY OF THE ROLE OF THE INTERFACE BETWEEN NIOBIUM FILMS AND COPPER RF RESONATORS

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Abstract

Niobium-coated copper resonators are usually produced with an oxide interface between the film and the substrate. This oxide has two sources: the passivation layer inevitably formed on the surface of the cavity after chemical preparation before coating, and the niobium oxide which builds up on the surface of the cathode when it is exposed to air, and is transferred to the cavity surface during coating. The oxide layer may influence both the purity and the structural properties of the film, and in turn its RF behaviour. To study its effect, some cavities have been coated with a special two-cathode sputtering system, allowing for a complete removal of both oxide layers by sputter-etching. For comparison, a few cavities have also been produced with the same coating system without sputter-etching, or with a controlled oxidation of the copper surface of the cavity after sputter-etching. Two cavities have also been produced without oxide interface using Kr and Ne as sputter gas instead of Ar.

Paper presented at the Eighth Workshop on RF Superconductivity
Abano, Italy, 6-10 October 1997

Geneva, Switzerland
December 1997

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1. Introduction

The technology of sputter deposition of niobium films onto copper resonators, developed for the 352 MHz superconducting accelerating cavities of LEP2 at CERN [1], is now well established. The maximum achieved values of Q and accelerating field are however lower than theoretical estimations would suggest, and to address these basic questions an extensive R&D programme has been undertaken. For this purpose, the LEP coating technology has been adapted to 1.5 GHz resonators, which are more easy to handle compared to resonators of a lower frequency [2].

An essential step in the LEP2 coating procedure is the chemical preparation of the surface of the cavity before coating [3]. In this process, the copper surface is covered by an oxide passivation layer, whose thickness can be estimated of the order of 10 nm. The niobium cathode, always kept in a clean room when not in use, has also on its surface an oxide layer, that is diluted in the first layers of the sputtered film. These oxide layers have an effect both on the impurity content and on the growth properties of the niobium film, thus influencing its RF performance.

2. Experimental procedure

For the purpose of completely removing any oxide contamination at the interface between copper and niobium, a special coating system has been employed (Fig. 1). The system is equipped with two electrodes, one of copper and one of niobium, on either side of the central part where the cavity is mounted. These electrodes can be introduced in the cavity by compressing long bellows, while in the rest position they are insulated from the central part of the coating system by gate valves and individually pumped to a vacuum of the order of 10^{-10} Torr.

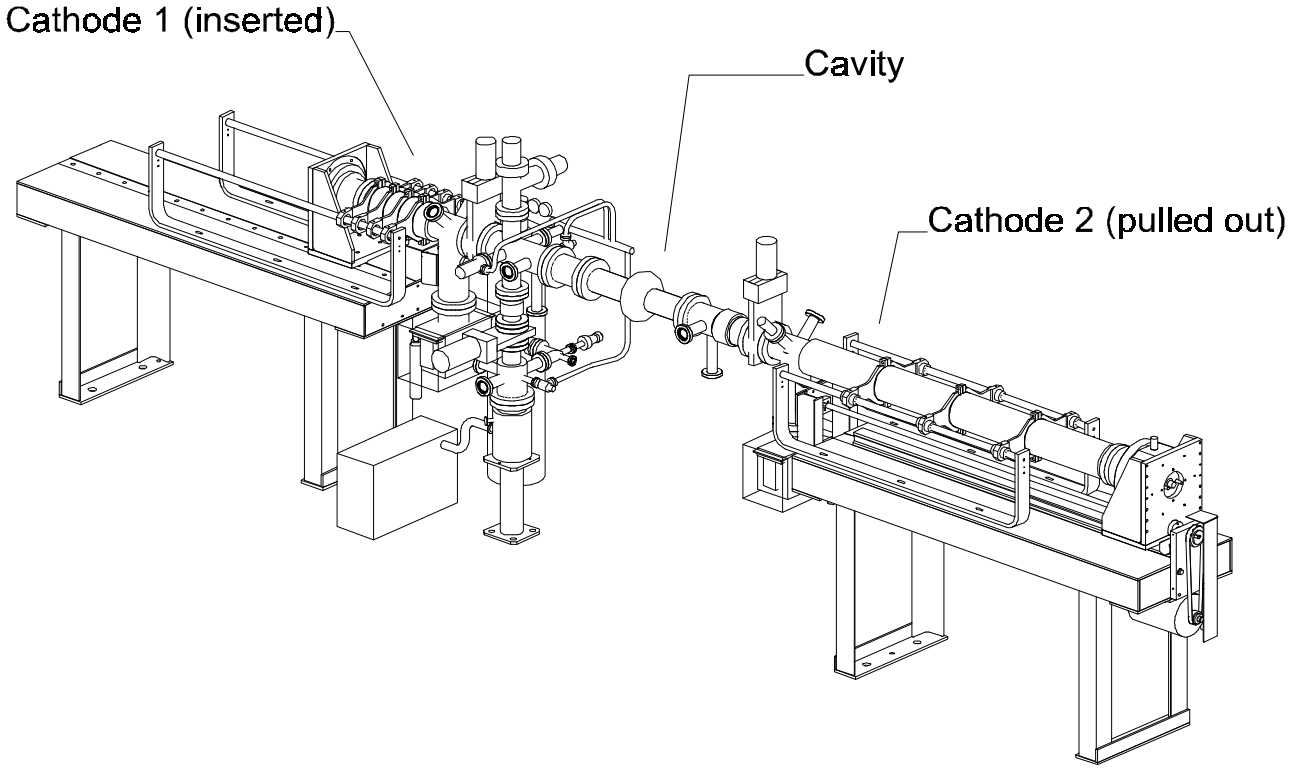

Fig. 1 : View of the coating system

The niobium electrode, negatively polarised to be operated as a sputtering cathode, is sputter-cleaned in a preliminary run by coating a dummy cavity, and then kept under vacuum to prevent any oxidation.

The copper cavity is mounted on the coating system after a standard chemical surface preparation, and baked-out at 150°C for 20 hours. The copper electrode is then introduced and set at a positive potential (anode) while the cavity is kept at ground potential, in an argon atmosphere. A plasma is ignited close to the cavity surface because of the different mobility of ions and electrons, resulting in an ion bombardment (etching) of the cavity surface (Fig. 2).

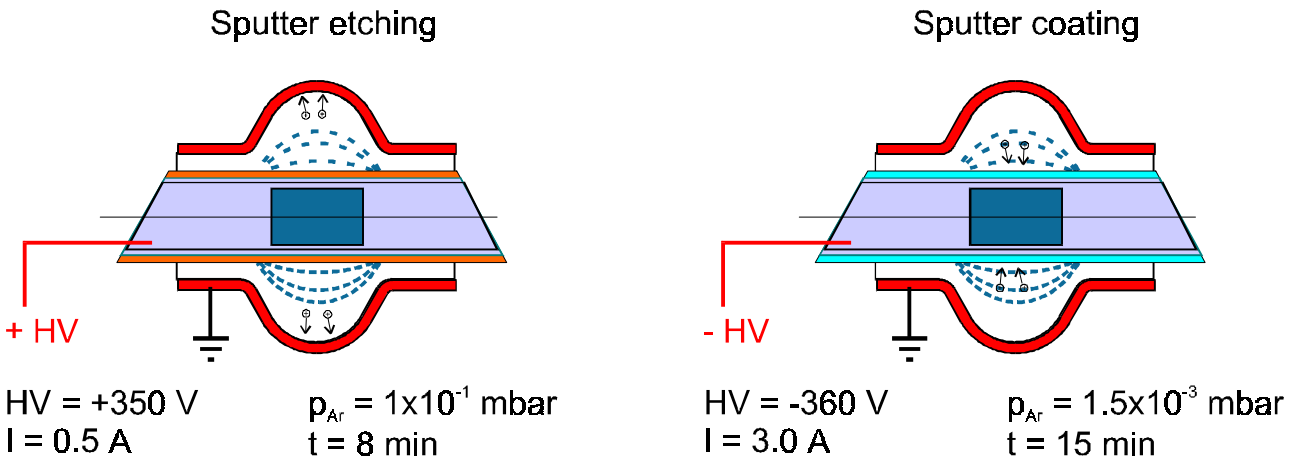

Fig 2 : Sputter-etching and sputter-coating. The parameters used for the two discharges are also listed.

A total cleaning dose of approximately 10^{18} ions/cm^2 (averaged over the entire cavity surface) is employed, subdivided into eight cycles of one minute each. The interval between each cycle is about five minutes, to allow the evacuation of the impurities produced. A large increase of the partial pressure of the desorbed gases, mainly CO and CO_2, is observed at the first etching cycle, while at the last cycle this rise is minimal (Fig. 3).

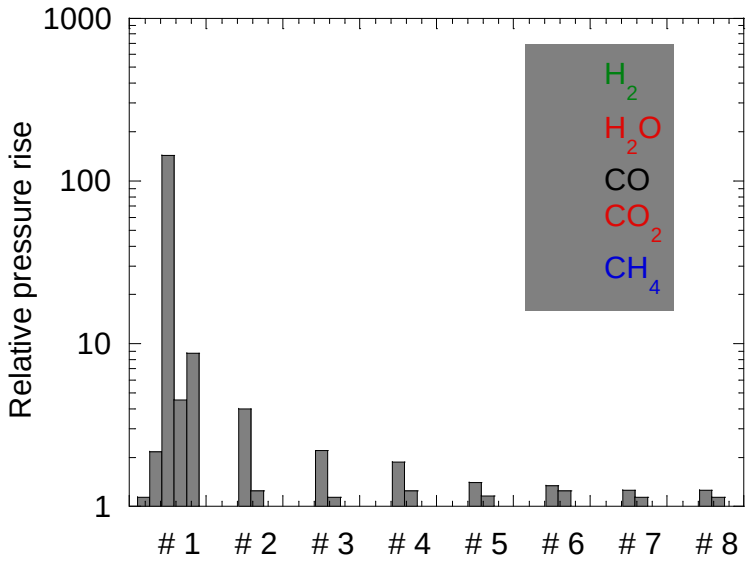

Fig. 3 : Relative pressure rise of the desorbed gases at each etching cycle.

Niobium coating then takes place immediately after etching, in the standard cylindrical magnetron sputtering configuration, with the niobium electrode (cathode) at negative potential. The gas pressure, as read from a ionisation gauge, and the sputtering parameters are the same for the three different sputter gases employed, namely neon, argon and krypton. The coating time is adapted to take into account their different sputtering efficiencies, in order to produce a coating thickness of 1.5 μm.

In a few cases the copper has been oxidised by a few hours' exposure to dust-free air after sputter-etching. In such cases the niobium coating takes place after a further bake-out of the cavity.

3. Experimental results

Samples have been cut from a cavity produced by sputter-etching the copper prior to niobium coating, and from a standard niobium-coated cavity for comparison (using argon as sputter gas in both cases). By dissolving the copper in nitric acid, it is possible to measure the RRR of the films. Standard cavities have on average a RRR of 11±1, while sputter-etched cavities have a RRR of 21±2. The same samples have also been analysed by Auger spectroscopy, and no difference in their bulk composition was observed at 1% sensitivity level. XRD spectra show that the standard film grows preferentially in the direction <1,1,0>, while the film of the sputter-etched samples is less oriented.

The surface resistance of the cavities has been measured and analysed in terms of BCS surface resistance R_{BCS}, residual resistance R_{res}, slope at low field $\Delta R_{res}/\Delta H_{RF}$, and dependence on trapped magnetic flux in presence of RF field. For the latter, the parametrisation $R_{fl} = (R_{fl}^{0} + R_{fl}^{1} H_{RF})H_{ext}$ has been used [4]. All these quantities are measured at 1.7 K, except for R_{BCS} of which the 4.2 K values are reported.

Hydroformed cavities that underwent sputter-etching of the copper surface have very high values of residual resistance and slope (Fig. 4), compared to spun cavities. Cavities with a controlled oxidation of the copper surface after sputter-etching differ minimally from cavities produced in the standard way, i.e. with the usual oxide interface due to chemical passivation. It must be noted that films prepared in the standard way under supposedly identical conditions exhibit a very large spread of residual resistance and slope.

The BCS surface resistance measured for cavities produced with different gases depends on the impurity content, which is dominated by the amount of sputter gas implanted into the film [5]. It takes however larger values in the case of sputter-etching (Fig. 5). In the case of argon and krypton this can be interpreted as an increased purity of the film, as suggested by measurements of the RRR and of the penetration depth [6].

The sputter-etching procedure is found to significantly increase the effect of trapped magnetic flux on the surface resistance (Fig. 6). Cavities where the copper has been oxidised after sputter-etching again are similar to standard cavities.

The change of sputter gas has also an influence on this measurement (Fig. 7). By replacing argon with neon for example, the effect is bigger than the sputter-etching alone.

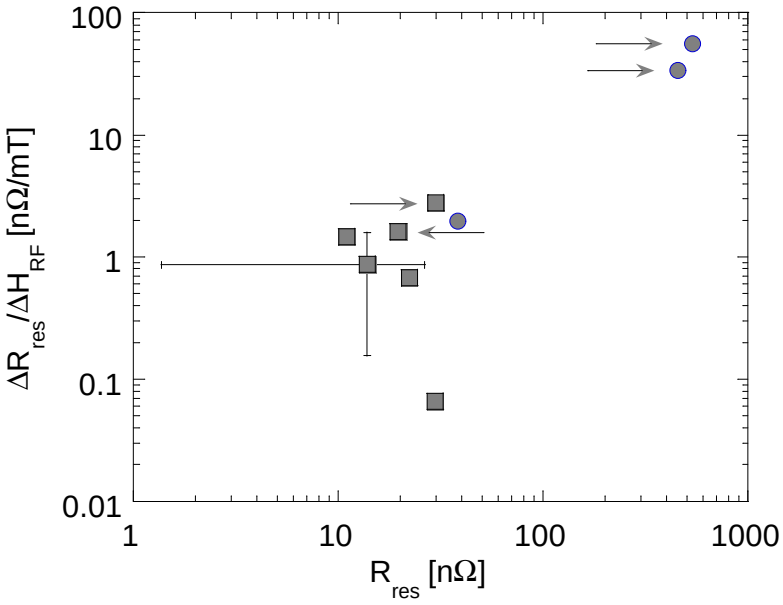

Fig. 4 : Residual resistance and its slope. Hydroformed cavities are indicated by dots, spun cavities by squares. The arrows indicate cavities that underwent sputter-etching as described in the text, compared to cavities with oxidation of copper after sputter-etching. The average of 28 cavities coated according to the standard procedure is also indicated with the error bars.

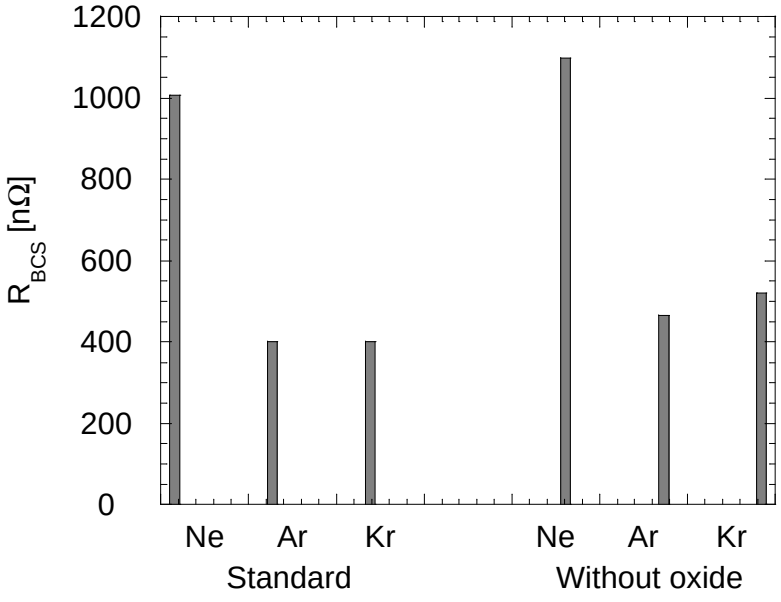

Fig. 5 : R_{BCS} for different discharge gases, with and without an oxide layer between copper and niobium

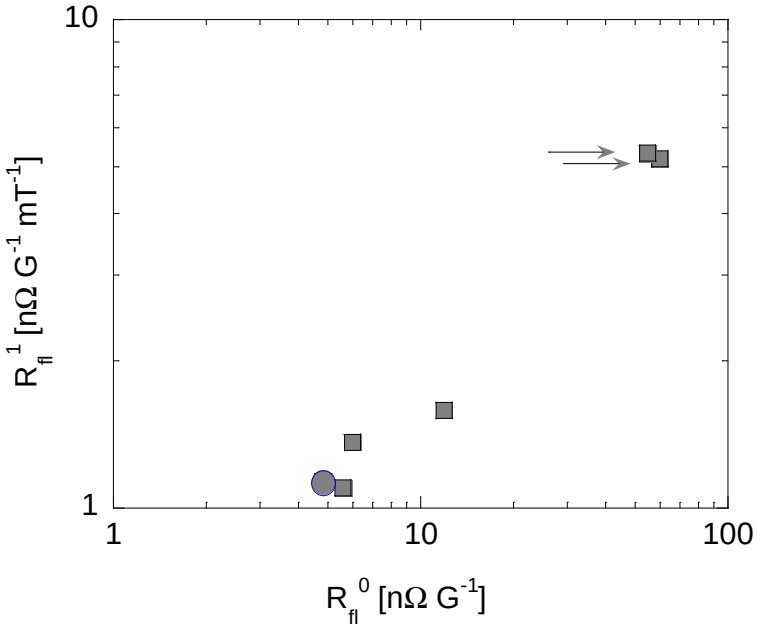

Fig. 6 : Effect of trapped magnetic flux on cavities coated using argon. Cavities without oxide interface are indicated by arrows. The average of 28 standard cavities is indicated by the dot, the error bars being smaller than it.

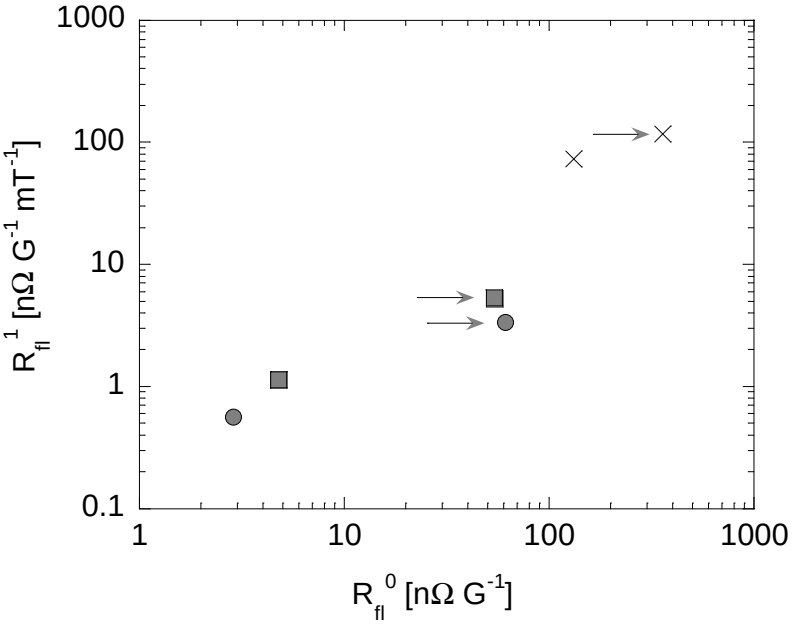

Fig. 7 : Effect of the change of sputter gas. Dots indicate krypton, squares argon and crosses neon. The arrows indicate cavities without the oxide interface.

4. Conclusion

A few 1.5 GHz niobium-coated copper cavities have been produced without an oxide interface between the film and the substrate, always present in the standard coating procedure. A big increase of residual resistance has been measured for hydroformed cavities treated in this way, while for spun cavities the effect is minimal. The RRR measurement and the R_{BCS} values suggest that films without oxide interface are cleaner. However, the understanding of the effect of the removal of the interface oxide layer on the fluxon induced losses requires more experimental data.

References

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