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RF SUPERCONDUCTING PROPERTIES OF THIN FILMS ON NIOBIUM*

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Summary

We are investigating the RF properties of thin films of materials which are known to have low secondary emission coefficients, such as NbC, NbN and TiN. Preliminary measurements on the latter material have been performed by depositing a 15 nm film on parts of a doubly re-entrant Nb cavity designed to favor electron multipacting which, in the uncoated cavity, occurs copiously between the posts' tips. The measurements performed with TiN films sputtered onto Nb indicate that the RF current losses are increased by the presence of the films while the dielectric losses are negligible, within the measurement sensitivity of the system. The electron multipacting cannot be excited between the posts coated with the material tested.

Introduction

Electron loading in superconducting cavities and structures has constituted a major problem in the application of superconducting technology to accelerators. In recent years new choices of cavity geometries which inhibit multipacting have been successfully applied in the construction of superconducting accelerator structures. The problem remains, however, of decreasing or eliminating the effects of multipacting in parts of the structures for which the geometry cannot be significantly changed, such as in higher-order-mode couplers. In these cases the suppression of multipacting can be achieved by coating the surfaces of interest with a material which must have both a low secondary electron emission coefficient and low RF losses, in order to maintain the original high unloaded Q of the cavity. Materials such as NbC, NbN, and TiN seem to be good candidates for this purpose.

A special niobium S-band cavity has been designed and built, which allows the testing of the RF properties of such films. The measurements so far performed indicate that multipacting is inhibited in the cavity coated with TiN, although the RF losses are somewhat increased by the presence of the films.

Surface Physics Studies

Experimental Apparatus:

Surface properties of TiN deposited on Nb were investigated in two UHV chambers, one for coating and processing Nb samples and the other for measuring the surface properties. These chambers are coupled together with an isolation valve. The transfer of the samples (1.59 cm diameter × .47 cm thick discs) between chambers under UHV conditions is accomplished by magnetically-coupled rods carrying the samples.

The Nb samples were sputter-cleaned and then sputter-coated with TiN. Electron bombardment heating of the samples was also available.

Various surface physics techniques can be applied in the analysis chamber (including x-ray photoelectron spectroscopy (XPS), Auger electron spectroscopy (AES), and ellipsometry), but the measurements of particular interest here are those of the secondary electron emission (SEE) yields 1) after coating, 2) after coating followed by exposure to air and 3) followed further by electron bombardment of the air-exposed coatings.

The Nb samples were made from low-Ta (< 350ppm) previously outgassed (2500K) rods. The degreased samples were mechanically polished with 5μ diamond paste and electropolished in 10% HF - 90% H_2SO_4 solution.

TiN is routinely used at SLAC (because of its heretofore apparently low SEE yield) to coat inter-cavity coupling slots, RF windows and RF coupling loops. In addition to sputter-coating the TiN, we also made coatings by Ti evaporation in a N_2 atmosphere. Their properties were similar to those of the sputtered films. Heating of the Nb substrate to 500^oC during TiN coating did not give significantly different results from coatings deposited onto ambient temperature substrates. The TiN film thickness for the data presented here is 14 nm.

Results:

Figure 1 shows the SEE yield of TiN/Nb under the various conditions expected to be encountered in real cavities. The XPS-AES analysis [Figs. 2(a), 3(a)] reveals that a small amount of oxygen is incorporated into the TiN/Nb layer during TiN deposition, possibly from H_2O vapor present in the gas phase.

The as-deposited yield [Fig. 1(a)] is very good; however, it degrades significantly upon overnight air exposure [Fig. 1(b)]. Such a condition (air exposure of the film) will be found in practice because the cavity will come up to air for installation, maintenance, or during a machine accident. XPS-AES showed the presence of mixed Ti-oxides (mostly TiO_2) on these air-exposed surfaces [Figs. 2(b), 3(b)]. The Nb 3d-core levels (not shown) of the substrate were typical of NbN in both the as-deposited and air-exposed cases.

TiN surfaces having these relatively high yields would apparently be of marginal use. In view of the extensive excellent experience in normal RF use of TiN coatings at SLAC, an attempt was made to further simulate conditions in the cavity which might lead to a lowering of the in-situ SEE yield. The major in-situ effect expected is electron bombardment of the coating. The TiN/Nb coating was electron-bombarded by rastering a 1 keV electron beam across the surface and periodically checking the yield, XPS and AES spectra. Figure 1(c) shows

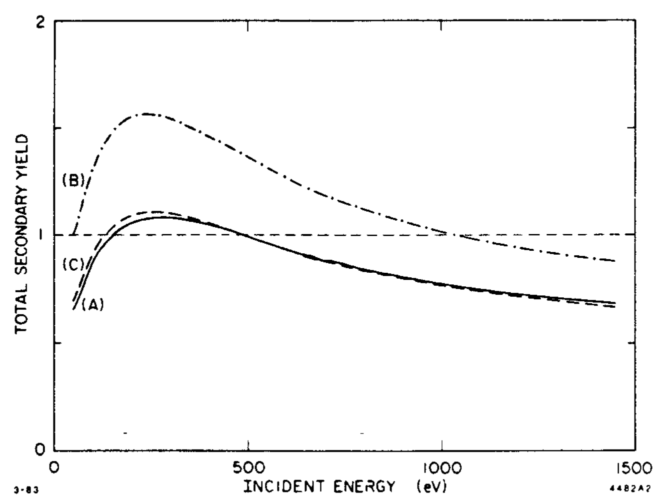

Fig. 1. Total SEE yield for 14 nm TiN/Nb. (a) As deposited, (b) after overnight atmospheric air exposure, (c) followed by electron beam exposure of 1.1 × 10^{18} electrons per cm^2.

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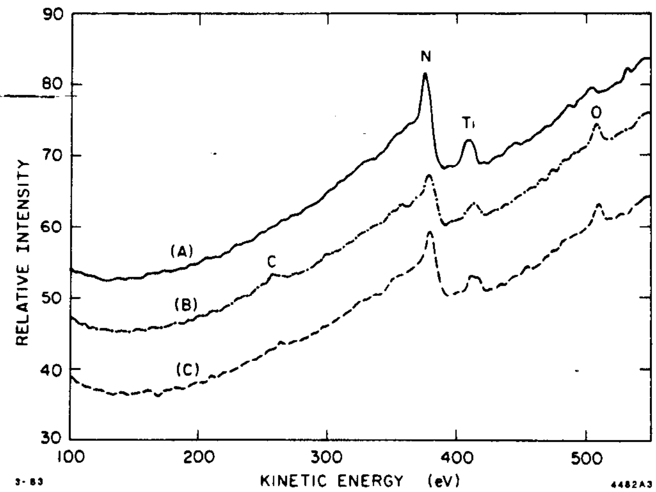

Fig. 2. AES energy distributions for the three cases of Fig. 1.

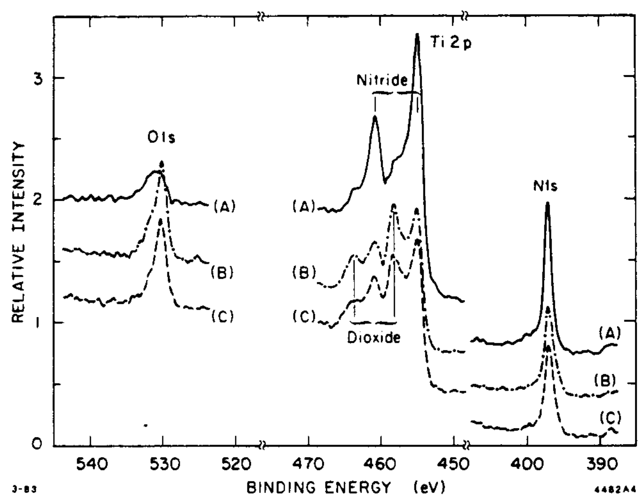

Fig. 3. XPS spectra for the three cases of Fig. 1. X-ray radiation is Mg-Kα at 1253.6 eV. Curves displaced vertically for clarity.

that the yield drops back to essentially initial values. Electron doses greater than about $3 \times 10^{17} cm^{-2}$ did not result in significantly different final yields. Thus, it would appear that electron-bombardment is responsible for the low in-situ SEE yield that is observed in actual use.

The electron-bombardment did not greatly alter the XPS and AES spectra [Figs. 2(c), 3(c)] as compared to the oxidized surface. A small change from TiO_2 to sub-oxides is observed using XPS, while an increase in the nitrogen and a decrease in the carbon surface concentrations is seen using AES. It seems reasonable to expect larger changes in the surface chemistry to account for the significant SEE yield change observed. If this contention is correct, then the XPS and AES may be relatively insensitive to the chemistry of the changes occurring in the secondary electron emitting layer. A possible explanation for this lack of sensitivity follows: The AES detection depth is only a few atomic layers but chemical information is difficult to extract from the spectra. XPS results are more easily interpreted but the information comes from depth further into the sample than that layer from which true secondary (< 40 eV) electrons emanate. This may be responsible for the lack of significant alteration in our AES and XPS spectra. Ellipsometry, contact potential difference and secondary ion mass spectrometry studies in progress may reveal the nature of this phenomenon.

RF Test Apparatus

The cavity used in the studies of the microwave properties of the thin film coatings is of the doubly re-entrant type. Its cross section is illustrated in Fig. 4. This cavity has the property of having the highest electric field in the gap between the posts, where the two-point multipacting is most likely to occur. The gap size was designed for a resonant behavior of the electrons which would acquire a kinetic energy of about 1 keV, energy for which the SEE yield is maximum. The surfaces at the posts' tips are therefore the only ones that need to be coated in order to test the presence or the absence of multipacting. The cavity can be disassembled at points where the surface magnetic field has a node, is such a way that the RF current losses at the joints can be minimized. The fields in this cavity were determined using the program SUPERFISH (see Fig. 5). With the small gap the proper potential difference between the posts is reached for very low power levels in the cavity, so that other types of breakdown do not interfere with the electron loading due to multipacting. Typical input power levels at which multipacting is observed range between 200 μW and a few mW, while the thermo-magnetic breakdown occurs in the cavity at an input power of about 200 mW or higher.

Since a wide range of the cavity's unloaded Q's is expected depending on the coatings' properties, the use of a variable coupling mechanism was necessary. This was achieved by using a coaxial transmission line with a sliding contact.

The electronics employed for the Q measurements uses the standard phase-locked-loop technique, with the local voltage-controlled crystal oscillator locked to the cavity frequency.

Results of the RF Measurements

Two sets of niobium posts have been used for the measurements. The cavity unloaded Q was first measured with both sets of posts without any coatings. Q_0 at 4.2K was about 2.5×10^7, very close to the BCS theoretical value 2.34×10^7. for this particular cavity geometry.

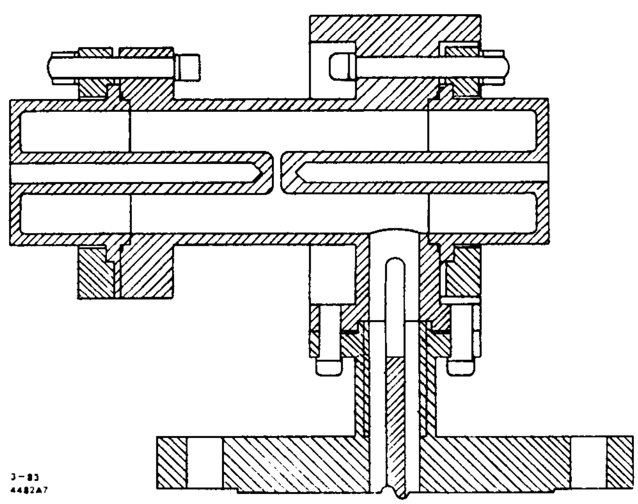

Fig. 4. Cross section of the coaxial cavity.

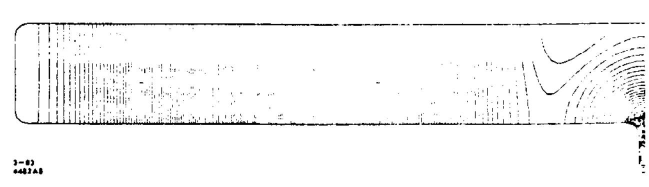

Fig. 5. Electric field lines computed with SUPERFISH (upper left quadrant only).

At lower temperatures (1.35K) the losses in the uncoated cavity were much larger than the theory predicted: Q_0 was limited to 6-7 $\times 10^7$, while the theoretical value should be 2.2×10^{10}. The excess losses may be ascribed to a slight offset of the cavity joints from the true current node.

The uncoated cavity tips easily gave rise to multipacting when the proper power was applied to the cavity: at the threshold a pattern like the one shown in Fig. 6 was observed in the reverse power (CW). Above the threshold much more stable loading of the cavity was achieved. This stable state is illustrated in Fig. 7, where a long RF pulse was applied to the cavity.

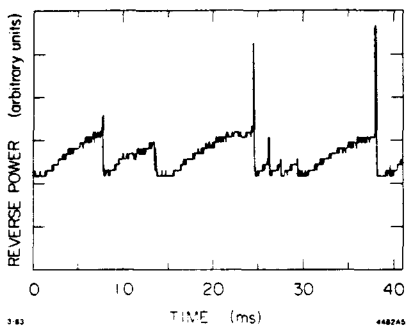

Fig. 6. Reverse power vs. time from the cavity undergoing occasional multipacting.

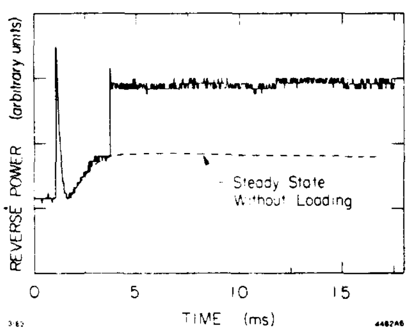

Fig. 7. Stable loading of the cavity due to multipacting.

The preliminary tests performed on TiN films 15 nm thick deposited on the tips as well as on the sides of the posts indicated that the multipacting phenomenon cannot be excited in the coated cavity. At 4.2K multipacting was never observed, but an instance of cavity loading was observed below 1.8K, when a superleak allowed helium into the cavity and possibly a plasma discharge was initiated between the posts. Tests performed on the same set of coated posts after the superleak was eliminated showed that the multipacting was not present at any temperature between 1.35K and 4.2K.

When only the end surfaces of the posts were coated with TiN the unloaded Q both at 4.2K and at 1.35K coincided with the Q_0's of the uncoated cavity. This fact indicates that, within the sensitivity allowed by the relatively low residual Q_0, the RF dielectric losses in the film could be considered negligible.

On the other hand, when the posts were coated also on parts of the lateral surface which included a current maximum Q_0 showed some degradation. At 4.2K it decreased from about 2.5×10^7 to 2.1×10^7 and at 1.35K it changed from 7.3×10^7 to 4.2×10^7. We estimate that the loss contribution of the coated parts represents between 30% and 40% of the losses which would be present if the whole cavity surface had been coated.

Although presently the low Q_0 does not allow us to set accurate values to the S-band current losses in TiN, there is evidence that at 1.35K they represent the dominant contribution to the cavity losses, while at 4.2K the niobium surface itself seems to determine the value of the unloaded Q.

Conclusions

The first set of measurements performed on the RF multipacting properties of an S-band doubly-reentrant niobium cavity has shown that multipacting is entirely suppressed when TiN films are deposited on selected regions of the surface. The cavity multipactors very easily without the coating. The films do not show any appreciable dielectric losses. The unloaded Q was the same for the uncoated cavity and when only the end surfaces of the posts were coated [$Q_0 = 2.5 \times 10^7$ (4.2K) and 7×10^7 (1.35K)]. Some current losses were observed when also part of the lateral surface of the posts was coated [$Q_0 = 2.1 \times 10^7$ (4.2K) and 4.2×10^7 (1.35K)]. These additional losses should not compromise the operation of superconducting Nb cavities at 4.2K when coated with TiN films.