CHARACTERISTICS OF TiN ANTI-MULTIPACTOR LAYERS REACHED BY TITANIUM VAPOR DEPOSITION ON ALUMINA COUPLER WINDOWS

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Abstract

A procedure of thin TiN layers generation on RF coupler surfaces was developed at DESY in order to reduce multipactor effects. Within last three years roughly 200 various components of couplers for Tesla Test Facility (TTF) have been TiN coated using titanium vapor deposition in ammonia. Surface layer properties which are essential for coupler performance, like secondary electron yield (SEY) and loss tangent have been studied. The loss tangent in RF field, measured for TiN coated cylindrical alumina coupler windows showed a considerable growth with rising layer thickness for thickness values higher than 9 nm. The chemical composition of a typical TiN antimultipactor layer was determined using XPS analysis. The received depth profiles revealed that titanium dioxide and titanium oxinitrides dominate TiN across the layer.

INTRODUCTION

A project aimed at reducing multipactor effects on vacuum-facing walls of RF couplers has been developed at DESY since 2000 . Titanium vapor deposition in low pressure ammonia has been successfully used for anti-multipactor protection of various coupler components like cylindrical, waveguide (planar) and coaxial windows, wave-guide sections and coax. line components. After reaching a basic vacuum with residual gas pressure below $5 \cdot 10^{-6}$ mbar, ceramic substrate surface is preheated to 120 – 170 ^{0}C due to infrared radiation emitted from a titanium wire (filament) heated to 1000 ^{0}C by dc current. Next constant ammonia flow is established at a pressure of 10^{-3} mbar under continuous pumping. Ti vapor is then deposited on the substrate within 30-60 s after rising the filament temperature to 1520 ^{0}C. Final Ti ->TiN conversion takes place after deposition at ammonia pressure of 100 – 300 mbar for ca. 10 hours.

So far TiN coating of roughly 200 components of currently used TTF2 and TTF3 coupler versions has been performed which resulted in visible reduction of multipactor, considerable decrease of needed preconditioning time (especially in the case of "cold" part of coax. line of TTF3 coupler for up to 1 MW peak power and wave-guide windows of TTF2 couplers) and improving power transmission. A positive effect of similar anti-multipactor measures applied for twenty TTF4 couplers (designed for "superstructure") has been also verified.

In several TTF3 couplers ceramic heating occurred (up to 75 ^{0}C) in cylindrical "warm" windows installed in the warm part of the coax. line which separate vacuum from ambient atmosphere. As potential reasons one can include on the one hand specific local RF field configuration resulting in excessive power loss in this area or power losses within TiN protective layers on the other. Therefore, measurements of loss tangent has been performed for these windows before and after TiN coating.

To shed more light on SEY values of evaporation TiN-coated layers on alumina which underwent RF conditioning, a series of SEY measurements were carried out on a coated sample, taking into account the impact of in-situ vacuum baking and electron bombardment. Main features of TiN layer chemical composition have been investigated using X-ray induced photoelectron spectroscopy (XPS).

ALUMINA PROPERTIES AND SAMPLE PREPARATION

Ceramic windows for TTF couplers at DESY are commonly made of AL-300 alumina. Its main properties are listed below.

Table 1: Properties of AL-300 alumina

Alumina content	97.6%	[1]
Density	3.76g/cc	[1]
Flexural strength	296 Gpa	[1]
Dielectric constant (1.43 GHz)	9.25-9.4	[2]
Dielectric loss tangent (1.43 GHz)	$(1\text{-}3)\cdot 10^{-4}$	[2]
Surface roughness R_a	1.1 μm	[2]

Four complete ceramic rings of TTF3 warm windows (external diameter 75 mm, thickness 6 mm, height 49 mm) were used as samples for loss tangent determination. The rings were TiN coated on the inner sides using the standard procedure described above after ultrasonic bath in ultra-pure water.

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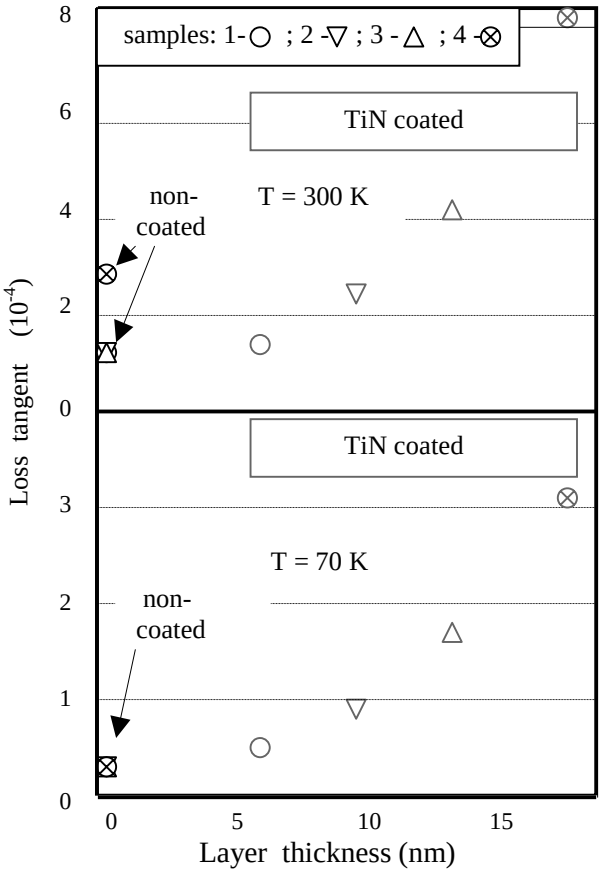

Figure 1: Effective loss tangent for cylindrical "warm" windows of TTF3 coupler as a function of surface TiN layer thickness at 300 K (the top part of the plot) and at 70 K.

Alumina samples of dimensions $8 \cdot 8 \cdot 3$ and $6 \cdot 20 \cdot 3.5$ mm^3 were cut out from another coupler window which had been TiN coated in a similar way up to 6 nm. They were used for SEY and XPS studies, respectively.

ESTIMATION OF RF POWER LOSSES

Design of couplers requires the knowledge of real ceramic window behavior described by relative dielectric constant and loss tangent. Since the latter can be changed by anti-multipactor coating of ceramic components, loss tangent measurements were done on warm cylindrical windows before and after TiN coating. Experimental setup designed for complex dielectric constant measurement included a cylindrical resonator excited in TM_{010} mode and computer programs which permit determination of fields and quality values (Q) of resonators with any dielectric objects [3].

Four samples have been tested before and after TiN coating on the inner cylindrical surface up to thickness of 6.5; 9.5; 12.5 and 17 nm at a room temperature (300 K) and at a temperature of 70 K. Layer thickness has been derived from filament temperature (measured with a pyrometer), geometry of the coating setup and deposition time. Layer thickness calibration vs deposition time has been checked using a quartz crystal thickness monitor.

Loss tangents values of the four cylinders before coating ranged from 10^{-4} to $3.5 \cdot 10^{-4}$ for resonance frequency of 1427.87 MHz. Coating to 6.5 nm-thick layer did not increase ohmic RF losses considerably, compared to non-coated surface. Further thickness growth led to roughly linear increase of loss tangent to reach $8 \cdot 10^{-4}$ for 17 nm-thick layer. In our practice TiN layer thickness is kept between 6 and 9 nm which guarantees effective reduction of multipactor effects and a loss tangent value comparable to that of non-coated element. The respective loss tangent values at 70 K are approximately three times lower than at a room temperature.

The measured effective loss tangents include the ohmic loss of the surface layer and dielectric loss of ceramic. The entire power loss in a coated element is given by:

$$P_{loss} = \int_{V+v} \pi f \varepsilon_0 \varepsilon (tg\delta) E_{peak}{}^2 dV$$

where f is the frequency, E_{peak} - maximum electric field (0.85 MV/m) in TTF3 coax. line at maximum power of 230 kW, pulse length 1.3 ms, repetition 2 Hz and V and v are ceramic and film volumes, respectively. Taking into account the measured loss tangent values and the data collected in Table 1 one can estimate upper limits for power losses in a single warm window with 9 nm-thick TiN layer as 15 W at 300 K and 3 W at 70 K. The respective values for non-coated window amount to 10 and 1 W.

SEY MEASUREMENT

Secondary electron emission measurement was performed using a standard apparatus composed of an electron gun capable of generating a 0.2 mm diam. beam of 50 to 5000 eV electrons and a Faraday cup (FC) with a channel to pass the primary electrons onto a studied sample. The gun was equipped with a deflection and scanning system. As a first step the primary beam is deflected on FC to measure its current I_p. Then the beam is passed through the channel towards the sample, normally to its surface. The secondary electron yield is obtained from:

$$SEY = 1 - I_T/I_p$$

where I_T is the total current to the sample. An electrometer with two isolated channels with charge/digit conversion was applied to record I_T and I_p.

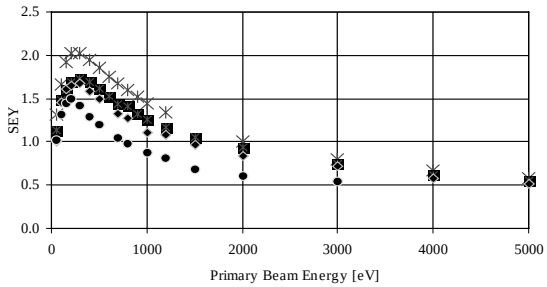

Figure 2: SEY vs primary electron energy (see the text).

Measurement of SEY as a function of primary electron energy was first performed on a TiN coated sample "as delivered" (the top curve in Figure 2), next - after in situ baking at 200 ^{0}C for 16 hours (the second curve from the top), followed by consecutive measurements after sample irradiation with 1100 eV electrons over a surface area of 5 · 5 mm^2 with a cumulated dose per unit area of 10^{-18} and 10^{-19} cm $^{-2}$ (the third and the fourth curve from the top, respectively).The bake-out and electron irradiation approach a typical coupler preconditioning process. Maximum SEY value decreased from 2.05 to ca. 1.7 as a result of baking (surface water desorption). Whereas the following e^- irradiation up to a dose of 10^{-18} cm^2 proved to be ineffective in reducing secondary emission, the last conditioning step (irradiation to 10^{-19} cm^2) gave visible maximum SEY drop to 1.5. SEY values remained bigger than one for primary energy between 50 eV nad 760 eV.

SURFACE CHEMISTRY

XPS studies of the alumina sample coated with 6 nm thick TiN layer were performed by use of ESCALAB 210 spectrometer . It is a constant energy analyser using MgKalfa radiation as an excitation source. $7 \cdot 10^{-9}$ mbar pressure is maintained in the spectrometer during measurement. The apparatus is equipped with an Ar^+ gun to sputter the studied surface.

XPS spectra were measured on "as received" sample and after each ion bombardment. After smoothing, satellite removing and non-linear background subtraction the spectra were fitted. The resulting depth profiles showed titanium compounds distribution across the surface layer. Table 2 includes contents of main atomic constituents of the film and TiN : TiO_xN_y : TiO_2 ratios at different depths from the starting sample surface. The depth is parametrized by J=SUM(I_{Ar}·t), where I_{Ar} is the Ar^+ current used for surface sputtering in consecutive steps and t - the sputtering time. The summation extends over all sputtering operations prior to measurement. At J = 180 microA·min substrate components (Al_2O_3) began to dominate titanium, showing layer-substrate interface.

Table 2: TiN surface layer composition

J microA · min	Contents Ti	N	O	C (atomic %)	TiN : TiO_xN_y : TiO_2 ratio
0	12	7.5	29	44	1 : 3.2 : 5.2
15	21	14.5	41	12	1 : 1.4 : 2.4
35	21.4	16	40	8.5	1 : 1.2 : 1.4
56	22	16.3	40	8	1 : 1.2 : 1.5
107	23.8	18	36	7.5	1: 0.98: 0.98
157	20.6	16	38	7	1 : 1.2 : 1.15
257	9.5	11.8	40	5.5	1 : 1.24 : 1.6

Apart from the top surface of the film, oxygen contents is close to 40 at. % across the whole layer . Ti/N ratio is much bigger than 1 at all depths above the interface. TiN concentration has minimum value (ca 2 at.%) on the starting surface because of high carbon presence due to surface contamination. It is also dominated by oxinitrides and titanium dioxide due to the oxidation by the atmospheric air. 1–2 nm thick surface TiO_2 layer is typical of TiN films after exposition to air, especially in a case of porous surfaces. TiN concentration reaches maximum at J =107 microA· min (equivalent to a depth of ca 3.5 nm) with the TiN : TiO_xN_y : TiO_2 ratio close to 1 : 1 : 1. At next (deeper) layers TiN contents is lower than those of titanium dioxide and oxinitrides which is due to TiN interaction with the substrate.

TiO_2 presence on the surface increases secondary emission. On the other hand relatively high TiO_xN_y concentration partially compensates for this effect and reduces RF losses due to high resistivity of grain boundaries [4].

ACKNOWLEDGMENT

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REFERENCES

[1] Data source: Wesgo Ceramics Hayward.
[2] Measured at DESY.
[3] B. Dwersteg, Z. Yu, "Measuring Complex Dielectricity Constant of Aluminum Oxide Window Ceramics for TTF-power Couplers at DESY", Proc. of the 10th Workshop on RF Superconductivity, Tsukuba, Japan, Sept. 2001, http://conference.kek.jp/SRF2001/ .
[4] Shigeru. Isagawa et al., "Coating Techniques and Improvement of High Power CW Klystrons for TRISTAN", EPAC'94, London, June-July 1994, p. 1912.