

# TITANIUM COATING OF CERAMICS FOR ACCELERATOR APPLICATIONS

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## Abstract

Titanium thin films can be deposited on ceramics, in particular alumina, without adherence problems. Even after air exposure their secondary electron yield is low compared to alumina and can be further reduced by conditioning or beam scrubbing. In addition, depending on the film thickness, titanium provides different surface resistances that fulfil requirements of ceramics in particle accelerators. Titanium thin films (MOhm range) are used to suppress electron multipacting and evacuate charges from ceramic surfaces. Thicker films (5-25 Ohm range) are applied to lower the surface resistance so that the beam impedance is reduced. In this contribution, we present the results of a development aimed at coating 2-meter long alumina vacuum chambers with a uniform surface resistivity by a dedicated DC magnetron sputtering configuration.

## INTRODUCTION

Ceramic windows are frequently used for transmitting Radio-Frequency (RF) power, while maintaining the separation between vacuum and atmospheric pressure. However, the electrically insulating materials used for the ceramic windows have a high Secondary Electron Yield (SEY), far above the typical value for air exposed metal surfaces, and can lead to multipacting. Part of the power of the RF is dissipated in the multipacting electrons reducing efficiency. In addition the electrons impinging on the walls can generate local heating, which in turn can lead to cracking due to thermal stresses [1]. In general lowering the SEY can be achieved by appropriate thin film coatings [2, 3]. For instance Non-Evaporable Getters (NEG) are an ideal candidate in an Ultra High Vacuum (UHV) environment, provided a baked-out to at least at 180 °C is possible (in addition, NEG thin films also pump most of the gas species present in UHV systems). On unbaked ceramics thin titanium films have proved their effectiveness also from the point of view of adhesion [4] and carbon coatings could be envisaged [3] as a solution in the future. In the case of RF windows the functional performance depends crucially on the correct electrical resistivity of the coating. Therefore in-situ resistance measurement during the thin film deposition process is systematically applied as a control method.

Ceramic vacuum chambers are also widely used for fast pulsed magnets, in order to avoid eddy current issues. Resistive titanium films are deposited inside these chambers to reduce the wall impedance perceived by the particle beam. In this case, as well, the surface resistance is measured during the coating process and after venting to air. Since the range of resistivity is tuned by the

thickness in the following two examples we present the so called “ultrathin” and “thin” titanium films.

## TITANIUM “ULTRATHIN” FILMS

RF ceramic  $\text{Al}_2\text{O}_3$  windows are coated by DC sputtering in a dedicated coating set-up. Ceramics are degreased and vacuum fired (2 hours at 800 °C under vacuum) prior to coating. A central titanium cathode is used in a cylindrical configuration to coat uniformly the inner surface of the ceramic window (Fig. 1). Prior to coating the system was pumped down to  $3 \times 10^{-7}$  mbar, without a bake-out. The coating takes 5 – 10 minutes at a plasma power of 50 W and Ar pressure of  $4 \times 10^{-2}$  mbar. The overall resistance ( $R_{\text{total}}$ ) is measured in situ during the coating process between the top and bottom metallic collars of the ceramic assembly. Special attention should be given to enable an appropriate electrical contact between the collar and the titanium thin film during the process.

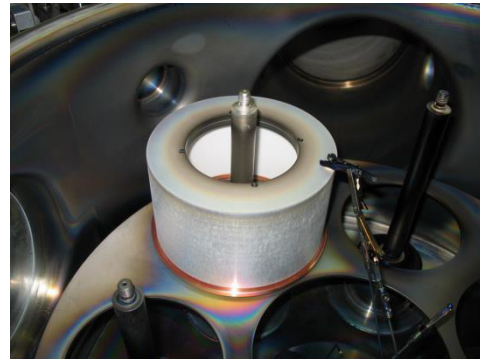


Figure 1: Cylindrical coating configuration for high resistivity coating of RF ceramic windows.

The square resistance ( $R_{\square}$ ), which is a shape independent quantity characterising the coating, can be calculated from  $R_{\text{total}}$  and the given geometry of the ceramic assembly (by multiplying times the length and dividing by the width of the rectangular developed surface). RF windows are generally coated with  $R_{\square} = 10\text{-}20$  MOhm, as measured in vacuum before venting. This is an empirically established value. By assuming the specific resistivity of bulk titanium this value would imply a thickness in the order of  $10^{-4}$  nm, which is not physical. In reality the coating probably forms clusters leading to a percolation path for the transport. As a consequence of the air exposure after coating, the  $R_{\square}$  increases to GOhm range values. Measurements on the resulting titanium coated  $\text{Al}_2\text{O}_3$  samples show a SEY reduction from 7 (for uncoated alumina) to 2 [6]. These values refer to samples that were transported in air from the coating system to the SEY measurement system.

Further reduction of SEY can be obtained by accumulating electron bombardment, i.e. by conditioning.

### TITANIUM “THIN” FILMS

For an external collaboration (MedAustron, Wiener Neustadt, Austria) CERN performed titanium thin film deposition on the inner wall of squared alumina chambers, 1.7 m long and 93.25 x 93.25 mm cross section, used for scanning magnets. The ceramic vacuum chamber requires a resistive layer, with a design  $R_{\square}$  in the range 20-200 Ohms after air exposure.

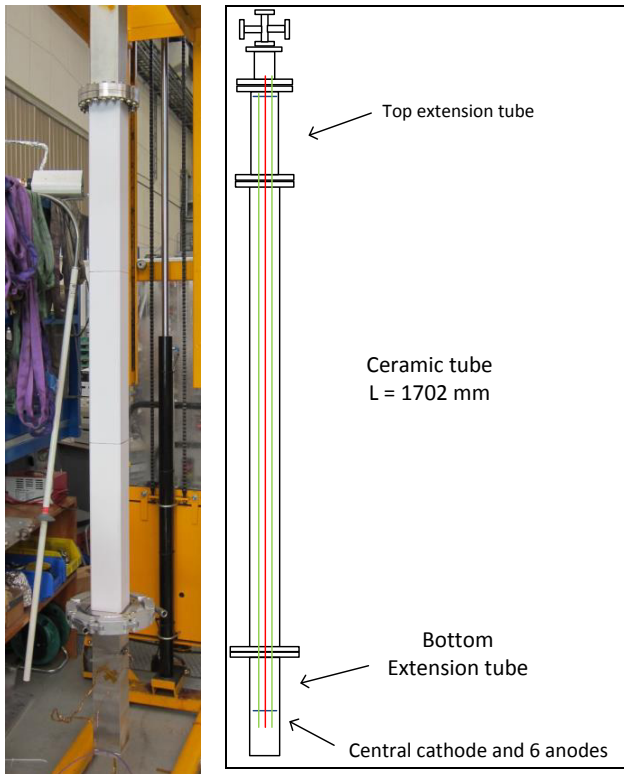


Figure 2: a) ceramic chamber in vertical position. b) scheme of coating assembly with extensions.

The ceramic chambers were cleaned and sintered at 1500 °C under atmospheric conditions prior to the brazing to flanges [7]. The coating was performed in DC cylindrical magnetron sputtering with the chamber in vertical position by using it as vacuum system for the process. Extensions with identical cross sections were mounted on top and bottom to avoid edge effects. Small copper substrates were mounted in the extension tubes, as close as possible to the ceramic chamber and were used to measure the coating thickness at the end of the process. In order to guarantee a uniform coating over the length and cope with the insulating properties of the ceramics, additional anodes were positioned around the central titanium cathode. In Fig. 3 a cross section of the cathode-anode-ceramic configuration is shown. The central cathode is composed of 3 twisted titanium wires of  $\varnothing$  3 mm. The anodes are made of Cu wires with  $\varnothing$  1.0 mm. In order to keep the cathode-anode configuration centred in

the ceramic tube, top and bottom centring plates hold the electrodes at a given position (Fig. 4 for bottom centring plate). Alumina parts were used to electrically isolate the cathode and the anode from the centring plates (which touched the inner wall of the squared extension tubes). The two centring plates were positioned in the 50 cm long extension tubes (Fig. 2a and 2b).

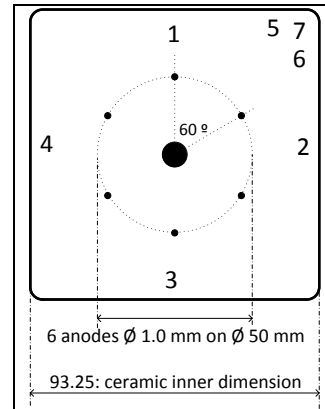


Figure 3; Cross section showing the cathode (central wire) and anodes (6 surrounding wires) in the ceramic chamber and positions (numbers) of the  $R_{\square}$  measurements.



Figure 4: Centring plate and weights for the anodes

In order to improve the purity of the titanium layer the coating assembly was pumped down and baked-out at 120 °C during 36 hours. Heating bands were enveloping the assembly and thermocouples placed at different positions, to assure a uniform bake-out of the assembly. Residual gas analysis was performed to validate the vacuum prior to coating. Final pressures before starting the coating were in the range  $2-3 \times 10^{-9}$  mbar. A 150 G magnetic field was provided by a long solenoid [8]. A Kr glow discharge was created at  $9 \times 10^{-3}$  mbar and the

uniformity of the power dissipation monitored by thermocouples mounted onto the external walls of the ceramic. The power was kept at 60 W, with the temperature rising only by 10 °C above room temperature and uniform along the 1.7 meter. The plasma was regularly interrupted to measure the resistance in vacuum between top and bottom flange of the ceramic chamber ( $R_{total}$ ). The process was resumed until the target resistance was achieved.

$R_{\square}$  of 20 to 200 Ohm after venting corresponds to a titanium coating thickness of 6 to 60 nm, if we take into account that the specific resistivity of the thin titanium films produced in this way is typically 3 times higher than for bulk titanium. Given the geometry of the chamber, the  $R_{total}$  of the coated ceramic tube can be considered equivalent to the resistance of 4.5 squares in series, which results in  $R_{total}$  of 90 to 900 Ohms. The chamber cross section is square, but the distribution of sputtered titanium will exhibit a cylindrical symmetry around the cathode axis. The central part of each wall (positions 1, 2, 3, 4 in Fig. 3) is expected to be covered with a thicker film than the corners (position 5, 6, 7 in Fig. 3). In order to remain in the specified range of resistance and to avoid a too thick coating at the central part of the wall or a too thin coating in the edges, the target value of  $R_{total} = 300$  Ohms was selected. Since this is defined as  $R_{total}$  after venting and the control measurements during coating were made in vacuum before any surface oxidation a reduced target value of 240 Ohm was chosen.

Table 1: Measured overall resistance  $R_{total}$ , calculated  $R_{\square}$ , and measured local  $R_{\square}$  by 4 point probe method.

Tube Nr	$R_{total}$ [Ohm]	$R_{\square avg}$ [Ohm]	$R_{\square 1-4}$ [Ohm]	$R_{\square 5-7}$ [Ohm]
2	302	67	72 +/- 28	199 +/- 64
3	349	78	79 +/- 20	139 +/- 36
4	268	61	55 +/- 15	100 +/- 31
5	284	63	67 +/- 38	149 +/- 74
6	294	67	69 +/- 33	98 +/- 55
7	313	77	97 +/- 27	225 +/- 95
8	310	69	84 +/- 37	108 +/- 54

In total 7 ceramic tubes were coated. The process time varied from 1h25' to 2h15'. After venting the resistance increased and additional oxidation in air during 1 night resulted in a stabilized resistance ( $R_{total}$ ). From this value the average  $R_{\square}$  for the tube could be calculated and was also measured locally by 4 point method [5] at 15 cm from the top and bottom flanges. Measurements were taken at the centre of tube wall (positions 1-4, fig 3) and close to the corners (5-7). An overview of resistivity values are in table 1. In the first prototype chamber  $R_{\square}$  was also measured at 45 cm from the flanges and the values were similar to those at 15cm. This measurement

is not without risk of scratching the film. For this reason they were not repeated for the whole series. The thickness of the titanium film on copper substrates placed in the extension tubes of the first 2 coating runs were measured by X-ray Fluorescence (XRF). The values vary between 0.08  $\mu\text{m}$  at the centre of the face and 0.04  $\mu\text{m}$  at the edges. As it is clear from the values in table 1, the measured  $R_{\square}$  fulfil the specifications for the required coating and show a sufficiently good reproducibility of the process.

## CONCLUSIONS

The method to produce ultrathin titanium coatings by DC magnetron sputtering with  $R_{\square}$  10-20 MOhm on ceramic parts is illustrated. These coatings induce a reduction of SEY and were successfully applied to hundreds of ceramic components, mainly for the LEP and LHC. Titanium films with  $R_{\square}$  of 60-80 Ohms were deposited on long rectangular ceramic chambers of scanning magnets with a dedicated DC cylindrical magnetron sputtering configuration. Total resistance measurements together with local surface resistivity measurements enable to control the coating process. The resulting values meet the specifications for the resistivity after venting.

## ACKNOWLEDGMENTS

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