TITANIUM NITRIDE COATING AS A MULTIPACTOR SUPPRESSOR

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Abstract

A particular interest is accorded, in LAL-Orsay, to the R&D and technology studies on RF power couplers for superconductive cavities. One of the most critical components of those devices is the ceramic RF window that allows the power flux to be injected in the coaxial line. The presence of dielectric window on a high power RF line has a strong influence on the multipactor phenomena. The most important method to reduce the multipactor is to decrease the secondary emission yield of the ceramic window. Due to its low Secondary electron Emission Yield (SEY), TiN thin film is used as a multipactor suppressor coating on RF ceramic coupler windows. In this frame work, TiN deposition was made by magnetron reactive sputtering. XPS and XRD analysis were performed to control the compositions and the stoechiometries of the obtained films. Coating thickness was optimized so that the TiN coating effectively reduces the SEY but does not cause excessive heating, due to ohmic loss. For this purpose, SEY measurements on covered and uncovered TiN Alumina substrates and multipactor level breakdown on TiN coated Copper substrates were performed for different deposit thicknesses.

INTRODUCTION

Ceramic windows are components of extreme importance in RF power coupler. They basically allow an RF power matching between air and ultra high vacuum parts. In superconductive accelerator technology, it permits also a transition between an ambient temperature medium and a cryogenic one. Alumina (Al_2O_3) is a common material for RF windows. Besides its high dielectric and mechanical strength, it is stable under thermal treatment and has a low out-gazing rate [1]. Nevertheless, it has a high secondary electron emission coefficient, which enhances the multipactor and limits the power coupler performances. One way to suppress the multipactor effect on Alumina ceramic windows is to coat it with a thin film material having a low Secondary Electron Emission Yield (SEY). Titanium Nitride is a good candidate for this purpose since its SEY is about 1.5 [2] rather than 7 for Alumina ceramic (97.6%) [3]. Moreover, the thickness of the coating must be carefully optimized: not too thin to lose its mutipactor suppressor characteristics, not too thick to increase the RF reflection coefficient on the window. A range of 7-15 nm thickness has been found a good compromise between these contradictory requirements [4].

TiN thin layers were deposited on stoechiometric sputtering mode. XRD and XPS analysis were performed

to control film stoechiometries and compositions. Layer thickness, in-situ monitored by a quartz crystal microbalance, was optimized so that the TiN coating effectively reduces the SEY but does not cause excessive heating, due to ohmic loos. Thus, SEY measurements on coated and uncoated Alumina substrates and multipactor level measurement on TiN coated copper substrates were performed.

EXPERIMENTS

TiN layers were deposited by reactive magnetron sputtering in a coating system developed in collaboration with Ferrara Ricerche Consortium-Italy. A detailed description of the machine was made in a previous paper [5].

Ti- TiN transition determination

Titanium to titanium nitride transition determination is studied at a given current value I=2 A, an argon flux rate Φ_{Ar}= 0.137 sccm, increasing gradually the N_2 flux rate. In the following, the curves presenting deposition rate and process pressure dependencies with N_2 flow rate variation are illustrated.
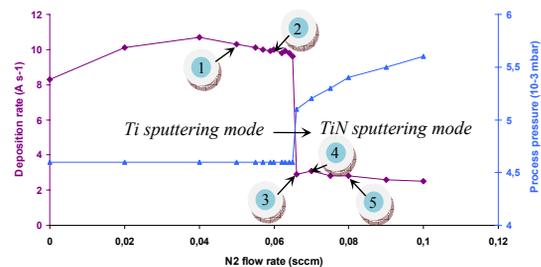
Figure 1: Deposition rate and process pressure variations with N_2 flow rate during Ti-TiN transition $I_{Sputtering}$ = 2A, Φ_{Ar} = 0.137 sccm.

Two important zones, corresponding to two different sputtering modes, can be distinguished when observing the curves. The first one called "Ti sputtering mode" where the deposit is metal rich, the second called "TiN sputtering mode" where the deposit is a combination between metal and nitride nearly stoechiometric. Those modes are separated by a sharp transition zone.

During titanium sputtering mode, and after a slight increase when we start introducing nitrogen (probably due to N_2 adsorption on quartz crystal surface covered with titanium), the deposition rate decrease gradually when increasing the N_2 flow rate till it incur a sharp drop in the transition zone. After that, the deposition rate is still almost the same in the TiN sputtering mode despite the continuous increase of the reactive gas flow rate.

The described deposition rate variation is due to the fact that the sputtering yield from a poisoned surface is less than the one from a pure metallic target. Thus, as the reactive gas partial pressure rise in the chamber, the target poisoning increases causing the decreases of deposition rate. The sharp drop observed during the transition corresponds to the entire target poisoned.

In the mean time, when observing the process pressure curve, we note that the value, stills constant during Ti sputtering mode, increases suddenly in the transition zone and still increases gradually during TiN sputtering mode. This is due to the fact that the amount of nitrogen firstly introduced in vacuum chamber was totally trapped in chamber wall, substrate and target surfaces coated with titanium. As long as we increase the N_2 flow rate, we tend to all surfaces "poisoning". Once this step reached, the nitrogen pressure increase rapidly in vacuum chamber (and thus process pressure as Argon flow rate is constant) indicating sputtering mode transition. Nitrogen added forward increase almost linearly the process pressure in TiN sputtering mode.

XRD analysis for stoechiometries determination

Several deposit samples are prepared in different sputtering conditions, corresponding to the two sputtering modes and the transition in the between (Figure 1). XRD plots permit the determination of the atomic lattice planes spacing d_{hkl} according to the Bragg's law:

$$d_{hkl} = \frac{n\lambda}{2\sin\theta}$$

(Where λ is the wave length of the x-ray source, for a Cu tube $\lambda=1.54056$Å)

Assuming that TiN_x crystallizes in a face centred cubic system; it is possible to calculate the lattice parameter for each deposit using the relation:

$$a_{TiN_x} = d_{hkl}\sqrt{h^2+k^2+l^2}$$

(Where (hkl) are Miller indices for diffraction plane)

From the obtained value of the lattice parameter, it is possible to calculate x, the N/Ti ratio, according to the relation below, valid in the range $0.6 < x < 1$ [6]:

$$a_{TiN_x} = 4.1925 + 0.0467\,x$$

The following table summarizes the results obtained:

Table 1: Stoechiometries determination of samples deposited in different sputtering mode

Sample	Ar (sccm)	N_2 (sccm)	I (A)	X (TiN_x)
Sample 1	0.137	0.05	2	0.78
Sample 2	0.137	0.060	2	0.73
Sample 3	0.137	0.066	2	0.97
Sample 4	0.137	0.070	2	1.02
Sample 5	0.137	0.08	2	0.90

The results in Table 1 fit well with the previous sputtering mode determination. Film Stoechiometries are close to 1 in the TiN sputtering mode, once we reach the transition. As this later is an instable zone, we will avoid to deposit in its corresponding conditions and stoechiometric deposits will be made beyond.

XPS analysis for film composition determination

An XPS profile of a 30 nm thick TiN sample has been realised by successive steps of acquisition and ion abrasion. A quick acquisition has been done first for carbon and oxygen to limit the sample surface recontamination and/or oxidation by molecules of residual vacuum. Abrasion speed is 3.5nm/min. A zalar rotation during ion abrasion has permit to minimize ceramic substrate roughness effect. Figure 2 shows profile curves of different elements (C, O, N, Ti and Al).
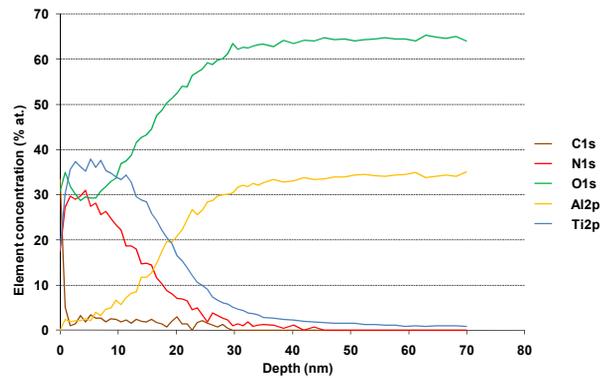
Figure 1: XPS profile of a 30 nm TiN deposit on Alumina substrate.

The first information that we can deduce from XPS profile is that the N/Ti ratio is about 0.8 at the sample surface, a little lower than the stoechiometric expected ratio. Gradually as we go depth, the atomic percentage of titanium and nitrogen decrease, till reaching zero. On the other hand, the atomic percentage of aluminium and oxygen, constant at the surface, increase gradually till reaching two levels when we achieve the bulk substrate material. These profile shapes are due to the substrate high roughness. If the Aluminium is almost absent in the sample surface (only 2-3%), a high percentage of oxygen is observed (nearly 30%). As titanium is a good getter for nitrogen and oxygen, the residual oxygen present in the vacuum chamber during sputtering process is in competition with reactive gas for titanium adsorption site occupancy. Thus, a particular attention should be paid to have the lowest base pressure before starting the sputtering process in order to minimize the amount of residual oxygen. Another source of deposit contamination by oxygen is the XPS method itself as the residual oxygen in analyse chamber could interact with the deposit during acquisition or ionic abrasion. A very low carbon atomic concentration (2-3%) was also observed in the deposit.

SEY measurements on TiN coated Alumina at different thicknesses

The current bias during the sputtering process influences significantly the TiN deposition rate. Thus, a current bias I= 2A gives a deposition rate $R_{Dep} = 2.6 \pm 0.2$ Ås^{-1}, while

I= 3A gives an R_{Dep} = 4.2 ± 0.2 Ås^{-1}. This sputtering rate difference may induce a deposit roughness change, which could have an incidence on the TiN SEY measured value.

Several TiN deposit samples of different thicknesses were prepared on alumina substrate, at two different current bias values (I= 2A and I= 3A). SEY measurements were performed thereafter, and the results are summarized in the following plots:
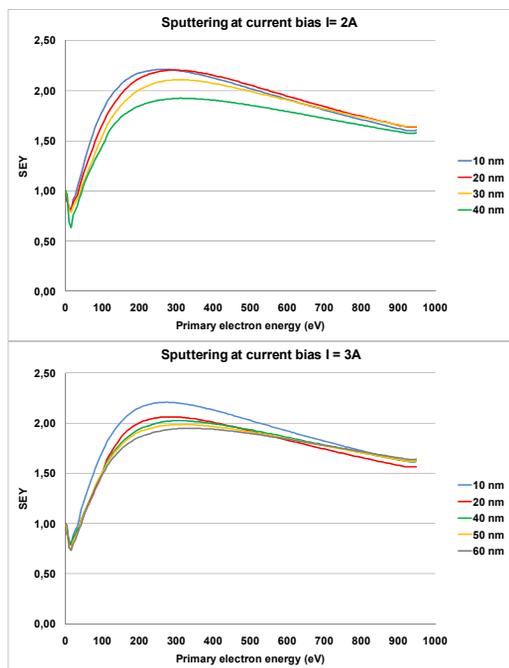
Figure 3: SEY measurements of TiN coated Alumina at different thicknesses and two different current biases.

An important decrease of the SEY from 8.7 for alumina to an average of nearly 2 for the coated samples was observed. This significant decrease is observed even for very thin TiN layer, which indicates the efficiency of the coating in its desired role. As the thickness of the deposit grows, the measured SEY decreases increasingly. However, this variation still slight compared to the one observed between coated and uncoated alumina. This is a good point that we can follow up when we have to measure coated ceramic resistivity. In fact, a trade off should be done between SEY and resistivity when optimizing deposit thickness. Roughness (current bias) seems to have no influence on the measured SEY values. This is probably due to the high substrate roughness like shown in the SEM photo below:
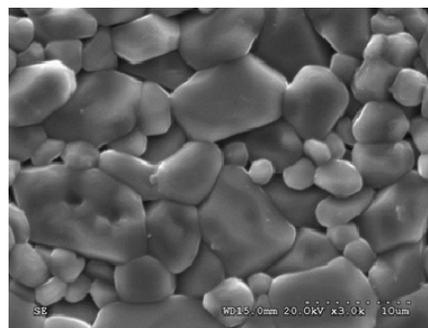
Figure 4: Scanning Electron Microscopy photo of alumina substrate.

Stoechiometric deposit conditions are now well defined. A particular attention must be paid to the base pressure before sputtering process start to avoid deposit contamination by residual oxygen.

TiN deposit, even in thin layer, shows a very good aptitude to lower SEY of alumina. This later decrease inversely with the thickness, but still insignificant regarding to the one observed between coated and uncoated alumina.

No influence of deposit roughness (current bias) on measured SEY values is observed due to the very high roughness of the substrate.

ACKNOWLEDGMENTS

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