

TITANIUM NITRIDE COATING AS A MULTIPACTOR SUPPRESSOR ON RF COUPLER CERAMIC WINDOWS*

W. Kaabi[#], H. Jenhani, A. Variola, LAL, Univ. Paris-Sud, CNRS/IN2P3, Orsay, France
G. Keppel, V. Palmieri^{‡§}, LNL/ INFN, Legnaro, Italy

Abstract

LAL-Orsay is developing an important effort on R&D and technology studies on RF power couplers for supraconductive 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 a 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 coefficient, TiN thin film is used as a multipactor suppressor coating on RF ceramic coupler windows. In this frame work, a TiN sputtering bench has been developed in LAL. The reactive sputtering of TiN needs the optimisation of the gas flow parameters, as well as the electrical one, to obtain stoichiometric deposit. XRD analysis was performed to control the film composition. The obtained measurements point out how the Nitrogen vacancy on the film can be controlled very well acting on the N₂ flow during the reacting sputtering process. As the coating thickness should be optimized so that the TiN effectively reduces the SEE coefficient but does not cause excessive heating due to ohmic loss, and before multipactor level breakdown and resistance measurements for different deposit thicknesses, a good control of film thickness is essential. In-situ deposit thickness measurements given by quartz crystal microbalance was compared to X-Ray reflectivity one. A very good agreement was found between those two methods pointing out an efficient thickness and deposition rate monitoring. Once those steps accomplished, an optimisation of deposition rate was performed in stoichiometric gas flow rate and electrical conditions.

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 (Al₂O₃) 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 multipactor 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].

In this frame work, a sputtering machine was developed in LAL, allowing very thin TiN layer coating on ceramic. The coupler operating conditions, the physical properties of TiN layer and alumina substrate in addition to windows geometries have defined the strict constraints that have been taken into account in the definition of the coating bench design. Along this study, stoichiometry and thickness of TiN deposits are the main parameters to be investigated. XRD analysis will permit film composition control while XR-reflectivity and quartz crystal microbalance measurements will allow thickness control.

EXPERIMENT

The Sputtering System

The conception and the development of the magnetron sputtering system were made in collaboration with Ferrara Ricerche Consortium-Italy in the frame of CARE program. An overview of the magnetron sputtering system is presented in Fig. 1.



Figure 1: Sputtering system Overview.

A detailed description of the sputtering bench was made in a previous paper [5].

*Work supported by CARE Program

[#]kaabi@lal.in2p3.fr

[‡]Padova University

[§]Ferrara Ricerche Consortium

Film Stoichiometry Determination

For an imposed current $I=3A$, and by maintaining the Ar flow rate constant at $f_{Ar} = 0.1$ sccm, several TiN_x layers of ~ 500 nm were deposited for different values of N_2 flow rate. XRD analyses are performed to control film stoichiometry. From the plot $I_r = f(2\theta)$ (where I_r is the relative intensity of diffracted X-ray and 2θ is the angle between X-ray source, substrate and detector), it is possible to determine spacing between the planes in the atomic lattice d_{hkl} by applying Bragg's law:

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

(Where λ is the wave length of x-ray source, in this case we have a Cu tube with a λ of 1.54056\AA).

Then, it is possible to calculate the lattice parameter for each deposit, assuming that TiN_x crystallizes in a face centred cubic system 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]:

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

Figure 2 shows XRD patterns for some TiN_x layers obtained according to the protocol cited below.

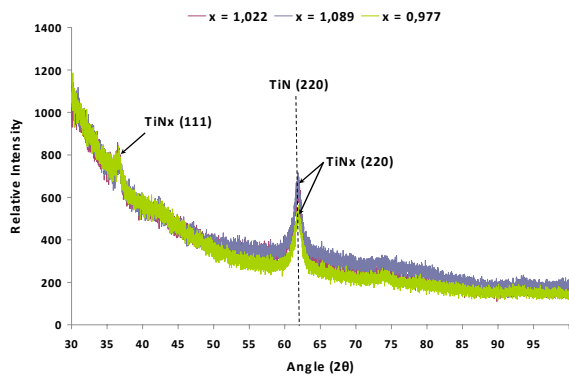


Figure 2: XRD plot for different TiN_x deposits compared to stoichiometric film.

Considering the sharp and relatively intense peak corresponding to (220) direction, we calculate x , the N/Ti ratio, for different deposited TiN_x films according to the method cited previously:

Table 1: Lattices Parameter and Correspondent Stoichiometry of Deposits at Different N_2 Flow Rate

Ar (sccm)	N2 (sccm)	I (A)	2θ	d (220) (\AA)	a (\AA)	x(TiNx)
0,10	0,11	3	61,8629	1,50115	4,2459	1,1423
0,10	0,12	3	61,8430	1,50028	4,2434	1,0896
0,10	0,13	3	61,8938	1,49917	4,2403	1,0223
0,10	0,14	3	61,9277	1,49843	4,2382	0,9775
0,10	0,15	3	62,1270	1,49410	4,2260	0,7153

Stoichiometric films are obtained for a N_2 flow rate between 0.13 and 0.14 sccm for a fixed Ar flow rate of 0.1 sccm and an imposed current of 3A. We note that increasing nitrogen flow rate causes the decrease of TiN lattice parameter, and consequently x the N/Ti ratio. This behaviour seems to contradict what was expected. In fact, according to the theory, an increase of nitrogen flow rate should increase the N/Ti ratio till reaching the stoichiometry; such results were obtained by Hugen et al. [7].

The behaviour difference of our experiments is due to the fact that we are already in sub-stoichiometric conditions. Thus, a further increase of nitrogen flow rate acts more on increasing poisoning phenomenon, and thus modifying process pressure and deposition rate, than modifying the film stoichiometry. A similar behaviour was reported by Huang and co-workers [8]. Concerning the little variation in sub-stoichiometric conditions, several studies have found that the phase can have a significantly greater lattice parameter that can reach 4.3\AA [9] (although than 4.24\AA). To explain this lattice expansion, it was assumed that two factors can be responsible. One is nitrogen entrapment in non equilibrium positions during film growth. The second is internal macrostrains [8].

Deposition Rate Variation with Process Pressure

Deposition rate is a very important parameter to consider in so far as it can influence the film morphology, its physical and mechanical properties, and even chemical composition since a low deposition rate of TiN could increase its oxidation. In our case, in addition to the cited reasons, the choice of deposition rate is also an operating criterion. In fact, as we need to deposit some nanometers of stoichiometric TiN, we should choose the appropriate value so that the process occurs in a reasonable time in a good process conditions.

Once the stoichiometric parameters are reached ($f_{Ar} = 0.1$ sccm, $f_{N_2} = 0.14$ sccm, $I = 3A$), we will now vary the flow rate for both gases keeping the ratio relative to stoichiometric conditions ($f_{Ar} / f_{N_2} = 0.71$). The aim is to vary process pressure without modifying stoichiometric conditions and see the influence of such variation on deposition rate. Results are shown in Fig. 3:

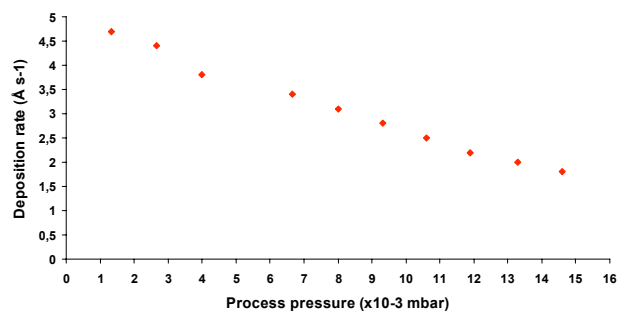


Figure 3: Deposition rate dependency with process pressure.

As we can see, dependence is quite linear, which means that even when process pressure change for a given argon and nitrogen flow rate setting (due to base pressure change or a better getter effect of the vacuum chamber's walls), we can estimate the deposition rate and so the time required for a given thickness obtaining just by process pressure reading.

Thickness Control of Stoichiometric Deposit

A quartz microbalance is integrated to the bench allowing an in-situ monitoring of deposition rate and film thickness. However, the quartz crystal placed at the internal of vacuum chamber, is oriented in such way that it receive sputtered atoms from both targets (Fig. 4).

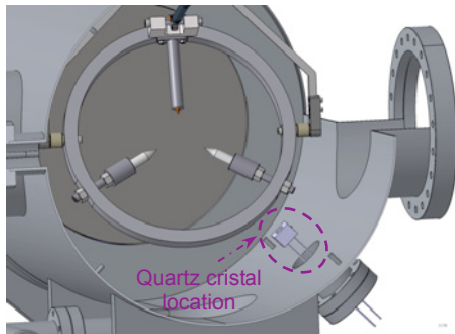


Figure 4: Section view of vacuum chamber with the localisation of quartz crystal holder.

This configuration permits a good monitoring of the deposition in internal and external faces of cylindrical samples, but may be not the samples receiving deposit just from one target. That's why, a correlation must be found between the film thickness estimated by microbalance and the real value obtained on the substrate.

Several thin deposits are made for different deposition rate and/or different time deposition, then analysed by X-ray reflectivity to measure their thickness and thus correlate them to the value given by microbalance.

A simulation soft allow as the determination of electronic density, substrate and film roughness, and layer thickness. If the first two film characteristics still somewhat approximated, the third one is very precise. In the Table 2 we present the results of the subsequent analysis making a comparison with the thickness estimated by the microbalance.

Table 2: Reflectivity Simulated Curve Results and Comparison Between Expected Thickness (μ -Balance) and Measured One

Expected thickness (nm)	Measured thickness (nm)	Density (g cm-3)	Roughness (nm)
16.2	16.2	4.4	1.9
23.4	23	3.48	2
36.0	37	3.4	3.9
71.1	72	3.09	7

A very good agreement is found between real thicknesses measured by X-Ray reflectivity and estimated one from microbalance. In fact, the difference between the two values doesn't exceed 3% in all exploitable range. Thus, the fact that quartz crystal emplacement allow the coating from both target is compensated by the position of our samples in front of the target. This result is very important since for a cylindrical window placed in the middle of the chamber, we can have the same deposit thickness in internal and external side of the cylinder receiving deposit from both target, and on the bottom and top grooves exposed to only one target. The movement of the sample holder will assure the difference of thickness between those two parts as it is required in sputtering machine specifications.

ACKNOWLEDGEMENTS

We acknowledge the support of the European Community-Research Infrastructure Activity under the FP6 "Structuring the European Research Area" programme (CARE, contract number RII3-CT-2003-506395).

We acknowledge M. Lacroix, Y. Peinaud, A. Thiebault, P. Lepercq, E. Herry, C. Prevost and B. Mercier for their precious help. A particular thanks to P.A. Besombes that follows all the administrative procedures for the TiN bench and X-Ray Diffractometer.

REFERENCES

- [1] Suharyanto, S. Michizono, Y. Saito, Y. Yanano and S. Kobayashi, Vacuum 81 (2007)
- [2] J. Lokiewicz, T. Fadina, J. Kula, A. Bilinski and Z. Yu, SRF 2003, DESY, Germany
- [3] N. Hilleret, Workshop on High-Power Couplers for Superconducting Accelerators Jefferson Lab, Newport News, Virginia, USA (2002)
- [4] J. Lorkiewicz, 10th Int. Workshop on RF Superconductivity, KEK, Tsukuba, Japan (2001)
- [5] A. Variola, W. Kaabi, H. Jenhani, P. Lepercq, G. Keppel, V. Palmieri and F. Strada, EPAC 08, Genova, Italy (2008)
- [6] S. Nagakura, T. Kusunoki, F. Kakimoto, Y. Hirotsu, Journal of Applied Crystallography 8 (1975)
- [7] J. H. Huang, K. W. Lu and G. P. Yu, Surface & Coating Technology, 191, (2005)
- [8] H. H. Huang, M. H. Hon, Thin Solid Films, 416, (2002)
- [9] V. Valvoda, R. Cerny, R. Kuzel Jr, J. Musil and V. Poulek, Thin Solid Films, 158-2 (1988)