Titanium Nitride Coating of RF Ceramic Windows by Reactive DC Magnetron Sputtering

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TITANIUM NITRIDE COATING OF RF CERAMIC WINDOWS BY REACTIVE DC MAGNETRON SPUTTERING*

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Abstract

LAL-Orsay is developing an important effort on R&D and technology studies on RF power couplers for superconductive cavities. These are complex and high technology devices due to their basic functions: vacuum and temperature separation form the environment to the cavity. One of the most critical components of high power couplers 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 in fact a strong influence on the multipactor phenomena, a resonant electron discharge that is strongly limiting for the RF components performances. 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 the framework of the EU program FP6 the LAL-Orsay and the LNL-Legnaro establish a collaboration to develop a coating bench that takes into account the different strong constraints on stoechiometry and film coating thickness given by coupler operating conditions. Reactive magnetron sputtering technology was chosen to obtain such deposit. A full description of a sputtering bench recently installed in LAL, and its main characteristics are given. Stoechiometric TiN films are obtained by optimization of reactive gas flow (N_2), for a given bias and a given ionisation gas flow (Ar). XRD analysis was performed to control film composition. From the data obtained, lattice parameter is calculated for each deposit and film stoechiometry is determined. XPS analysis of stoechiometric film had shown the existence of oxygen and carbon mainly in the surface. However, it shows also that the ratio Ti/N in atomic percentage is equal to 1.

INTRODUCTION

Ceramic windows are components of extreme importance in HF 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 RF 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]. At the same time 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].

In the frame of coupler research program in LAL, the topic of "Coating and Surface Studies" has been recently created. This activity belongs to coupler conditioning studies; its goal is to ameliorate surface properties, to avoid as well as possible the occurrence of multipactor phenomena and thus the decrease of conditioning time.

EXPERIMENT

Coating system description

The technology chosen for TiN coating of RF ceramic windows is the reactive DC magnetron sputtering. For this purpose, a sputtering bench has been developed with the collaboration of the consortium Ferrara Ricerche-Italie in the frame of CARE program.

Figure 1: Magnetron sputtering system overview.

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As sputtering is a vacuum process, the machine is equipped with a pumping system (turbomolecular and scroll pumps) that allows achieving a base pressure of 10^{-8} mbar in the vacuum chamber, before the introduction of ionisation gas (Argon) and reactive gas (Nitrogen) and the starting of coating process.
In the both sides of vacuum chamber, the machine is equipped with a 10 inch titanium disc target of high quality (grade 2, minimum 99.7 % Ti). Two rotary magnet packs are placed just behind the targets to increase plasma density at their surface, and thus ameliorate sputtering yield (Figure 2-B). A special rotating sample holder was designed to permit uniform deposition on cylindrical ceramic windows (Figure 2-C). The machine permits also the RF etching of the substrate (Figure 2-D), a pre-treatment step in order to remove particle contamination, as it is not possible to clean ceramic with solvent due to the high porosity of the material and the possibility of solvent trapping. RF etching allows also enhancement of TiN adhesion by increasing substrate surface roughness.

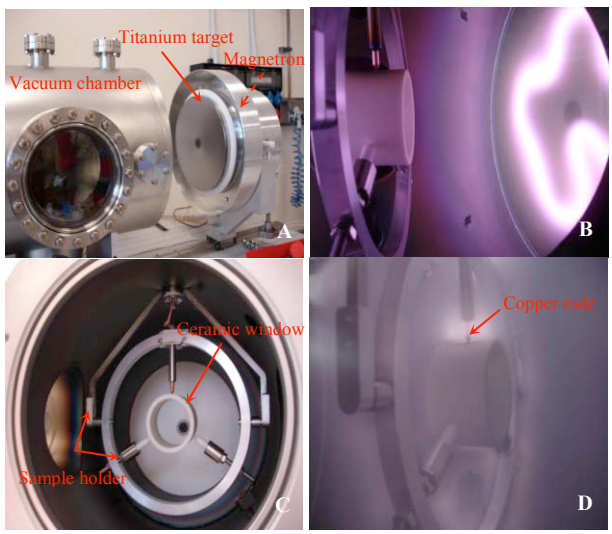

Figure 2: Components of sputtering machine
(A) Titanium disc Target and vacuum chamber; (B) plasma confinement in target surface by rotary magnetron; (C) Sample holder; (D) RF etching.

The coating thickness optimisation is an important point to consider. Thus, TiN film should be thick enough to reduce significantly multipactor when a coated ceramic window is placed in an RF field, but this thickness should not be excessive to avoid heating due to ohmic loss [5]. A crystal quartz microbalance integrated to the bench allows following the deposit thickness and the deposition rate during the process.
In order to obtain a stoechiometric TiN, reactive sputtering process needs the optimisation of gas and electrical parameters. Thus, the bench includes a mass flow controller for gas process (Argon) and reactive gas (Nitrogen), in addition to an adaptable power supply for each target.

Results and discussions

During this parameter optimisation step, all deposits were made on 10x10mm quartz substrates. The latter are cheaper than alumina substrates and suitable for XRD analysis.
For a given bias and by maintaining constant Ar flow, the N_2 flow variation leads to different stoechiometry TiN_x films. XRD analysis was performed to control film stoechiometry. From the plot $I = f(2\theta)$ (where I is the 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 1.54056Å)
Since TiN_x crystallize in a face centred cubic system for $0.605 \leq x \leq 0.999$ [6], 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 planes)
Thus, by comparing the obtained values with the lattice parameter of stoechiometric TiN ($a_{TiN} = 4.239$ Å [7]), we can check the right reactive gas flows (N_2) for a given electrical parameter and gas process (Ar).
Furthermore, from the obtained value a_{TiN_x}, it is possible to calculate x, the N-Ti ratio, by the relation [6]:

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

thus, obtained the right stoechiometry of the film.
In the following table, we summarize the results obtained for several deposit at the same applied current of 3A and the same Argon flow of 0.1 sccm.

Table 1: lattices parameter and correspondent stoechiometry of deposits at different N_2 Flow

Ar (sccm)	N2 (sccm)	I (A)	2θ	d (220) (Å)	a (Å)	x(TiNx)
0,10	0,11	3	61,8629	1,5012	4,2459	1,1423
0,10	0,12	3	61,8430	1,5003	4,2434	1,0896
0,10	0,13	3	61,8938	1,4992	4,2403	1,0223
0,10	0,14	3	61,9277	1,4984	4,2382	0,9775
0,10	0,15	3	62,1270	1,4941	4,2260	0,7153

Stoechiometric films obtained at these parameters have a deposition rate of 0.9 $Å s^{-1}$. It is possible to modify this value by acting on substrate bias and/or gas parameters. However, this rate seems suitable, especially when we should deposit only few nanometers in a reasonable time.

Figure 3 shows XRD graph, $I = f(2\theta)$, for some deposition represented in table 1.

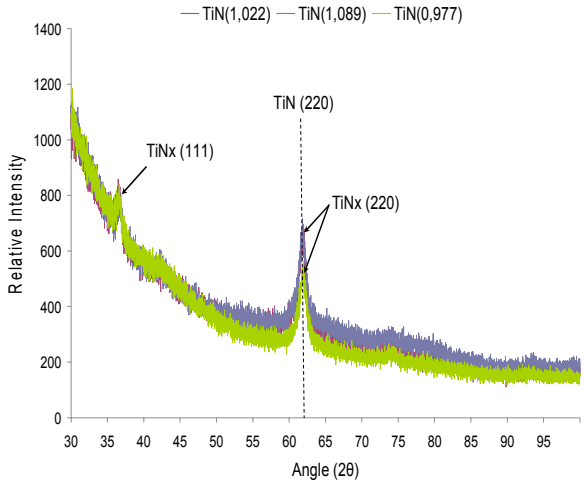

Figure 3: XRD plot for different TiN_x deposits-comparison to stoechiometric film

Spacing between the planes in the atomic lattice d_{hkl} is obtained by analyzing the peak (220) for each deposited film. The deposits seem have preferential orientation with the plane (220) parallel to the substrate surface. Lattice parameter a_{TiNx} and x given in table 1 are also calculated according to the same plane.

An XPS analysis of a stoechiometric film (deposited by an other magnetron sputtering system into Alumina ceramic disc substrate) shows that, in addition to titanium and nitrogen atoms, a relatively high atomic percentage of oxygen and carbon is present at different depths. The results of this analysis are shown in the table 2:

Table 2: element atomic percentage and Ti to N ratio in a stoechiometric deposit at different depth.

	C	O	Ti	N	Ti/N
1st abrasion (≈ 12 nm)	15.1	15.8	35.2	33.8	1,04
2nd abrasion(≈ 60 nm)	6.8	14.6	39.4	39.2	1
3rd abrasion(≈ 120 nm)	6.6	13.2	39.9	40.3	0,99

In spite of carbon and especially oxygen atom presence in different depth of the stoechiometric film, the atomic ratio of Ti to N is close to 1. This indicates that reactive process during sputtering is well optimised. The high oxygen and carbon atomic percentage can be explained by the fact that the vacuum chamber was not clean enough. In the case of our bench, results will certainly be better.

CONCLUSIONS

In the framework of a joint research activity an innovative design of a sputtering coating bench has been realised by Ferrara ricerche consortium, LNL-Legnaro and LAL-Orsay. The goal is to be able to coat different geometry of Alumina windows (planar and cylindrical) with nanometric and stoechiometric TiN films. A first phase of the machine design was successfully worked out. A particular rotating system that allows better coating uniformity in the cylindrical windows has been implemented. The bench has been realised and tested in Orsay on different samples with a thick deposition. This was provided to first study the process and than to analyse the film. The first results show a very good stoechiometric ratio between Titanium and Nitrogen. The coating velocity deposition has been roughly defined and it will provide in the future the process parameters to define the procedure to obtain the nanometric films.

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