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Molybdenum sputtering activity at INFN - LNF

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Abstract

As known the SALAF Group activity is presently dedicated to set up an X-band, accelerating structure, using different materials and methods. Here we report the tests made by sputtering molybdenum thin film on different samples, finalized to achieve a real X-band copper-molybdenum sputtered, three cells standing wave accelerating structure.

EXPERIMENTAL ACTIVITY

Sputtering has proven to be a successful method of coating a variety of substrates with thin films of electrically conductive or non-conductive materials. One of the most striking characteristics of sputtering is its universality. Since the coating material is passed into the vapour phase by a physical rather than a chemical or thermal process, virtually any material can be deposited.

According to our experience, preliminary studies show that molybdenum thin films deposited by RF magnetron sputtering, are friendly (and therefore less sensitive to the device) to obtain a better adhesion to the substrate than those deposited by DC magnetron sputtering with the same operational conditions, like argon pressure and RF power and also that the grain sizes of the deposited films grows by increasing of the same parameters [1].

To this end, a sputtering benchmark has been realized [Fig. 1]. To obtain a homogeneous and very tight bond molybdenum film on copper substrate several tests have been done, so that a procedure as been developed.

The device is equipped with a pumping system (turbo molecular and scroll pumps) to reach a base pressure up to 10^{-7} mbar in the vacuum chamber, before introducing an ionization gas (Argon) by means of mass flow controllers and to start of coating process. Magnetron set-up coming with a 2 inch molybdenum disc target of high quality (grade 2, minimum 99.7 % Mo). The DC glow discharge of the substrate is used as a pre-treatment step, in order to remove a possible contamination of water and to allow an enhancement

of molybdenum adhesion by increasing substrate surface roughness. The process starts introducing Argon at pressure of about 10^{-2} mbar and glow discharge was stabilized at 1.5 A resulting in a potential of about 200V between the central molybdenum anode and the grounded vacuum chamber, so that Argon ions act like a broom on this. After that the power supply is switched to RF and the coating process starts.
By using a RF (13,56 MHz, max 1KW) power supply at 60W, the coating took place at room temperature with a deposition rate of about 30nm/min. The deposition thickness has been measured by FILM thickness monitor.

Fig 1 Sputtering set-up.

To evaluate the surface quality and to understand the morphological aspects we used an Atomic Force Microscopy (AFM) as already reported [2]**.**
In figure 2 we show one surface of the machined copper with a roughness of about 70 nm before the molybdenum sputtering process. Clear undulations of the surface due to the step of lathe machine (~700 nm) with many overlapping spikes appear.

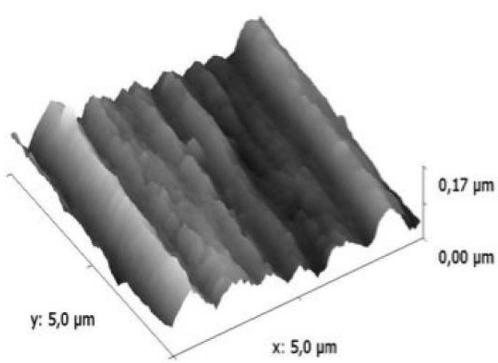

Fig. 2: AFM image of the surface of a copper sample before the molybdenum sputtering.

In figure 3 the same spikes disappear after a deposition of a ~100nm Molybdenum film. The film on the copper surface acts like a smooth layer improving the roughness of the surface.

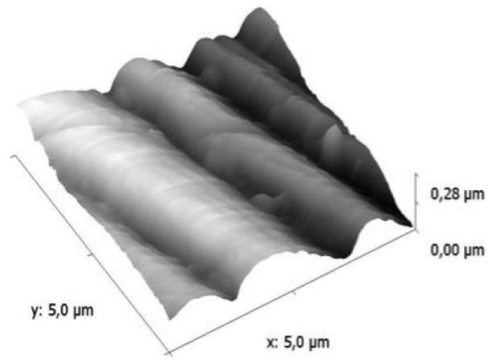

Fig. 3: AFM image of deposited molybdenum on copper by the sputtering technique

Actually, seems that the initial low roughness of the Cu machined surface is preserved or improved by Mo sputtering.
In order to test this statement some dedicated sample has been prepared.
In particular molybdenum film has been deposited on copper and glass substrate according to the following table:

	Moly Thickness	Deposition time	Magnetron Power	Argon pressure
Copper substrate	300nm	600 sec	60W	$2 \cdot 10^{-2}$ mbar
Copper substrate	600nm	1200sec	60W	$2 \cdot 10^{-2}$ mbar
Copper substrate	900nm	1800sec	60W	$2 \cdot 10^{-2}$ mbar
Glass substrate	300nm	600sec	60W	$2 \cdot 10^{-2}$ mbar
Glass substrate	600nm	1200sec	60W	$2 \cdot 10^{-2}$ mbar
Glass substrate	900nm	1800sec	60W	$2 \cdot 10^{-2}$ mbar

Using Rutherford Back Scattering technique [3], chemical composition and thickness of samples made on copper substrate will be analyzed for a cross check.
In particular, preliminary tests made on copper disc sample sputtered with 300nm of moly and then annealed at 300 ºC for 2 hours, in order to fix the film on the substrate achieving a homogeneous coating and a reasonable contact force between the Cu and the Mo layer, show that the film is uniformly contaminated vs. depth by oxygen (about 30%). It is shown in Fig. 4 and it is not clear if the contamination is due to air storage after deposition run or if it is due to the coating process.

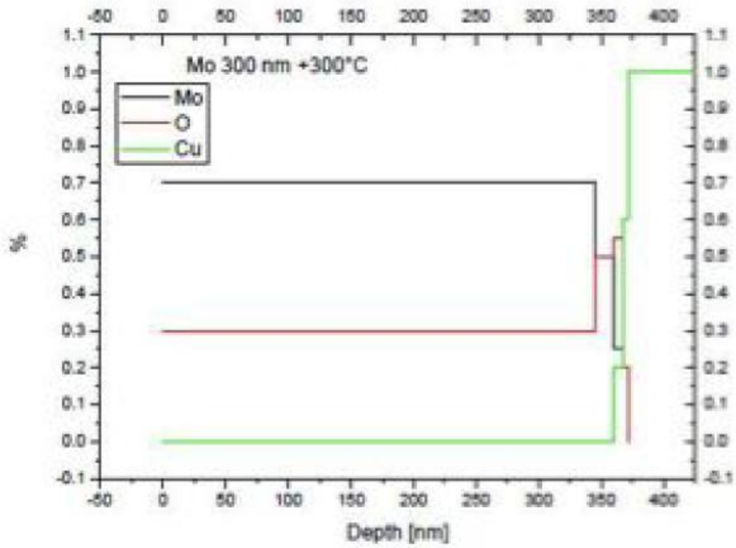

Fig. 4: Chemical composition of a 300 nm molybdenum film deposited on a Cu disk and treated at 300 ºC for 2 hours.

Anyway, studies are in progress in order to understand this statement and to reduce the film oxygen contamination which could be intolerable.
Tests with Atomic Force Microscope are also in progress [4] on samples made on glass substrate in order to evaluate if the film roughness quality is preserved by sputtering process. Further characterizations and cross-checks are in progress and they will be described in a forthcoming paper.
RF tests on glass substrate samples are in progress to measure the resistance of the film[4,5], too.
The measured quantity is the microwave reflection coefficient that is the complex ratio between the reflected wave and the incident one, measured at the instrument port. After proper calibration, resistance data could be derived.
In these measurements a crucial point is the film deposition thickness. From a first preliminary result, the film thickness measured with RBS technique is in agreement with RF resistivity measurements with respect thickness estimations made in Frascati. However, a global analysis and related experimental results will be described in a dedicate paper.

CONCLUSIONS

The technological R&D on Mo coatings on Cu is a promising technique for increasing the accelerating gradient of accelerators at higher frequencies.
In this contribution we describe tests made on different substrate and with different molybdenum thickness, deposited by Magnetron sputtering, in the framework of realize an X-band accelerator structure.
Using Rutherford Back Scattering technique chemical composition and thickness of samples made on copper substrate will be analyzed for a cross check .
Tests with Atomic Force Microscope are also in progress on samples made on glass substrate in order to check the film roughness quality and related deposition thickness.

Finally, the RF resistivity measurements will be completed as soon as possible.
Detailed RF tests on glass substrate samples are in progress in order to measure the resistance of the film and the related data will be published in a dedicated paper

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