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A procedure to obtain a very low Mo films with oxygen contamination

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Abstract

In order to determine the maximum sustainable gradients in normal conducting RF powered particle beam accelerators operating at 11.424 GHz with extremely low probability of RF breakdown, we are investigating the possibility of using the Mo coating method.
Molybdenum films were grown by rf magnetron sputtering technique on glass and sapphire substrates at room temperature. The sputtering parameters were optimized specifically addressing the growth of oxygen free Mo layers. An almost oxygen free content in the thin Mo films has been checked by Rutherford Back Scattering (RBS) technique. The chemical properties characterization of the coatings film have been carried out at Diamond Light Source (UK) with the XANES (X-ray Absorption Near Edge Structure) technique.
In this paper a procedure to obtain a very low Mo films oxygen contamination is described. Some hints about other applications of the molybdenum coatings are also presented.

Introduction

Technological advancements are strongly required to fulfill the demands of new accelerators devices with highest accelerating gradients reliability for the future collider([1], [2]).
An intense technological activity is therefore committed to realize X-band accelerating structures using different materials and methods.
In the frame of the collaboration with INFN-LNF/SLAC/KEK concerning breakdown studies about RF high gradient accelerating structures working at 11.424 GHz, an extensive R&D activities concerning the molybdenum coatings are in progress.
Materials with a higher fusion point seem to be good candidates to fabricate the accelerating structures working at 11.424 GHz with a high accelerating gradient. A sintered Mo bulk brazed RF structure has been realized at LNF and tested at SLAC [3].
The breakdown rate of the brazed Mo structure is higher than that of coppers structure for same RF parameters. From the investigation of the sintered molybdenum bulk brazed section some technological problems have been detected. As a result, the main issues for sintered molybdenum bulk are: 1. long time for machining the cavity; 2. 300 nm surface roughness using ‘tungsten carbide’ tools; 3. gas contamination; 4. an uneven loading stress in the brazed region [3]. For these reasons we are investigating the possibility of applying a Mo films, too.

Sputtering technique procedure at the *Thin film Laboratory* of CNR (Institute of Complex Systems, ISC)

The Mo thin films were grown by rf magnetron sputtering technique on different insulating substrates, such as Corning 7059 glass, Al_2O_3 (0001) and Al_2O_3(11-20), starting from a high purity (99.995%) Mo target. The sputtering system is a custom apparatus equipped with a turbo molecular pump backed by a dry scroll pump: the primary pump is used to rough out the chamber to a pressure low enough for use of a secondary turbo molecular pump. The use of a dry mechanical pump instead of an oil sealed mechanical pump avoids any oil back-streaming, thus improving the sputtered films quality by controlling the gas contamination. The apparatus is equipped with four target (4 inches diameters), four gas inlets and two sample holders; the films can be sputtered on cold samples or on hot samples (up to 500°C). The sample holders can be moved during the sputtering process at a variable rate, in order to improve the film uniformity. Before each deposition, the target was pre-sputtered for 30 min in order to remove any surface contamination layer, being the sputtered film deposited on the shutter surface. The sputtering process was performed in ultrapure Ar gas atmosphere and the substrates were positioned on a cold sample holder. In order to optimize the sputtering parameters, two different sputtering power were tested, 60 and 150 W.
A series of preliminary runs was made in order to select the sputtering parameters suitable for low oxygen Mo films. The Mo film optimized sputtering parameters are listed in the following table:

Background Pressure substrate type Substrate Temperature	$\sim 2 \cdot 10^{-7}$ torr Corning glass ; sapphire (0001) and (11-20); silicon (001) 20°C
Presputtering pressure Presputtering power Presputtering gas Presputtering pressure	4mTorr 100 w ultra pure Argon (23 sccm) 4 mTorr
Sputtering pressure Sputtering power Sputtering gas	4 mTorr 60 w (150w) ultra pure Argon (23 sccm)
Film thickness	3000 Å; 6000 Å; 9000 Å.

Table 1 shows the sputtering apparatus from Ionvac Process srl at the *Thin Film Laboratory* of the Institute of Complex Systems-CNR.

Figure 1: the RF magnetron sputtering apparatus from IONVAC PROCESS srl.

The glass substrates were carefully cleaned before being put inside the chamber in an ultrasonic bath containing an hot aqueous alkaline solution and then were carefully rinsed by ultra-pure deionized water; finally the samples were dried by an Ar flux. The sapphire substrates were pre-cleaned in class 10 room as provided by the manufacturer, and housed individually. Two different sputtering RF power values, 60 and 150 Watt, were applied, corresponding to two different deposition rate (and obviously deposition time): the Mo films grown on glass were sputtered at 60 Watt, while the Mo films grown on Al_2O_3 (11-20) and Al_2O_3 (0001) were sputtered at 150 Watt. At the present, preliminary investigations on the Mo films grown on glass reveal that the films have a quite low Oxygen content and the Mo films grown on sapphire substrates show an oxygen content lower than some percent. This different oxygen content may be due to a possible surface contamination from the glass substrates: although the high quality of the Corning 7059 glass substrates, an oxygen migration from the slide to the Mo film may be induced by the sputtering process, as a consequence of the locally increased temperature of the slide surface. Moreover, the lower Mo sputtering power used for the glass substrates and hence the longer layer deposition time increases the probability of film contamination from the residual gasses.

The RBS (Rutherford Back Scattering) measurements [4] performed on the Mo films sputtered on glass and sapphire were compared with the simulation of a pure Mo film of equal thickness: the acquired spectra show that there is no detectable O_2 contamination on the films surface (no signal in correspondence of O_2 surface channel indicated by the label) assessing the optimization of the sputtering parameters. However, details about the RBS characterization will be also described in a forthcoming paper.

The chemical properties estimated with the XANES technique have been completed [5] and they will be discussed in another dedicated paper, too.

Moreover, samples resistivity measurements at room temperature as function of the frequency up to 20 GHz are in progress.

Other applications of the Mo films

A further analysis of the structural properties of the Mo films deposited on different crystallographic cuts of sapphire are in progress, in order to evaluate the influence of the substrate on the structural properties of the films. Measurements of the Mo RF resistivity vs temperature are also in progress in order to investigate the reliability of the Mo films-based electroacoustic devices able to work under extreme conditions (temperature up to 900°C). The ability of molybdenum to withstand high temperature and harsh environments, together with its excellent mechanical and acoustical characteristics, make it the ideal candidate for the development of electronic devices able to operate under extreme conditions for application to the telecommunications and sensing fields. Electroacoustic devices based on Aluminium Nitride (AlN) piezoelectric films, such as thin film bulk acoustic wave resonators (TFBAR), employ the Molybdenum films to make the top and bottom electrode thanks to its low electrical resistivity, its acoustic impedance, and its structural characteristics that promote the growth of AlN films highly c-axis oriented [6]. The solidly mounted resonators (SMR) use a Bragg reflector consisting of alternating high and low acoustic impedance layers of quarter-wavelength thickness to mechanically insulate the resonator from the substrate. Typical materials with high acoustic impedance are metals such as Tungsten (W), Platinum (Pt), Molybdenum (Mo) or Gold (Au); materials with low acoustic impedance are, for example, Titanium (Ti) or Aluminum (Al). Titanium-Molybdenum is an example of a sequence of stacked layers having alternating low and high acoustic impedance. Further studies are in progress to test the temperature effects (900°C in air) on the sheet resistance and the structural properties of the Mo films[7] and [8].

Conclusions

Thin Mo films has been produced with almost no oxygen contamination, but surely satisfactory for the applications in the RF accelerating structures.

Resistivity measurements at room temperature as function of the frequency up to 20 GHz of the molybdenum coatings at room temperature will be completed in the coming days.

The chemical properties of the sputtering films have been characterized with RBS, XANES methods. The global samples characterization will be discussed in detail in a dedicated forthcoming paper as soon as possible, too.

References

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