

Comparison of different Zr deposition techniques on CNT thin films

F. FIORENTINO^{(1)(*)}, M. TODARO⁽²⁾, U. LO CICERO⁽²⁾, L. SCIORTINO⁽²⁾,
E. ALAIMO⁽¹⁾, F. D'ANCA⁽²⁾, G. DI CICCIA⁽²⁾, S. VARISCO⁽²⁾, A. GULIZZI⁽²⁾,
C. SAITTA⁽²⁾, P. T. TÖRMÄ⁽³⁾, B. HAQ⁽⁴⁾, H. LIPSANEN⁽⁵⁾ and M. BARBERA^{(1)(**)}

⁽¹⁾ *Università degli Studi di Palermo - Palermo, Italy*

⁽²⁾ *INAF, Osservatorio Astronomico di Palermo - Palermo, Italy*

⁽³⁾ *Ametek Finland Ltd - Espoo, Finland*

⁽⁴⁾ *Canatu Oy - Vantaa, Finland*

⁽⁵⁾ *Aalto University - Espoo, Finland*

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Summary. — In recent years, CNT pellicles have been investigated as suitable materials for filters for high-energy astrophysics missions, and more recently specifically for EUV missions, where Zr is a relevant material due to its transmittance in this band. In this work, we present the comparison of two different types of zirconium depositions on CNT pellicles, and manage to obtain a complete filter with performances compatible with the typical requirements for EUV missions.

1. – Introduction

In recent years, carbon nanotube (CNT) thin film pellicles have been investigated [1] within ESA-funded research contracts (Ametek Finland Oy. prime contractor) as an alternative substrate for optical blocking filters aimed at high-energy astrophysics applications. Such filters, essential for the proper operations of high-energy astrophysics observatories [2], generally consist of a thin metallic film that filters the out-of-band incoming radiation and a structural substrate that allows for the necessary mechanical resistance, usually made of polymeric material, *i.e.*, polyimide as the state-of-the-art.

Although generally a good choice for high-energy astrophysics missions, polymers might not be the best choice for missions that observe in the extreme ultraviolet (EUV), due to their high absorbance in those wavelengths and to the tendency to degrade over time under UV radiation [3]. CNT pellicles, on the contrary, offer high EUV transmittance, great mechanical resistance at low material density and stability over time, and

(*) E-mail: federico.fiorentino@unipa.it

(**) E-mail: marco.barbera@unipa.it

have been investigated for other high-energy applications such as EUV nanolithography [4]; however, metal coating of such pellicles is challenging due to their nano-porous structure and the thermal properties of the free-standing films. More recently, similar pellicles have also been investigated for applications in EUV astrophysics missions [5], where zirconium is generally the coating of choice in the 5–20 nm band.

In this work we compare the results of two different kinds of zirconium physical vapor depositions (PVD) on CNT thin films, one performed with Direct Current (DC) Magnetron Sputtering and one with electron beam (e-Beam) evaporation. The coated pellicles have been characterized with UV-VIS transmission spectroscopy and EUV-soft X-Ray transmission spectroscopy, to evaluate their optical performances. Scanning Electron Microscopy (SEM) imaging has also been performed, to evaluate the quality of the metal depositions and support the interpretation of the spectroscopic results.

2. – Materials and methods

The CNT films used to produce the filters have been manufactured by Canatu Finland Oy through floating catalyst chemical vapor deposition, and consisted of a random network of single wall (SW) CNT bundles. After their collection, the pellicles have been transferred on small aluminum rings, 1.5 mm in thickness and with an internal diameter around 16 mm, before the metal coatings.

Both investigated PVD deposition techniques, namely e-Beam evaporation and DC Magnetron Sputtering, have been performed in an Angstrom PVD deposition chamber at the Micronova research center, Espoo, Finland. For the DC Sputtering, the plasma was generated by excitation of argon gas at a pressure of ~ 4 mTorr, and the deposition rate was ~ 2 Å/s. The sputter target was a $> 99.5\%$ pure Zr disc from Angstrom Engineering. The base pressure of the deposition chamber before the deposition was $\sim 4 \cdot 10^{-7}$ Torr. A total of 200 nm of Zr has been deposited on the pellicles. For the e-Beam evaporation, the chamber pressure during the deposition was $4.3 \cdot 10^{-8}$ Torr, the deposition rate was ~ 2 Å/s. The target consisted of $> 99.5\%$ pure Zr pellets from Angstrom Engineering, the crucible was a FABMATE liner. A total of 230 nm of Zr has been deposited on the pellicles. In both cases, along with the CNT pellicles small silicon wafer chips have also been coated, as references for the deposition.

UV-VIS transmission spectroscopy in the 190–900 nm range was performed with a Perkin Elmer Lambda 1050+ dual beam spectrophotometer. For each sample, three measurements were repeated by rotating the sample around the axis parallel with the beam and then averaged to compensate for any possible inhomogeneity in the samples. EUV transmission spectroscopy in the 50–250 eV (5–25 nm) range was performed at the Metrologie beamline of SOLEIL synchrotron, Paris.

Scanning Electron Microscopy (SEM) images have been acquired for both types of samples. The acquisitions have been carried out using a Zeiss Supra 40 scanning electron microscope available at the premises of Micronova research center.

3. – Results and discussion

Figure 1 reports the transmission curves for both types of filters. On the left are the UV-VIS transmission curves: the e-Beam coated sample performs visibly better in attenuating the visible light than the sputtering coated one, with a transmission 10^{-4} lower in almost all the band. On the right are the EUV transmission curves: the e-Beam coated

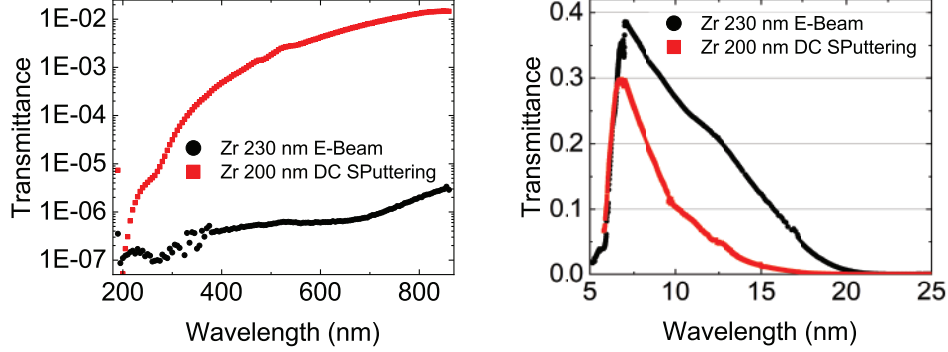


Fig. 1. – Out-of-band and in-band transmission curves of the e-Beam coated (black dot) and DC Sputtering coated (red square) samples. Left: transmission curves in the 190–860 nm out-of-band range. The y axis is in log scale. Right: transmission curves in the 10–25 nm in-band range.

sample is up to 157% more transmissive, despite the thicker Zr deposition. These differences in performances can be explained by looking at the SEM scans reported in fig. 2. The DC Sputtering coated sample is shown on the left, and the e-Beam coated sample, on the right. Next to them, in the green boxes, are shown the corresponding coatings on the silicon wafer chips used as references. The DC Sputtering deposition shows grains irregular in size and not very compact, and also the presence of some big agglomerates. A similar consideration can be made for the corresponding silicon deposition, where the grains are sized more evenly but there are still empty spaces between them. The e-Beam deposition, on the other hand, appears more compact, with well defined grains, more similar in size, and without the presence of agglomerates. The deposition on the silicon is very similar. This difference in appearance can justify the differences in performances reported in fig. 1: a less compact film can scatter light through the empty spaces, and the increased surface area allows for more oxidation of the coating that further reduces the optical performances, since zirconium oxide is quite transmissive in the visible range. Moreover, as the oxygen is absorbing in the EUV, an increase in oxygen content lowers the transmission in that range. The e-Beam coating, being more compact and uniform, blocks more light in the UV-VIS range and due to lower oxidation is more transmissive in the in-band region.

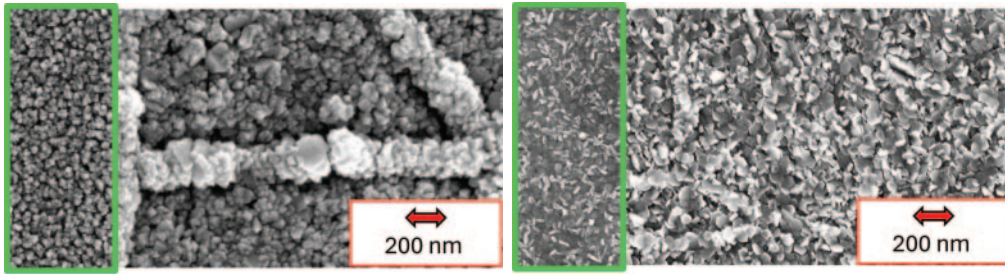


Fig. 2. – SEM scans of the DC Sputtering coated sample (left) and the e-Beam coated sample (right), at 200 $\times$ magnification. In the green boxes on the left the scans of the corresponding deposition on the silicon wafer chips are reported.

These findings are opposite to what is usually found in literature, where DC Sputtering is generally denser and less granular than e-beam; this could be justified assuming that the thermal radiation from the melted Zr during e-Beam evaporation heated the pellicles aiding the process, while the plasma pressure during the Sputtering process was too high and resulted in a poor film quality. It is in fact known that the temperature of the substrate and the pressure of the process can be directly correlated with the density and the microscopic structure of the metal film [6, 7].

Although the e-Beam coatings performed better both in EUV transmission and UV-VIS attenuation, the yield was low ($\sim 50\%$), likely due to the thermal stress.

4. – Conclusions

In this work, a comparison between two different metal deposition methods performed on two CNT pellicles of the same kind has been carried out. UV-VIS and X-Ray transmission spectroscopy have been used to evaluate the performances of the two kinds of coated pellicles as filters, and SEM imaging allowed to observe the microscopic topology of the depositions and to explain the differences in performances. With e-Beam evaporation an optical blocking filter compliant with typical EUV mission requirements has been achieved, however it has been observed that this deposition technique has a low production yield, probably due to thermal stress, and is not reliable for final production. The DC Sputtering did not produce comparable results, probably due to non optimized deposition parameters during the process. These results are still preliminary, and further investigation is still ongoing.

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