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# Preparation and characterisation of High Oriented Pyrolytic Graphite backed targets for the NUMEN project

Antonio Massara<sup>1\*</sup>, Maria Fisichella<sup>1</sup>, Martina Ursino<sup>1</sup>, Vittoria Capirossi<sup>2,3</sup>, Francesco Cappuzzello<sup>1,4</sup>, Manuela Cavallaro<sup>1</sup>, Franck Delaunay<sup>1,4,5</sup>, Federico Pinna<sup>2,3</sup>, Alberto Rovelli<sup>1</sup> for the NUMEN collaboration

<sup>1</sup>Istituto Nazionale di Fisica Nucleare, Laboratori Nazionali del Sud, Catania, Italy

<sup>2</sup>Department of Applied Science and Technology, Politecnico di Torino, Torino, Italy

<sup>3</sup>Istituto Nazionale di Fisica Nucleare, Sezione di Torino, Torino, Italy

<sup>4</sup>Dipartimento di Fisica e Astronomia “Ettore Majorana”, Università di Catania, Catania, Italy

<sup>5</sup>LPC Caen, Normandie Université, ENSICAEN, UNICAEN, CNRS/IN2P3, Caen, France

**Abstract.** The equipment for target production and characterisation at the Laboratori Nazionali del Sud – INFN (Catania, Italy) is described. In particular, the CACTUS system used for target thickness and non-uniformity measurements is detailed. First trials in view of the production of Cadmium targets, under development for the NUclear Matrix Elements for Neutrinoless double  $\beta$  decay (NUMEN) project, are reported and discussed.

## 1 Introduction

The target laboratory at Laboratori Nazionali del Sud (LNS) of INFN at Catania has over thirty years of experience in target preparation for nuclear physics experiments and interdisciplinary physics. Users are not only local scientists but also researchers from other universities and laboratories worldwide. A list of the targets produced at LNS and their respective production techniques are given in [1].

In the present paper, the production and characterisation of a Cd target prototype, recently realised for the NUMEN project experiments [2-4], will be discussed in detail together with a brief description of the devices available at LNS for target preparation and characterisation.

## 2 LNS equipment for target preparation

Physical Vacuum Deposition (PVD) is a common method in the preparation of targets for nuclear experiments. PVD systems deposit thin films by a process in which a solid target material is vaporized, transported in vacuum and condensed on a substrate. PVD processes include thermal evaporation, electron beam (e-beam) evaporation, and sputtering. At the LNS laboratory, two evaporation systems are present: a classical L300 Thermal Bell Jar (TBJ) evaporator and a L560 Leybold-Heraeus, equipped, respectively, with a resistive source and an e-beam source. Thermal evaporation is typically used for materials which have a melting point lower than 1500 °C, whereas the e-beam gun is well suited for materials with high melting point

temperatures and/or which could alloy or chemically react with crucible materials causing a contamination of the deposited film.

### 2.1 Thermal Bell Jar Evaporator

The L300 TBJ evaporator (Figure 1), with a power of 1.1 kW, has a stainless steel base and a glass bell jar with a diameter of 300 mm and a height of 350 mm.



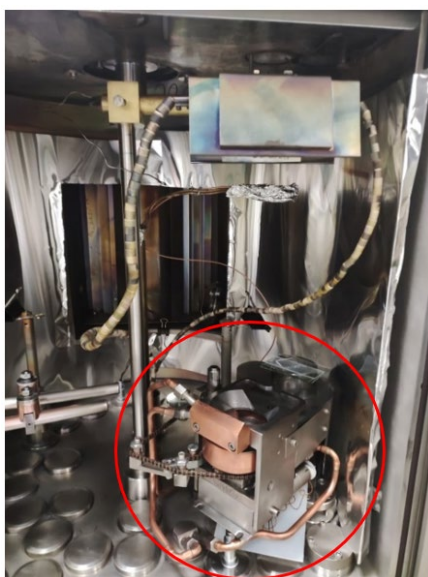
**Fig. 1.** View of the L300 Thermal Bell Jar evaporator. It is possible to distinguish the glass cylinder on which the target frame holder is placed and the quartz balance at the central upper position.

\* Corresponding author: [massara@lns.infn.it](mailto:massara@lns.infn.it)

It is equipped with two pockets for material deposition. Source materials are melted by resistive heating in metal evaporation boats (Ta, W, Mo) or ceramic crucibles. Evaporation rates are manually controlled by varying the output power of a power supply. Deposition rates are monitored with a quartz crystal microbalance. Using glass cylinders of different heights, as shown in figure 1, it is possible to vary the distance between the source and the substrate (on which the evaporated material will be deposited) in a range from 80 to 220 mm.

## 2.2 L560 Leybold-Heraeus Low Vacuum Evaporator

The L560 evaporator at LNS (see figure 2) is equipped with a resistive heating source as well as with a 6 kW 270° electron-beam gun, with a 4 pocket evaporation source. It is possible to choose which one must be used according to the properties of the material to be evaporated. The evaporator is equipped with a quartz microbalance crystal and, as in the case of the L300 evaporator, it is possible to set the distance between the source and the substrate using glass cylinders of different heights. Moreover, the evaporator has a halogen heating element to set the temperature of the substrate, if needed. Indeed, as is well known [5], that heating the substrate during the deposition can support the diffusion of the material on the substrate surface, thus avoiding the formation of islands or typical structures. The quartz and the heating system are mounted on movable arms so that they can be coupled with both systems. In the bottom part of figure 2, one can see a typical set up for an e-beam evaporation (inside the red circle): the 8-cm<sup>3</sup> hearth with its cooling system (to maintains the source temperature lower than 100 °C), the microbalance and the heating elements. On the left, the electrodes for thermal evaporation are also visible.



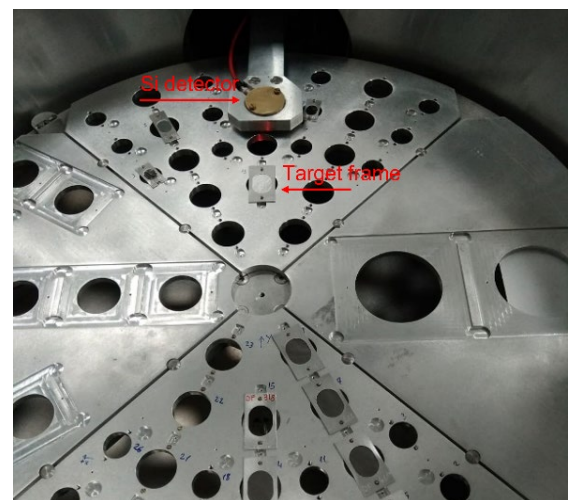
**Fig. 2.** Internal view of the L560 evaporator. The setup is prepared for e-beam evaporation. Inside the red circle one can see the e-beam hearth, the quartz balance and the heating elements. On the bottom left part the electrodes for thermal evaporation are also visible.

## 2.3 Rolling mill

A cold rolling mill is also available for preparing metal targets with thicknesses greater than 1 μm. It consists of two hardened and polished stainless steel rollers of 125 mm face width by 85 mm diameter, driven by a motor. The metal foil is progressively thinned by passing between these two rollers until it reaches the desired thickness.

## 3 New system for target characterisation at LNS

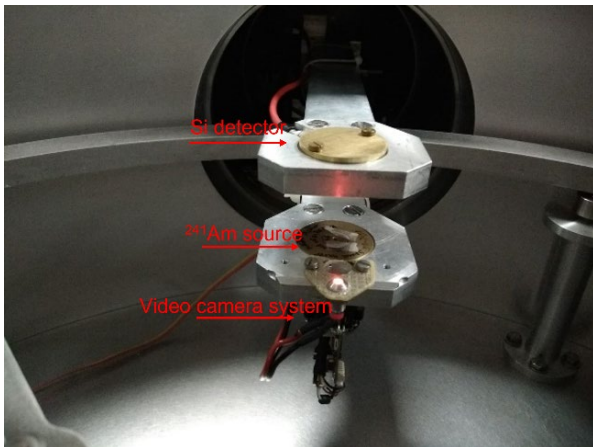
The target thickness is checked during the evaporation using the quartz microbalance of the evaporator. To have a more accurate value of the single target thickness, Alpha Particle Transmission (APT) measurements are usually performed. Recently, the LNS target laboratory has been equipped with an apparatus named Chamber for Alpha-particle Characterisation of target Thickness and Uniformity by Scanning (CACTUS). The system, which uses the APT technique, was entirely developed at LNS. It is a cylindrical chamber of about 1 m in diameter containing four sectors where it is possible to place the targets to be characterised (see Fig.3). Each sector has been designed for different target frame geometries. The measuring system (see Fig. 4) consists of a moving probe with a special head containing a <sup>241</sup>Am α-source (55.5 kBq unsealed source) and an ion implanted silicon detector, 100 μm thick and with a diameter of 25 mm<sup>2</sup>. In front of the source and the detector two collimators, respectively, of 6 mm and 3 mm of diameter are mounted. The distance between the two collimators is 33 mm, while the distance between a target and the detector collimator is about 6 mm. According to this geometry, the dimension of the alpha spot in the target will be about 3 mm. The head has been designed to allow the sector movement in the space between the source and detector.



**Fig. 3** Internal view of the CACTUS chamber. The four plates to place the target frames and the top part of the radial probe are visible.

The radial probe movement and the sector rotation allow positioning the head over the target to be charac-

terised. A video camera, also located on the head, guarantees precise automatic target positioning with a precision within 1 mm. Control loop and automated procedures ensure the measurement of many targets without opening the vacuum chamber. The CACTUS system allows to investigate the target thickness on the whole target surface, making possible a study of the uniformity between different areas of the target (“global” uniformity). An ORTEC/AMETEK easy-MCA multichannel analyser with the MAESTRO software is used for data acquisition.



**Fig.4** Close-up view of the measuring probe. The source and the detector are placed in the bottom and top part, respectively, of the moving arm. In the foreground the LED-video camera system is also visible.

The measured target thickness is the average value over a spot surface, defined by collimators placed in front of the source and the detector. In measurements reported in the following, the geometry of the system was such that the alpha spot on the target was about 4 mm.

## 4 Targets for NUMEN experiments

In this section, preliminary results concerning the Cadmium target production for the NUMEN (Nuclear Matrix Element for Neutrinoless double beta decay) experiments [2-3] are reported. Targets for these experiments will be made with isotopes suitable as candidates for neutrinoless double beta decay studies (for example <sup>48</sup>Ca, <sup>76</sup>Ge, <sup>76</sup>Se, <sup>82</sup>Se, <sup>96</sup>Zr, <sup>100</sup>Mo, <sup>106</sup>Cd, <sup>116</sup>Cd, <sup>110</sup>Sn, <sup>124</sup>Sn, <sup>128</sup>Te, <sup>130</sup>Te, ...). They have to be deposited on High Oriented Pyrolytic Graphite (HOPG) [6], which thanks to its high thermal conductivity (1950 Wm<sup>-1</sup>K<sup>-1</sup>) could help dissipating the high power released by very intense <sup>18</sup>O and <sup>20</sup>Ne beams up to 10<sup>13</sup>pps used for these experiments. According to thermal calculations, the minimum value of HOPG for efficient heat dissipation is about 450 μg/cm<sup>2</sup>. To maintain a reasonable resolution on the energy of reaction products, the target thickness will range from 250 to 500 μg/cm<sup>2</sup>, depending on the material to be evaporated. Moreover, both the backing and the target have to be uniform, to avoid degrading the energy resolution on the reaction products by addi-

tional straggling effects. For this reason, the APT measurements are also performed on the backing before the evaporation process to determine its thickness and uniformity. Furthermore, the characterisation of the HOPG substrates provided by different companies are being characterised and the results will be presented elsewhere. In the present report, the focus will be on the cadmium target manufacture and characterisation.

### 4.1 Cadmium target production

The required thickness for the cadmium layer deposited films was about 500 μg/cm<sup>2</sup>. Natural cadmium material was used, not to waste isotopically enriched material. The evaporation was performed at LNS using the L300 evaporator with the following working conditions and geometry:

- 1.5 - 2g Cd deposited on Mo boats;
- distance source - backing equal to 250 mm;
- Evaporation rate: 0.2 Å/s.

At the end of the evaporation procedure, about 1g of Cd was used and 8 targets with an area of 12 mm x 15 mm were produced.

In the same evaporation both 450-μg/cm<sup>2</sup> thick HOPG, and thin (70 μg/cm<sup>2</sup>) amorphous carbon, manufactured by OPTIGRAPH and Yissum respectively, were used as backings to study the possible effects due to different substrate. Since it is well known that the cadmium does not stick well on carbon substrates, a 0.1 μg/cm<sup>2</sup> bismuth buffer was deposited on the backing to improve the cadmium adhesion. The first trial did not give good results: The cadmium thickness measured by APT resulted thinner than the expected one, measured by the quartz microbalance. The average thicknesses of the targets produced are reported in the first two rows of Table 1. A different behaviour has been observed if the backing was an amorphous carbon foil. The amount of cadmium material deposited on pyrolytic graphite is about half of the one deposited on amorphous carbon. Literature [3] suggests that the best result for evaporated Cd targets is reached if Cd is deposited on a cooled backing.

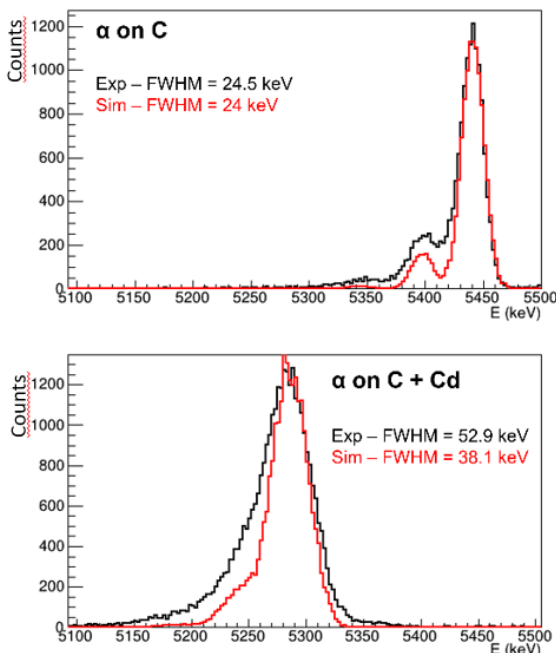
Evaporation procedure	Backing-Type	Cd thickness (μg/cm <sup>2</sup> )
Single-step	HOPG (450 μg/cm <sup>2</sup> )	170
Single-step	C (70 μg/cm <sup>2</sup> )	300
2-step	HOPG (450 μg/cm <sup>2</sup> )	140
2-step	C (70 μg/cm <sup>2</sup> )	474
2-step	HOPG (2.25 mg/cm <sup>2</sup> )	370

**Table 1.** Evaporation procedure (see text for details), type and nominal thickness of the backing (from the manufacturer), and average thickness of the cadmium target obtained.

Since the evaporator systems at LNS do not offer the possibility to cool the substrate, a new trial was performed by evaporating the Cadmium in two steps. In the first one, the cadmium material is evaporated till the half

of required amount is deposited. Then follows an intermediate phase to reach room temperature before the second cadmium evaporation step to reach the final thickness. The deposition thickness is monitored using the evaporator quartz crystal microbalance. The starting conditions were identical to the ones described above. In this second target series, in addition to HOPG and amorphous C backings similar to those used in the first test, 2.25-mg/cm<sup>2</sup> thick HOPG, substrates, produced by PANASONIC, were used.

In the last three rows of Table 1, results obtained in this 2-step evaporation are reported. The thickness of the cadmium evaporated on the 450 µg/cm<sup>2</sup> HOPG is similar to the ones of the single-step procedure. On the contrary, the two-step procedure improved the deposition on amorphous carbon: A result very close to the expected one has been obtained. Concerning the third type of substrate, the 2.25-mg/cm<sup>2</sup> thick HOPG, a thicker deposition has been obtained compared to the case of a thinner HOPG substrate, but the thickness is still lower than the expected one. This seems to indicate that the surface of the foil plays an important role in the deposition process. Further studies are necessary to understand the different behaviour of cadmium on C and HOPG. In particular, scanning electron microscopy analysis to study the backing surface has already been planned.



**Fig. 5.** (Colour online) Experimental (black histogram) and simulated (red histogram) energy spectra of  $\alpha$ -particles passing through an amorphous carbon backing alone (top) and through a C+Cd target (bottom). The simulated spectra are obtained with the SRIM [8] code.

For the targets deposited on the amorphous carbon, with the expected thicknesses, a detailed analysis has been performed to study the uniformity of the cadmium

deposition. Thanks to the CACTUS system, the thickness has been measured in several regions across each target, to investigate its dependence on the position: Differences inside 2% have been observed between the measured points, confirming a good global uniformity of the deposition. The local uniformity (i.e. the uniformity inside the area covered by the alpha-particle spot, 3mm) of the cadmium layer was extracted following the procedure described in [7], by comparing the Full Width at Half Maximum (FWHM) of the experimental spectrum with the FWHM of a spectrum simulated assuming a uniform target with the measured average thickness. In figure 3, the residual energy spectra for alpha-particles crossing the carbon backing alone (top) and the C+Cd target (bottom) are reported. The black histogram is the experimental spectrum, and the red histogram is the result of a SRIM simulation [8]. The local non-uniformity of the backing has been estimated to be less than 10%, while for the cadmium layer a local non-uniformity of about 25% has been determined.

## 5 Conclusions

The devices available at the LNS target laboratory allow to produce several types of targets. The new CACTUS system allows a complete characterisation of the target in term of thickness, local and global non-uniformities. Obviously, the development of new targets requires multiple trials and in addition to APT other types of analysis can help to better understand the deposition process. Examples of very useful analyses are given by scanning electron microscopy, to study the morphology of the sample surface, and Rutherford backscattering spectroscopy, to investigate the purity of the target.

Regarding the development of cadmium targets discussed in this report, further studies are necessary to obtain targets with the required thickness. A particular analysis must be devoted to the surface of the HOPG backing.

## References

1. <https://www.lns.infn.it/en/targets.html>.
2. F. Cappuzzello et al, Eur. Phys. J. A (2018) **54**: 72
3. F. Cappuzzello et al. International Journal of Modern Physics A 36, (2021) 2130018
4. P. Finocchiaro et al. Universe 6 (2020) 129
5. Stolarz, J. Radioanal Nucl. Chem. (2014) 299:913-931
6. F. Pinna, Acta Polonica B, 51, 3 2020
7. M. Fisichella, A. Shotton et al., Phys. Rev. C **92** 064611 (2015)
8. J. Ziegler, "SRIM code" [<http://www.srim.org>]