# PROCEEDINGS OF SCIENCE



# Status of the HOLMES experiment: commissioning of the ion implanter .

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© Copyright owned by the author(s) under the terms of the Creative Commons Attribution-NonCommercial-NoDerivatives 4.0 International License (CC BY-NC-ND 4.0). The neutrino mass determination is an open issue in particle physics. The study of the endpoint of beta decay is the best experimental way to provide a model-independent measurement. The HOLMES experiment aims to measure directly the neutrino mass with a calorimetric approach studying the <sup>163</sup>Ho electron-capture decay. The very low Q-value (2.8 keV), the half-life (4570 y) and the proximity of the endpoint to M1 resonance make the <sup>163</sup>Ho decay a very good choice. However, there are two critical steps to be considered for the realization of the experiment. The first step is embedding of the source isotope inside the cryogenic microcalorimeters so that the energy released in the decay process is entirely contained within the detectors, except for the fraction taken away by the neutrino. The second one is the rejection of <sup>166</sup>Ho radioactive isotope that could produce false signal in the region of interest. Taking into account these two requirements, a dedicated implanter with a sputter ion source, an acceleration section (up to 50 keV) and a magnetic dipole (for ion selection and beam focusing) has been designed and developed. The implanter calibration and performance have been evaluated using <sup>63</sup>Cu/<sup>65</sup>Cu and <sup>197</sup>Au beams. Currently, different holmium compounds are being tested to find the candidate with the best efficiency in the sputter process. This work will show the status of the machine development and commissioning.

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#### 1. Holmes experiment

The aim of the HOLMES experiment is the study of end point of electron capture (EC) of <sup>163</sup>Ho to perform a direct measurement of neutrino mass with a calorimetric approach (source embedded in the detector)[1]. <sup>163</sup>Ho decay is a optimal choice because of its very low Q-value (2.8 keV) and its half-life (4570 y). These properties consent to have a reasonable activity and sufficient statistics, reduce the pile-up probability and not alter the heat capacity of detector. The measure will be performed by 500 Transition Edge Sensor (TES) based microcalorimeters (8x64 array), read out by a microwave multiplexed rf-SQUID system[2], to reach an accuracy of the order of eV. In the next sections we will present the activities concerning the <sup>163</sup>Ho production, purification and implantation, and the preliminary results from implanter commissioning.

#### 2. Holmium production and purification

The <sup>163</sup>Ho is not a natural isotope but is produced with neutron irradiation of <sup>162</sup>Er. In the process a lot of impurities and contaminants are produced and needed to be removed. The first step, a chemical separation using an ion exchange chromatography procedure, is performed at Paul Scherrer Institute (PSI, Villigen, CH) and reduces the impurities with an efficiency  $\epsilon > 98.6\%$ . [3] The chemical separation cannot remove other holmium isotope contaminations. In particular it is crucial to remove the <sup>166m</sup>Ho, a beta emitter produced during the neutron irradiation with half-life of about 1200 y and could induce background below 5 keV. To isotopic selection and <sup>163</sup>Ho embedding into the detector, a dedicated ion implanter has been developed and commissioned in Genoa's INFN laboratories.

## 3. Ion implanter

The ion implanter has been produced by DANFYSIK[4] and consists of different parts (fig.1):

- 1. The main component of the implanter is a sputter ion source. It consists of a small vacuum chamber where a flux of ionized argon hits a target, kept at a potential difference up until 600 V, and sputter the target material. The ionization is obtained via electrons emitted by a filament (thermionic effect). These electrons ionize also the sputtered material that diffused from an hole and enter in region where an electrical potential accelerate them until a maximum energy of 50 keV. All parts of the ion source are water cooled but the sputter target can reach a temperature higher than 1000 °C.
- 2. A magnetic dipole and a steering magnet. The first one has a bending radius of 46 cm and is capable to reach field intensity up to 1.1 T. It will perform the isotopic separation. The second one is a small magnet located near the exit of the ion source. It has a bending radius of 573 m, reaches field intensity up to 0.02 T and will correct possible vertical misalignments of the ion beam.
- 3. A focusing electrostatic triplet and a X-Y steering magnet needed to refocus and scanning the beam. They are not yet installed.



Figure 1: The scheme of the implanter.

- 4. A slit system and a faraday cup placed near the beam focus to cut the beam tails and to measure its intensity.
- 5. A target vacuum chamber where holmium will be implanted into the detectors. In the chamber a smaller argon sputter source will allow a simultaneous gold evaporation to control the <sup>163</sup>Ho concentration and avoid saturation. It will be used also to deposit a final gold layer of about 1  $\mu m$  to encapsule Ho for a full containment of the decay products.

The whole implanter operates at a vacuum level better than  $10^{-6}$  mbar and the beam spot is expected around 4.5 mm FWHM in the focal point. The machine is designed to reach a separation between  ${}^{163}Ho/{}^{166}Ho$  better than 5  $\sigma$ .

## 4. Sputter targets production

The implanter system (see next section) needs a sputter target with an annulus geometry. The chemical purified Ho will arrive from PSI in oxide or acid form but, to avoid chemical shifts of the end point, metallic holmium is needed. Two different techniques are used to produce the target:

- a sintered target, made by a mixture of fine grained metal powders (≤ 40 μm) of Ti(36%), Ni(41%), Sn(18%)and Ho(5%) pressed at 350 bar/cm2 and heated at 950 degree in a low vacuum environment for 2 days. Nichel and tin to create an intermetallic compound with holmium (HoNiSn) that is scattered in a matrix of a second intermetallic compound of nichel, titanium and tin (Ti<sub>2</sub>Ni<sub>2</sub>Sn). To obtain metallic holmium powder, a reduction-distillation (RD) procedure is performed in a dedicated evaporation chamber with an efficiency more than 70% [5]. Alternative metal powders are under investigation to obtain a RD procedure directly inside the ion source (yttrium, zirconium) using the high temperature reached by the sputter target and maximize the efficiency.
- a copper bulk target covered with a gold film (100 nm) where a thin layer of Ho compound is deposited. This procedure is performed by PSI using a molecular plating procedure that



Figure 2: Copper peaks (right) and molybdenum (left) with (red) and without (black) sputtering voltage turned on.



Figure 3: Measure of beam current as function of steering magnet (left) and slit opening (right).

consists in an electrodeposition of holmium compounds (oxides, hydroxides, carbonates and others) from an organic solvent by applying high voltages (100 - 600 V). This technique can achieve yields close to 100% for many lanthanides as well as holmium. Different targets with different holmium compounds are under investigation to maximize the capability of sputter to break the molecule and extract metallic holmium.

## 5. First tests

Preliminary tests were done using dummy copper targets but without holmium. The extraction potential was fixed at 25 kV and several scans of materials are done to calibrate the system. The copper and molybdenum (present in support structure of the sputter target) isotope peaks are shown in fig.2. We estimated an expected separation for holmium 163 and 166 around 19 mm at 25 keV with a peak sigma better than half a.m.u.. Several tests are done with steering magnet and we found a vertical misalignment of around 2 mm near the focal point. Several tests are done with slit system and we estimated a gaussian beam profile with  $\sigma_x \approx \sigma_y \approx 1.35 \text{ mm}$  (fig3).

## 6. Preliminary tests with natural holmium

Preliminary test with natural holmium ( $^{165}$ Ho) has been performed using both a molecular plating target with holmium hydroxide (Ho(OH)<sub>3</sub>) and a sintered target of Ti,Ni,Sn,Ho.

• Molecular plating target test. The test showed a clear peak at 165 a.m.u. but the beam current is quite low (100 nA) and decreasing to zero in some minutes (fig4). This behavior is under investigation and tests with different compounds are ongoing.



**Figure 4:** <sup>165</sup>Ho peaks with a molecular plating sputter target. Different colors corrispond to different sputter voltage but also different times.

• Sintered target test. The test showed a clear peak at 165 a.m.u with a lower beam current (40 nA) but more stable in time. An unexpected peak at 164 a.m.u was present and we melted the target after 2 hours of data taking. Other materials to improve thermic proprieties are under investigation and a deep analysis to understand 164 peak is ongoing.

#### 7. Conclusions

The commisioning of the Holmes ion implanter is completed and the system showed good performances. Various calibration procedures have been established. Two types of sputter target with natural holmiumn has been produced and tested. The measurments showed clear peaks at 165 a.m.u. but ceam intensity and stability are not yet satisfactionary. New tests with different sputter targets are on going and the first implantion of adioactive <sup>163</sup>Ho are currently expected for the end of 2022.

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