ECT* workshop: Determination of the absolute electron (anti)-neutrino mass

^{163}Ho distillation and implantation for HOLMES experiment

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HOLMES is an experiment to directly measure the neutrino mass relying on a calorimetric technique. This approach avoids several systematic uncertainties usually present in spectrometers, where the usage of a source external to detector and the decays to excited states can affect the measurement. ^{163}Ho is chosen as source for the very low Q-value (2.8 keV) of its electron capture decay and its half life (4570 y). These features guarantee to have enough statistics close to the end point with a limited amount of radioactive material – thus leaving the detector thermodynamic properties unaltered. ^{163}Ho will be produced by means of neutron irradiation of a $^{162}Er_2O_3$ sample at the Institute Laue-Langevin (Grenoble, France). This process produces not only ^{163}Ho but also other Ho isotopes and contaminants which can spoil the measurement. In particular the metastable ^{166m}Ho has a beta decay with half life of about 1200 y which can induce a very dangerous background below 5 keV. Therefore the sample will be purified in 2 steps: first of all, a chemical separation will be performed at Paul Scherrer Institute (Villigen, Switzerland). Then, a magnetic spectrometer based mass separation will be done at the INFN laboratory of Genova. The removal of ^{166m}Ho is critical for Holmes so a dedicated system is being set up in Genova INFN section. The system is designed to achieve an optimal mass separation for ^{163}Ho and consists of two main components: an evaporation chamber and an ion implanter. In the evaporation chamber Holmium will be reduced in metallic form, using the reaction $2Y+Ho_2O_3 \rightarrow Y_2O_3+2Ho$ and then used to produce a metallic target for the ion implanter source. The ion implanter consists of five main components: a Penning sputter ion source, an acceleration section, a magnetic/electrostatic mass analyzer, a magnetic scanning stage and a focusing electrostatic triplet. In this contribution we describe the R&D work done on the Ho reduction system and the implanter machine.

Holmium production and reduction

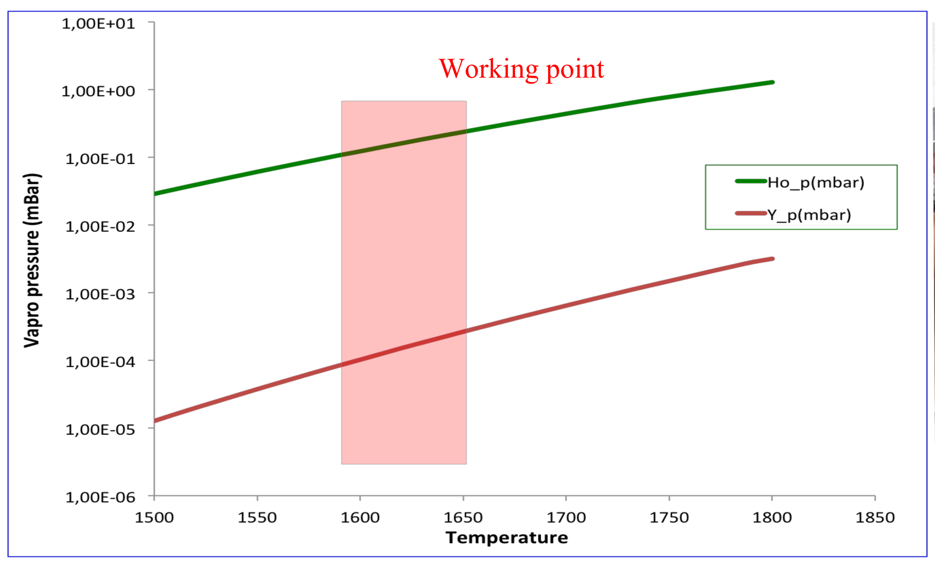
^{163}Ho will be produced by neutron irradiation of Er_2O_3 enriched in ^{162}Er at the ILL (Grenoble, France).
The impurities and the contaminants will be chemically removed at PSI (Villigen, Switzerland).
The purified sample will arrive in Genova in oxide form (Ho_2O_3). The others holmium isotopes, and in particular $^{166(m)}$Ho will be removed by means of an ion implanter.
In order to avoid chemical shifts of the end-point, only Ho in metallic chemical form must be embedded in the detectors. Due to the ΔG higher than the Ho's one, metallic Yttrium can be used for Ho reduction by means of the reaction:

$$\mathrm{Ho_2O_3} + 2\mathrm{Y(m)} \rightarrow 2\mathrm{Ho(m)} + \mathrm{Y_2O_3}$$

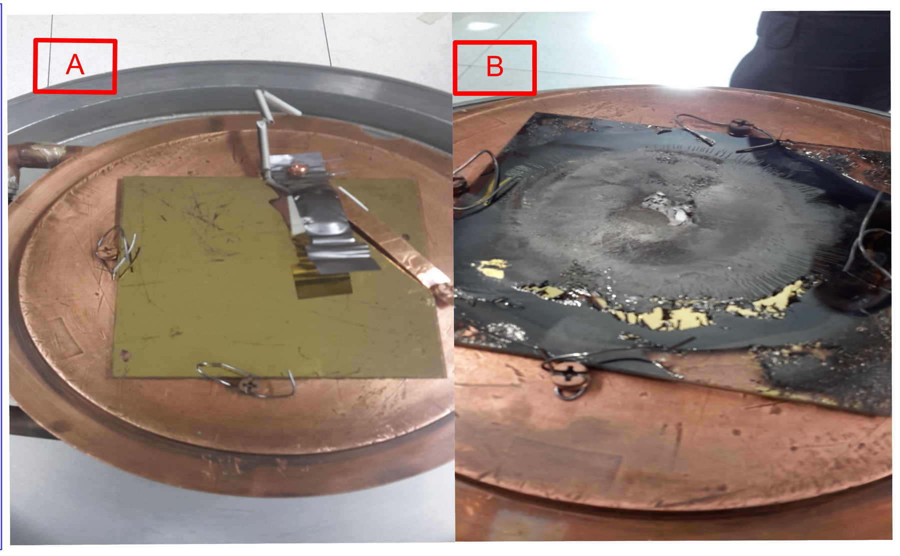
Metal	Melting Point (°C)	Oxyde form	ΔG (kJ/mol)
Ho	1460	Ho_2O_3	-1791.1
Y	1526	Y_2O_3	-1816.2

Distillation process

A quartz substrate is used for Ho evaporation because of its high resistivity to thermal deformation.
A gold thin film has been deposited on quartz by thermal evaporation to easy remove Ho film from substrate. The high reflectivity of gold help to avoid excessive heating of substrate too. The crucible is heat up to 1600 °C to melt Yttrium and speed up the reaction. The Y vapor pressure is three order of magnitude lower than Holmium one this working point. This condition minimizes Y contamination. The distillation efficiency is ~ 80%, determined as the ratio between the mass of the condensed Ho on the substrate and the missing mass in the crucible.



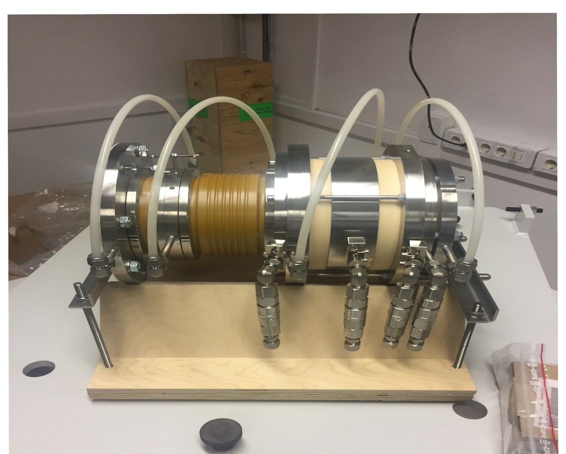
Ho and Y vapour pressure

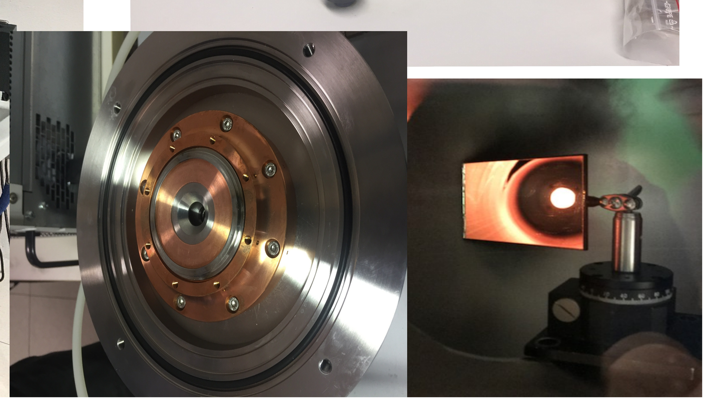


A) Quartz substrate coated with gold before Ho evaporation
B) Quartz substrate after Holmium evaporation

Ion implanter

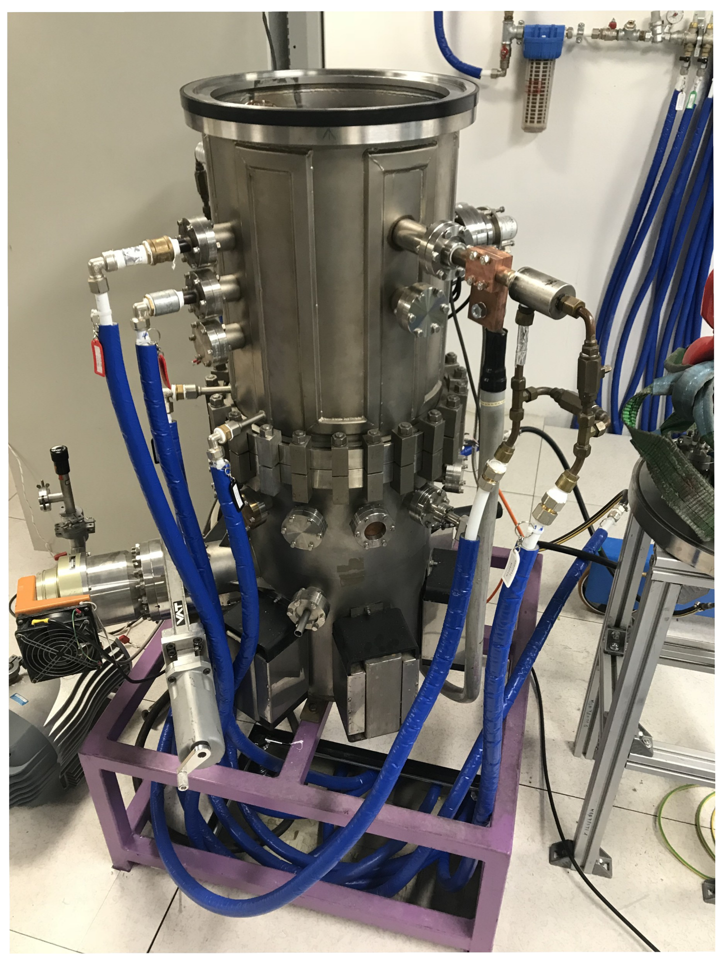
A dedicated ion implanter is now under comissioning phase in Genova's laboratory and will be used to remove contamination of holmium isotopes different from ^{163}Ho as well as other impurities.
The ion implanter consists of five main components:
- an argon penning sputter ion source;
- an acceleration section to reach the beam energy of 50 KeV;
- a magnetic/electrostatic mass analyzer with magnetic field up to 1.1 Tesla;
- a magnetic scanning stage;
- a focusing electrostatic triplet (not installed yet).

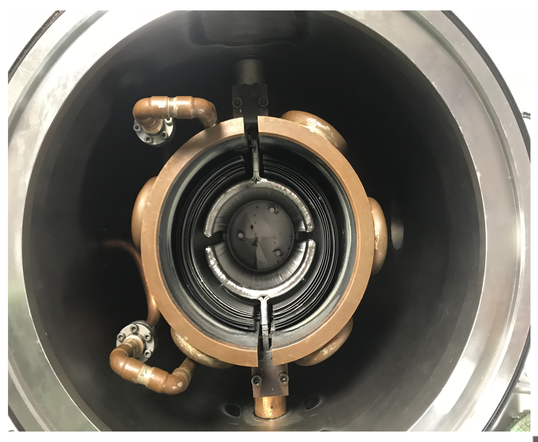


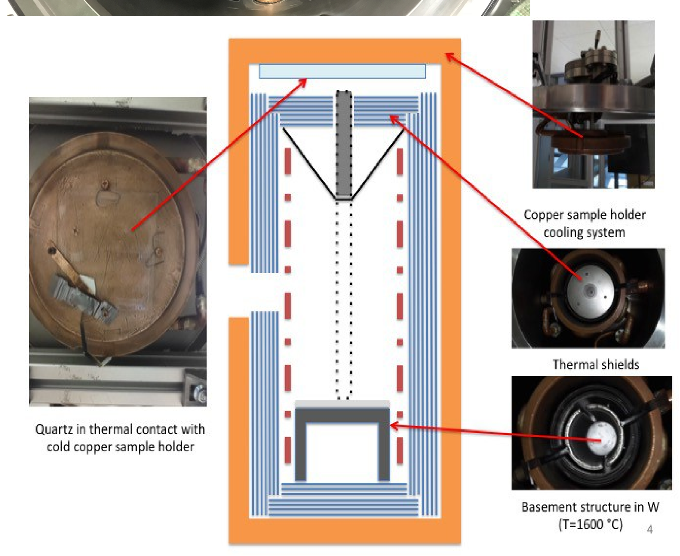


Knudsen cell for Holmium reduction

A dedicated evaporation chamber (Knudsen cell) has been assembled for Ho reduction. The oxide sample is put in a small alumina crucible placed inside a cylindrical oven that could reach temperatures as high as 2000°C. The oven is thermally isolated by nine W shields from a water cooled copper support. A hole is bored on each shield in a such a way to allow the evaporated Ho flowing from the crucible to a substrate fixed on the top of the cooled copper support. The whole system is set in a vacuum chamber which could reach pressures as low as 10^{-8} mBar.



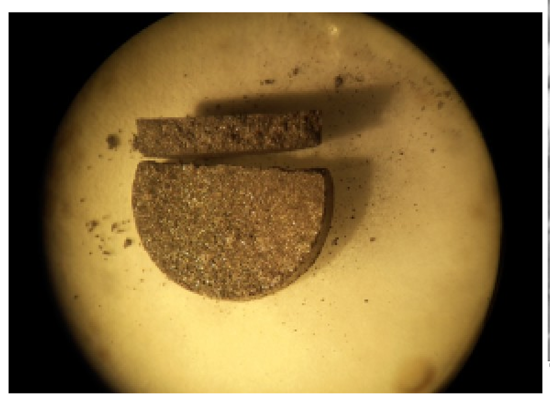




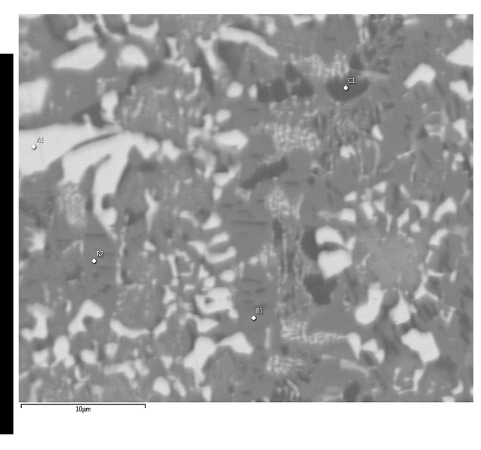
Sputter target production

The sputter ion source needs a metallic cathode containing the ^{163}Ho. We decided to realize a sintered sputter target, including Ho(5%) in a metallic mixture of Ti(36%), Ni(41%) and Sn(18%) fine grained powder (< 40 µm).
The compound is arc melted in argon atmosphere than milled and pressed at 350 bar/cm². The obtained target is heated at 850 °C pressure 10^{-2} mbar for 4 days to improve the mechanical proprieties of the sintered.
The crystallographic measurements and SEM-EDS analysis show two different phases: a Ti_2Ni_2Sn matrix with homogeneously distributed islands of HoNiSn.

Dedicated press for the target



Example of sintered



SEM images of metallic matrix. The light gray zones are HoNiSn "islands".

Conclusions

- The procedure to distillate holmium is tested. Few refinements are needed.
- A metallic sintered sputter target has been obtained using Ti,Ni,Sn and Ho. In the final target the analysis show a Ti_2Ni_2Sn metallic matrix with HoNiSn islands uniformly distributed.
- The implanter machine is now in commissioning phase.
- The implantations test are foreseen for the incoming months.

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