^{163}Ho distillation and implantation for HOLMES experiment

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Abstract

HOLMES is an experiment to directly measure the neutrino mass with a calorimetric approach. The calorimetric technique eliminates several systematic uncertainties usually present in spectrometers where the external source and the decays to excited states affect the measurement. ^{163}Ho is chosen as source for its very low Q-value (2.8 keV), the proximity of the end-point to resonance M1 and its half life (4570y). These features are optimal to reach simultaneously a good activity to have sufficient statistics in the end-point and a small quantity of ^{163}Ho embedded in the detector to not alter significantly its heat capacity. ^{163}Ho will be produced with neutron irradiation of enriched ^{162}Er2O3 at the Institute Laue-Langevin (Grenoble, France), and chemical separated at Paul Scherrer Institute (Villigen, Switzerland). It will arrive at INFN laboratory of Genova in oxide form (Ho2O3) with traces of others Ho isotopes and contaminants not removable using chemical methods. In particular the metastable ^{166m}Ho has a beta decay with half life of about 1200y which can induce background below 5 keV. The removal of these contaminants is critical for Holmes so a dedicated system is being set up by our group from the 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+Ho2O3 → Y2O3+2Ho and 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 procedures, under continuous refinement, for the Holmium evaporation process, the metallic target production and the status of the ion implanter.

Holmium production and distillation

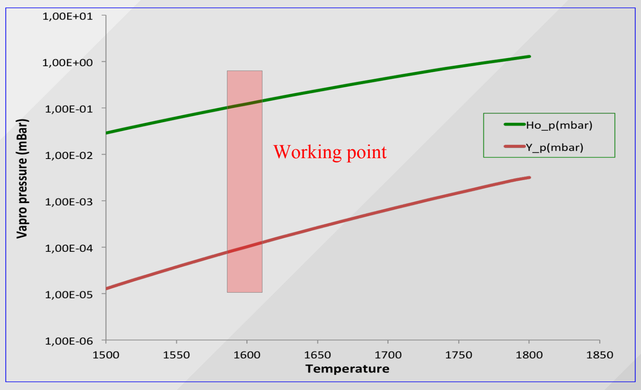
^{163}Ho will be produced by neutron irradiation of Er_2O_3 enriched in ^{162}Er at the Institut Laue-Langevin (ILL, Grenoble, France). The impurities and the contaminants will be removed chemically at the Paul Scherrer Institute (PSI, Villigen, Switzerland). The purified sample will arrive in Genova in oxide form (Ho_2O_3). The others holmium isotopes, and in particular ^{166}Ho(m), will be removed in the ion implanter. To avoid chemical shifts of the end-point, only holmium in the metallic chemical form must be introduced. Due to the higher ΔG of Yttrium, holmium could be reduced in metallic form using the following reaction:

Metal	Melting Point (°C)	Oxyde	ΔG (kJ/mol)
Holmium	1460	Ho_2O_3	-1791.1
Yttrium	1526	Y_2O_3	-1816.2

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

Distillation process

The substrate used for the collection of the distilled Ho is made of quartz because of its high resistivity to thermal deformation. A thin layer of a low degassing thermal conducting paste is used to improve the thermal conductance between the quartz substrate and the cooled copper cap. 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 tantalum 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 at working point. This condition minimizes Y contamination. The distillation efficiency is around 73%, determined as the ratio between the mass of the condensed Ho on the substrate and the lost mass on the crucible.

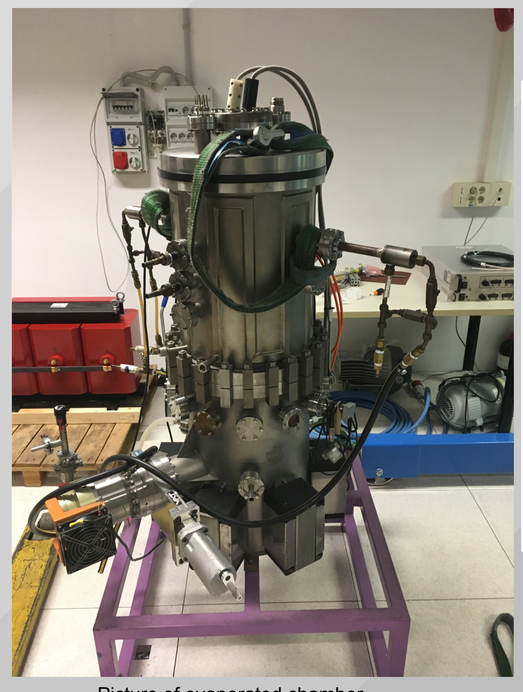


Vapor pressure of Holmium and Yttrium

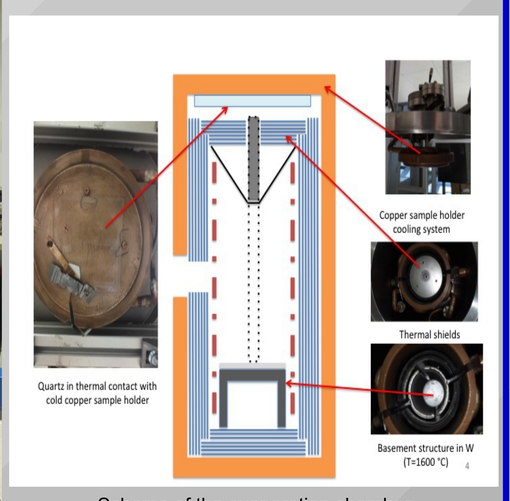
A) Quartz substrate coated with gold before Ho evaporation
B) Quartz substrate after Holmium evaporation

Evaporation chamber for holmium

A dedicated evaporation chamber is necessary in order to distillate Ho. The oxide sample is put in a small tantalum cylindric crucible inside an hot zone that could reach temperatures as high as 2000°C. The hot zone is thermally isolated by nine tungsten layer by a water cooled copper box. The upper part of the shield is holed allowing the evaporated Ho flows from the crucible to a substrate fixed on the top of the cooled copper box. The whole system is set in a vacuum chamber which could reach pressures as low as 10^{-8} mBar.



Picture of evaporated chamber



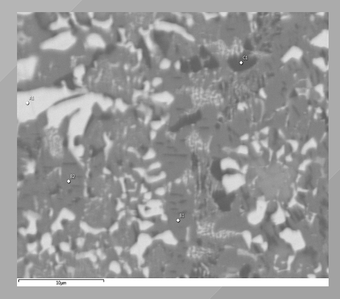
Scheme of the evaporation chamber.

Sputter target production

The sputter ion source of the implanter needs a metallic cathode containing the ^{163}Ho. We decided to realize a sintered sputter target. Ho(5%) is included in a metallic mixture of Ti(36%), Ni(41%), Sn(18% fine grained powder (< 40 μm). The compound is arc melted in argon atmosphere than milled and pressed at 350 bar/cm^2. 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 Ti2Ni2Sn 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.

Holmium implanter

A dedicated ion implanter 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 until 1.1 Tesla;
- a magnetic scanning stage (not yet mounted);
- a focusing electrostatic triplet (not yet mounted).

A) Sputter ion source with acceleration section;
B) A copper sputter target before the closing of the ion source;
C) Argon plasma and cathode filament light.

Picture of the implanter “under construction”

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 Ti2Ni2Sn metallic matrix with HoNiSn islands uniformly distributed.
- The implanter arrived at Genova at the beginning of 2017 and it is under installation and construction.
- The first test implantations (without radioactive material) are planned at the end of this year. The implantation of the final arrays of Holmes is planned for 2018.

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