

Fabrication of an Absorber with ¹⁶³Ho implanted for the Neutrino Mass Searches in the HOLMES project

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¹⁶³Ho Electron Capture Decaying isotope as probe for direct neutrino mass search, is presently the most appealing option for a calorimetric measurement in the framework of the micro calorimeter technology that has been developed. A small amount of ¹⁶³Ho atoms per detector, at a level of 10^{11} - 10^{13} , is enough for making high statistics measurements with micro-calorimeters with "standard" metallic absorbers. TES micro calorimeter with gold absorber containing ¹⁶³Ho isotopes can achieve spectral resolution as good as the ones developed for X-ray astrophysics. Respect to the former TES calorimeter they can be fabricated with in order to minimize the absorber volume, optimize the absorber-TES coupling in order to achieve a higher signal rise time. But the most important issue is the chemical forms in which Ho can be incorporated in the Absorber. As already stated, we show that the most easy method for ¹⁶³Ho production is the neutron irradiation of enriched ¹⁶²Er samples, which is typically in for of oxide, therefore the final product is composed mainly by Ho and Er oxides. The high precision required by the neutrino mass measurement need the knowledge of the chemical status of the ¹⁶³Ho, in order to study the effect of the chemical bound on the end-point energy. Ho-oxide and Ho-metal in metal absorber are here considered. Both are incorporated in metal absorber and studied form the chemical an structural point of view.

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1. Introduction

The HOLMES experiment [1] aims to measure the neutrino mass with an accuracy of 0.4 eV by studying the calorimetric spectrum of ¹⁶³Ho electron capture (EC decay) near the end point. The ¹⁶³Ho will be contained in a metallic absorber (Bi:Au, [2]) thermally coupled with a transition edge sensor (TES). An embedding system has been designed and set-up at the Genoa INFN laboratory: it consists of a holmium evaporation chamber to produce the metallic target and an ion implanter to embed the isotope in the absorber.

 163 Ho is produced via neutron radiation of Er₂O₃ powder enriched at 30% in 162 Er

$$^{162}Er(n,\gamma)^{163}Er \to ^{163}Ho + v_e,$$
 (1.1)

where ¹⁶³Er decay via EC with a half-life of 75 min and $\sigma(n,\gamma)$ is ~20 barns for thermal neutrons. The irradiation will be performed at the nuclear reactor of the Institute Laue-Langevin (ILL, Grenoble, France) with a thermal neutron flux of about 1.3 10¹⁵ n/s/cm² [1]. The isotope is produced in an oxide form (Ho₂O₃), which could modify the end-point shape. In order to avoid this effect we need to reduce the holmium in the metallic form by means of a reduction-distillation technique [3, 4] which will be described in section 2.

2. Holmium reduction distillation technique and results

Holmium oxide is mixed with metallic Y in a Knudsen cell and the system is heated at temperature of $\sim 1700^{\circ}$ C. In this condition oxygen is captured by metallic Y via the reaction:

$$Ho_2O_3 + 2Y \longrightarrow Y_2O_3 + 2Ho, \tag{2.1}$$

because the Y standard enthalpy of oxide formation $(\Delta_f H^o)$ is lower than Ho one. The operation temperature is chosen in such a way to have a Ho partial pressure higher than oxides and metallic Y ones [5, 6]. Thus it is possible to collect the evaporated material by simply using a cold plate. This test is done using Ho₂O₃ powder with natural isotope composition. The cell is fixed between two rheophores, inside a vacuum chamber (pressure 10^{-6} mbar) and, in order to measure the temperature, a type C thermocouple is placed in thermal contact with the Knudsen cell.

The condensed material (show in figure 1) is analyzed by X-ray Photoelectrons Spectrometry (XPS) to establish the chemical state obtained for its compound [7].

Figure 1: Condensed material on the slide after reduction distillation, weight 10 mg



The strongest peak of the holmium is the 4f shell and for the yttrium is the 3d shell (both at $\sim 160 \text{ eV}$). For this peak the background has a strongly dependence form shake-up peak: these are multielectron processes in which an atom is left in an excited state following a photoionization or Auger electron process, so that the outgoing electron has a characteristic kinetic energy slightly smaller than that of the parent photoelectron. To analize the chemical composition of the distilled material we have used the linear superposition hypothesizing that the spectrum measured from the distilled material (M), for each energy (E_i), is the linear sum of the spectra: holmium oxide (Ho_{oxide}), yttrium oxide (Y_{oxide}), metallic holmium (Ho_{metallic}) and metallic yttrium (Y_{metallic}) (shown in figure 2,3):

$$M(E_i) = \alpha Ho_{metallic}(E_i) + \beta Ho_{oxide}(E_i) + \gamma Y_{metallic}(E_i) + \delta Y_{oxide}(E_i)$$
(2.2)



Figure 2: Oxide and metallic holmium 4f shell spectra. In both spectra ara showed the shake up peak



Figure 3: Oxide and metallic Yttrium spectra. In both spectra ara showed the shake up peak

As it is shown in figure 4 the XPS analysis on the distilled material showed a spectrum compatible with the metallic Ho one. In fact the linear superposition shows a presence of metallic holmium larger than 90%, the other compound is holmium oxide (in figure 1 are shown the metallic holmium and in figure 4 is shown the distilled material XPS spectra and the linear superposition comparison).

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Molecular Formula	Chemical weight
$\operatorname{Ho}(m)$	$0.947 {\pm} 0.004$
Ho ₂ O ₃	$0.036 {\pm} 0.004$
Y(<i>m</i>)	$4 \ 10^{-11} \pm 4 \ 10^{-5}$
Y ₂ O ₃	$4 \ 10^{-12} \pm 6 \ 10^{-5}$

 Table 1: Distilled material chemical composition

3. Conclusion

As it was shown in section 2, we are able to produce metallic holmium starting from oxide holmium powder. This is a preliminary set-up and it allows us to think a new dedicated vacuum



Figure 4: Comparison between distilled material vs linear superposition

chamber to improve this reduction-distillation technique. At the moment we are designing a new high vacuum chamber able to reach a pressure down to 10^{-10} mbar. In this way the oxide contamination, due to the vacuum in the current chamber, will drop close to zero. In this new system we think to use a pyrometer to measure the temperature of the oven, in which the Knudsen cell will be placed, in order to know with a higher precision this parameter. Another important parameter is the efficiency of the process. We plane to measure it in different temperatures in order to reach the highest value and to use those parameters for the reduction and distillation of the ¹⁶³Ho.

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