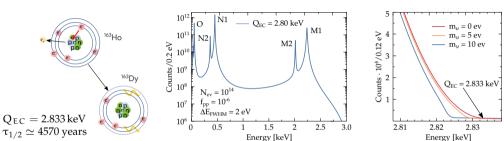
# An overview of the status of HOLMES, an experiment for measuring the neutrino mass

Elena Ferri

University of Milano-Bicocca and INFN of Milano-Bicocca on behalf of HOLMES collaboration







 $^{163}\text{Ho} + e^{-} \rightarrow \ ^{163}\text{Dy}^{*} + \nu_{e}(\text{E}_{c}) \quad \text{electron capture from shell} \geqslant \text{M1}$ 

- Calorimetric measurement of Dy atomic de-excitations (mostly non-radiative)

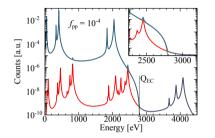
 $\Rightarrow$  measurement of the entire energy released except the  $\nu$  energy

- proposed for the first time by A. De Rujula e M. Lusignoli in 1982 Phys. Lett. 118B (1982) 429 Nucl. Phys. B219 (1983) 277-301
- rate at the end point depends on  $(Q E_{M1})$ : the proximity to M1 resonance peak enhances the statistics at the end point (i.e. sensitivity on  $m_{\nu}$ )
- Searching for a tiny deformation caused by a non-zero neutrino mass to the spectrum near its end point

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### $S(E_c) = \left[N_{ev}(N_{EC}(E_c, m_v) + f_{pp} \times N_{EC}(E_c, 0) \otimes N_{EC}(E_c, 0)) + B(E_c)\right] \otimes R_{\Delta E}(E_c)$



Nev	: total number of events
$N_{EC}(E_c, m_v)$	: <sup>163</sup> Ho spectrum
B(E)	: background energy spectrum
$R_{\Delta E}(E_c)$	: detector energy response function
fpp	: fraction of pile-up events
$R_{\Delta E}(E_c)$	: detector energy response function
$\Delta E$	intervall of energy

more details on Eur. Phys. J. C 74 (2014) 3161

- Pulse pile-up occurs when multiple events arrive within the temporal resolving time of the detector
- Unresolved pile-up events close to the end-point impairing effect on the end-point measurement
- The <sup>163</sup>Ho pile-up events spectrum is quite complex and presents a number of peaks at the end-point
- To resolve pile-up:
  - Detector with fast signal rise-time  $\tau_{\texttt{rise}}$
  - Pile-up recognition algorithm (i.e. Wiener filter, Singular Value Decomposition)

## The HOLMES experiment (ERC-2013-AdG no. 340321)

-

The  $m_{\nu}$  statistical sensitivity has:

- Strong dependence on statistic:  $\Sigma(m_\nu) \propto {N_{events}}^{1/4}$
- **Strong** dependence on pile-up:  $f_{pp} \simeq A_{EC} \cdot \tau_{res}$

(A  $_{E\ C}$  : pixel activity,  $\tau_{\rm res}$ : time resolution)

- Weak dependence on energy resolution  $\Delta E$ ;

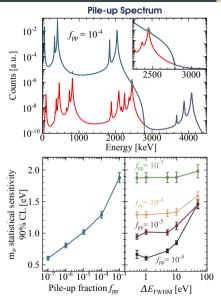
Multiplaxable detectors with fast response are required

### HOLMES

Neutrino mass determination with a sensitivity as low as  $\approx 1 \text{ eV}$ 

- Microcalorimeters based on Transition Edge Sensors with  $^{163}\mathrm{Ho}$  implanted Au absorber
- Pixel activity of  $A_{E\,C} \sim 300\,$  Bq/det
- Energy resolution: O(eV)
- Time resolution:  $\tau_{\rm res} \sim 3 \ \mu s$  (  $\tau_{\rm rise} = 10 20 \ \mu s$  );
- 1000 channels for  $3\cdot 10^{13}\,$  events collected in  $T_M=3\,years$

more details on Eur. Phys. J. C (2015) 75: 112



#### Production

<sup>163</sup>Ho production from <sup>162</sup>Er neutron activation

 $^{162}$ Er(n, $\gamma$ ) $^{163}$ Er  $\sigma_{\text{therm}} \approx 20b$  $^{163}$ Er +  $e^- \rightarrow^{163}$ Ho +  $\nu_e$   $\tau_{1/2} \approx 75$ m

- <sup>162</sup>Er irradiation at ILL nuclear reactor @ Grenoble: high thermal n flux
- cross section burn up  ${}^{163}$ Ho (n,  $\gamma$ )  ${}^{164}$ Ho not negligible (~200 b)
- ${}^{165}$ Ho  $(n, \gamma)^{166}$  m Ho  $(\beta, \tau_{1/2} \sim 1200 \mu)$  from Ho contamination or  ${}^{164}$ Er

### Purification

Chemical purification @ PSI before and after the irradiation

- radiochemical separation with ion-exchange chromatography
- efficiency better than 79%
- Expected  $^{166 m}$  Ho contamination fraction:  $\sim 10^{-3}$

Tb 161

#### Sample processed

Enriched Er<sub>2</sub>O<sub>3</sub> samples irradiated @ ILL, pre and post processed @ PSI:

- 25 mg, 55 days irradiation,  $A(^{163}Ho) \sim 5 MBq$
- 150 mg, 53 days irradiation,  $A(^{163}Ho) \sim 38 MBg$
- 544 mg, 50 days irradiation,  $A(^{163}Ho) \sim 120 MBg$
- \*  $\sim 100$  MBg enough for R&D and 500 pixels

Tm 163 1.81 h <sup>6</sup> <sub>β</sub> + γ 104; 69; 241; 1434; 1397	Tm 164 5.1 m 2.0 m h (h 2.0 m h 2.0	Tm 165 30.06 h <sup>6</sup> β <sup>+</sup> γ 243; 47; 297: 807	Tm 166 7.70 h <sup>6</sup> β <sup>+</sup> 1.9 γ779; 2052; 184; 1274	Tm 167 9.25 d	$\begin{array}{c} Tm \ 168 \\ 93.1 \ d \\ \epsilon; \beta^+ \\ \beta^ \\ \gamma \ 196; 816; \\ 447. \end{array}$
Er 162 0.139	Er 163 75 m	Er 164 1.601	Er 165 10.3 h	Er 166 33.503	Er 167
σ19 σ <sub>n. α</sub> <0.011	β <sup>+</sup> γ (1114) 9	ar 13 σn, α <0.0012	е по у	σ3+14 σ <sub>n.α</sub> <7E-5	ty 208 σ 650 σ σ σ 3Ε-6
Ho 161 6.7 s 2.5 h	Ho 162 68 m 15 m	Ho 163	Ho 164	Ho 165 100	Ho 166 1200 a 28.80 h
γ 28; 78 e <sup>-</sup>	e <sup>+</sup> 1.e <sup>+</sup> γ 165; 1520; 283; 937e <sup>+</sup>	ly 210 no y	by 37; 57 e <sup></sup> b <sup>-</sup> b <sup>-</sup> 1.0 y 97; 73 e <sup></sup> b <sup>-</sup>	σ 3.1 + 58 σ <sub>n, α</sub> <2E-5	0.07 γ 184; 1.9 810; 712 γ81 φ 3100 θ <sup>m</sup>
Dy 160 2.329	Dy 161 18.889 9 600	Dy 162 25.475	Dy 163 24.896	Dy 164 28.260	Dy 165 1.3 m <sup>by 106; e<sup>-</sup></sup> <sup>β<sup>-</sup>0.9; 1.0 <sup>y</sup> 515 (382)</sup>
u <sub>n.u</sub> <0.0003 Tb 159	σ <sub>6, n</sub> <1E-6 Tb 160	a 170 Tb 161	σ <sub>n. α</sub> <2E-5 Tb 162	1610 + 1040 Tb 163	Tb 164

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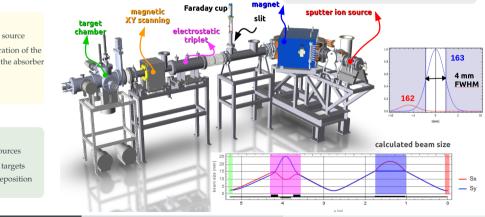
## Ion implanter

**Ion implanter** designed to embed Ho inside the detectors absorbers and to perform a mass separation of the  $^{163}$ Ho from the other contaminants.

- extraction voltage 30-50 kV  $\rightarrow$  10-100 nm implant depth
- <sup>163</sup>Ho/<sup>166</sup><sup>m</sup>Ho separation better than 10<sup>5</sup>

#### Main components:

- Ar penning sputter ion source
- magnetic dipole mass analyzer (B  $m \alpha x = 1 T$ )
- faraday cup and slit
- target chamber for Au co-evaporation



#### Au co-evaporation:

- to fully encapsulate the source
- to compensate the saturation of the <sup>163</sup>Ho concentration in the absorber
- to avoid oxidation
- heat capacity

#### Target chamber:

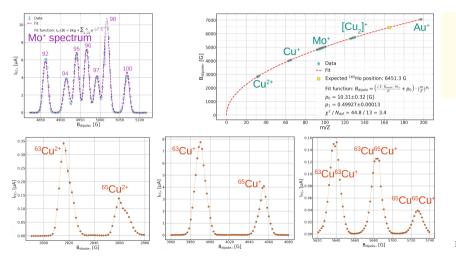
- 4 COMIC microwave sources
- 4 Ar beams hit on 4 Au targets

 $\rightarrow$  4 in order to increase the deposition rate and uniformity

## Ion implanter calibration



#### Magnetic field vs mass-to-charge ratio calibration with Cu, Au and Mo peaks.



- Cu/Au from sputter target/holder
- Mo from the anode
- The source produces also multiple-ionized and dimeric ions from the same material, which can also be used for calibration

for more details Mariia Fedkevych's talk @ NuMass 2022



#### Efforts are put to build the most suitable target for the Ho sputtering

 $\rightarrow$  different techniques for target fabrication are tested

#### Molecular plating

Electrodeposition of Ho complexes in an organic solvent at high voltages with high uniformity and efficiency (>90%)

#### Drop-on-demand inkjet printing

put droplets of solution containing compound and let solvent evaporate to deposit the dissolved compound

#### Sintered targets

Ho(NO<sub>3</sub>)<sub>3</sub> in a metallic mixture of Zr and Y fine-grained powder preparade pressed at 350 bar/cm<sup>2</sup> and baked at 950°C

#### **Coupled** reduction

Ho reduction and diffusion into backing material due to thermodynamically favourable formation of intermetallic compound





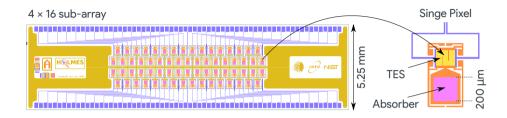




#### With sintered target we obtained the best current-stability:O(200) nA over 15 h!

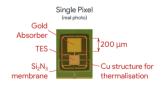
ena	



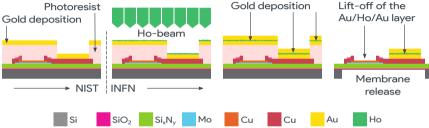


- Mo/Cu TES coupled to Gold absorbers where <sup>163</sup>Ho will be ion-implanted
- 2  $\mu$ m Gold thickness for full e/ $\gamma$  absorption
- Side-car design to avoid TES proximitation effect
- Thermal conductance G engineering for  $\tau_{\mbox{\tiny decay}}$  control
- $4\times 16$  linear sub-array designed for high implant efficiency and low parasitic L
- Optimized design for high speed and high resolution:

Specs @ 2.8 keV :  $\Delta E_{FWHM} \simeq 3-4\,eV$  ,  $\tau_{rise} \simeq 10\,\mu s$  ,  $\tau_{decay} \simeq 100\,\mu s$ 







### <sup>163</sup>Ho isotopes embedded in metallic absorbers (through ion-implantation)

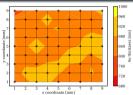
- Fabrication in two steps:
  - $\blacktriangleright\,$  NIST: TES fabrication with 1  $\mu m$  Au absorber
  - + INFN:  $^{163}\text{Ho}$  implantation, final deposition of 1  $\mu\text{m}$  Au and SiN membrane release
- final micromachining step definition in progress
  - $\Rightarrow$  KOH vs DRIE machining

## HOLMES: detectors fabrication process (cont.)



### Au deposition

- 1µm of Au deposited
  - with Ion beam sputter system
  - at rate of around 52 nm/h  $\rightarrow$  about 20 h for 1 $\mu$ m
  - gold thickness uniformity  $\rightarrow\,\sigma_t\,/\,t\sim 4\%$



#### Lift-off

Removal of the resist mask (7 µm thickness)

- sample in acetone at  $40^{\circ}$ C for 24 h

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After the lift-off, the Au deposited remains only on the absorber:

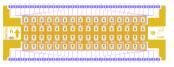
 $\rightarrow$  Minimal crowning and almost isotropical deposition thanks to the 4 ion beam sources



#### Membrane release

#### кон

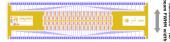
- Anisotropic wet etching
- Requires more spacing between pixels
- Sucessfully tested



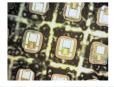
### DRIE

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- Silicon Deep Reactive Ion Etching
- Best for close packing
- High implant efficiency
- Not yet tuned

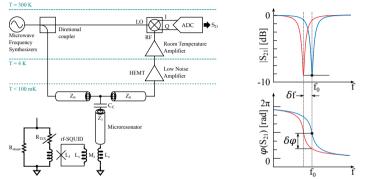




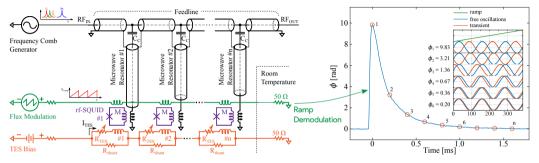




### HOLMES TESs readout is based on microwave rf-SQUID multiplexing



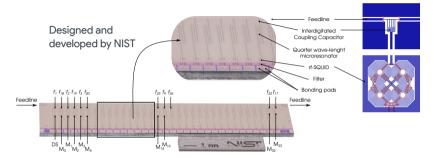
- rf-SQUID inductively coupled to a dc-biased TES and to a high-Q superconducting  $\lambda/4$ -wave resonator
- Change in TES current  $\Rightarrow$  change in the input flux to the SQUID
- The rf-SQUID transduces a change in input flux into a variation of resonant frequency and phase
- Each micro-resonator can be continuously monitored by a probe tone



- By coupling many resonators to a single microwave feedline it is possible to readout multiple detectors
- Sensors are monitored by a set of sinusoidal probe tones (frequency comb)
- At equilibrium, the resonator frequencies are matched to the probe tone frequencies, and so each resonator acts as a short to ground
- The ramp induces a controlled flux variation in the rf-SQUID, which is crucial for linearizing the response
- Large multiplexing factor (> 100) and bandwidth, currently limited by the digitizer bandwidth



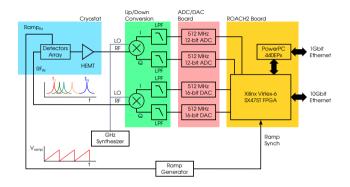
### The core of the microwave multiplexing is the multiplexer chip



- Superconducting 33 quarter-wave coplanar waveguide (CPW) microwave resonators covering 500 MHz in the 4-8 GHz frequency range
- 200 nm thick Nb film deposited on high-resistivity silicon ( $\rho > 10 \text{ k}\Omega \cdot \text{cm}$ )
- each resonator has a trombone-like shape with slightly different length
- 2 MHz bandwidth per resonator
- separation beetween resonances 14 MHz (to prevent cross-talk)
- resonance depth greater than 10 dB
- squid equivalent noise less than  $2\mu\varphi_o/\sqrt{Hz}$

### HOLMES DAQ with the ROACH2





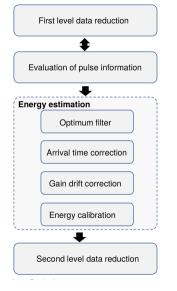
- Software Defined Radio with the open system ROACH2 (Casper collaboration)
- ADC BW 550 MHz
- real time pulse reconstruction
  - $\rightarrow$  at the moment readout available for 64 channels

Multiplexing factor proportional to the target rise time

- $n_{\text{TES}}\approx 3.4\cdot\tau_{\text{rise}}$
- requiring  $\tau_{\text{rise}}=10 \mu s$

### Event reconstruction





- Robust analysis is mandatory for achieving the expected microcalorimeter intrinsic energy resolution.
- The data from each pixel need to be processed separately.

#### Watson toolkit

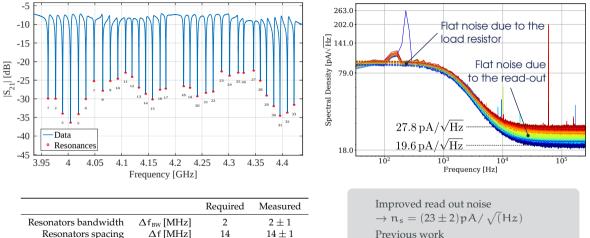
- Software for low temperature detector data analysis
- Object oriented programming. Written in python (numpy and scipy)
- Fast, easy to read, easy to fix code
- GUI with QT5 for handy day to day operations
- Data are stored in hdf5 (hierarchical, filesystem-like data format)Anisotropic wet etching





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## Multiplexing: characterization results



Previous work

$$\rightarrow$$
 n<sub>s</sub> = (26 ± 7)pA/ $\sqrt{(Hz)}$ 

more details on IEEE TAS 31 (2021) 5, 2100205

All the microresonator parameters match the HOLMES specification

 $\Delta S [dB]$ 

> 10

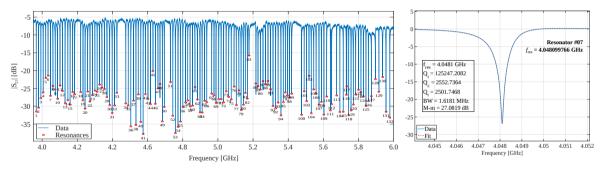
Resonators depth

 $29\pm 6$ 

## Multiplexing: characterization results (cont.)



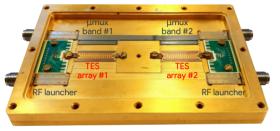
Forward transmissiom S<sub>21</sub> of 4 different band chips wired in series and an example of resonce fit

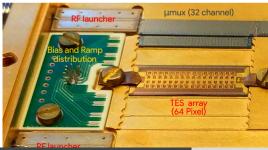


Four µmux in series are able to cover a wide frequency range from 4 to 6 GHz

### HOLMES: test on the processed detectors



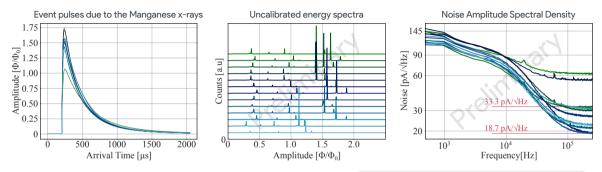




- Holder designed to host 128 Channels:
  - $2 \times (4 \times 16)$  sub arrays
  - ► 4× µmux multiplexer chips with 4 bands
- 8 holders will cover the entire HOLMES in its final configuration (1024 channels);
- Preliminary low temperature tests performed with fully processed arrays (with KOH):
  - detector with (1 μm) absorber at NIST
  - absorber finalized (1 μm) at MIB
  - wet etching at MIB
- 32+32 TES pixels bonded (half of the available)
- Absorbers without the <sup>163</sup>Ho implanted
- New SDR firmwares for 16 and 32 channels: 16-channel version fully operational 32-channel version under testing
- New up/down-conversion system fully operational

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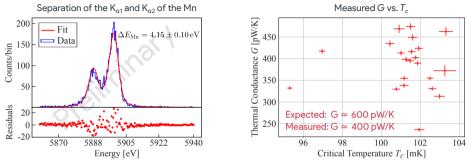


- non implanted detectors with KOH membrane release
- 13/16 working detectors (3 detectors with problematic resonators)
- Calibration run performed with a primary  $^{55}\mathrm{Fe}$  source faced to different targets
- X-ray fluorescence emission lines:

<sup>55</sup>Mn (5.9 keV) <sup>40</sup>Ca (3.7 keV) <sup>40</sup>Cl (2.6 keV) <sup>27</sup>Al (1.5 keV)

Measured read out noise  $n_{s} \sim (19-33)\,pA/\sqrt{Hz}$ 

- Compatible with the previous prototypes Eur. Phys. J. C (2019) 79:304
- Two channels with higher noise due to not optimal rf-SQUID oscillations



For the best detector:  $\Delta E_{Mn} = 4.15 \pm 0.10 \, eV @ 5.9 \, keV$ 

- Energy resolution in the (4 6) eV range @ 5.9 keV
   Large spread probably due to the large G dispersion different G ⇒ different working point
- $\tau_{rise} \simeq 20~\mu s$  and  $\tau_{fall} \simeq 300~\mu s$  longer fall time due to lower thermal conductance G

KOH vs DRIE machining

- same energy resolution and rise time
- longer decay time and larger coupling dispersion

## Background



The count rate at the ROI is very low (0.26 counts/eV/day/det @ [2650,2833]eV)  $\rightarrow$  the fraction of background signals must be kept as low as possible

#### Background

#### 1. Pile-up

 $\rightarrow$  the main background source for pixel with A  $_{E\,C}\sim$  300 Bq and  $\tau_R\sim$  1.5  $\mu s.$  (0.8 counts/eV/day/det @ ROI)

#### 2. Internal radionoclides

 $^{166\,\mathrm{m}}\,\mathrm{Ho} \rightarrow \mathrm{expected}$  count rate <0.01 counts/eV/day/det @ ROI

#### 3. Natural radioactivity

Smooth and almost flat background @ ROI except for  ${}^{40}$ K

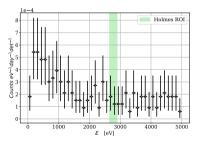
#### 4. Cosmic rays

GEANT 4 simulation  $5x10^{-5}$  counts/eV/day/det @ [0,4000] eV

3. and 4. can be comparable or even overcome the pile-up rate if the  $^{163}$ Ho activity per pixel is too low.

#### Background measurement

Single interaction in a pixel produces a background spectrum which seems to be monotonically decreasing.



0.0001 counts/eV/day/det @ HOLMES ROI  $\rightarrow$  lowering with a muon veto

### Conclusion

- A powerful tool to determine the effective electron-neutrino mass is the calorimetric measurement of the energy released in <sup>163</sup>Ho electron capture (EC)
- The HOLMES experiment will performe a direct measurment of the neutrino mass by using microcalorimenters with absorber <sup>163</sup>Ho-implanted
- Ion implanter is working as expected. The production of a proper sputter target is almost ready!
- The software for analysis and signal processing of microcalorimeters events is up and running!
- For reading out the 1024 detectors, HOLMES will use the microwave multiplexing read-out
  - All the microresonator parameters match the HOLMES specification
- Transition edge sensors with Au absorber where the <sup>163</sup>Ho will be ion-implanted
  - Tested and tuned the final array fabrication processes
  - TES characterization with a fluorescence source without Ho
  - The performances (energy and time resolution) required by HOLMES are achieved
- The first phase of the HOLMES experiment is expected on the last quarter of 2022: a low dose implantation of a 2x32 pixel array