The HOLMES low activity implantation

G.Gallucci on behalf of HOLMES collaboration

NuMass 2024 - Determination of the absolute electron (anti)-neutrino mass

Genova, 26th Feb – 1th Mar

Outline

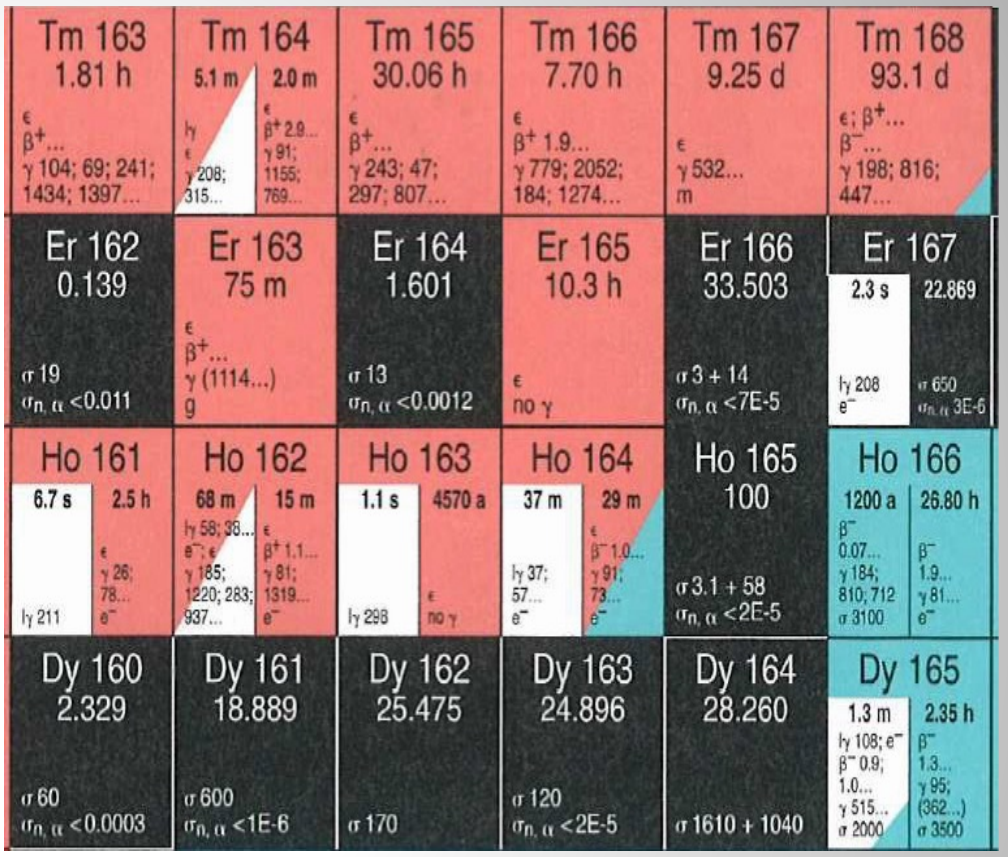
- Holmium implanter and ion source
- Holmium ion sputter target
- Implanter calibration and beam commissioning
- First low activity implantations

^{163}Ho production and separation

^{163}Ho is produced by neutron irradiation of Er_2O_3 enriched (30%) in ^{162}Er at the Institute Laue-Langevin (ILL, Grenoble, France).

$^{162}Er\ (n,\gamma)\ ^{163}Er \quad \sigma_{thermal} \approx 20b$

$^{163}Er \rightarrow ^{163}Ho + \nu_e \quad \tau_{1/2}^{EC} \approx 75min$



Two types of contaminants:

Other elements than Ho : ^{170}Tm and ^{171}Tm, both beta emitters, are produced by irradiation of ^{168}Er and ^{170}Er. ^{159}Dy could be produced from ^{158}Dy impurities. Also, the presence of stable elements like residual erbium (164, 166, etc...) or dysprosium (163, 164, etc...) are not suitable. Radiochemical separation with ion-exchange resins in hot-cell at PSI (efficiency > 80%).

Holmium Isotopes: residual of ^{165}Ho, but in particular the irradiation of ^{165}Ho present in the sample (as impurity or from irradiation of ^{164}Er and Dy isotopes) creates ^{166m}Ho, β^- with Q = 1.855 MeV, $\tau_{1/2}$ = 1200y. vThe ratio between activity A(^{163}Ho)/A(^{166m}Ho) = 100 ~ 10000.

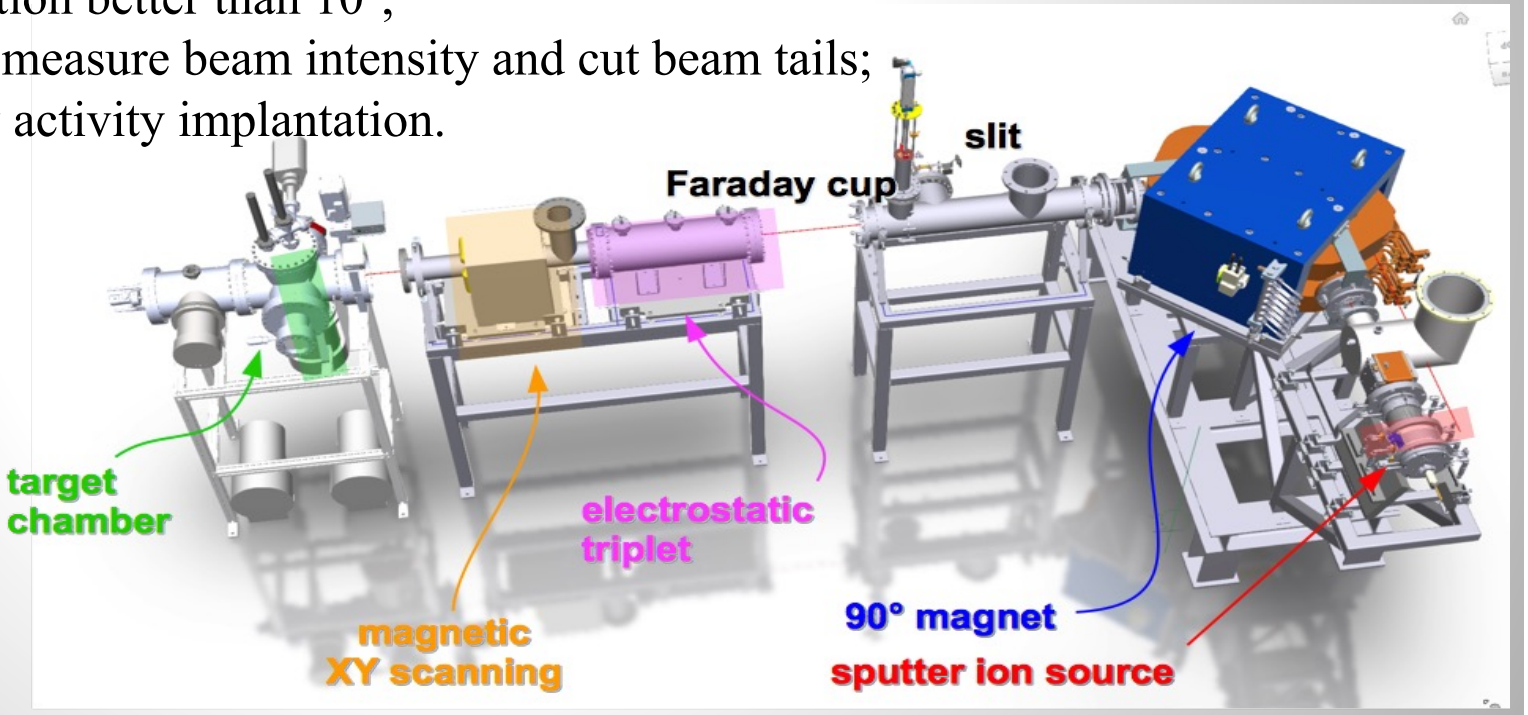
Implanter

A dedicated ion implanter to remove contamination of holmium isotopes different from ^{163}Ho and other impurities.

1. **an argon sputter ion source** to create the ion beam;
2. **an acceleration section** to reach a maximum beam energy of 50 KeV;
3. **a steering magnet** to correct vertical shift of beam;
4. **a magnetic dipole mass analyser** with a field up to 1.1 Tesla and capable to guarantee ^{163}Ho/^{166m}Ho separation better than 10^5;
5. **a Faraday cup and a slit** to measure beam intensity and cut beam tails;
6. **a target holder** used for low activity implantation.

There are three parts not yet mounted in the actual configuration:

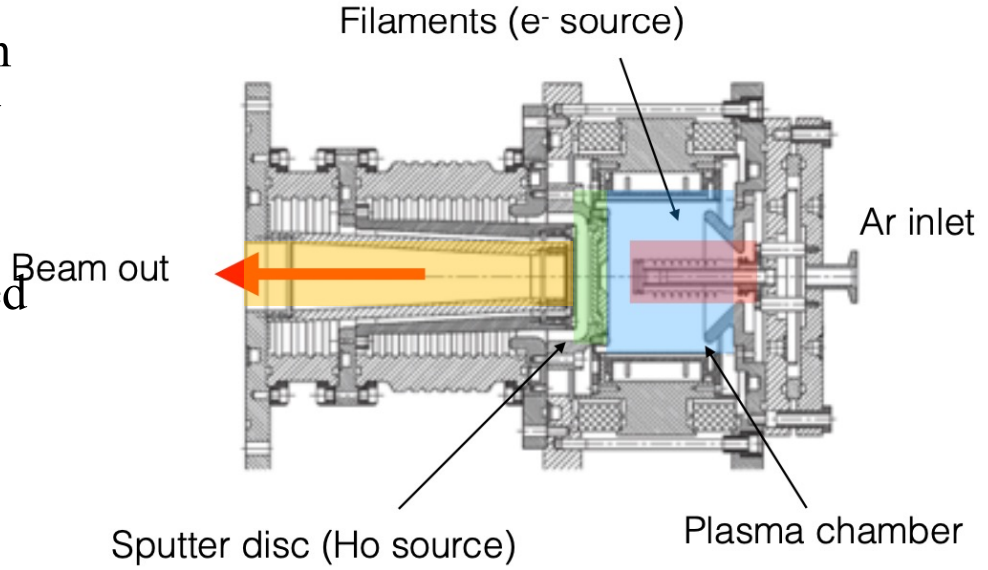
- a magnetic scanning stage;
- a focusing electrostatic triplet;
- a target chamber for implantation and gold co-evaporation.

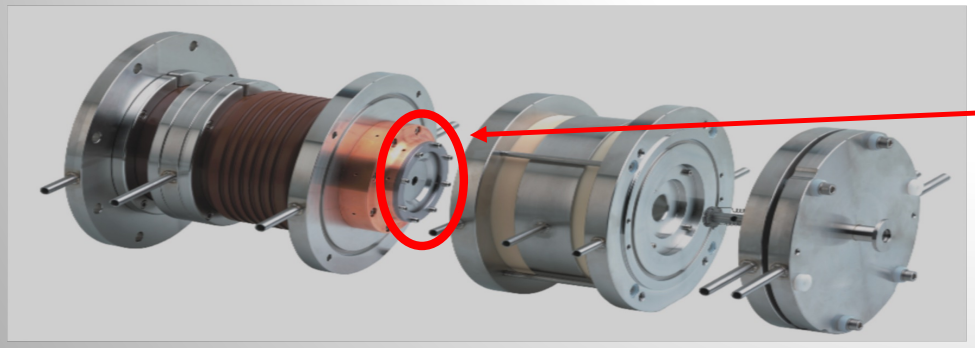


The sputter ion source

- A controlled argon flow enters inside the sputter chamber and is ionized by electrons emitted for thermionic effect from a four filaments. The ionization is increased by a magnetic field and could be tuned by an electrostatic potential. When the ionization is sufficient, a “quiet and stable” Ar plasma is created.
- The Ar plasma ions are attracted by a negatively-biased (max 600 V) metal disk (sputter target) in front of the outlet hole and sputter the disk populating the plasma with atoms from the target. The plasma diffuses through the hole and enters in the acceleration section (Extraction potential 20-50kV).

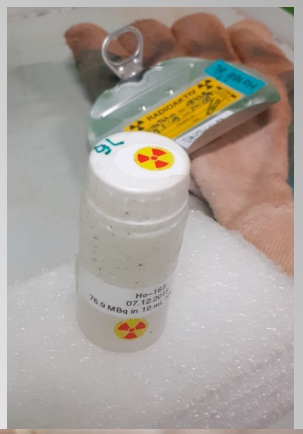
All parts are water cooled but the sputter target reaches temperatures more than 1000 ^{O}C.





The sputter target is ring-shaped and consists in a support done by Ti or Mo and the real target. We tried different techniques to create the target and selected the best one.

Sputter Target



We need a metallic target containing the ^{163}Ho. The holmium, after chemical purification, is in nitric acid form ($^{163}Ho(NO_3)_3$) with PH<4 to avoid adhesion to the vial wall. In collaboration with PSI and Chemistry Department of Genova University we tested different 4 different solutions.

Sintered target

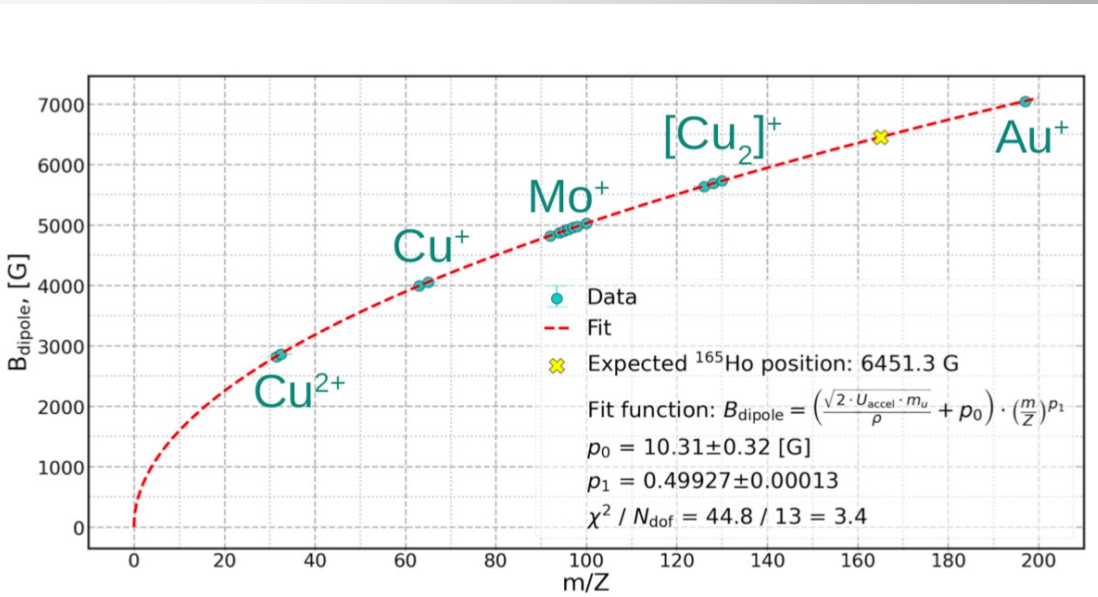
Different metal fine grained powders (Zr, Bi, …) in a Mo support are compressed at ~ 200 bar/cm^2 and baked at 950 ^{O}C for 2 hours in high vacuum (<10^{-6} mbar).

The acid solution of Ho ((Ho$(NO_3)_3$ is dripped and dried in the obtained final target.

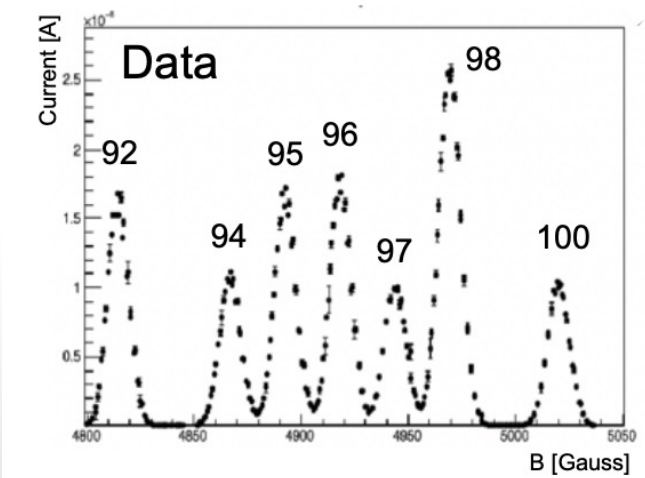
The final sputter target consists of a Zr/Bi (98%/2%) sintered matrix with Al powder to increase adhesion at target/support interface. The Ho solution is "dripped" and dried in a dedicated facility in Genova radioactive source bunker.

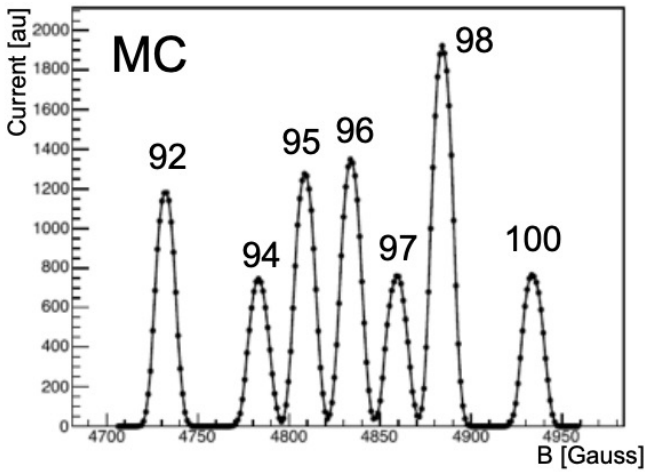
Implanter calibration

The implanter is calibrated using the different materials inside the chamber and the target. They allowed for a magnetic field vs mass-to-charge ratio calibration. A small misalignment was found and considered during implantation process.



Multiple isotopes element (like Mo) are used to extract beam size **(σ ~ 1.3 / 1.5 mm)** and cross check MC simulation reliability



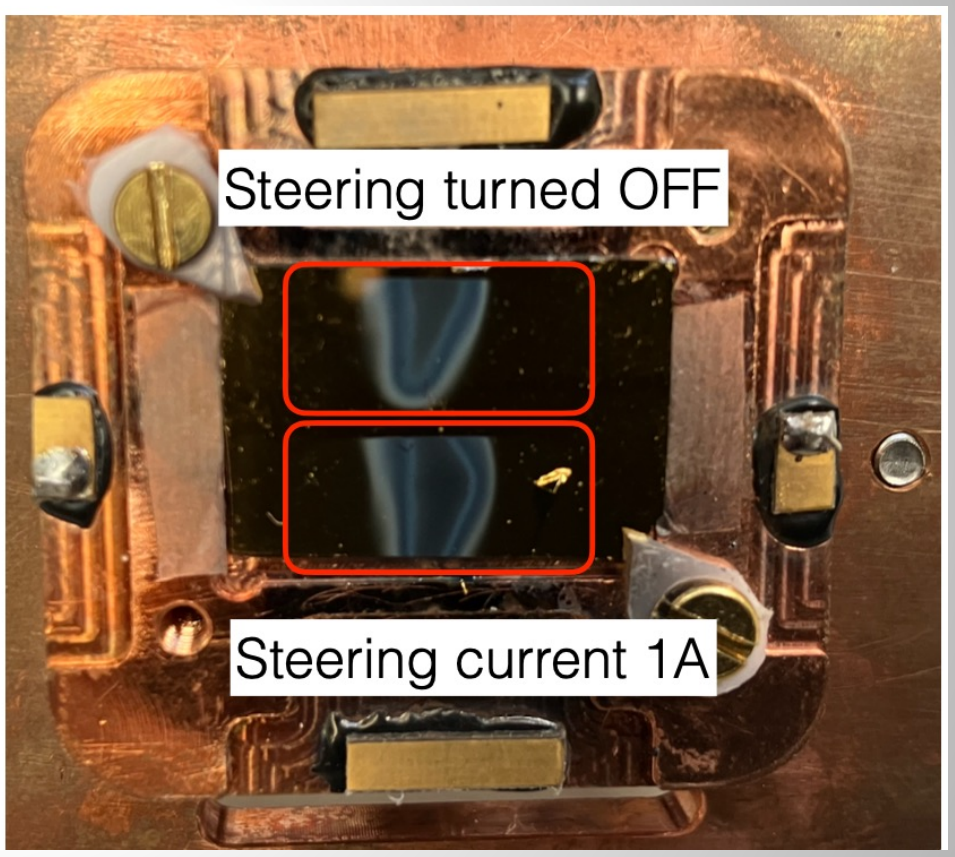


Beam spot and vertical position

In the current implanter setup, the only diagnostic tool is a Faraday Cup, which is not the best tool for alignment purposes.

Beam profile and alignment were checked with high current implantation runs using gold plated silicon substrate as targets and looking for the beam “shadow”.

We found beam profile is not exactly gaussian - mainly in y direction. This is under study with MC simulation.

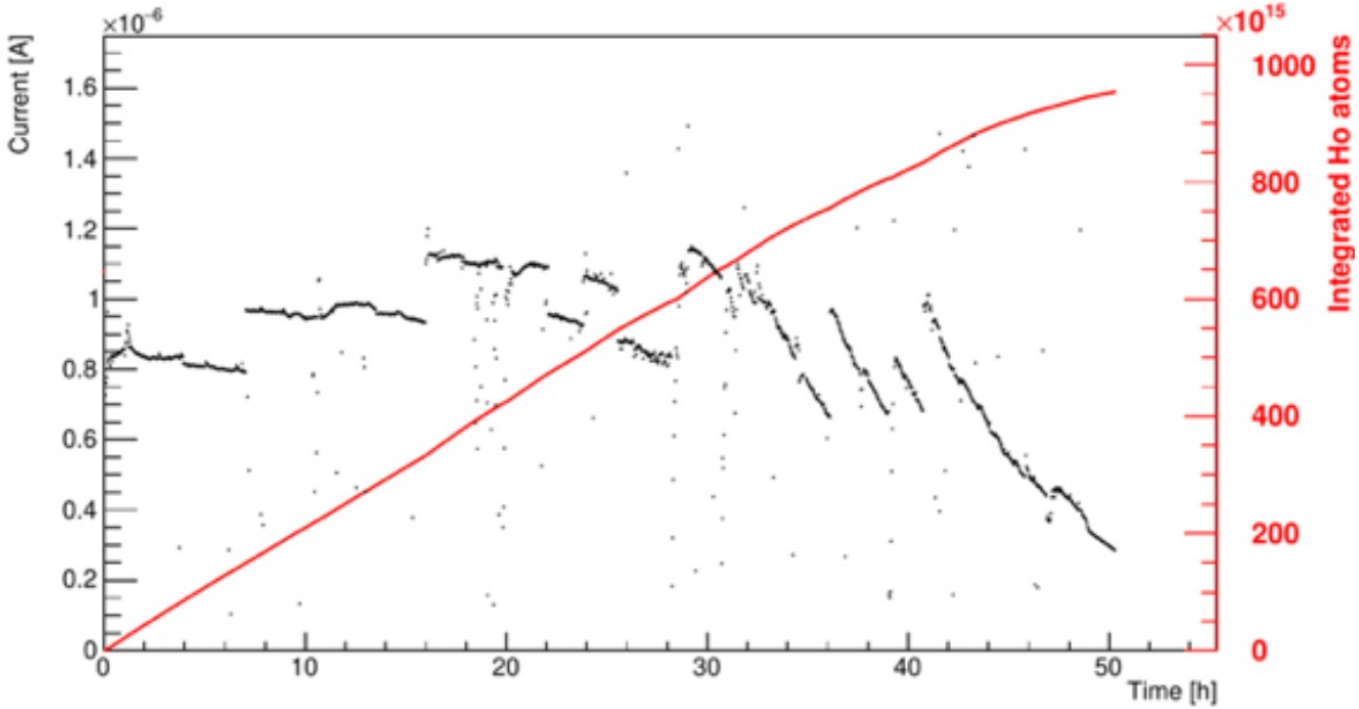


Extraction efficiency

- Extraction efficiency is evaluated using ^{165}Ho loaded target by acquiring long run (>50h running time) up to a total consumption of the target and comparing integrated charge with ^{165}Ho content.

- An efficiency ε ~ 0.2 % was found. It is low but enough to proceed with first implantation.

- Studies are ongoing to improve tl source extraction efficiency.



Jumps in beam current plot are due to different source configuration (sputtering and discharge voltage)

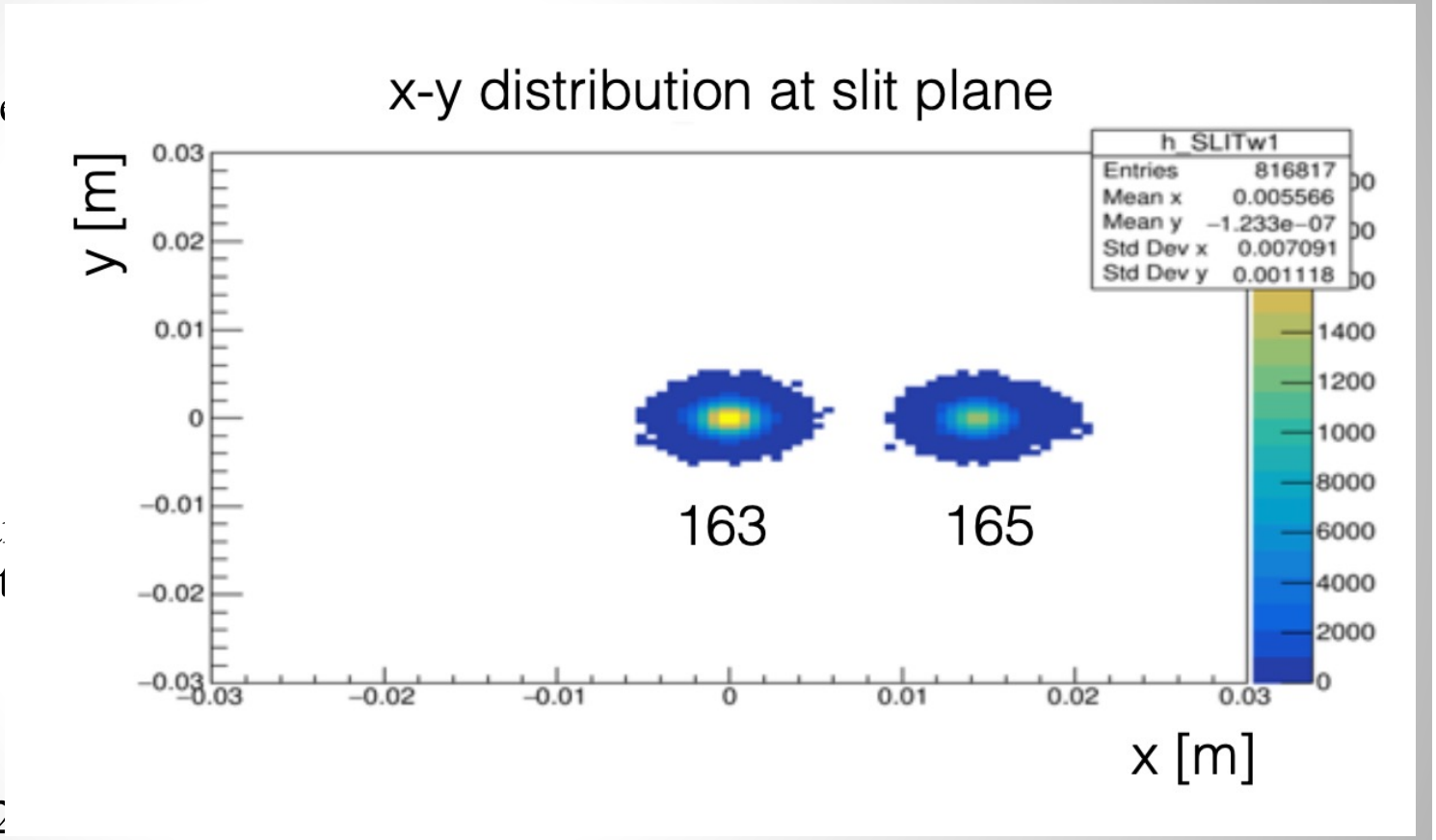
Expected Separation

- Our solution contains $^{163}Ho/^{165}Ho/^{166m}Ho$ with relative abundance: 60/40/0.1

- 163 vs 165/166 a.m.u. separation evaluated by MC simulation.

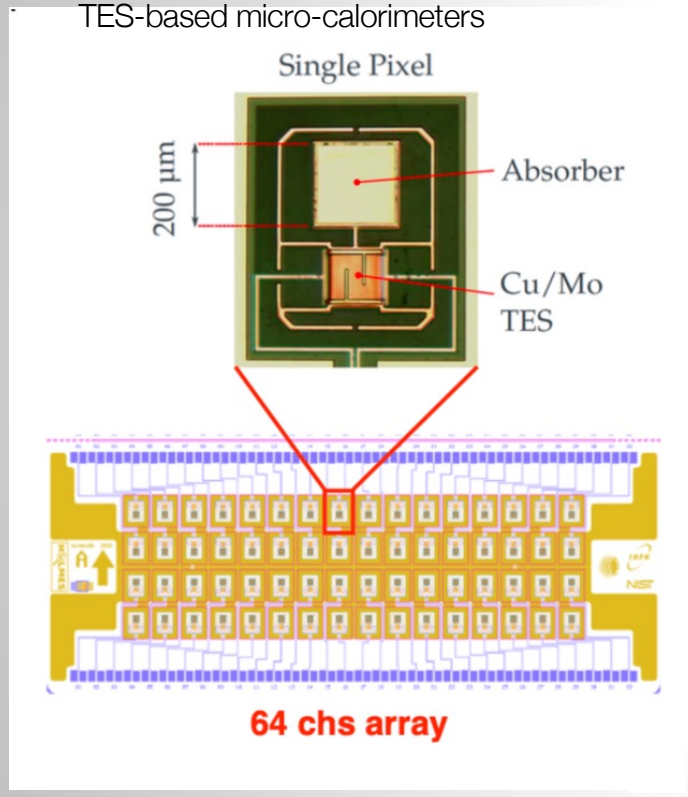
- The distance between ^{165}Ho a ^{163}Ho, at slit plane, is expected t be about 15 mm.

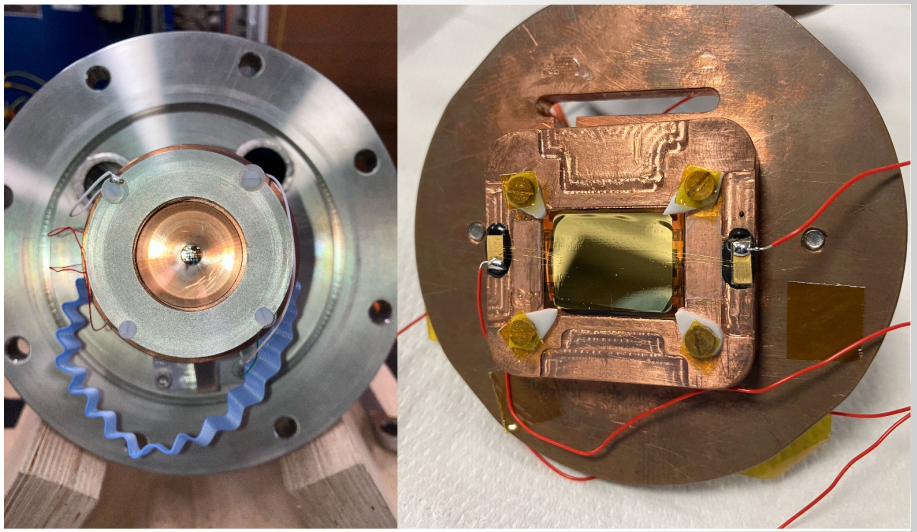
- The distance between ^{166m}Ho and ^{163}Ho is expected to be ~ 22 mm.

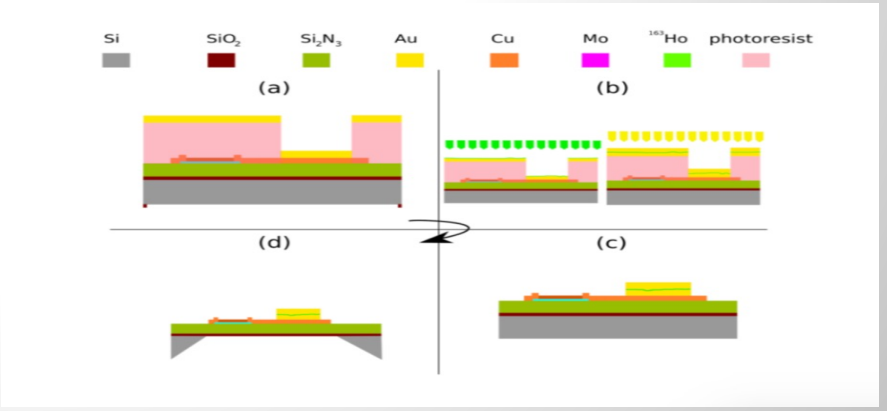


Remainder: HOLMES detectors

Each Holmes chip has 64 pixels and a 32 pixels chain is implanted in each run. For the first implantations, a dummy support is used. The chip in covered of gold but the not-absorber part are masked by photoresist.



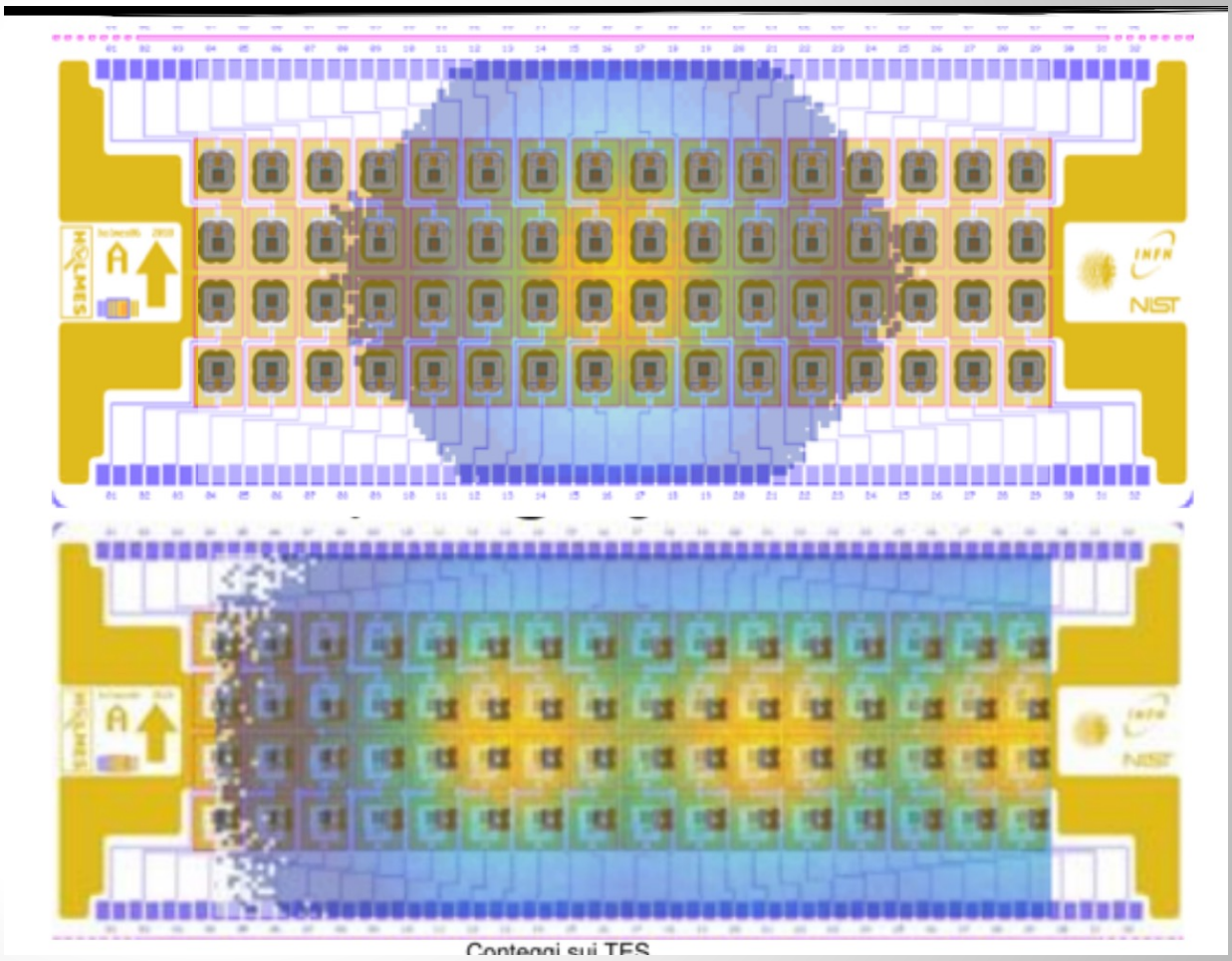




After the implantation, the chip goes to Milan for "sealing" with gold, removing mask and suspension. (see L.Origo talk)

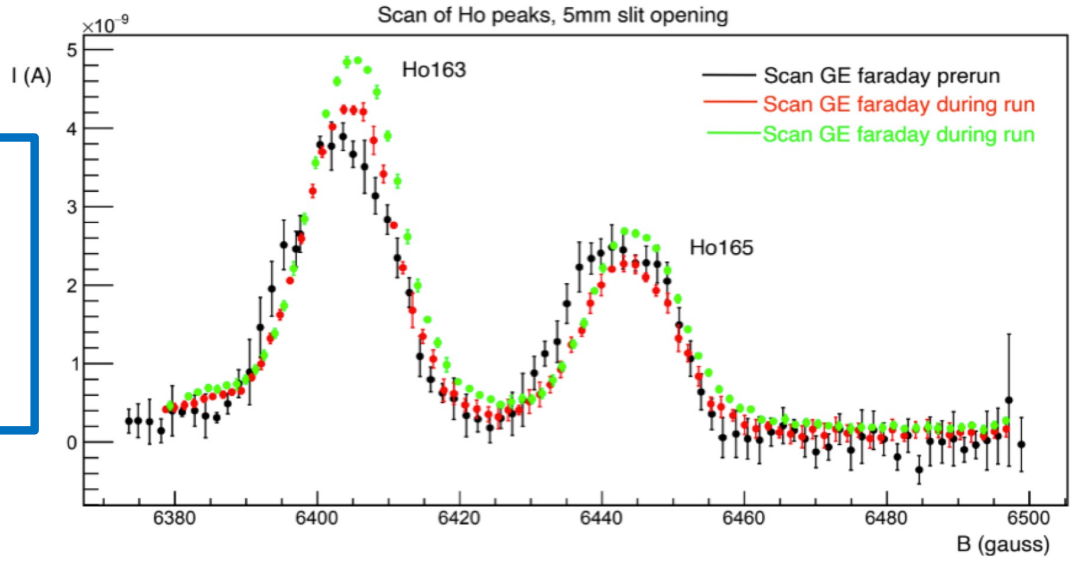
First implantations

- Two chips implanted with 25keV Ho beam, four chains with four different geometries for evaluate characteristic of implantation, beam profile, Ho effect on detector properties, etc…
  - single spot in the center of the array,
  - multiple (3 positions) spots
  - multiple (4 positions) spots
  - Vertical position
- Geometrical efficiency was evaluated by means of MC simulations.

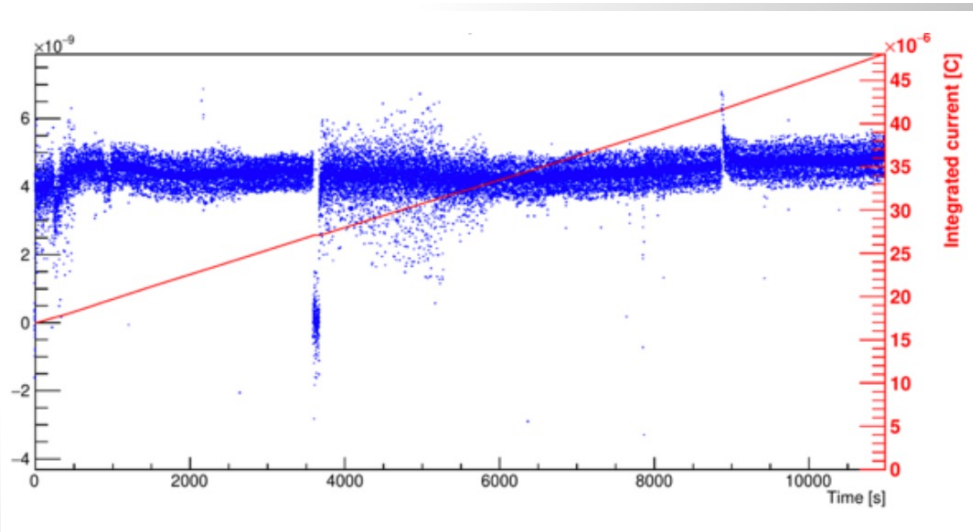


First implantations (2)

We evaluate the mass separation between 163/165 during implantation run and it was ~45 G corresponding to ~ 15 mm, as expected.



The dummy target holder was designed in "Farady cup form" and we are able to measure the beam current during the implantation.
For single spot, beam current of 5 nA for 3 hours.
Expected activity ~2Bq on central TES.



First implantation (3)

Single spot implanted chain:

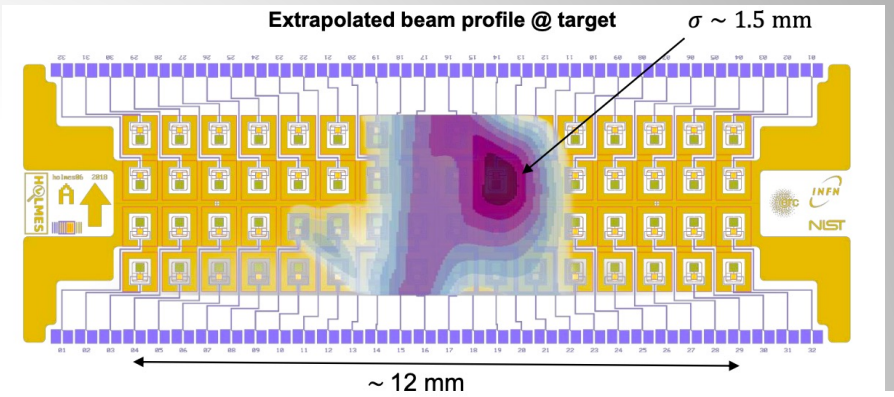
- Max activity ~ 1 Bq factor 2 discrepancy with respect

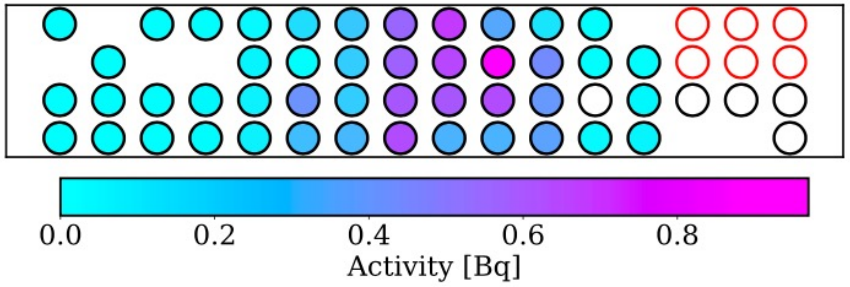
to expectation.

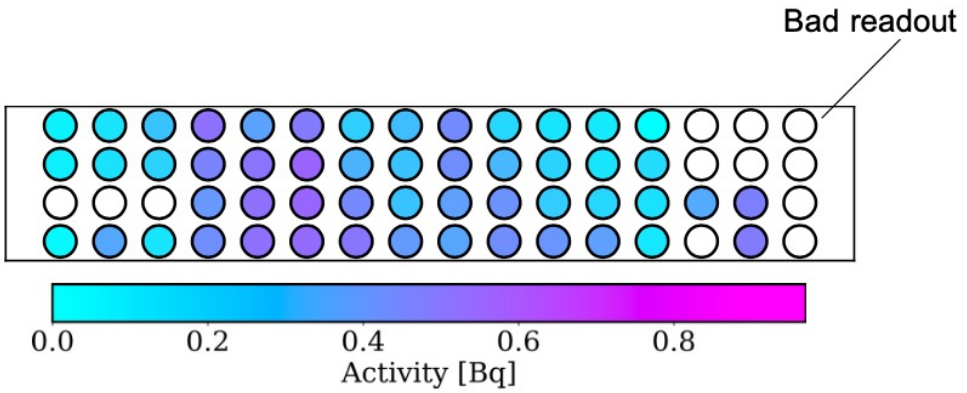
- Mean activity ~0.24 Bq, total activity ~ 10 Bq
- From activity map, extracted beam size ~ 1.5mm, in good agreement with expectation.

Multiple spots and vertical implanted under studies.
From preliminary measurements of 3-spot chain we obtain a mean activity ~ 0.3 Bq and a total activity > 15.4 Bq.

Activity discrepancy with respect to expectation was found is under investigation.



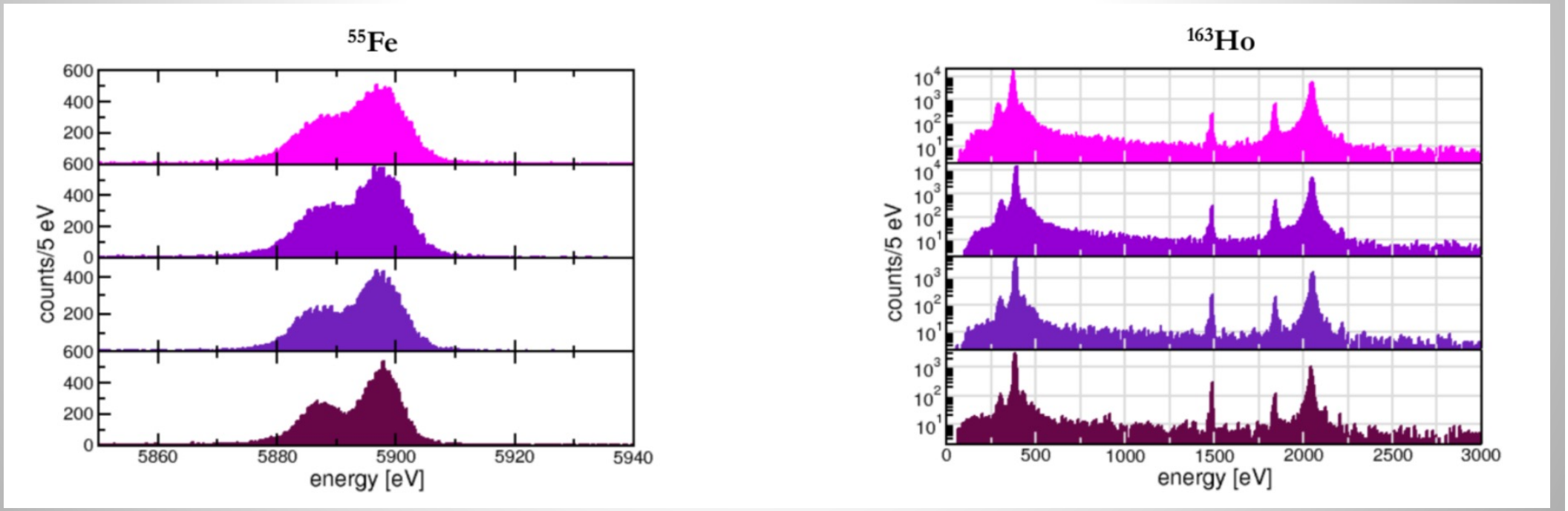




First implantation (4)

Preliminary ^{163}Ho spectrum obtained and TES characterization, timing and energy resolutions studies are on going (see L. Origo talk).

TES #	ΔE_{FWHM} @6 keV [eV]	^{163}Ho activity [Bq]
13	8.36 ± 0.09	0.97
17	7.78 ± 0.08	0.55
19	7.12 ± 0.08	0.21
21	5.76 ± 0.07	0.11



Calibration source

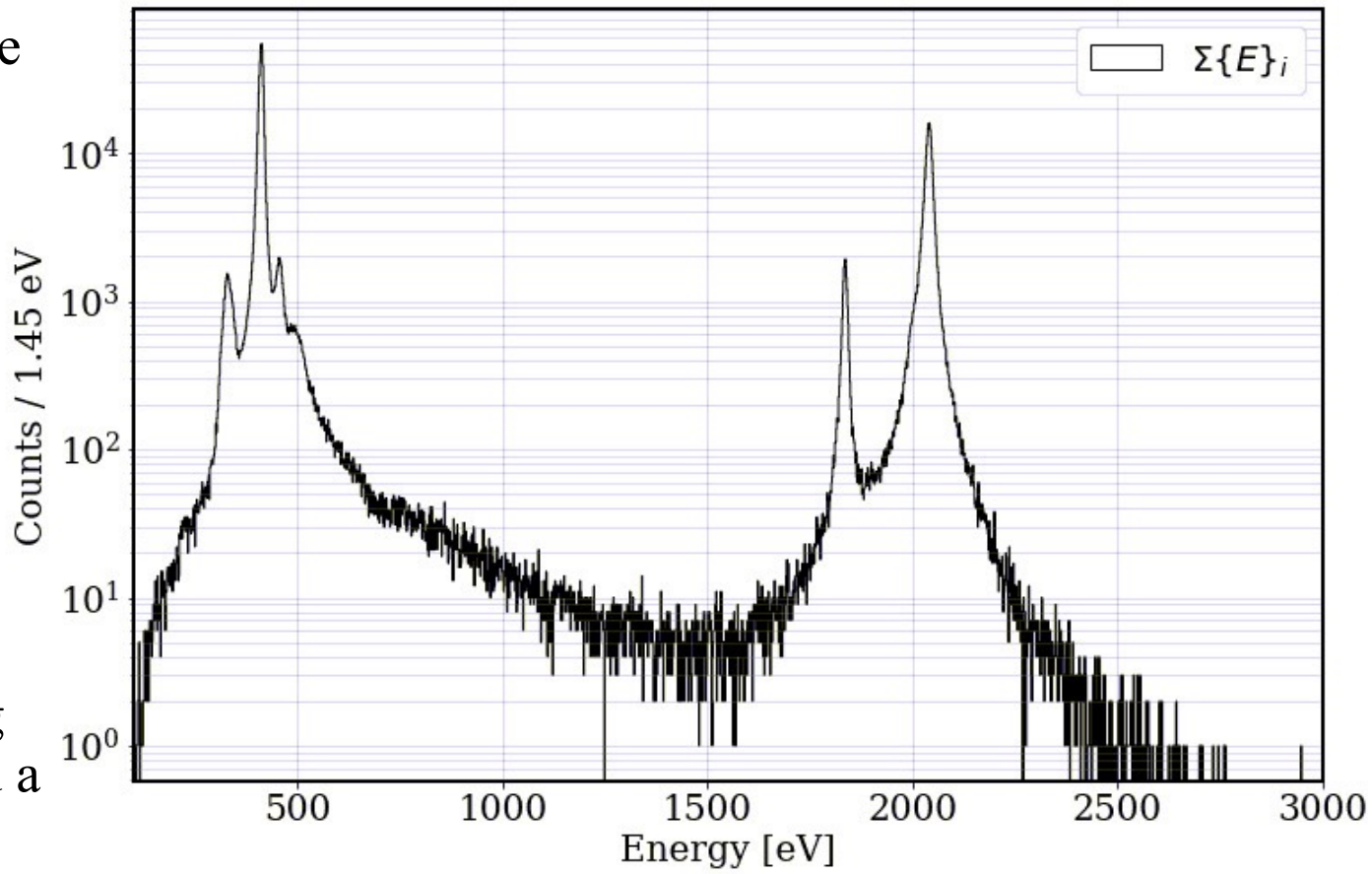
^{163}Ho spectra

Conclusions

- Four different geometry low activity implantations are done and under studied.
- Good agreement between excepted and measured characteristics.
- A discrepancy in implanted activity is under study.

Next step

- Upgrade of the implanter facility by adding a focusing stage, diagnostic system and a co-evaporation chamber.



Thank you for attention

Backup

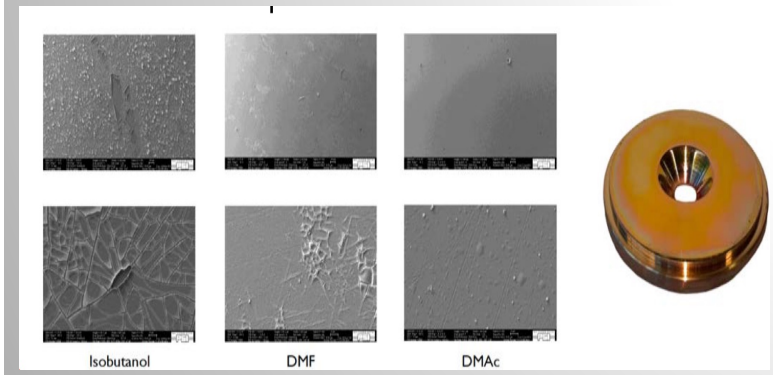
Sputter Target

In collaboration with PSI we tested three different solutions.

Molecular plating

Deposition of Ho complexes in an organic solvent at high voltages on a substrate (Cu-Au or others).

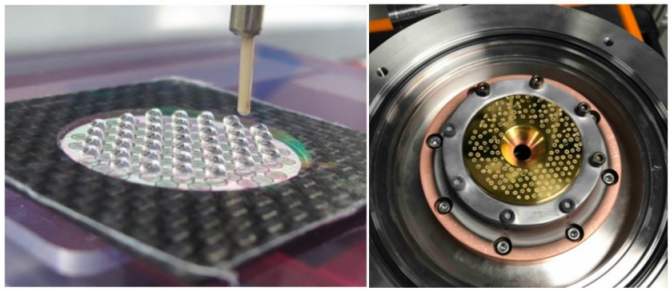
- High yield (> 90%);
- Very uniform layer;
- Different Ho compounds available;
- Uniformity function of compounds;
- Ho concentrated in surface;
- Possible decomposition of compounds.



Drop-on-demand inkjet printing

Deposition of Ho complexes in an organic solvent using a dedicated pipe (Cu-Au or others).

- Very high yield (> 99%);
- Good uniformity ;
- Different Ho compounds available;
- Ho concentrated in surface;
- Possible decomposition of compounds.



Molecular plating + coupled reduction

After a molecular plating the target is baked a high temperature in a reducing environment. The Ho is reduced and diffuses in the material with formation of intermetallic compound.

- High yield;
- Good uniformity;
- Concentration in the target;
- Intermetallic;
- Expensive materials for substrate (Pt, Pd);
- Thin substrate (~25 μm);
- Mechanical system to attach to the final support.