

Enclosing of Ho-163 in absorber of large TES arrays for the **HOLMES** experiment

Giovanni Gallucci
on behalf of the HOLMES
collaboration

Determination of the absolute electron (anti)-neutrino mass
ECT* 26-30 March 2018 Trento , Italy



Outline

163-Holmium production;
Chemical purification.

Reduction/distillation of Ho

- Evaporation chamber
- Sputter target production

Mass separation and implantation

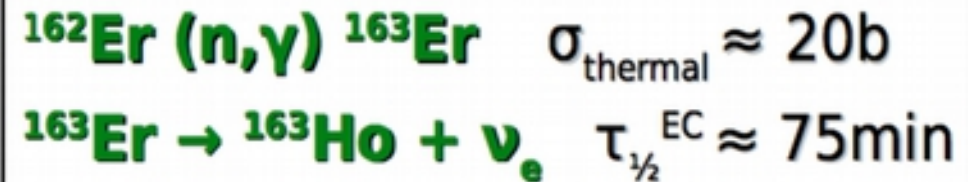
- Magnet
- Ion sputter source
- Target chamber

163-Holmium production

Er 162 0.139 e 19 $\sigma_{n,\gamma} < 0.011$	Er 163 75.0 m p ⁺ ... γ (1114...) 0	Er 164 1.601 e 13 $\sigma_{n,\gamma} < 0.0012$	Er 165 10.36 h e no γ	Er 166 33.503 e 3 + 14 $\sigma_{n,\gamma} < 7E-5$	Er 167 2.209 s 22.869 IT 208, e ⁺ $\sigma_{n,\gamma} < 3E-4$
Ho 161 6.76 s IT 211, e ⁺ γ 26, 103	Ho 162 2.48 h IT (10), e ⁺ γ 26, 94... e ⁺ γ 11, 1... γ 81, 1220 1319	Ho 163 15.0 m 1.00 s 4570 s IT 298 no γ	Ho 164 36.4 m 29 m IT (46), e ⁺ γ 37, 57... γ 91, 73	Ho 165 100 e 3.1 + 58 $\sigma_{n,\gamma} < 2E-5$	Ho 166 1132.6 s 26.824 h γ 184, 810 γ 12... e 3100 γ 81... e ⁺
Dy 160 2.329 e 60 $\sigma_{n,\gamma} < 0.0003$	Dy 161 16.600 e 600 $\sigma_{n,\gamma} < 1E-6$	Dy 162 25.475 e 170	Dy 163 24.896 e 120 $\sigma_{n,\gamma} < 2E-5$	Dy 164 26.260 e 1610 + 1040	Dy 165 1.257 m 2.334 h IT 108... γ 99... γ 515... e 2000 γ 95 (362...) e 3600

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

Thermal neutron flux at ILL: $1.3 \times 10^{15} \text{ n/cm}^2/\text{s}$



Contaminants:

- 1) Other elements (residual Er, Rare earth contaminants, decay products, etc...);
- 2) Holmium Isotopes, in particular $^{166\text{m}}\text{Ho}$, β^- - Q = 5.97 keV, $\tau_{1/2} = 1200\text{y}$

$$A(^{163}\text{Ho})/A(^{166\text{m}}\text{Ho}) = 100 \sim 1000.$$



- 1) Chemical purification at PSI;
- 2) Isotopic separation using an implanter.

Holmium chemical purification

The Er/Ho mixture is subjected to a radiochemical separation with ion-exchange resins in hot-cell at PSI.

Efficiency $\approx 79\%$ (preliminary) .

The Ho is in oxide form (Ho_2O_3) in acid solution ($\text{pH} < 4$) to avoid adhesion to the vial wall.



Three different batches are produced:

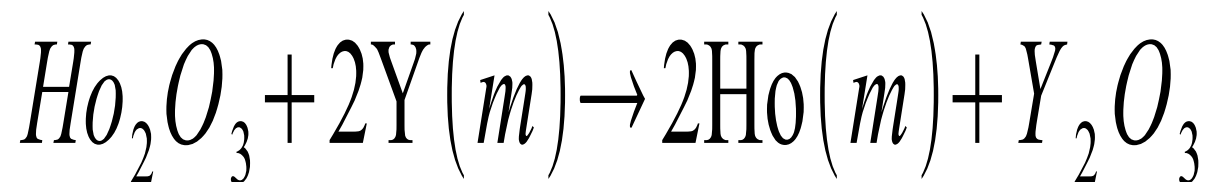
- 1) 25 mg irradiated for 55 days (2014), $A(^{163}\text{Ho}) \approx 5\text{MBq}$ ($A(^{166\text{m}}\text{Ho}) \approx 10\text{kBq}$)
- 2) 150 mg irradiated for 50 days (2015), $A(^{163}\text{Ho}) \approx 38\text{MBq}$ ($A(^{166\text{m}}\text{Ho}) \approx 37\text{kBq}$)
- 3) 540 mg irradiated 50 days (2017), $A(^{163}\text{Ho}) \approx 130\text{MBq}$ ($A(^{166\text{m}}\text{Ho}) \approx 180\text{kBq}$) (not yet purified at PSI)

Reduction/Distillation Process (1)

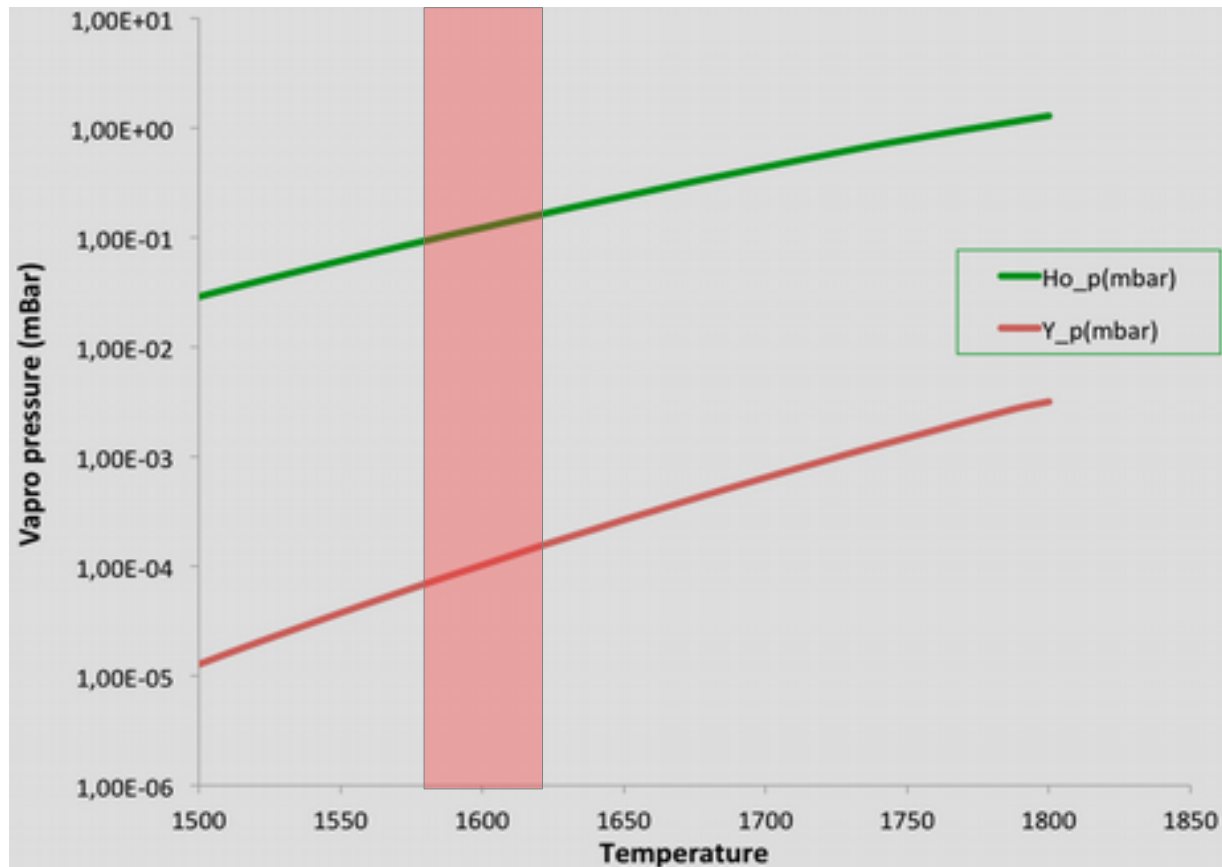
Only holmium in the metallic chemical form must be introduced:

- Increase ionization efficiency in metallic form;
- Metallic form for sputter ion target .

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



Reduction/Distillation process (2)

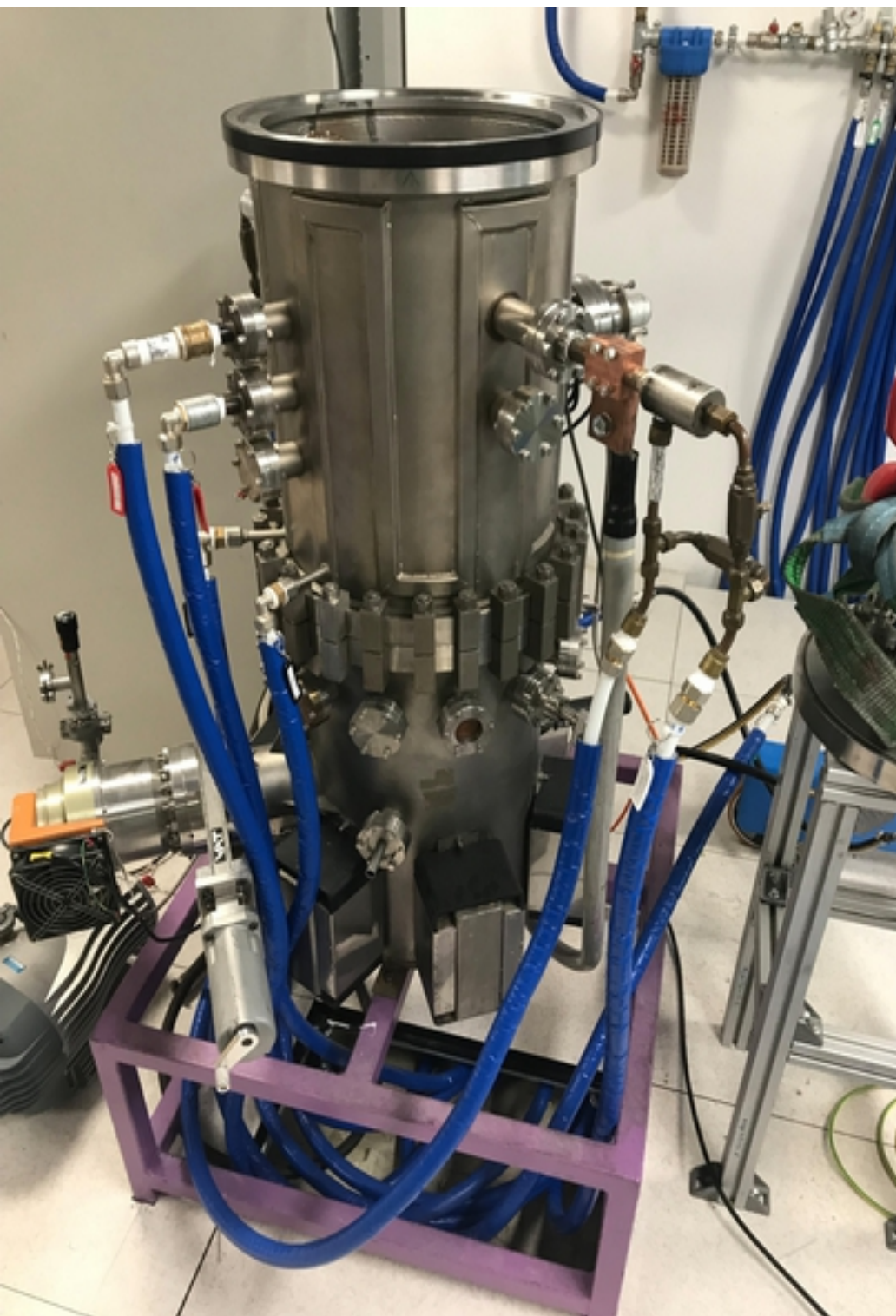


- Holmium Oxide powder is mixed with metallic Yttrium (lump).
- The mixture is heat up to about 1600 °C (Y melting point).
- When the Y wets the oxide the reaction starts.
- Metallic Ho has a high vapour pressure at this temperature and evaporated.
- Ho condensates on a cold substrate.

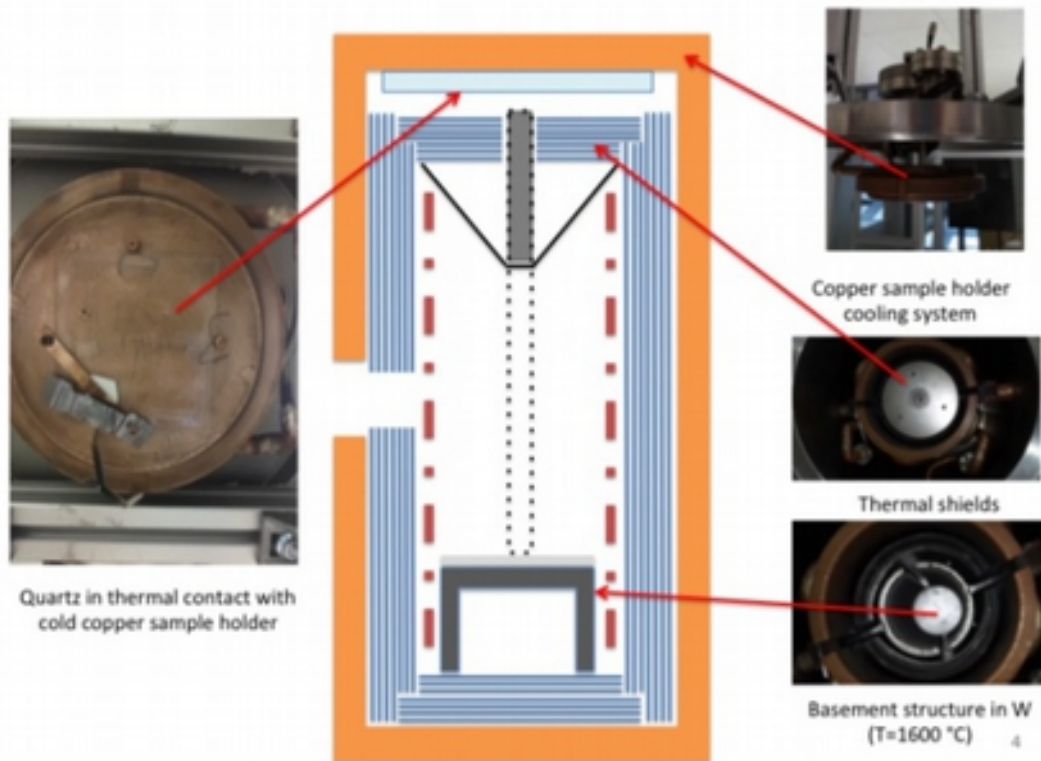
Oxide (Y and residual Ho) have a negligible vapour pressure.
The Y vapor pressure is three order of magnitude lower than Ho one at working point.

We use a dedicated evaporation chamber to the whole process.

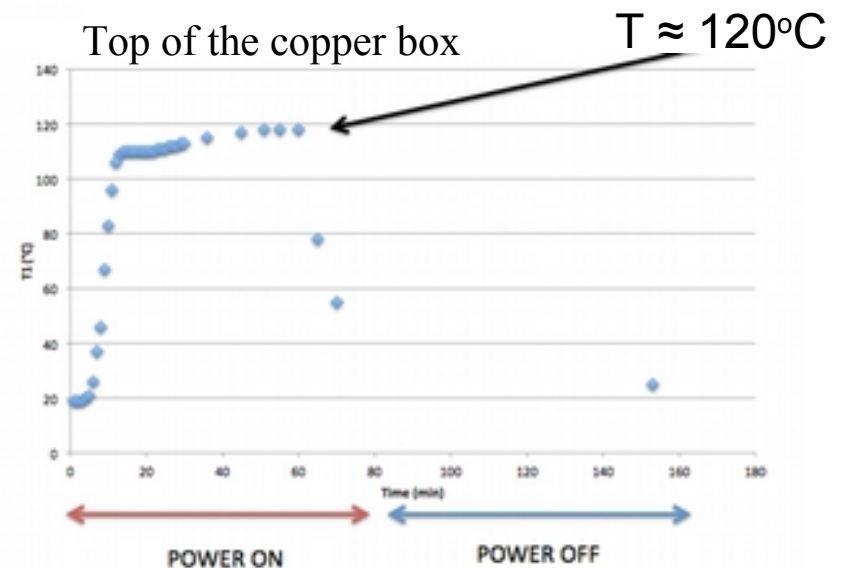
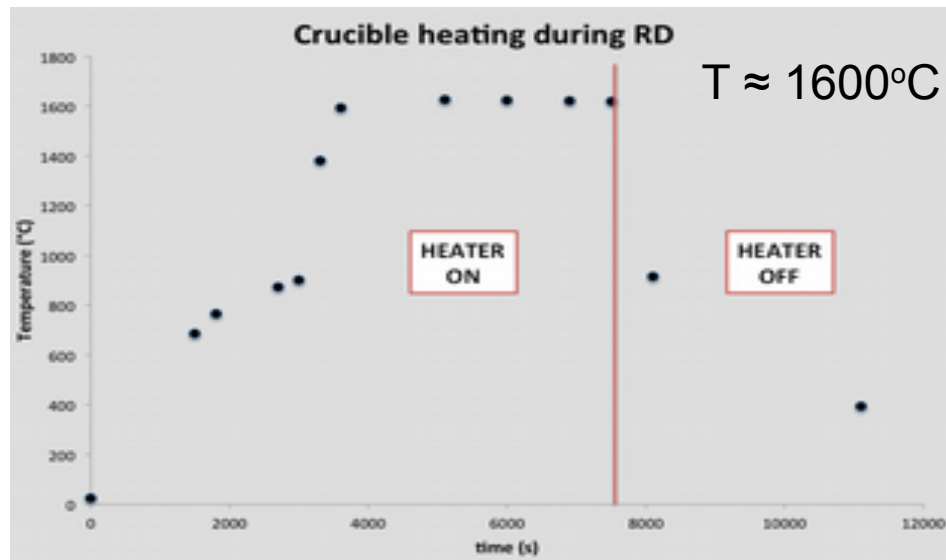
Evaporation chamber



Evaporation Chamber (1)

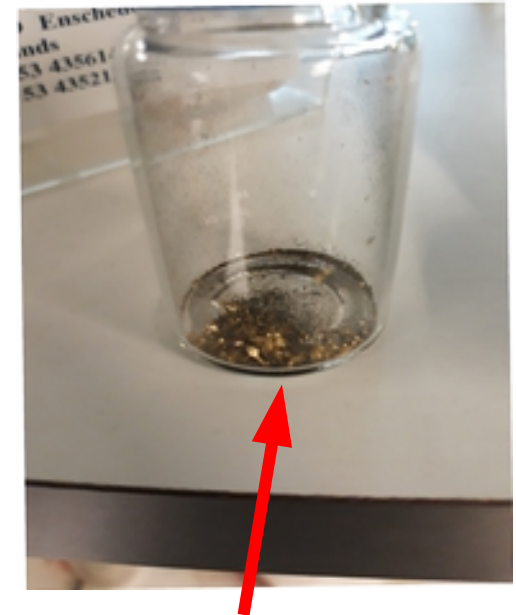
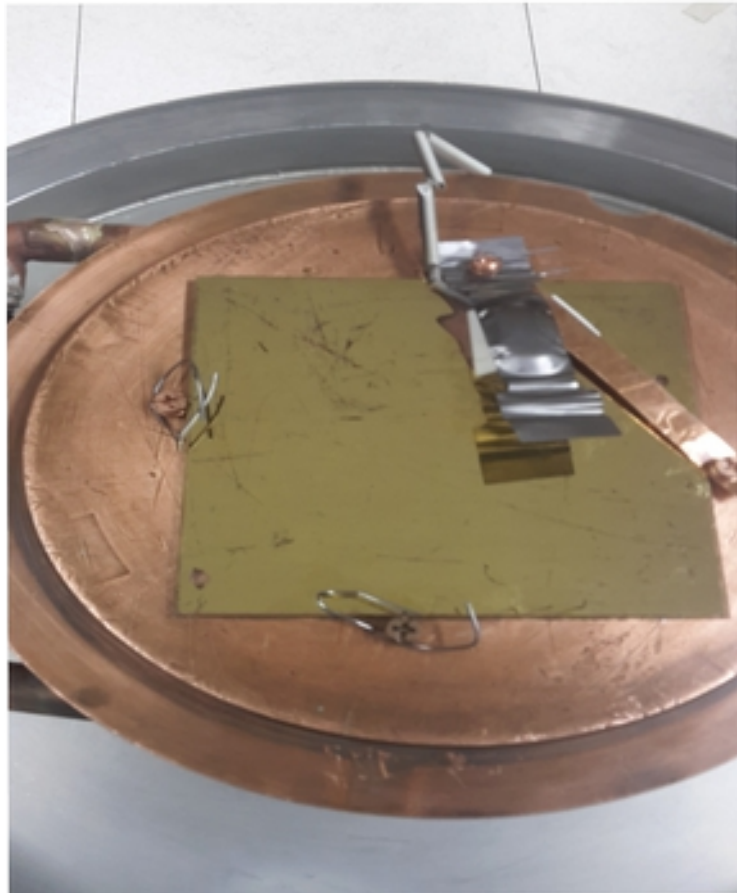


- The hot zone is thermally isolated by nine tungsten layer.
- The external copper box is water cooled.
- The upper part of the shield is holed allowing the evaporated Ho flows from the crucible to a substrate fixed on the top of the copper box.
- The whole system is set in a vacuum chamber which could reach pressures as low as 10^{-8} mBar.



Evaporation chamber (2)

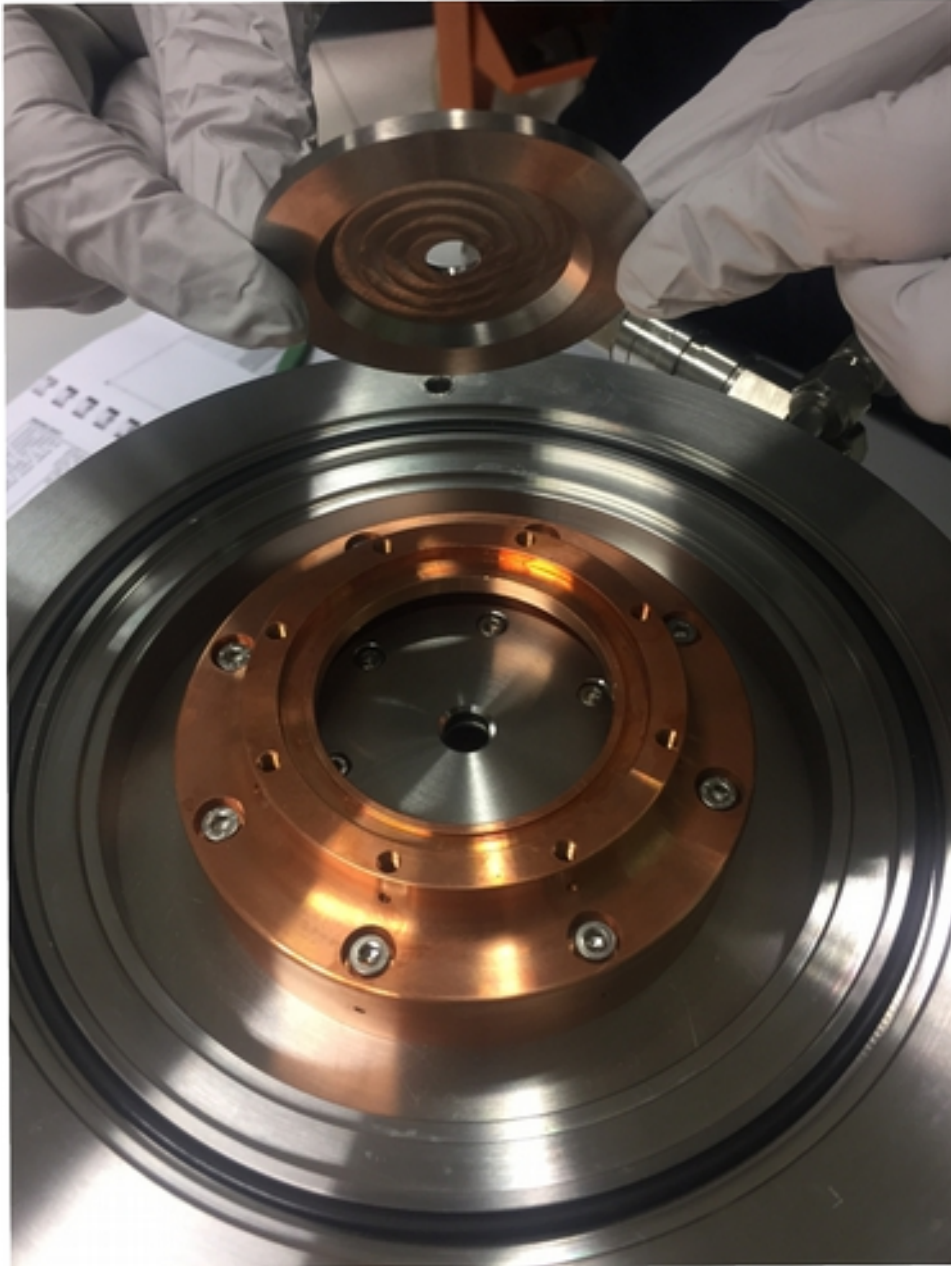
- The substrate used for the collection of the distilled Ho is made of quartz because of its high resistivity to thermal deformation.
- A thin layer of a low degassing thermal conducting paste is used to improve the thermal conductance between the quartz substrate and the cooled copper cap.
- A gold 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.



Ho + Au powder

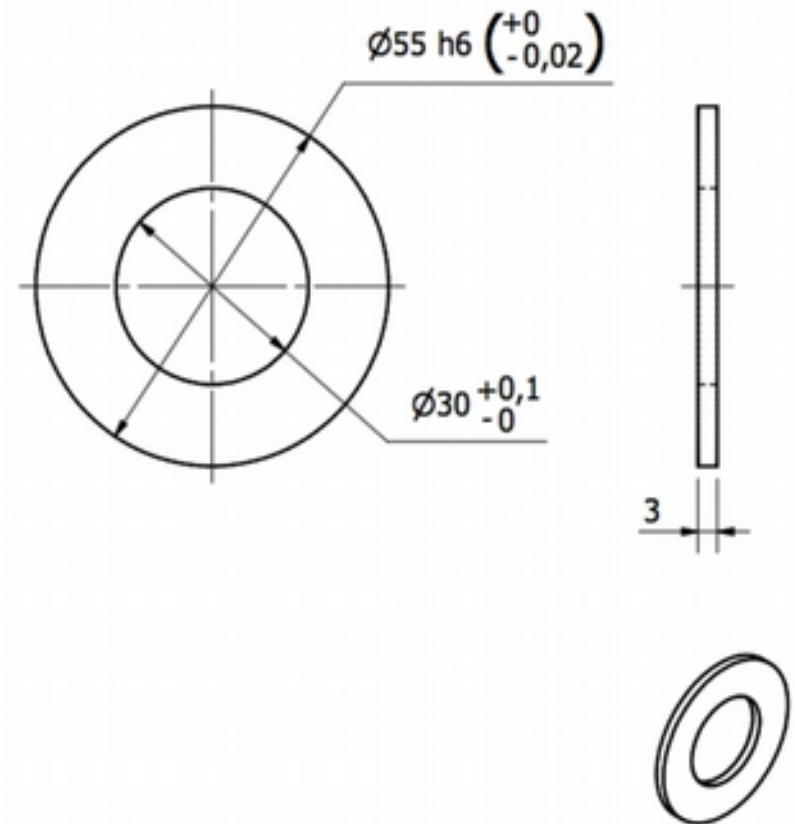
Efficiency \approx 70%.

Sputter Target (1)



The sputter ion source of the implanter needs a metallic cathode.

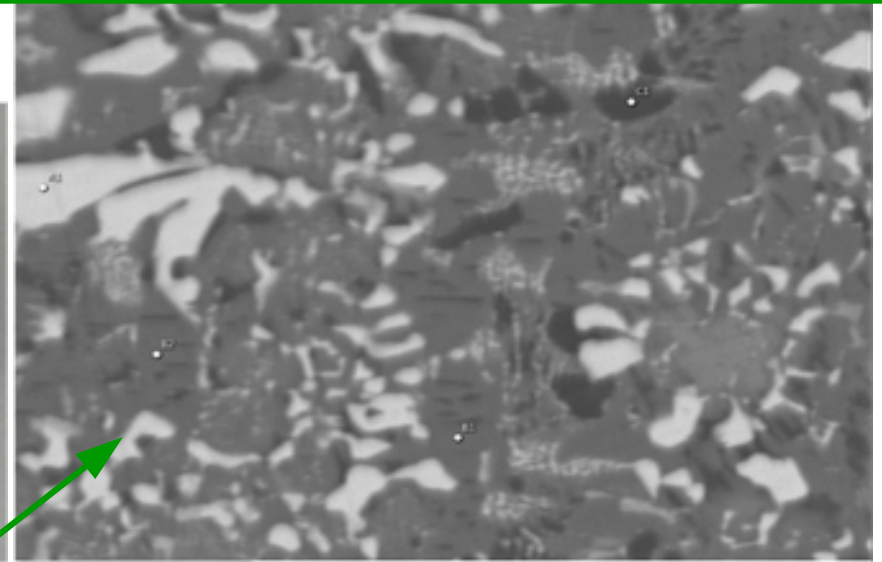
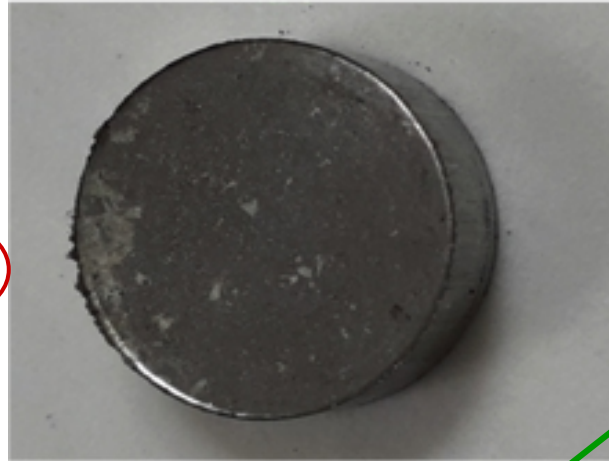
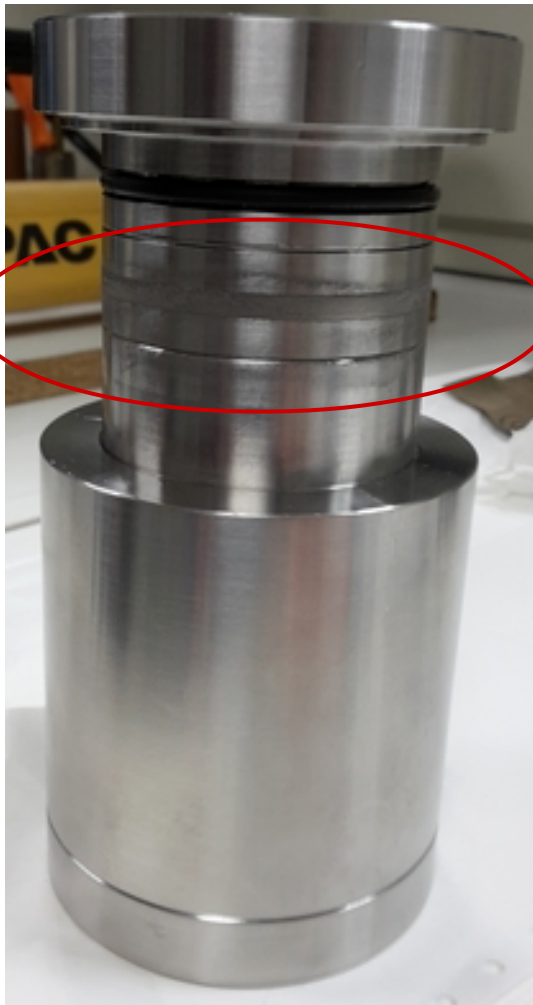
We need turn the holmium powder into a metallic disk.



Sputter Target (2)

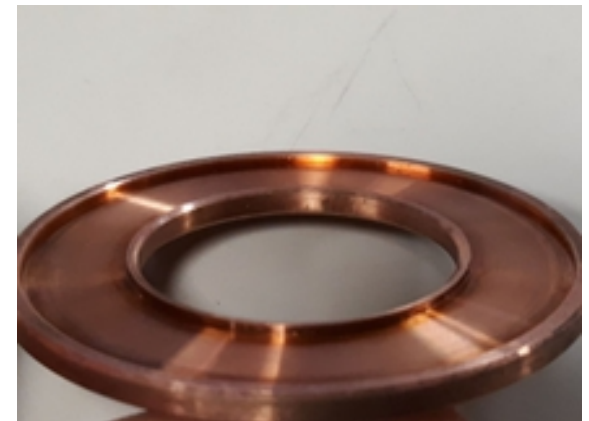
In collaboration with prof. Manfrinetti (from Chemistry Department of Genova University) we decide to realize a sintered sputter target.

- Ho(5%) is included in a metallic mixture of Ti(36%), Ni(41%), Sn(18%) fine grained powder ($< 40 \mu\text{m}$).;
- Pressed at 350 bar/cm²;
- Heated at 850 °C pressure 10^{-2} mbar for 4 days to improve the mechanical proprieties of the sintered.

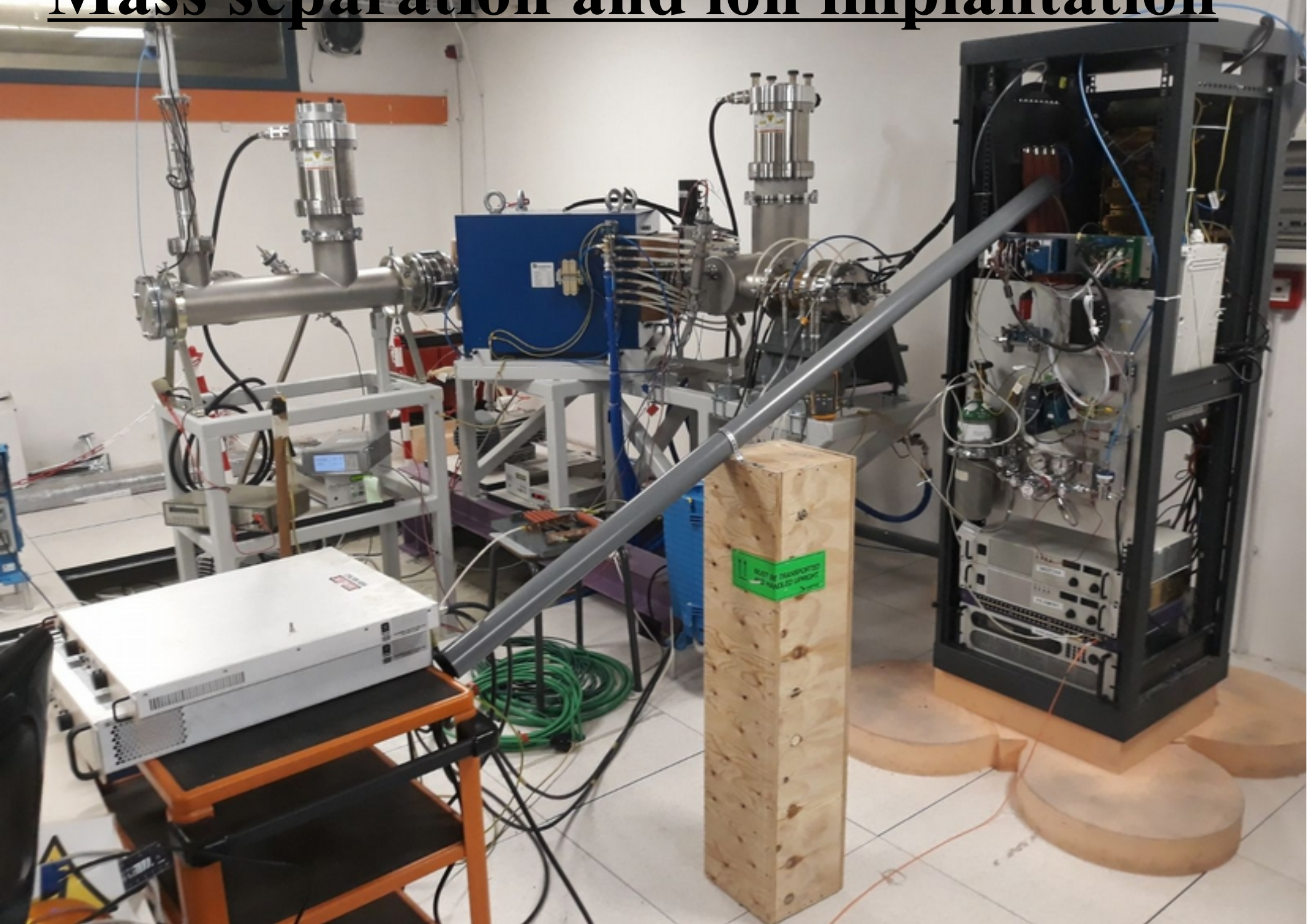


The crystallographic measurements and SEM-EDS analysis show a $\text{Ti}_2\text{Ni}_2\text{Sn}$ matrix with homogeneously distributed islands of HoNiSn .

Efficiency $> 95\%$



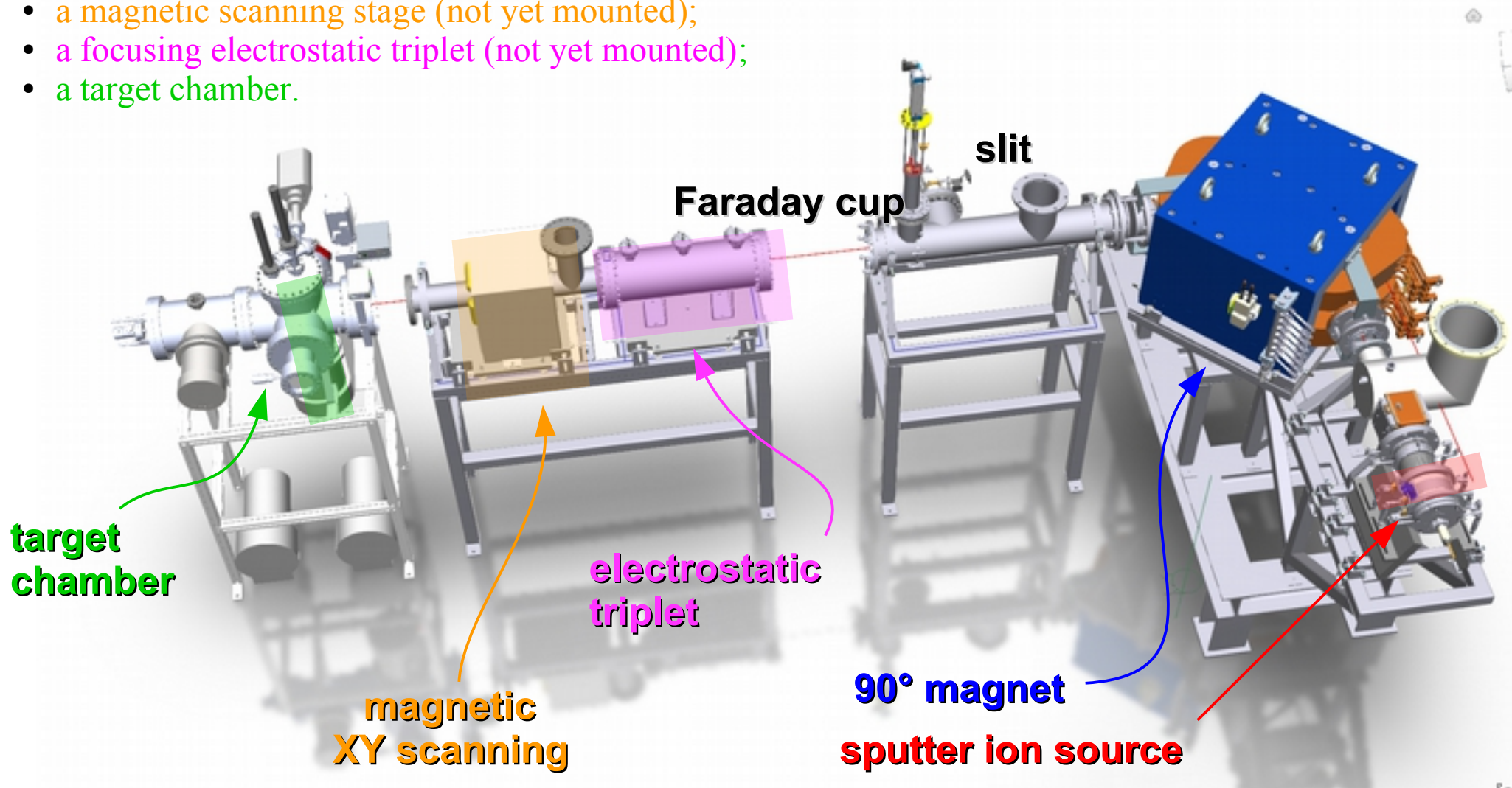
Mass separation and ion implantation



HOLMES mass separation and ion implantation

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 six main components:

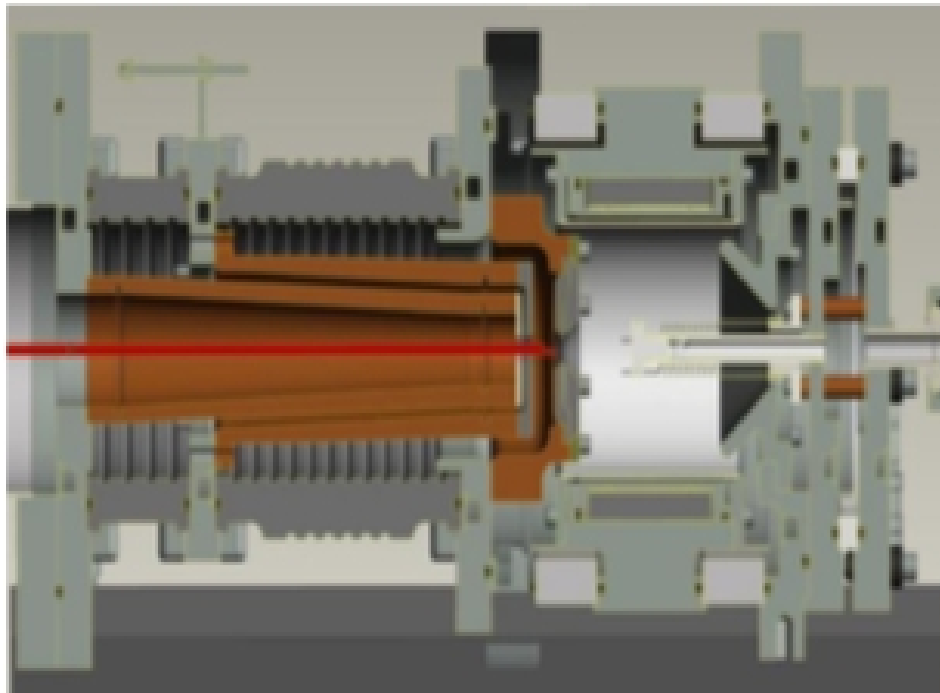
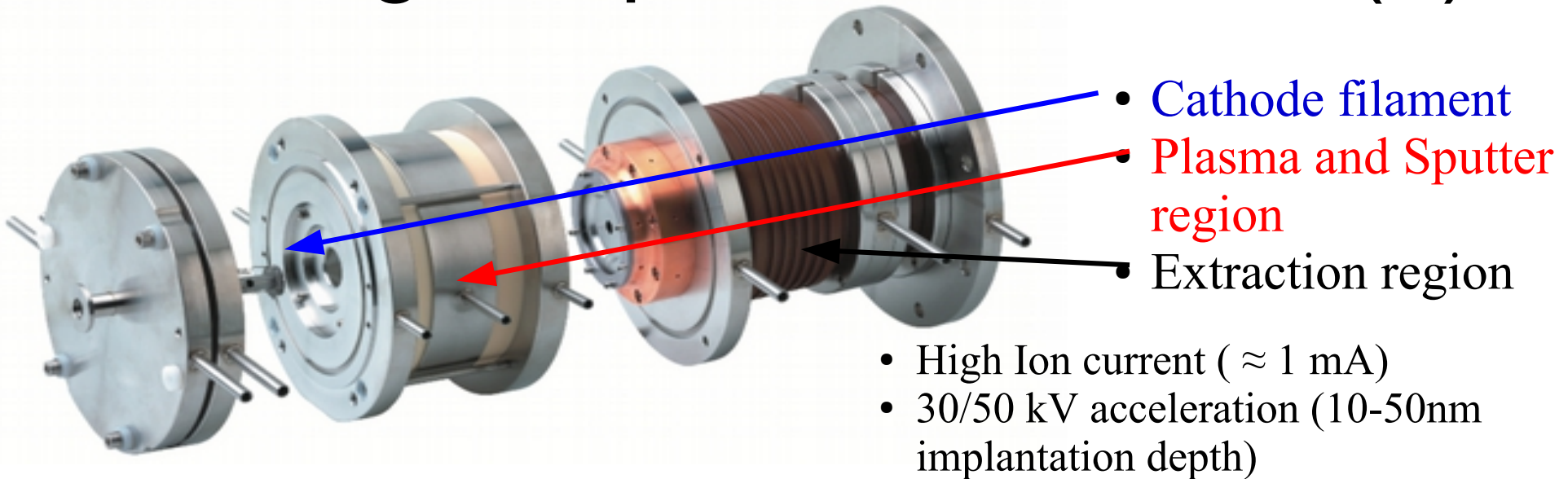
- an argon sputter ion source;
- an acceleration section to reach the beam energy of 50 KeV;
- a magnetic/electrostatic mass analyzer with magnetic field until 1.1 Tesla;
- a magnetic scanning stage (not yet mounted);
- a focusing electrostatic triplet (not yet mounted);
- a target chamber.





Sputter Ion Source

Argon Sputter IonSource (1)

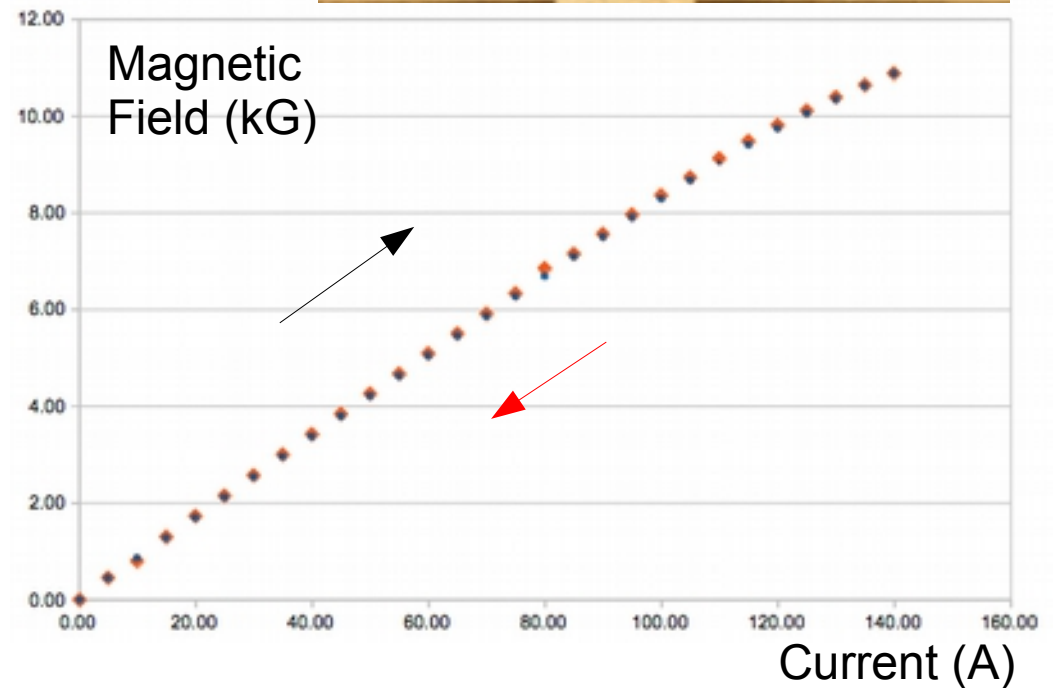
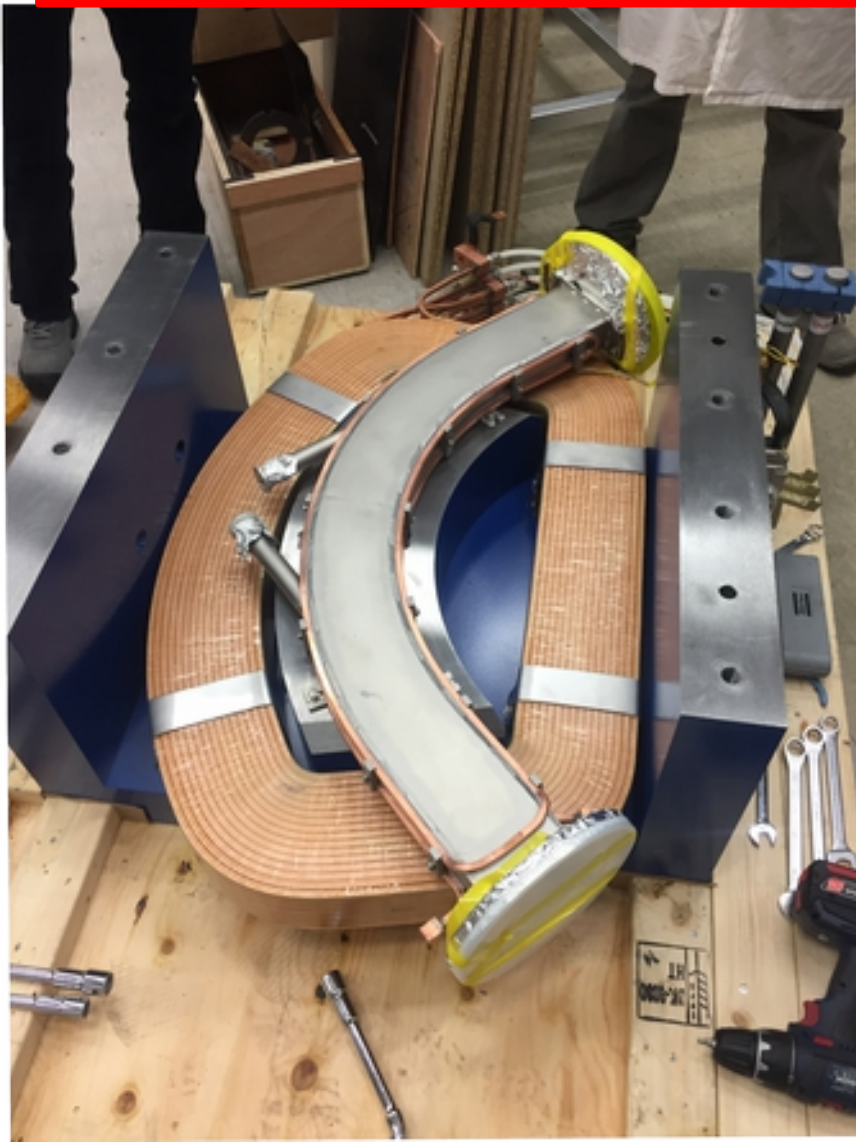


Efficiency $\approx 5\%$
reasonable assumptions
on the (unknown)
process efficiency

The Magnet

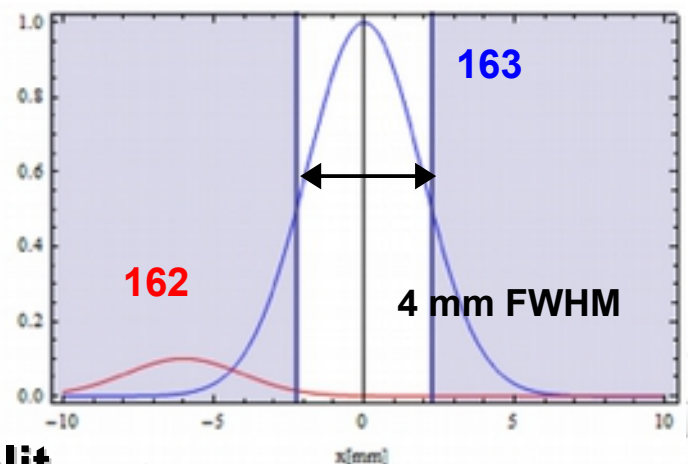
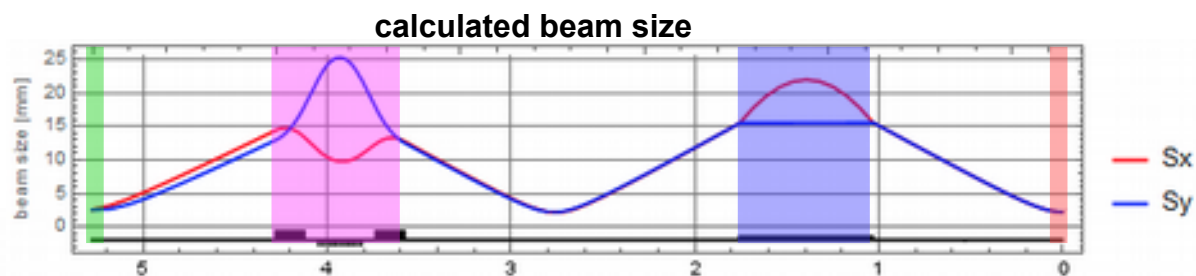
The magnet arrived in Genova end of the 2016.
Installation ended in April 2017.

- Induction until 1.1 Tesla
- Bending radius 46 cm
- Deflection Angle 90 degree

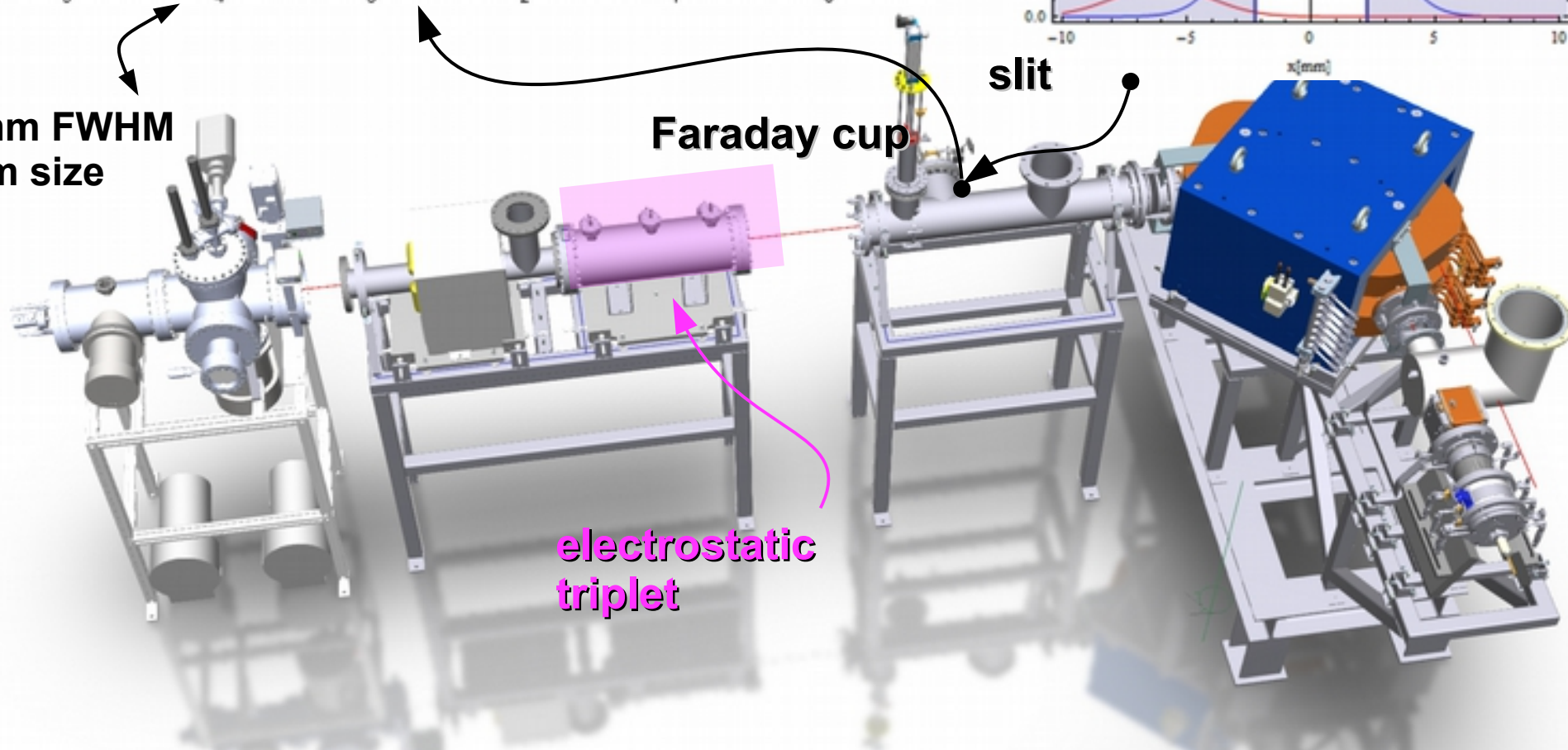


Beam spot and electrostatic triplet

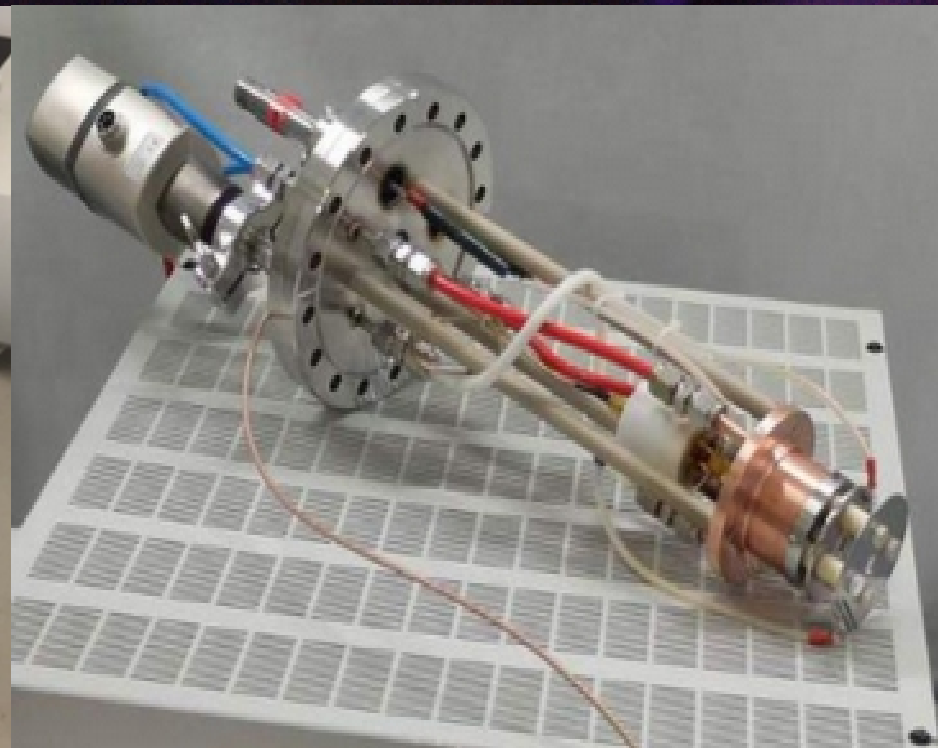
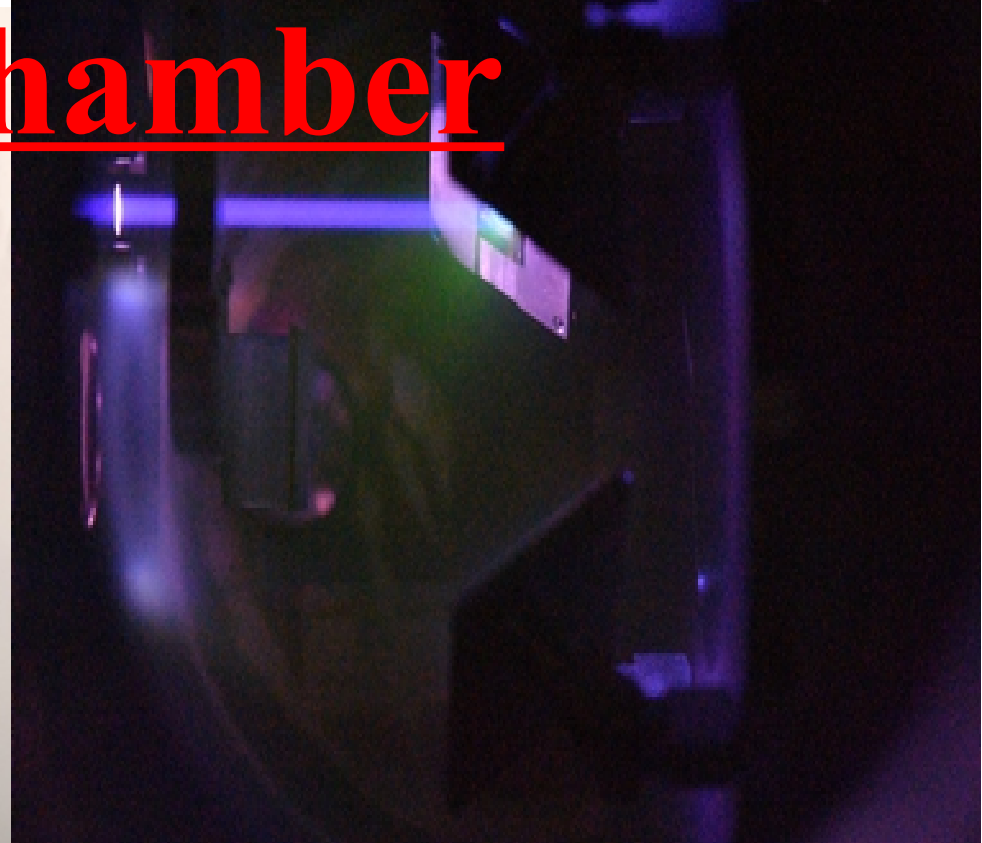
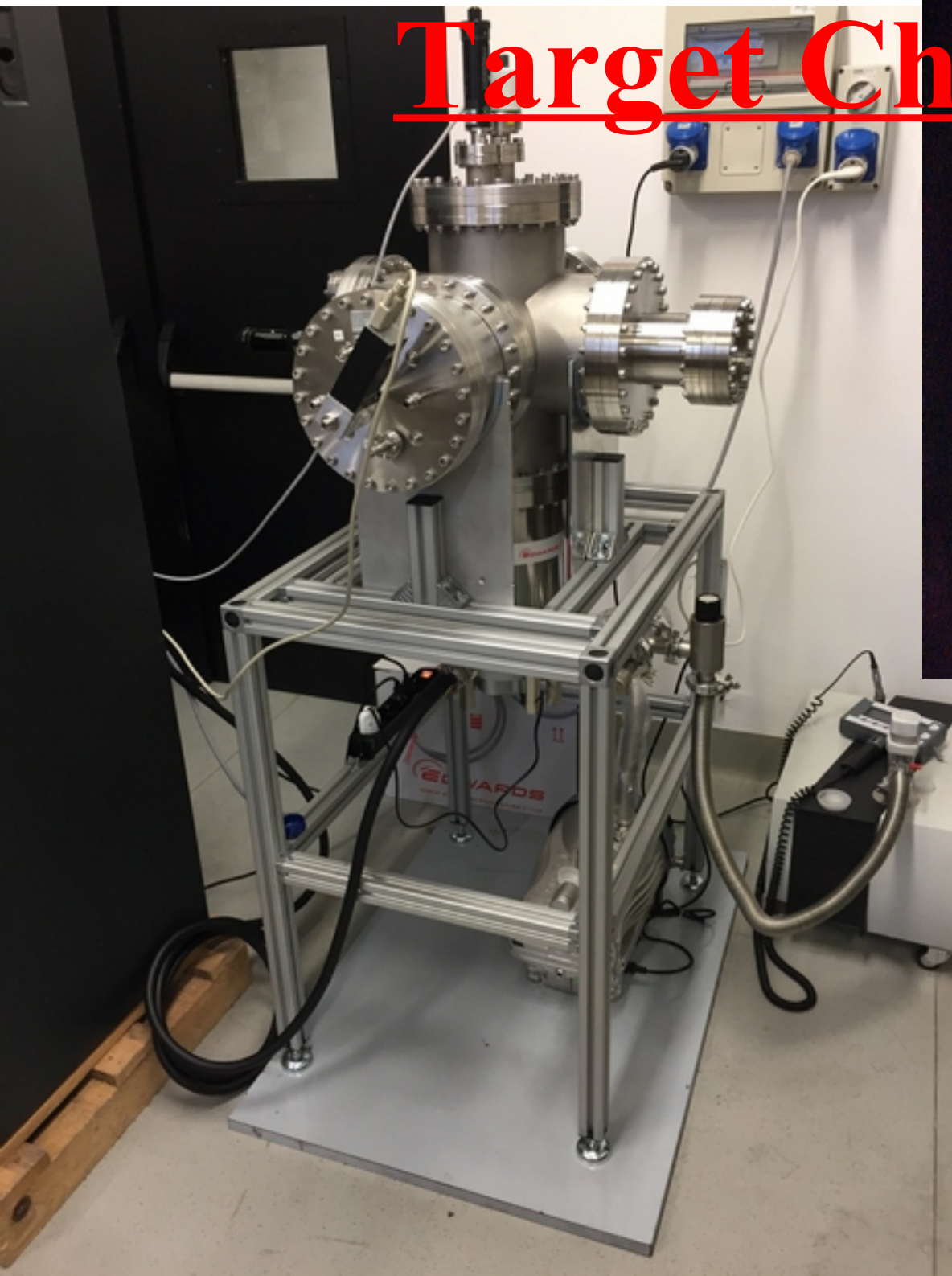
- $^{163}\text{Ho}/^{166\text{m}}\text{Ho}$ separation better than 10^5



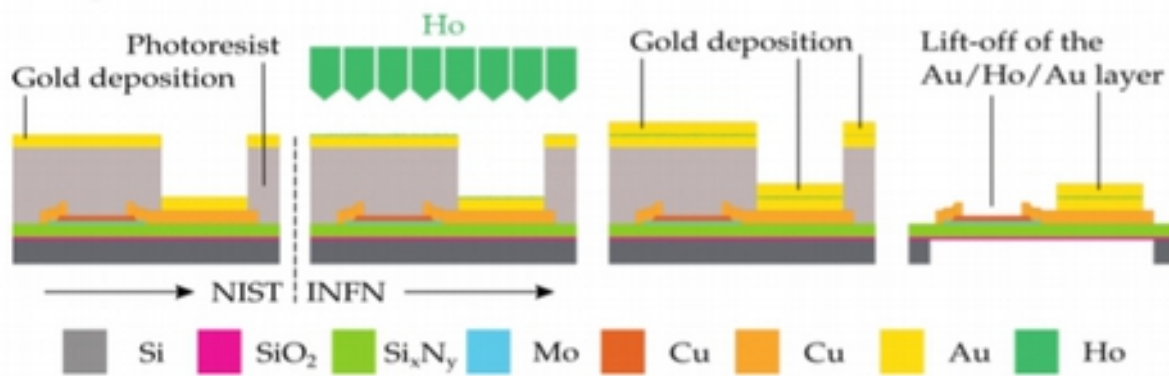
≈4 mm FWHM
beam size



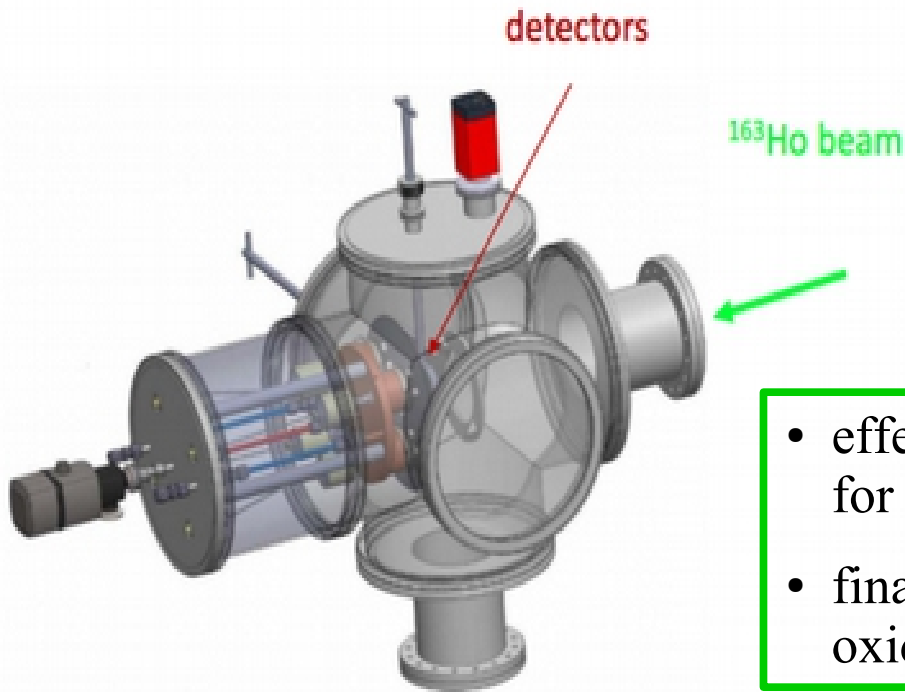
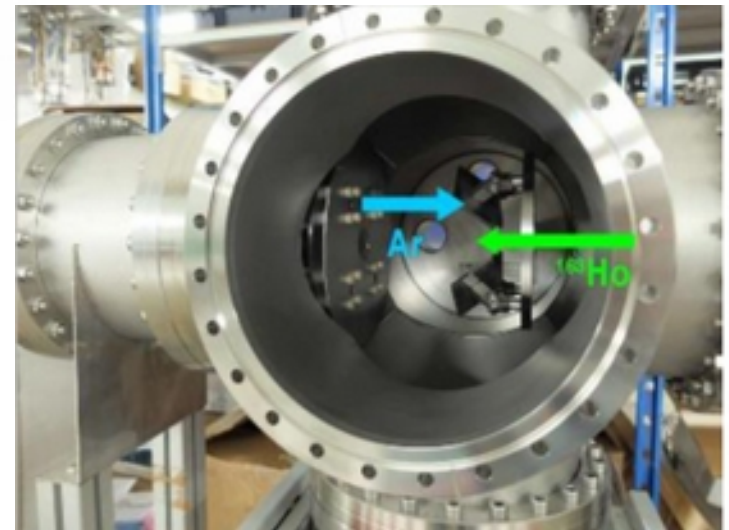
Target Chamber



Target chamber



Activity for single pixel 300 Bq but ^{163}Ho concentration in absorbers saturate because ^{163}Ho sputters off Au from absorber.



- effect compensated by Au co-evaporation (also for heat capacity reasons)
- final 1 mm Au layer deposited in situ to avoid oxidation

Efficiency $\approx 3.5\%$

Summary

Two batches of ^{163}Ho (175 MBq) are ready at PSI.
They will be moved to Genova in the months.

The procedure to distillate holmium is tested. Some refinements are needed.

A metallic sintered sputter target has been obtained using Ti, Ni, Sn and Ho. A $\text{Ti}_2\text{Ni}_2\text{Sn}$ metallic matrix with HoNiSn islands uniformly distributed.

The implanter arrived at Genova at the beginning of 2017 installation and construction are mainly finished.

Every part has been individually tested. Integration and first tests in progress.

Target chamber test in progress at Milano.

Integration at Genova (June/July 2018).