

Status of the HOLMES neutrino mass experiment

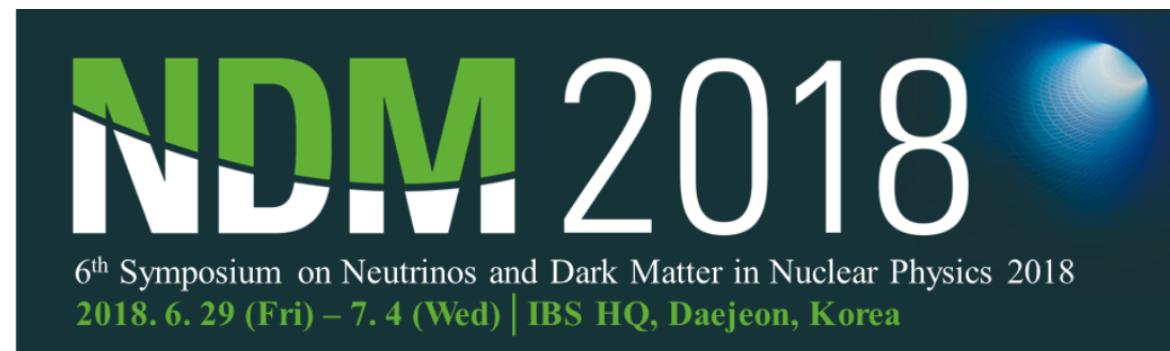
Angelo Nucciotti

on behalf of the HOLMES collaboration

Università di Milano-Bicocca e INFN - Sezione di Milano-Bicocca



Istituto Nazionale di Fisica Nucleare





- ^{163}Ho decay calorimetry and neutrino mass measurement
- HOLMES experiment goal and design
- HOLMES tasks status
 - isotope production and chemical purification
 - isotope mass separation and embedding
 - single detector design
 - detector read-out and DAQ
 - detector array fabrication
- conclusions and perspectives

Electron capture calorimetric experiments

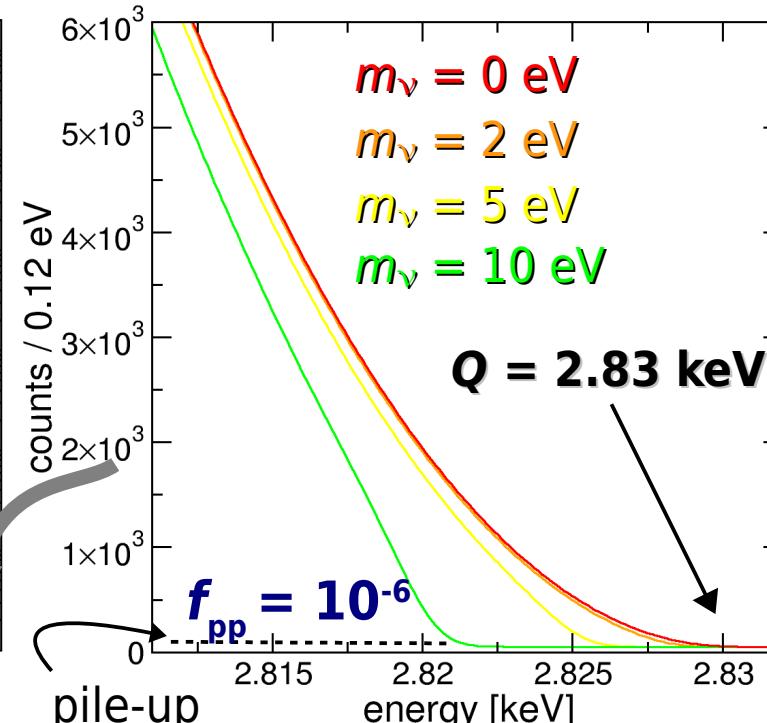
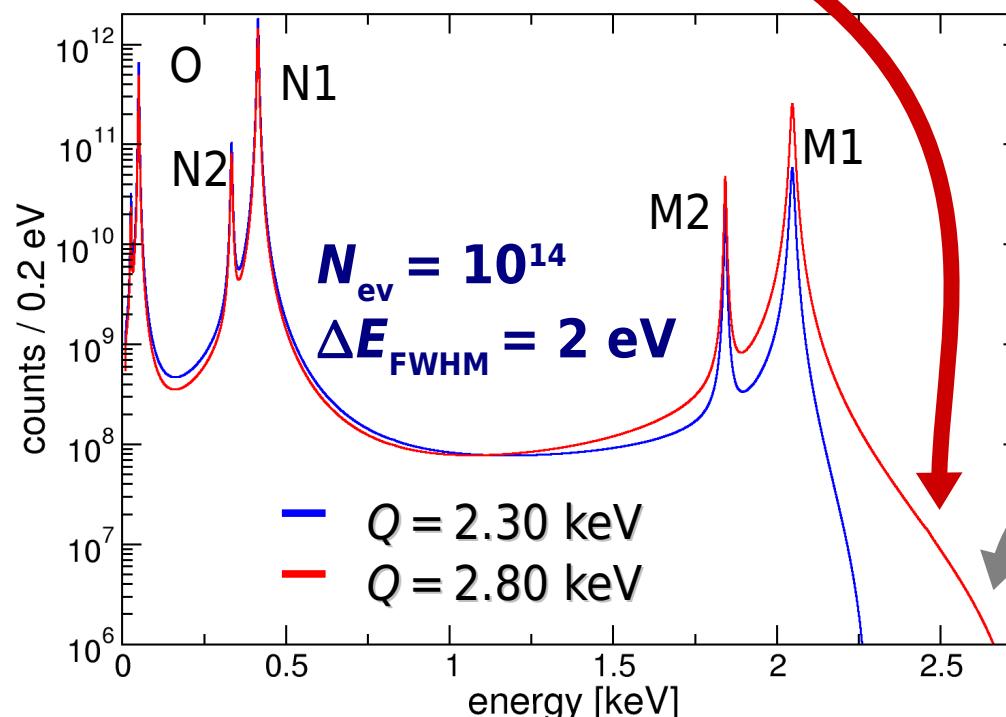


electron capture from shell $\geq M1$

A. De Rújula and M. Lusignoli, Phys. Lett. B 118 (1982) 429

- calorimetric measurement of Dy atomic de-excitations (mostly non-radiative)
- $Q = 2.83 \text{ keV}$ (determined with Penning trap in 2015)
 - end-point rate and ν mass sensitivity depend on $Q - E_{M1}$
- $\tau_{1/2} \approx 4570 \text{ years} \rightarrow 2 \times 10^{11} \text{ }^{163}\text{Ho} \text{ nuclei} \leftrightarrow 1 \text{ Bq}$

$$N(E_c) = \frac{G_\beta^2}{4\pi^2} (Q - E_c) \sqrt{(Q - E_c)^2 - m_\nu^2} \times \sum_i n_i C_i \beta_i^2 B_i \frac{\Gamma_i}{2\pi} \frac{1}{(E_c - E_i)^2 + \Gamma_i^2 / 4}$$

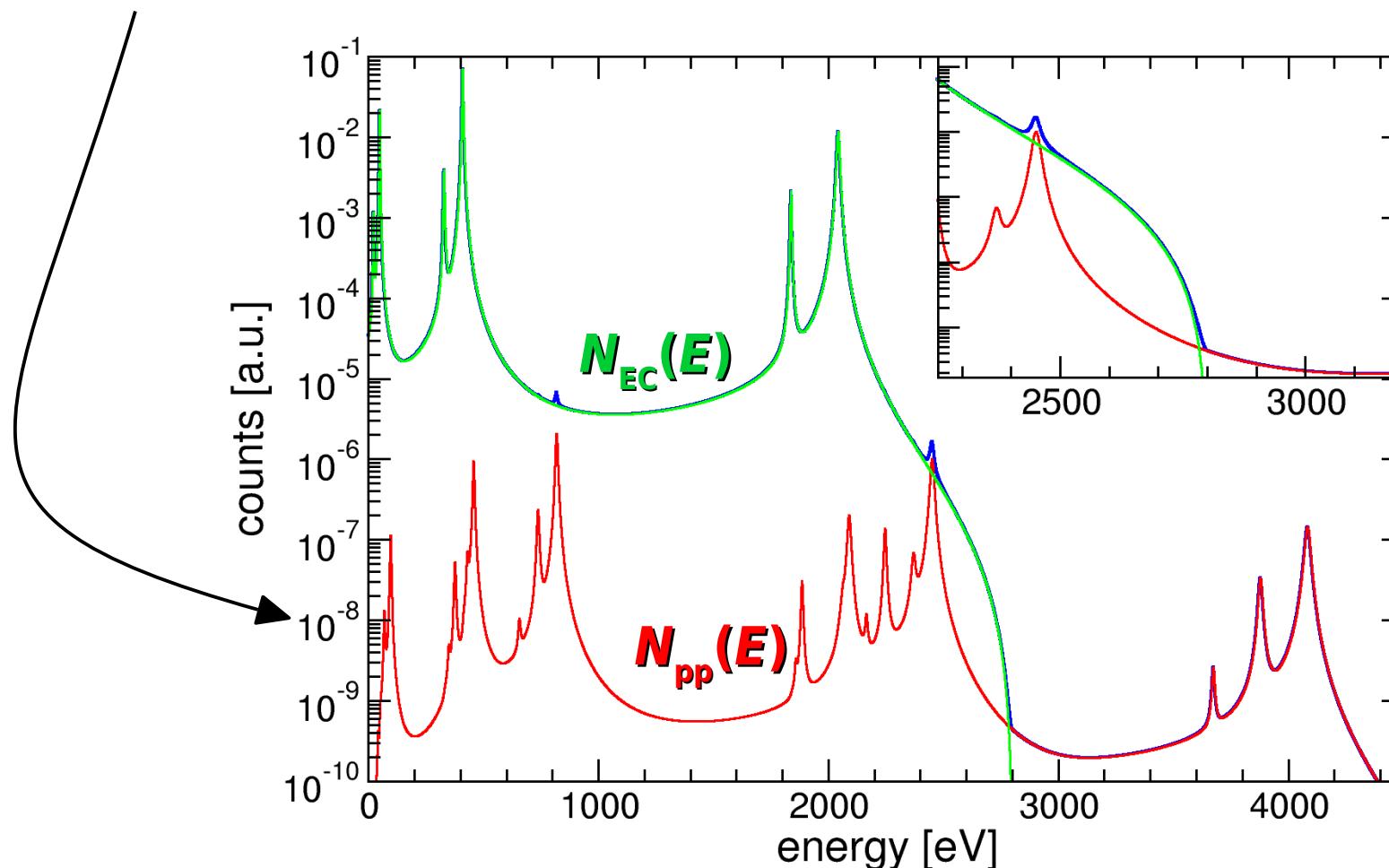


Electron capture calorimetric experiments



- calorimetric measurement \leftrightarrow **detector speed is critical**
- accidental coincidences \rightarrow complex pile-up spectrum
 - $N_{pp}(E) = f_{pp} N_{EC}(E) \otimes N_{EC}(E)$ with $f_{pp} \approx A_{EC} \tau_R$

A_{EC} EC activity per detector
 τ_R time resolution (\approx rise time)



$$Q = 2800 \text{ eV}$$
$$f_{pp} = 10^{-4}$$

$N_{EC}(E)$ without higher order processes (shake up / shake off)

Montecarlo simulations: ^{163}Ho sensitivity potential

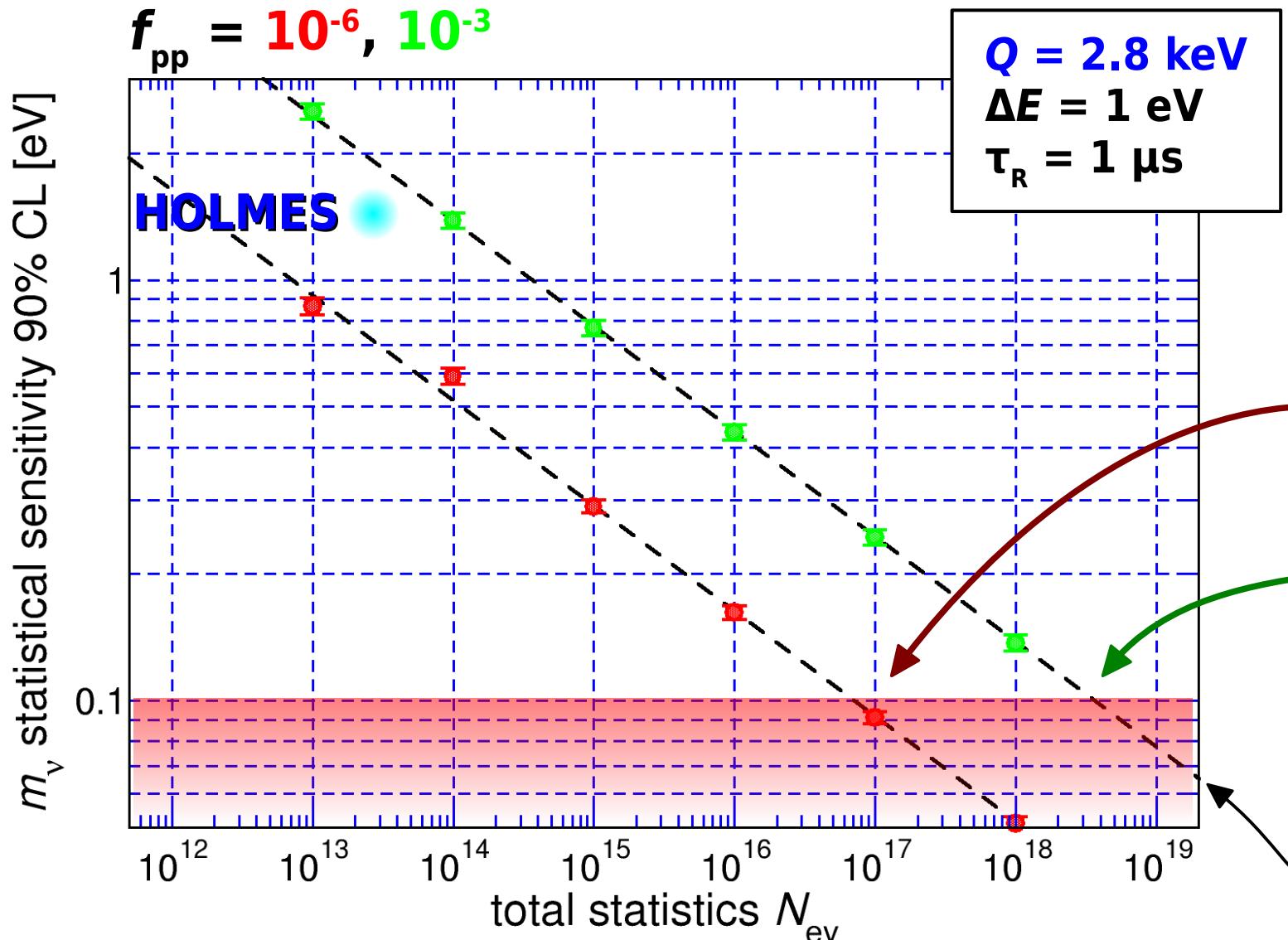


m_ν sensitivity mostly depends on: ▲ total statistics $N_{\text{ev}} = A_{\text{EC}} N_{\text{det}} t_M$

▼ pile fraction $f_{\text{pp}} = A_{\text{EC}} \tau_R$

t_M measuring time
 N_{det} number of detectors

$$f_{\text{pp}} = 10^{-6}, 10^{-3}$$

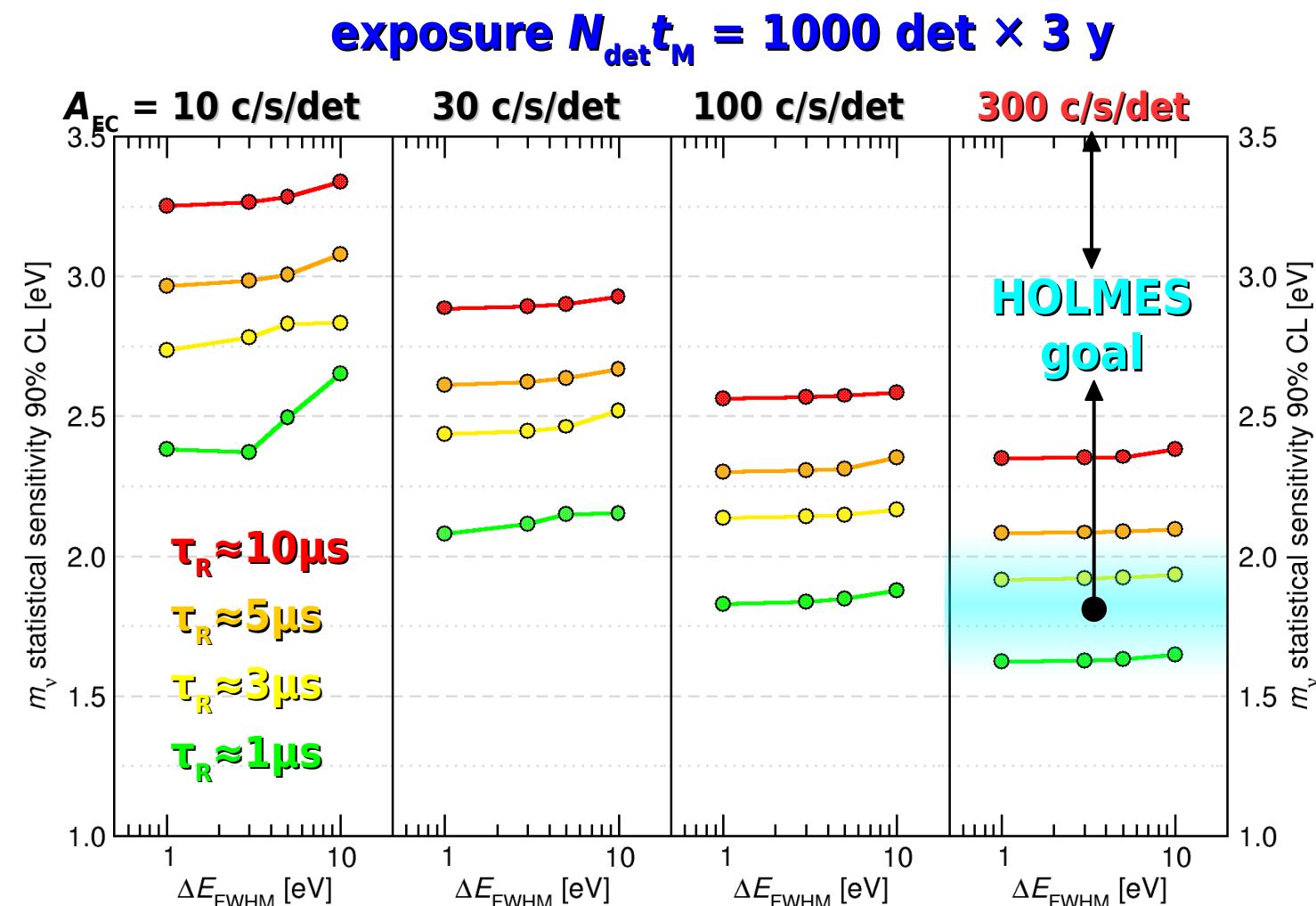


goal

- direct neutrino mass measurement: m_ν statistical sensitivity around 1 eV
- prove potential and scalability:
 - ▶ assess EC spectral shape
 - ▶ assess systematic errors

baseline

- low T microcalorimeters with implanted ^{163}Ho
 - ▶ 6.5×10^{13} atom/det $\rightarrow A_{\text{EC}} = 300 \text{ c/s/det}$
 - ▶ $\Delta E \approx 1 \text{ eV}$ and $\tau_R \approx 1 \mu\text{s}$
- 1000 channel array
 - ▶ $6.5 \times 10^{16} {}^{163}\text{Ho}$ nuclei $\rightarrow \approx 18 \mu\text{g}$
 - ▶ 3×10^{13} events in 3 years



5 years project started on February 1st 2014 (now extended by 1 year)

HOLMES collaboration



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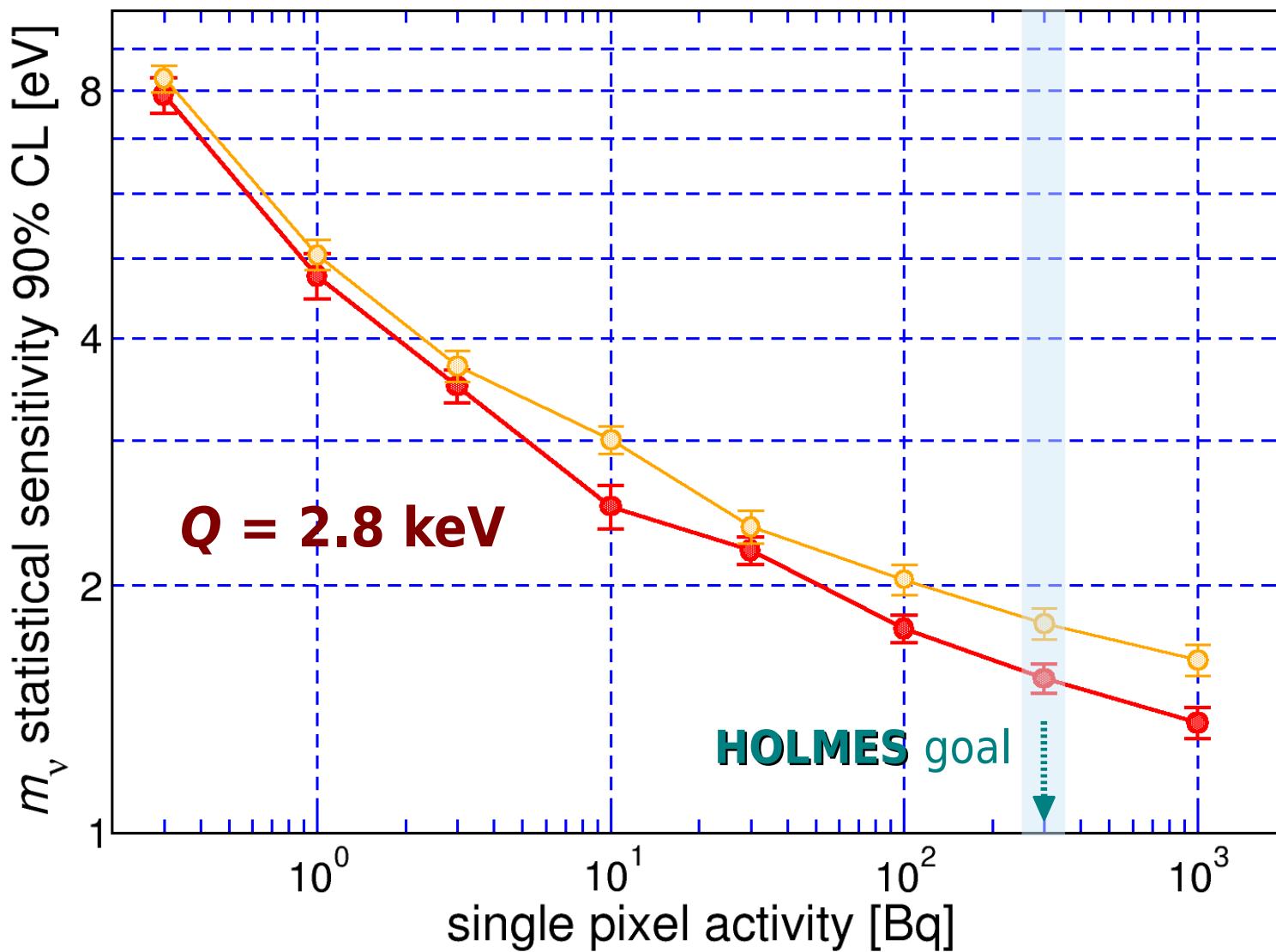
U.Koester

<http://artico.mib.infn.it/holmes>

Statistical sensitivity and single pixel activity



exposure $N_{\text{det}} t_M = 1000 \text{ det} \times 3 \text{ y}$



$\Delta E = 1 \text{ eV}$
 $\tau_R = 1 \mu\text{s}$

$\Delta E = 3 \text{ eV}$
 $\tau_R = 3 \mu\text{s}$

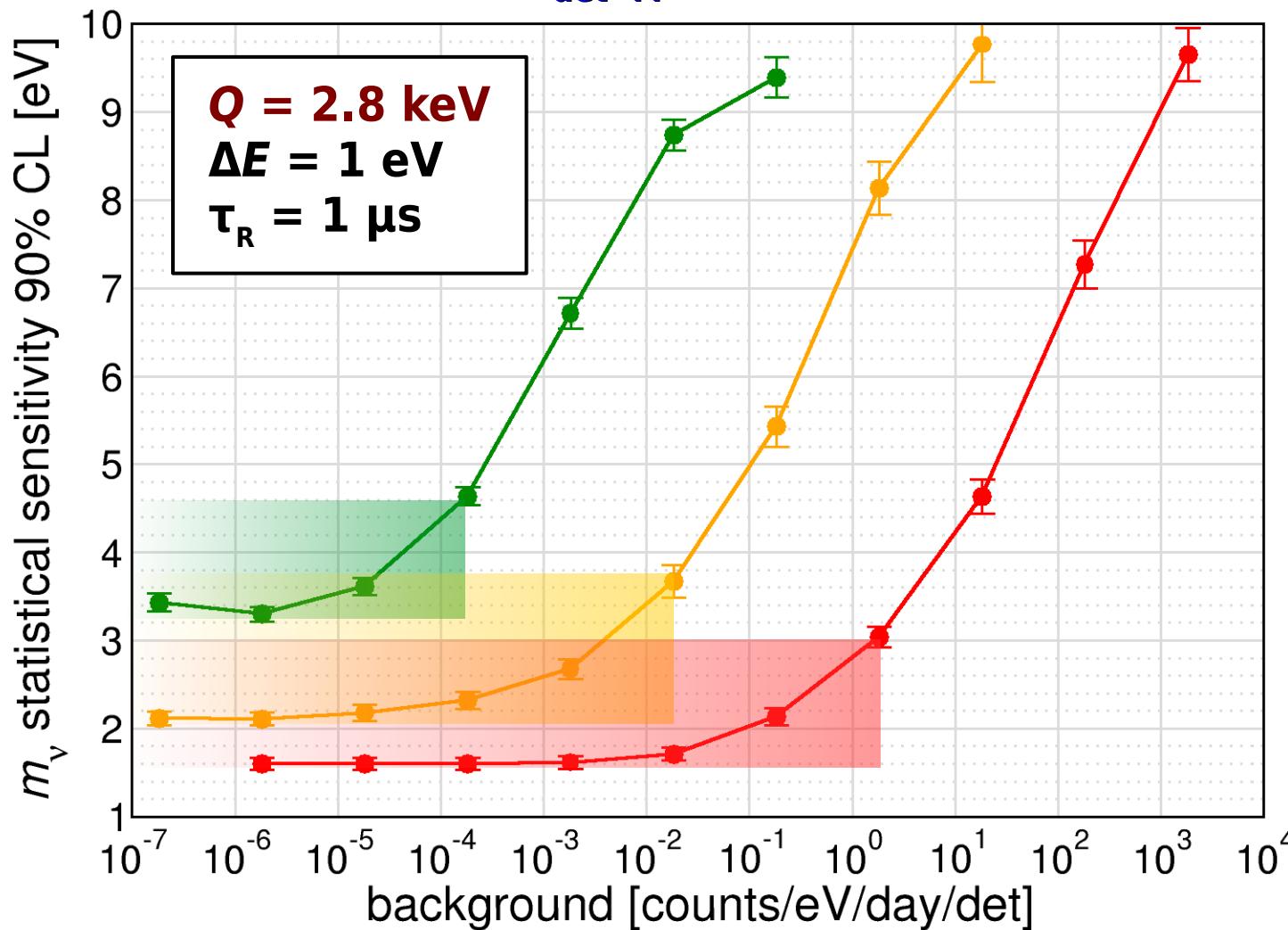
high activity → robustness against (flat) background

$A_{\text{EC}} = 300 \text{ Bq} \rightarrow bkg < \approx 0.1 \text{ counts/eV/day/det}$

Effect of background on sensitivity



exposure $N_{\text{det}} t_M = 1000 \text{ det} \times 3 \text{ y}$



$$A_{\text{EC}} = 3 \text{ Bq/det}$$
$$f_{\text{pp}} = 3 \times 10^{-6}$$

$$A_{\text{EC}} = 30 \text{ Bq/det}$$
$$f_{\text{pp}} = 3 \times 10^{-5}$$

$$A_{\text{EC}} = 300 \text{ Bq/det}$$
$$f_{\text{pp}} = 3 \times 10^{-4}$$

pile-up background \approx average rate $\langle r_{\text{pp}} \rangle$

$$\langle r_{\text{pp}} \rangle = A_{\text{EC}} f_{\text{pp}} / 2Q = 1.5 \times 10^{-4} \quad 1.5 \times 10^{-2} \quad 1.5 \text{ c/eV/day/det}$$

Low energy background



- environmental γ radiation
- γ , X and β from close surroundings
- cosmic rays
 - ▷ GEANT4 $\rightarrow bkg \approx 5 \times 10^{-5} \text{ c/eV/day/det (0 - 4 keV)}$

HOLMES target

for $A_{EC} = 300 \text{ Bq}$

$$bkg < \approx 0.1 \text{ c/eV/day/det}$$

Au pixel $200 \times 200 \times 2 \mu\text{m}^3$

MIBETA experiment: $300 \times 300 \times 150 \mu\text{m}^3$ AgReO₄ crystals at sea level
 $bkg(2-5\text{keV}) \approx 1.5 \times 10^{-4} \text{ c/eV/day/det}$

• internal radionuclides

- ▷ ^{166m}Ho (β^- , $Q = 1.8 \text{ MeV}$, $\tau_{1/2} = 1200 \text{ y}$, produced along with ^{163}Ho)
- ▷ GEANT4 $\rightarrow bkg \approx 0.5 \text{ c/eV/day/det/Bq}(^{166m}\text{Ho})$
- ▷ $A(^{163}\text{Ho}) = 300 \text{ Bq/det}$ ($\leftrightarrow \approx 6.5 \times 10^{13} \text{ nuclei/det}$)
 $bkg(^{166m}\text{Ho}) < 0.1 \text{ c/eV/day/det} \rightarrow A(^{163}\text{Ho})/A(^{166m}\text{Ho}) > 1500$
 $\rightarrow N(^{163}\text{Ho})/N(^{166m}\text{Ho}) > 6000$

^{163}Ho production by neutron activation



$^{162}\text{Er} (\text{n},\gamma) ^{163}\text{Er}$ $\sigma_{\text{thermal}} \approx 20\text{b}$
 $^{163}\text{Er} \rightarrow ^{163}\text{Ho} + \nu_e$ $\tau_{1/2}^{\text{EC}} \approx 75\text{min}$

Tm 163 1.81 h	Tm 164 5.1 m	Tm 165 30.06 h	Tm 166 7.70 h	Tm 167 9.25 d	Tm 168 93.1 d
ϵ β^+ ... γ 104; 69; 241; 1434; 1397...	ϵ β^+ 2.9... γ 91; 208; 315...	ϵ β^+ ... γ 243; 47; 297; 807...	ϵ β^+ 1.9... γ 779; 2052; 184; 1274...	ϵ γ 532...	ϵ ; β^+ ... β^- ... γ 198; 816; 447...
Er 162 0.139	Er 163 75 m	Er 164 1.601	Er 165 10.3 h	Er 166 33.503	Er 167 2.3 s 22.869
σ 19 $\sigma_{n,\alpha} < 0.011$	ϵ β^+ ... γ (1114...)	σ 13 $\sigma_{n,\alpha} < 0.0012$	ϵ γ no γ	σ 3 + 14 $\sigma_{n,\alpha} < 7E-5$	γ 208 σ 650 $\sigma_{n,\alpha} 3E-6$
Ho 161 6.7 s 2.5 h	Ho 162 68 m 15 m	Ho 163 1.2 4570 a	Ho 164 37 m 29 m	Ho 165 100	Ho 166 1200 a 26.80 h
ϵ γ 26; 78... ϵ^- γ 211	ϵ β^- ; ϵ γ 185; 1220; 283; 937...	ϵ β^+ 1.1... γ 81; 1319... ϵ^- γ 298	ϵ γ 37; 57... ϵ^- γ 37;	ϵ β^- 1.0... γ 91; 73... ϵ^- σ 3.1 + 58 $\sigma_{n,\alpha} < 2E-5$	β^- 1.9... 810; 712 σ 3100
Dy 160 2.329	Dy 161 18.889	Dy 162 25.475	Dy 163 24.896	Dy 164 28.260	Dy 165 1.3 m 2.35 h
σ 60 $\sigma_{n,\alpha} < 0.0003$	σ 600 $\sigma_{n,\alpha} < 1E-6$	σ 170	σ 120 $\sigma_{n,\alpha} < 2E-5$	σ 1610 + 1040	β^- 1.3... 1.0... γ 95; 515... σ 2000

- **HOLMES needs $\approx 200 \text{ MBq}$ of ^{163}Ho**

with *reasonable* assumptions on the (unknown) global embedding process efficiency...

- ^{162}Er irradiation at **ILL nuclear reactor** (Grenoble, France)

- ▶ thermal neutron flux at **ILL**: $1.3 \times 10^{15} \text{ n/cm}^2/\text{s}$
- ▶ **burn up** $^{163}\text{Ho}(\text{n},\gamma)^{164}\text{Ho}$: $\sigma_{\text{burn-up}} \approx 200\text{b}$ (preliminary result from **PSI** analysis)
- ▶ $^{165}\text{Ho}(\text{n},\gamma)$ (mostly from $^{164}\text{Er}(\text{n},\gamma)$) $\rightarrow ^{166\text{m}}\text{Ho}$ (β , $\tau_{1/2} = 1200\text{y}$) $\rightarrow A(^{163}\text{Ho})/A(^{166\text{m}}\text{Ho}) = 100 \sim 1000$

- chemical pre-purification and post-separation at **PSI** (Villigen, CH)



HOLMES source production

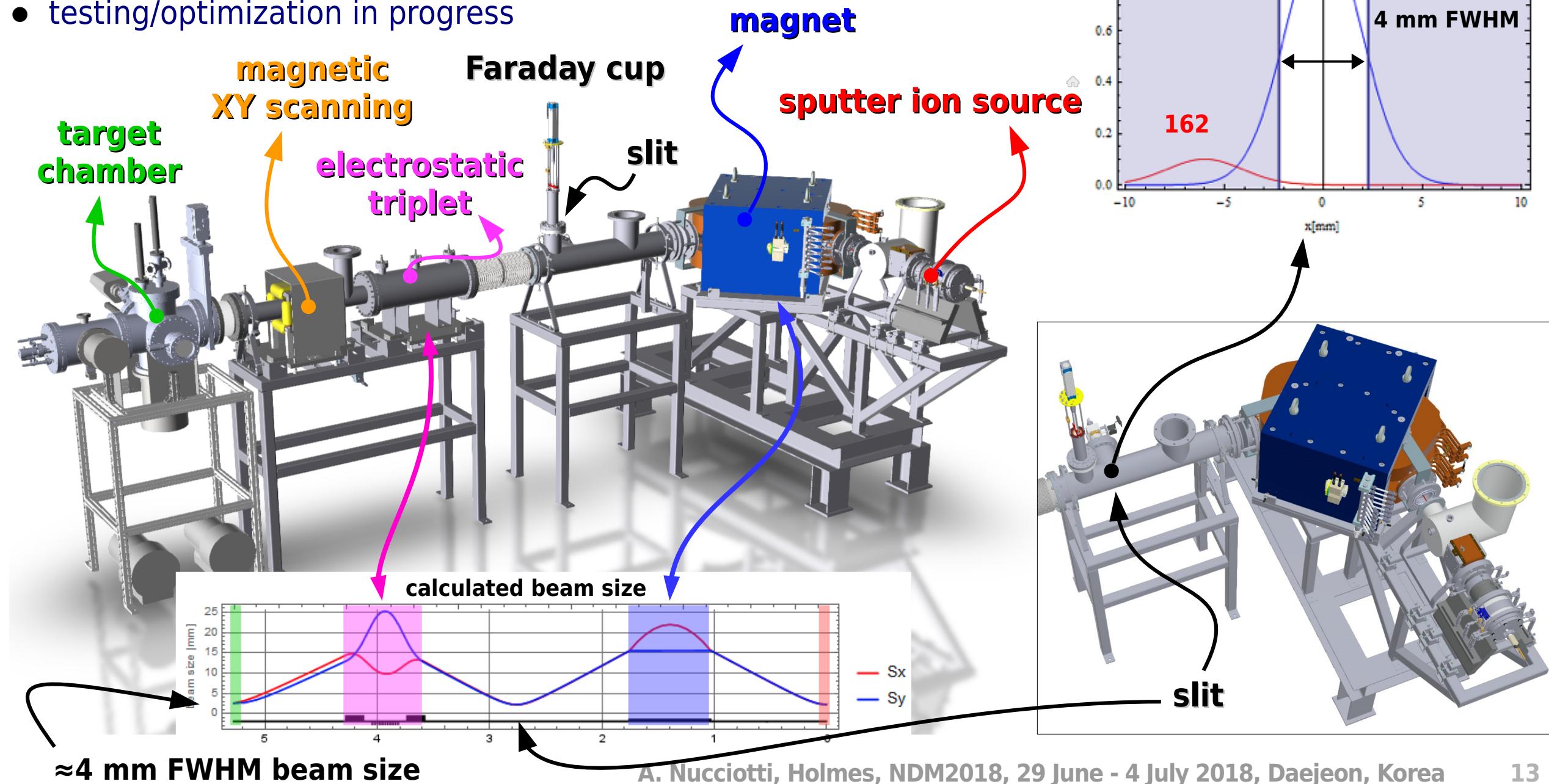
- **enriched Er_2O_3** samples irradiated at **ILL** and pre-/post-processed at **PSI**
 - ▶ 25 mg irradiated for 55 days (2014) → $A(\text{Ho}^{163}) \approx 5 \text{ MBq}$ ($A(\text{Ho}^{166m}) \approx 10 \text{ kBq}$)
 - ▶ 150 mg irradiated for 50 days (2015) → $A(\text{Ho}^{163}) \approx 23 \text{ MBq}$ ($A(\text{Ho}^{166m}) \approx 37 \text{ kBq}$)
- **Ho radiochemical separation** with ion-exchange resins in hot-cell at **PSI**
 - ▶ efficiency $\geq 79\%$ (preliminary)
- **540 mg of 25% enriched Er_2O_3** irradiated 50 days at **ILL** in 2017 (separation in progress)
 - ▶ $A(\text{Ho}^{163})_{\text{theo}} \approx 100 \text{ MBq}$ (enough for R&D and 500 pixels) ($A(\text{Ho}^{166m}) \approx 180 \text{ kBq}$)





HOLMES mass separation and ion implantation

- extraction voltage 30-50 kV → 10-100 nm implant depth
- ^{163}Ho / $^{166\text{m}}\text{Ho}$ separation better than 10^5
- testing/optimization in progress



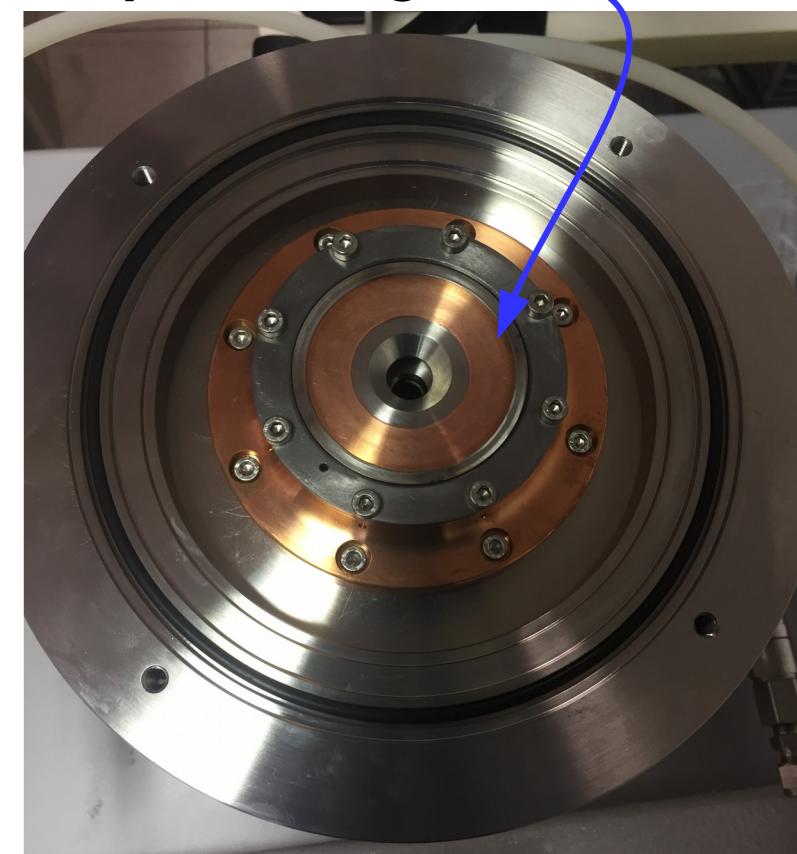
HOLMES ion implantation system testing



sputter ion source



sputter target

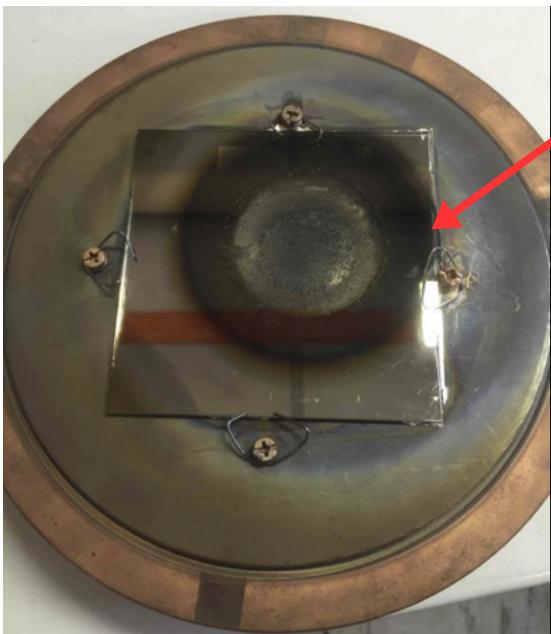


- tests in progress
 - ▶ without focussing
 - ▶ with natural Ho
- triplet and scanning stages ready to be installed

Ion source sputter target production / 1



- metallic holmium sputter target for implanter ion source
- enriched $\text{Er}_2\text{O}_3 \rightarrow \text{Ho}_2\text{O}_3$
- thermoreduction/distillation in furnace
 - ▶ $\text{Ho}_2\text{O}_3 + 2\text{Y(met)} \rightarrow 2\text{Ho(met)} + \text{Y}_2\text{O}_3$ at $T > 1600^\circ\text{C}$
- new furnace set-up in 2016
- work in progress to
 - ▶ optimize the process
 - ▶ measure efficiency ($\approx 70\%$, preliminary)

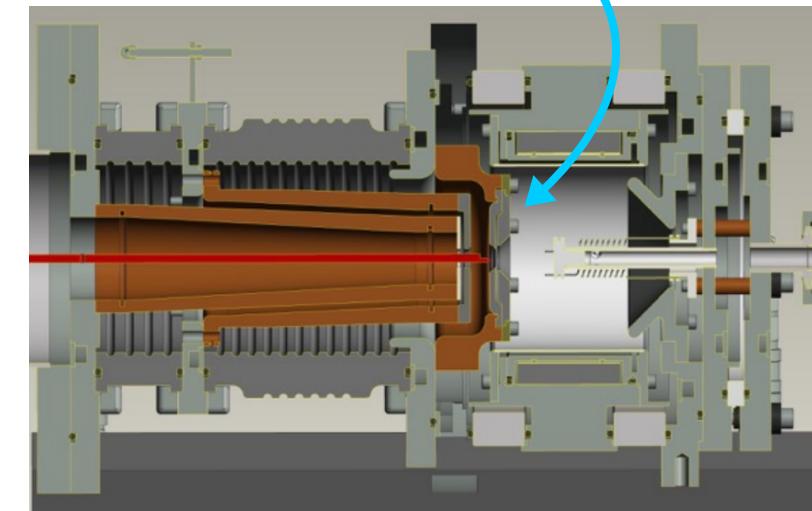
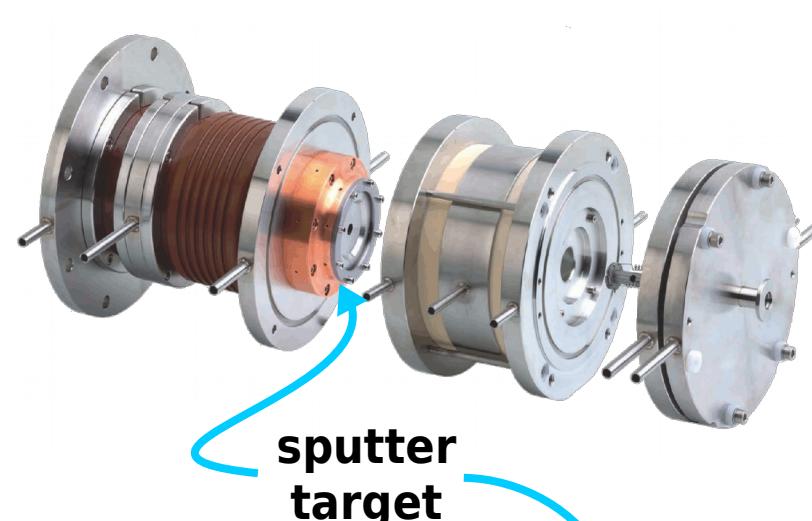


Ion source sputter target production / 2



■ metallic holmium sputter target for implanter ion source

- ▶ work is in progress to produce the sputter target
- ▶ sintering Ho with other metals



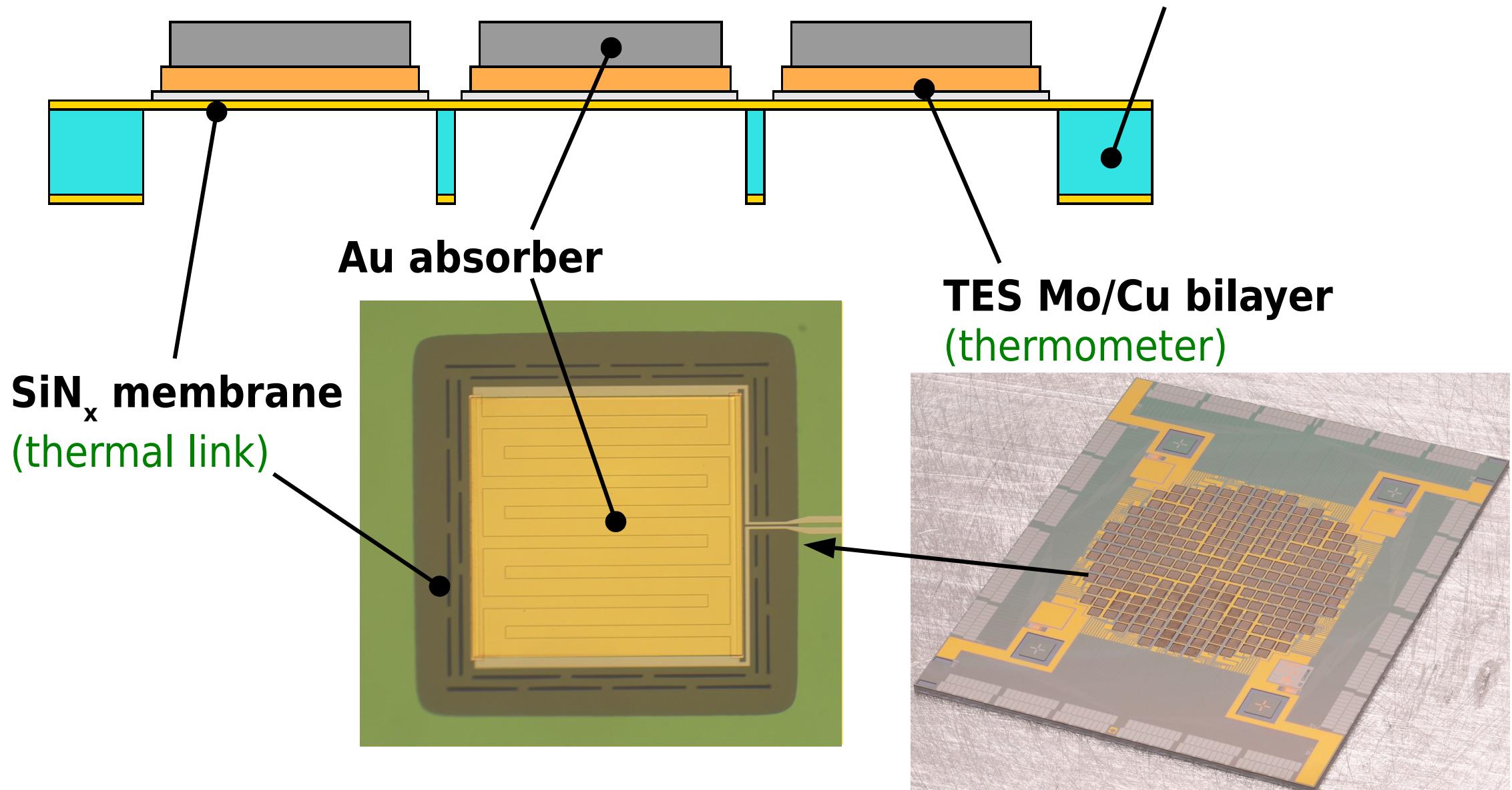
high pressure
+
heat treatment



TES low temperature microcalorimeters



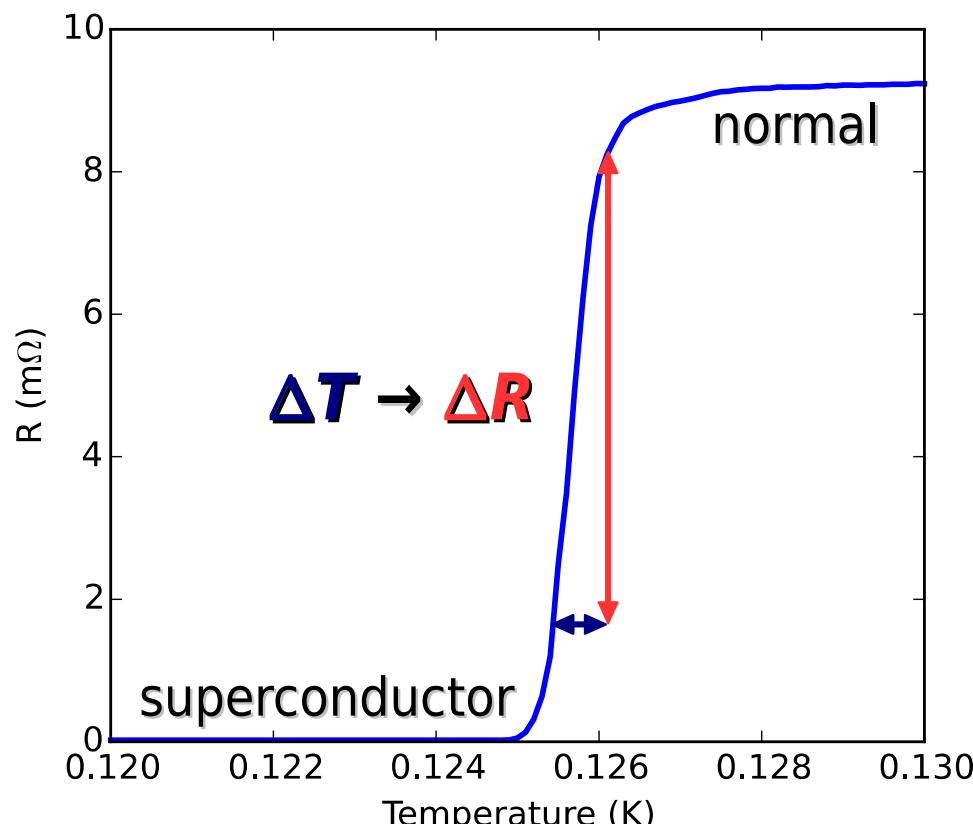
NIST TES arrays for X-ray spectroscopy



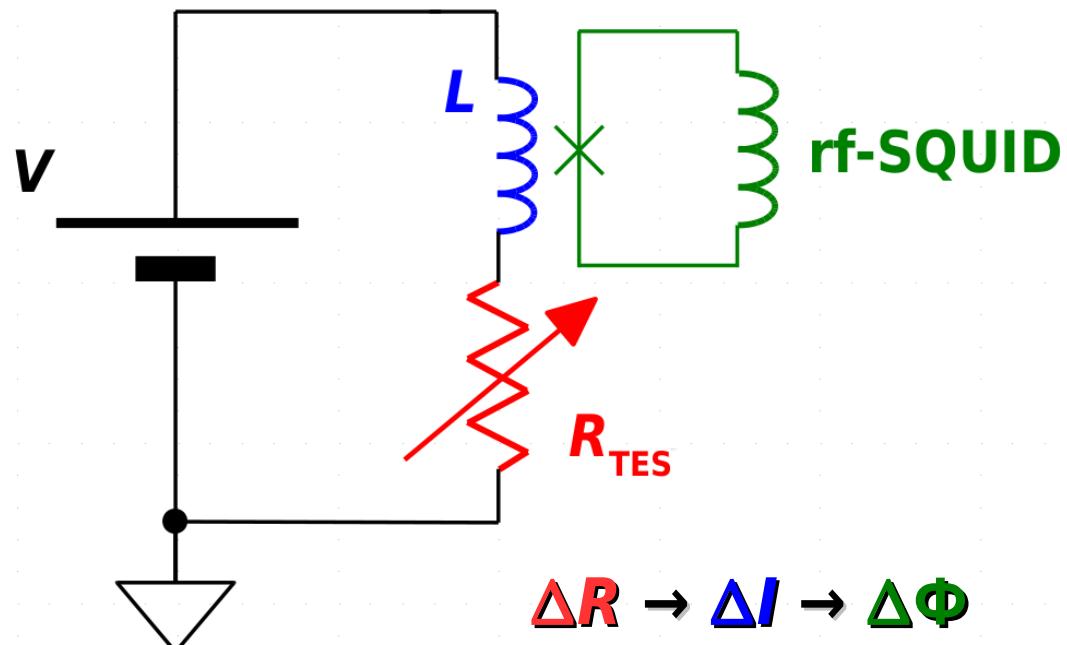
Superconducting transition edge sensors (TES)



- superconductor thin films operated inside the phase transition at T_c
 - ▶ metal-superconductor bilayers → tunable T_c (20÷200 mK) : Mo/Cu, Ti/Au, Ir/Au, ...
- high sensitivity $T_dR/(RdT) \approx 100$ → **high energy resolution**
 - ▶ as thermal sensors → thermodynamical fluctuation limited → $\sigma_E^2 \approx \xi^2 k_B T^2 C$
- strong electron-phonon coupling → **high intrinsic speed**
- low impedance → SQUID read-out → **multiplexing for large arrays**



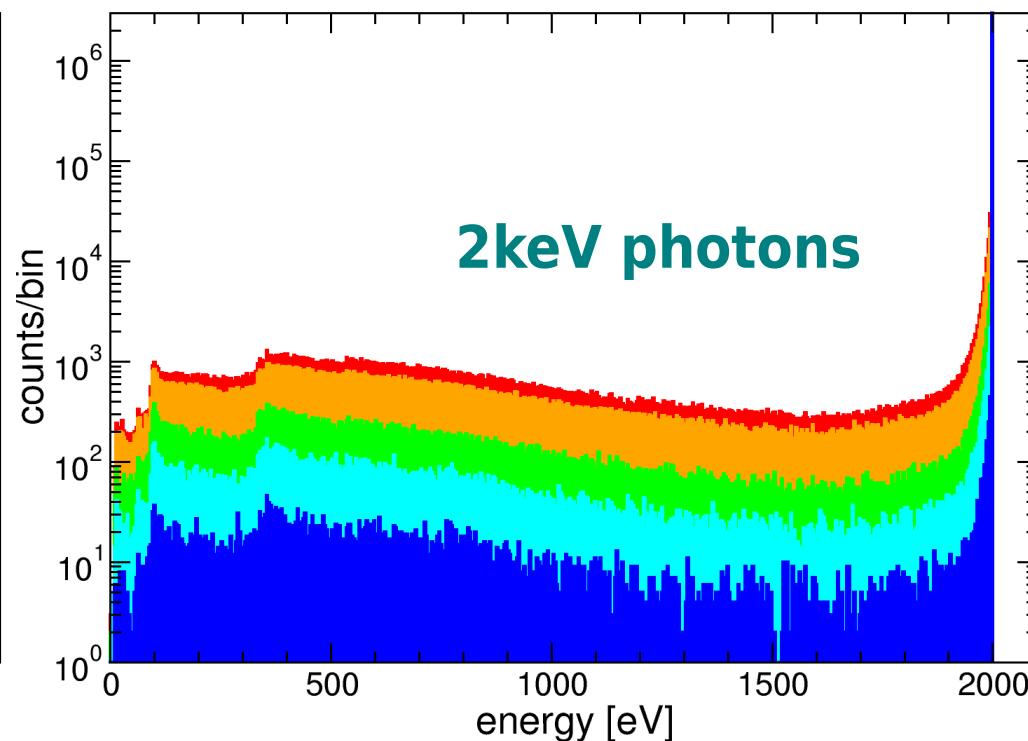
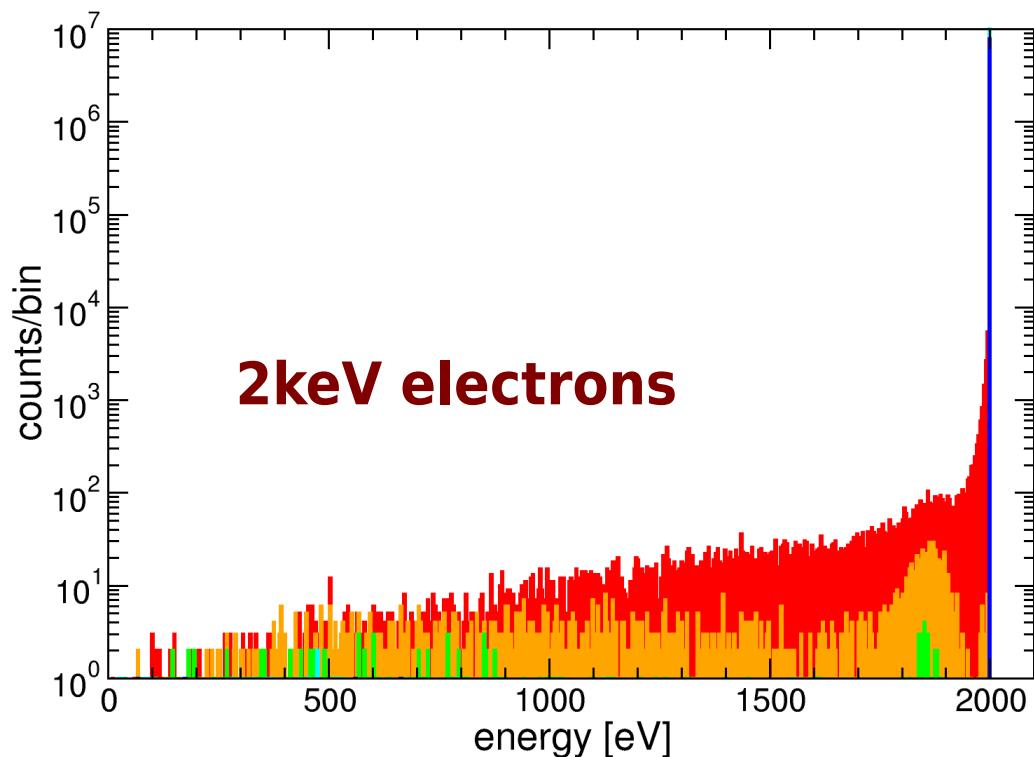
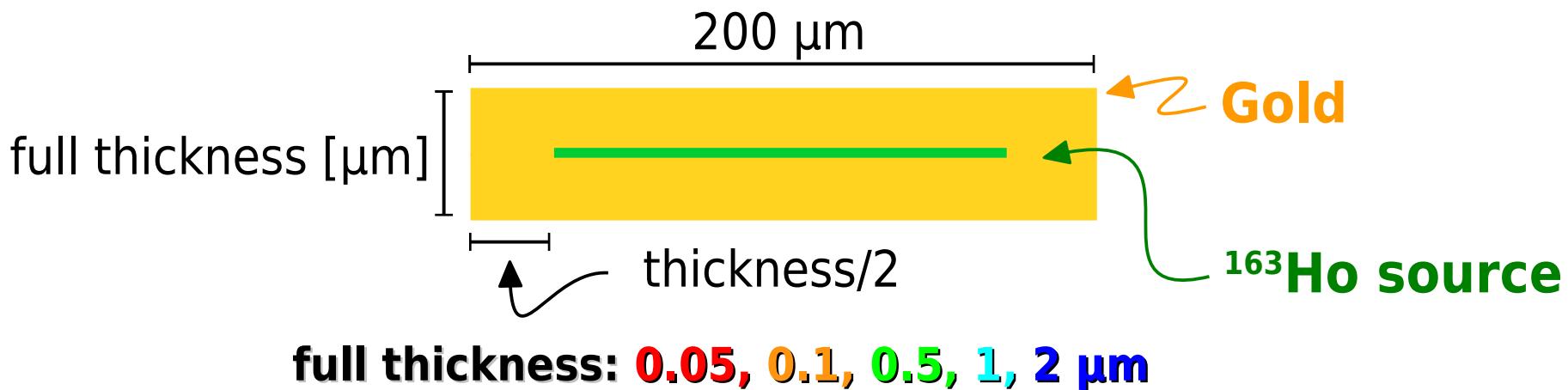
TES read-out: constant voltage bias



TES absorber design: stopping EC radiation / 1



Geant4 + LowEnergyEM MC simulation

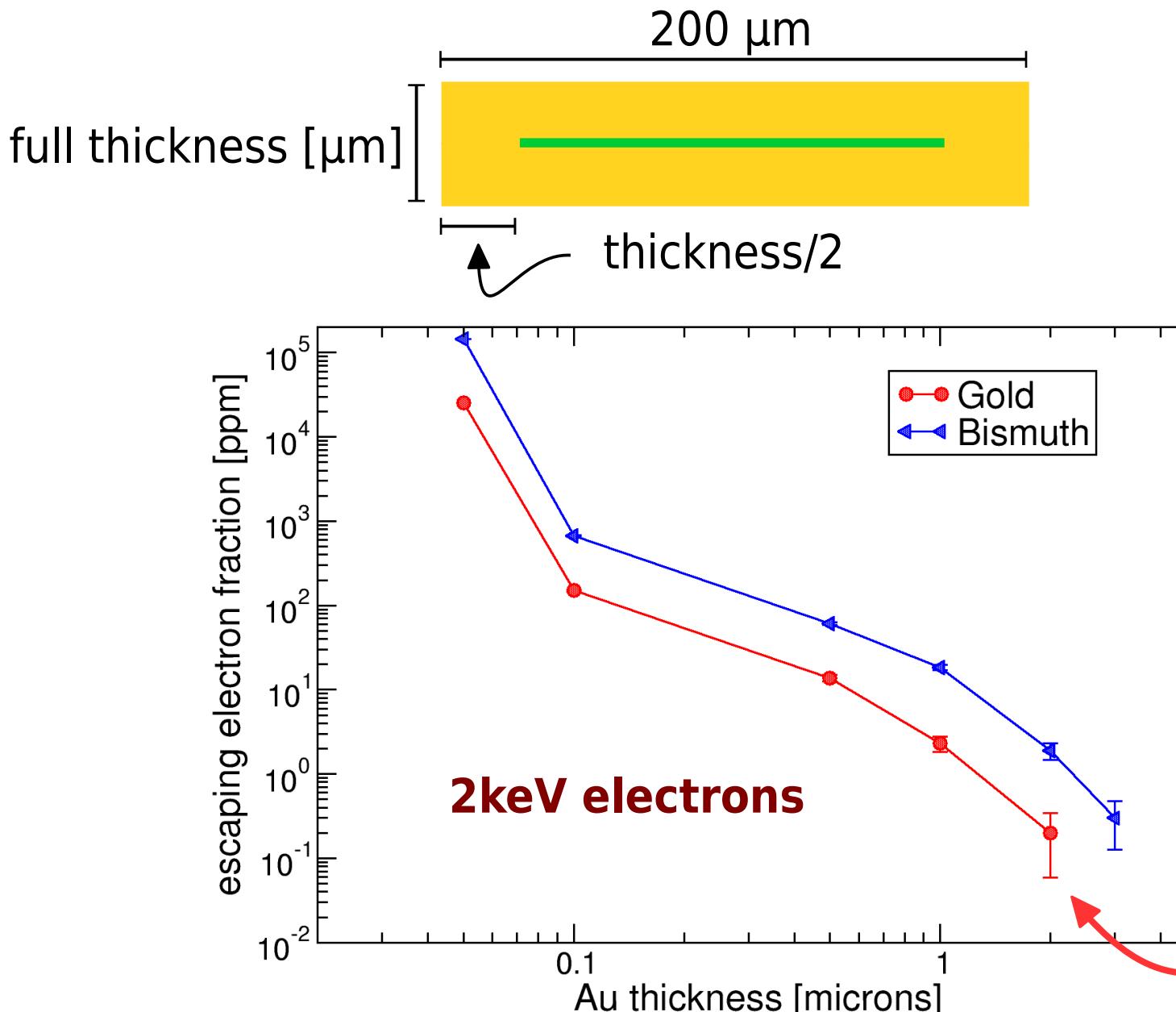


$$I_x \approx 10^{-5} I_e$$

TES absorber design: stopping EC radiation / 2



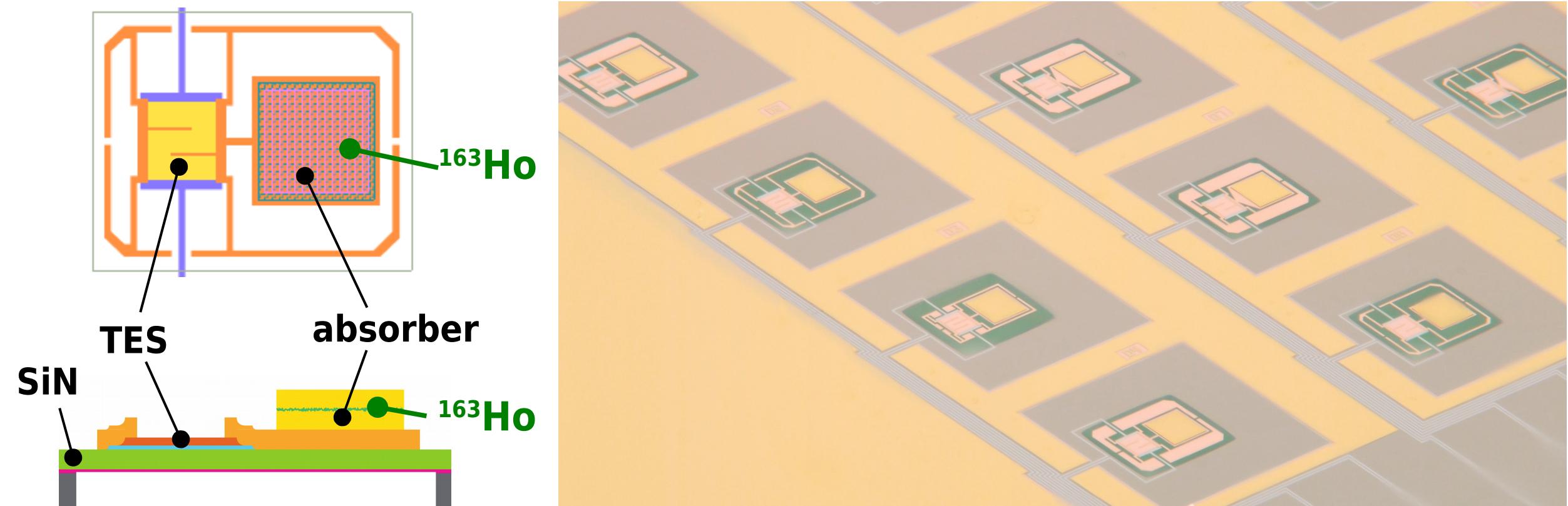
Geant4 + LowEnergyEM MC simulation



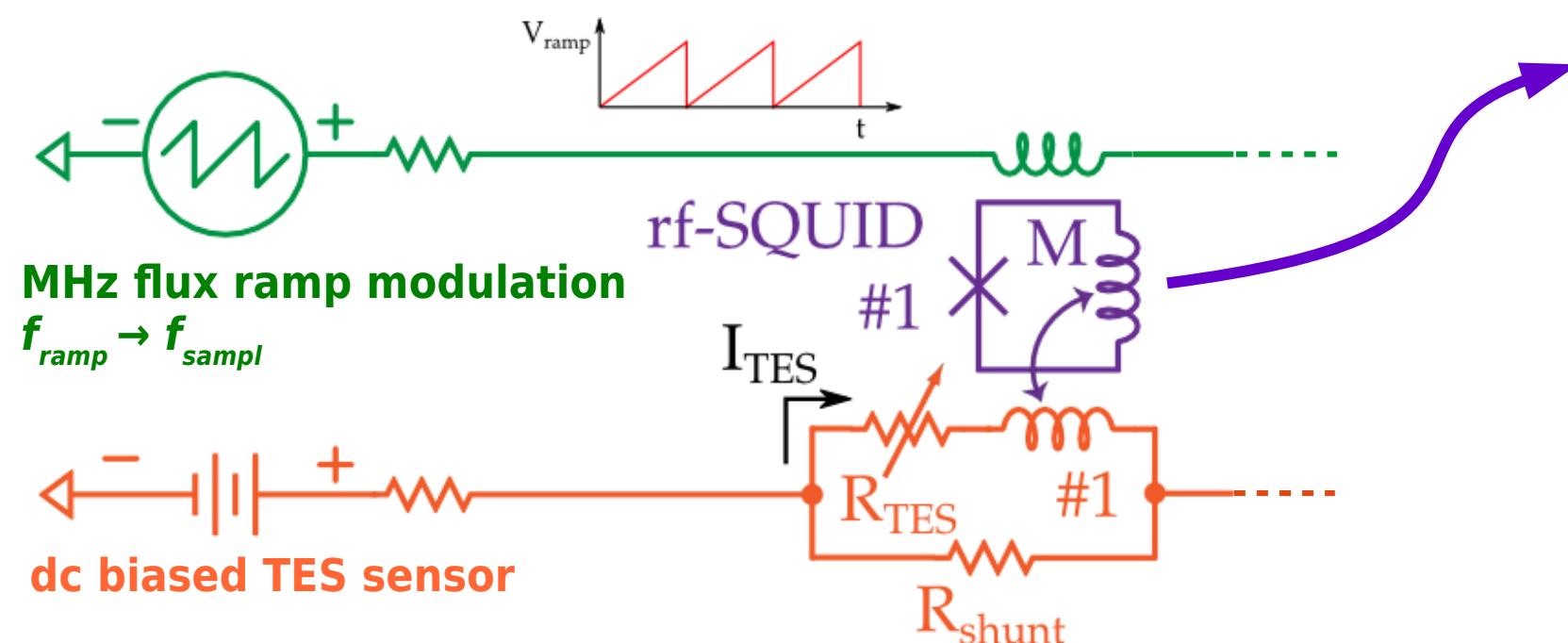
HOLMES pixel design and test



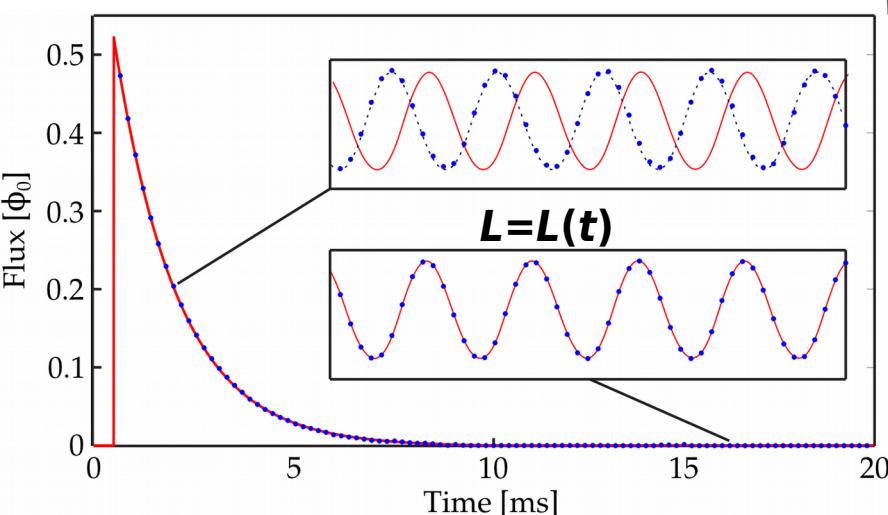
- optimize design for speed and resolution
 - ▷ **specs @3keV : $\Delta E_{FWHM} \approx 1\text{eV}$, $\tau_{rise} \approx 10\mu\text{s}$, $\tau_{decay} \approx 100\mu\text{s}$**
- **2 μm Au** thickness for *full* electron and photon absorption
- **side-car** design to avoid TES proximitation and G engineering for τ_{decay} control
- **NIST** designed and fabricated 4×6 arrays of **TES prototypes** for optimization **w/o ^{163}Ho**



HOLMES array read-out: rf-SQUID

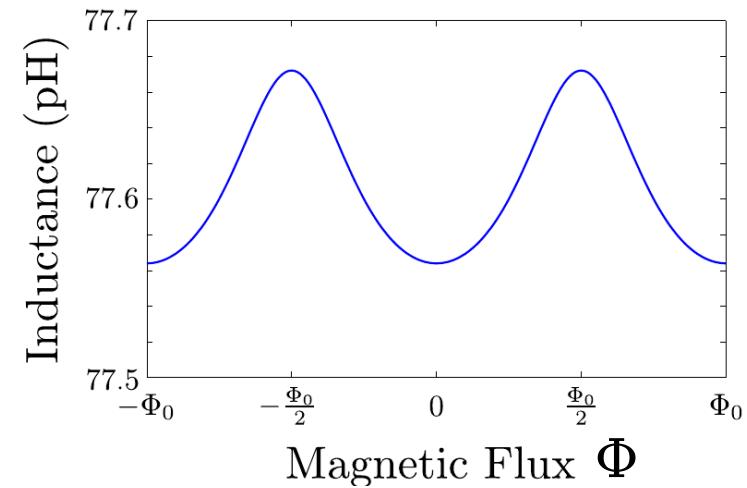
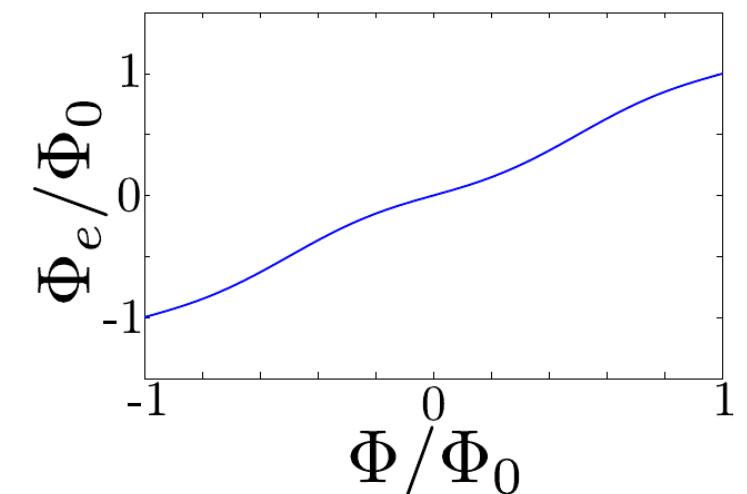


$$\Delta T \rightarrow \Delta R_{\text{TES}} \rightarrow \Delta I_{\text{TES}} \rightarrow \Delta \Phi_e \rightarrow \Delta \Phi \rightarrow \Delta L$$

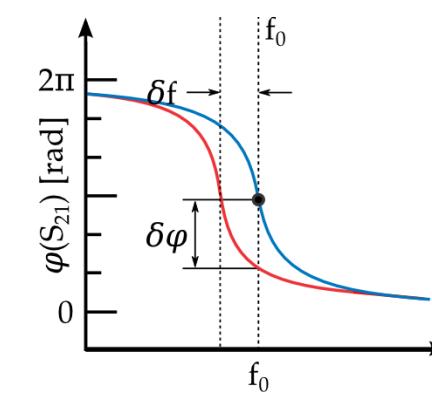
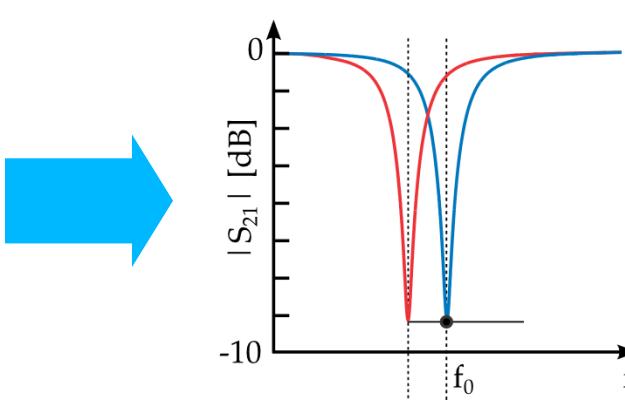
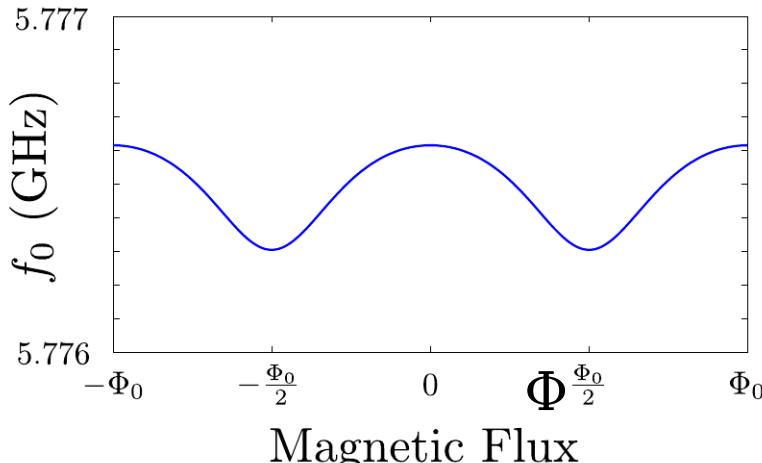
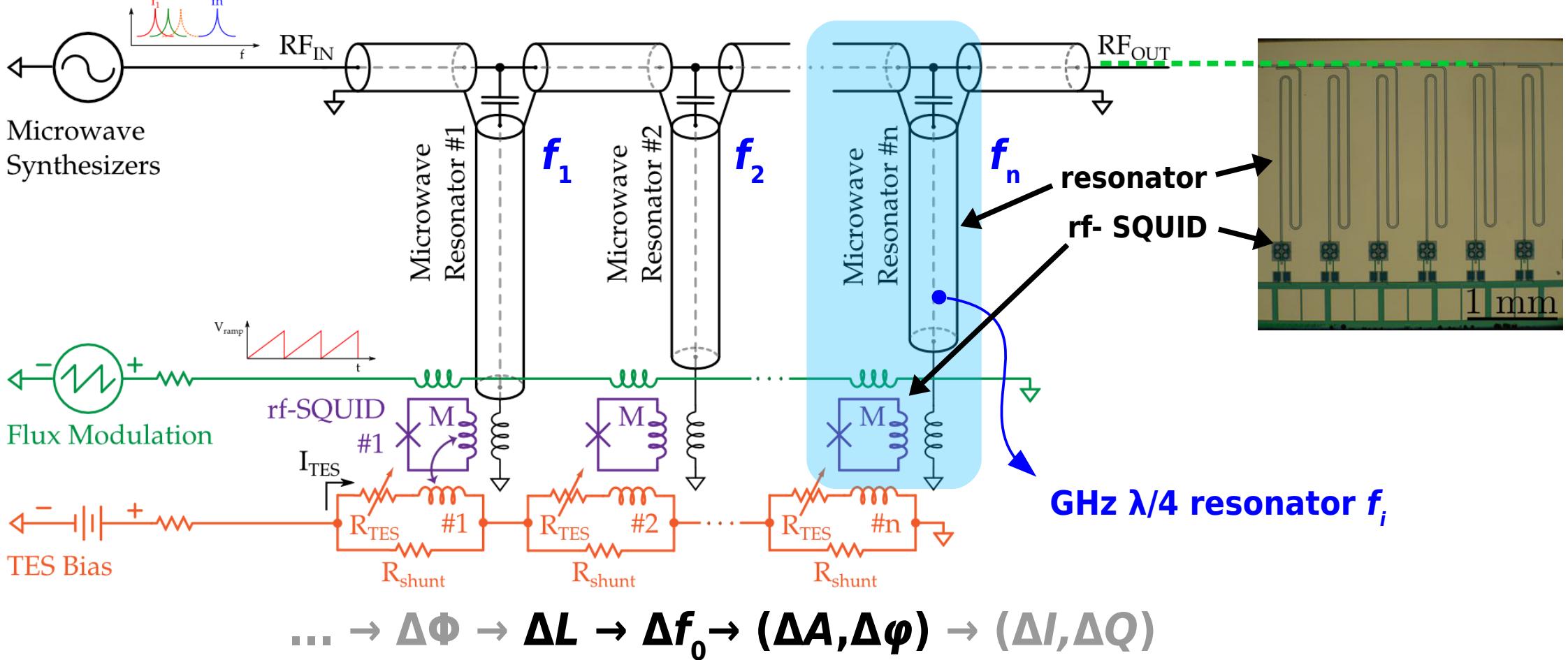


- $\Phi_e = \Phi_{\text{TES}} + \Phi_{\text{ramp}}$
- **flux-ramp modulation**
 - phase modulated MHz signal $L(t)$
 - linearized non-hysteretic rf-SQUID response
 - avoid low frequency noise sources

non-hysteretic rf-SQUID
 Φ_e vs. Φ and L vs. Φ responses



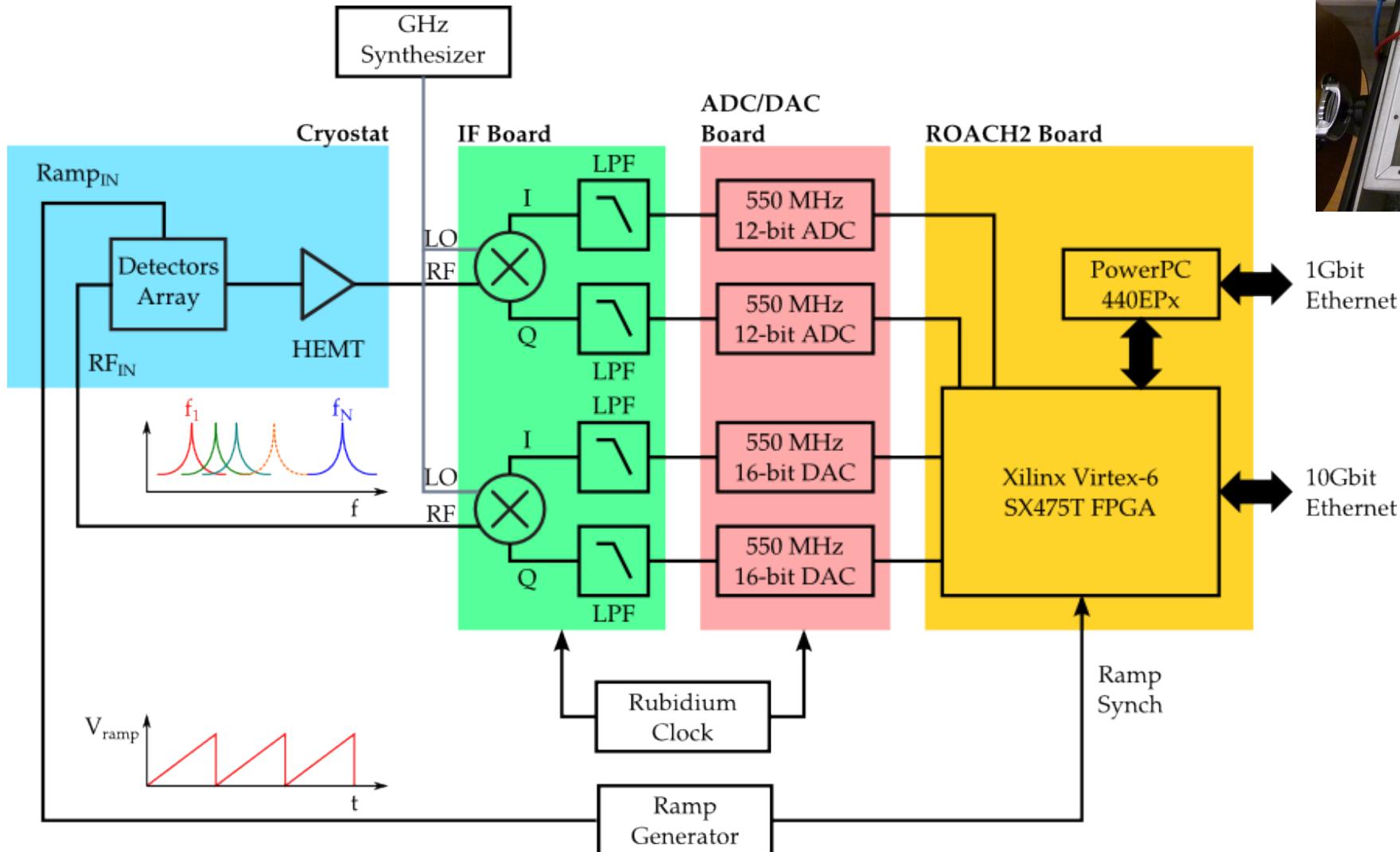
HOLMES array read-out: microwave multiplexing



HOLMES DAQ: Software Defined Radio



- base-band tone generation (0-512MHz)
- up- / down-conversion (base-band → 4-8 GHz → base-band)
- base-band tone IQ de-modulation (0-512MHz)
- rf-SQUID phase signal de-modulation by Fourier analysis

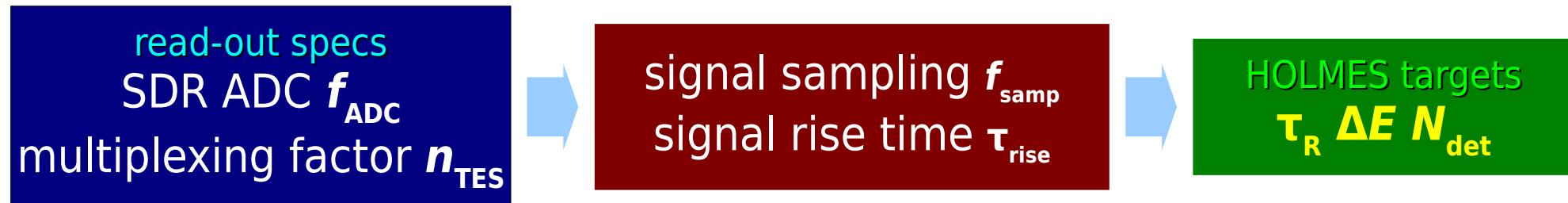


HOLMES detector design



design mostly driven by **read-out bandwidth** requirements

- TES microwave multiplexing with rf-SQUID ramp modulation + Software Defined Radio (SDR)



$$f_{\text{samp}} \geq \frac{R_d}{\tau_{\text{rise}}} \approx \frac{5}{\tau_{\text{rise}}} \quad \text{detector signal sampling (signal BW)}$$

$$f_{\text{res}} \geq 2 n_{\Phi_0} f_{\text{samp}} \quad \text{flux ramp modulated signal BW (resonator BW)}$$

$$f_n \geq g_f f_{\text{res}} = \frac{2 R_d g_f n_{\Phi_0}}{\tau_{\text{rise}}} \quad \text{microwave tones separation } (g_f \geq 7)$$

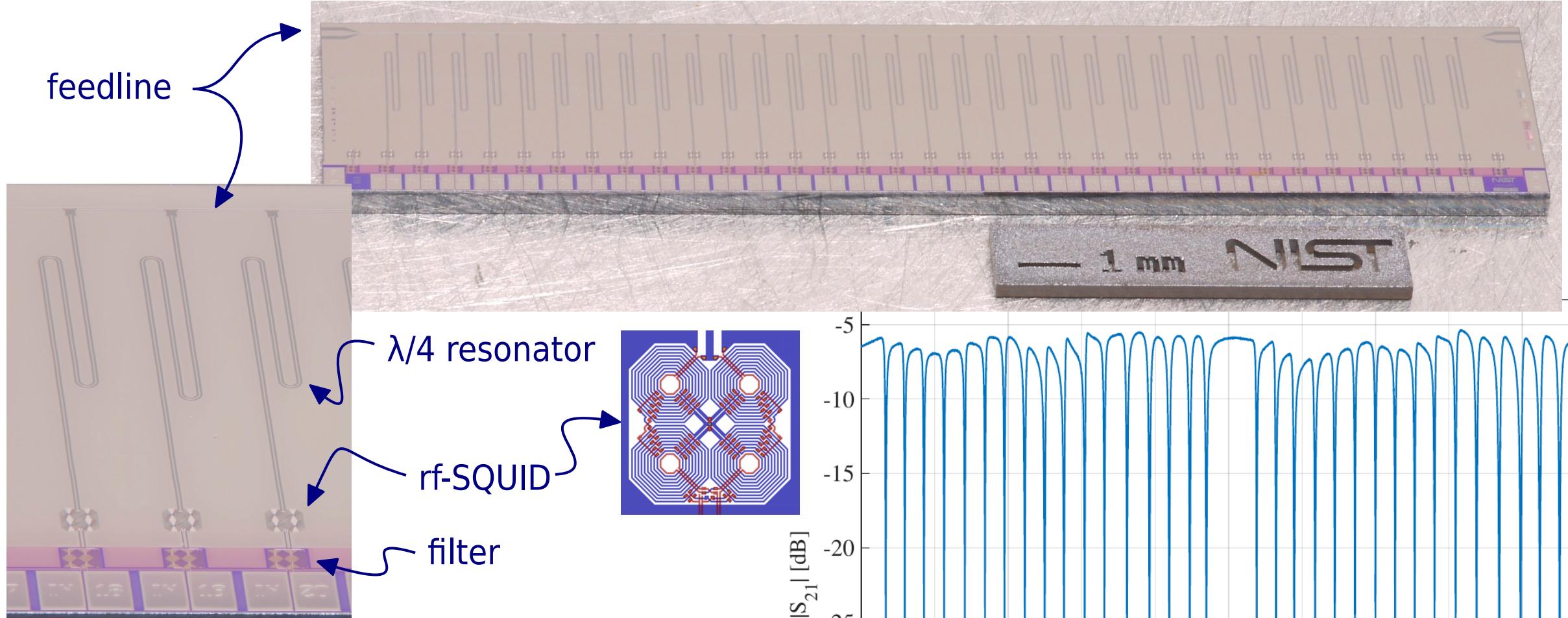
multiplexing factor →

$$n_{\text{TES}} = \frac{f_{\text{ADC}}}{f_n} \leq \frac{f_{\text{ADC}} \tau_{\text{rise}}}{2 R_d g_f n_{\Phi_0}} \approx \frac{f_{\text{ADC}} \tau_{\text{rise}}}{140}$$

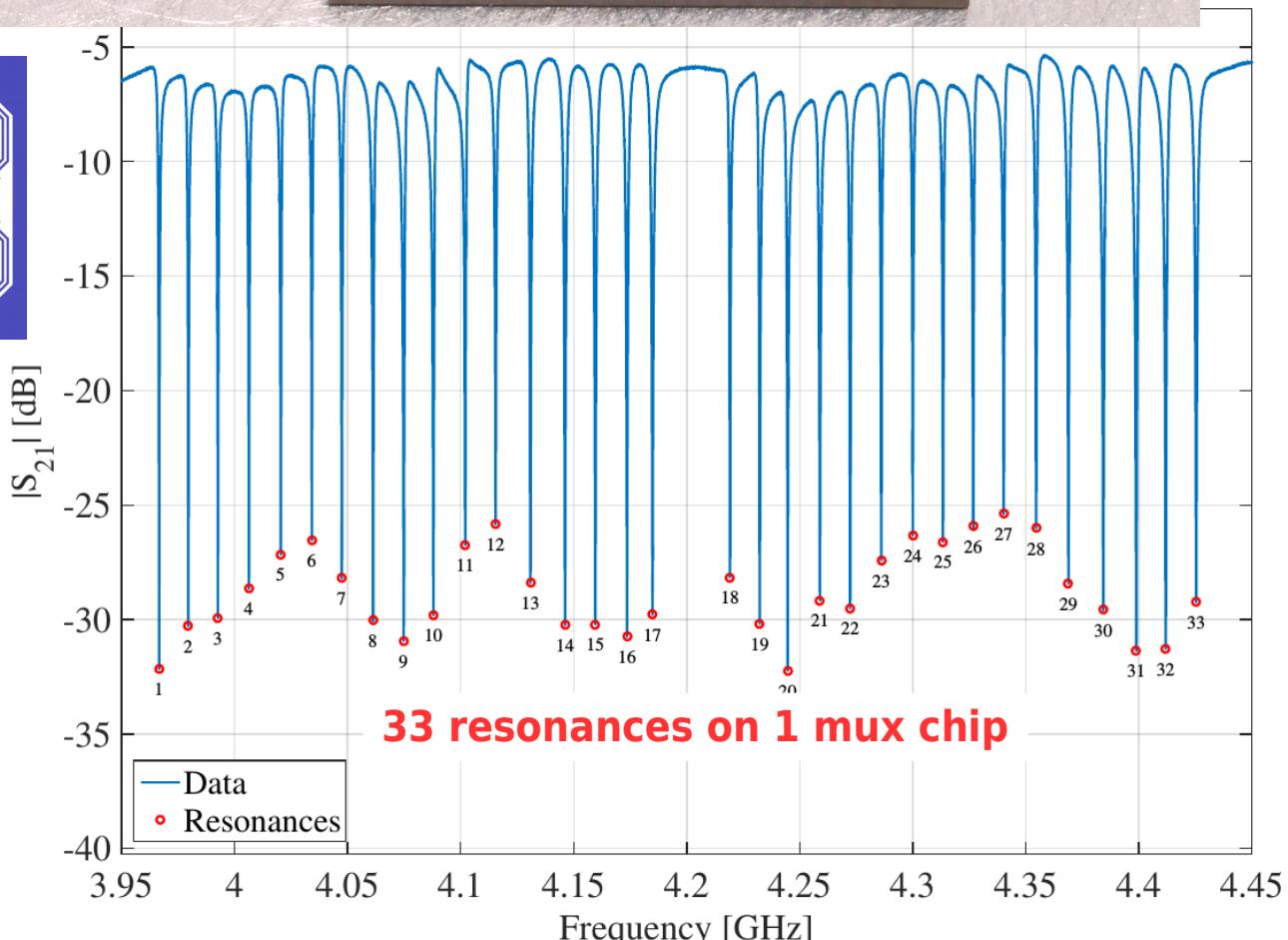
for fixed $f_{\text{ADC}} = 512 \text{MHz}$ and $n_{\text{TES}} = 32 \leftrightarrow \tau_{\text{rise}} \approx 10 \mu\text{s}$ with $f_{\text{samp}} = 0.5 \text{MHz}$

→ check for slew rate, τ_R and ΔE ...

HOLMES μ wave multiplexed TES read-out



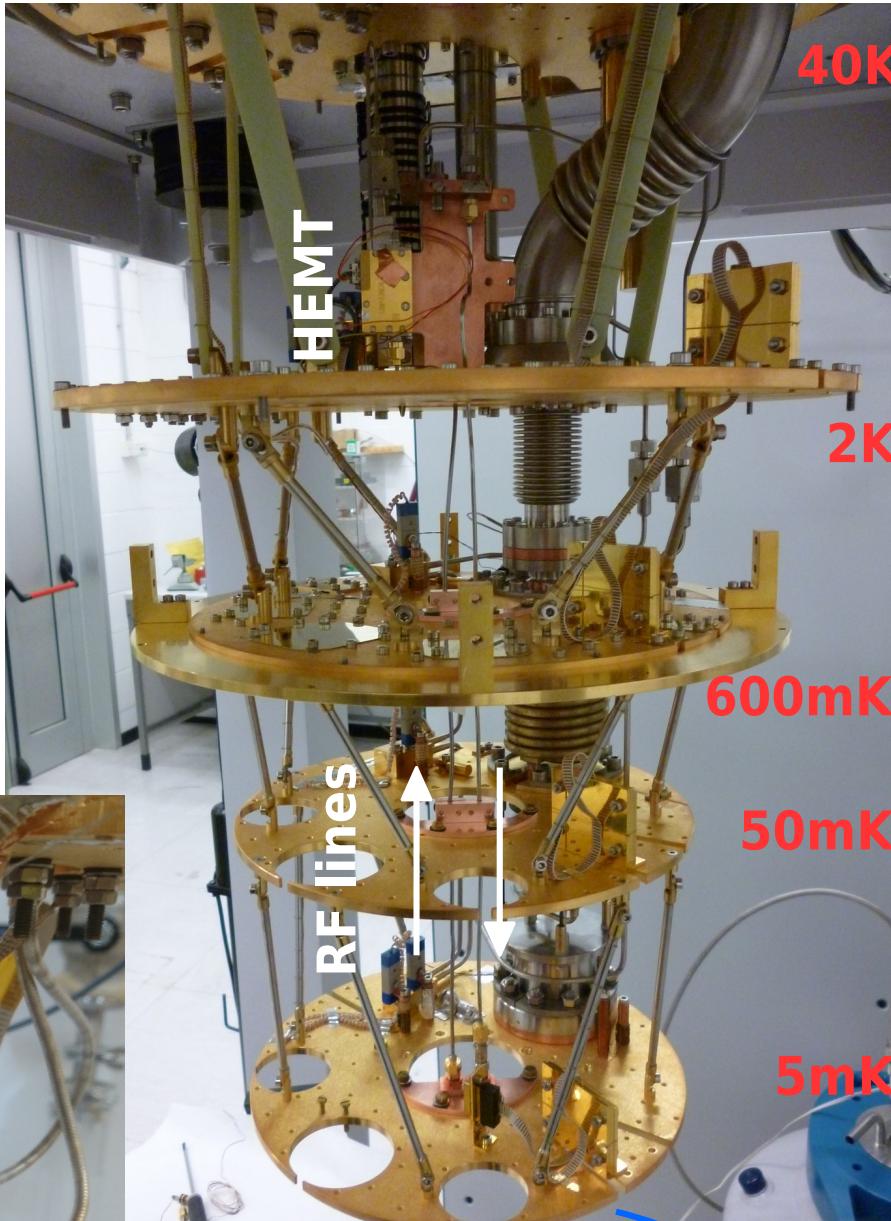
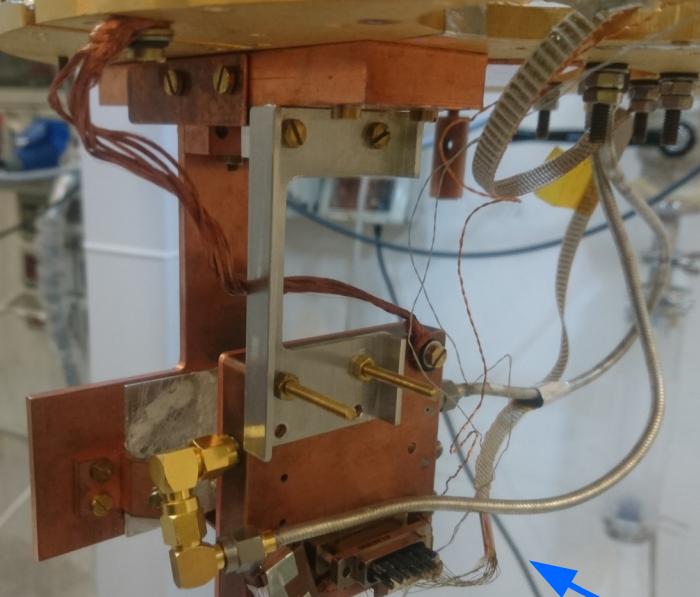
- **μ MUX17A** optimized for **HOLMES**
- 33 resonances in 500 MHz
 - ▶ width $f_{\text{res}} = 2 \text{ MHz}$
 - ▶ separation 14 MHz ($g_f = 7$)
- squid noise $< \approx 2 \mu\Phi_0/\sqrt{\text{Hz}}$



Cryogenic set-up



detector holder
mounted with
calibration source

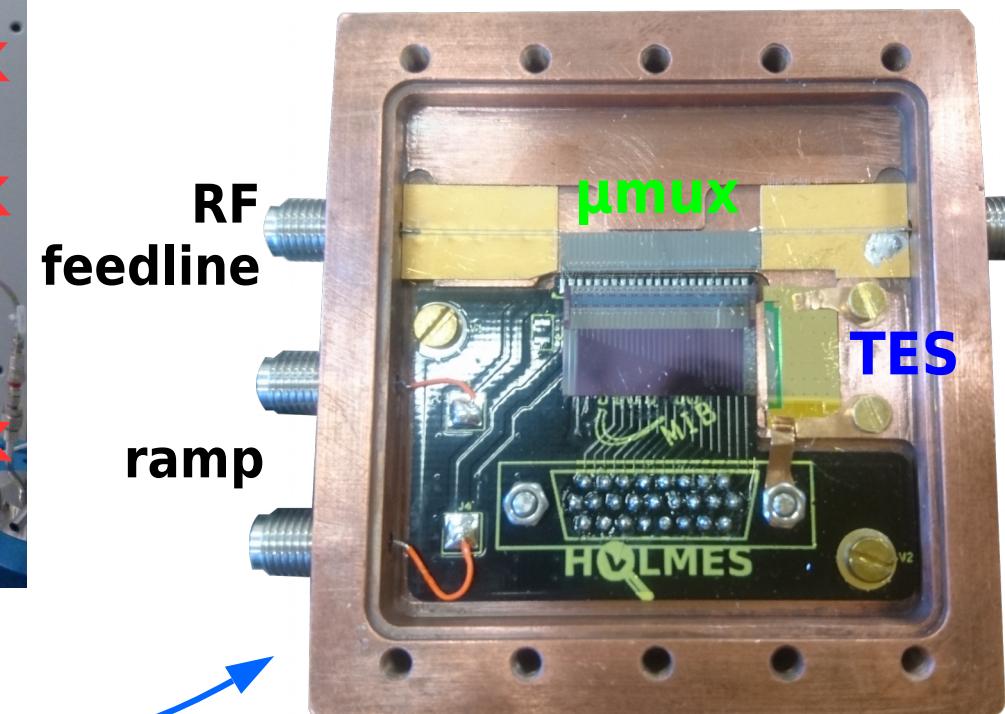


A. Nucciotti, Holmes, NDM2018, 29 June - 4 July 2018, Daejeon, Korea

LHe-free dilution fridge

1 HEMT + 2 coax RF lines
→ **8** μmux chips
→ **256** detectors

detector holder

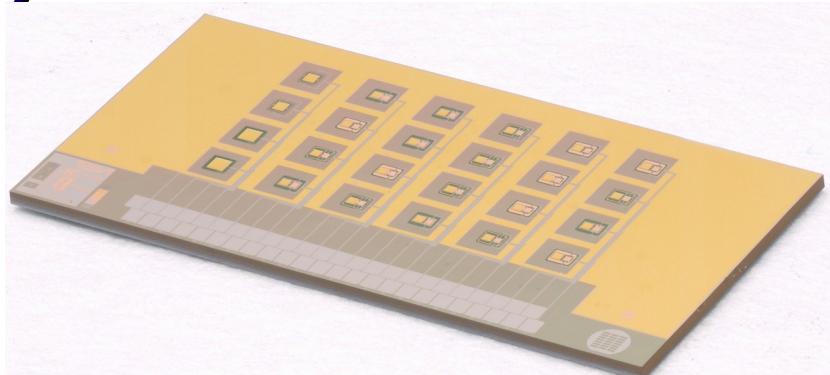


TES pixel testing with HOLMES DAQ / 1

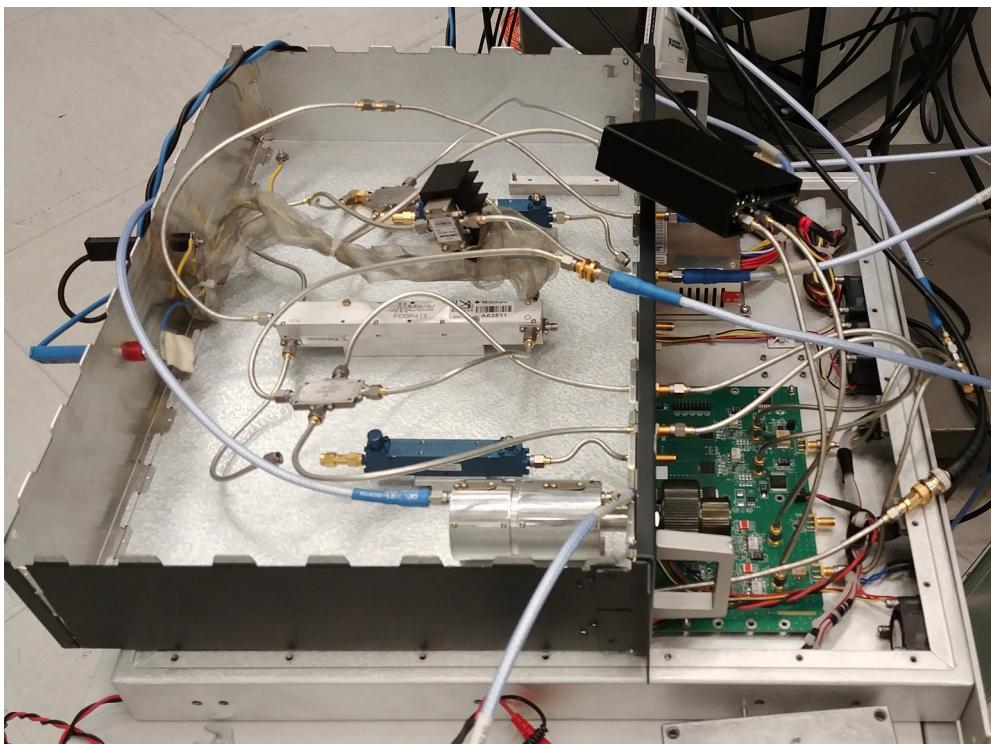


ROACH-2 based Software Defined Radio

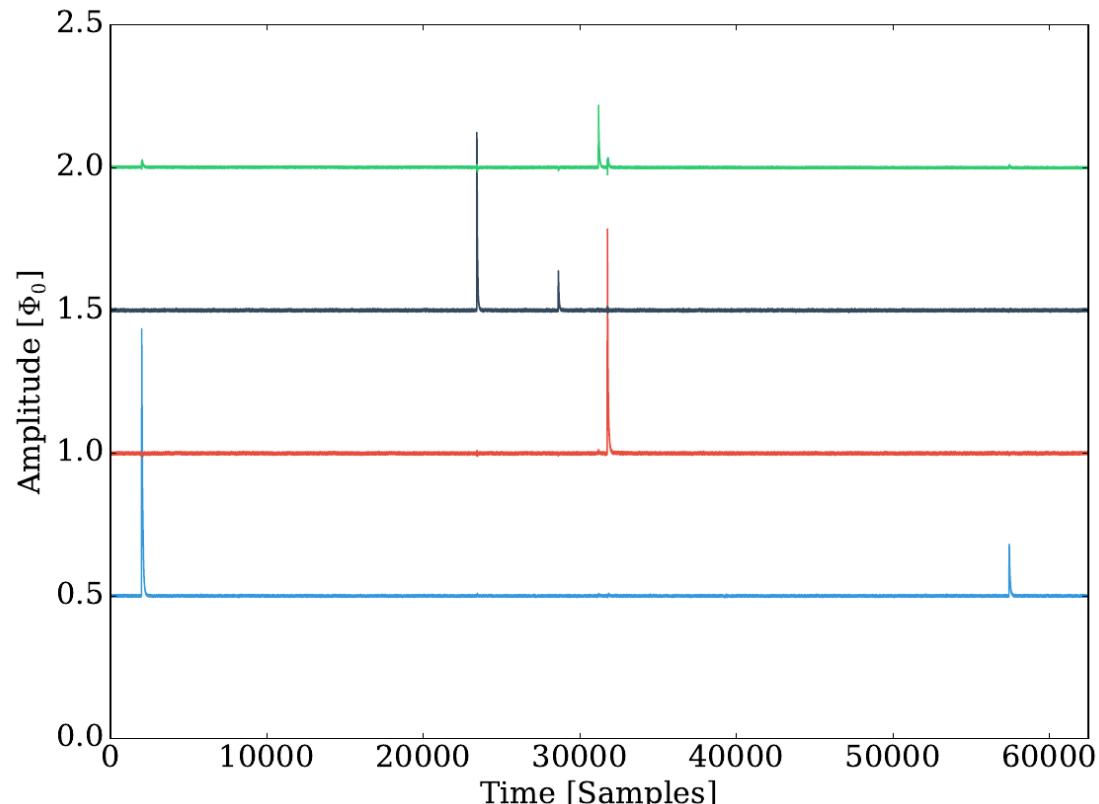
- ADC (550 MS/s 12bit) / DAC (1 GS/s 16bit)
- discrete components IF circuitry (up- / down- conversion)
- $n_{\Phi_0} = 2$, $f_{\text{samp}} = 500$ kS/s
- 16 ch firmware from NIST (uses only half of available ADC bandwidth)
- 4 HOLMES prototypes acquired \leftrightarrow limited by available tone power
- **goals:** check algorithms, noise, ΔE , τ_R and slew rate



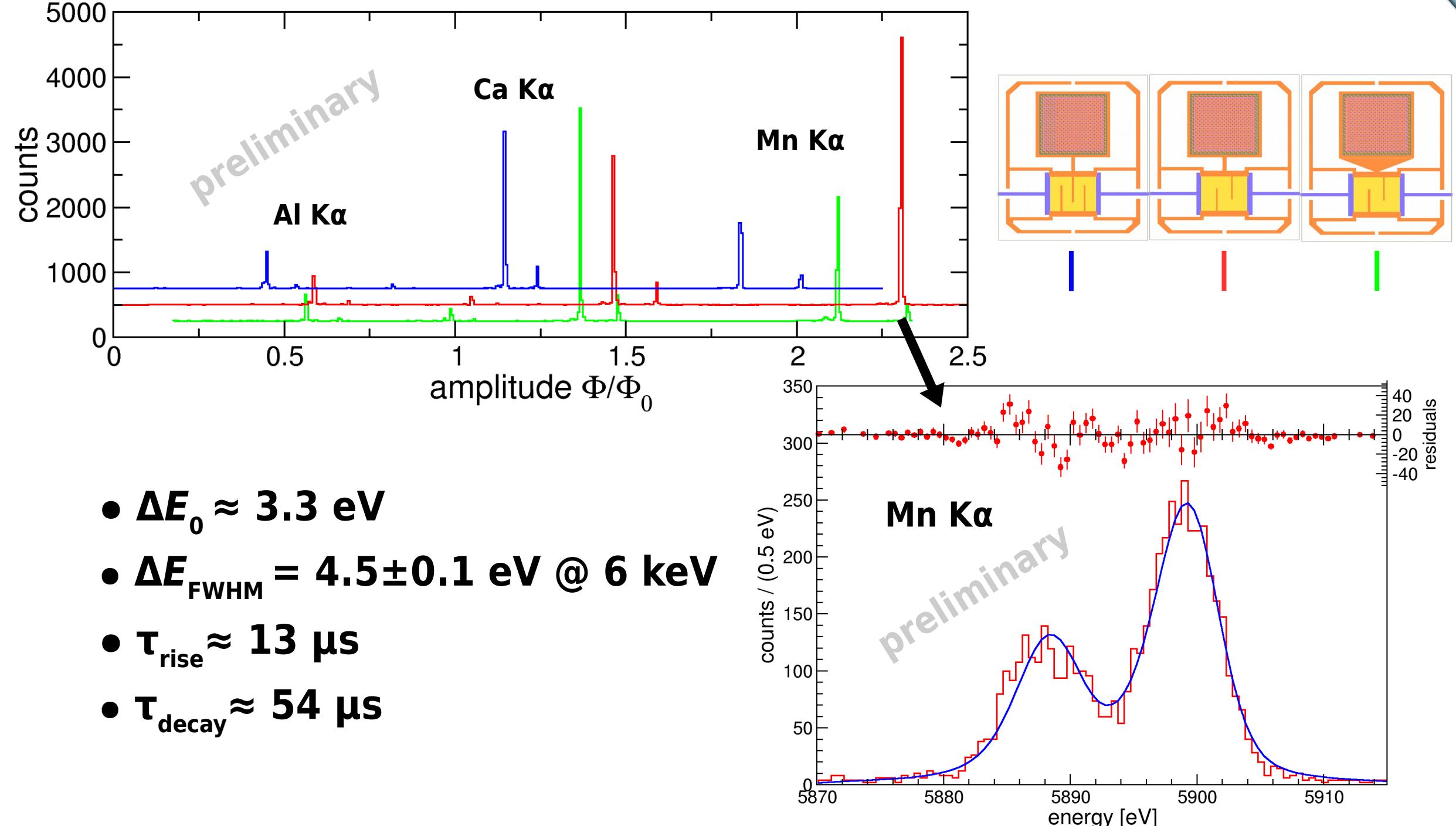
HOLMES prototypes



ROACH + IF circuit



TES pixel testing with HOLMES DAQ / 2



Rise time pile-up

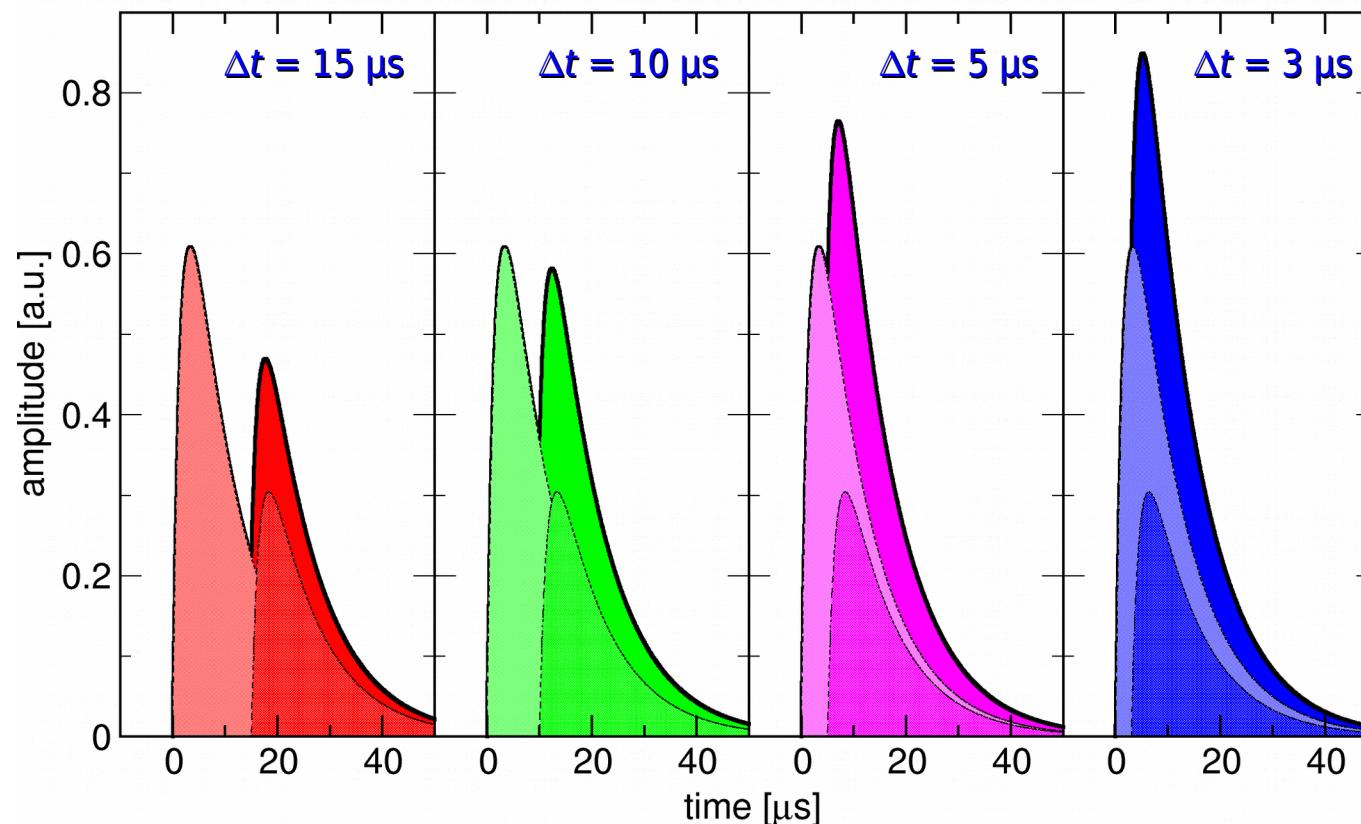


simple pulse model

$$A(t) = A_0(e^{-t/\tau_{decay}} - e^{-t/\tau_{rise}})$$

2 pulses with:

- $\tau_{rise} = 1.5 \mu s$
- $\tau_{decay} = 10 \mu s$
- $A_2/A_1 = 0.5$



resolving time $\tau_R \approx$ pulse rise time τ_{rise}

Detector time resolution



- for subsequent (Δt) events with energy E_1 and E_2 : time resolution $\tau_R = \tau_R(E_1, E_2)$

$$N_{pp}(E) = A_{EC} \int_0^{\infty} \tau_R(E, \epsilon) N_{EC}(\epsilon) N_{EC}(E - \epsilon) d\epsilon$$

- **Montecarlo pile-up spectrum simulations**

- ▷ event pairs with $E_1 + E_2 \in [2.4 \text{ keV}, 2.9 \text{ keV}]$ (drawn from ^{163}Ho spectrum), $\Delta t \in [0, 10\mu\text{s}]$
- ▷ pulse shape and noise from NIST TES model, sampled with f_{samp} , record length, and n bit

- **process with pile-up detection algorithms:**

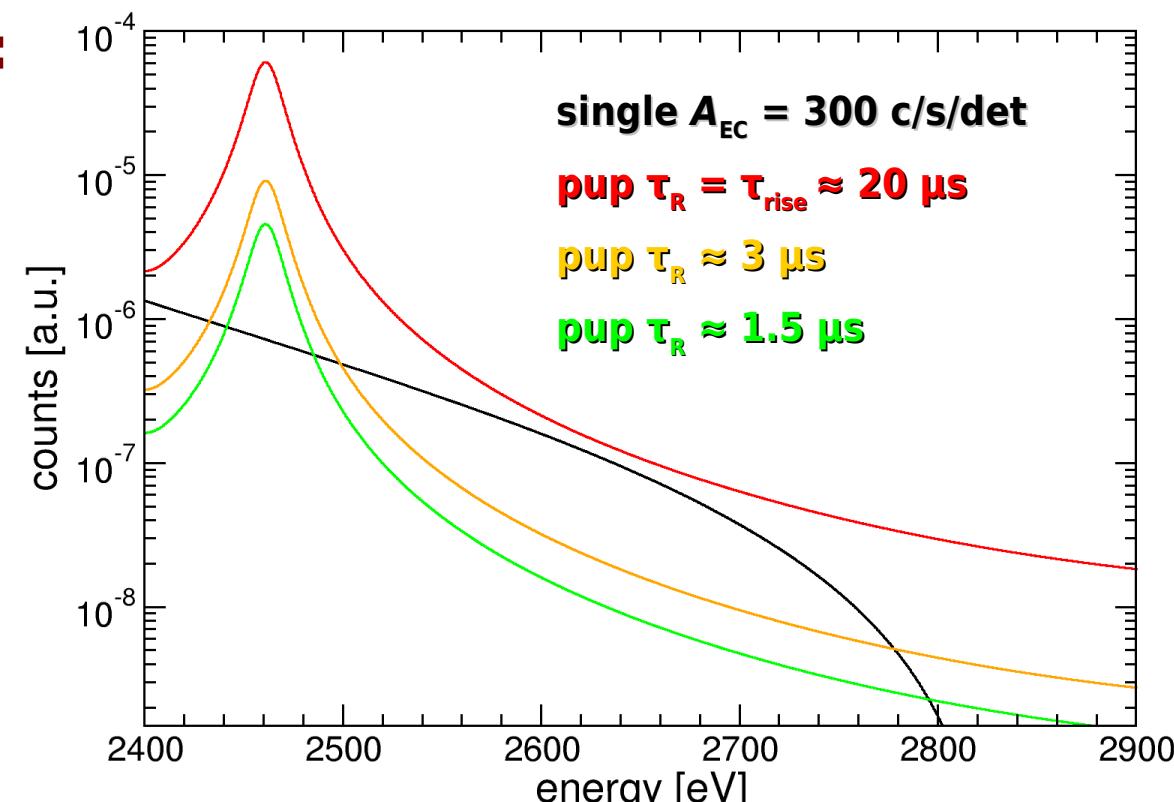
- ▷ for $f_{\text{samp}} = 0.5\text{MHz}$, $\tau_{\text{rise}} \approx 20\mu\text{s}$

- **Wiener Filtering**

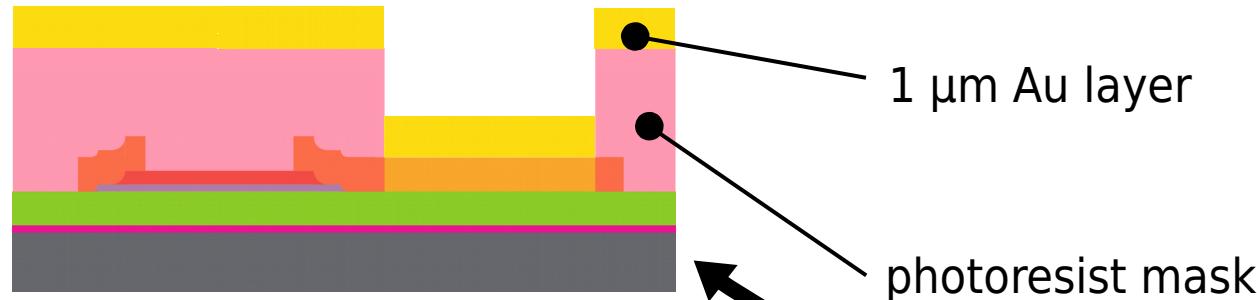
- $\tau_R \approx 3 \mu\text{s}$

- **Singular Value Decomposition**

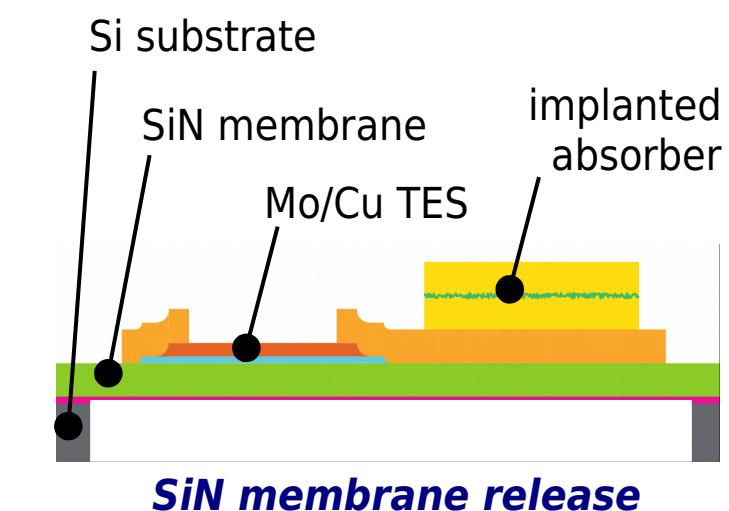
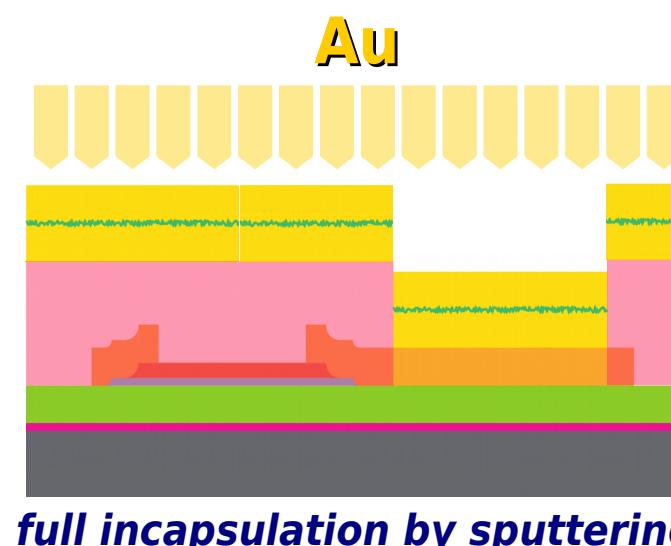
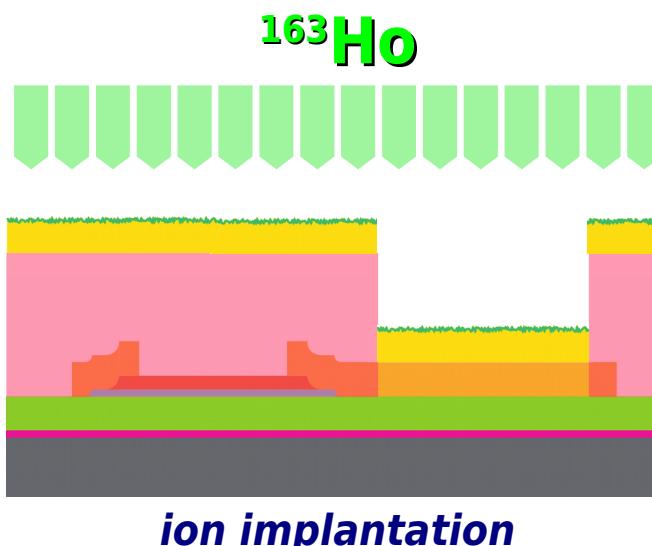
- $\tau_R \approx 1.5 \mu\text{s}$



HOLMES array design and fabrication



- TES array fabrication after first steps at **NIST**
- **^{163}Ho** implantation and final 1 μm **Au** layer deposition
- final micromachining step definition in progress
- **4×16 sub-array** for low parasitic L and high implant efficiency

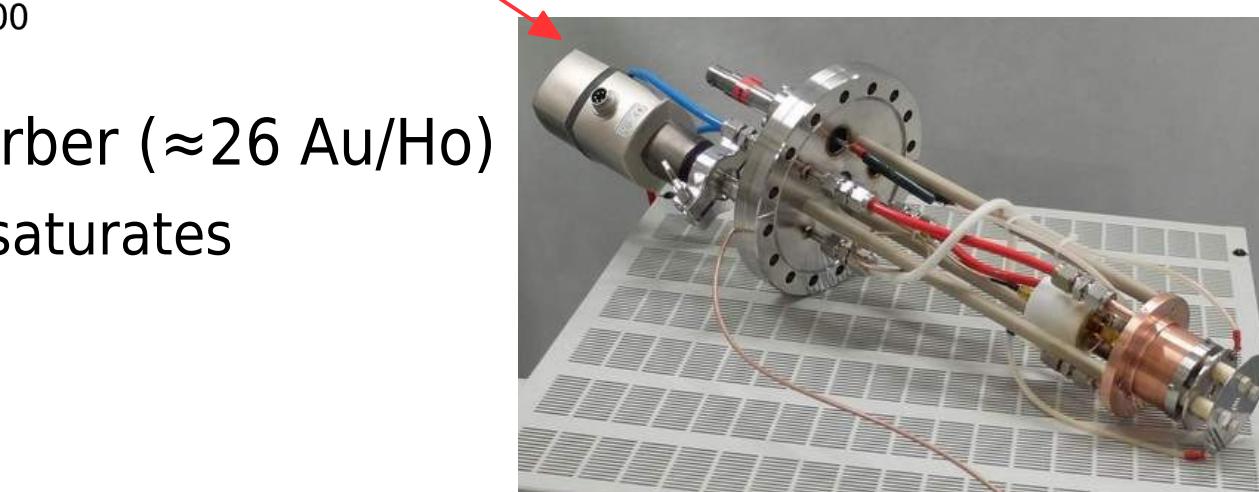
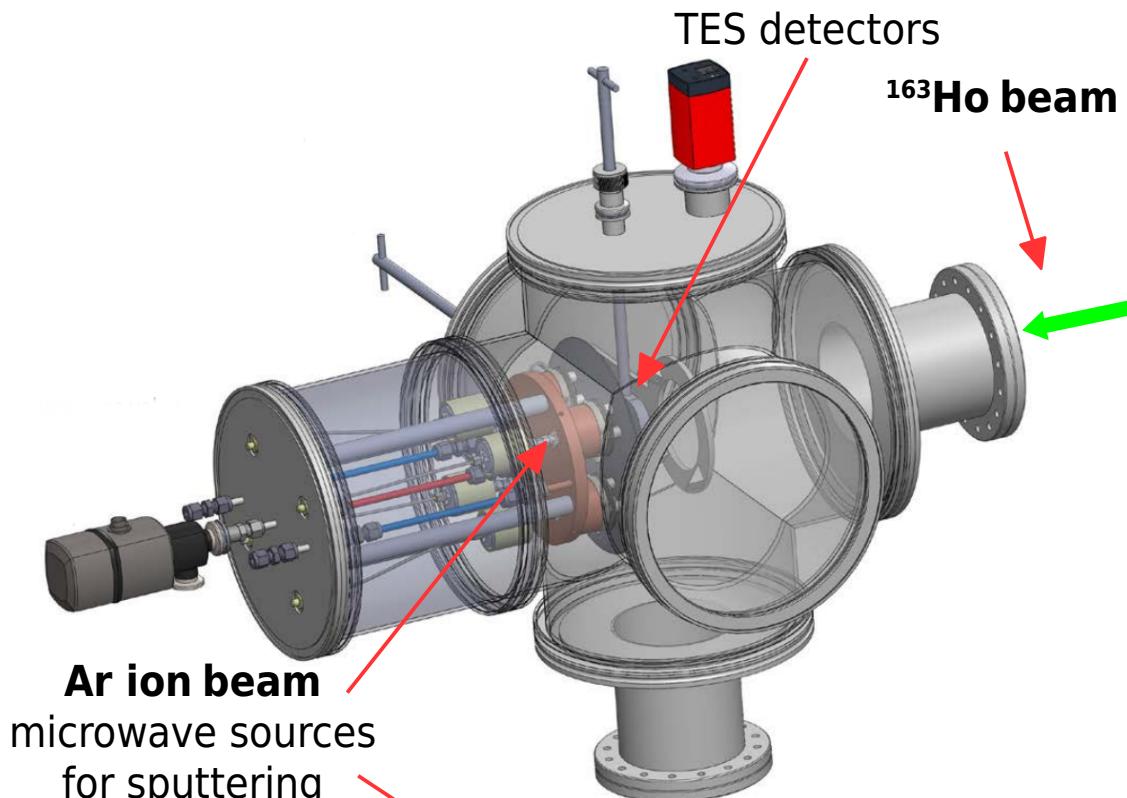
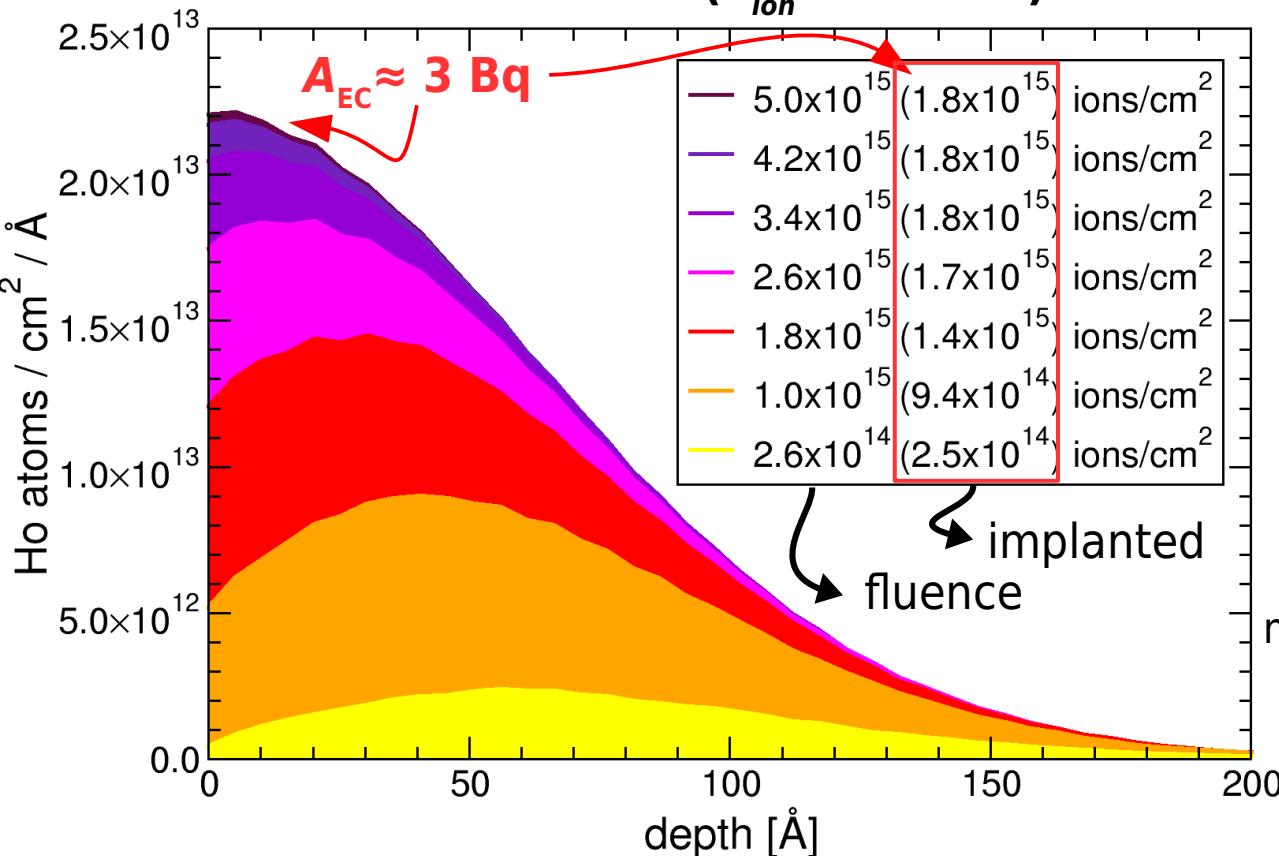


Target chamber for absorber fabrication / 1



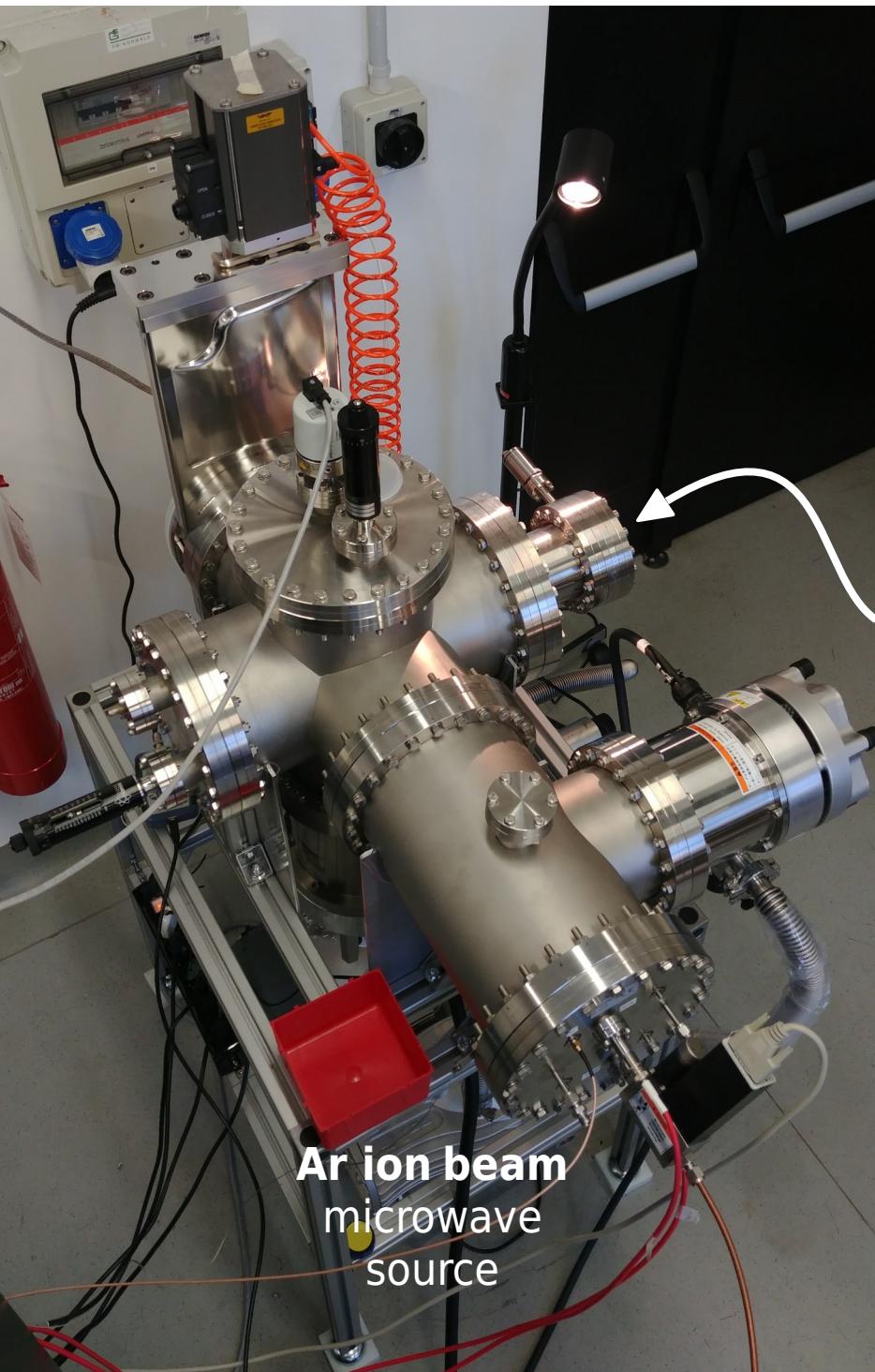
ion implant simulation with SRIM2013

^{163}Ho ions on Au ($E_{\text{ion}} = 50$ keV)

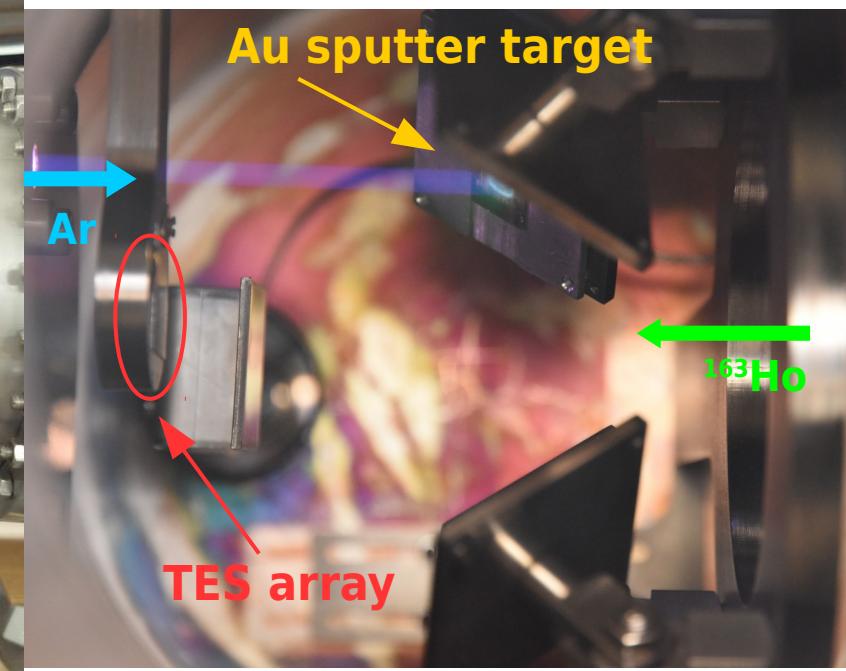
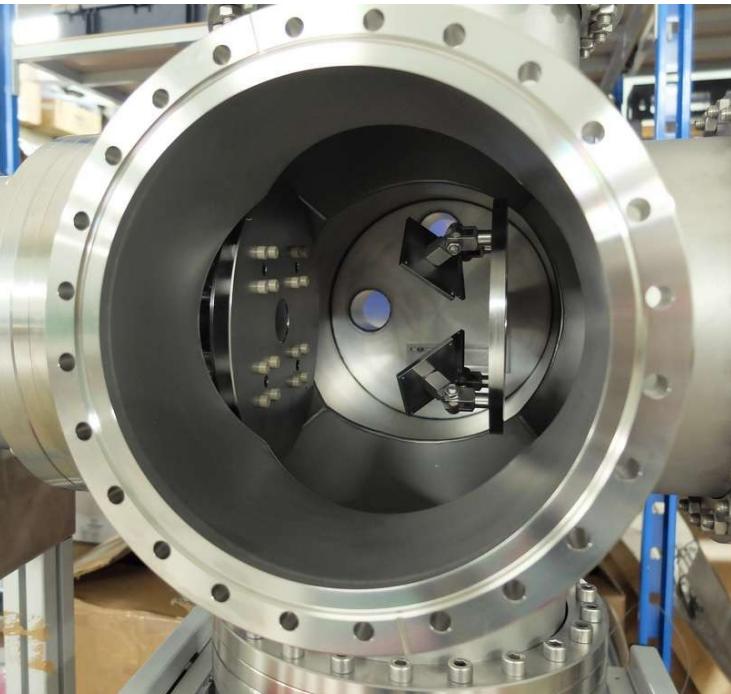


- ^{163}Ho ion beam sputters off Au from absorber (≈ 26 Au/Ho)
 - ▶ implanted ^{163}Ho concentration in absorber saturates
 - ▶ compensate by Au co-evaporation
- final 1 μm Au layer in situ deposition
 - ▶ to prevent Ho oxidization

Target chamber for absorber fabrication / 2



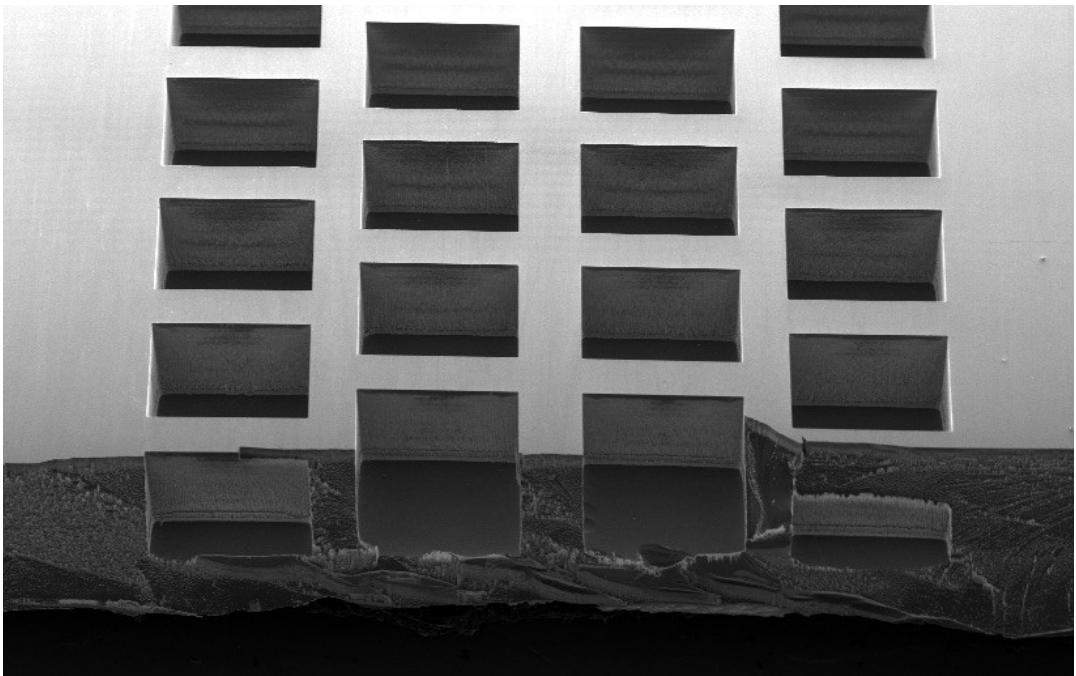
- Ion Beam Sputtering system for on-line deposition
- up to 4 ECR ion beam sources
- testing / optimization in progress with 1 ECR source
 - ▶ Au deposition rate control and maximization
 - ▶ Au film quality and uniformity characterization



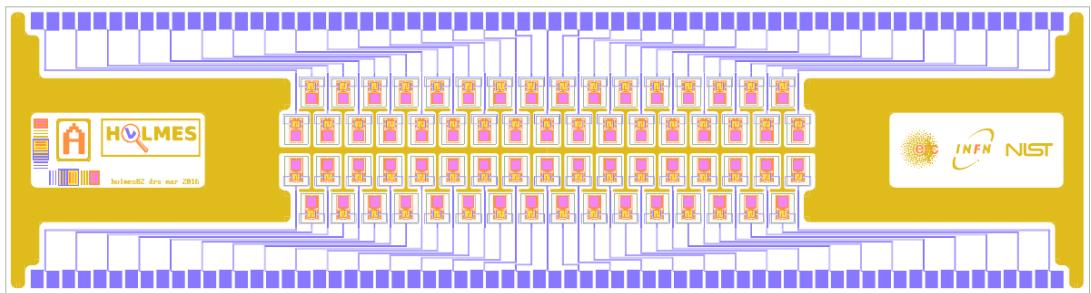
Detector array fabrication



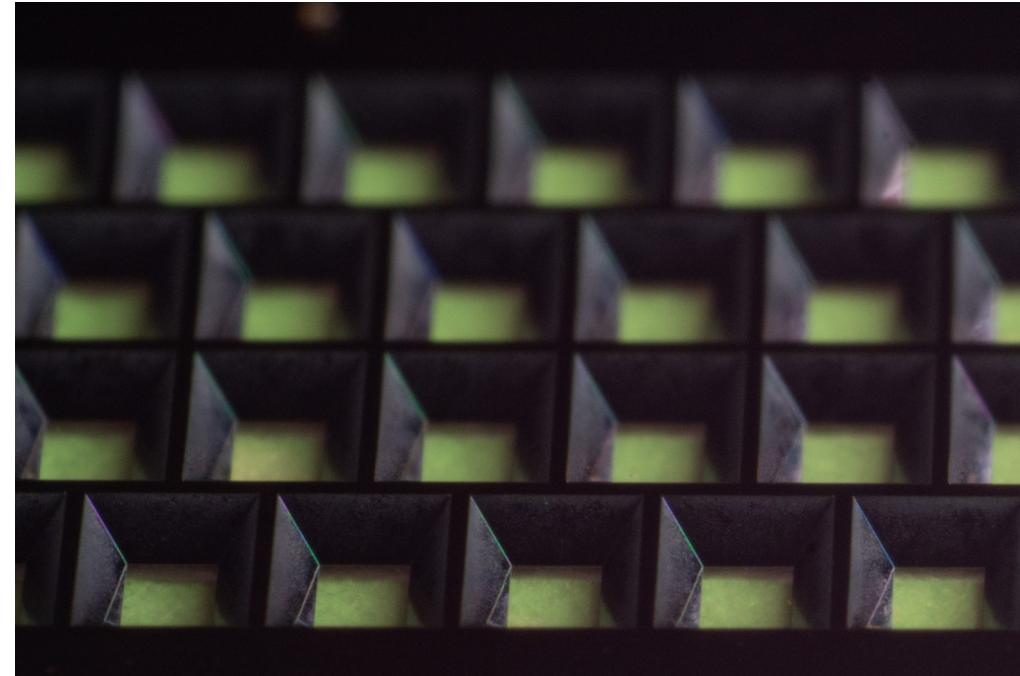
two options for membrane release (i.e. final array fabrication step)



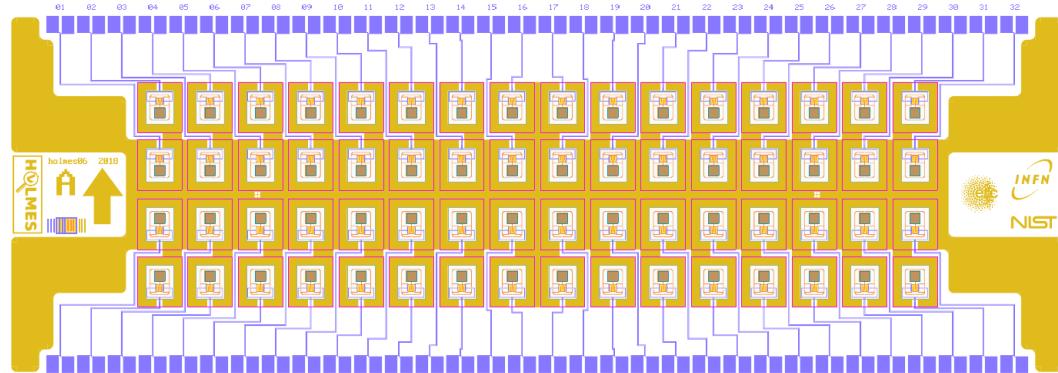
- Silicon Deep Reactive Ion Etching (DRIE)
- best for close packing and high implant efficiency
- not yet properly tuned → work in progress



A. Nucciotti, Holmes, NDM2018, 29 June - 4 July 2018, Daejeon, Korea



- Silicon KOH anisotropic wet etching
- requires more spacing between pixels
- successfully tuned → **HOLMES baseline**



Conclusions

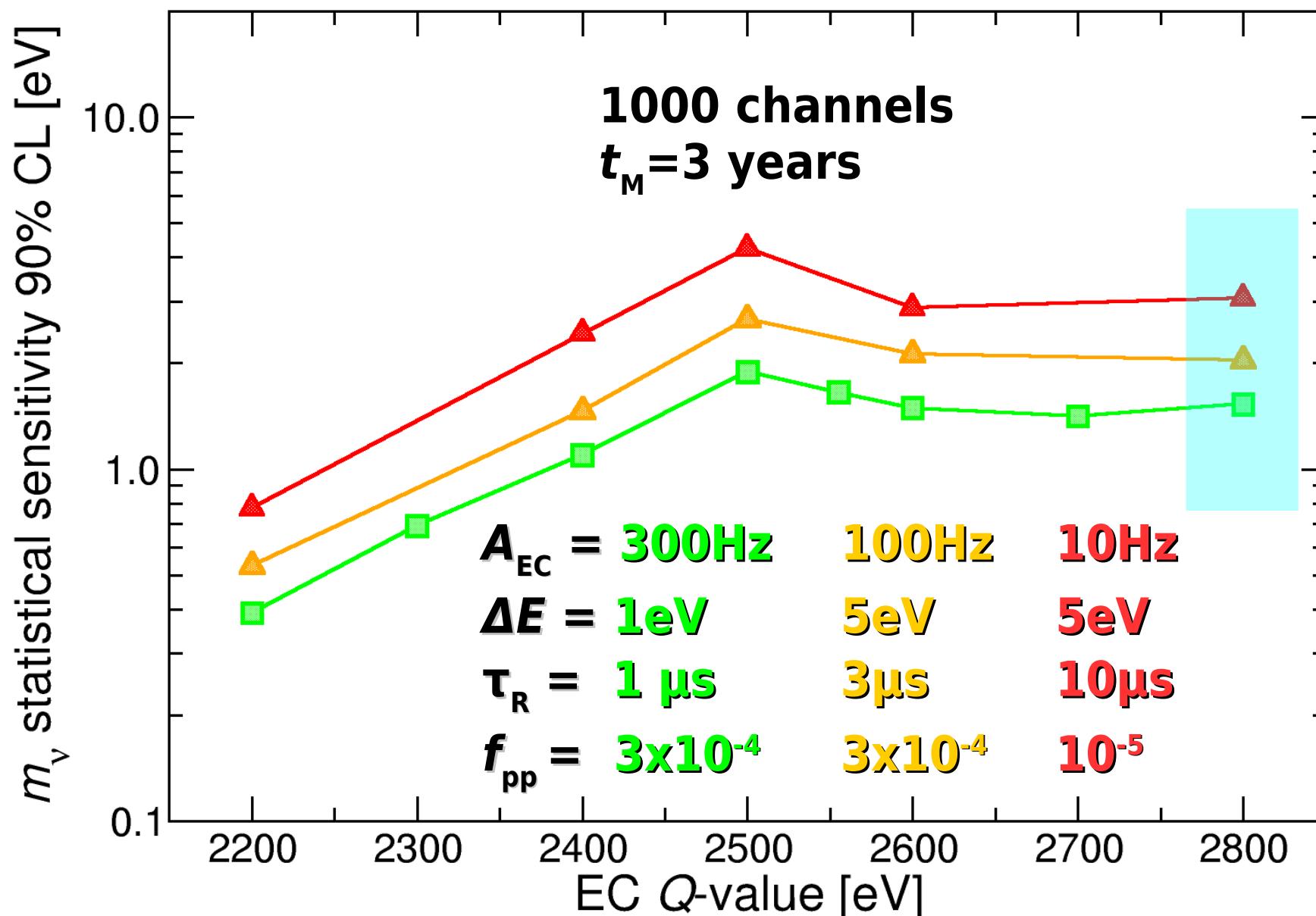


- first detector arrays are being fabricated at NIST
- first ion implantation tests with ^{163}Ho before the end of 2018
 - ▶ first not-optimized ion implanted detectors late in 2018
- ^{163}Ho implanted activity optimized during 2019
 - ▶ first high ^{163}Ho activity array running in 2019
 - ▶ 1 month data taking can provide a m_ν statistical sensitivity $\approx 10 \text{ eV}$
 - ▶ full array deployment will follow

Backup...



Worst case scenarios...



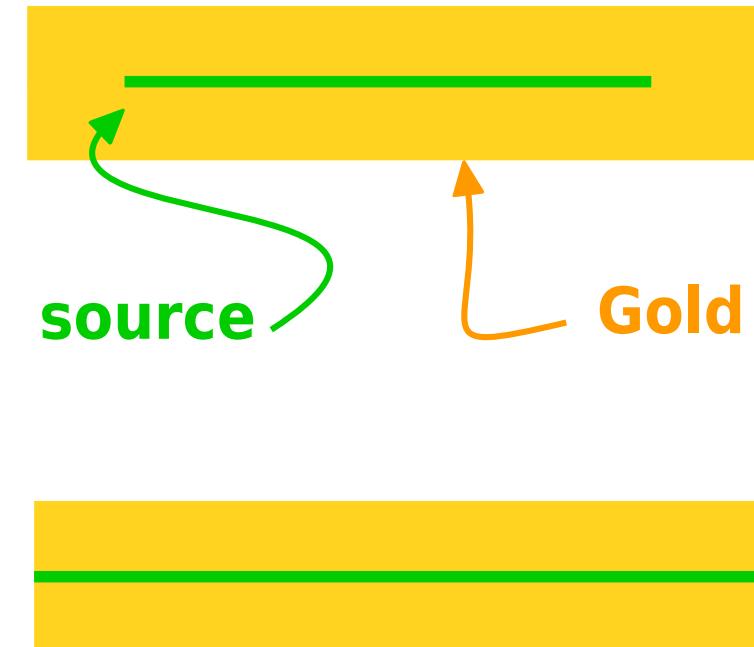
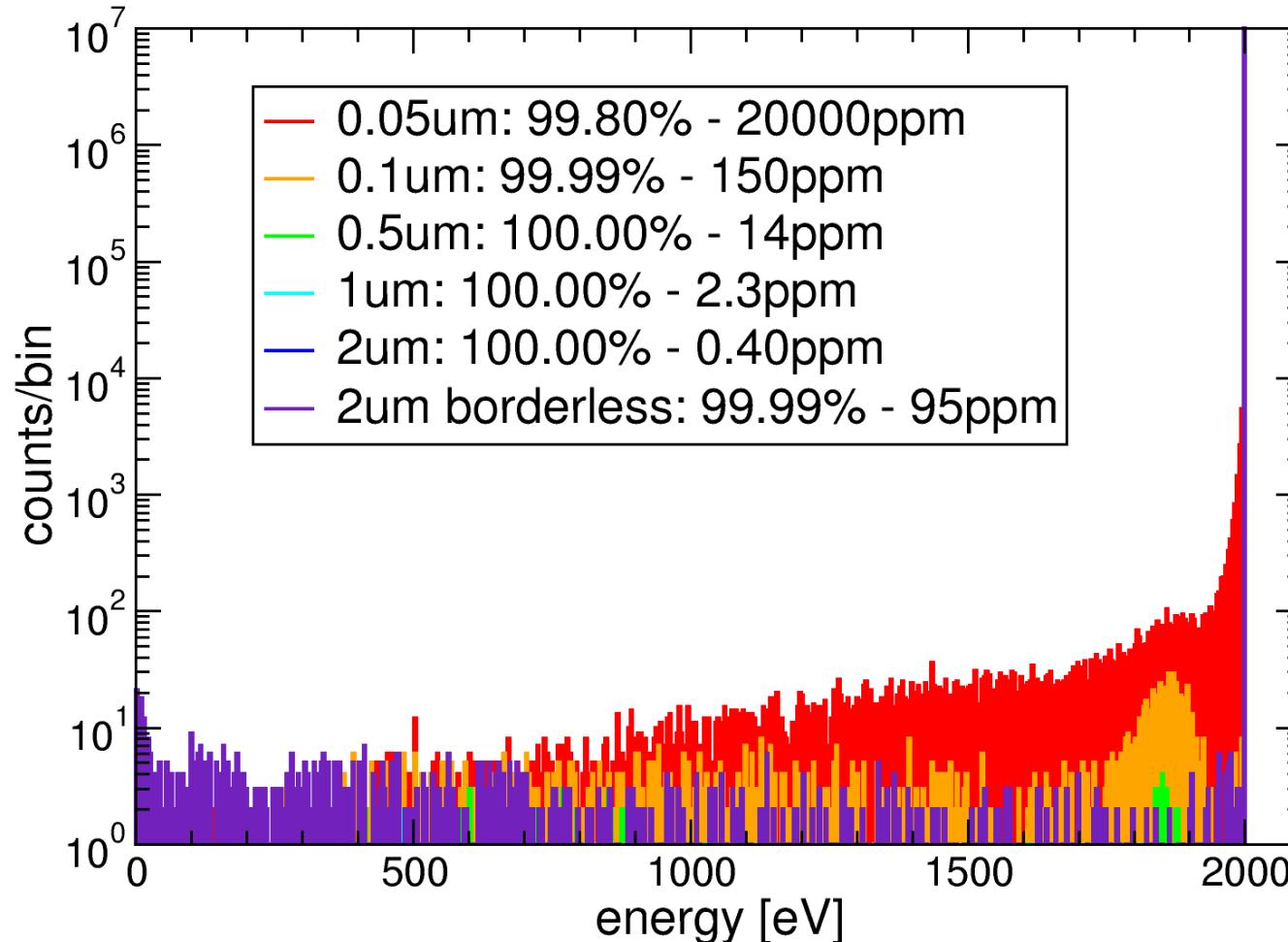
TES absorber design: stopping EC radiation / 1 b



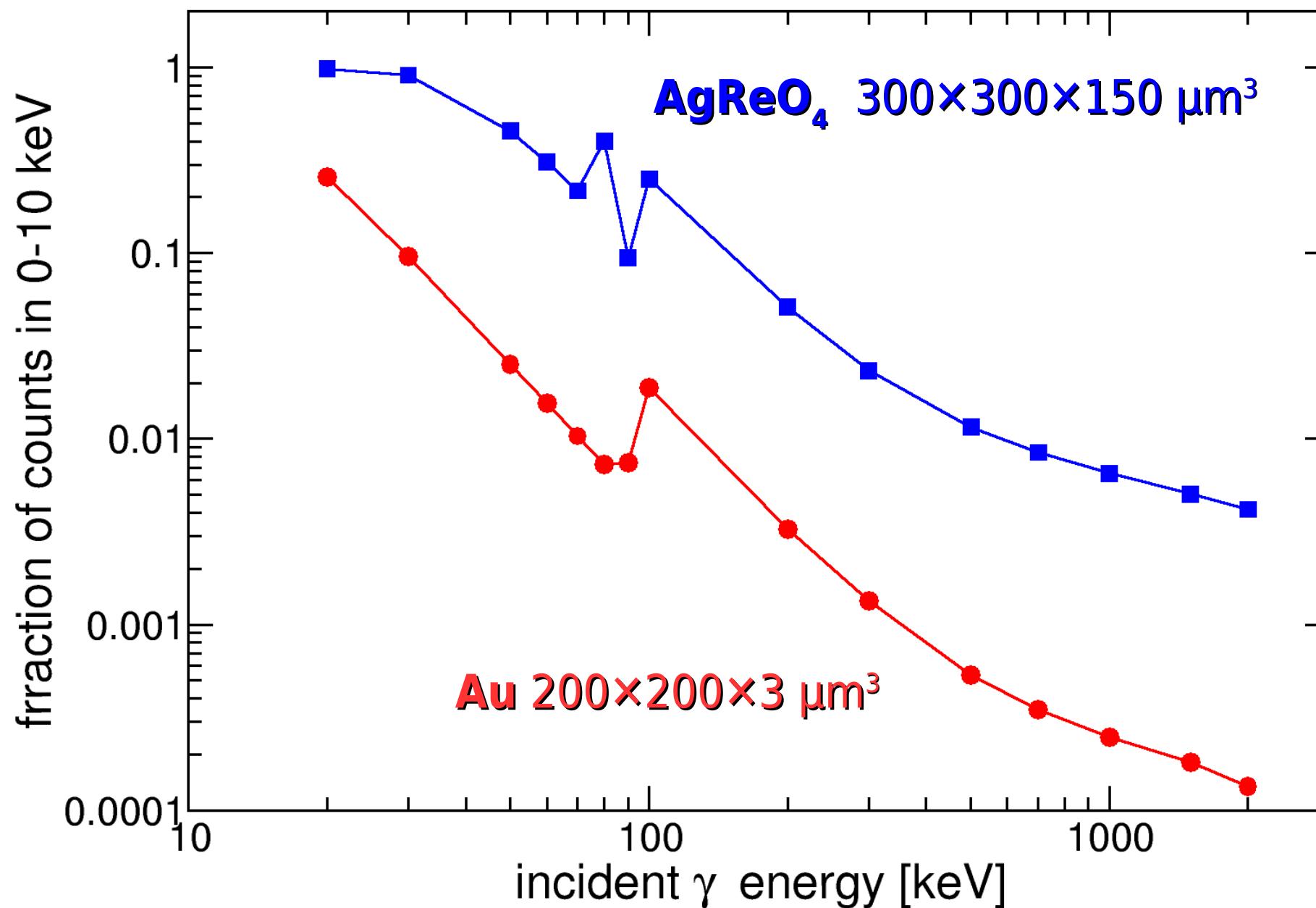
Geant4 + LowEnergyEM MC simulation

2keV electrons

full thickness: 0.05, 0.1, 0.5, 1, 2, 2 μm



Low energy background: γ sources



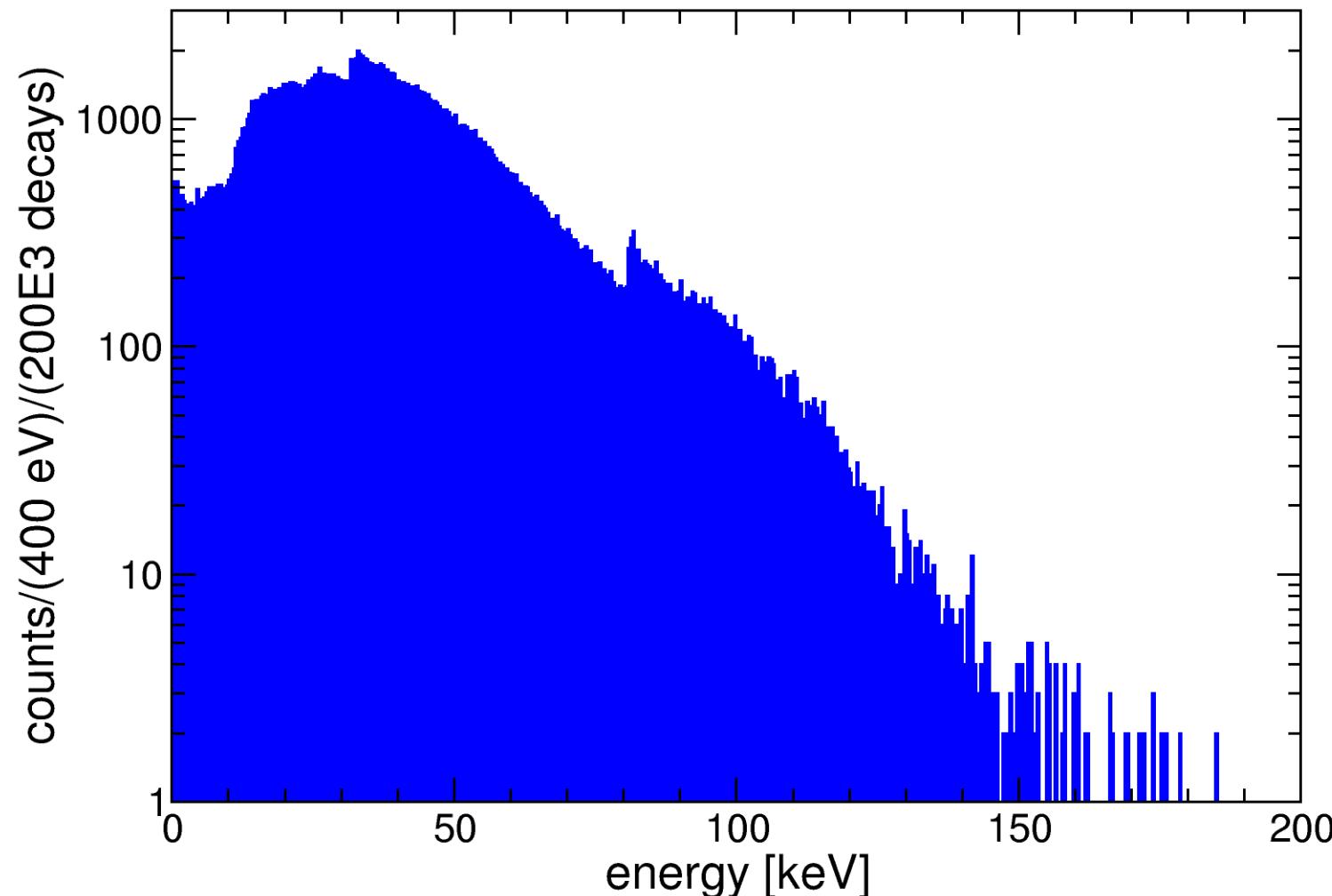
^{166m}Ho background

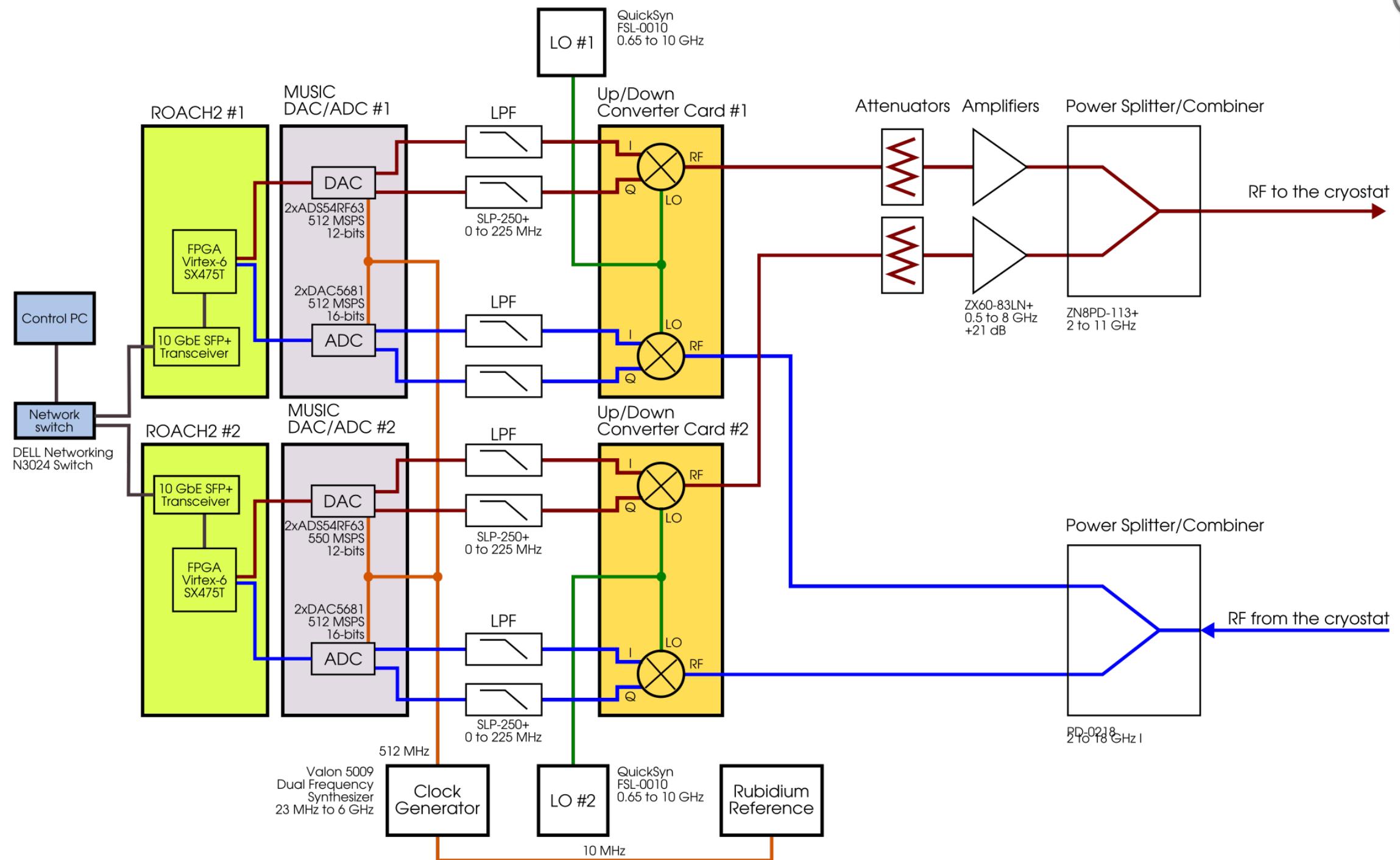


Geant4 + LowEnergyEM

$2 \cdot 10^5$ events

full
thickness
[μm]



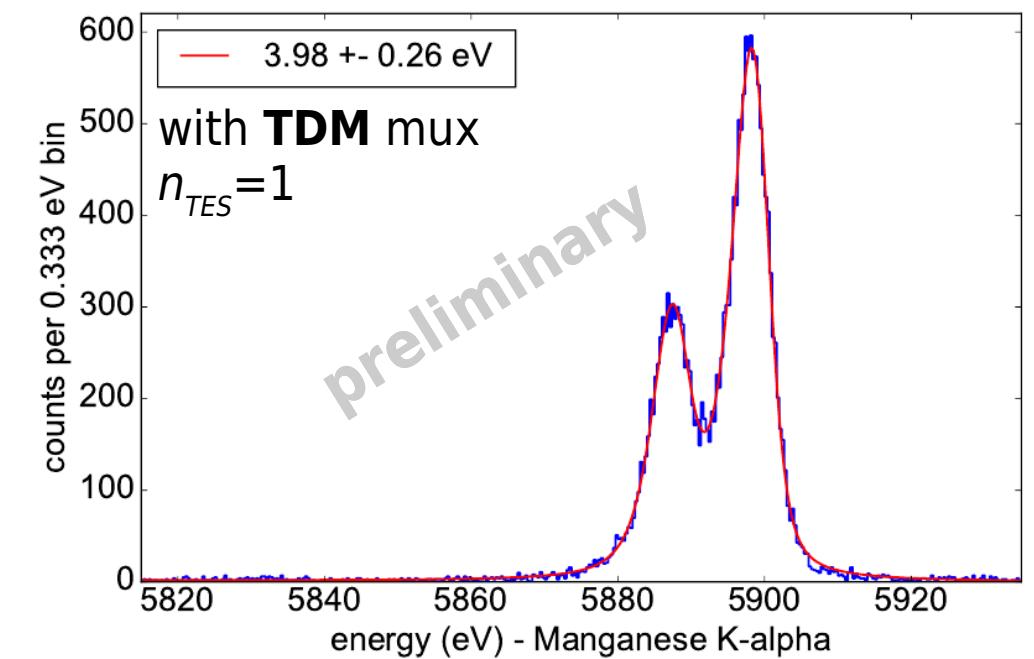
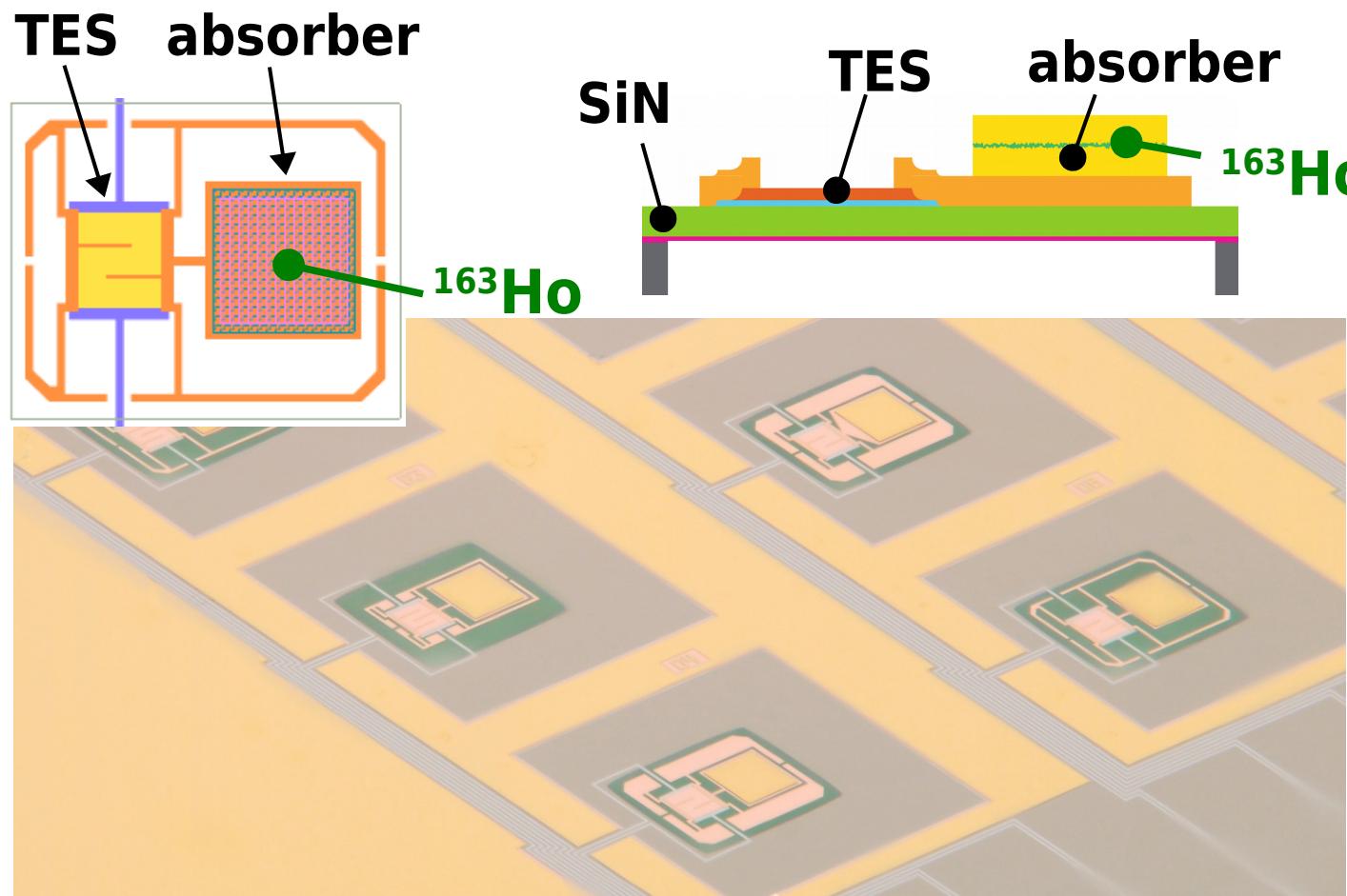


HOLMES pixel design and test



- optimize design for speed and resolution
 - ▷ specs @3keV : $\Delta E_{FWHM} \approx 1\text{eV}$, $\tau_{rise} \approx 10\mu\text{s}$, $\tau_{decay} \approx 100\mu\text{s}$
- 2 μm Au thickness for full electron and photon absorption
- side-car design to avoid TES proximitation and G engineering for τ_{decay} control

TES prototypes w/o ^{163}Ho : fabrication & test @ NIST



- ▷ $\Delta E_{FWHM} \lesssim 4\text{ eV}$ @ 6 keV ($\rightarrow \approx 3\text{ eV}$ @ Q_{EC})
- ▷ $\tau_{rise} \approx 6\ \mu\text{s}$ (with $L=38\text{nH}$ \rightarrow to be slowed)
- ▷ $\tau_{decay} \approx 130\ \mu\text{s}$ (still tunable)