Developments for low background experiments

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GDR DI2I

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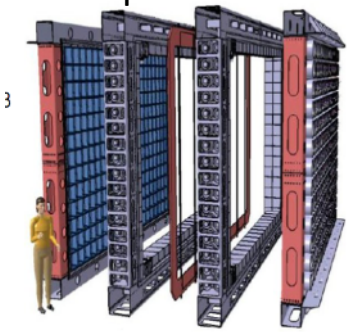
What means Low background experiments

- Looking for rare events:
  - Fondamental Physics
    - Proton decay
    - Nuclear structure
    - Neutrinon double beta decay ßßv0
    - Dark matter
  - Other disciplines
    - Low radioactivity measurement
    - Logical test failure in nano/micro electronics
    - Qubits (Quantum computing)
    - Biology
- Protected from any irradiation
  - Cosmic rays
  - Natural irradiation
  - Radon
- Located in the Underground Laboratory (UL)
- Experiment components are radiopure

Example of the experiments

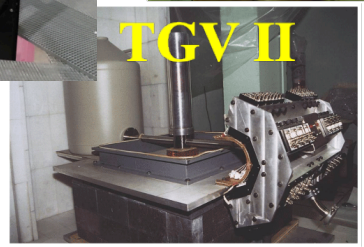
- Fondamental Physic

SuperNemo

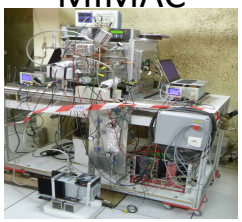


EDELWEISS

NEWS_G (SEDINE)

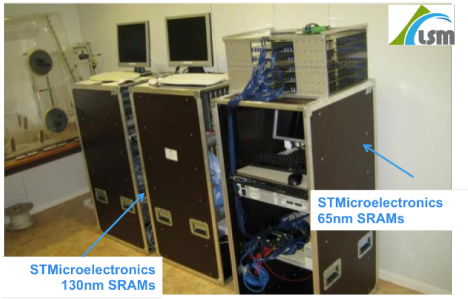


MIMAC



DAMIC_M

- Other disciplines



Gamma spectrometry

- Then …… TESSERACT ; Qubits

Common part of LB experiments

- Material selection
- Low radioactive components such as:
  - Electronics components
  - Shielding
    - ==> Standard Lead
    - ==> Ancient Lead
  - Detector structures/support
    - ==> **Copper mainly**
      - Commercial production ≈ 5N (99.999%) ≈ 0.1-10 mBq/kg
      - **Electroform Copper** ≈ 6N (99,9999%) ≈ 10-100 nBq/kg

Electrolytic Cell and Electroplating

- Electrolytic cell driven by **oxidation and reduction** reactions
- Current supplied to drive reactions
- Desired result is generally **the reduction of ions** from the electrolyte to form atoms on the cathode surface
- Mass deposited proportional to current supplied:

$$M = \frac{m_r \int I(t)\,\mathrm{d}t}{zF}$$

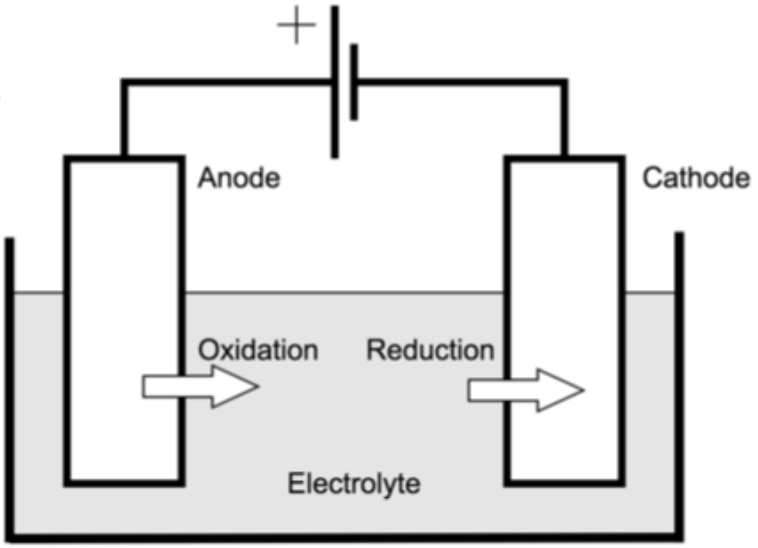
M – mass

m_r – molar mass

$I(t)$ – current as function of time

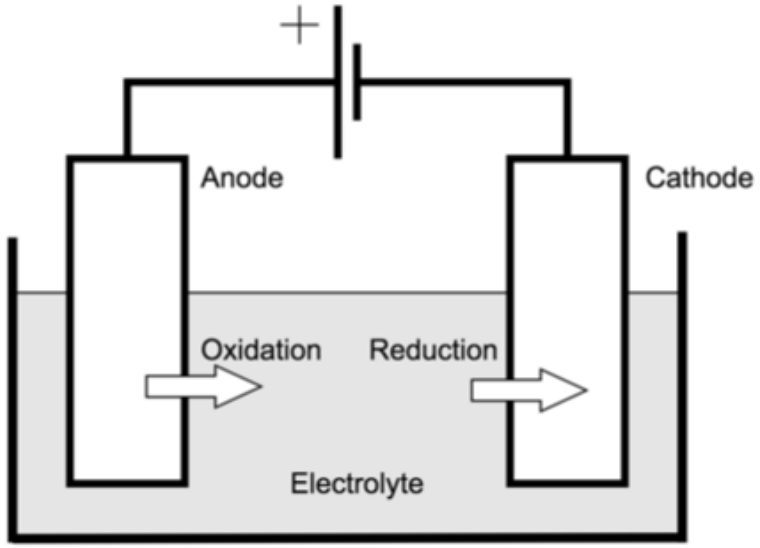
z – number of electrons transferred in reduction reaction

F – Faraday Constant ($= e\,N_A$)



Electroplating Copper

- Some ions reduce more readily than others – **reduction potentials**
- Voltage between anode and cathode limits **electroplating of some species**
- Copper benefits from '**electrowinning**' – high reduction potential +0.34 V
- Reduction potential of:
  - Uranium: -1.80 V
  - Thorium: -1.90 V
  - Lead: -0.44 V



- **All lower than copper;** refined during electroplating

Why don't impurities plate too?

- Which reaction proceeds determined by standard **cell potential**:

$$E^0_{cell} = E^0_C - E^0_A$$

E^0_{cell} - standard cell potential

E^0_C - standard cathode reduction potential

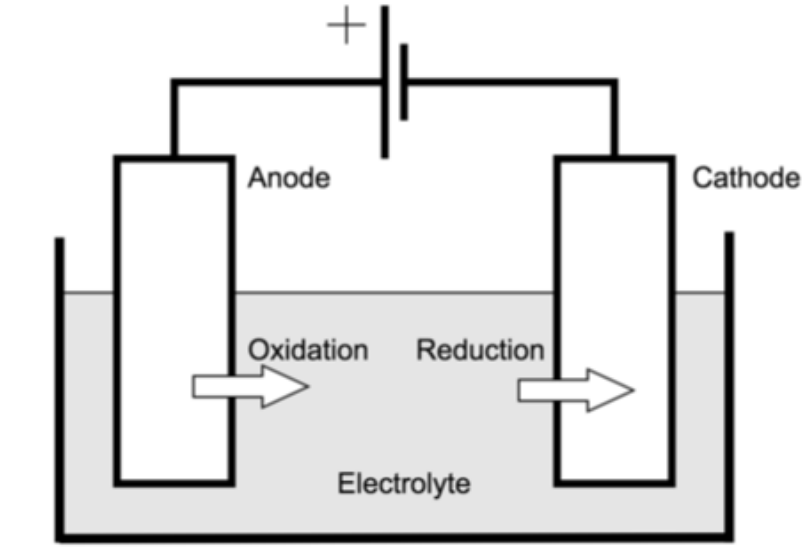
E^0_A – standard anode reduction potential

- Related to Gibbs Free Energy:

$$\Delta G^0 = -z\,F\,E^0_{cell}$$

  - If $\Delta G^0 < 0$ reaction is spontaneous

  - If $\Delta G^0 > 0$ need extra energy input



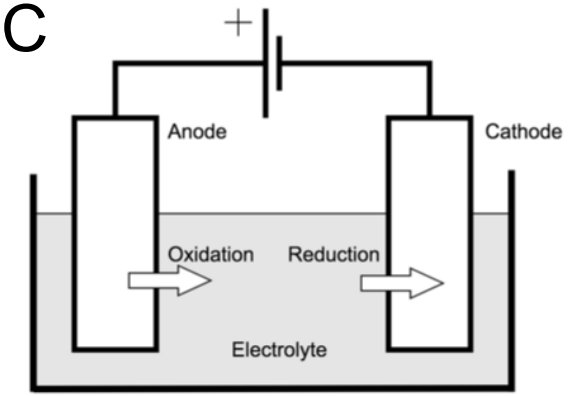
An Example

E^0_{cell} - standard cell potential

E^0_C - standard cathode reduction potential

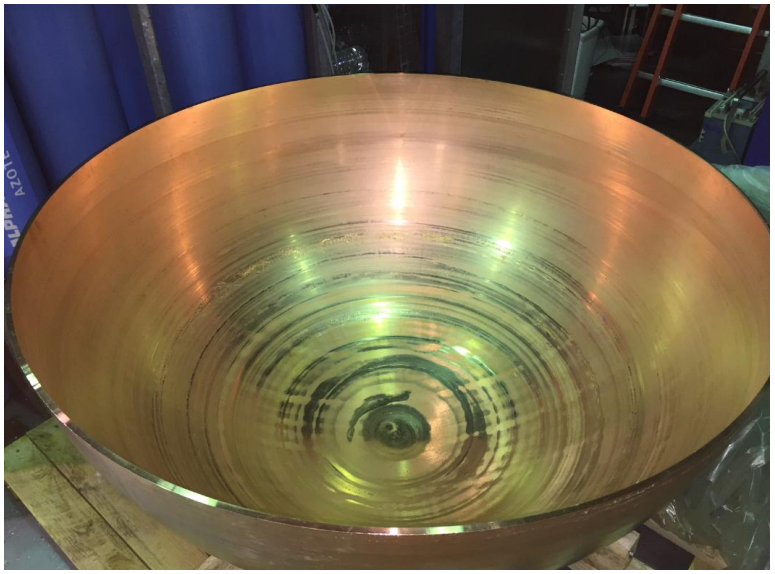
E^0_A – standard anode reduction potential

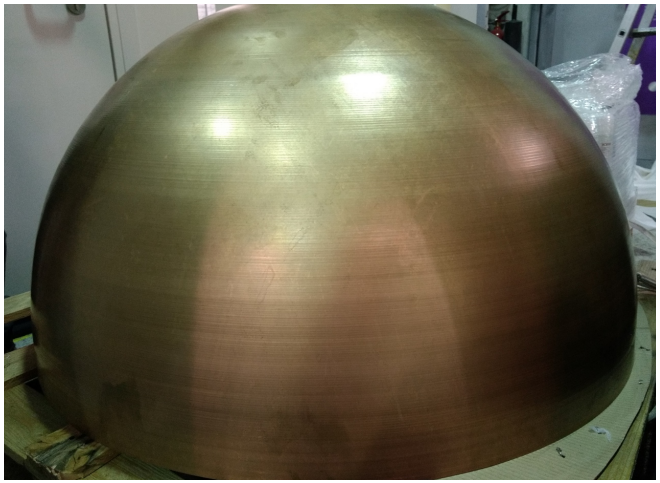
- Example of solution containing U^{3+} and Cu^{2+}, with a Cu anode:
  - U^{3+} to U
    - E_Cell= -2.138 V → Requires energy Cu^{2+} to C
  - Cu^{2+} to Cu
    - E_Cell= 0 V → In equilibrium
- **Cu^{2+} reduction** will occur at lower potential
- Still require a potential difference between electrodes to **overcome energy losses**



Detector Hemispheres

- Two 140 cm diameter hemispheres
- 4N copper (99.99% pure)
- Plates purchased and then spun into hemispheres
- 110 cm diameter hemisphere also spun as the anode for electroplating





Preparation Procedure

- Cleaned with domestic detergent
- **Sanded** – different sanding for each hemisphere – but final stage with silicon carbide paper
- Estimated ~10-100 μm removed by sanding (~500 μm second hemisphere)
- Cleaned with **Micro90** detergent (1%) in deionised water
- Inner hemisphere received same treatment for first use – just Micro90 second time

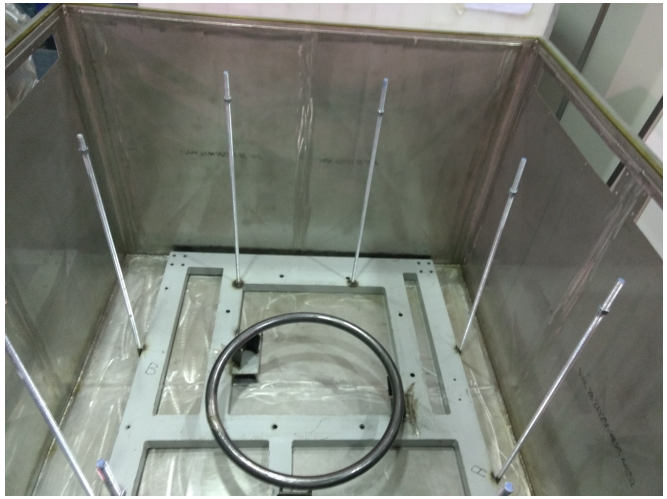
Chemical Etching

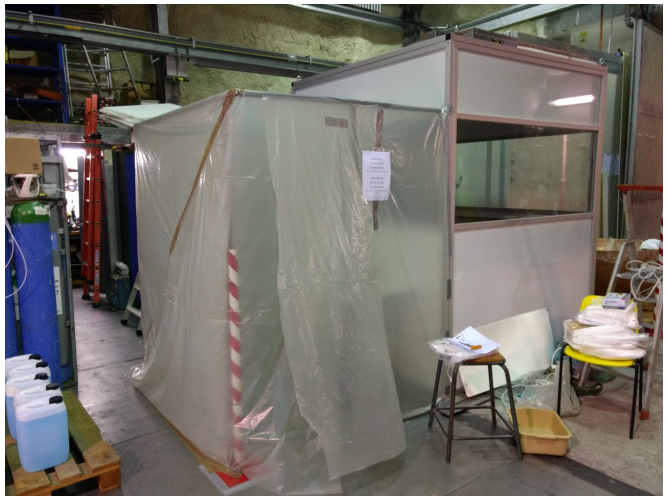
- Surface **chemically etched** with 3 % H_2O_2, 1 % H_2SO_4 in deionised (DI) water
  - Shown to be effective etchant while less aggressive than some alternatives

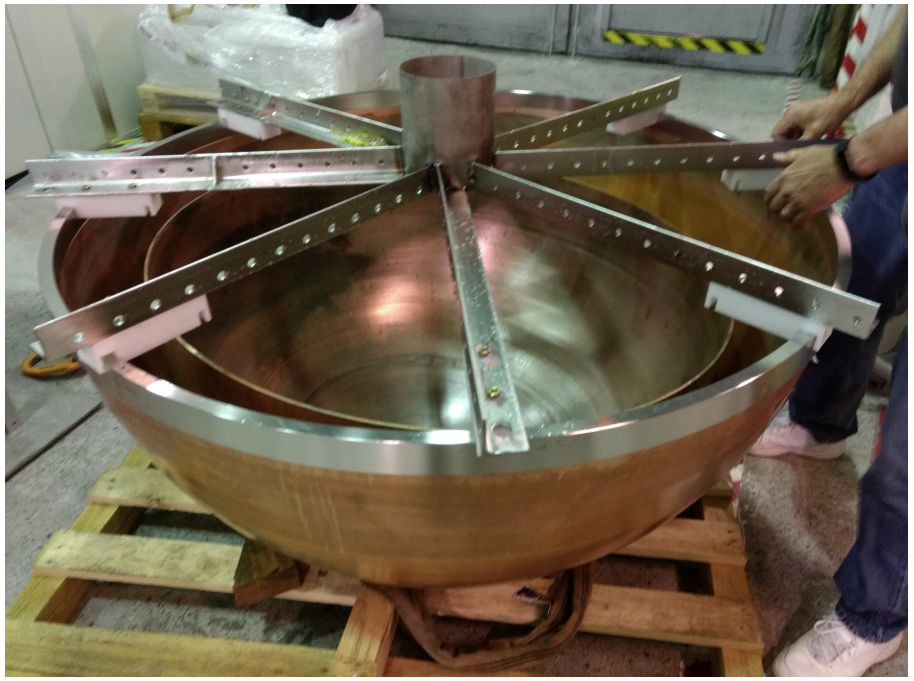
-To remove any trapped particulates from spinning process or sanding, and make surface more smooth

- Estimate **5-10 µm** removed during etching

Cleanroom



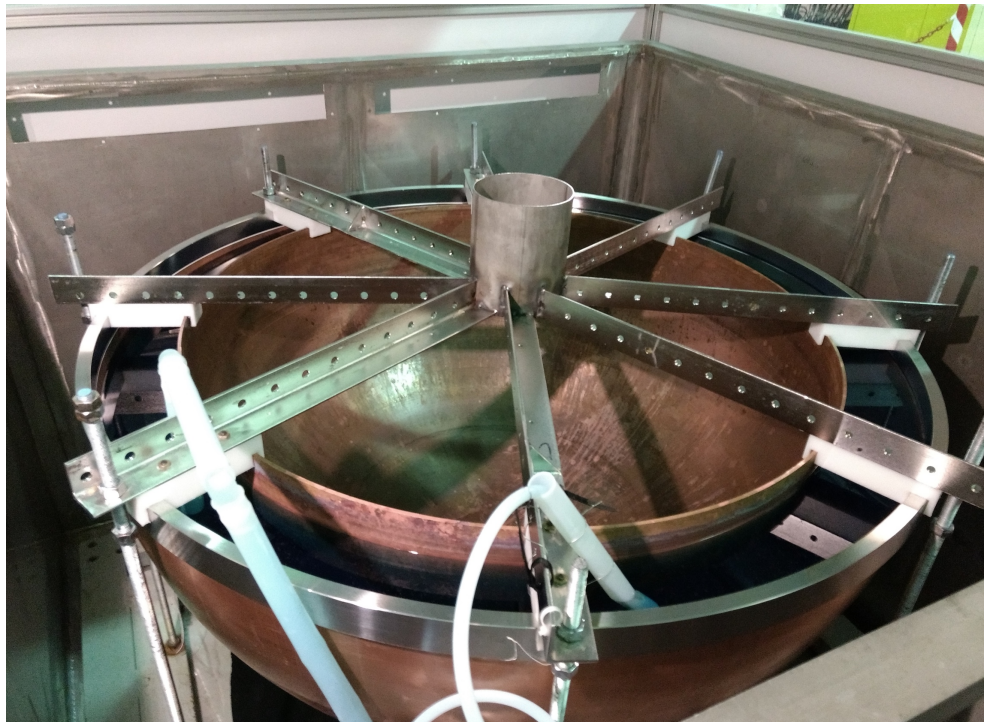




Process carried out in cleanroom to minimise contamination and particulates

Electrolyte

- First hemisphere filled with 80 l electrolyte from SEDINE electroplating, 5.5 l H_2SO_4 and topped up to ~300 l with DI water
- Second hemisphere used ~275 l first-hemisphere electrolyte, 2 l H_2SO_4 and up to ~300 l with DI water
- Both have some $CuSO_4$ in solution already
- **Pump and filter** used to move electrolyte and remove particulates

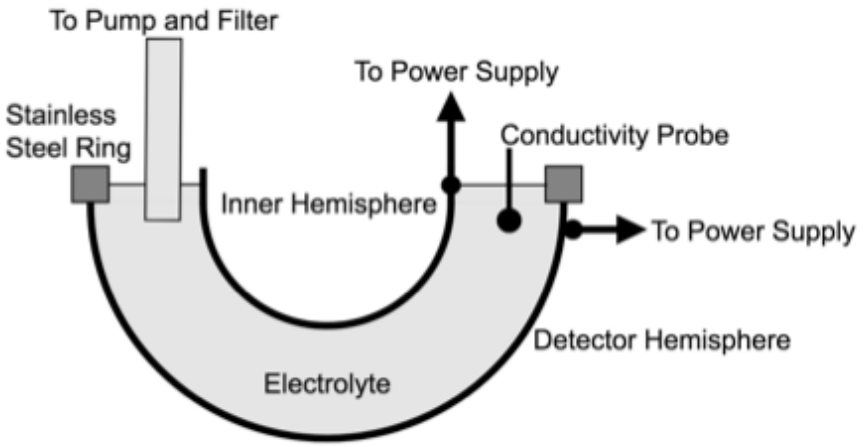


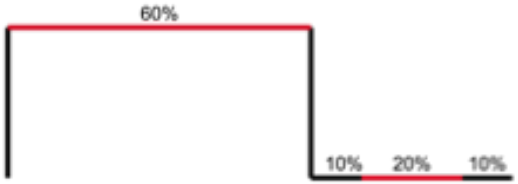
Electropolishing

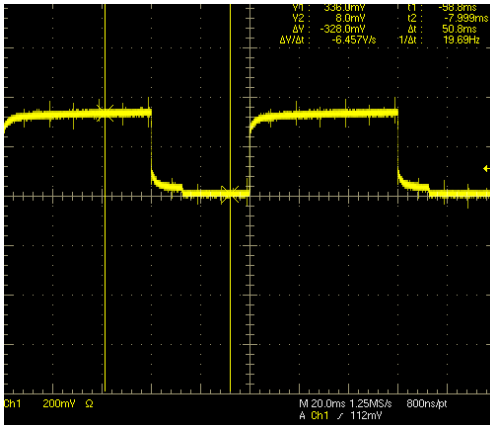
- Electropolishing serves several purposes
  - Removes layer from hemisphere without chemical or mechanical attack
  - Preferentially **removes high spots** from surface
  - **Increases concentration** of CuSO4 in electrolyte
- **Waveform** used to polish
  - **+ve = detector hemisphere**

voltage>inner hemisphere

- First (second) hemisphere 21.2±0.1 μm (28.2±0.1 μm) polished





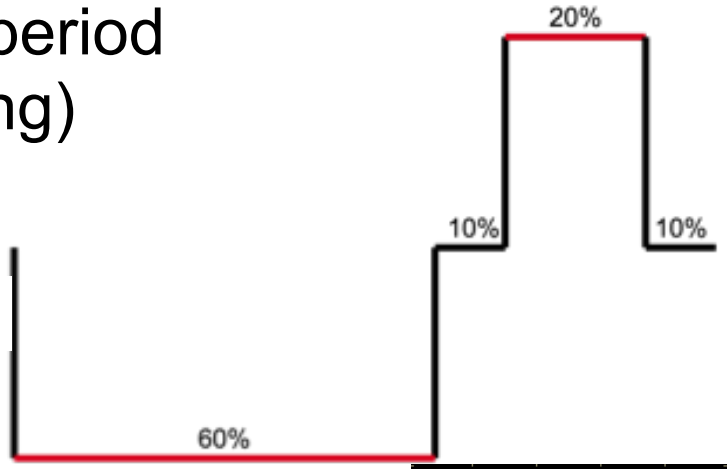


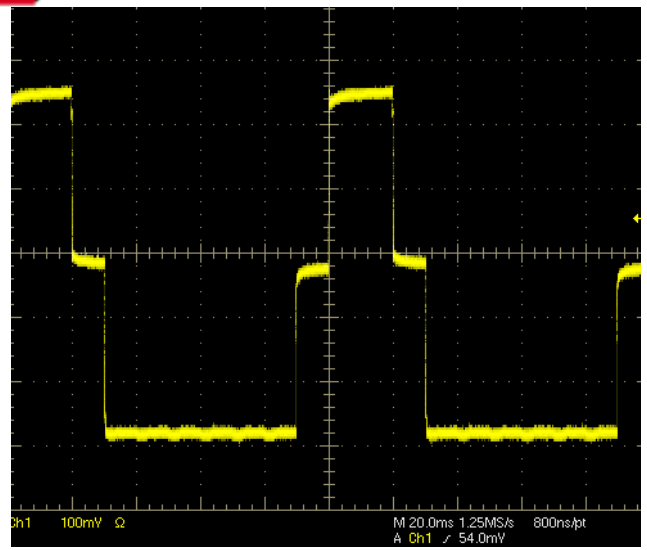
Why Pulse-Reverse Plating?

- Waveform for plating with relaxation period and/or reverse bias (effectively polishing)

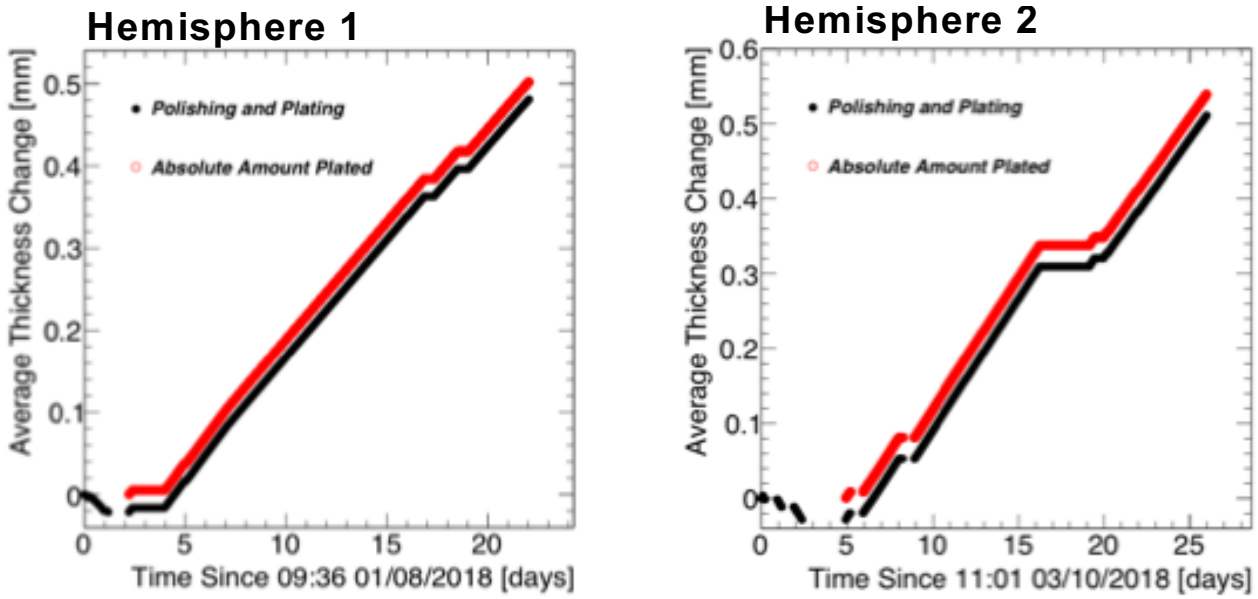
- Benefits compared to DC plating:

  - – **High points** are preferentially **removed** in reverse bias section
  - – Greater **uniformity** of plate – relaxation period allows diffusion of ions
  - – Higher density copper plate





Electroplating



- Plated for approximately 20 days for both hemispheres
- 0.27 to 0.3 V amplitude in forward and reverse directions of plating
  - Established value for copper plating
- In total first (second) hemisphere plated 502.1 ± 0.2 μm (539.5 ± 0.2 μm)

Results

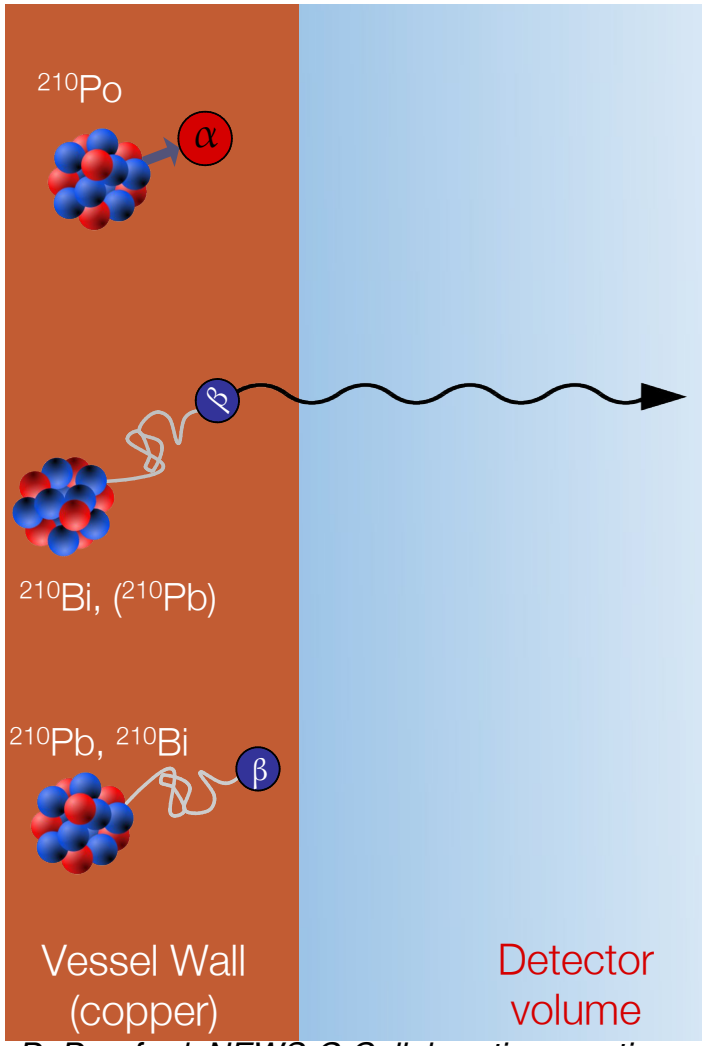
- Drained electrolyte and rinsed surface with DI water
- Passivated with 1% $C_6H_8O_7$ in DI water for 30 min-1 hour
  - Prevents further oxidisation
- Rinsed again with DI water
- Stored in plastic ready for electron beam welding

- First hemisphere had many nodules – frequent and 0.5-1.5 mm in height
- Second hemisphere had very few nodules

Hemispheres 1st / 2nd

- Second hemisphere has less nodules. Main difference with first hemisphere:
  - **Higher initial $CuSO_4$** concentration – already used for one hemisphere
  - But also:
  - **More sanding** – ensure uniform surface before beginning
  - **Pump outlet moved** daily – improve mixing and spread excess-plating
  - Improved electrical isolation from container
  - Maybe higher H_2SO_4 concentration (to be determined by analysis)
  - Solution filtered for longer before beginning and between polishing and plating
  - Inner hemisphere had layer removed from polishing already

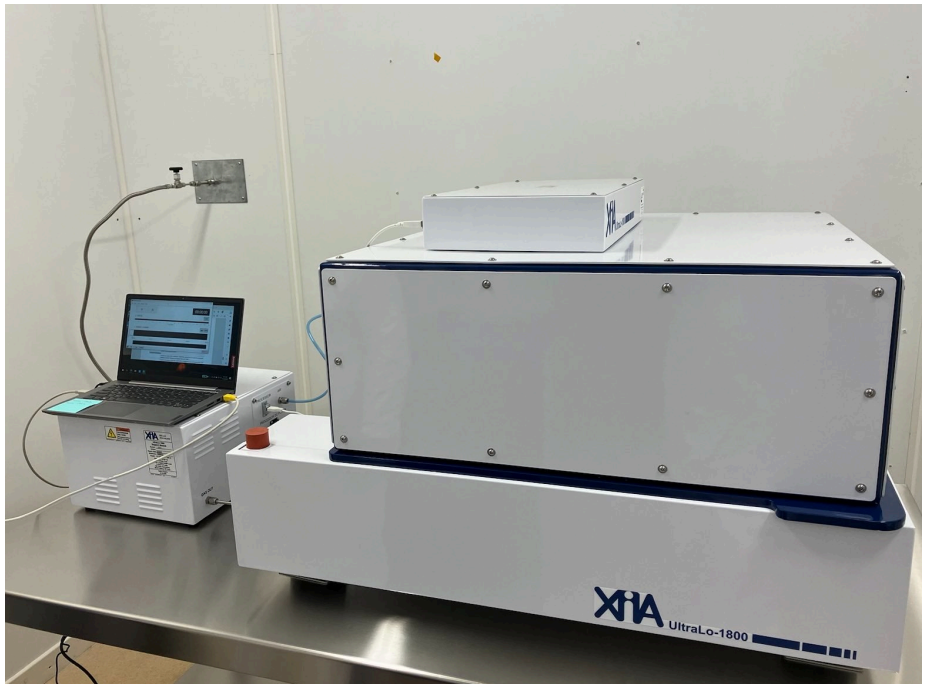
Then, surface contamination

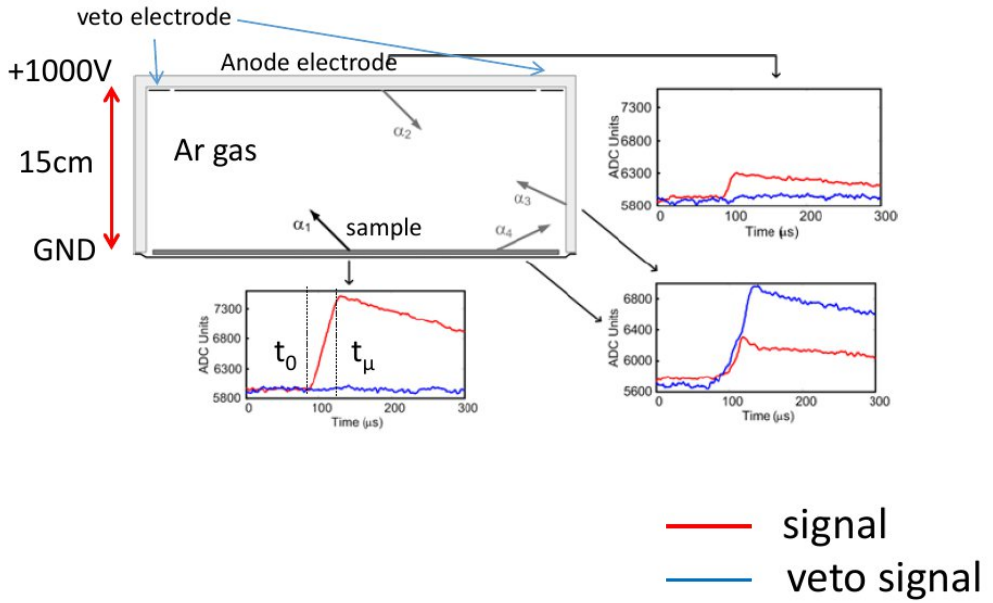


- 210Pb is a long-lived radio-impurity found in copper
- Most radiation is stopped inside the copper but…
  - Bremsstrahlung x-rays (~keV) from 210Pb and 210Bi β- decay in the copper escape, travel through whole volume

- *D. Durnford. NEWS-G Collaboration meeting*

XIA, Ultralo-1800 Alpha counter

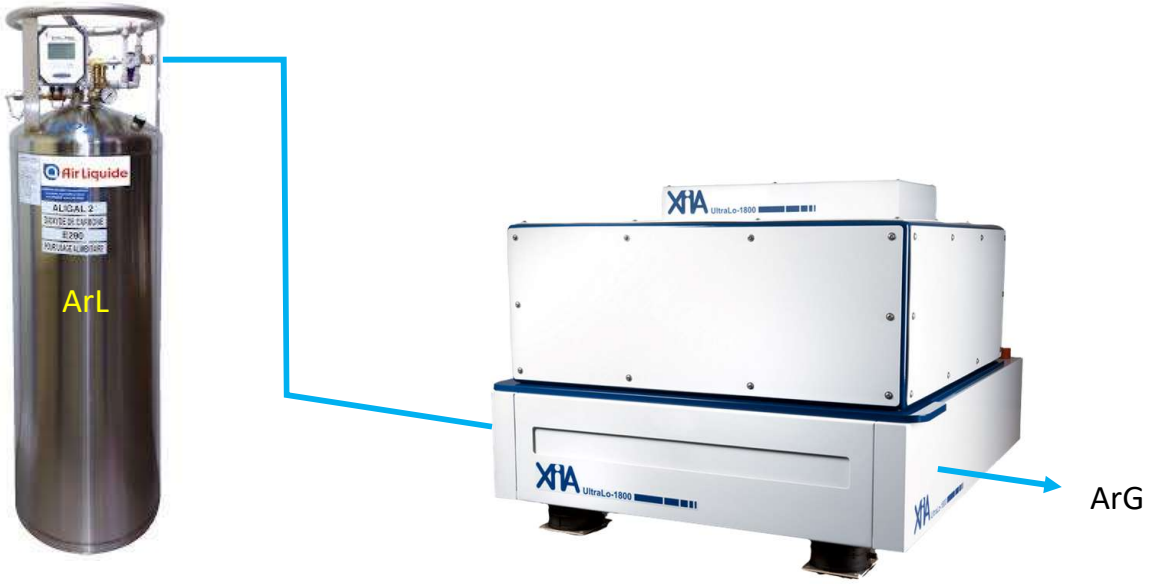




- Installation in a Cleanroom @ LPSC for commissioning run
- Final installation @ LSM

- By measuring rise time (t_μ - t_0), surface event can be distinguished from event in Ar gas or celling

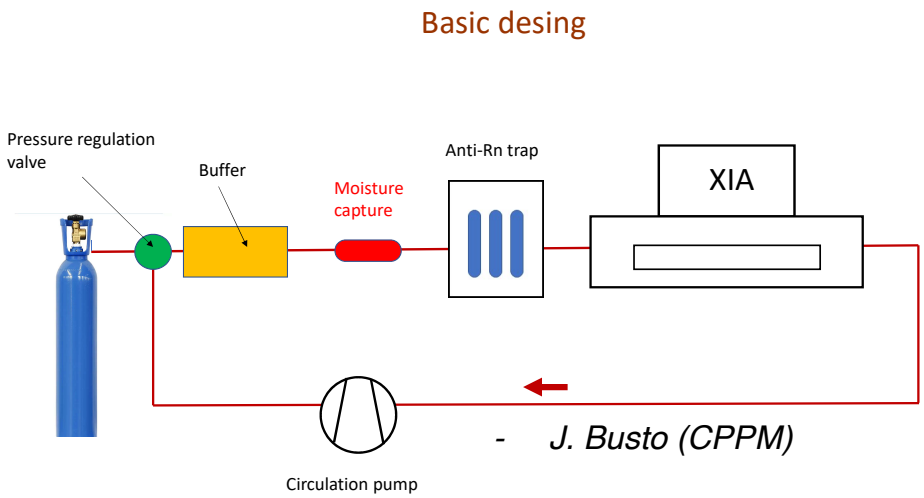
How XIA operate



Flushing : ~ 700 L (45 min)
Mesurement : ~300 L/ h

Proposition : replace Liquid-Ar by Gas-Ar and operate in a closed circuit with an anti-Rn purification system and very low moisture.

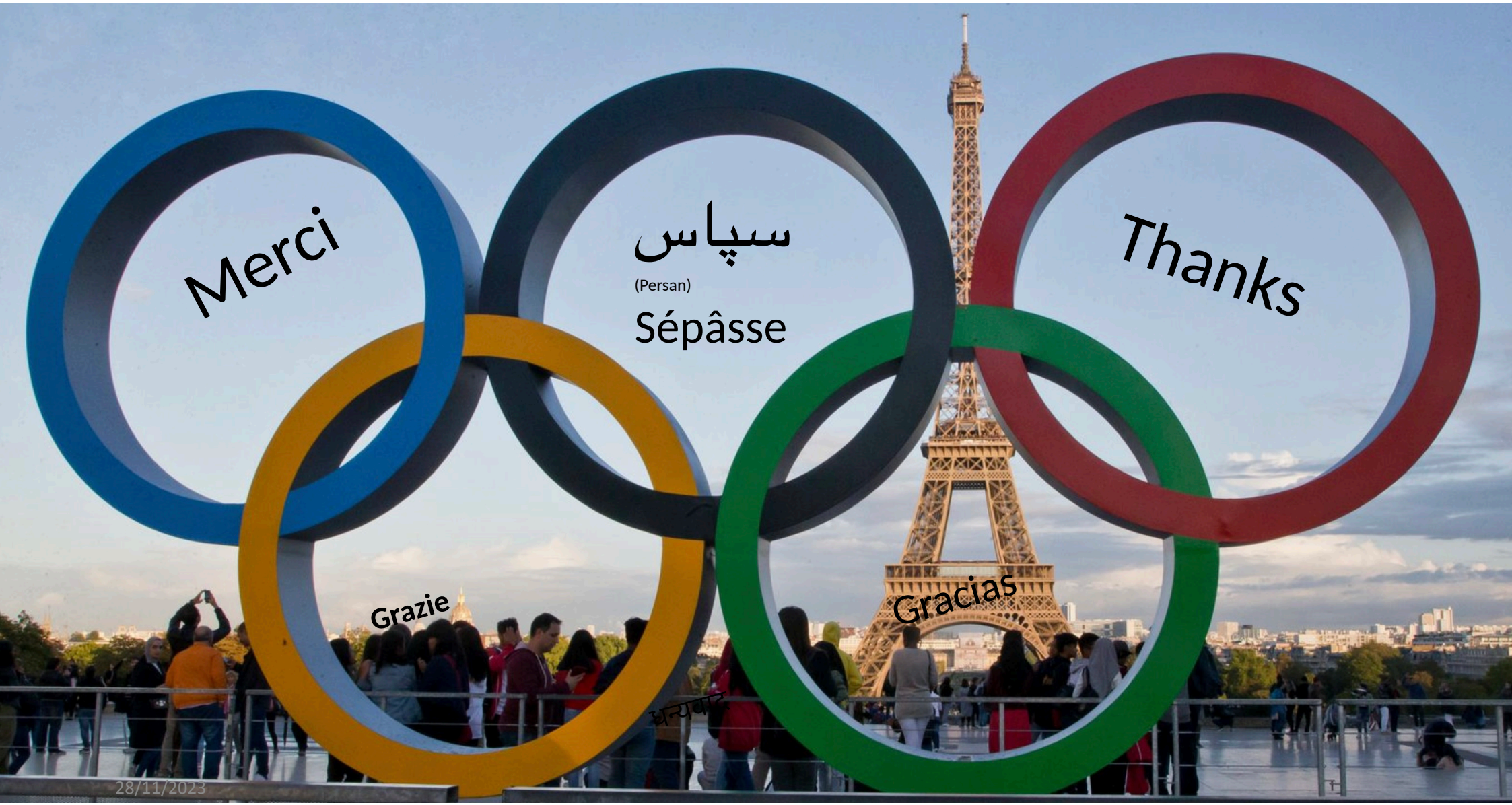
Using XIA in a closed circuit



- **Development of GPS: Gas Purification System**
- Manufacture is finished
- Pre-Installation @LPSC
- To be operational after confirming the XIA “normal” operating mode

Conclusion

- Radiopurity of off-the-shelf copper not sufficient – Solution: electroplate 0.5 mm ultra-pure copper layer to remove alpha particle component
- Electrode potential can be selected to plate only copper but not U, Th and Pb
- Surface preparation is critical – sanding, cleaning and etching
- Electropolished ~20 μm from surface and electroplated ~500 μm ultra-pure copper
- Difference in surface quality between the two hemisphere
- Measurement of the surface is necessary by an Alpha Counter
- For the cost and environment reason, gas purification and circulation system is welcome =>if the XIA operation mode is respected


Merci
سپاس
(Persan)
Sépâsse
Thanks
Grazie
धन्यवाद
Gracias