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Preparation and characterization of ^{10}B targets at JRC-Geel

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Lansing

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Outline

- EC - JRC.G2 - Target Preparation Laboratory
- Boron targets - Thermal evaporation
- New equipment - Design
- Implementation - Characterization
- Results
- Conclusions - Outlook

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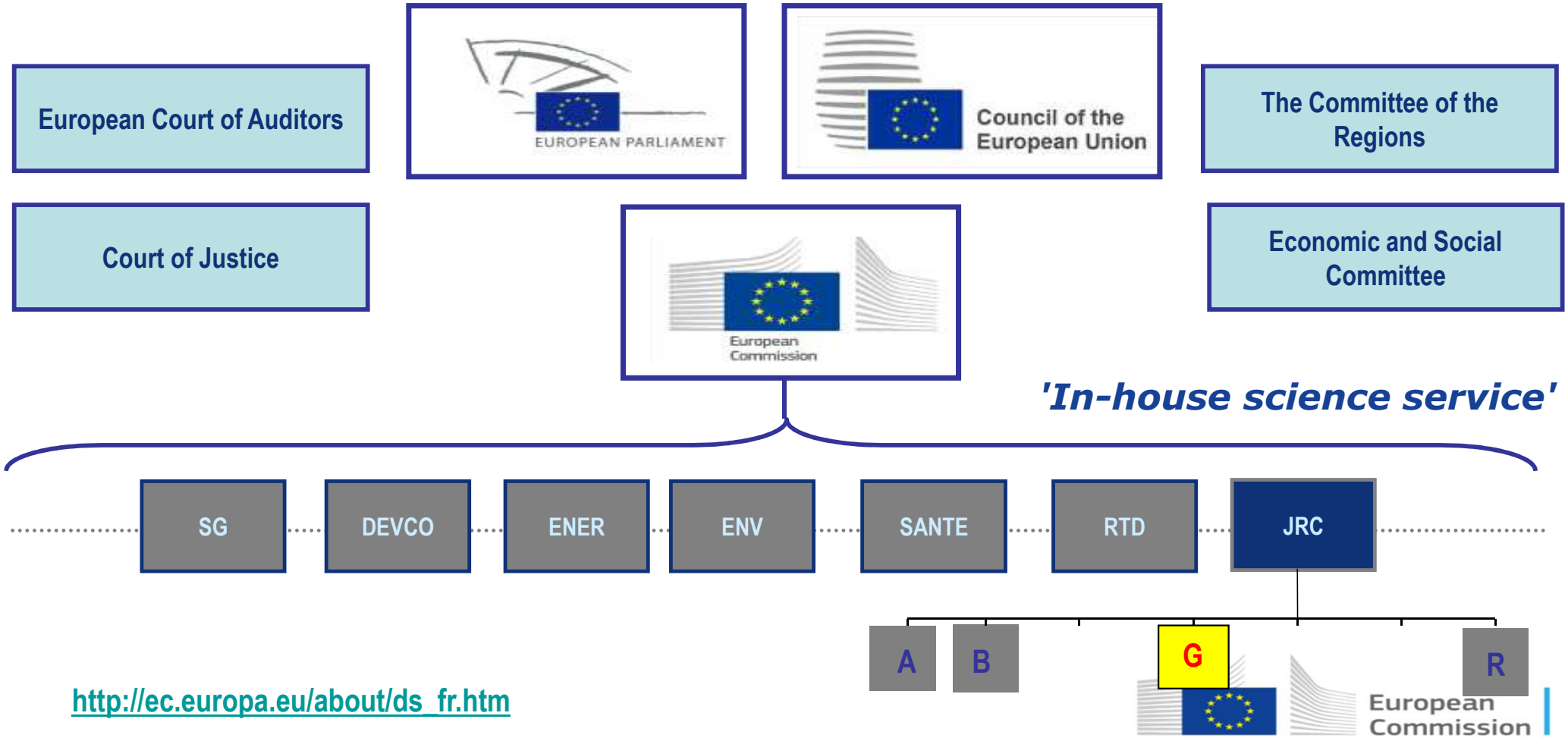
Panorama of the European Union

INTDS 2018

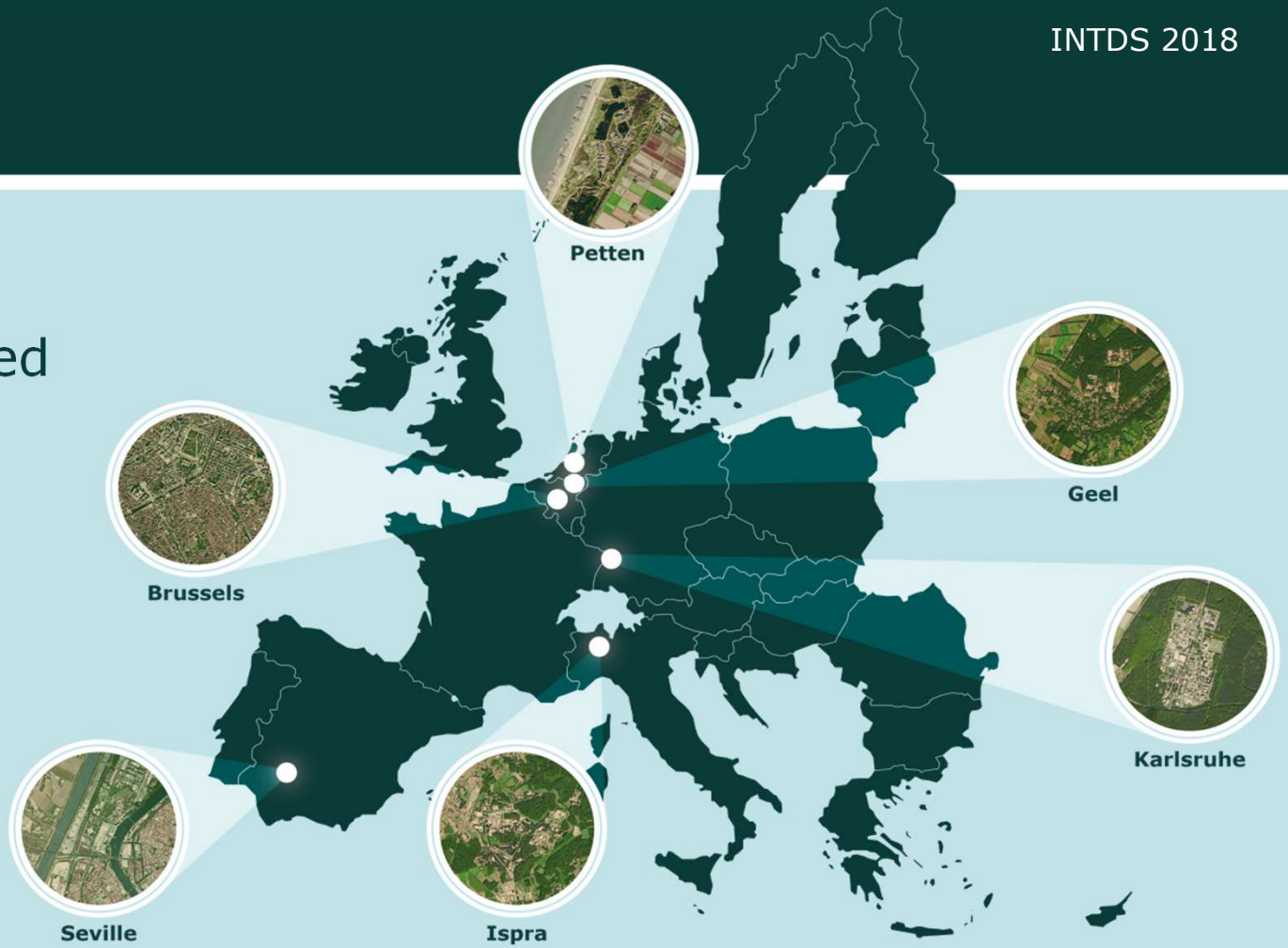


http://ec.europa.eu/about/ds_fr.htm

JRC sites

Headquarters in Brussels and research facilities located in **5 Member States:**

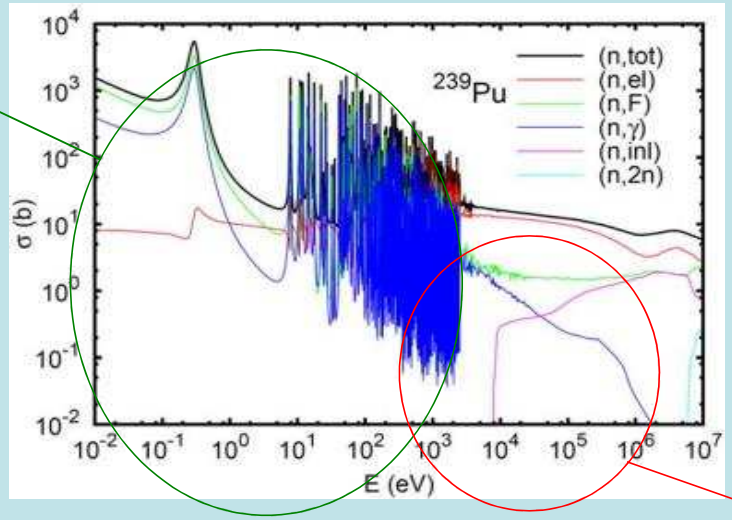
- Belgium (Geel)
- Germany (Karlsruhe)
- Italy (Ispra)
- The Netherlands (Petten)
- Spain (Seville)



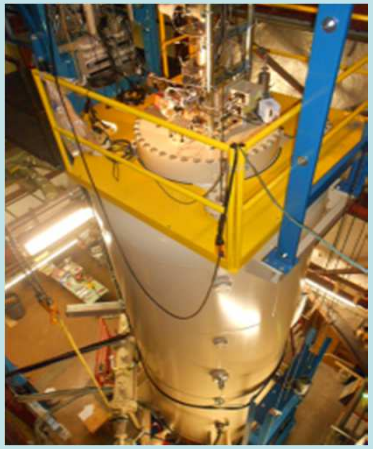
Nuclear facilities at JRC-Geel

GELINA and MONNET: nuclear data research

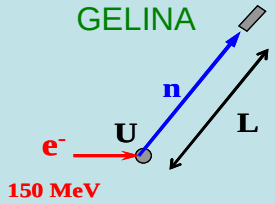
Time-of-flight measurements



MONNET

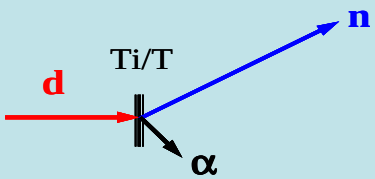


GELINA



energy resolution

Mono-energetic neutron beams



Nuclear targets

For neutron-induced measurements (mainly actinides)

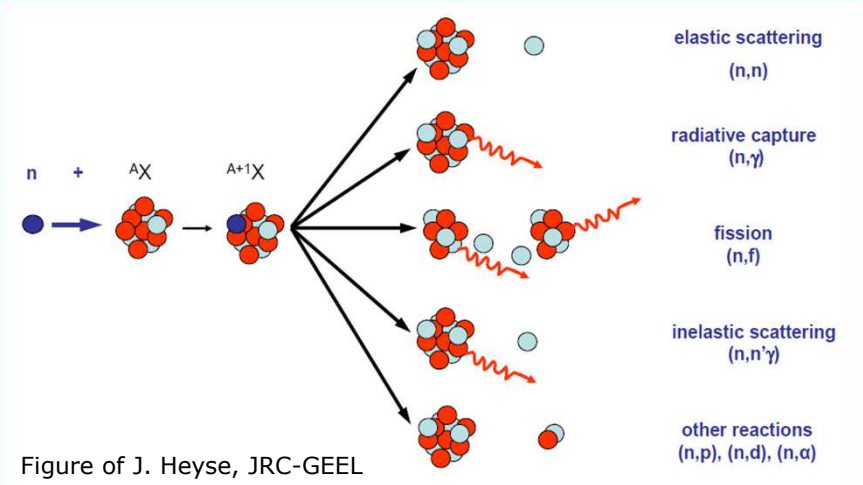
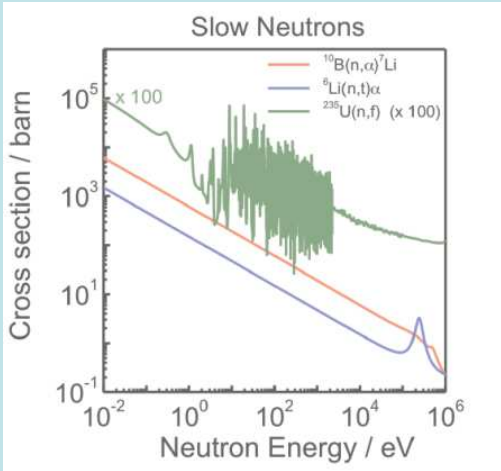


Figure of J. Heyse, JRC-GEEL

For **neutron flux monitoring (^{10}B)**

TABLE I. Nuclear reactions for thermal neutron detection

Reaction	Isotope abundance (%)	Cross section (barn) at 1.8 Å	Q-value (MeV)
^{3}He(n,p)^{3}H	0.014	5330	0.76
$^{10}B(n,\alpha)^{7}$Li	19.9	3838	2.31(94%) 2.79(6%)
$^{6}Li(n,\alpha)^{3}$H	7.6	947	4.78
^{14}N(n,p)^{14}C	99.63	1.91	0.62
^{235}U(n,3n)ff	0.72	680.9	≈200



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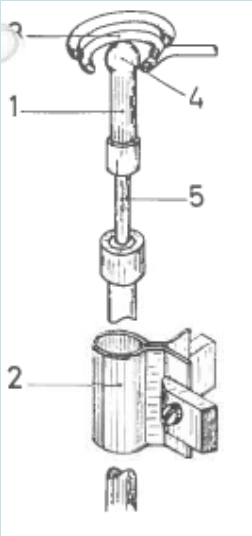
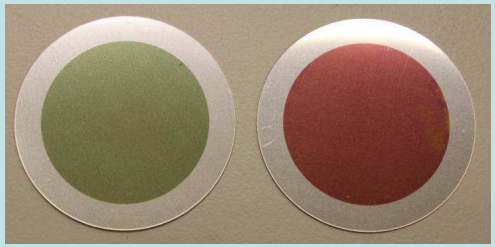
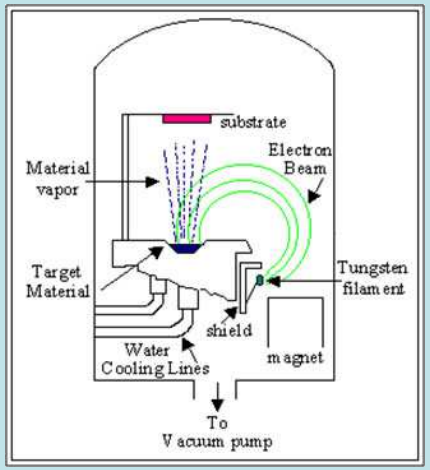
Boron target preparation



- Start of TP activities in 1961 (>20 staff)
- Start of vacuum ^{10}B deposition in 1967 with high frequency induction
- Start of vacuum ^{10}B deposition in 1988 with e-beam evaporator (Leybold Univex 450) in view of neutron lifetime experiment → reference targets extensively characterized by ICP-MS and by irradiation in BR1 research reactor:



Metallic Boron: melting point of 2349 K and evaporation heat of 509 kJ/mol



- After reorganisation during nineties: ^{10}B project stopped, e-beam evaporator stored (3 staff)
- After 'nuclear renaissance' (2000, 5 staff): renewed interest for nuclear data → need for ^{10}B targets as flux monitor →restart with Leybold e-beam evaporator

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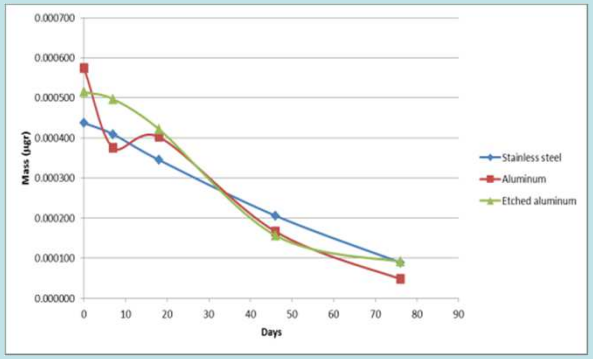
Boron target preparation

- Several deposits were prepared with Leybold e-beam for TOF experiments → milky white deposit (weighable), poor signal → unstable?
- Monitoring of mass of deposit → unstable



- Variation of parameters: material (3 batches), substrate → no improvement
- Reduced vacuum and e-beam power→ poor adsorption

  → New equipment required

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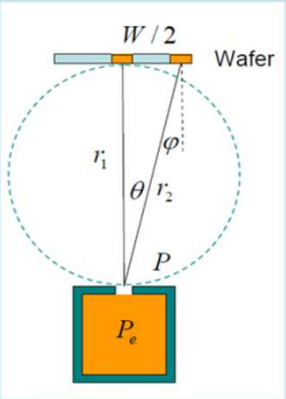
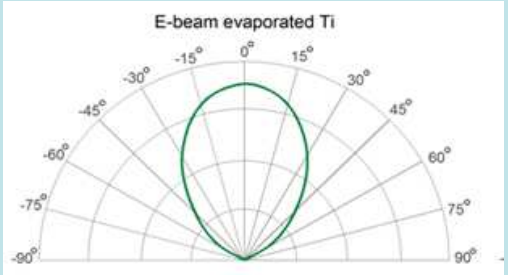
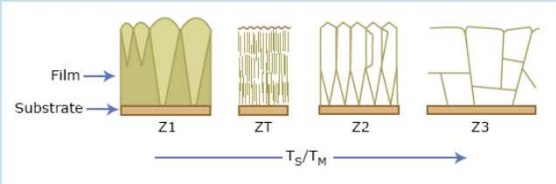
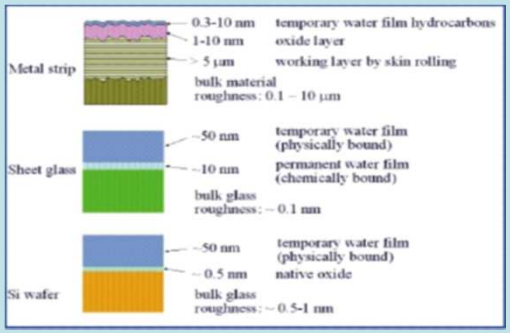
Boron target preparation

- New equipment: public procurement, techspecs based on experience and new technological possibilities
  - → Good vacuum (<10^{-6} mbar) and e-beam power
  - → High frequency sweeping of e-beam
  - → Vertical movement for larger samples or faster evaporations
  - → Multiple pockets for thicker deposits or sandwich, W liner
  - → Plasma cleaning: in-situ removal of hydrocarbons/moisture for better adhesion, especially for thin fragile backings (Al Mylar)
  - → Quartz heating (SZM)
  - → MiniLab E080A from Moorfield Nanotechnology ©









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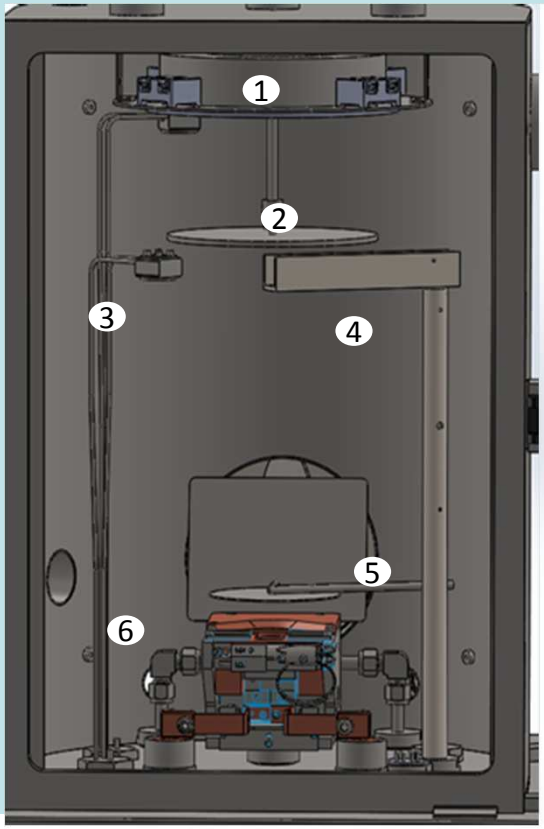
Design

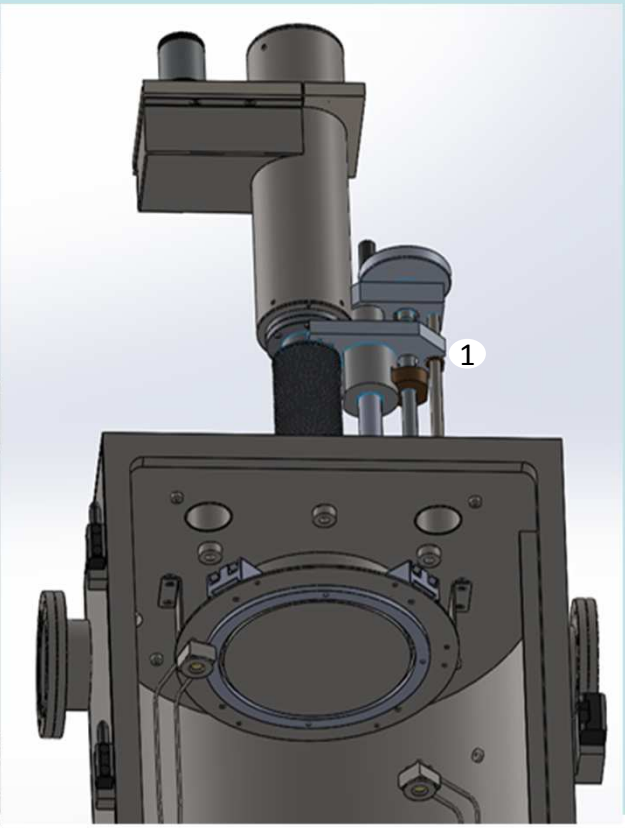
D-shaped chamber with vacuum better than 5.10^{-7} mbar

1. Quartz heating (500°C), thermocouple
2. Rotating substrate stage with Z-shift: 30-40 cm
3. Two quartz crystals for thickness measurement
4. Glow discharge (Ar)
5. Pneumatic shutter
6. evaporation source of 6 kW, 10 kV power supply, 0-500 mA emission current, 6 water cooled pockets of 7 cm^3 with automated indexer, W liner

→ Approval of design before construction

→ FAT before installation





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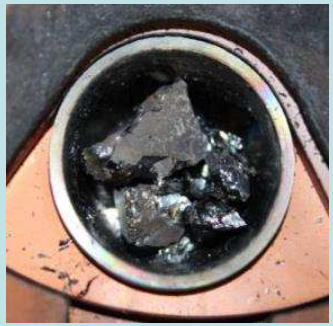
Testing, implementation and characterization



- Natural metallic boron (assumed 19.9% ^{10}B, no certificate)
- 0.5 mm Al backing ∅ 50 mm, spot size ∅ 30 mm
- In-situ plasma cleaning
- Factory acceptance test: 4 targets at premises of supplier
  - → Validation of equipment, training, determination of settings
  - → Differential substitution weighing, SEM/EDX
- Tests at JRC-Geel: 5 targets of different thicknesses
  - → Implementation, calibration of thickness monitor
  - → Differential substitution weighing, comparative TOF measurement with ^{10}B and ^{235}U standards, SEM/EDX
- 2 targets for n-TOF experiment in CERN, 0.025 mm Al Mylar ∅ 120 mm, spot size ∅ 80 mm, 8 µg $^{10}B \cdot cm^{-2}$ (weighing not possible)

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Results

W!

Target	e-beam power (mA)	Deposited mass (g)	Areal Density, weighing (µg/cm2)	Areal Density B10, weighing and natural abundance (µg/cm2)	Areal Density B10, TOF (µg/cm2)	SEM	EDX
FAT 1 and 2	200	-	-	-	-	-	-
FAT 3	30 -40	0.000142	20.2	4.0		Some dust particles, layer follows Al morphology	B, Al, O, Fe traces, no W
FAT 4	30-40	0.000260	36.8	7.3		Some dust particles, layer follows Al morphology	B, Al, O, Fe traces, no W
Test 5	30-40	0.000039	5.6	1.1			
Test 4	30-40	0.000079	11.1	2.2			
Test 3	30-40	0.000154	21.8	4.3			
Test 2	30-40	0.000289	40.8	8.1	7.0		
Test 1	30-40	0.000527	74.5	14.8	12.7	Some dust particles, layer follows Al morphology	B, Al, O, Fe traces, no W
TP2017-10-01	30-35	no weighing	8.0 according monitor	2.8	2.9		
TP2017-10-02	30-35	no weighing	22.2 according monitor	8.0	8.0		

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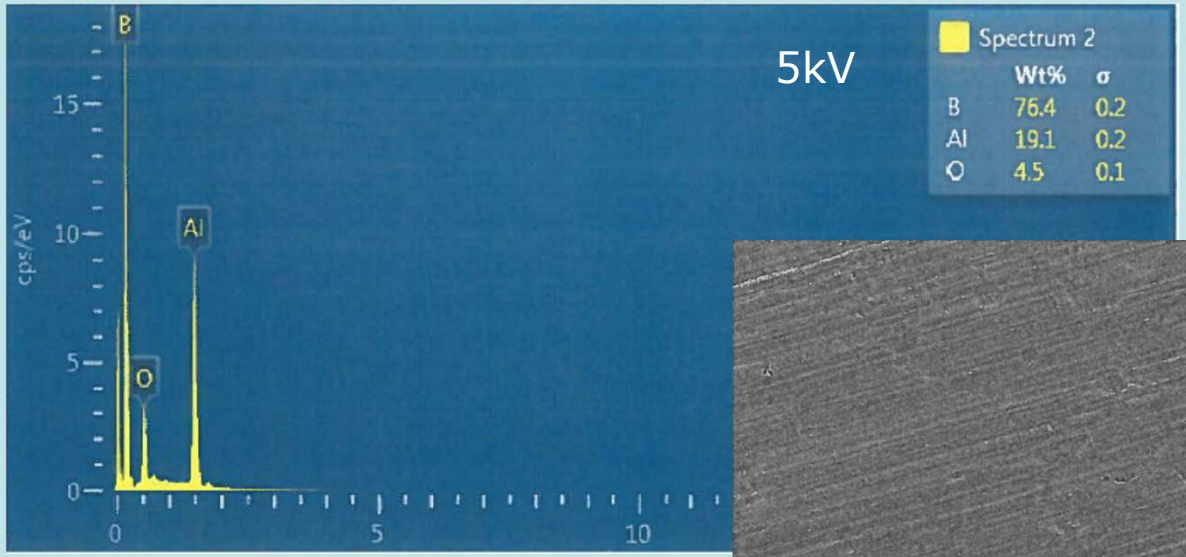
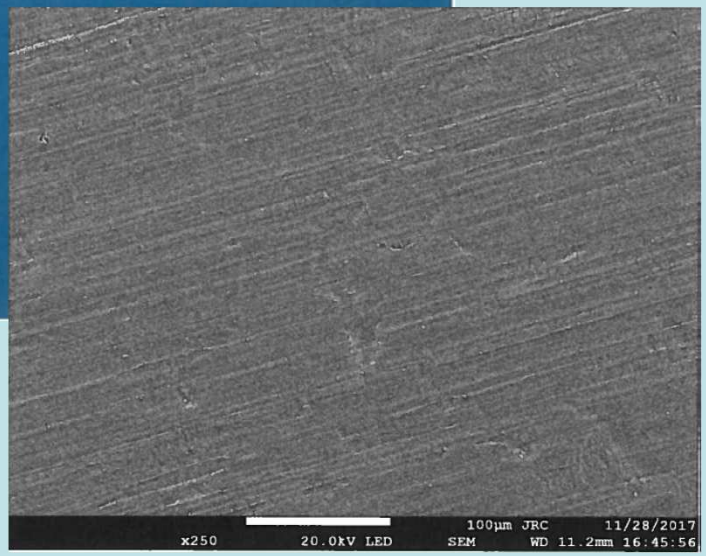
Results: EDX FAT4; SEM FAT3

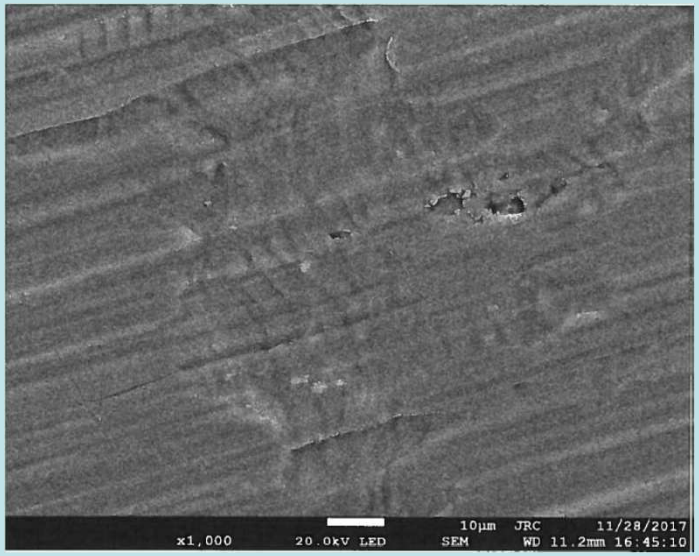


5kV

x250



x1000



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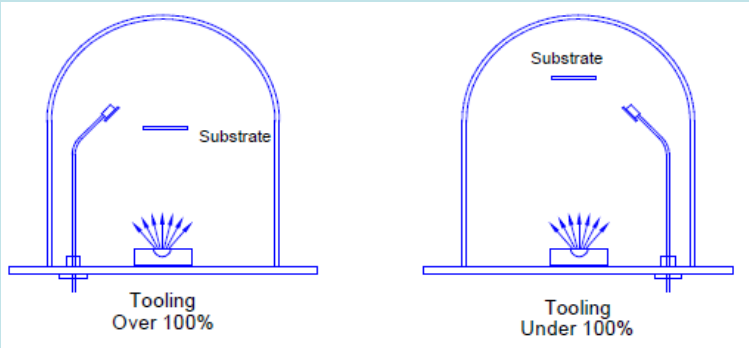
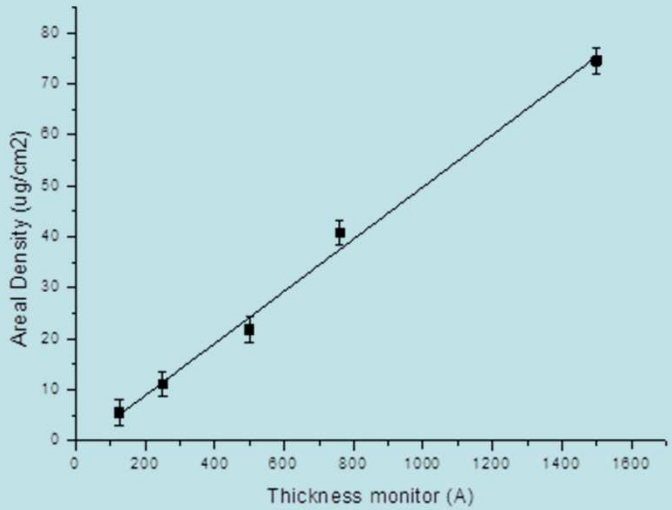
Results

B Target	Measured thickness monitor (Å)	Measured total mass layer (g)	Calculated areal density (µg/cm2)	Calculated thickness (nm)	Calculated Tooling Factor
test 5	125	0.000039(18)	6(3)	24(11)	188(85)%
test 4	250	0.000079(17)	11(2)	47(10)	188(43)%
test 3	500	0.00[illegible]	[illegible]	92(10)	184(24)%
test 2	760	0.0[illegible]	[illegible]	172(11)	227(20)%
test 1	1500	0.0[illegible]	[illegible]	314(11)	210(15)%

$$F_m = F_i \frac{T_m}{T_i}$$





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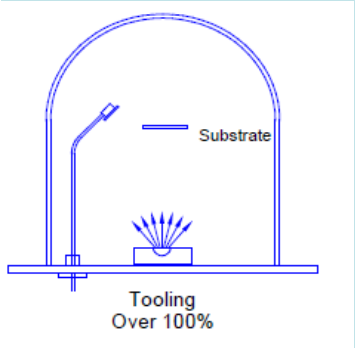
Results



Moving craters→ varying TF

→ Other techniques required for accurate thickness measurement

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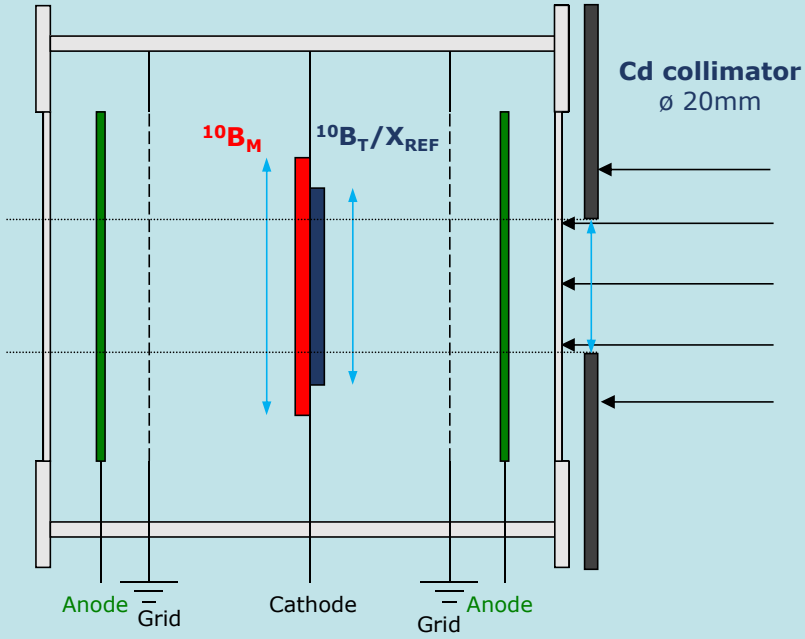
TOF measurements



- Characterisation of samples with thermal neutrons at GELINA TOF facility
- Double-sided Frisch-gridded ionisation chamber (IC)
- 2 samples back-to-back on IC Cathode
  - Side 1: Fixed neutron flux monitor ($^{10}B_M$)
  - Side 2: Test sample ($^{10}B_T$) or Reference sample (X_{REF})
- ø 20mm Cd collimator to have well defined thermal neutron beam
- Reference samples used (X_{REF})
  - ^{10}B (TP2012-00-01): 8.766(138) µg/cm^2
  - ^{10}B (TP2012-00-03): 17.096(264) µg/cm^2
  - ^{235}U (NS0009-A): 110.889(66) µg/cm^2
- Reactions of interest
  - $^{10}B(n,\alpha)^7Li$: detection of α and ^7Li in IC
  - $^{235}U(n,f)$: detection of fission fragments in IC

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Results

Target	e-beam power (mA)	Deposited mass (g)	Areal Density, weighing (µg/cm2)	Areal Density B10, weighing and natural abundance (µg/cm2)	Areal Density B10, TOF (µg/cm2)	SEM	EDX
FAT 1 and 2	200	-	-	-	-	-	-
FAT 3	30 -40	0.000142(14)	20.2(8)	4.0(2)		Some dust particles, layer follows Al morphology	B, Al, O, Fe traces, no W
FAT 4	30-40	0.000260(13)	36.8(24)	7.3(5)		Some dust particles, layer follows Al morphology	B, Al, O, Fe traces, no W
Test 5	30-40	0.000039(18)	5.6(25)	1.1(5)			
Test 4	30-40	0.000079(17)	11.1(25)	2.2(5)			
Test 3	30-40	0.000154(18)	21.8(26	4.3(5)			
Test 2	30-40	0.000289(18)	40.8(30)	8.1(6)	7.0		
Test 1	30-40	0.000527(19)	74.5(40)	14.8(9)	12.7	Some dust particles, layer follows Al morphology	B, Al, O, Fe traces, no W
TP2017-10-01	30-35	no weighing	8.0 according monitor	2.8 calculated according monitor	2.9		
TP2017-10-02	30-35	no weighing	22.2 according monitor	8.0 calculated according monitor			

ID-MS of B start material

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Results

Target	e-beam power (mA)	Deposited mass (g)	Areal Density, weighing (µg/cm2)	Areal Density B10, weighing and natural abundance (µg/cm2)	Areal Density B10, TOF (µg/cm2)	SEM	EDX
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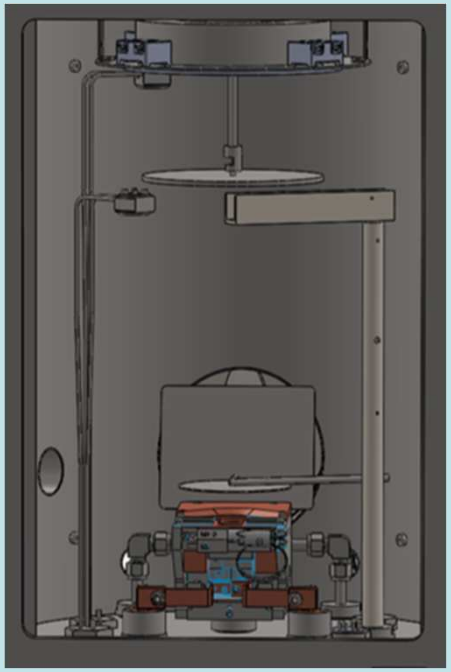

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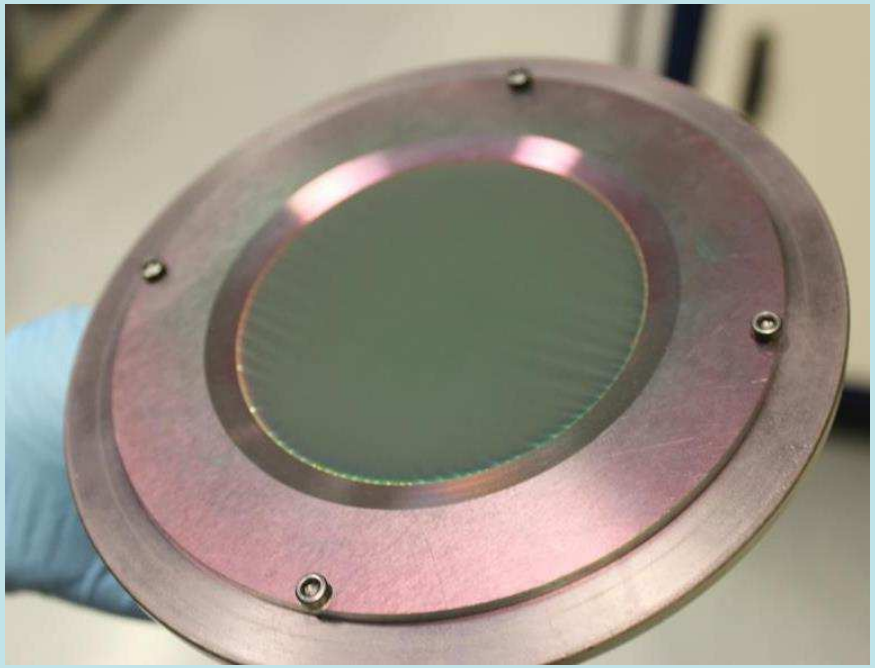
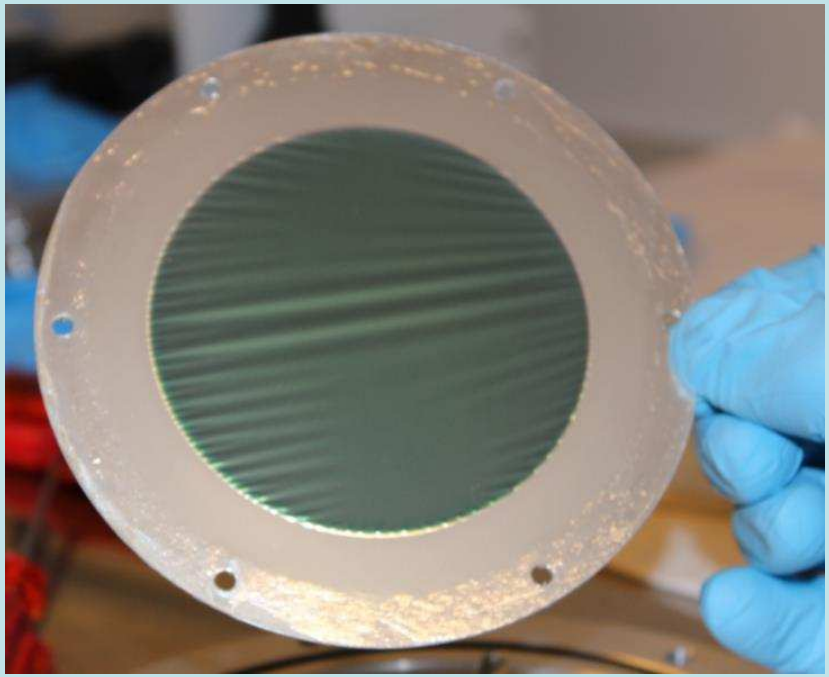
Results





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Conclusion

- A new e-beam evaporation system customized according to the specific needs for the deposition of thin boron layers was installed at the TP laboratory
- Successfully implemented and calibrated by conducting several evaporation runs
- Characterization shows good quality of the layer (morphology and impurities)
- Thickness measurement only with quartz crystal is indicative
- Isotopic composition of starting material requires more characterization
- First ^{10}B targets made for TOF experiment in CERN
- After almost 20 years of technical incapability, the target preparation laboratory is once again able to prepare high quality boron targets which are of great importance for the neutron flux monitoring in the frame of nuclear data research.
- E-beam evaporation of U_3O_8?

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Any questions?

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