ITALIAN NATIONAL AGENCY FOR NEW TECHNOLOGIES, ENERGY AND SUSTAINABLE ECONOMIC DEVELOPMENT

**Academia-Industry Matching Event**

**Second Special Workshop on Neutron Detection with MPGDs**

# Thin film Boron deposition for GEM-based neutron detectors


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**Nuclear Technologies Laboratory**

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**CERN, 16-17 March 2015**

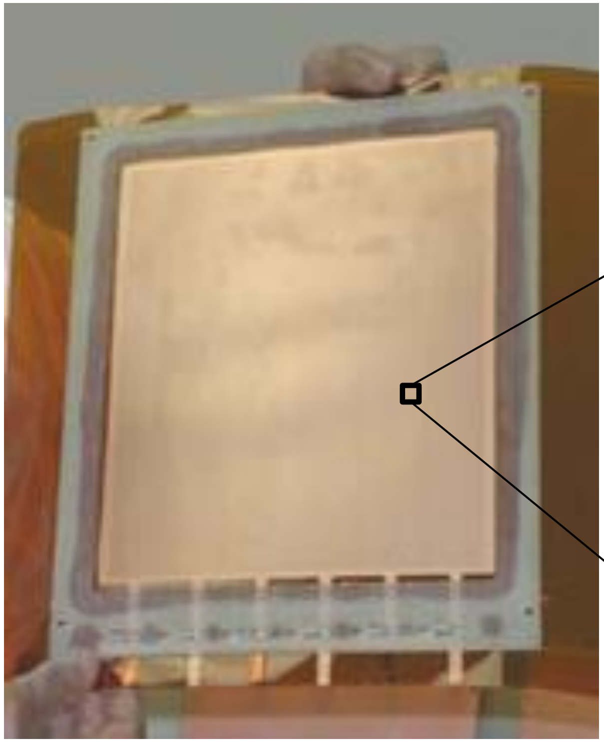
# outline

- ✓ Side-on GEM-based thermal neutron detectors

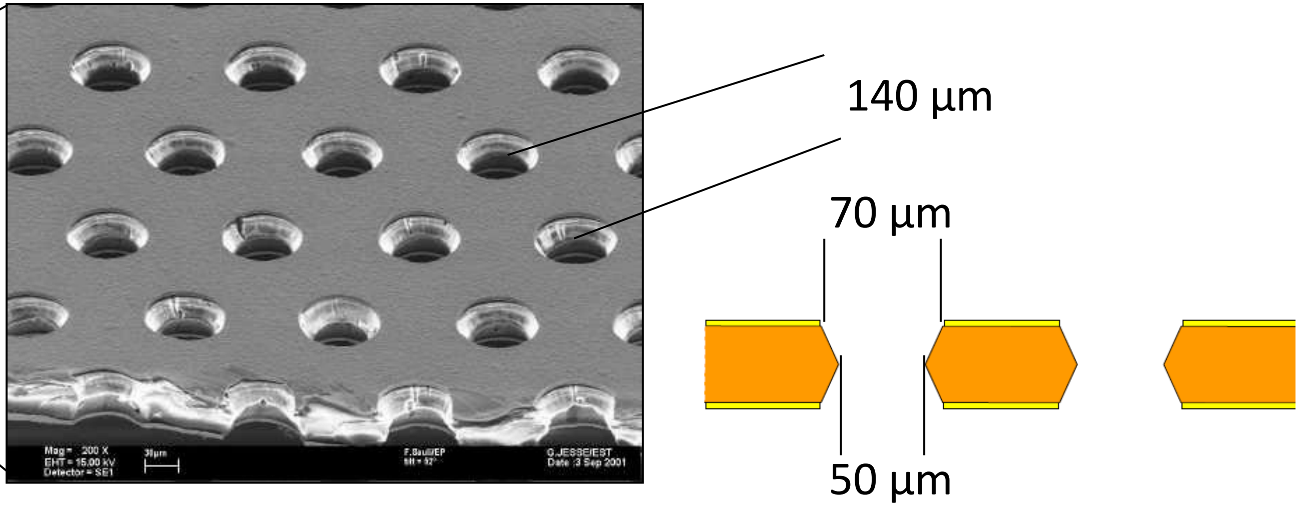
- ✓ Pure $^{10}B$ (rather than $B_4C$) deposition activity
  - ✓ Deposition facilities
  - ✓ Characterization tools

- ✓ A brief overview on results and perspectives

# Gas Electron Multipliers (GEM)

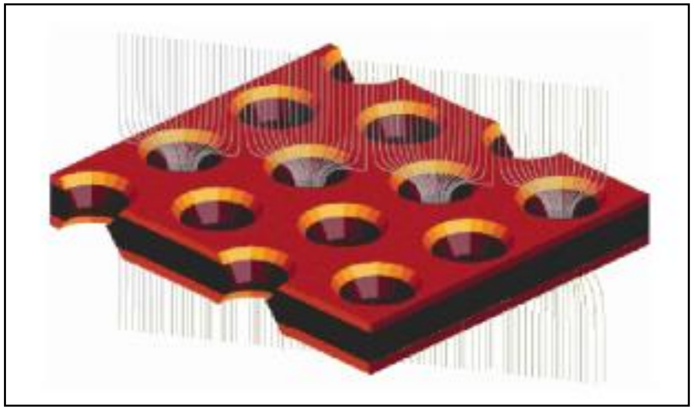


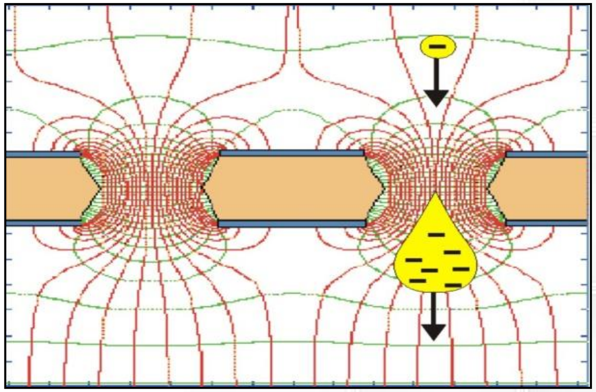
A Gas Electron Multiplier (F.Sauli, NIM A **386**, 531) is made by 50 μm thick kapton foil, copper clad ( 5 μm thick) on each side and perforated by an high surface-density of bi-conical channels;




Applying a potential difference (typically between 300 and 500 volts) between the two copper cladding, an high intensity electric field is produced inside the holes (80-100 kV/cm).

GEM is used as a proportional amplifier of the ionization charge released in a gas detector.

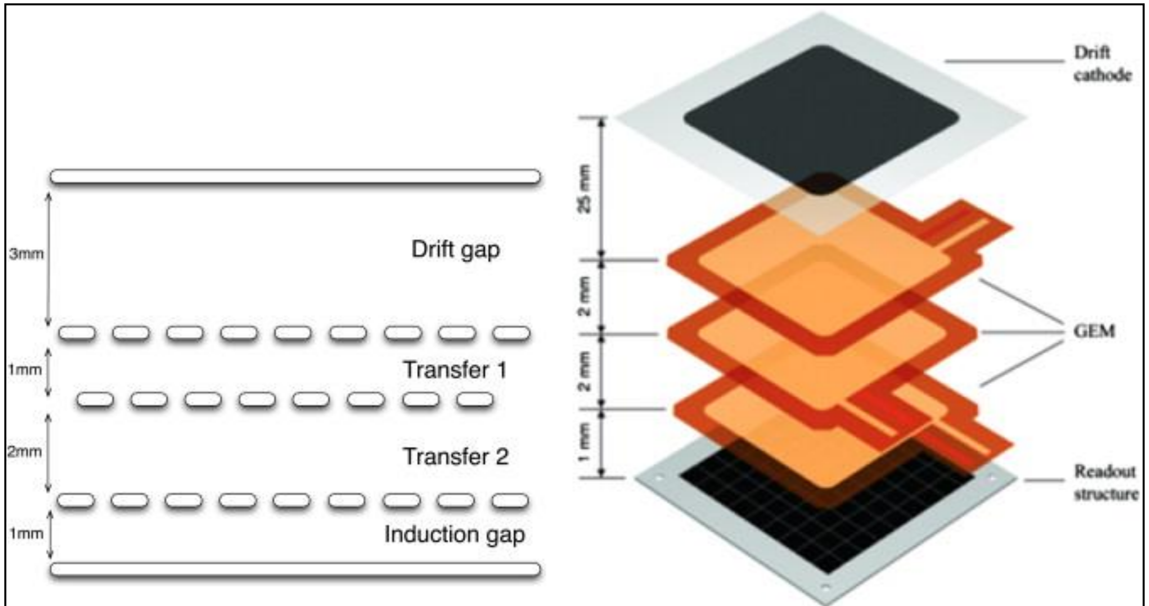


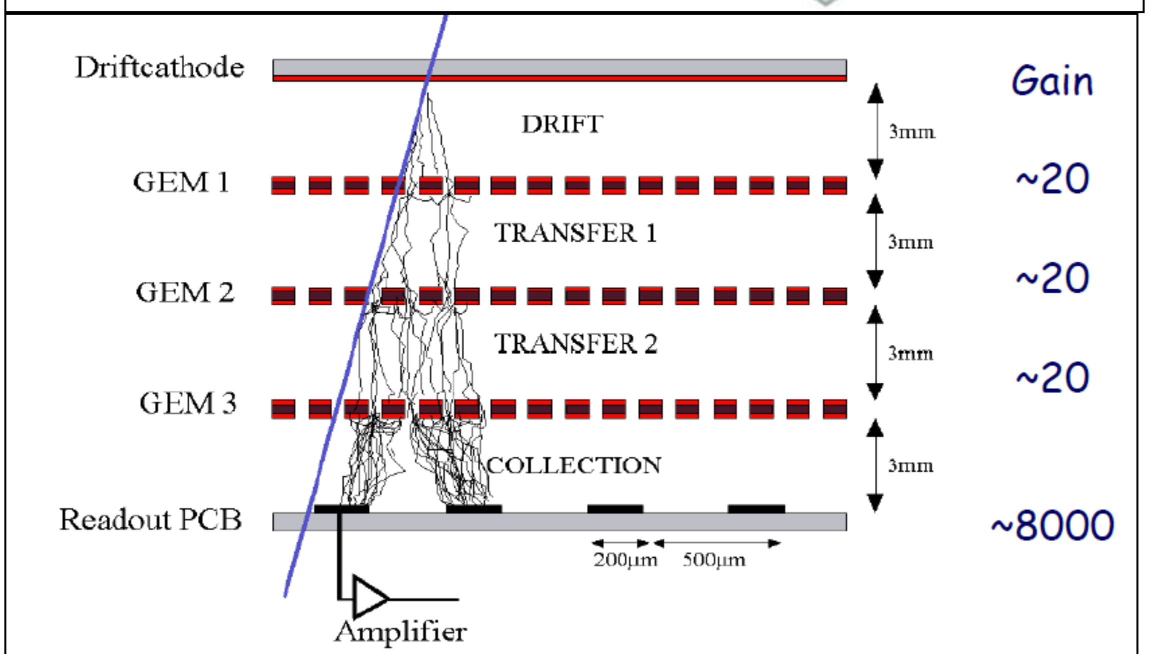


# Gas Electron Multipliers (GEM)

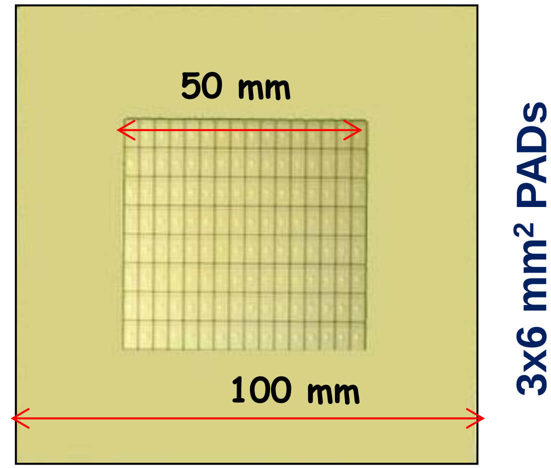
## Triple GEM configuration

Layout of a typical Triple GEM detector constructed with standard 10 x 10 cm²

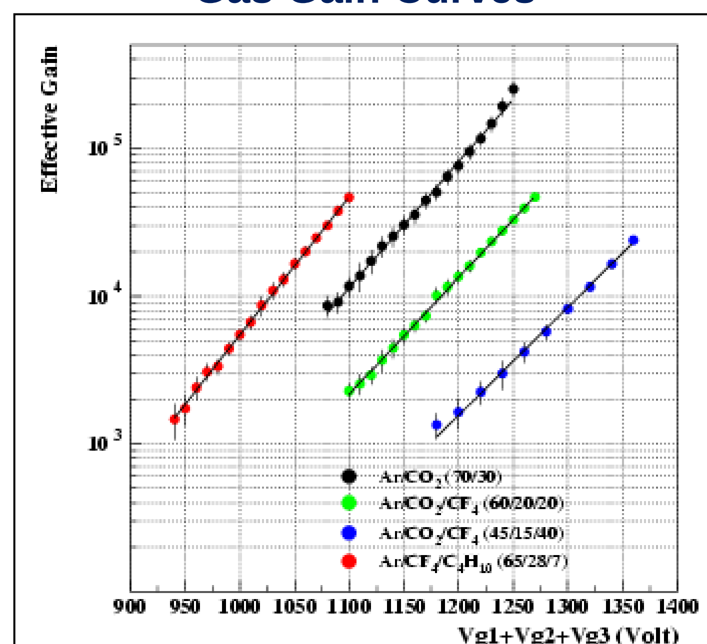



The anode has 128 pads. Each PAD can have a different geometry depending on detector applications.

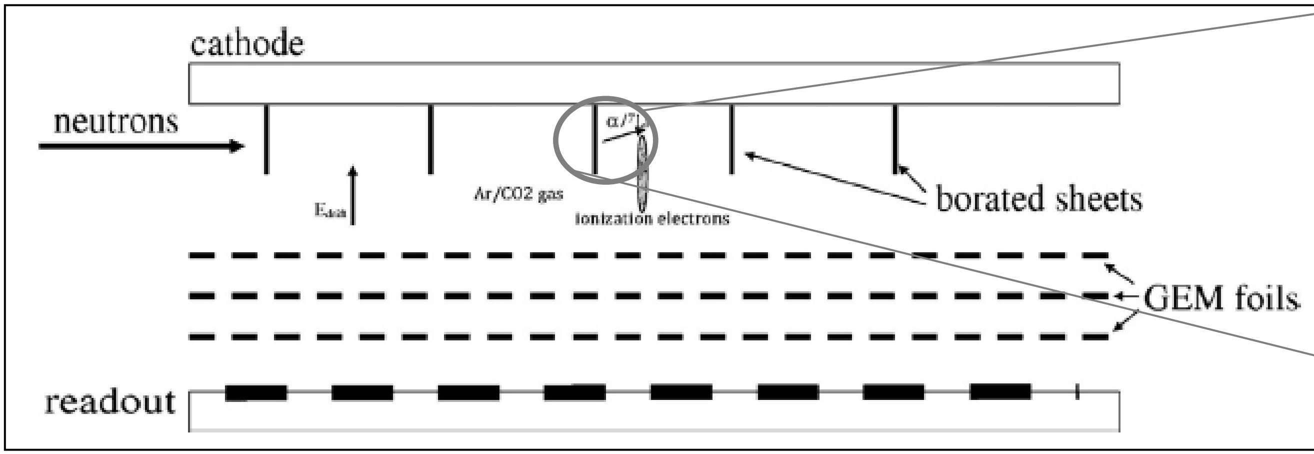


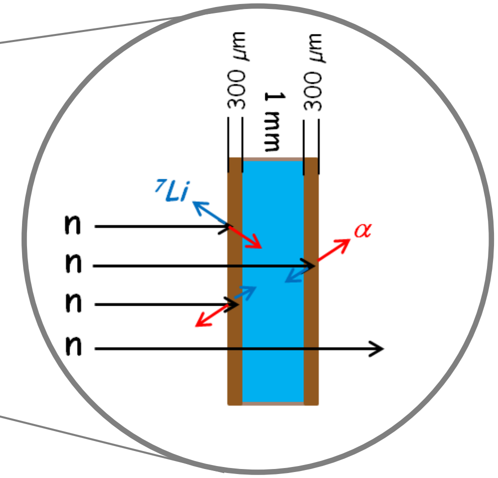

### Gas Gain Curves

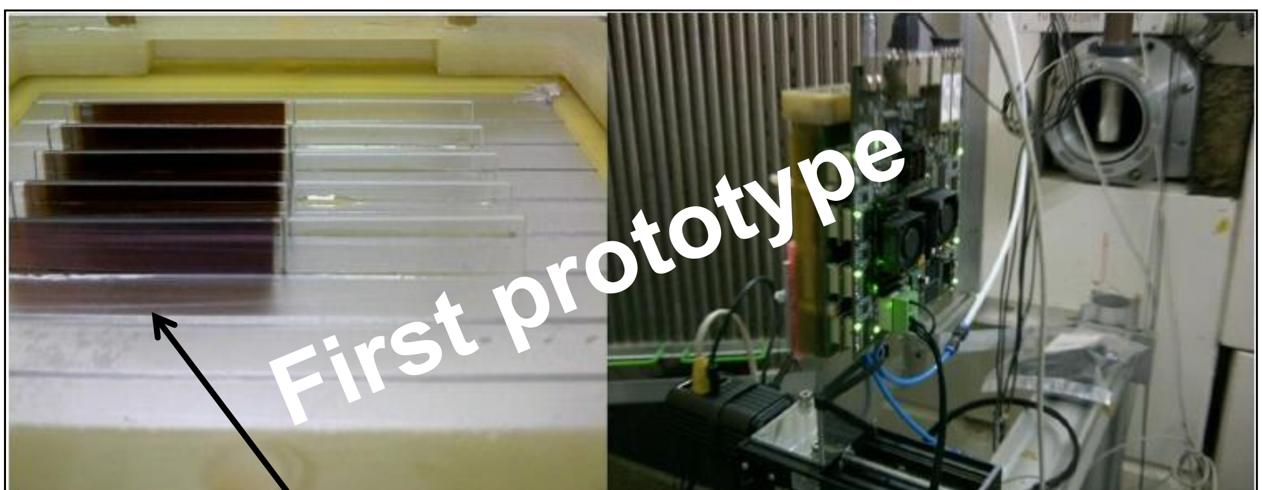


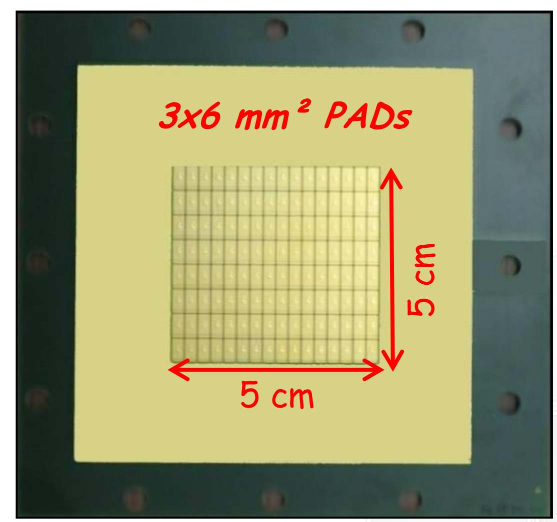
# The sideon GEM (S-GEM) configuration for thermal neutrons

The idea was to insert a sequence of borated strips attached to the aluminum cathode.
The drift region was extended and the detector was equipped with a side window.




Boron Reaction: $^{10}B + n \longrightarrow \alpha + {}^{7}Li + 2.79$ MeV ( 6 %)

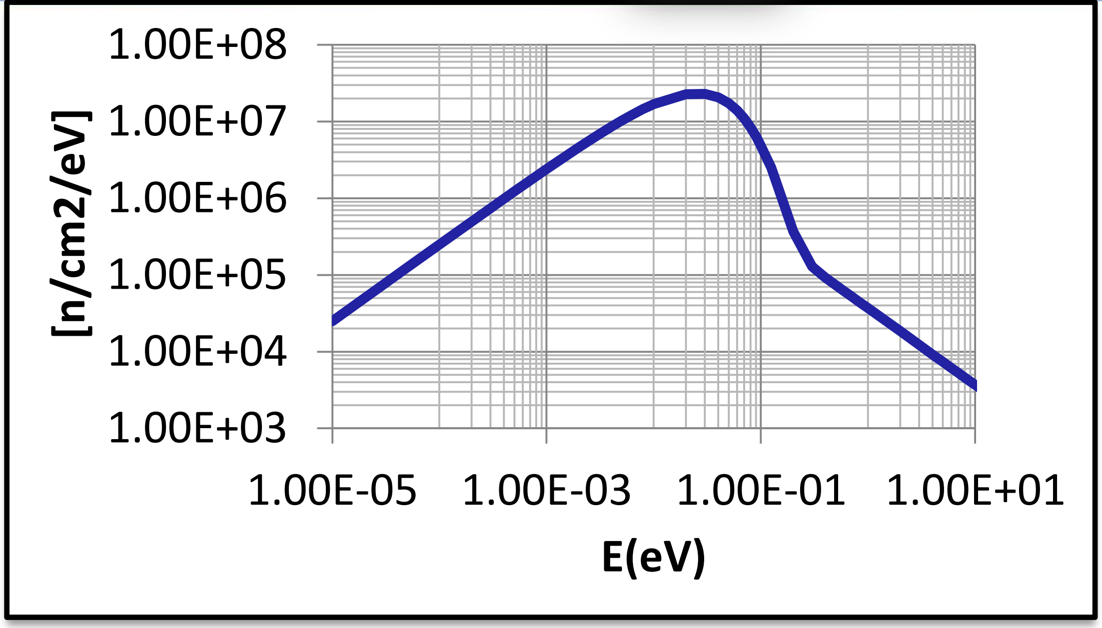
$\longrightarrow \alpha + {}^{7}Li^{*} + 2.31$ MeV + $\gamma$ (478 keV) ( 94%)

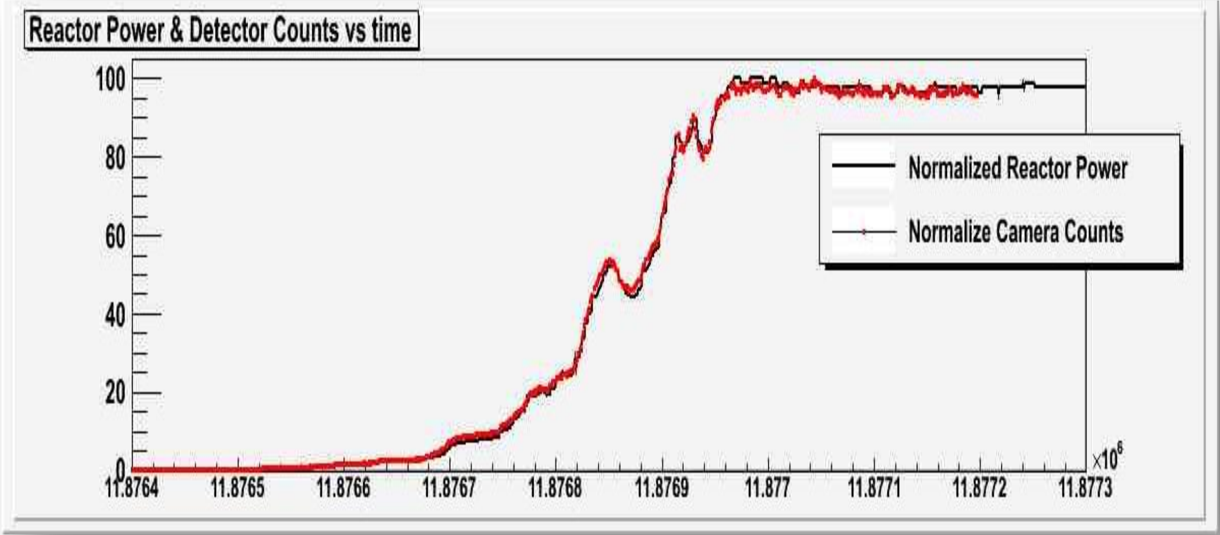
**Boron deposited by electron beam (thermal evaporation)**

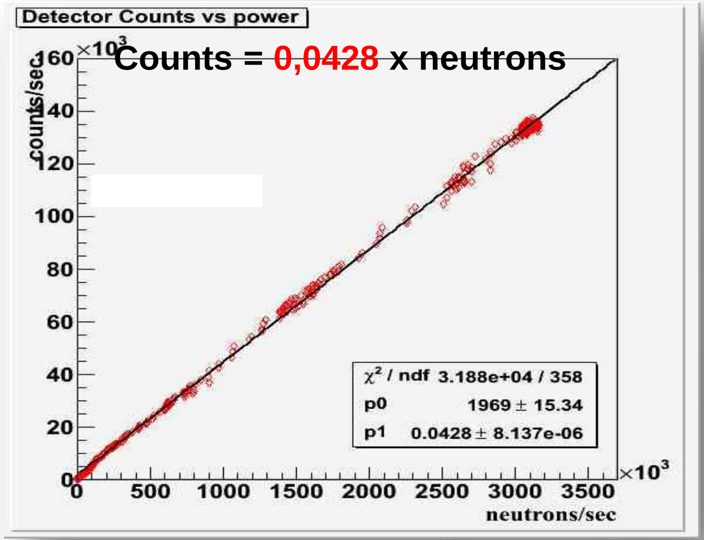
# Tests @ TRIGA reactor (ENEA)

**TRIGA Power**: 1 MW (variable)
**Beam line**: radial channel
**Flux @ 1 MW**: $\phi$ = (2,3 ± 0.2) x$10^6$ n$cm^{-2}s^{-1}$
**Spectrum**: Maxwell-Boltzmann at 25 meV
**FWHM** ~ 70 meV

The flux was changed varying the reactor power from 100 W to 1 MW.
Side-on GEM detector was able to follow these variations.



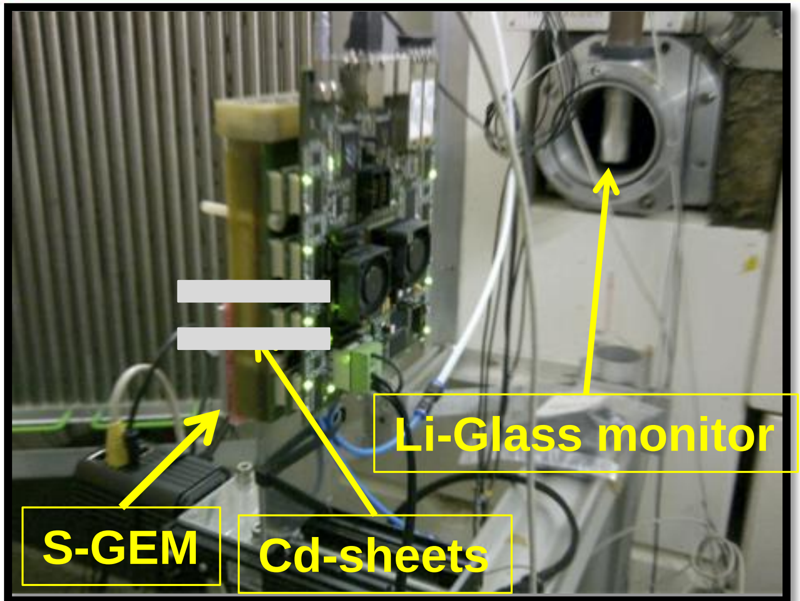




Knowing the incident flow and the detector active area of we estimated an **efficiency** of **4.3 ± 0,5%,** over the whole range of reactor thermal energies.

# Test @ ISIS: ToF measurements

## Beam line: ROTAX

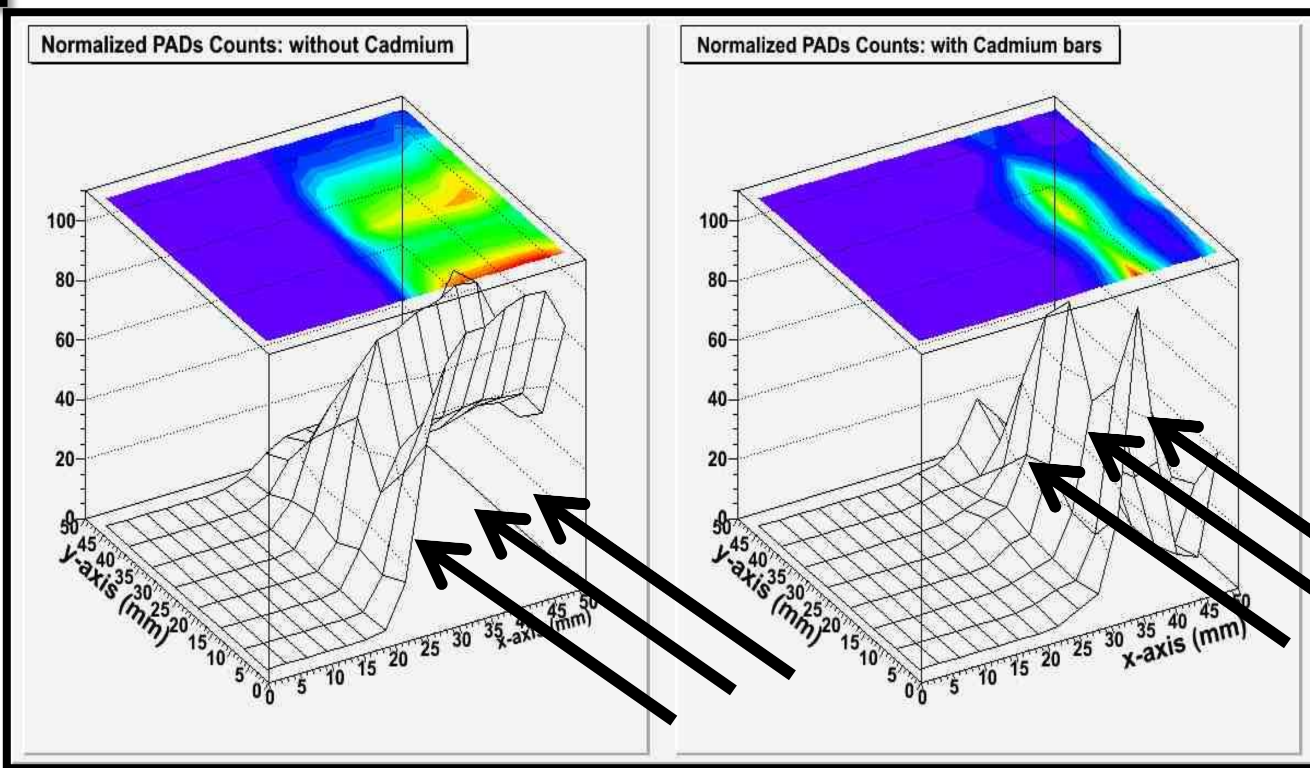



Stand alone detection system (not ISIS DAE)

An external trigger starts the detector acquisition of the detector which records data in a time window of 1 ms.

Reconstructed ToF spectrum from **S-GEM** measurement is in agreement with that obtained from the beam monitor of **ROTAX**.

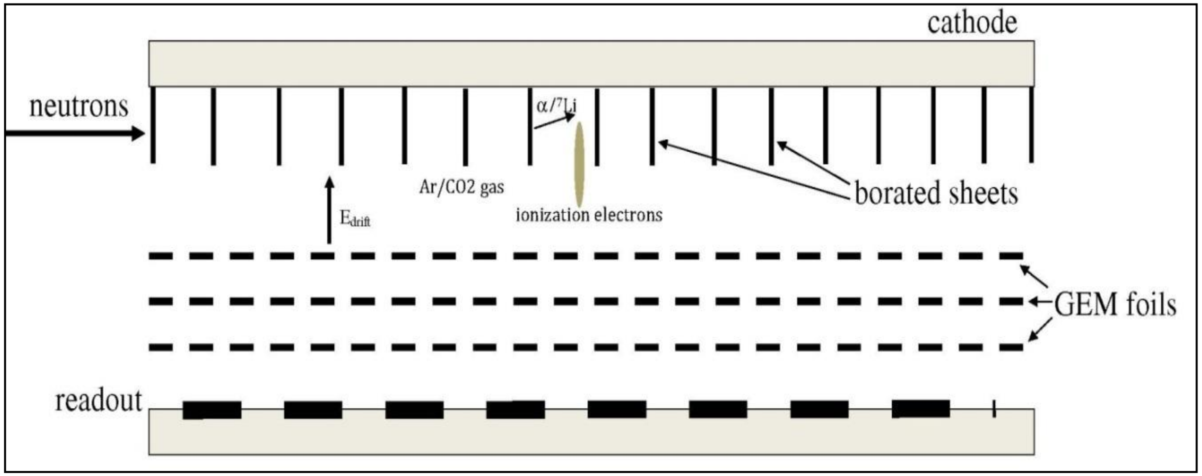
2D intensity plots and the corresponding contour plots relative to the measurements with and without the cadmium sheets in front of the S-GEM. In the right panel, the counts are present in the region between the two absorbing Cd sheets.

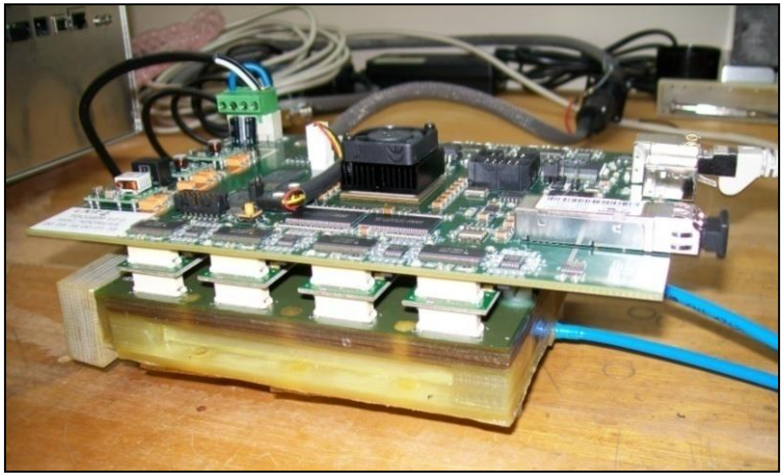


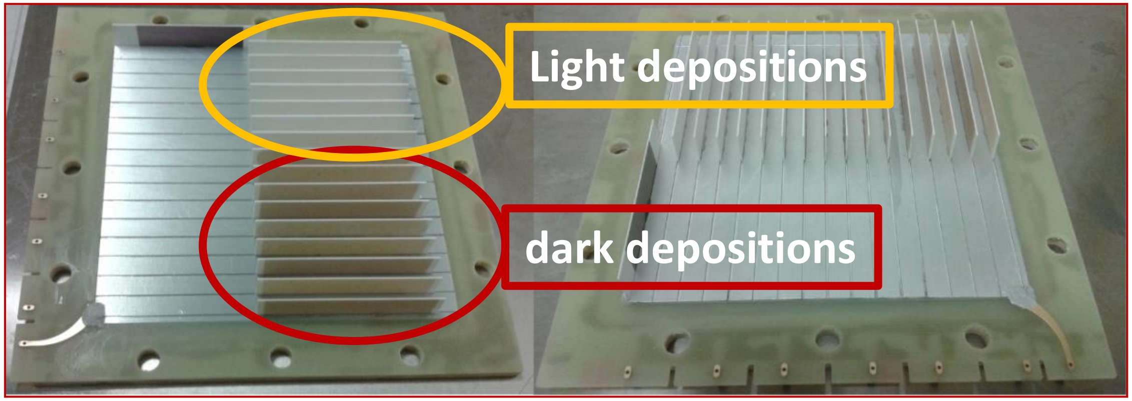

Signals from gamma-rays (10B/Cd and background) are effectively rejected at the chosen bias seen before.

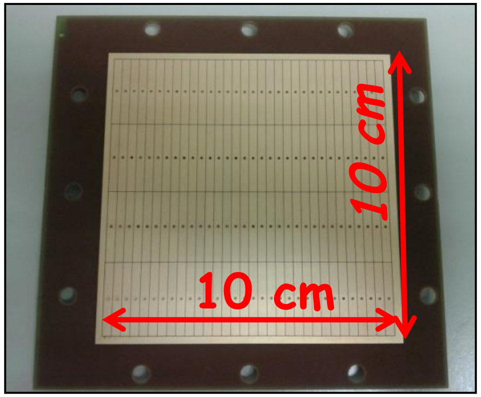
# The S-GEM second prototype

16 borated strips to obtain higher efficiency.





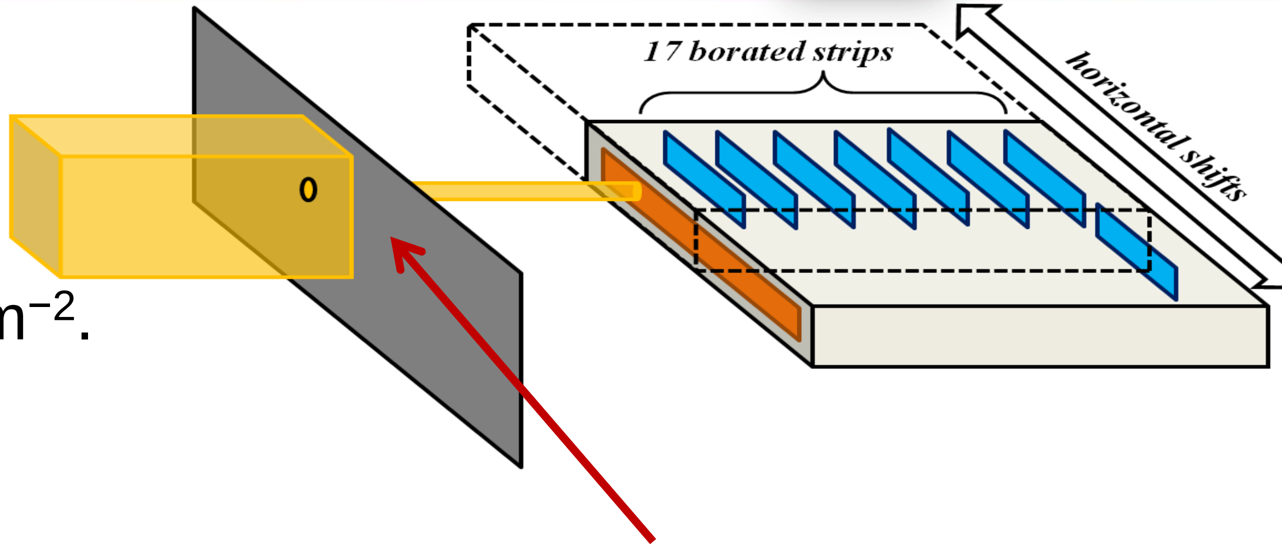

Anode with PADs having different geometry.
32x4 PADs each PAD is 3x24 mm².

**ceramic strips ($Al_2O_3$) 400 μm thick.**

Detector window is placed on the side of the anode with 32 PADs. In this way detector is able to measure also position of small spot beams impinging on the side-on window.

# Tests @ HFIR Oak Ridge National Laboratory (ORNL, US)

**HFIR Power**: 85MW
**beam line**:CG1A
(detector test station)
**estimated flux**: $2 \times 10^6$ n $cm^{-2}$.
$\lambda_P \sim$ 4.23 Å (**E**~4.57 meV)
**FWHM** ~0.11 Å
**irradiation area:** $5 \times 5$ $cm^2$
**angular divergence**: < 1°




borated aluminum mask with a hole 2,5 mm in diameter

**Comparison with $^3$He gas tube at pressure**

| | S-GEM | $^3$He Tube |
|---|---|---|
| Overall mean counts [$s^{-1}$] | 1863 | 6011 |
| Background mean counts [$s^{-1}$] | 21 | 1586 |
| Signal/Background | 87.7 | 2.8 |
| Efficiency [%] | 31 | 99 |

# Deposition activity

From boron powder (enriched boron-10) solid targets for e-beam thin film deposition were produced at ENEA Frascati Research Centre.

**Possible air contamination during targets sinterization.**

Indeed darker depositions were obtained by using raugh gross powder (prototype 1-glass sheets) targets rather than targets sinterized from fine powder.

We asked support to a private industry to produce sputtering targets in controlled environment and high-T and high-P to have less porous targets.

Columbus Superconductors

Columbus Superconductors SpA is a **world leader in cutting-edge magnesium diboride ($MgB_2$) technology** and the transformation of this superconducting material into long, versatile and **highly reliable superconducting wires**.

The company is vertically integrated, from R&D to applications and from production to sales.

Columbus is equipped with a comprehensive system for the processing of boron related powders and bulks

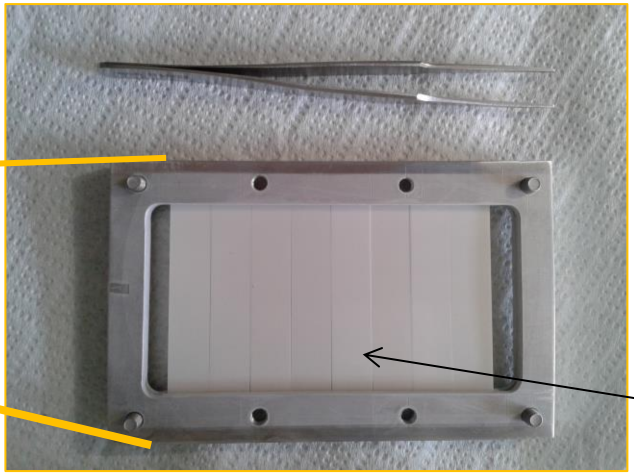



Pellets are now realized and analyzed before shipment, both with natural boron as well as $^{10}B$

First boron targets will be shipped to INFN during this week

# Deposition facilities
(ENEA Frascati)

9 sheets can be simultaneously deposited on both sides



Alumina sheet
10 × 50 × 0.5 mm

High Vacuum systems e-beam device for film depositions in the superconductivity Laboratory of the **Fusion Department** of the **ENEA** Frascati research Centre

**Evaporation system**: Thermionics 10 kW (10 kV, 1 A), 4 crucibles (25 cc)

**$d_{T\text{-}S}$** = 30 cm

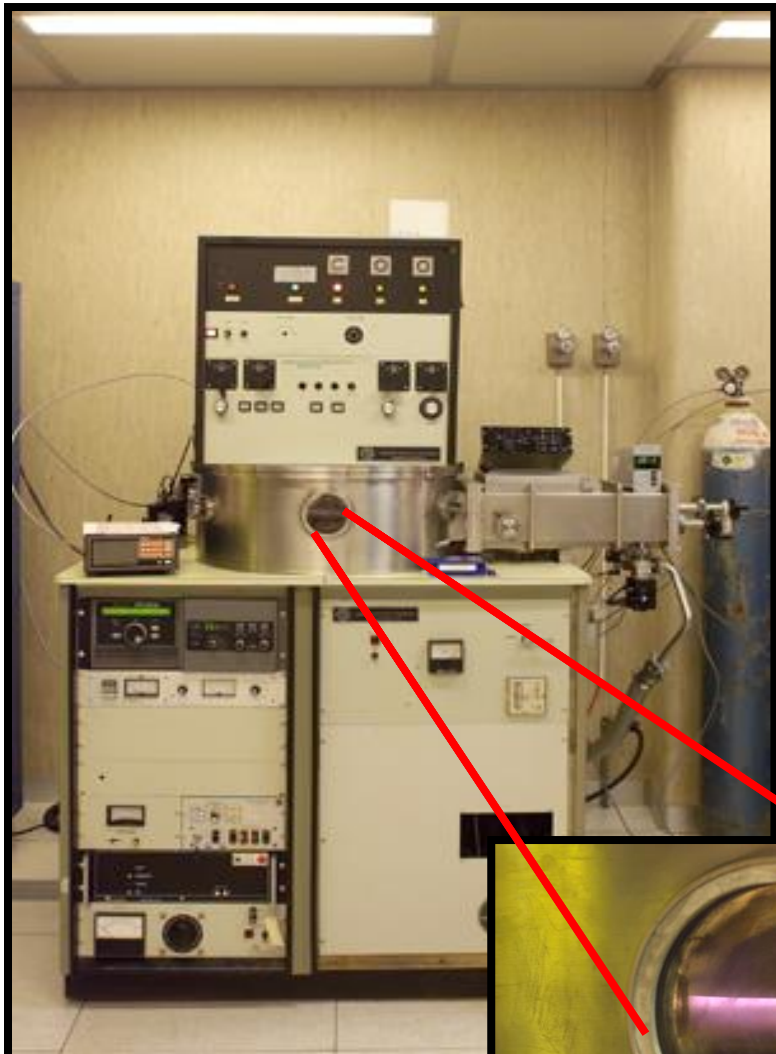
**Maximum growth rate**: 0.75 nm/s

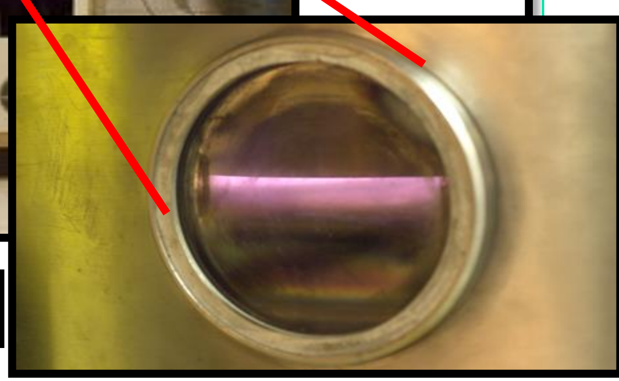
**Thickness uniformity**: > 90% on 90 × 50 $mm^2$ area

# Depostion facilities
## (ENEA Casaccia)

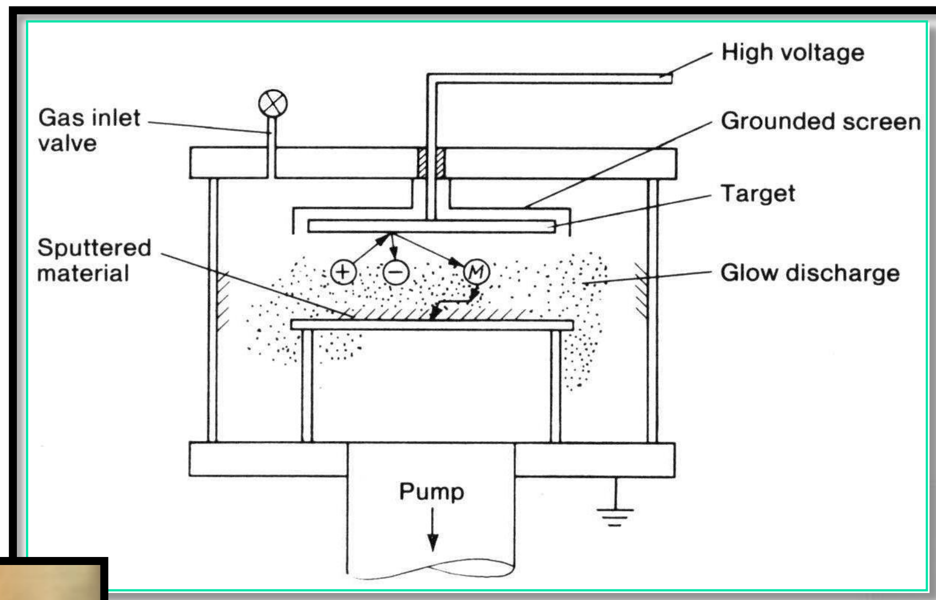
## *Deposition systems*: *radiofrequency sputtering*

- Radiofrequency sputtering system with 3 cathodes was used (target diameter 6.5 inches)
- Well suited for thin film deposition over 100 $cm^2$ surfcaes





**Glow discharge**

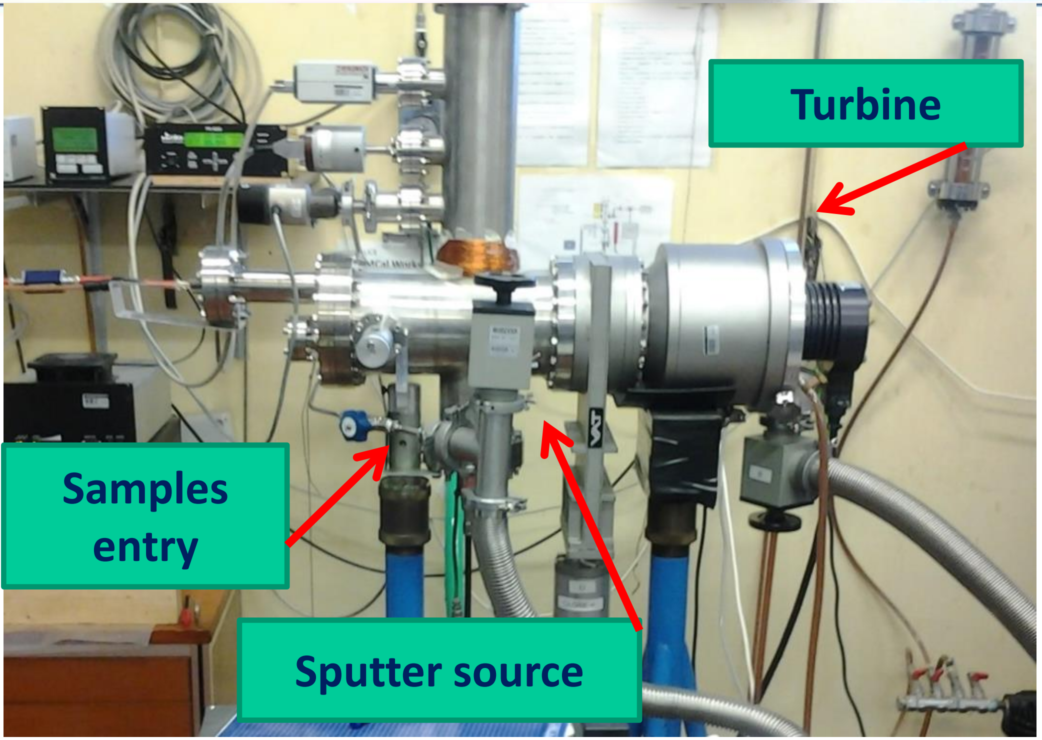


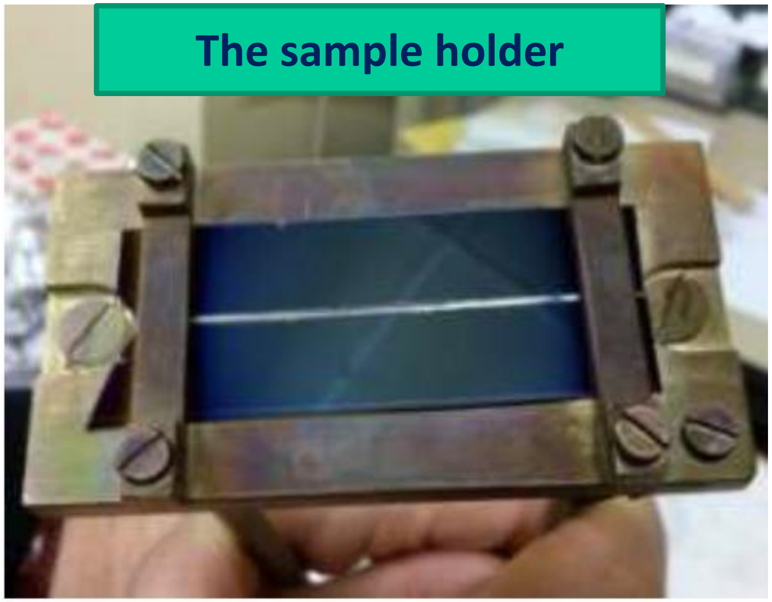

**Typical working pressure ~$10^{-3}$ mbar**

# Deposition facilities
(INFN-LNF)

## Sputtering deposition device

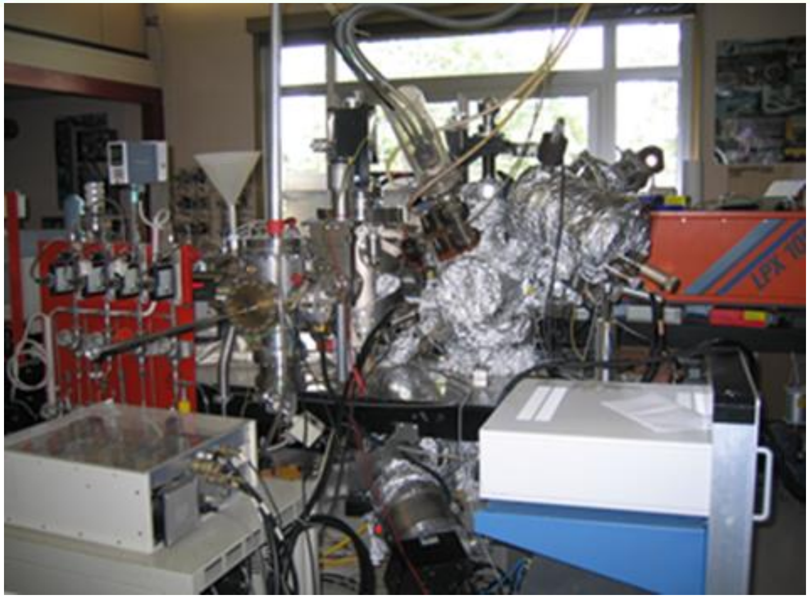
High Power Impulse Magnetron Sputtering (HPIMS) sputtering. The sputtering source is a MAK 2'' (50,8 mm).
Since October 2014 we are performing depositions of boron on rectangular silicon sheets (50 mm x 10 mm).
A control unit attached measures the thickness of the deposition by the oscillation frequency of a crystal placed inside the chamber.




| Features | Our settings |
|---|---|
| **Power** | 100 W - RF |
| **Distance target-substrate** | 50 mm  to 120 mm |
| **Thickness of the deposition** | 500 nm t0 1200 nm |
| **Deposition Rate** | 8 nm /min |
| **Vacuum** | 10E-6 mbar |
| **Pressure Argon** | 10E-3 mbar |

# Characterization tools
## (ENEA Frascati)

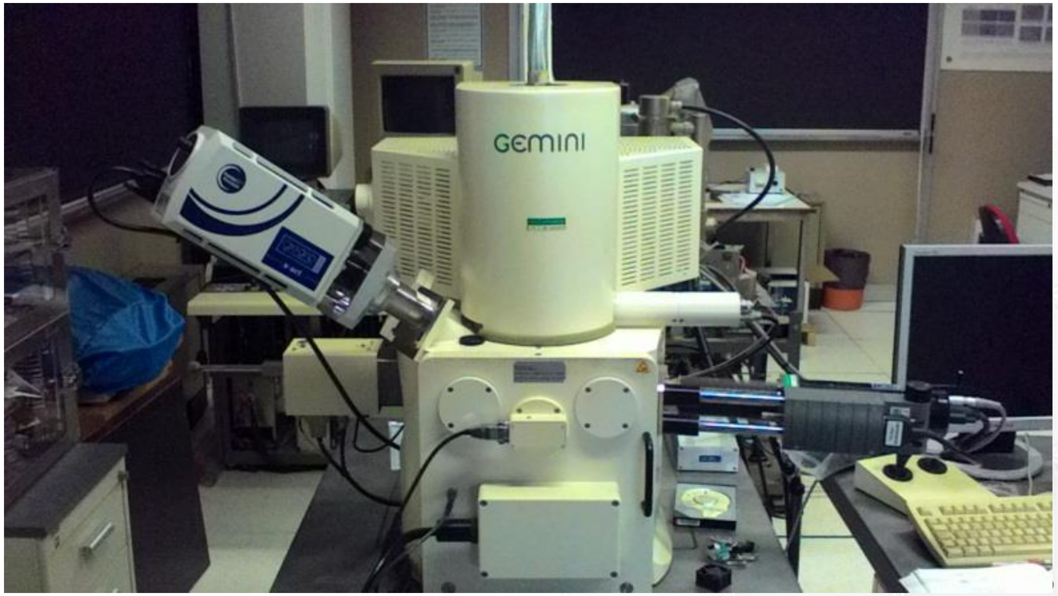


The XPS acquisition system operates in an ultra-high vacuum system with $4 \times 10^{-8}$ Pa base pressure.

It is equipped with a VG Al Kα monochromatized x-ray source and a CLAM2 hemispherical analyser.

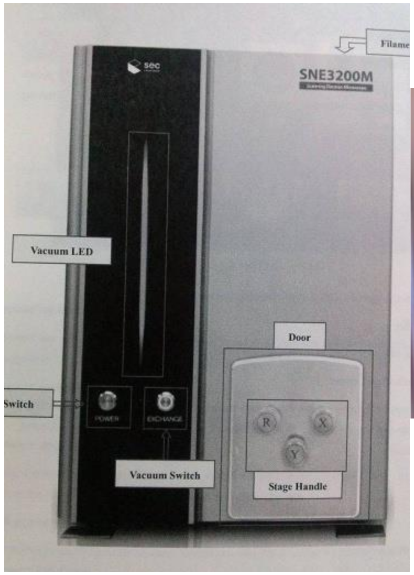
Morphological caracterization was performed with a Leo 1525 field emission scanning electron microscope (SEM) equipped with in-lens annular detector

Atomic Force Microscope



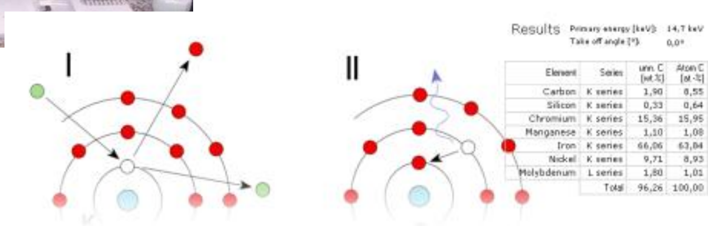
# Characterization tools
## (INFN-LNF)

### *Scanning Electron Microscope equipped with a X-flash detector*

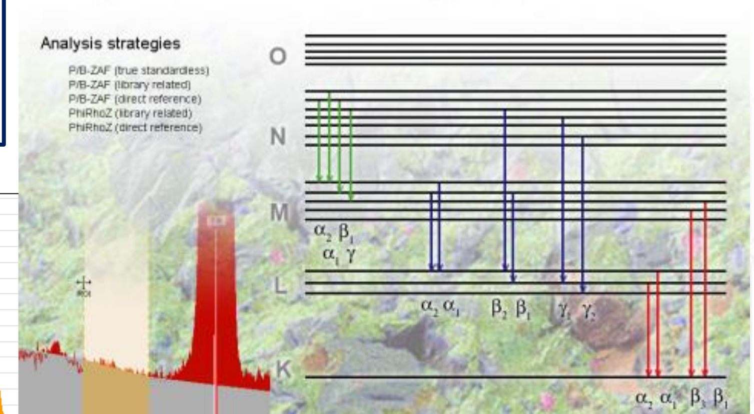


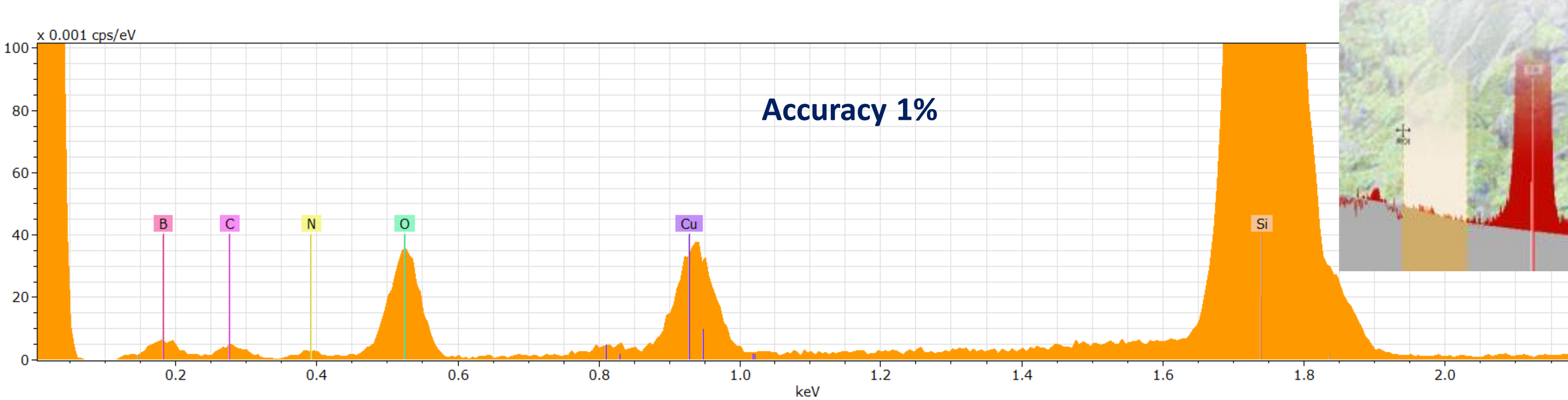



SE and BSE provide images of the specimen, revealing surface structure and topology
The X-ray spectometer provides detailed information about chemical composition.




The energy of the characteristic X-rays is nearly independent of the chemical bonding state of the affected atoms; the electron probe microanalysis (EPMA) is element sensible.
In the resulting energy dispersive spectrum X-rays correspond to visible peak to identify.



# Characterization tools
## (Univ. Roma Tre)

**Leica DCM-3D**

**non contact profilometer**

**confocal and interferometric profilometry**

The very low depth of field intrinsic of confocal microscopy is used to make z-scan maps of a sample;

2 nm z resolution at 150x magnification, 0,95 NA;

Use of a Mirau interferometer to measure even steps as small as 0,1 nm;

Lateral resolution diffraction limited, same as a conventional blue light microscope.

# Characterization tools
## (Univ. Roma Tre)

**Keysight G200 Nanoindenter**


Indenter using a three-sided diamond tip to obtain depth profiled elastic modulus and hardness plots;

2 measuring heads, a high displacement resolution (10 pm) and an ultrahigh resolution one (0,2 pm);

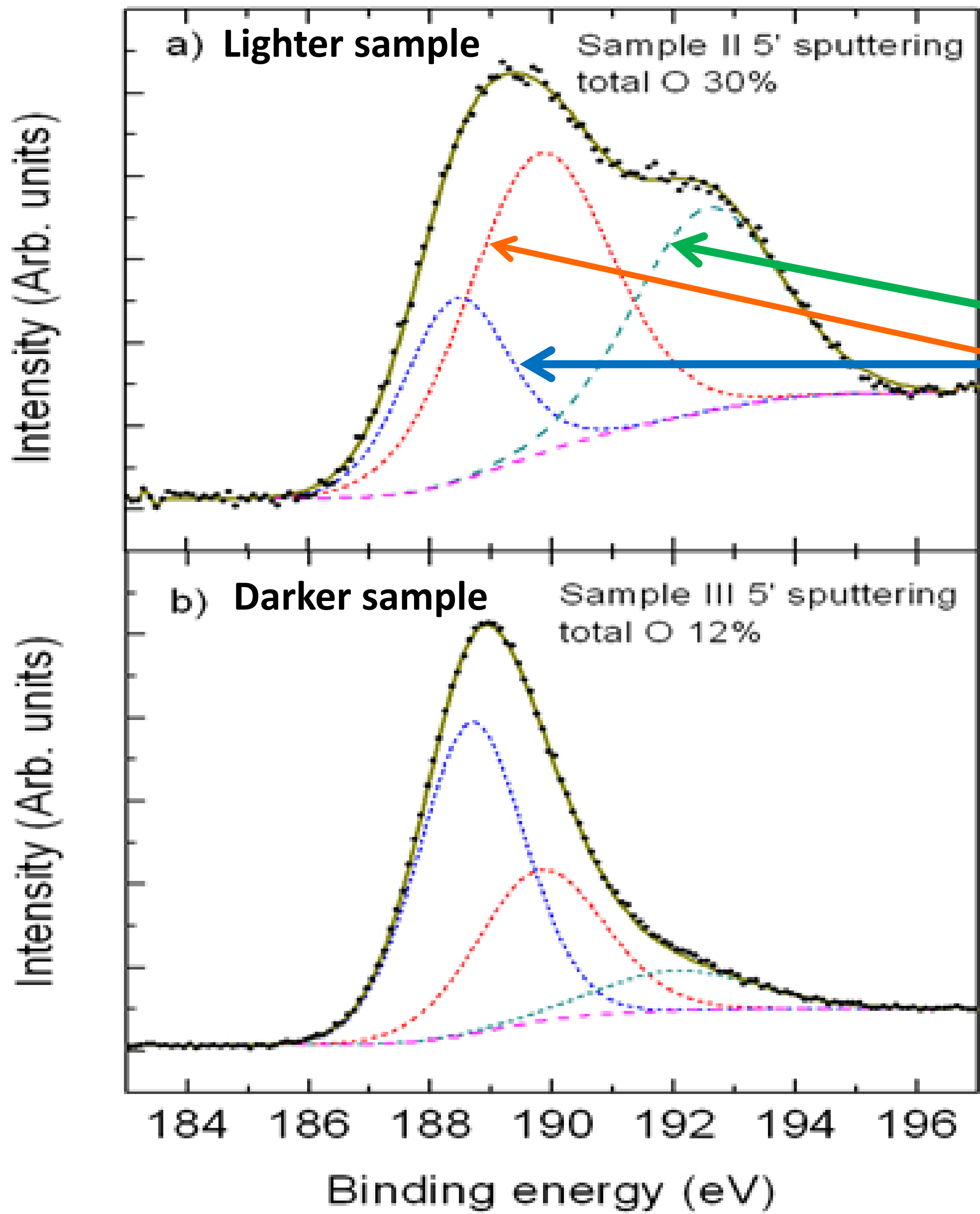
High speed mode capable of one indentation/sec;

High resolution head capable of scratch testing, lateral force resolution 2 µN, normal force resolution 0,05 µN.

# A brief overview on preliminary results

# X-Ray Photoelectron Spectroscopy-XPS

## surface chemico-physical analysis




**XPS spectra of depositions**

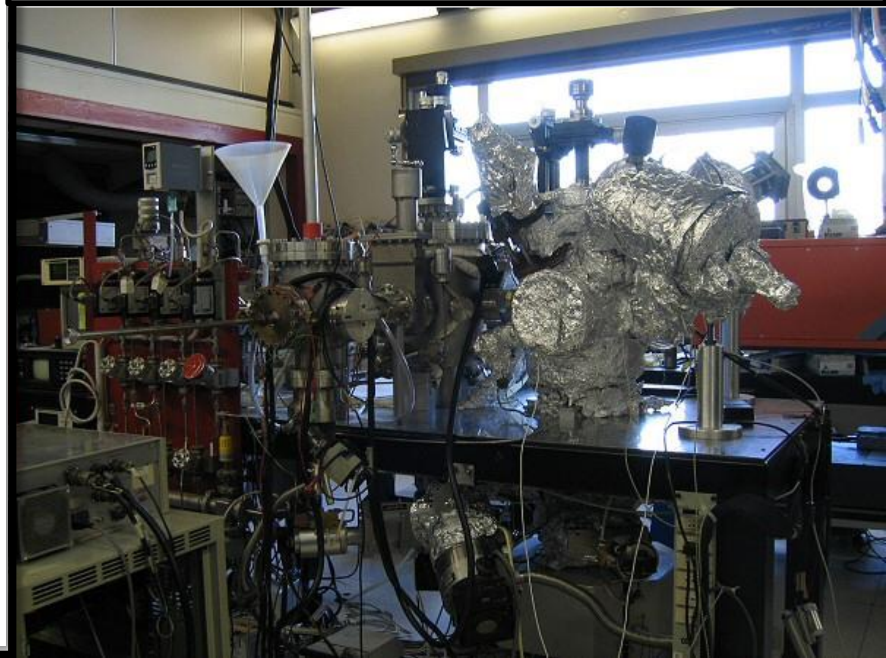
$E_B$ scan around the boron peak (tables) 3-components fit indicate chemical composition made of:

**metallic Boron**

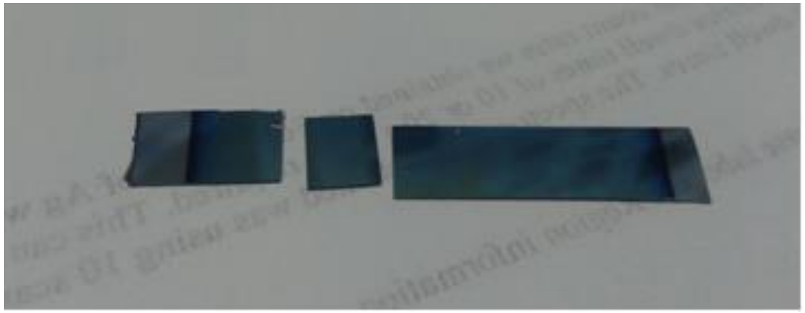
**$B_2O_3$**

**$B_xO_y$**

A pure element would appear as a Gaussian distribution



# A new set of depositions
## INFN sputtering system

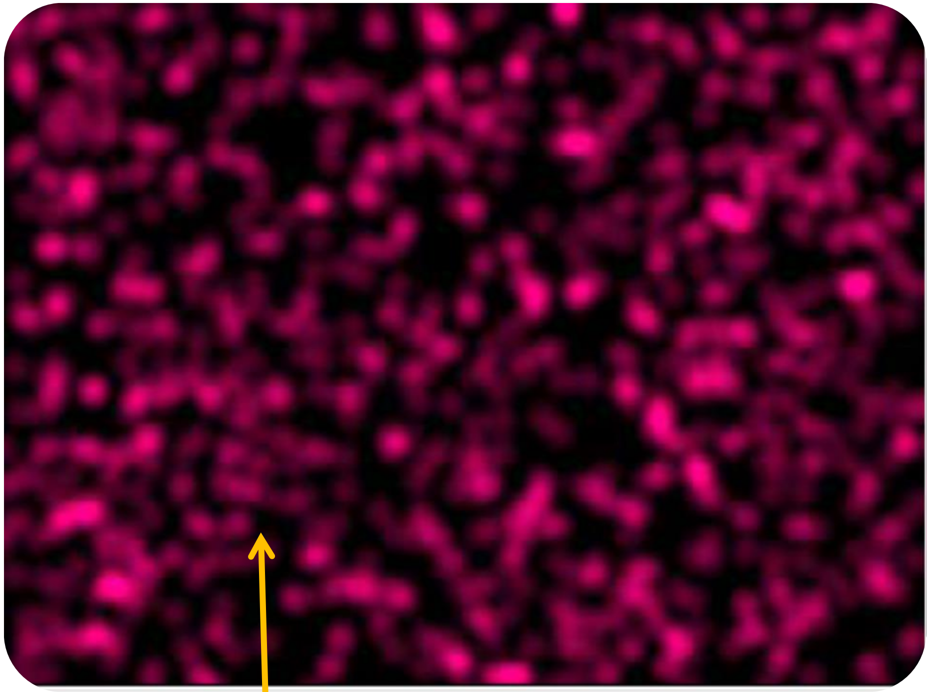


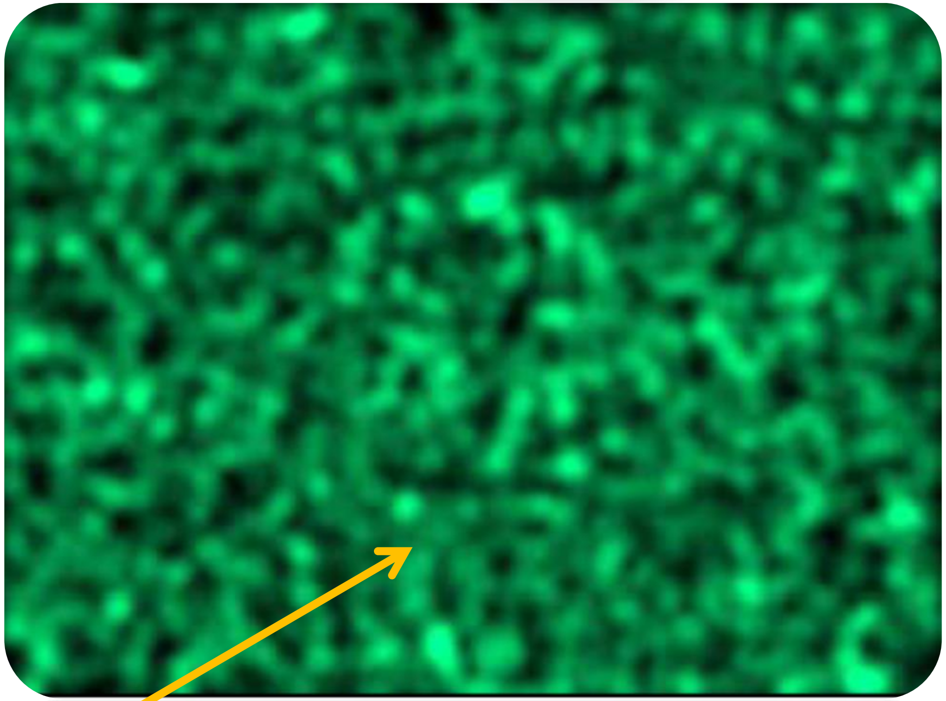
- We have been completed over 40 depositions on silicon by changing:
  - Type of silicon used,
  - Target-sample distance
  - Sample pre-treatment (etching, heating...)
  - B film thickness.
- Analyses were conducted using profilometry, SEM, optical microscope (500 x) and tape test to evaluate the adhesion “on the fly”

- The figure on the right shows a particularly homogeneous coating (500X) of a deposition from 1 μm
- The profilometer confirms the presence of a coating of 600 nm of boron
- Further research will be carried out to stabilize the oxidation reactions of boron. Oxidation leads to a strong degradation of the deposition.

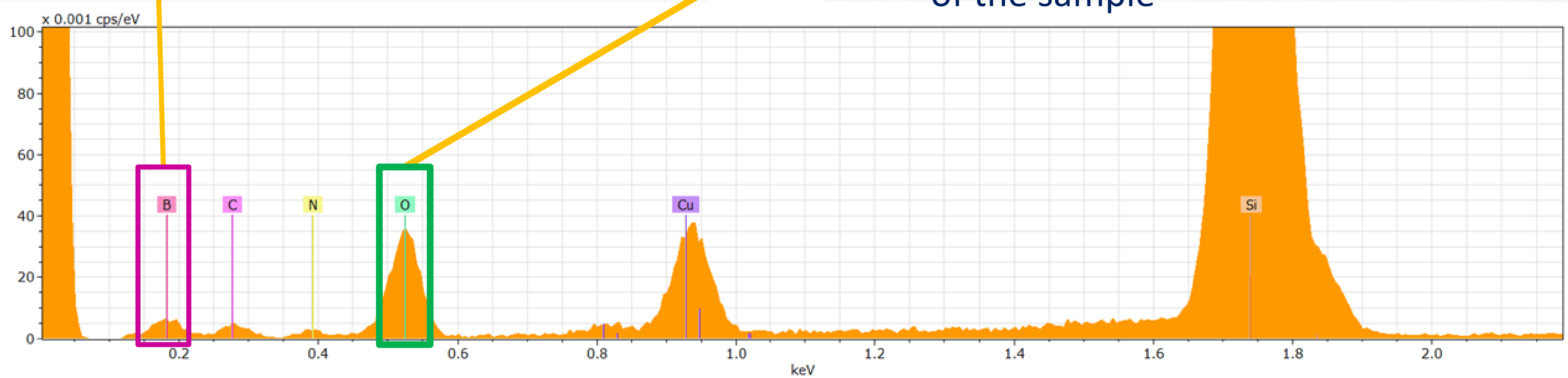
# SEM + X-flash detector

## Microanalysis tool





Fixing x-ray energy one obtains an intensit 2D map scanning a given area of the sample



# Conclusions

✓ We developed a side-on GEM-based thermal neutron detectors to reach high efficiency (50%) @ 25 meV neutron energy.

✓ The main activity to reach the goal is the deposition of **pure $^{10}B$** films (not an easy task) rather than $B_4C$ onto proper substrates (glass as a first attempt, alumina, aluminum and silicon).

✓ We started with e-beam but we are using/will use sputtering and Pulsed Laser Deposition

✓ We are using targets sinterized in a controlled environment and due (P,T) conditions to provide not porous target reducing internal oxidation.

✓ We are supported after deposition by several and complementary tools for mechanical and chemico-physical analysis of the deposited films

GEM SIDEON IS BEING DEVELOPED WITHIN A COLLABORATION OF PEOPLE COMING FROM DIFFERENT INSTITUTIONS AND WITH DIFFERENCE COMPETENCES

**Side-On GEM Collaboration network (deposition and on beam testing)**

# Collaboration

**Integrated Interface Activity**

AGENZIA NAZIONALE PER LE NUOVE TECNOLOGIE, L'ENERGIA E LO SVILUPPO ECONOMICO SOSTENIBILE

**E. Aza (CERN)**
**E. Bemporad (Roma Tre)**
**S. Cappello (INFN-LNF)**
**G. Celentano (ENEA)**
**G. Claps (ENEA)**
**G. Croci (CNR)**
**A. Grilli (INFN-LNF)**
**S. Loreti (ENEA)**
**R. Moscatelli (Roma Tre)**
**F. Murtas (INFN-LNF)**
**A. Pietropaolo (ENEA)**
**L. Quintieri (ENEA)**
**D. Raspino (ISIS)**
**R. A. Riedle (Oak Ridge)**
**M. Renzelli (Roma Tre)**
**A. Rufoloni (ENEA)**
**A. Santoni (ENEA)**
**E. Soldani (INFN-LNF)**
**A. Sytchkova (ENEA)**
**A. Vannozzi (ENEA)**
**A. Viticchiè (INFN)**

**Ongoing activity involves:**

- **Italian Institute of Technology**
- **Columbus**

**Some results already published**
**S-GEM tests:**

- NIMA A **729** (2013) 117–126
- EPL, **105** (2014) 22002

**Deposition analysis**

- Surf. Coat. Tech. in press (march 2015)

**Color legend**

| | |
|---|---|
| **Black**: | test on beam |
| **Red**: | mechanical characterization |
| **Yellow:** | Chemico-physical analyss |
| **Green**: | MC simulation |
| **Violet:** | deposition |