



UNIVERSITÉ  
CAEN  
NORMANDIE

# IRRADIATION AND CHARACTERIZATION FOR MATERIAL SCIENCE AT CIRIL-GANIL FACILITY

Platform for welcoming interdisciplinary researches at GANIL, CIRIL  
Research center on Ions, Materials and Photonic, CIMAP



Dr Clara GRYGIEL

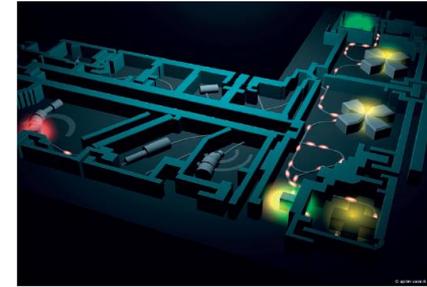


IRRSUD beamline coordinator  
Deputy director of EMIR&A network

[grygiel@ganil.fr](mailto:grygiel@ganil.fr)



GANIL campus



GANIL + SPIRAL1

**1983:** First experiment

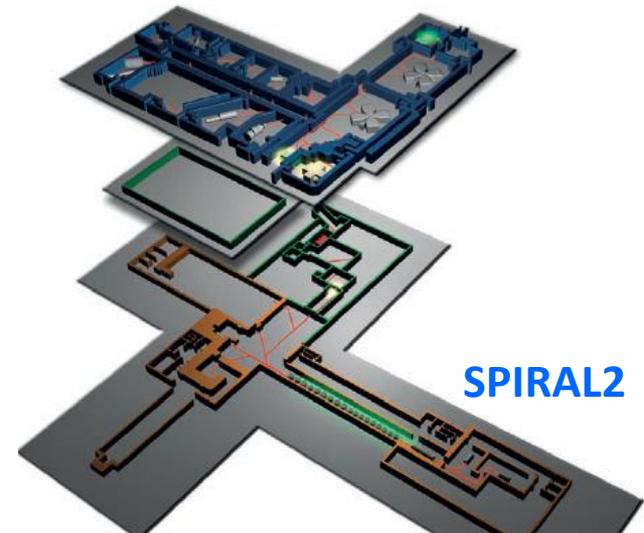
➤ 39 years of running

**Users:** 600 physicists (50% foreigners)

**Beam:** about 5500 hours per year

2000 hours per year as pilot # High Energy  
+ 1000 hours in parallel: Medium Energy  
+ 1500 hours in IRRSUD  
+ 1000 hours in ARIBE

*Average numbers*



SPIRAL2



*Run the accelerator facility and Nuclear physics research*

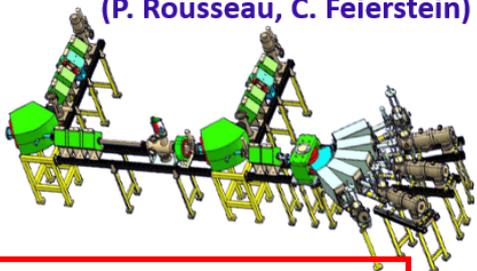


*Run interdisciplinary physics research  
(materials science, astrophysics, atomic physics and radiobiology)*

# BEAMLINES FOR INTERDISCIPLINARY RESEARCH

## Out of INB : ARIBE

(P. Rousseau, C. Feierstein)

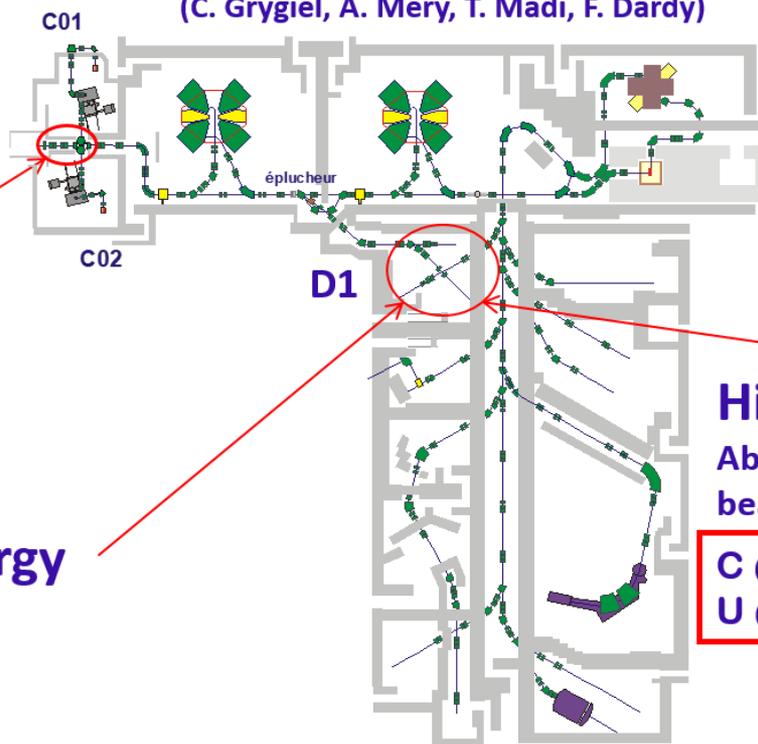


From He<sup>+</sup> to Xe<sup>31+</sup> 18 kV

## CIRIL platform (J. Rangama, F. Ropars)

### INB : IRRSUD+SME+HE

(C. Grygiel, A. Méry, T. Madi, F. Dardy)



### IRRSUD

≈ 1 MeV/A

### Medium Energy

≈ 10 MeV/A

D1

### High Energy

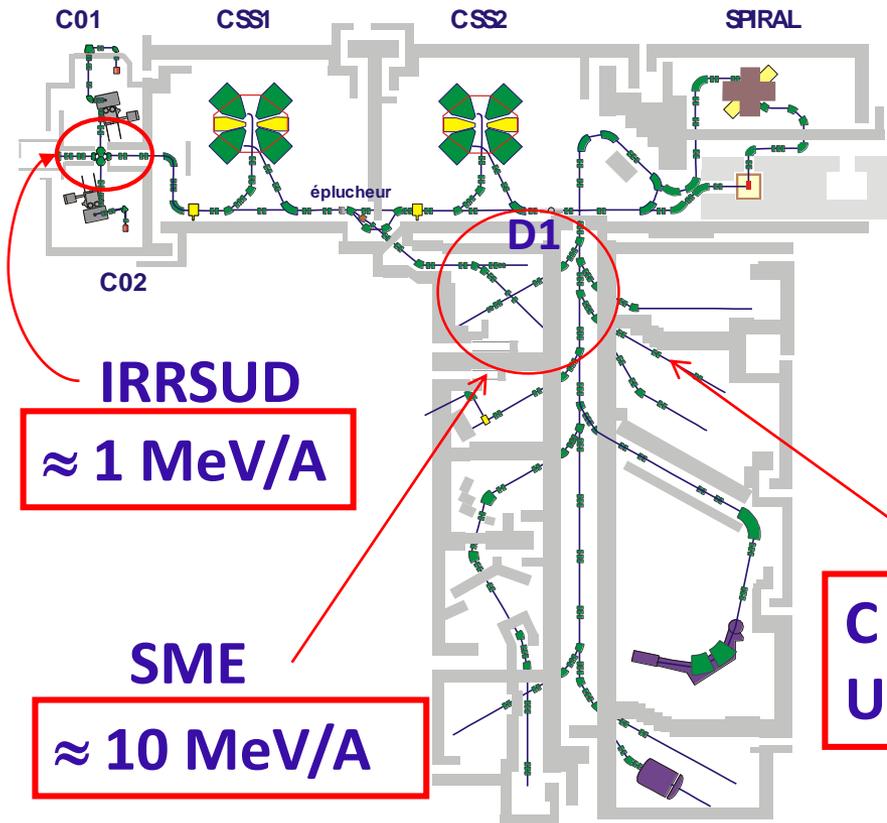
About 10% of pilot beam

C @ 95 MeV/A

U @ 24 MeV/A

# HIGH ENERGIES WITH CYCLOTRONS

## Heavy ions at higher energies (from C to U)



flux up to  $10^{10}$  ions.cm<sup>-2</sup>.s<sup>-1</sup>

Rp to some hundred microns

Continuous measurement of flux

Beam sweeping up to 30cm<sup>2</sup>

IRRSUD

≈ 1 MeV/A

SME

≈ 10 MeV/A

HE

C @ 95 MeV/A

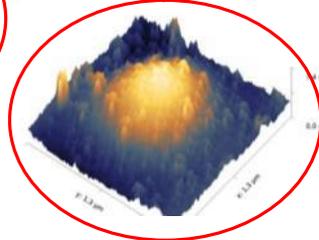
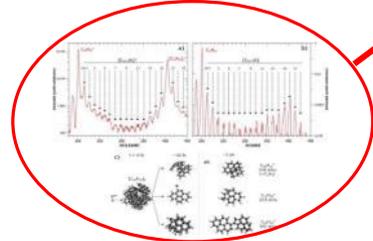
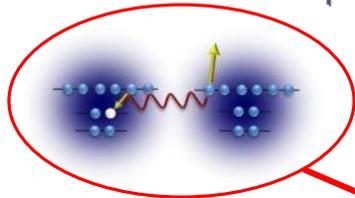
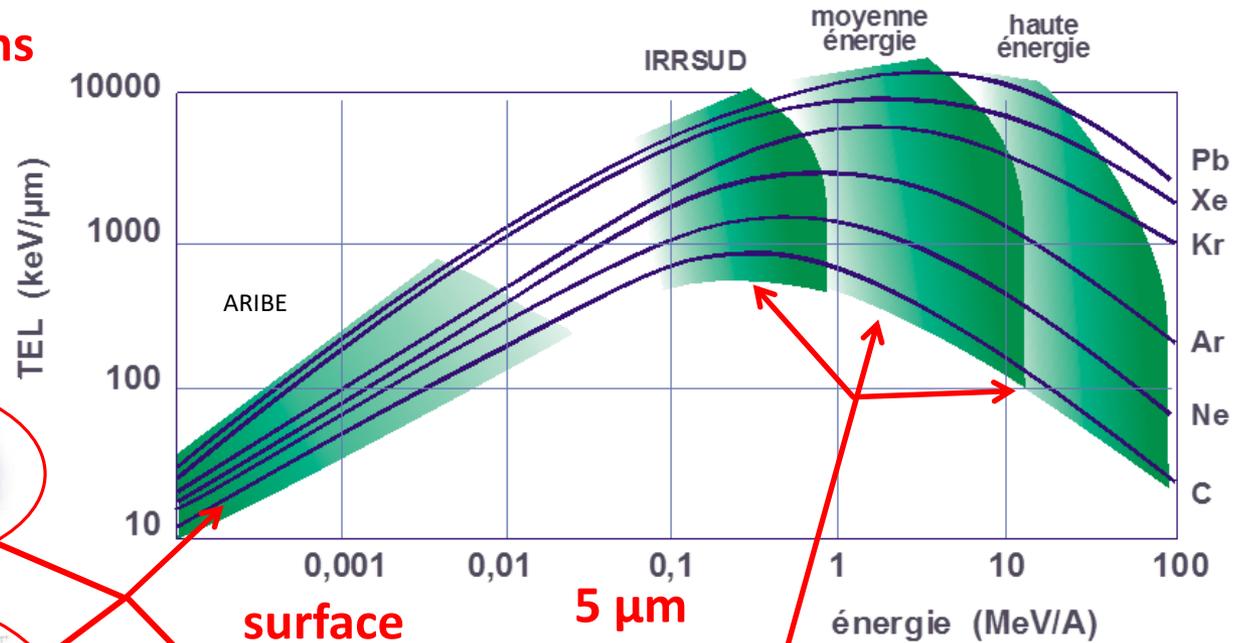
U @ 24 MeV/A

# INTERDISCIPLINARY PHYSICS AT GANIL

## ENERGY RANGE

Highly charged ions  
Potential energy

Swift heavy ions  
Electronic excitations



surface

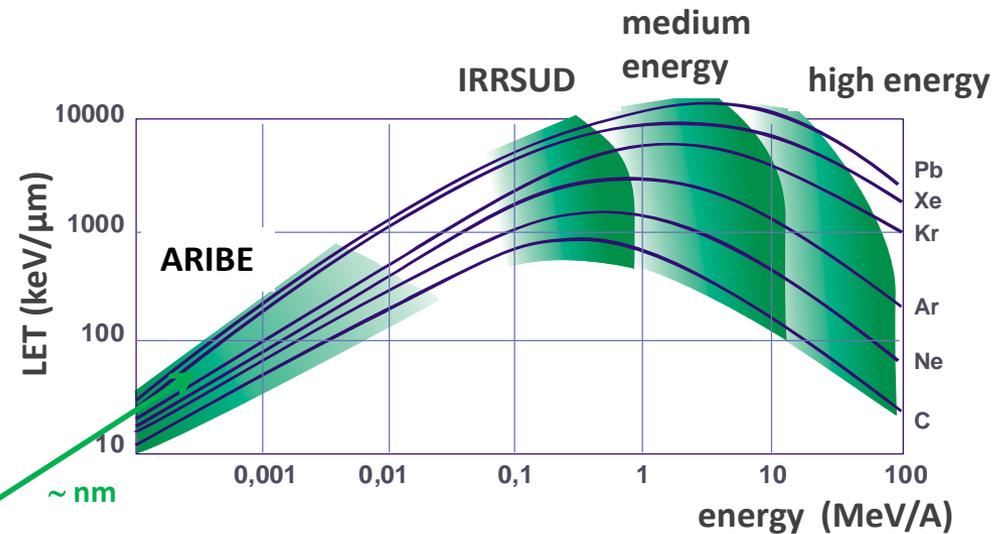
5 μm  
> 10<sup>15</sup> cm<sup>-2</sup>



100 μm

1500 μm  
10<sup>13</sup> cm<sup>-2</sup>

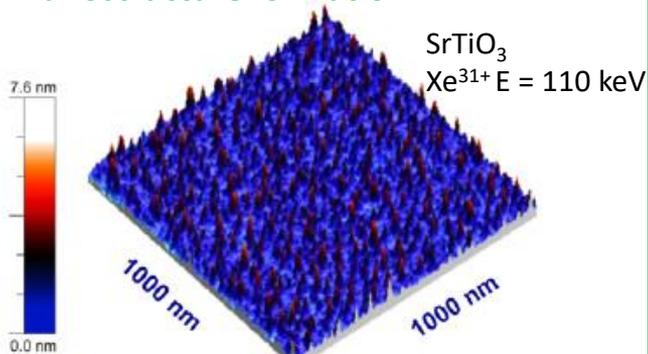
# GANIL FACILITY BEAMLINES VS MATERIAL MODIFICATIONS



## Potential energy deposition $E_p$

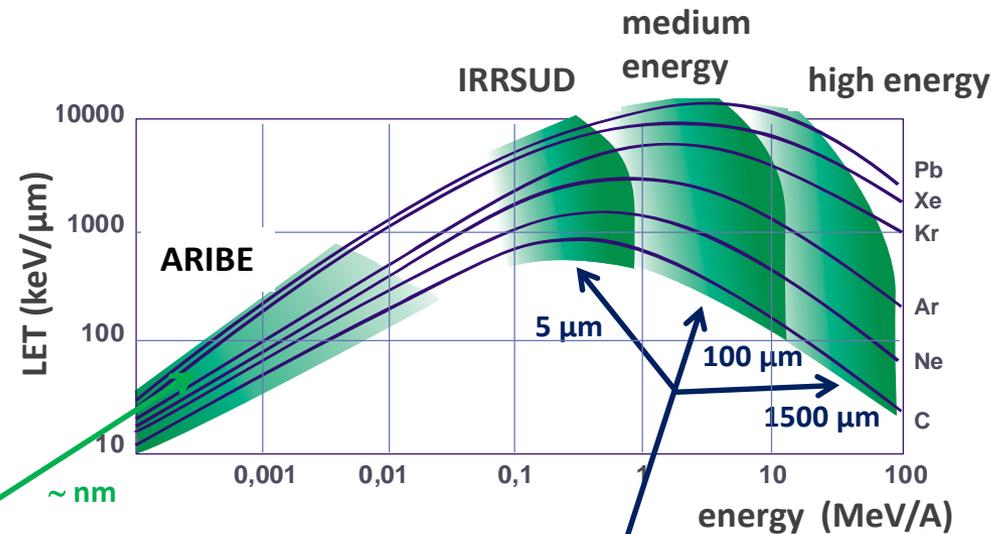
Capture, decay, emission, sputtering

>> Nanostructure formation



El Said et al, NIMB 269-1234(2011)  
Aumayr et al, JPCPM 23-393001(2011)

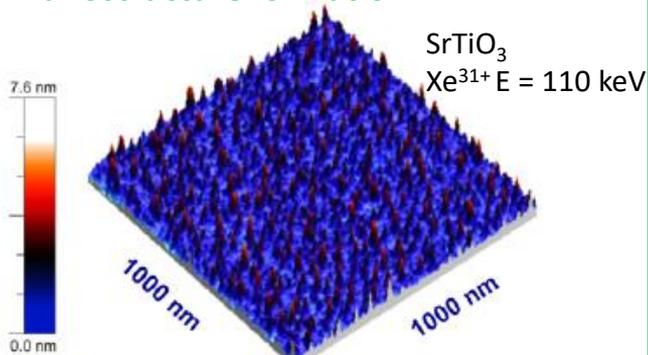
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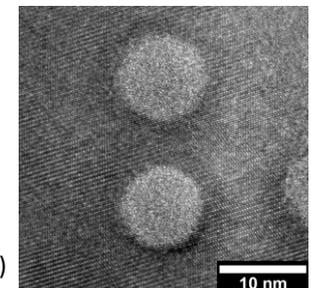
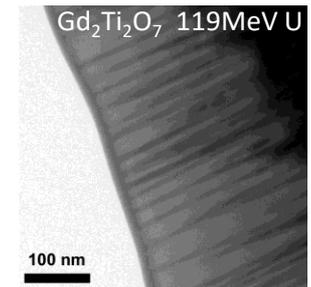
## Electronic energy loss $S_e$

Defect formation by radiolysis

Collective effect: atomic motions by intense electronic excitations -> extended defects

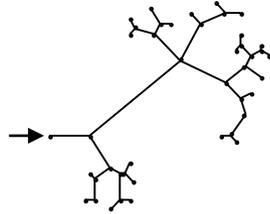
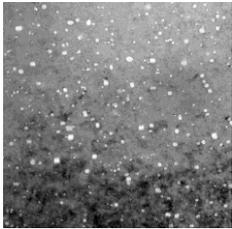
>> Track formation, disorder, phase transitions, surface nanostructures

Sattonnay et al, Acta Mat 60-22 (2012)



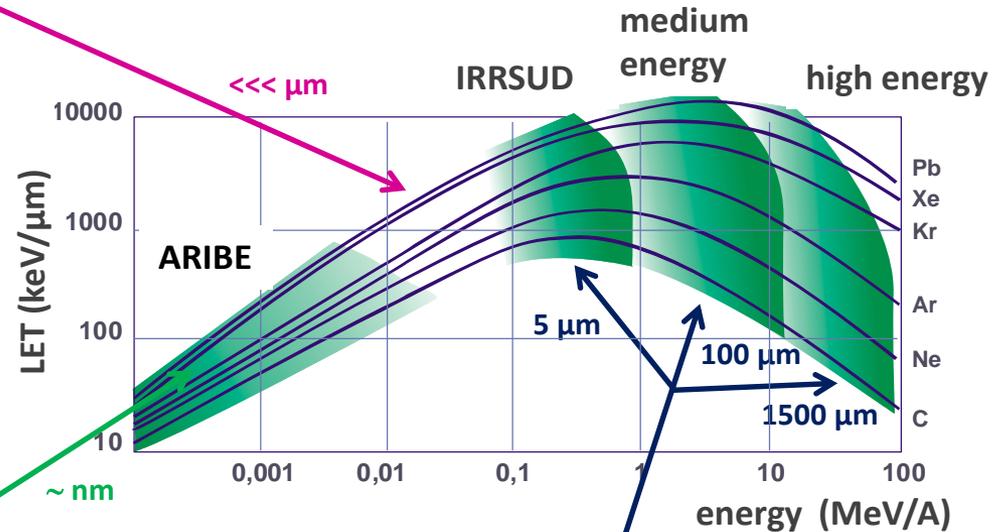
# GANIL FACILITY BEAMLINES VS MATERIAL MODIFICATIONS

## Nuclear energy loss $S_n$



Frenkel pairs, Displacement cascades

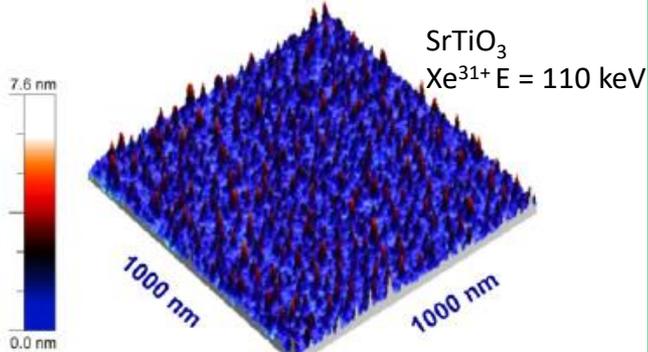
>> Cavities, bubbles, dislocation loops, segregation, precipitation, amorphization, phase transitions



## Potential energy deposition $E_p$

Capture, decay, emission, sputtering

>> Nanostructure formation



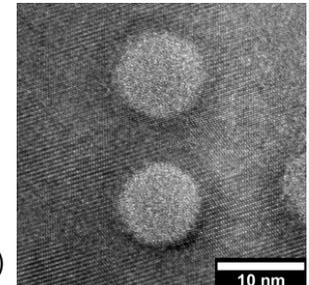
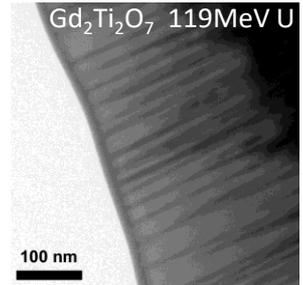
El Said et al, NIMB 269-1234(2011)  
Aumayr et al, JPCPM 23-393001(2011)

## Electronic energy loss $S_e$

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Collective effect: atomic motions by intense electronic excitations -> extended defects

>> Track formation, disorder, phase transitions, surface nanostructures



Sattonnay et al, Acta Mat 60-22 (2012)

# IRRSUD : SOME NUMBERS

SRIM

$^{36}\text{Ar}^{10+}$   
 0,98 MeV/A  
 # 35 MeV



*average flux, RT:  $5^e9$  ions/cm<sup>2</sup>.s  
 In 24H : fluence  $4,3^e14$ ions/cm<sup>2</sup> at IRRSUD  
 (Rastered beam on 25cm<sup>2</sup>)*

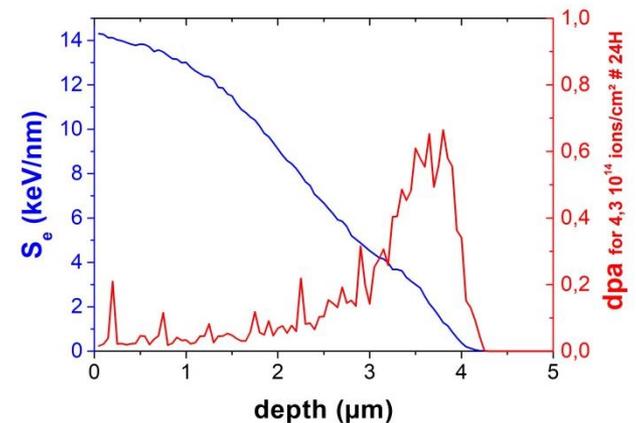
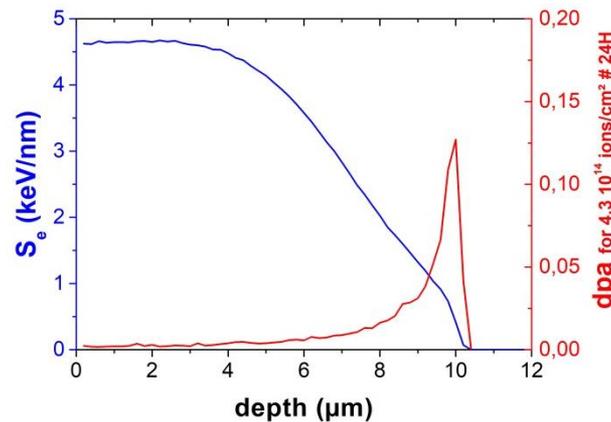
*Max:  $5e10$ - $1e11$ ions/cm<sup>2</sup>.s*

C 1,8g/cc

Rp	10,4 μm
Se	4,61 keV/nm
Sn	0,0079 keV/nm
ENSP surface	584

W 19,35g/cc

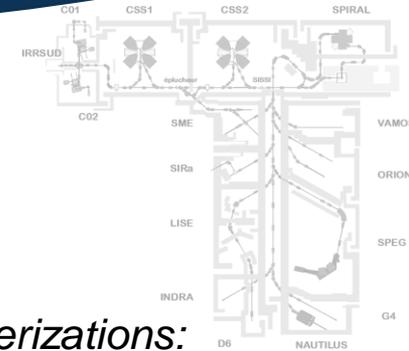
Rp	4,2 μm
Se	14,24 keV/nm
Sn	0,0417 keV/nm
ENSP surface	341



# IN-BEAM CONDITIONS

**Ions:** C to U *multicharged*

**Energy:** keV up to 0.3 – 100 MeV/A



*Online setups for material characterizations:*

**Beam scanning**

**Temperature**  
8 - 1300K

**Incidence**  
90° - 0°

**Gas partial pressure**

**+ Nuclear materials**

**XRD**

ALIX IRRSUD

**Absorption spectro**

FTIR  
UV-Vis



CASIMIR 15-300K  
CESIR RT  
DETAIL up to 1400K

**Emission spectro**

**Gas release**

FTIR  
Mass spectroscopy

**Sputtering**

TOF or catchers  
AODO (X-Y TOF SIMS in situ LEED Auger)

**External user setups**

ex: *MiniMecaSiC* (CEA-DEN) or Raman (Orléans)

# GAS RELEASE



## GRAL

**FTIR absorption of the gas mixture**

Small hydrocarbons (up to 3 Carbons)

*→ Track creation at high stopping power in organic compounds*

Picq et al, NIM B 151 - 76 (1999)



## CIGAL

**mass spectrometry**

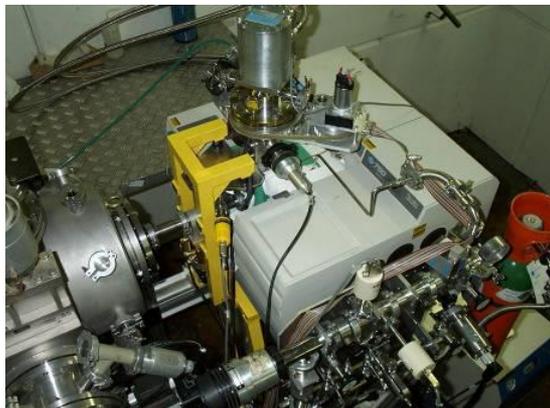
H<sub>2</sub> formation, O<sub>2</sub> consumption, CO, CO<sub>2</sub>

*→ Gas production during nuclear waste storage of organic compounds contaminated with actinides as Pu (gloves, ...)*

Dely et al, NIM B 236 - 145 (2005)

# ABSORPTION SPECTROSCOPY

Vibrational spectroscopy: **FTIR**,  $8000 - 400 \text{ cm}^{-1}$  Electronic spectroscopy: **UV-vis**  $200 - 900 \text{ nm}$



## CASIMIR

15 K - 300 K

- Nitrides
- Polymers, fundamental mechanisms
- Effects in solids relevant to astrophysics and astrochemistry

Sall et al, EPL 102- 26002 (2013)

Balanzat et al, NIM B 105 - 1 (1995)

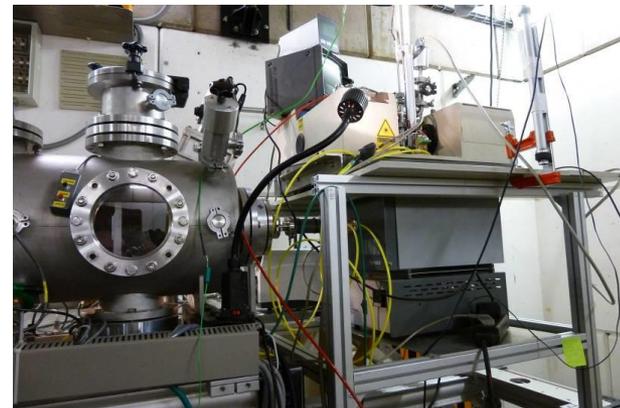
Pilling et al, Astronom & Astrophys 509 – A87 (2010)



## CESIR

Room temperature

Melot et al, NIM B 208 - 345 (2003)



## DETAIL

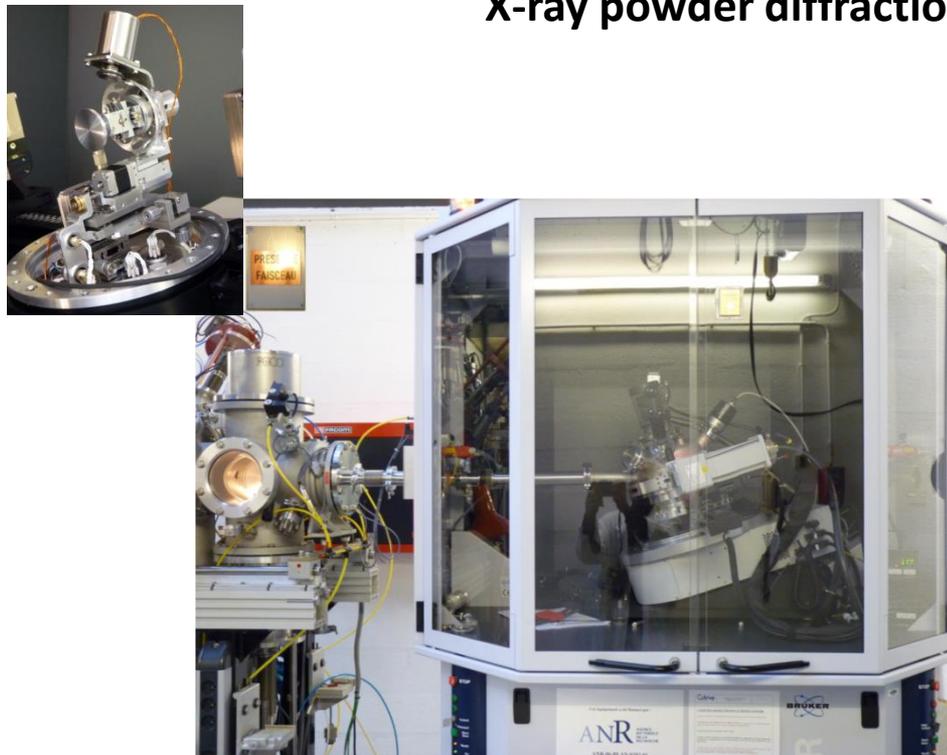
High temperature & FTIR  
Up to  $1100 \text{ }^\circ\text{C}$

## CHESOI

Special low volume chamber for rare gas atmospheres ( $^{18}\text{O}_2$ )

# STRUCTURAL - MICROSTRUCTURAL ANALYSES

## X-ray powder diffraction



“ALIX” setup

Only on IRRSUD

grazing incidence diffraction  
simultaneous or sequential

→ *Phase transition & structural modifications*

→ *Kinetics studies*

Grygiel et al, RSI 83-013902 (2012)  
Sattonnay et al, Acta Mat (2013)  
Grygiel et al, Acta Mat 140, 157 (2017)

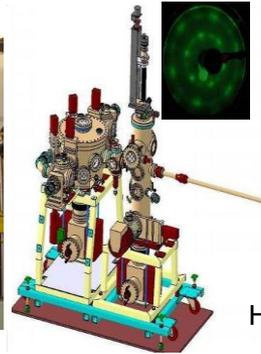
*CHEXPIR, on ME not anymore available*

# SPUTTERING

High energy



Low energy



**AODO**  
(X-Y TOF SIMS)

UHV  
In-situ LEED - Auger

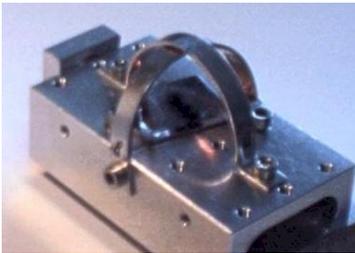
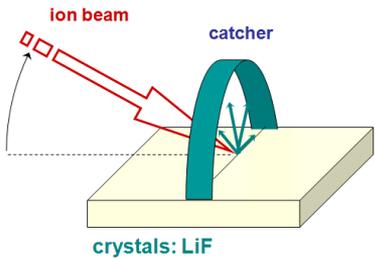
Hijazi et al, NIM B 269 – 1003 (2011)

Time of flight

charged particles

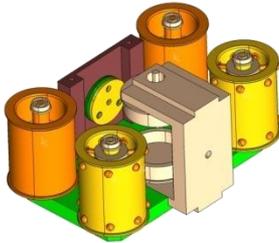
Catcher

neutrals



Ex-situ analysis by RBS, ERDA, ...

Toulemonde et al, NIM B 212 – 346 (2013)



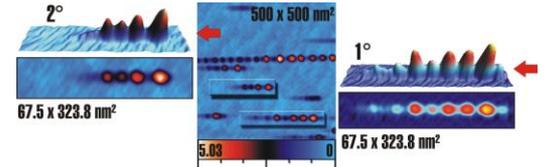
In-situ Auger

Salou et al, RSI 84-095115 (2013)

# USER'S INSTRUMENTATION



## In-situ UHV AFM



→ Creation of multiple nanodots by single ions, foldings on 2D

Akcoeltek et al, Nature Nanotech 2 - 290 (2007)

H Lebius, M Schleberger Nature Nanotech 2, 290(2007), New J Phys 10-053007(2008)

E Gruber et al, J. Phys.: Condens. Matter 28, 405001 (2016); 30, 285001 (2018)

Collab Prof M. Schleberger, Uni. Duisburg-Essen, Germany

## In-situ traction on SiC fibers



Jankowiak et al, NIM B (2013)

# POST-IRRADIATION EXPERIMENT TECHNIQUES

## Structural Characterizations:

- High Resolution X-Ray Diffraction  
D8 discover Bruker, Cu anode  
4-circles, in-plane geometry
- Transmission/Scanning Electron Microscopy and FIB  
ARM JEOL 200kV equipped with EELS (Gatan)  
HELIOS NANOLAB 660 FIB  
PIPS II Gatan
- Raman spectroscopy



## Surface Characterizations:

- Atom Force Microscopy  
NX10 Parks Systems



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-- and many others...

# CIMAP-GANIL FACILITY

## 4 Beam lines/4 Energy ranges

- Access through call for proposals (iPAC, EMIR&A, RADIATE)
- Accepted proposals: beamtime during 3 years in backlog
- 2023: 4/5 months of beamtime (march -> july)
- Beamline renovation work: IRRSUD 2022, ME 2023, HE 2024
- Cyclotron renovation at GANIL (not yet scheduled)
- Beamtime free for scientific collaborations
- In-situ and Post-irradiation techniques are available  
PIE: various costs depending on techniques (mainly for MEB/FIB/TEM)

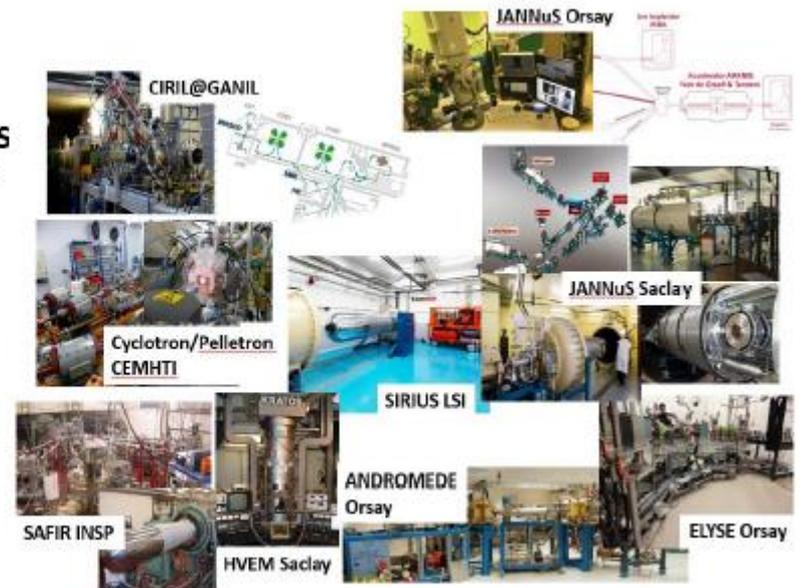


# IRRSUD AND ME BEAMLINES PART OF EMIR&A

## EMIR&A

French Network of Accelerators  
for Irradiation and Analysis of  
Molecules and Materials

<http://emir.in2p3.fr>



Director: Dr Nathalie MONCOFFRE

Deputy directors: Dr Marie-France BARTHE  
Dr Clara GRYGIEL



# EMIR&A NETWORK



**Research Federation (FR)** of CNRS, linked mainly to 3 Institutes :  
Institute of Physics (INP), Institute of Nuclear and Particle Physics (IN<sub>2</sub>P<sub>3</sub>) and Institute of Chemistry (INC)

**Research Infrastructure** on the MESRI National roadmap



**15 accelerators, 11 ion and electron irradiation platforms on 6 sites**

From fundamental research to Applications

- Irradiation effect in materials and molecules (including radiolysis)
- Ion beam analysis



# EMIR&A NETWORK

- **Orléans**, CEMHTI -, UPR CNRS/INC, Université d'Orléans  
Cyclotron 45 MV, Pelletron 3 MV
- **Caen**, CIMAP -, UMR CEA/DRF/IRAMIS, CNRS/INP, ENSICAEN, Univ. Caen-Normandie  
SME, IRRSUD
- **Orsay** IJCLab - UMR CNRS/IN2P3, Université Paris-Saclay  
JANNuS –Orsay : IRMA 190 keV implantor, ARAMIS, Tandem-VdG 2 MV  
Andromède, 4 MV Pelletron  
ICP –Université Paris-Saclay  
ELYSE, 9 MeV electrons
- **Palaiseau**  
LSI - UMR CEA/DRF/IRAMIS, CNRS/INP, École Polytechnique  
SIRIUS 2.5 MeV electrons
- **CEA Saclay**  
JANNuS Saclay: EPIMETHEE, JAPET, PANDORE  
HVTEM 1.2 MeV electrons  
ALIENOR 10 MeV electrons  
LEEL Microbeam
- **Paris-Sorbonne Université, INSP**  
SAFIR, 2.5 MV VdG

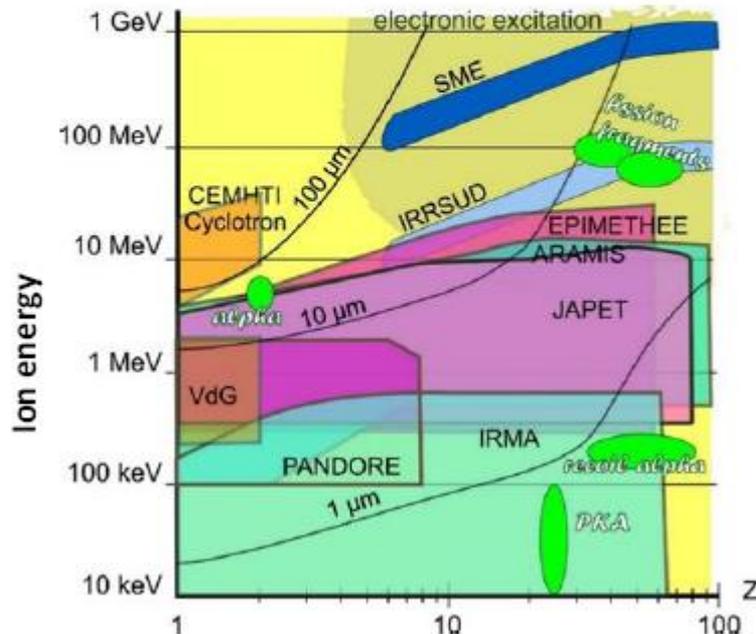


# EMIR&A NETWORK

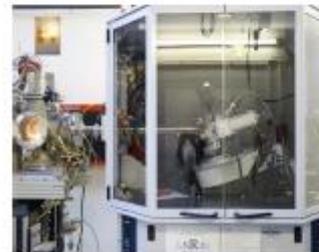
These platforms are highly complementary for irradiation and material analysis



**Irradiation:** These installations cover a very wide range Z - energy  
 $Z = 1 \rightarrow 92$  and 10 keV to GeV



**in situ characterisation:** They offer original and high-performance online instrumentation:  
 transmission electron microscopy, Raman spectroscopy, X-ray diffraction, optical spectroscopy, ion beam analysis techniques (Rutherford backscattering, nuclear reactions, ...).



*in situ* XRD diffractometer  
 IRRISUD-GANIL



TEM Jannus-Orsay  
 (© C. Baumier  
 (IJCLab))

<https://emira.in2p3.fr/>

# EMIR&A NETWORK

## EMIR&A

Set of accelerators and instrumentations made available to **users everywhere in the world**, after evaluation by an international scientific committee.

→ EMIR&A : <http://emir.in2p3.fr/>

- One call for proposal per year (mid-October)
- Pump priming possibility for Ion Beam Analysis proposals



# Thank you for your attention

*Ciril*



<http://cimap.ensicaen.fr/>



<https://www.ganil-spiral2.eu/>



<https://emira.in2p3.fr/>