



The JANNUS-Orsay / SCALP platform

Presentation at the Scientific Council of the National Institute of Nuclear Physics and Particle Physics (IN2P3)

Tuesday 25th February 2020, Paris

SCALP Synthesis and Characterization using ion **AcceLerators** for **Pluridisciplinary** research

JANNUS Joint **Accelerators** for **Nanoscience** and **Nuclear Simulation**



Operations manager: Dr Cyril Bachelet
Scientific leader: Dr Aurélie Gentils

Outline

Description of the platform

Equipment

Governance structure

Networks

Beam time allocation

Resources

Distribution of users and scientific themes

Scientific issues

Nuclear materials

Nuclear physics and astrophysics

Radionuclides production for health

Miscellaneous

Perspectives

The JANNuS-Orsay / SCALP platform



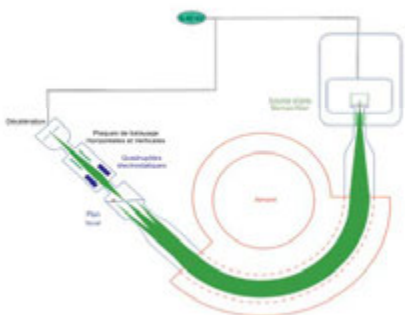
Joint Accelerators for Nanoscience and Nuclear Simulation

✓ Home-made ion accelerators

ARAMIS SINCE 1987
2 MV Tandem – Van de Graaff

SNICS negative ion source:
500 keV < E < 11 MeV ; 10 nA < i < 10 μA
Penning source @ HV : i < 20 μA
200 keV < He < 3.6 MeV ; 200 keV < H < 1.8 MeV

IRMA SINCE 1979
190 kV ion implanter
10 - 570 keV
up to 20 mA



SIDONIE SINCE 1967
50 kV isotope separator
50 eV – 150 keV
up to 20 mA, M/ΔM > 1000

Ion Beam Analysis
RBS, RBS/C, ERDA,
PIXE, μPIXE, PIGE

Ion implantation / irradiation
LN₂ → 1000°C

**in situ RBS/C
and ion impl.**
LN₂ → 600°C

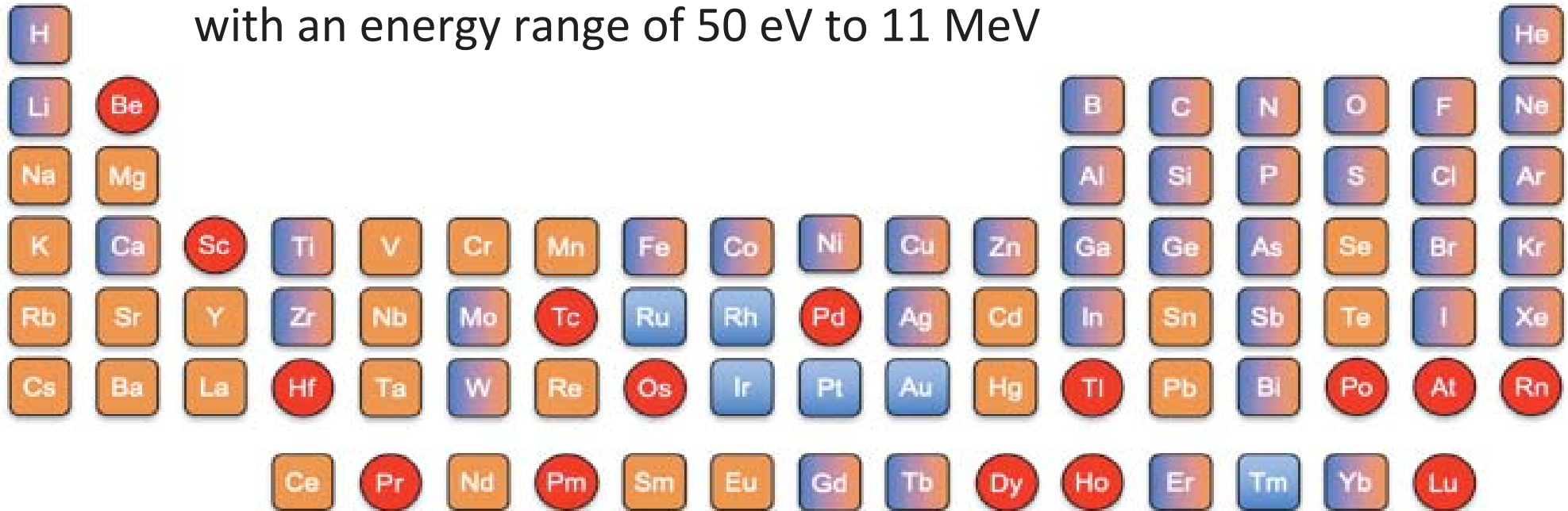
IN SITU DUAL ION BEAM TEM
SINCE 1980 UPDATED IN 2007
TRANSMISSION ELECTRON MICROSCOPE
200 kV FEI Tecnai G² F20 Twin
Resolution: 0.27 nm
Magnification range: x 70-700 000
EDX, GIF (EELS, EFTEM...), STEM
-170°C up to 1000°C

✓ *In situ* measurements using IBA and TEM with one or two ion beams



Large diversity of ions available (nature, energy)

71 chemical elements available,
with an energy range of 50 eV to 11 MeV



ARAMIS
Available with ARAMIS

IRMA + SIDONIE
Available with IRMA and SIDONIE

ARAMIS
Available with ARAMIS, IRMA and SIDONIE

Not available



100 eV

1 keV

10 keV

100 keV

1 MeV

10 MeV



A unique *in situ* dual ion beam TEM

Transmission Electron Microscope

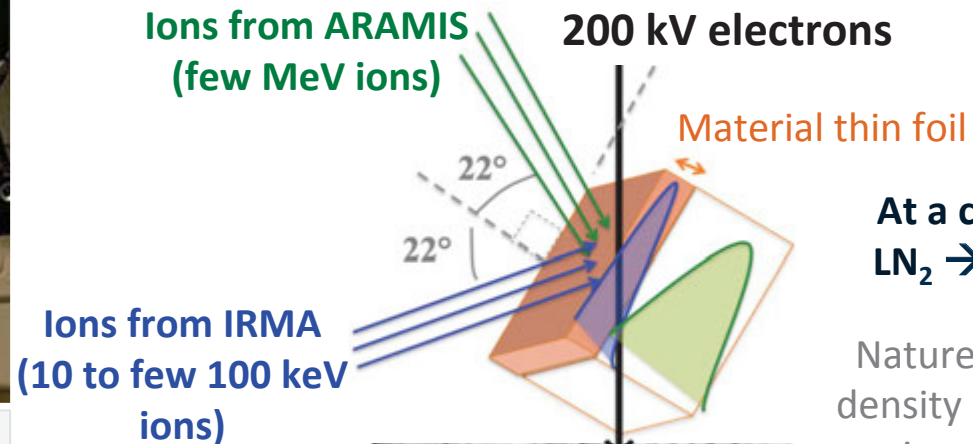
***In situ* observation of the atomic scale microstructure of materials submitted to one or two ion beams**

- ✓ Dynamical observation (above 100 keV N ions)
- ✓ Raster scanned beam
- ✓ Ion beam dosimetry inside TEM: continuous flux measurement ≤ 3 cm sample, accuracy $< 5\%$



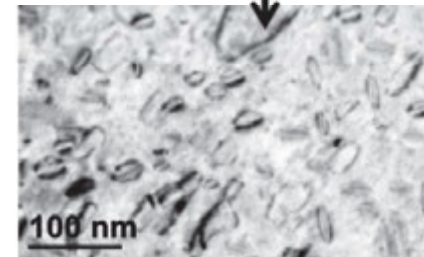
© C. Baumier (JCLab)

- 200 kV FEI Tecnai G² 20 TWIN
- **Electron source:** LaB₆ filament
- **Spatial resolution:** 0.27 nm
- **Analytical techniques :** EDX (Energy Dispersive X-rays), EELS (Electron Energy Loss spectroscopy), EFTEM (Energy Filter TEM), STEM (Scanning TEM), HAADF (High Angle Annular Dark field)

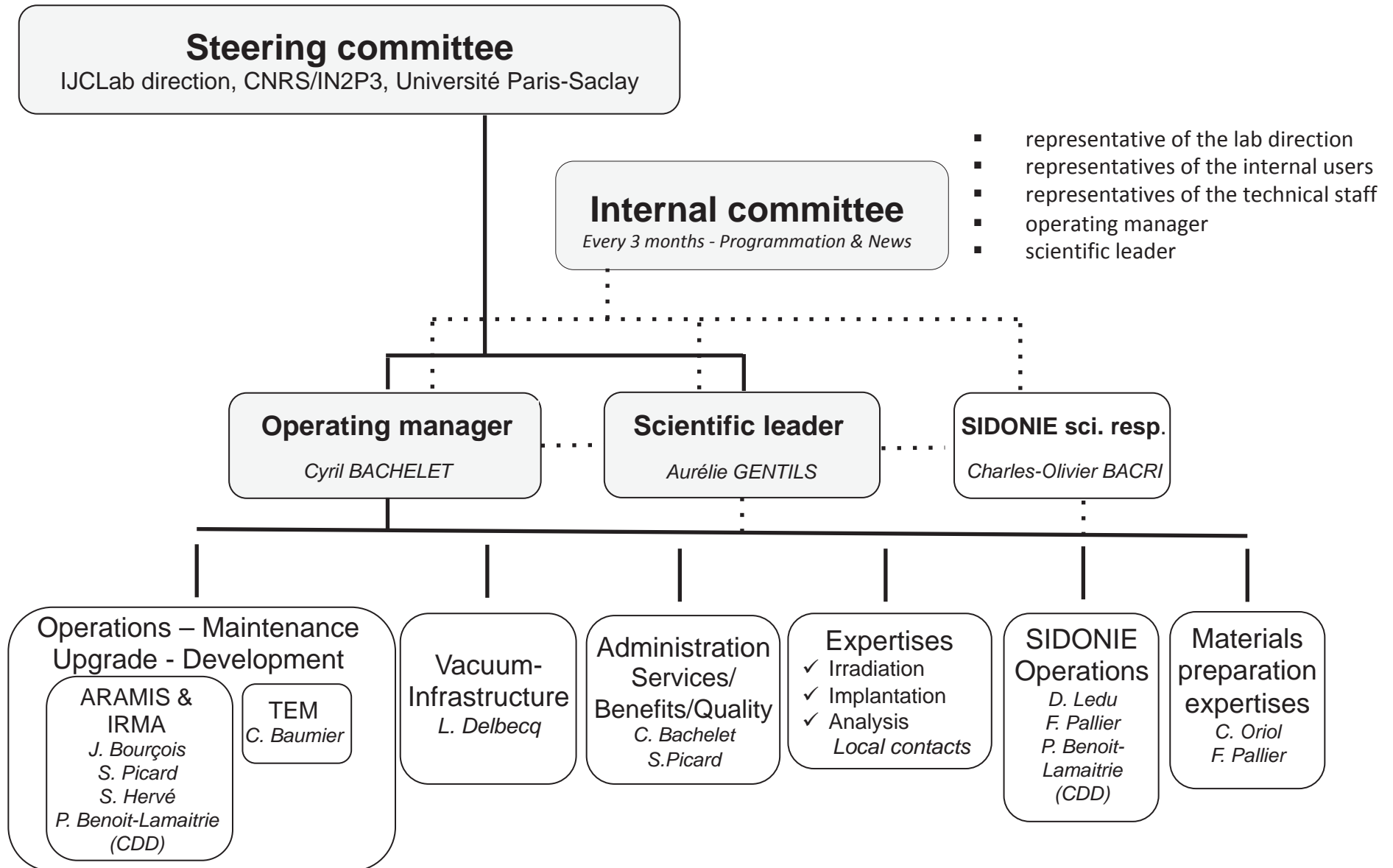


At a chosen T
LN₂ → 1000°C

Nature, size and density of defects, impurities, nanoprecipitates, crystallographic structure, chemical composition....

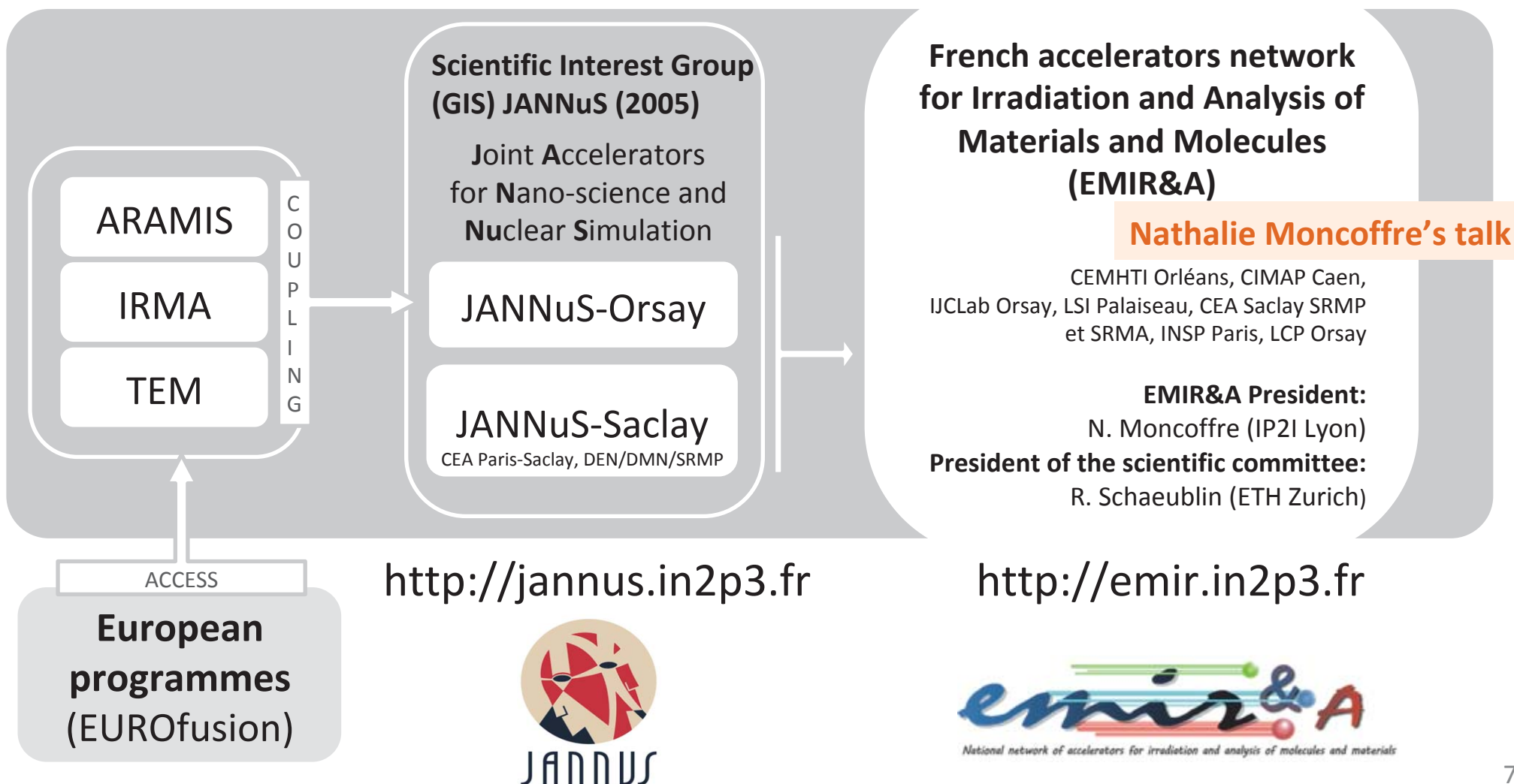


Governance structure of JANNuS-Orsay / SCALP

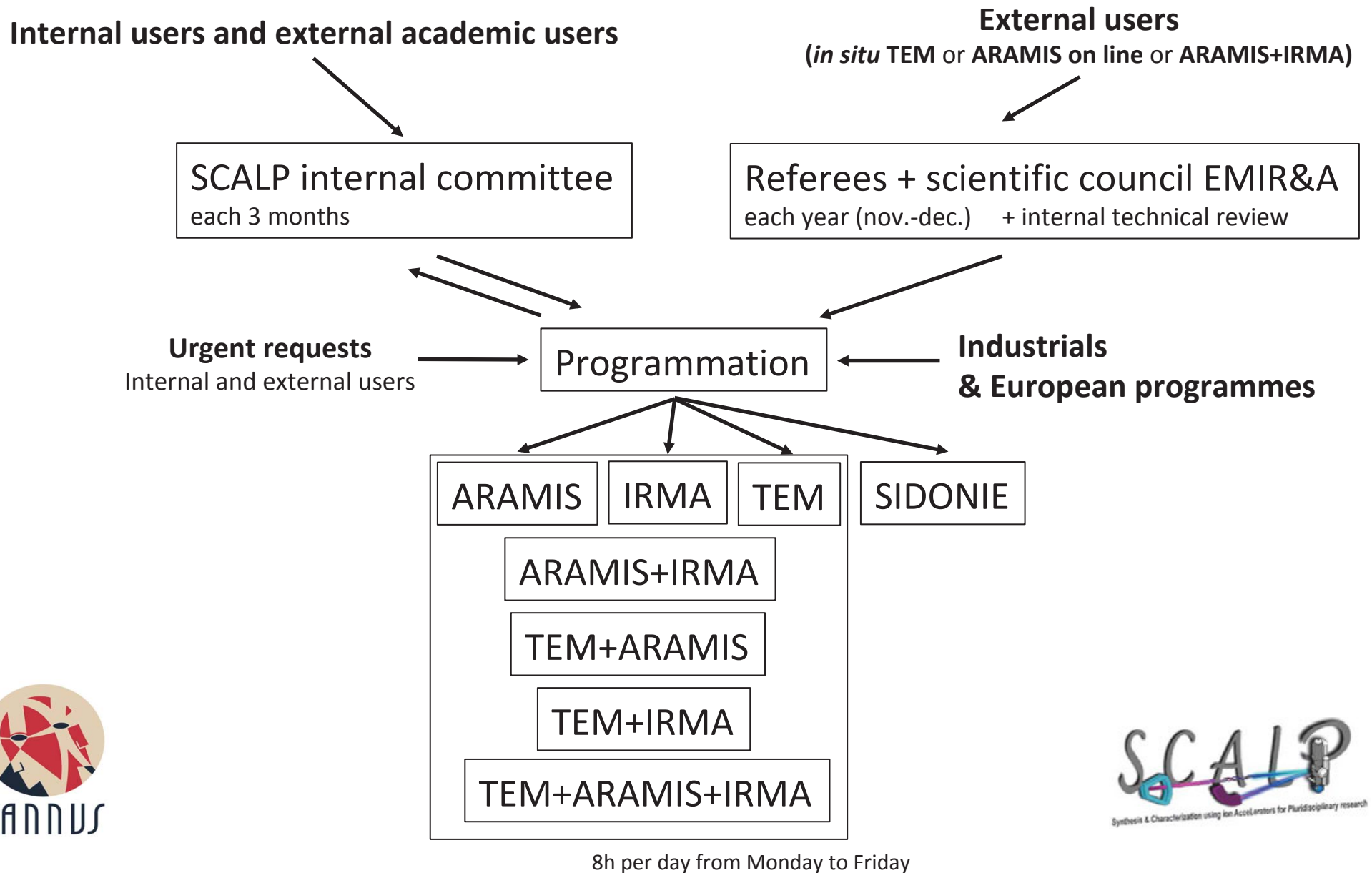


Networks

JANNUs is a founding member of **EMIR** French Accelerators network (2009)
CNRS Federation (FR 3618) since 2014
EMIR&A since 2019



Beam time allocation: proposal procedure



Resources

Human resources: approx. 11 FTE (IN2P3)

Operations and dev. technical staff	9.4 FTE
Engineering pole technical help	≈ 1 FTE
Local contacts and scientific leading	≈ 1 FTE

Financial model:

- ✓ ~ 85 % of own resources coming from chargeable services for industries and external users
- ✓ Annual operating cost is in average 85-115 k€ per year

Example – average on the last 3 years :

Annual costs 106,7 k€
Operation costs 93,1 k€
Developments 13,6 k€

Income :

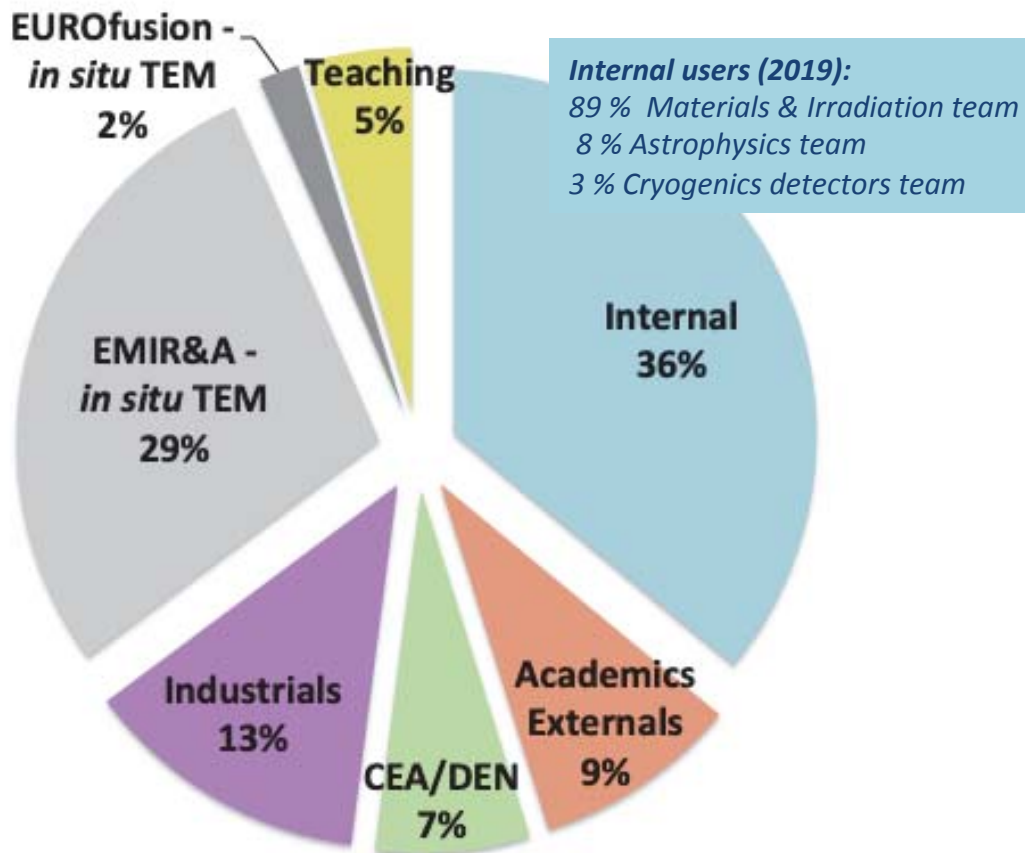
IN2P3 15 k€ / year
+ chargeable services

*Strongly dependant on
economical context*

Distribution of users

ion beam modification and ion beam analysis of materials

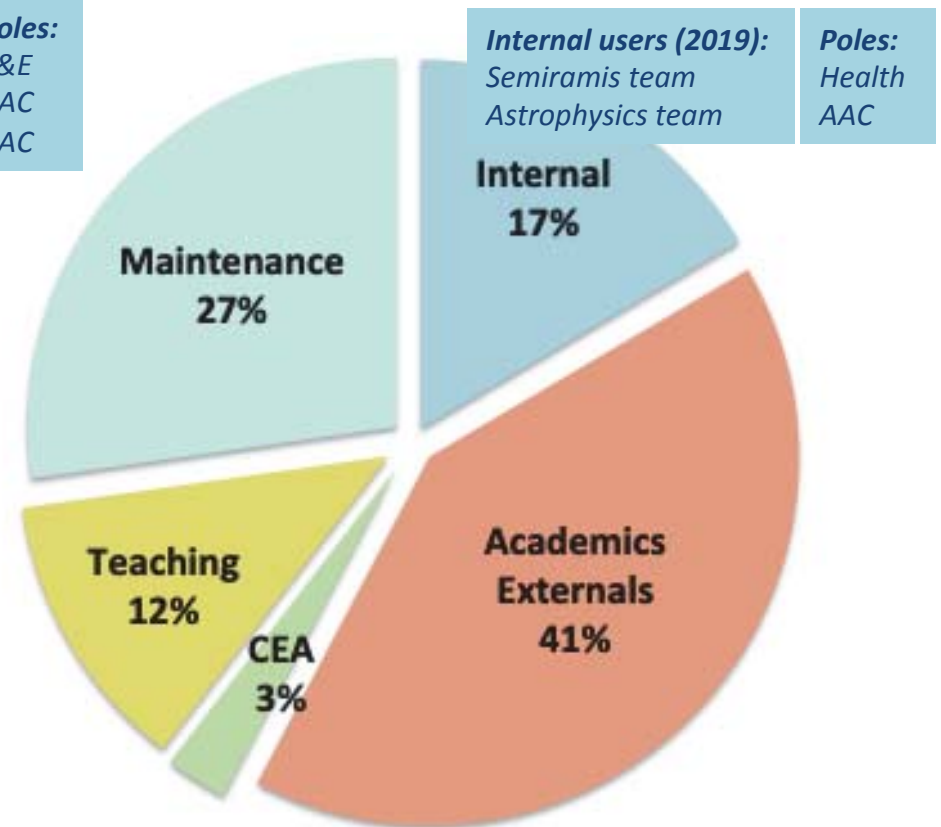
IRMA-ARAMIS-TEM JANNuS



Year 2019
 170 days of beam time, 130 distinct experiments, 30 users

isotopes and targets production

SIDONIE

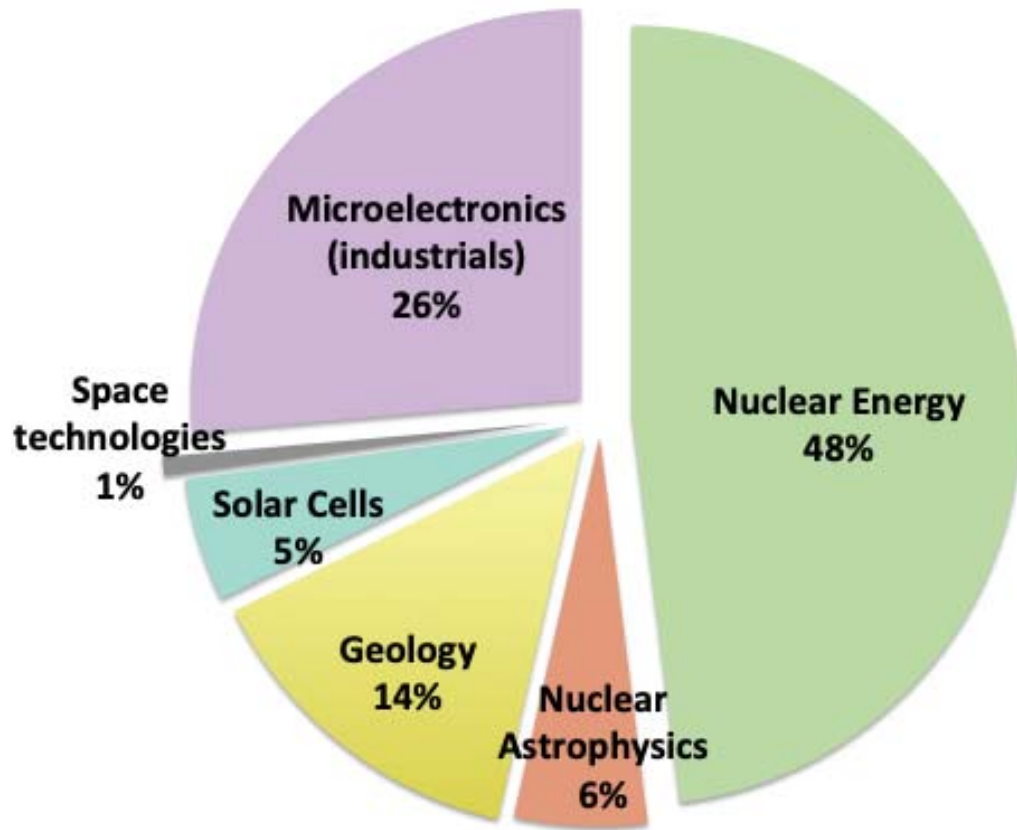


Years 2017-2019
 36 days of beam time per year in average

Overview of scientific themes

ion beam modification and ion beam analysis of materials

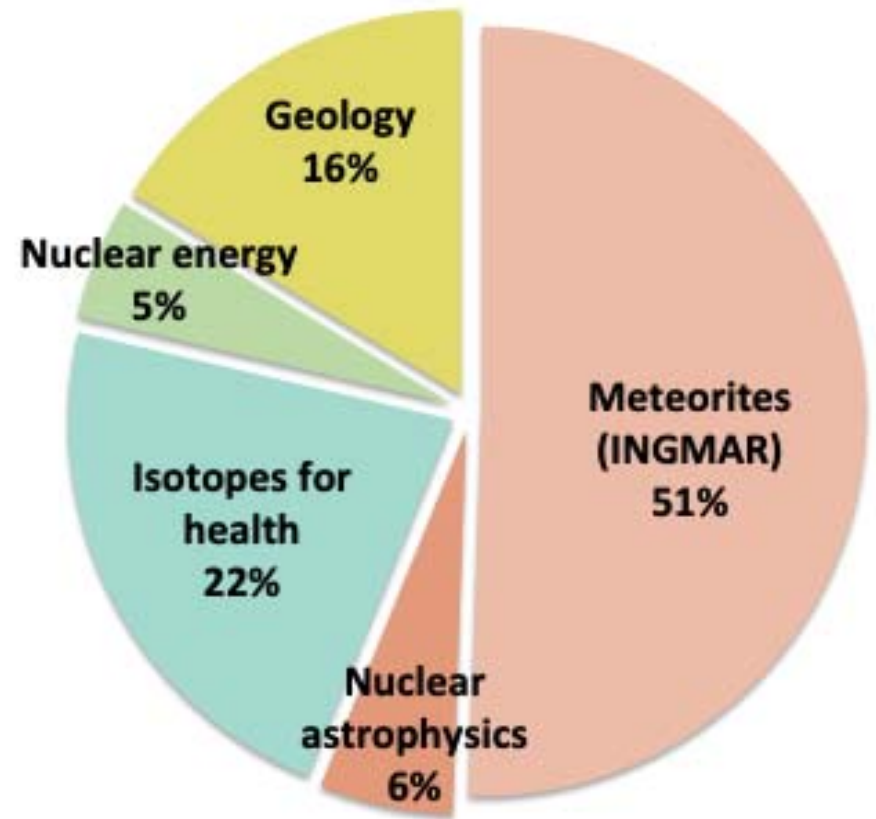
IRMA-ARAMIS-TEM JANNuS



Year 2019
170 days of beam time, 130 distinct experiments, 30 users

isotopes and targets production

SIDONIE



Years 2017-2019
36 days of beam time per year in average

Scientific issues – some examples

Materials for nuclear energy

Irradiation of austenitic steels – *in situ* TEM + ARAMIS

Light gas in ODS steels – *in situ* TEM + IRMA

Ion beam synthesis of nano-oxides - IRMA and TEM

Irradiation of UO_2 - *in situ* RBS/C analysis using IRMA+ARAMIS

Astrophysics

Meteorites and ices - INGMAR setup on SIDONIE

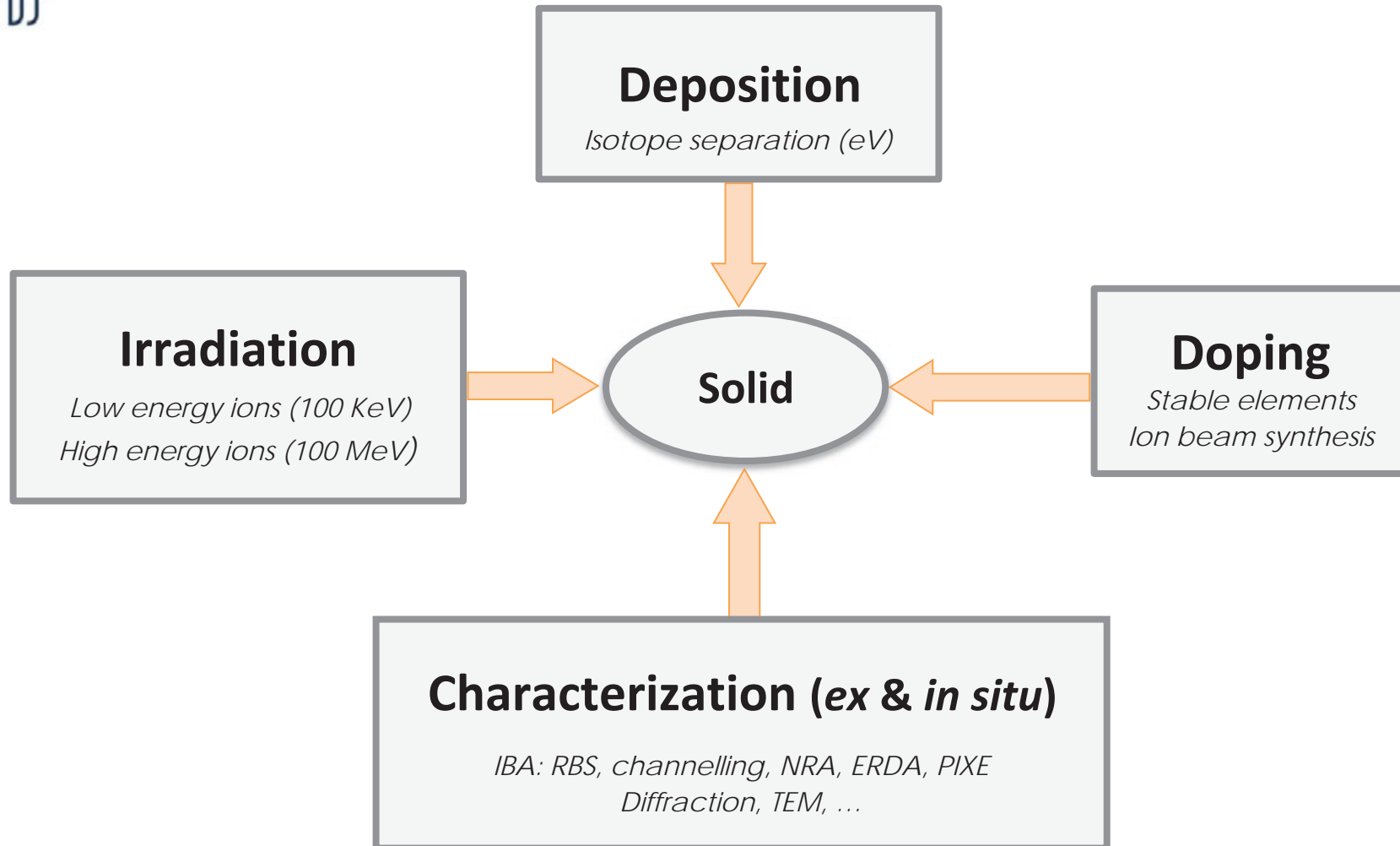
Radionuclides production for health

Production of Gd for theragnostic – SIDONIE

Miscellaneous

Helium in geological minerals - ERDA analysis on ARAMIS

SNEEL effect - RBS/C analysis on ARAMIS



**Irradiation-induced defects, atomic diffusion,
physical and chemical properties, etc, using JANNuS / SCALP facility**

Behaviour under irradiation of model austenitic steels

Objective: get a better understanding of the effect of solutes, Ti and Cr, on the irradiation behaviour of austenitic structure, i.e. Face Centered Cubic (FCC) structure



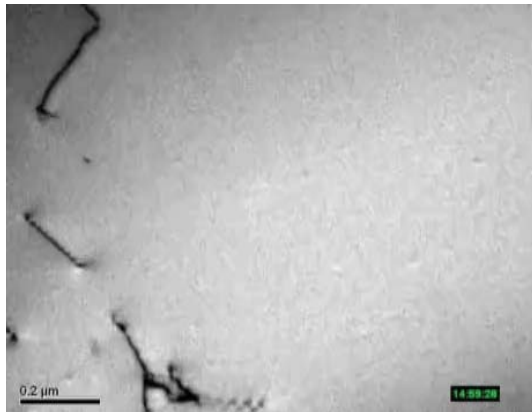
In situ TEM irradiation with ARAMIS (JANNuS-Orsay)
2 MeV Ni ions, $\Phi = 4 \times 10^{11}$ ions.cm⁻².s⁻¹ during 3'40''
0.07 dpa (displacements per atom), RT

Clear influence of an addition of Cr or Ti on loop growth and mobility

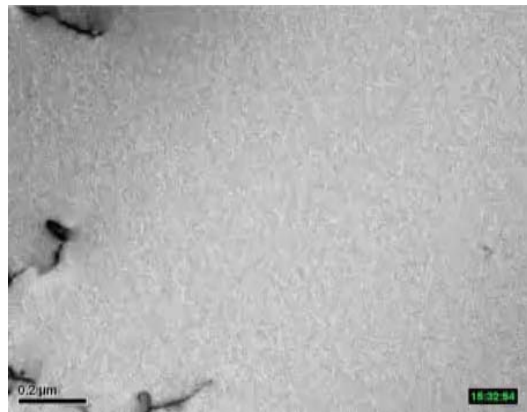
PhD thesis of Kan Ma, 2017-2020, unpublished results

x16 play speed

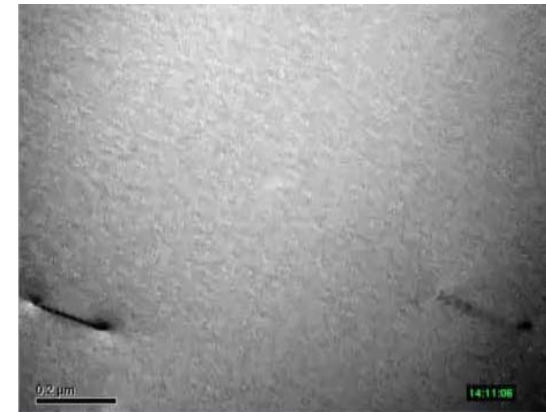
Ni



Ni-0.4Cr



Ni-0.4Ti



Kan MA¹, Marie LOYER-PROST¹, Thomas JOURDAN¹,
Brigitte DECAMPS², Anna FRACZKIEWICZ³, Frédéric PRIMA⁴

- ¹ CEA Paris-Saclay, DEN/DANS/DMN/SRMP
- ² Université Paris-Saclay, CNRS/IN2P3, IJCLab
- ³ CNRS/UMR5307/ENSMSE/LGF
- ⁴ IRCP/CNRS/UMR 8247-Chimie Paristech



The authors thank the RMATE (CEA) and NEEDS project (ARMADA) for their financial support; the JANNuS-Orsay team for the irradiations and their welcome. This experiment was supported by the EMIR French accelerator network.

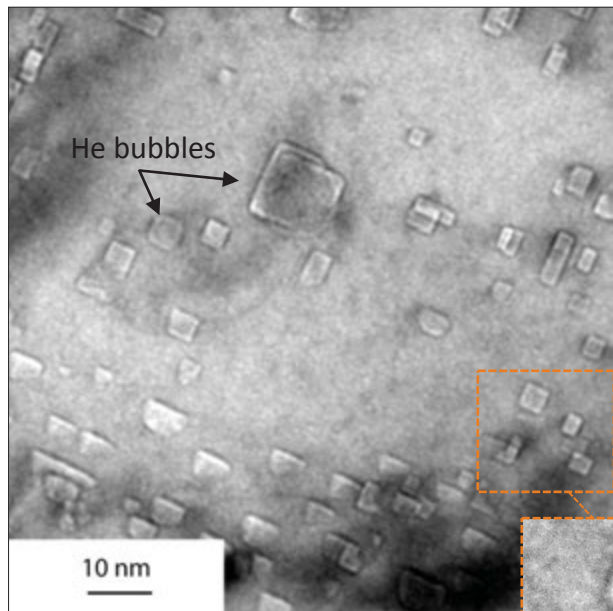
Synergetic effect of He and H gas in ODS FeCr EUROFER steels

Influence of light gas accumulation in Oxide Dispersed Strengthened (ODS) FeCr steels microstructure ?
Application to fusion reactor conditions (He+H)

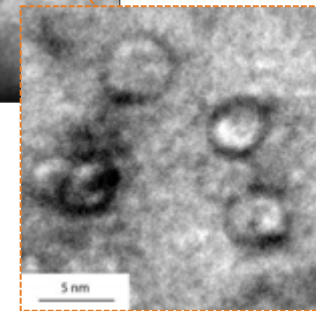
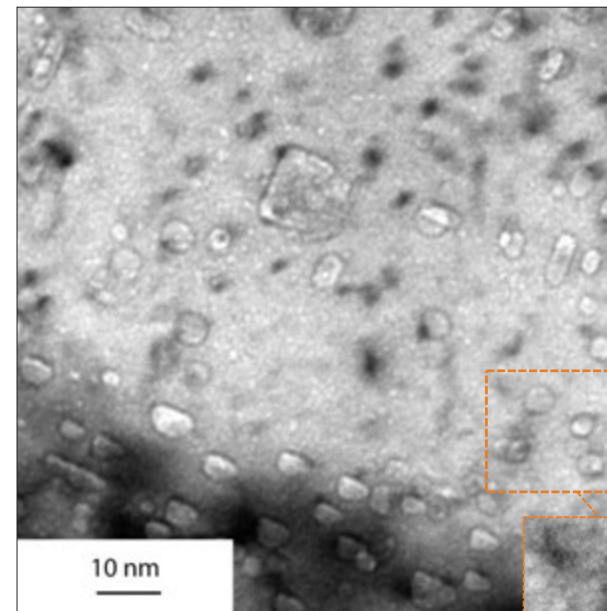
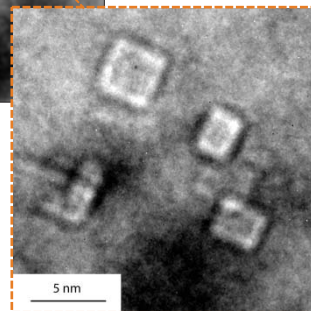
- ✓ He/H ion implantation results in pronounced evolution of the cavities shape associated with all types of the sinks
- ✓ Possible changes could be associated with H accumulation on the inner surface of the cavities



He⁺, 550 °C, up to $1 \times 10^{16} \text{cm}^{-2}$ (IRMA)



H⁺
 $1 \times 10^{17} \text{cm}^{-2}$
RT
→
in situ
TEM + IRMA



unpublished results

Olga Emelianova PhD thesis, 2020

Université Paris-Saclay / National Research Nuclear University MEPhI

Co-supervision: A. Gentils (IJCLab), M. Ganchenkova and V.A. Borodin (NRNU MEPhI et NRC Kurchatov Inst., Moscow, Russia)

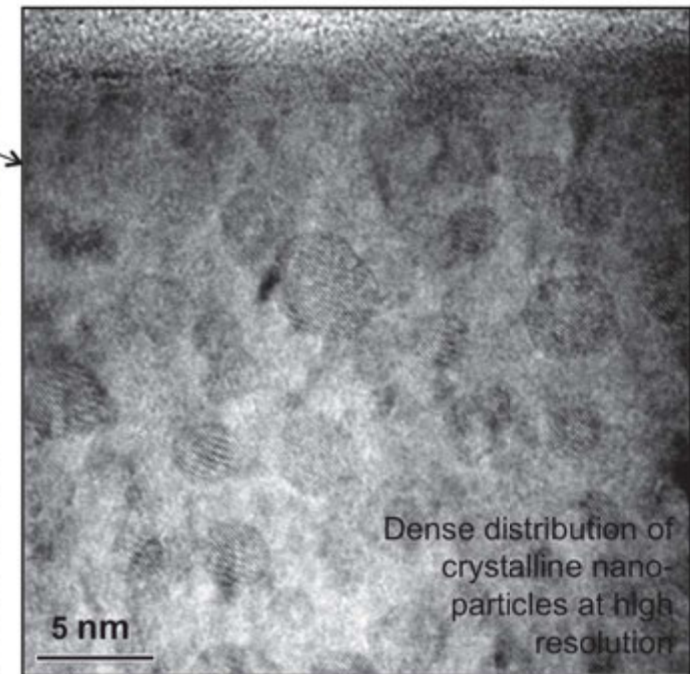
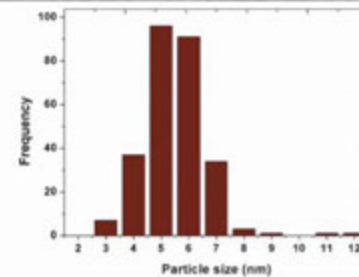
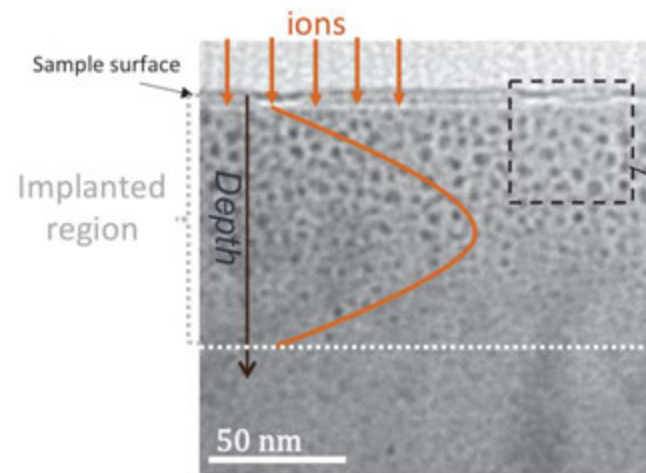
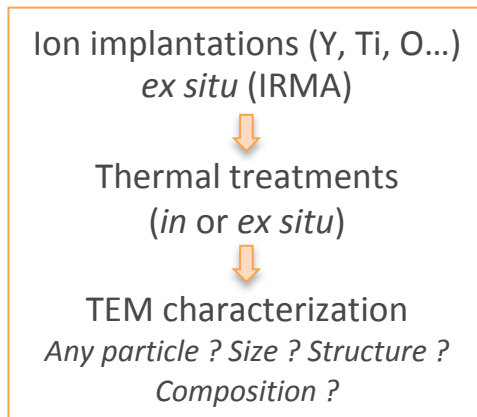
Fundings: NRNU MEPhI, Univ Paris-Sud, AP ODS IN2P3, FR FCM, Renatech

Ion beam synthesis of (Y,Ti) nano-oxides in high purity FeCr

Optimisation of the Oxide Dispersed Strengthened (ODS) steels industrial fabrication

Application to future reactors (Gen IV fission, fusion)

- Model experiments with high purity Fe-10Cr alloy (size, structure, chemistry of the nano-oxides)
- Understanding the involved mechanisms on a nanometric scale



Y + O ions, annealed 1100°C

- Average particle size ≈ 6 nm
- Particle density $\approx 10^{23} \text{ m}^{-3}$
- Crystallographic structure (not shown): *fcc* and *bcc* Y₂O₃-type structure

unpublished results

Martin Owusu-Mensah PhD thesis, 2019

Université Paris-Saclay

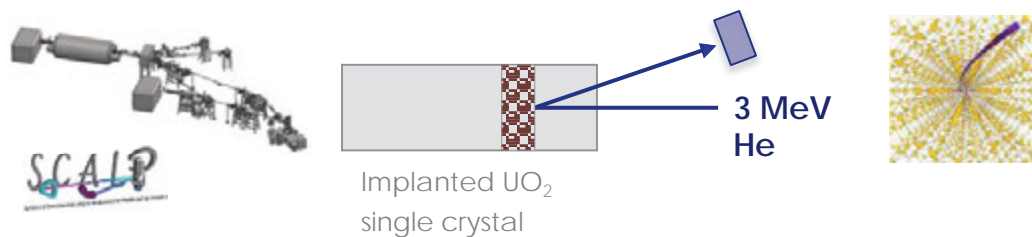
Co-supervision: A. Gentils and S. Jublot-Leclerc (IJCLab)

Coll. CEA Saclay, IM2NP Marseille, NCSU USA, NRC Kurchatov Inst. Russia

Fundings: Univ Paris-Sud (ED PHENIICS), AP ODS IN2P3, FR FCM, Renatech, METSA

Experimental simulation of High Burnup Structure (HBS) in UO₂

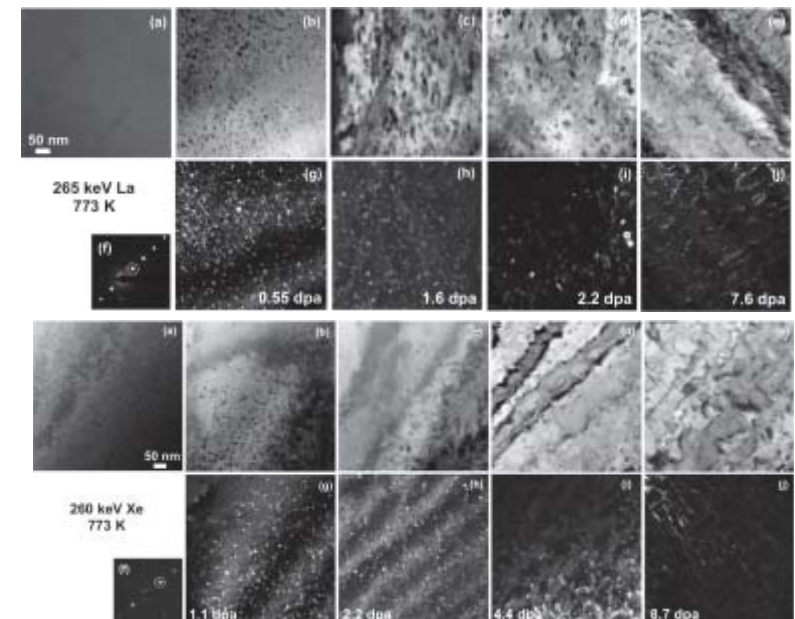
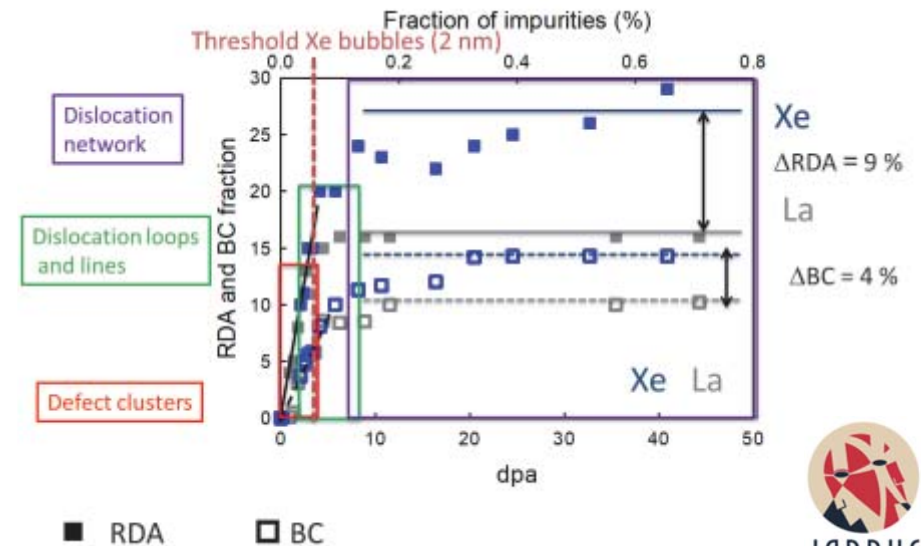
- Formation mechanisms of the HBS at the fuel periphery (high porosity, small grain size; local increase of the Pu content)
- Parametric approach : burnup, T, chemistry of impurities, radiation defects and damage
- UO₂ single crystal as model system
- In situ* irradiation/RBS-C or TEM at 773 K
- Two steps in the fuel destabilisation
 - First is ballistic (radiation damage): same dpa for Xe and La, same evolution (clusters, dislocations, network)
 - Second: dramatic role of FP solubility – polygonization induced by nanometer-sized gas bubbles



PhD thesis of Yara Haddad (2014-2017) - Supervisor F. Garrido (CSNSM)

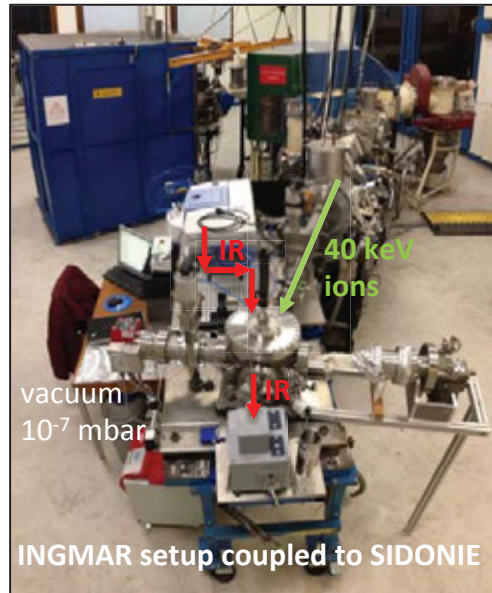
Y. Haddad, L. Delauche, A. Gentils, F. Garrido, NIM B 435 (2018)

Y. Haddad, T.H. Nguyen, A. Gentils, L. Nowicki, C. Bachelet, J. Bourçois, S. Picard, S. Resnouf, F. Garrido, to be submitted



Simulating asteroid surface alteration process

INGMAR setup : Irradiation de Glaces et Météorites Analysées par Réflectance VIS-IR



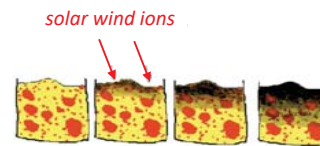
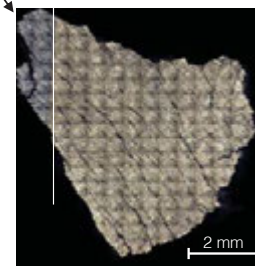
Visible et IR in situ spectroscopy
(0.4 – 15 μm , transmission and reflectance)

Study of surfaces alteration
from cosmic radiation and/or solar wind
 Interpretation of spectra obtained on ground or onboard instruments

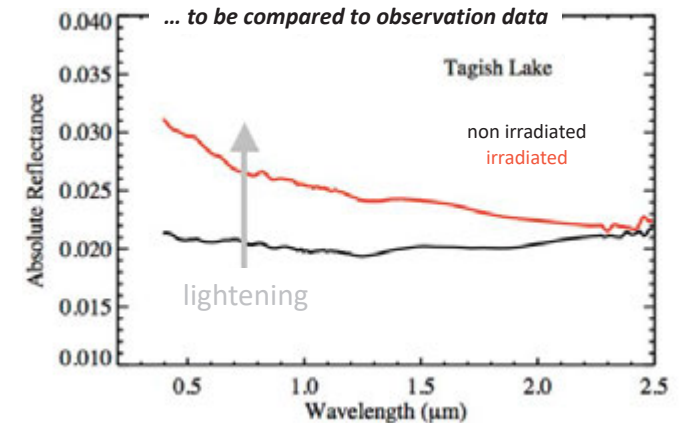
Amorphization: modification of the structure of the grains
 Radiolysis: variation of the chemical composition
 Sputtering: erosion

Brunetto et al. 2014 (Icarus)
 Lantz et al. 2015 (A&A)
 Lantz et al. 2017 (Icarus)
 Brunetto et al. 2018 (PSS)
 Lantz et al. 2018 (Icarus)

Non irradiated Irradiated
 (He^+ 20 keV ; $6 \cdot 10^6$ ions/cm²)



Pattern of asteroid surface



Radionuclides production for health physics

PRISM PROJECT

PRODUCTION D'ISOTOPES ET SÉPARATION POUR LE MÉDICAL

ISOTOPE PRODUCTION AND SEPARATION FOR THE MEDICAL SECTOR

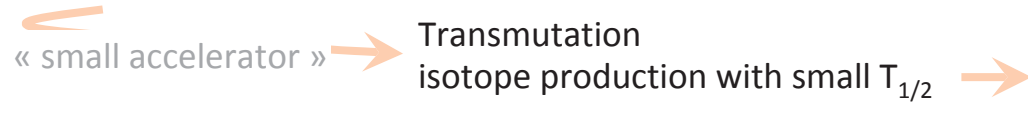
Theranostic :

One element (same chemistry) ...

with at least 2 isotopes (therapy and diagnostic)

Development of a complementary method to produce pure isotope for the medical sector

Pure stable isotope production



The Right Drug to the Right Patient for the Right Disease at the Right Time with the Right Dosage

TEP imaging

Tb 152 -70730 (40)	Tb 161 -67472 (3)
4.2 m 3+	17.5 h 2-
2.287 190 377	6.906 d 32+
α 0.7 (1.07)	β 0.52 0.46 0.590
γ 484 811	γ 26 49 75 57 88
427 533	105 106 77 w...
414	96.5

β^- therapy

Tb 149 -71500 (5)	Tb 155 -71259 (12)
4.16 m 130-	4.118 h 12+
α 0.7 (1.86)	α 0.7 (1.41)
γ 796 851	1 78
184 632	7 352 163
775	389 652
α 3.999	α 3.907

α therapy

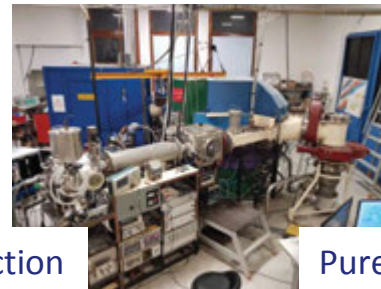
$^{155}\text{Gd}(p,n) ^{155}\text{Tb}$ production at ARRONAX

SPECT imaging

σ measurement

Pure ^{155}Gd production

Proof of concept experiment on SIDONIE



TEP imaging

Cu 64 -65420.8 (14)	Cu 67 -67300 (8)
12.700 h 1+	61.83 h 32-
α 0.6514	β 0.395 0.484 0.577
γ 1346	10 180
β 0.5778 α γ	γ 185 93* 91 500
α < 6000	394 209

β^- therapy

radioimmuno therapy

$^{68}\text{Zn}(\gamma,p) ^{67}\text{Cu}$ production at ALTO

σ measurement

Pure ^{68}Zn production

FIRST RESULT (2019)

(coll. ARRONAX, ILL) part of K. Kamalakannan M2 internship (2019)

« proof of concept » experiment

^{155}Gd production of $^{155}\text{Gd}(p,n)^{155}\text{Tb}$ cross section measurement



$^{155}\text{Gd}^{1+}$ target

$3 \cdot 10^{18}$ at/cm² on 0.95 cm²

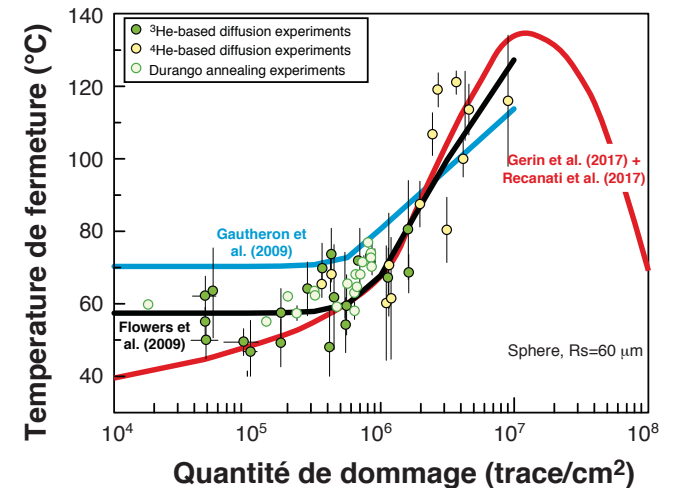
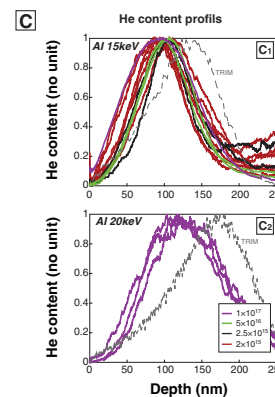
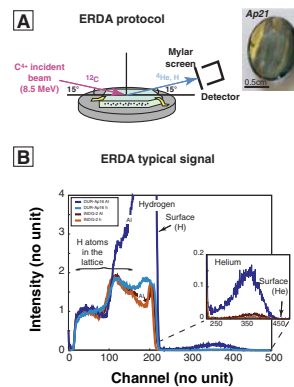
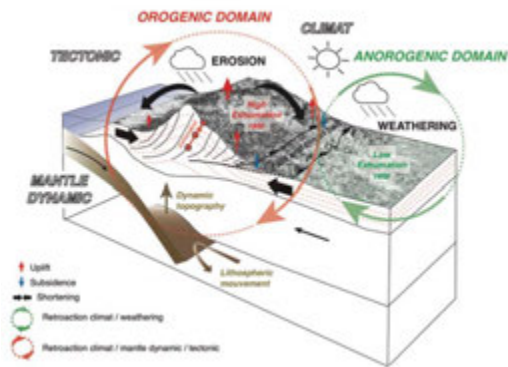
≈ 40 irradiation hours

C.O. Bacri *et al.*
coll. with



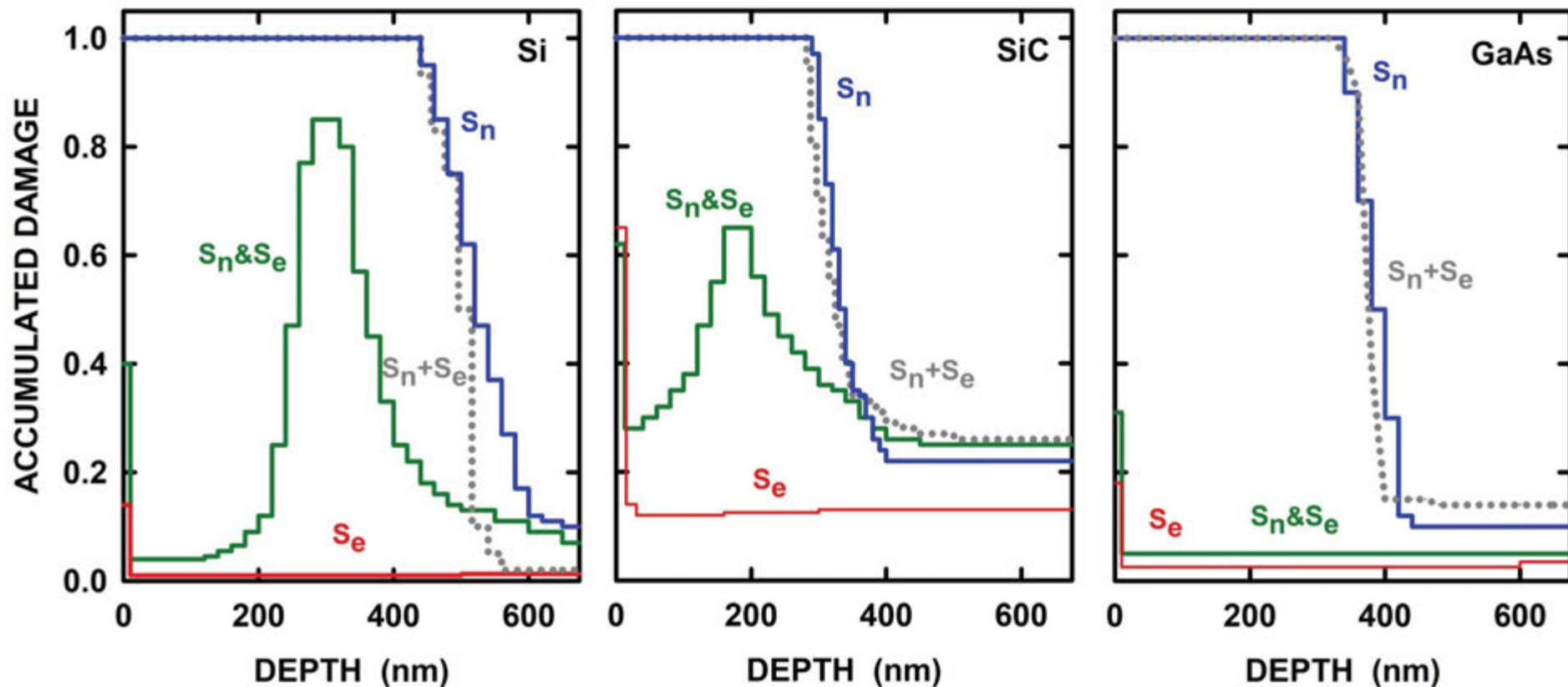
Helium diffusion in apatite for thermochronology (U-Th)/He

- Method based on the production rate and diffusion of He
- Age measurement based on the accumulation of He during a rock-dependant time-temperature
- Role played by radiation-induced defects on the He diffusion simulated by 20-keV He implantation
- Measurement of He diffusion by ERDA (8.5-MeV C, ARAMIS)



SNEEL – Synergy between Nuclear & Electronic Energy Losses

- Solid irradiated with either single or dual beam
- SNEEL leads to a strong healing by S_e of the damage created by S_n
- SNEEL mostly observed in semiconductors but also in MgO, KTaO_3
- Important result for applications: synergistic S_n/S_e effects lead to a strong reduction of the damage production in irradiated devices

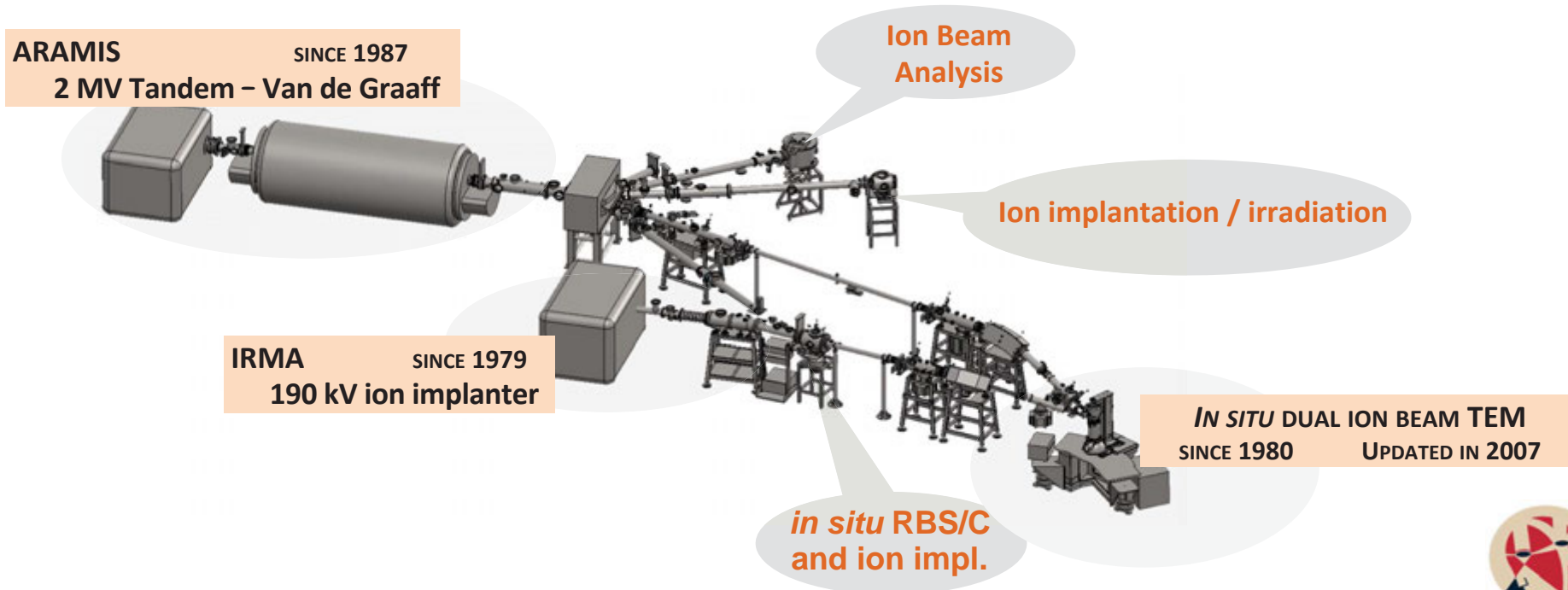


Damage profiles from RBS/C spectra on solids irradiated with either low-energy (S_n), high-energy (S_e) or dual ($S_n \& S_e$) ion beams

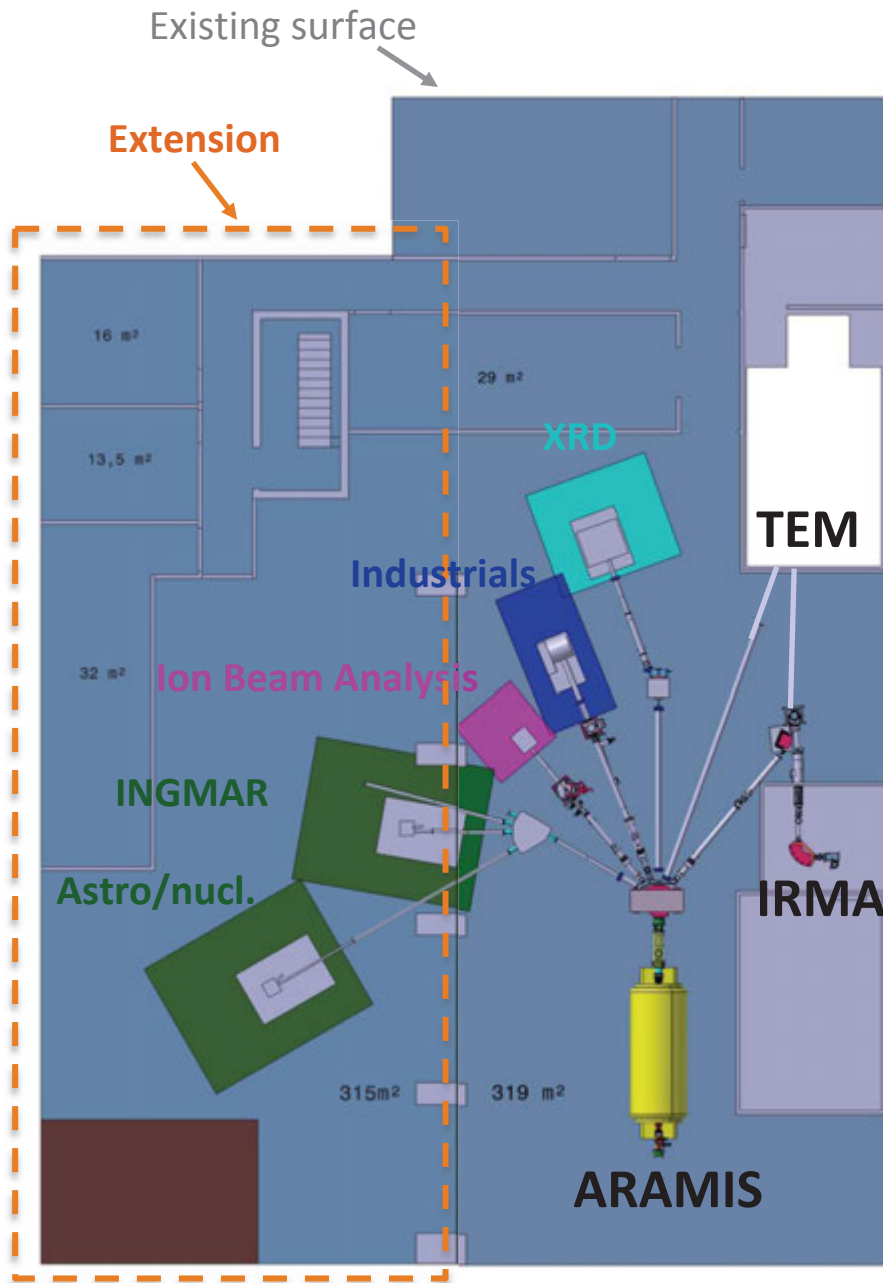
Perspectives

Extension of the experimental hall (ARAMIS-IRMA-TEM)

- ✓ Creation of an *in situ* XRD beam line
- ✓ Need for a new *in situ* TEM



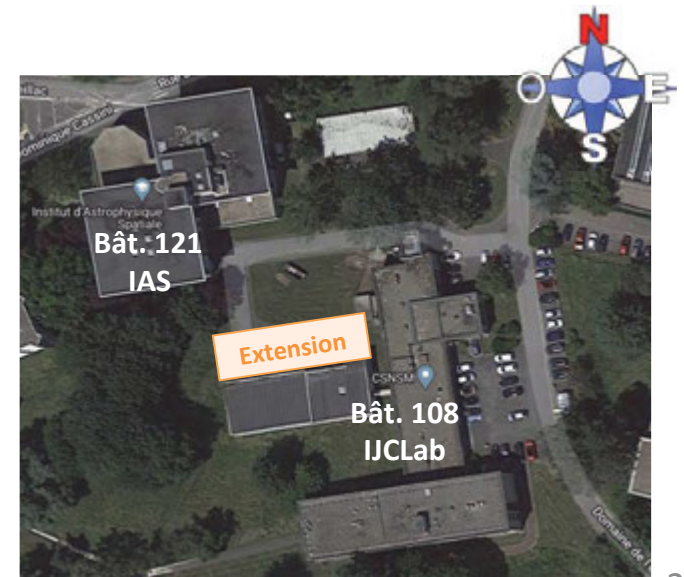
Perspectives – Building extension



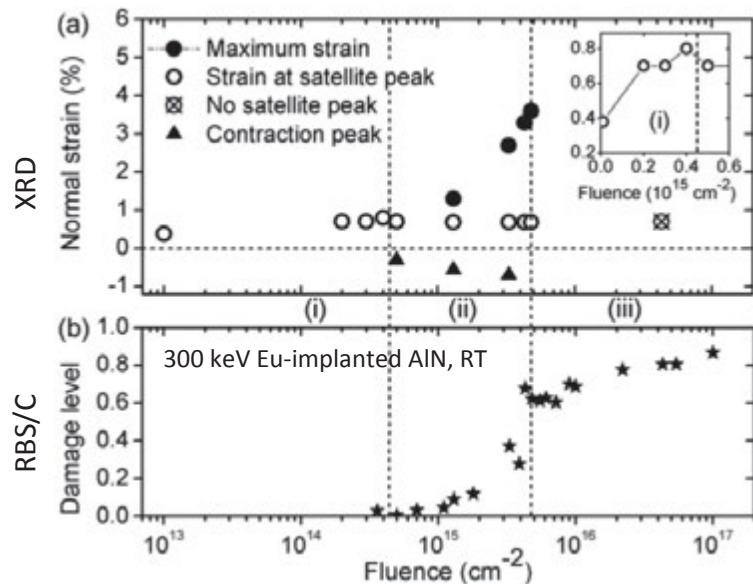
- ✓ **New ion beam lines on ARAMIS**
 - *In situ* X-Ray Diffraction line
 - γ analysis line (astro/nucl. physics)
 - Infra-Red *in situ* Spectrometry (INGMAR setup)
 - ...
- ✓ **Practical work with dedicated beam time and data analysis room (Masters MNE, GI, OSAE, ...)**
- ✓ **Industrials - Valorization**



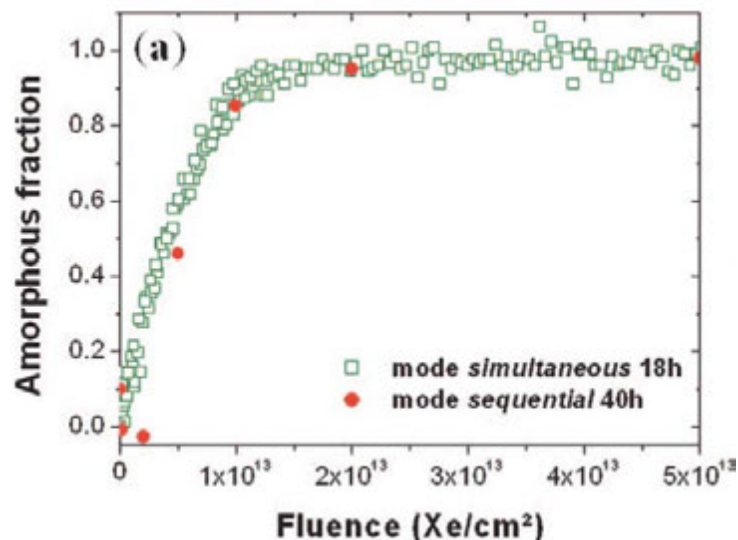
CPER-P210



Perspectives – Need for an *in situ* X-Ray Diffraction (XRD) setup



S. Leclerc et al., *J. Appl. Phys.* **112** (2012) 073525



C. Grygiel et al., *Review of Scientific Instruments* **83** (2012) 013902

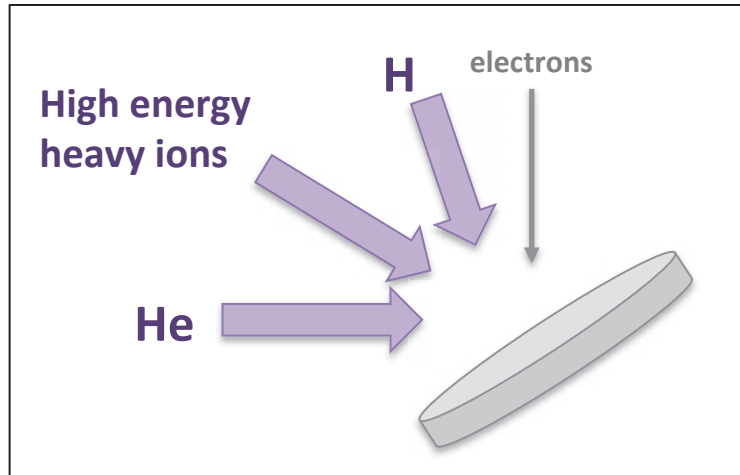
Use of X-Ray Diffraction to characterize structural ion-induced modifications

- ✓ Phase transformation (amorphization, ...)
- ✓ Elastic strain (sensitivity to point defects)
- Complementary to TEM and RBS at low damage level
- Non destructive method

In situ measurement with ion irradiation

- ✓ Inhomogeneity of the specimens, high cost, low availability
- ✓ Kinetics vs. fluence (more precision)
 - More reliability on the interpretation
- ✓ Kinetics possible at a chosen temperature
- ✓ Time saving

Perspectives – Need for a new *in situ* Transmission Electron Microscope (TEM)



Synergetic effects → 3 ion beams needed

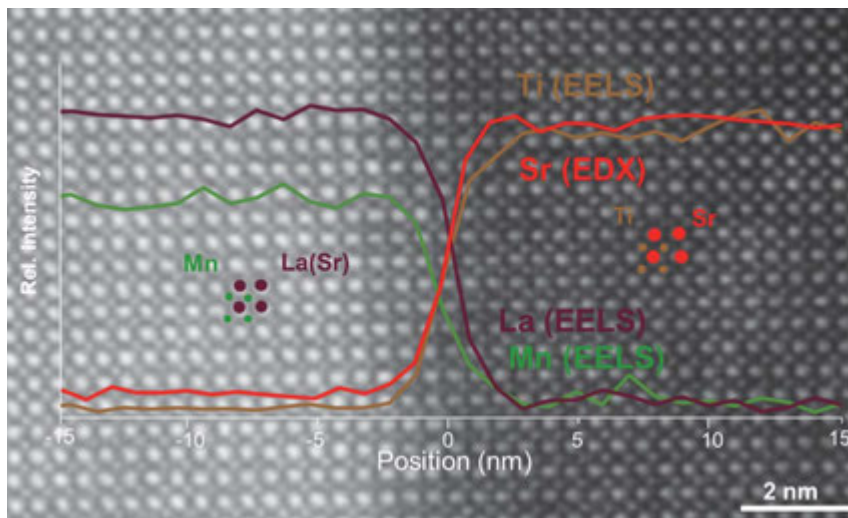
- ✓ Unique in the world ! (fluence accuracy)
- ✓ Simultaneous observation and irradiation for low energy light ions (not possible today)
- ✓ Materials for fusion (He + H + irradiation)

Higher performances for structural and chemical characterization

- ✓ High resolution (HRTEM) → interfaces and nanostructured materials, precise identification of nanostructures and defects
- ✓ Highly-resolved elemental analysis (STEM-EDX/EELS, STEM-HAADF, ABF-STEM, iDPC, ...)

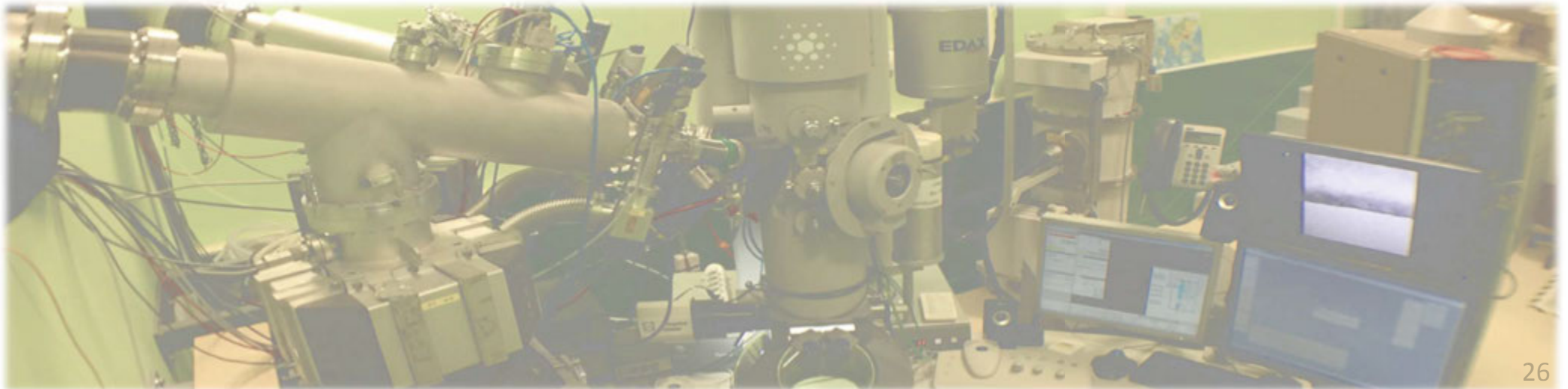
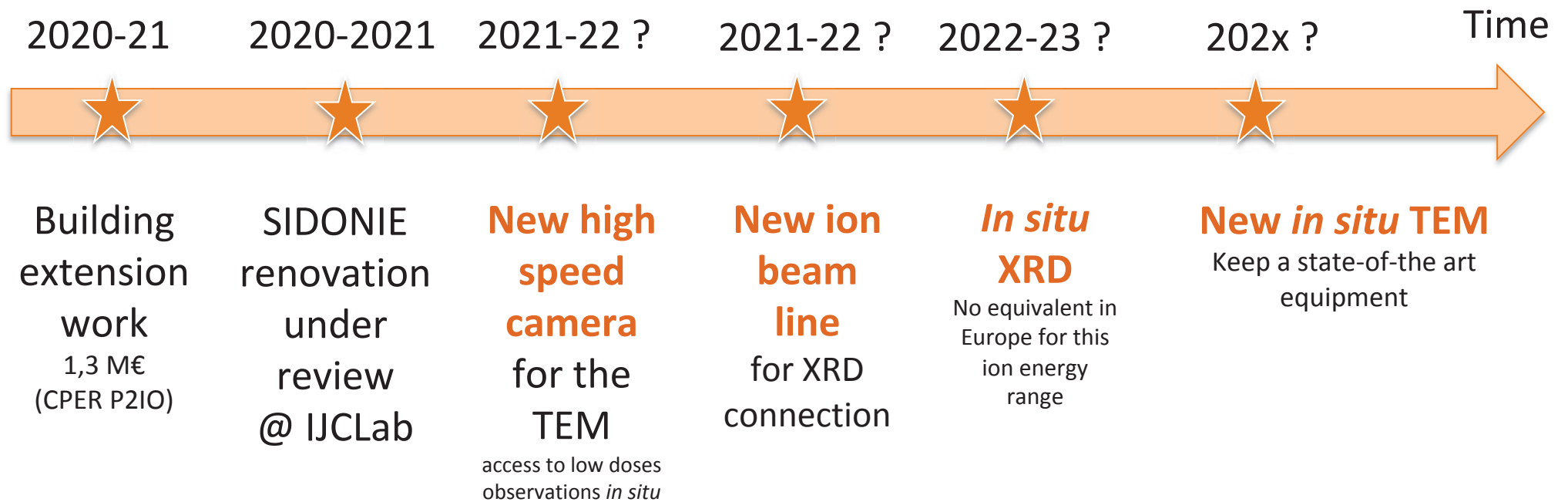
Keep the existing microscope (LaB₆ filament)

- ✓ Ideal for conventional observations and dislocation-like defects imaging (becomes rare)



HAADF-STEM image of a $\text{La}_{1-x}\text{Sr}_x\text{MnO}_3/\text{SrTiO}_3$ interface with the individual atomic columns well resolved across the interface. Overlaid is an EELS/EDX intensity profile across this interface.
P.M. Leufke and D. Wang et al., *Thin solid films* 520 (2012) 5521-5527

Perspectives – *equipment needs*



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