IJCLab and its contribution to HerHEA project

Meeting #1, 13/06/2024

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EUROfusion, ENR-MAT.02.VTT-T001
2024-205

https://indico.euro-fusion.org/event/3152/

In situ TEM coupled to 190 kV IRMA ion implanter and 2 MV ARAMIS accelerator

JANNUS Orsay

mosaic

https://mosaic.ijclab.in2p3.fr

- which compositions of alloys ?

  To be chosen : binary -WMo, WTa, WV-, ternary –WTaV- and WTaMoNbV ?
  Pre-analysis of the specimens ?

- thin foil preparation dev. / optimization

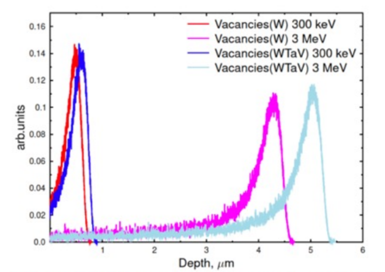


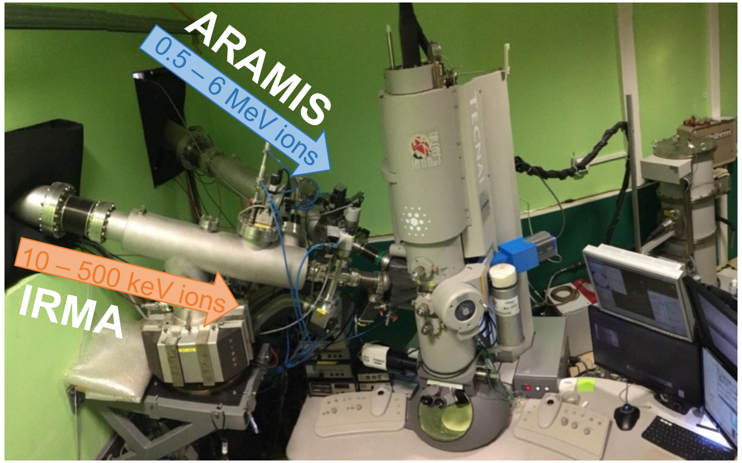
Fig. 2 Depth distributions of vacancy generated by 300 keV and 3 MeV He ions in W and WTaV calculated within the binary collision approximation. From MF Barthe

- ions parameters (similar as for some *ex situ experiments* ?)

  Helium, 10-20 keV

  One condition: influence of a coupled few MeV irradiation ?

- implantation temperature : RT and 500°C

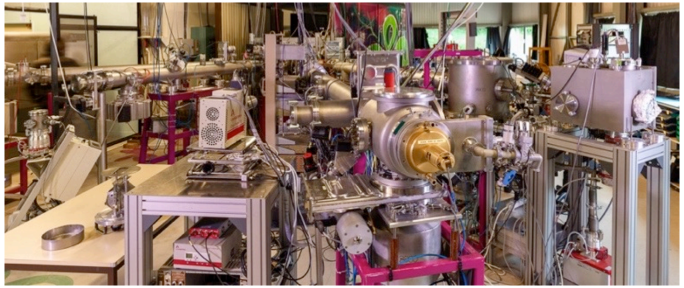


- Observation of He bubbles nucleation and growth *vs* fluence

« The evolution of created defects (and, in particular, the size and density of cavities, voids and bubbles) will be monitored in different compositions vs the fluence at room temperature and 500°C), for selected parameters chosen **with the help of simulations**. »

M3.1 *In situ* TEM experiments performed (Links to D3.1) June 2025

The JANNuS-Orsay experimental hall : ARAMIS, IRMA, *in situ* TEM



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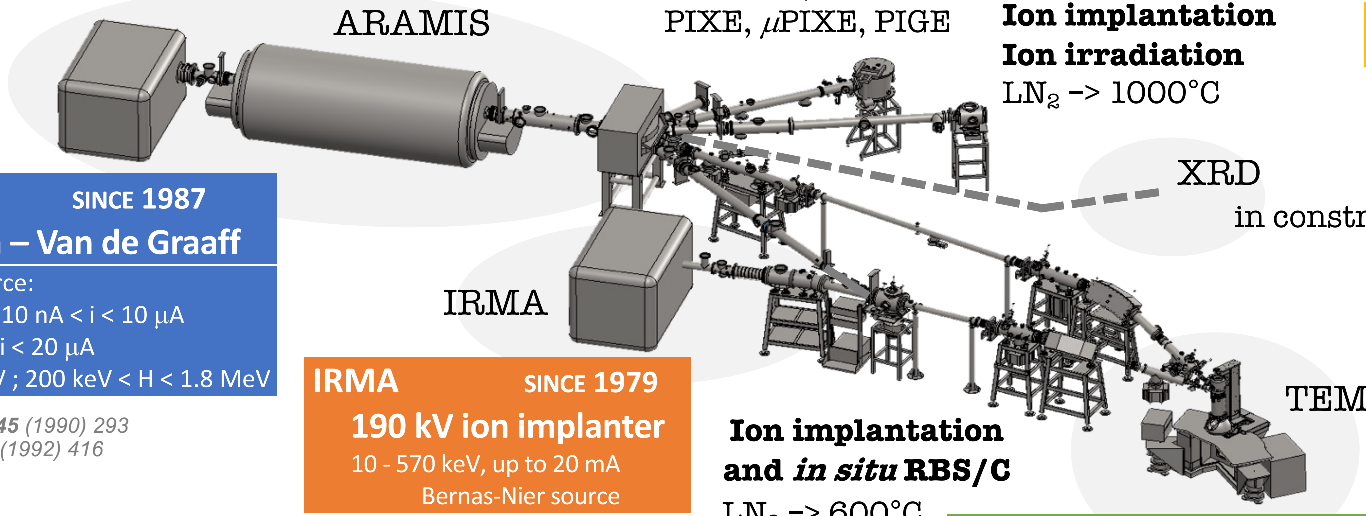


ARAMIS SINCE 1987

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

*E. Cottereau et al., NIMB **45** (1990) 293*
*H. Bernas et al., NIMB **62** (1992) 416*

IRMA SINCE 1979

190 kV ion implanter

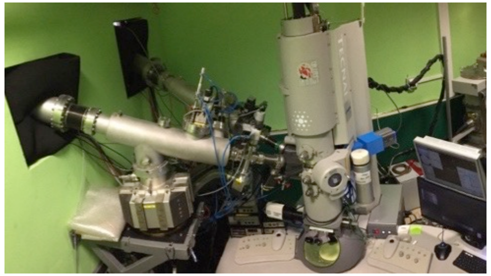
10 - 570 keV, up to 20 mA
Bernas-Nier source

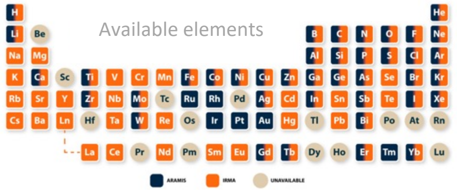
*J. Chaumont et al., NIMB **198** (1981) 193*

IN SITU DUAL ION BEAM TRANSMISSION ELECTRON MICROSCOPE

SINCE 1980 UPDATED IN 1994, 2007

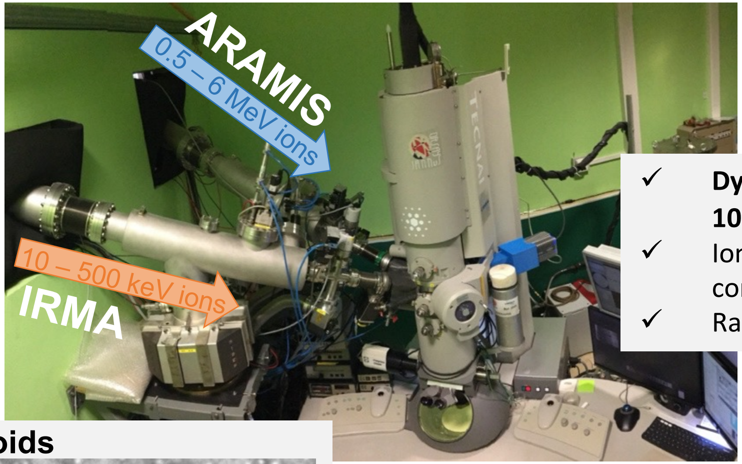
*M.-O. Ruault et al., J. Mater. Res. **20** (2005) 1758*
*A. Gentils et al., NIMB **447** (2019) 107*





Home-made ion accelerators

A unique *in situ* dual ion beam Transmission Electron Microscope (TEM)

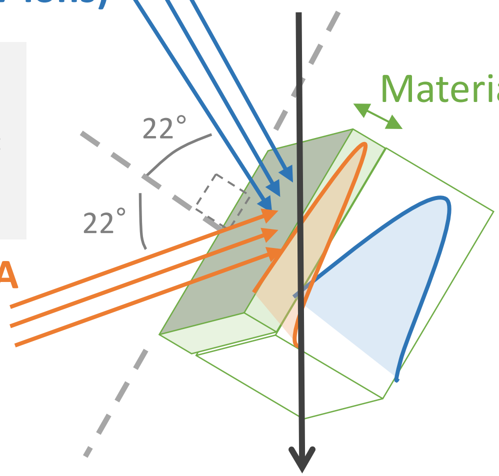


- ✓ **Dynamical observation (above 100 keV N ions)**
- ✓ Ion beam dosimetry inside TEM: continuous flux measurement
- ✓ Raster scanned ion beam(s)

Ions from ARAMIS (few MeV ions)

200 kV electrons

Ions from IRMA (10 to few 100 keV ions)



Imaging, Diffraction, and Chemical Analysis

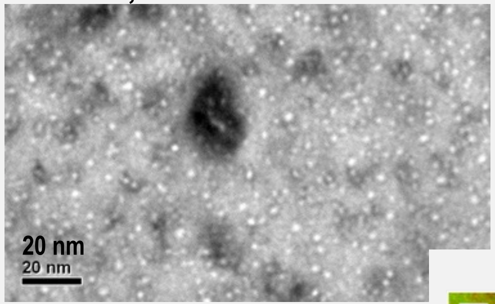
200 kV FEI Tecnai G^2 20 TWIN
Electron source: LaB_6 filament
Spatial resolution: 0.27 nm
Magnification range: x 70 – 700 000

At a chosen T
LN_2 → 1000°C

Techniques available:
Conventional TEM
Scanning TEM
EDX, EELS, EFTEM, HAADF

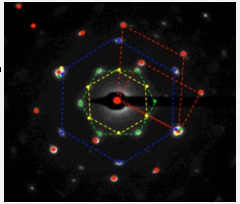
***In situ* observation of the atomic scale microstructure evolution of materials submitted to single/dual ion beams :**
role of defects (nature, size, density), impurities, nanoprecipitates, crystallographic structure, chemical composition….

Bubbles, voids

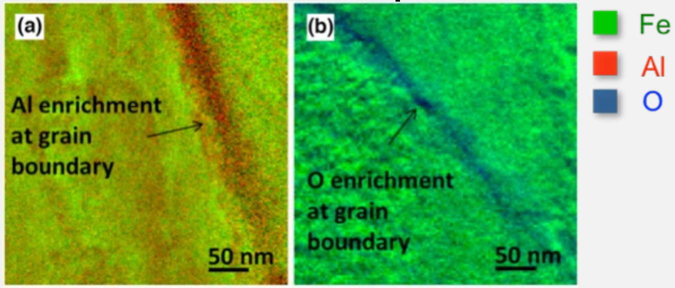


© A. Gentilis (IJCLab)

Crystallo-graphic structure

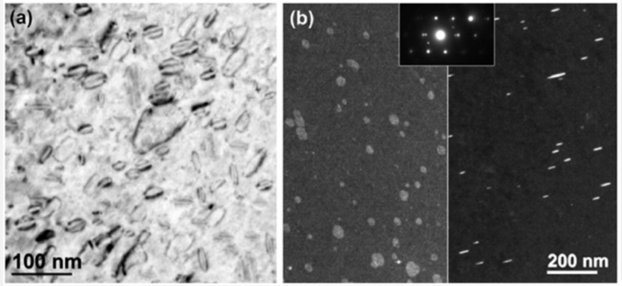


Elemental maps



C. Zheng, A. Gentils, *et al.*, NIMB **365** (2015) 319

Defects: dislocations, dislocation loops, …



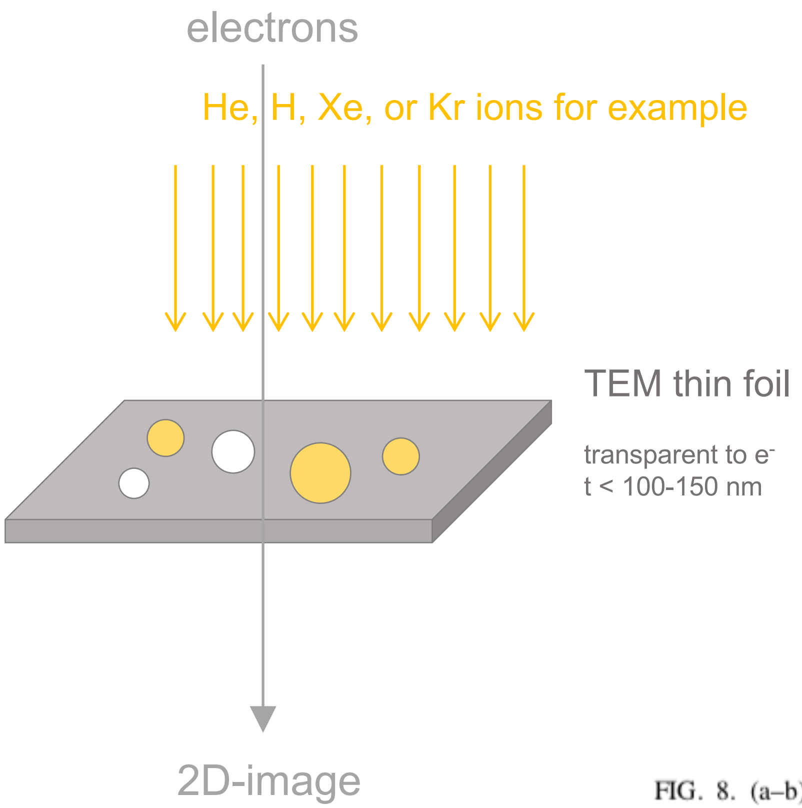
S. Jublot-Leclerc *et al.*, JNM **480** (2016) 436

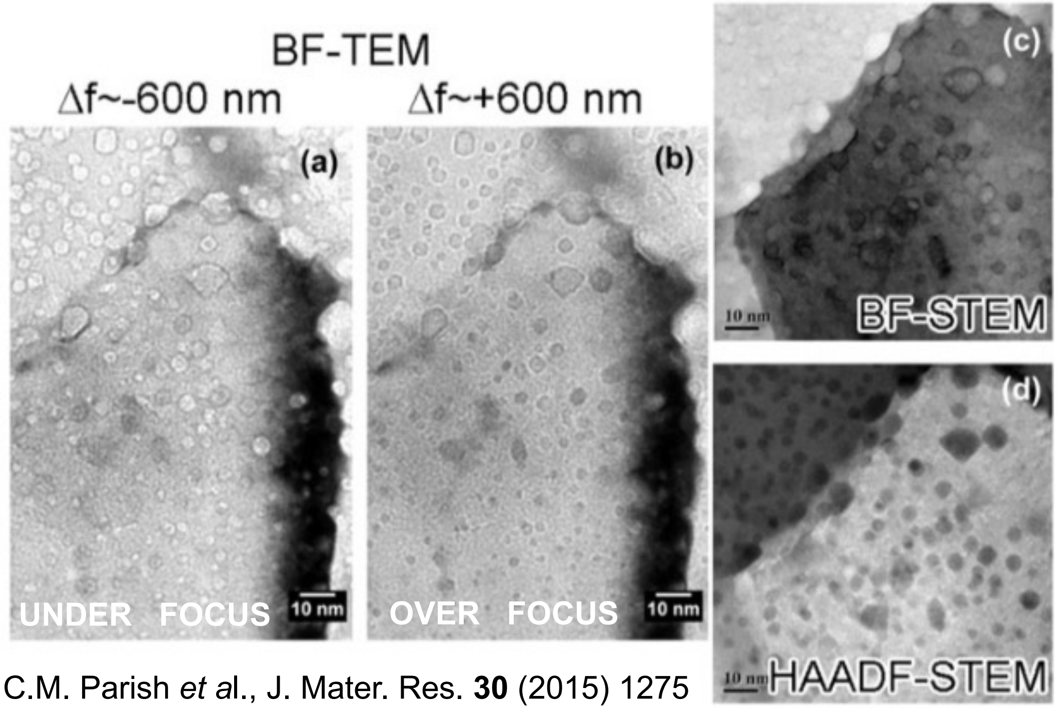
IJCLab Irène Joliot-Curie Laboratoire de Physique des 2 Infinis
mosaic
JANNuS
emir&a Réseau national d'accélérateurs pour l'irradiation et l'analyse des molécules et matériaux

Observing gas bubbles with a TEM

Cavities = voids or gas-filled bubbles

- Conventional use of out-of-focus bright field imaging (Fresnel fringes)
- Also possible using Scanning TEM (HAADF) , Z contrast
- Limitations below 0.8 nm diameter approx., depending on microscopes





C.M. Parish *et al*., J. Mater. Res. **30** (2015) 1275

FIG. 8. (a–b) Fresnel contrast under/over focus TEM images of NFA 14YWT implanted with helium. (c–d) BF and HAADF-STEM. (a–b) $\alpha_{TEM} < 1$ mrad, $\beta_{TEM} \sim 5$ mrad. (c–d) $\alpha_{STEM} \sim 15$ mrad, $\beta_{BF} \sim 16$ mrad, $\beta_{HAADF} \sim 55$ mrad.

Experimental simulation of alpha decay damage in apatite

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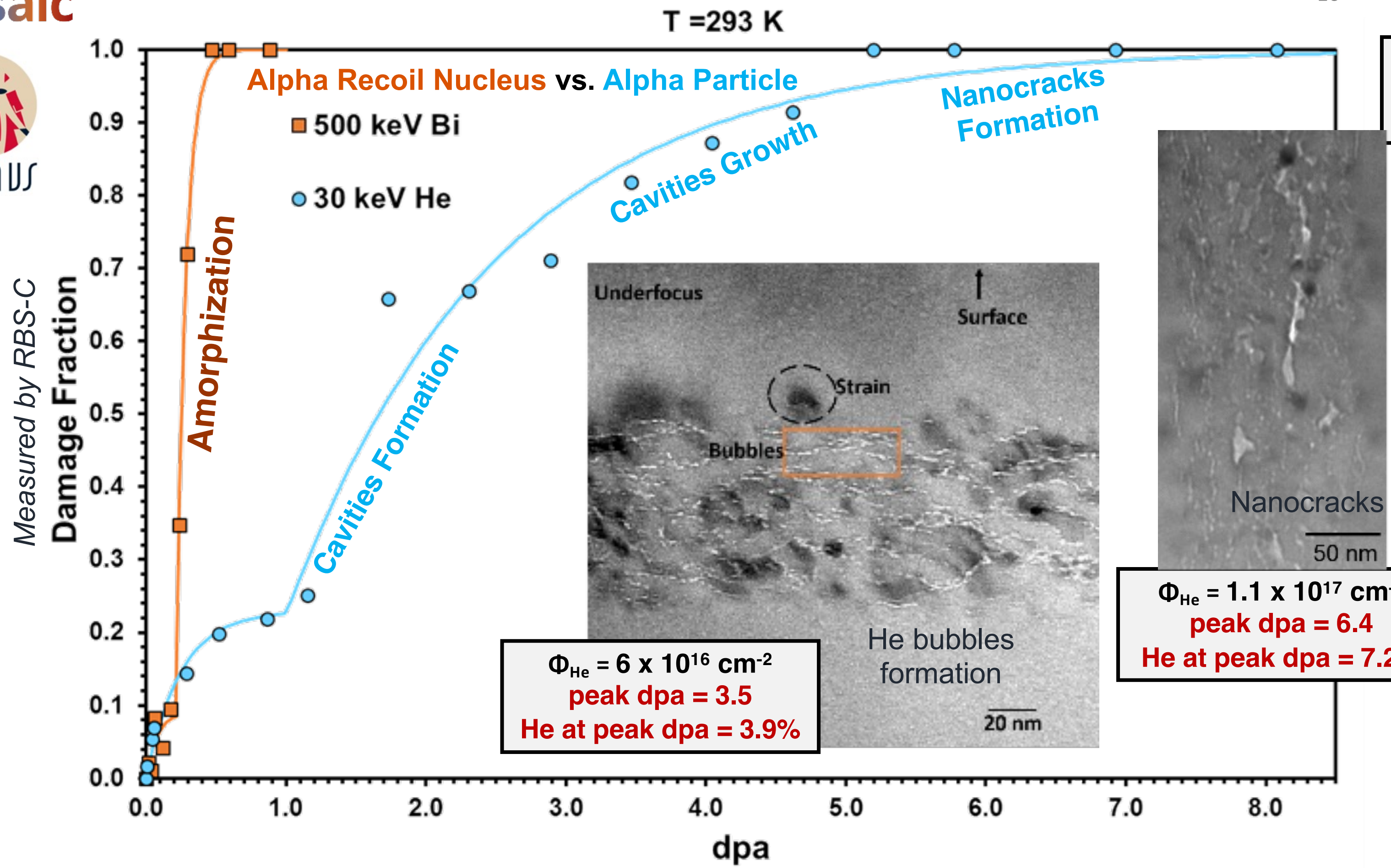
He ion-implanted fluoroapatite

Applications : Nuclear waste management and thermochronology

Fluorapatite, $Ca_{10}(PO_4)_6F_2$: most abundant apatite in nature

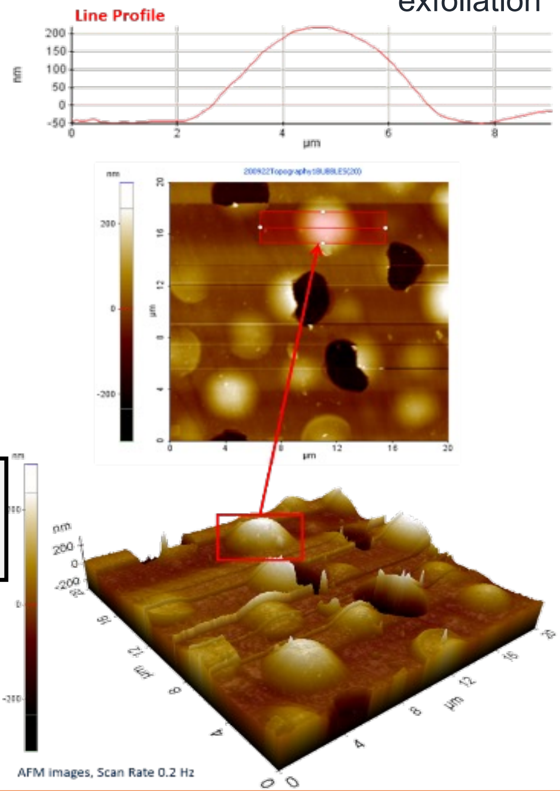


Φ_{He} = 6 x 10^{16} cm^{-2}
peak dpa = 3.5
He at peak dpa = 3.9%

Φ_{He} = 1.1 x 10^{17} cm^{-2}
peak dpa = 6.4
He at peak dpa = 7.2%

Φ_{He} = 1.4 x 10^{17} cm^{-2}
peak dpa = 8.1
He at peak dpa = 9.2%

Surface exfoliation



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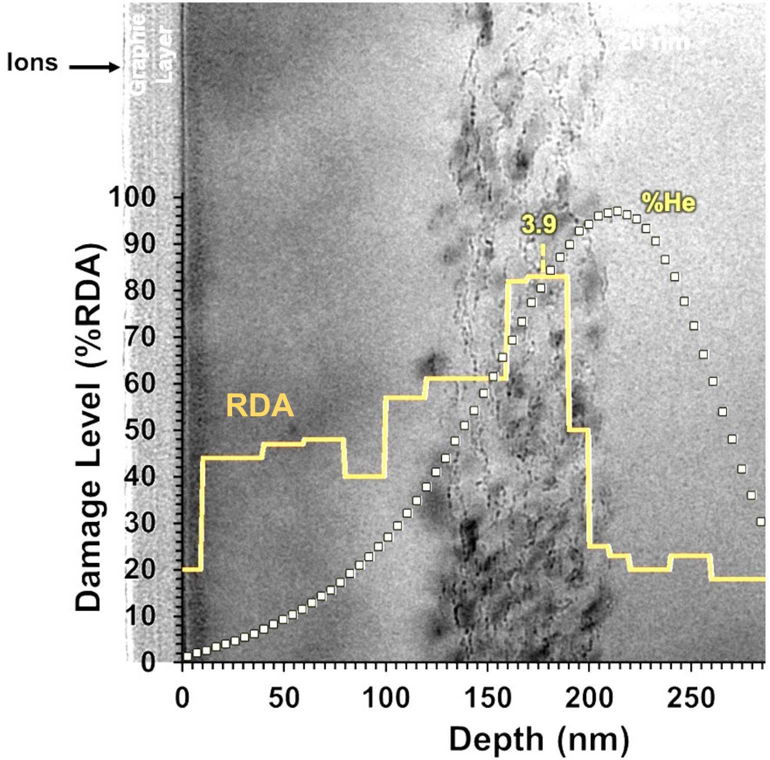
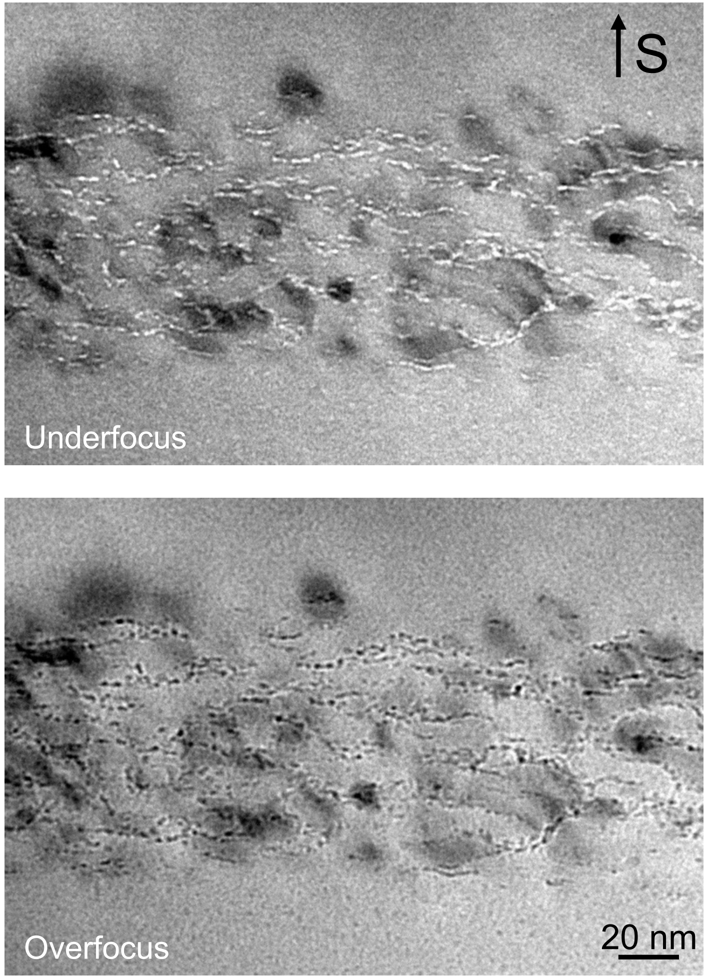
Φ_{He} = 6 x 10^{16} cm^{-2}
peak dpa = 3.5
He at peak dpa = 3.9%

Fluorapatite, $Ca_{10}(PO_4)_6F_2$
implanted with

30 keV He at 293 K

He bubbles formation
observed by TEM





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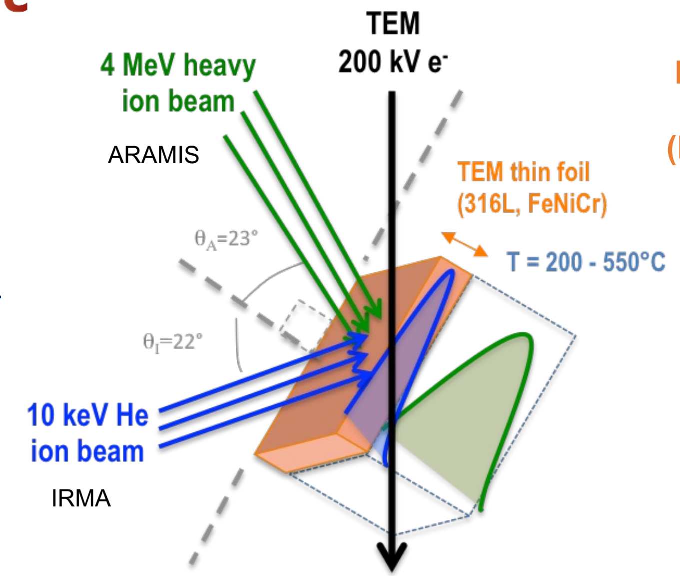
Agence Nationale de la Recherche ANR

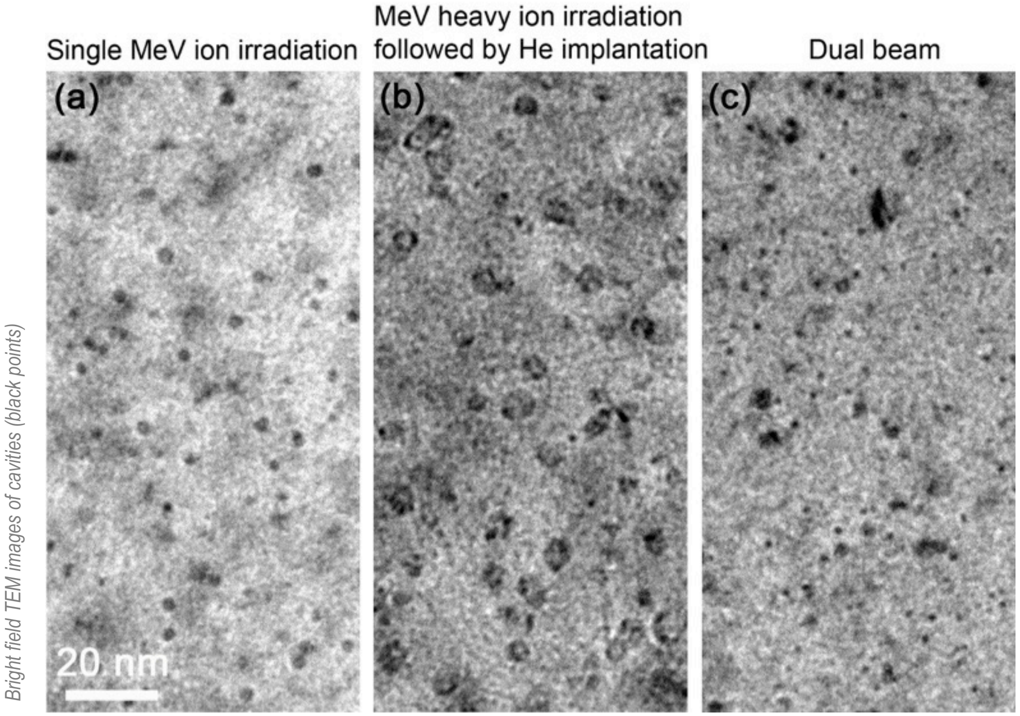
Synergetic effects with dual ion beam: concomitant helium accumulation and damage creation - Effects on bubble nucleation



Helium + irradiation in austenitic steels (PWR vessel internal components)

Imaging of populations of cavities induced by single or dual ion beam at different T (200, 450 and 550°C) and fluences

Cavities (voids, bubbles)



Industrial CW 316L steel – 550°C

He when injected in dual beam enhances the cavity nucleation, unlike when injected after damage

S. Jublot-Leclerc *et al.*, J. Nucl. Mater. **494** (2017) 240

Synergetic effects with dual ion beam : example in austenitic steels

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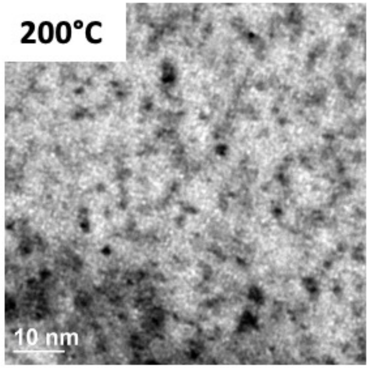
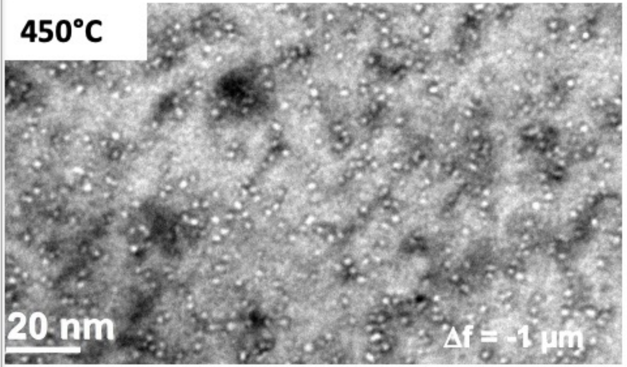
Simulation of helium transmuted from nickel and boron (100 - 300 appm He/dpa in our study – "extreme" case)
10 keV He^+ in 316L with a flux $\phi = 5\times10^{11}\ cm^{-2}.s^{-1}$ @ JANNuS-Orsay

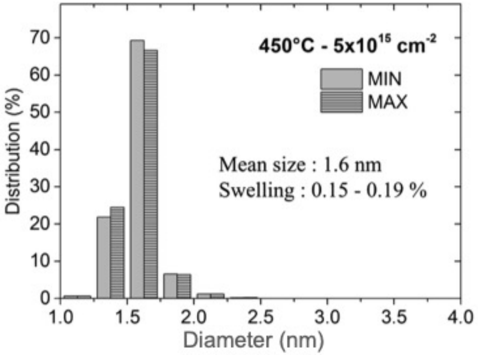
Temperature effects on He bubbles



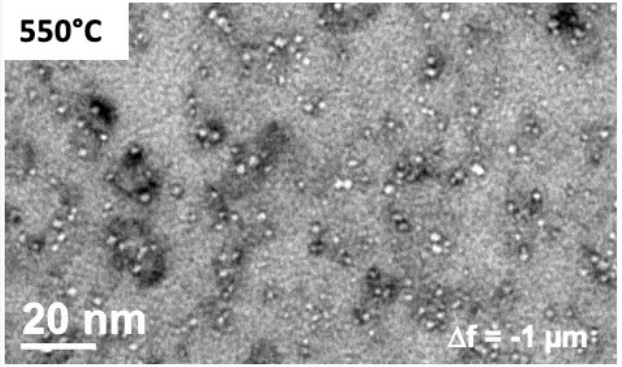
5×10^{15} He.cm^{-2}

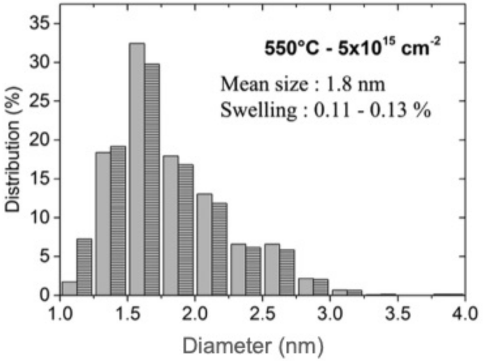
Tiny bubbles (maximal size = 0,8 nm) lost in the grainy contrast of an amorphous oxide layer on top of the specimen : NOT COUNTABLE !





Broadening of the size distribution of bubbles with increasing T°C





At each T°C, strain contrasts are observed around bubbles -> **overpressurized bubbles** (P > 0,5 – 0,75 GPa according to [1])

[1] B. Cochrane, P.J. Goodhew, Phys. Status Solidi A 77 (1983) 269

Light gas (He,H) accumulation in ODS FeCr steels

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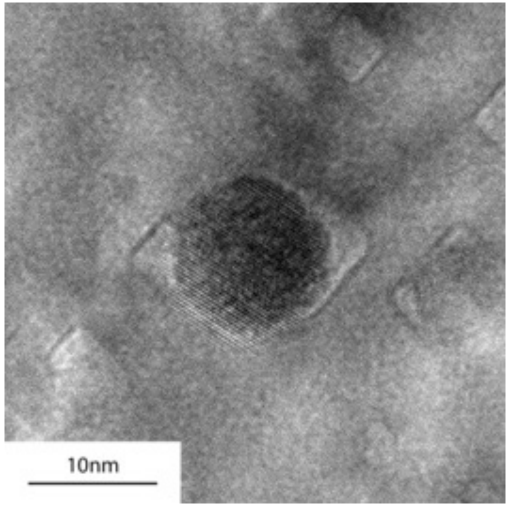
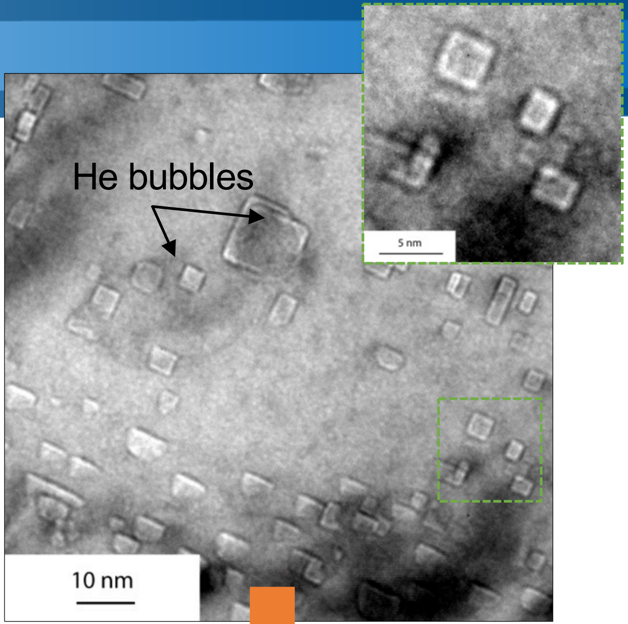


He bubble around a nano-oxide in ODS-Eurofer

O.V. Emelyanova *et al.*, Nucl. Mater. Energy **35** (2023) 101456
and J. Nucl. Mater. **545** (2021) 152724

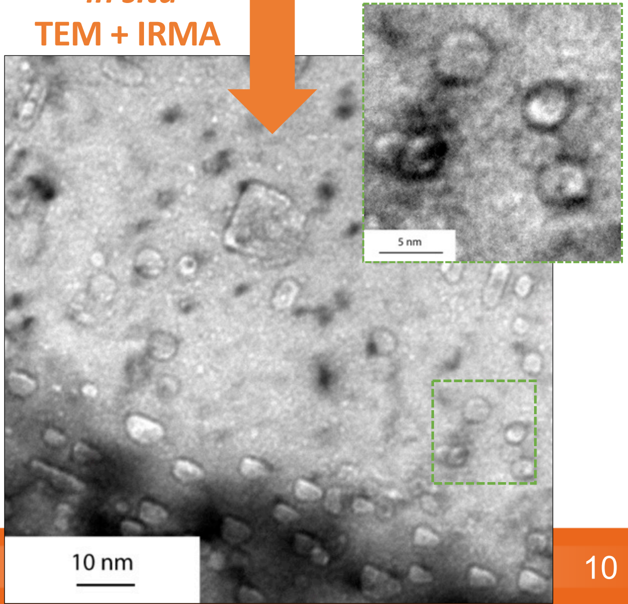
- ✓ He/H ion implantation results in pronounced evolution of the cavities shape associated with all types of the sinks
- ✓ Possible changes could be associated with H accumulation on the inner surface of the cavities

He^+, 550 °C, $1x10^{16}cm^{-2}$

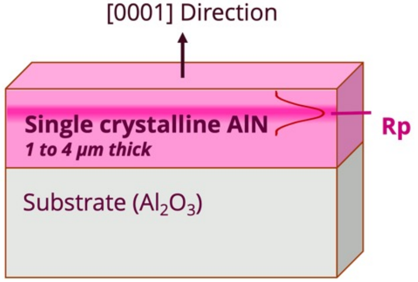


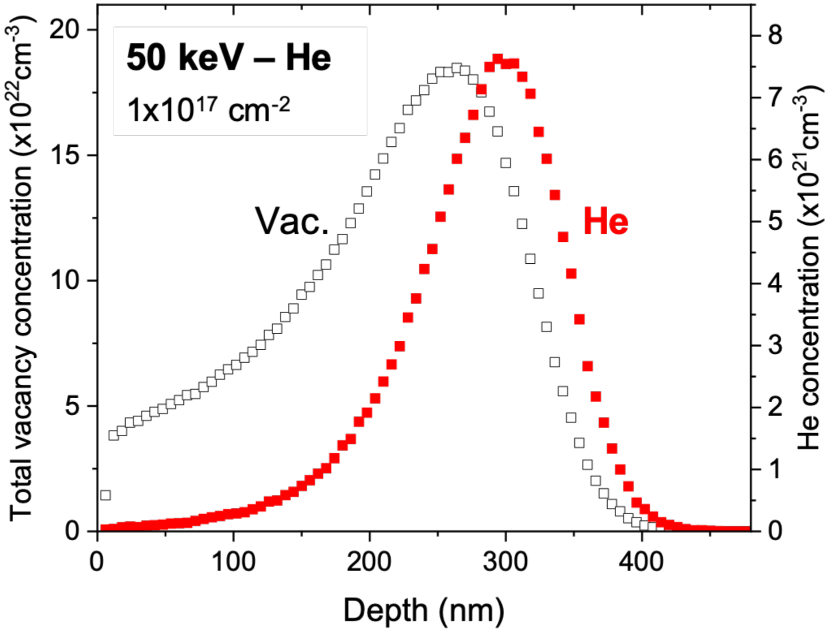
in situ
TEM + IRMA

He^+, 550 °C, $1x10^{16}cm^{-2}$
+
H^+, RT, $1x10^{17}cm^{-2}$



Effect of helium injection on the microstructural evolution of AlN

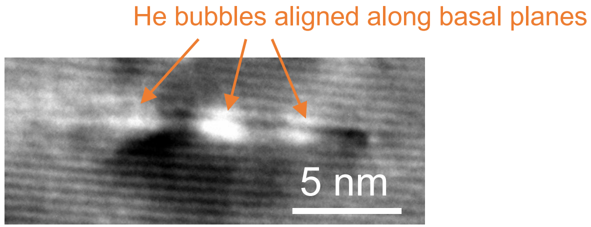




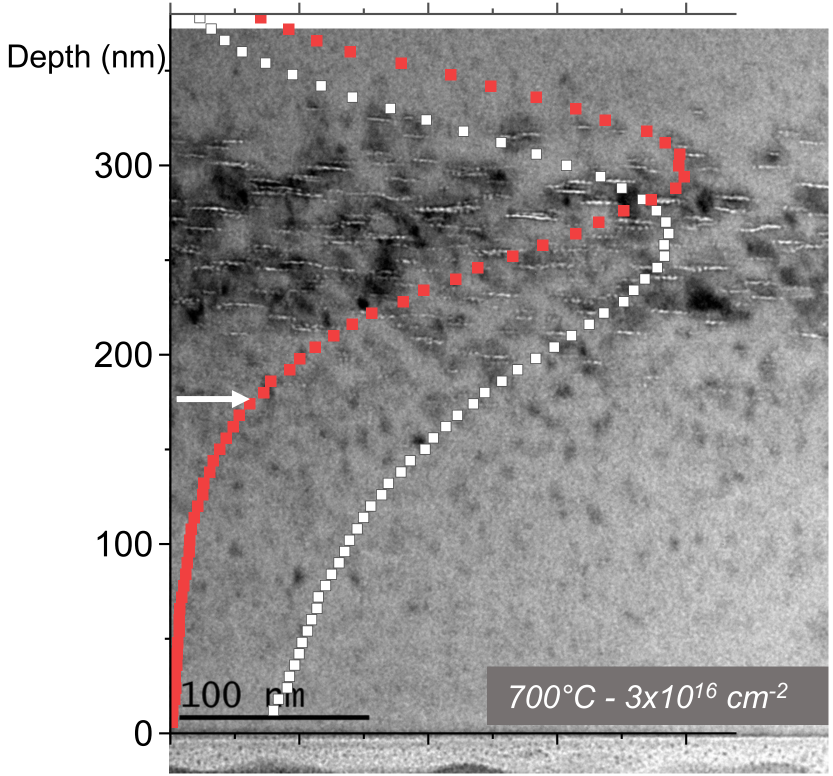
HRTEM

Higher magnification

B = [11$\bar{2}$0]



Bubbles associated with BSF



S. Jublot-Leclerc et al, submitted to Journal of the European Ceramic Society (2024)

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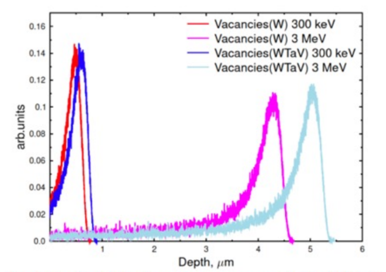

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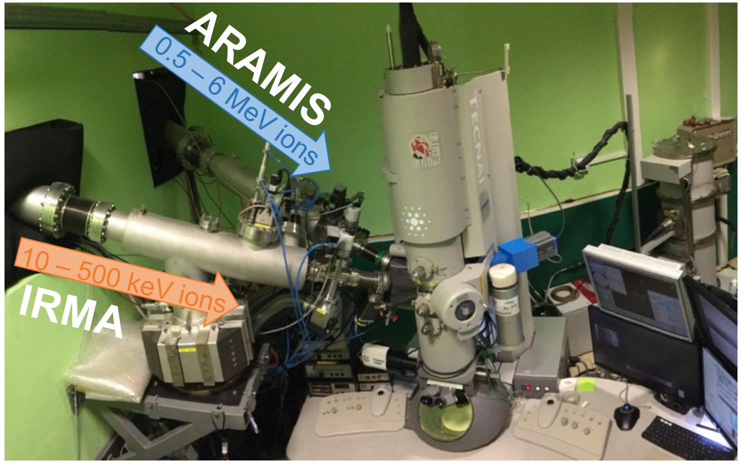

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Thank you !