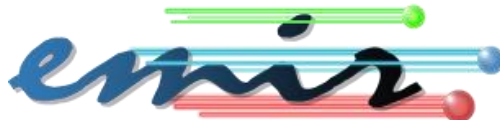




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# INFLUENCE OF FLUX AND COMPOSITION ON LOOP'S NATURE IN NICKEL DURING ION IRRADIATION

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# Industrial context

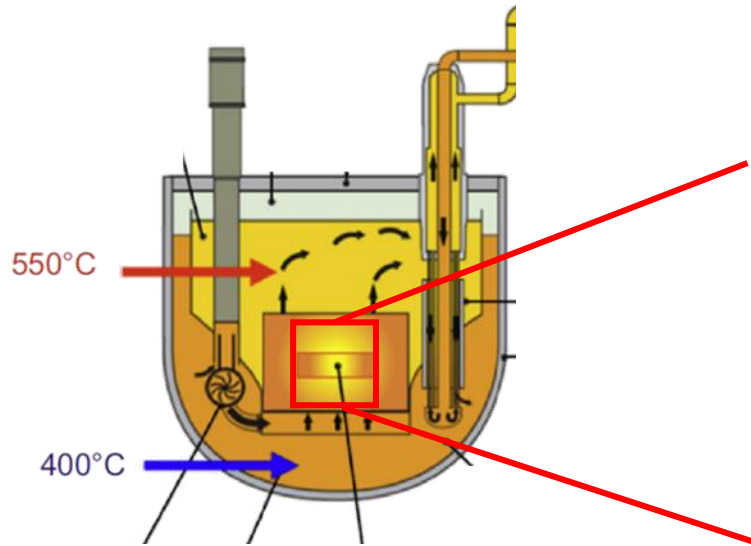
Development of next generation reactors (Sodium-cooled Fast Reactor, SFR-Gen IV)

Huge challenge for fuel cladding materials = harsh environmental conditions

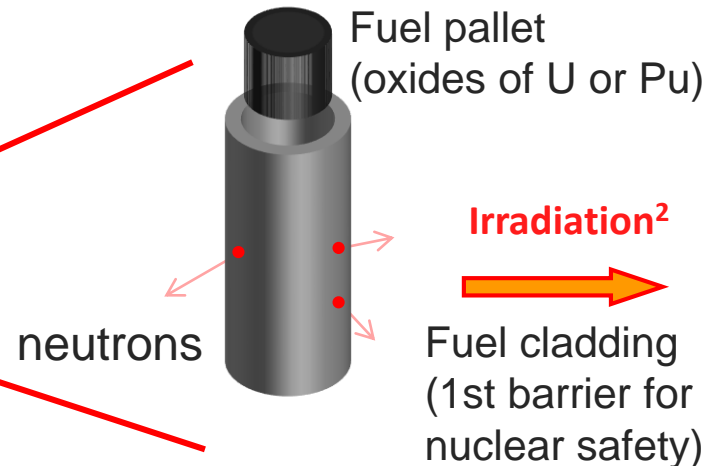
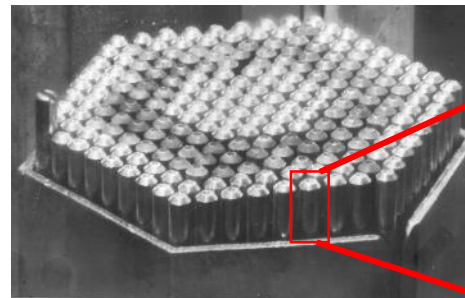
- High irradiation > 100 dpa (displacement per atom)
- High temperature 450-600°C

Austenitic Materials

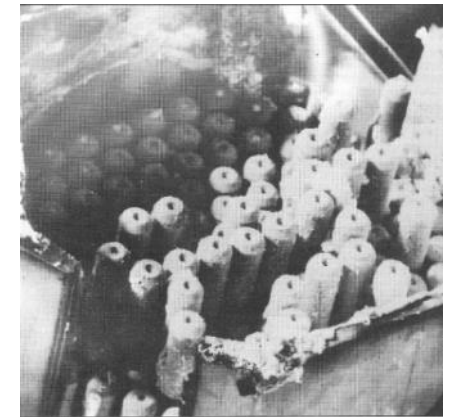
- Foreseen as the first candidate for fuel cladding of SFR-Gen IV
- Well-known major issue : 3D volume extension => irradiation-induced **void swelling**



Typical SFR diagram with nominal temperature<sup>1</sup>

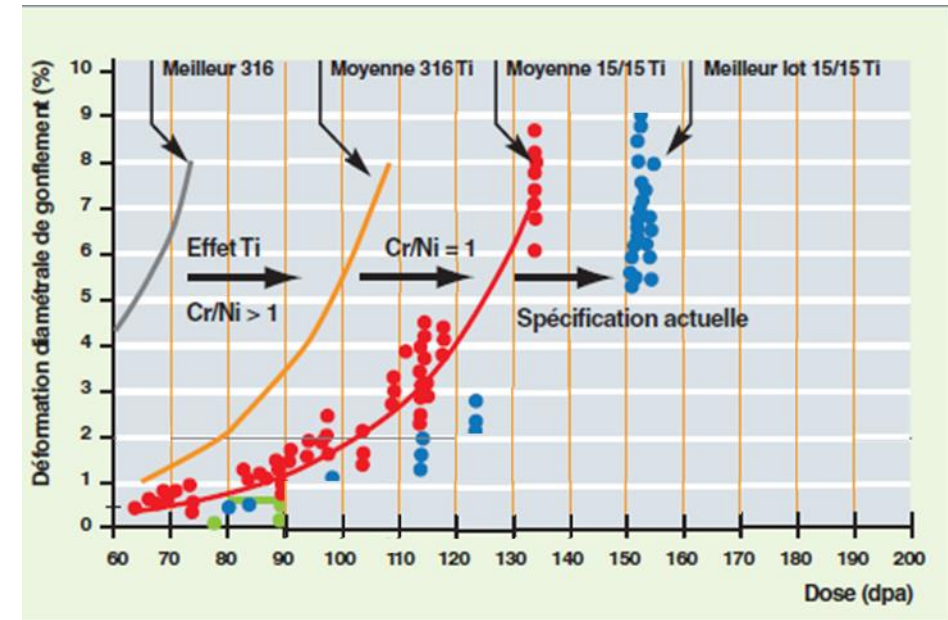
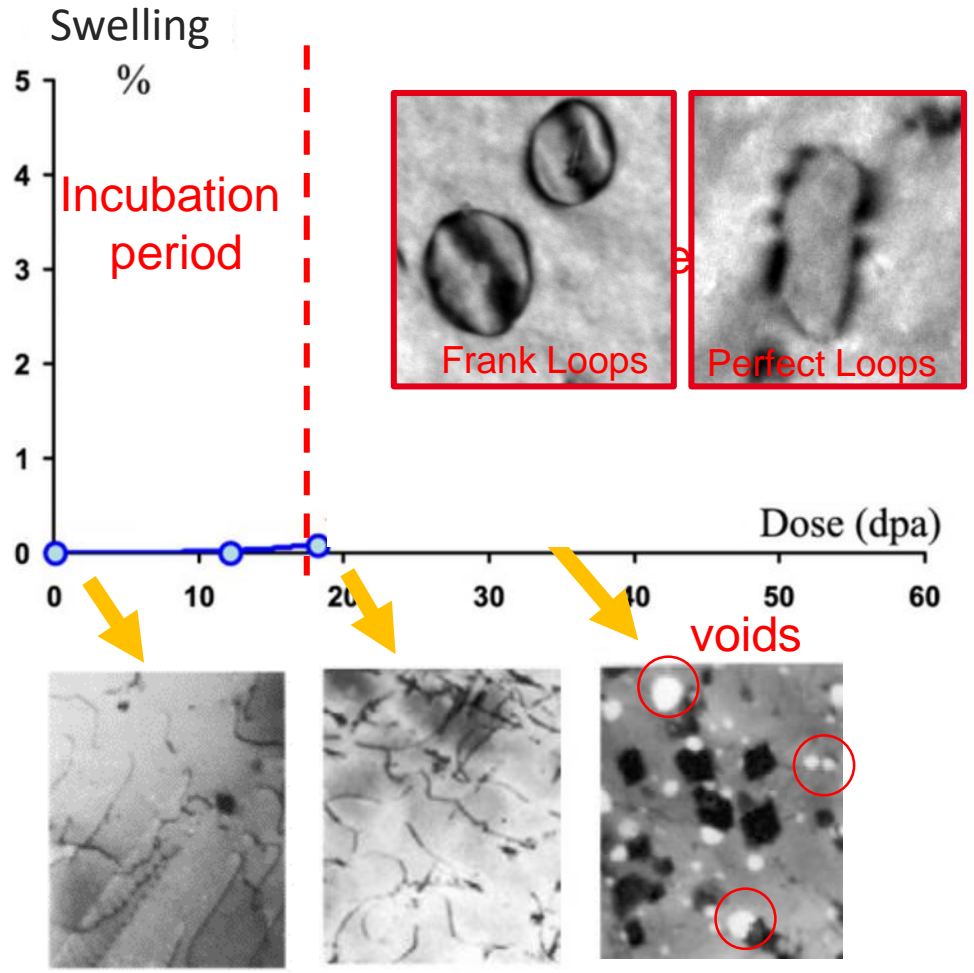


Swelling of austenitic stainless steels in SFR<sup>2</sup>



# Radiation-induced void swelling in austenitic steels

Swelling of 316 ASS in function of irradiation dose at 600°C in Phénix<sup>1</sup>.



Swelling of different cladding candidates of Gen-IV reactors<sup>3</sup>

➤ Influence of composition ?

# Approach

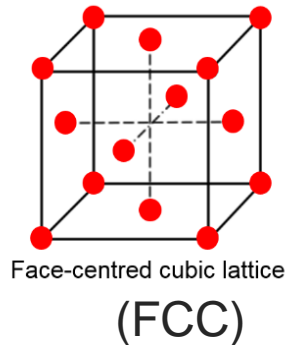
## 1. Objective

Better understand the mechanisms governing the early-stage irradiation behavior of austenitic structure (FCC) structure

## 2. Approach

### ■ Simplification of studied materials

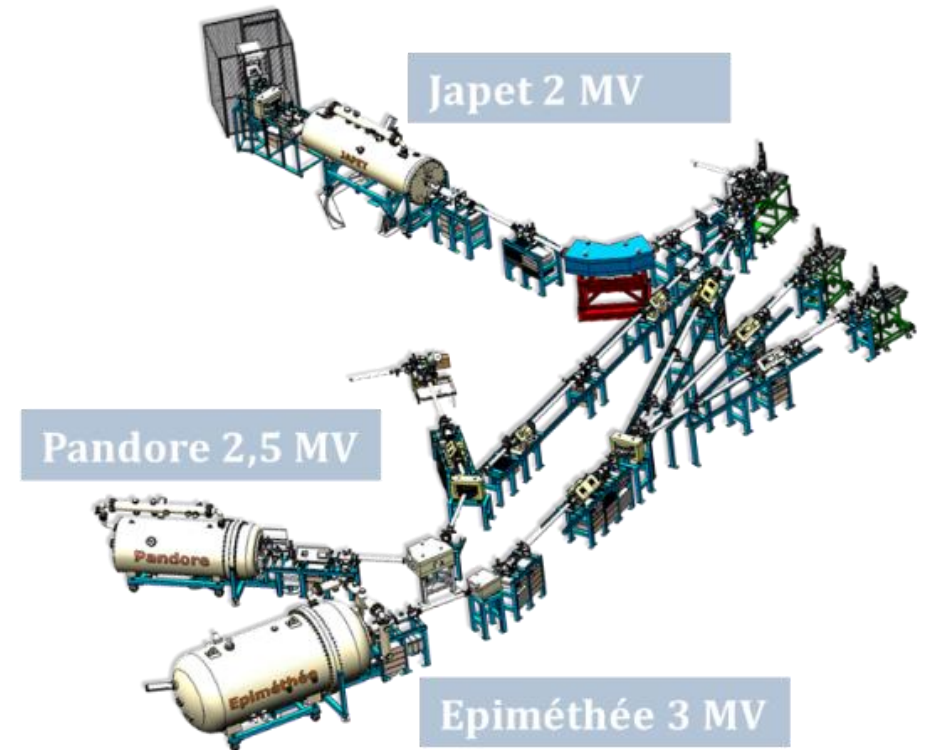
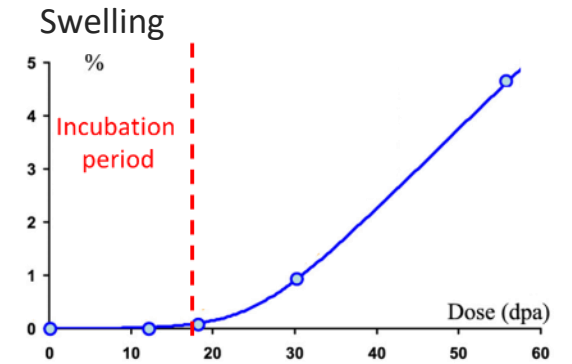
Ni and nickel alloys (Ni<sub>0,8</sub>Ti)  
→ FCC structure



### ■ Simplification of experiment conditions

- Ion irradiations on JANNuS platforms
- Good control of various flux and irradiation temperatures

→ Influence of flux ?

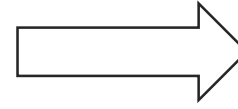
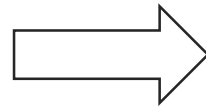
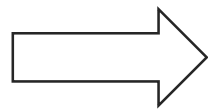
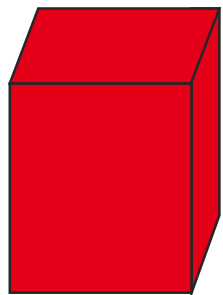


# Approach – sample preparation

Ni - Prepared at Ecole des Mines St. Etienne

Element Wt%(ppm)	C	S	O	N	Ti/Cr	Ni
Ni	8	2	3	2	\	Bal.

Ni<sub>0,8</sub>Ti (99,2% Ni + 0,8% Ti) - Prepared in CEA Saclay

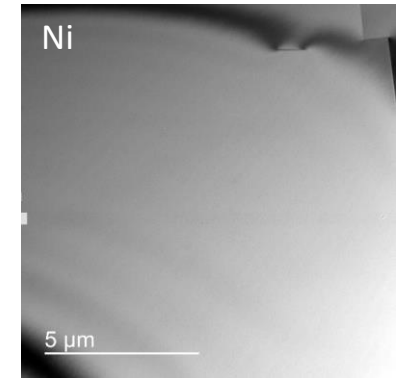


Mechanical polishing  
100 μm

3 mm disc  
punching

Heat treatment  
(annealing 1000°C)

Electrolytic  
polishing



Microstructure characterization  
before irradiation

Irradiations

Observations by TEM,  
Thin foil characterization



# Approach – irradiations



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JANNuS facilities Saclay and Orsay



Material	Platform	Temperature	Flux
Ni	JANNuS Orsay ARAMIS Ni <sup>2+</sup> 2MeV	450°C	<b>High</b> 3.10 <sup>-4</sup> dpa/s
Ni	JANNuS Saclay Japet Ni <sup>2+</sup> 5MeV	450°C	<b>Intermediate</b> 6.10 <sup>-5</sup> dpa/s
Ni	JANNuS Saclay Epiméthée Fe <sup>9+</sup> 22,5MeV	450°C	<b>Low</b> 6,7.10 <sup>-6</sup> dpa/s
<b>Ni 0,8 Ti</b>	JANNuS Orsay ARAMIS Ni <sup>2+</sup> 2MeV	450°C 510°C ; 560°C	<b>High</b> 3,1-3,5.10 <sup>-4</sup> dpa/s

## Iradina:

- SRIM like, quick calculation,  
 $E_{\text{displacement}} = 40 \text{ eV}$



# 1 ■ Influence of flux

# High flux

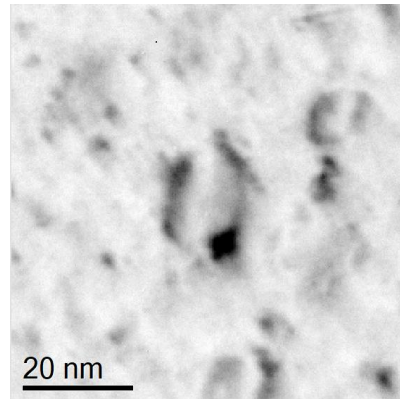
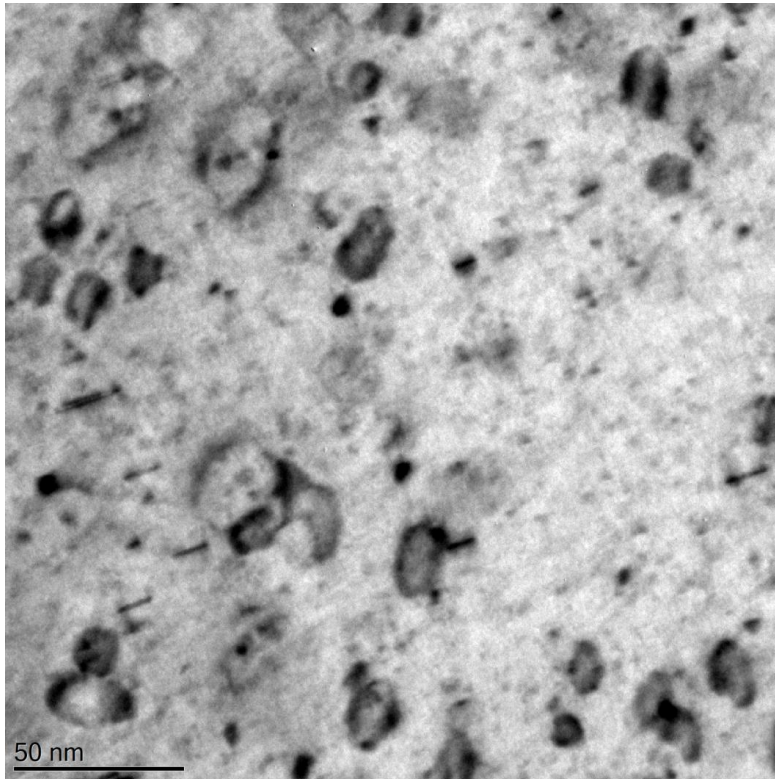
Ions = Ni<sup>2+</sup> 2MeV (JANNuS Orsay)

Ion flux = 4e11 ions/cm<sup>2</sup>/s ⇔ **G = 3.10<sup>-4</sup> dpa/s**

T = 450°C

Dose = 0.18 dpa

$g = [0\bar{2}0]$  ←



→ Vacancy type loops



?



# High flux

- ▶ At steady state, **standard rate theory** predicts a radius variation of a vacancy loop :

$$\frac{dR}{dt} = \frac{\Omega}{b} \left( Z_{l,v} - Z_{l,i} \frac{k_v^2}{k_i^2} \right) D_v C_v < 0$$

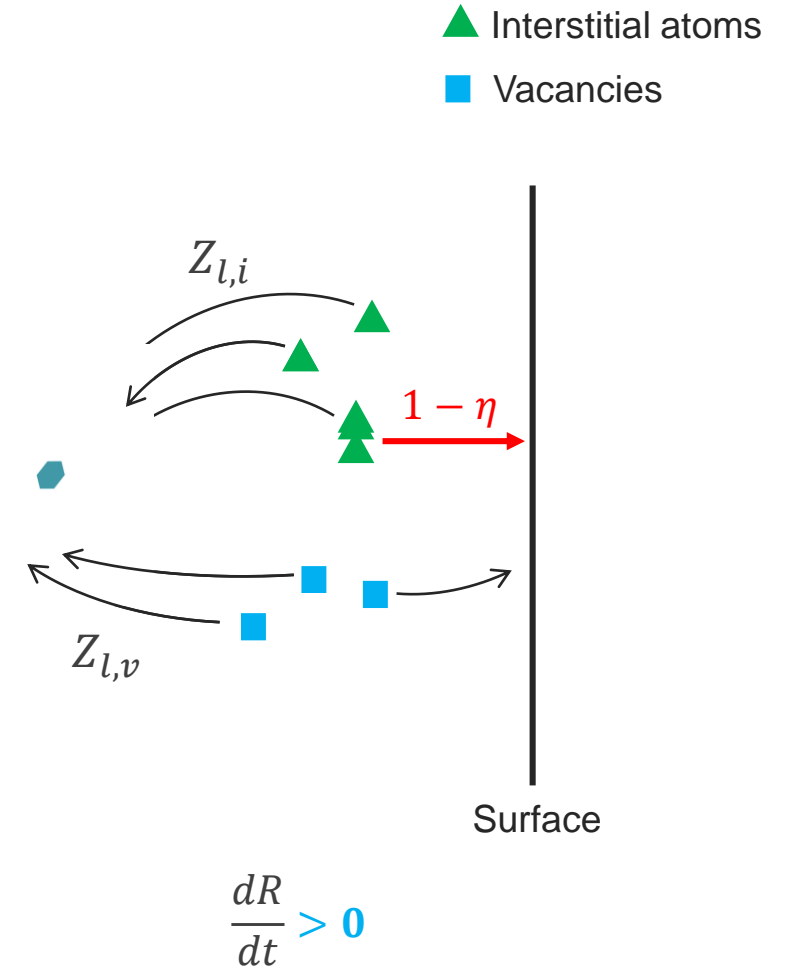
- ▶ **Vacancy-type loops should not grow**

- ▶ Introduction of production bias  $1 - \eta$  (surface effect)

$$\frac{dR}{dt} = \frac{\Omega}{b} \left( Z_{l,v} - Z_{l,i} \frac{k_v^2}{k_i^2} \right) D_v C_v + \frac{\Omega Z_{l,i}}{b k_i^2} G(1 - \eta)$$

Dislocation bias term  $< 0$

Production bias term  $> 0$



K. Ma, B. Décamps, T. Jourdan, M. Loyer-Prost & al., *Acta Materialia* 212 (2021)\*

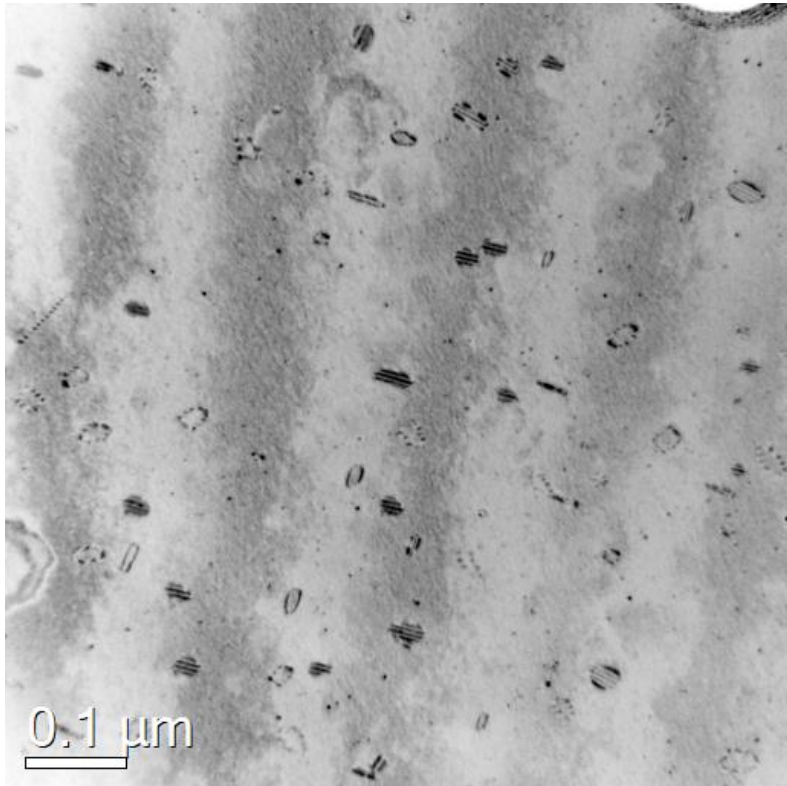
# Low flux

Ions = Fe<sup>9+</sup> 22,5 MeV (JANNuS Saclay)

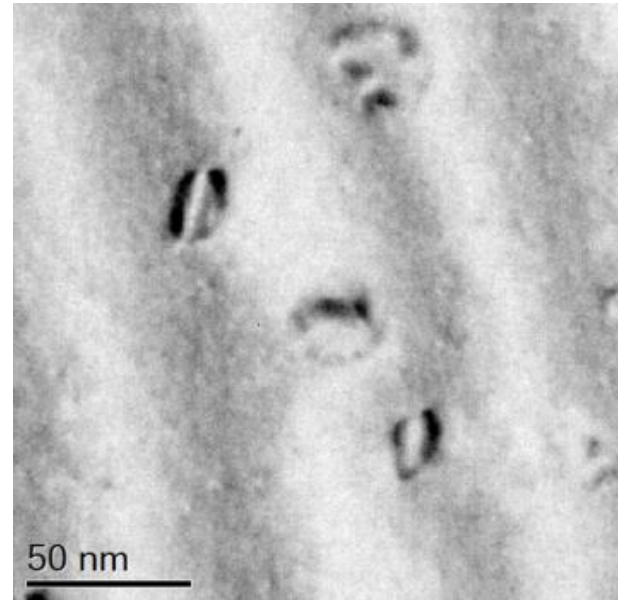
Ion flux = 7.93e10 ions/cm<sup>2</sup>/s ⇔ **G = 6,7.10<sup>-6</sup> dpa/s**

T = 450°C

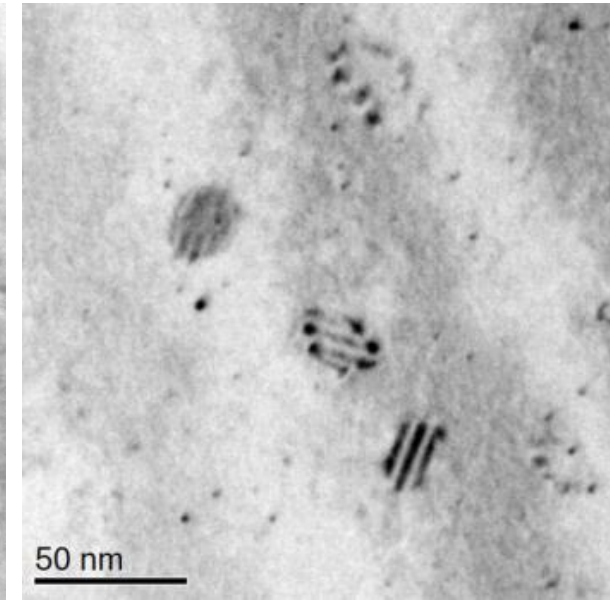
Dose = 0.06 dpa



$g = [200]$  ↗



$g = [\bar{2}00]$



→ Interstitial type loops

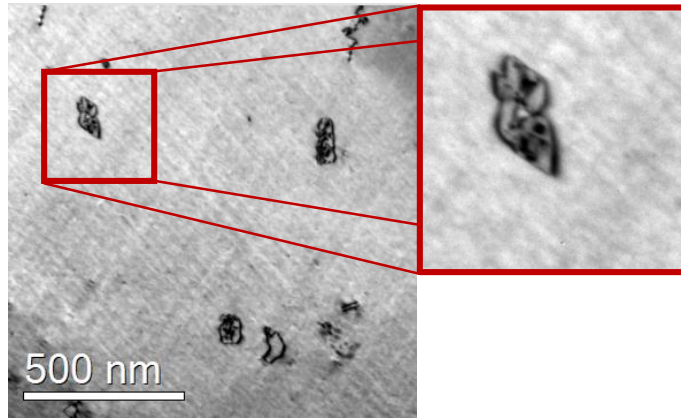
# Intermediate flux

Ions = Ni<sup>2+</sup> 5MeV (JANNuS Saclay)

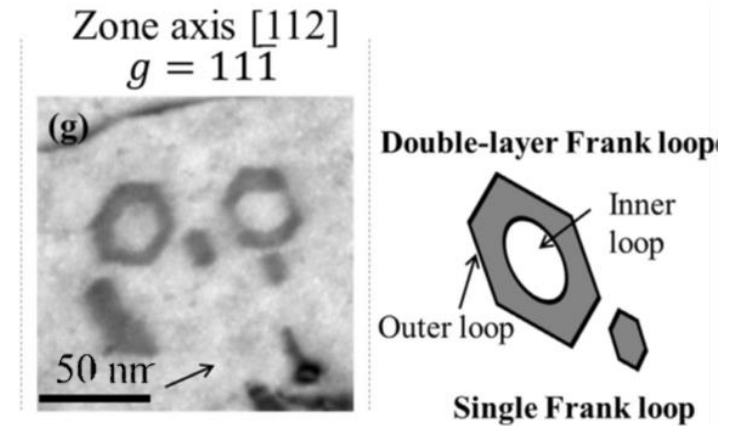
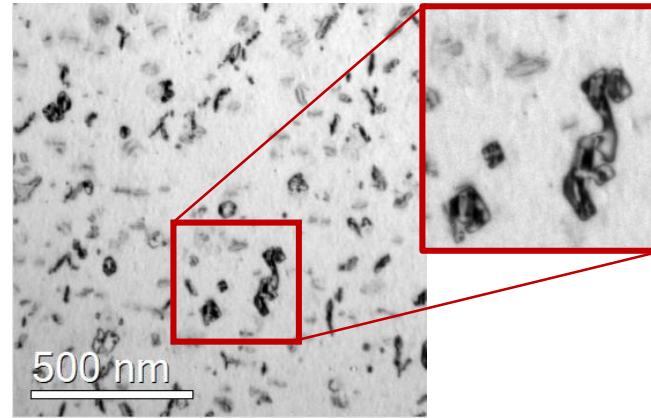
Ion flux = 2.2e11 ions/cm<sup>2</sup>/s ⇔ **G = 6.10<sup>-5</sup> dpa/s**

T = 450°C

Dose = 0.06 dpa

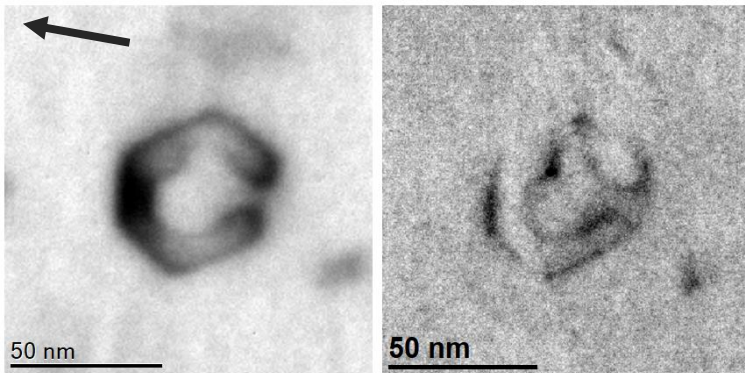


Dose = 0.7 dpa

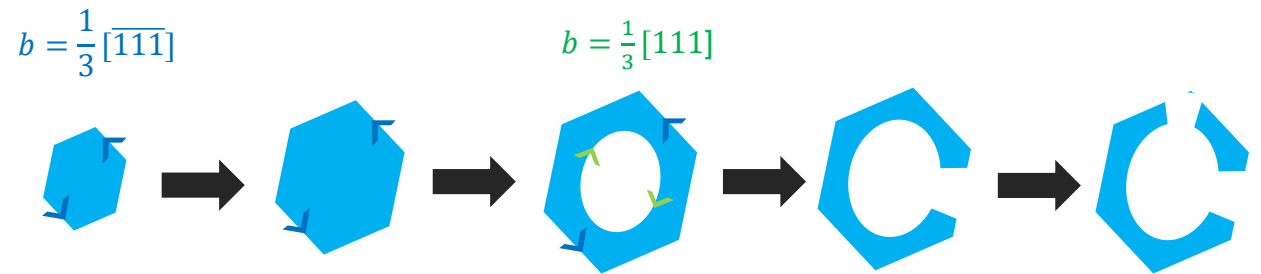


*K. Ma & al., Inversion of dislocation loop nature driven by cluster migration in self-ion irradiated nickel. Scripta Materialia 208 (2022).*

Z = [121], g = [111]



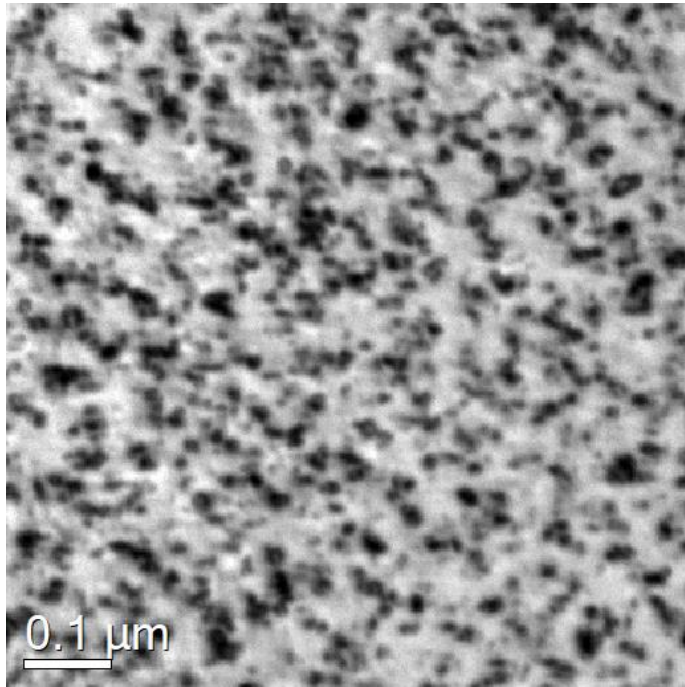
→ Vacancy type loop



# To sum up

0.06 dpa, 450°C

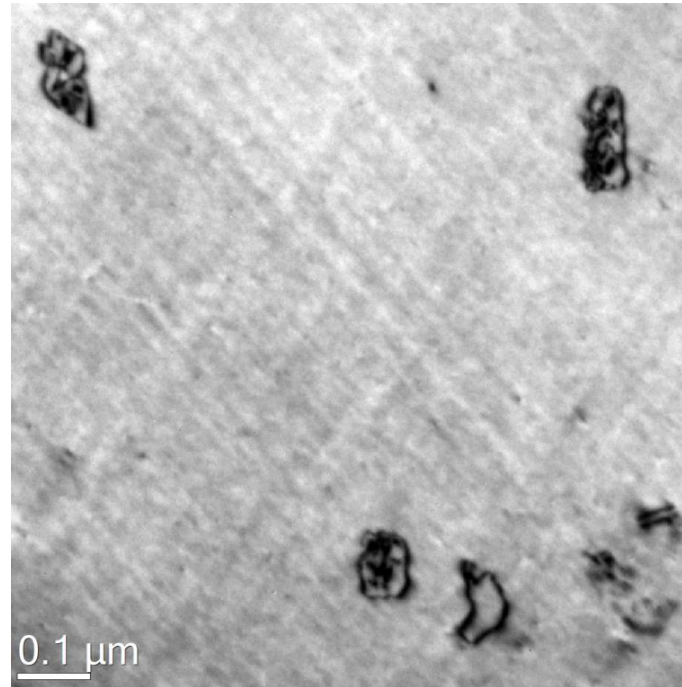
High flux  
 $3 \cdot 10^{-4}$  dpa/s



t ~ 200 nm

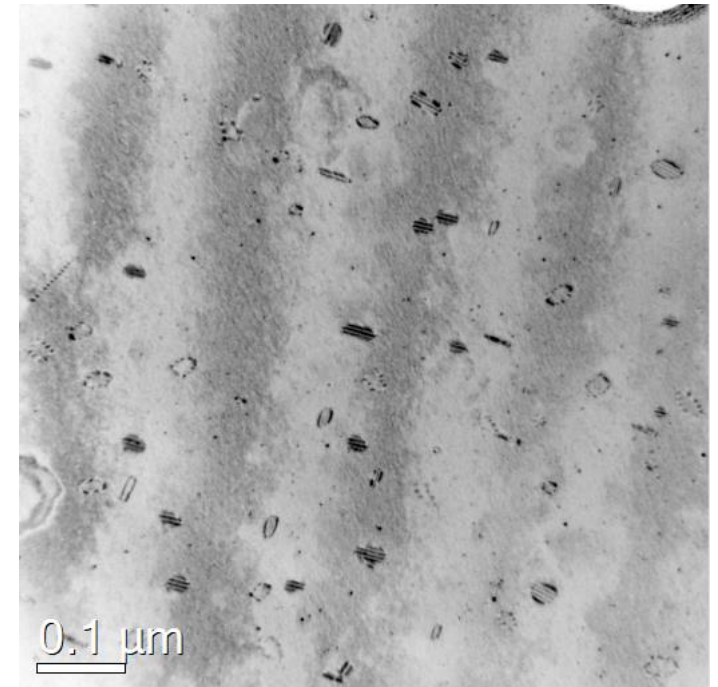
Vacancy type

Intermediate flux  
 $6 \cdot 10^{-5}$  dpa/s



t ~ 200 nm

Low flux  
 $6.7 \cdot 10^{-6}$  dpa/s



t ~ 80 nm

Interstitial type



# 2 ■ Influence of composition



# Influence of composition

Ions =  $\text{Ni}^{2+}$  2MeV (JANNuS Orsay)

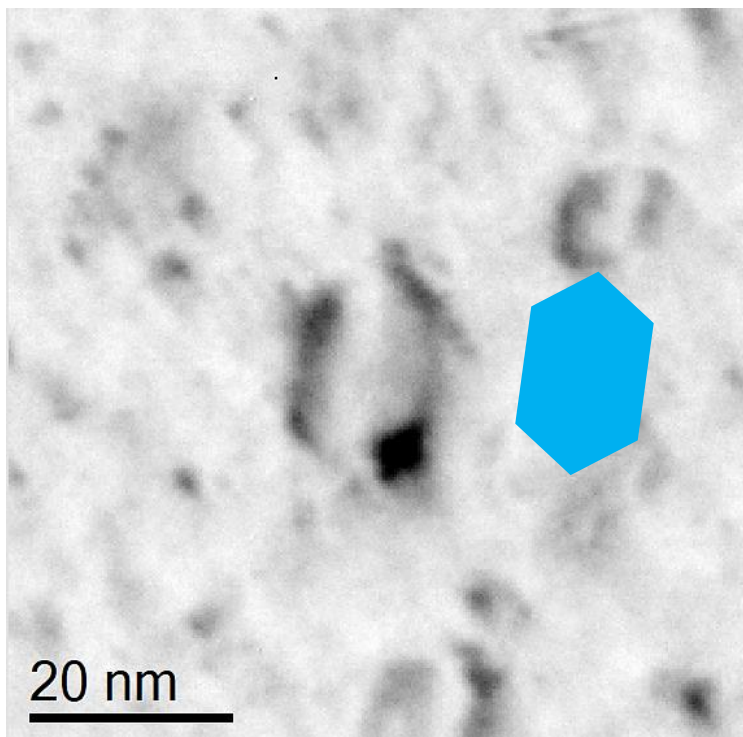
Ion flux =  $4 \times 10^{11}$  ions/cm<sup>2</sup>/s  $\Leftrightarrow$  G =  $3 \cdot 10^{-4}$  dpa/s

T = 450°C



**Ni**

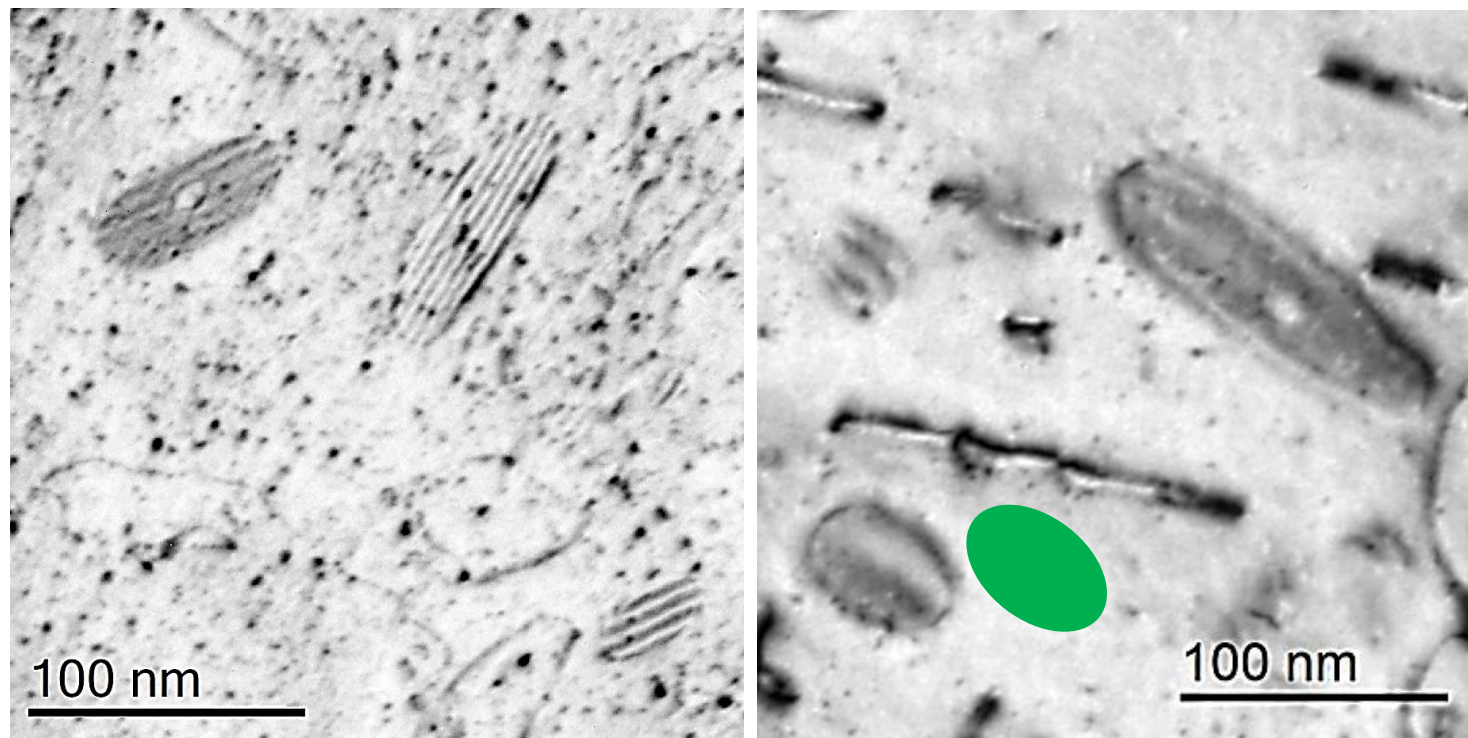
0,18 dpa



→ Vacancy type loops

**Ni<sub>0,8</sub>Ti**

0,66 dpa



→ Interstitial type loops

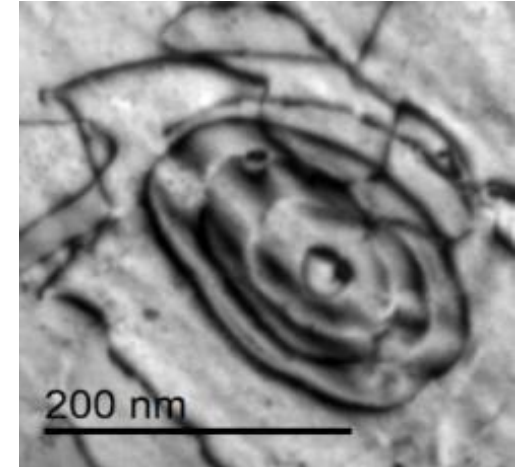
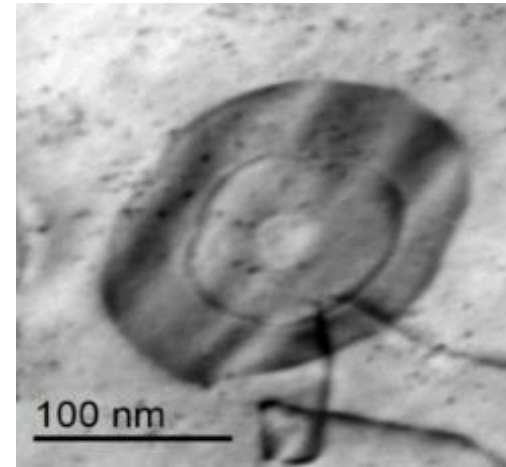
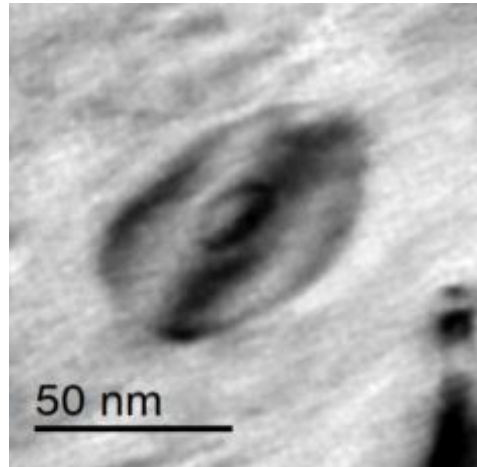
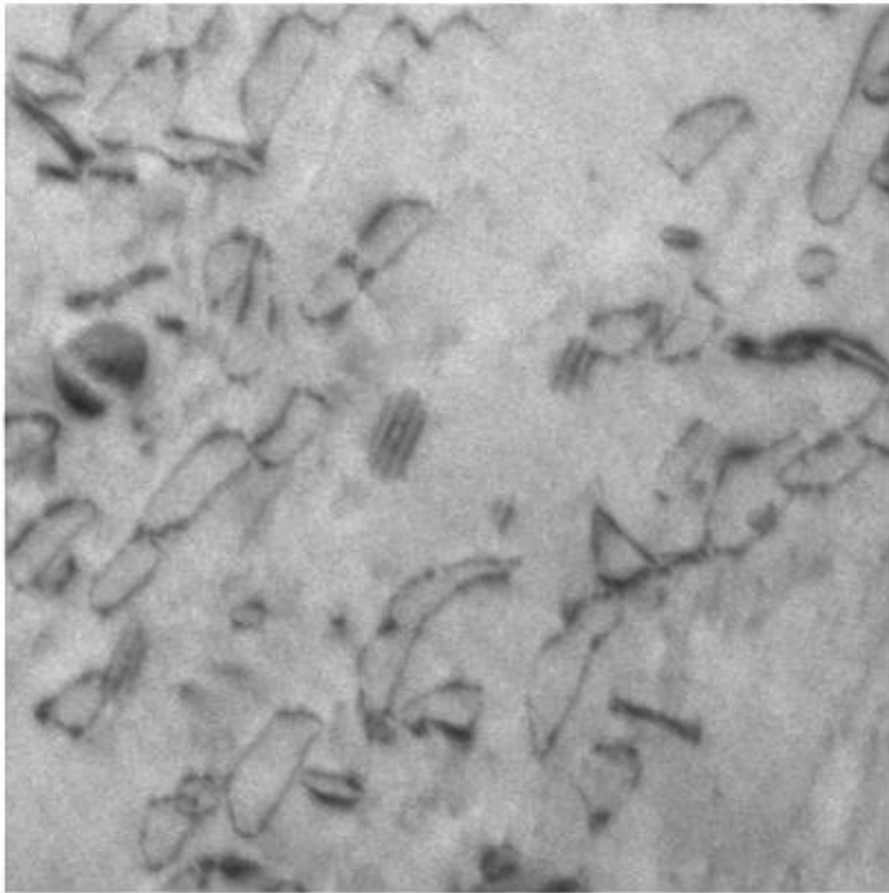
→ 1-  $\eta$  lower in Ni<sub>0,8</sub>Ti



# Multi-layer loops



560°C



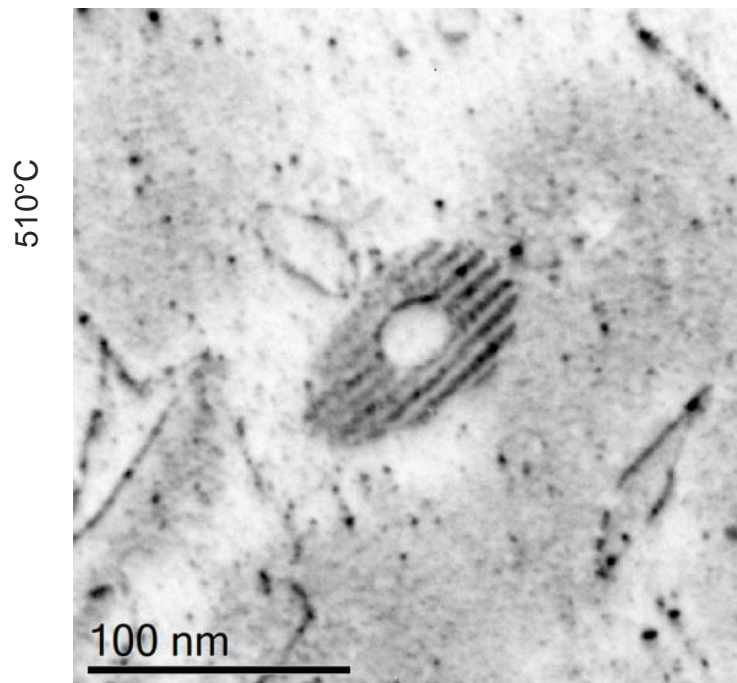
→ Very close to each other

→ Same Burgers

# Frank loop with perfect crystal inside

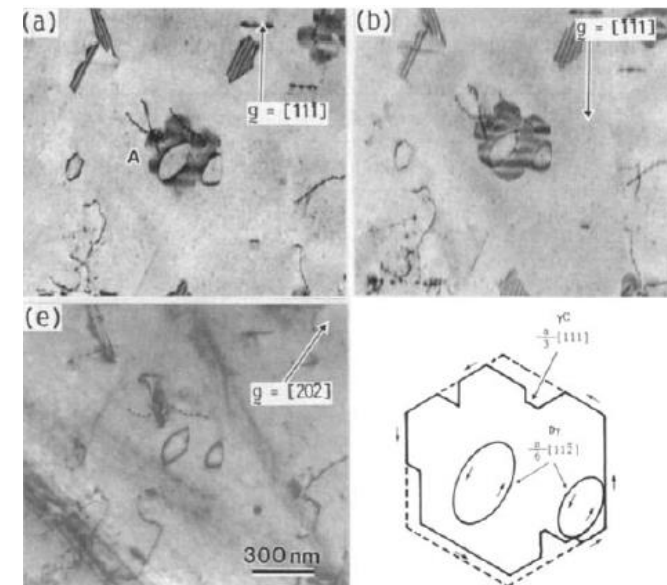



 faulted/unfaulted



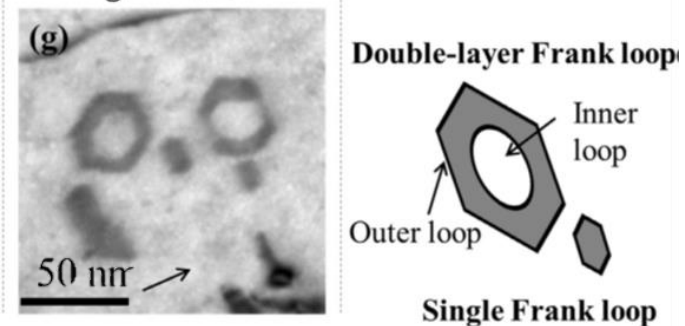
→ Shockley partial ?

→ Inner vacancy loop ?



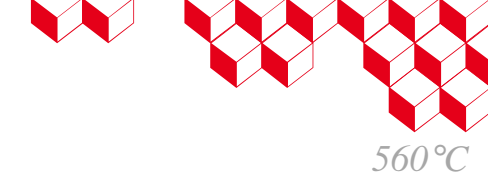
Suzuki et al., Philosophical Magazine A (1992) 1309

Zone axis  $[112]$   
 $g = 111$

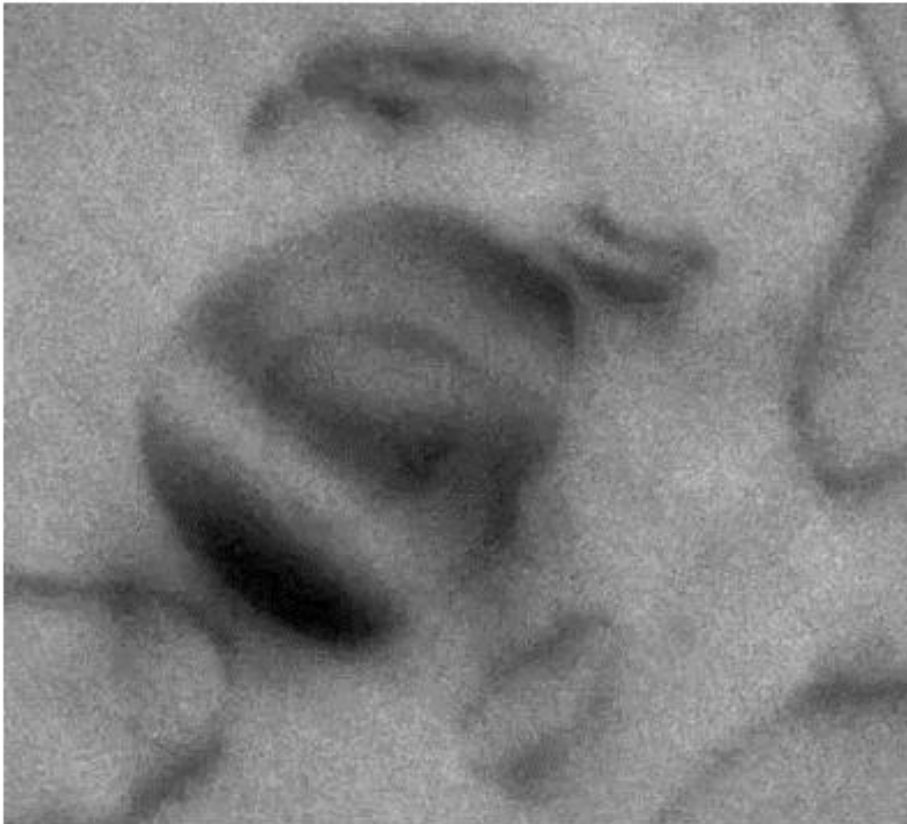


K. Ma & al., Scripta Materialia 208 (2022).

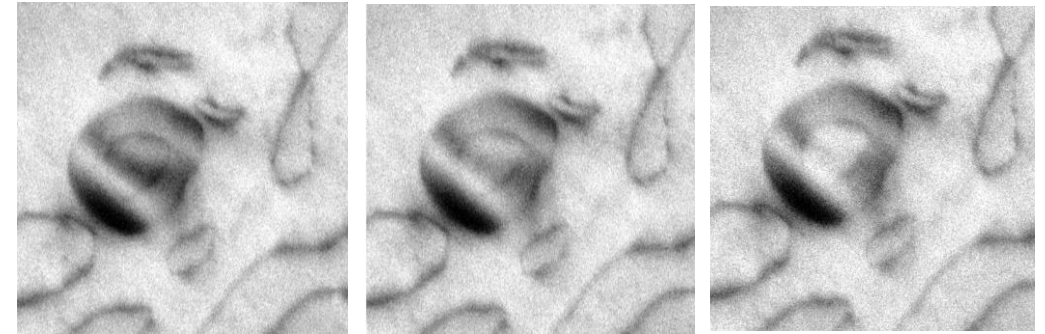
# Frank loop with perfect crystal inside



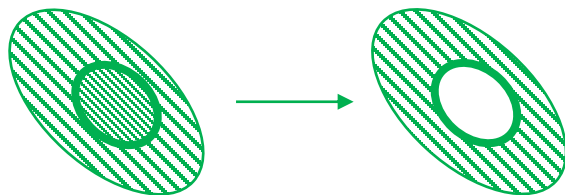
560°C



[020]



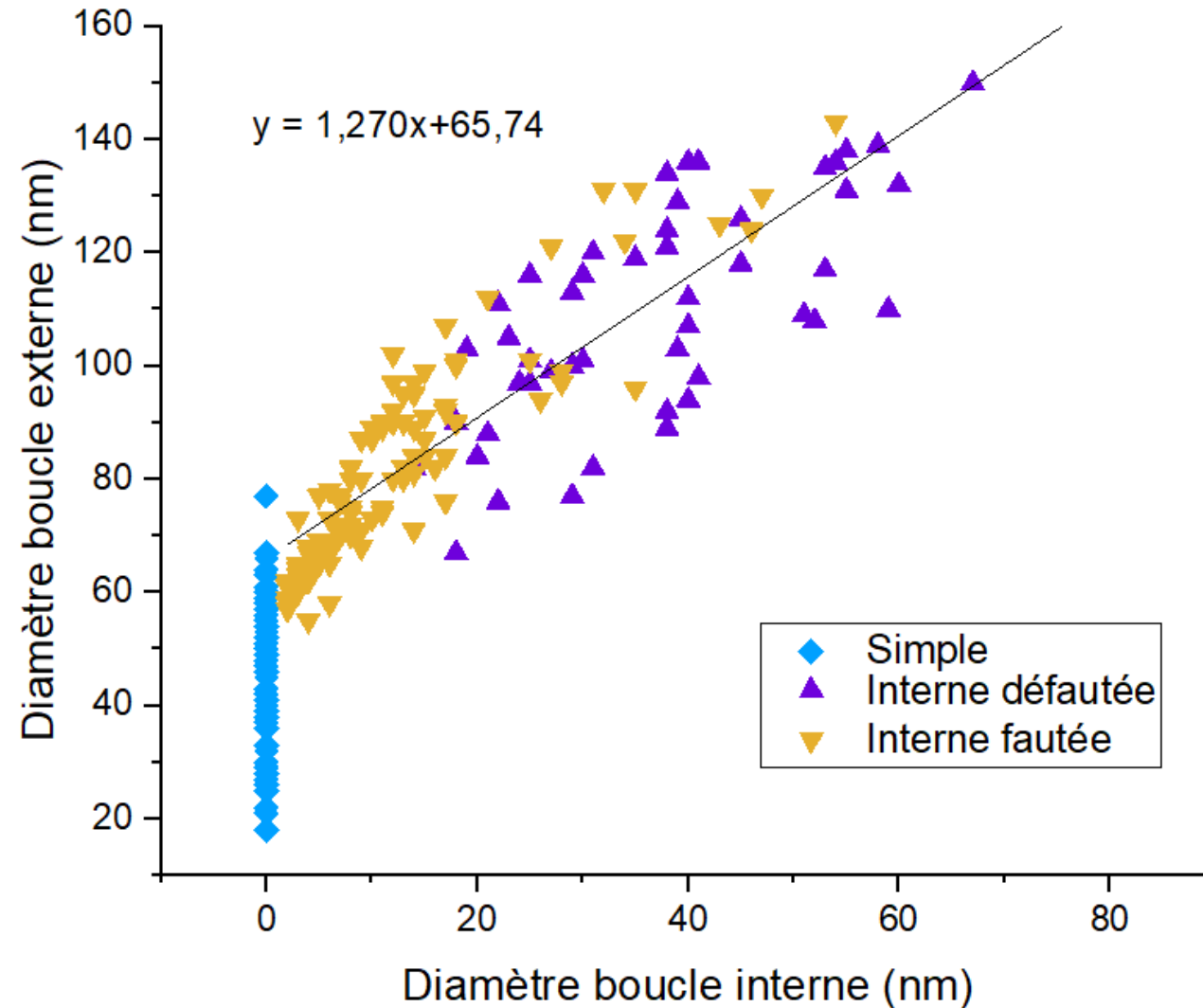
$\Delta t = 0,25s$



→ Shockley partial ?

→ Inner vacancy loop ?

# Frank loop with perfect crystal inside



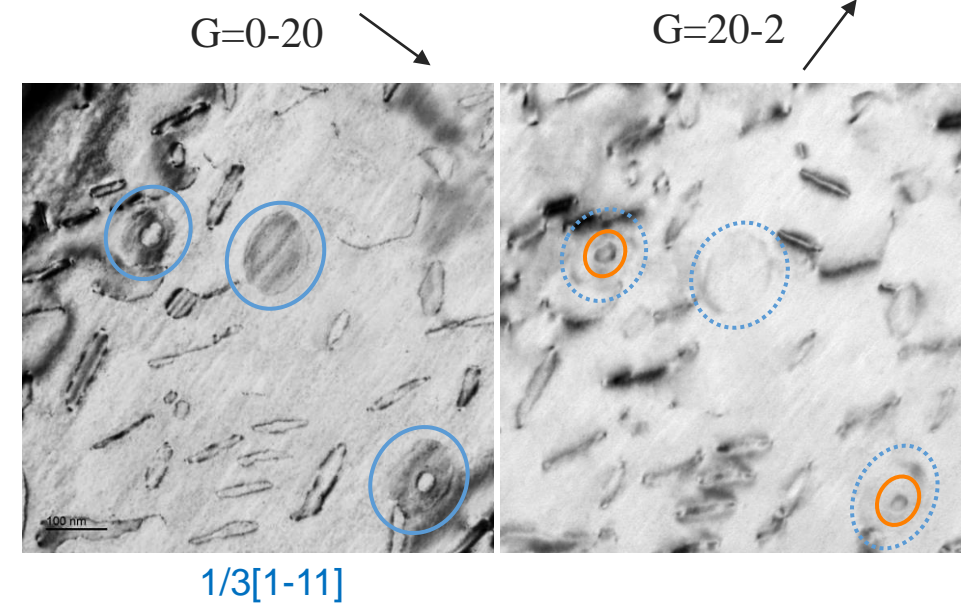
# Frank loop with perfect crystal inside



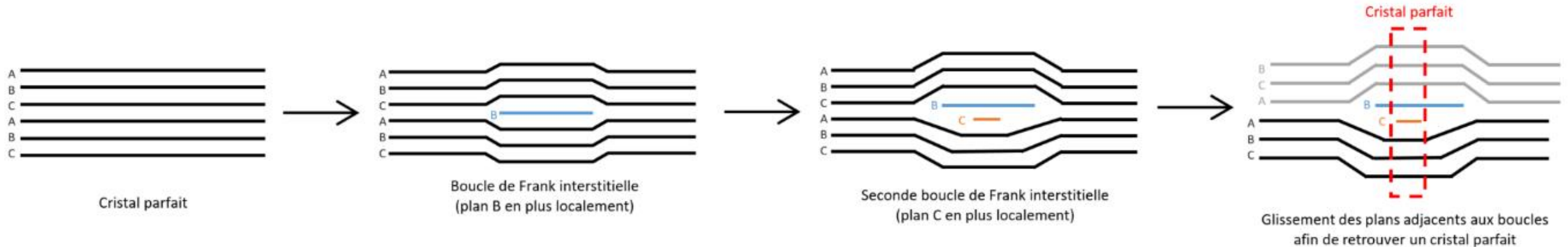
Visibility of external and internal loops (560°C)

→ internal loop Burger vector =  $1/a[1-12]$

$$2/3[1-11] + 1/6[-112] = 1/2[1-12]$$



Mechanism of internal loop unfaulting by Shockley partial





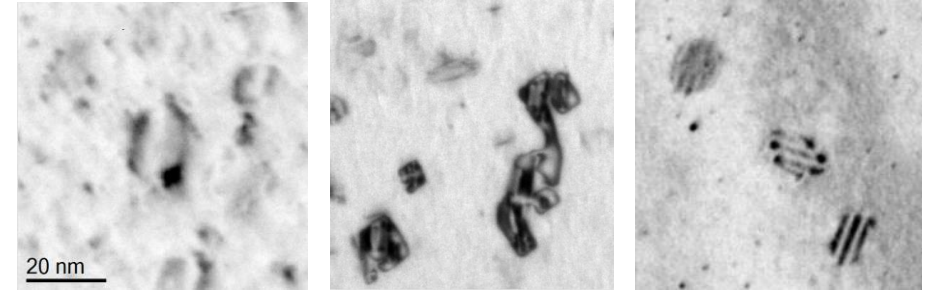


# 3 ■ Conclusion

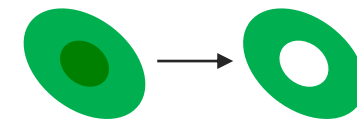
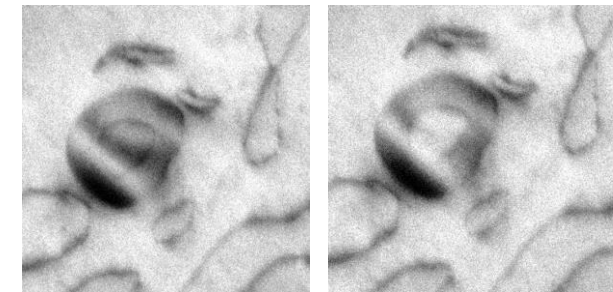


# Conclusion

- Flux variation can change loop's nature
- Complex microstructure for intermediate flux
- Relation between shape and nature's loop for Frank loop



- Titanium significantly reduces interstitial losses at surfaces
- Multiple loops can unfault partially, becoming faulted/unfaulted

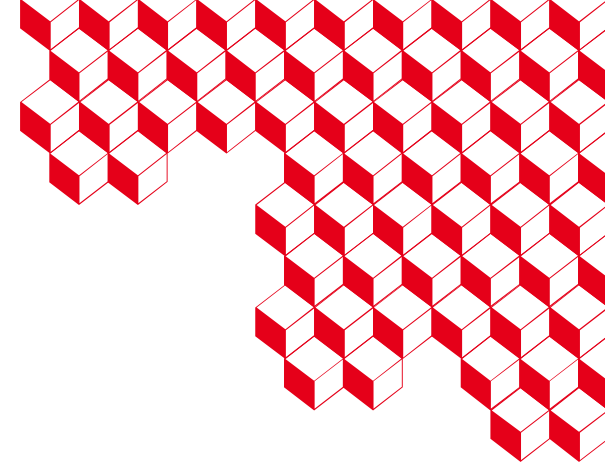


# Prospects

- Further study on multiple loops
- Removal of temporal variation of the flux : effect of defocused beam



isds



# Thank you

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The JANNuS-Orsay and JANNuS-Saclay teams for the irradiations and their welcome.

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