Journées MOSAIC
September 25th 2024

PhD student – Antoine Dartois (2022 – 2025)

In-situ quantitative characterization of microstructures in iron-based alloys from binary $Fe_{60}Ni_{40}$ to HEA – $Fe_{37}Ni_{34}Cr_{16}Mn_{13}$

Supervisors : Estelle Meslin[1], Maylise Nastar[1], Brigitte Décamps[2], in collaboration with Anna Fraczkiewicz[3]

1: CEA/ISAS/DRMP/S2CM/SRMP

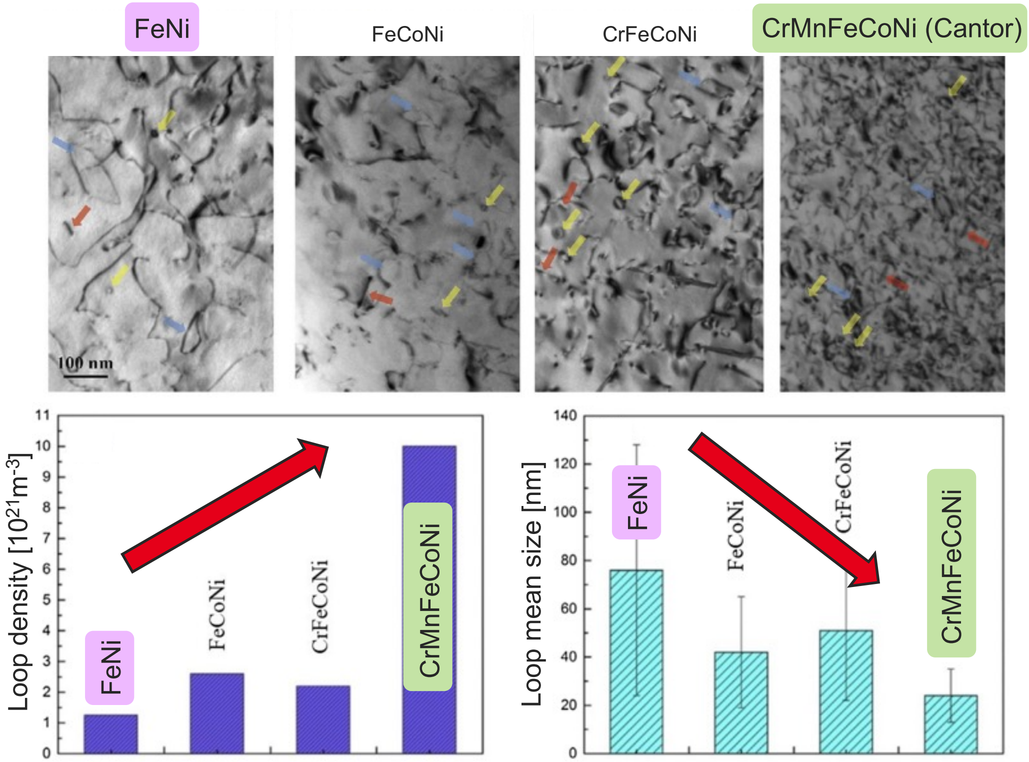
2 IJClab/IN2P3

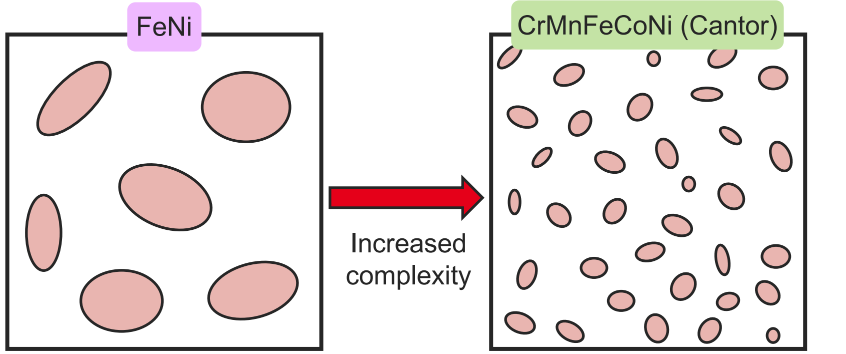
3 EMSE

Controversial results on HEA microstructure under irradiation

Lu et al, Acta mat (2017)

3 MeV Ni at 773 K up to 38 dpa





Low density of large loops

Higher density of smaller loops

✗ Discrepancy

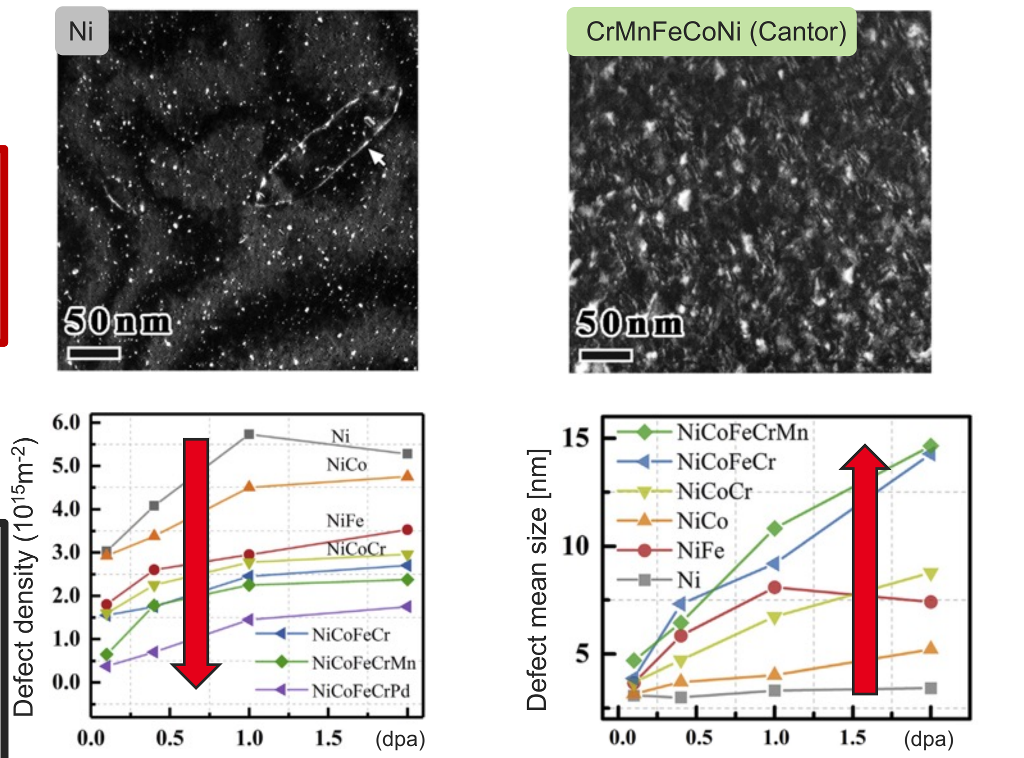
High impact of irradiation conditions and dose on microstructure

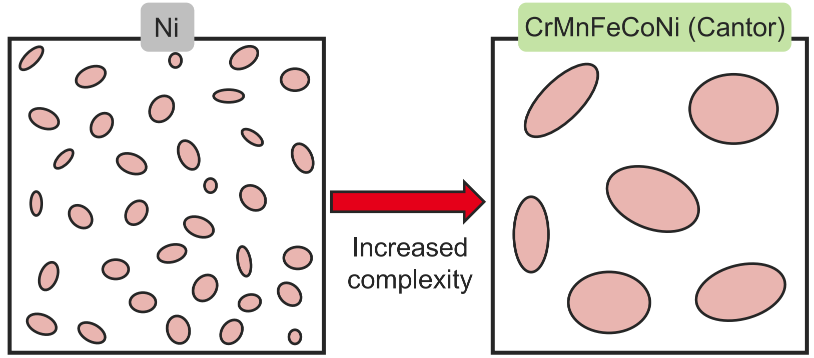
? Questions

- What about HEAs without Co?
- How differently do HEAs and stainless steels behave?

Shi et al, JNM (2018)

1 MeV Kr at 773 K up to 2 dpa



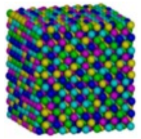


High density of small loops

Lower density of larger loops

Experimental details

3 compositions



ES1

1 — Model high-entropy alloy **$Fe_{37}Ni_{34}Cr_{16}Mn_{13}$**

2 — Industrial austenitic steel **316L**
$Fe_{\sim 62}Ni_{\sim 12}Cr_{\sim 17}$
& (Mo, Mn, Si)

3 — Model binary alloy **$Fe_{60}Ni_{40}$**

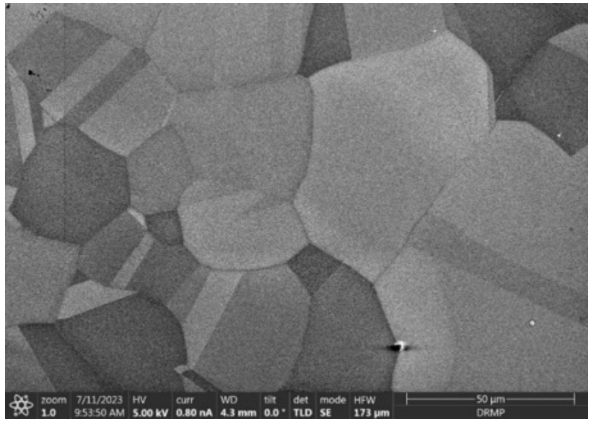
Alloy fabrication

- **High purity raw materials** (< 20 ppm of C, N, O, S)
- Cold crucible melting
- Hot forging (about 1000 °C), thermomechanical treatment homogenization (1000 °C / 2h)

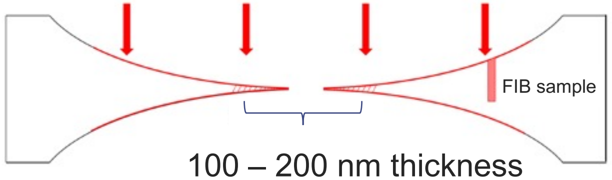
Sample preparation

Thin foils for irradiation:

- Mechanical polishing
- Thermal annealing
- Electro-polishing
- Focused ion beam

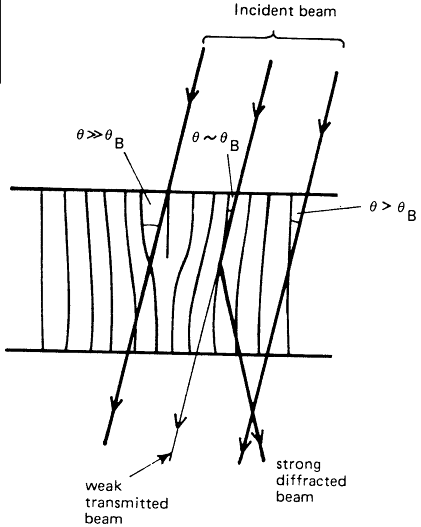


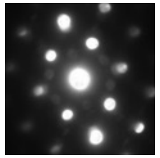
SEM – $Fe_{37}Ni_{34}Cr_{16}Mn_{13}$



Conventional TEM – defect analysis

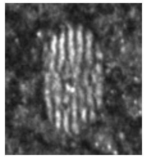
Bragg law:
$$n\lambda = 2d \sin \theta_B$$





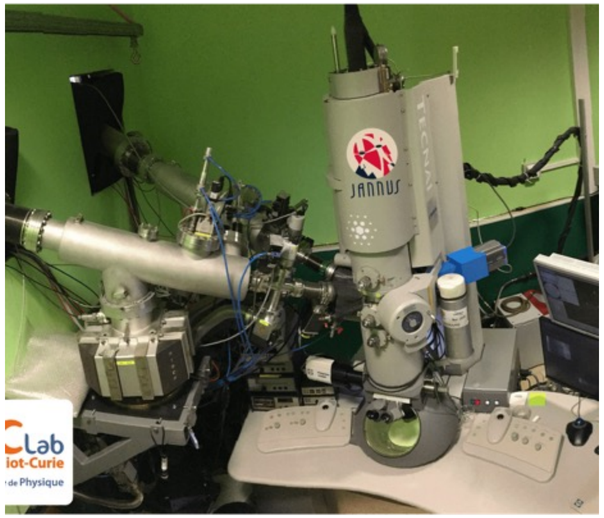
Diffraction pattern

Bright field (BF)

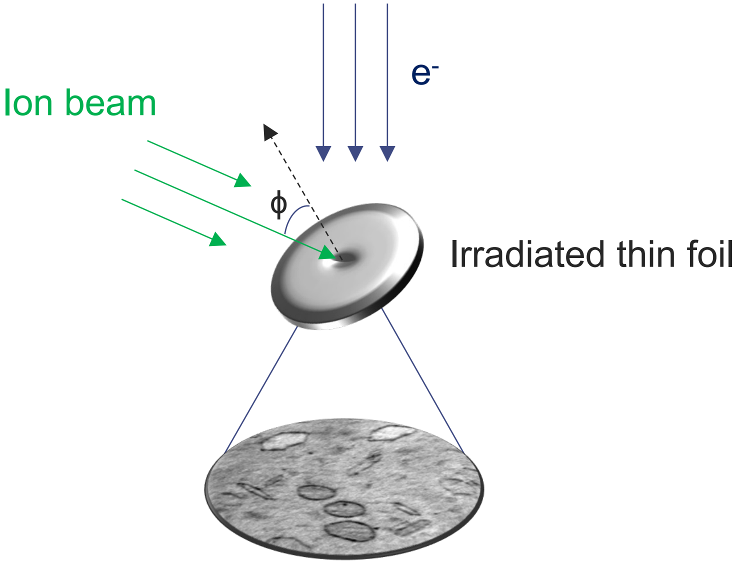


Weak-beam dark field (WBDF)

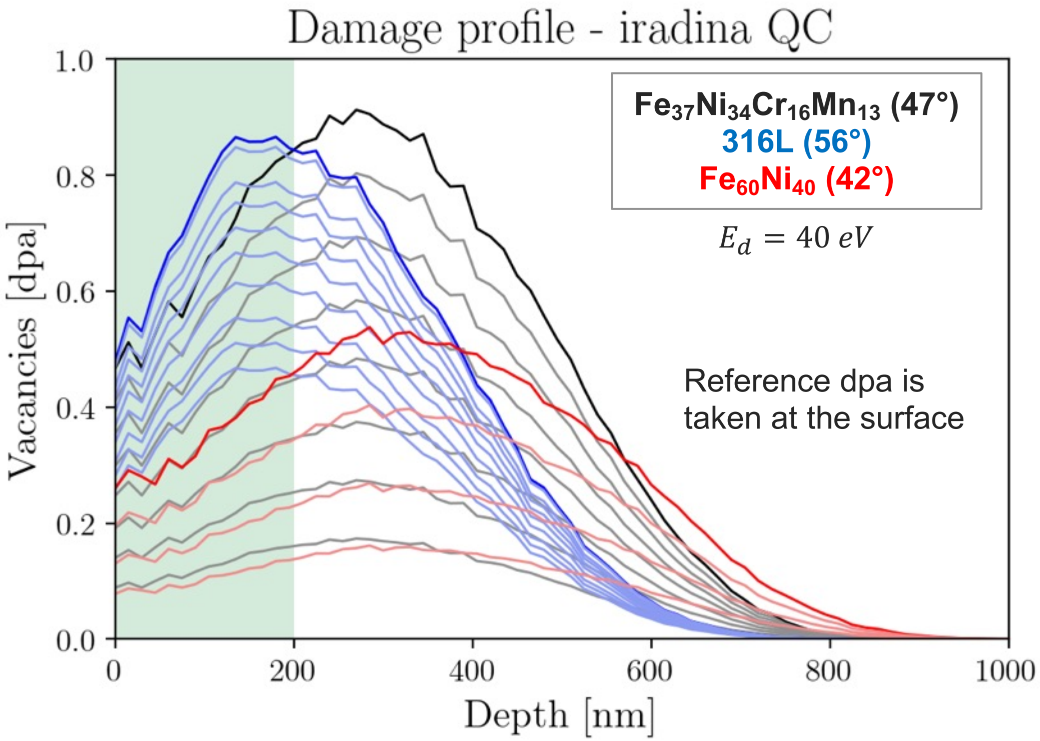
Model in-situ ion irradiations to simulate neutron irradiation



Temperature
550 °C



Material	Ion	G [dpa/s]	Ion beam incidence angle (ϕ)
$Fe_{37}Ni_{34}Cr_{16}Mn_{13}$	Fe^{+} 2 MeV	1,9E-4	47°
316L	Fe^{+} 2 MeV	1,5E-4	56°
$Fe_{60}Ni_{40}$	Ni^{+} 2 MeV	2,2E-4	42°

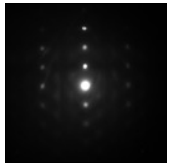


Microstructure evolution under irradiation

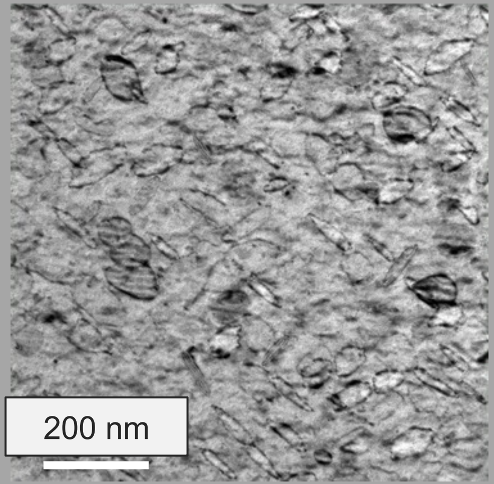
$Fe_{37}Ni_{34}Cr_{16}Mn_{13}$

0.44 dpa

Z = [012]
g = [200]



Loop size: **++**
Density: +
Growth: +



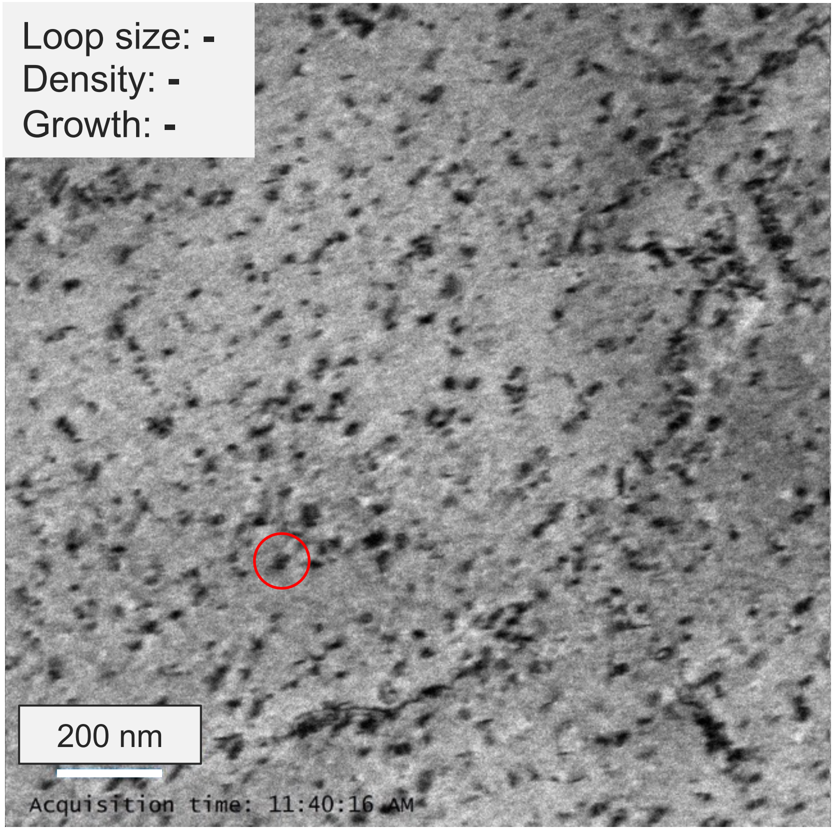
Conventional TEM / BF

316L

0.54 dpa

Z = [341]
g = [1-11]

Loop size: **-**
Density: **-**
Growth: **-**



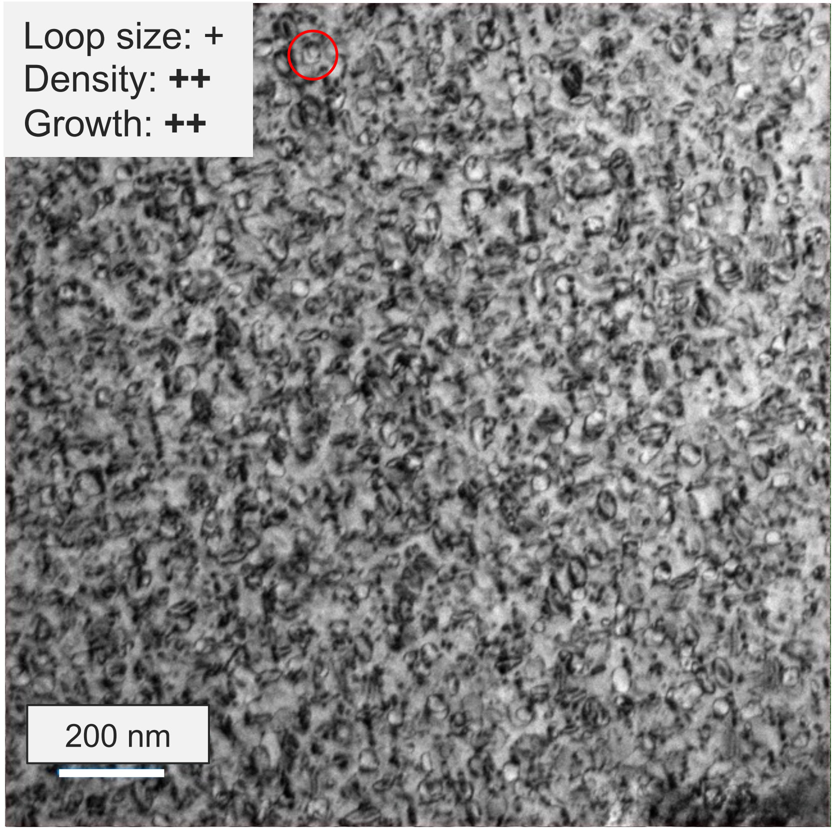
Conventional TEM / BF

$Fe_{60}Ni_{40}$

0.28 dpa

Z = [001]
g = [200]

Loop size: +
Density: **++**
Growth: **++**



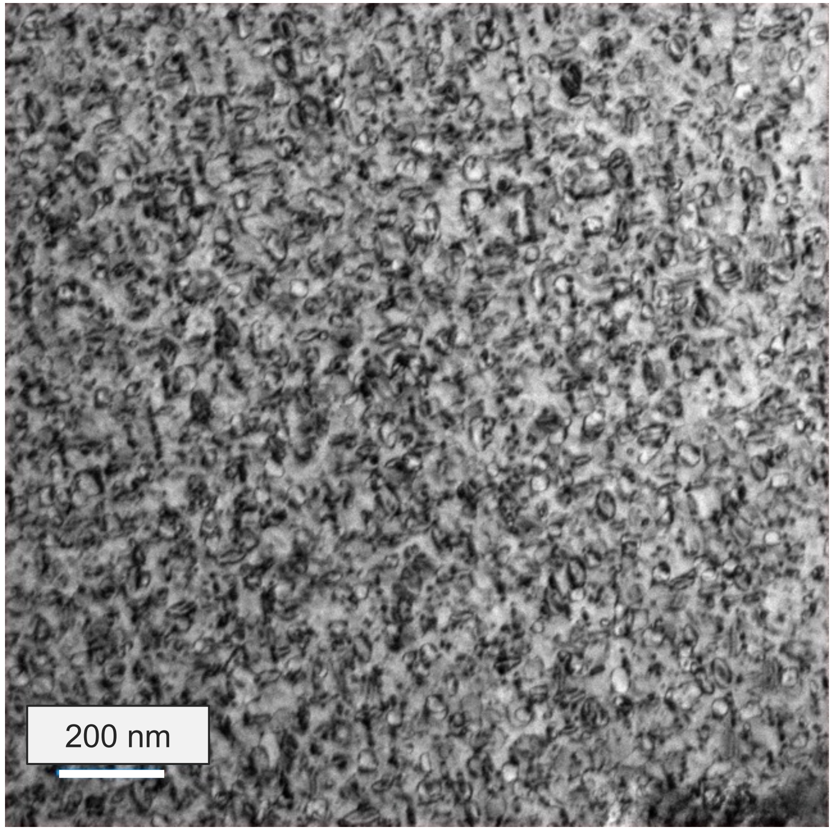
Conventional TEM / BF

Method – Loop size distribution

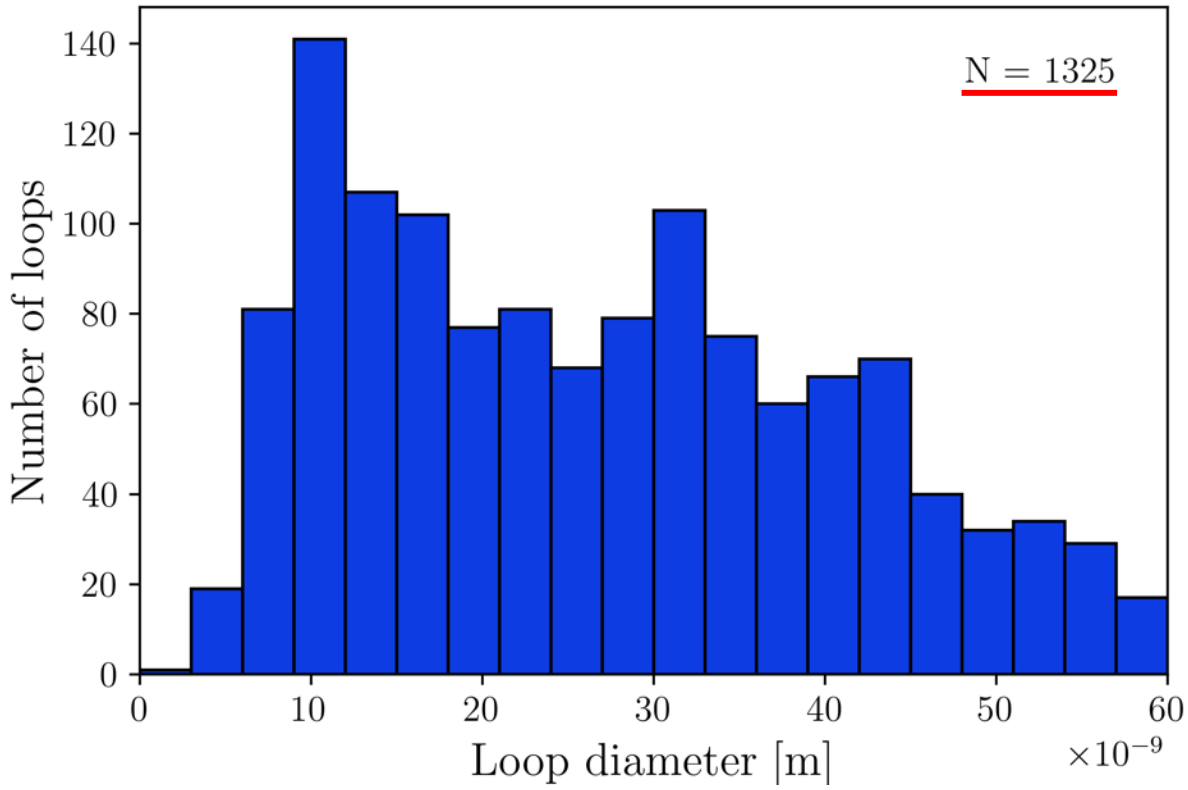
example for $Fe_{60}Ni_{40}$

0.28 dpa

Z = [001]
g = [200]

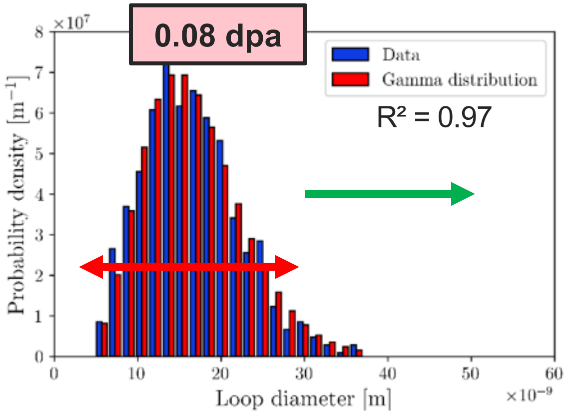


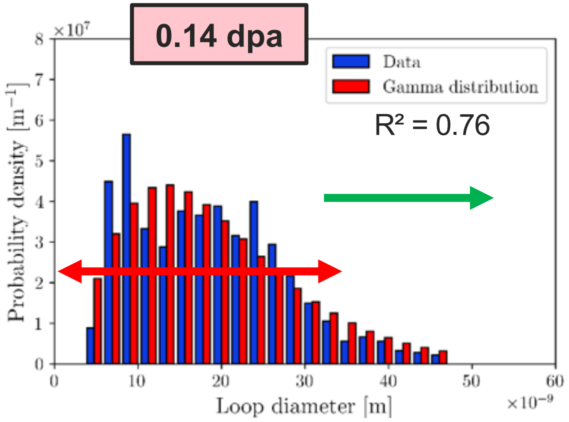
Conventional TEM / BF

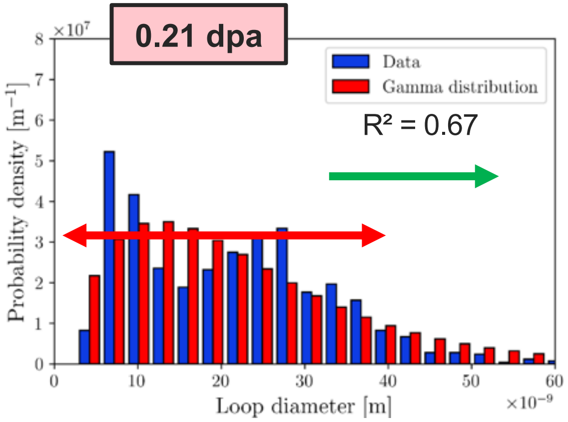


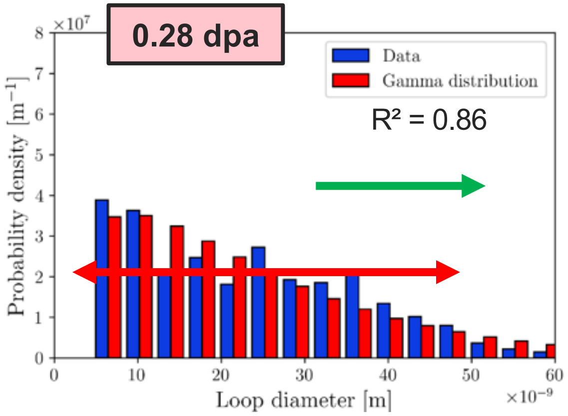
Method – Gamma distribution fit

example for $Fe_{60}Ni_{40}$









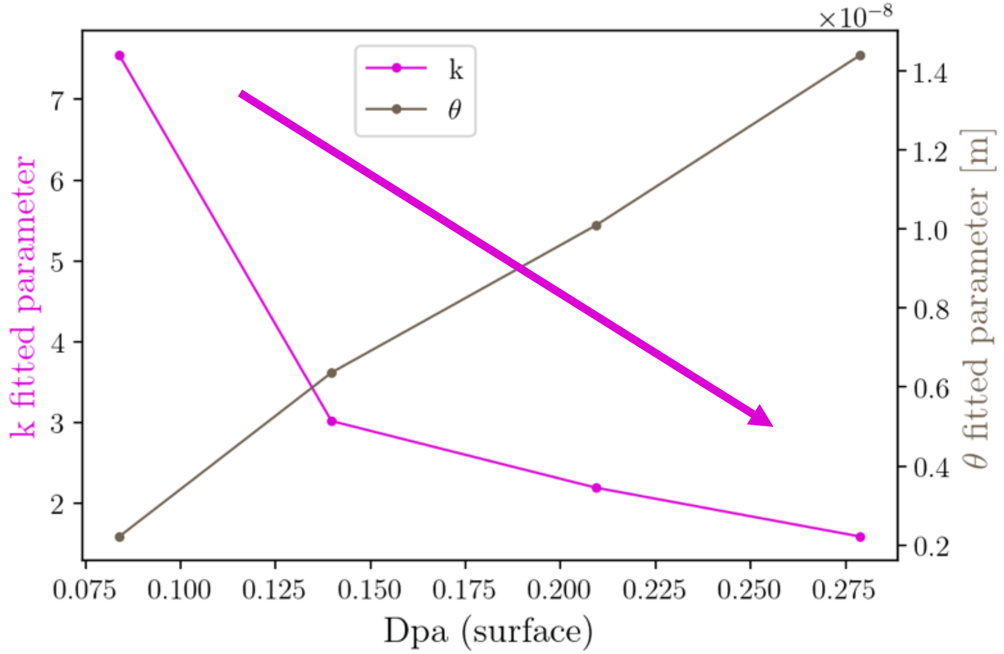
As damage increases… ❖ Distribution shift and spreading ↔

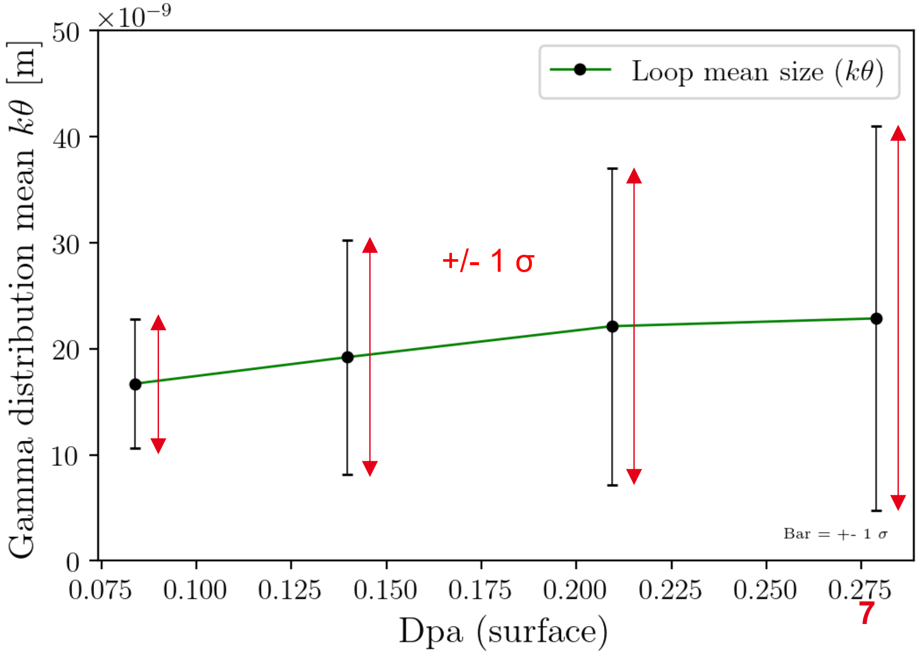
Gamma distribution

$$X \sim \Gamma(\mathbf{k}, \boldsymbol{\theta})$$

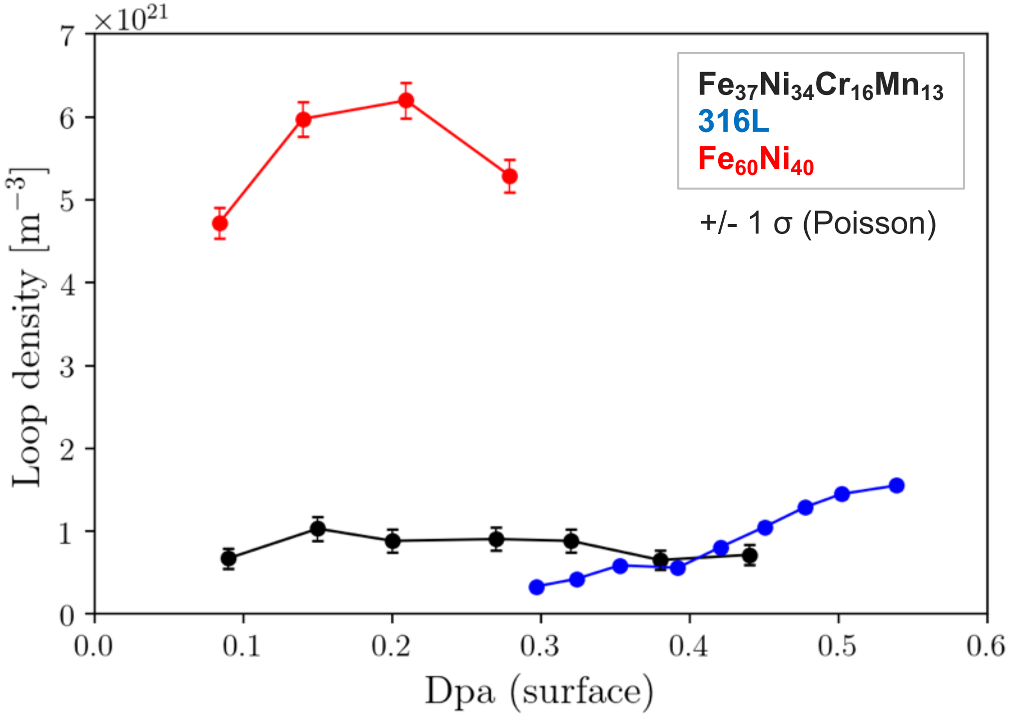
$$f(x; k, \theta) = \frac{x^{k-1} e^{\frac{-x}{\theta}}}{\Gamma(k)\theta^k}$$

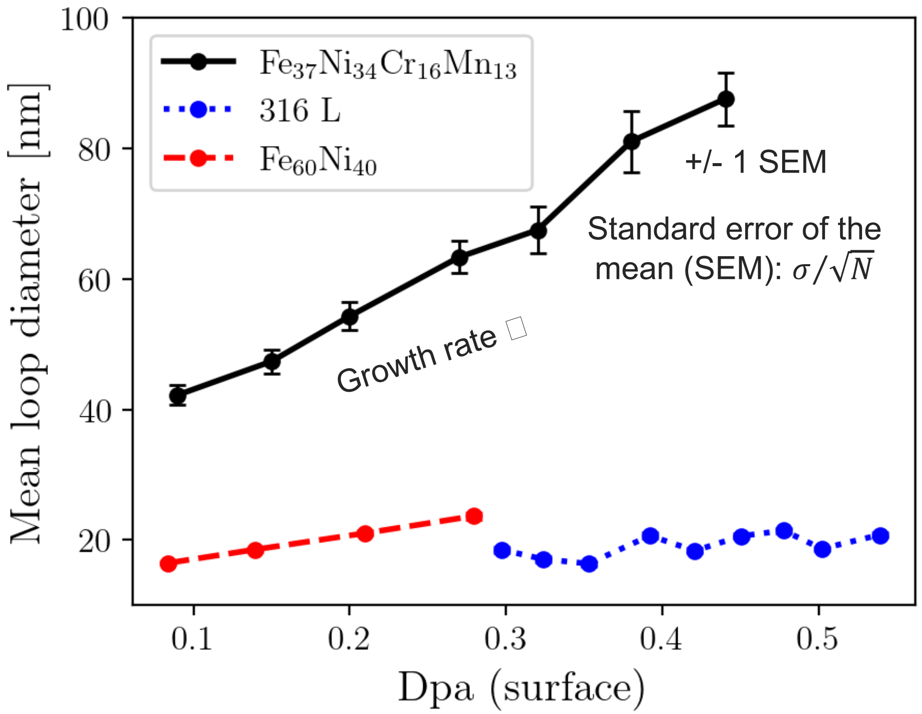
k: shape parameter
θ: scale parameter
$\mu = k\theta$
$\sigma^2 = k\theta^2$





In-situ loop nucleation and growth rates





1 **Loop nucleation rates**

$Fe_{60}Ni_{40}$ > $Fe_{37}Ni_{34}Cr_{16}Mn_{13}$ > 316L

	$Fe_{37}Ni_{34}Cr_{16}Mn_{13}$	316L	$Fe_{60}Ni_{40}$
a [m/dpa]	1,33^E-7	1,38^E-8	3,67^E-8
b [m]	2,82^E-8	1,34^E-8	1,34^E-8
R^2	0,99	0,40	1,00

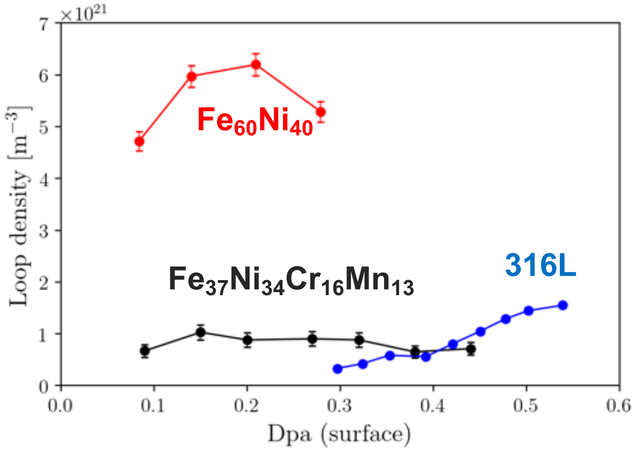
2 **Loop growth rates**

$Fe_{37}Ni_{34}Cr_{16}Mn_{13}$ > $Fe_{60}Ni_{40}$ > 316L

In-situ loop nucleation and growth rates

1 Loop nucleation rates

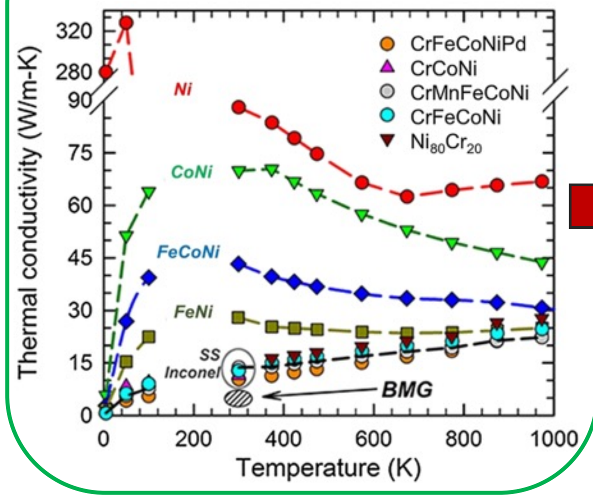
$Fe_{60}Ni_{40}$ > $Fe_{37}Ni_{34}Cr_{16}Mn_{13}$ > 316L



Possible interpretation

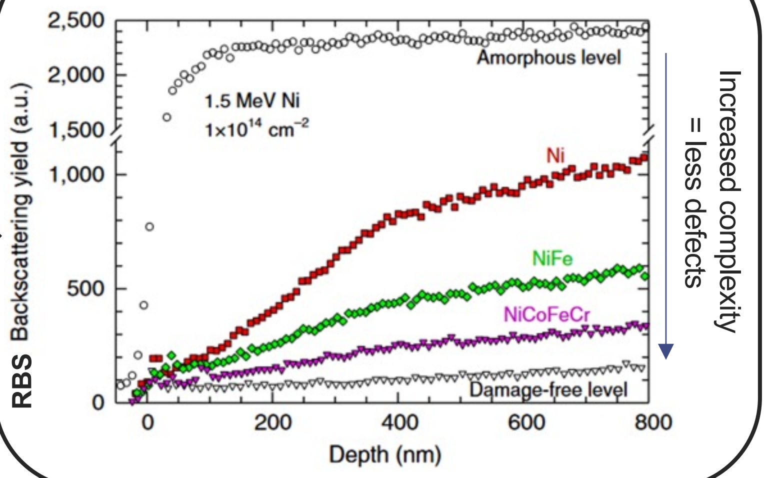
Cluster production within the displacement cascade is reduced in **$Fe_{37}Ni_{34}Cr_{16}Mn_{13}$** and **316L**
A self-recovery tendency due to decreased thermal conductivity?

Slower energy dissipation
⇒ more recombination



Jin et al. (2018)

Reduced damage



Zhang et al, Nature Com (2015)

2 Loop growth rates

$Fe_{37}Ni_{34}Cr_{16}Mn_{13}$ > $Fe_{60}Ni_{40}$ > 316L

Material	Distribution growth rate (nm/dpa)	Individual growth rate (nm/dpa)
$Fe_{37}Ni_{34}Cr_{16}Mn_{13}$	133 ± 7	154 ± 17
316 L	14 ± 6	68 ± 8
$Fe_{60}Ni_{40}$	37 ± 0.3	97 ± 13

Kiritani (1994)

$$\frac{dr}{dt} = \frac{2}{b}(Z_i - Z_l)\sqrt{\frac{GV_{at}D_{ol}}{R_{il}}}\exp(-\frac{E_v^m}{2k_bT})$$

$$\frac{dr}{dt} \propto \exp(-\frac{E_v^m}{2k_bT})$$

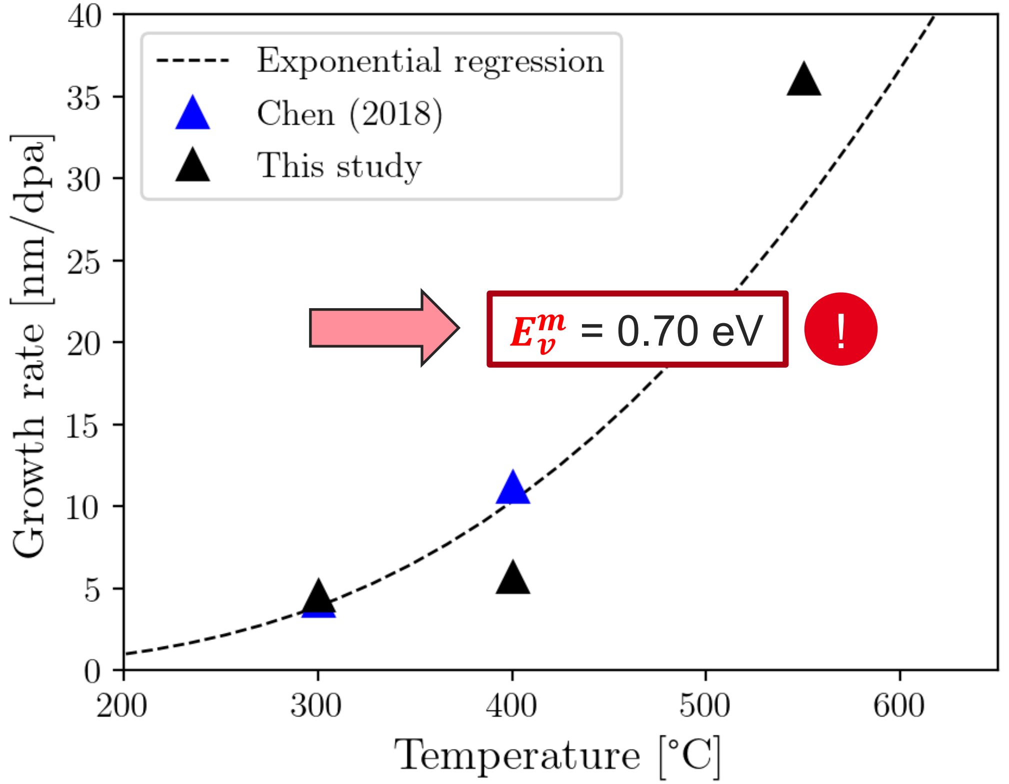
Possible interpretation

Enhanced migration of vacancies in **$Fe_{37}Ni_{34}Cr_{16}Mn_{13}$**
Lower E_v^m ?

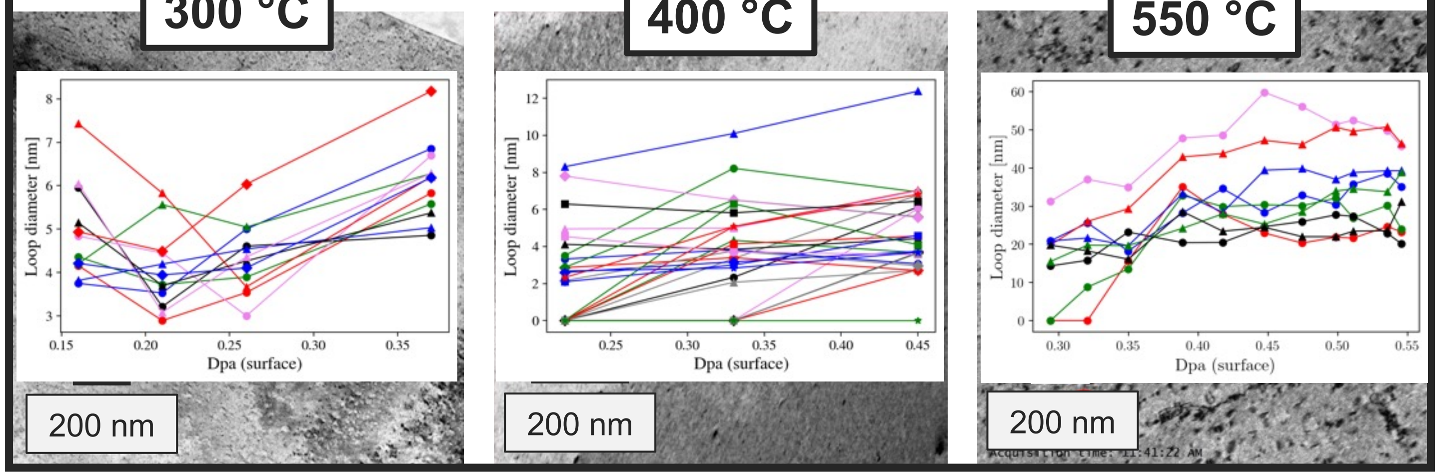
Estimation of E_m^V in 316L – Kiritani model

Kiritani (1994)

$$\frac{dr}{dt} = \frac{2}{b}(Z_i - Z_l)\sqrt{\frac{G V_{at} D_{ol}}{R_{il}}}\exp\left(-\frac{E_v^m}{2k_bT}\right)$$



This study



Chen (2018)

300 °C

400 °C

Contents lists available at ScienceDirect

Acta Materialia

journal homepage: www.elsevier.com/locate/actamat

Full length article

Investigation of interactions between defect clusters in stainless steels by in situ irradiation at elevated temperatures

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[b] Nagaoka University of Technology, Department of Nuclear System Safety Engineering, 1603-1 Kamitomioka, Nagaoka-shi, Niigata, 940-2188, Japan
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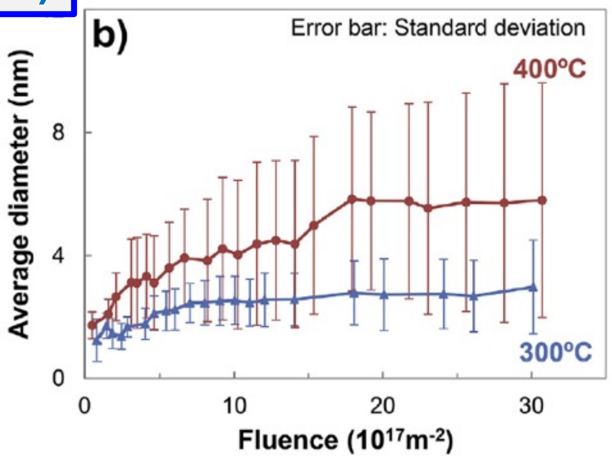
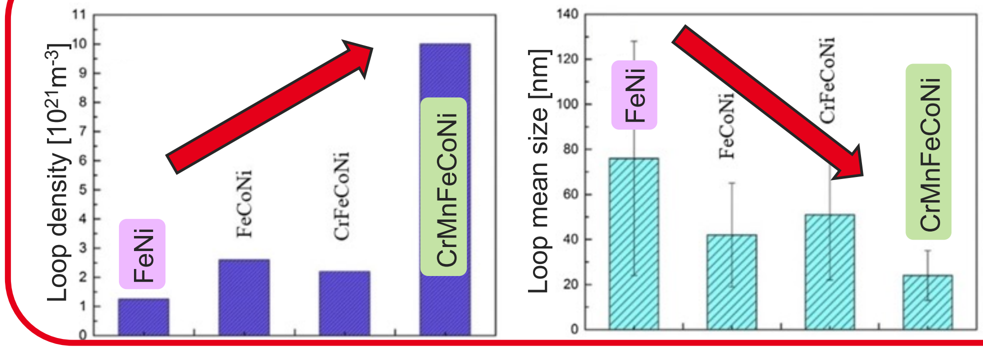


Fig. 4. (b) Average size of defect clusters as function of irradiation fluence.

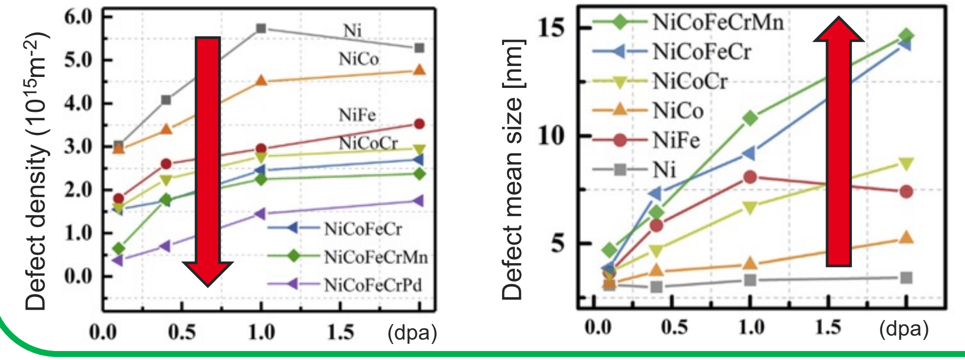
Conclusions

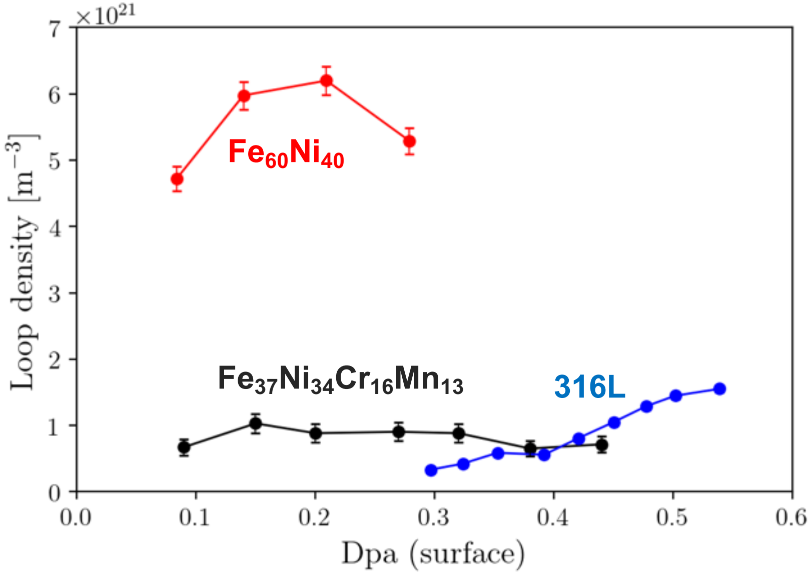
- Quantitative and statistical study of **$Fe_{37}Ni_{34}Cr_{16}Mn_{13}$**, **$Fe_{60}Ni_{40}$** and **316L**, in-situ irradiated using Ni/Fe 2 MeV ions at 550°C
- Significant differences in **damage kinetics** between **$Fe_{37}Ni_{34}Cr_{16}Mn_{13}$**, **$Fe_{60}Ni_{40}$** and **316L**:
  - Loop density: **$Fe_{60}Ni_{40}$** > **$Fe_{37}Ni_{34}Cr_{16}Mn_{13}$** > **316L**
    - *One interpretation: Thermal conductivity affecting the cluster formation within the cascade?*
  - Loop growth rate: **$Fe_{37}Ni_{34}Cr_{16}Mn_{13}$** > **$Fe_{60}Ni_{40}$** > **316L**
    - *One interpretation: Faster migration of vacancies?*

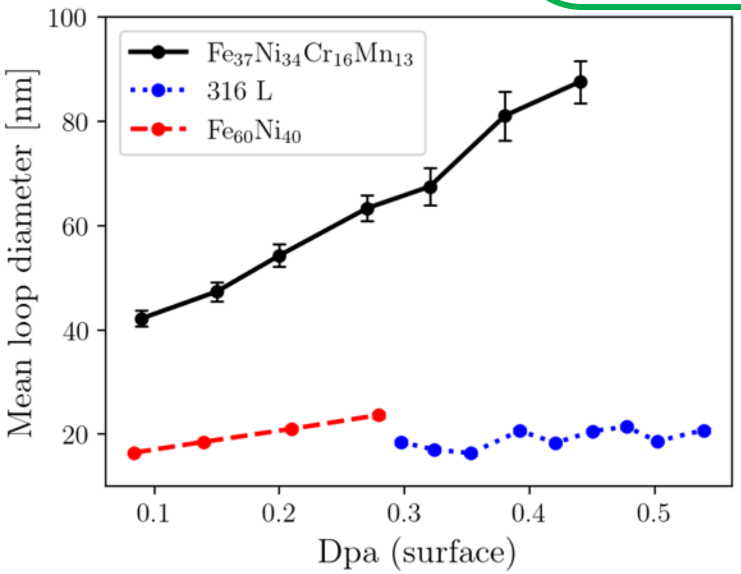
Lu et al, Acta mat (2017)



Shi et al, JNM (2018)







Thank you for your attention!

Special thanks to JANNuS Orsay team