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In Situ Transmission Electron Microscopy Investigation of Microstructural Evolution in Complex F-M Steels under Ion Irradiation

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Outline

Motivation

- Lack of irradiation data for F-M steels that are amongst the candidates for in-core applications in Sodium-Cooled Fast Reactor.

Method

- In-situ investigation of microstructure evolution of NF616 and HCM12A under Kr irradiation at various temperatures.

Results

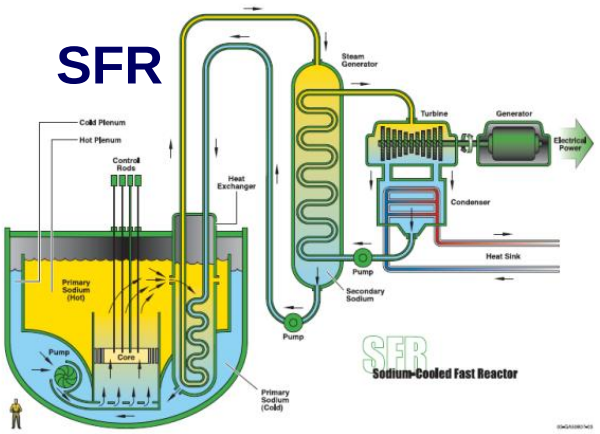
- As-fabricated microstructure of NF616 and HCM12A
- In-situ heavy ion irradiations of NF616 and HCM12A between 50K and 673K.
- Transmission Electron Microscope (TEM) images recorded at certain dose points
- Quantitative analysis: Defect density, average defect size and defect size distributions.

Conclusions

Introduction

Gen-IV Reactors (Sodium Fast Reactor)

- Waste minimization
- High efficiency
- Improved economics



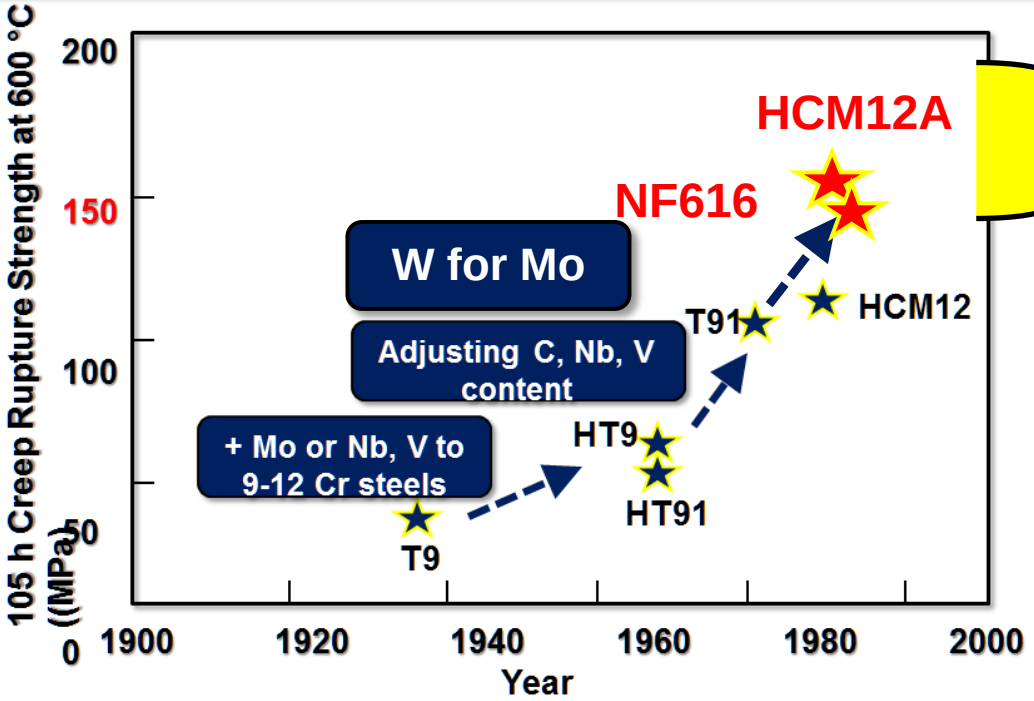
Material Challenges (Fuel Cladding and Internals)

- High operating temperatures
- Intense radiation fields
- Higher doses

(A Technology Roadmap for Generation IV Nuclear Energy Systems, 2002)

F-M alloys

- High swelling resistance
- Good microstructural stability
- Good thermal properties



(Data from R. Viswanathan , 2001)

Third Generation F-M Alloys

- NF616 and HCM12A
- Primary candidates for SFR
- **Lack of irradiation data**

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Objective

- Investigate the microstructure evolution in NF616 and HCM12A alloys under heavy ion irradiation at various temperatures with Transmission Electron Microscope (TEM).

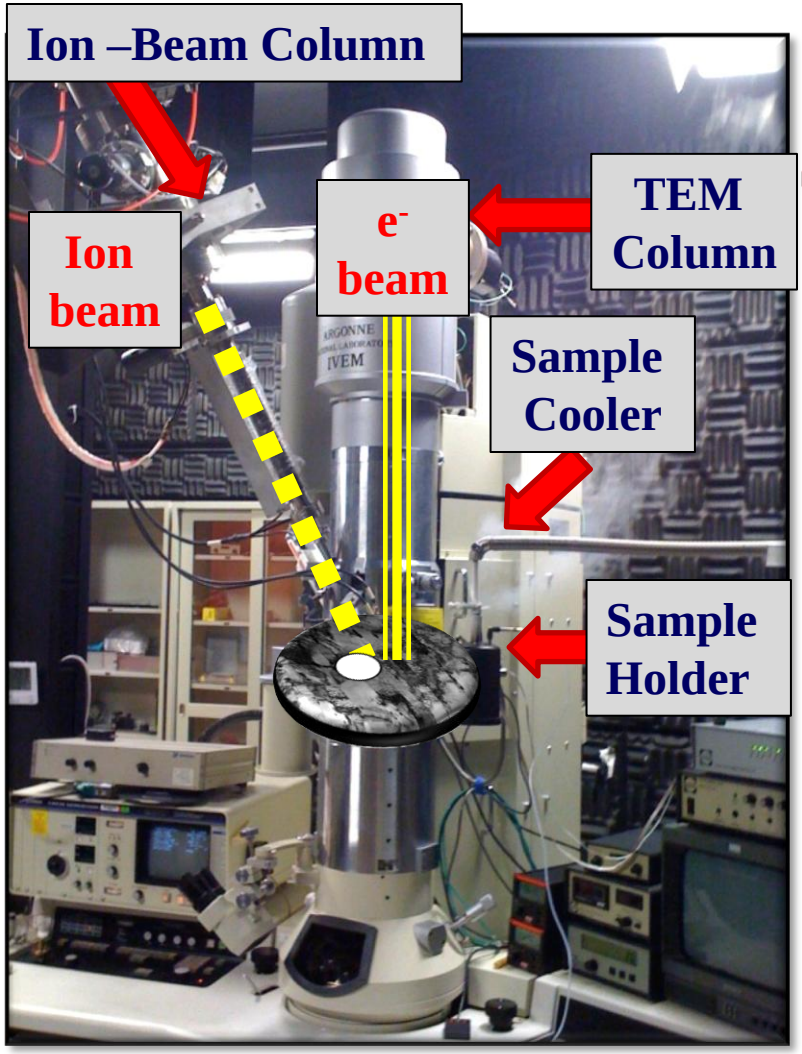
- Observe the damage evolution as it develops:

  - Stability of starting microstructure
  - Irradiation induced defect microstructure:

    Defect cluster concentration and size distribution as $f(Dose, T)$
  - Interaction of defect clusters with each other and with pre-existing microstructure.

In-Situ Ion Irradiations of NF616 and HCM12A at Argonne National Laboratory

IVEM at ANL



Experimental Matrix

Ion: 1 MeV Kr^{++}

T: 50K, 473K, 573K and 673K

Dose: 0 to ~9 dpa with ~0.8 dpa intervals to perform microscopy

SRIM

1 dpa ≈ 3.48x10^{14} ions/cm^2

Dpa/s ≈ 1.44 x10^{-3}

2.44 disp/ion-A

Input:

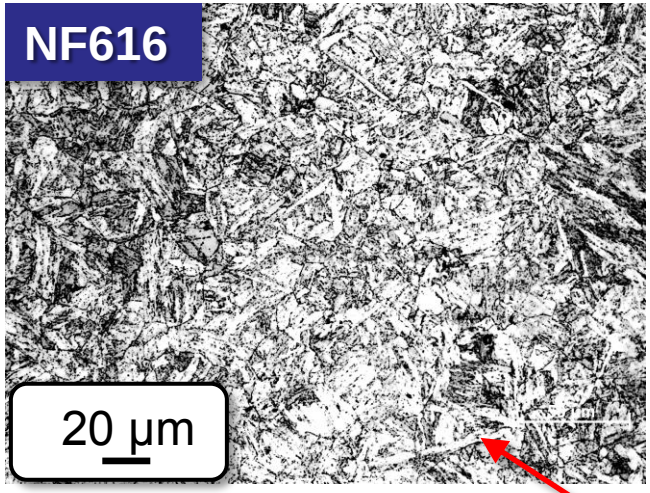
1 MeV Kr^{++} ions

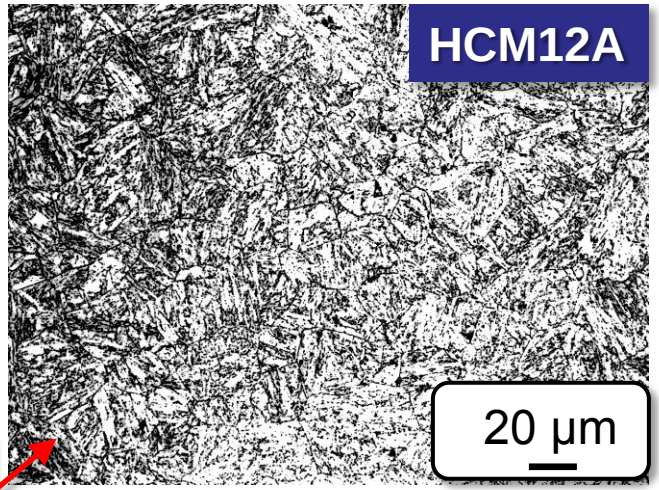
E_d=40 eV for Fe and Cr and 24 eV for C

Angle of incidence=15°

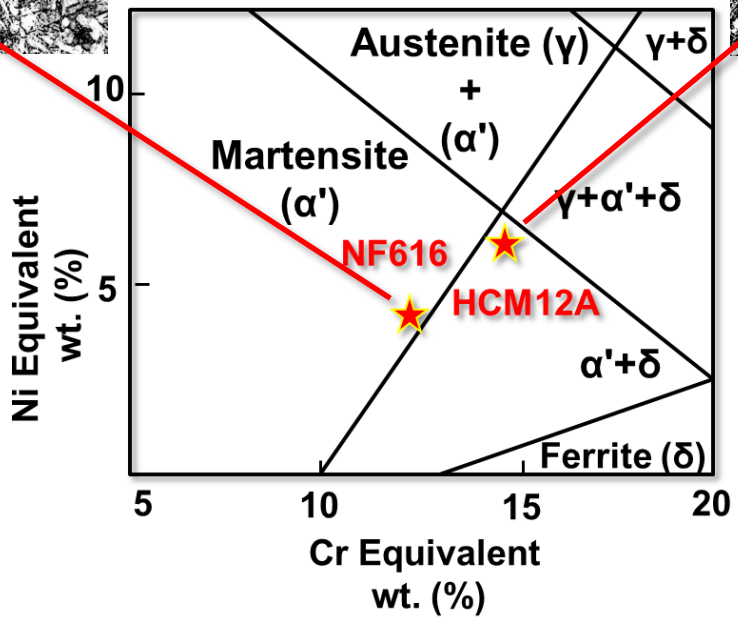
Flux =0.5x10^{12} cm^{-2}s^{-1}

As- fabricated Microstructure





- Tempered martensite



NF616 (P-92)

Heat treatment:

1. 1343 K for 2h, AC
2. 1043 K for 2h, AC

HCM12A (P-122)

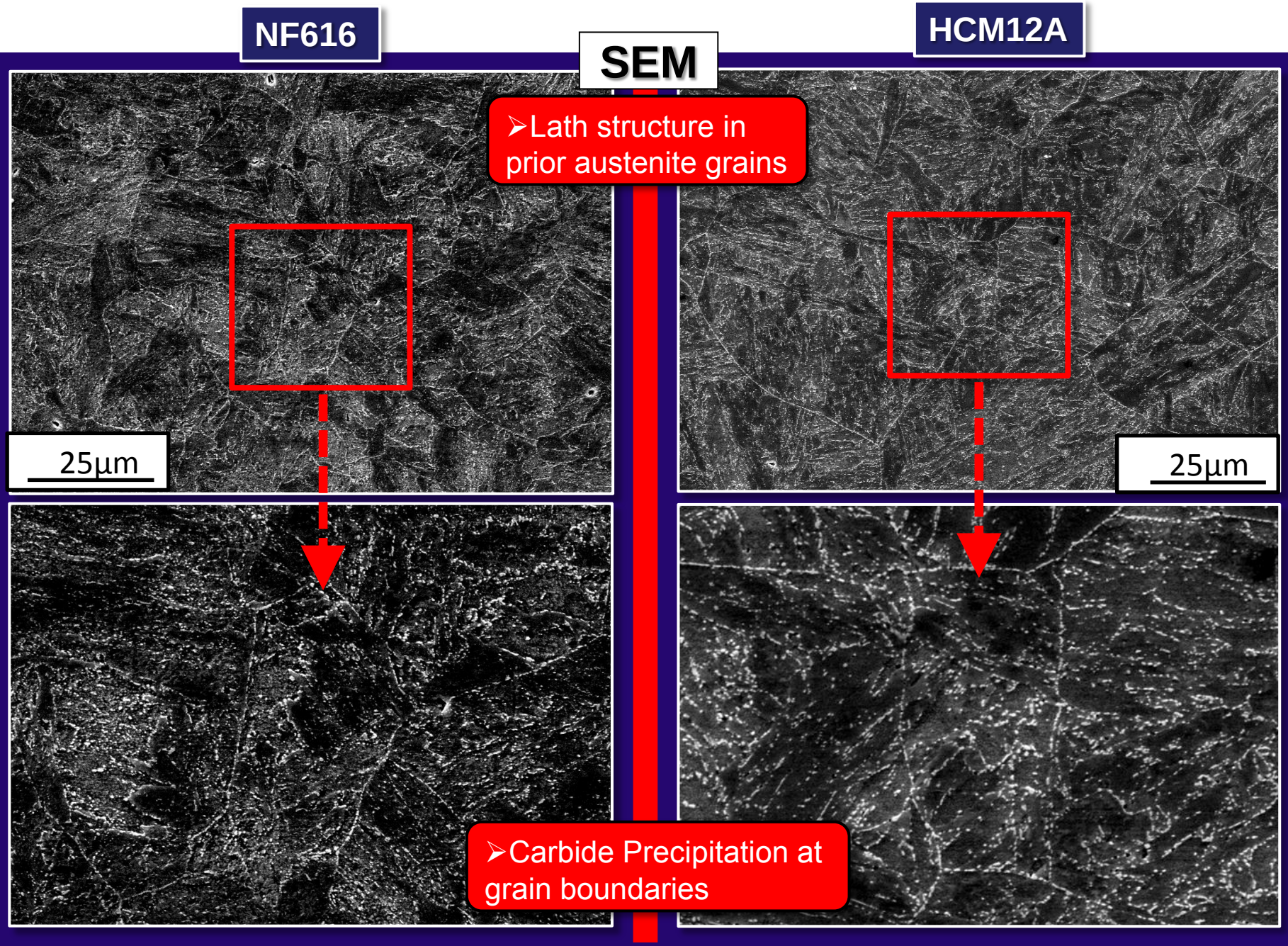
Heat treatment:

1. 1323 K for 1h, AC
2. 1043 K for 45 min, AC

Alloy	C	Cr	Fe	Si	P	S	V	N	Mn	Ni	Cu	Nb	Mo	W	Al
HCM12A	0.1	10.8	Bal.	0.3	0.02	0.002	0.2	0.1	0.6	0.4	1.0	0.05	0.3	1.9	0.001
NF616	0.1	8.8	Bal.	0.1	0.01	0.003	0.2	-	0.5	0.2	-	0.06	0.5	1.9	0.005

Alloys provided by Japan Atomic Energy Agency

Microstructure

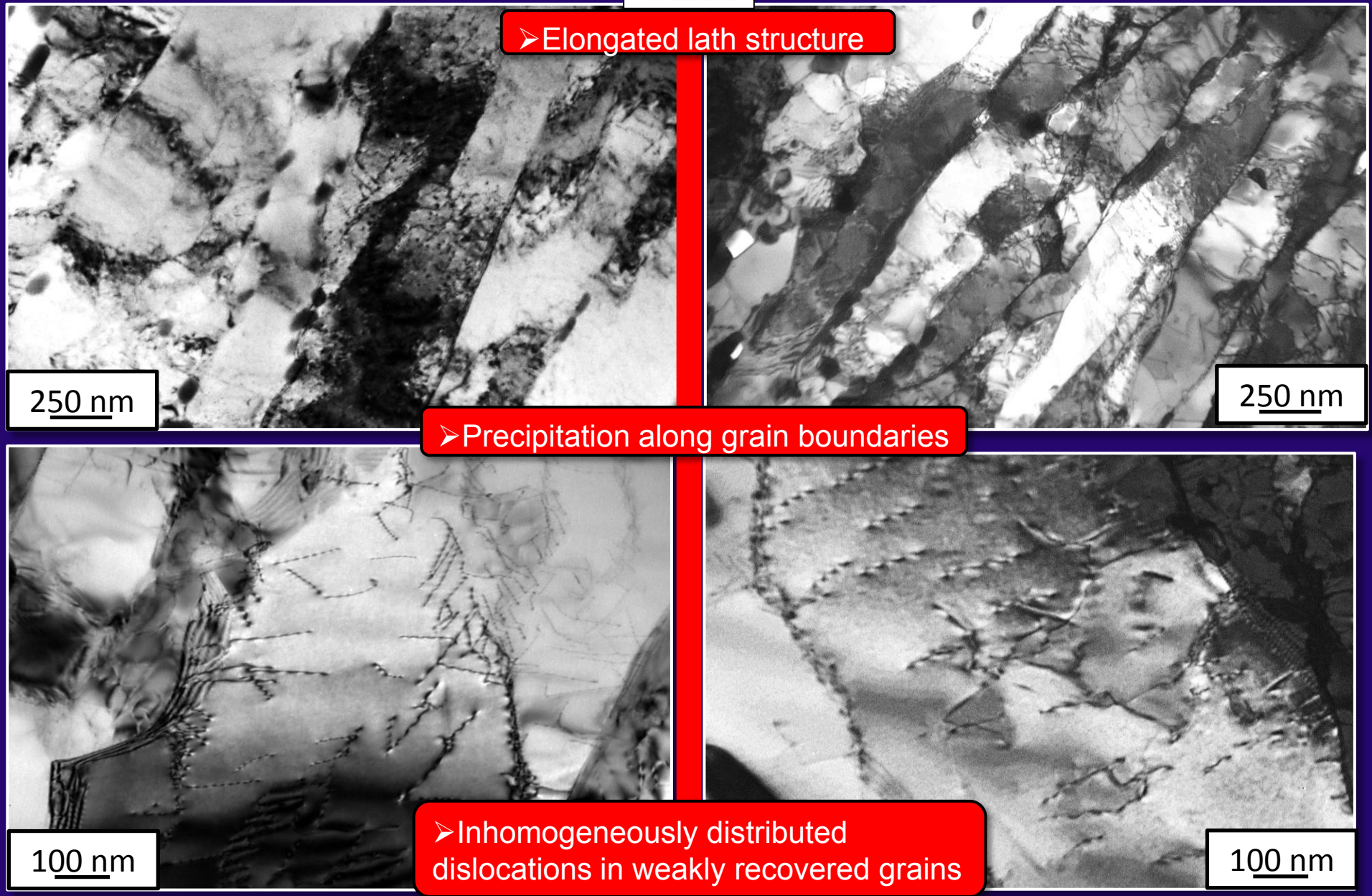


Microstructure

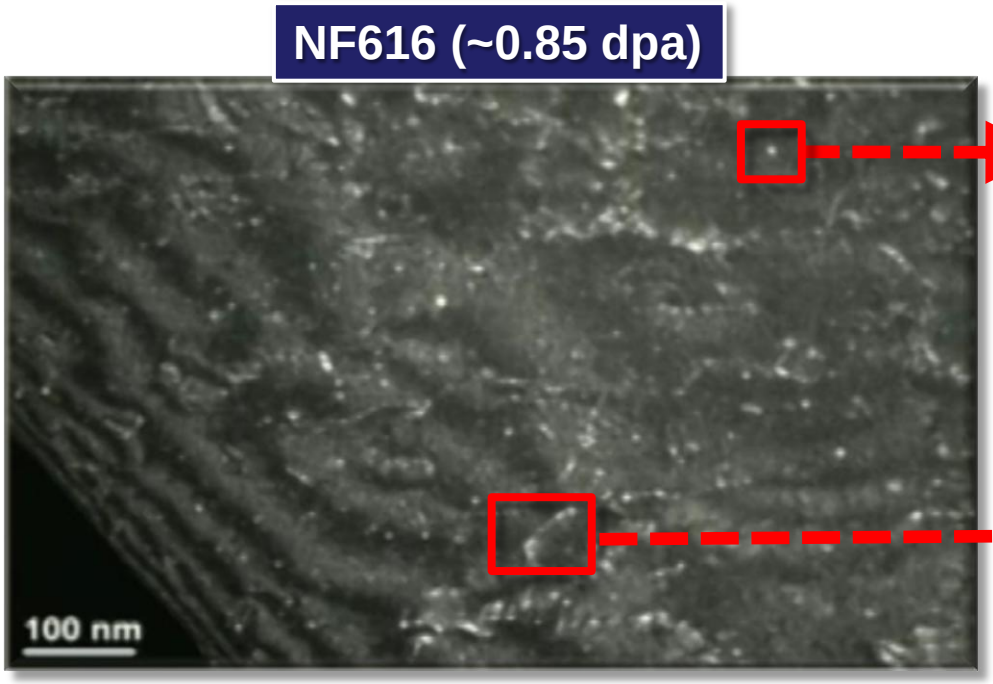
NF616

TEM

HCM12A



General Characteristics of Microstructure Evolution in NF616 and HCM12A ≤ 573 K

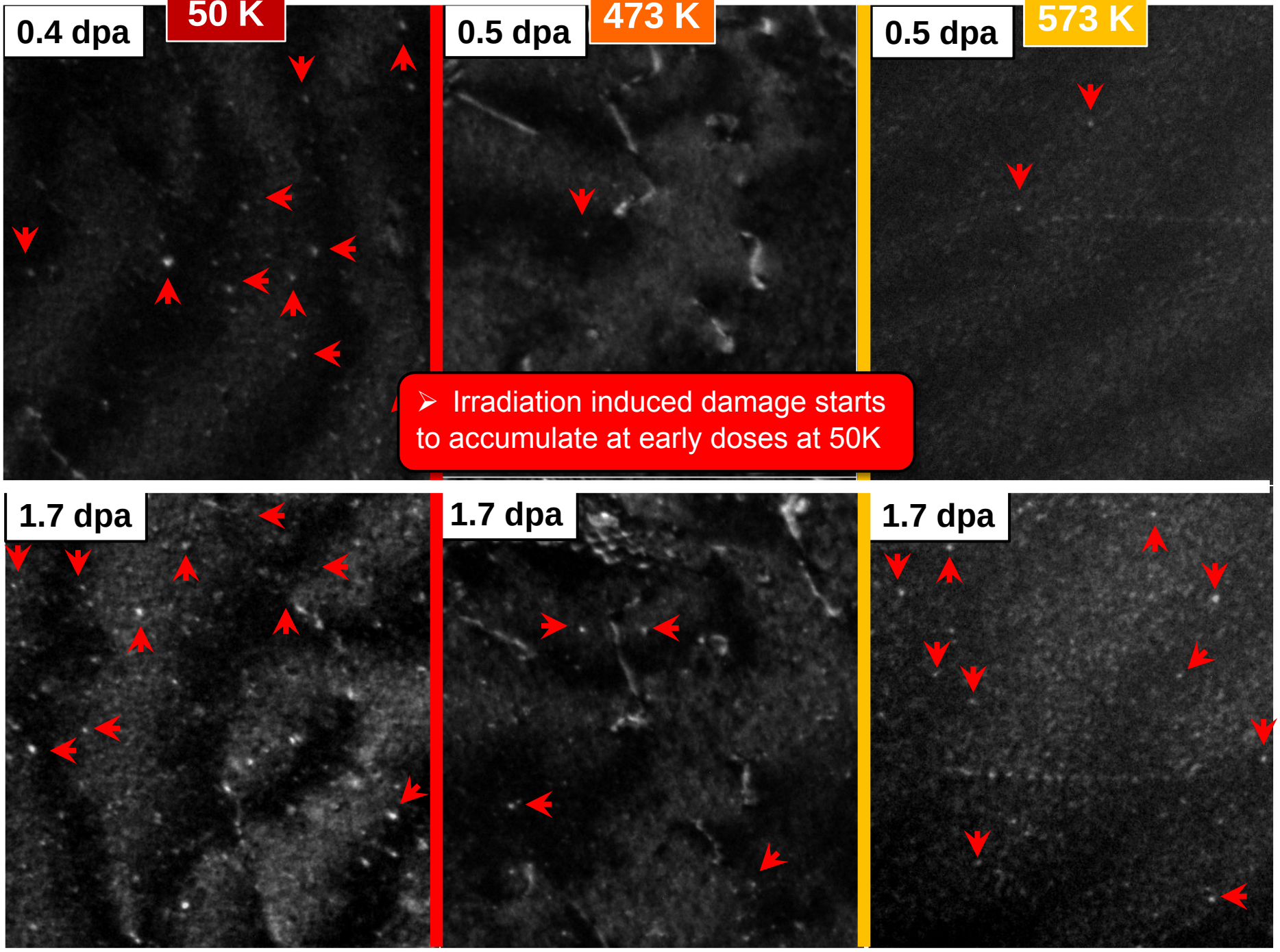


Irradiation-induced Defect Clusters

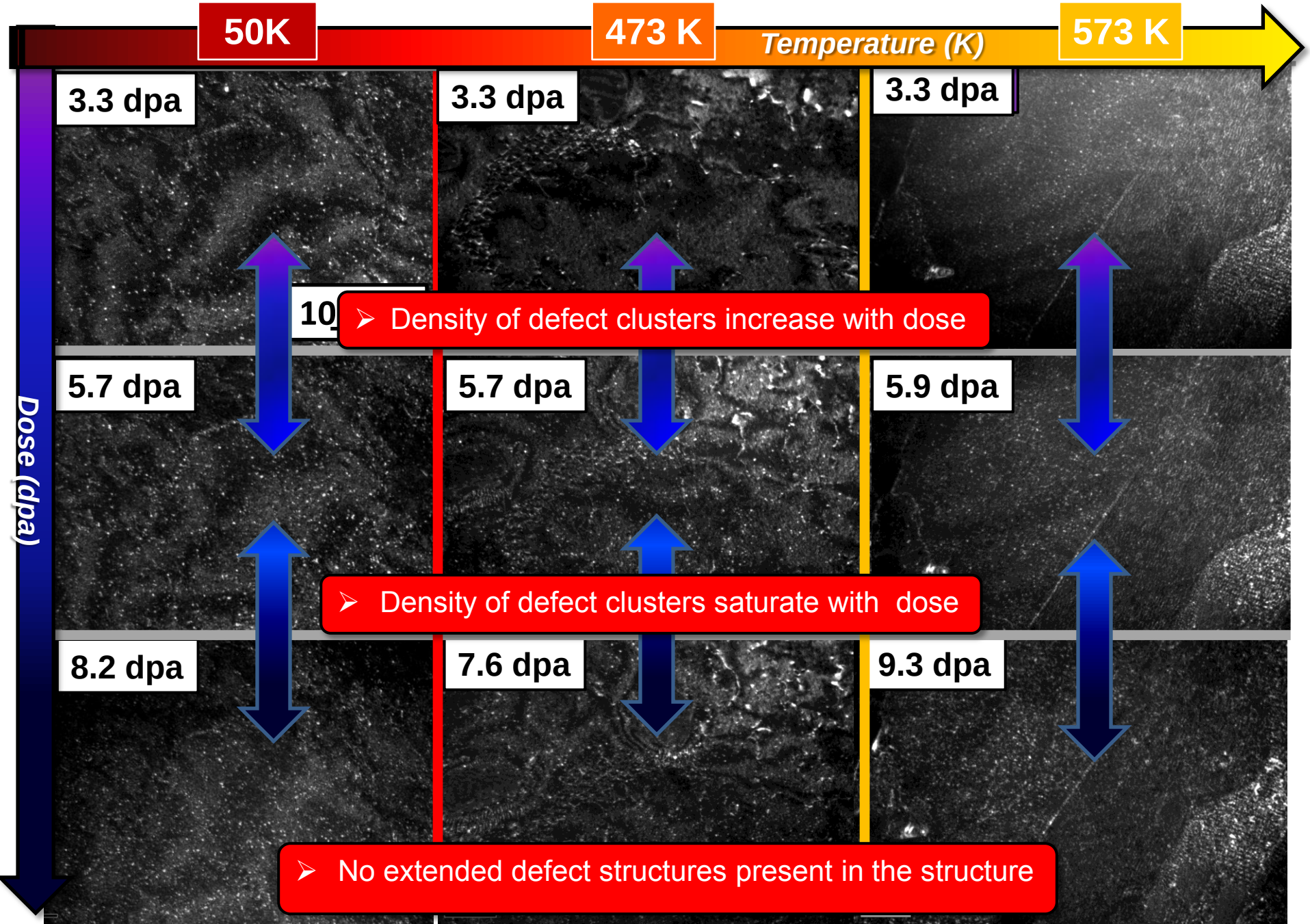
Pre-existing dislocations

Video:
- 373K
- 0.85 →1 dpa
- 5X faster

- Defects clusters (white dots) form and gradually increase in density with slightly different kinetics (saturation of defect density reached at ~6 dpa).
- No defect cluster growth observed.
- Defect clusters flicker even at 50 K under the ion beam → Shock wave due to cascade impact causes limited movement of clusters.

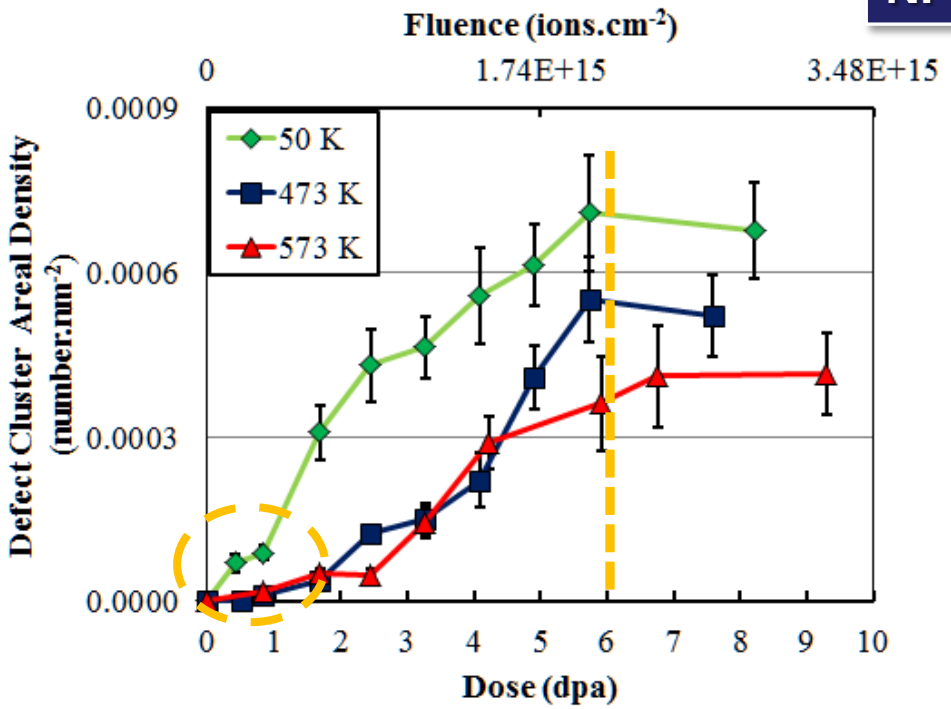

0.4 dpa
50 K
0.5 dpa
473 K
0.5 dpa
573 K
➢ Irradiation induced damage starts to accumulate at early doses at 50K
1.7 dpa
1.7 dpa
1.7 dpa

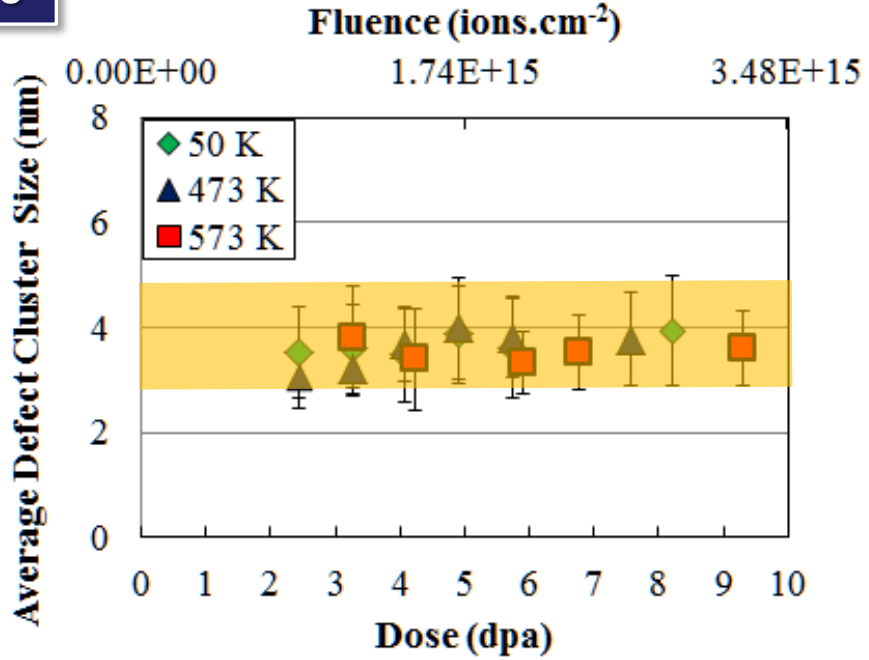
In-Situ Irradiation of NF616 ≤ 573 K (High Doses)



Defect Cluster Density and Average Cluster Size Vs. Dose and Temperature

NF616

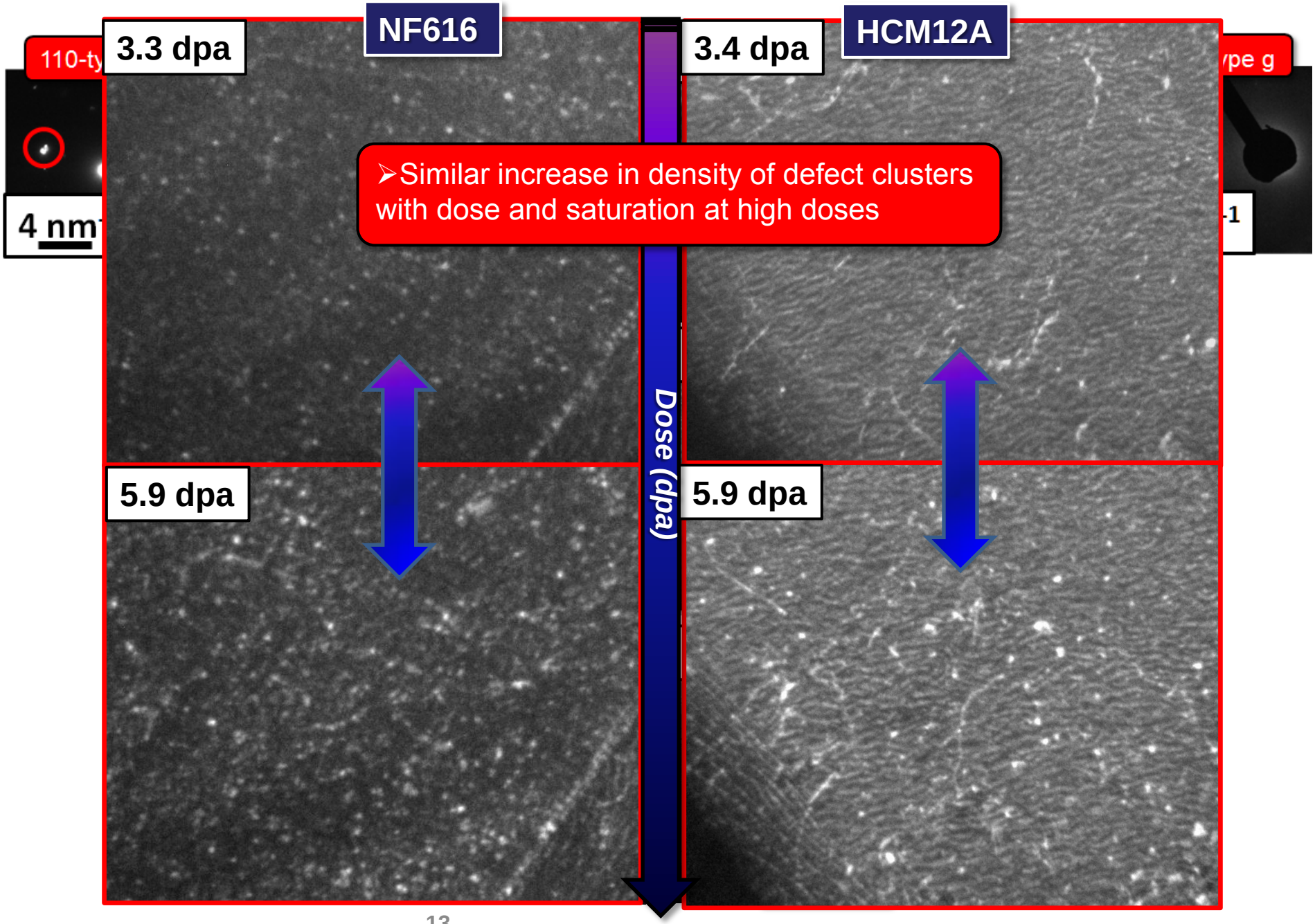




- Defect cluster density increases and tends to saturate at ~ 6dpa
- Saturation level of defect density decreases as temperature increases
- Defect accumulation starts at lower doses at 50K

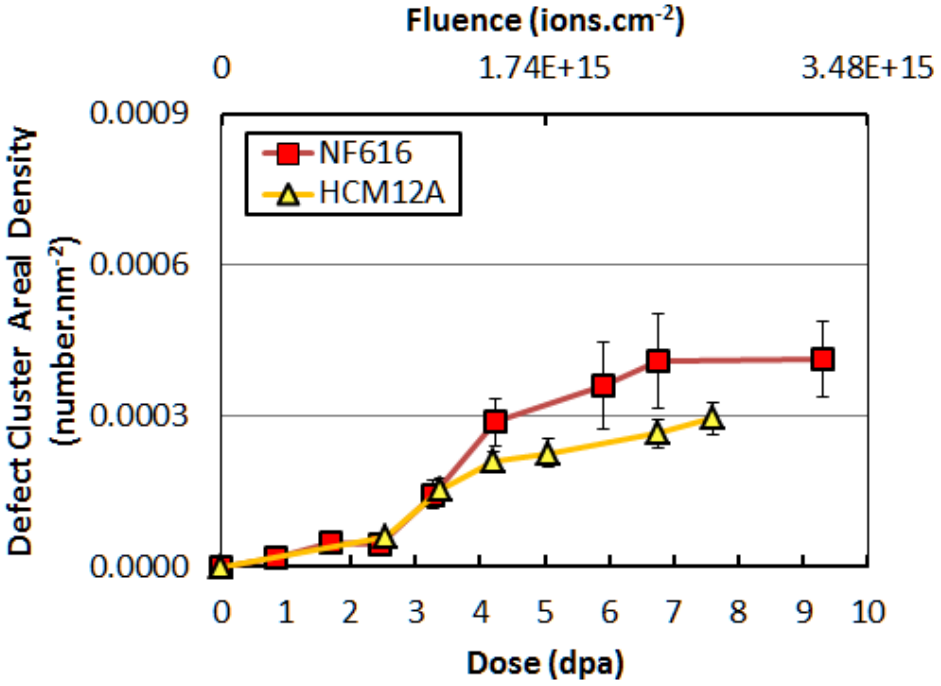
- Average size between 3 and 4 nm
- Average defect size does not depend on temperature and dose below 573K.

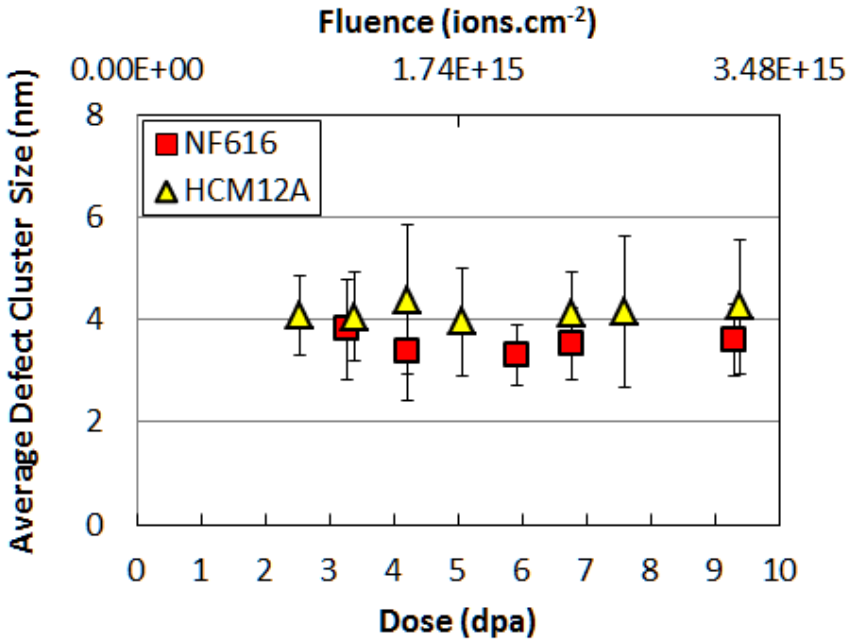
In-Situ Irradiation of HCM12A and NF616 at 573 K



Defect Cluster Density and Average Cluster Size Vs. Dose and Temperature

NF616 and HCM12A at 573 K

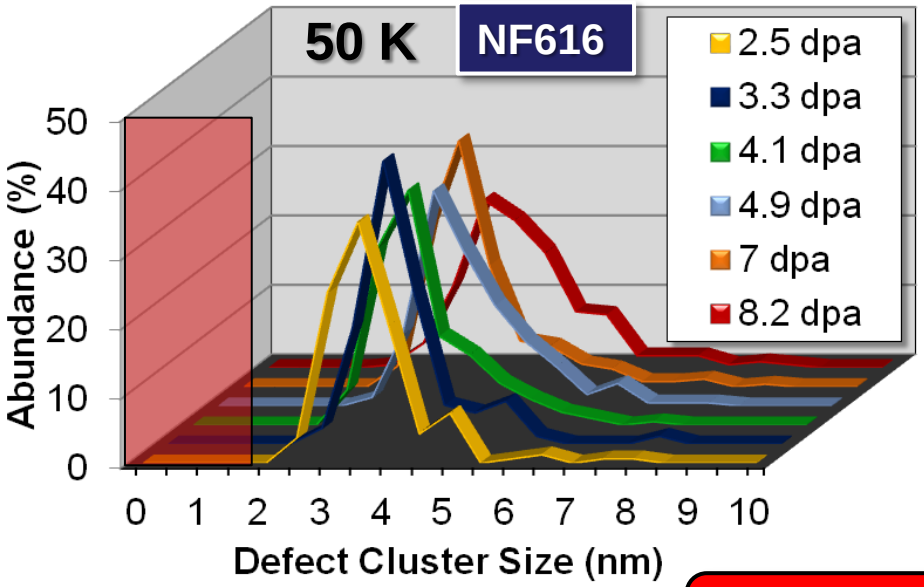


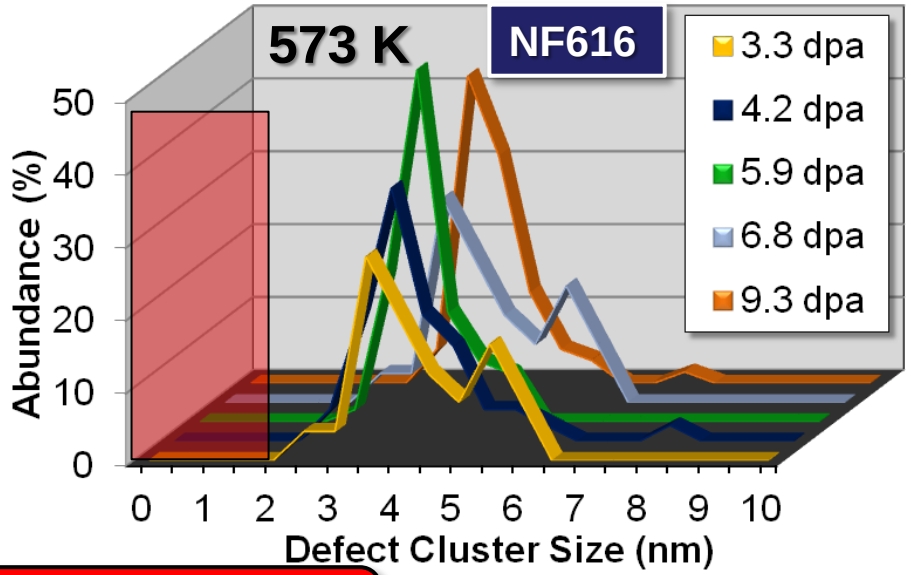


- Defect density values of HCM12A and NF616 are in good agreement at 573 K

- Average defect sizes are similar in NF616 and HCM12A at 573 K and do not depend on dose

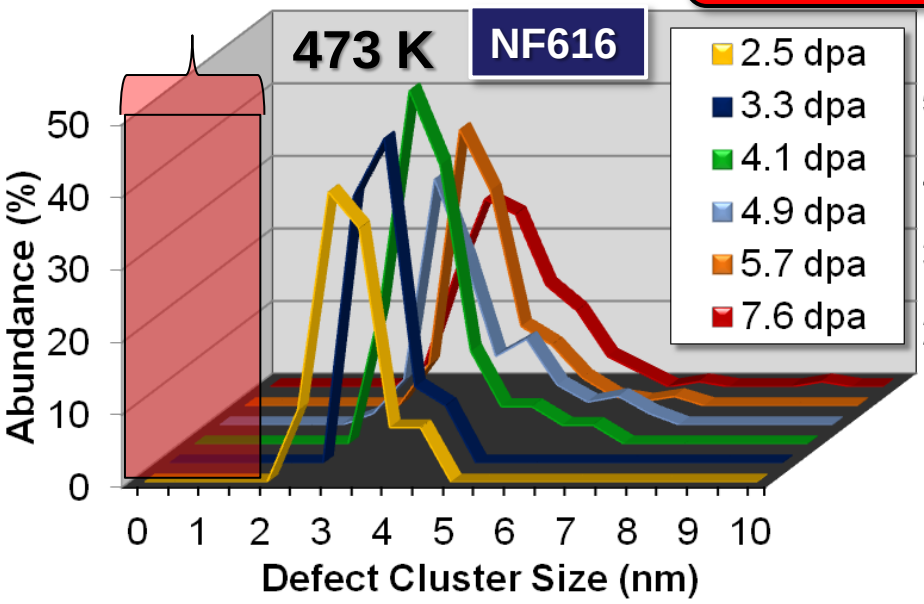
Defect Cluster Size Distributions

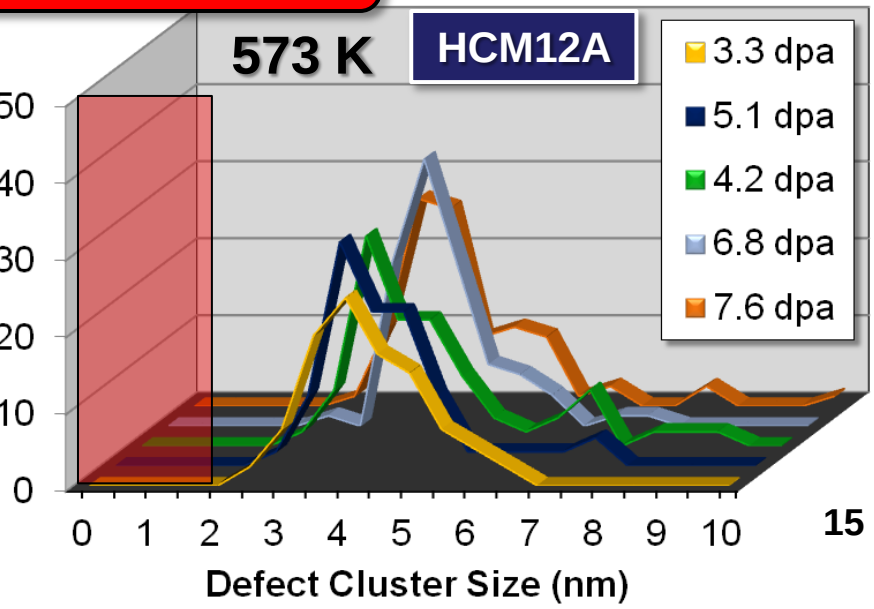




<resolution limit of TEM

- No significant shift in size distributions with dose

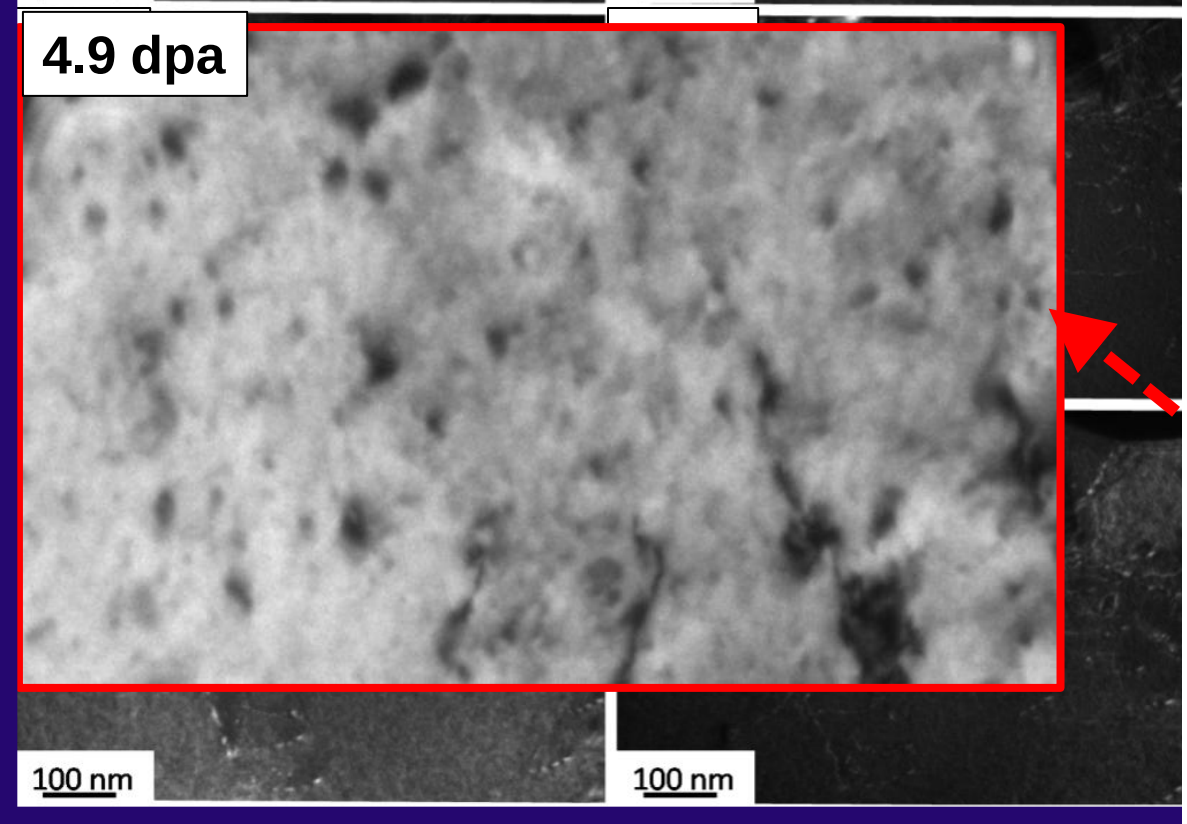




In-situ Irradiation of NF616 at 673 K

Area 1

- Defects start to appear in between 2.5 and 4.1 dpa
- Large defect clusters were observed above ~4 dpa
- Defect clusters are more mobile at 673 K

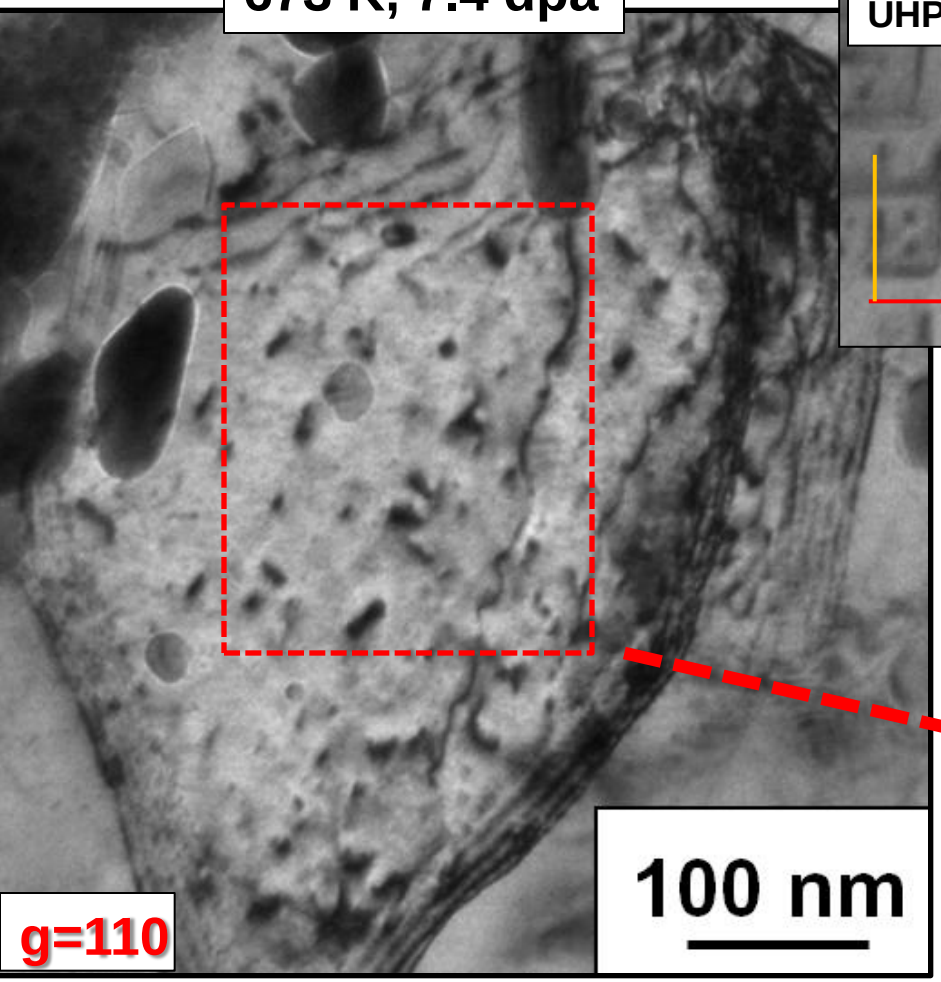


- Two Different areas were followed at 673 K
- Area #1 includes a grain with a high pre-existing dislocation density
- No defect accumulation observed up to 7.4 dpa

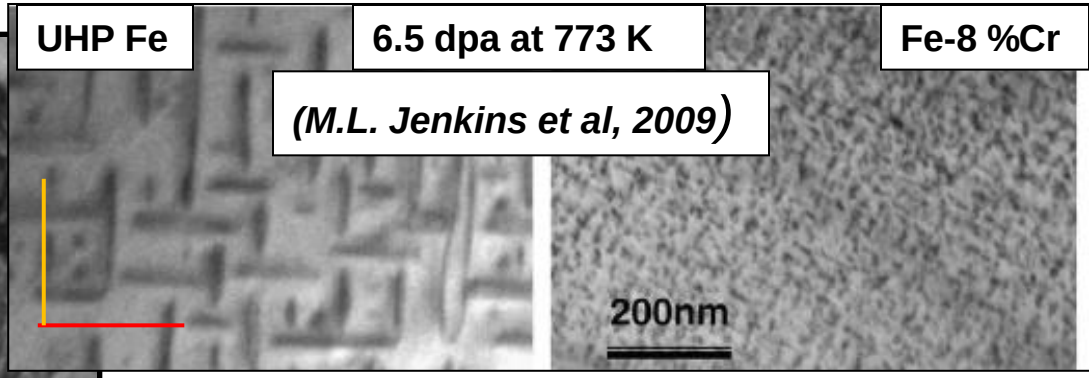
High Dose Defect Structure in NF616 at 673 K

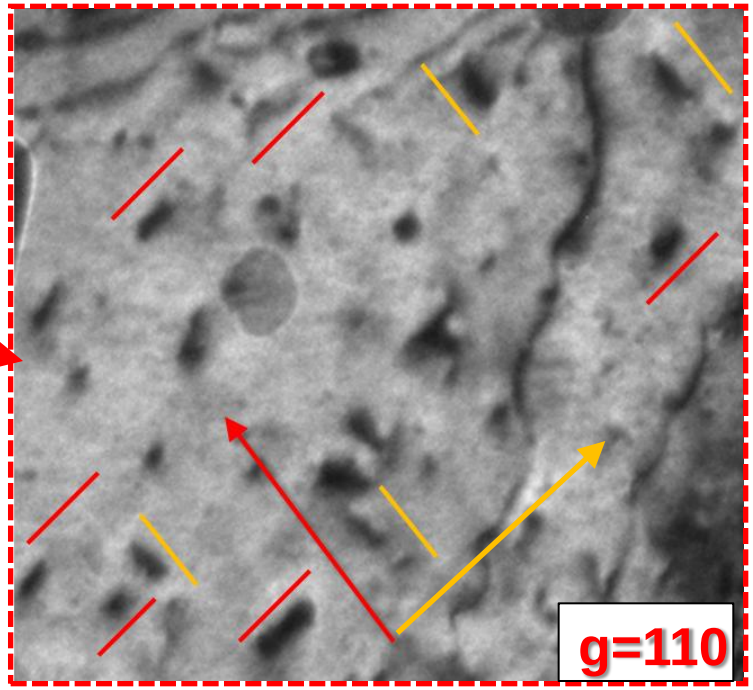
Area 3

673 K, 7.4 dpa



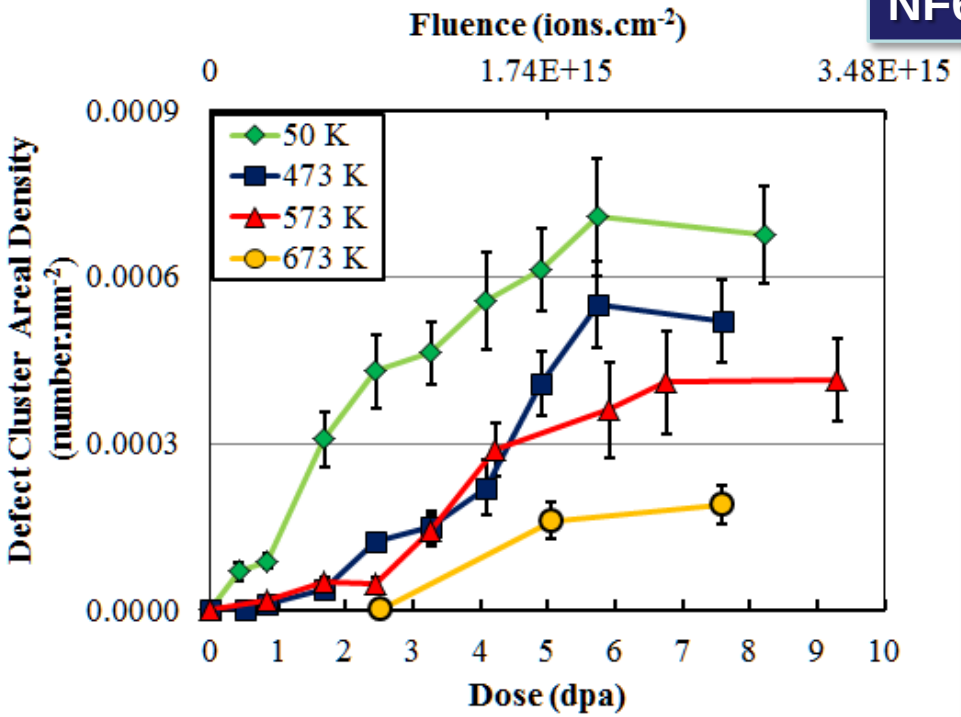
2 families of 100 aligned defects
Observed in pure Fe and Fe-Cr alloys

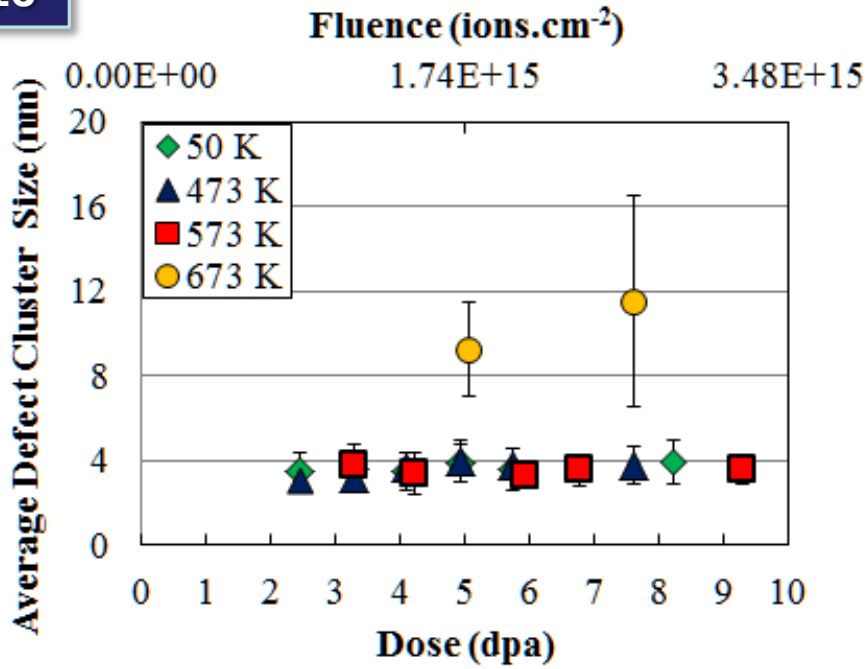




Defect Cluster Density and Average Cluster Size Vs. Dose and Temperature

NF616





- Lowest defect density was observed at 673 K
- Defect accumulation starts between 2.5 and 5 dpa at 673 K

- Larger clusters are seen at 673 K
- Coalescence of defect clusters at 673 K

Conclusions

In-situ irradiations of NF616 and HCM12A were performed with 1 MeV Kr ions between 50 K and 673 K.

- Similar microstructure evolution was observed in HCM12 and NF616 at 573 K (further analysis in process).
- Defect density increases with dose and saturates at ~6 dpa
- Saturation density decreases with temperature.
- Onset of defect accumulation shifts to higher doses with temperature.
- Size of defects is not a function of temperature ≤ 573 K
- Development of larger defect structures and a more pronounced effect of existing microstructure was observed at 673 K.

Results of in-situ investigations will be quantitatively compared with results of cluster dynamics calculations conducted at University of California Berkeley to model irradiation induced microstructure evolution in these complex F-M alloys.

Thank you for your attention

Acknowledgements

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