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In-situ Heavy-ion Irradiations of bcc Fe

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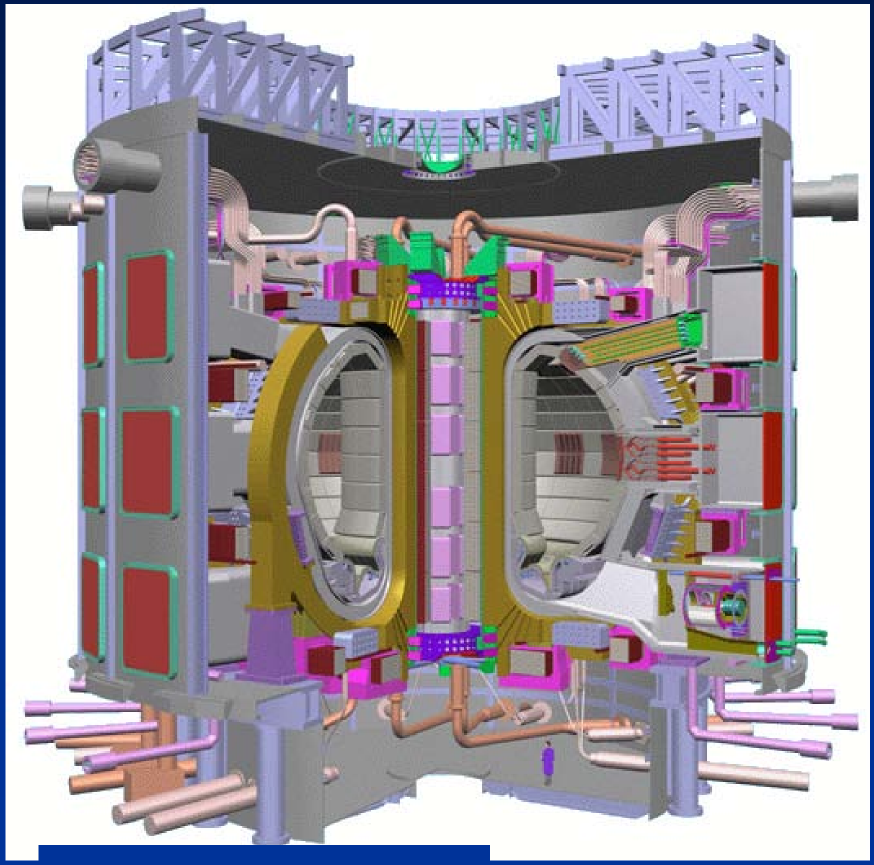
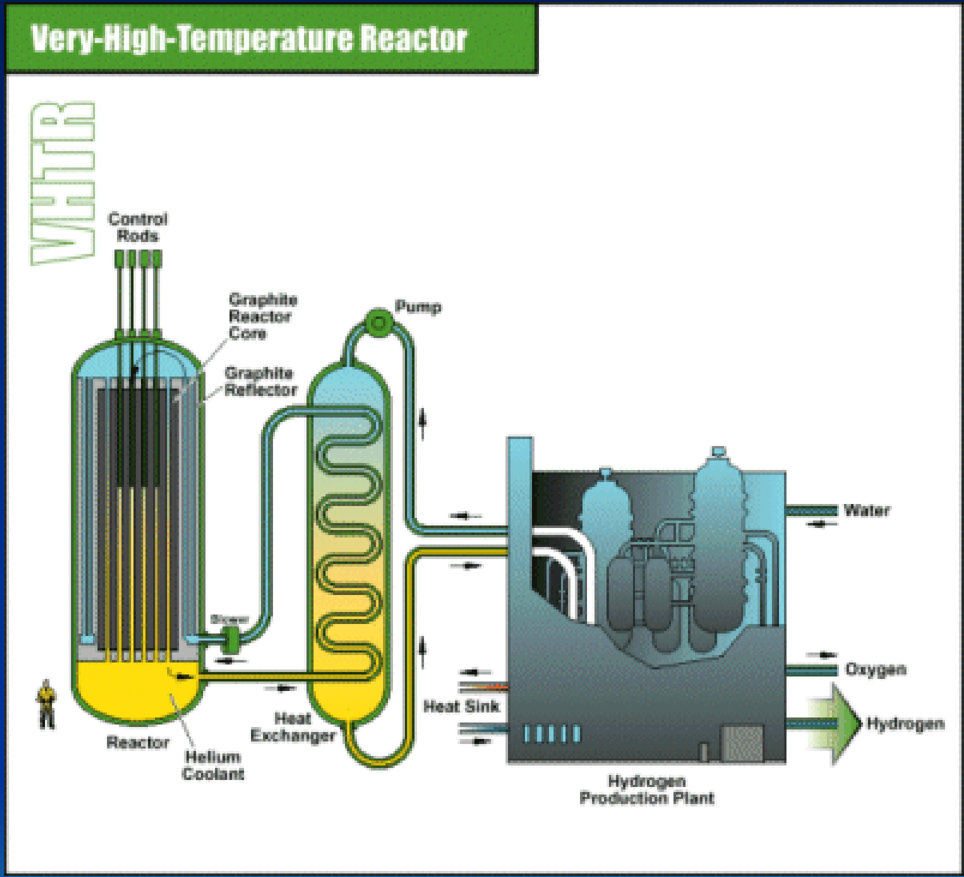
Background



Fusion - ITER

Nov, 2006 - Cadarache, France



Generation IV - VHTR

Materials problems in Fusion & Advanced Fission reactors

Materials - crucial to the success of future reactors.

The operating environment - extremely demanding.

Operating temperatures ~ up to	600°C
Stresses up to	300MPa
Radiation damage	~100 dpa
Transmutation	He + H

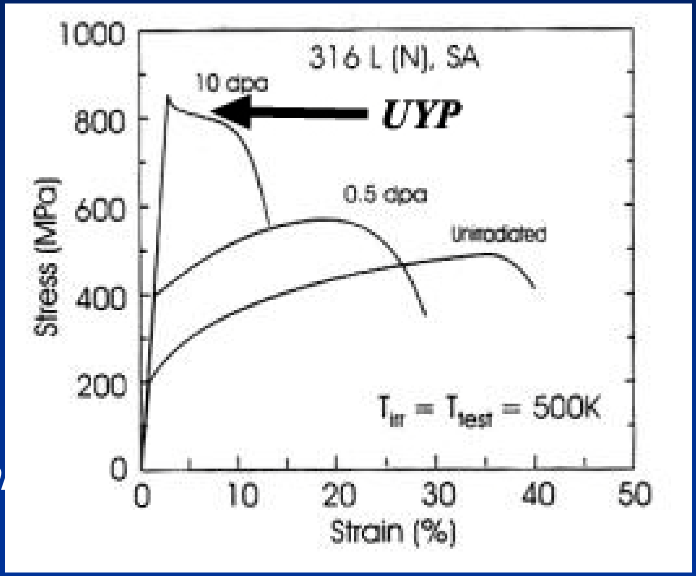
Structural materials must maintain adequate strength and toughness, while suffering minimal dimensional changes through swelling and creep.

No known materials meet these criteria so far.

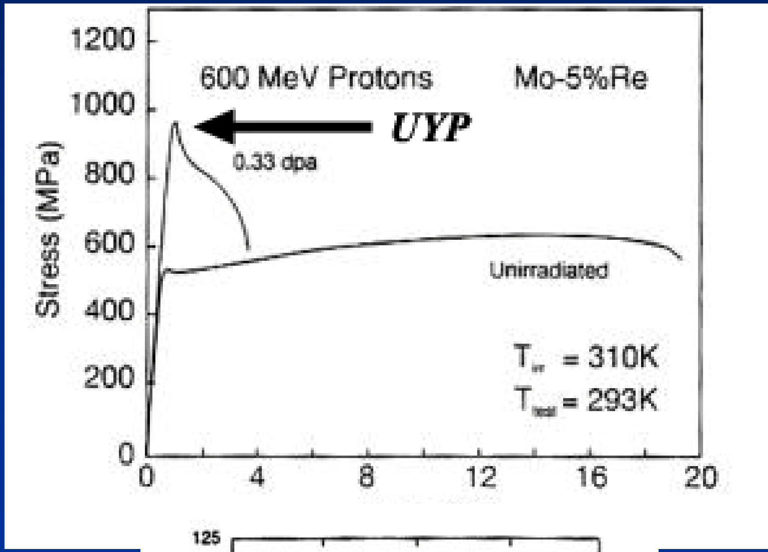
Candidate materials include steels based on iron ~9% chromium alloys, vanadium alloys and tungsten.

In all materials, irradiations induce a degradation of the mechanical properties:

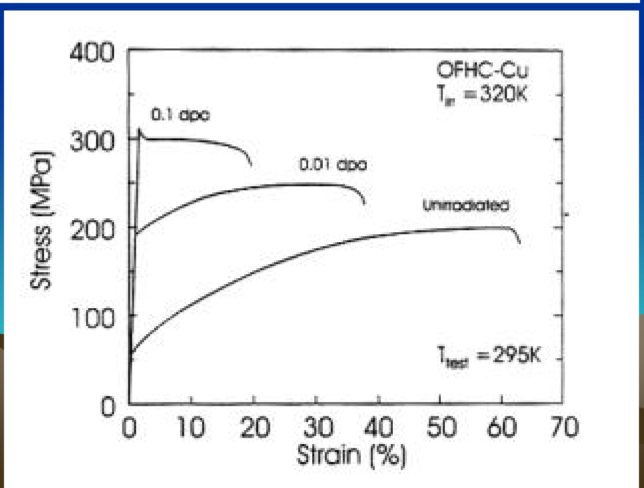
- hardening (increase in yield stress)
- decrease in ductility
- plastic instability (upper yield point + softening)



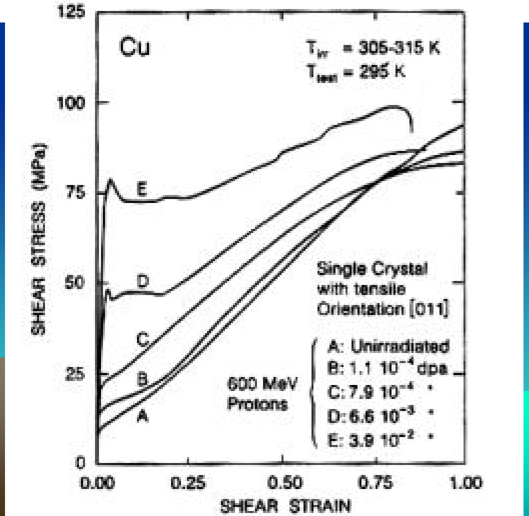
FCC steel



BCC Mo



Polycrystal Cu



Single crystal Cu

Literature

C English, M Jenkins & B Eyre, Nature 1976
After heavy ion (W) irradiation in Fe, the vacancy loops were found with both 1/2a<111> and a<100>.

D Gelles, J Nucl. Mat. 1982
The dislocation structures in the alloy series included dislocations with a<100> and a/2 <11 1> Burgers vectors; the former predominating in the lower chromium alloys, over the temperature ranges from from 400°C to 450°C.

I Robertson, etc., J Nucl. Mat. 1987
Most of loops are a<100> and maybe vacancy type at RT.

Y Konobeev, etc., J Nucl.. Mat. 2005
All loops are a<100> and voids are found at 400°C.

J Chen, etc., Acta Mat. 2007
Two sets of dislocation loops were identified, both interstitial in nature, with Burgers vectors b = a<100> and b = 1/2a<111>, and habit planes of (100) and (111), respectively, at temperatures from 300°C to 500°C.

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Experiments

IVEM-Tandem

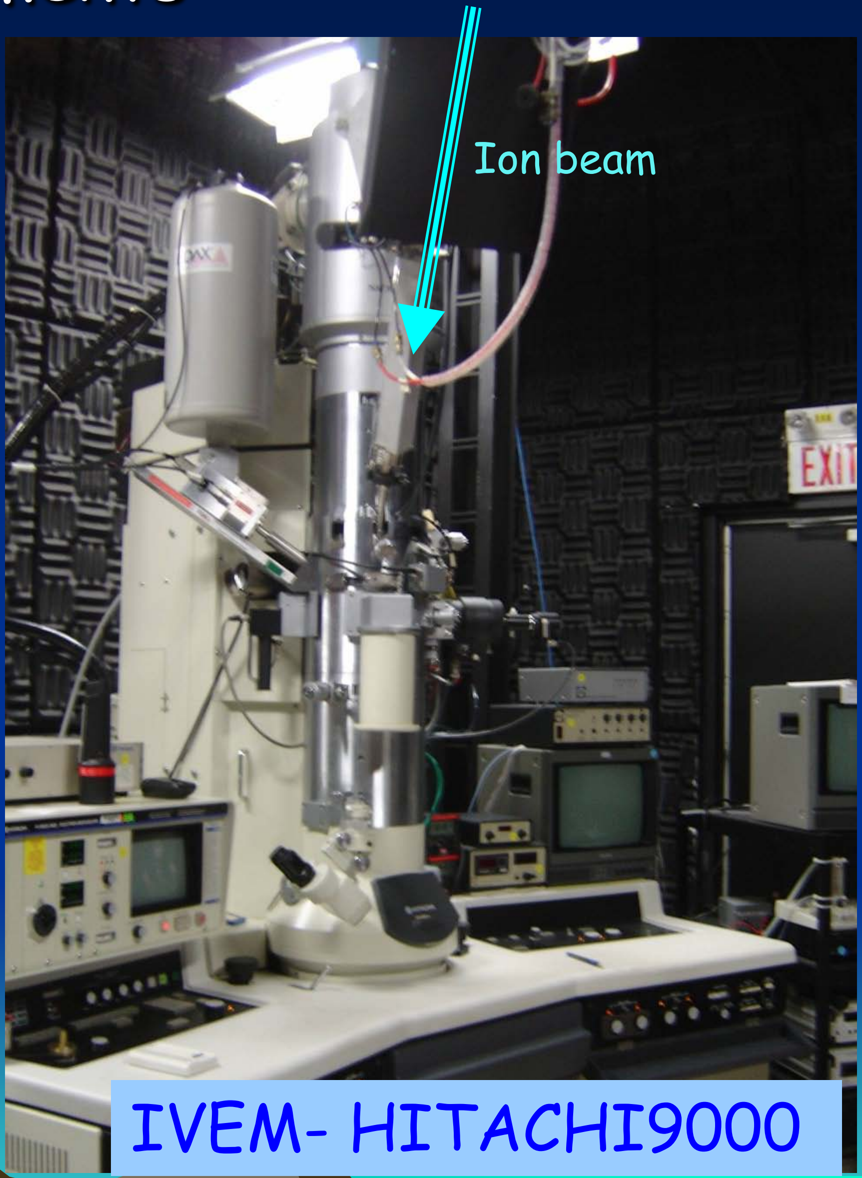
Transmission electron microscope interfaced with two ion accelerators for in situ ion beam studies involving ion implementation and/or ion damage.

Accelerating Voltages 100-300 kV
Goniometer Tilts +/-45deg. (X-Axis); +/-30deg. (Y-Axis)
Video Capabilities:
Gatan Model 622 Image Intensified Camera on column axis
VHS Video Tape Format (including S-VHS)

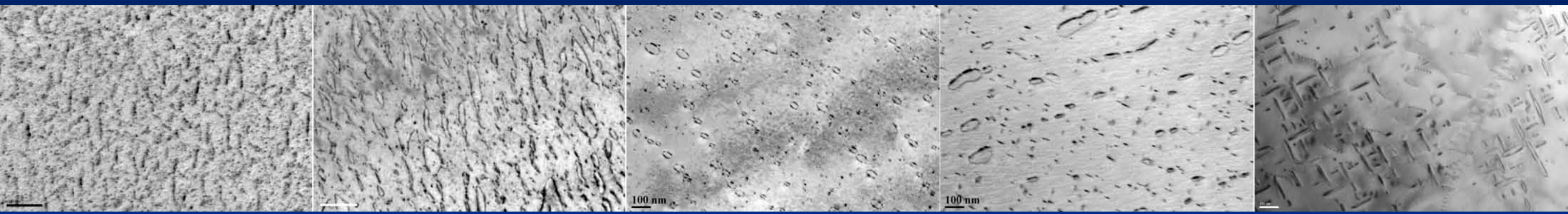
Specimen holders
Cooling and heating from 15-1200K
Straining from 300-600K
Electrical biasing experiments from 300-800K.

Angle between Incident Electron and Ion Beam: 30deg
Ion Beam Diameter at Specimen Position: ~1.5mm

Ion Irradiation Conditions (Limited by Bending Magnet)
650kV Accelerator--All ions to 600 kV
Tandem--He to 2 MV; Ne to 1.8 MV; Ar to 0.9 MV ; Kr to 0.4 MV (singly charged ions)



Irradiation induced Microstructures



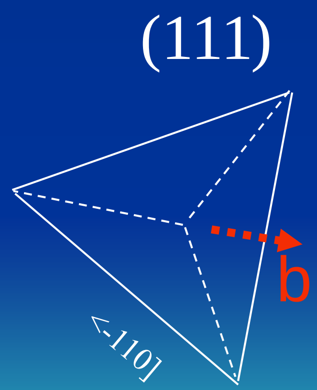
RT	300 ºC	400 ºC	450 ºC	500 ºC
½<111> loop clusters along <110> direction	½<111> large finger loops with big shear components	½<111> large circular loops with shear components	½<111> large circular loops plus <100> edge-on loops	Only <100> edge-on loops

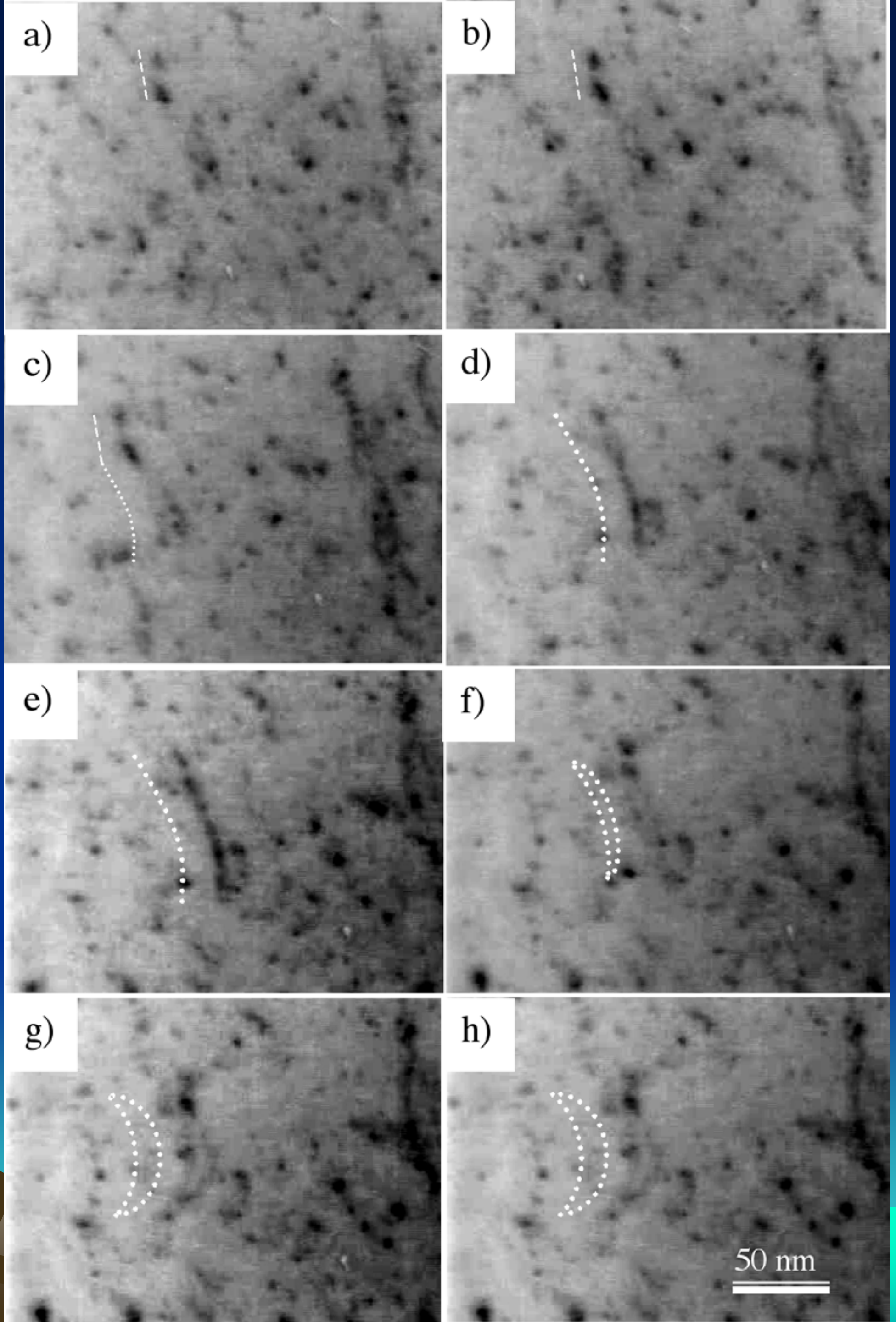
100 nm

In-situ irradiation

In ultra-pure Fe at 300°C

Dose increment: 6~10 dpa



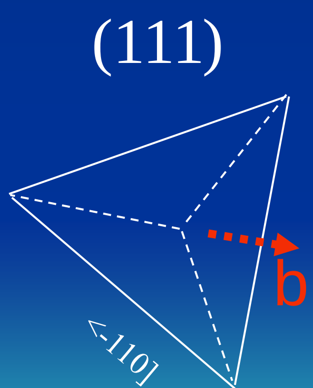


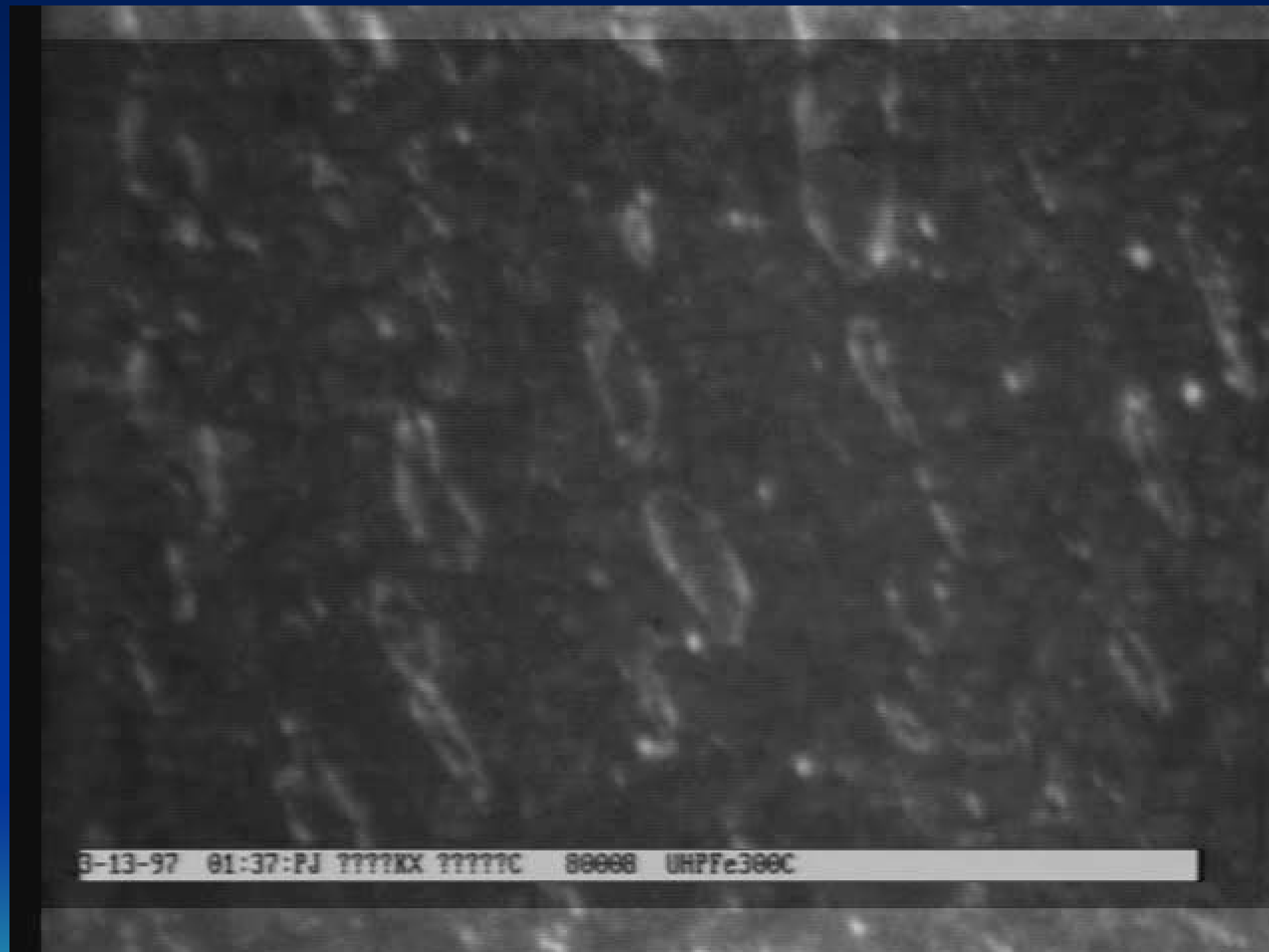
Z Yao, etc Phil. Mag. (2008)

In-situ irradiation

In ultra-pure Fe at 300°C – these are interstitial loops with **b** = ½ [-111]

25 nm

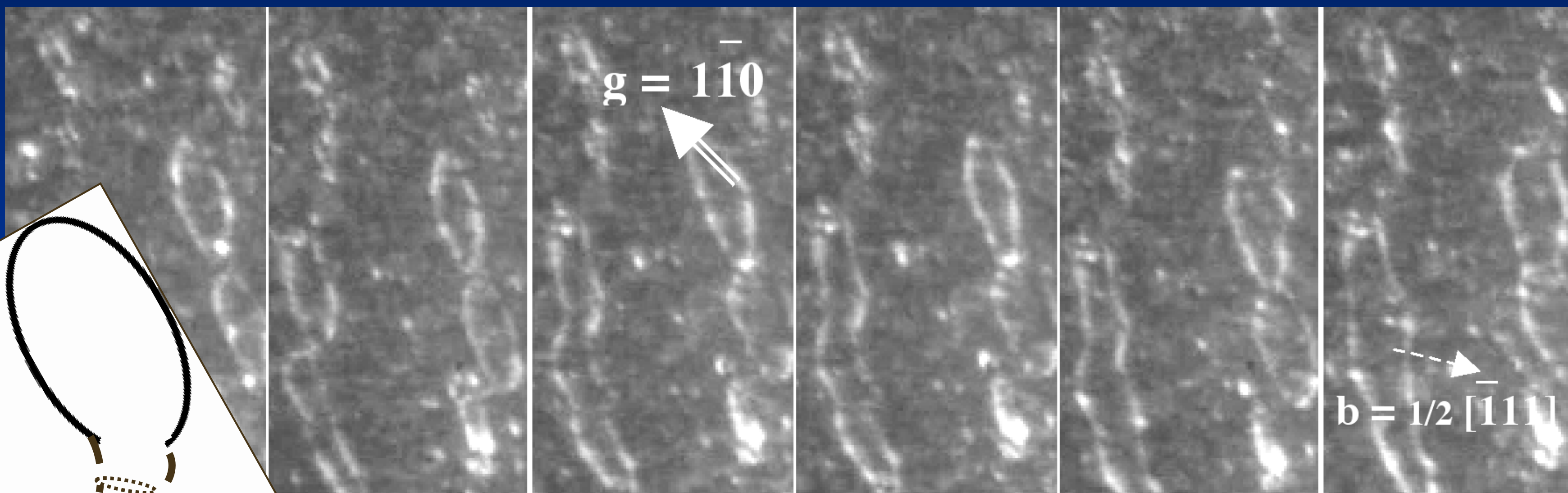


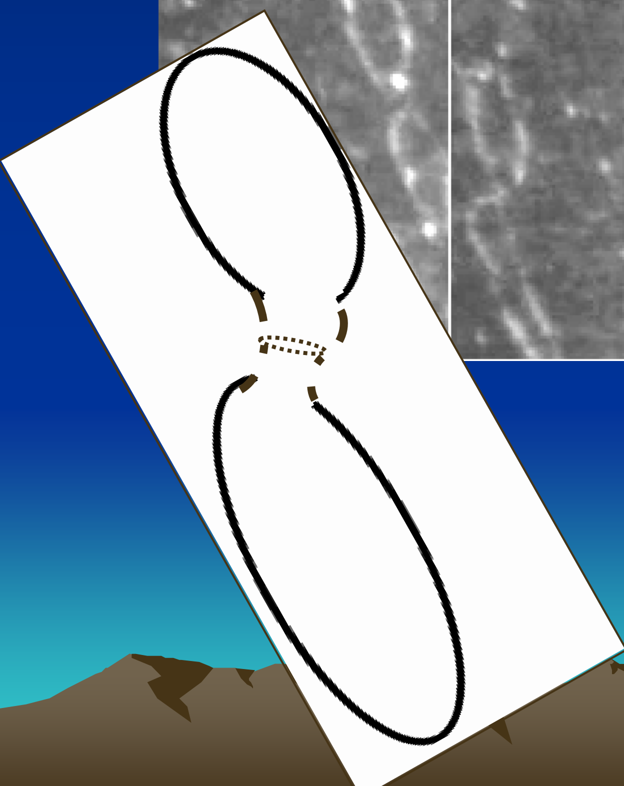


Dose increment: 6~10 dpa; viewed 40 x real time

Z Yao, etc   Phil. Mag. (2008)

Loop coalescence and growth

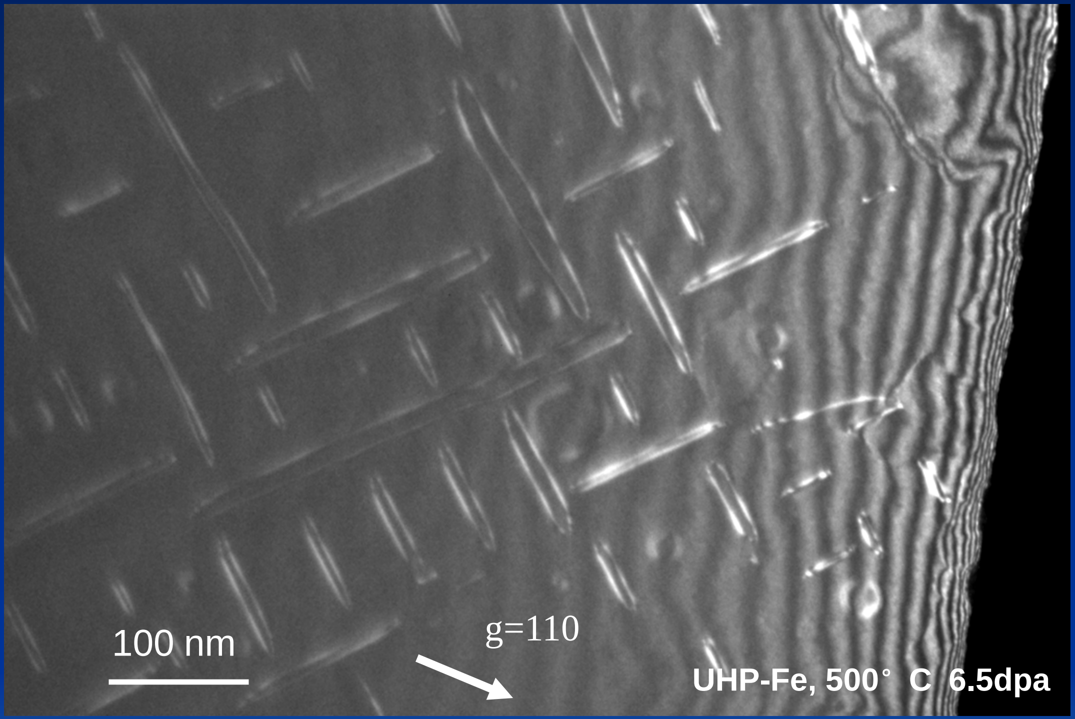




M Jenkins, Z Yao, M Kirk   Journal of Nuclear Materials (2009)

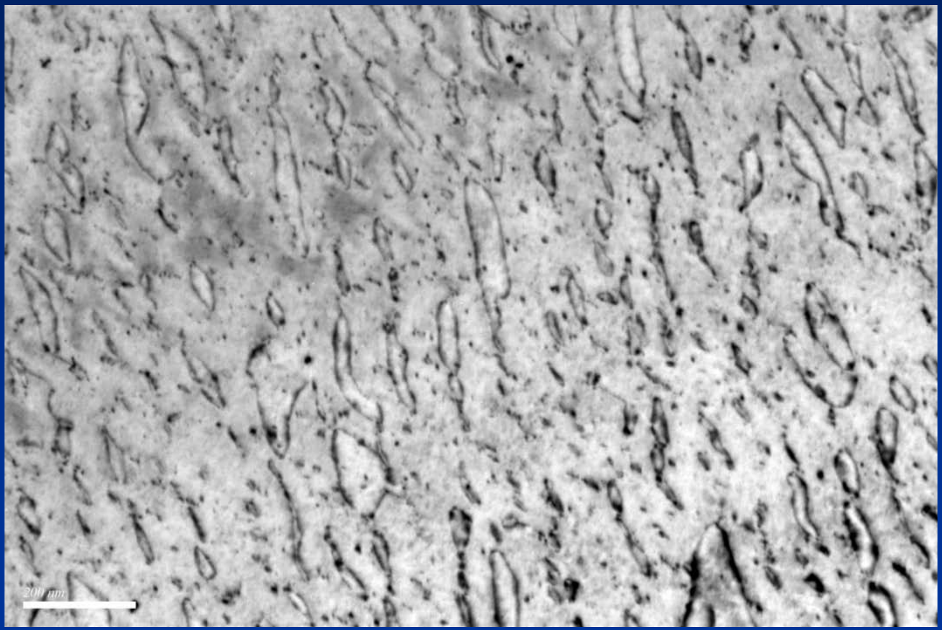
In-situ irradiation

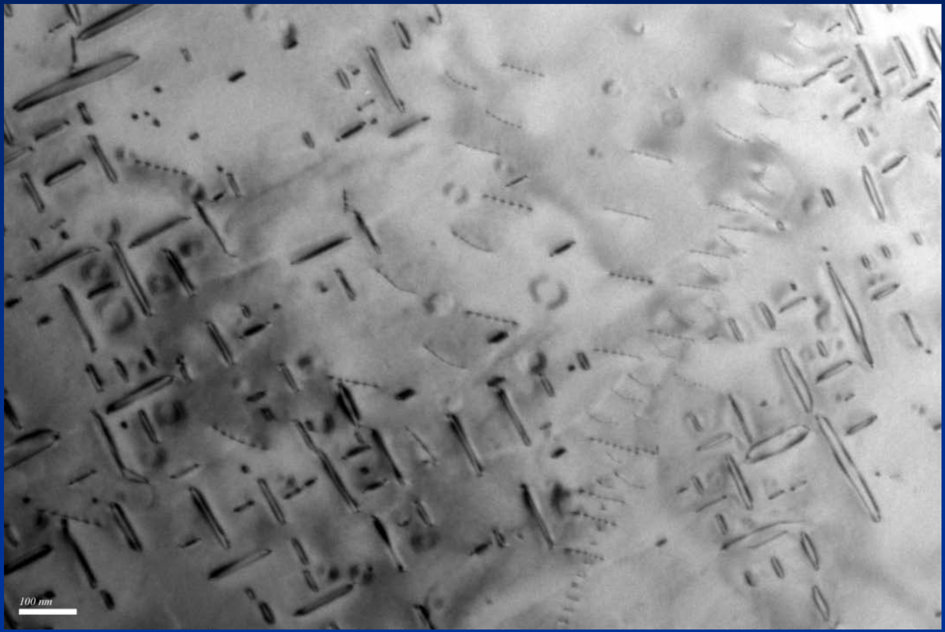
In ultra-pure Fe at 500°C – there are only interstitial loops with b = ½ <100>



M Jenkins, Z Yao, M Hernandez, M Kirk Journal of Nuclear Materials (2009)

What is going on?





½<111> loops predomination
At 300° C

Only **<100> loops**
At 500° C

UHP-Fe irradiated to dose: 1x10^{19} ions m^{-2}

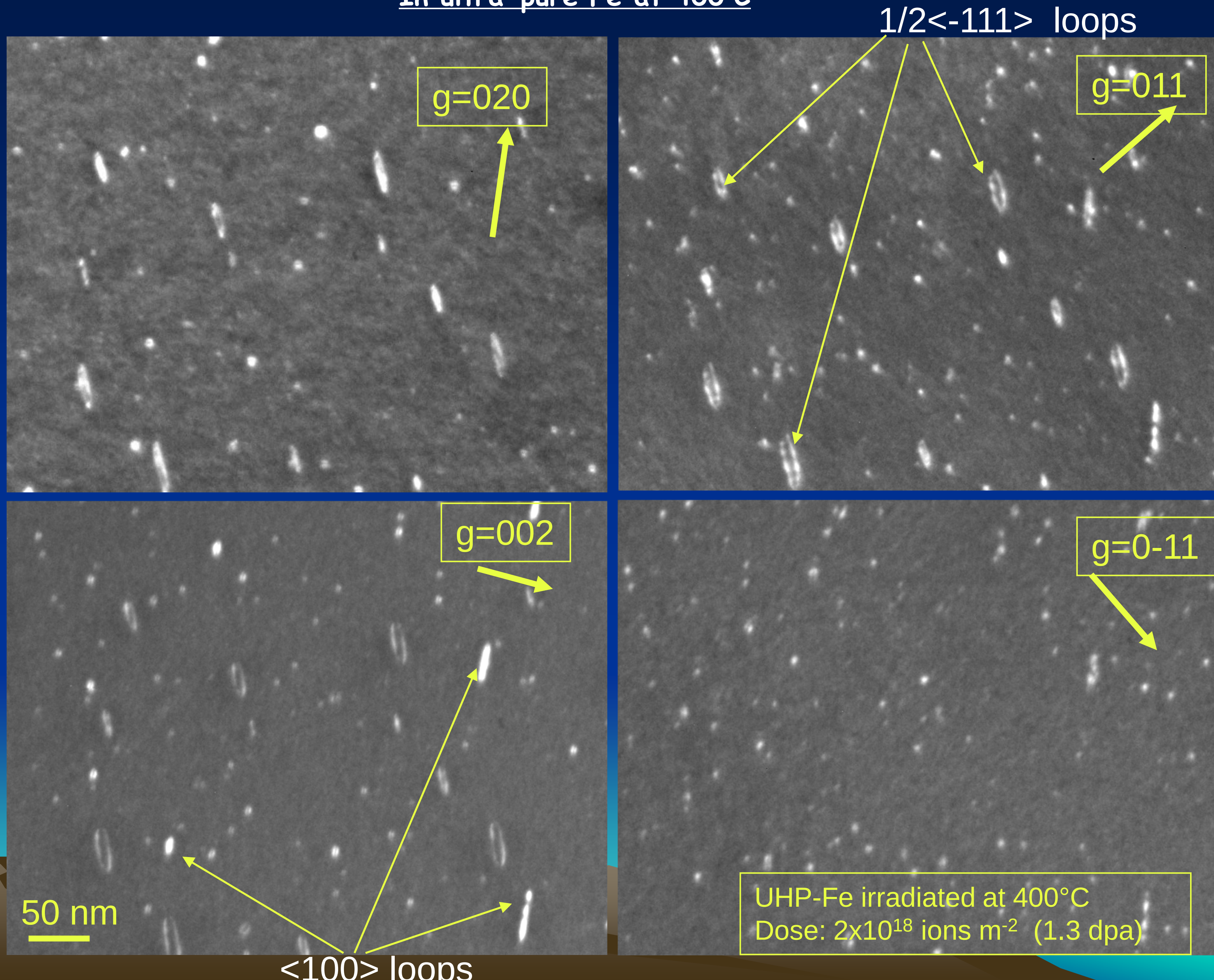
M Jenkins, Z Yao, M Kirk Journal of Nuclear Materials (2009)

Where are 1/2<111> loops going to?

1. They might lose to surface, but we should see their formation initially?
1. If irradiation was performed in <110> foil, do they form and stay at 500 $^\circ$C.
1. Temperatures between 300°C and 500°C.

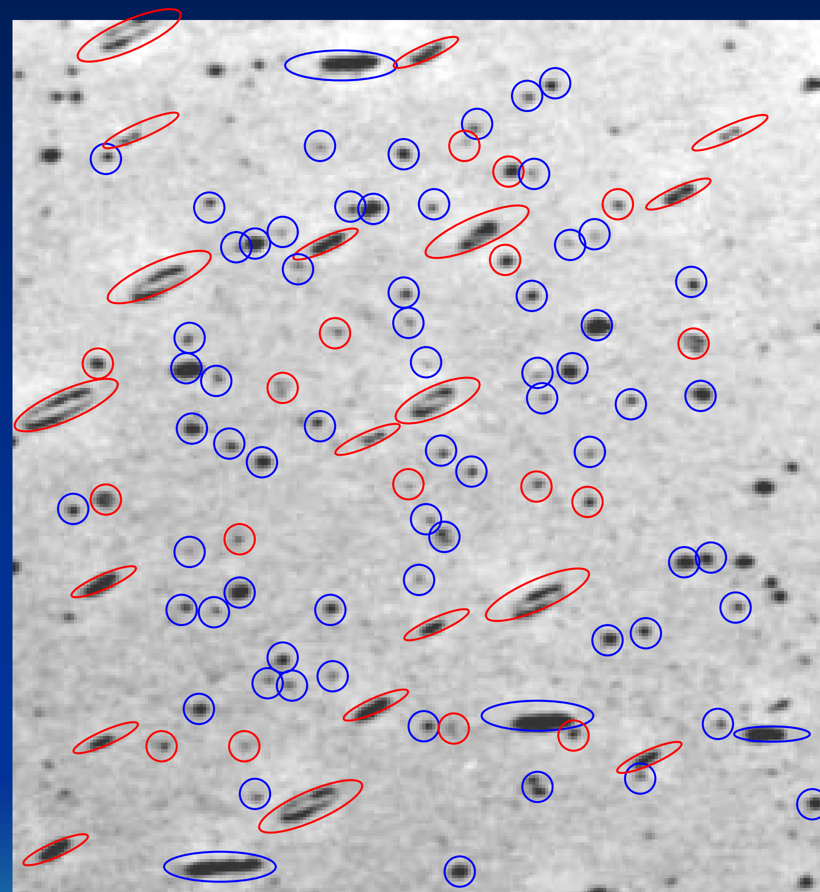
In-situ irradiation

In ultra-pure Fe at 400°C



In-situ irradiation

In ultra-pure Fe at 400°C



50 nm

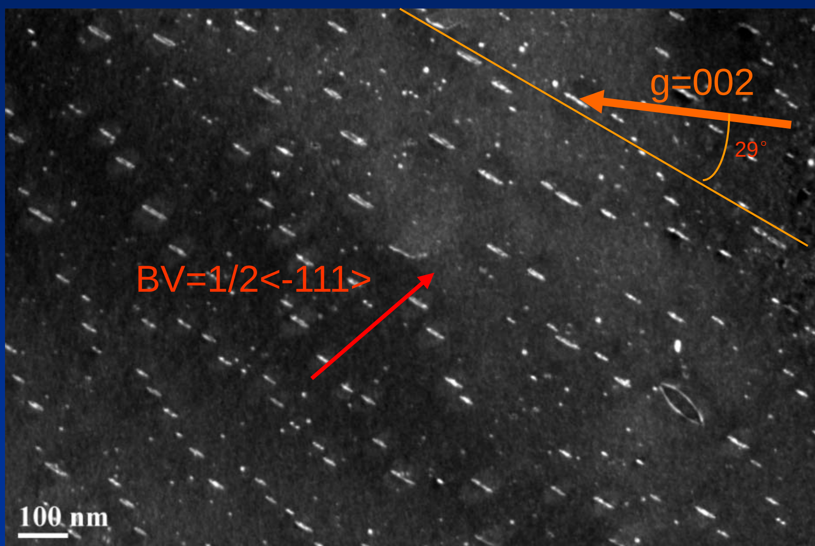
Burgers Vector 1/2<-111>

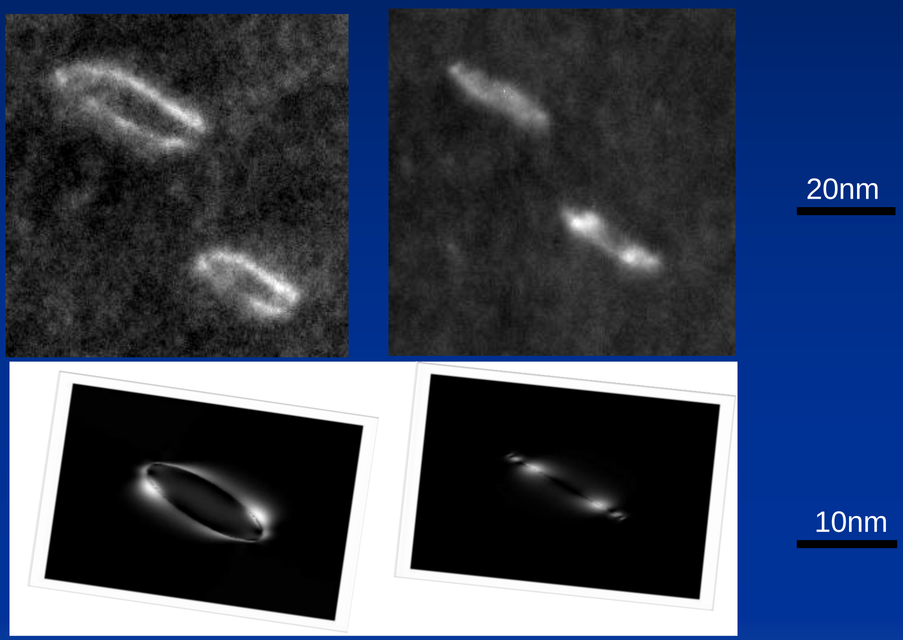
Burgers Vector <100>

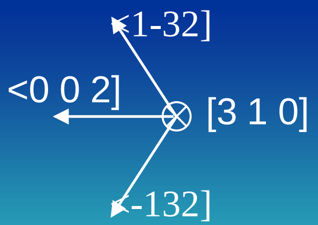
Habit Plane of 1/2<111> loops

<u>In ultra-pure Fe at 400°C</u>

Pole (110) (310)



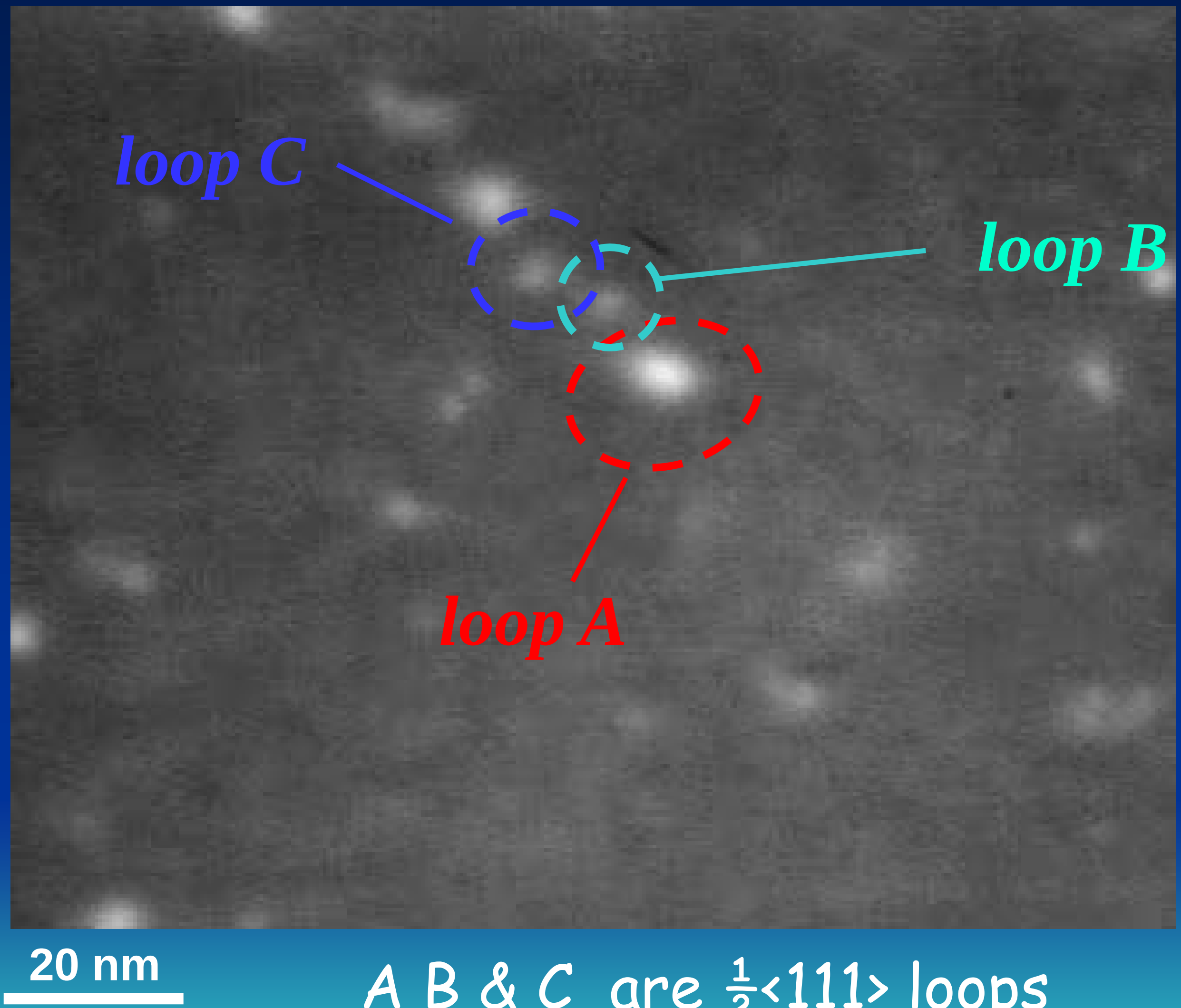




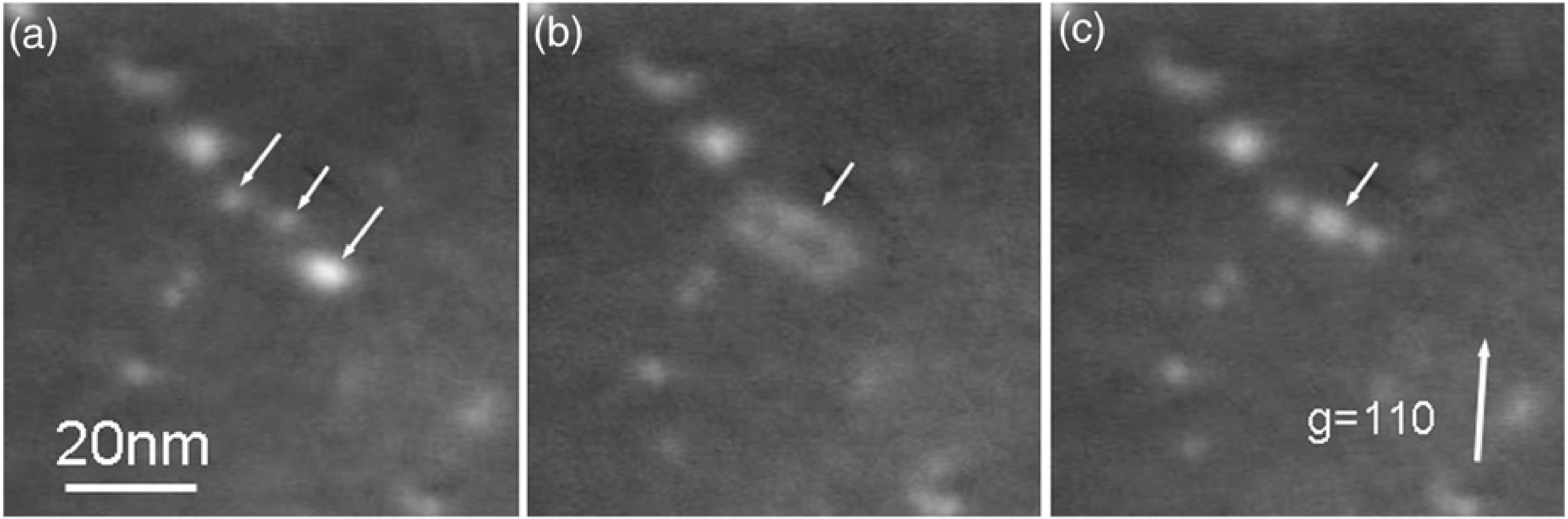
b=1/2<-111>, HP=<-132>

In-situ irradiation

In ultra-pure Fe at 450°C – these are interstitial loops with b = ½ [-111]



A B & C are ½<111> loops



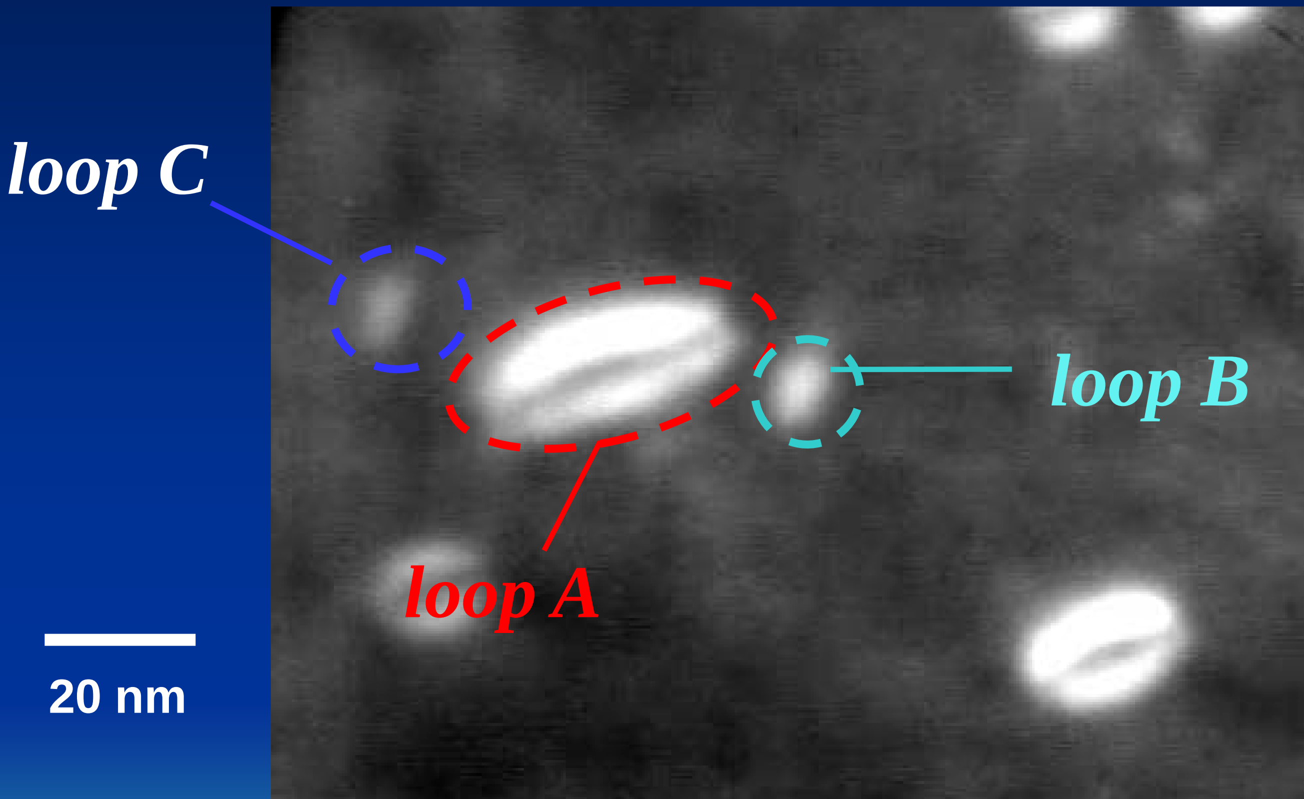
This occurred soon after ion irradiation ceased, as the specimen was being examined in the TEM and was still at 400°C. Three small ½<111> loops towards the centre of (a) coalesce into a single ½<111> loop in (b). The loop is seen to rotate to new habit plane (c) and continues to hop.

Where are 1/2<111> loops going to?

1. They might be going to surface, but we should see their formation?
1. If irradiation was performed in <110> foil, do they form and stay at 500 °C.
1. Temperatures between 300 °C and 500 °C.

In-situ irradiation

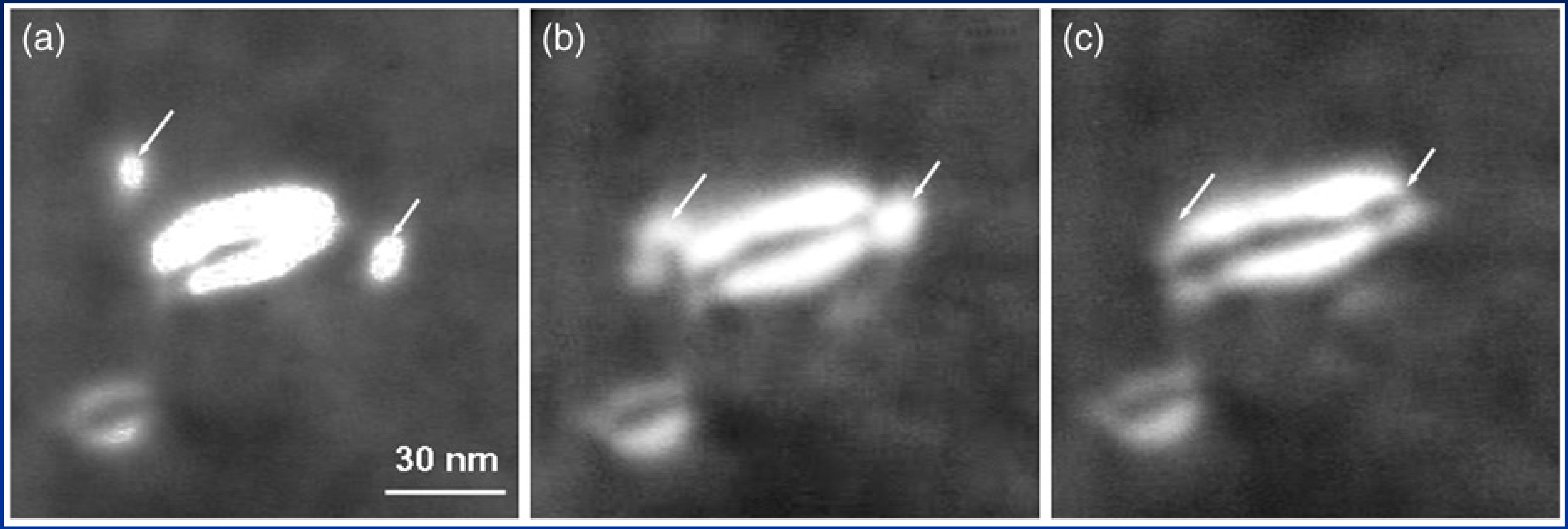
In Single Crystalline <110> Fe at 465°C



A <100> loop

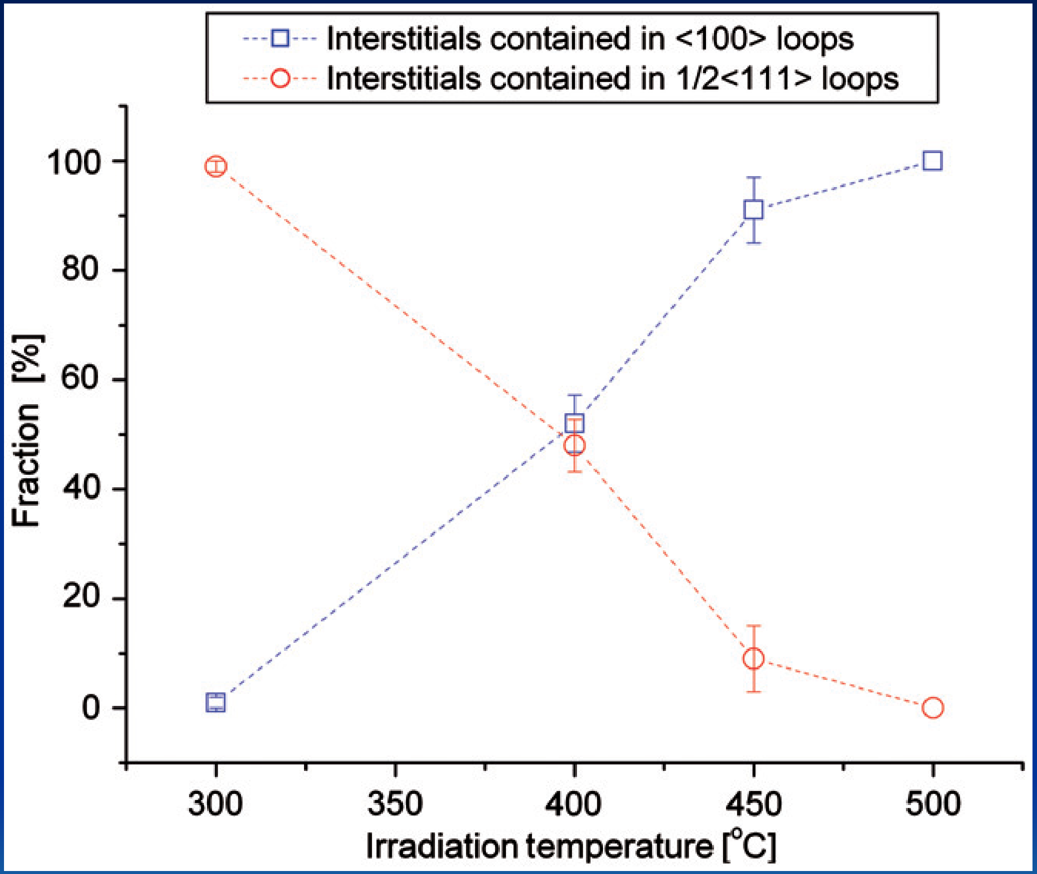
B $\frac{1}{2}$<111> loop

C $\frac{1}{2}$<111> loop



The large loop at the centre of the micrograph with b <100> subsumes two smaller ½<111> loops.

Transition



Estimates of the fraction of interstitials contained in loops of the two types.
The error bars do not take account of systematic errors due to loss of ½<111> loops to the surface.

Summary

TEM investigations of damage evolution have been carried out in electron transparent foils of ultra high pure Fe (UHP) and Fe-Cr alloys produced by in-situ heavy ion irradiations at room temperature, 300 °C, 400 °C, 450 °C, 465 °C, 482 °C and 500 °C in the Argonne IVEM-Tandem Facility.

1. At temperatures below 500 °C, loops formed with a mixture of 1/2<111> and <100> Burgers Vectors. At 500 °C, only <100> loops formed. 1/2<111> loops start to be absorbed or evaporated at 465 °C.

2. 1/2<111> interstitial loops are remarkably mobile. In contrast, <100> loops are much more sessile. They don't hop but may move to coalesce or may be lost at the surface.

3. Extensive microstructures formed at high doses by loop growth and coalescence.

4. The in-situ TEM experiments provide the most direct evidences for Modellers.

Acknowledgments

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