The transmission electron analysis (TEM) of dislocation loops in T91 steels from MEGAPIE and TWIN-ASTIR irradiation programs

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Outline

- ❑ MEGAPIE experiment
- ❑ MIRE irradiation program
- ❑ Post irradiation experiments of T91 steel
  - ✓ Transmission electron microscopy
- ❑ Comparison with the results of FeCrC alloys

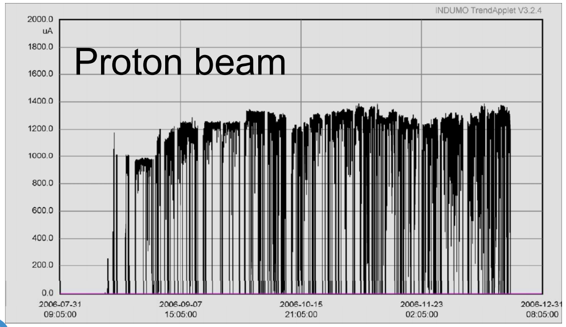
Characterization of irradiation induced damage

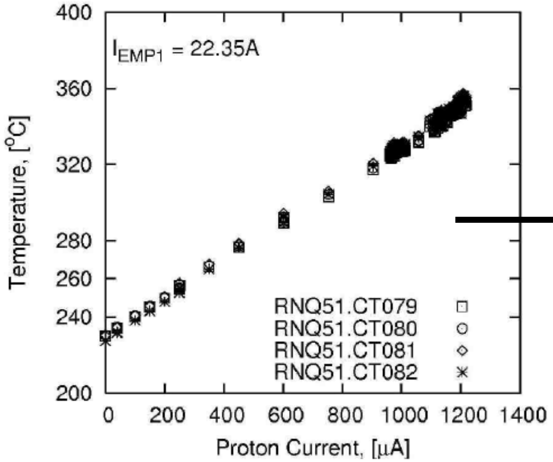
Dislocation loop distribution

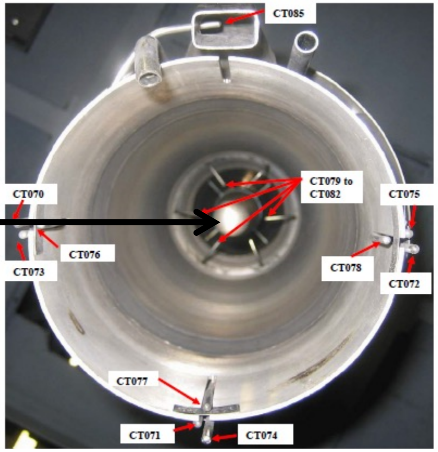
MEGAPIE and MIRE irradiation programs

MEGAPIE experiment

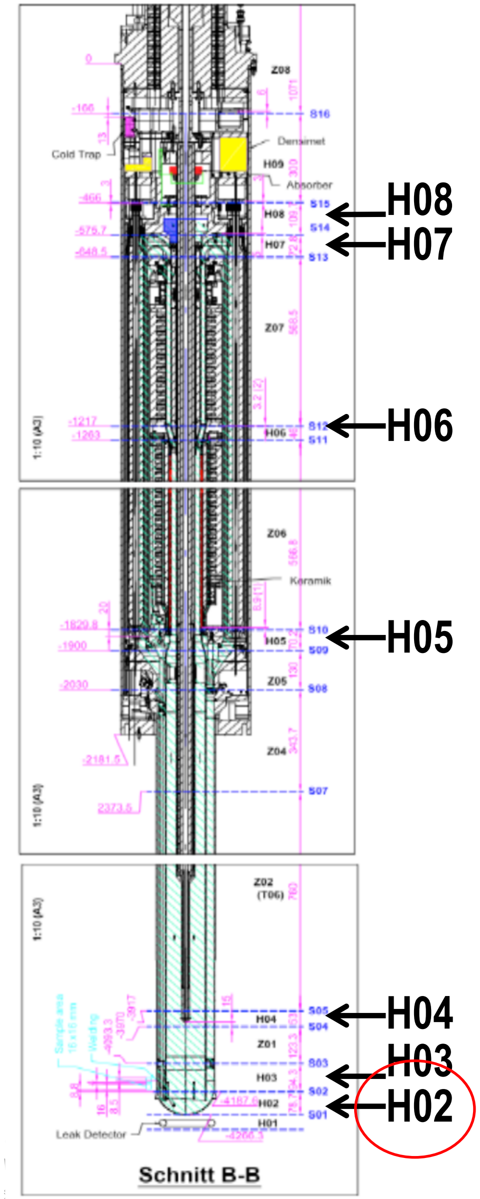
- Materials: T91, 316L
- Irradiation: High-E protons + spallation neutrons
- Doses: 0-7 dpa - window
- Environment: LBE
- Temperatures: 230-350°C
- Specimens extracted: Flat tensile, TEM discs, plates, bars



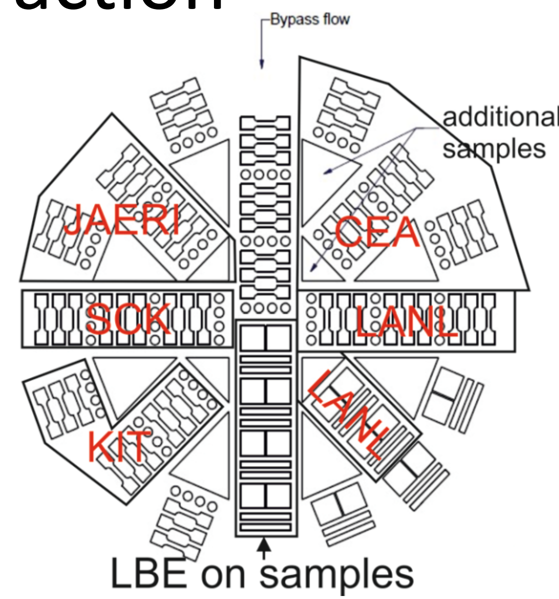


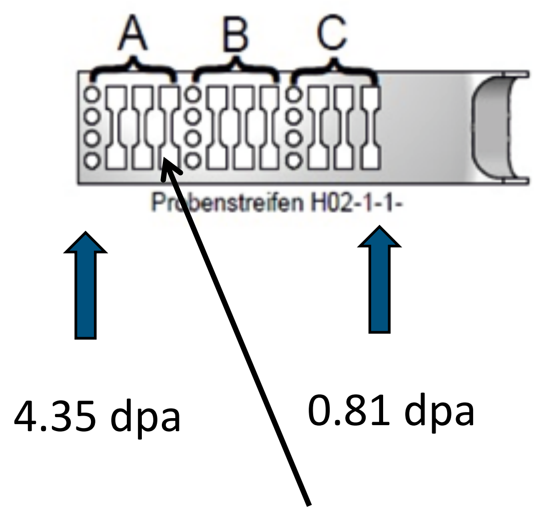


MEGAPIE sample extraction



H02 - The Beam Entrance Window (Calotte)





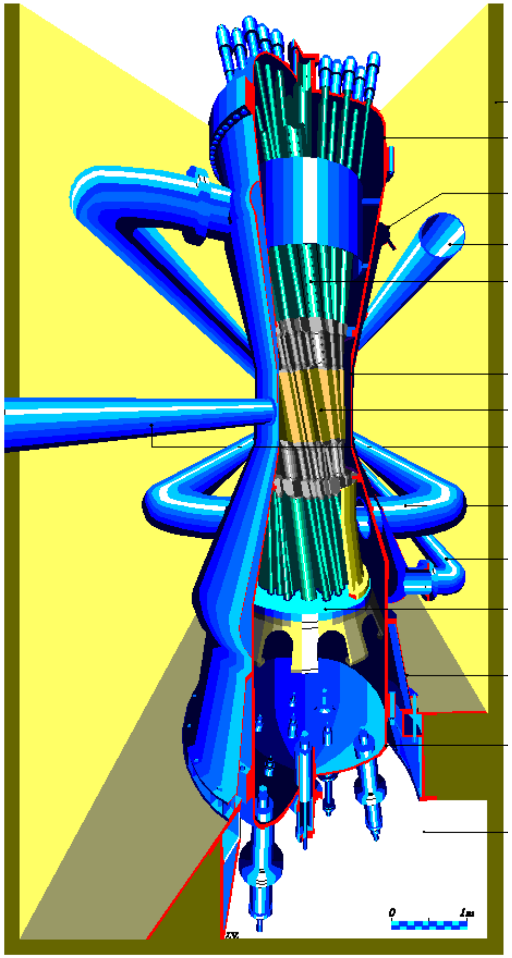
M. J. Konstantinovic et al., JNM 468 (2016) 228

Neutron irradiation MIRE - Cr

Materials	Temperature (^0C)	Dose (dpa)		
Fe-2.5% Cr-C	300	0.06	**0.6**	**1.0**
Fe-5% Cr-C	300	0.06	**0.6**	**1.0**
Fe-9% Cr-C	300	0.06	**0.6**	**1.0**
Fe-12% Cr-C	300	0.06	**0.6**	**1.0**
T91	300	0.06	**0.6**	**1.0**

Neutron flux = 7.4×10^{13} n cm^{-2} s^{-1}.

BR2 reactor in Mol



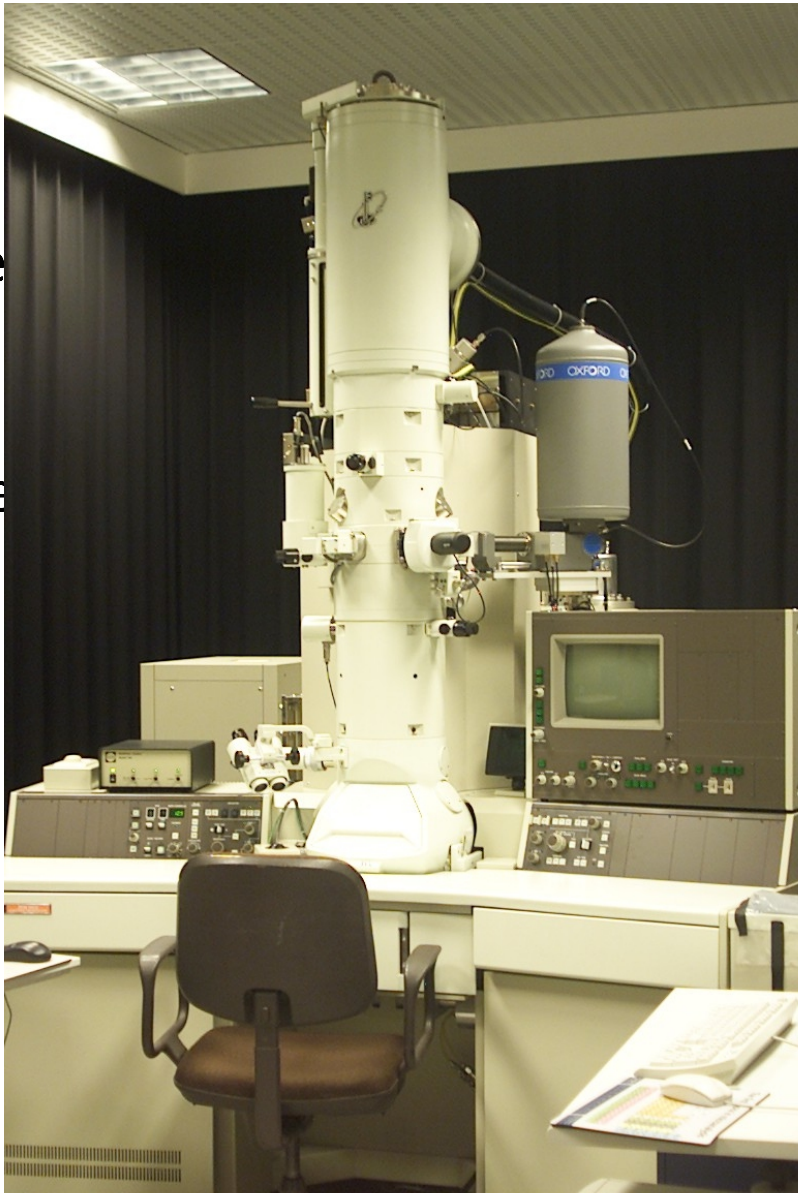
TEM of MEGAPIE and MIRE T91 samples

TEM at SCK•CEN

Mechanical polishing on SiC pape
in hot cell

Electrochemical polishing in fume
hood

JEOL 3010 instrument
Operating at 300 kV
Placed in controlled area

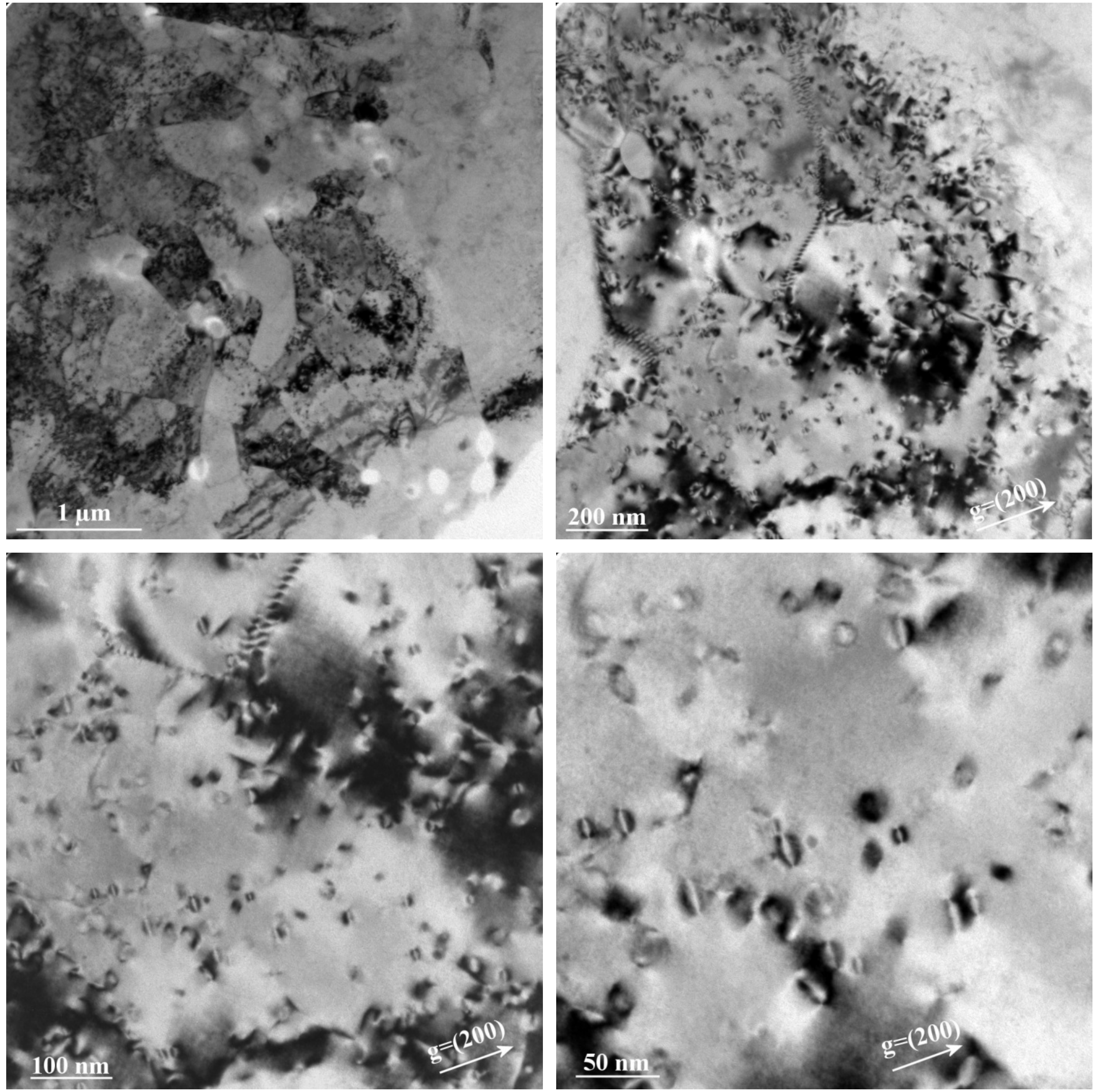


TEM-Loops

H-02-13-A

Dose 4.35 dpa

T_{irr}= 320-350 ^{0}C



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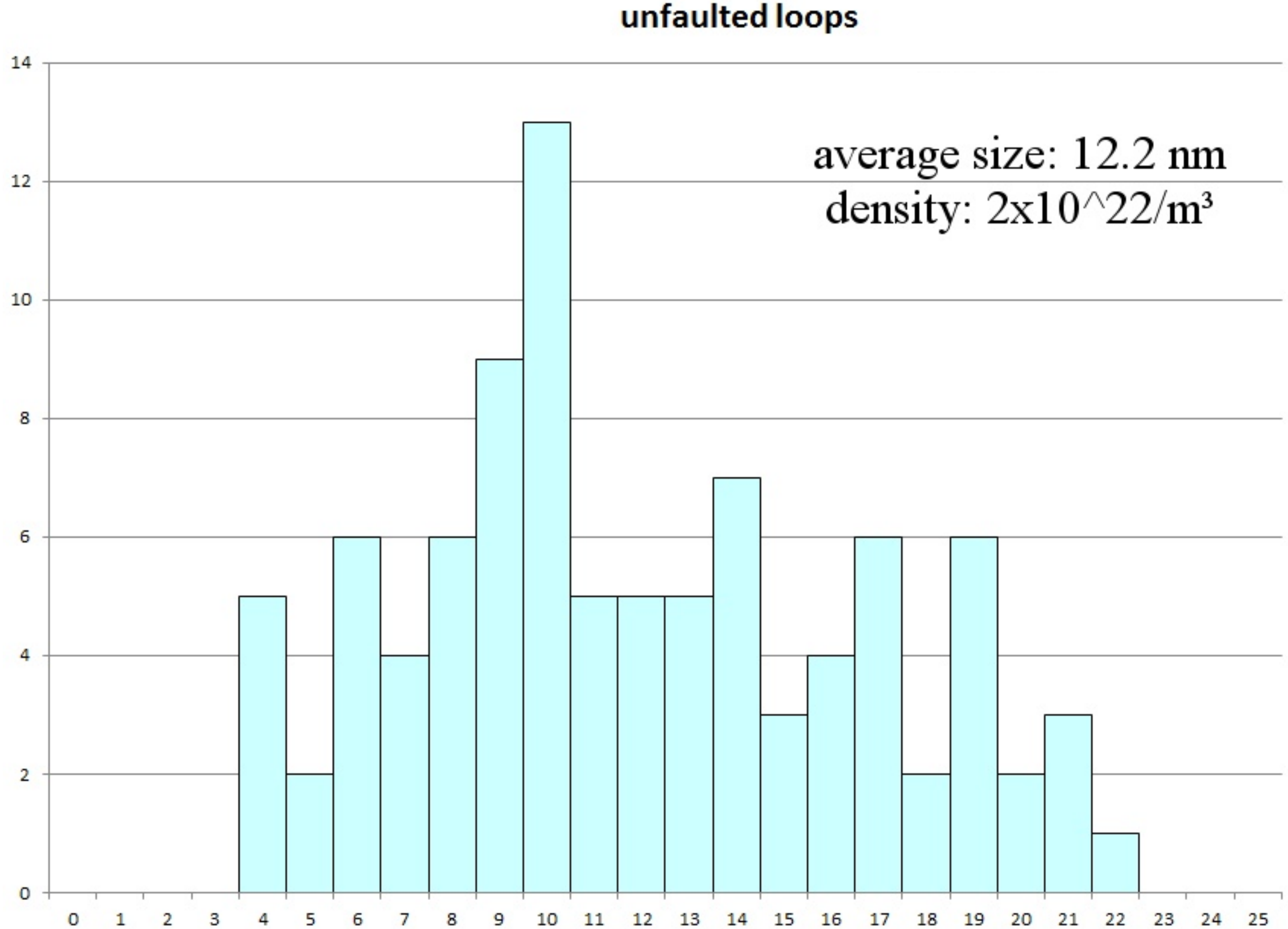
TEM-Loops

Sample: H-02-13-A, Dose 4.35 dpa, T_{irr}= 320-350 ^{0}C

Type <100>

Type <111>
0-7 %

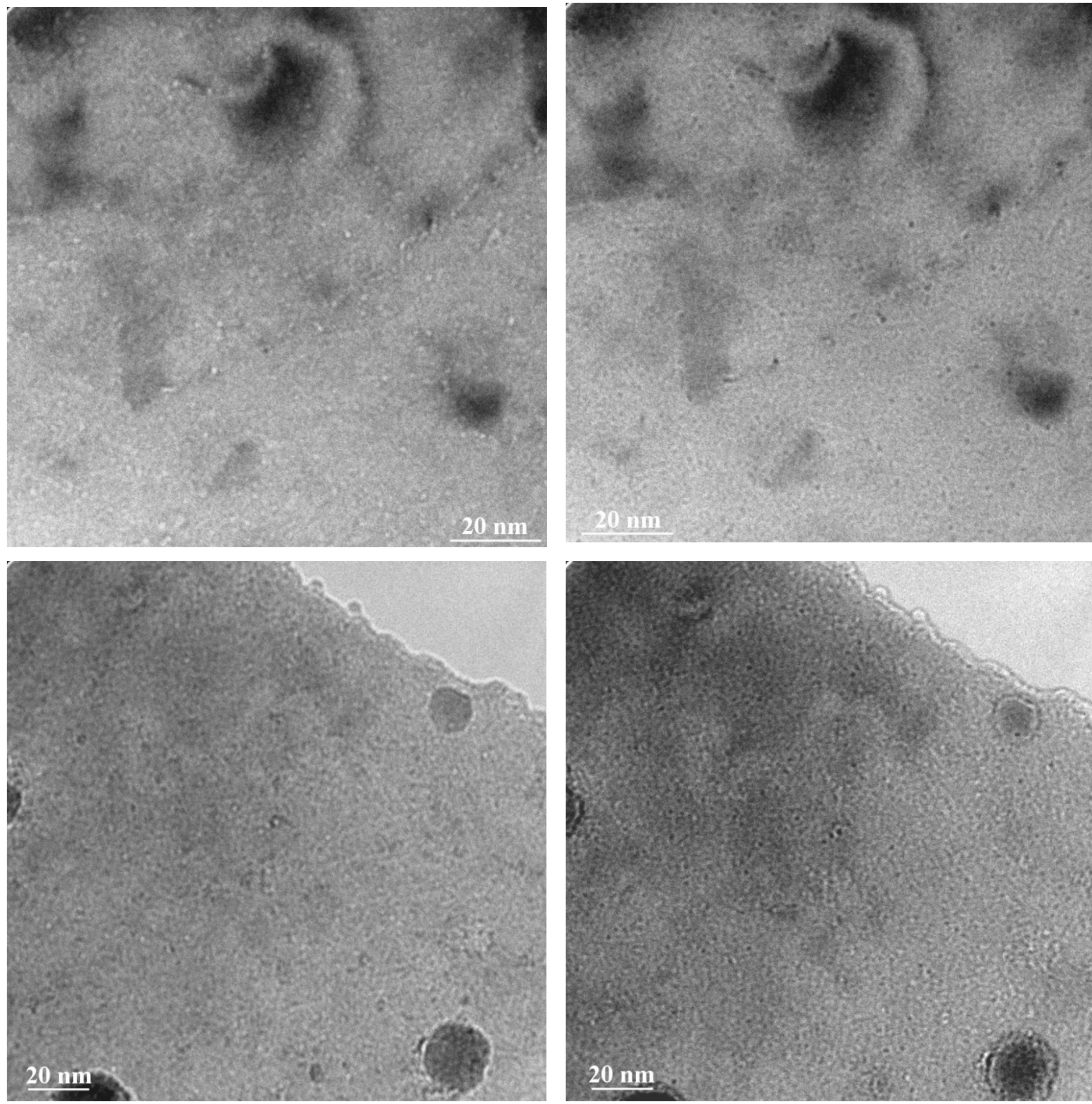


TEM-Voids

H-02-13-A

Dose 4.35 dpa

T_{irr}= 320-350 ^{0}C



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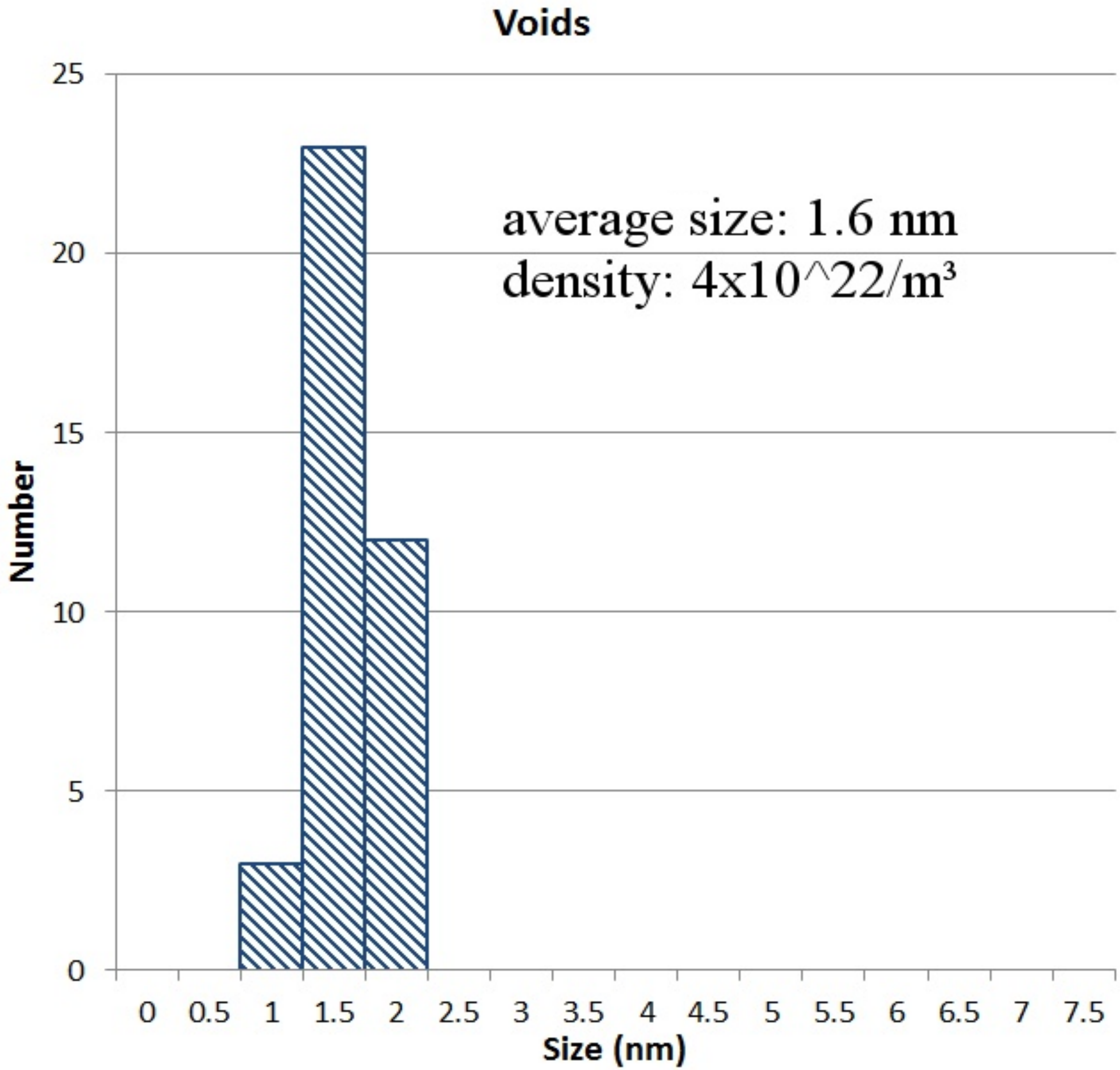
TEM-Voids

H-02-13-A

Dose 4.35 dpa

T_{irr}= 320-350 ^{0}C



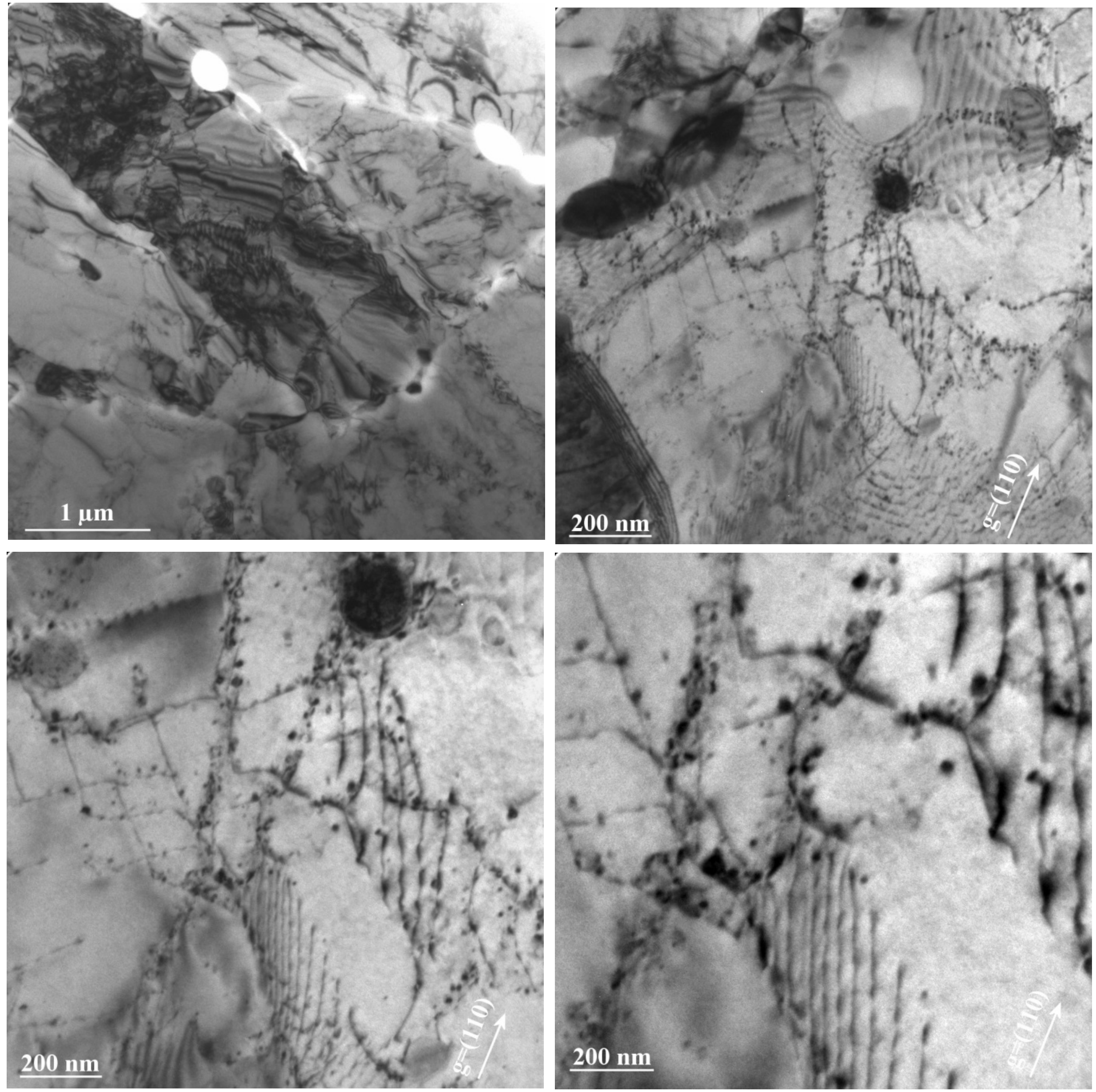
TEM-Loops

H-02-13-C

Dose 0.88 dpa

T_{irr}= 320-350 ^{0}C

Dislocation density – order/two orders of magnitude higher in comparison with H-02-13-A



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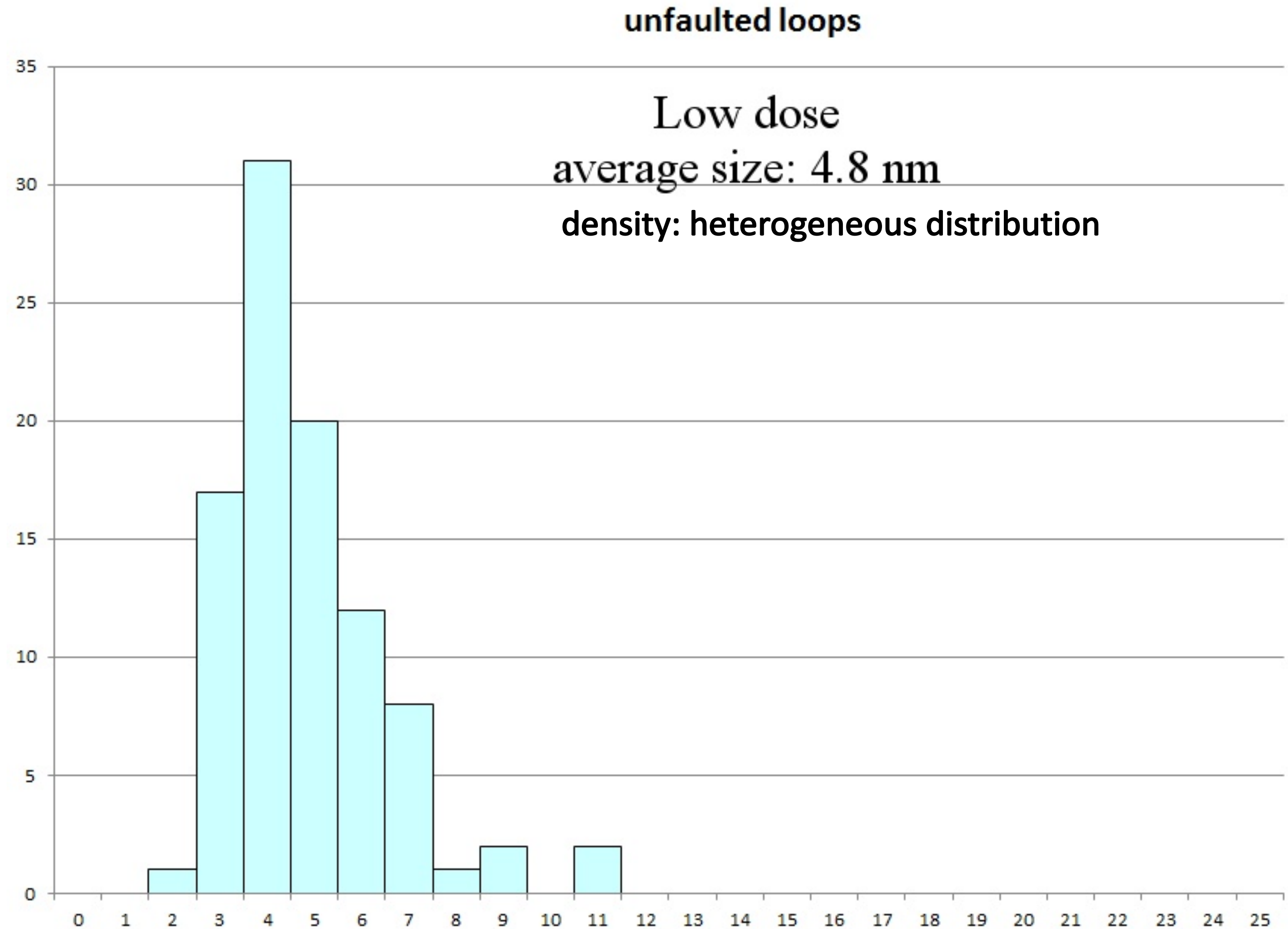
TEM-Loops

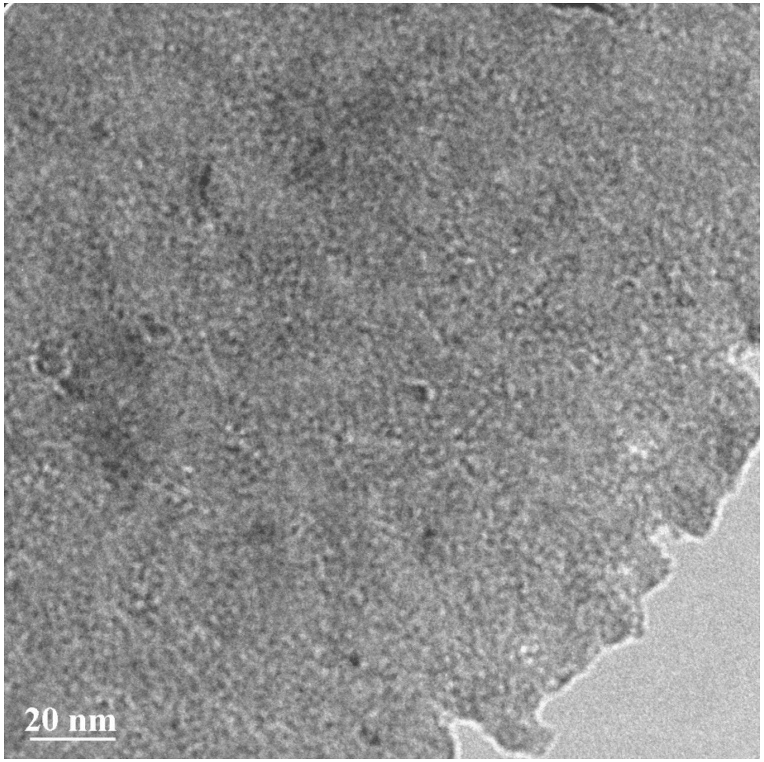
Sample: H-02-13-C, Dose 0.88 dpa, T_{irr}= 320-350 ^{0}C

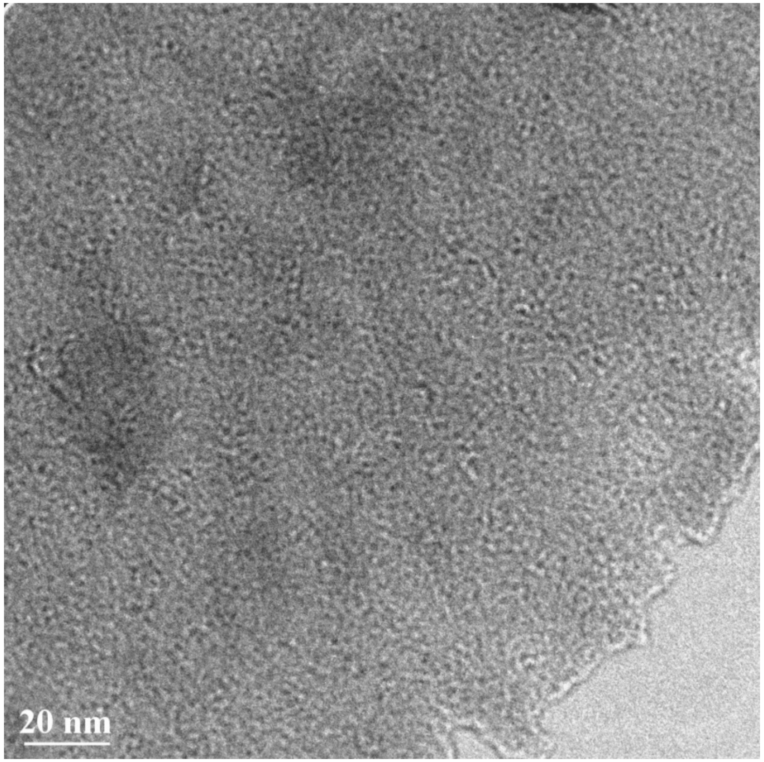


TEM-Voids

H-02-13-C , Dose 0.88 dpa, T_{irr}= 320-350 ^{0}C

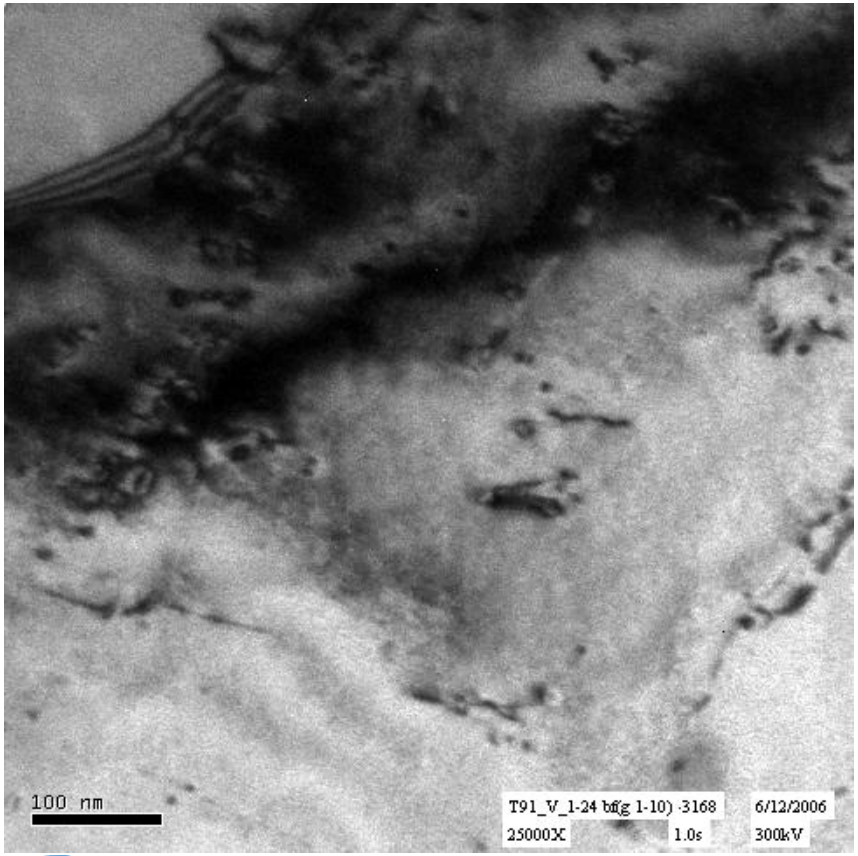
No Voids

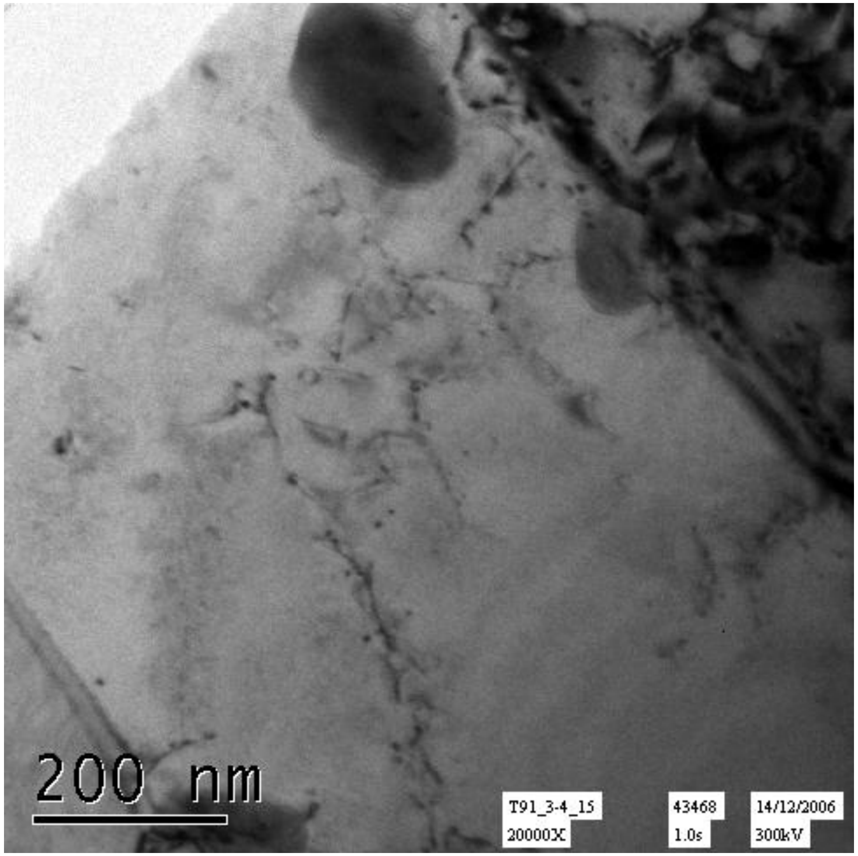




TEM-Loops MIRE irradiation-BR2 T_{irr}= 300 ^{0}C

Dose 0.6 & 1.0 dpa





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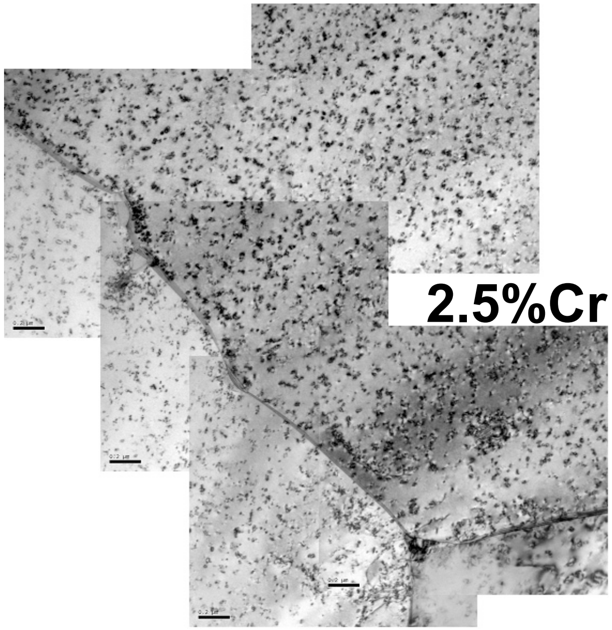
Homogeneous versus heterogeneous loop distribution

- T91 MEGAPIE : heterogeneous (low dose) homogeneous (high dose) loop distribution
- Heterogeneous loop distribution seems to be a common feature of neutron irradiated FM steels

What is causing the homogeneous loop distribution?

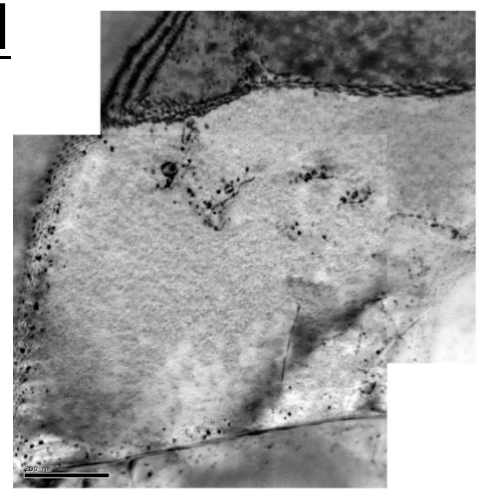
Homogeneous versus heterogeneous loop distribution

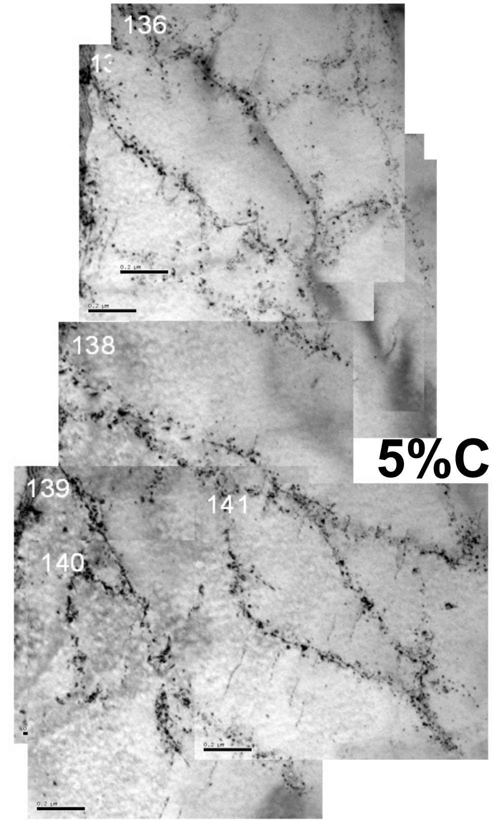
TEM



2.5%Cr

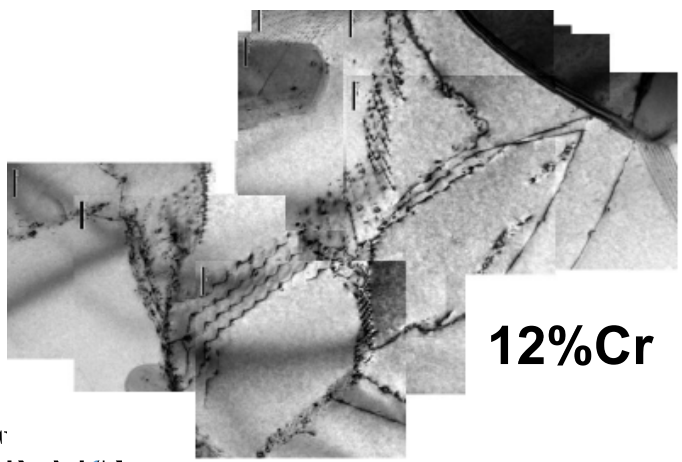
9%Cr





5%Cr

0.6 dpa



12%Cr

2.5%Cr homogeneous loops distribution!
5-12%Cr heterogeneous loop distribution

M. Hernández Mayoral, et al. JNM 474 88 (2016)

M. J. Konstantinovic et al Phys. Status Solidi A, 1–7 (2016)

Summary of TEM results

	Fe2.5Cr MIRE		Fe9Cr MIRE		T91 MIRE		T91 MEGAPIE	T91 MEGAPIE
Dose (dpa)	0.6	1.0	0.6	1.0	0.6	1.0	0.88	4.35
Spatial distribution of loops	**homogeneous**	**homogeneous**	heterogeneous	heterogeneous	heterogeneous	heterogeneous	heterogeneous	**homogeneous**
Average loop diameter (nm)	13 (12.1*)	16	7 (5.3*)	13	7	10	4.8	12.2
Number density of loops (x $10^{21} m^{-3}$)	5.5*		2.2*				?	20
Average void size (nm)	No(?)	1-2	No	No	No	No	No	1.6

Matijasevic et al., JNM 377 (2008) 147
* M. Hernández-Mayoral et al., JNM 474 (2016) 88

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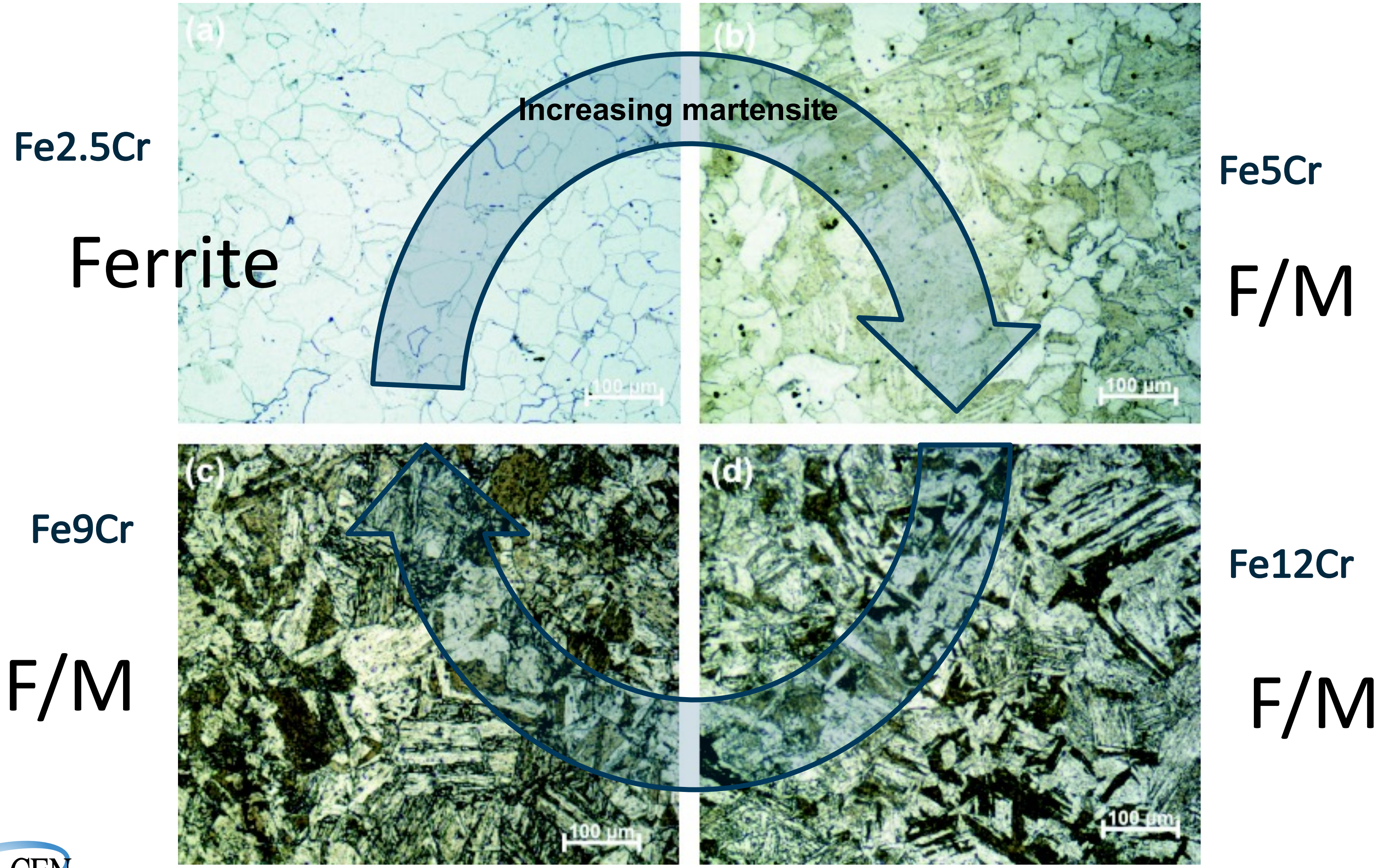
FeCrC alloys initial microstructure



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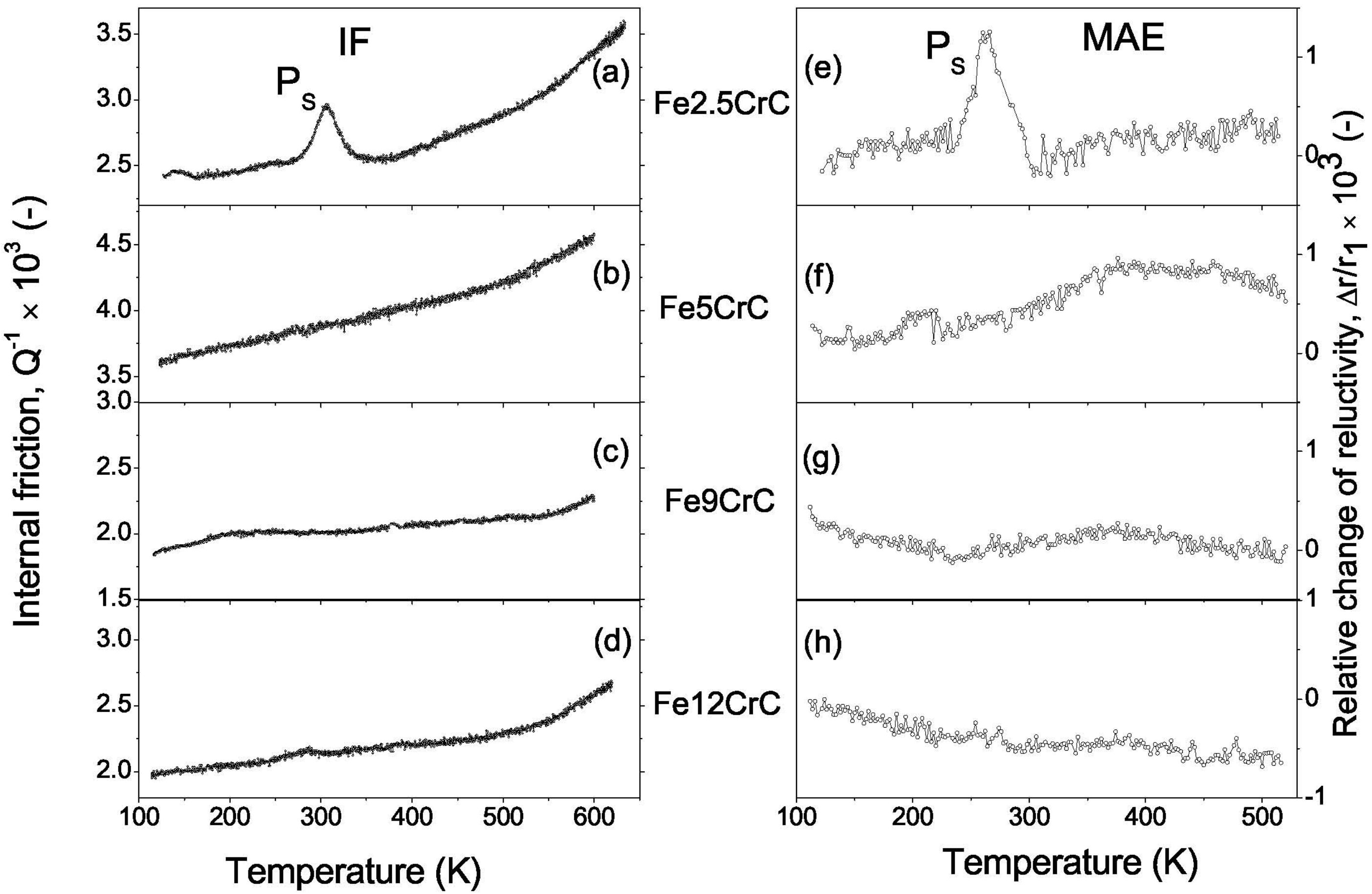
Carbon distribution in initial structure – FeCrC alloys



IF & MAE - Only in 2.5%Cr C atoms are uniformly distributed in the matrix

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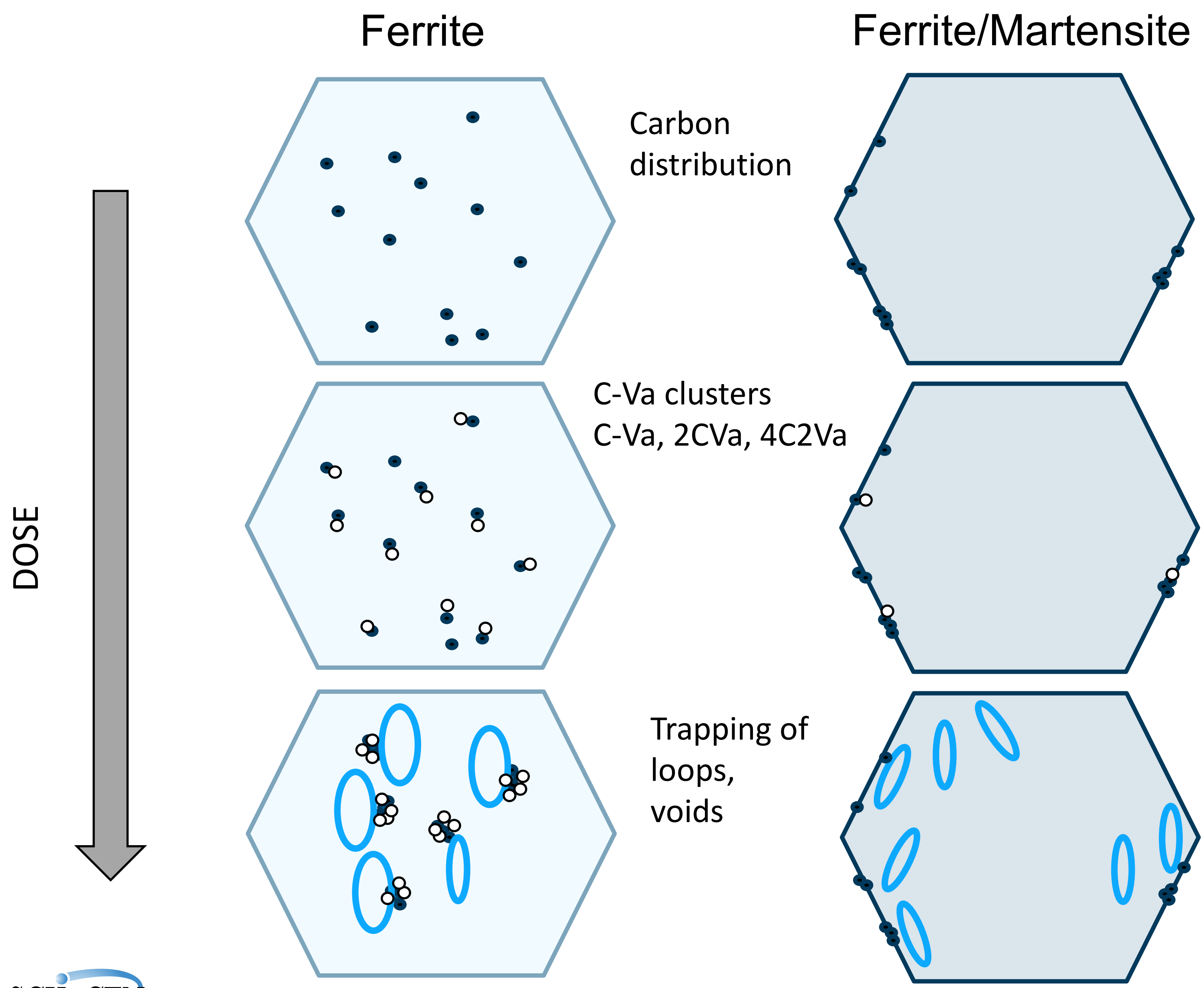

Ferrite
Ferrite/Martensite
Carbon
distribution
C-Va clusters
C-Va, 2CVa, 4C2Va
Trapping of
loops,
voids
DOSE

- The appearance of voids in the TEM spectra seems to be associated with homogeneous loop distribution?

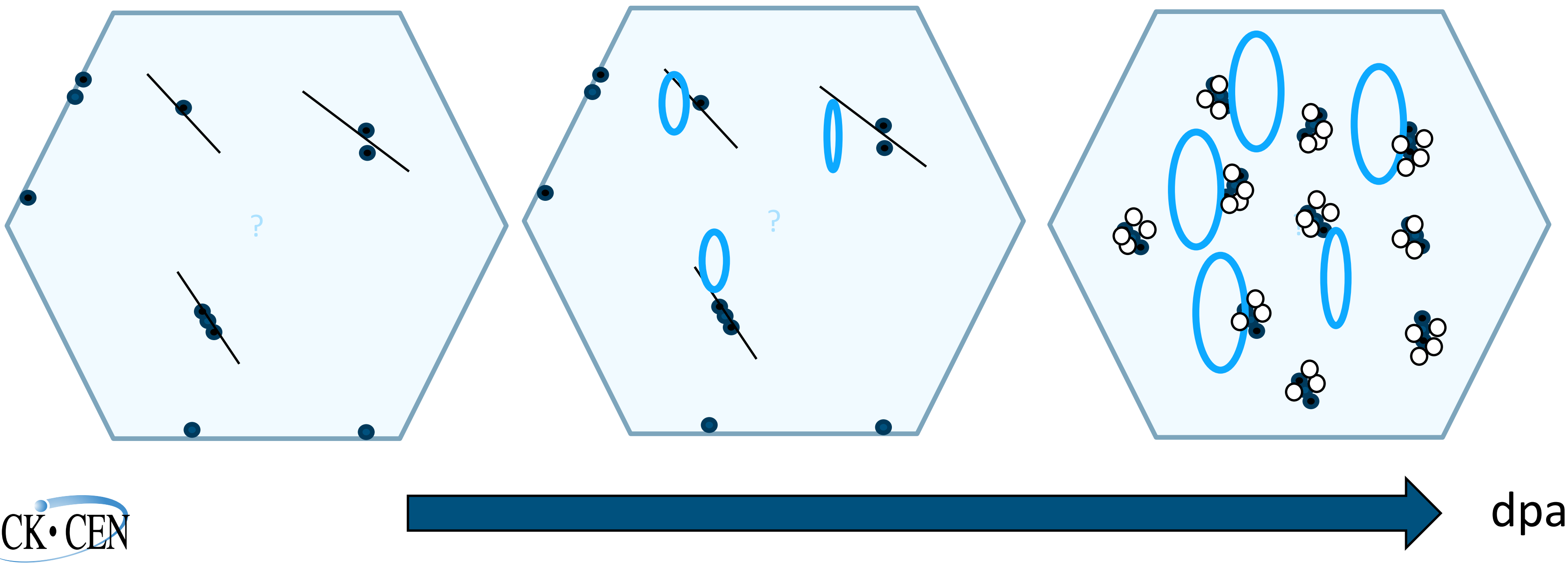
Reduction of swelling in FM alloys ⟺ heterogeneous loop distribution

Do they have the same origin?

Formation of small VC clusters - act as pinning points for both vacancy and interstitial clusters allowing them to grow

T91- high dose. What is the origin of homogeneous loop distribution?

1. Loop type
2. Solute clusters & precipitates
3. Carbon distribution
4. Dislocation density



Modeling results: OKMC parameterization

Types of defects:

1. Point defects: I, V
2. 3D-clusters: I_N (N=2-7), V_N
3. 1D clusters: <111> & <100>
4. Carbon – "C"
5. C_N-V_M
6. C-$<111>_N$
7. 2C-$<111>_N$

Types of reactions:

1. $I_N + V_M$ = recombination
2. I+I; V+V = mobile clusters
3. V + C = immobile clusters
4. <111> + C_N-V_M = immobile clusters + recombination
5. <111> + <111> = <111>/<100>
6. <111> + <100> = <111>/<100>
7. <111>/<100>/C-<111> + V_N/I_N = recombination/growth

Mobile objects	Immobile objects
I_1 – I_4: 3D	> V_5
V_1 – V_5 : 3D	V-C_2
Carbon interstitial: 3D	C-loop: traping energy 1eV
$\frac{1}{2}$<111> loops : 1D	2C-loop: traping energy 2 eV
<100> loops: 1D (or immobile)	

1. Description of I, V, I_N and V_N : Fu et al. Nature 4 (2005) 68
2. V_N-C_M interaction : D. Terentyev et al. J. Phys. Cond. Matter 24 (2012) 385401
3. Carbon-<111> interaction: D. Terentyev et al. J. Nucl. Mater. 408 (2011) 272.
4. ½<111> Loop mobility: Osetsky Phil. Mag. 83 (2003) 61
5. Loop – loop interaction: Phys. Rev. Lett. 110 (2013) 265503

Modular Monte Carlo MMonCa

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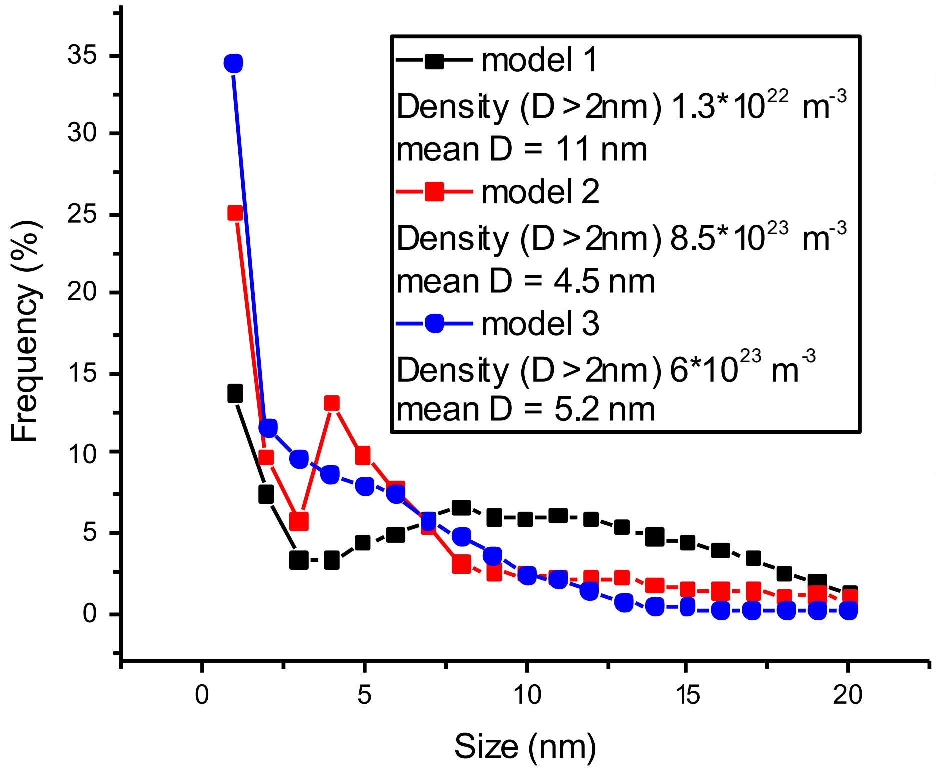
Modeling results: OKMC parameterization

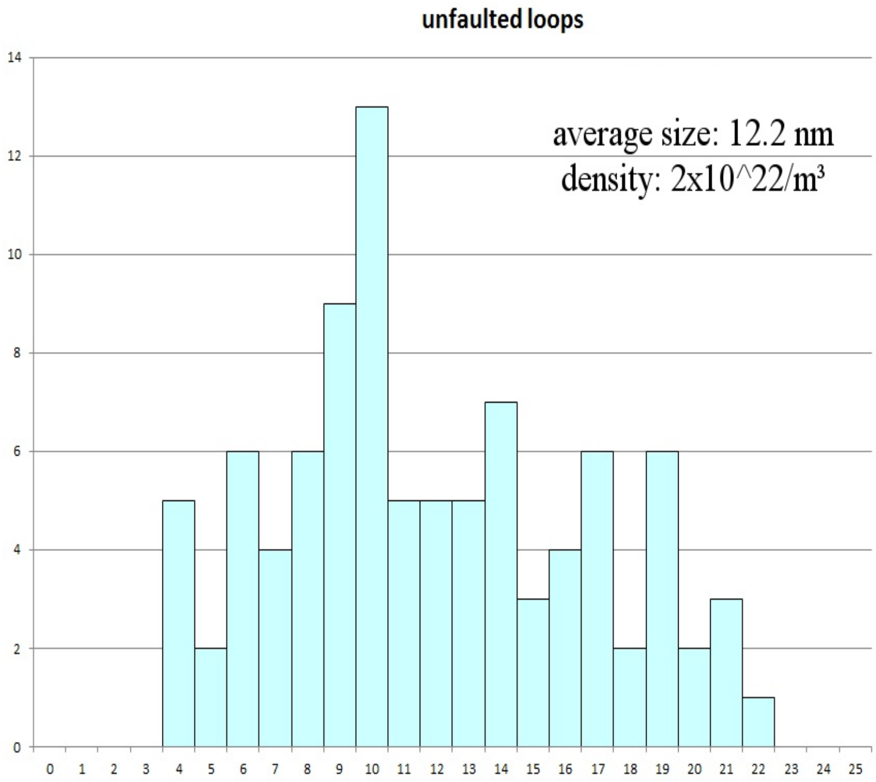
Three models are proposed to narrow down principle mechanisms responsible for microstructural evolution

- Model 1
  - **Loop+Loop = Transformation into <100> or <111> is thermally activated reaction**
  - **Binding energy Loop-C = 1 eV; Loop-2C = 2 eV & being mobile**
  - **Migration energy of <100> loops is 1 eV**
- Model 2
  - **<100> loops are immobile**
- Model 3
  - **Loop-2C & Loop-C are immobile**

TEM-Loops

Sample: H-01-13-A, Dose 4.35 dpa, T_{irr}= 320-350 ^{0}C





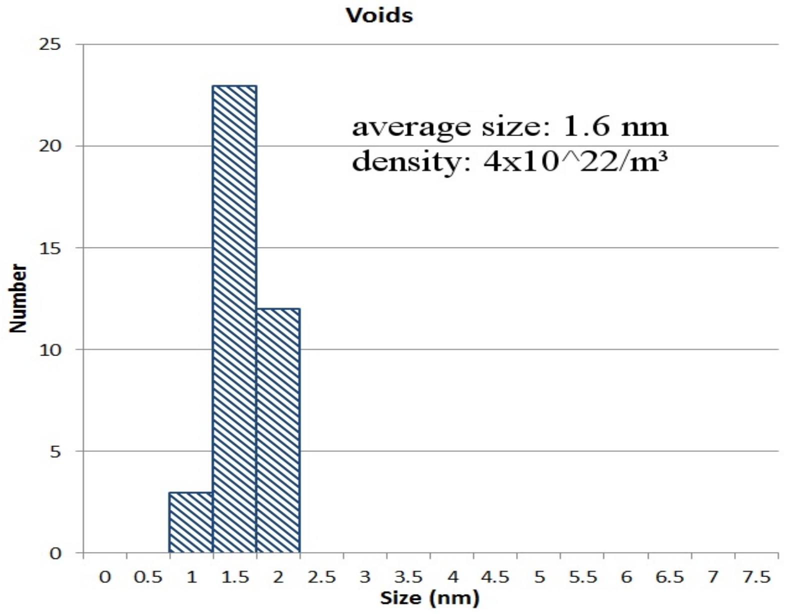
- Model 1 gives best agreement
- Model 2 & Model 3 do not predict correctly both mean loop size and density
- Large density of small loops represent TEM invisible loops

TEM-Voids

H-01-13-A

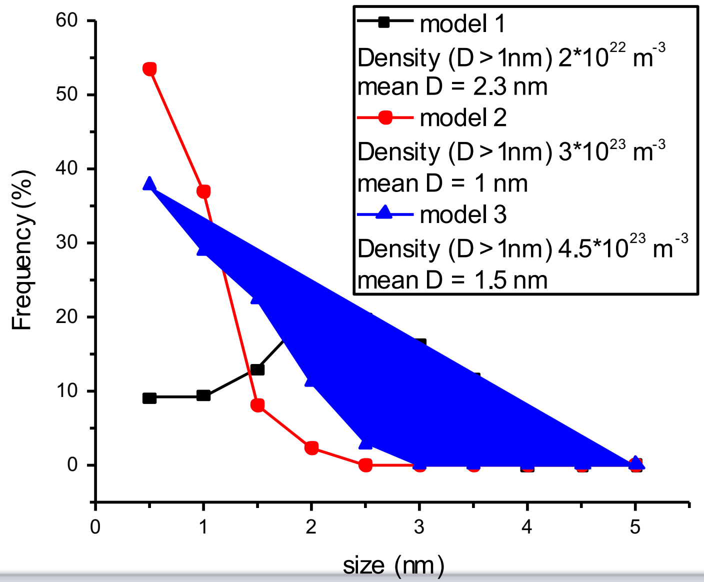
Dose 4.35 dpa

T_{irr}= 320-350 ^{0}C



- Model 1 gives reasonable agreement

- Model 2 & Model 3 essentially overestimate the density of voids



Modeling results

- Results at 0.88 and 4.35 dpa at 335 ^{0}C (taken as average) can be reproduced if
  - ✓ One assigns diffusion to <100> loops with Em = 1 eV
  - ✓ One assigns loop-loop reaction as 111+111 = 100; assuming that 111+111 = 111, does not reproduce the emergence of loop density
  - ✓ One account for C_2-V trapping with binding energy of 2 eV
    - ✓ 1 eV does not give a good trend
  - ✓ Loops that sunk at GB and dislocation lines remain trapped there (i.e. disappear from OKMC treatment)
  - ✓ C-V trapping is the must to observe voids
  - ✓ Key to observe loops: to include Carbon trapping & reduce the pre-factor of diffusion coefficient with loops size as $\sim N^{-0.64}$

Conclusions

- TEM is performed on MEGAPIE samples
- Heterogeneous and homogeneous distribution of dislocation loops are observed in low (0.88 dpa) and high (4.35 dpa) dose samples, respectively
- Visible voids appear together with the occurrence of homogeneous loop distribution
- The results are compared with FeCr alloys indicating that Carbon distribution plays important role for dislocation loop distribution.
- Object kinetic Monte Carlo modeling indicates:
  - Best agreement with experimental data is obtained assuming that carbon-vacancy clusters pin loops strongly reducing their diffusion
  - Delay in the formation of large loops inside grains is due carbon being distributed at the dislocations and grain boundaries as well as due to strong sink strength offered by high dislocation density
  - Once dislocations are removed, carbon is released back to the matrix. Formation of carbon-vacancy clusters and defect trapping allowing them to grow.

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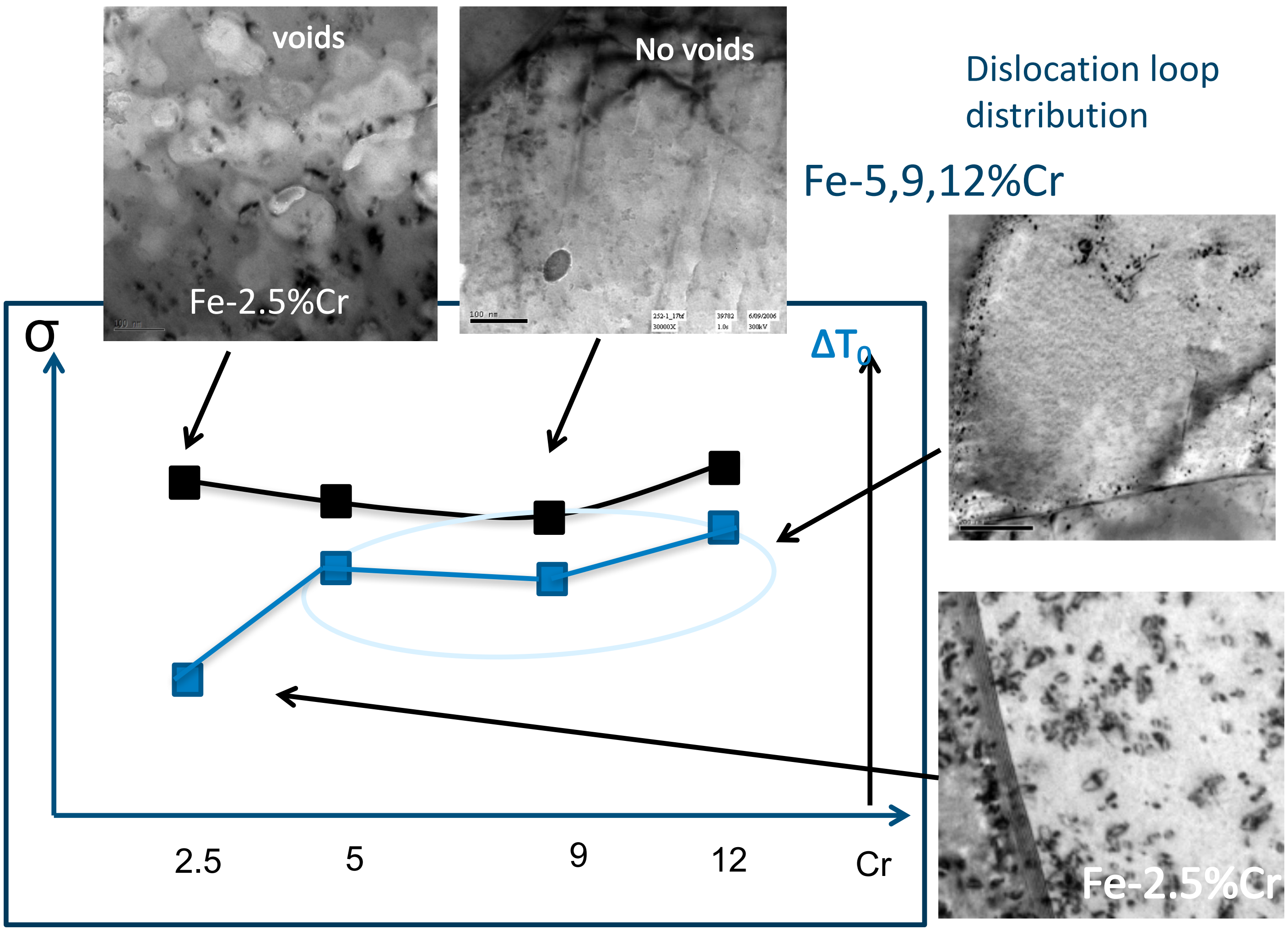
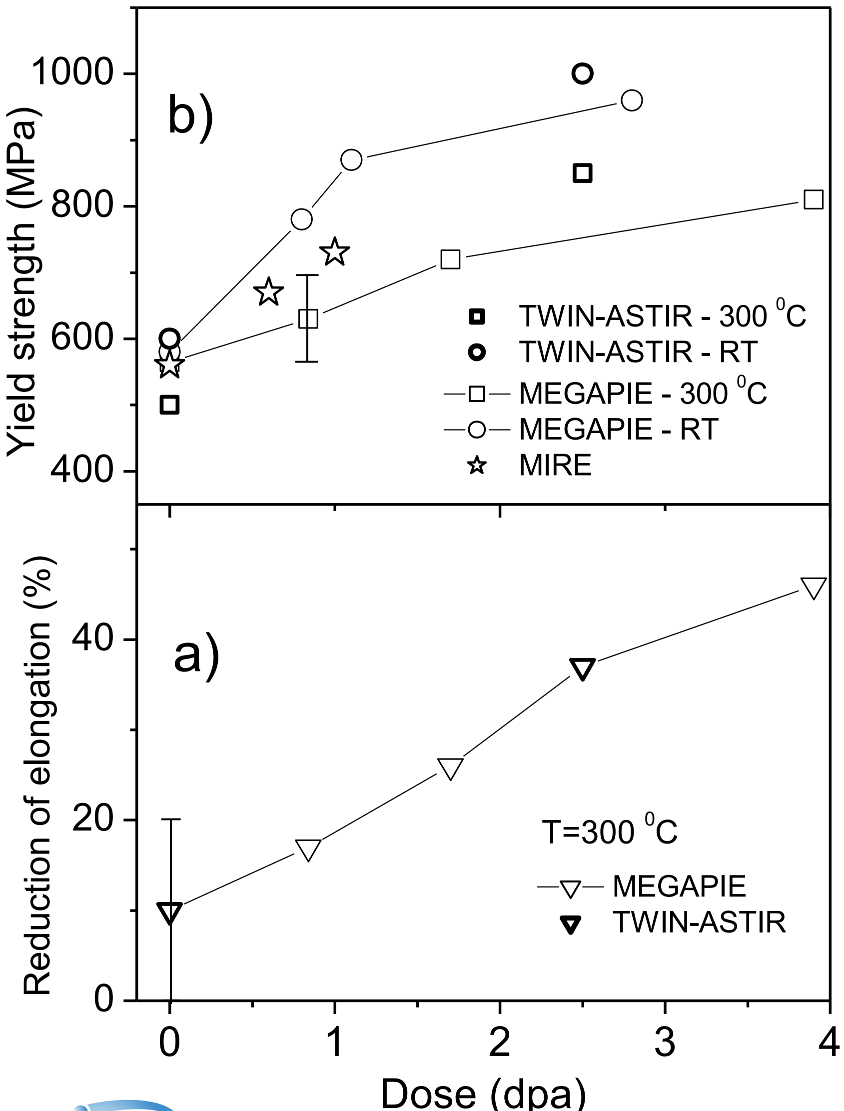
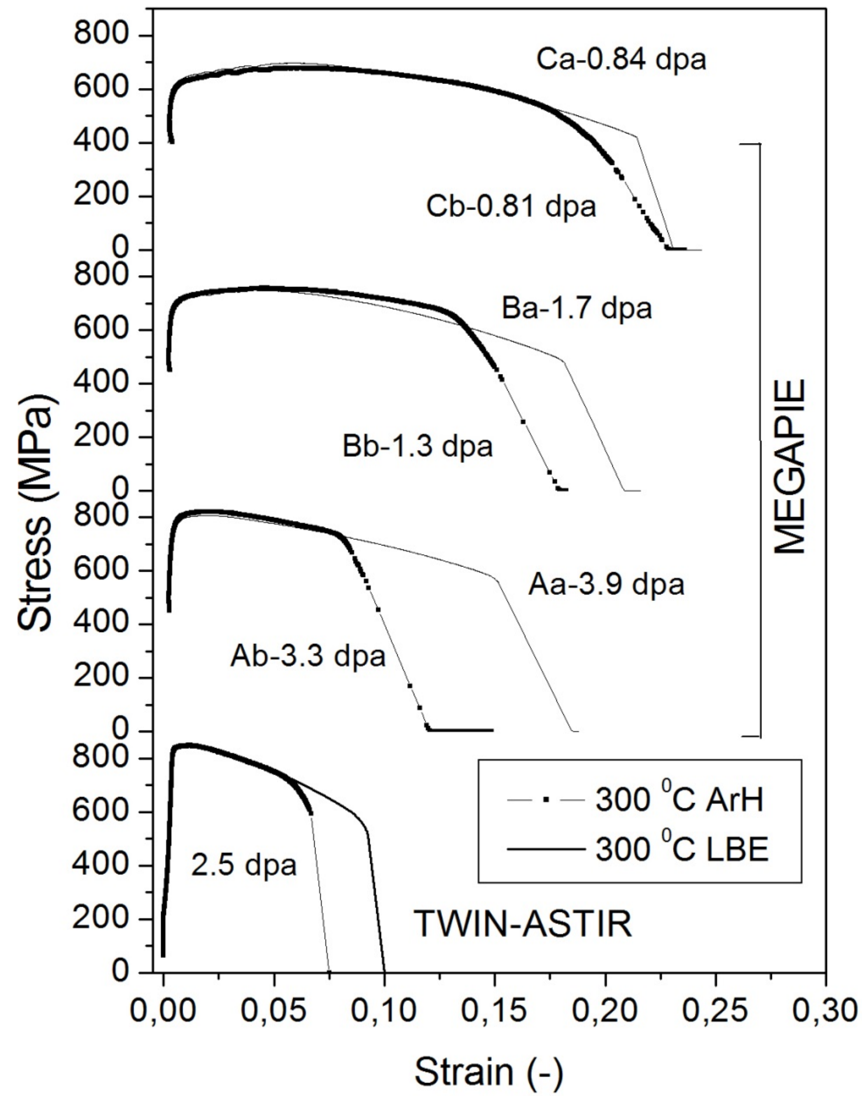
Defect properties of neutron irradiated FeCrC alloys



M. J. Konstantinovic et al., Physica Status Solidi A, 2016

Summary of the tensile tests





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