Heat Treatment Effects on Precipitation in Irradiated HT9 Steel

T.M. Kelsy Green[1], Li He[2], Todd Allen[1]

[1]University of Michigan-Ann Arbor
[2]University of Wisconsin-Madison

Materials in Nuclear Energy Systems
October 7, 2019
Baltimore, Maryland

NUCLEAR ENGINEERING & RADIOLOGICAL SCIENCES
UNIVERSITY OF MICHIGAN

HT9 Properties and Performance for Advanced Reactors

- 12Cr-1Mo ferritic/martensitic (F/M) steel with minor solute additions
- Pros: high-dose resistance to void swelling, high thermal conductivity, high strength up to ~500℃
- Cons: prone to radiation-induced embrittlement from the secondary phase α'

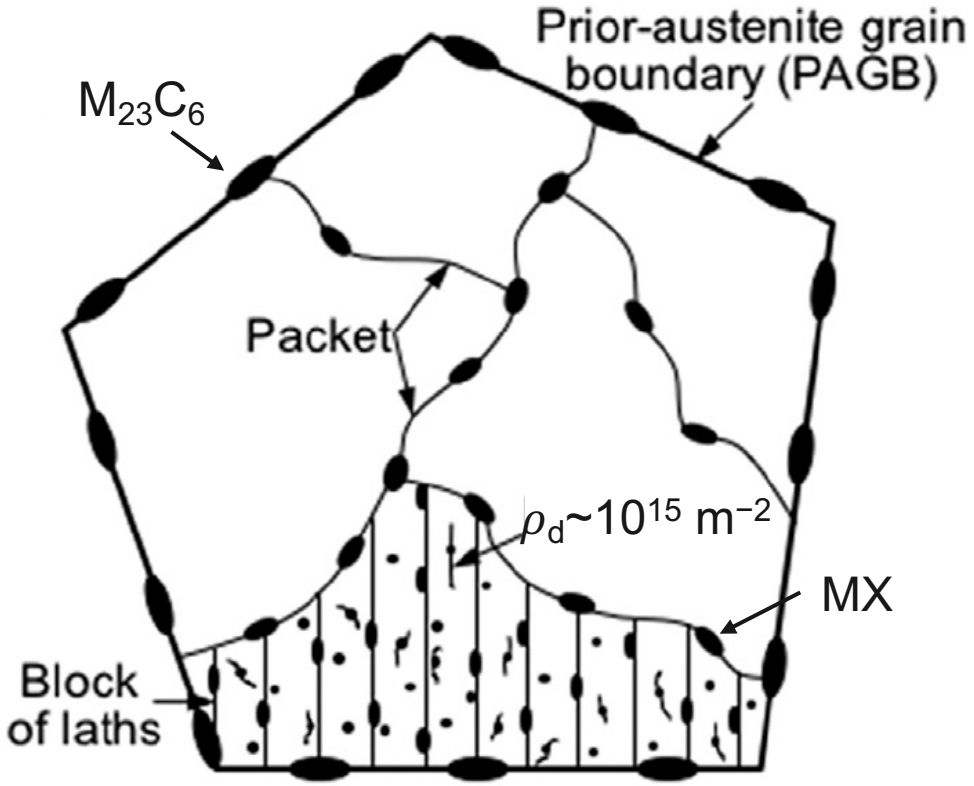


Figure 1. Microstructure of normalized and tempered HT9, taken from [2].

[1] Ronald L. Klueh and Donald R. Harries / High Chromium F/M Steels for Nuclear Applications
[2] B.H. Sencer et al. / Journal of Nuclear Materials 393 (2009) 235–241
[3] L. Tan et al./ Journal of Nuclear Materials 493 (2017) 12-20

Heat-to-Heat Variability of HT9

- 12Cr-1MoVW steel samples with two different heat treatments were irradiated in EBRI-II

HT1	1038℃ / 0.08hr / AC + 760 ℃ / 0.5hr / AC
HT2	1038℃ / 0.5hr / AC + 760 ℃ / 2.5hr / AC

- Mechanical properties of HT9 change rapidly as a function of temperature between 400-450°C with properties varying as a function of processing conditions

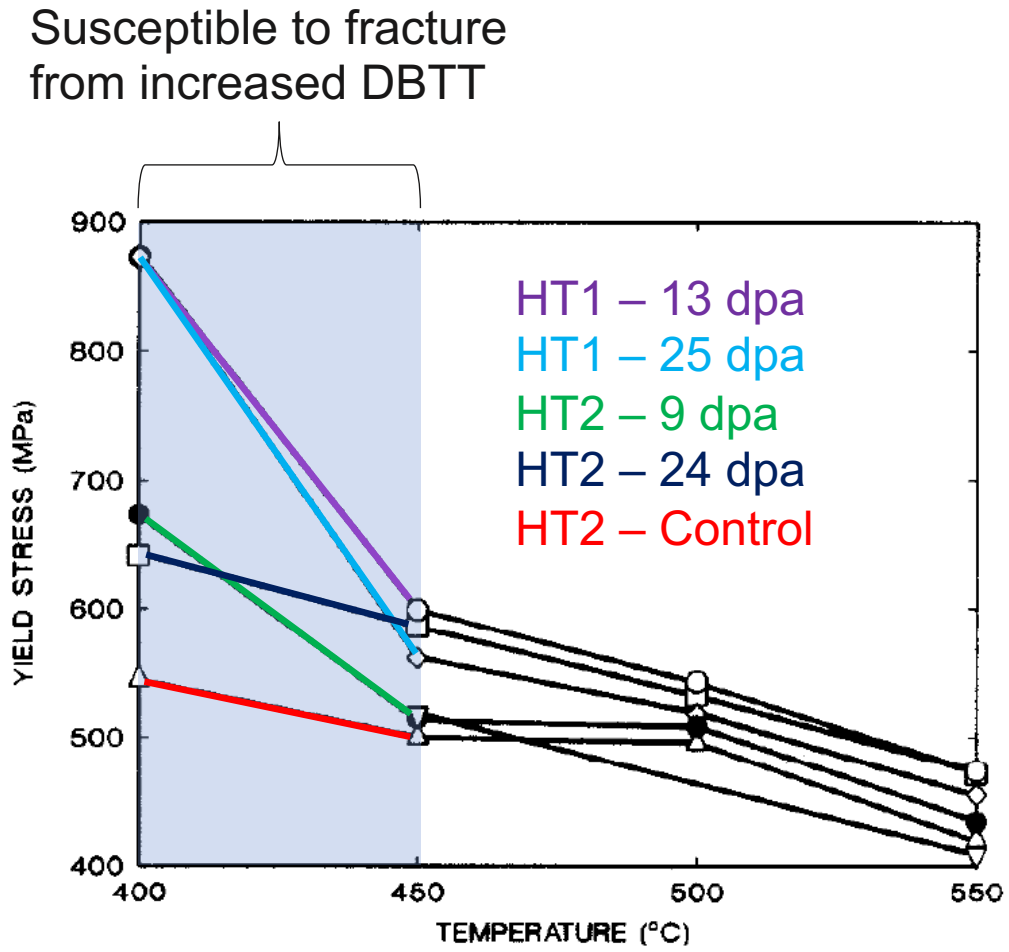
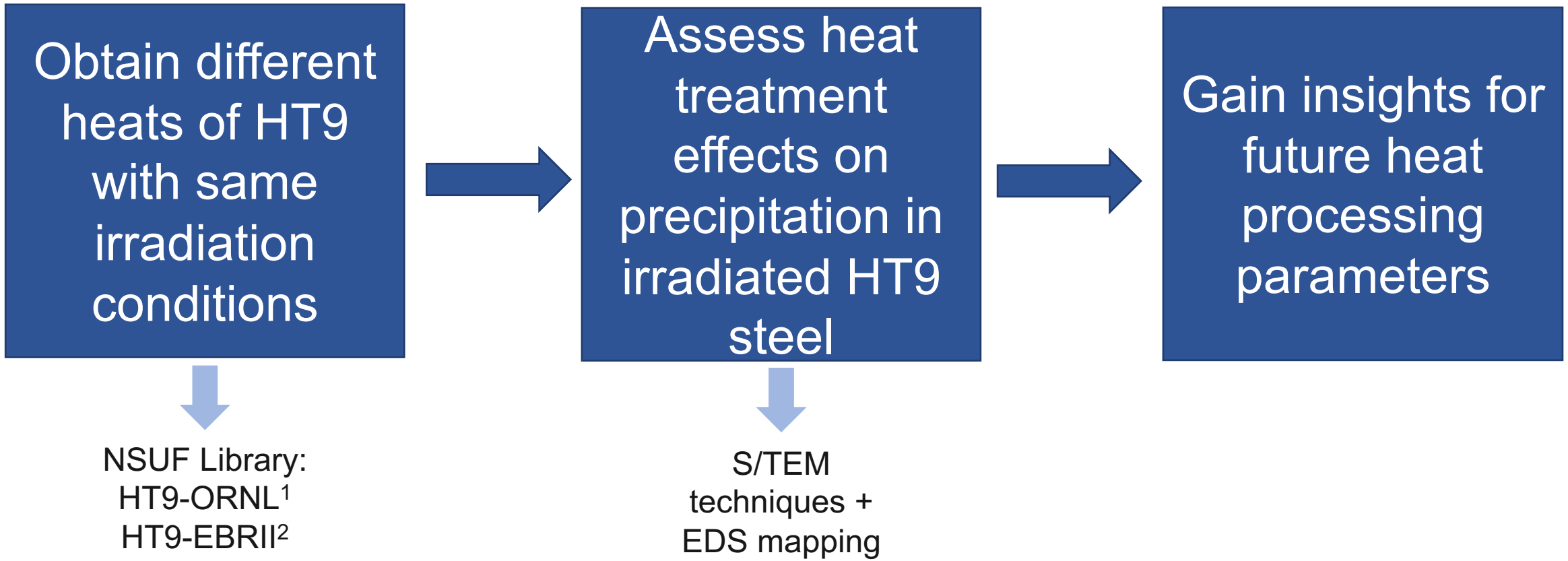


Figure 2. Comparison of the yield stress behavior of 12Cr-1MoVW steel given two different heat treatments [1]. Temperature of tensile tests were conducted at $T_{irradation}$.

[1] R.L. Klueh and J.M. Vitek / Journal of Nuclear Materials 182 (1991) 230-239
[2] Porter, D. L. , Fuel Test Plan for ALMR, June 23, 1994.

To better understand the heat-to-heat variability of HT9's radiation response in the low dose regime from 400-450℃



NSUF Library Identification: [1] KGT 1755, ID 113-08-331, [2] KGT 1776, ID 112-08-331, [3] KGT 1728, ID 111-08-331

Three variables: composition, normalization, and tempering

HT9-EBRII

Cr	11.92
Mo	1.01
W	1.04
Ni	0.92
Mn	0.76
Si	0.39
V	0.22
C	0.22
N	?
Cu	-
Other	-

Normalization: 1040℃, 1 hour
Tempering: 740℃, 0.75 hour

HT9-ORNL

Cr	11.63
Mo	1
W	0.52
Ni	0.5
Mn	0.52
Si	0.22
V	0.3
C	0.2
N	0.047
Cu	0.04
Other	Al,P,S,Ti,Co

Normalization: 1040℃, 0.5 hour
Tempering: 760℃, 1 hour

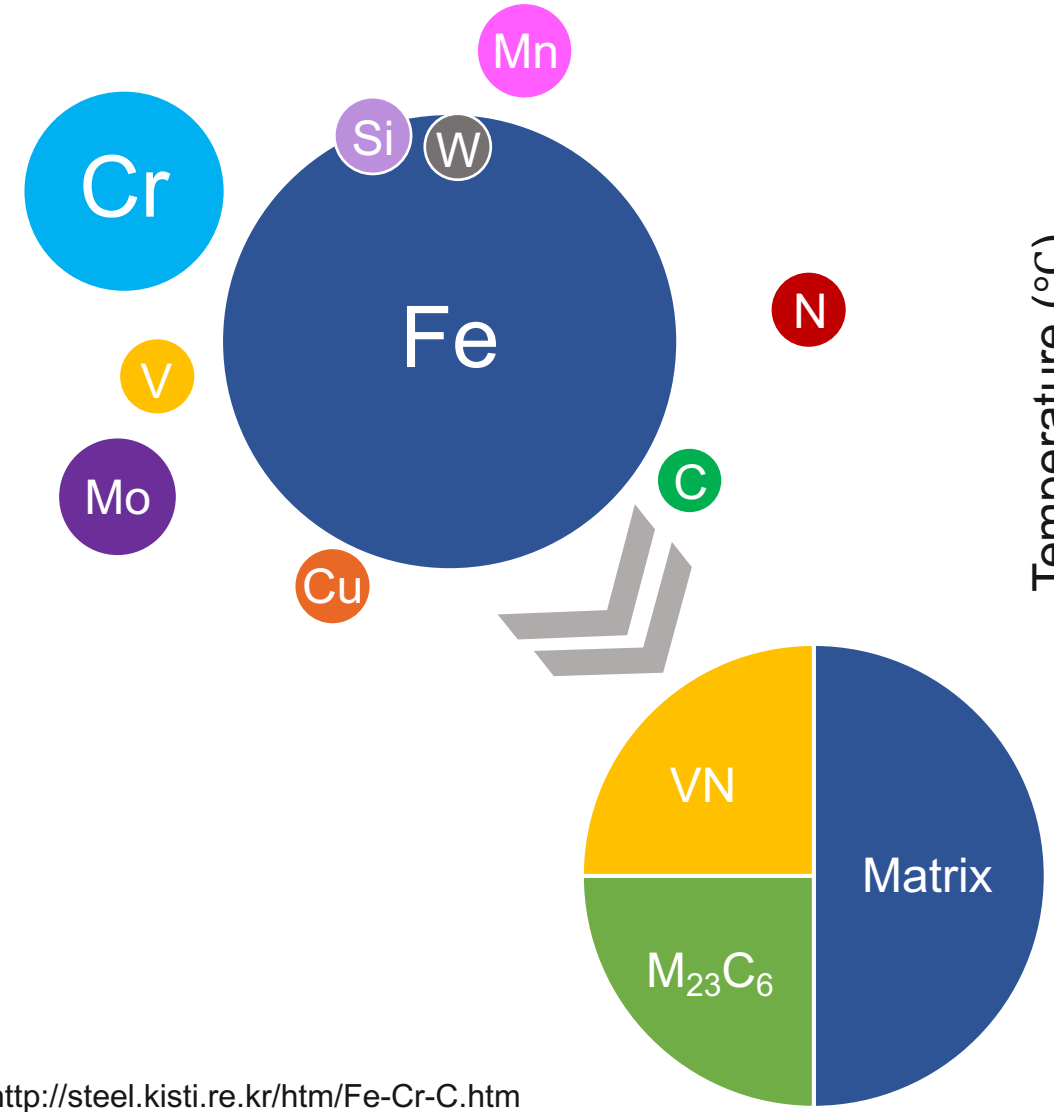
HT9-LANL

Cr	12.1
Mo	0.97
W	0.58
Ni	0.59
Mn	0.61
Si	0.41
V	0.34
C	0.171
N	0.031
Cu	0.025
Other	O,Al,P,S,Ti,Co

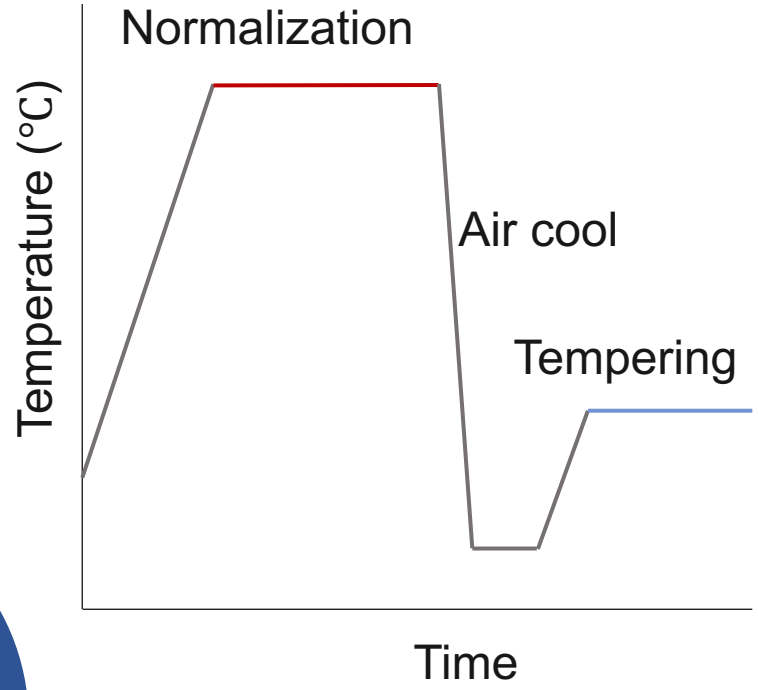
Normalization: 1060℃, 1 hour
Tempering: 730℃, 2 hour

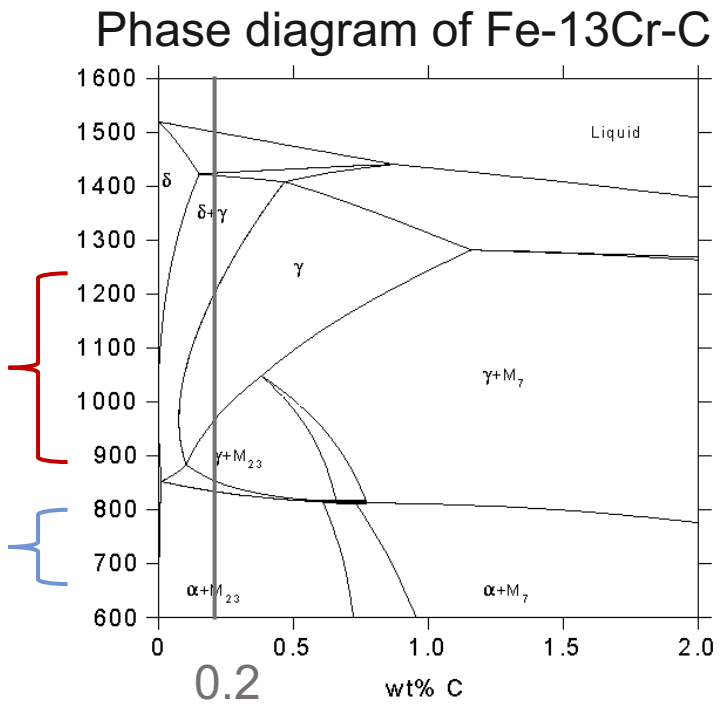
Heat-Treated Microstructure

Composition



Normalization & Tempering





http://steel.kisti.re.kr/htm/Fe-Cr-C.htm

All samples received the same irradiation history

Facility

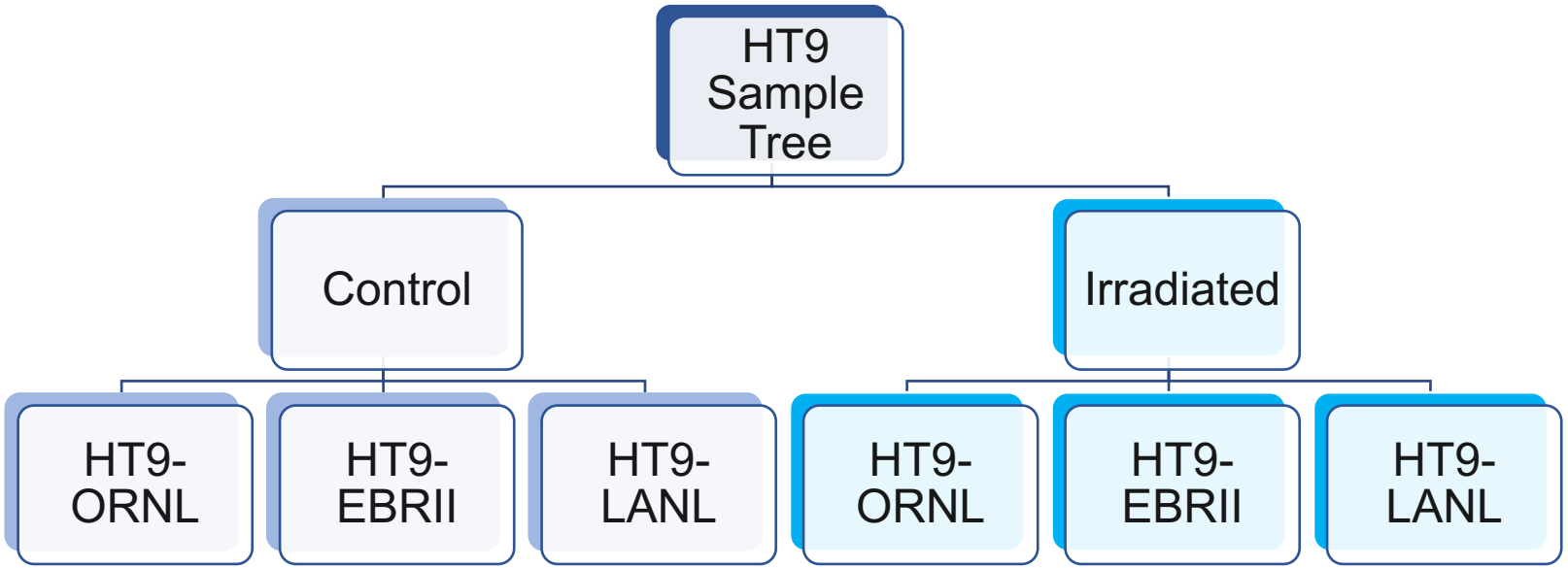
Advanced Test Reactor (ATR)

Location

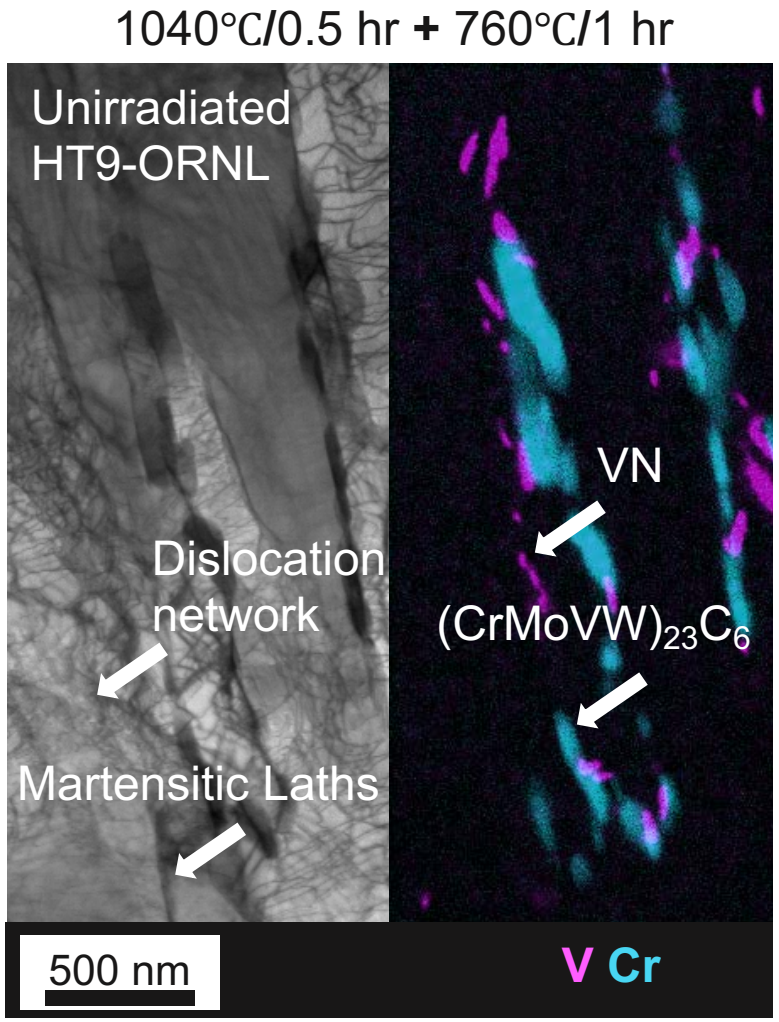
side-by-side in the East Flux Trap

Irradiation Parameters

430°C
8 dpa
5×10^{14} n/cm^{2}s
~10^{-7} dpa/s



Heat-Treated Microstructure



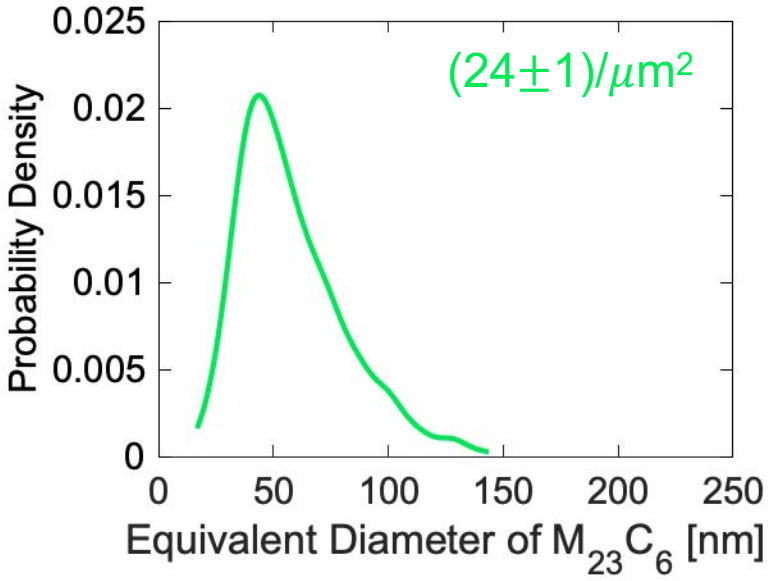
- $M_{23}C_6$ carbides distributed on grain boundaries
- MX nitrides distributed on and within grain boundaries
- Inhomogeneous distribution of precipitates in the microstructure

Figure 2. STEM BF micrograph and corresponding EDS map showing the qualitatively typical lath and dislocation network present in all samples.

Heat-Treated $M_{23}C_6$ Size Distributions

Control HT9-EBRII

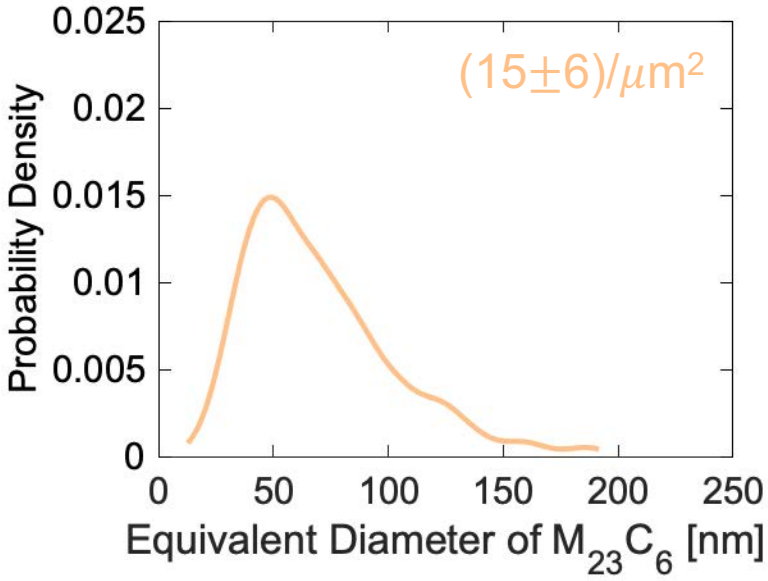
1040°C/1 hr + 740°C/0.75 hr



Mean = 58±24 nm
N = 606

Control HT9-ORNL

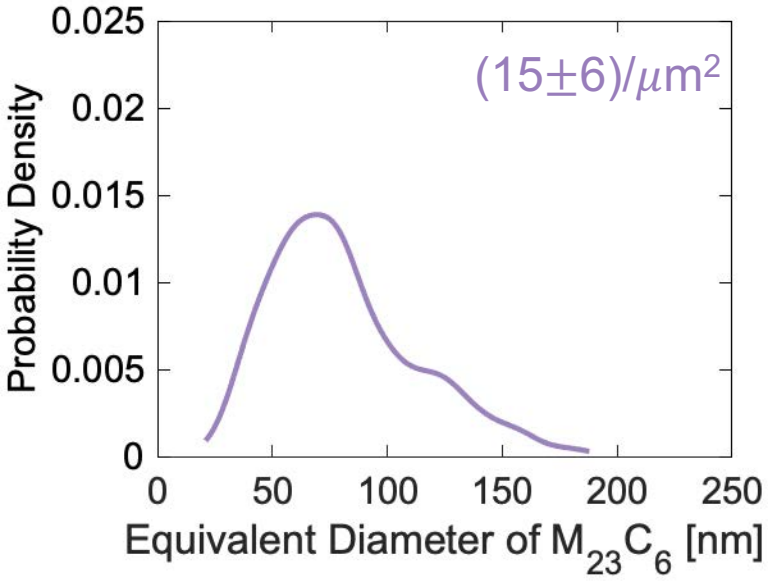
1040°C/0.5 hr + 760°C/1 hr



Mean = 70±33 nm
N = 541

Control HT9-LANL

1060°C/1 hr + 730°C/2 hr



Mean = 81±32 nm
N = 494

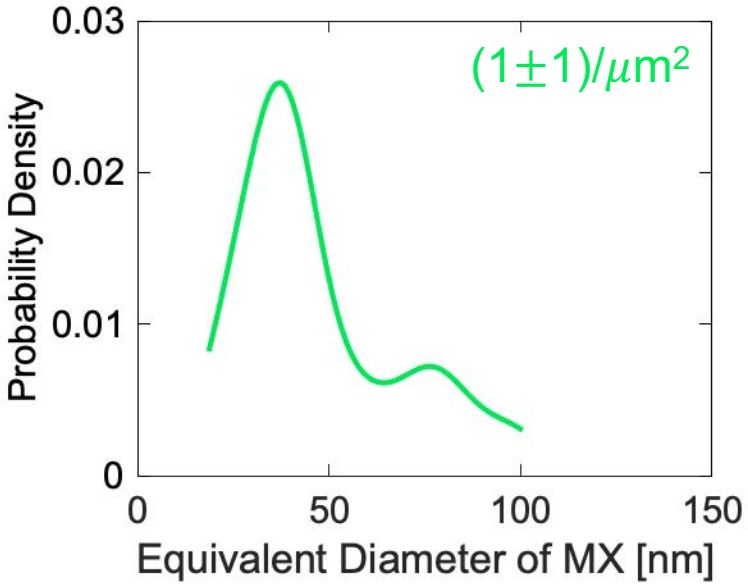
Increasing Tempering Time & Increasing Mean Size

Reported error is sample standard deviation

Heat-Treated VN Size Distributions

Control HT9-EBRII

1040°C/1 hr + 740°C/0.75 hr

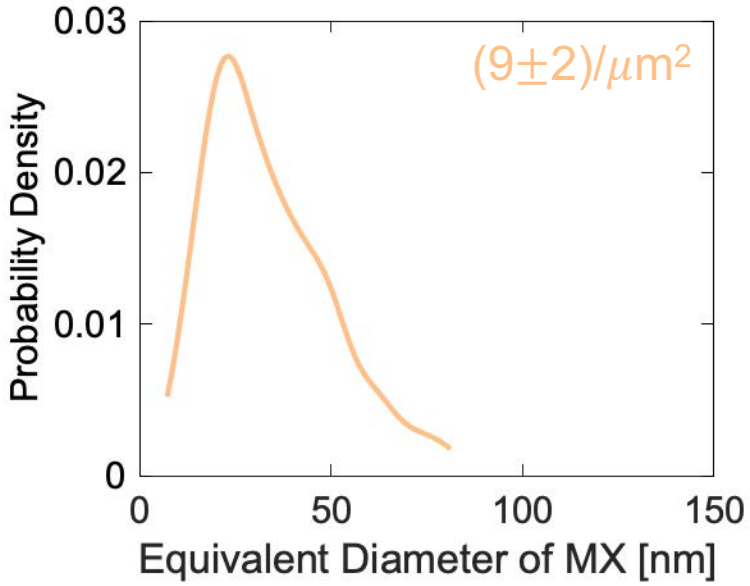


Mean = 48±22 nm
N = 30

0.22wt% V
? wt% N
0.22wt% C

Control HT9-ORNL

1040°C/0.5 hr + 760°C/1 hr

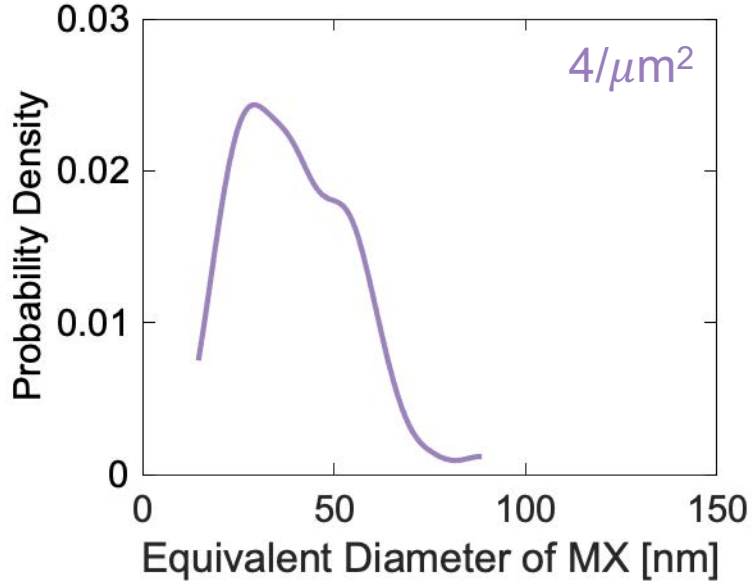


Mean = 34±16 nm
N = 323

0.30wt% V
0.047wt% N
0.20wt% C

Control HT9-LANL

1060°C/1 hr + 730°C/2 hr



Mean = 40±15 nm
N = 134

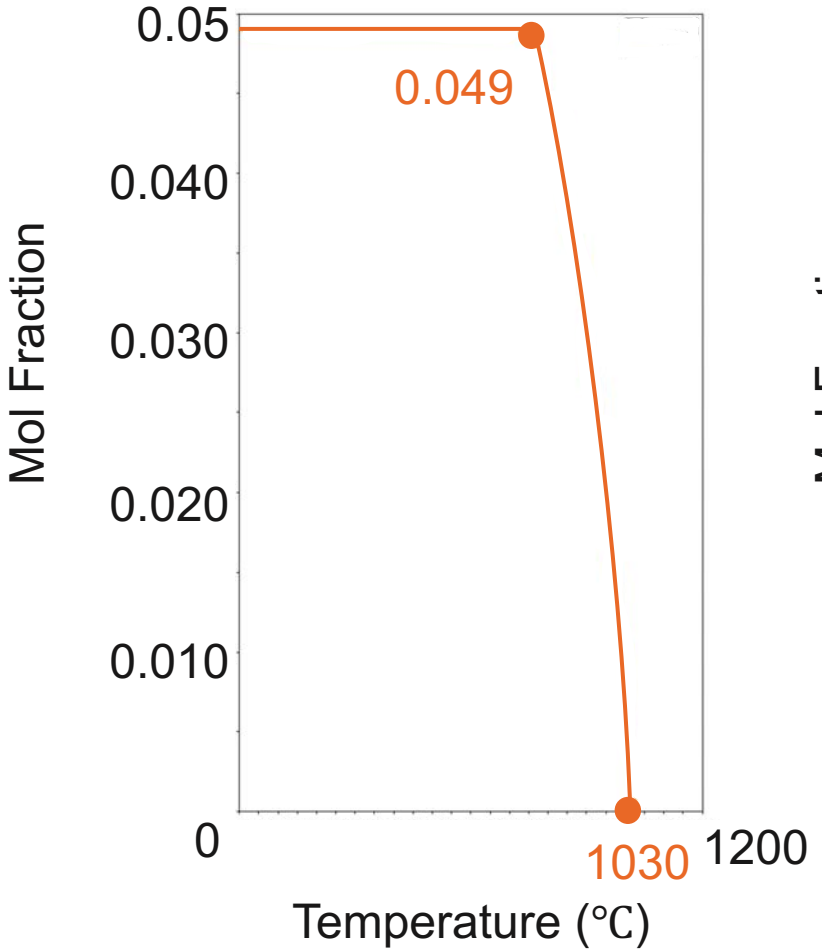
0.34wt% V
0.031wt% N
0.171wt% C

$M_{23}C_6$ Phase Fraction Diagrams

Carbides fully dissolved during normalization in all samples

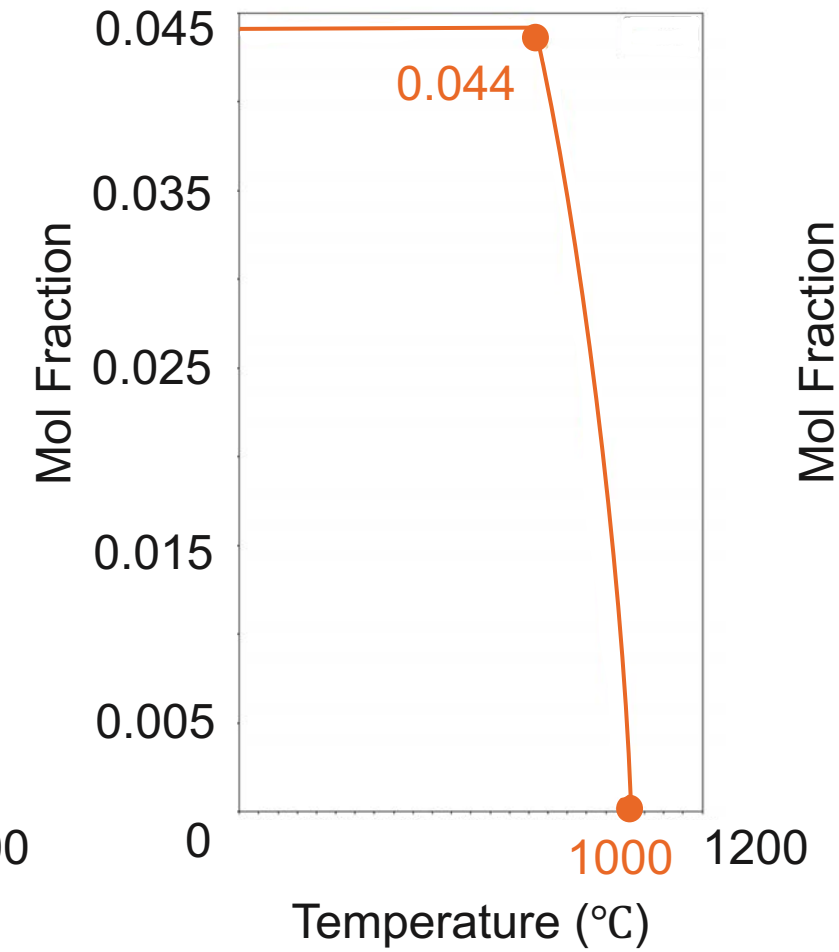
Control HT9-EBRII

1040℃/1 hr + 740℃/0.75 hr



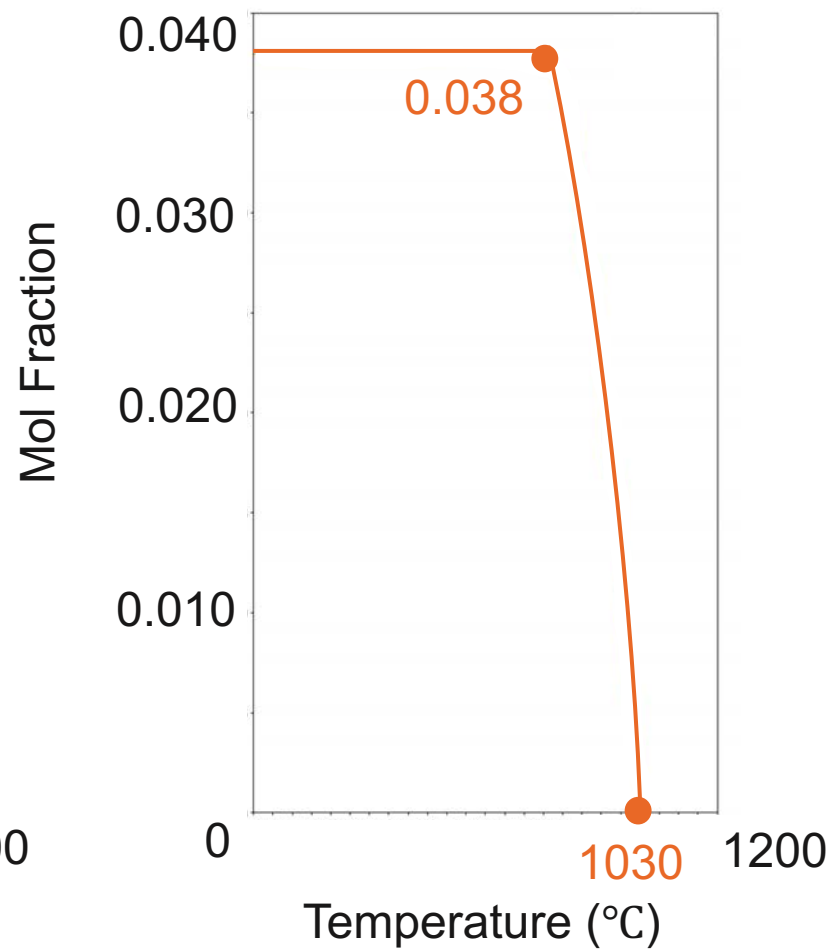
Control HT9-ORNL

1040℃/0.5 hr + 760℃/1 hr



Control HT9-LANL

1060℃/1 hr + 730℃/2 hr

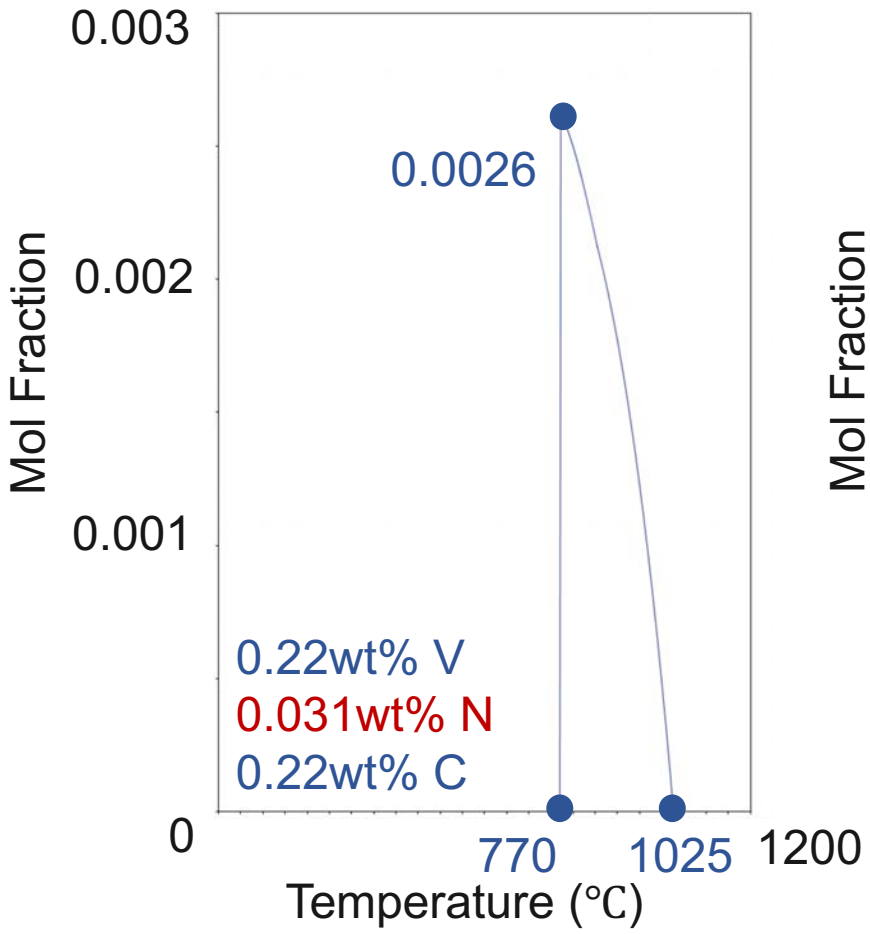


ThermoCalc

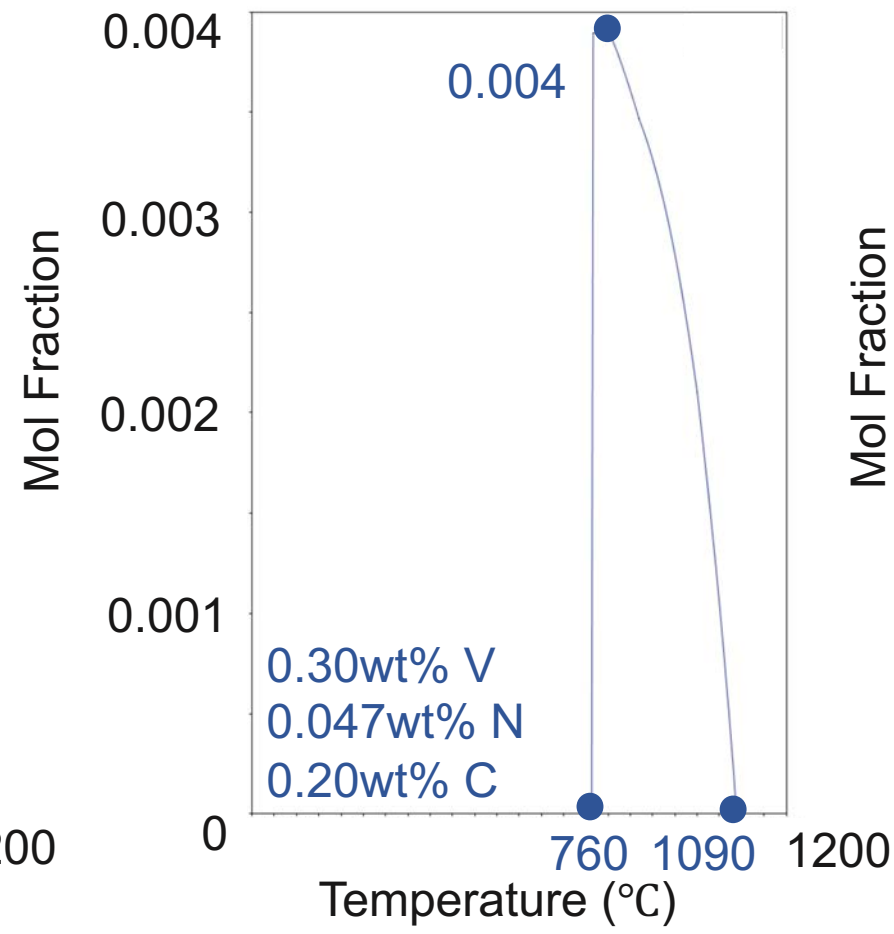
VN Phase Fraction Diagrams

N was key for increasing VN phase fraction

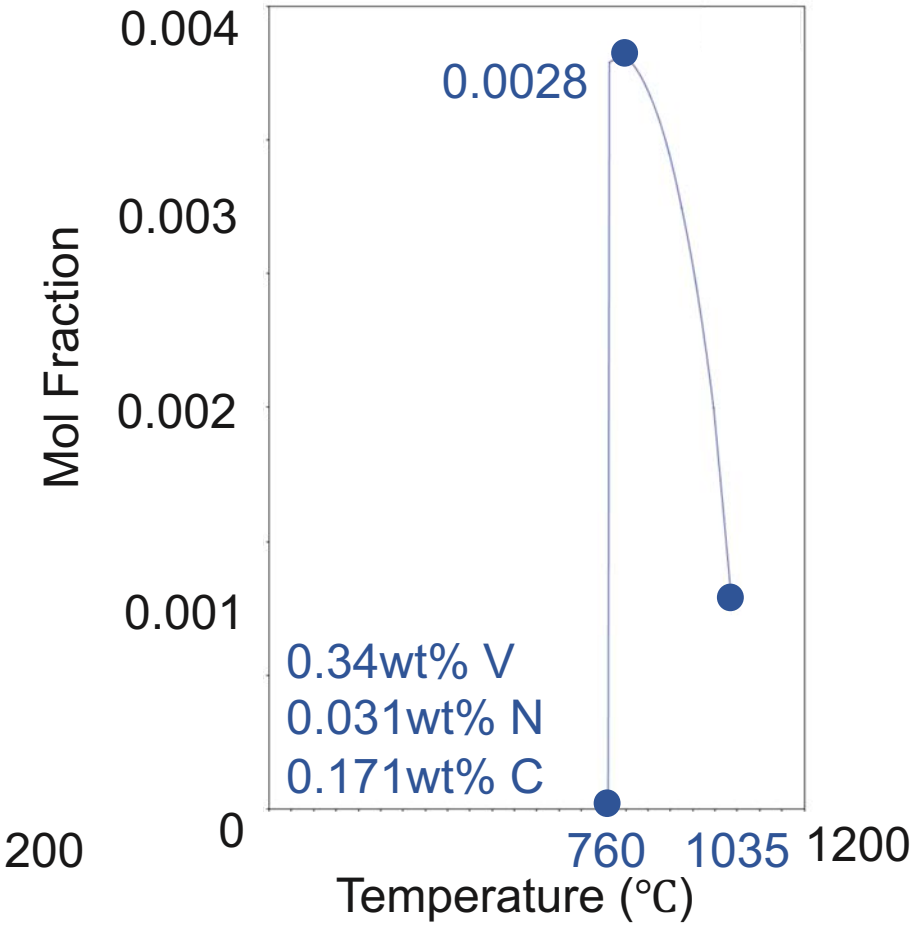
Control HT9-EBRII
1040℃/1 hr + 740℃/0.75 hr



Control HT9-ORNL
1040℃/0.5 hr + 760℃/1 hr



Control HT9-LANL
1060℃/1 hr + 730℃/2 hr

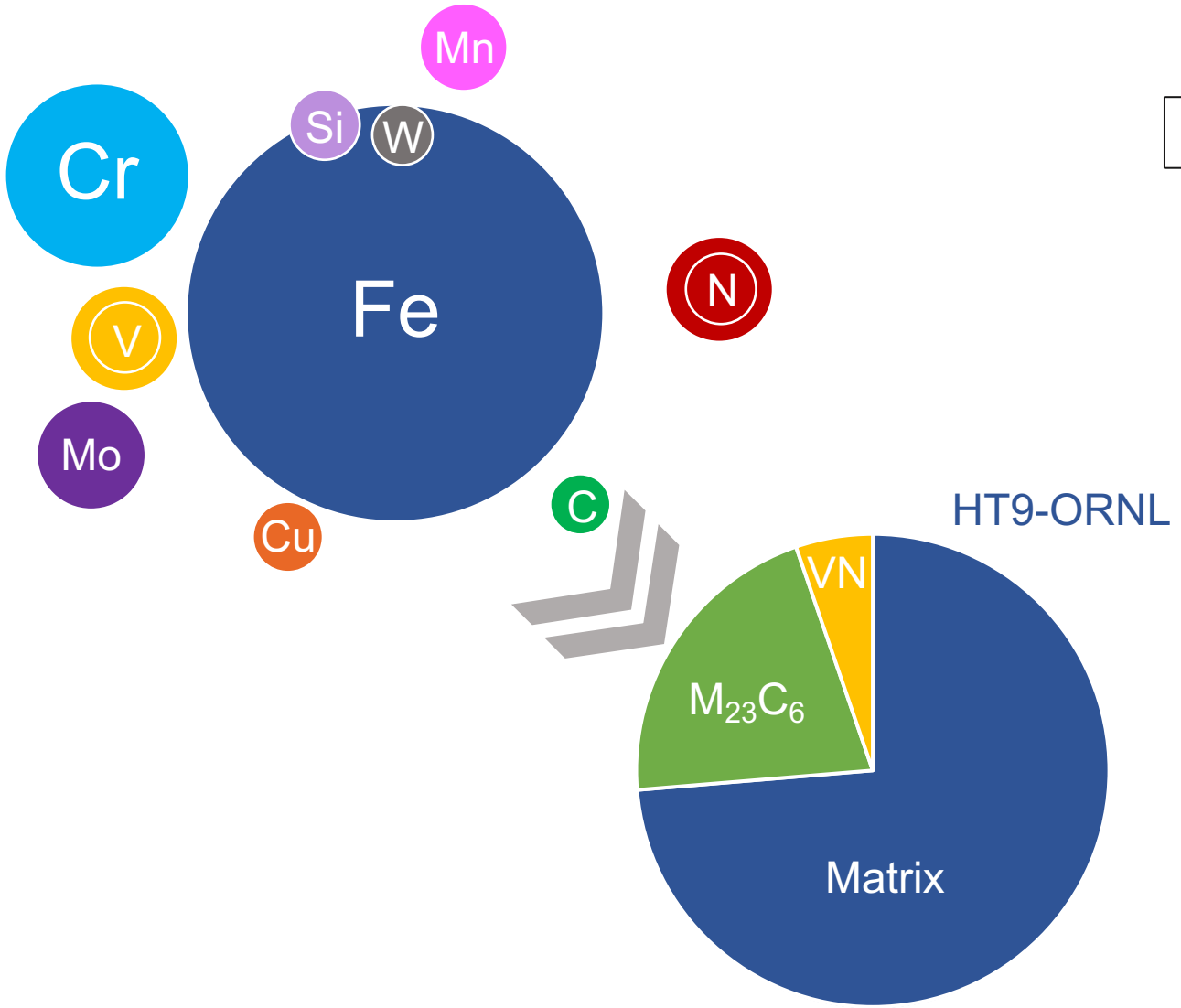


VN did not dissolve during normalization

ThermoCalc

Heat-Treated Microstructure Summary

Composition



Normalization & Tempering

Dissolution of precipitates

Reduction in dislocation density
Growth of carbides during tempering

HT9-EBRII $M_{23}C_6$

HT9-LANL $M_{23}C_6$

Irradiated Microstructure

- Irradiation did not alter tempered martensitic microstructure
- Caused formation of dislocation loops and black spot defects

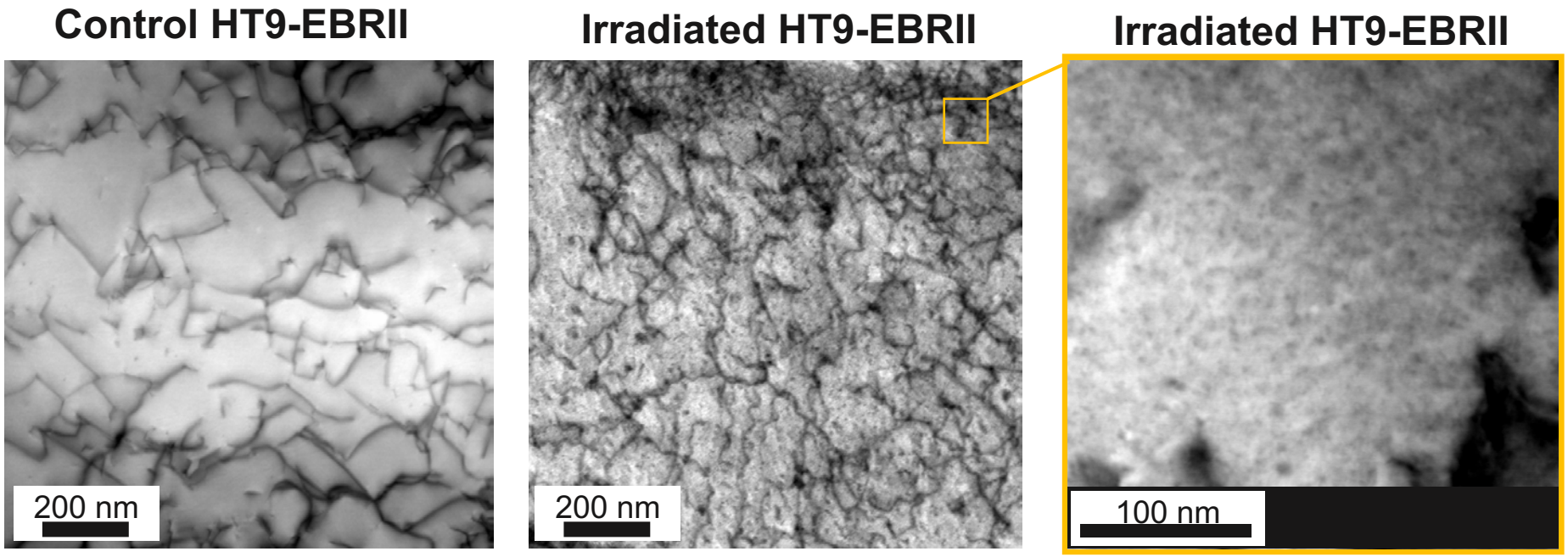


Figure 3. STEM BF micrographs showing representative change in dislocation structure induced from irradiation in all samples.

Irradiated Microstructure

- V is found enriched at dislocations in HT9-LANL and HT9-ORNL after irradiation

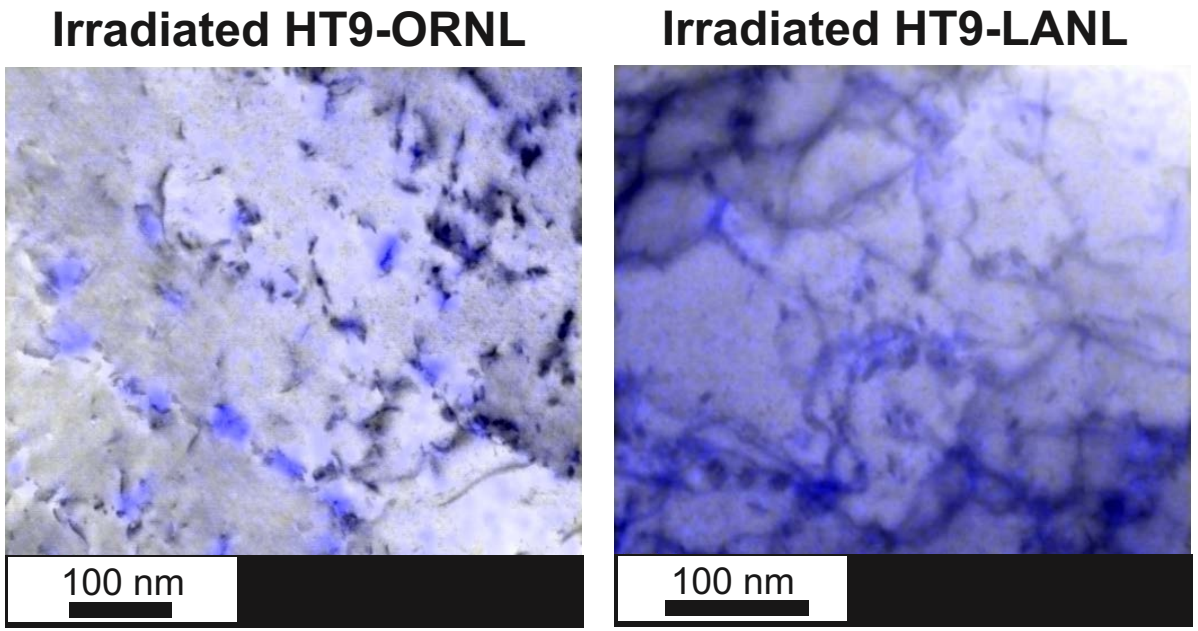
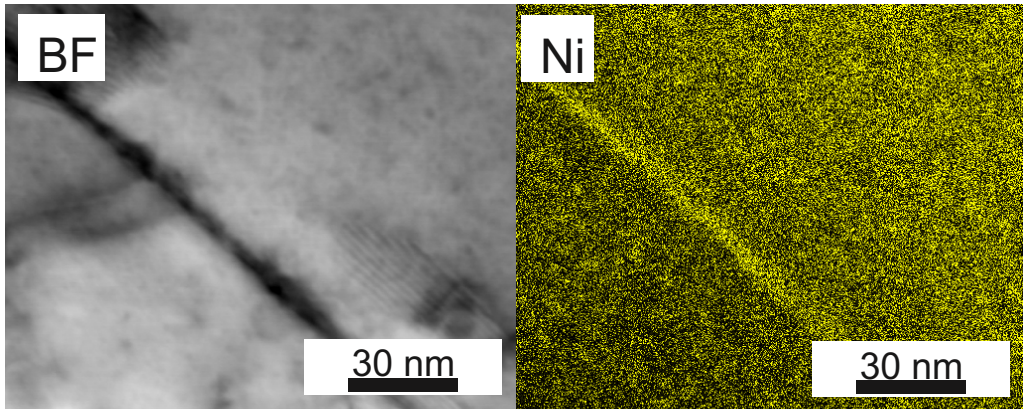


Figure 3. Overlap of STEM BF images and EDS maps in irradiated HT9-ORNL and HT9-LANL showing V-enriched dislocation loops. Image plane is {110}.

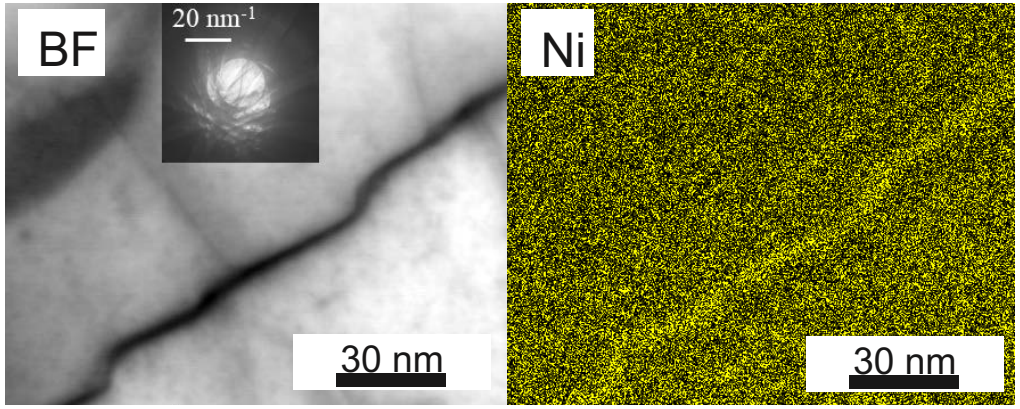
Radiation Induced Segregation

Radiation induced segregation of Ni at grain boundaries in all irradiated conditions

Irradiated HT9-ORNL

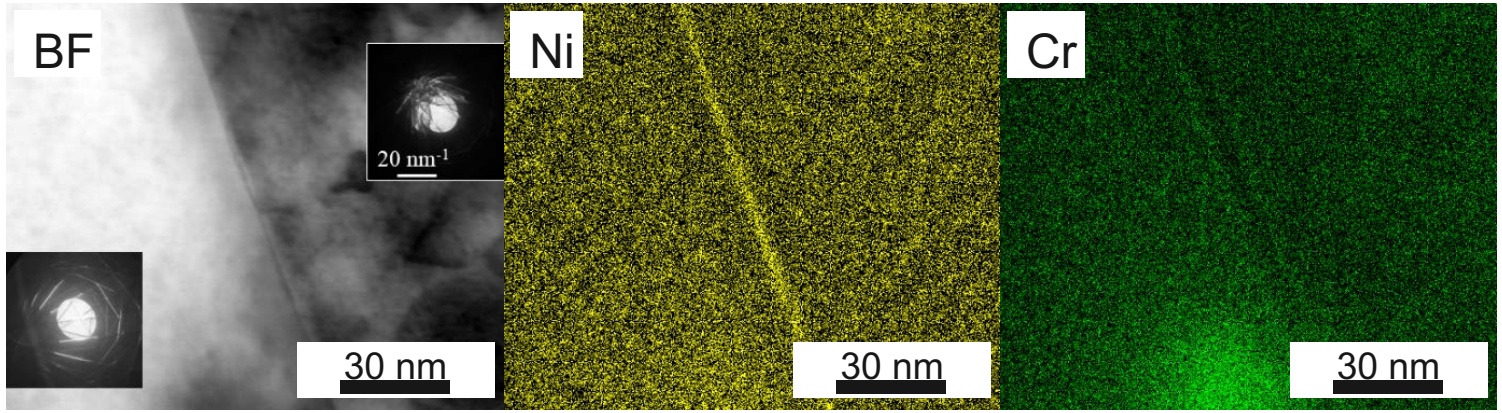


Irradiated HT9-LANL



Cr depleted at grain boundaries in irradiated HT9-EBRII as well

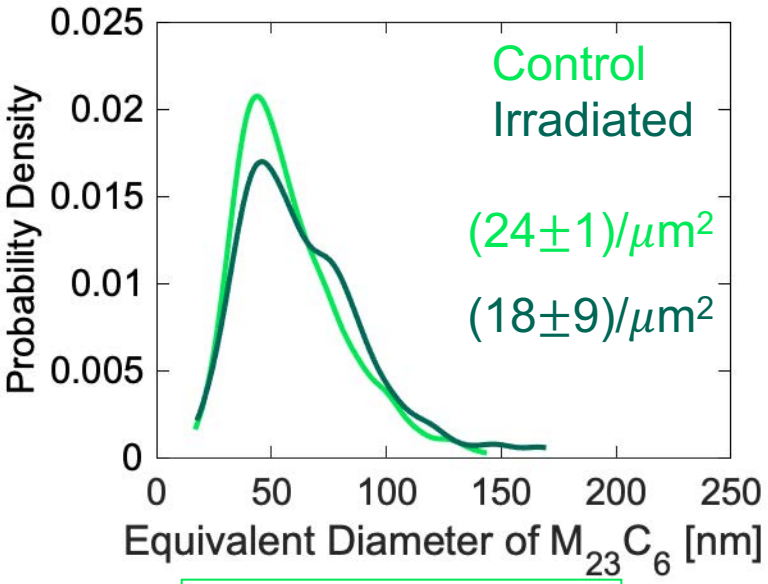
Irradiated HT9-EBRII



Irradiation had an insignificant affect on $M_{23}C_6$ precipitation size distribution → ***Minor Solute Stabilization***

Control HT9-EBRII

1040°C/1 hr + 740°C/0.75 hr



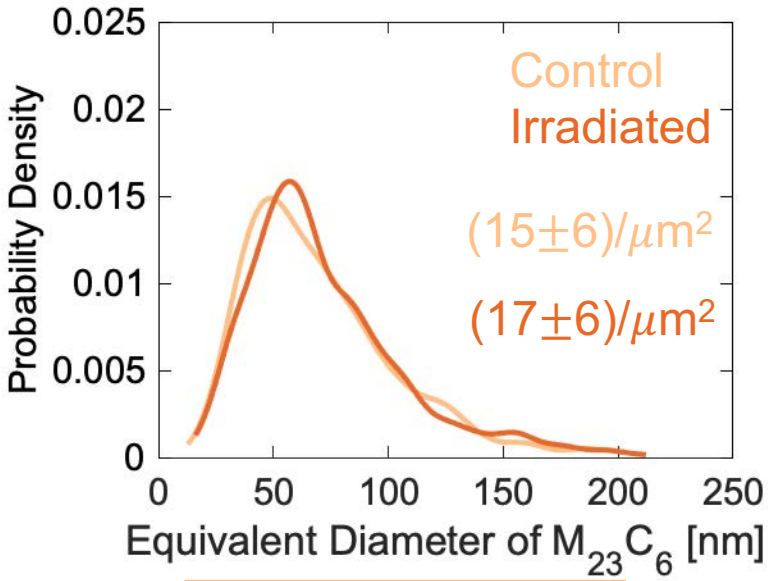
Mean = 58±23 nm
N = 606

Mean = 64±28 nm
N = 552

ΔMean = 6 nm

Control HT9-ORNL

1040°C/0.5 hr + 760°C/1 hr



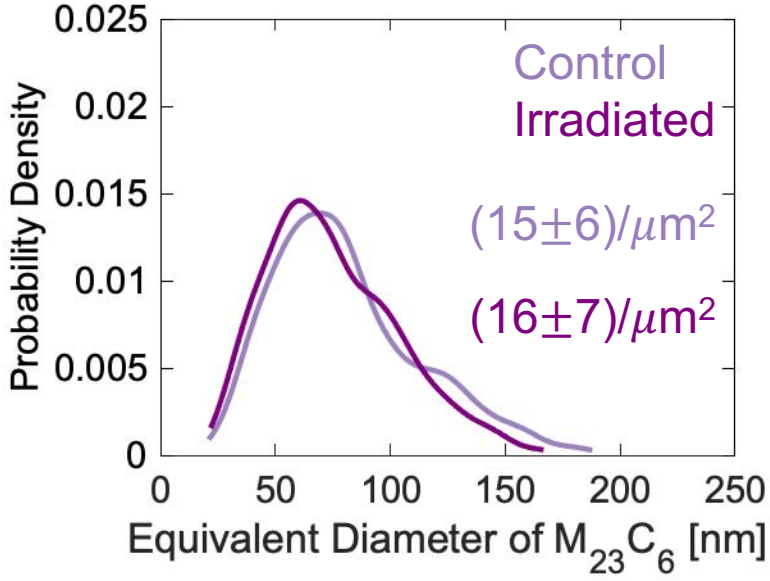
Mean = 70±33 nm
N = 541

Mean = 72 ±34 nm
N = 781

ΔMean = 2 nm

Control HT9-LANL

1060°C/1 hr + 730°C/2 hr



Mean = 81±32 nm
N = 494

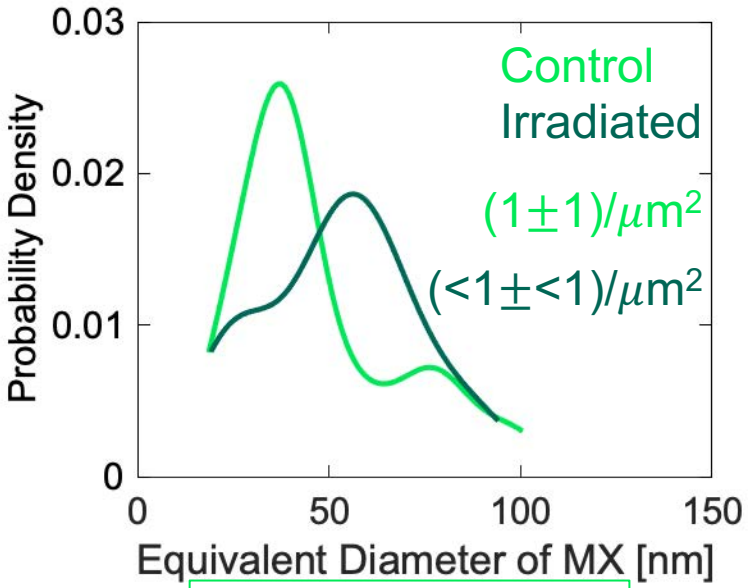
Mean = 75 ±28 nm
N = 639

ΔMean = 6 nm

Irradiation caused growth of MX precipitates in HT9-LANL and HT-9 ORNL → ***Radiation Enhanced Diffusivity***

Control HT9-EBRII

1040℃/1 hr + 740℃/0.75 hr



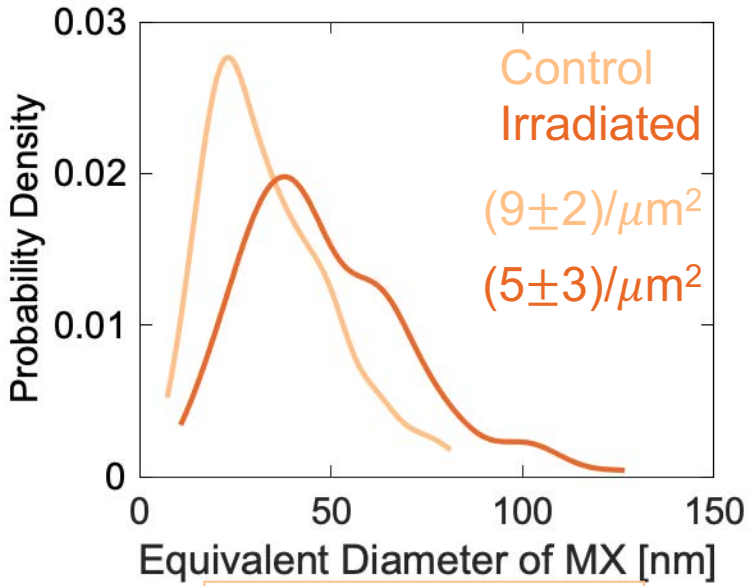
Mean = 48±22 nm
N = 30

Mean = 52±20 nm
N = 28

ΔMean = 4 nm

Control HT9-ORNL

1040℃/0.5 hr + 760℃/1 hr



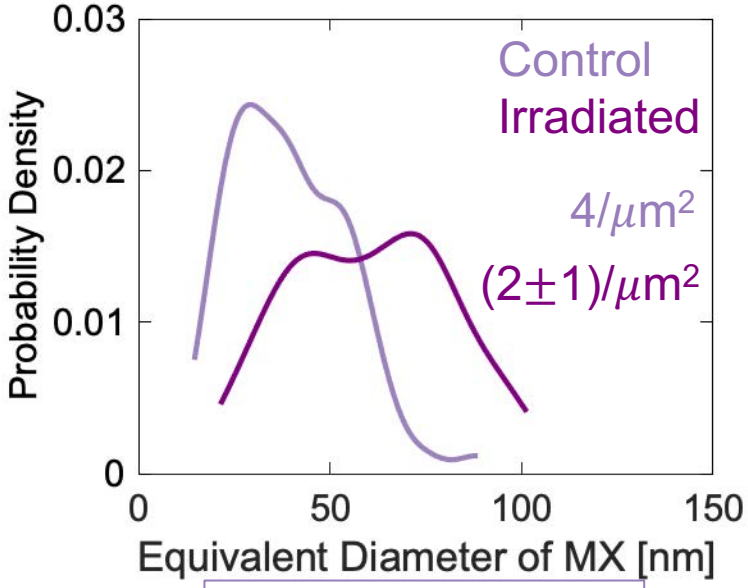
Mean = 34±16 nm
N = 323

Mean = 49±22 nm
N = 157

ΔMean = 15 nm

Control HT9-LANL

1060℃/1 hr + 730℃/2 hr



Mean = 40±15 nm
N = 134

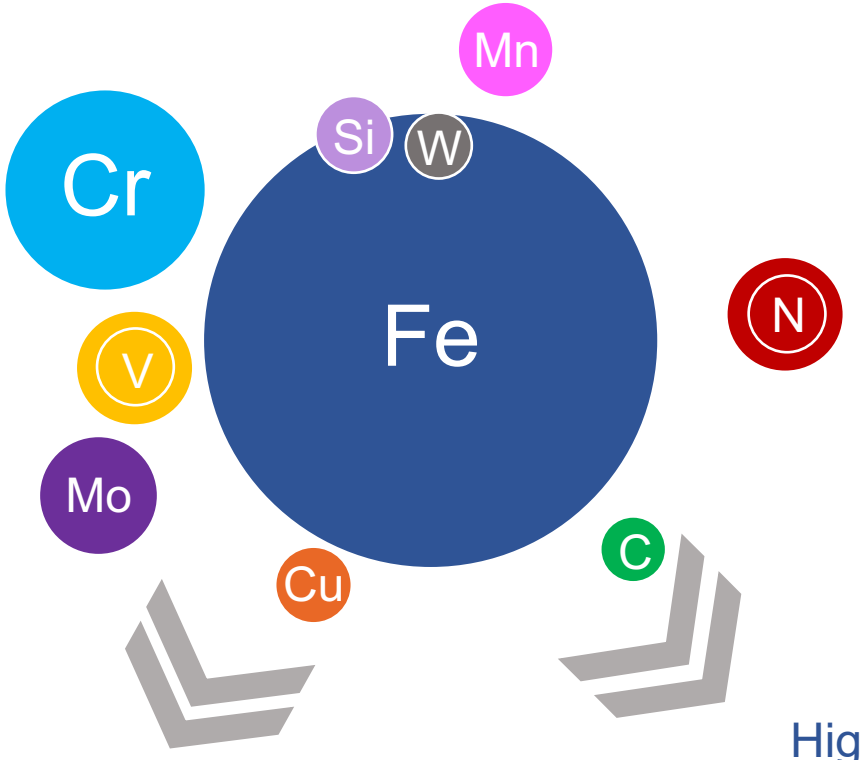
Mean = 60±21 nm
N = 73

ΔMean = 20 nm

L. Tan/Journal of Nuclear Materials 493 (2017) 12-20

Radiation Response of the Heat-Treated Microstructure

Composition



Minor solute stabilization of $M_{23}C_6$

Higher concentrations of V and N initially cause greater VN phase fraction

Normalization & Tempering

Dissolution of precipitates

Future Work: Effect of dislocations?

VN + 

Reduction in dislocation density
Growth of carbides during tempering

HT9-EBRII $M_{23}C_6$

HT9-LANL $M_{23}C_6$

Acknowledgements

- Nuclear Science User Facilities Rapid Turnaround Experiment, NSF Project 17-1047
- Thank you to INL staff for help preparing samples and performing electron microscopy
  - Dr. Brandon Miller
  - Dr. Lingfeng HE
  - Dr. Xiang Liu