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Development of nanosized carbide dispersed advanced radiation resistant austenitic stainless steel (ARES) for Generation IV systems

Dr. Ji Ho Shin

KAIST

Republic of Korea

11 May 2022

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Meet the Presenter

Dr. Ji Ho Shin recently completed his PhD at the Korea Advanced Institute of Science and Technology (KAIST) in the field of nuclear materials on the subject of “Development of nano carbide dispersed advanced radiation resistant austenitic stainless steels (NC-ARES) for reactor internals”.

His PhD focuses on the development of next-generation nuclear in-core materials, including Small Modular Reactor (SMR), Sodium Fast Reactor (SFR), and fusion reactor to demonstrate the superior radiation resistant features.

He is currently a post-doctoral fellow in the Korea Atomic Energy Research Institute (KAERI). He was the popular vote winner of the 2021 Pitch your Gen IV research competition.

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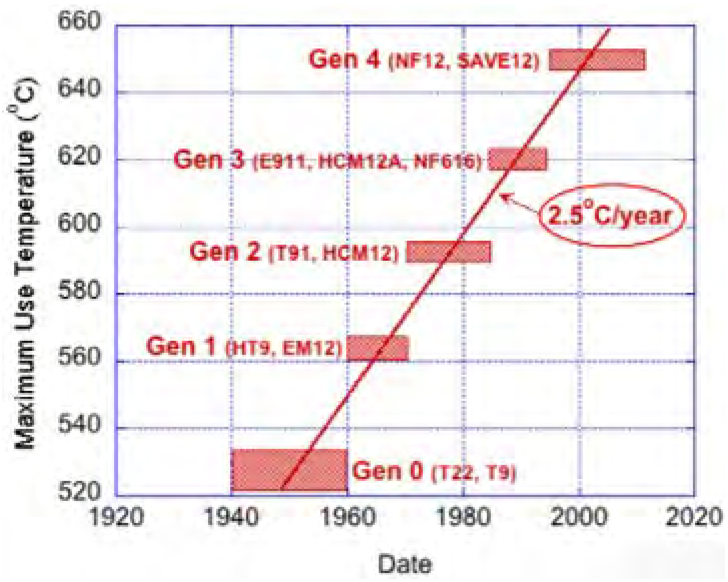
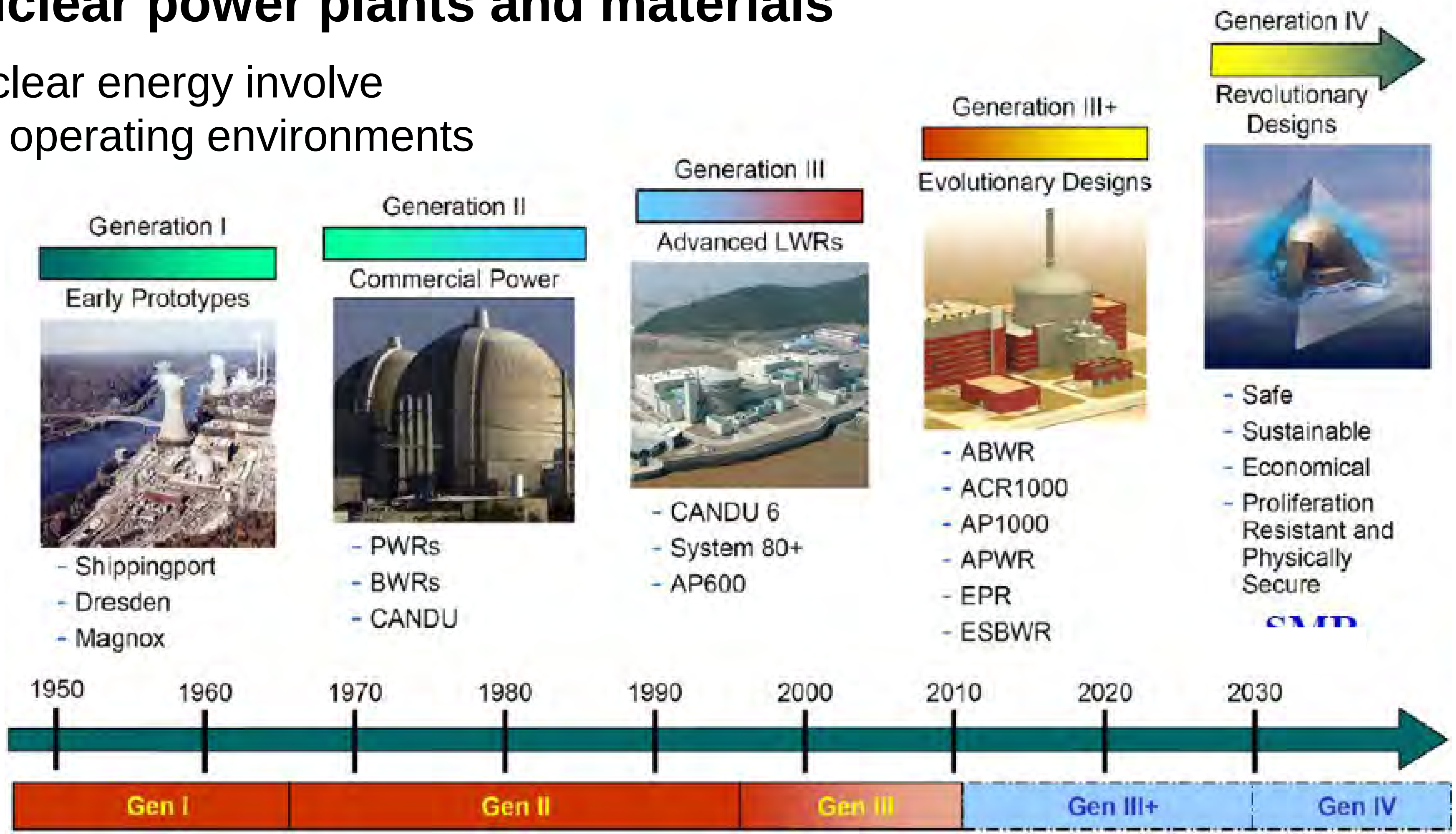
Background

❑ Brief history of nuclear power plants and materials

- Future goals for nuclear energy involve even more extreme operating environments



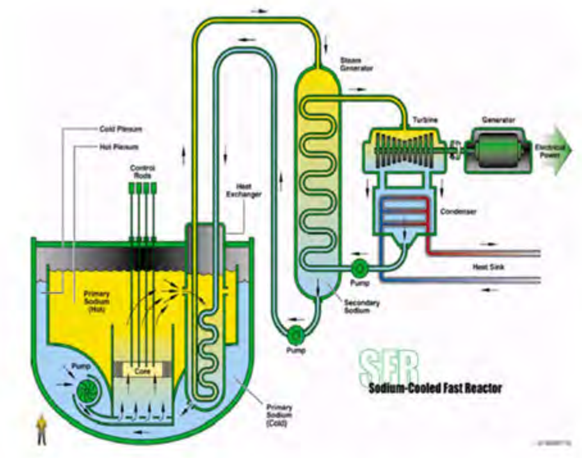
▲ Historical development of improved high-temp. steels [1]



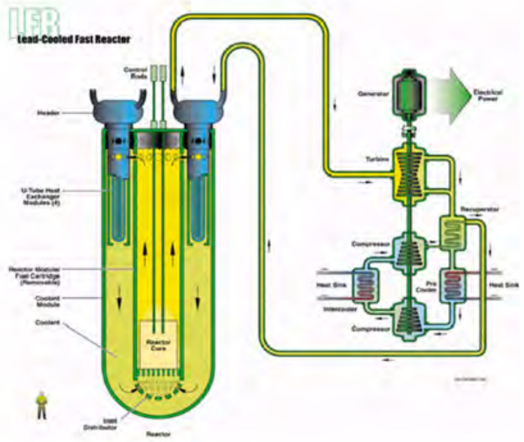
▲ Generation IV roadmap from Argonne National Laboratory (wikipedia)

Background

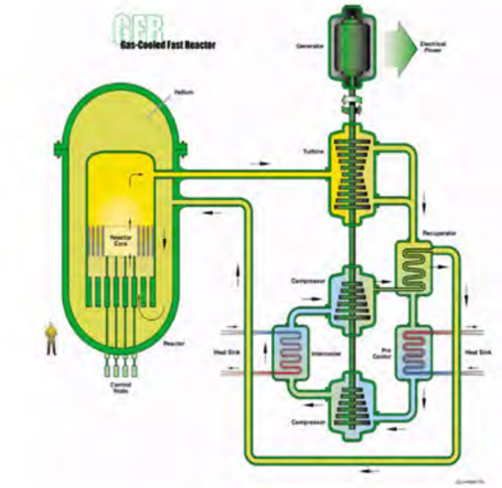
❑ Generation IV Forum: selection of *six nuclear systems*



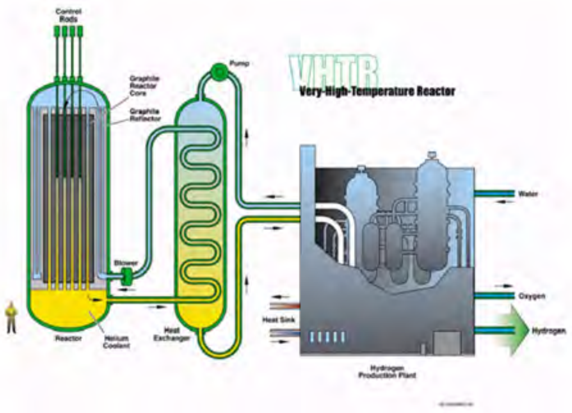
▲ Sodium Fast Reactor



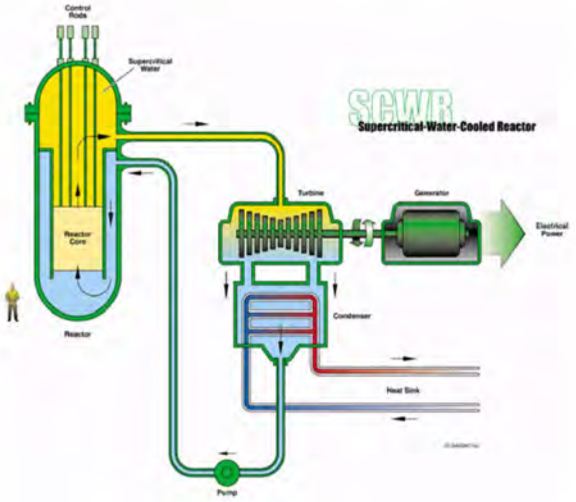
▲ Lead Fast Reactor



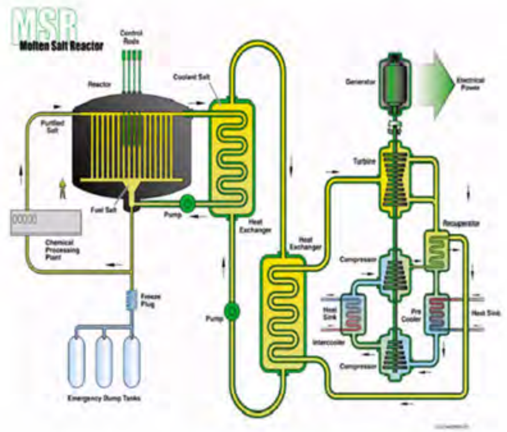
▲ Gas Fast Reactor



▲ Very High Temperature Reactor



▲ Supercritical Water-cooled Reactor

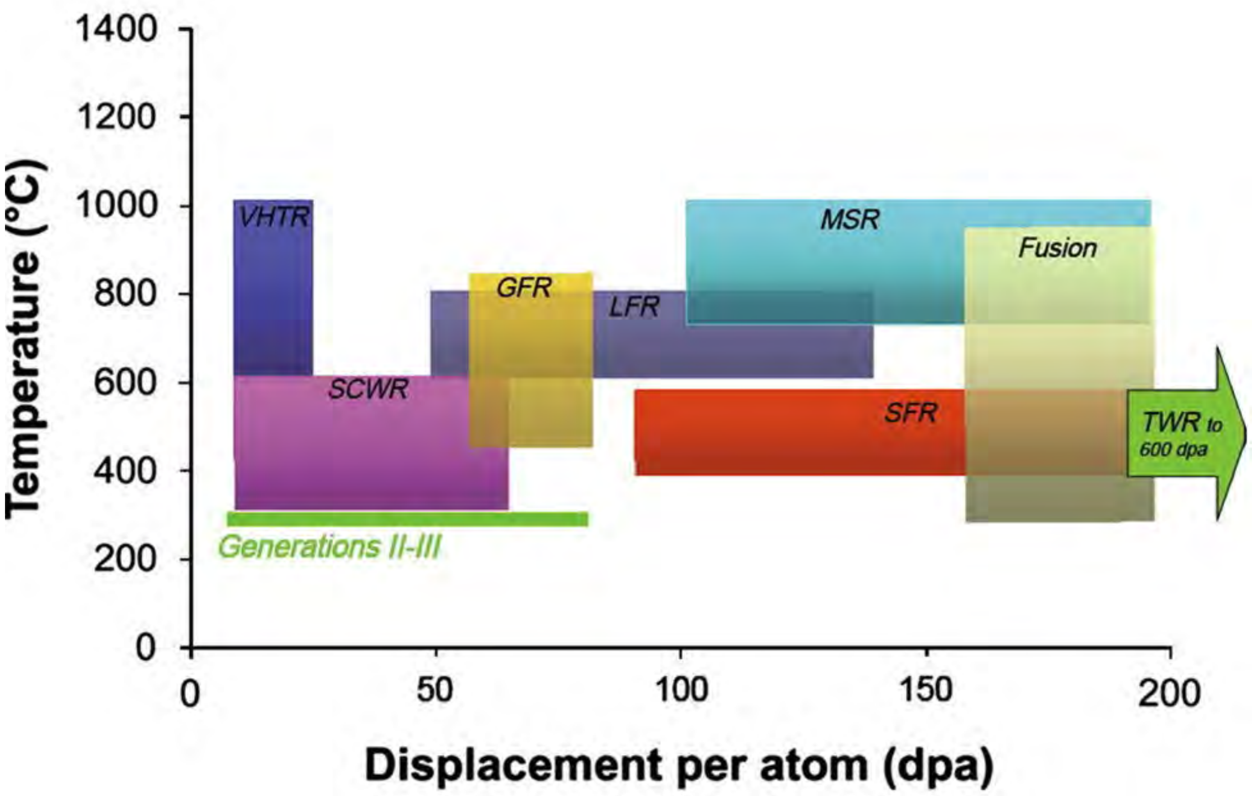


▲ Molten Salt Reactor

[1] G.R. Odette et al., Annu. Rev. Mater. Res. 38 (2008) 471
[2] S.J. Zinkle et al., JNM 417 (2011) 2

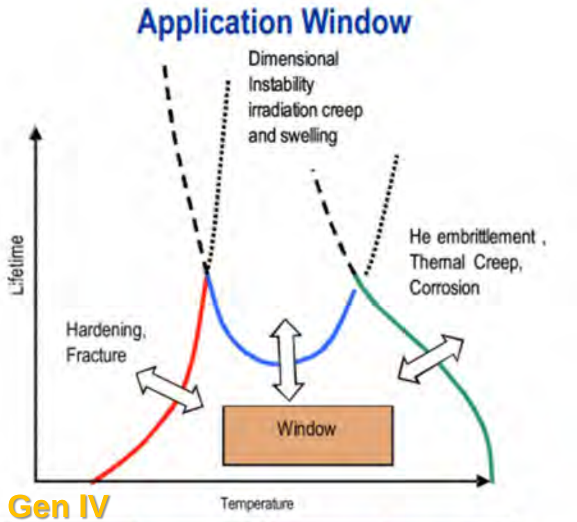
Operating Conditions

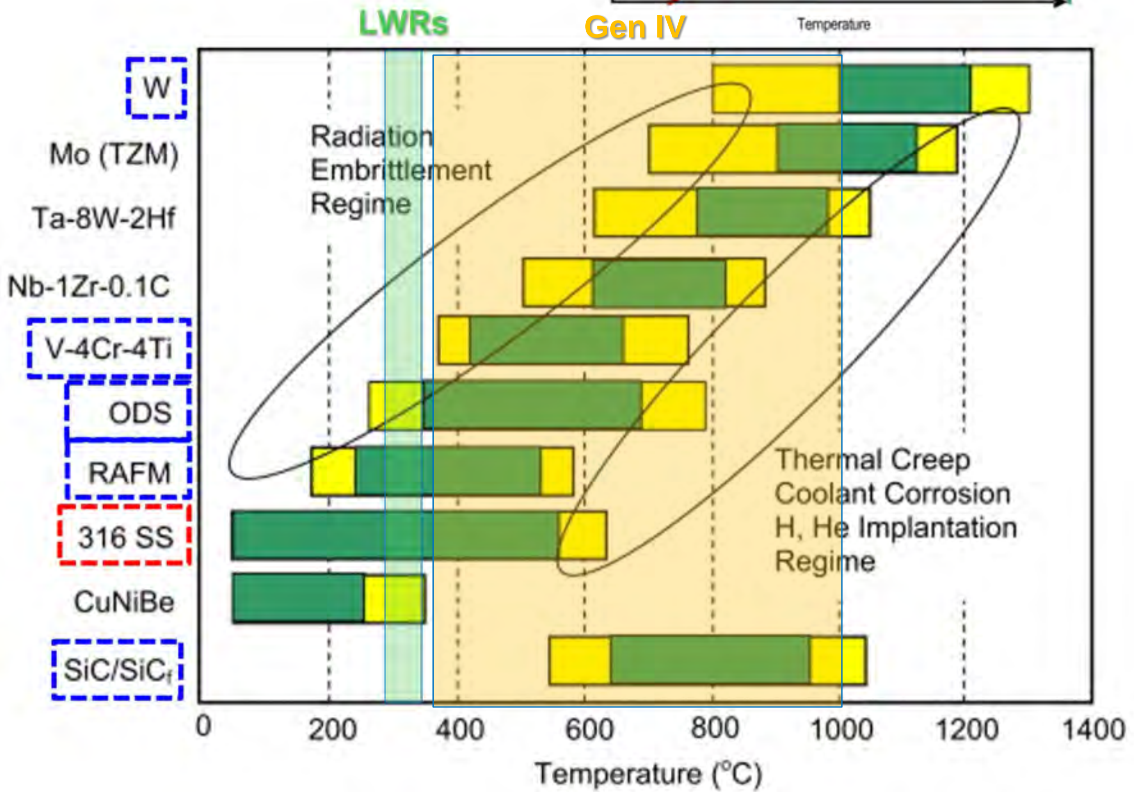
❑ Comparison of structural materials environments



▲ Schematic of the temperature-dpa requirements for various reactors [1]

- VHTR = very high temperature reactor / SCWR = supercritical water reactor
- GFR = gas fast reactor / LFR = lead fast reactor / MSR = molten salt reactor
- SFR = sodium fast reactor / TWR = traveling wave reactor
- Generations II-III = present day light water reactors



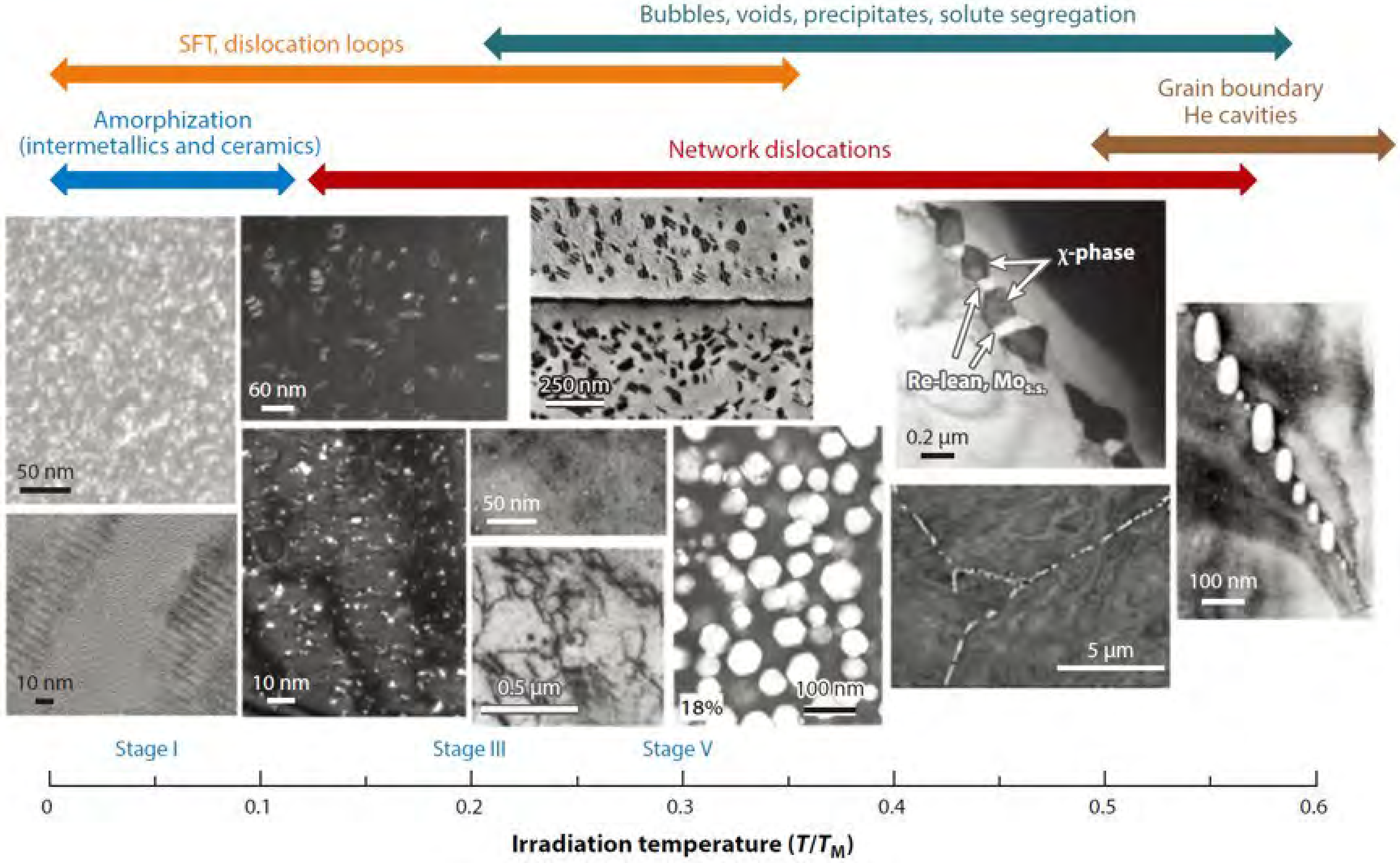


▲ Operating temperature windows of some candidate reactor materials for the neutron irradiation giving rise to 10–50 dpa. [2]

S.J. Zinkle et al., Annu. Rev. Mater. Res. 44 (2014) 241

Radiation Damage in Materials

- Representative microstructures in irradiated materials

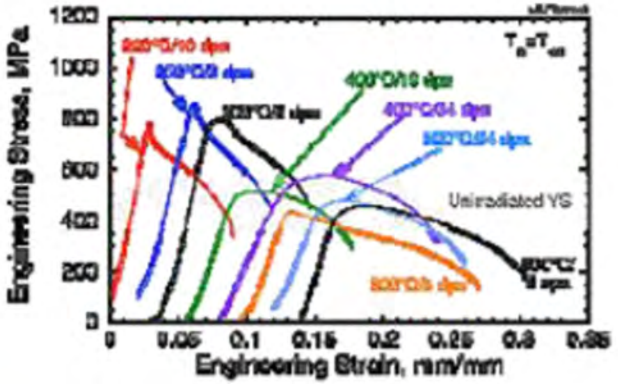


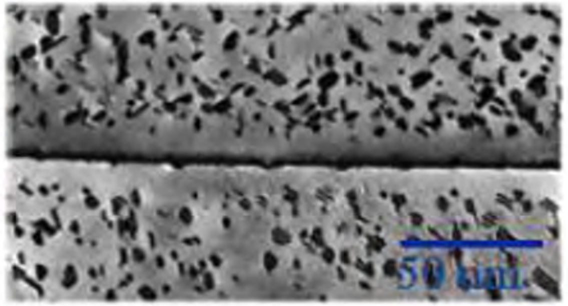
S.J. Zinkle, Phys. Plasmas 12 (2005) 058101

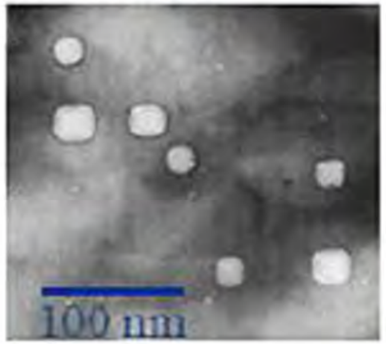
Radiation Damage in Materials

❑ Radiation induced degradation in structural materials

1. **Radiation hardening and embrittlement**
   - <0.4 T_M, >0.1 dpa
2. **Phase instability from radiation-induced precipitation**
   - 0.3-0.6 T_M, >10 dpa
3. **Irradiation creep**
   - <0.45 T_M, >10 dpa
4. **Volumetric swelling from void formation**
   - 0.3-0.6 T_M, >10 dpa
5. **High temperature He embrittlement**
   - >0.5 T_M, >10 dpa







Introduction

❑ Generation IV requirements and technical challenges

– **The four priority areas of technology or requirements to focus on are:**

- development of sustainable nuclear energy
- maintaining or increasing competitiveness
- improving and enhancing safety and reliability
- ensuring proliferation resistance and physical protection

– **The material and material supply needs for the new Generation IV reactors are expected to:**

- build on Generation II and III experiences + Fusion
- feature new materials developments
- use established industrial processes + new processes
- require Codes and Standards developments in parallel

System	Neutron spectrum	Coolant	Outlet temperature (°C)	Fuel cycle	Size (MW_e)
VHTR (very-high-temperature reactor)	Thermal	Helium	Up to 1000	Open	250–300
GFR (gas-cooled fast reactor)	Fast	Helium	850	Closed	1200
SFR (sodium-cooled fast reactor)	Fast	Sodium	500–550	Closed	50–150 300–1500 600–1500
LFR (lead-cooled fast reactor)	Fast	Lead	480–570	Closed	20–180 300–1200 600–1000
MSR (molten salt reactor)	Thermal/fast	Fluoride salts	700–800	Closed	1000
SCWR (supercritical water-cooled reactor)	Thermal/fast	Water	510–625	Open/closed	300–700 1000–1500

▶ Overall design characteristics of Generation IV systems [1]

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▶ Need for high performance alloys (e.g. ODS, FMS, advanced alloys

Requirements for Materials in Future Nuclear Systems

- **Extent operation lifetime: 60 (or 80+) years**
- **Fast neutron (+ high fluence) damage (fuel and core materials)**
  - Effect of irradiation on microstructure, phase instability, precipitation
  - Swelling growth, hardening, embrittlement
  - Effect on tensile properties (yield strength, UTS, elongation…)
  - Irradiation creep and creep rupture properties
  - Hydrogen and helium embrittlement
- **High temperature resistance (SFR > 550°C, V/HTR > 850-950°C)**
  - Effect on tensile properties (yield strength, UTS, elongation…)
  - High temperature embrittlement
  - Effect on creep rupture properties
  - Creep fatigue interaction
  - Fracture toughness
- **Corrosion resistance (primary coolant, power conversion, H2 production)**
  - Corrosion and stress-corrosion cracking (IGSCC, IASCC, hydrogen cracking & chemical compatibility…)

[1] V.D. Rusov et al., Sci. and Tech. Nucl. Inst. 2015 (2015) 1
[2] Tanigawa, IEEE symp. On Fusion Engineering, June, 2019
[3] M. Seitz et al., Nucl. Mater. Ener., 13 (2017) 90

New Materials for Generation IV Reactors

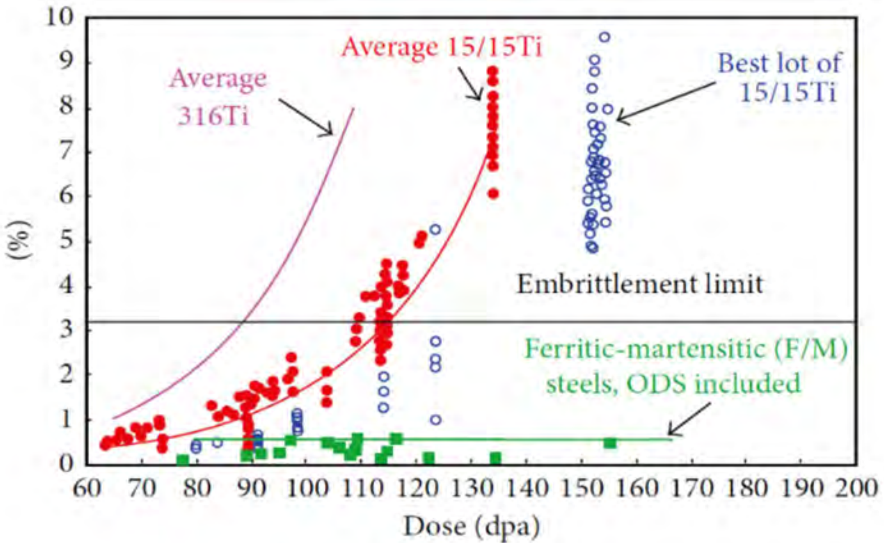
❑ SFR internal material: F/M steel

– Advantage of FMS (& FM-ODS)

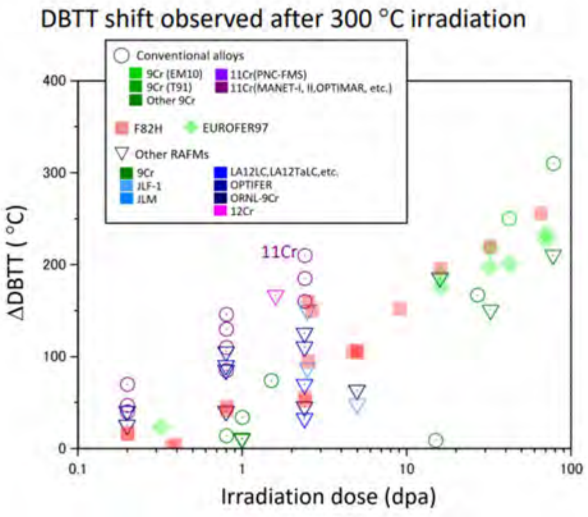
1) Low-activation (RAFM)
2) High radiation resistance (Swelling resistance)

– Drawback of FMS (& FM-ODS)

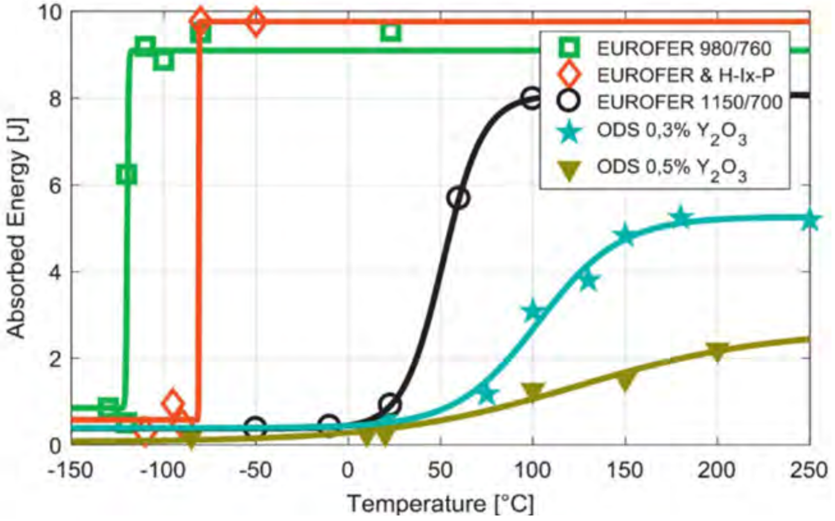
1) Radiation embrittlement: DBTT
2) Low corrosion resistance
3) Low creep resistance at high T
   - Improvement by FM-ODS
4) Productivity (FM-ODS)



▲ Swelling of austenitic SS with FMS and ODS [1]



▲ Irradiation embrittlement of FMS: DBTT shift [2]



▲ Impact property of FMS and ODS [3]

Goal of This Study

❑ Why austenitic stainless steel?

– **FMS (& FM-ODS) vs. Austenitic stainless steels**

	Activation	Radiation resistance		H embrittlement	Corrosion	Property at High T	Productivity	Thermal stress
		Embrittlement	Swelling					
FMS	Good	Poor (DBTT)	Good	Poor	Poor	Fair	Possible	Small
FM-ODS		Worse				Good	Impossible	
Austenitic SS (316)	**Poor (Ni)**	**Good**	**Poor**	**Good**	**Good**	**Good**	**Possible**	Large

**Increase the poor swelling resistance of austenitic SS

Contents

ARES: Advanced radiation Resistant austenitic stainless Steels

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Topic I

Development of ARES alloys

High density of uniformly distributed nanosized carbides in austenitic SS

[1] V.D. Rusov et al., Sci. and Tech. Nucl. Inst. 2015 (2015) 1

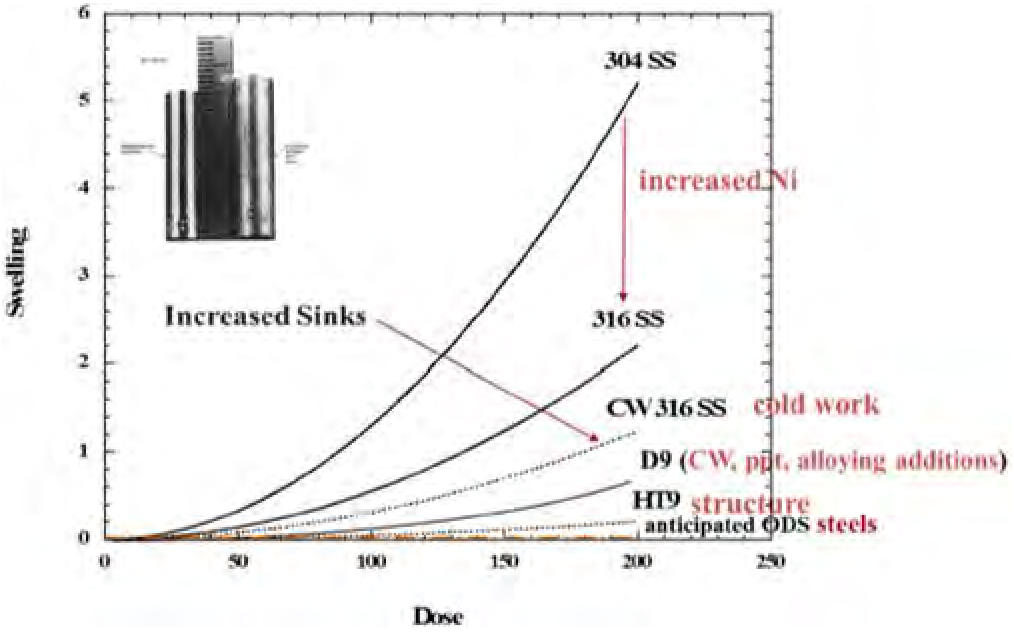
Alloy Design Strategy

❑ Radiation resistant characteristics

- High Ni (+Cr) content
- Low Si content
- High CSL fraction
  - GBE (grain-boundary engineering)
  - Difficult in large section material
- High SFE
  - SFE controls the nature of slip

◆ Ferritic or Ferritic-martensitic alloys
  - BCC alloys (swelling rate: ~0.2 %/dpa) are more resistant FCC alloys (~1.0%/dpa)
  - Resistant to localized corrosion, but less resistant to general corrosion

- High Schmid & Low Taylor factor
  - Minimize slip on slip systems to avoid localized deformation ($\tau = m\sigma$)
- Small grains
- Cold working
- Precipitates
- Low inclusion density

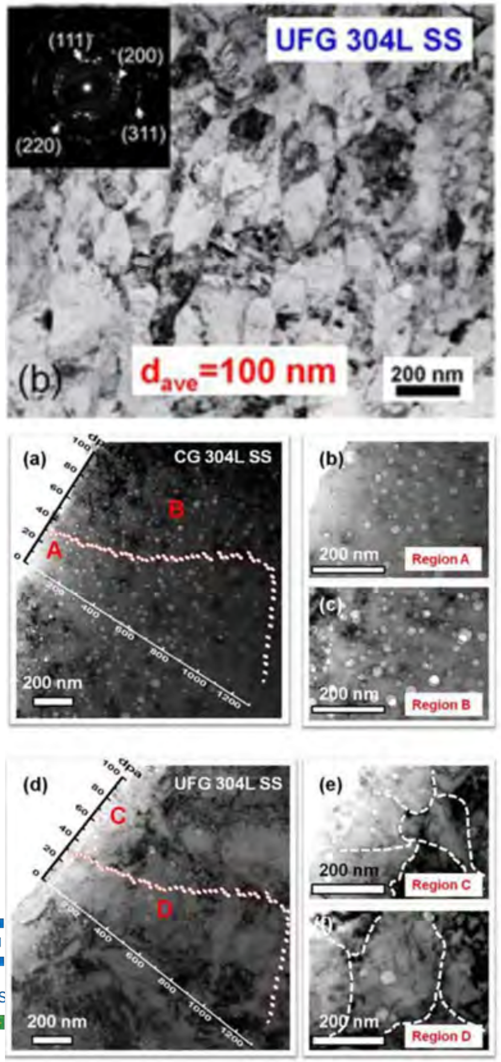


▲ Swelling of austenitic SS with FMS and ODS [1]

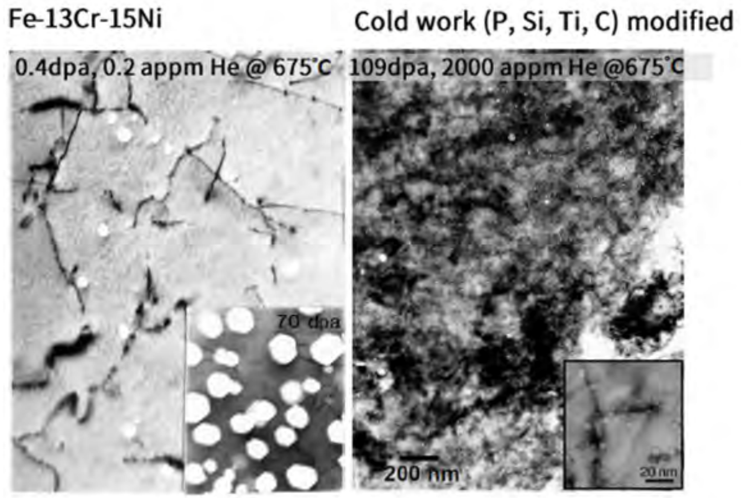
Overview of Recent Developments

[1] C. Sun et al., Sci. Rep. 5 (2015) 7801
[2] E.H. Lee et al., Phil. Mag. A 61 (1990) 733
[3] G.R. Odette et al., Annu. Rev. Mater. Res. 38 (2008) 471
[4] E.J. Pickering et al., Int. Mater. Rev. 61:3 (2016) 183
[5] L. Tan et al., JOM 68 (2016) 517

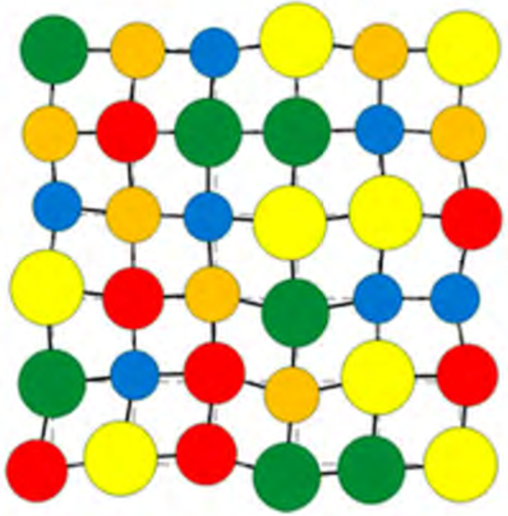
Nano-grain [1]



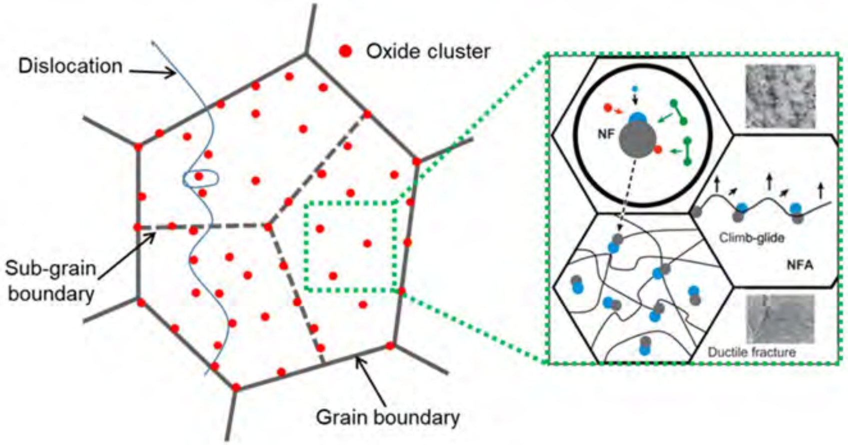
Dislocation+Nano precipitates [2]



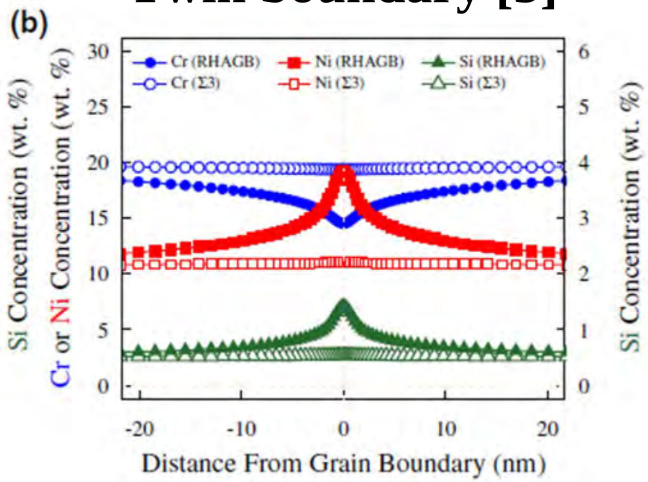
HEA [4]



FMS or ODS [3]



Twin boundary [5]



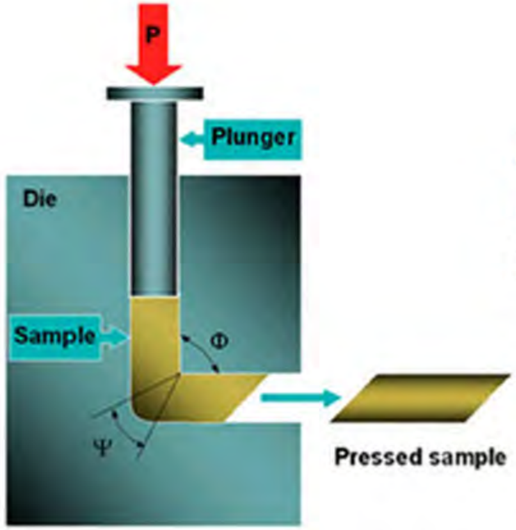
Focus of Research

[1] C. Sun et al., Sci. Rep. 5 (2015) 7801
[2] G.R. Odette et al., Annu. Rev. Mater. Res. 38 (2008) 471
[3] B. Rouxel et al., EPJ Nuclear Sci. Technol. 2 (2016)
[4] Y. Xu et al., Materials 11 (2018) 1161

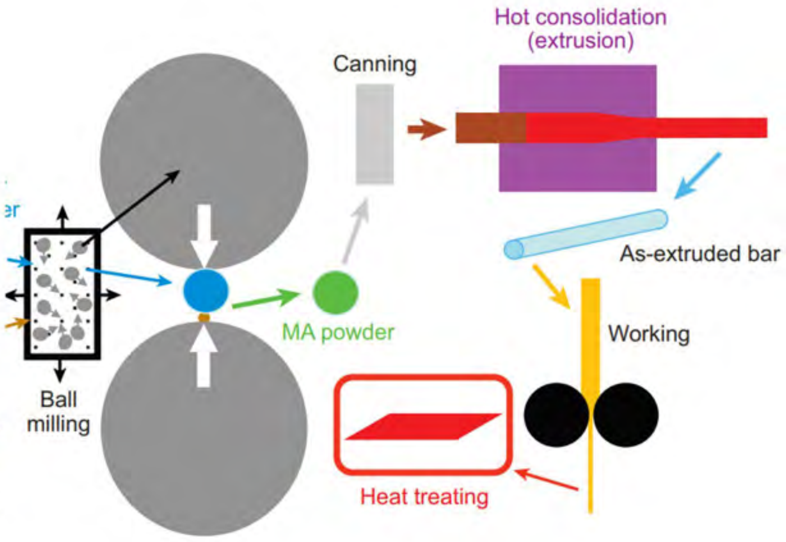
❑ Limitations of application

1. Complex manufacturing & mass production

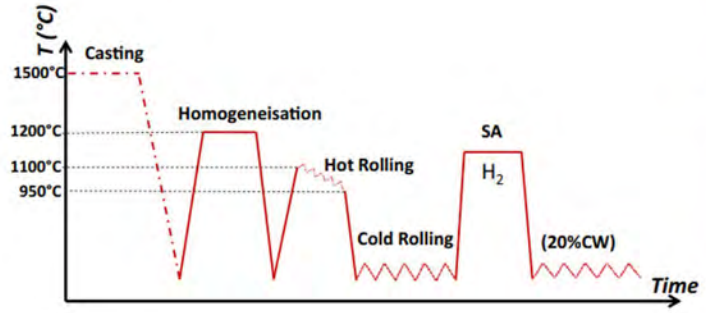
2. Crystallographic texture + heterogeneous ppt.



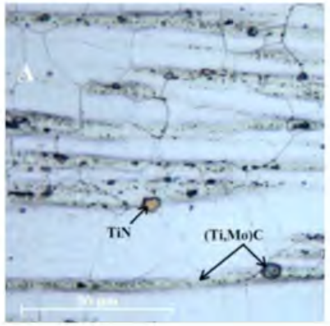
▲ Equal channel angular pressing [1]



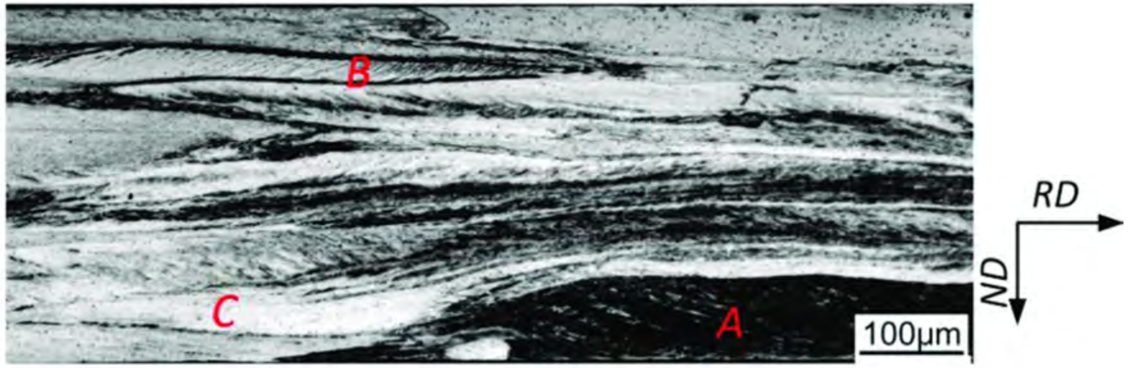
▲ Powder metallurgy [2]



▲ TMP of 15-15 Ti [3]

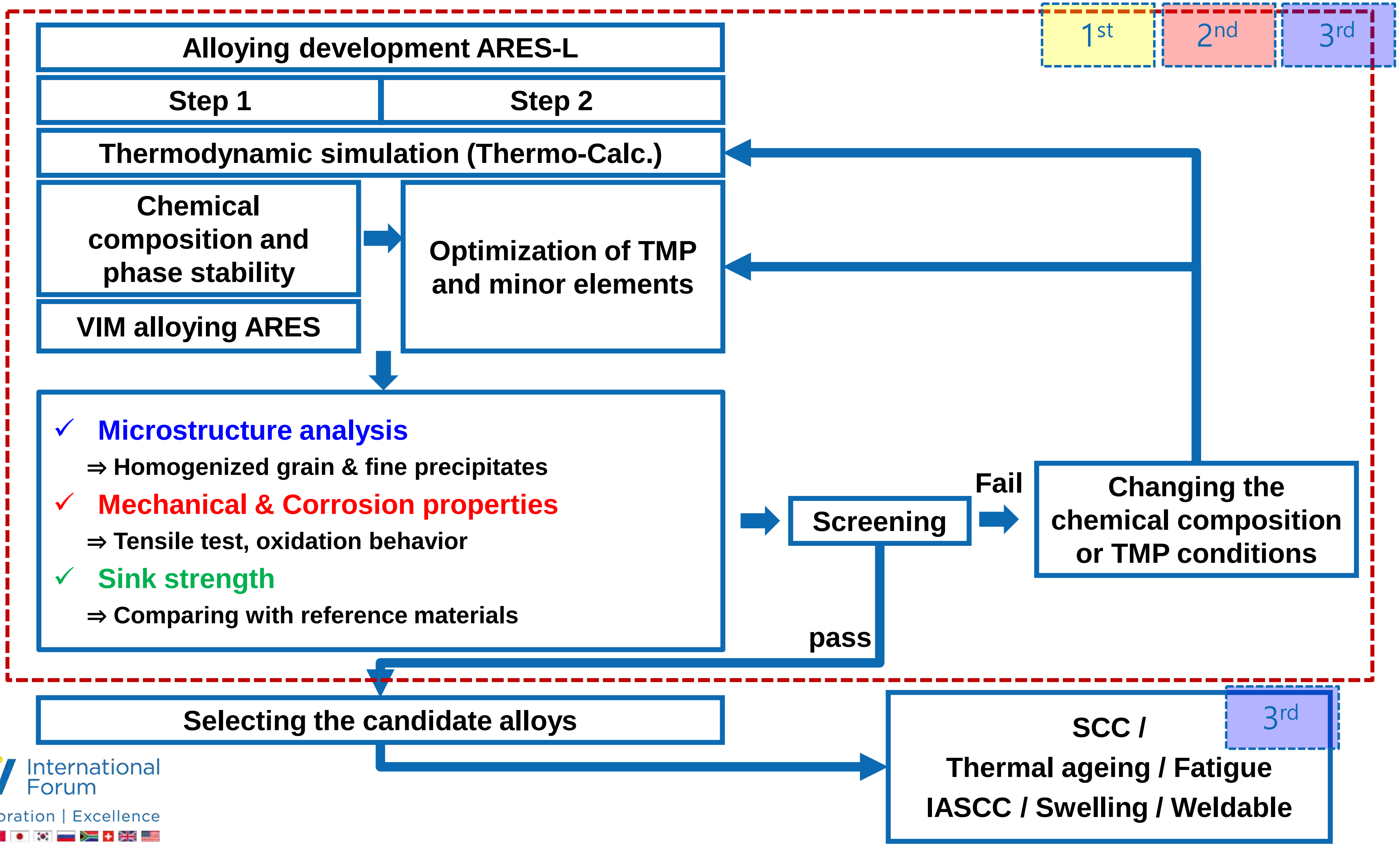


▲ Solution annealed 15-15 Ti [3]



▲ One-stage of cold rolled sheet [4]

Detailed Plan for ARES for In-core Materials



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[1] Bhadeshia et al., ISIJ International (2001) 41:626-640
[2] Porter et al., Phase Trans. in Metals and Alloys, Taylor & Francis Group (2004)

Motivation

❑ Control the minor elements to form the precipitates

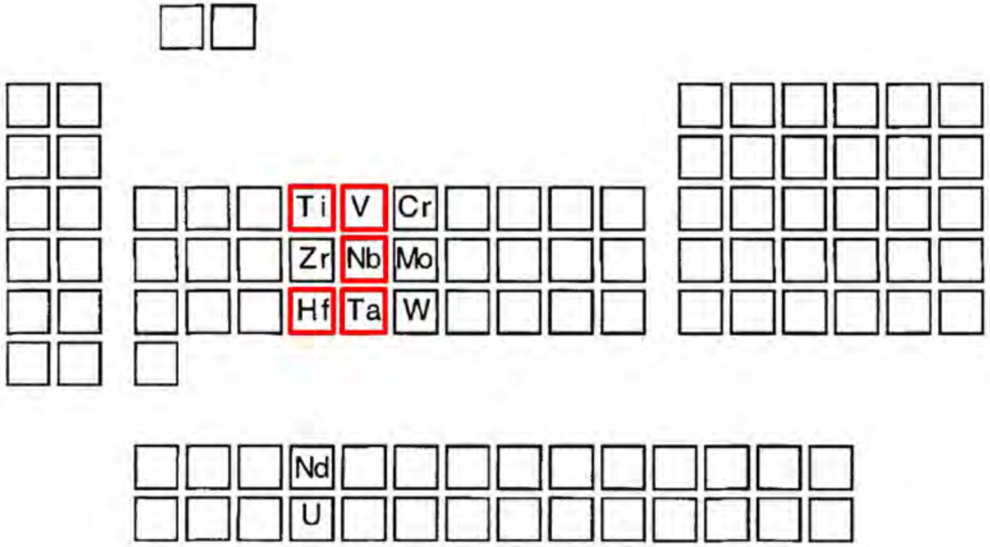
– **The fineness of the dispersion depending on the activation energy barrier (ΔG*)**

  • Free energy of formation of the ppt., Interfacial energy, Misfit

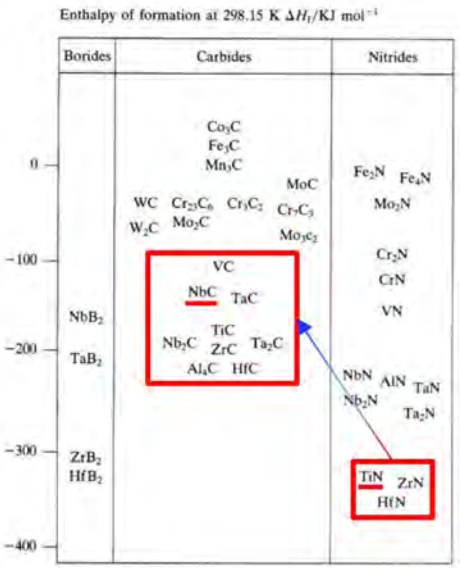
– **Solubility of precipitation particles in the austenite increasing in the order –**

  • Nitrides: TiN → NbN → AlN → VN

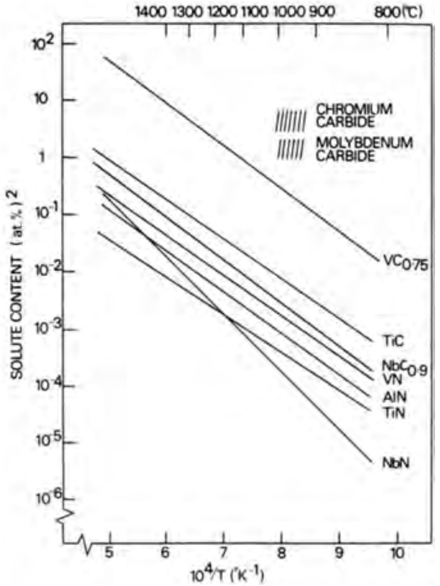
  • Carbide: NbC → TiC → VC



▲ The periodic table showing the positions of strong carbide-forming elements [1]



▲ Enthalpies of formation of carbides, nitrides and borides [2]

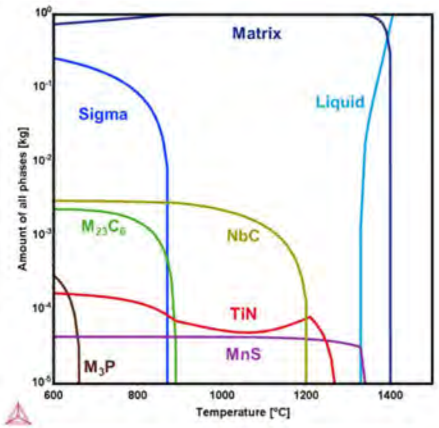


▲ Solubility product of carbides and nitrides in austenite as a function of temp. [2]

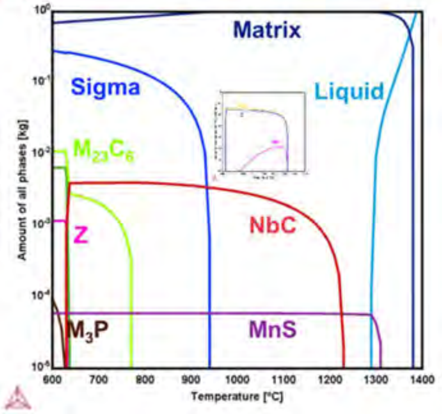
Alloy Design – Thermo-Calc. (3rd Phase)

❑ Alloy design process - simulation

ARES-6



ARES-8



- Thermodynamic simulation (Thermo-Calc)
- Data base: TCFE9 (steels/Fe-alloys v9.0), MOBFE3(steels/Fe-alloys mobility v3.0)
- Forming the fine Ti-rich ppt. (Ti(C,N)) – ARES-6
  - ✓ Absolute Ti composition: ~0.02 wt.%
  - ✓ Ti / N ratio ≤ 3.42 → Nitrogen: ~80ppm (~0.008 wt.%)
- Forming the fine NbC
  - ✓ ARES-6: Niobium = 0.27 wt.%, C= 0.042 wt.%
  - ✓ ARES-7, 8: Niobium = 0.45 wt.%, C= 0.035 wt.%
- Stability of precipitates
  - ✓ Reducing the diffusion coefficient
    - Slowing coarsening rate of ppt. to the slow rejection or combining of X atom from the precipitates ⇒ by adding Mn and Mo elements
    - ARES-7, 8: manganese = ~3.5 wt.%
    - ARES-8: ~0.8 wt.%
  - ✓ Reducing the interfacial energy (By reducing the elastic misfit energy)
- SCC(IASCC) resistance steel: Cr↑, Ni↑(Fully austenitic SS)

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[1] ARES-6: KR 10-1943591 (registration), PCT/KR2018/014845, US 16476597 (application)
[2] ARES-7, 8: KR 10-2292016 (registration), PCT/KR2019/017159, US 17045267 (application)

History of Alloy Design (Overview)

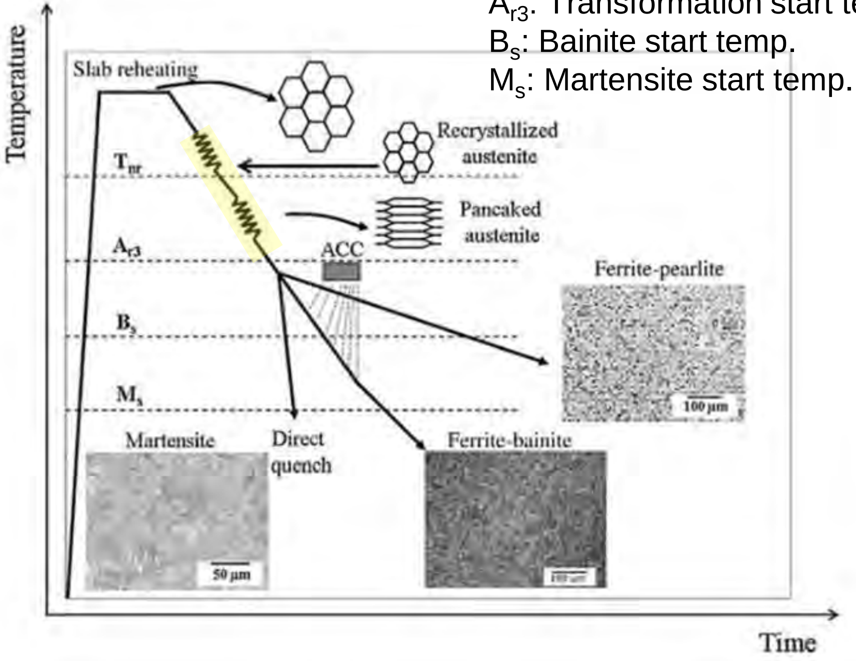
	Wt.%	Fe	Cr	Ni	C	Mn	P	S	Si	Nb	Ti	N	Mo
1st	ARES-1	Bal.	18.26	13.90	0.031	1.96	0.0440	0.0320	0.83	0.31	0.87	0.060	-
1st	ARES-2	Bal.	21.81	20.41	0.033	2.05	0.0500	0.0300	1.4	0.30	0.69	0.100	-
2nd	ARES-3	Bal.	24.25	20.91	0.014	1.5	0.0071	0.0037	1.25	0.098	0.015	0.012	-
2nd	ARES-4	Bal.	24.03	21.2	0.013	1.5	0.0049	0.0020	1.27	0.097	0.023	0.011	-
2nd	ARES-5	Bal.	23.72	21.09	0.012	1.49	0.0034	0.0011	1.25	0.099	0.022	0.008	-
3rd	**ARES-6**	**Bal.**	**24.13**	**21.07**	**0.042**	**1.32**	**0.0100**	**0.0020**	**0.23**	**0.27**	**0.023**	**0.008**	-
3rd	**ARES-7**	**Bal.**	**24.03**	**20.88**	**0.035**	**3.41**	**0.0062**	**0.0021**	**0.21**	**0.45**	-	**0.100**	-
3rd	**ARES-8**	**Bal.**	**24.12**	**20.94**	**0.034**	**3.44**	**0.0053**	**0.0022**	**0.21**	**0.46**	-	**0.100**	**0.77**

Newly Developed TMP

[1] R.L. Dalcin et al., Int. J. Eng. Tech. 8 (2019) 324
[2] J.H. Shin et al., MSE:A 775 (2020) 138986

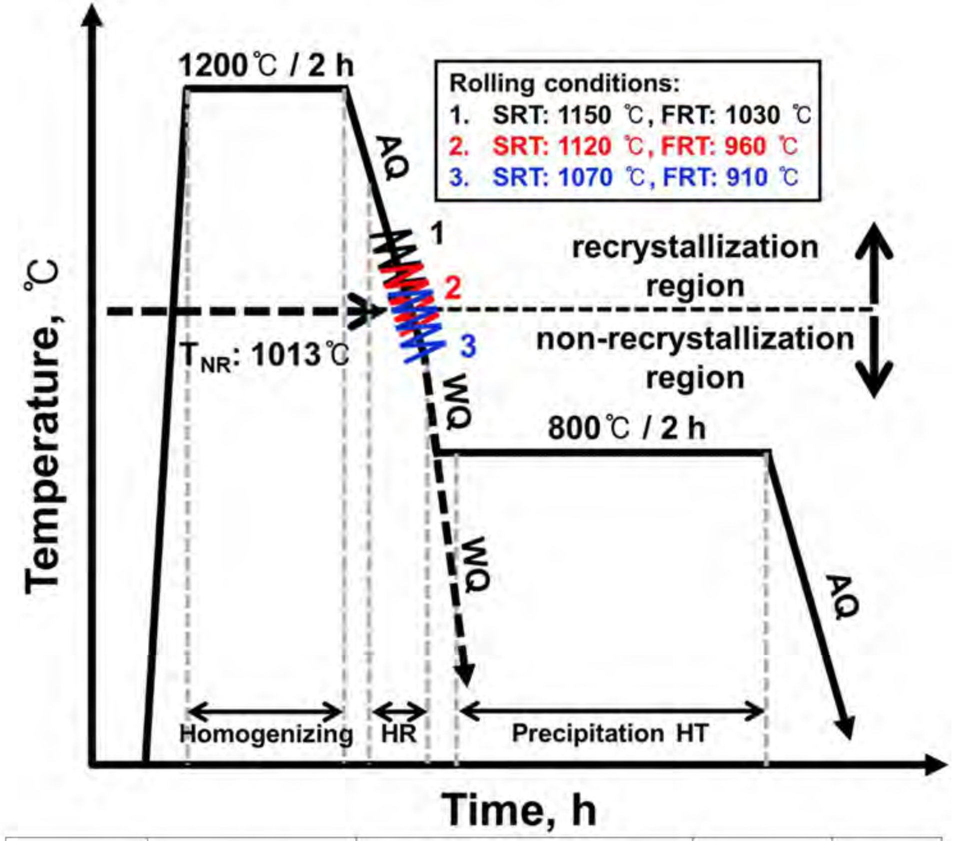
- **Motivation**

T_{NR}: non-recrystallization temp.
A_{r3}: Transformation start temp.
B_s: Bainite start temp.
M_s: Martensite start temp.



▲ Schematic of microstructure evolution in ferritic steels [1]

- **Hot rolling (+T_{NR})**
  - Uniformly distributed disl.
  - Equiaxed grains
- **Precipitation HT**
  - Nanosized precipitates

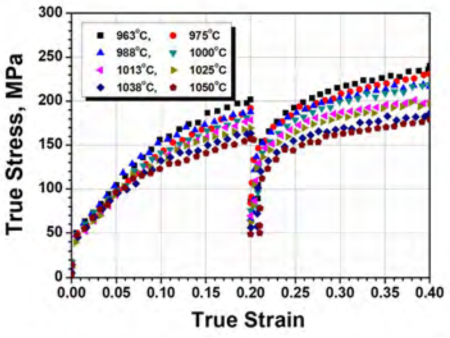


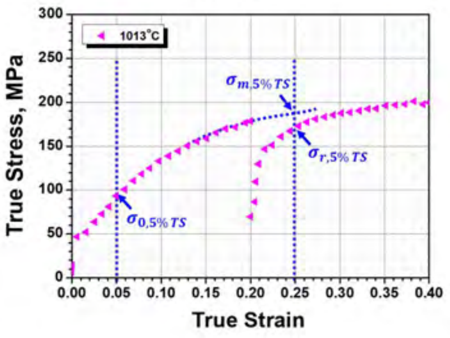
	TMP schedule, °C		Hot rolling process (total 6 passes)		Grain size, µm	LAGB[b] Fraction
	SRT	FRT	Over the T_{NR}[a]	Under the T_{NR}		
B61HR	1150	1030	6	0	10.5 (3.2)[c]	0.11
B62HR	1120	960	4	2	9.1 (3.9)	0.39
B63HR	1070	910	2	4	15.3 (9.2)	0.62

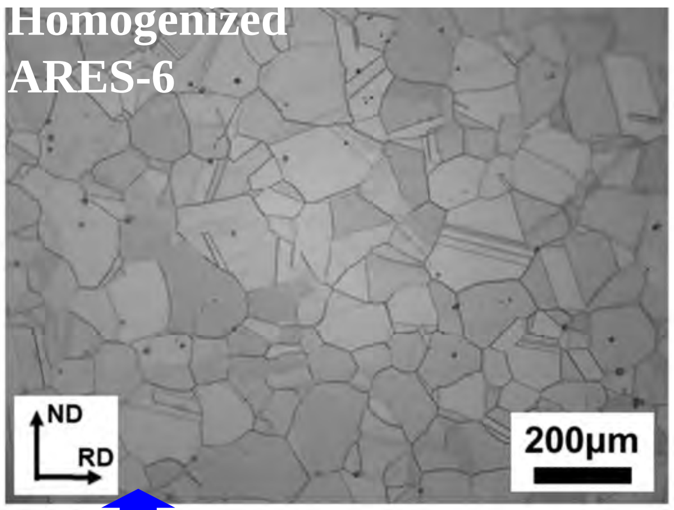
[a] T_{NR} represents the non-recrystallization temperature
[b] LAGB (2° ≤ θ < 15°)
[c] The number in parentheses represents the standard deviation of the grain size.

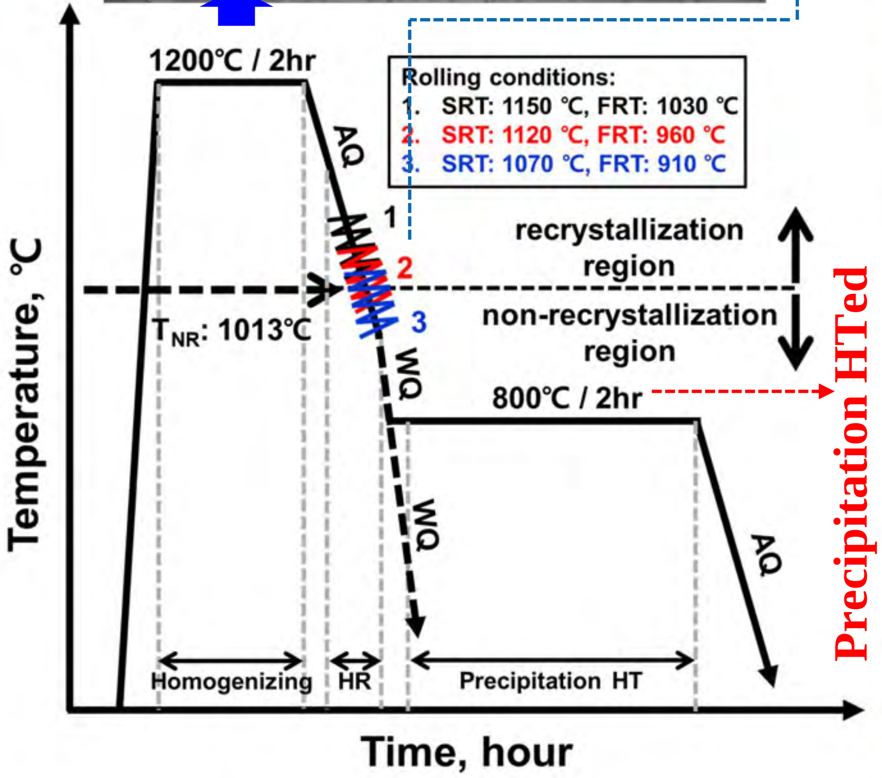
Effect of TMP on Microstructure Evolution

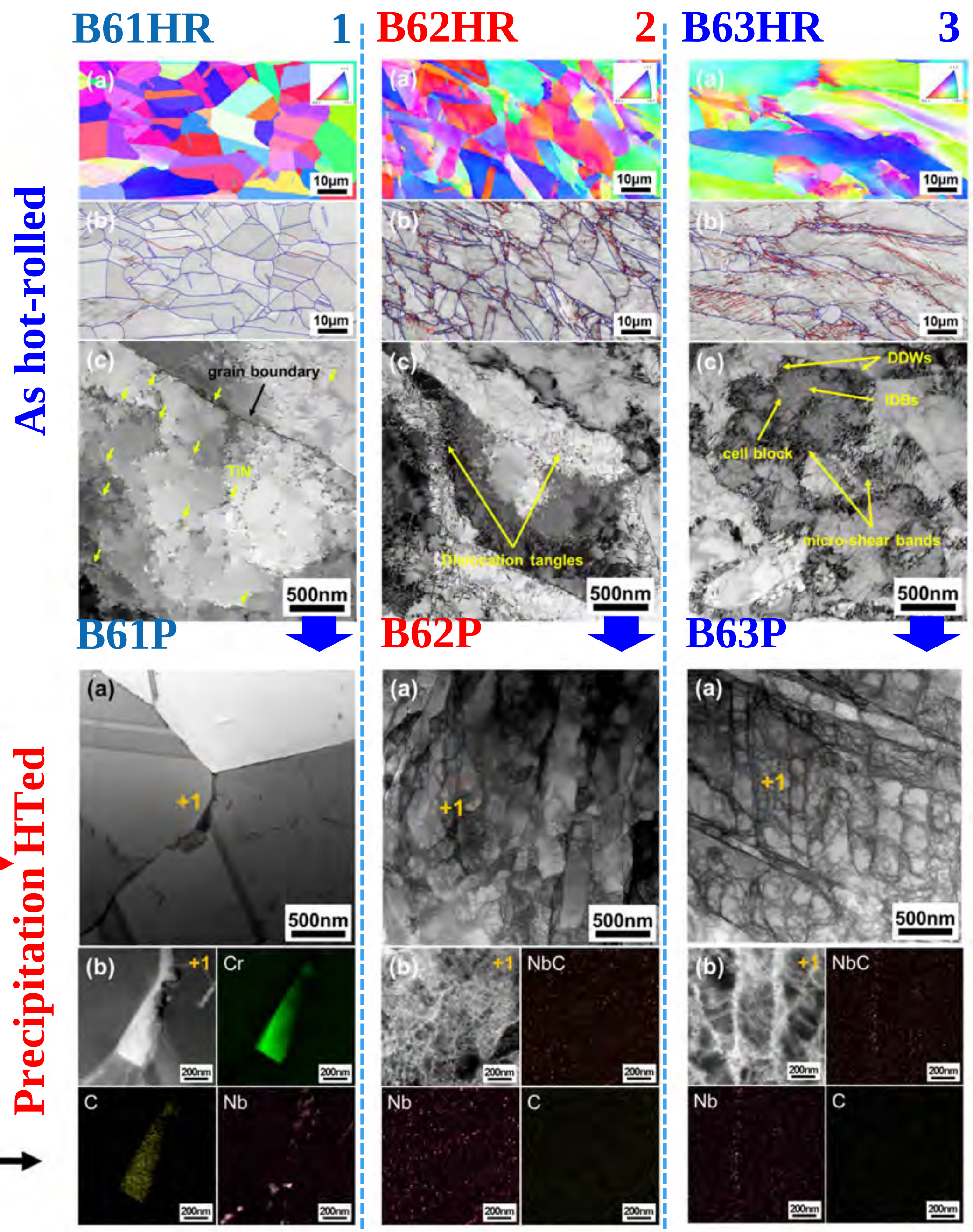
▼ Determination of non-recrystallization temp. (T_{NR})







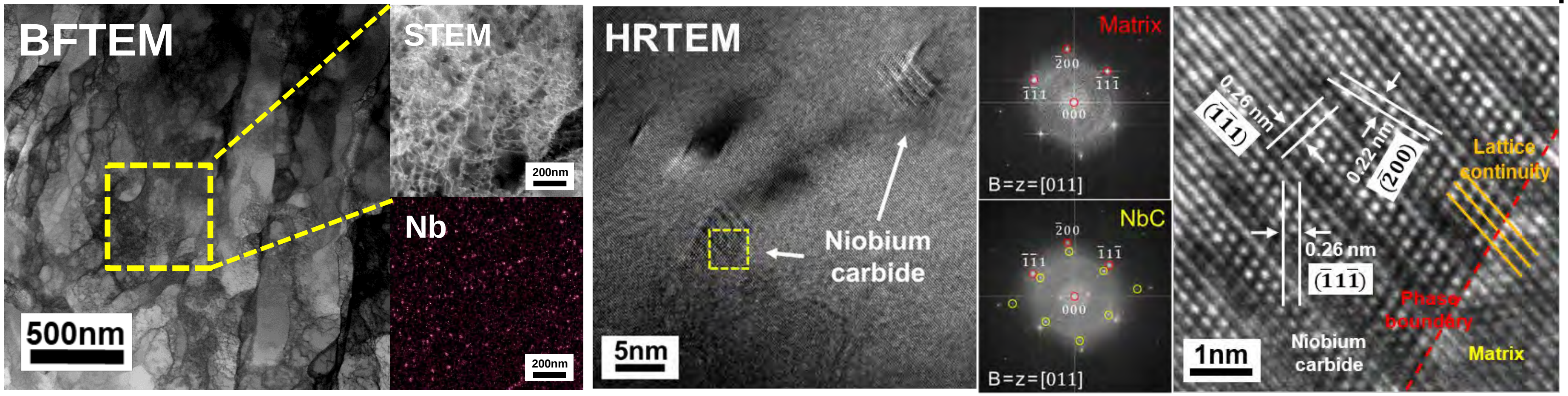




Microstructure after Precipitation HT

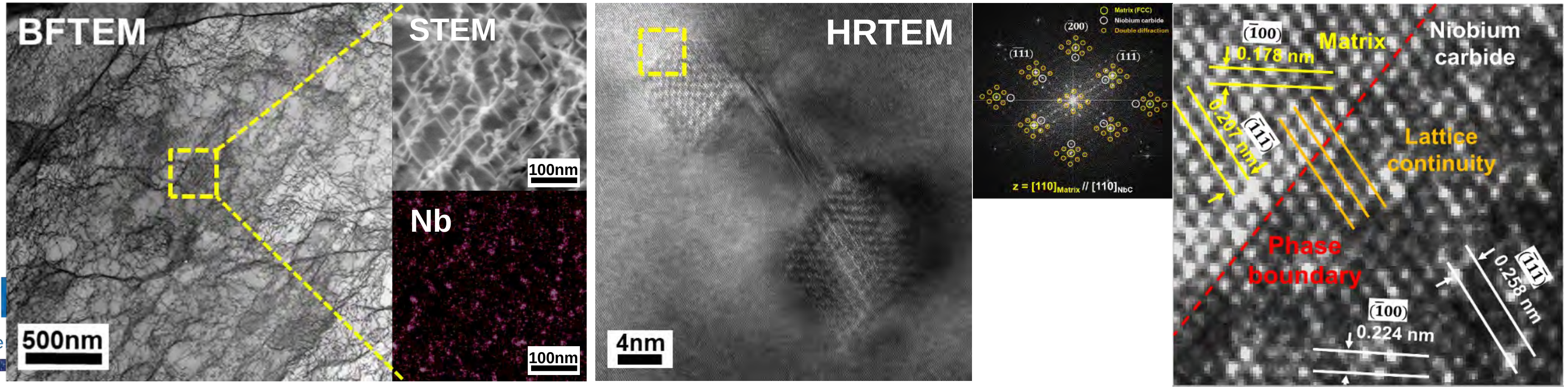
❑ ARES-6P (HT condition: 800 ℃ / 2hr)

Cube on cube relationship



❑ ARES-7P (HT condition: 750 ℃ / 2hr)

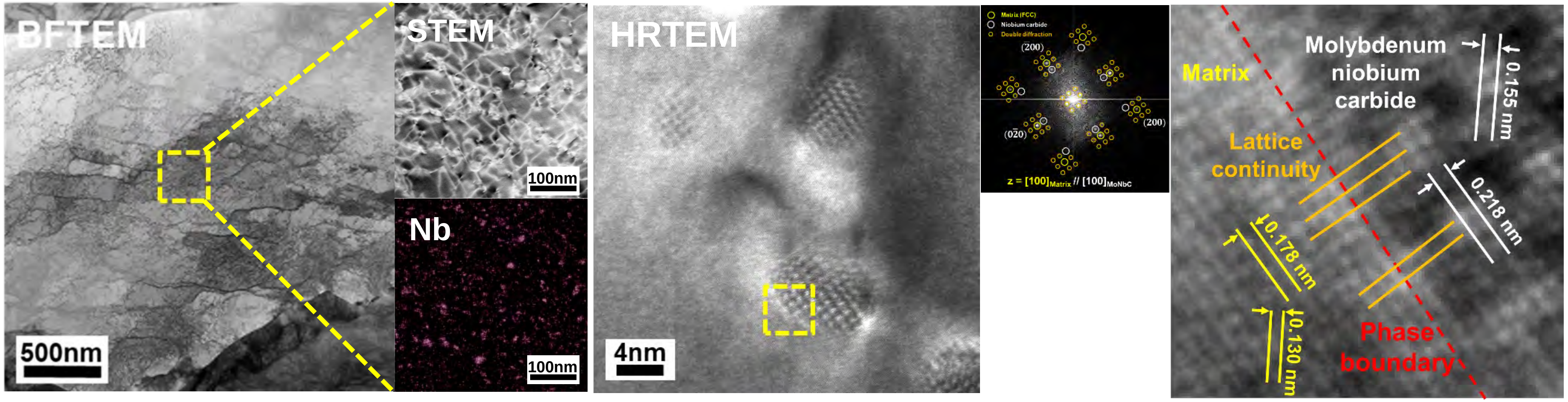
Cube on cube relationship



Microstructure after Precipitation HT

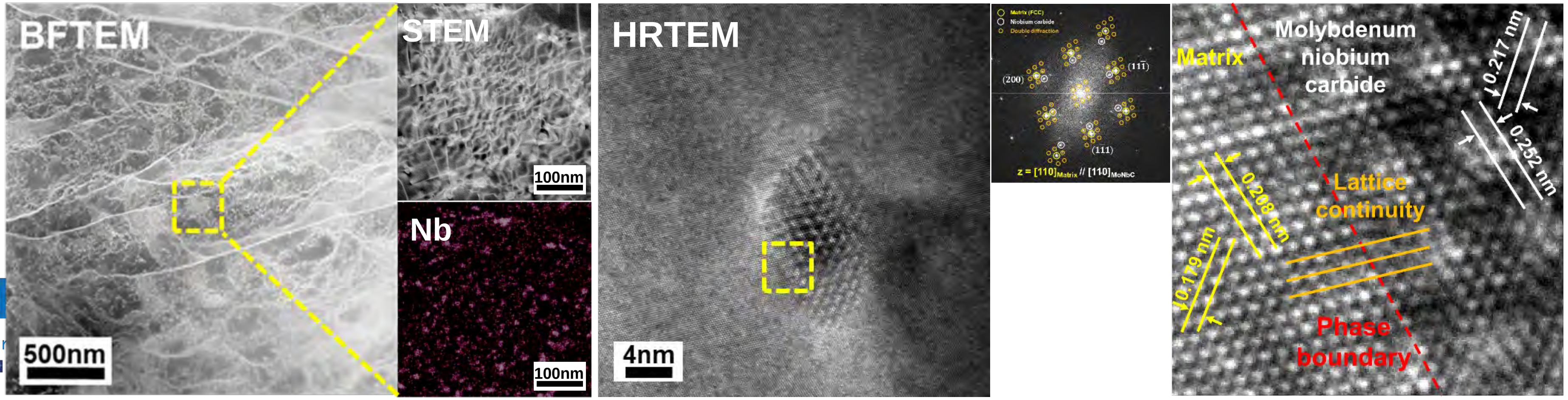
❑ ARES-8·P (HT condition: 750 ℃ / 4hr)

Cube on cube relationship



❑ ARES-8P (HT condition: 800 ℃ / 4hr)

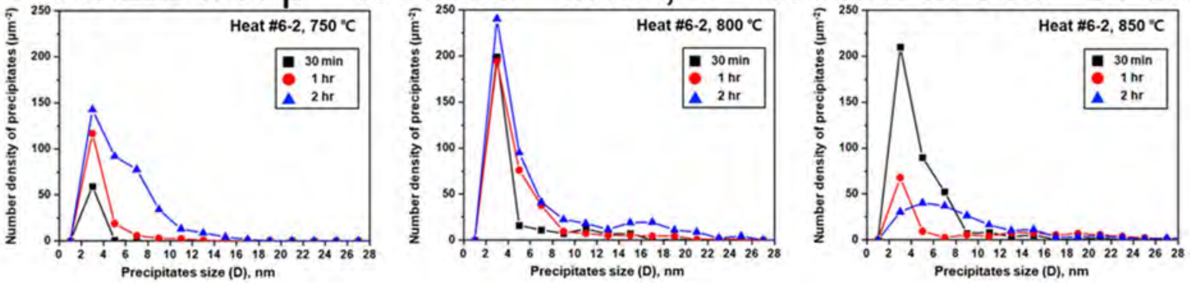
Cube on cube relationship



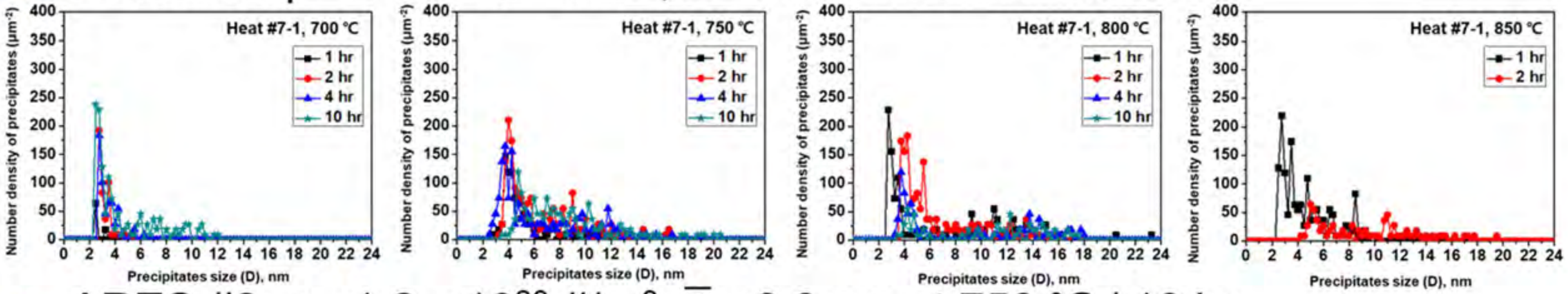
Microstructure after Precipitation HT

❑ Precipitation analysis for the ARES alloys

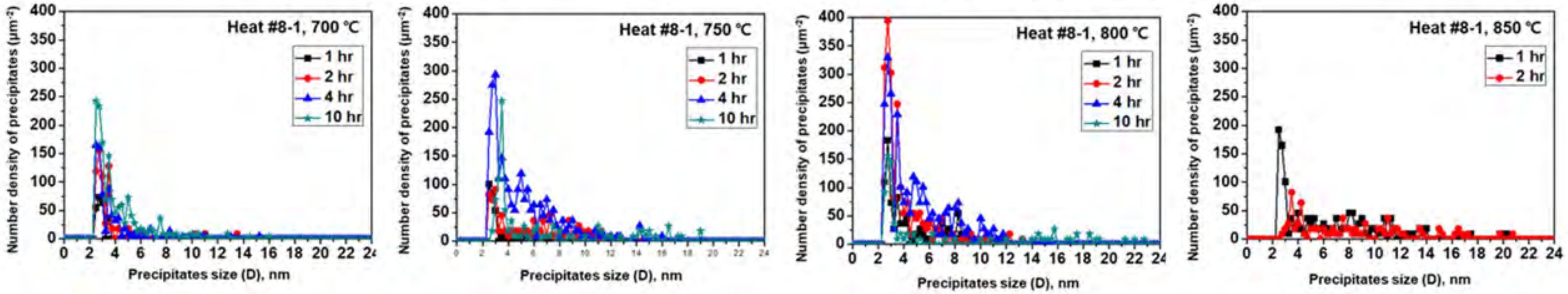
- ARES #6: ρ= 1.1 x 10^{22} #/m^3, $\overline{D}$= 8.4 nm at 800 ℃ / 2 hr

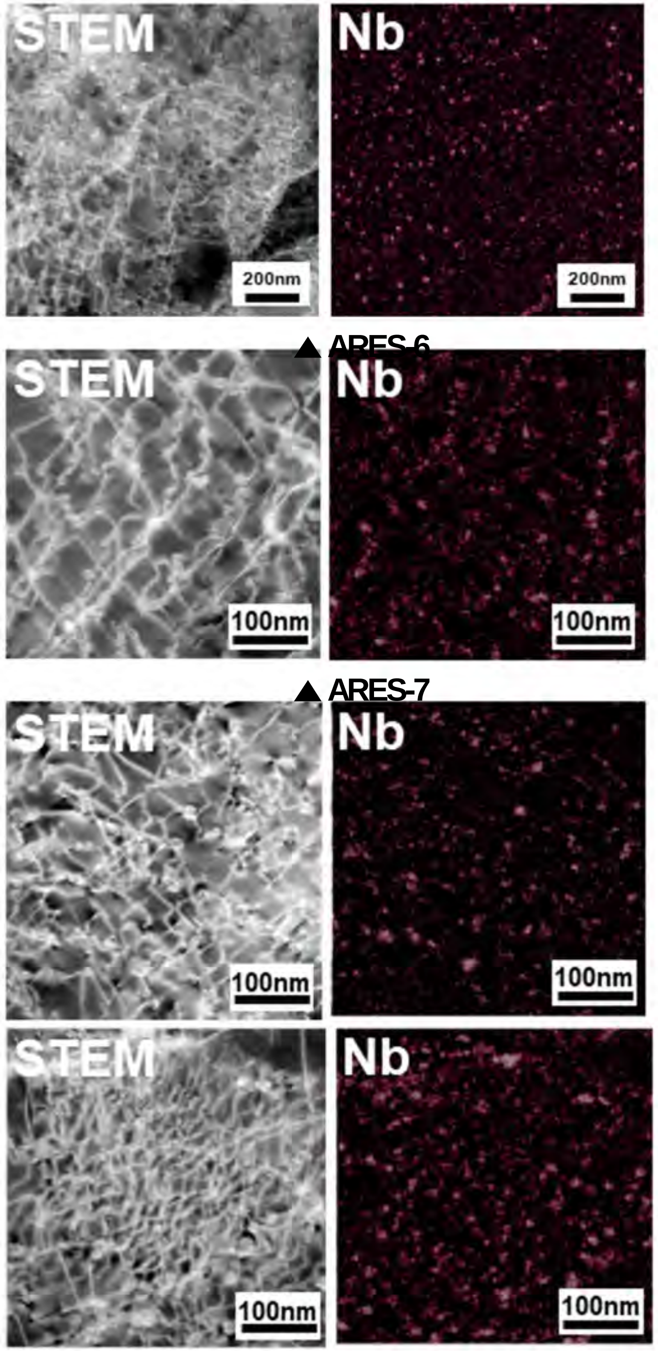


- ARES #7: ρ= 6.8 x 10^{22} #/m^3, $\overline{D}$= 7.8 nm at 750 ℃ / 2 hr



- ARES #8: ρ= 1.2 x 10^{23} #/m^3, $\overline{D}$= 6.0 nm at 750 ℃ / 10 hr
  ρ= 1.3 x 10^{23} #/m^3, $\overline{D}$= 5.8 nm at 800 ℃ / 4 hr





▲ ARES-6

▲ ARES-7

▲ ARES-8

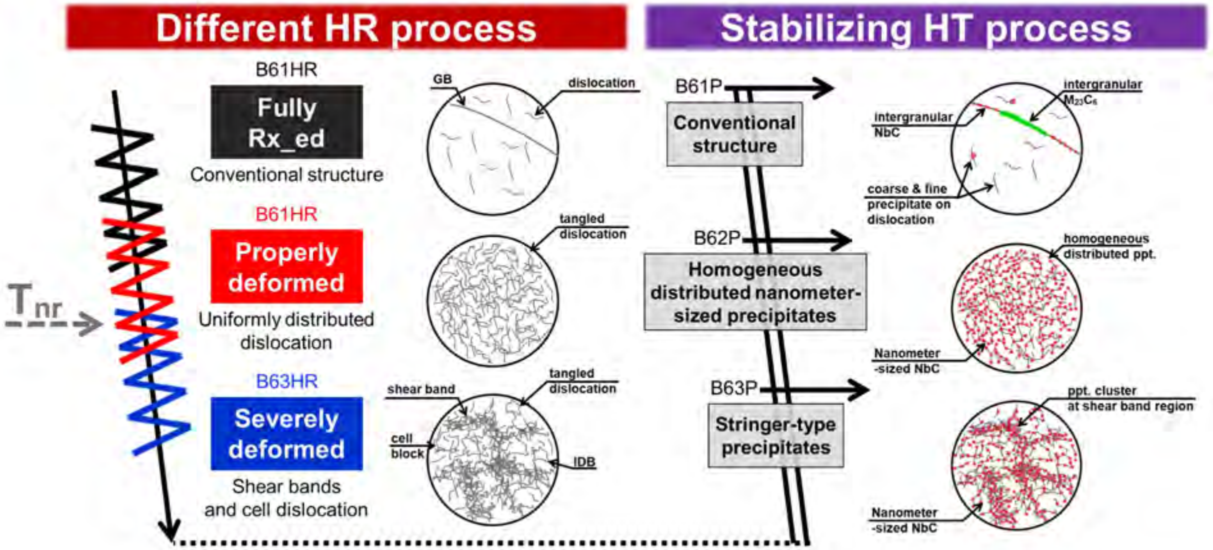
Summary

[1] Patent: KR 10-1943591
[2] B. Rouxel et al., EPJ Nuclear Sci. Technol. 2 (2016)
[3] J.H. Shin et al., MSE:A 775 (2020) 138986
[4] K. Dawson et al., J. Nucl. Mater. 464 (2015) 200

❑ Development of ARES alloy (nanosized precipitates)

- Newly designed chemical composition
  - High IASCC or SCC resistance ⇒ High Cr, Ni (fully austenite phase)
  - High radiation resistance ≪ Nanosized precipitates ⇒ control the minor element ⇒ Nb, Ti, Mo, Mn, C, N
- Newly developed thermo-mechanical processing

Precipitate	Stabilized 347 SS [1]	CW15-15 Ti [2]	ARES [3]	FM-ODS [4]
Diameter	20–100 nm	~ 2nm	<10 nm	<10 nm
Density (m^{-3})	$<10^{20}$	(locally) 3–6 x 10^{22}	10^{22} – 10^{23}	10^{22} – 10^{23}



◀ Schematic of the TMP and the resulting microstructures of the ARES [1]

Topic II

Radiation resistance of ARES alloy

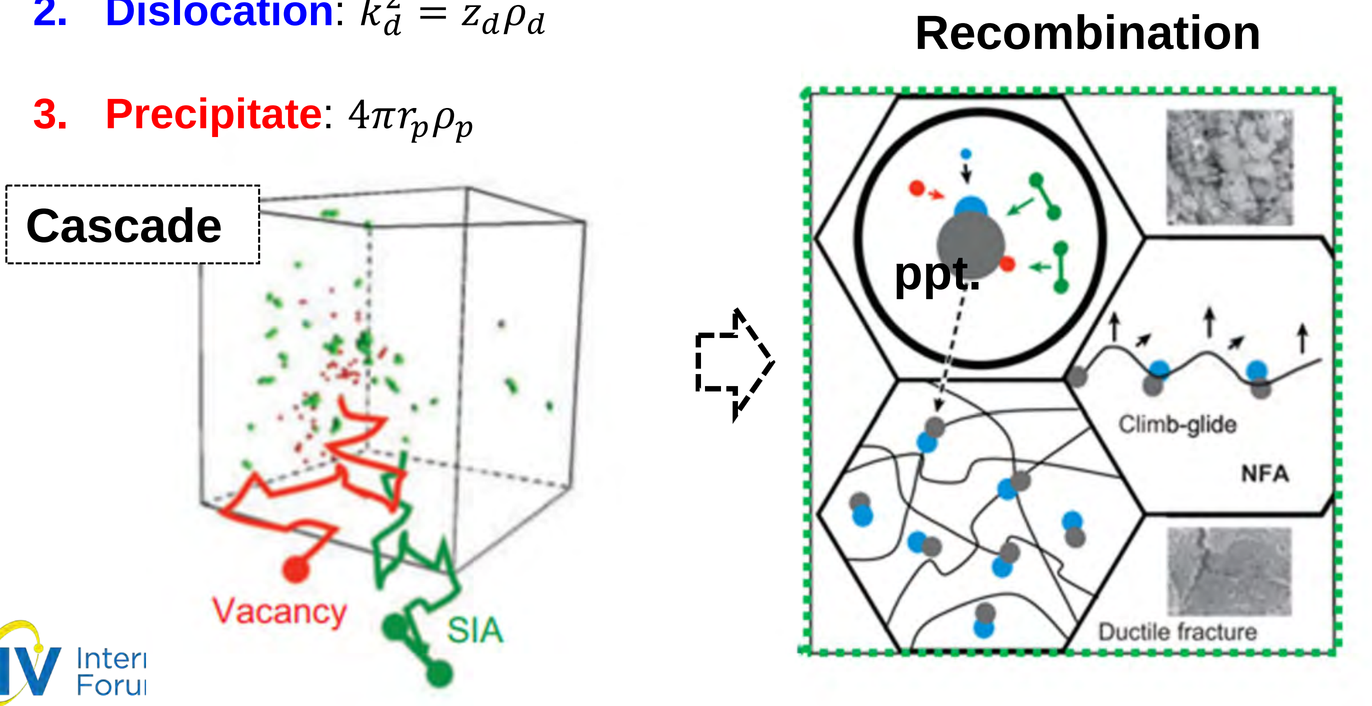
1) Void swelling
2) Radiation-induced hardening

[1] G. Was, Fundamentals of radiation materials science: metal and alloys (springer, 2016)
[2] G.R. Odette et al., Annu. Rev. Mater. Res. 38 (2008) 471
[3] C. Sun et al., Sci. Rep. 5 (2015) 7801

Effect of Defect Sinks on Radiation Resistance

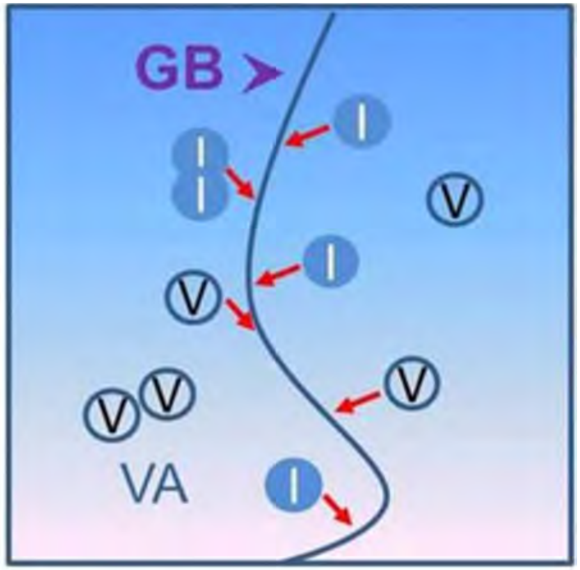
- Trapping or Annihilating point defects
- Typical sink sites [1]

1. Grain boundary: $k_{gb}^2 = 24/d^2, d < 10^{-3} cm$ or $k_{gb}^2 = 6k/d^2, d > 10^{-3} cm$
2. **Dislocation**: $k_d^2 = z_d \rho_d$
3. **Precipitate**: $4\pi r_p \rho_p$



▲ Schematic of cascade production of vacancies and self-interstitial atoms (SIA), and self-healing mechanism along the precipitate [2]

Effect of defect sinks for radiation-induced defects

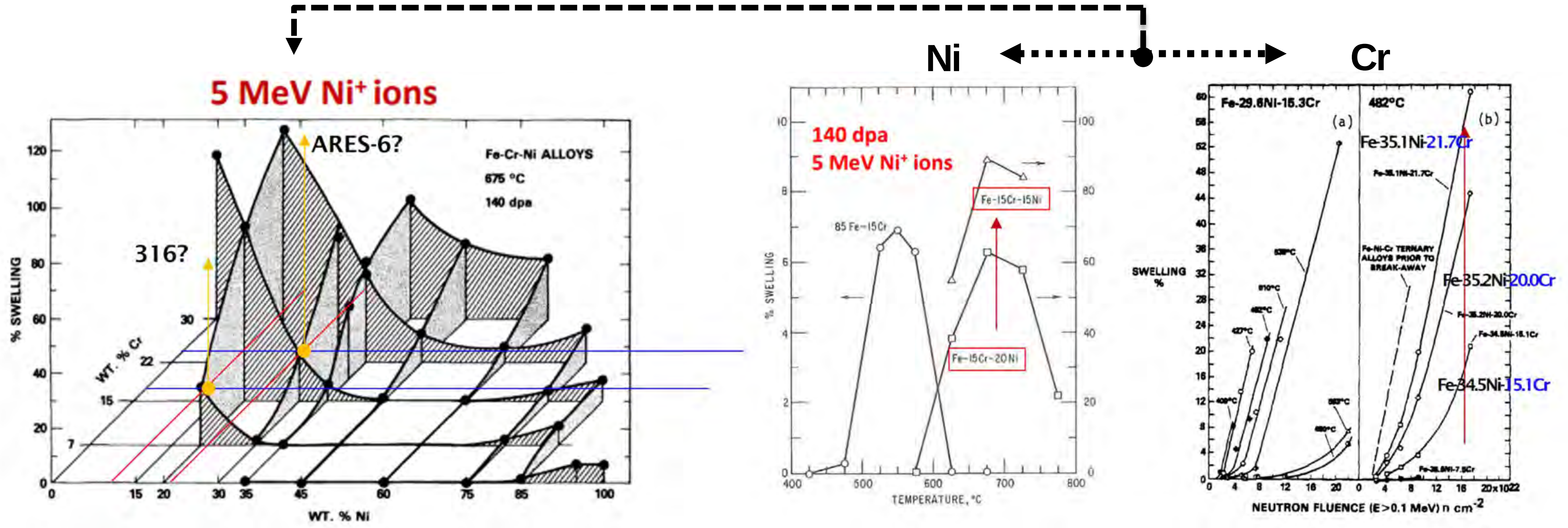


▲ Schematic of interstitials and vacancies migrate towards the grain boundaries [3]

[1] F.A. Garner et al., in Radiation-Induced Changes in Microstructure: 13th International Symposium (Part I), ASTM STP 955.
[2] B. Esmailzadeh et al., in Effects of Radiation on Materials: Twelfth International Symposium, ASTM STP 870
[3] S.B.Krivit et al., 2011, Nuclear Energy Encyclopedia, Wiley, Hoboken, NJ

Effect of Major Elements on Radiation Resistance

– Both the nickel and chromium concentrations are known to affect vacancy mobility in Fe-Cr-Ni alloys [1, 2]



[W. G. Johnston et al., J. Nucl. Mater. 54 (1974) 24.]

[F. A. Garner, DAFS Ouarterly Report, DOE/ER- 0046/14 (Aug. 1983) 133, to be published in Proc. of AIME Symp. on Tailoring and Optimizing Materials for Nuclear Applications, Feb. 1984, Los Angeles.]

1. The high Ni-v binding energy ($E_{v\text{-}Ni}$=0.26eV) => decrease vacancies mobility: act as recombination sites for punctual defects or as nucleation sites for cavities
2. The low Cr-v binding energy ($E_{v\text{-}Cr}$=0.06eV) =>increase vacancies mobility: act as depletion at boundaries [3]

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[1] C. Sun et al., Material Science Engineering (2015) 5:7801
[2] K. Yoshikawa et al., Journal of Materials Engineering and Performance (1988) 10:69-84
[3] S. Balaji et al., Journal of Nuclear Materials (2015) 467:368-372
[4] S.J. Zinkle et al., Nuclear Fusion 57 092005 (2017) 17

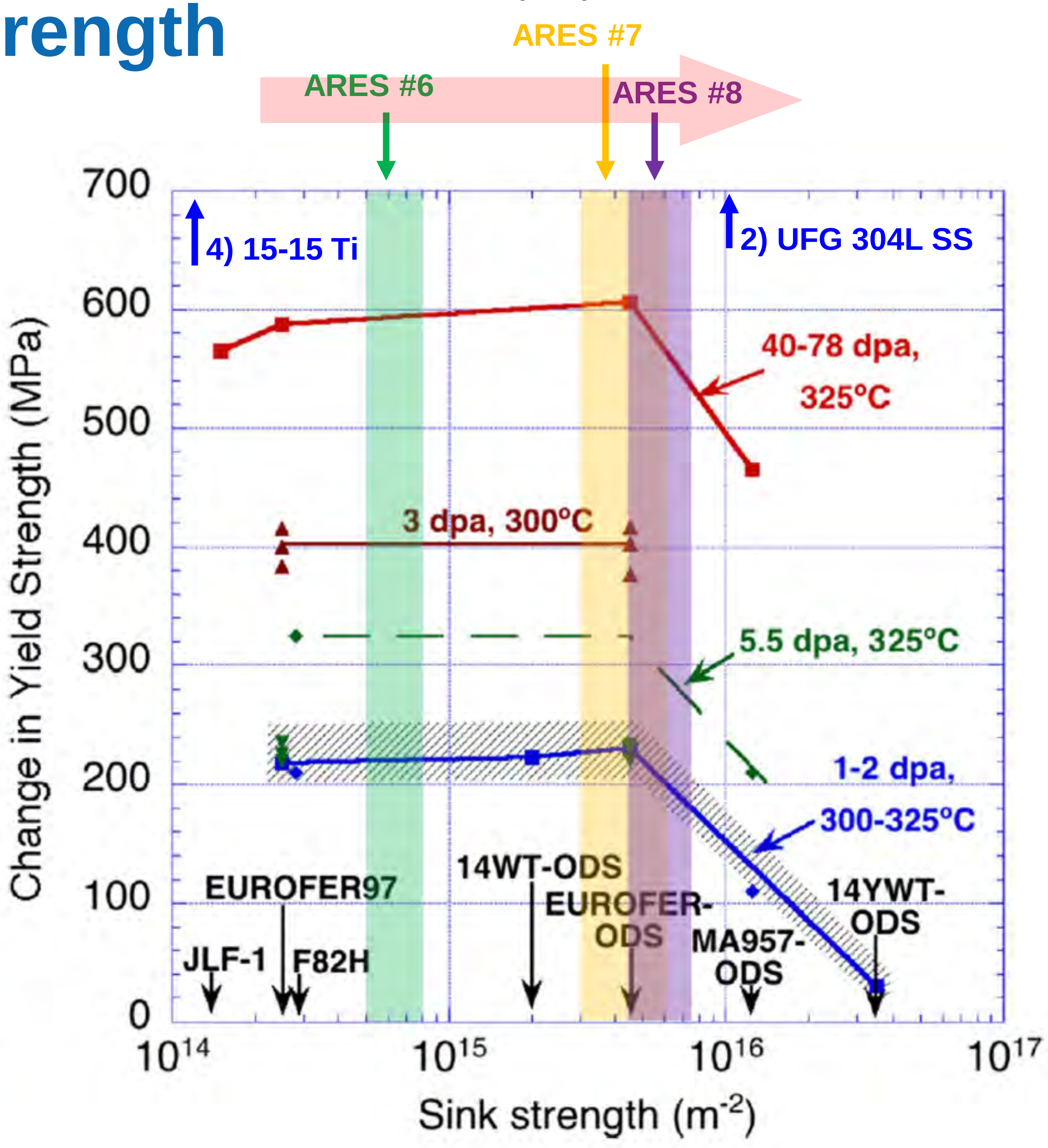
Qualitative Analysis Result: Sink Strength

– Result of calculation

	Density (#/m³)	Mean radius (nm)	Sink strength (m⁻²)
ARES #6	1.1×10^{22}	4.2	5.8×10^{14}
ARES #7	6.8×10^{22}	3.9	3.3×10^{15}
ARES #8·	1.2×10^{23}	3.0	4.5×10^{15}
ARES #8	1.3×10^{23}	2.9	4.7×10^{15}

– Comparing a sink strength w/ the reference alloys

1) CG 304L SS: 5.00×10^{12} /m² [1]
   - Grain diameter: ~35 μm
2) UFG 304L SS: 1.10×10^{16} /m² [1]
   - Grain diameter: ~100 nm
3) TP347H SS: 1.45×10^{13} /m² [2]
4) 15-15 Ti (D9): 1.39×10^{14} /m² [3]



▲ Effect of initial sink strength on the low temperature radiation hardening behavior of fission reactor irradiated FMSs [4]

Objective

Evaluation of the radiation resistance of ARES containing uniformly distributed nanosized NbC carbides under heavy ion irradiation of the ARES alloy

Radiation experiment in the target neutron environment → Investigation of the radiation resistance characteristics of ARES

Conducting ion irradiation to emulate neutron irradiation

Compared to commercial austenitic SS in terms of
: void swelling, radiation hardening
under low (8.5 dpa) & high (200dpa) dose

Measurement of void size and density, and calculation of void swelling → Effect of void swelling resistance on nanosized precipitates

Measuring the radiation induced hardening by nano-indentation → Evaluation of hardening mechanisms after radiation compared to commercial SS

Effect of Precipitates on Swelling Resistance

- Initial microstructure and chemical composition
- Materials: ARES-6 and 316 SS

	Fe	Cr	Ni	Mn	Si	Nb	C	Ti	N	Mo
316 SS	Bal.	17.09	10.28	0.58	0.56	-	0.080	-	-	2.1
ARES–6	Bal.	24.13	21.07	1.32	0.23	0.27	0.042	0.023	0.008	-

▲ **Chemical composition of the 316 SS and ARES-6 (ICP-AES, C/S-KS D 1804/1803)**

- Thermo-mechanical processing

	Thermo-mechanical processing				Grain size (µm)	LAGB[a] fraction	Precipitates	
	Homogenizing	Hot rolling SRT[a]	Hot rolling FRT[b]	Post heat treatment			Mean diameter (nm)	Density ($x10^{22}$ /m^3)
316 SS	-	-	-	Solution annealing[c], 1050°C/2hr	92.7±17.1	~0.02	N/A	
ARES-6SA	1200°C/2hr	1120°C	960°C	Solution annealing, 1100°C/1hr	47.2±4.1	~0.02	N/A	
ARES-6HR	1200°C/2hr	1120°C	960°C	X	9.1±3.9	~0.39	N/A	
ARES-6P	1200°C/2hr	1120°C	960°C	Precipitation heat treating, 800°C/2hr	10.3±4.4	~0.41	8.4	1.1±0.3

[a] SRT represents the starting rolling temperature
[b] FRT represents the final rolling temperature
[c] Presenting only final heat treatment condition
[d] LAGB: 2° ≤ θ < 15°

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J.H. Shin et al., J. Nucl. Mater. 564 (2022) 153678

Void Swelling Resistance Under Low Dose Condition (~8.5 dpa)

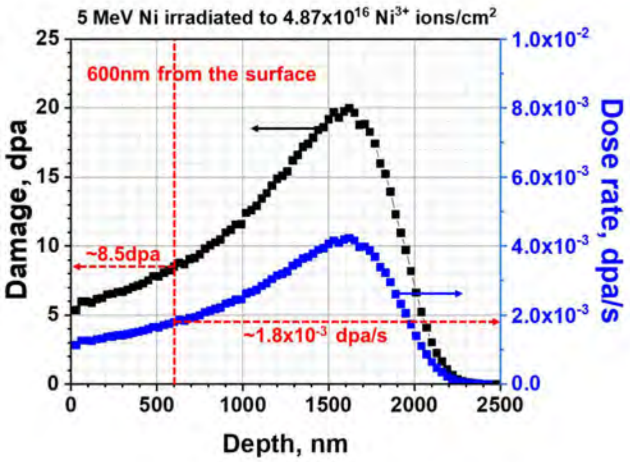
– Irradiated by MIT

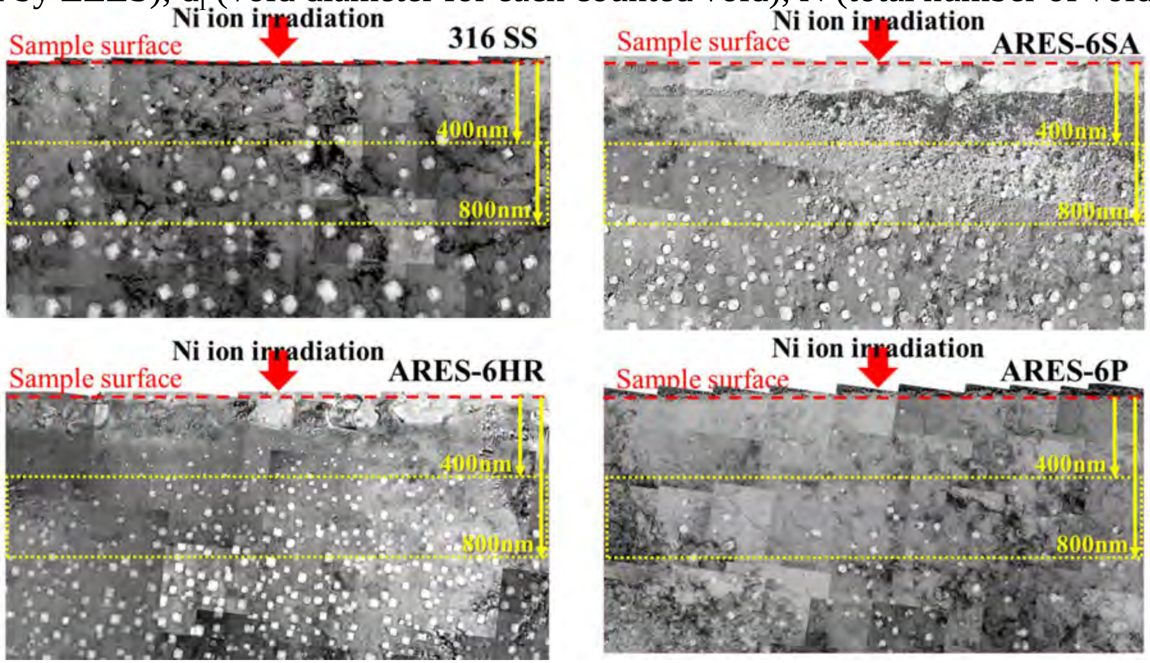
- 1.7 MV Tandem ion accelerator (5 MeV Ni^{3+} ions at ~500 ℃)
- Target damage: **~8.5 dpa** at 600 nm from the surface (1.8×10^{-3} dpa/s)

– Calculation depth profiles of the radiation damage by SRIM

– The equation of void swelling: $S(\%) = \dfrac{\frac{\pi}{6}\sum_{i=1}^{N} d_i^3}{A\ X\ t - \frac{\pi}{6}\sum_{i=1}^{N} d_i^3}\ X\ 100$

- A (observed area), t (sample thickness measured by EELS), d_i (void diameter for each counted void), N (total number of voids counted in each area)





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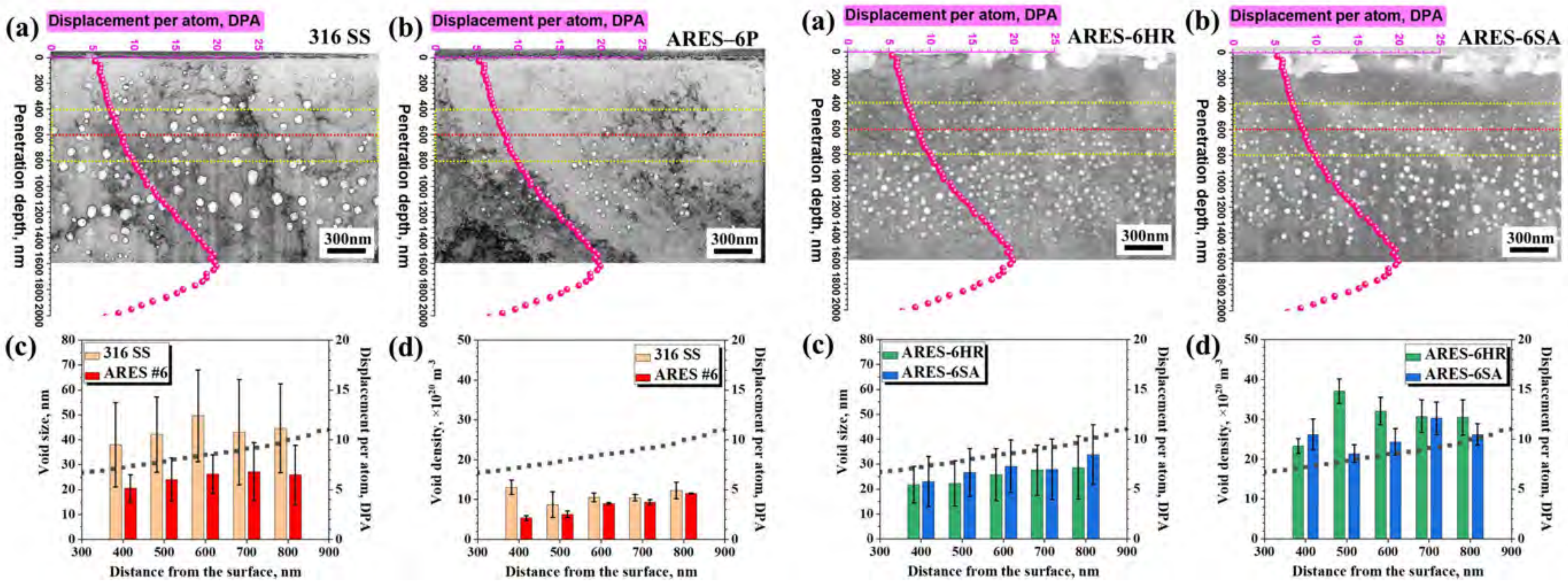
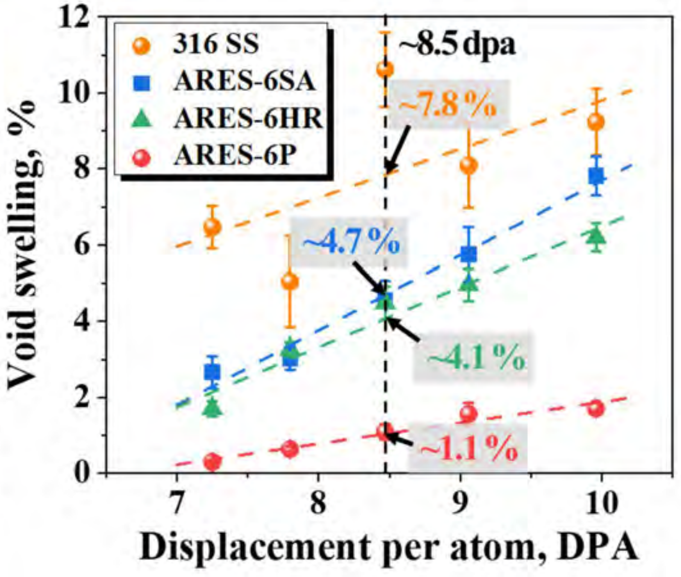
Void Swelling Resistance Under Low Dose Condition (~8.5 dpa)

– Quantification of the void swelling





- ARES-6P >> ARES-6HR & SA >> 316 SS
- A large amount of nanosized **precipitates** ⇒ **dominant factor**
- High Ni contents can contribute somewhat
- However, dislocation itself would not provide effective sink sites

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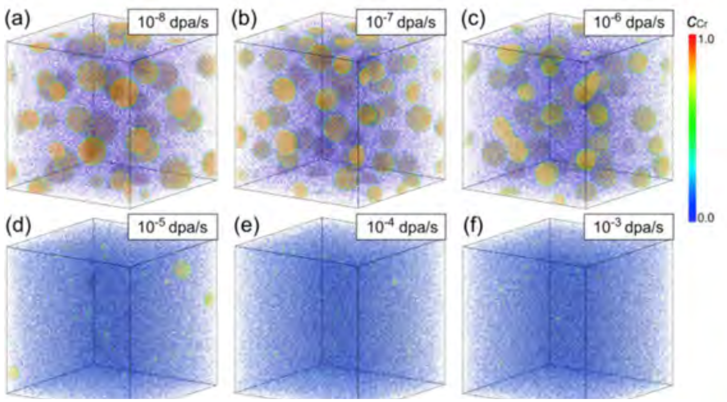
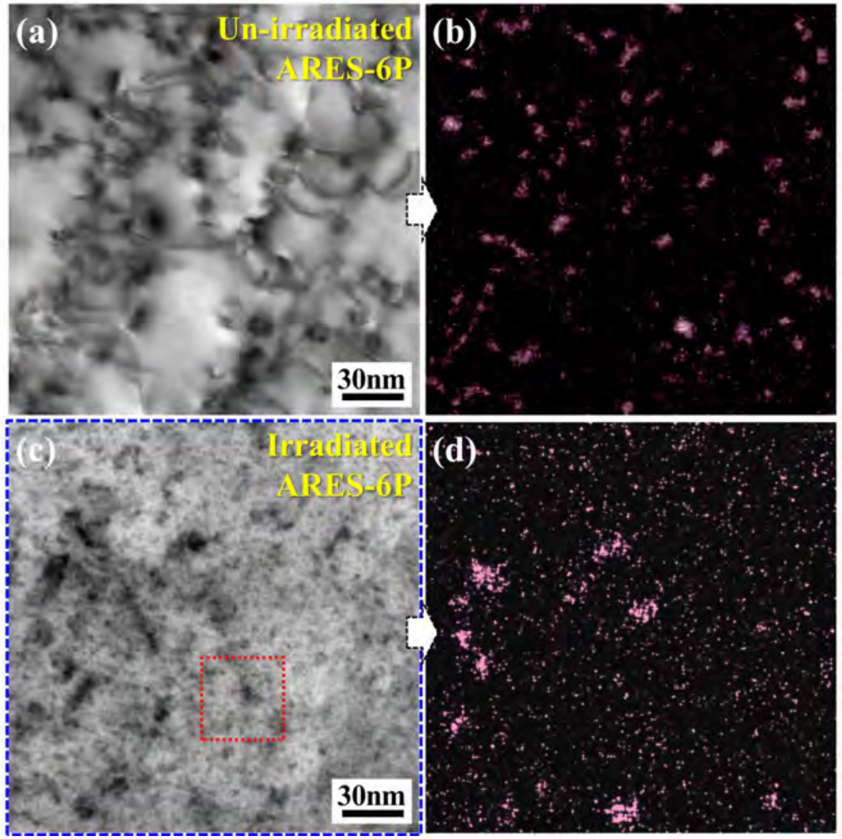
[1] J.H. Shin et al., J. Nucl. Mater. 564 (2022) 153678
[2] J.-H. Ke et al., Acta Mater. 164 (2019) 586

Stability of Nanosized NbC Precipitates

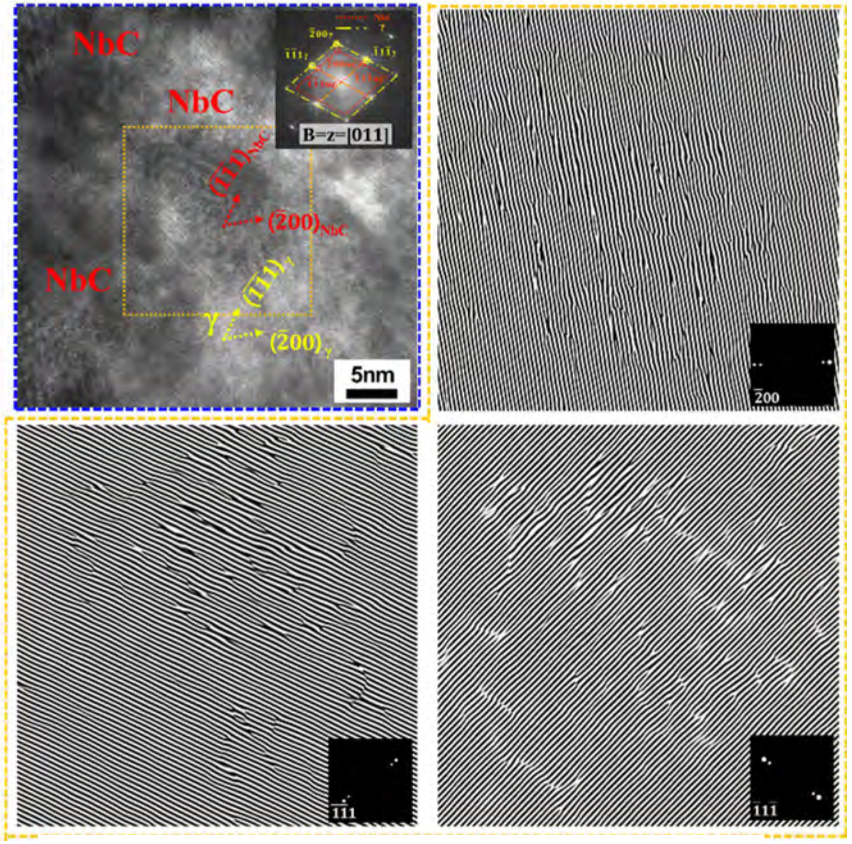
– Microstructural instability caused by extreme conditions? [1]
  - Decrease the volumetric number density
  - Increase the average size



▲Simulation results showing α′ precipitation in Fe-15Cr at 300 ℃ irradiated to 10 dpa depending on the dpa rate [2]



▲ Simulations for neutron by heavy ion irradiation
» including strong cascade mixing



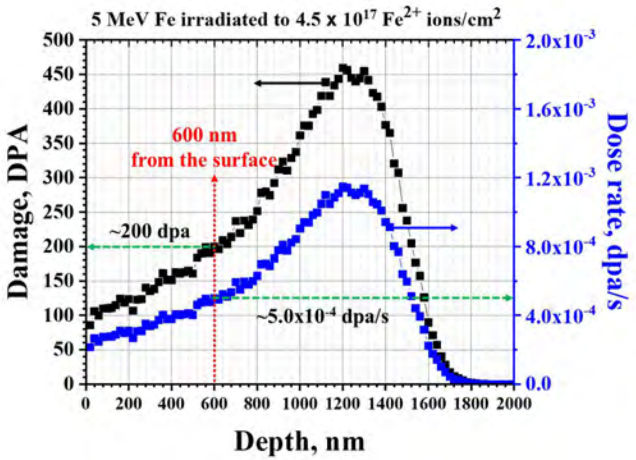
▲ TEM analysis of nanosized NbC precipitates after ion irradiation

J.H. Shin et al., J. Nucl. Mater. (2022) Under-review

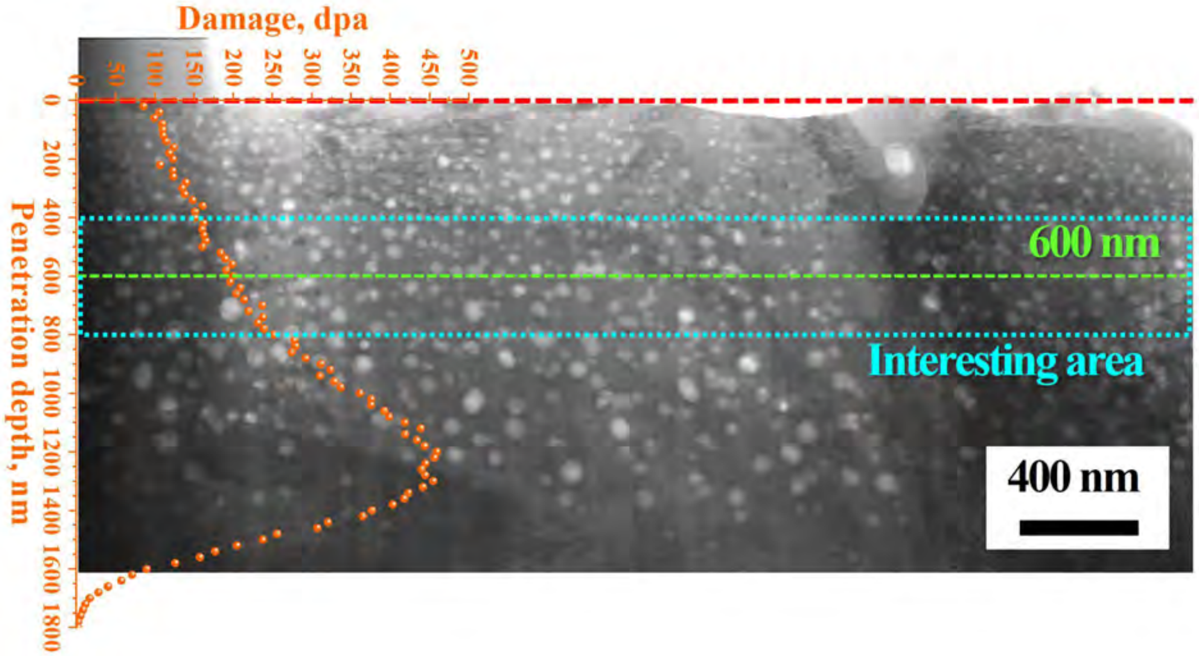
Void Swelling Resistance Under Low Dose Condition (~200 dpa)

– Irradiated by Texas A&M
  - 1.7 MV Tandem ion accelerator (5 MeV Fe^{2+} ions)
  - Target damage: **~200 dpa** at 600 nm from the surface (5.0 × 10^{-4} dpa/s) ⇒ dose rate effect
  - Test temperature: 500 ℃ and 575 ℃ ⇒ temperature effect

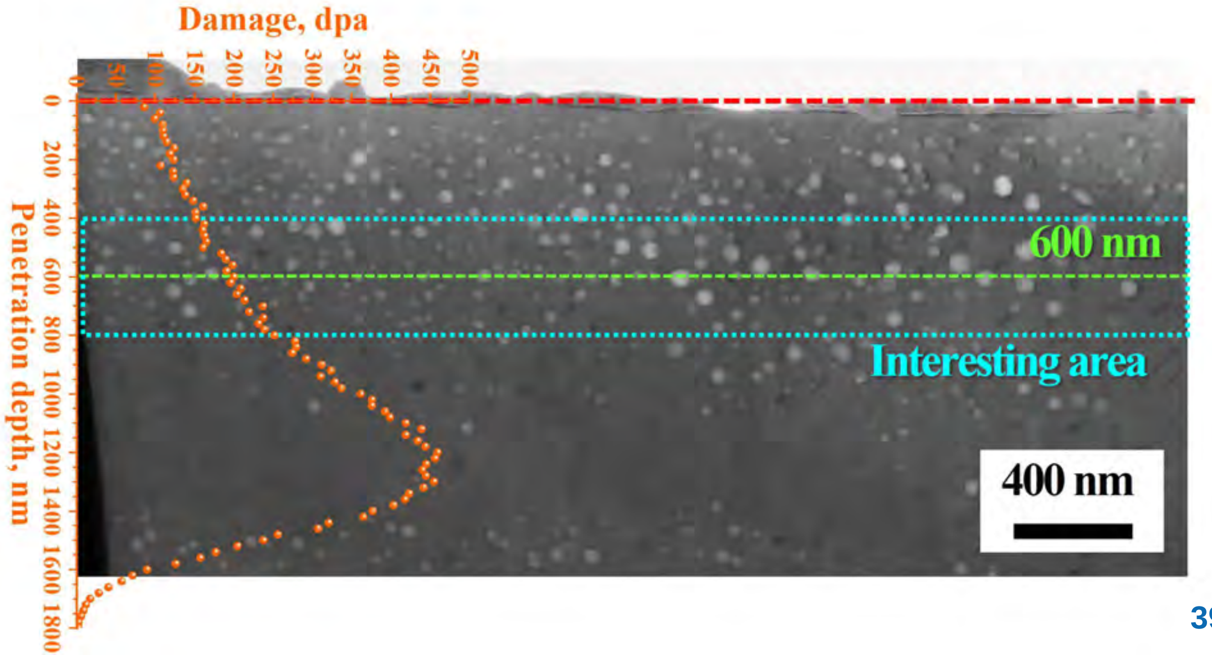
– Calculation depth profiles of the damage by SRIM



SS 316 / Irradiated at 500 °C



ARES-6P / Irradiated at 500 °C



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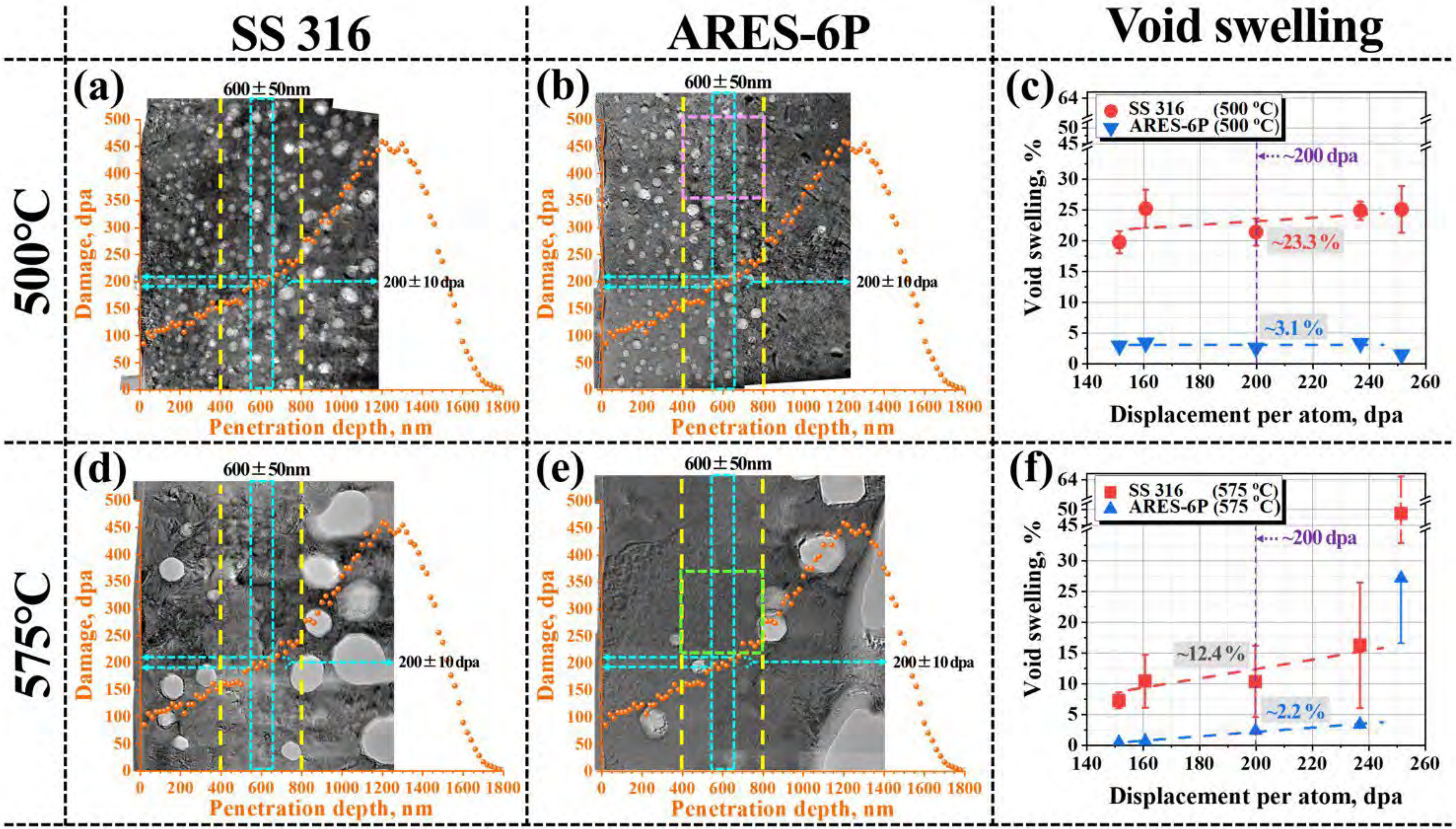
Void Swelling Resistance Under Low Dose Condition (~200 dpa)

– Quantification of the void swelling



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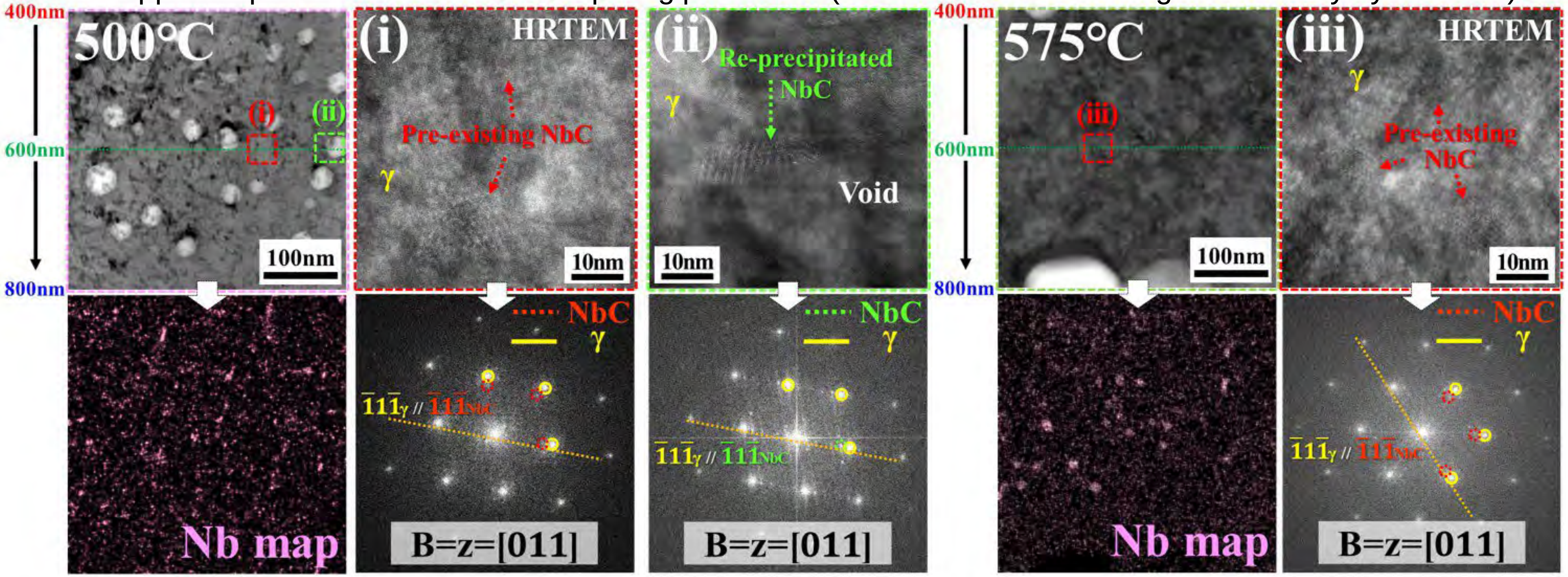
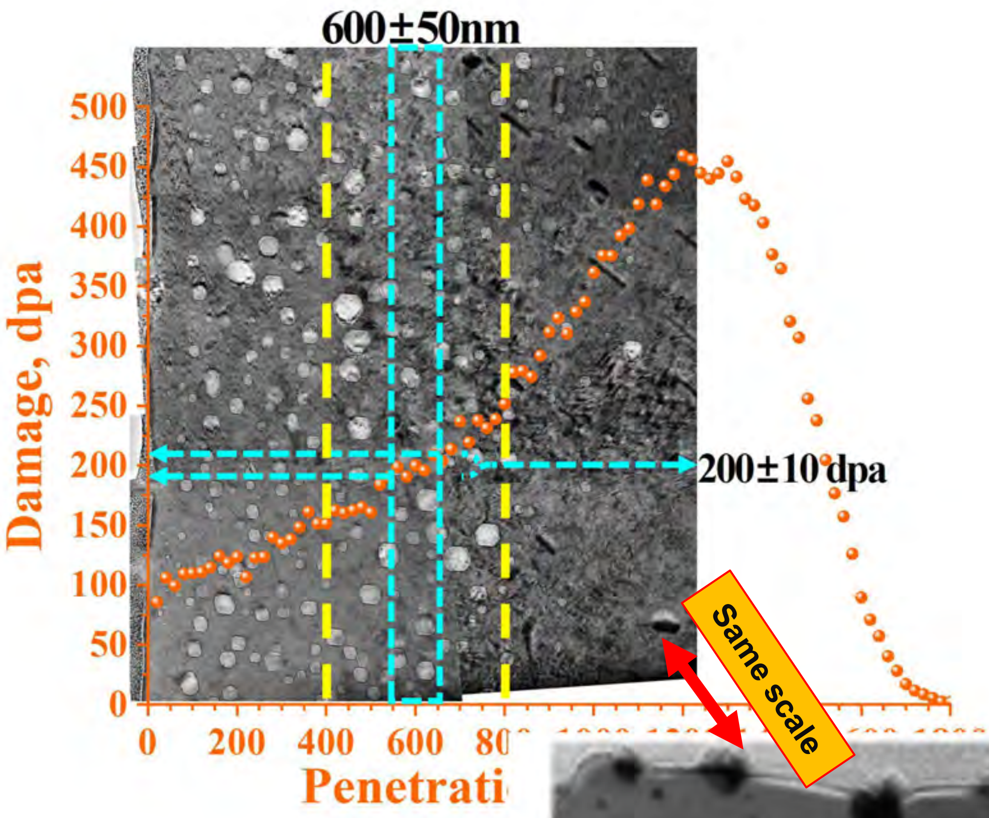
Stability of Nanosized NbC Precipitates

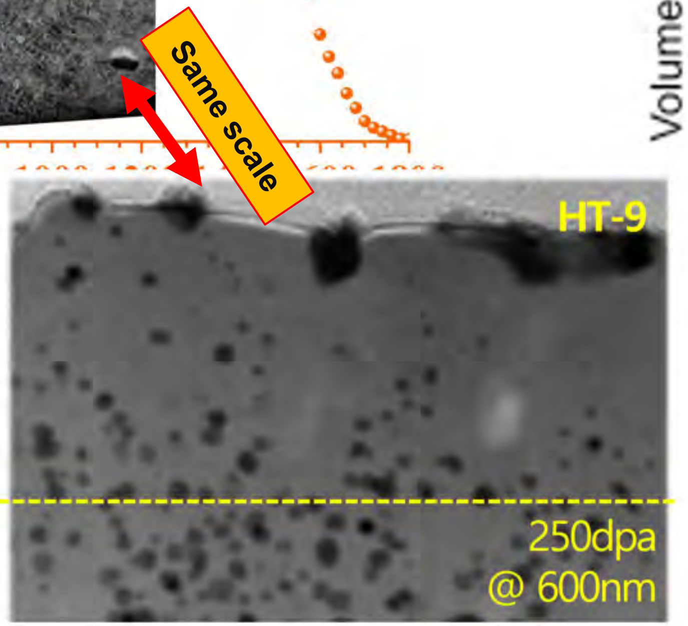
- BFTEM & Nb map – regions from 400 nm to 800 nm
  - $\bar{D}_{NbC}^{500℃} = \sim 6.3\ nm, \bar{\rho}_{NbC}^{500℃} = \sim 3.1 \times 10^{22} m^{-3}$
  - $\bar{D}_{NbC}^{575℃} = \sim 7.7\ nm, \bar{\rho}_{NbC}^{575℃} = \sim 0.9 \times 10^{22} m^{-3}$ // $\bar{D}_{NbC}^{initial} = \sim 8.4\ nm, \bar{\rho}_{NbC}^{initial} = \sim 1.1 \times 10^{22} m^{-3}$
  - Similar microstructural features with initial microstructure
- Pre-existing precipitates: away from the void, re-precipitated precipitates: nearby void
  - Cube-on-cube orientation relationship: with $[011]_{\gamma} \parallel [011]_{NbC}$ and $(\bar{1}1\bar{1})_{\gamma} \parallel (\bar{1}1\bar{1})_{NbC}$
  - Non-apparent phase boundaries ⇒ competing processes (dissolution due to mixing vs. recovery by diffusion)



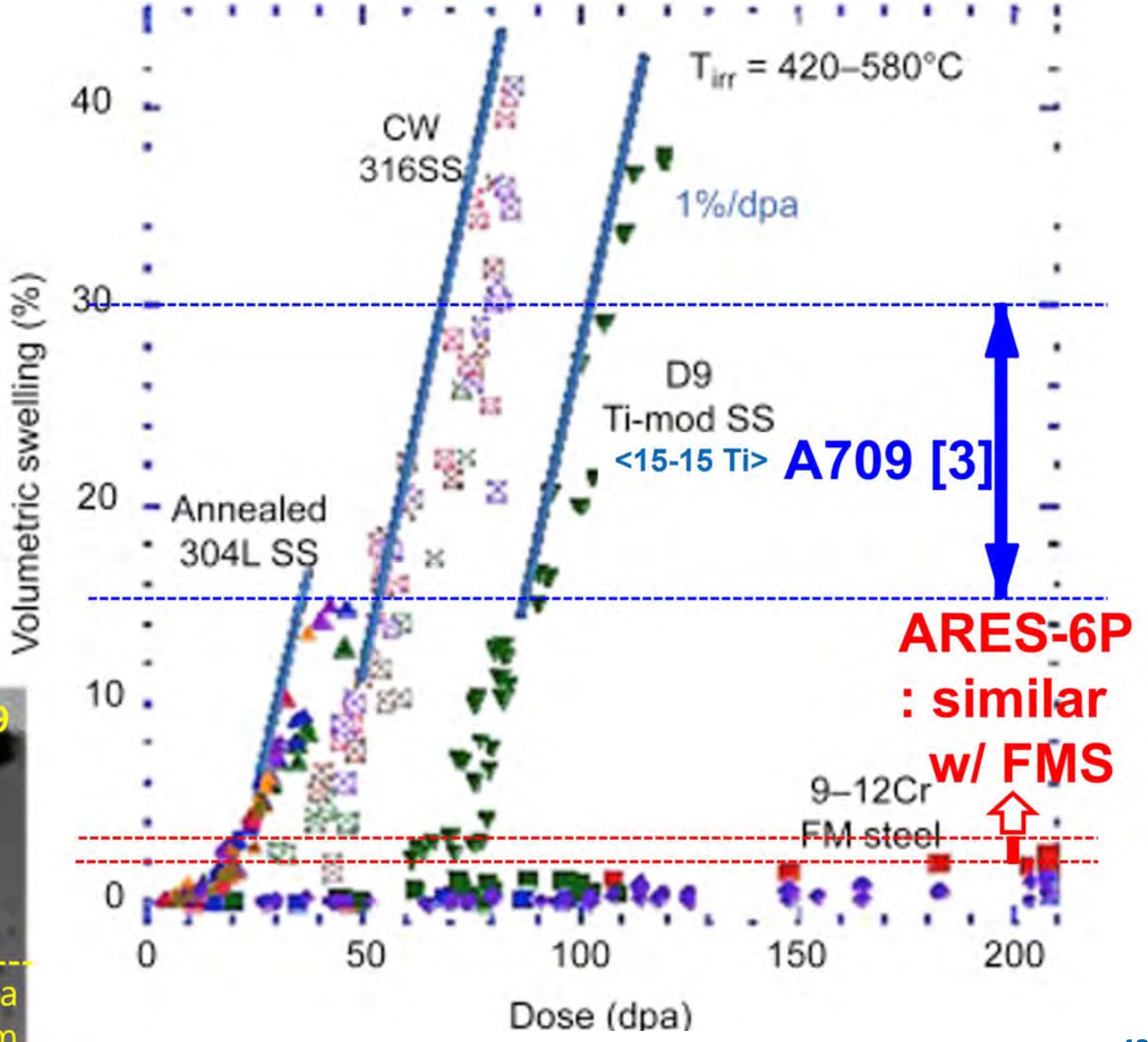
[1] E. Getto et al., JNM 480 (2016) 159-176
[2] J.L. Seran et al., Structure naterials for Gen. IV nuclear reactors (2017) 285-328
[3] H. Kim et al., JNM 527 (2019) 151818

Swelling Comparison





▶ HAADF images of HT9 10 appm He and irradiated with 5 MeV Fe^{2+} at 460 °C [1]



▲ Swelling measured on samples taken from FFTF fuel pin or duct [2]

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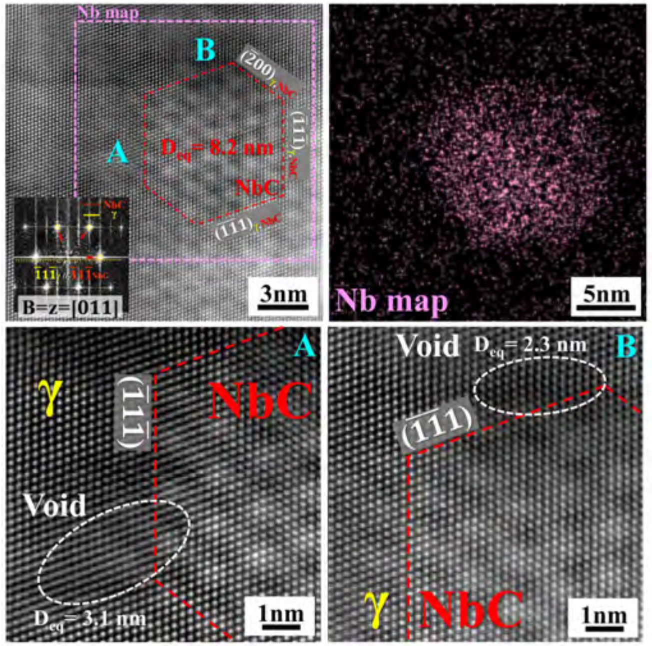
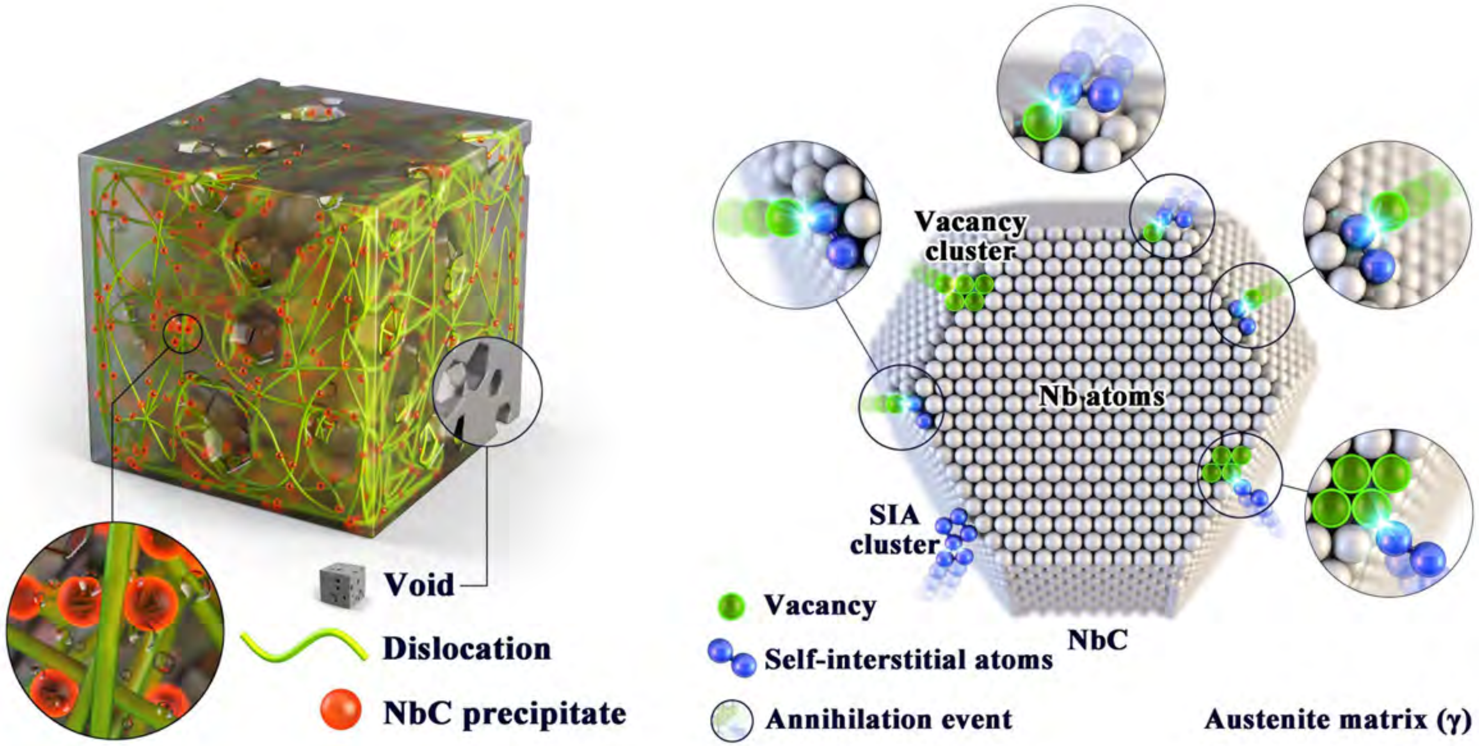
Nature of the Nanosized NbC Precipitates

- The nanosized precipitates: neutral defect sinks for **trapping** and **annihilating** radiation-induced defects
  - Suppression of void formation
  - or, small size of voids: far below the size needed for them to convert to unstably growing
- The **primary mechanism** of inhibition of void swelling
  - Dynamic evolution of radiation-induced defects along the **precipitate-matrix interfaces** at elevated temperature





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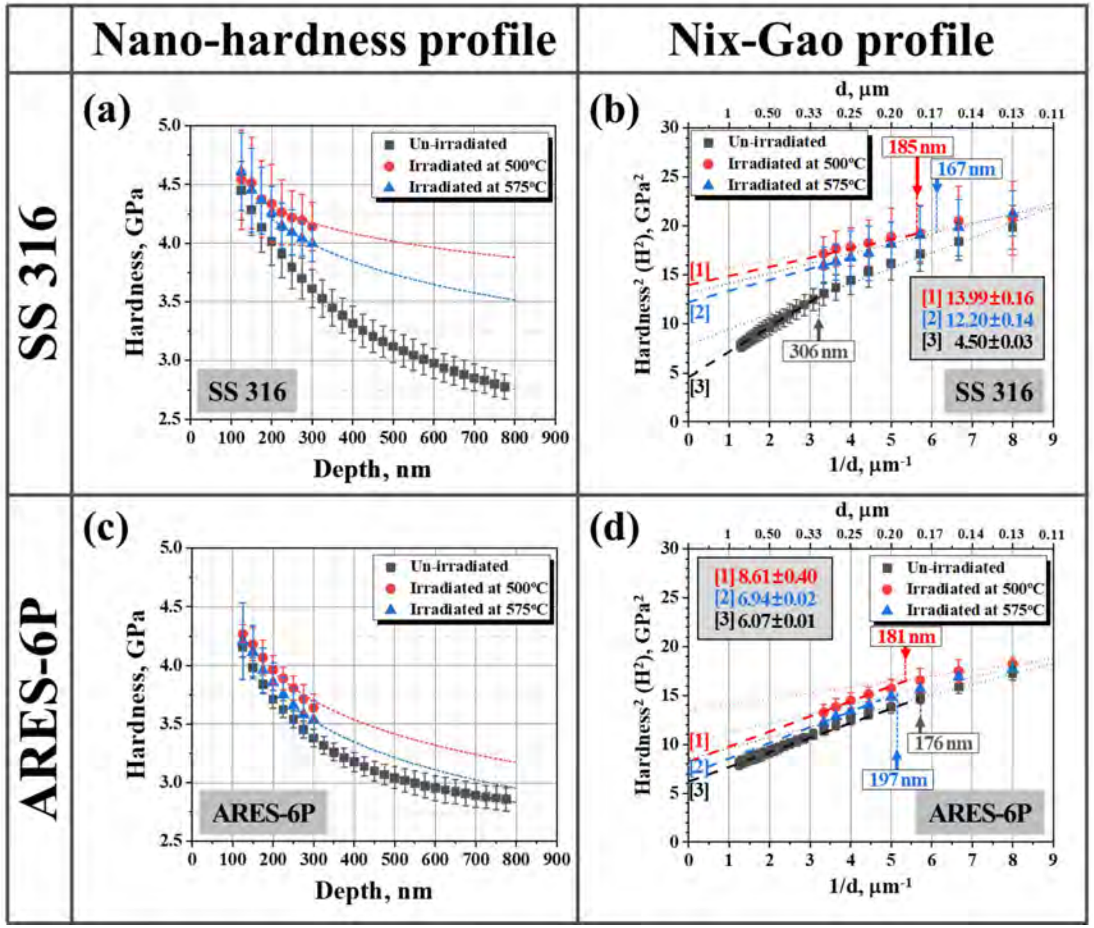
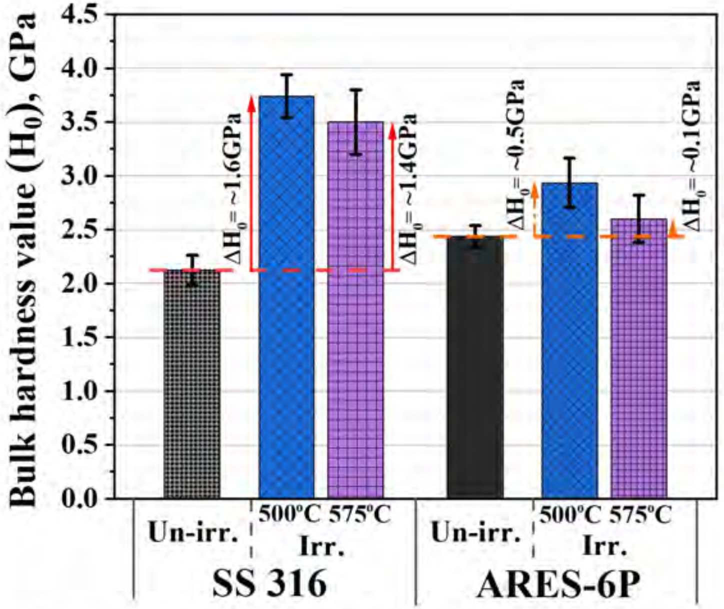
Evaluation of Radiation Hardening

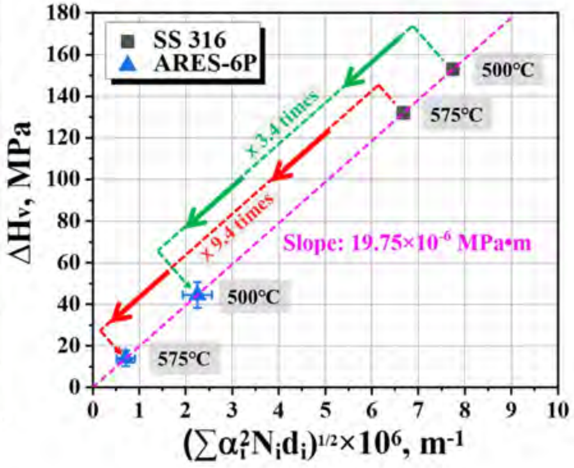
- Indentation (Berkovich tip) depth
  - 300nm for irra. / 800nm for un-irra.

Nix-Gao plot: H^2 vs. 1/d



- ARES-6P: enhanced irradiation hardening resistance
  - Absolute bulk hardness ($H_{0,ARES-6}$)
  - Bulk hardness change (ΔH_0)





Sample			Nano-hardness (Nano-indentation)				Vickers hardness (ASTM E92)
			H_0^2, GPa²	H_0, GPa	H_v, MPa	ΔH_v, MPa	H_v, MPa
SS 316	Un-irra.		4.5	2.1	199	-	188
	Irra.	500°C	14.0	3.7	352	153	-
		575°C	12.2	3.5	331	132	-
ARES-6P	Un-irra.		6.1	2.5	232	-	219
	Irra.	500°C	8.6	2.9	277	45	-
		575°C	6.9	2.6	246	14	-

Summary

❑ Evaluation of the radiation resistance of ARES

- The ARES (newly developed) and 316 SS (reference) were irradiated with heavy ion to emulate neutron irradiation
- ARES shows superior void swelling resistance than 316 SS in both irradiation tests (MIT, Texas A&M)

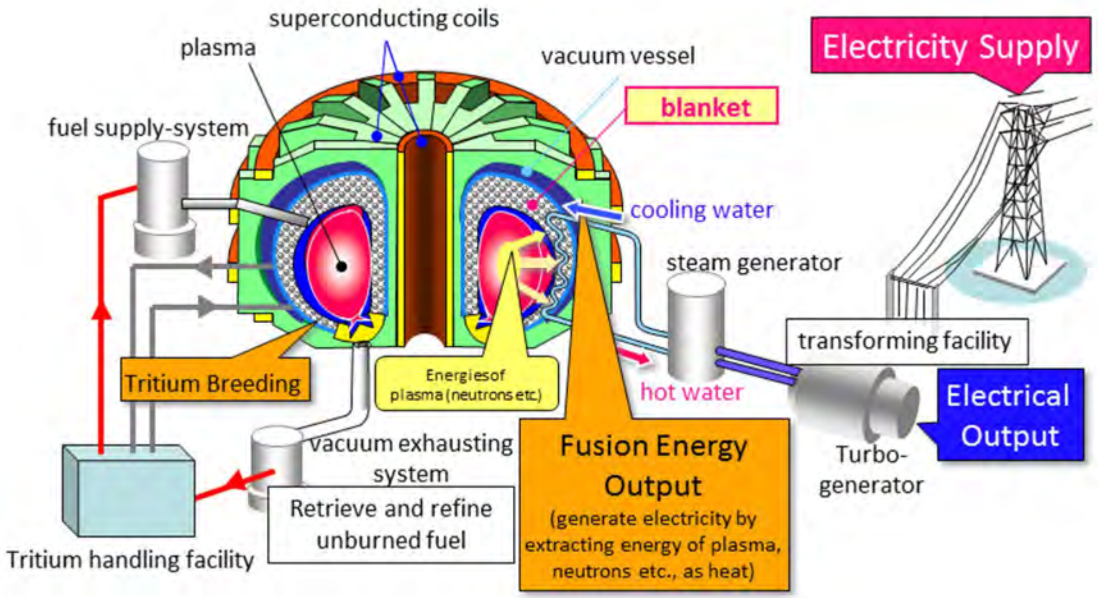
	Dose (dpa)	Dose rate (dpa/s)	NbC stability	Remarks
Low dose (MIT)	8.5	1.8×10^{-3}	X (∵ dose rate effect)	Demonstrate void swelling resistant factor : NbC precipitate (+Ni contents)
High dose (Texas A&M)	200	5.0×10^{-4}	Maintained	Superior void swelling resistance (similar with FMS)

- Outstanding radiation hardening resistance in ARES alloy
  - Nano-indentation tests were conduct to evaluate radiation hardening (ARES vs. 316 SS)
  - Small amount of hardening (ΔH_0) after the irradiation: relatively value
  - Lower hardness value: absolute value

Heidinger, Accelerator related Fusion prospect in EU, APAE kick-off meeting, 2015/16, London

Application & Further Works

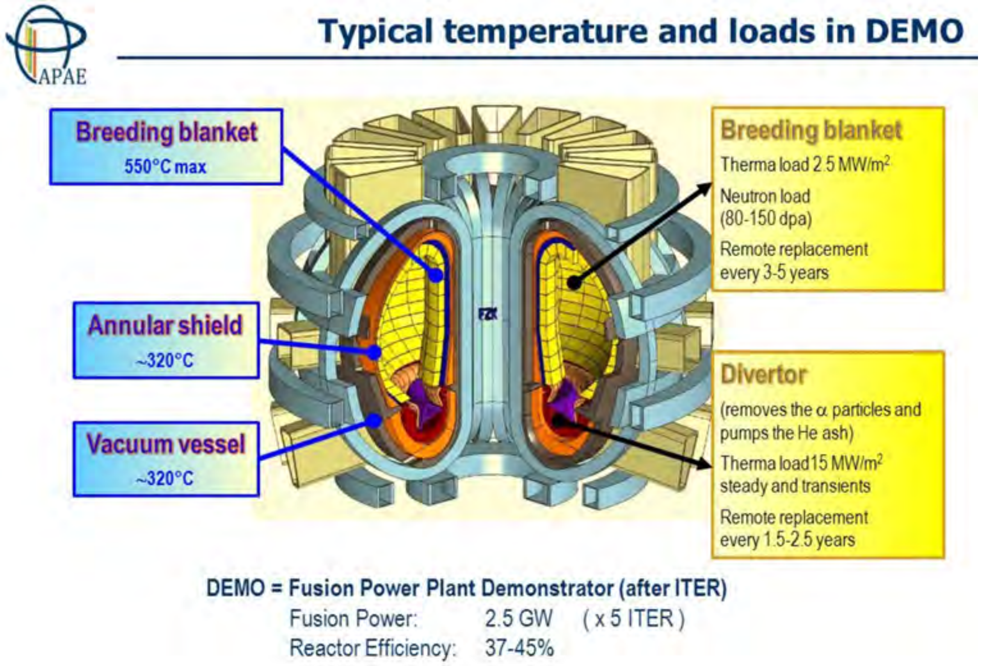
❑ Fusion reactor



❑ Operating condition

- Temperature: ~300 – ~700 ℃
- Damage: ~150 – ~200 dpa

❑ Operating condition of DEMO



Need to maintain good performance under the high energy neutron flux and heat flux

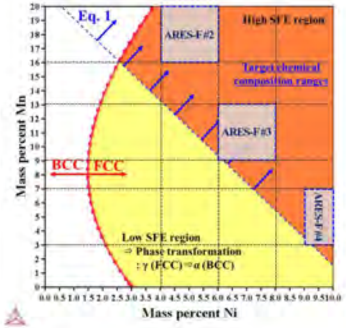
GEN IV International Forum
Expertise | Collaboration | Excellence

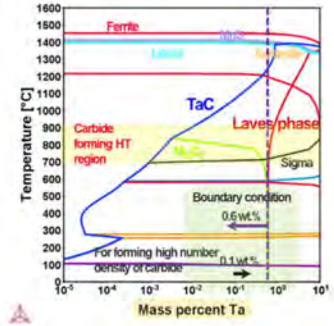
Development of ARES-F (for fusion) Alloys

❑ Requirement of fusion reactor blanket material

1) Low activation
2) Radiation resistance up to 200 dpa
3) High temperature properties, long-term thermal stability
4) Productivity for mass production

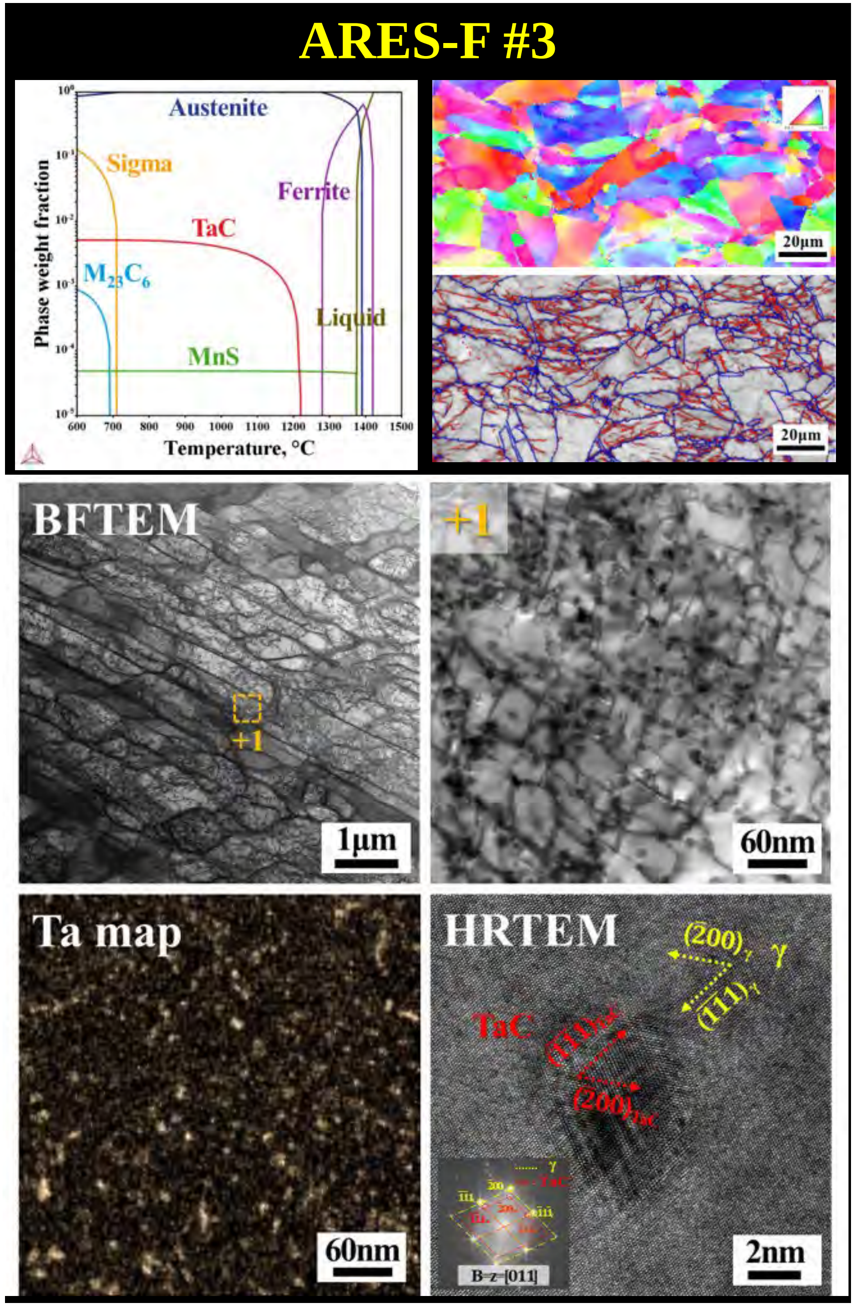
❑ Development of ARES-F alloys





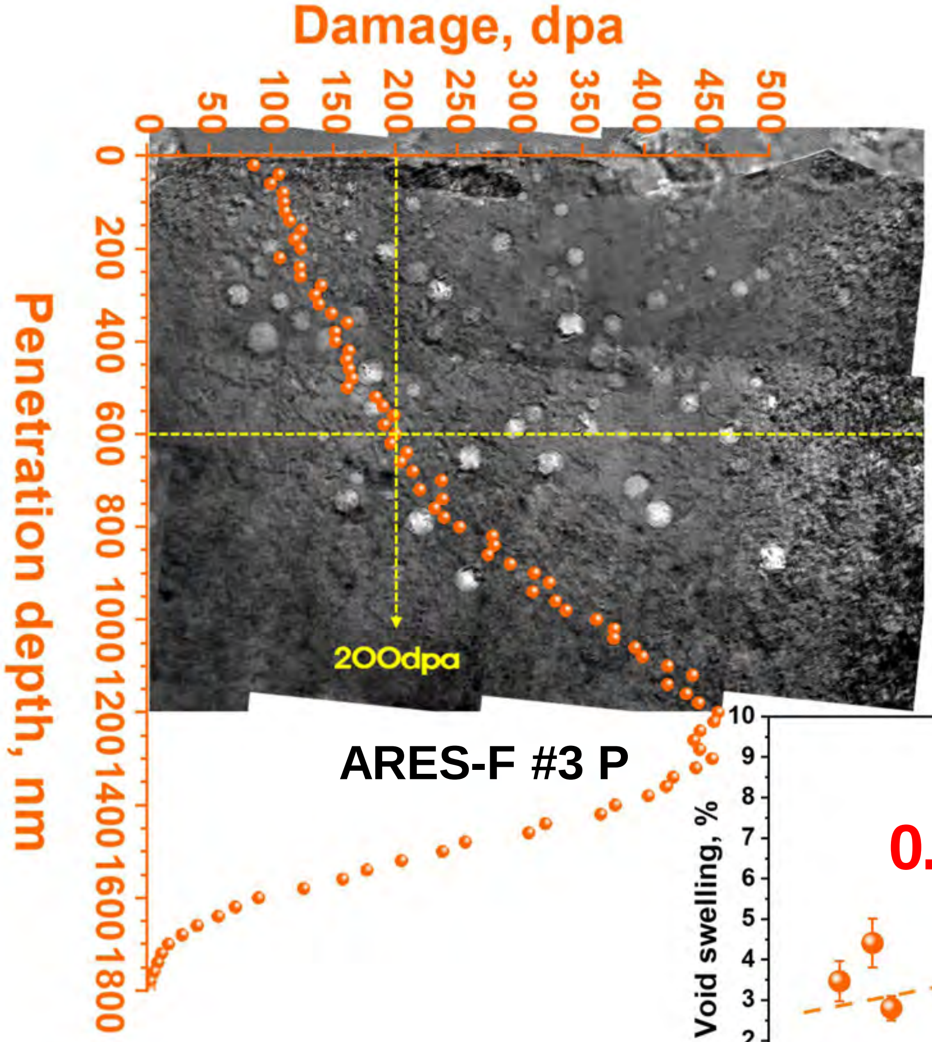
Cr weight percent: 15 wt.% (Fix)

	Ni wt.%	Mn wt.%	Ta & C	Fraction of TaC	
ARES-F #1	5 wt.%	8.3 wt.%	Ta: 0.4 wt.% C: 0.03 wt.%	0.45 wt.%	Ref. alloy
ARES-F #2	3~5 wt.%	16~20 wt.%	Ta: 0.1~0.2 wt.% C: 0.01~0.02 wt.%	〈 0.2 wt.%	Activation resistance
ARES-F #3	6~8 wt.%	9~13 wt.%	Ta: 0.1~0.5 wt.% C: 0.01~0.05 wt.%	〈 0.5 wt.%	⇕
ARES-F #4	9~10 wt.%	3~7 wt.%	Ta: 0.1~1.0 wt.% C: 0.01~0.1 wt.%	〈 1.0 wt.%	Fraction of precipitation, or density

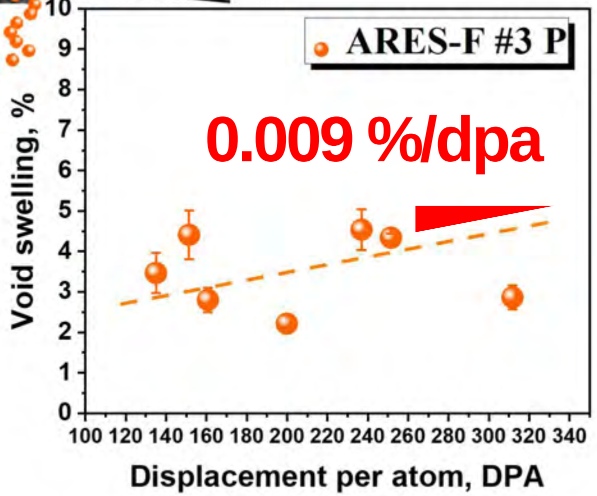


[1] J.L. Seran et al., Structure naterials for Gen. IV nuclear reactors (2017) 285-328

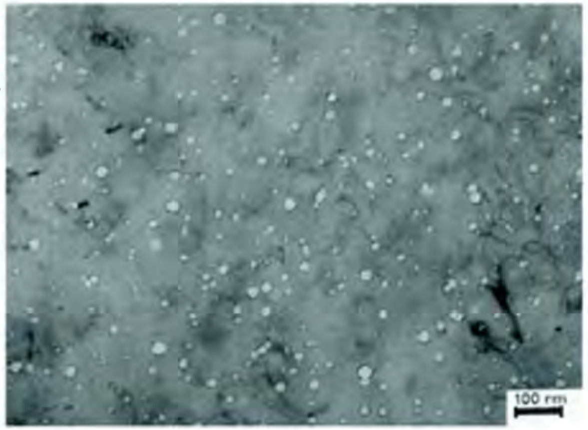
Radiation Resistance of ARES-F



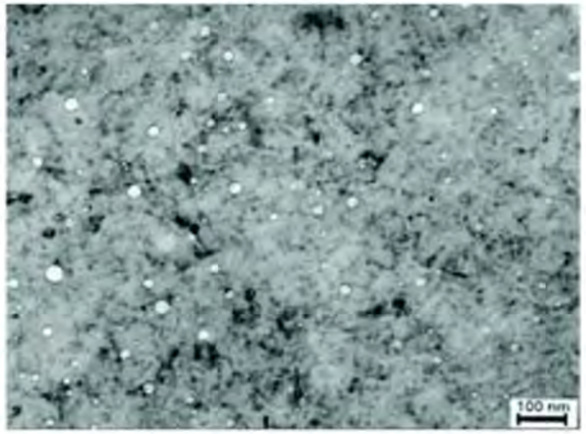
Same scale



6~7% swelling @BN-10 & BN-350 (IAEA No. NF-T-4.2)

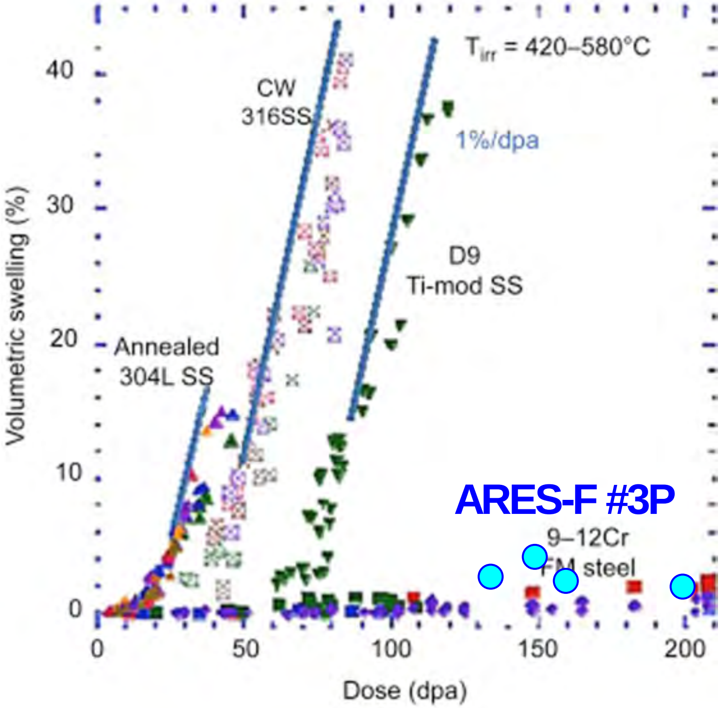


89 dpa, 352°C



46 dpa, 420°C

▶ Swelling measured on samples taken from FFTF fuel pin or duct [1]



Thanks for your attention!!

Upcoming Webinars

Date	Title	Presenter
15 June 2022	Nuclear Waste Management Strategy for Molten Salt Reactor Systems	Dr. John Vienna and Dr. Brian Riley, PNNL, USA
27 July 2022	A Gas Cherenkov Muon Spectrometer for Nuclear Security Applications	Mr. Junghyun Bae, Purdue University, USA
31 August 2022	China’s Multi-Purpose SMR-ACP100 Design and Project Progress	Dr. Song Danrong, Nuclear Power Institute of China