Development of reduced activation austenitic stainless steel containing high density of nanosized precipitates for fusion energy application

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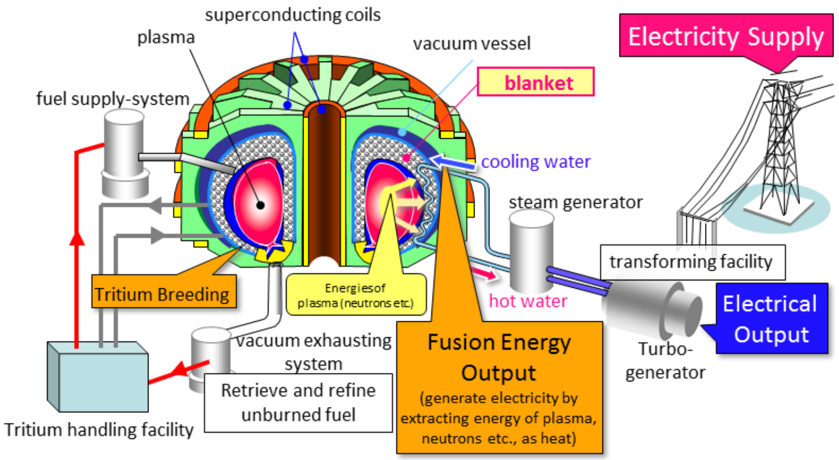
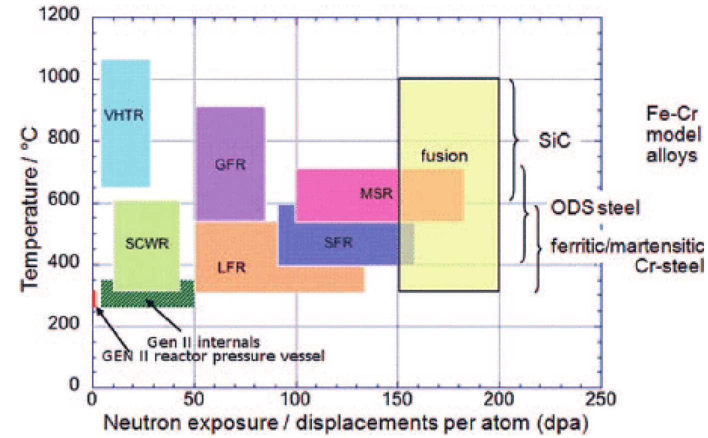
Introduction

❑ Schematic of Fusion Reactor

- High temperature & high neutron irradiation environment





▲ Operating condition for various nuclear reactors [1]

❑ Operating condition for blanket

- Temperature: ~300 – ~700 ℃
- Neutron irradiation: ~150 – ~200 dpa

❑ Requirement for blanket structural materials

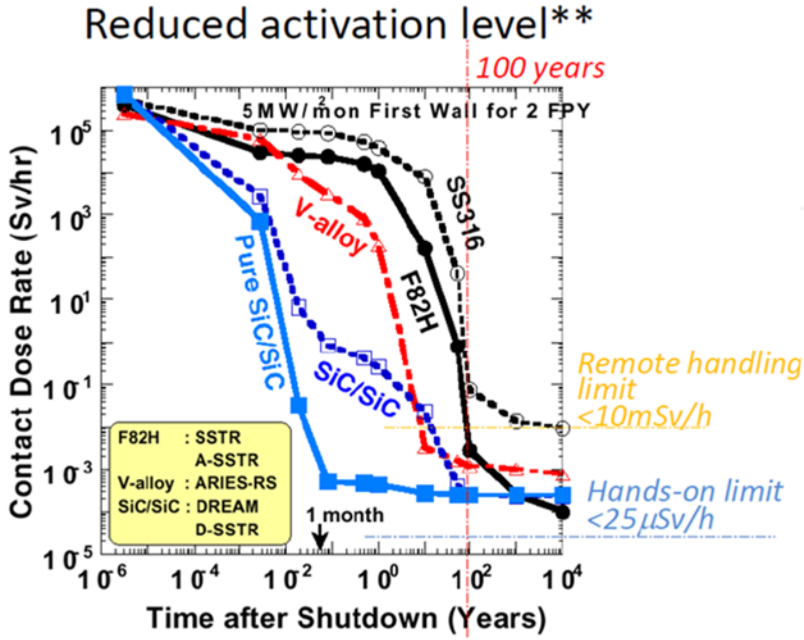
- **Low activation material**
- Good high-temperature mechanical property
- Good irradiation resistance (e.g. radiation embrittlement & void swelling resistance)
- Compatibility with chemicals (coolant, breeding material)

Alloy development motivation

[1] J. Seki et al., J. Jucl. Mater. 1791 (1998) 258
[2] V.D. Rusov et al., Sci. and Tech. Nucl. Inst. 2015 (2015) 1

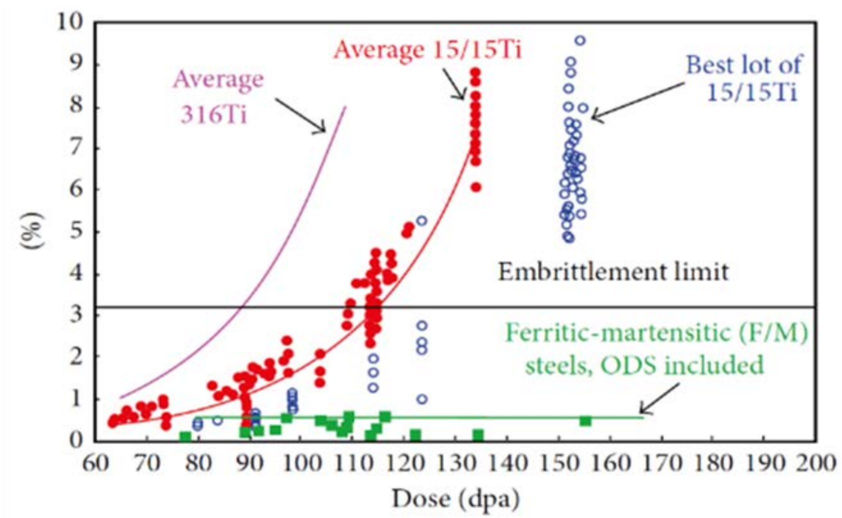
❑ Candidate material for blanket in nuclear fusion reactor

	Reduced activation	Irradiation resistance: Embrittlement	Irradiation resistance: Swelling	Corrosion resistance	High temperature property	Magnetic property
FMS&FM-ODS	Good	Poor	Good	Poor	Good	Ferromagnetic
Austenitic SS (316)	Poor	Good	Poor	Good	Good	Paramagnetic



▲ Activity after shutdown [1]

▲ Comparison between FMS&FM-ODS and austenitic SS (316)



▲ Void swelling resistance for various alloys [2]

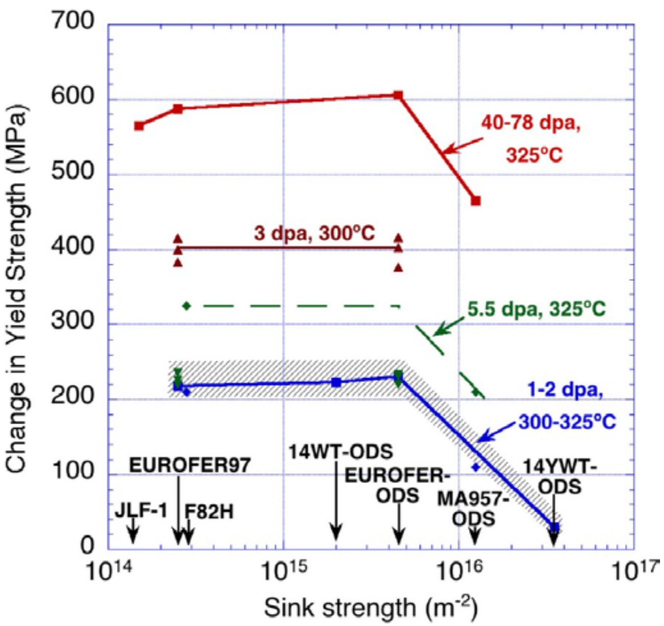
Development of Advanced radiation Resistant austenitic stainless Steel for Fusion reactor in-core materials (ARES-F)

Alloy development motivation

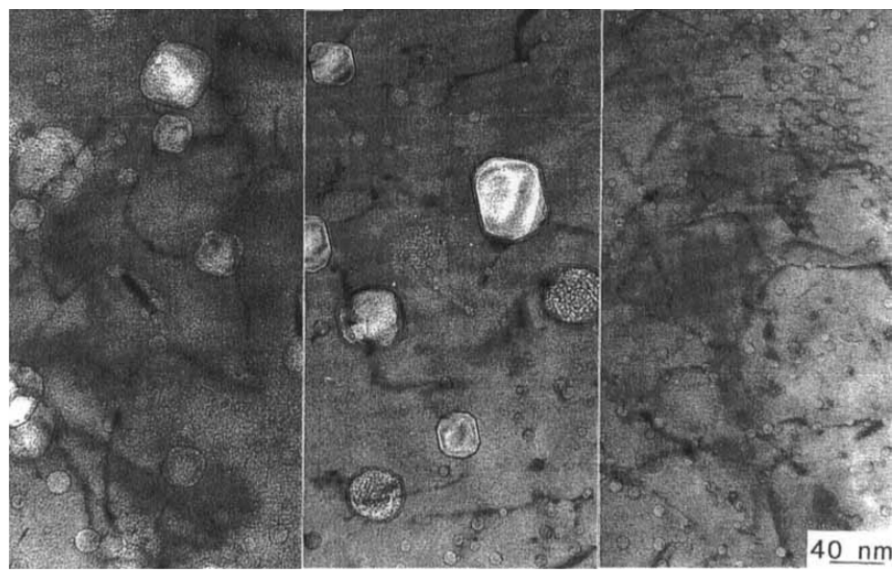
[1] S.J. Zinkle et al., Nuclear Fusion 57 092005 (2017) 17
[2] E.H. Lee et al., Phil. Mag. A 61 (1990) 733

❑ Enhancing irradiation resistance of ARES-F

- Trap He to avoid void swelling
- Recombination of radiation-induced defects
- High sink strength : Ability to absorb the radiation damage
  - Precipitate sink strength → $S_{ppt} = 4\pi r \rho_{ppt} [m^{-2}]$
  - Dislocation sink strength → $S_{dis} \propto \rho_{dis} \ [m^{-2}]$
- Good thermo-mechanical properties at severe environment (Fusion reactor)



▲ Effect of sink strength on radiation damage [1]



Increasing number density of precipitates

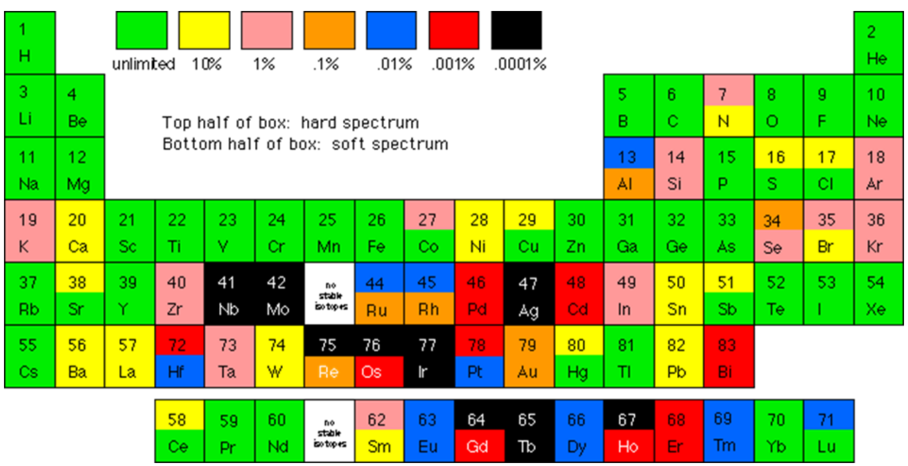
▲ Void swelling resistance by precipitation [2]

Alloy design – Chemical composition

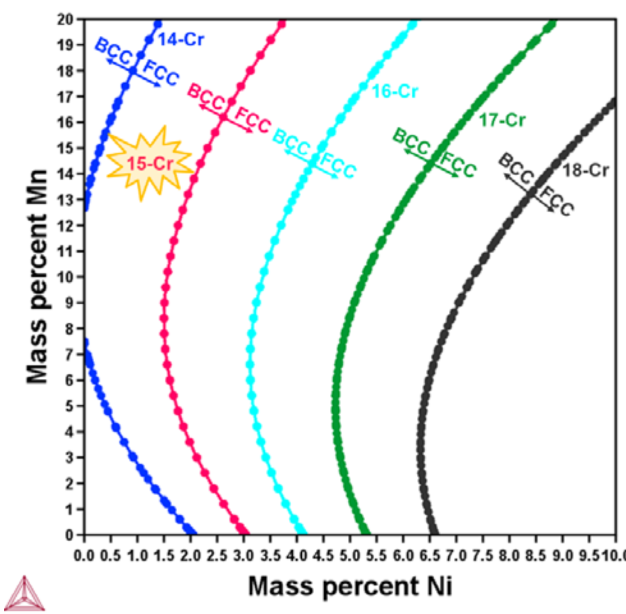
[1] S. J. Zinkle, Fusion Sci. and Tech. 64 (2013) 65

❑ Chemical composition for **ARES-F**

- Limitation on content for reduced activation
  - Replacement of **Ni** with **Mn**
  - Consideration of long-term waste disposal
- Alloy design based on thermodynamic simulation modeling (Thermo-Calc.)
  - Database : TCFE-9 (steels/Fe-alloys v9.0) & MOBFE3 (steels/Fe-alloys mobility v3.0)
  - Fe-Cr-Ni-Mn system
    - ☐ **Cr > 15 wt.%** for enough corrosion resistance



▲ **Reduced activation elements based on the long term waste disposal criteria [1]**



▲ **Ni-Mn balance for stable austenitic matrix based on Thermo-Calc.**

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Alloy design – Chemical composition

❑ Chemical composition for ARES-F

[1] R.E. Schram et al., Metallurgical Transactions A. (1975) 1345
[2] A.F. Padilha et al., ISIJ International. (2002) 325

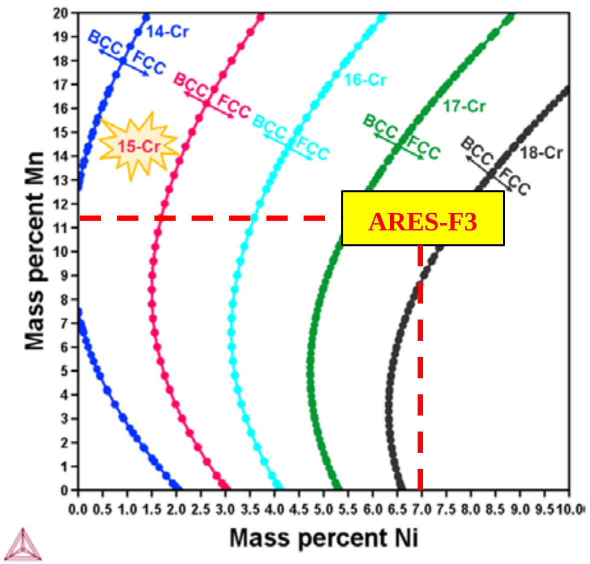
- Stable austenitic matrix with Fe-Cr-Ni-Mn system
  - Stacking fault energy $[mJ \cdot m^{-2}]$ – [1]

    → $\gamma_{SF} = -53 + 6.2(wt.\%Ni) + 0.7(wt.\%Cr) + 3.2(wt.\%Mn)$
  - Martensite formation temperature [℃] – [2]

    → $M_s = 1302 - 42(wt.\%Cr) - 61(wt.\%Ni) - 33(wt.\%Mn) - 28(wt.\%Si) - 1667(wt.\%C + wt.\%N)$
- Optimization of key element chemical composition
  - Reference alloy : 304 SS & 316 SS
  - Stacking fault energy: $> 30\ mJ \cdot m^{-2}$
  - Martensite formation temperature: $< -200℃$

	Fe	Cr	Ni	Mn	Stacking fault energy $[mJ \cdot m^{-2}]$	Martensite formation temperature [℃]
304 SS	Bal.	18.29	8.06	1.05	13.1	-133.4
316 SS	Bal.	17.09	10.28	0.58	44.5	-211.0
ARES-F3	Bal.	15	7	11.3	37.1	-200.2

▲ Chemical composition and corresponding stacking fault energy and martensite formation temperature of 304 SS, 316 SS and ARES-F3



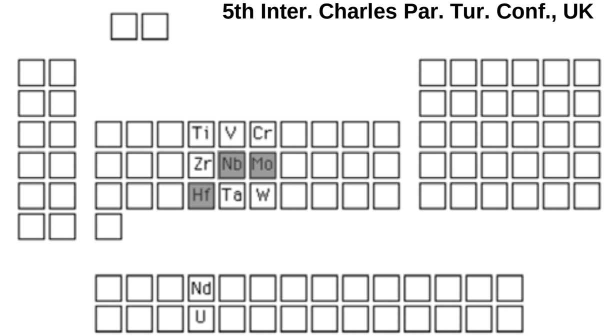
▲ Ni-Mn balance for stable austenitic matrix based on Thermo-Calc.

Alloy design – Chemical composition

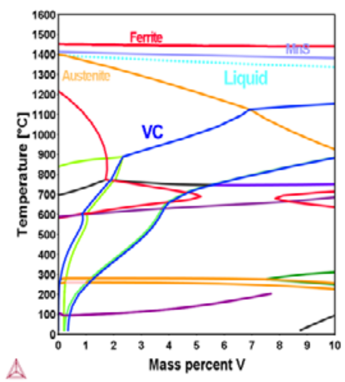
❑ Precipitation for ARES-F3

- Standard for minor alloying element for precipitation
  - Easy to control by heat treatment (**carbide** vs. nitride)
  - Low diffusivity → Large number of fine precipitates
  - Matrix stability → Thermo-Calc. simulation
- Vanadium carbide
  - Unstable matrix
- Zirconium carbide
  - Unstable matrix
- Tantalum carbide
  - **Stable matrix**

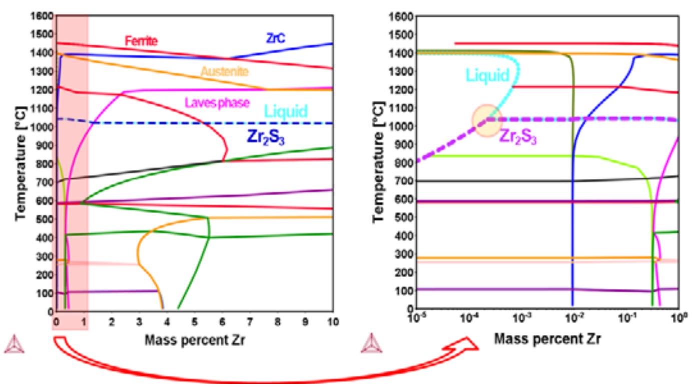
[1] H.K.D.H Bhadeshia, (2000) Proc. of 5th Inter. Charles Par. Tur. Conf., UK



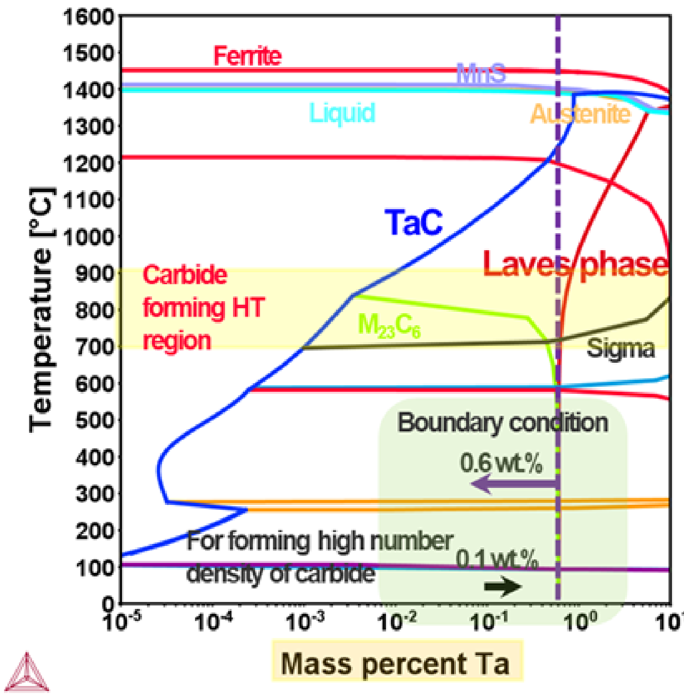
▲ Strong carbide forming element [1]



▲ Phase diagram for VC precipitate



▲ Phase diagram for ZrC precipitate



▲ Phase diagram for TaC precipitate

Alloy design – Chemical composition

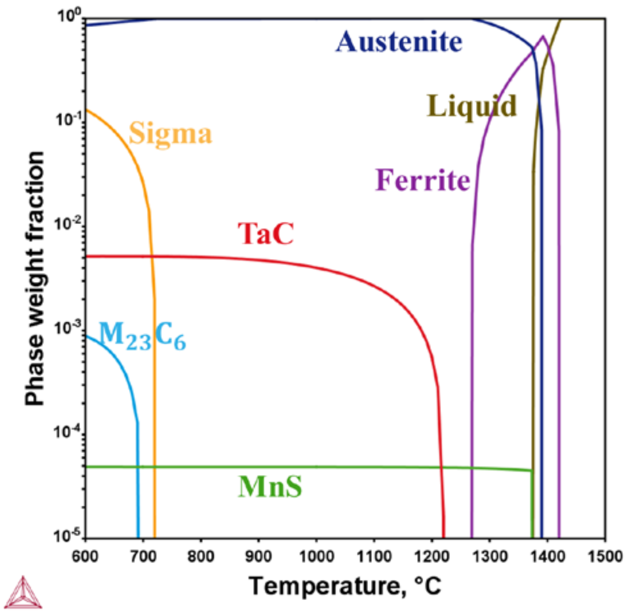
ARES-F3

- Austenitic matrix + TaC precipitates
- Low activation material with high sink strength
- Target chemical composition : Fe – 15Cr – 7Ni – 11.3Mn – 0.45Ta – 0.04C (wt.%)

	Fe	Cr	Ni	Mn	Ta	C	Si	P	S
ARES-F3	Bal.	15.13	7.17	11.16	0.48	0.039	0.22	0.001	0.002

▲ Inductively Coupled Plasma Atomic Emission Spectroscopy (ICP-AES) for ingot of ARES-F3 (wt.%)

- Thermodynamic simulation modeling (Thermo-Calc.)
  - TCFE-9 (steels/Fe-alloys v9.0)
  - MOBFE3 (steels/Fe-alloys mobility v3.0)
  - Minimization of Si content to prevent TaC coarsening
  - P & S content → impurities



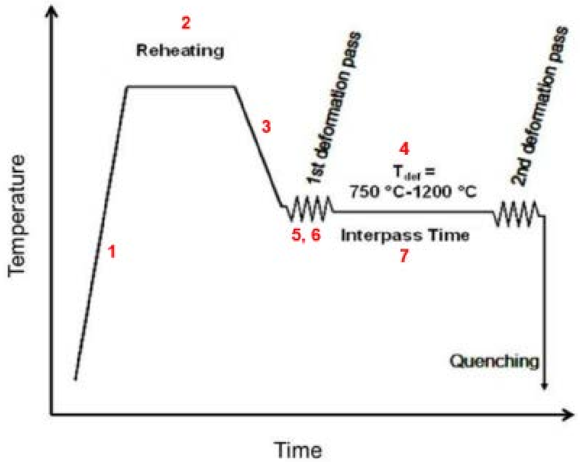
▲ Calculation of phase fraction using Thermo-Calc.

Alloy design – Thermo-mechanical processing

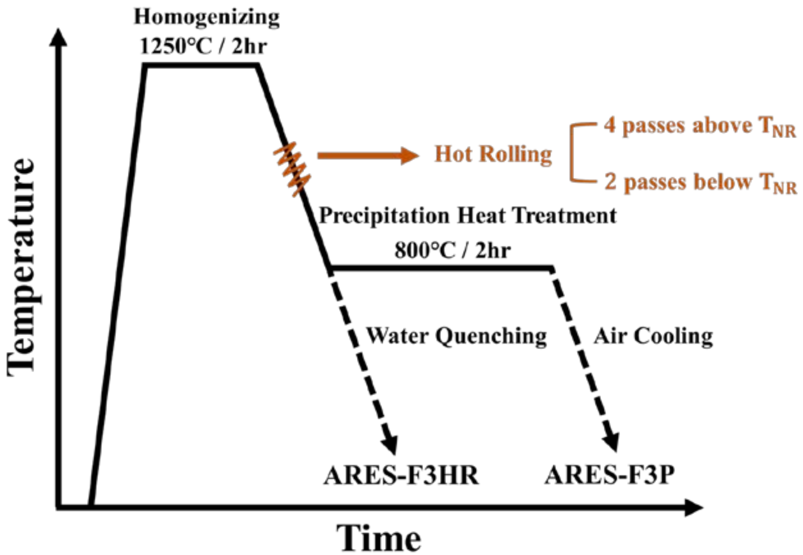
[1] C. N. Homsher, Colorado School of Mines (2012)

❑ Thermo-mechanical processing (TMP) for TaC precipitation

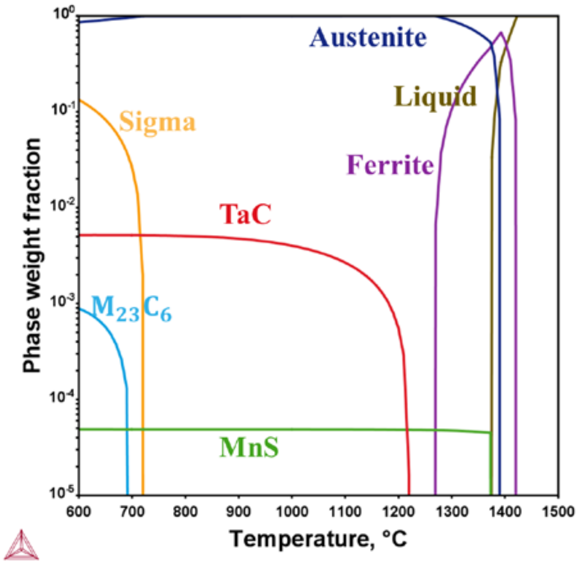
- Formation of pre-existing dislocations by hot-rolling
  - Utilizing the non-recrystallization temperature (T_{NR})
  - Double-hit deformation test
  - Nucleation site for TaC precipitates
- Precipitation heat treatment
  - Ferrite forming temperature (~1270℃)
  - TaC forming temperature (~1220℃)



▲ Schematic of double-hit deformation test [1]



▲ Schematic of TMP for TaC precipitation

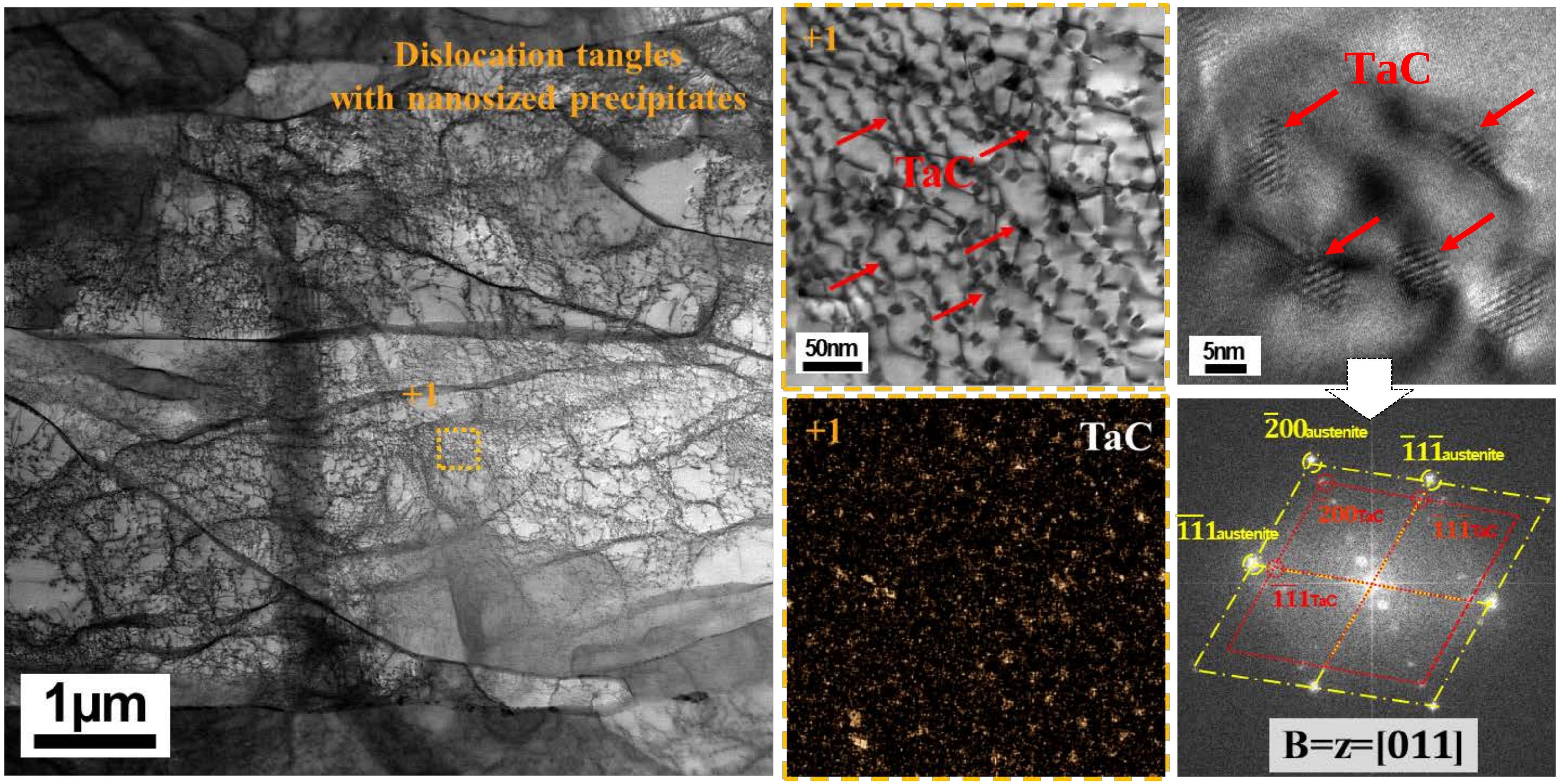


▲ Calculation of phase fraction using Thermo-Calc.

Alloy design – Thermo-mechanical processing

❑ Microstructure of ARES-F3P

- High number density of TaC precipitates near pre-existing dislocation
- $\bar{\rho}_{\mathrm{ppt}} = \sim 1.7 \times 10^{23}\ m^{-3}$
- $\bar{D} = \sim 5.7\ nm$



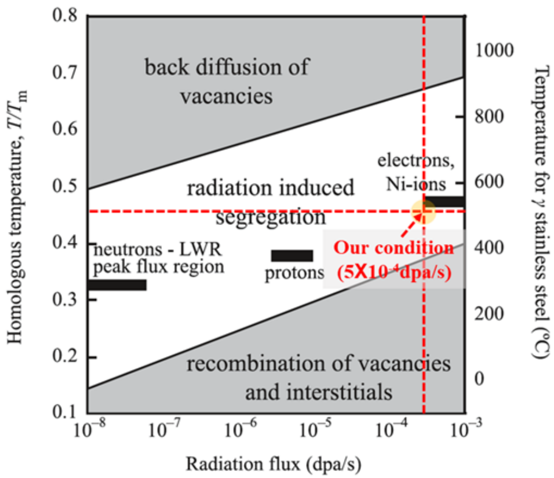
▲ TEM and EDS mapping images of ARES-F3P

Irradiation test condition

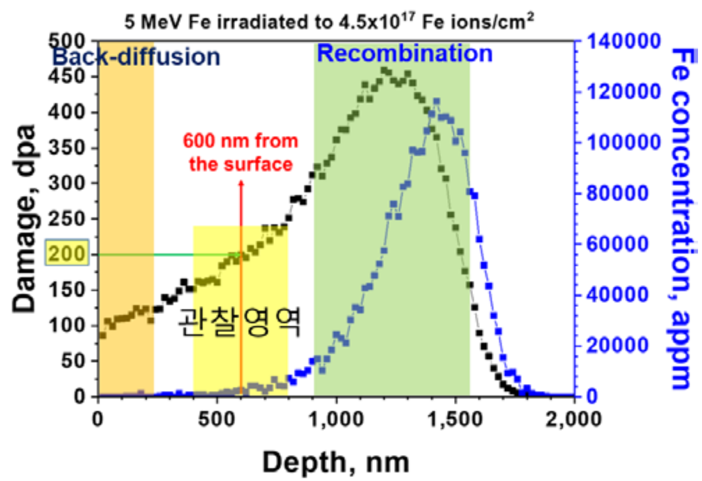
[1] G. Was, Fundamentals of Radiation Materials Science

❑ Heavy ion irradiation for simulation of neutron irradiation

- Materials
  - ARES-F3P
  - Commercial 316 SS (Reference alloy)
- Irradiation condition
  - Stopping and Range of Ions in Matter (SRIM) simulation
  - 40eV displacement energy in Kinchin-Pease model (K-P model)
  - Targeted damage : **200 dpa** / Dose rate : **5×10^{-4} dpa/s** at 600nm
  - **500℃** static defocusing beam with **Fe^{2+} ion**



▲ Heavy ion irradiation conditions [1]



▲ Depth profile of radiation damage (dpa) based on SRIM simulation under K-P model

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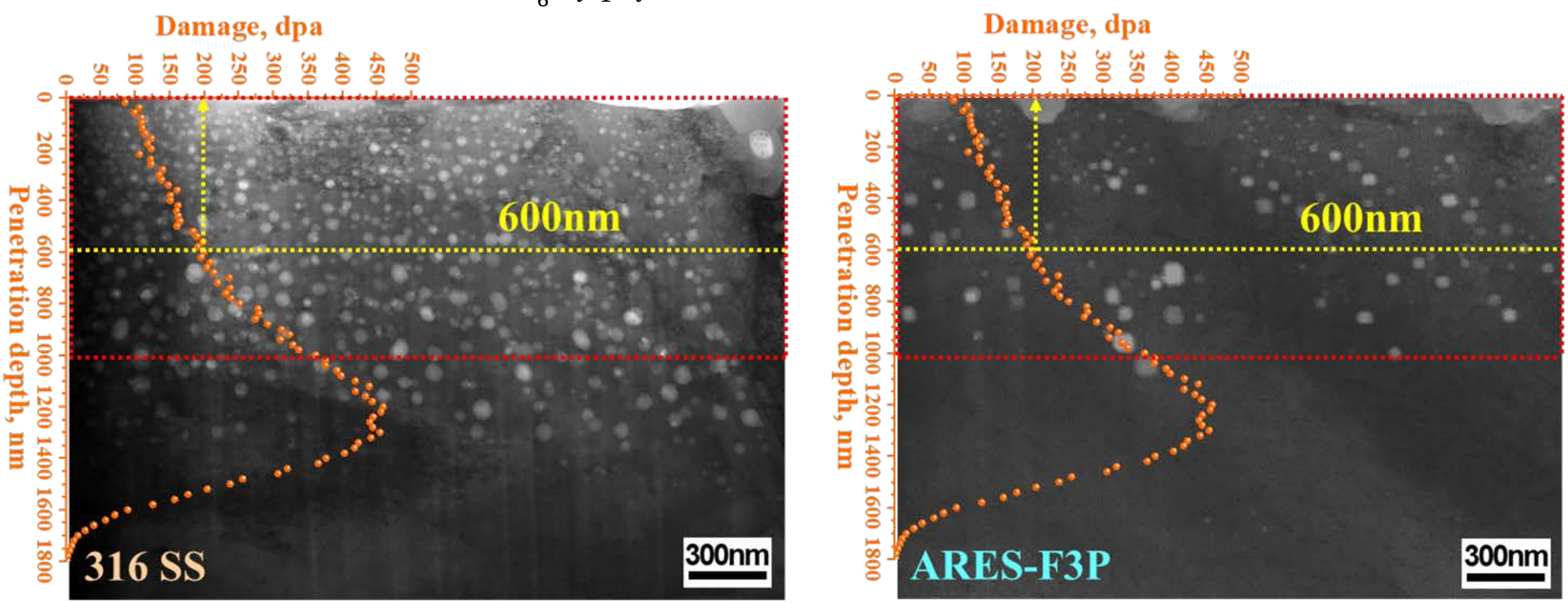
Void swelling resistance

[1] Z. Fan, Materialia 9 (2020) 100603

❑ Void swelling measurement

- High-magnitude BFTEM images → Mainly 400 ~ 800 nm from the surface
- Void size & Void density measurement by Image-J software
- Void swelling (%) $= \dfrac{\frac{\pi}{6}\sum_{i=1}^{N} d_i^3}{A\times t - \frac{\pi}{6}\sum_{i=1}^{N} d_i^3} \times 100$ - [1]

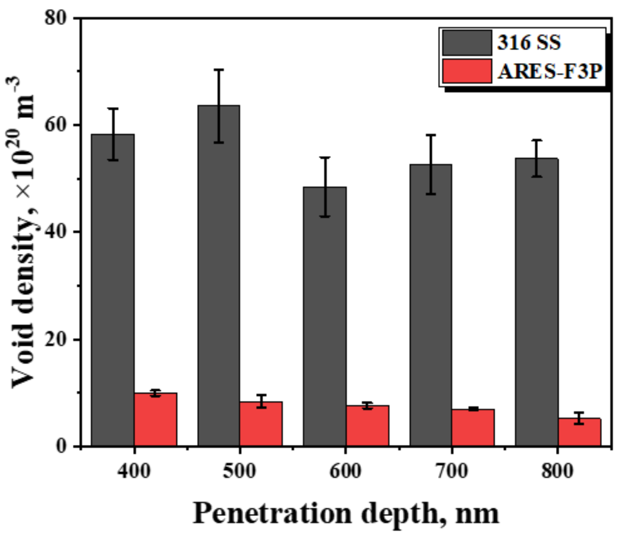


▲ Cross-sectional BFTEM images showing the voids in irradiated 316 SS and ARES-F3P

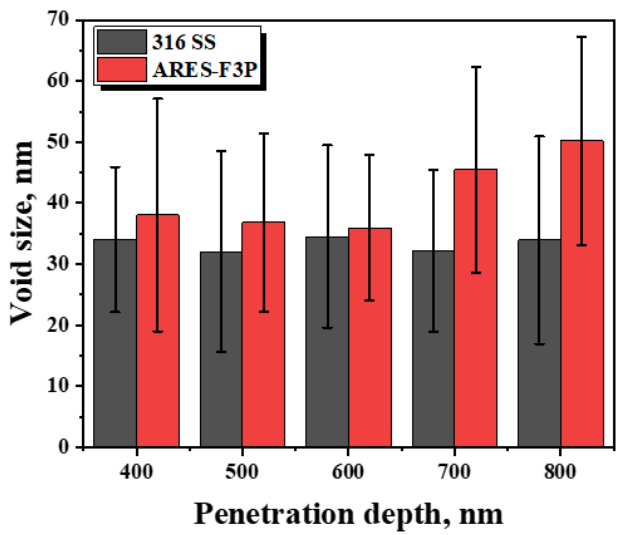
Void swelling resistance

❑ Void swelling measurement

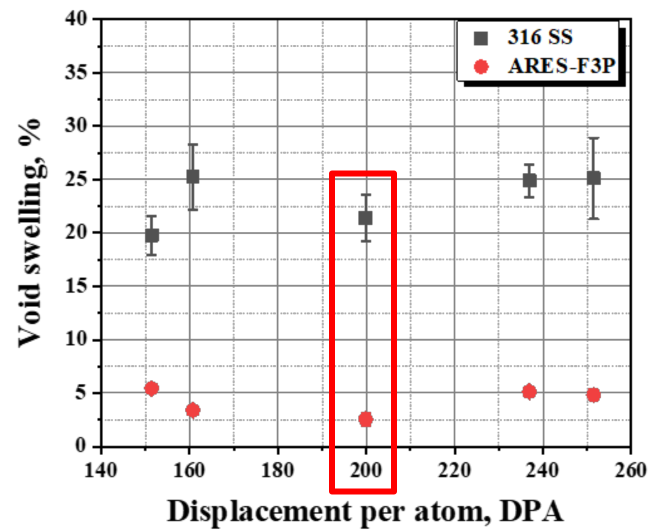
- High number density of voids for 316 SS
- Similar Void size
- **Much larger void swelling for 316 SS than ARES-F3P** at 400 ~ 800 nm from the surface



▲ Average void density of 316 SS and ARES-F3P



▲ Average void size of 316 SS and ARES-F3P



▲ Average void swelling of 316 SS and ARES-F3P

Uniformly distributed fine TaC precipitates in austenitic matrix shows superior void swelling resistance

Summary

❑ Development of ARES-F3

- Fe-Ni-Mn-Cr system for low activation material
- High radiation resistance compared to commercial austenitic SS
  - Fully austenitic matrix
  - Uniformly distributed fine precipitates
- Formation of TaC precipitates near pre-existing dislocations through TMP
  - Hot-rolling based on double-hit deformation test
  - Precipitation heat treatment
  - $\bar{\rho}_{ppt} = \sim 1.7 \times 10^{23}\ m^{-3}$, $\bar{D} = \sim 5.7\ nm$

❑ Void swelling resistance

- Heavy ion irradiation (~200 dpa: high damage level)
  - Radiation damage and dose rate selection based on SRIM under K-P model
  - Void size and number density measurement → Void swelling
  - Comparing ARES-F3P with reference 316 SS → Superior void swelling resistance in ARES-F3P

Energy for Earth !!

Thank you!

Appendix: Irradiation hardening

❑ Irradiation hardening resistance by nano-indentation

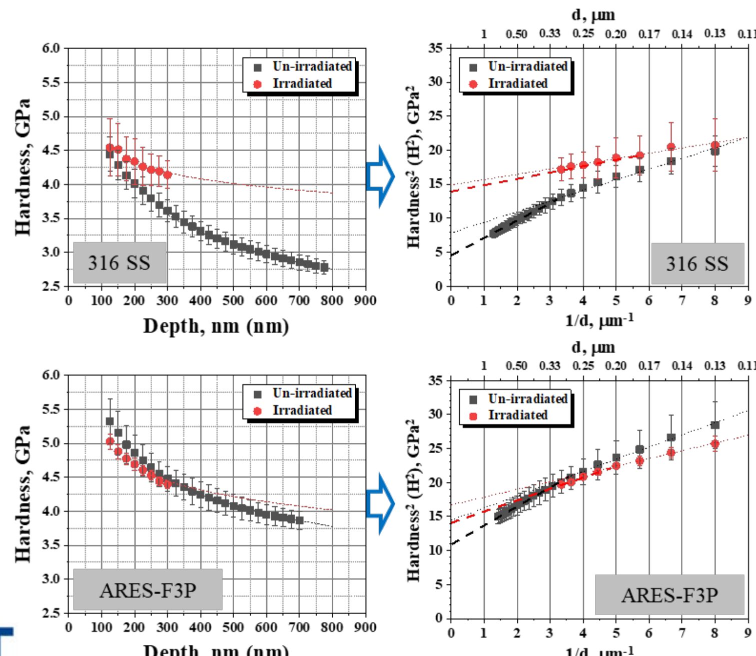
- Materials
  - ARES-F3P
  - Commercial 316 SS (Reference alloy)
- Nano-indentation
  - Small-scale evaluation method due to **shallow penetration depth** of ion irradiation
  - Orowan dispersed barrier hardening (DBH) model
  - **Much larger irradiation hardening for 316 SS than ARES-F3P**

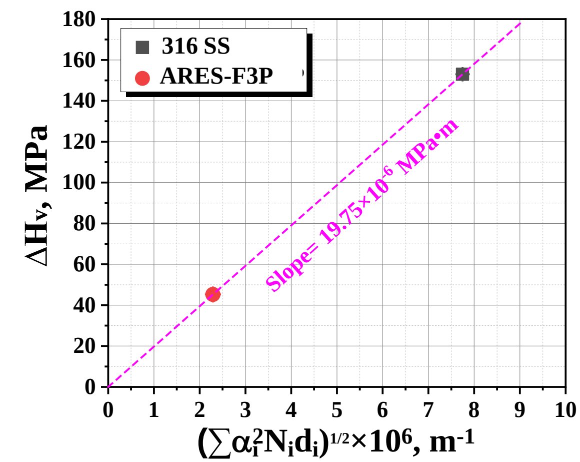
$$\Delta H_v = 0.0945 \Delta H_0 \ (GPa)$$

$$\Delta \sigma_y = 3.03 \Delta H_v \ (MPa)$$

$$\Delta \sigma_{y,i} = M \alpha_i \mu b \sqrt{N_i d_i} \ (MPa)$$

$$\Delta H_v = \frac{M \mu b}{3.03} \left(\sum \alpha_i^2 N_i d_i\right)^{1/2} \ (MPa)$$





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