COLLEGE OF ENGINEERING
NUCLEAR ENGINEERING & RADIOLOGICAL SCIENCES
UNIVERSITY OF MICHIGAN

The irradiation and aging effect in FeCrAl oxide dispersion strengthened steels

Zhexian Zhang
zxzhang@umich.edu
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MICHIGAN ENGINEERING
UNIVERSITY OF MICHIGAN

Zhexian Zhang

Research Fellow,
Nuclear Engineering & Radiological Sciences
University of Michigan, Ann Arbor

Guest Researcher
Materials Science and Engineering
University of Tennessee, Knoxville

Education:
- PhD, Energy Science, Kyoto University, Japan
- MS, Materials Science and Engineering, USTB, China
- BS, Materials Science and Engineering, USTB, China

Research interest:
- Nuclear materials
- Irradiation damage
- Advanced microscopy
- Physical metallurgy

Technology:
- TEM
- EBSD
- Nano-indentation
- ….

Extended interest:
- computational material science
- machine learning

Researched topics:
- Ion-irradiation effects in pure tungsten
- Helium effects in FeCrAl ODS steels
- Ion and neutron irradiation in multiple steels
- Aging embrittlement in FeCrAl ODS steels
- Advanced TEM techniques
- High entropy alloy for fission and fusion applications

Fukushima Daiichi

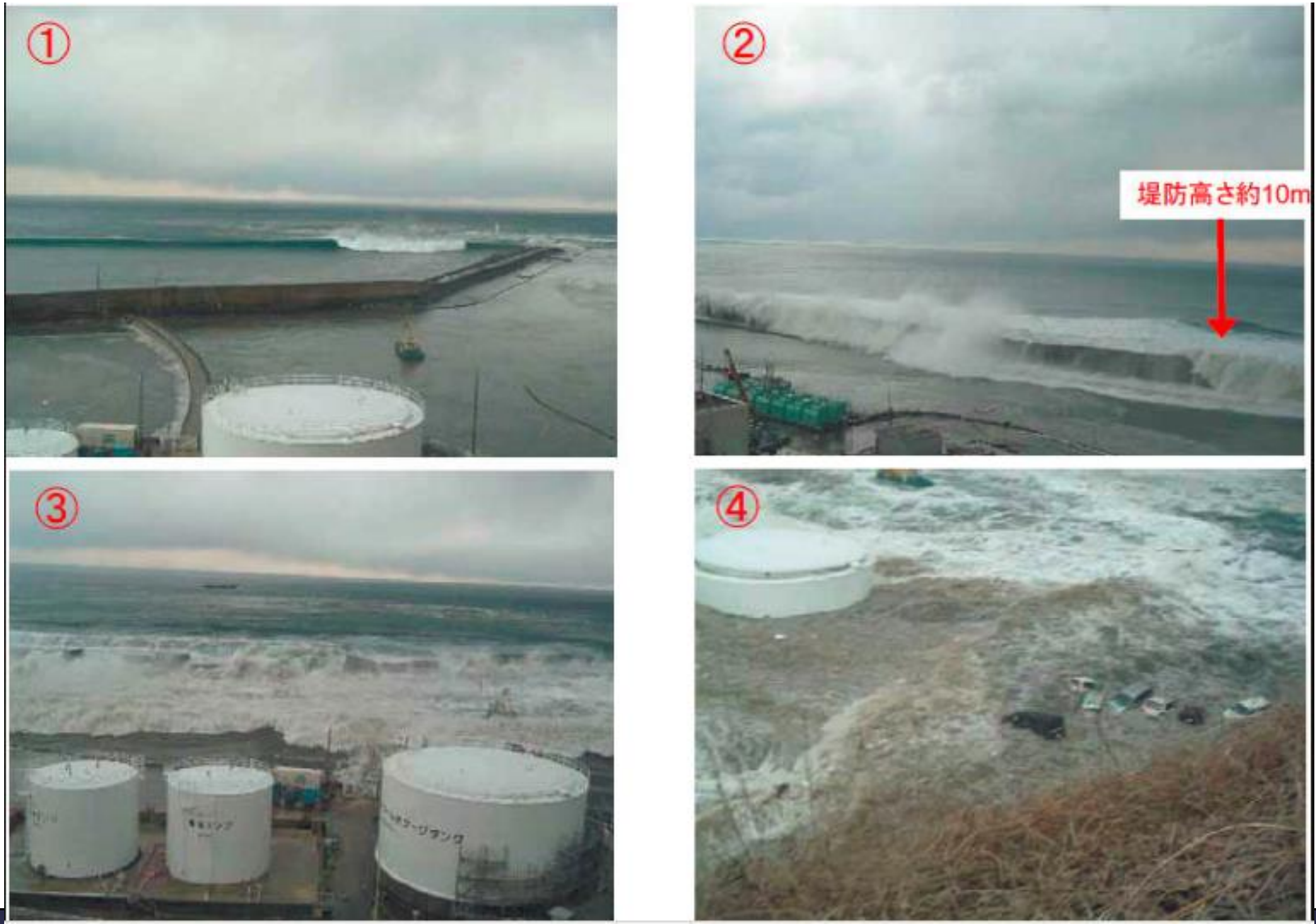
March 11th, 2011

March 12th-15th 2011

April, 2011

January, 2018

September 6th , 2023



Fukushima Daiichi

March 11th, 2011

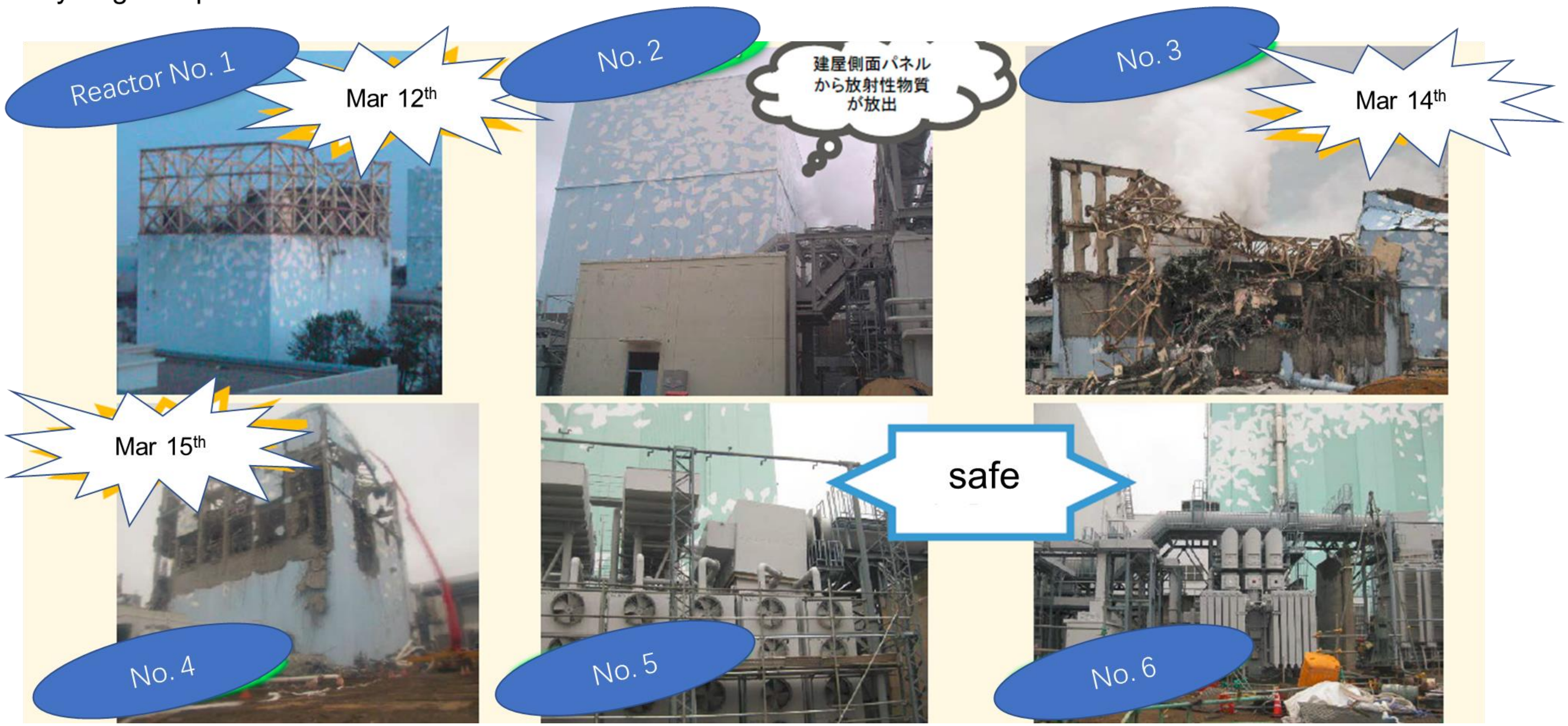
March 12th-15th 2011

April, 2011

January, 2018

September 6th , 2023

Hydrogen explosion



Fukushima Daiichi

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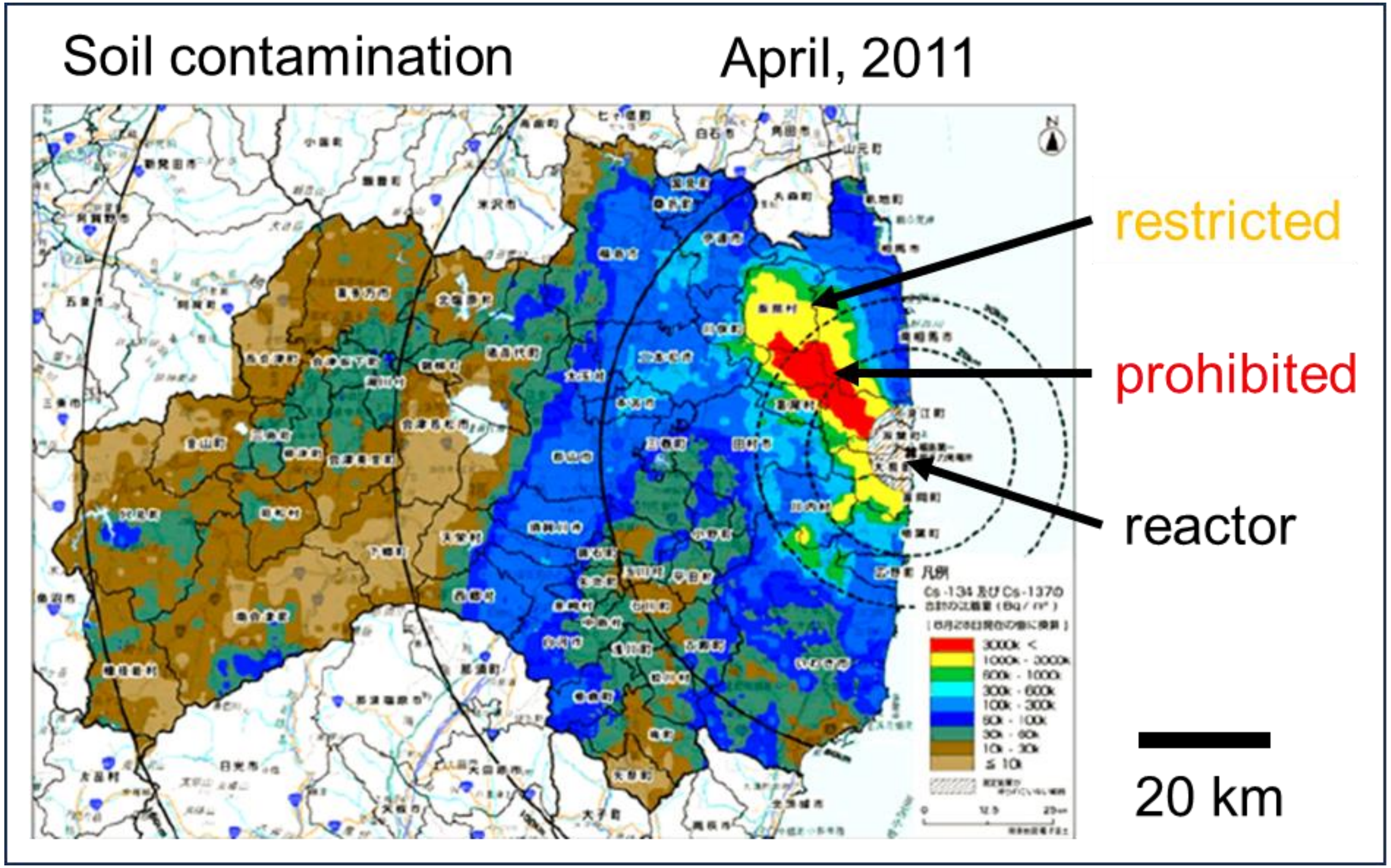

Fukushima Daiichi

March 11th, 2011

March 12th-15th 2011

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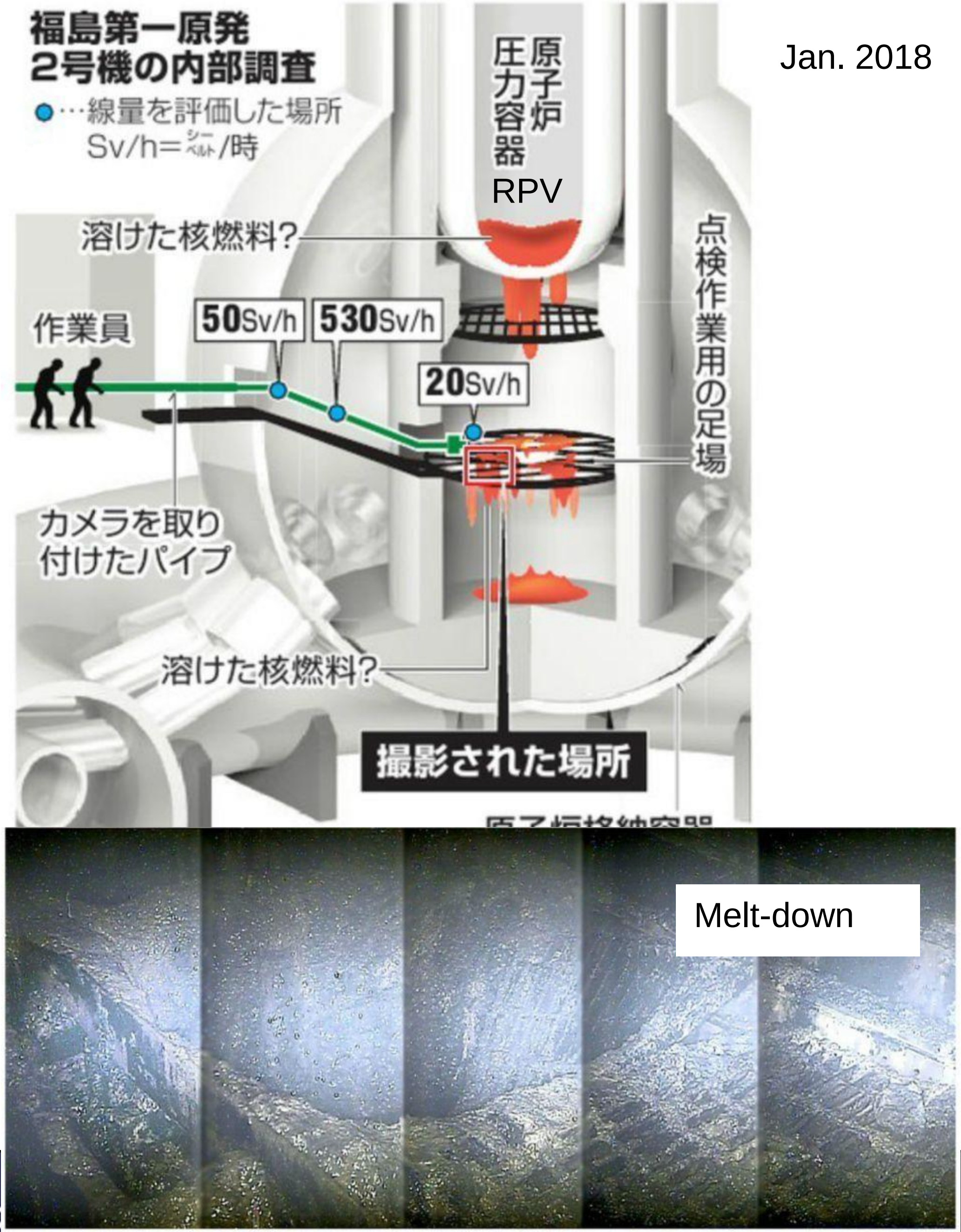
January, 2018

September 6th , 2023

Jan. 2018



福島第一原発２号機の原子炉圧力容器直下の様子。作業用足場には溶けた核燃料とみられる黒い塊がこびりつく。足場の一部は落ち、約１㍍四方の穴が開いていた＝東京電力が動画から切り出した画像をつなぎ合わせて提供

Fukushima Daiichi

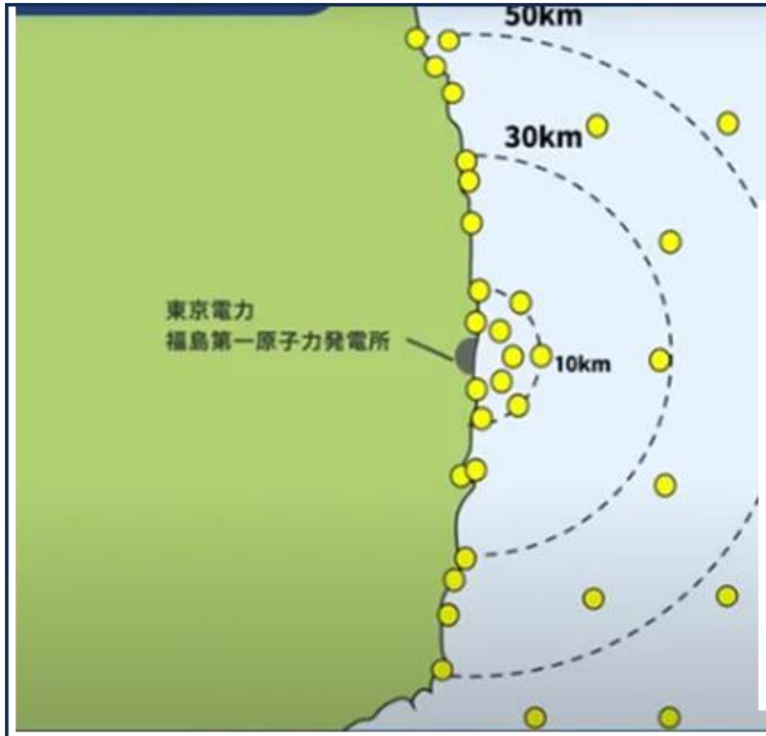
March 11th, 2011

March 12th-15th 2011

April, 2011

January, 2018

September 6th , 2023



Sept. 6th, 2023

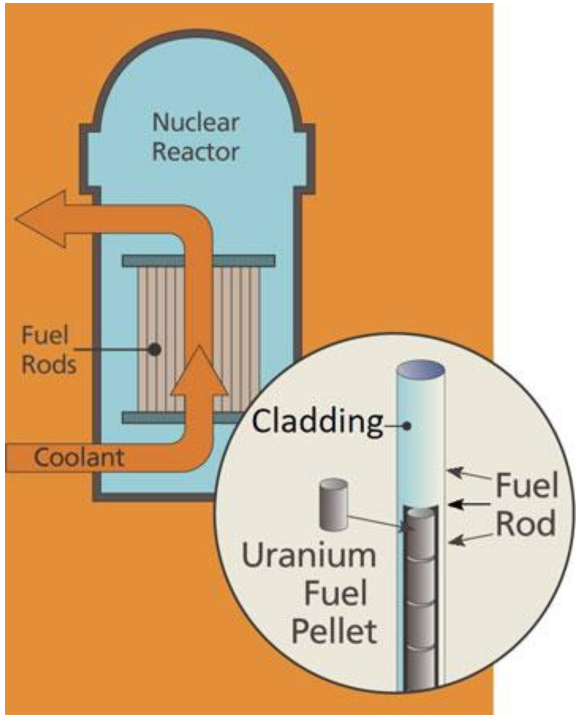
Tritium in Sea

測点	採取日	採取層	Depth(m)	核種	(Bq/L)
E-S1	2023/09/06	表層	1.5	H-3	< 8
E-S3	2023/09/06	表層	1.5	H-3	< 8
E-S5	2023/09/06	表層	1.5	H-3	< 8
E-S10	2023/09/06	表層	1.5	H-3	< 8
E-S10	2023/09/06	底層	12.7	H-3	< 7
E-S13	2023/09/06	表層	1.5	H-3	< 8
E-S15	2023/09/06	表層	1.5	H-3	< 8
E-S16	2023/09/06	表層	1.5	H-3	< 7

Part I: Fission nuclear reactor

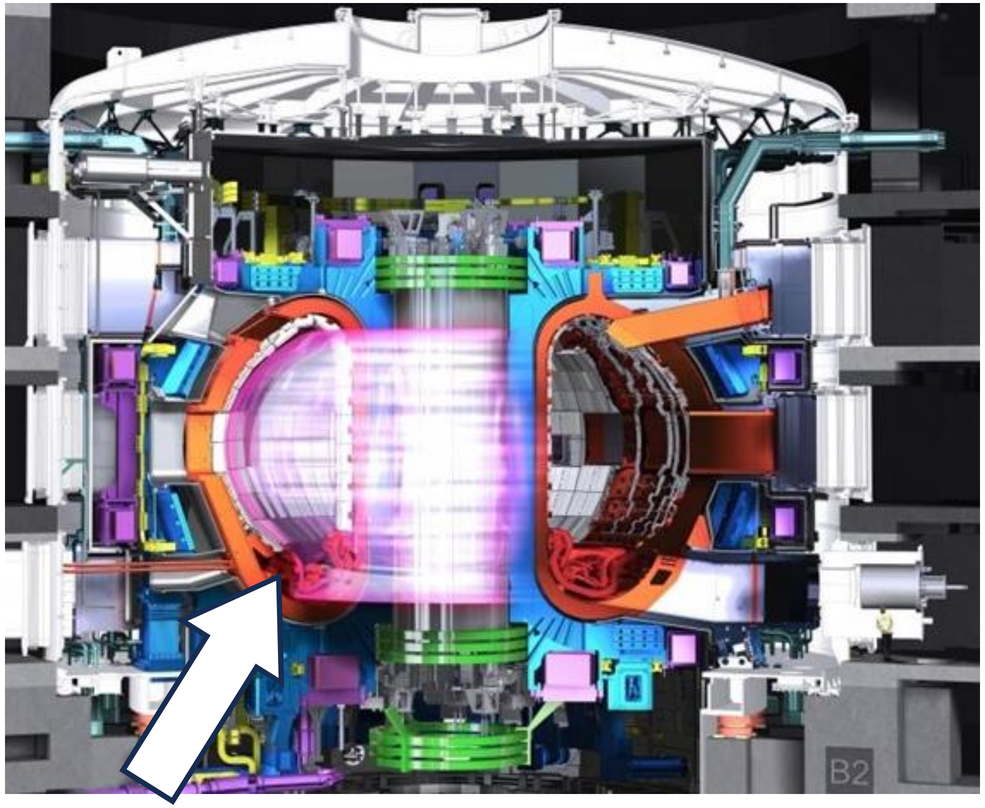
the near-term ATF concepts:

1. Chromium-coated cladding
2. Doped pellets
3. FeCrAl cladding



https://www.nrc.gov/reactors/power/atf/technologies/chrom-clad.html

Part II: Fusion nuclear reactor



divertor *https://www.iter.org/proj/inafewlines*

Element and researches

Accident Tolerance
Replaced with Zr alloys
→ Light Water Reactor

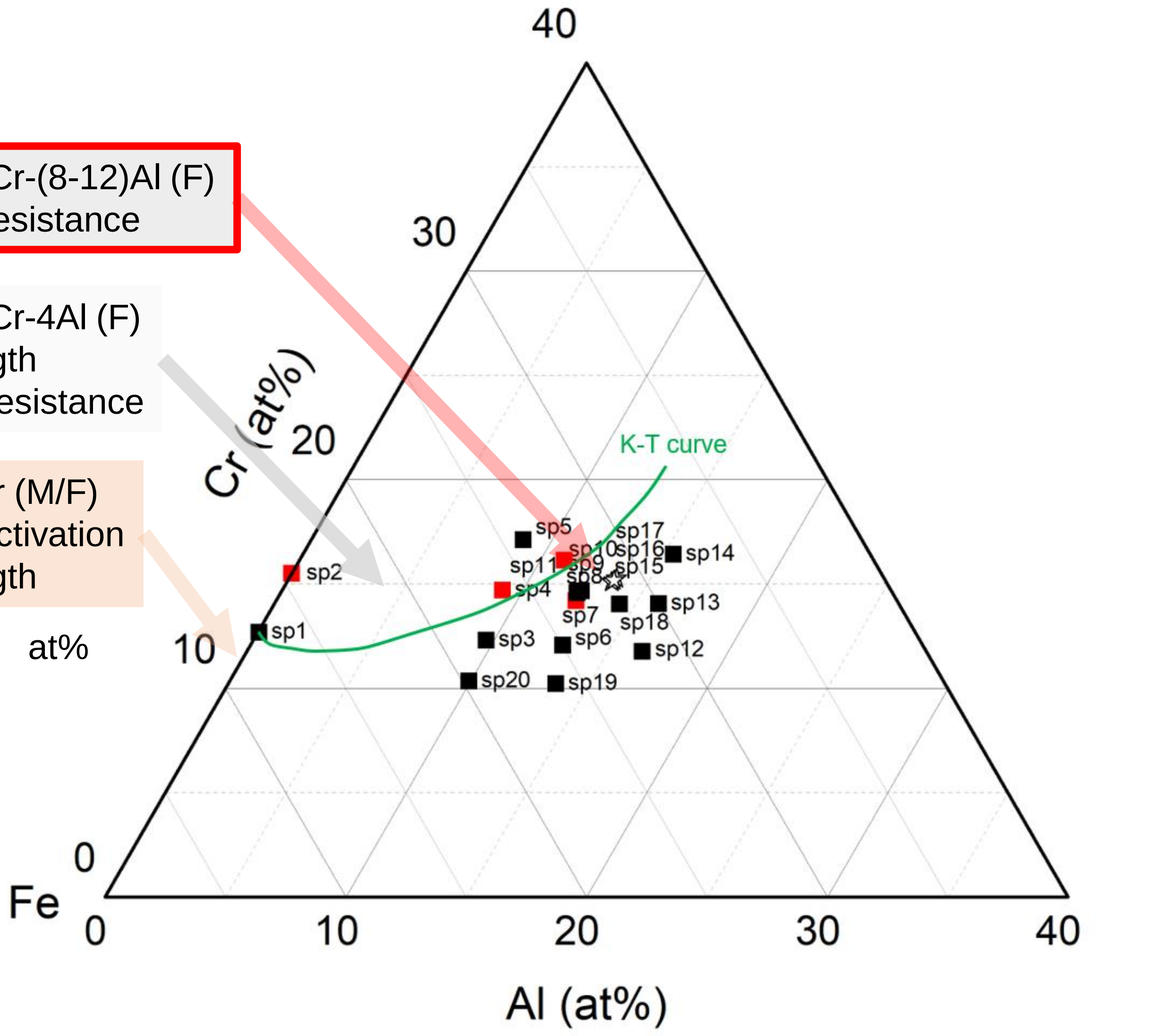
High Burn-up
Replaced with auste-nitic stainless steels
→Fast Reactors

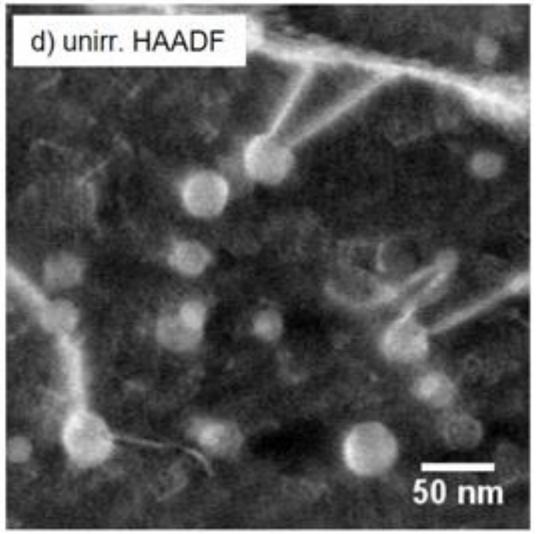
Long Lifetime
Replaced with RAFS
→Fast Reactor
→Fusion Reactors

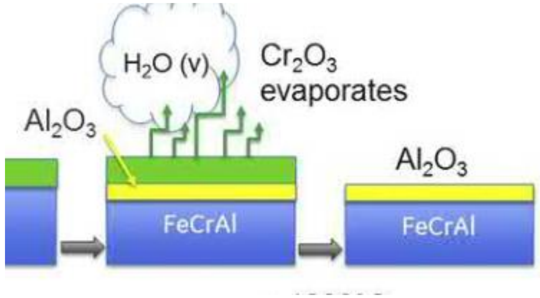
Fe-(14-15)Cr-(8-12)Al (F)
Oxidation resistance

Fe-(14-15)Cr-4Al (F)
High Strength
Corrosion resistance

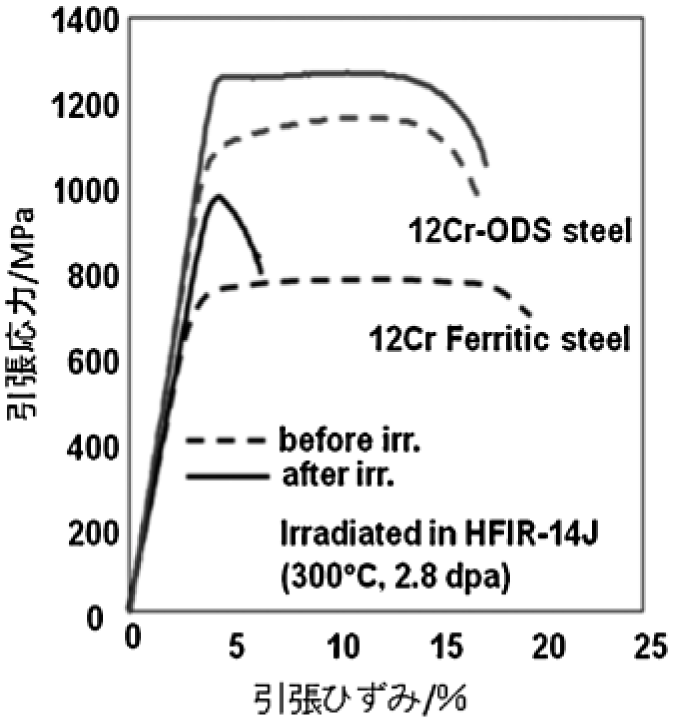
Fe-(9-12)Cr (M/F)
Reduced Activation
High Strength





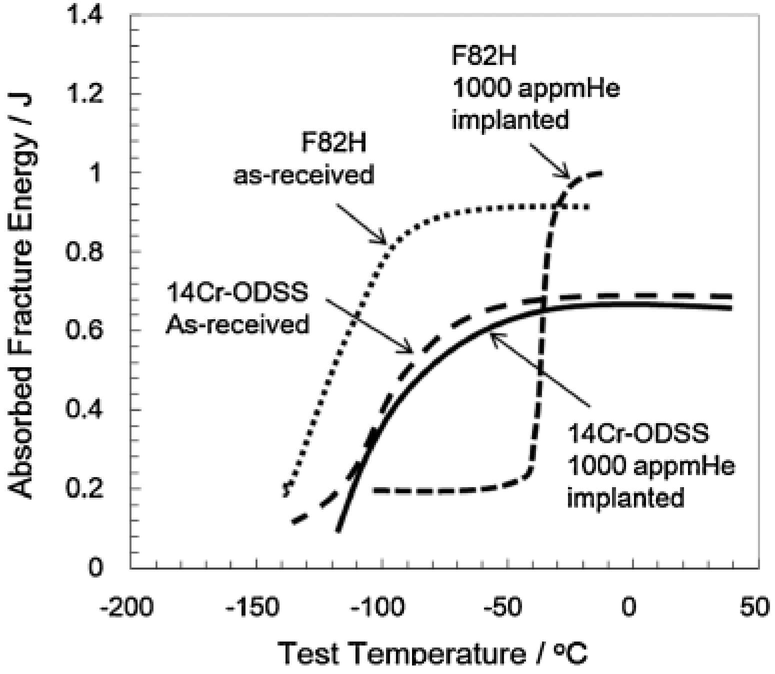


RAFS vs ODSS



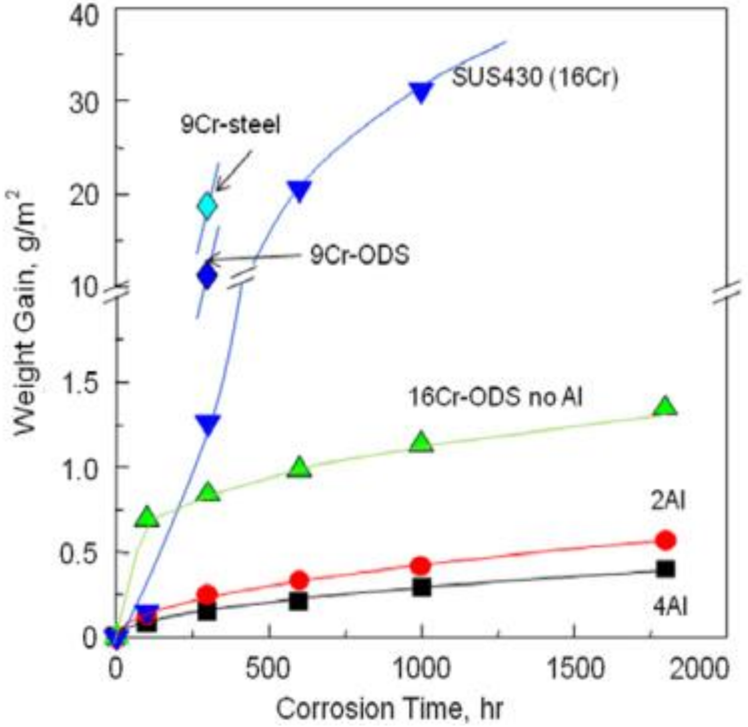
Ductility

F82H vs ODSS



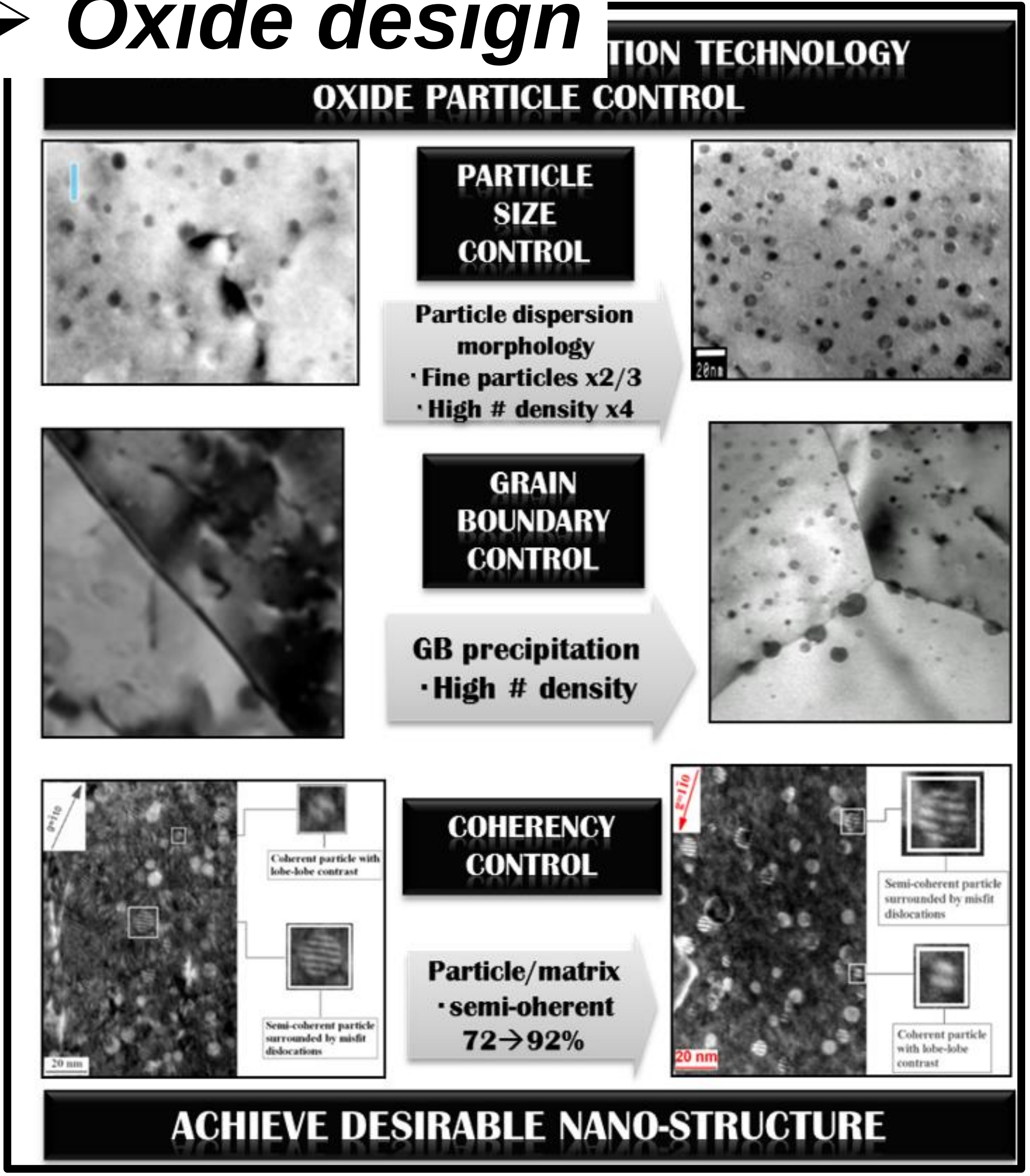
DBTT

Al-free vs Al-added

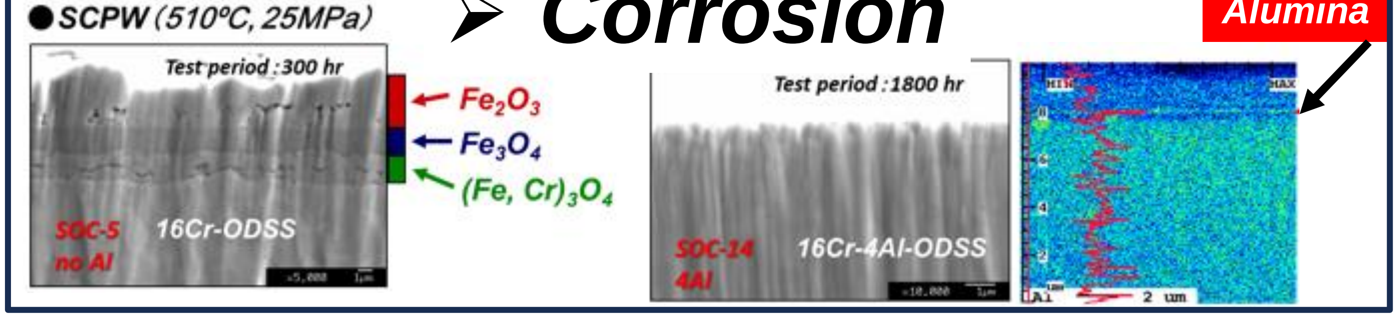


Weight gains after corrosion tests in SCPW (783K, 25MPa)

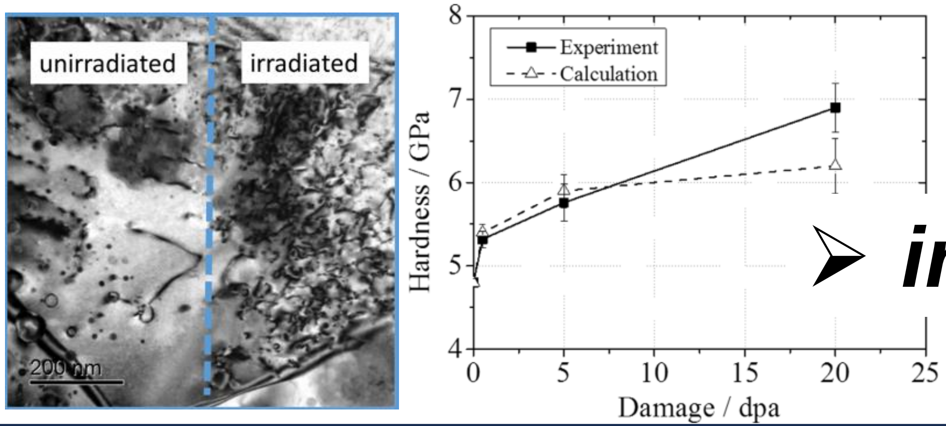
➢ Oxide design



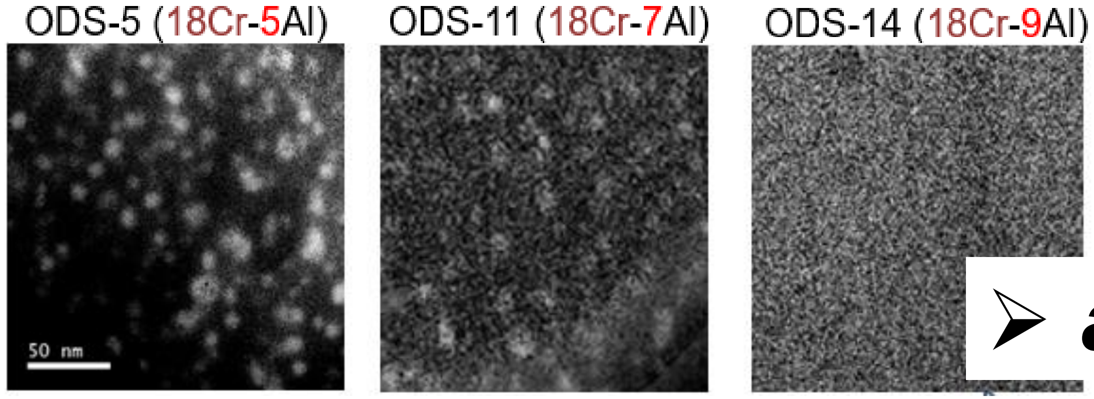
➢ Corrosion



➢ irradiation



➢ aging



Kyoto University

Akihiko Kimura

- Corrosion
- Ion-Irradiation
- Aging
- Joining

Hokkaido University

Shigeharu Ugai

- Manufacturing
- Metallurgy
- Creep

Japan Atomic Energy Agency

Shinichiro Yamashita

- Neutron irradiation

Nippon Nuclear Fuel Development, Co. Ltd

Kan Sakamoto

- Fuel compatibility

Kimura Lab

• Synthesis & fabrication	Mechanical alloying, arc-melting, rolling, heat-treatment, polishing
• Joining	Friction stir welding, hot isostatic pressing
• Mechanical test	Nano-indentation, multi-temperature tensile, small punch, Charpy, hardness
• Microscopy and spectrum	TEM, FIB, EBSD, SEM, XRD, EPMA, AES, TDS, AFM, ion-mill…
• Irradiation	DuET: Fe, Si, He
• Corrosion	Super critical water loop with different DO and DH
• Simulation	Assoc. Prof. Morishita group

Main topics:

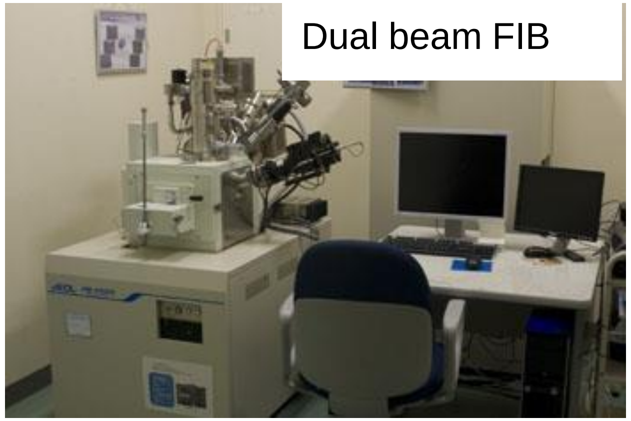
Nuclear materials: F82H, FeCrAl ODSS, tungsten, stainless steels, RHEA

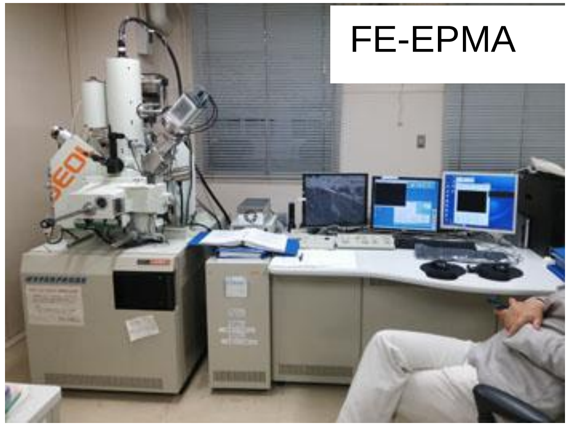

JEM 2200F


JEM 2010


Hitachi FB 2200


FE-SEM


Dual beam FIB


FE-EPMA


Super critical water loop

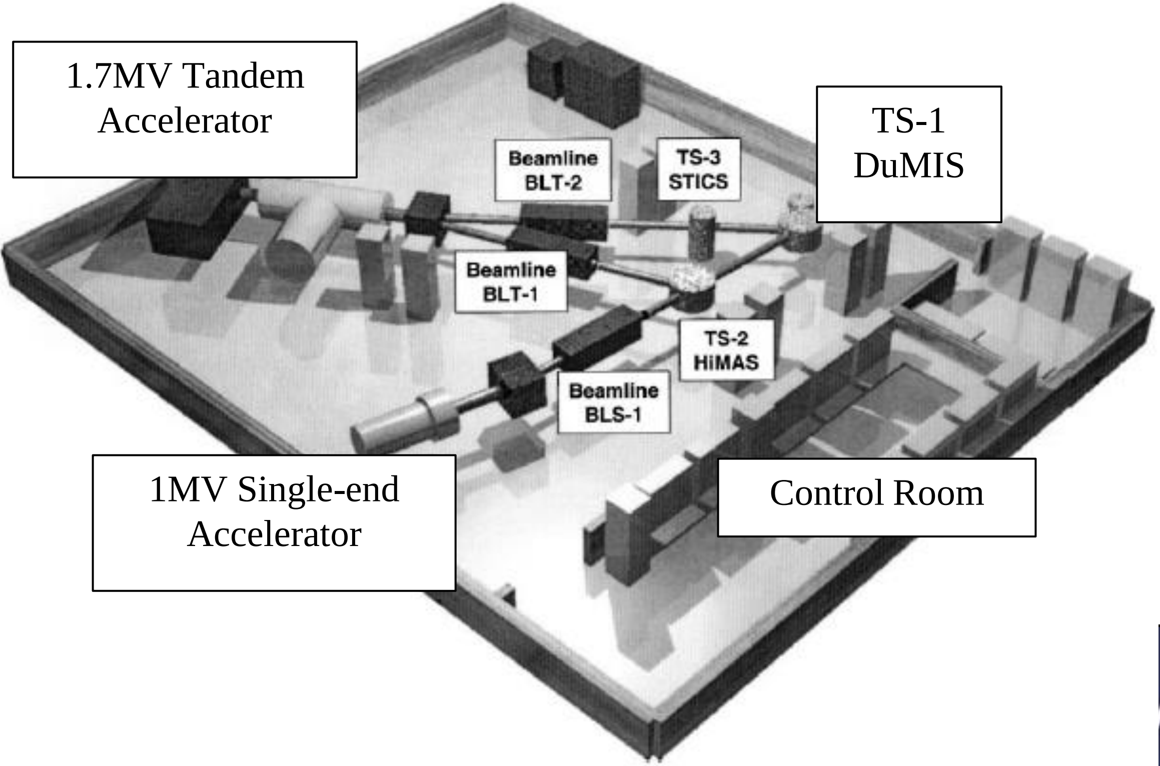

Charpy


Tensile, high T


Gentle mill


FE-AES

DuET,
Kyoto University



Ion type: 6.4 MeV Fe^{3+}
(+/- 1MeV He^{+})

Raster scanned:
X: 1000 Hz
Y: 300 Hz

Vacuum < 5×10^{-8} Torr

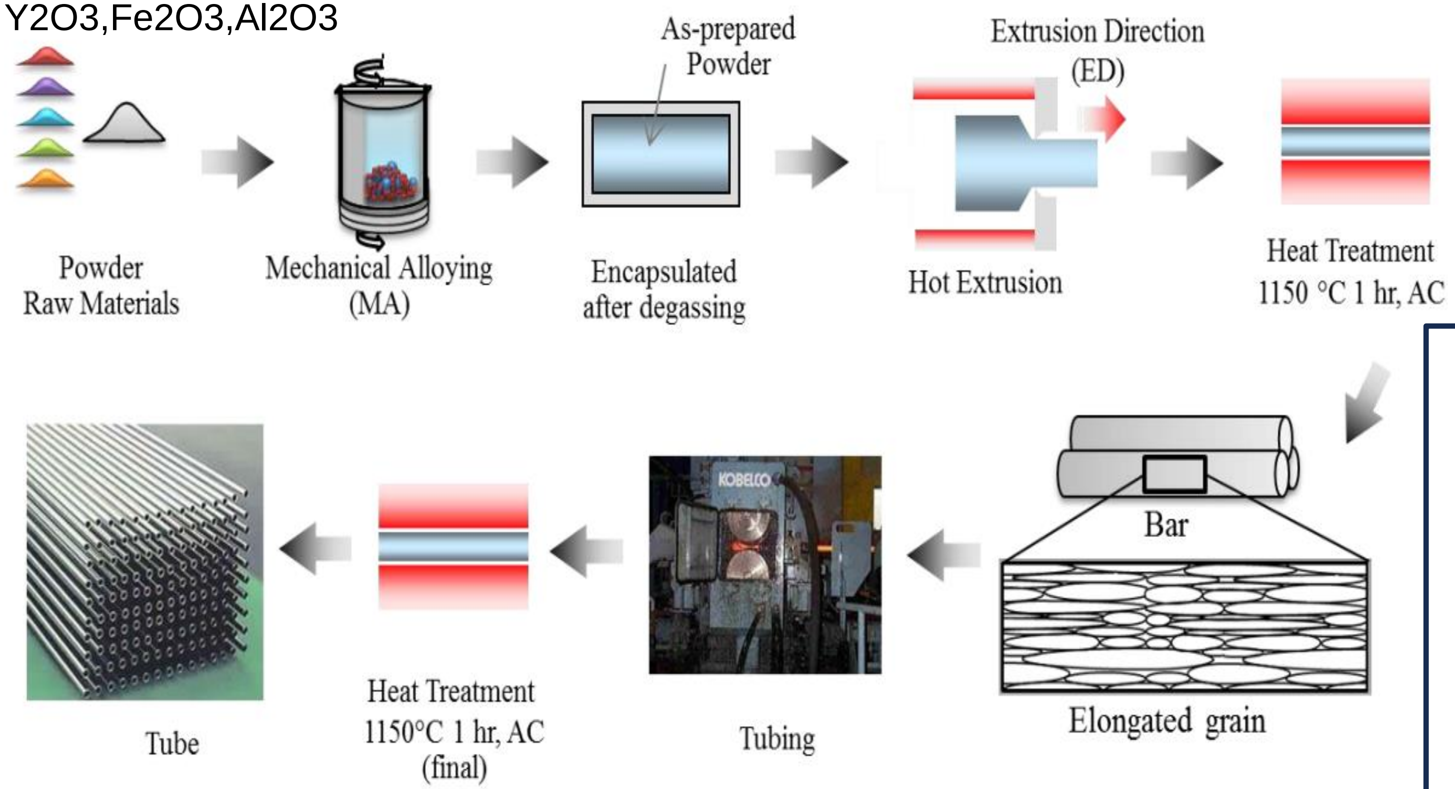
Heater type:
2kw infrared UHV

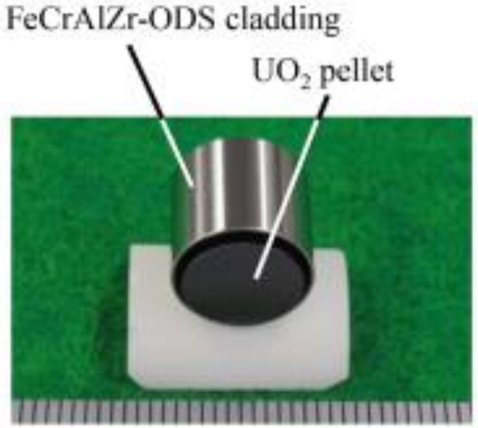
Temperature monitor:
high resolution thermography

Temperature rage:
RT ~ 1800 °C

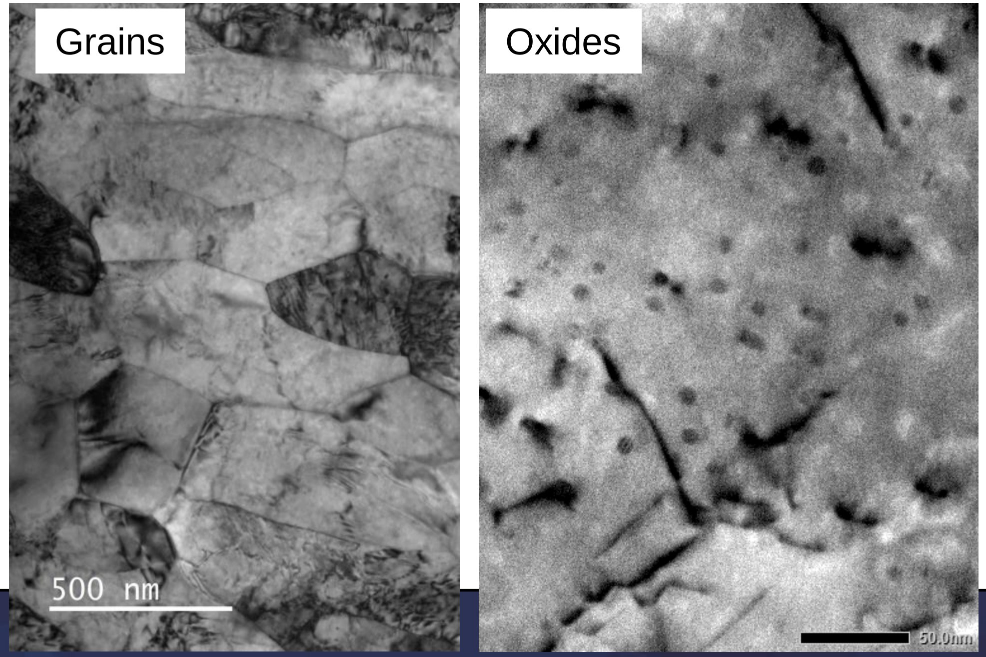
FeCrAl ODS steels

Fe,Cr,FeY,
Y2O3,Fe2O3,Al2O3





Sakamoto, JNM, 2021



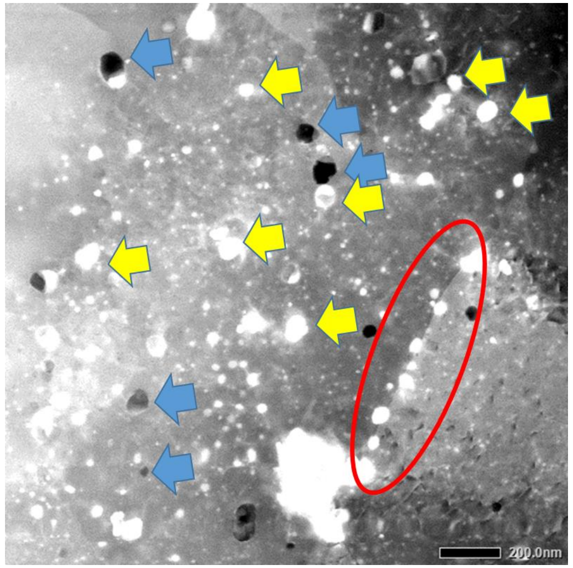
Zhang, JIM presentation, 2017

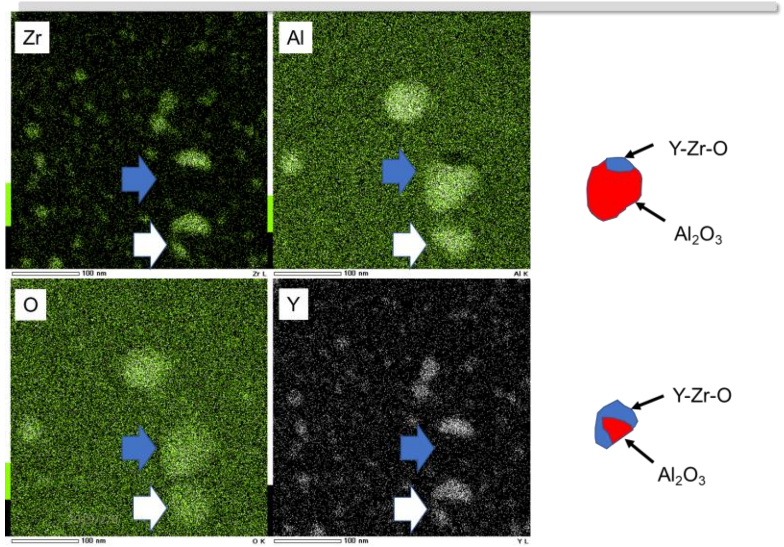
Oxides

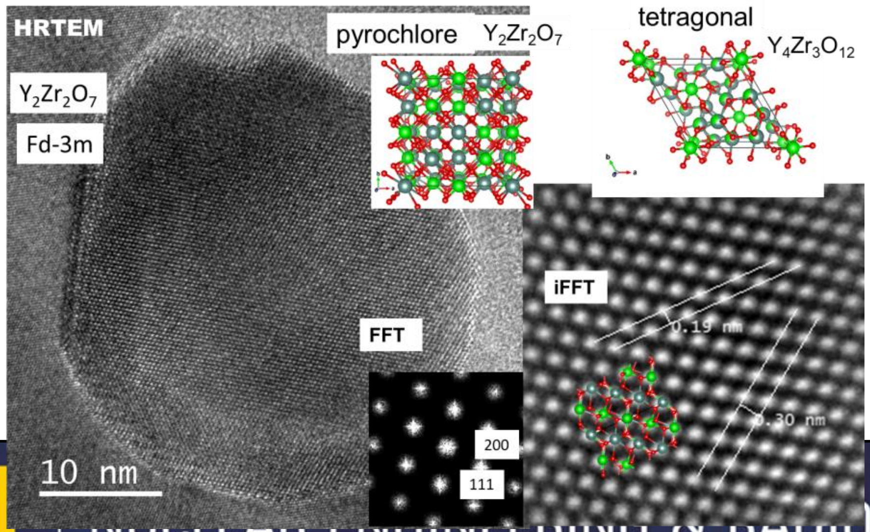
Zhang, JIM, 2017

Zr-added FeCrAl ODS steels

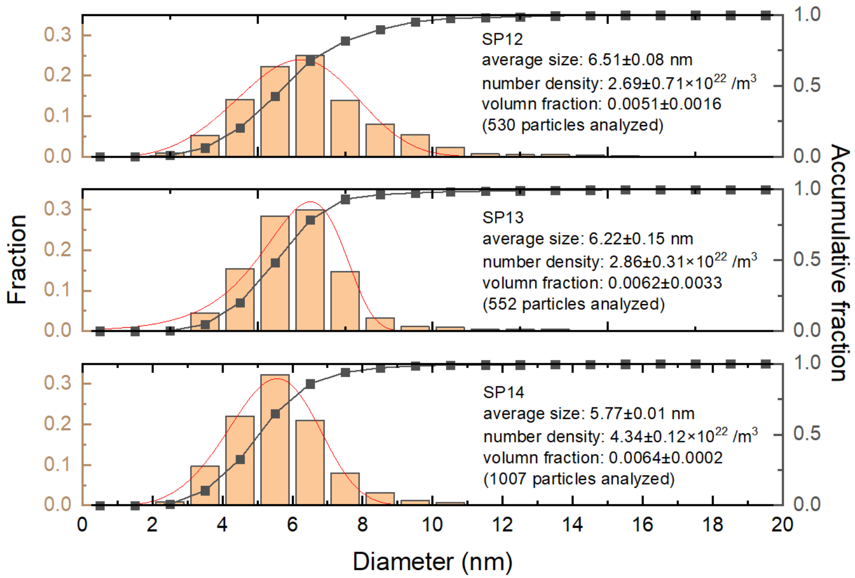
STEM-HAADF



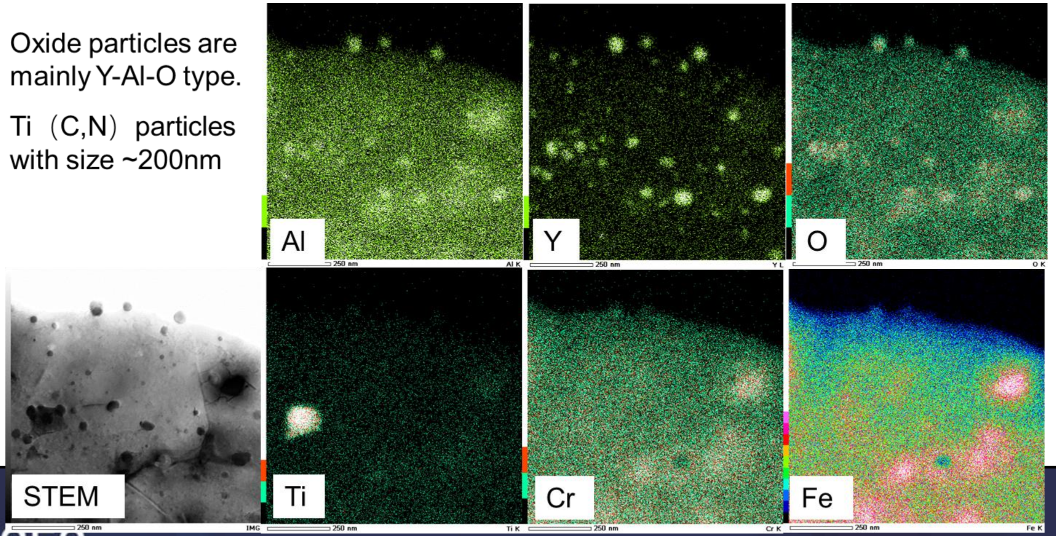




Zr-free FeCrAl ODS steels

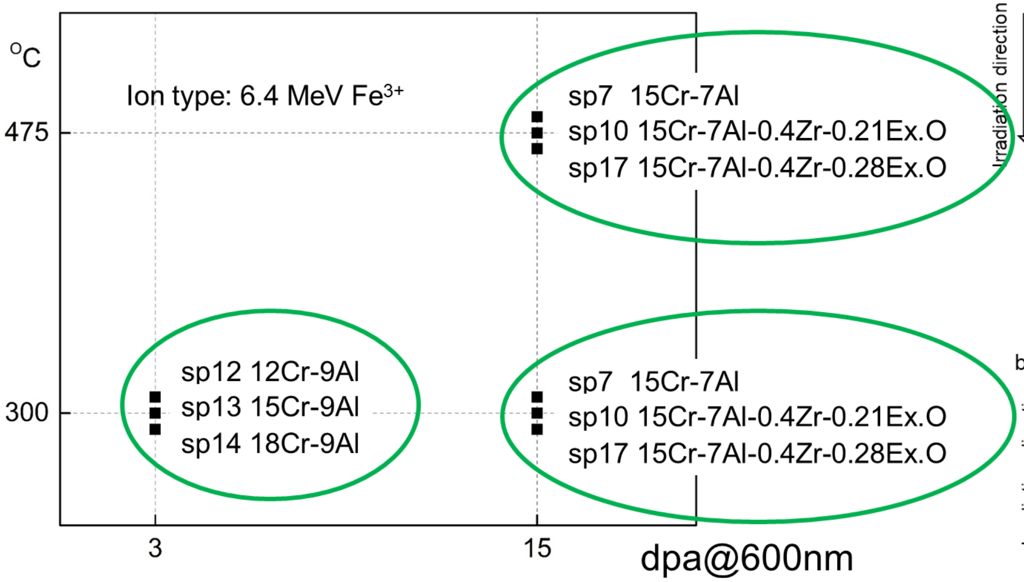


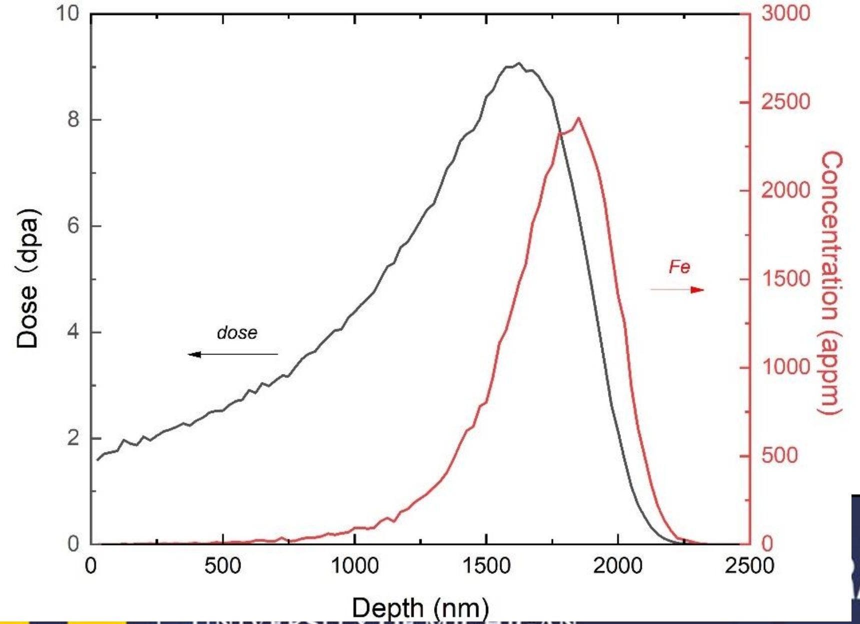
- Oxide particles are mainly Y-Al-O type.
- Ti（C,N）particles with size ~200nm

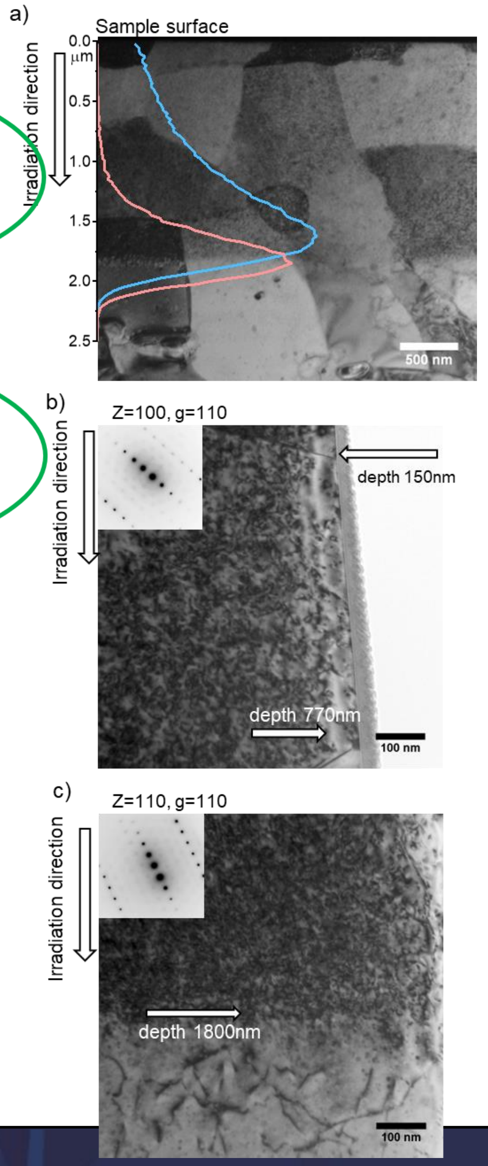


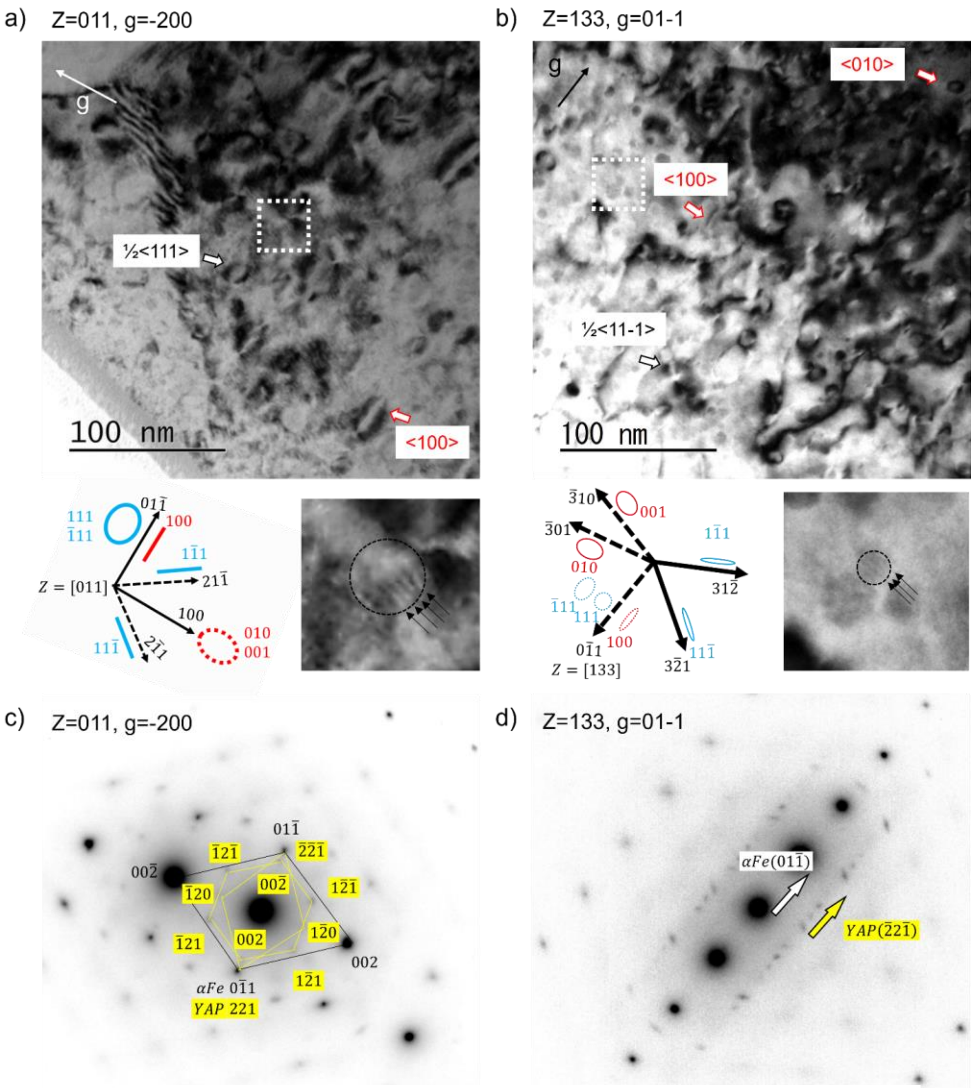
Irradiation and microscopy

Zr-free FeCrAl ODS steels





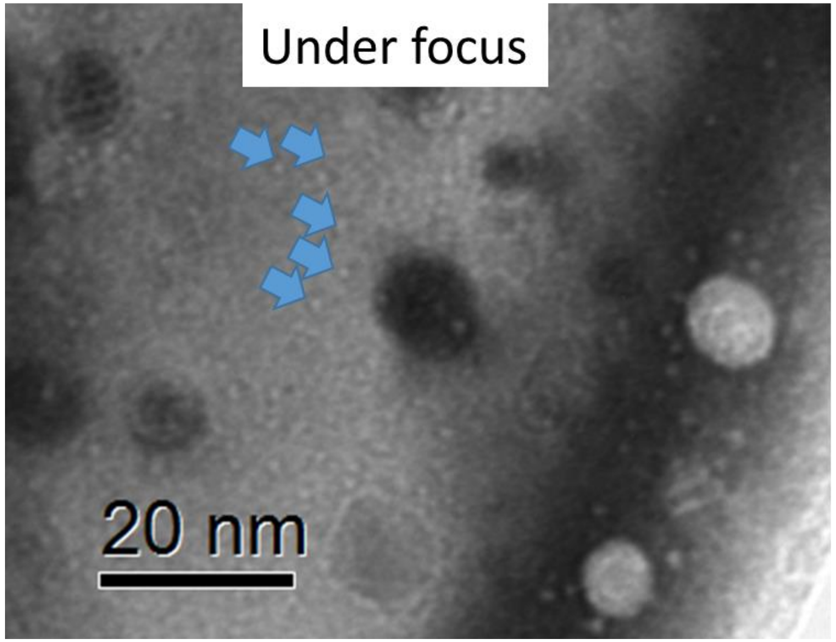


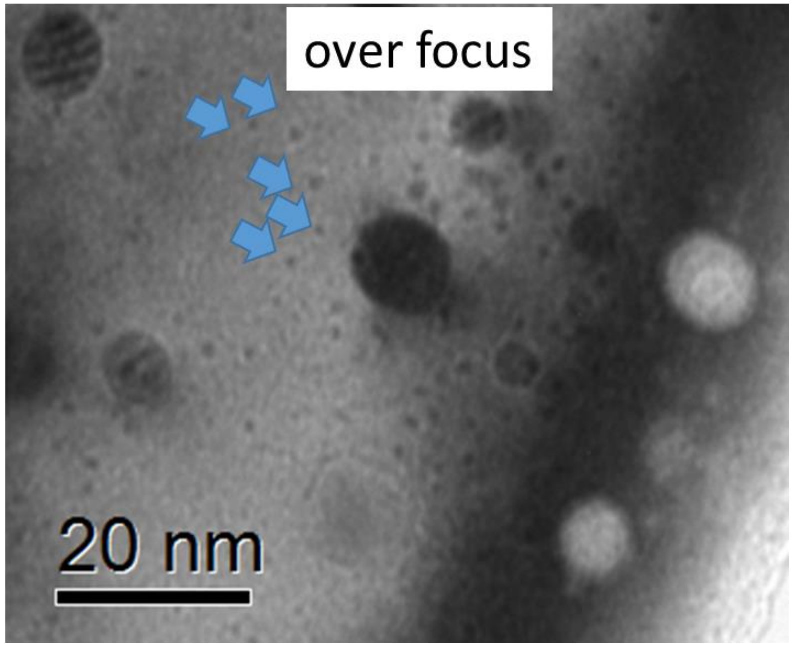


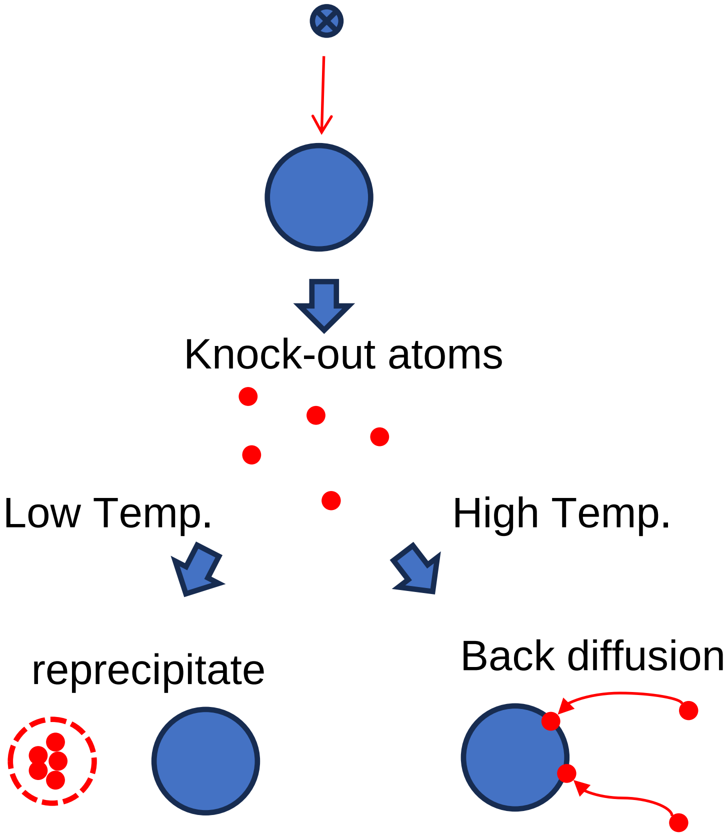
αFe(001)//YAP(1-20) and αFe[100]//YAP[210].

Oxides stability

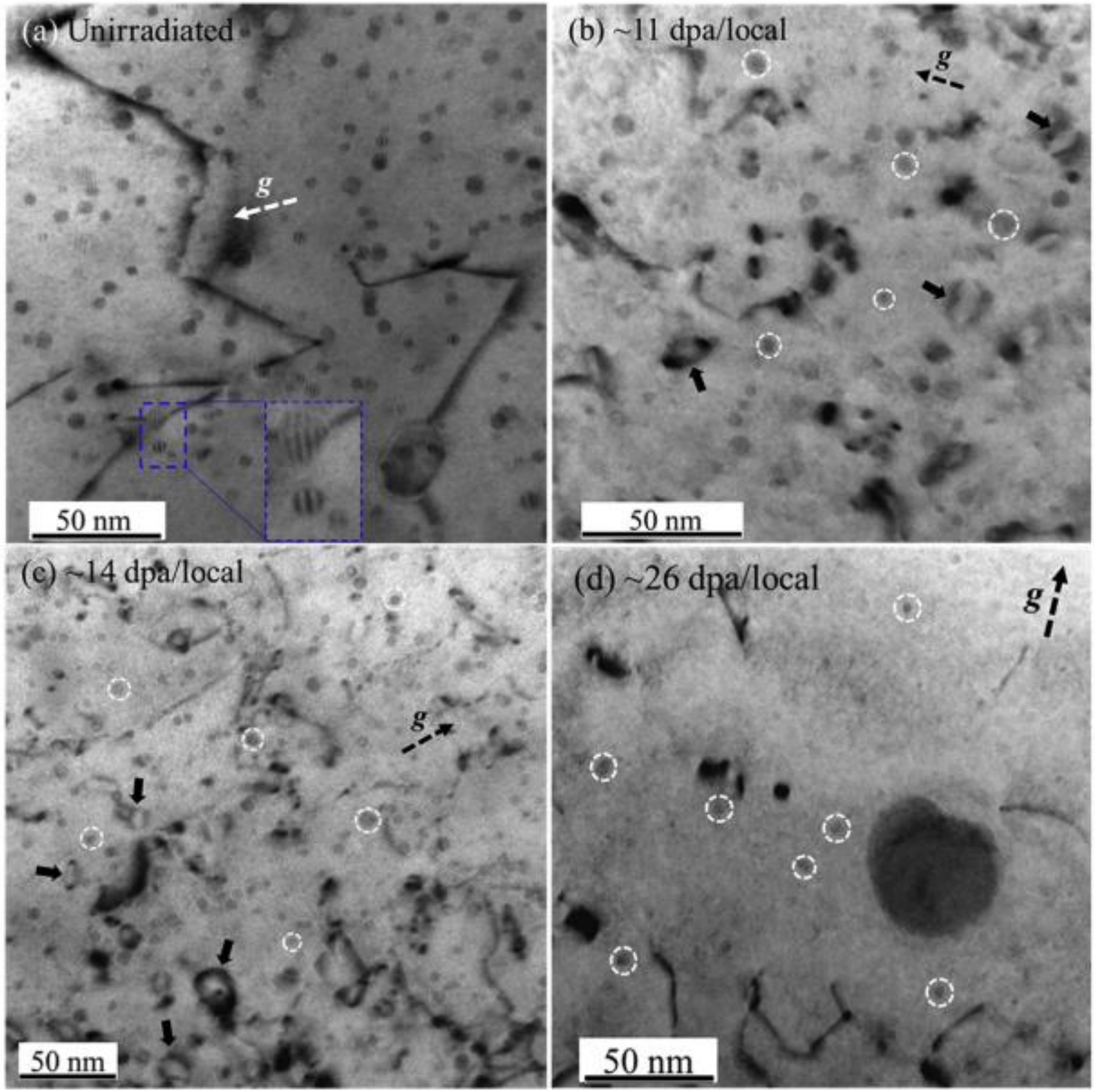
Zr-added FeCrAl ODS steels



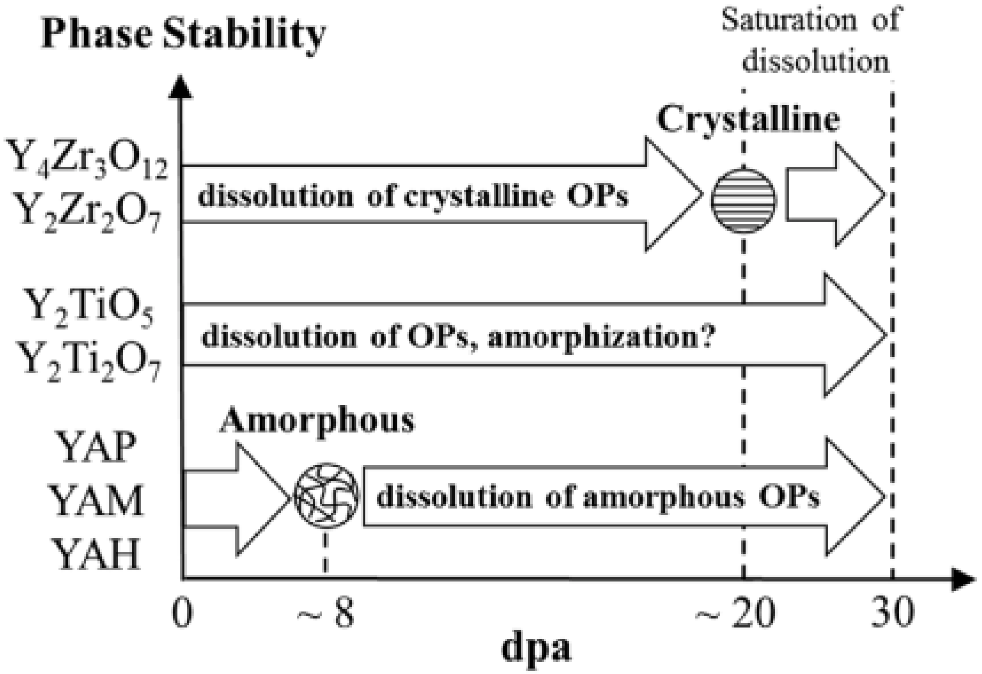




- Satellite precipitates at 300°C
- Not occurred at 475°C



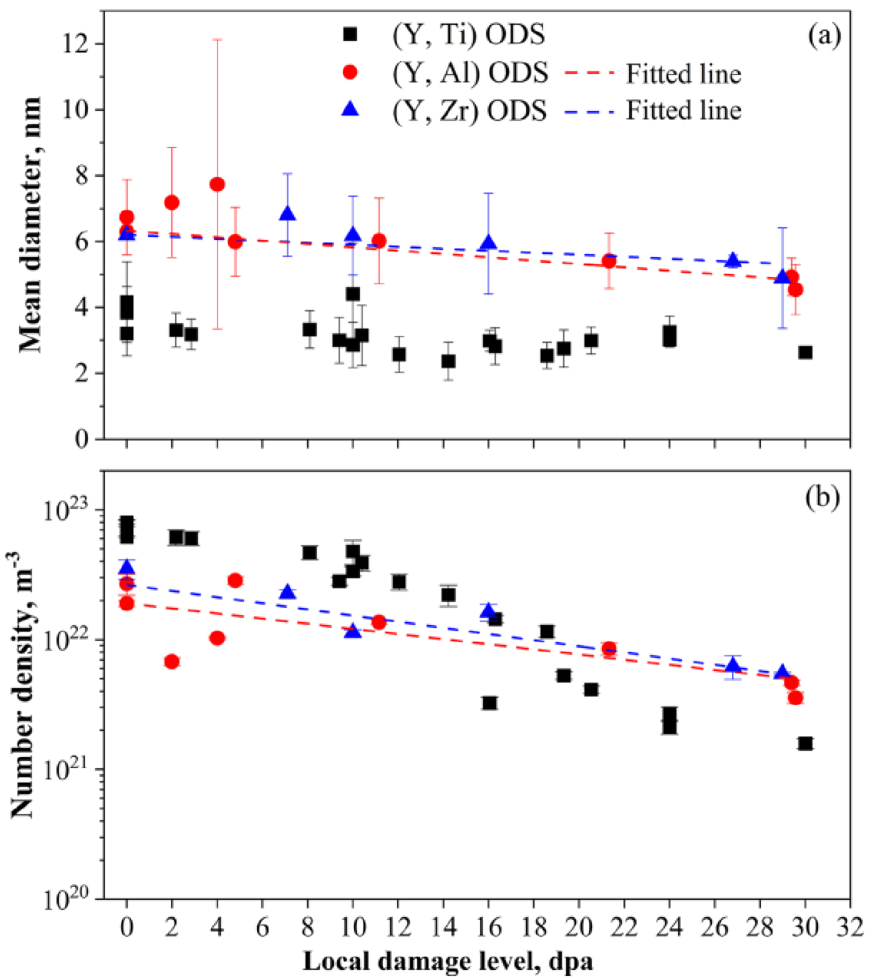
200°C



Zr-added FeCrAl ODS steels

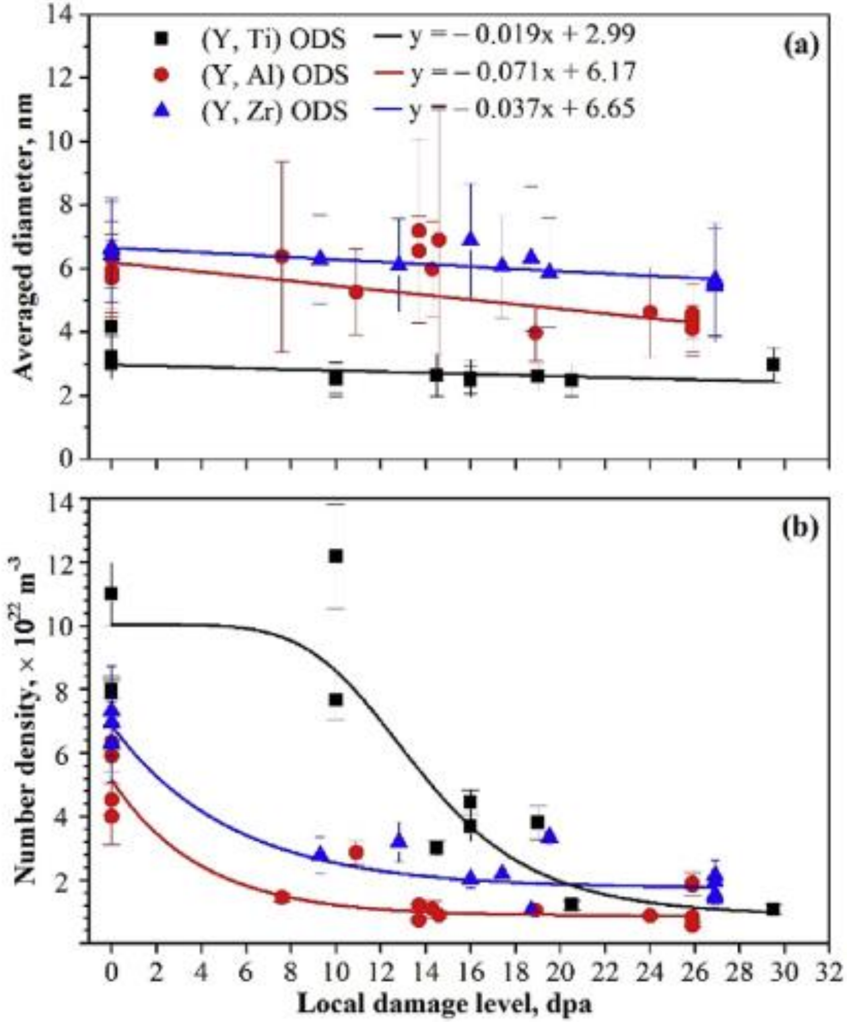
Song et al, JNM 2020

30°C



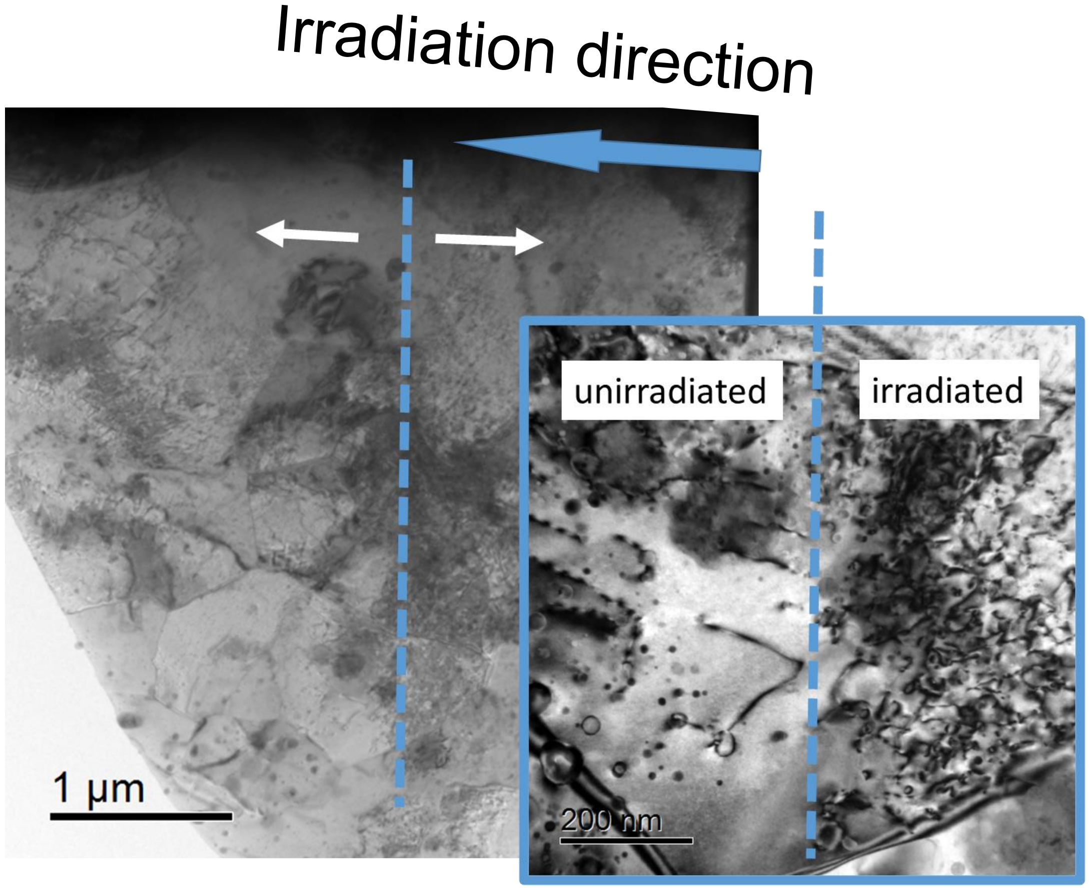
Song Acta Materialia, 2022

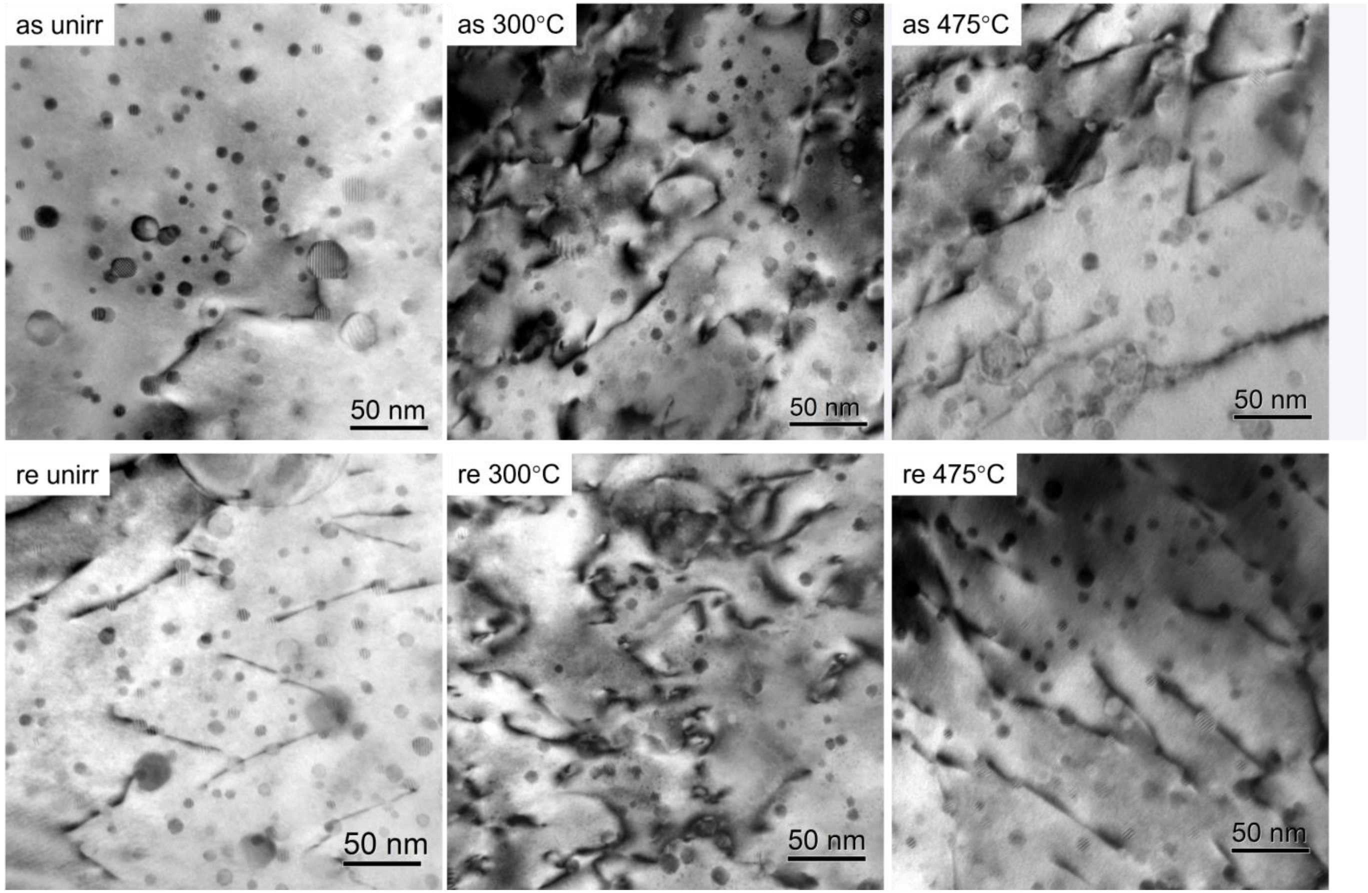
200°C



Song JNM, 2022

Zr-added FeCrAl ODS steels

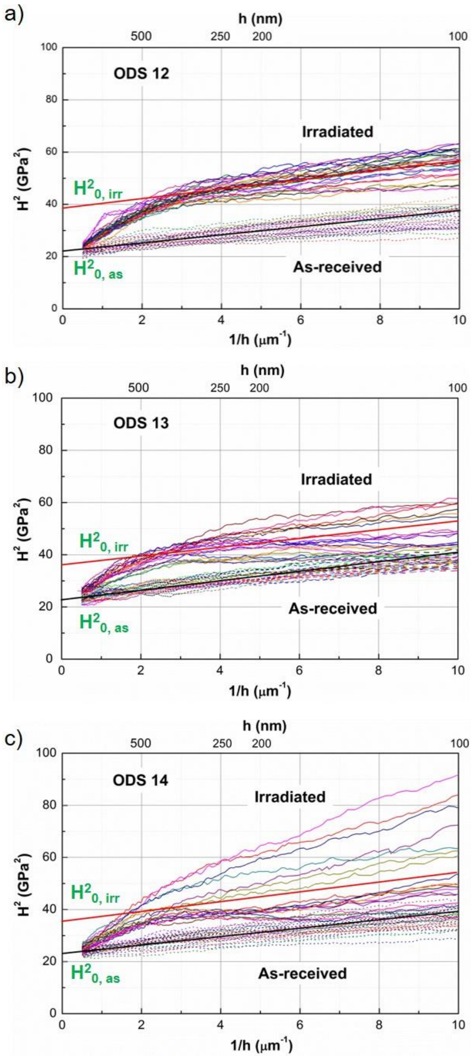


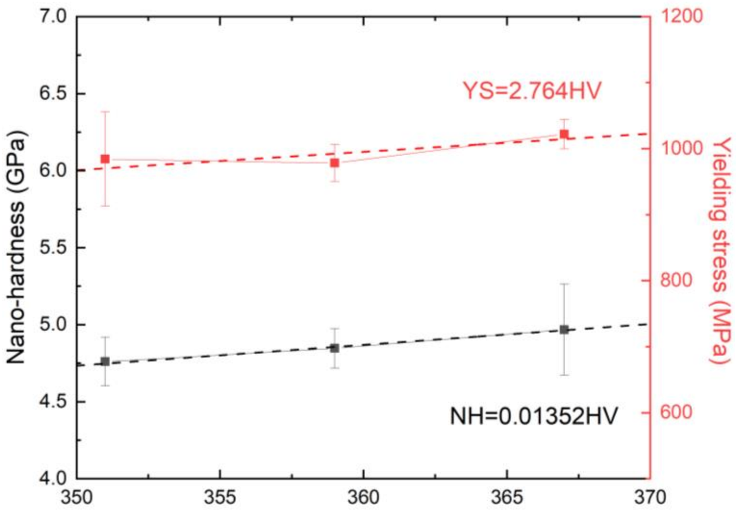


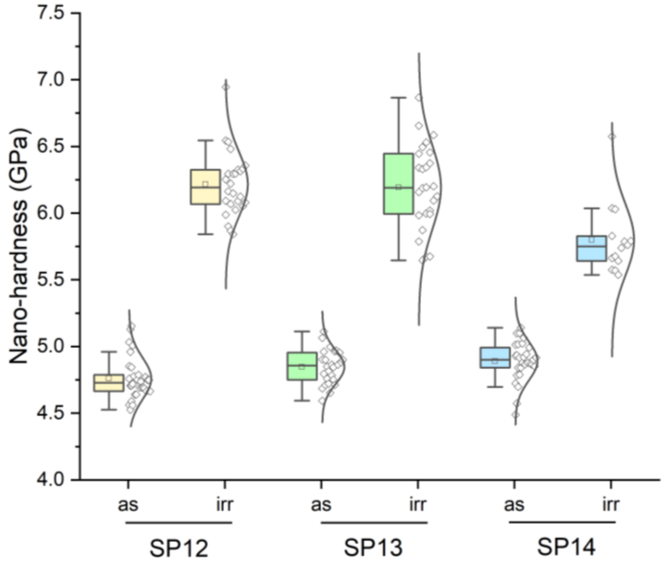
Irradiation hardening

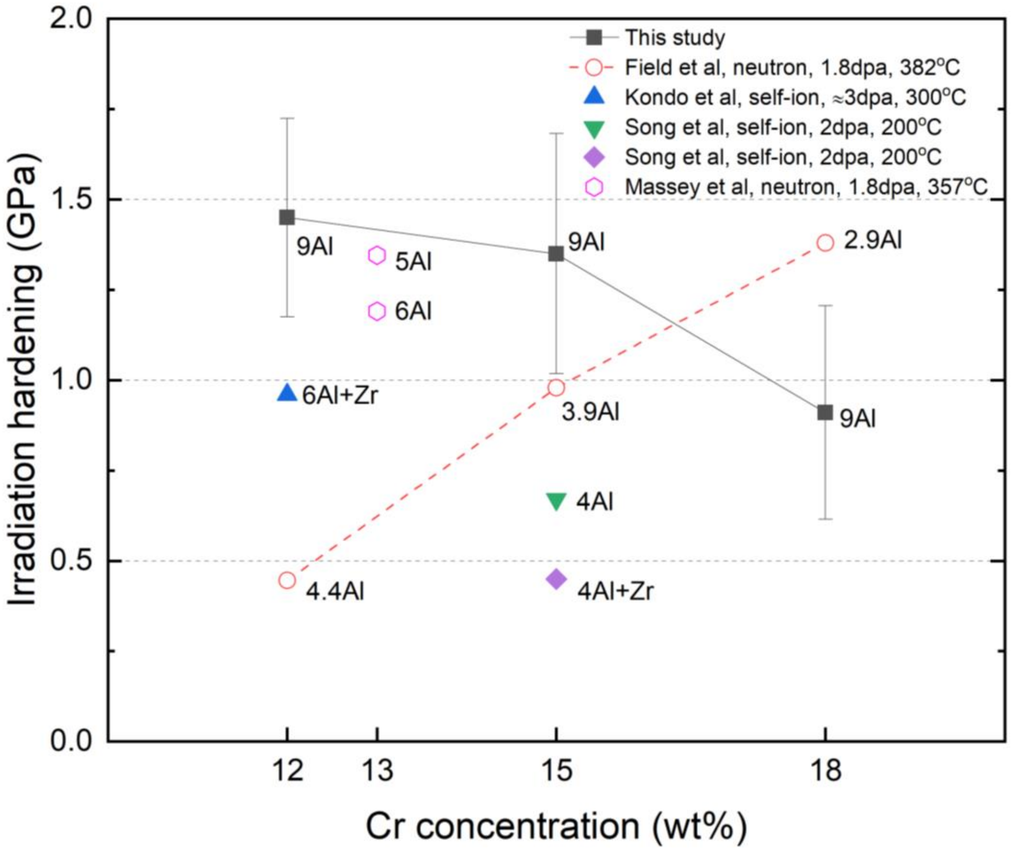
FeCrAl neutron irradiation Cr ΔH

FeCrAl ODS self-ion irradiation Cr ΔH

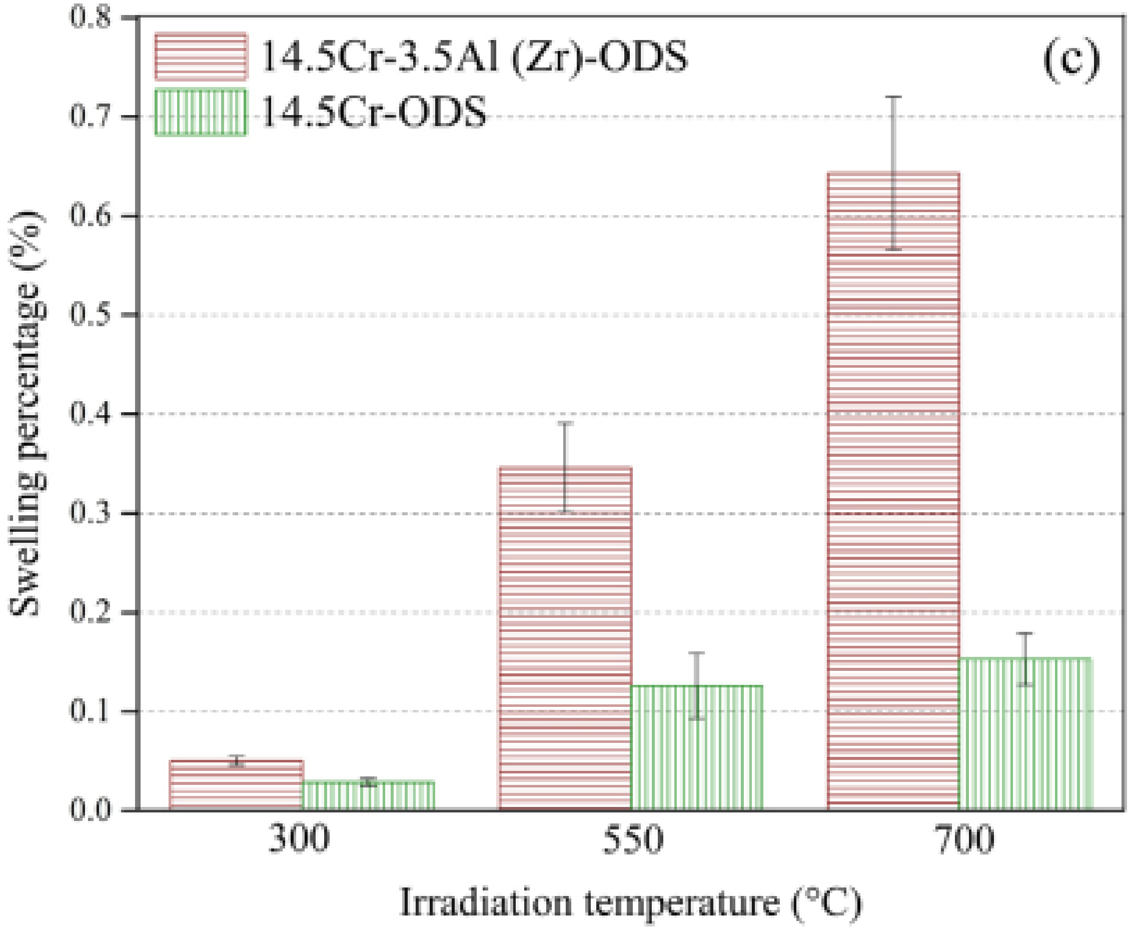


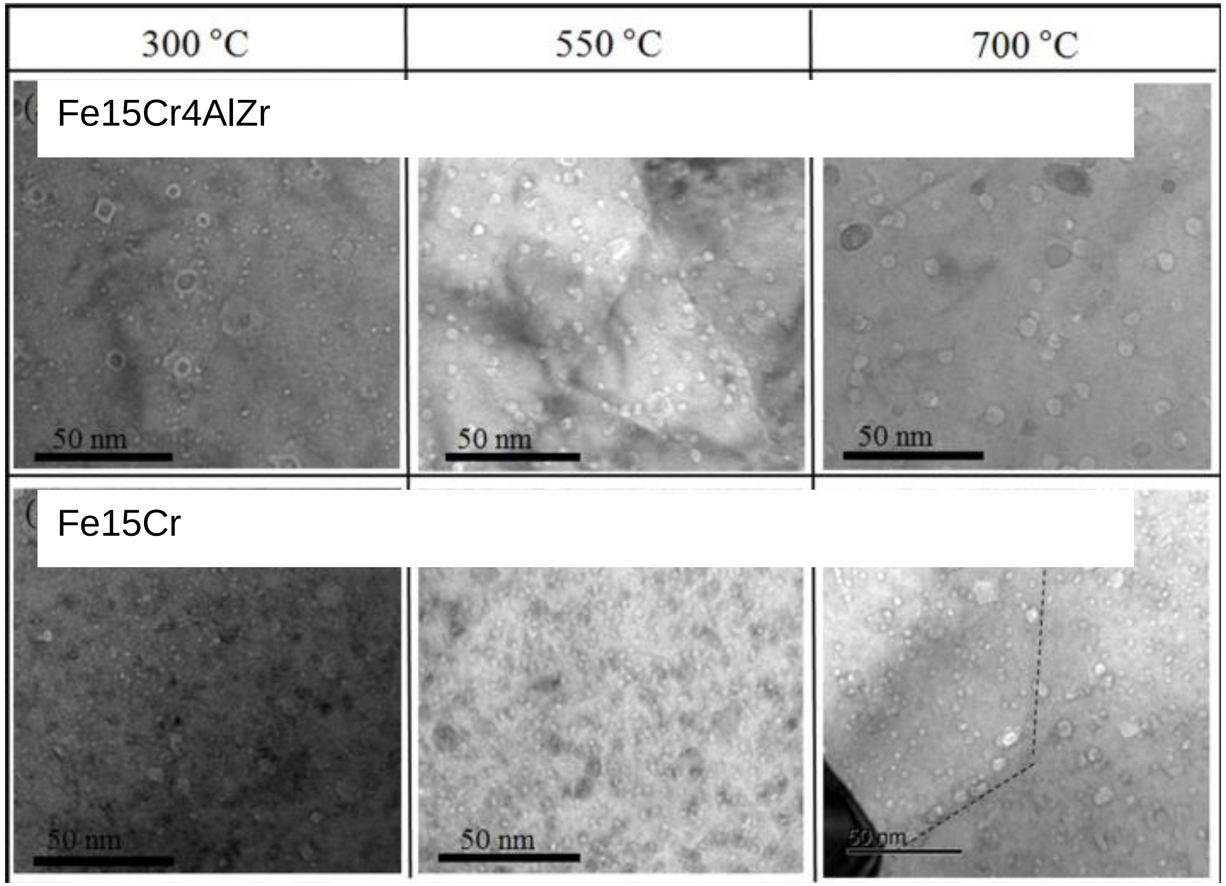






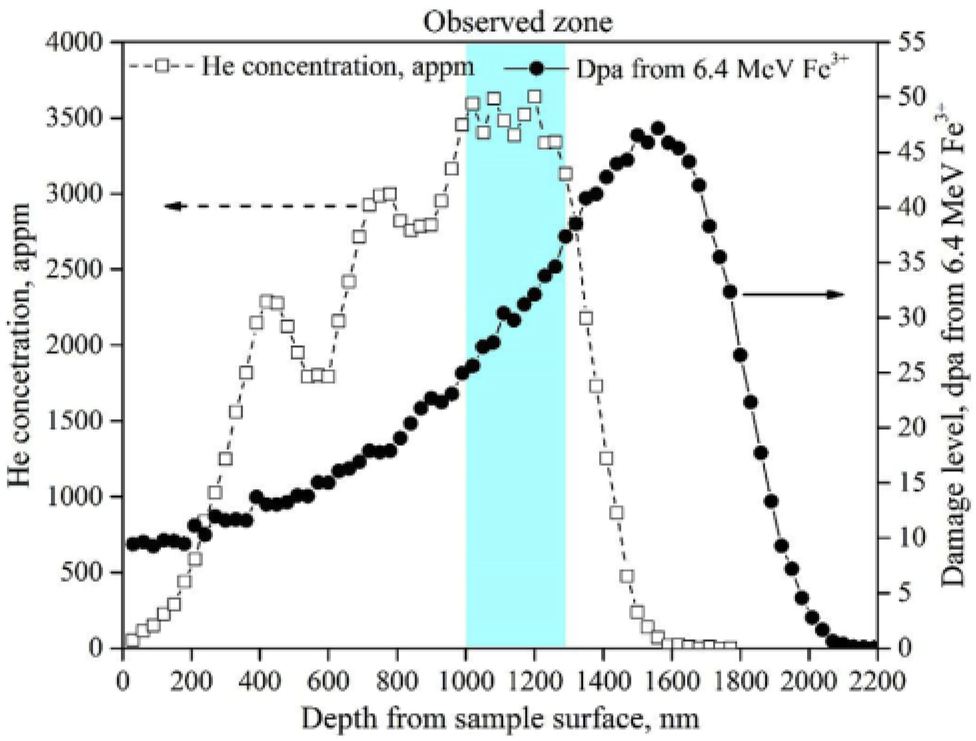
2) Helium embrittlement

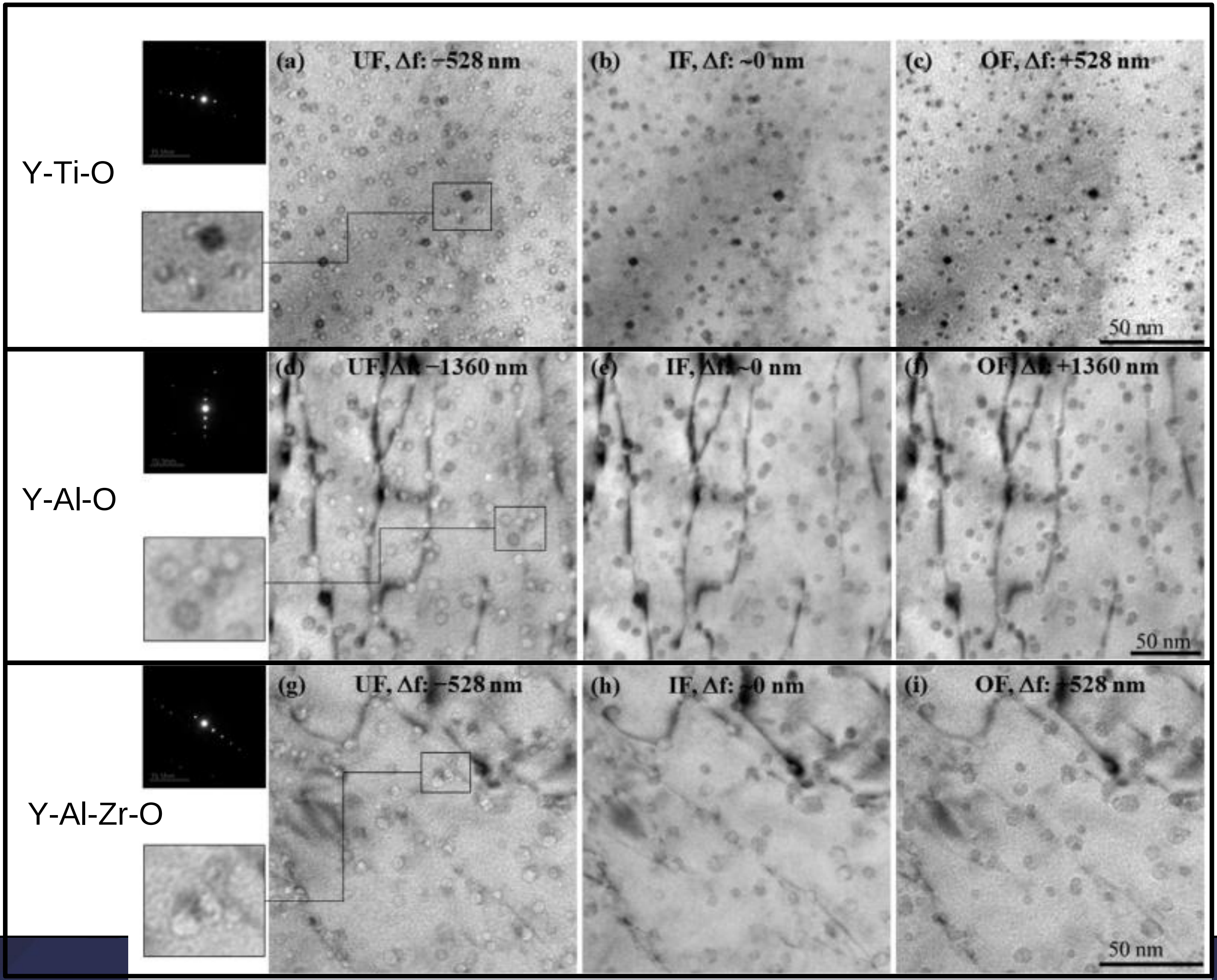




Dual-beam 550ºC

Song, JNM, 2018

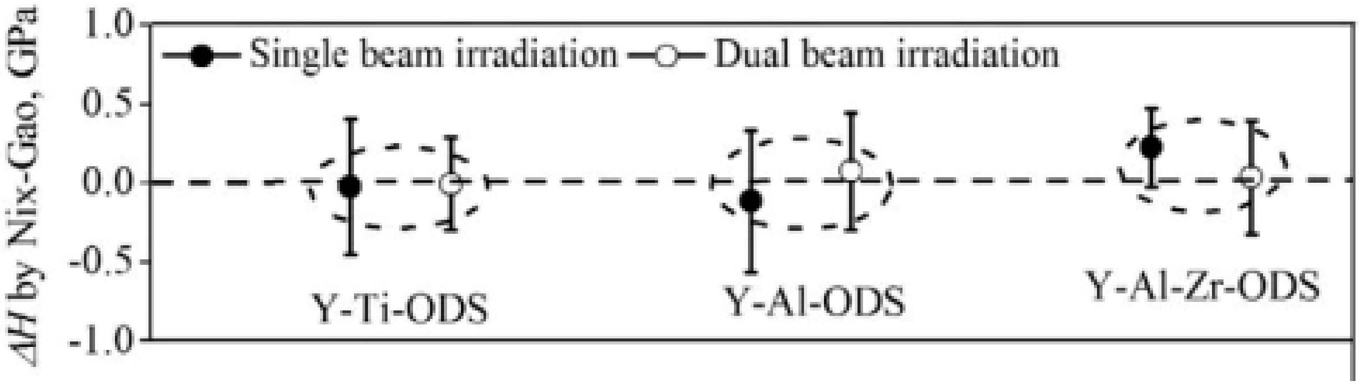




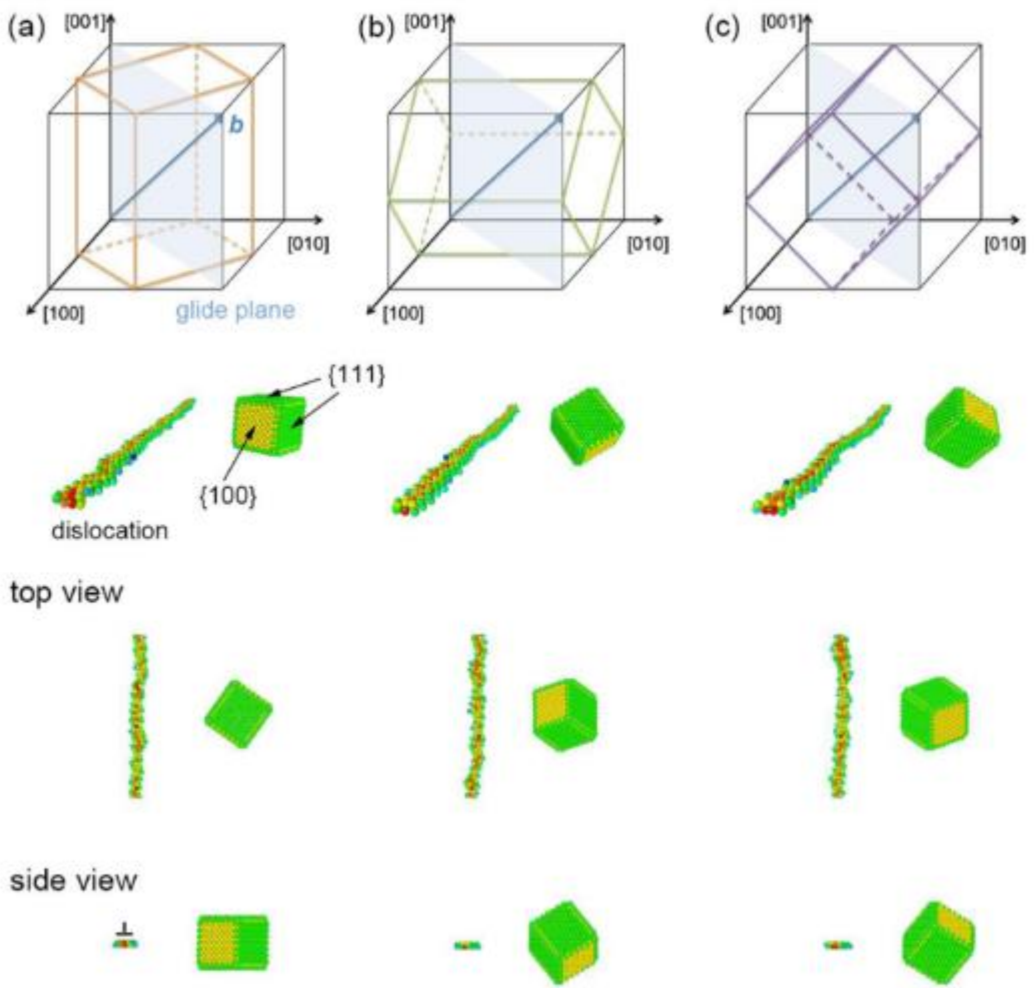
550ºC

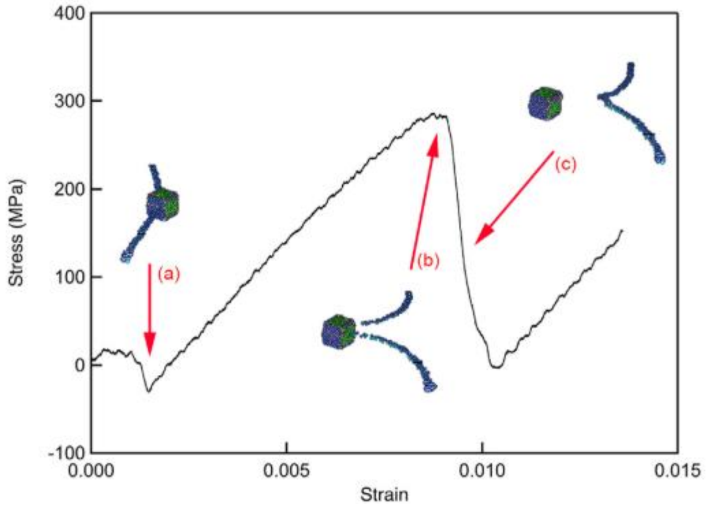
Table 3
Mean diameter (d_b), number density (N_b) of He bubbles, swelling ($\Delta V/V$), the number (T) of counted bubbles and the fraction (F) of bubbles adjacent to oxide particles in the depth range of 1000–1300 nm under dual beam irradiation.

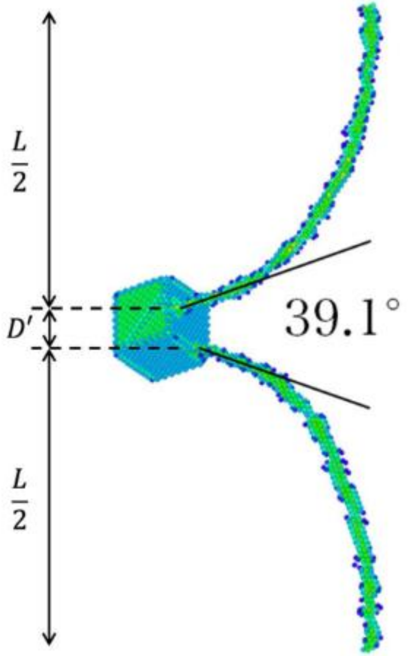
	Y-Ti-ODS	Y-Al-ODS	Y-Al-Zr-ODS
d_b, nm	2.8 ± 0.1	6.6 ± 1.0	4.5 ± 0.2
N_b, m^{-3}	$(1.1 \pm 0.1) \times 10^{23}$	$(2.7 \pm 0.2) \times 10^{22}$	$(3.6 \pm 0.6) \times 10^{22}$
$\Delta V/V$, %	0.13 ± 0.02	0.53 ± 0.19	0.20 ± 0.04
T; F, %	287; 73.7	194; 81.4	129; 82.2



Yabuuchi, JNM, 2023







$$\tau_c = \alpha \frac{\mu b}{L}$$

and

$$\alpha = \cos \frac{\theta_c}{2}$$

Table 1
Strength factor for each void type and the average apparent obstacle diameter.

		Strength factor, α (angle, θ_c [°])	Apparent obstacle diameter, D' [nm]
Facet type-a	Max.	0.8 (3.9)	3.0
	Ave.	0.78 (18)	
Facet type-b	Max.	0.8 (0)	1.9
	Ave.	0.77 (27.5)	
Facet type-c	Max.	0.8 (0)	2.2
	Ave.	0.78 (23.2)	
Spherical	Max.	0.8 (0)	2.1
	Ave.	0.79 (23.2)	

Irregular distribution

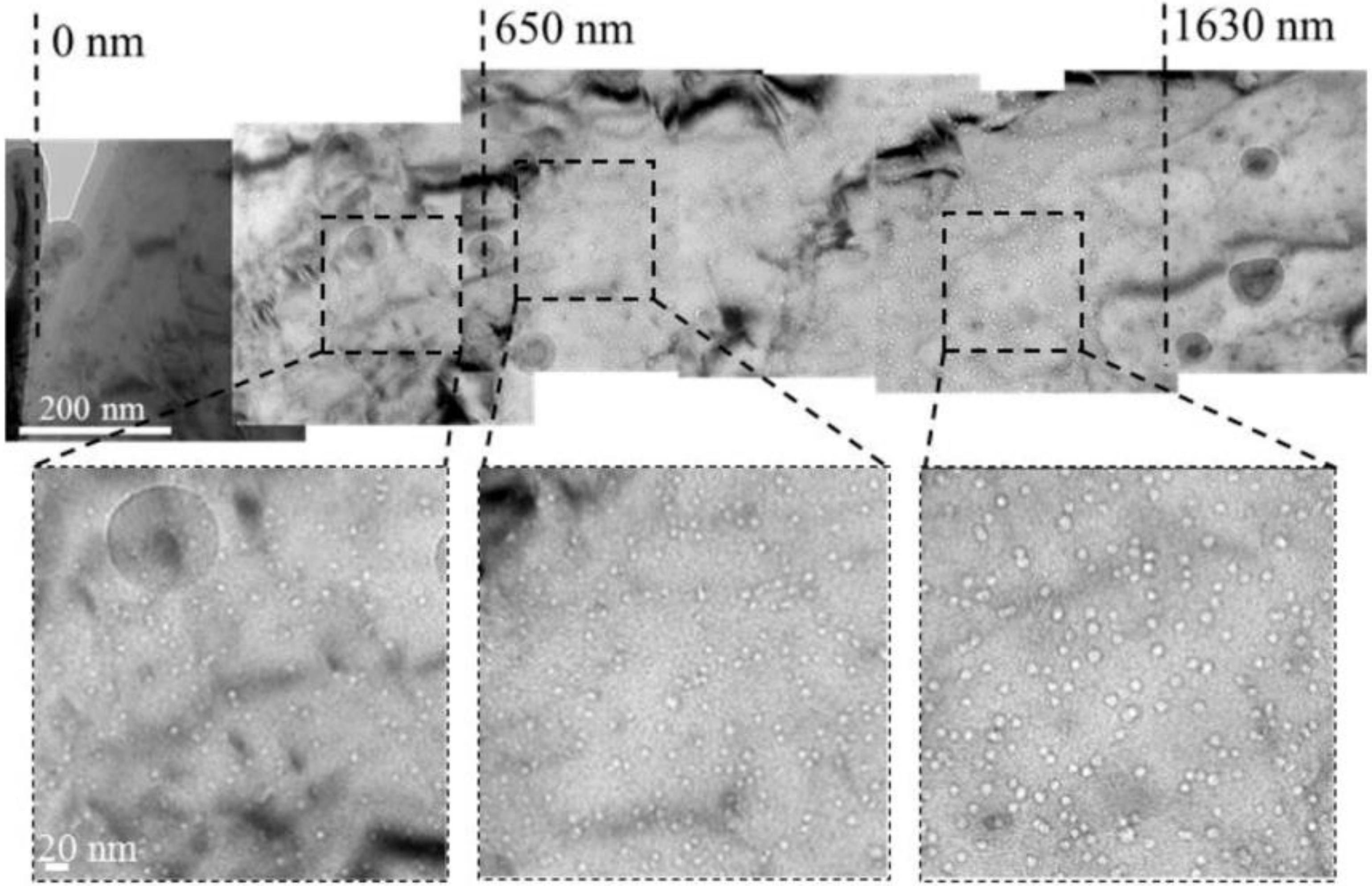
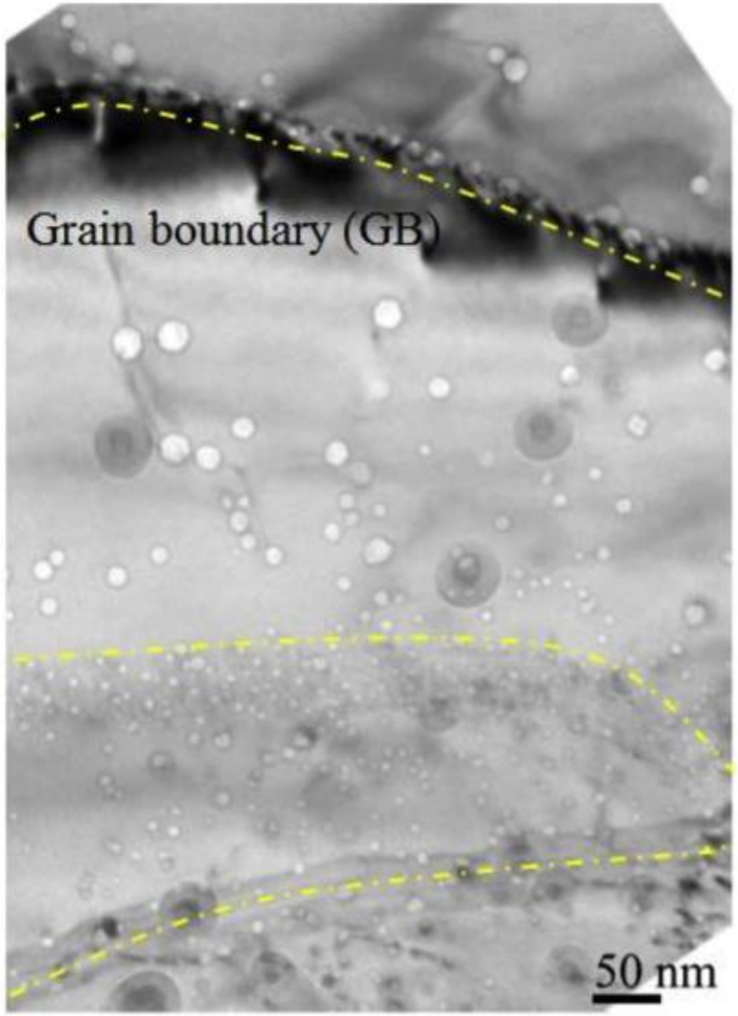


Fig. 3. He bubble distribution in Y-Al-ODS steel after dual-beam irradiation.



Irradiation + annealing

300^{o}C + 800^{o}C/100h

3500 appm He / 0.2 dpa

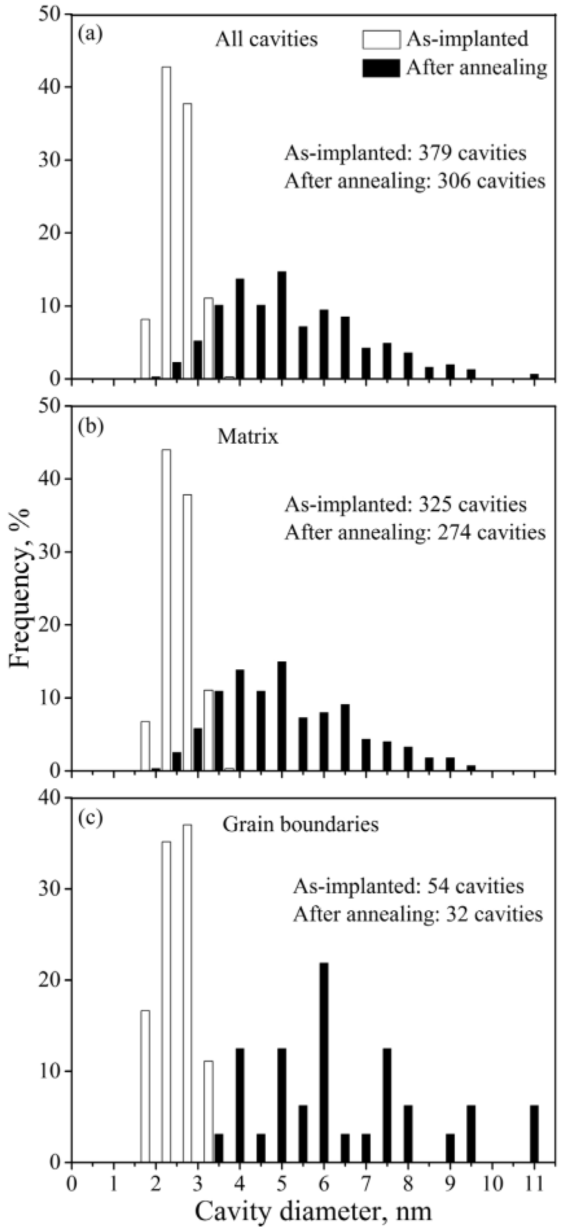


Fig. 6. Size distributions for all the cavities (a), cavities in the ferrite matrix (b) and cavities at the grain boundaries (c) in the specimens implanted with He at 300 °C before and after thermally aging at 800 °C/100 h in the depth range of roughly 1000–1300 nm (3500 appm He/0.2 dpa).

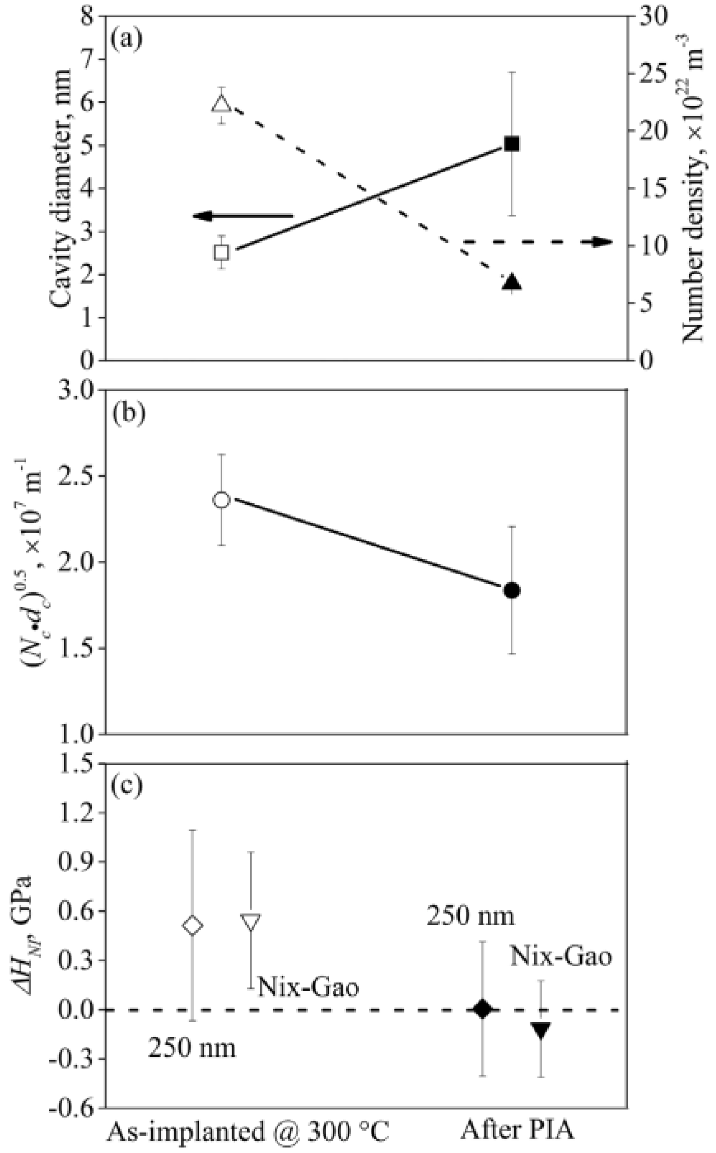
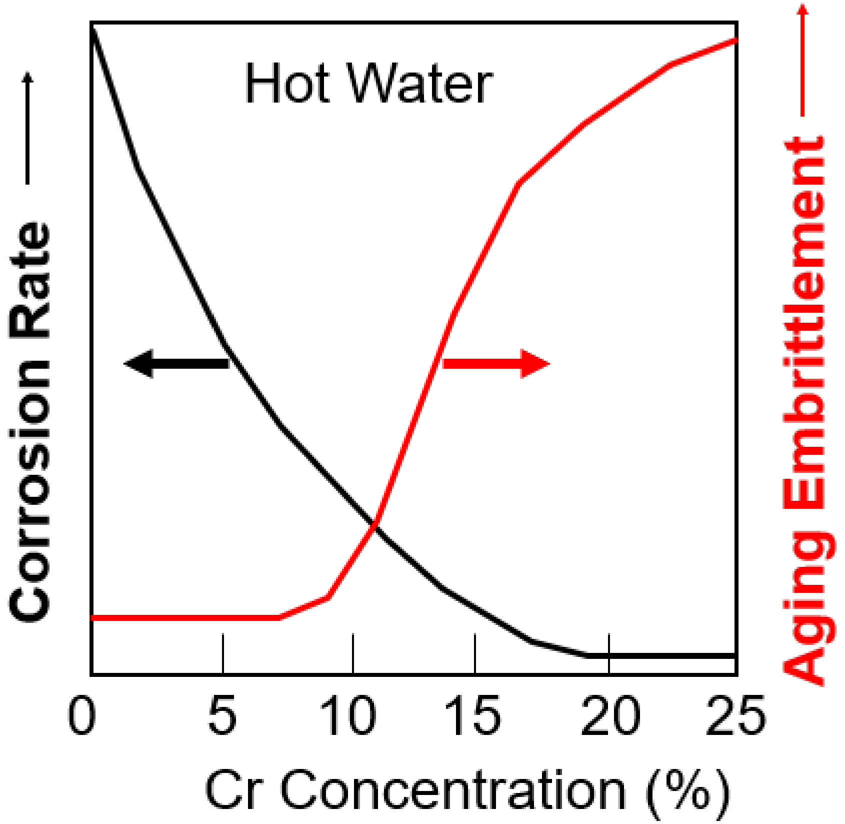


Fig. 8. (a) Diameter, d_c, number density, N_c of cavities; and (b) $(d_c \times N_c)^{0.5}$ for the as-implanted at 300 °C and the PIA; (c) The hardening before and after He-implantation at 300 °C and the hardness difference between the specimens after PIA and the un-implanted but thermally aged at 800 °C/100 h.

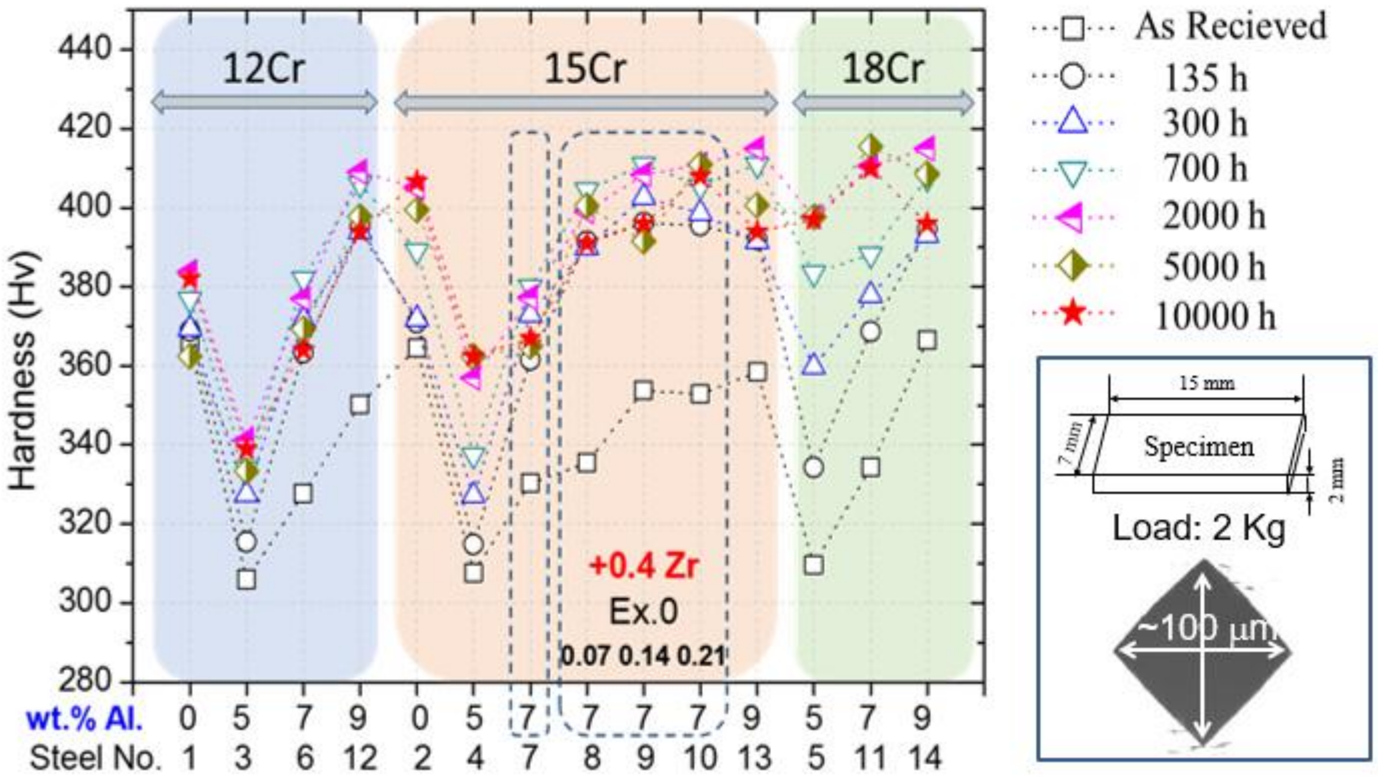
Song, 2018, NME

3) Aging

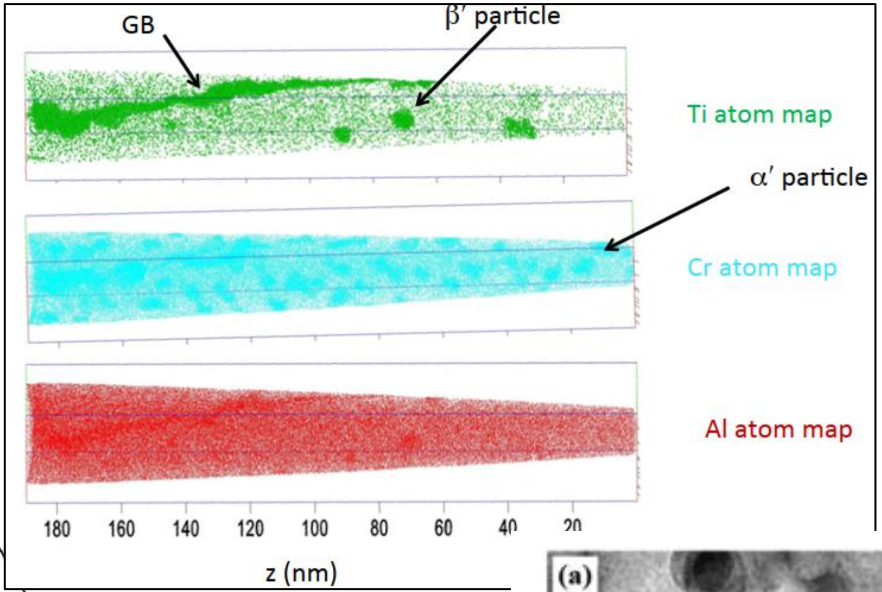
Tradeoff Issue!!

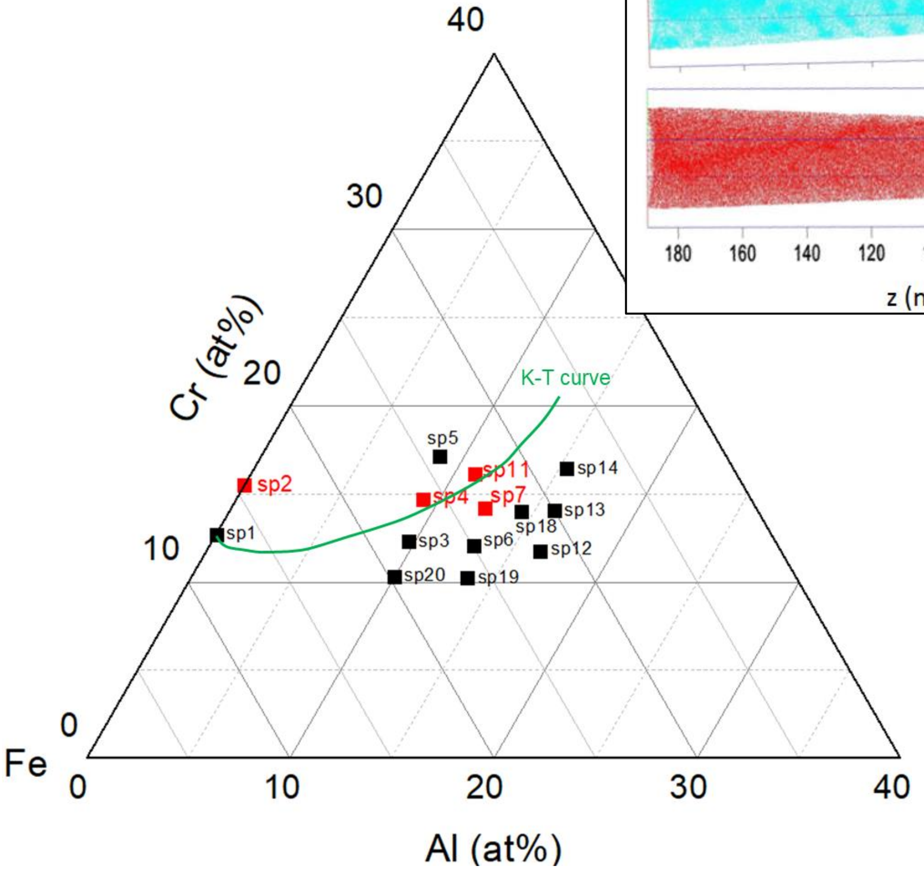


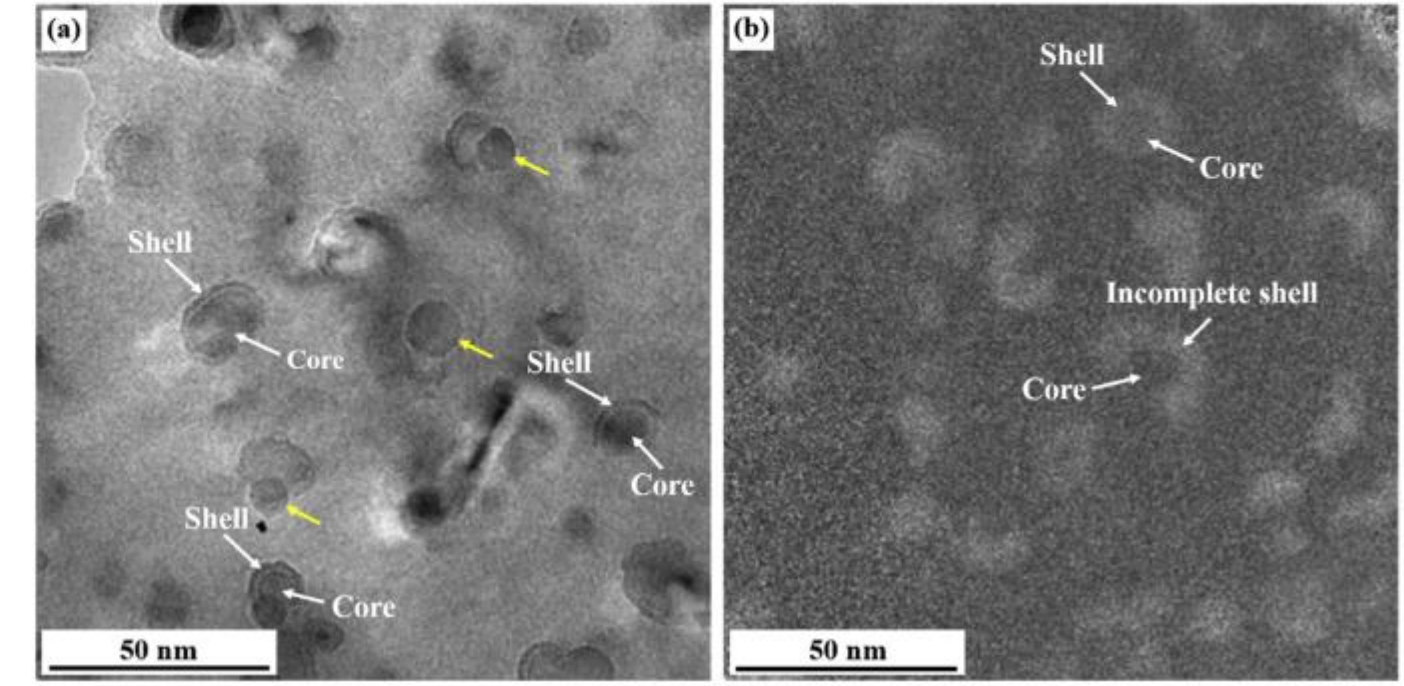
Vickers Hardness Measurements



475°C aging


GB
β′ particle
Ti atom map
α′ particle
Cr atom map
Al atom map
180
160
140
120
100
80
60
40
20
z (nm)


40
30
20
10
0
Cr (at%)
Fe
0
10
20
30
40
Al (at%)
K-T curve
sp1
sp2
sp3
sp4
sp5
sp6
sp7
sp11
sp12
sp13
sp14
sp18
sp19
sp20


(a)
Shell
Core
Shell
Core
Shell
Core
50 nm
(b)
Shell
Core
Incomplete shell
Core
50 nm

Simulated tubing process and aging

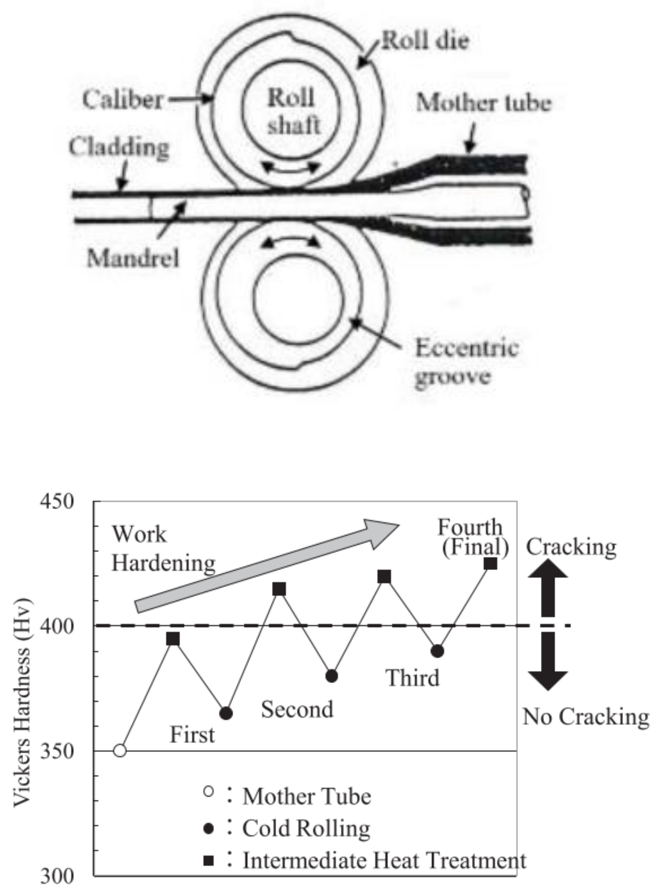
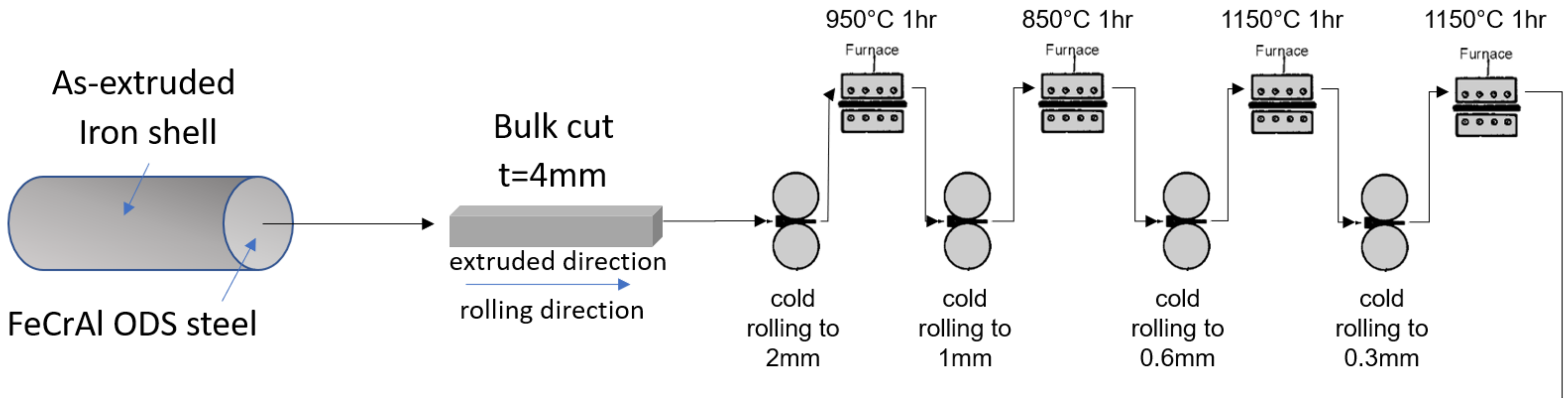
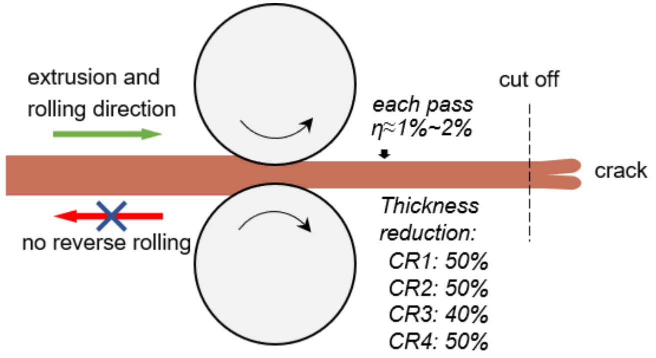
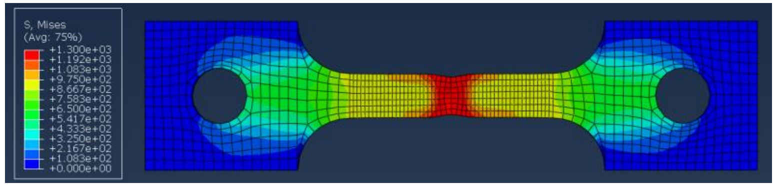
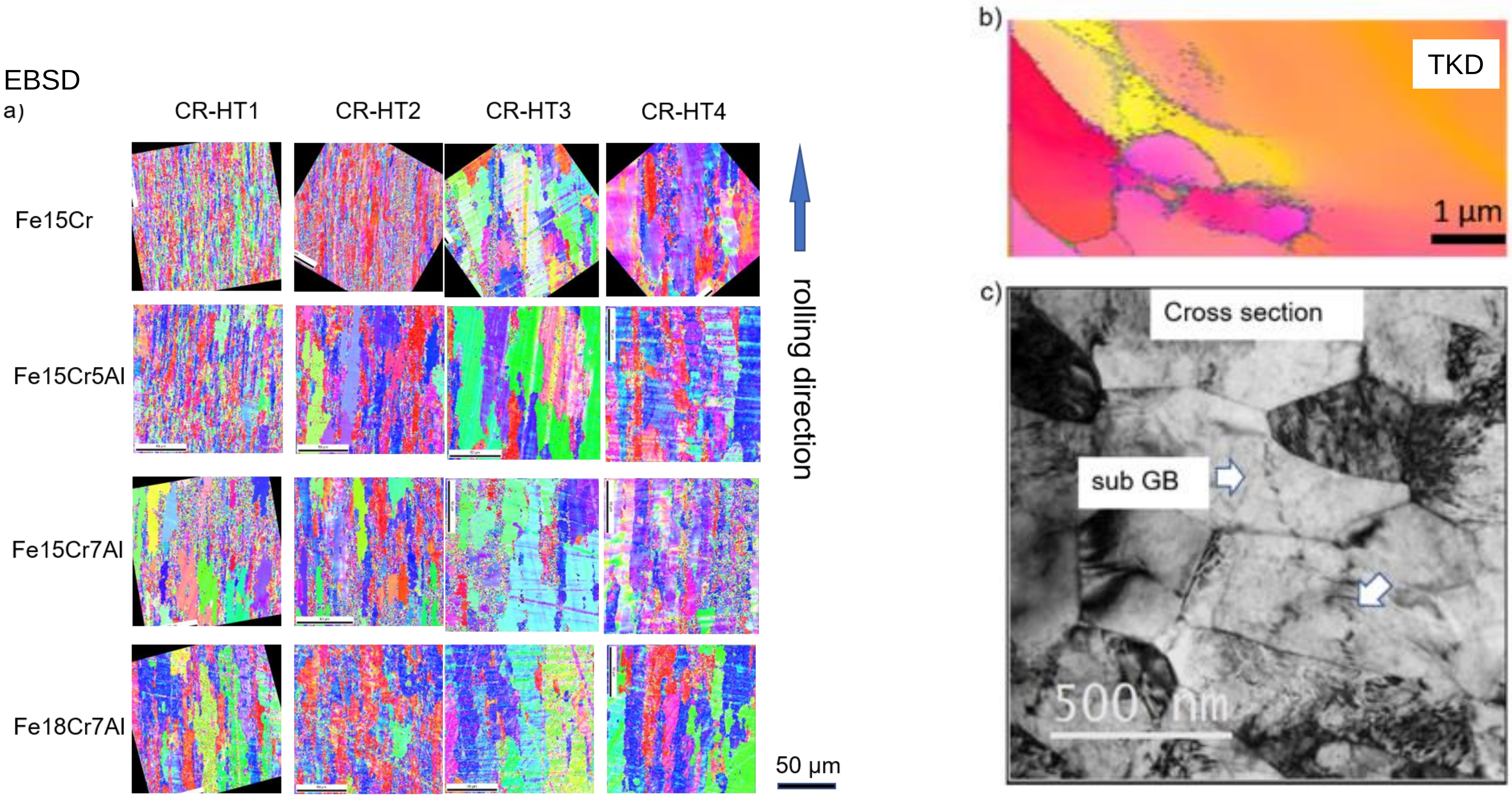


Figure 2. Typical increase in hardness during cold-rolling of 12Cr ODS ferritic steel cladding.

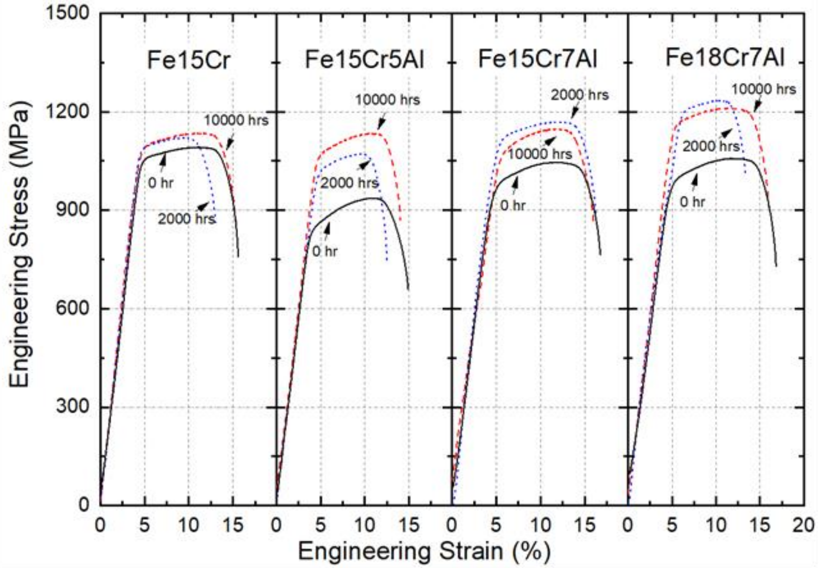


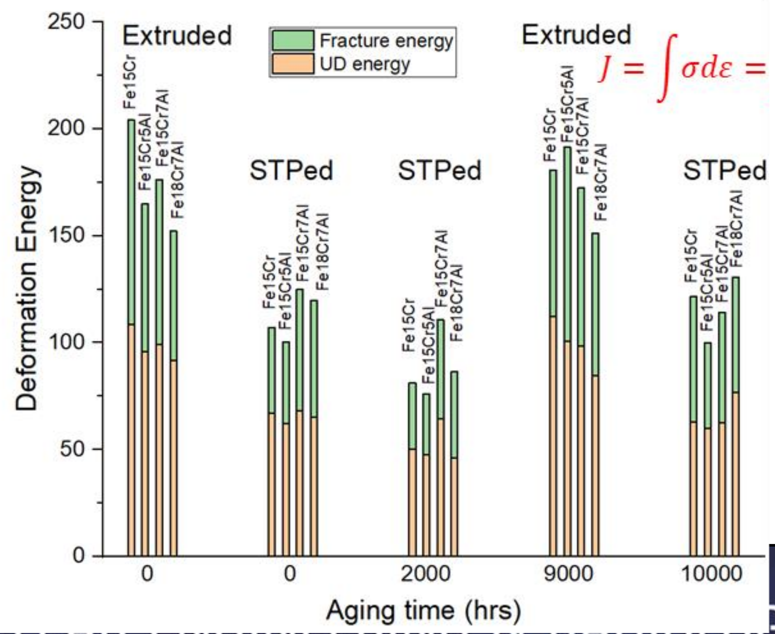




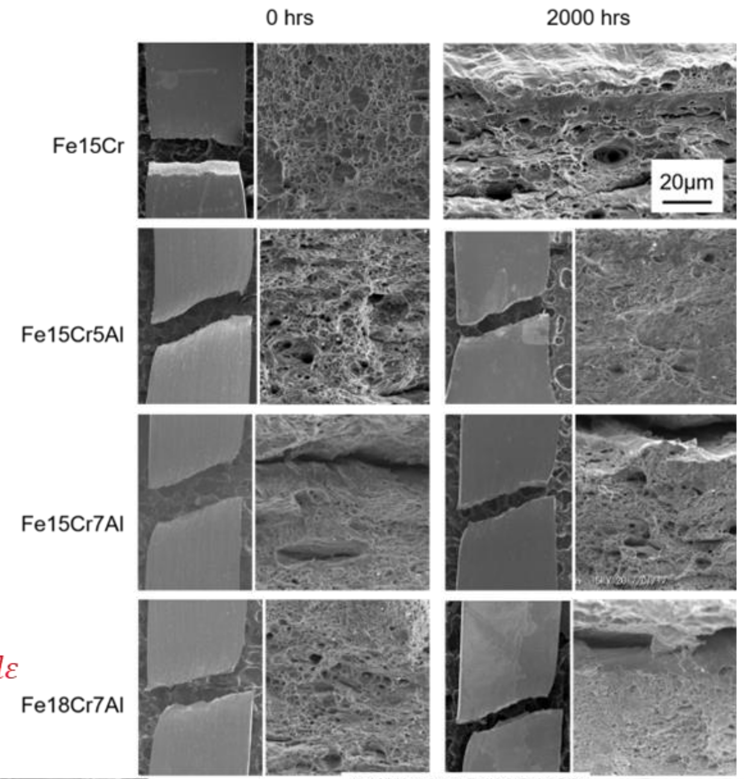

EBSD
a)
CR-HT1
CR-HT2
CR-HT3
CR-HT4
Fe15Cr
Fe15Cr5Al
Fe15Cr7Al
Fe18Cr7Al
rolling direction
50 μm
b)
TKD
1 μm
c)
Cross section
sub GB
500 nm

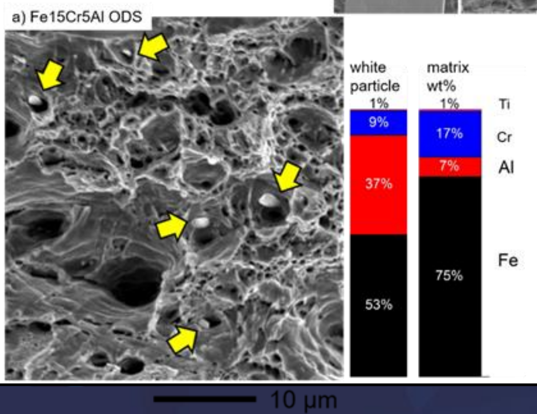
Tensile tests

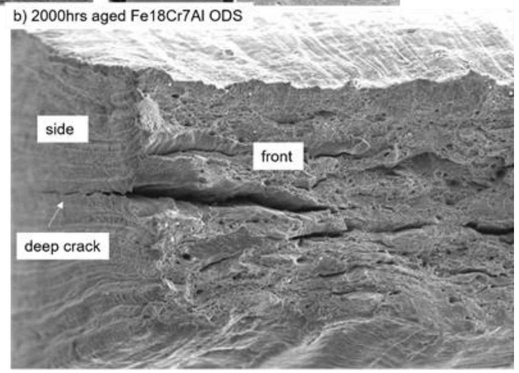


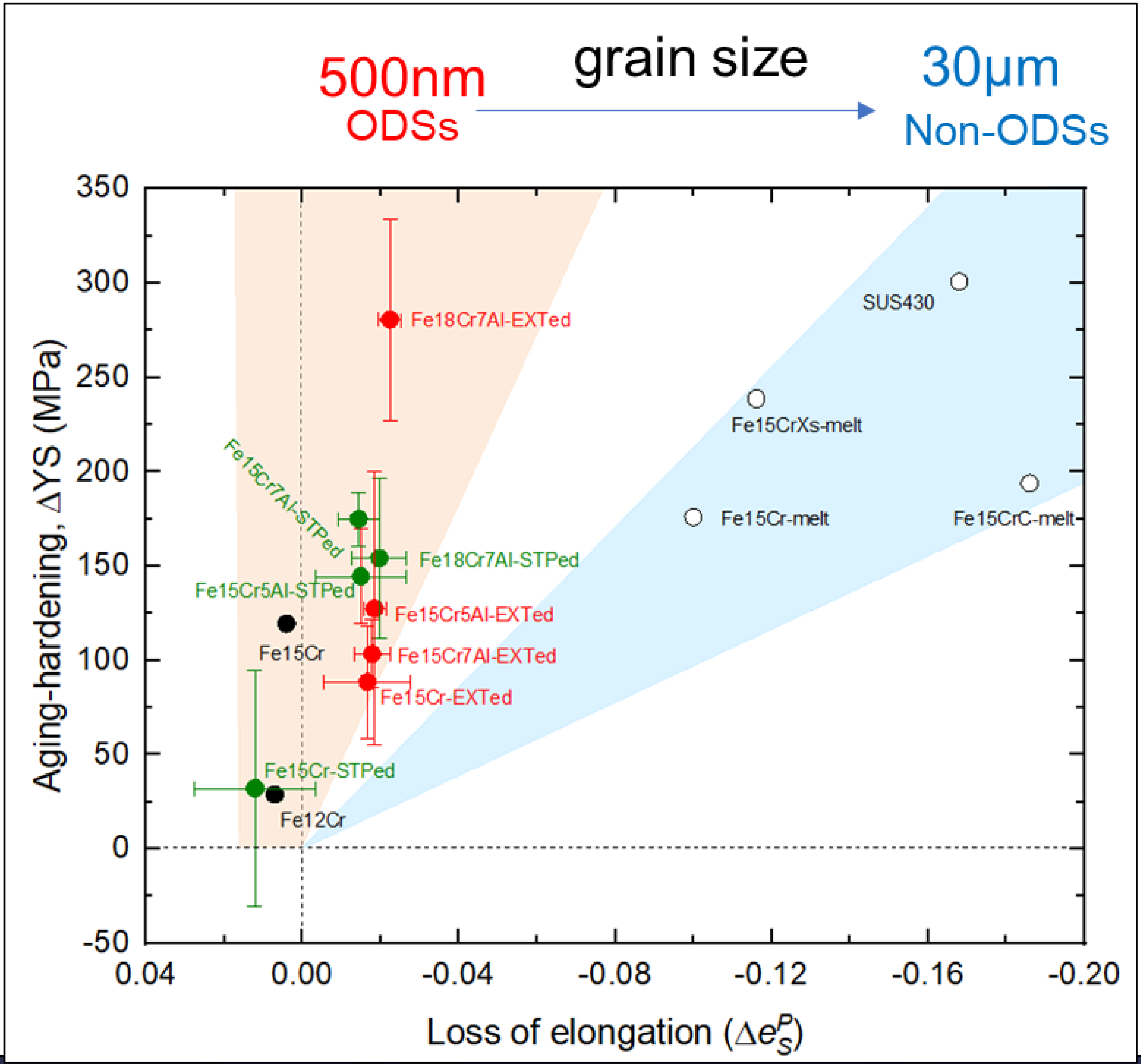


$$J = \int \sigma d\varepsilon = \int_0^{UE} \sigma d\varepsilon + \int_{UE}^{TE} \sigma d\varepsilon$$



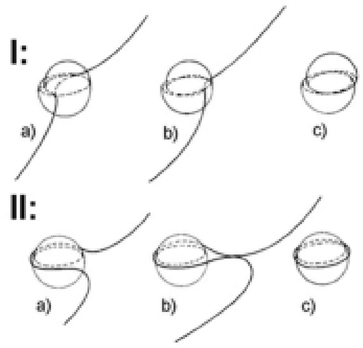






Excellent resistance of aging embrittlement

Aging hardening



I Cut-through:

$$\Delta\sigma_{chs} = \psi(12/\pi)^{\frac{1}{2}}\gamma_s^{\frac{3}{2}}(f/Gb)^{\frac{1}{2}}(1/r)$$ Chemical strength

$$\Delta\sigma_{cohs} = \psi\alpha(\varepsilon G)^{\frac{3}{2}}(2rf/Gb)^{\frac{1}{2}}$$ coherent strength

$$\Delta\sigma_{ms} = 0.0055\psi(\Delta G)^{\frac{3}{2}}(2f/G)^{\frac{1}{2}}(r/b)^{-1+\frac{3m}{2}}$$ modulus strength ← dominant

$$\Delta\sigma_{\alpha'}\text{ (predicted)} = \Delta\sigma_{chs} + \Delta\sigma_{cohs} + \Delta\sigma_{ms}$$ Linear summation

$$r = K_r t^{1/3}$$

II Bow-bypass: Orowan

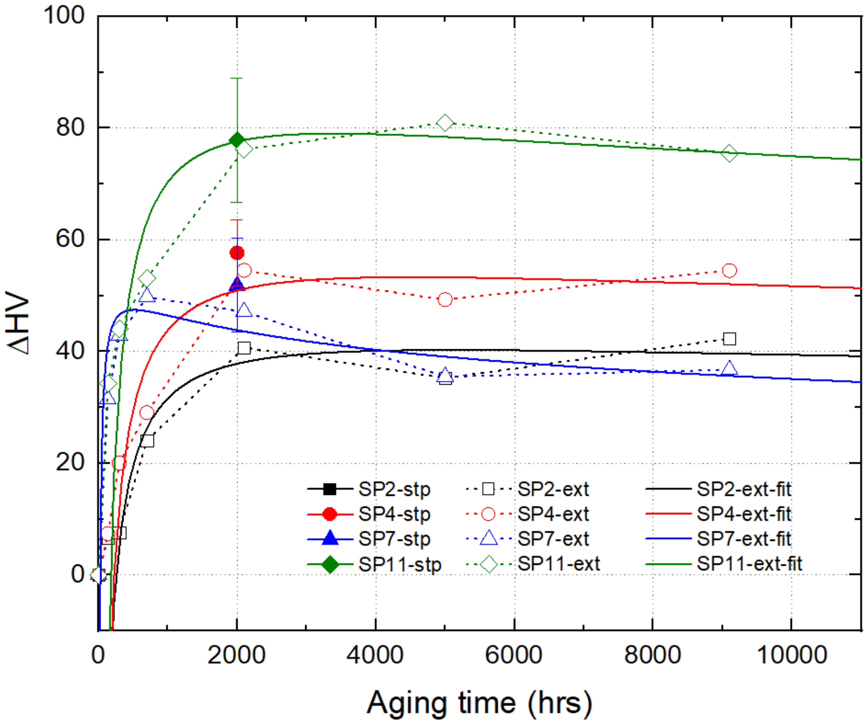
$$\Delta\sigma_{\beta'} = 0.1Gb\frac{f^{1/2}}{r}\ln\left(\frac{r}{b}\right)$$

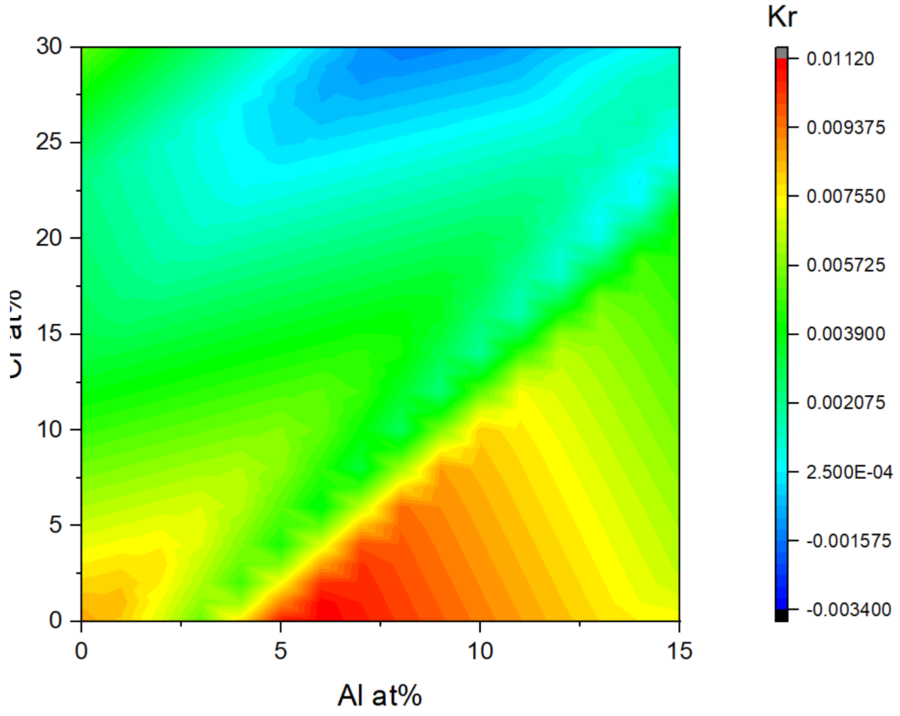
$$\Delta\sigma = 0.1Gb\frac{f^{1/2}}{K_r}t^{-1/3}\ln\frac{K_r t^{1/3}}{b}$$

$$\boxed{\Delta\sigma \cdot t^{\frac{1}{3}} = a + k\ln t}$$

$$\dot{\Delta\sigma} \cdot t^{\frac{1}{3}} + \Delta\sigma \cdot \frac{1}{3}t^{-\frac{2}{3}} = \frac{k}{t}$$

$$\boxed{t_{max} = exp(3 - a/k)}$$





Summary

FeCrAl ODS steels

- Ion-irradiation embrittlement — Irradiation hardening, oxide stability

  Temperature dependence, Cr-Al dependence, Ti,Al,Zr oxide

- Helium effect — Swelling, hardening

  Smaller swelling due to grain boundary and oxide surfaces

- Aging embrittlement — precipitates

  Smaller loss of ductility compared to non-ODS steels

- Corrosion — oxidation

  Super excellent corrosion resistance