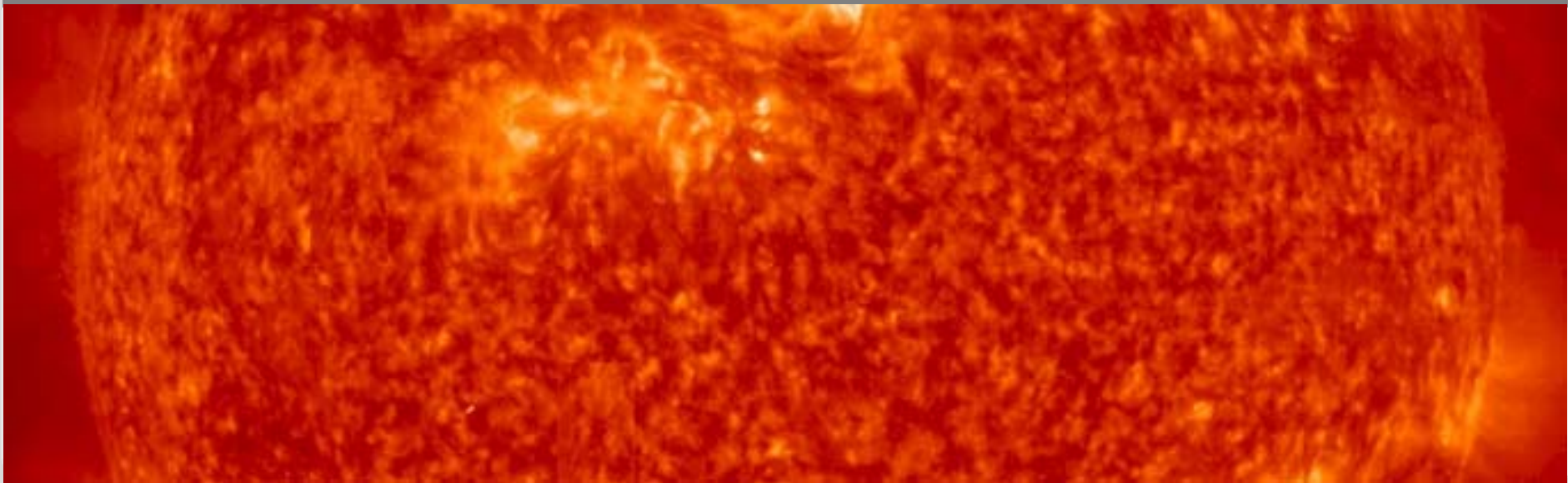


Material Constraints due to neutron irradiation and degradation

Anton Möslang

Institute for Applied Materials

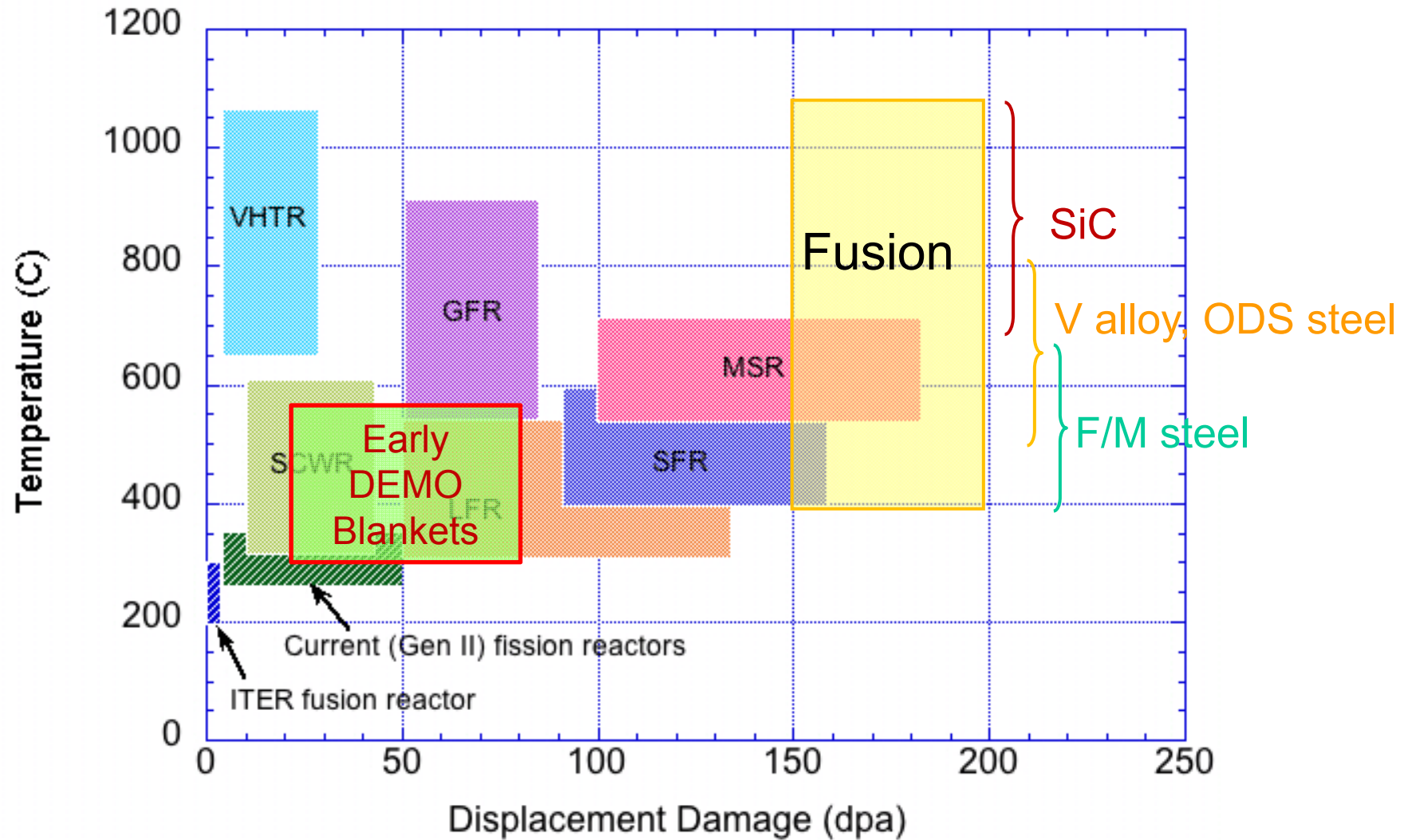


Outline

- Materials challenges: fusion – fission – spallation
- Neutron irradiation: Examples of progress and issues
 - Reduced activation ferritic/martensitic steels
 - Oxide dispersion strengthened steels
 - W alloys: I-1, I-3, O-1, O-2: Yesterday
I-7, O-5, O-6, I-8: Today
- Technical Readiness and database maturity
- Role of materials in fusion roadmaps,
Need for fusion specific irradiation source

Gen IV and Fusion reactors pose severe materials challenges

S.J. Zinkle & J.T. Busby, Mater. Today 12 (2009) 12



Requirements for “in vessel” structural materials



Fission – Fusion – Spallation: Three different irradiation loadings

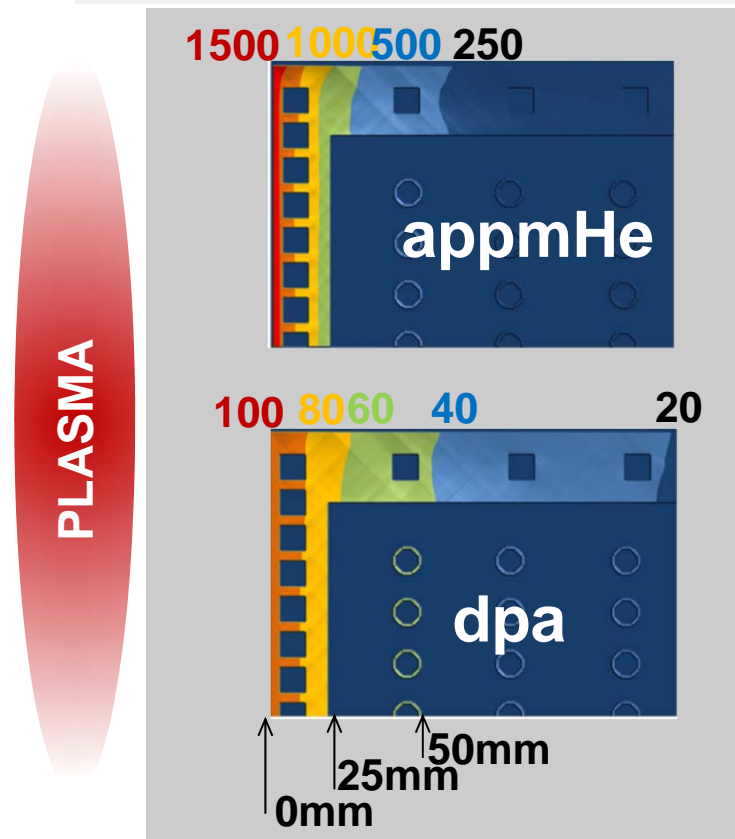
	Fission (Gen. I)	Fission (Gen. IV)	Fusion (DEMO-PROTO)	Spallation (MYRRHA)
Structural alloy T_{\max}	<300°C	500-1000°C	550-1000°C	400-600°C
Max dose for core internal structures	~1 dpa	~30-150 dpa	~50-150 dpa	≤60 dpa/fpy
Max transmutation helium concentration	~0.1 appm	~3-10 appm	~500-1500 appm (~8 times more for SiC)	~2000 appm/fpy
Particle Energy E_{\max}	<1-2 MeV	<1-3 MeV	<14 MeV	several hundred MeV

Materials R&D towards:

- improved irradiation resistance
- enhanced temperature window
- convincing compatibility with coolants

Displacement damage and He production in Blankets

Helium production (appm) for
100 dpa at plasma facing side



H. Tanigawa, E.Wakai 2012

- ❑ “Only” the first few centimeters have a high He/dpa ratio
- ❑ In addition this part of the blanket carries the highest thermo-mechanical loads
- ❑ Therefore,
 - fission reactor irradiations are still meaningful for a significant fraction of in-vessel components
 - Nevertheless, a dedicated fusion neutron source is indispensable, but has to focus on plasma-near materials and loading conditions

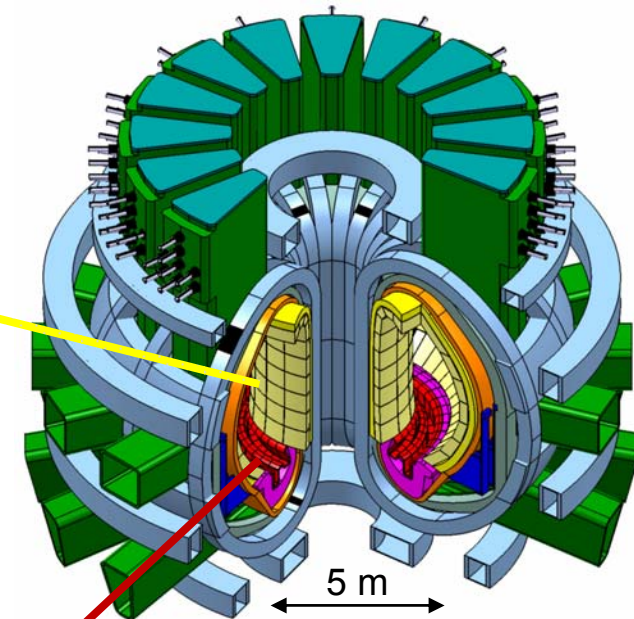
Fusion Power Plants: Structural Material Challenges

Blanket: ≤ 30 dpa/yr, ≤ 2.5 MW/m²

- Plasma Facing Materials
- Reduced Activation Structural Materials:
 - RAFM Steels (EUROFER, F82H) 350-550 °C
 - RAFM ODS Steels 300-650 °C
- Functional Materials
 - Neutron Multipliers (Be), Li ceramics

Divertor: ≤ 10 dpa/yr, 10-15 MW/m²

- Refractory alloys (e.g. W-materials)
 - 850-1100 °C → ~600 - 1300 °C
- Nano-scaled RAF(M)-ODS Steels
 - 350-650 °C → ~300 - 800 °C



- Materials challenges: fusion – fission – spallation
- **Neutron irradiation: Examples of progress and issues**
 - **Reduced activation ferritic/martensitic steels**
 - Oxide dispersion strengthened steels
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Need for fusion specific irradiation source

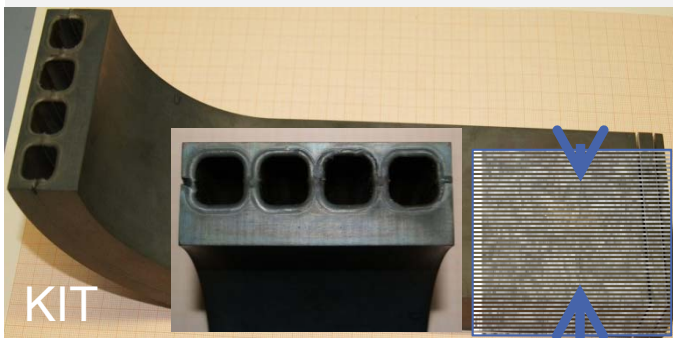
Recent past and present:

Qualification of RAFM steels EUROFER & F82H mod

Eurofer Blanket Module



ODS steel, diffusion welding



Main Achievements

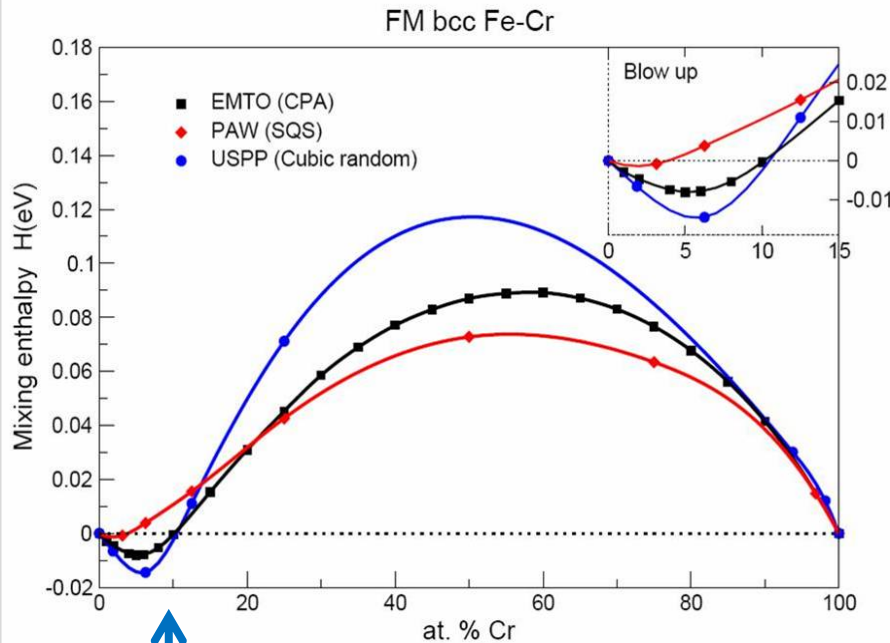
- ❑ Meanwhile ~35 tons of EUROFER & ~50 tons of F82H mod delivered
- ❑ Broad based Qualification Programme, including joining technologies and corrosion
- ❑ Fission neutron based materials data up to ~70 dpa
- ❑ Implementation of EUROFER database into the RCC-MRx code for ITER Test Blanket Modules ongoing

Future Mission

- ❑ Increase **upper operation temperature** and improve **neutron/He/H** embrittlement
- ❑ by fine tuning of alloying elements

Why RAFM steels are based on 9wt% Cr?

S. Dudarev et al, UKEA, 2006



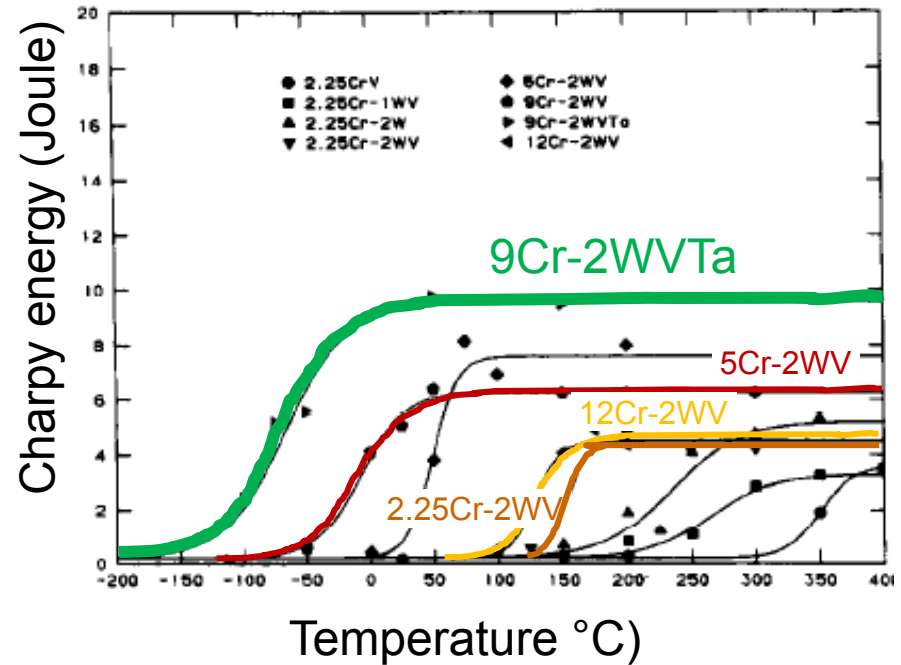
9-10%Cr

is favorable

Above 10% Cr: formation of Cr precipitates (σ -phase, α')

R. Klueh et al, J. Nucl. Mat. (1996) 336

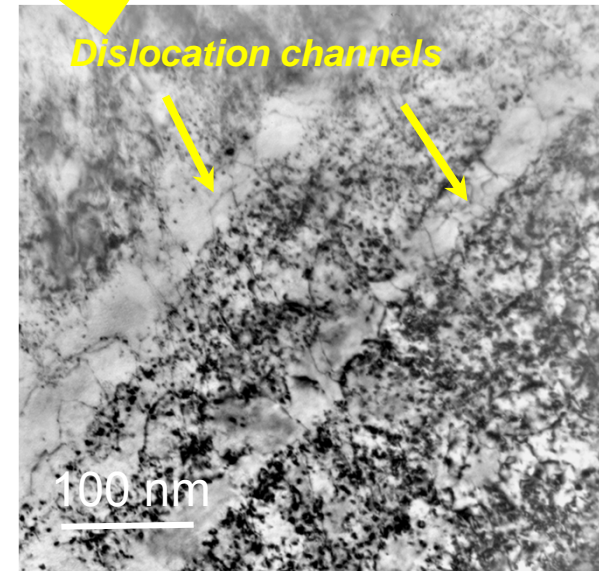
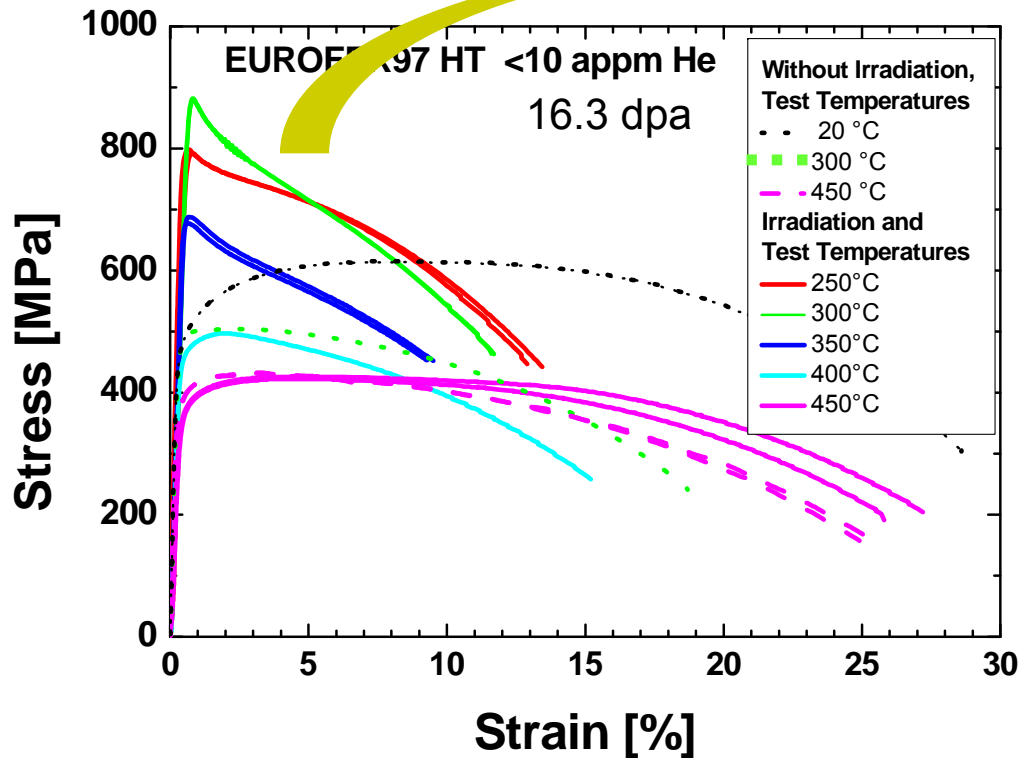
Neutron irradi., 20-24 dpa, $T_{irr} = 365 \text{ }^\circ\text{C}$



Experimentally verified:

9Cr-(1-2)WV-Ta steels have superior aging and irradiation properties

RAFM 8-10%CrWVTa-Steels without Helium: Tensile results after neutron irradiation

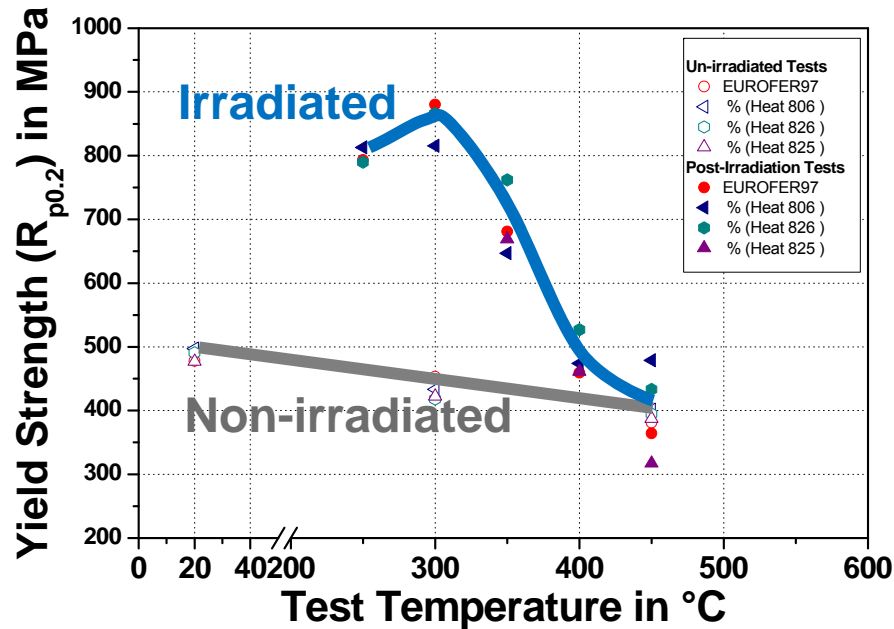


E. Materna-Morris, et al. JNM 386(2009) 422

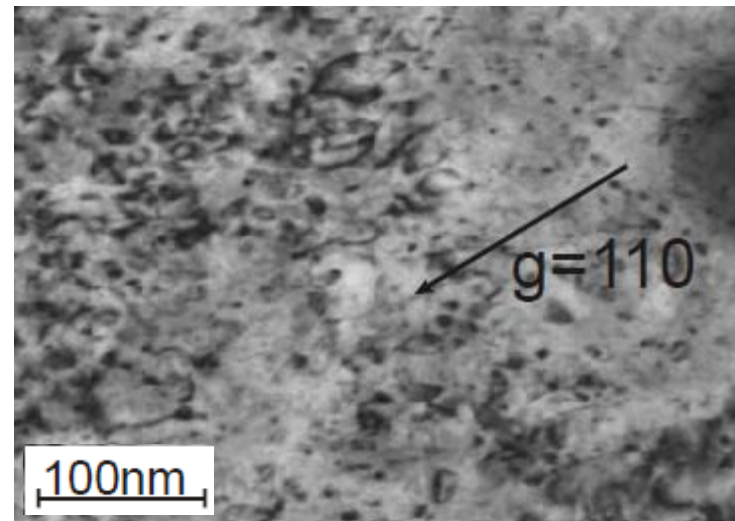
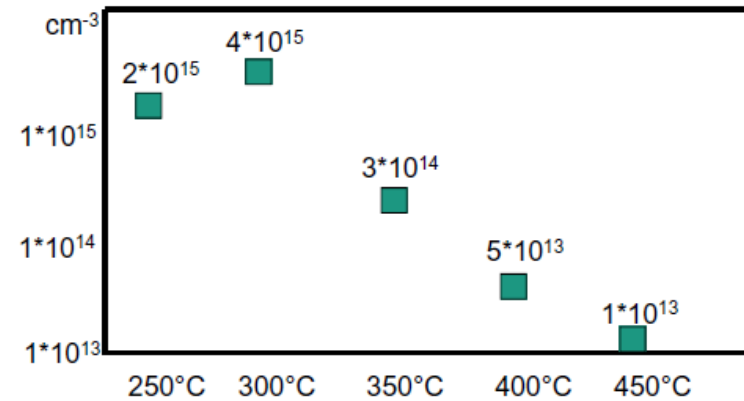
N. Hashimoto et al., Fus.Sci.Tech. 44 (2003)

- Below ~400 °C: - Strain localization due to dislocation channeling
 - Despite the very small uniform elongation rupture stress still high

Substantial irradiation induced hardening below $T_{irr} \sim 420^\circ\text{C}$ by interstitial type defects



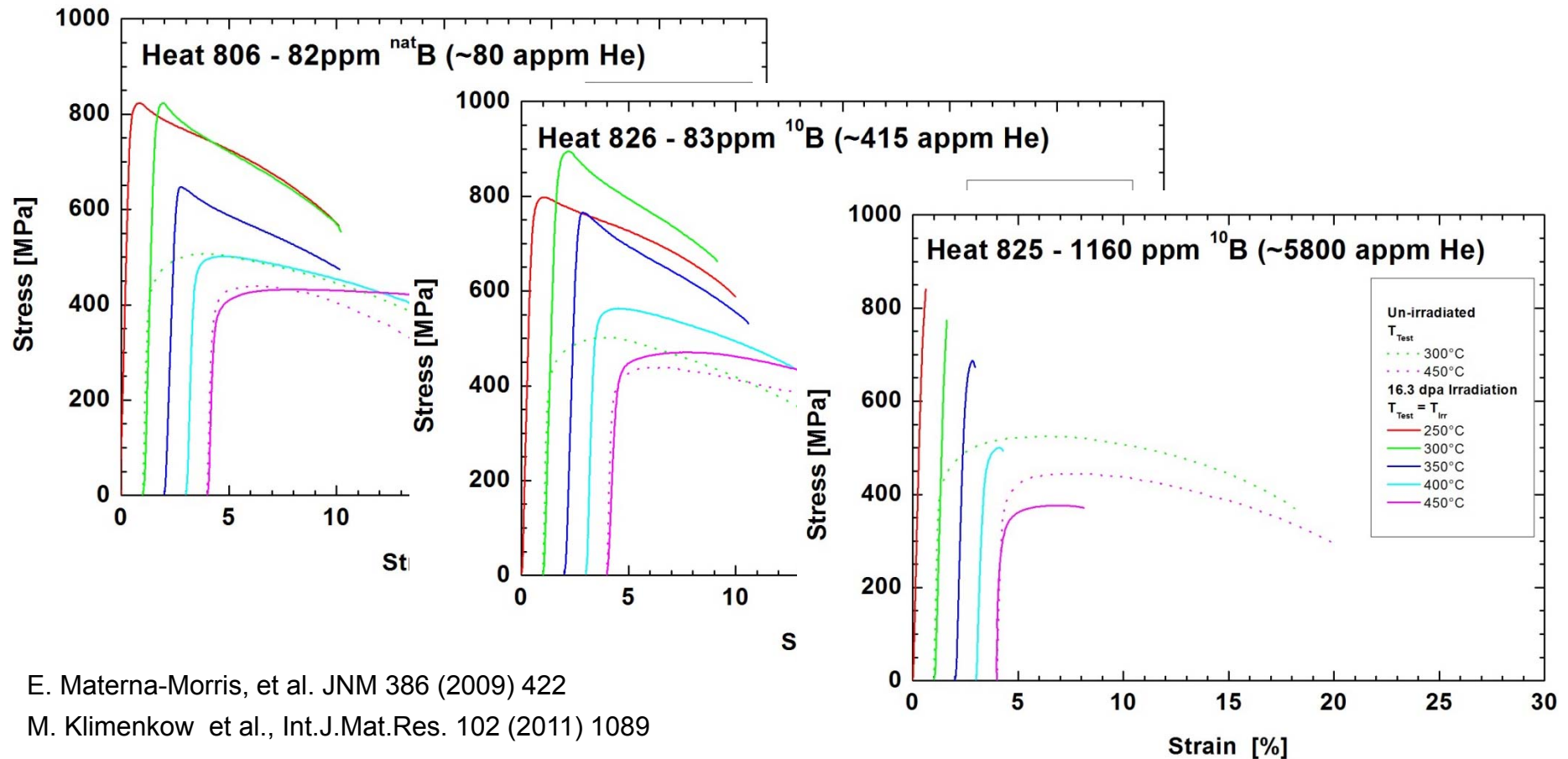
Concentration of irradiation induced defects



$T_{irr} = 300^\circ\text{C}$:

- Homogeneous distribution of point defects and dislocation loops
- Dislocation loops:
 - $\frac{1}{2}\langle 111 \rangle$ Burgers vector
 - 5-25nm diameter

Ductile or brittle? The importance of strain rate $\dot{\epsilon}$: Example: Eurofer, 16 dpa, B-doped, $\dot{\epsilon} \approx 10^{-3} \text{s}^{-1}$



E. Materna-Morris, et al. JNM 386 (2009) 422

M. Klimenkow et al., Int.J.Mat.Res. 102 (2011) 1089

- ≤ 415 appm He: Almost no effect on tensile properties at small strain rates
- 5800 appm He: Entirely brittle fracture; total loss of plasticity

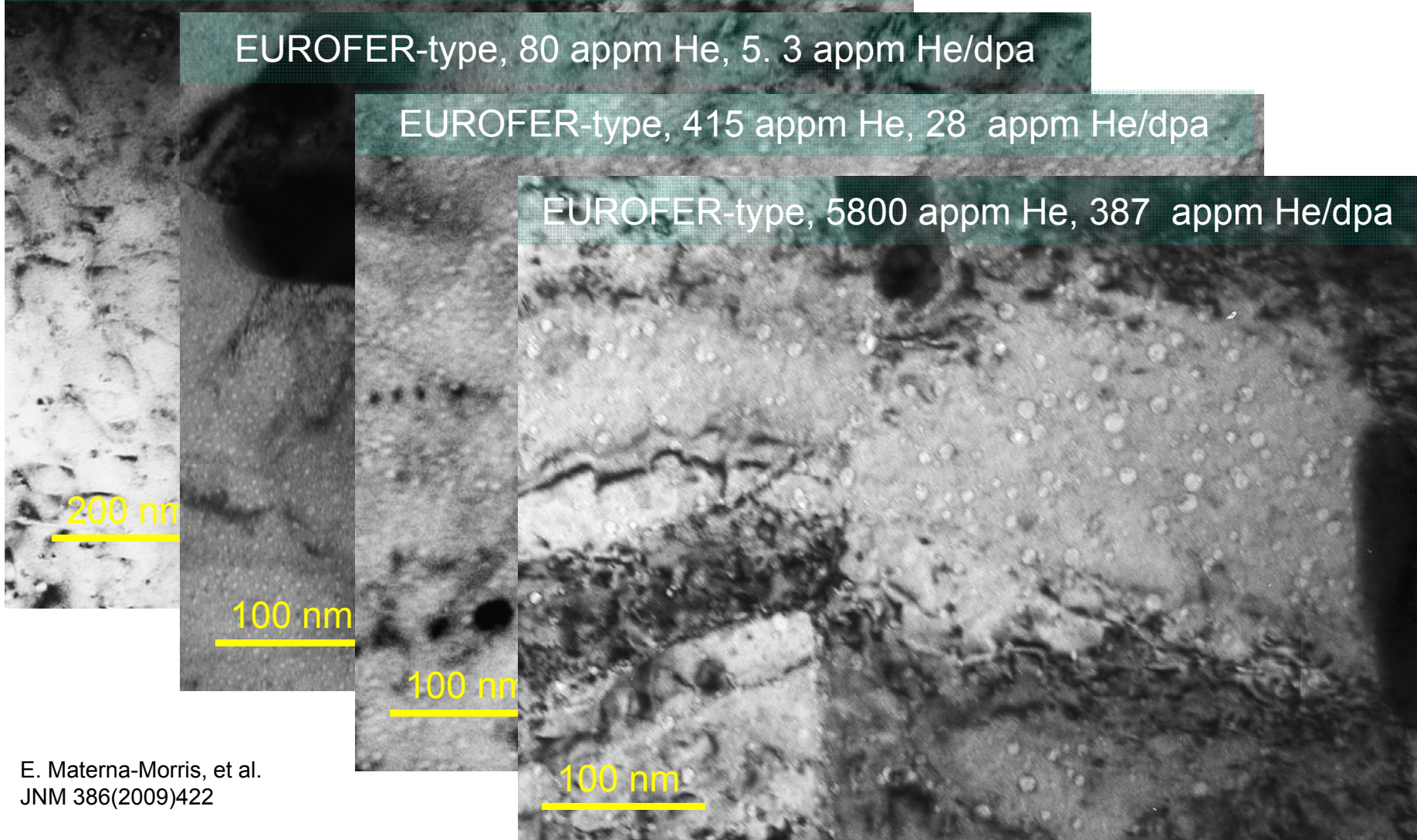
EUROFER, 15 dpa neutron irradiation at 250 °C

EUROFER, <10 appm He, <1 appm He/dpa

EUROFER-type, 80 appm He, 5.3 appm He/dpa

EUROFER-type, 415 appm He, 28 appm He/dpa

EUROFER-type, 5800 appm He, 387 appm He/dpa

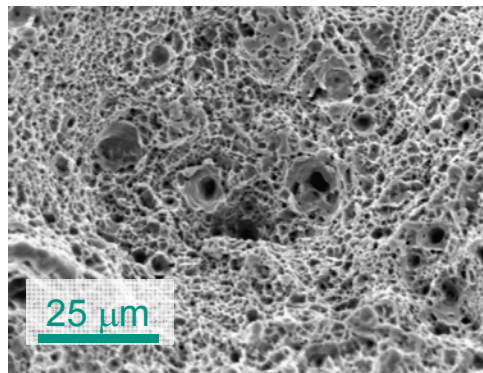
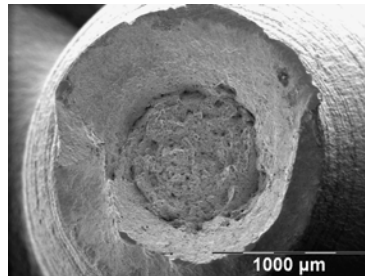


E. Materna-Morris, et al.
JNM 386(2009)422

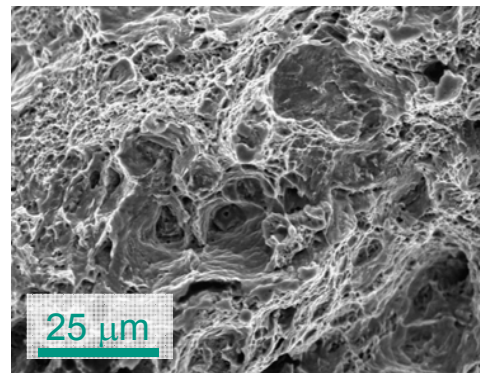
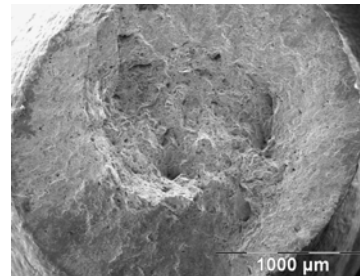
Fracture behavior of tensile tested EUROFER

16 dpa, $T_{irr} = T_{test} = 300 \text{ }^\circ\text{C}$

EUROFER97, <10 appm He



EUROFER-type, 415 appm He

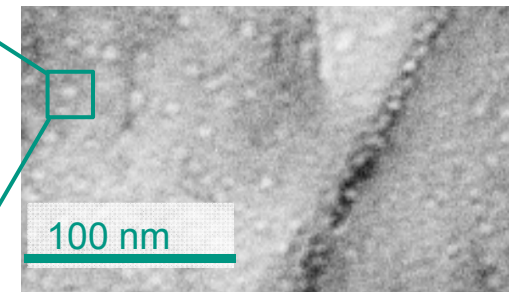
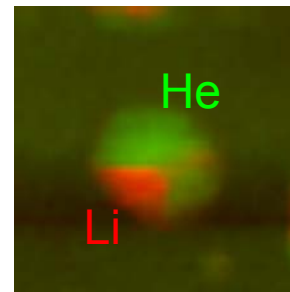


$n + {}^{10}\text{B} \rightarrow {}^4\text{He} + {}^7\text{Li} + 2.7895 \text{ MeV}$
 range of He (1.0 MeV): 1.6 μm
 range of Li (1.8 MeV): 2.0 μm



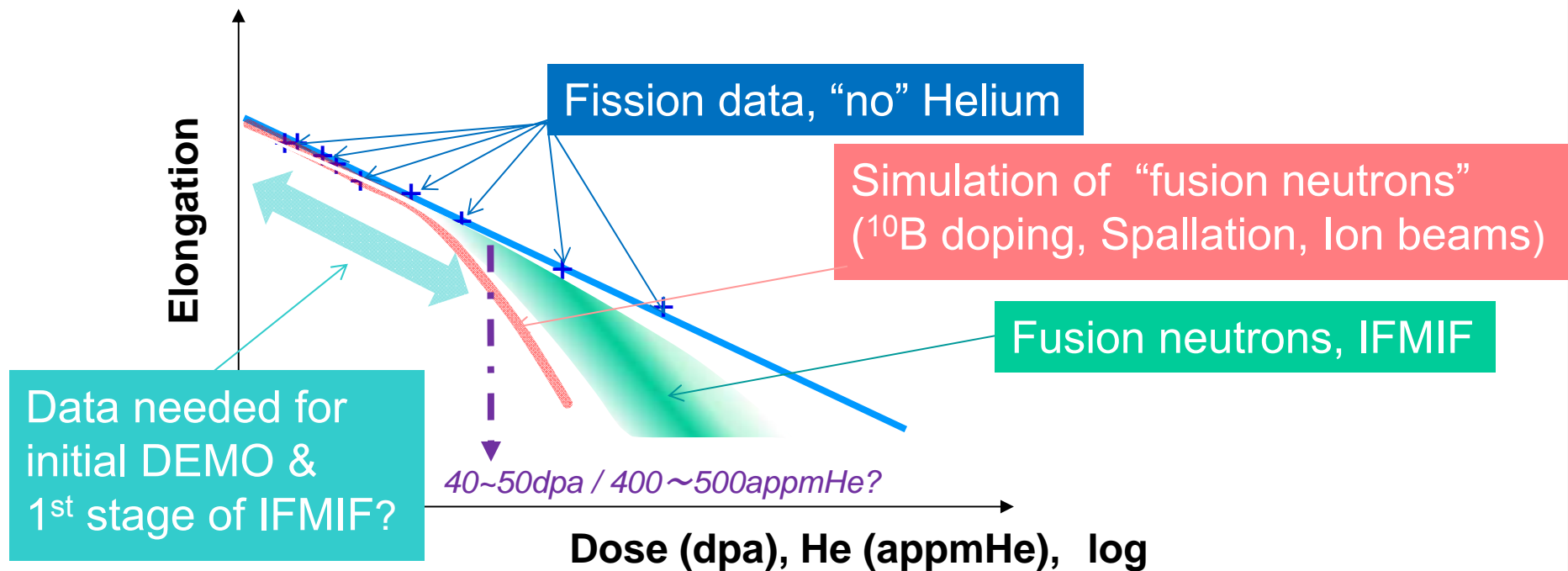
E. Materna-Morris et al., JNM 386-388 (2009) 422-425
 M. Klimenkov et al., Micron 46 (2013) 51-56

■ ${}^{10}\text{B}$ -doping: He and Li effects cannot really be decoupled



Possible steps towards a fusion design code: Tensile properties as example

In the style of H. Tanigawa, E. Wakai

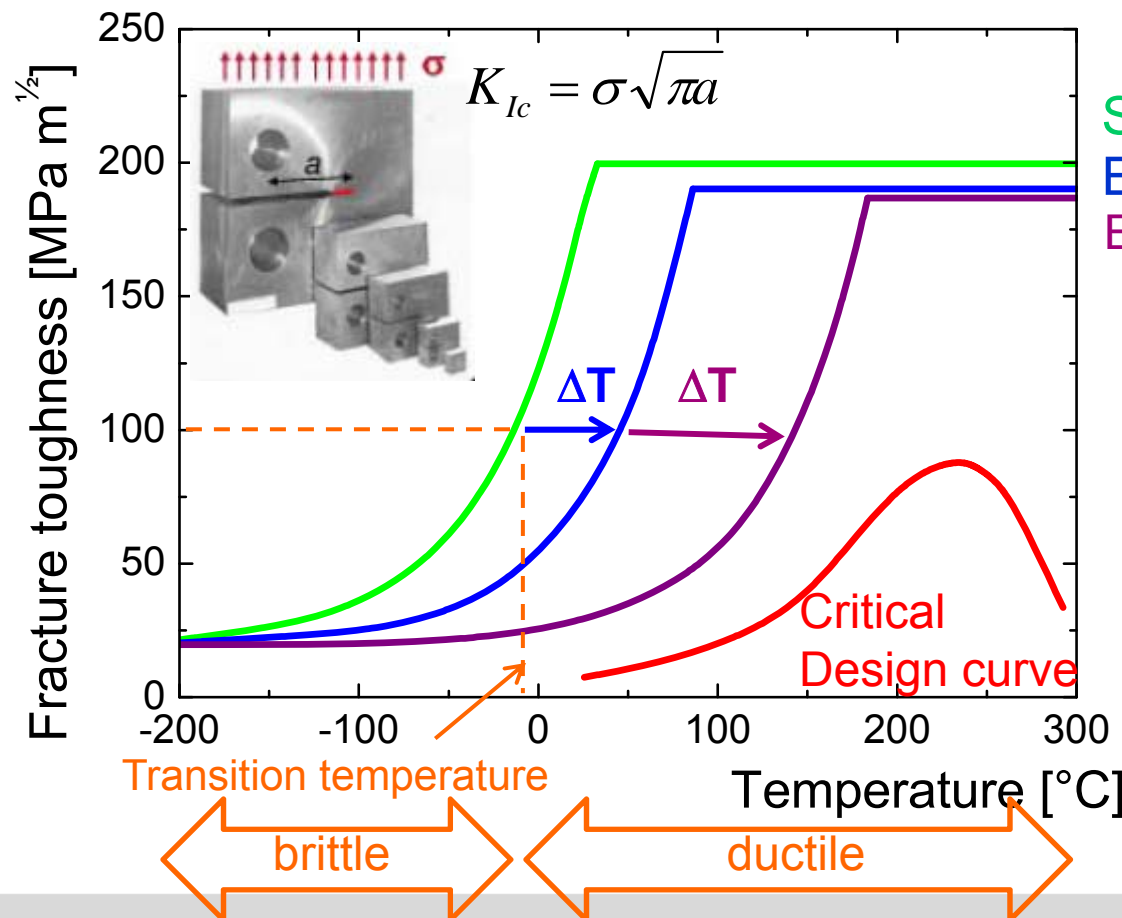


- Small strain rate: Critical condition 40~50 dpa / 400~500 appm He?
- This might be also the parameter window for initial DEMO and 1st stage of IFMIF and related design code development

Ductile or brittle?

Indispensable for safety, economy & life-time prediction

The accurate prediction of the ductile-to-brittle-transition temperature shift is fundamental for ensuring the structural integrity of reactor pressure vessels (Fission) and of blanket/divertor (Fusion)



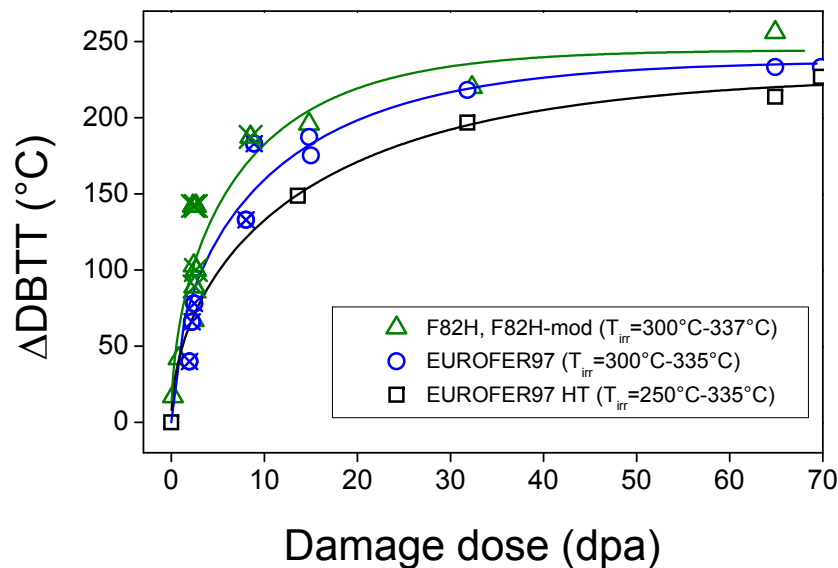
Start of life
End of life, predicted
End of life, 40 years, real?

Fracture toughness specimens are indispensable in all test matrixes

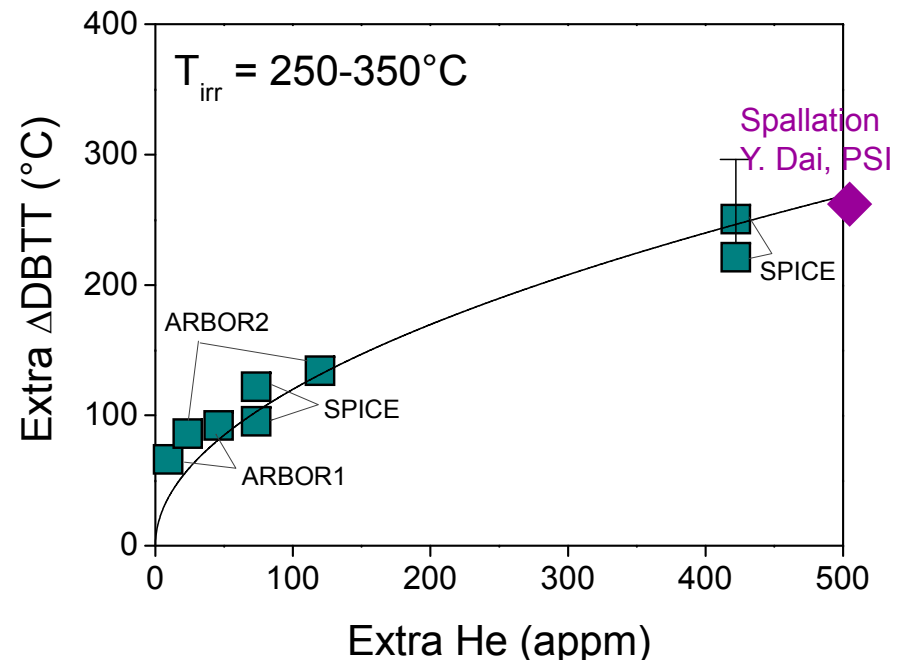
Ductile or brittle? The importance of strain rate $\dot{\epsilon}$: Example: Eurofer, 16 dpa, B-doped, $\dot{\epsilon} \approx 10^2 \text{s}^{-1}$

E. Gaganidze et al., J. Nucl. Mater. 417 (2011)93-98

EUROFER, <10 appm He

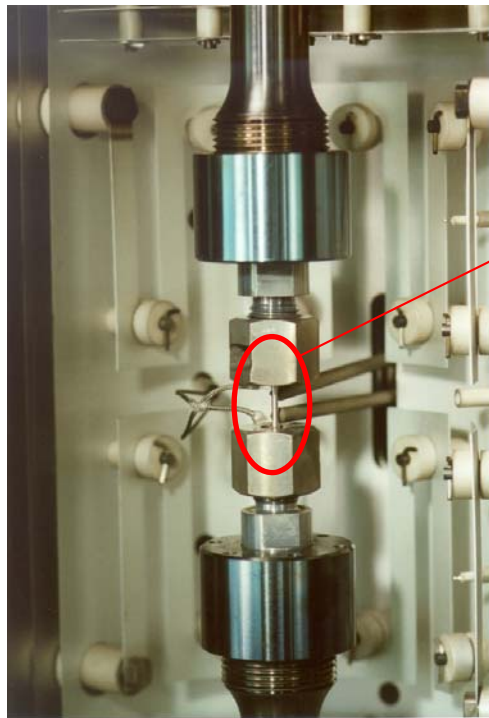
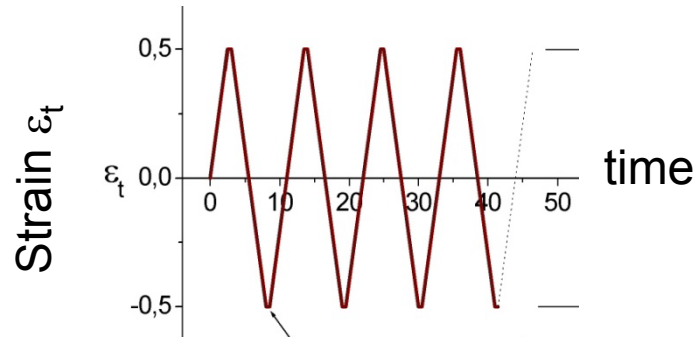


EUROFER, 10-500 appm He

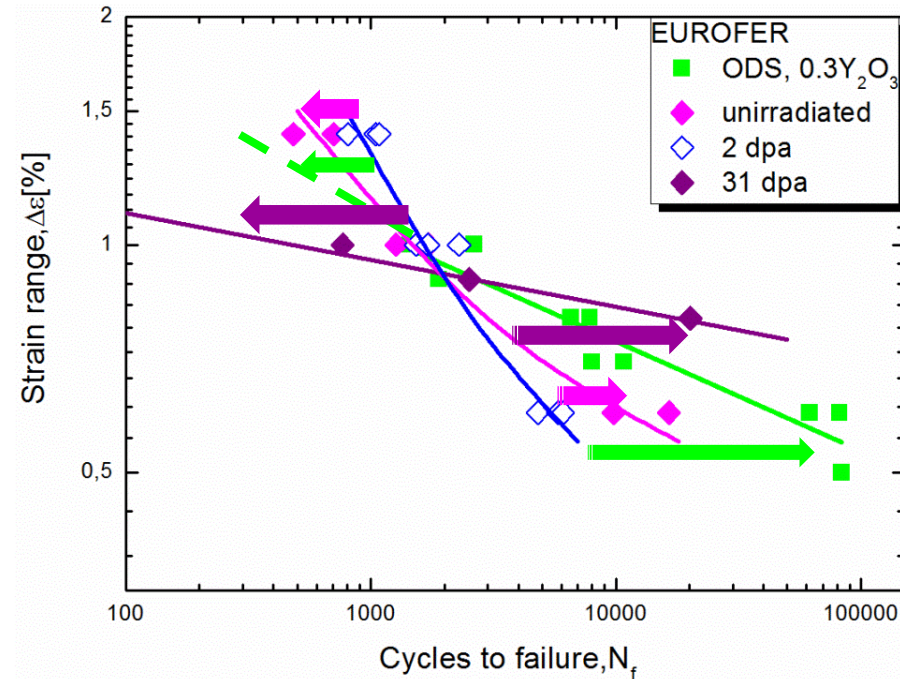


- ❑ High strain rates: Helium effects are an outstanding issue; Saturation??
 - ➡ He determines the lower operation temperature in DEMO blankets (~350°C).
- ❑ B-doping & Spallation neutrons are too aggressive
 - ➡ intense fusion n-source indispensable

Fatigue: Irradiation can lead to shorter or longer lifetime



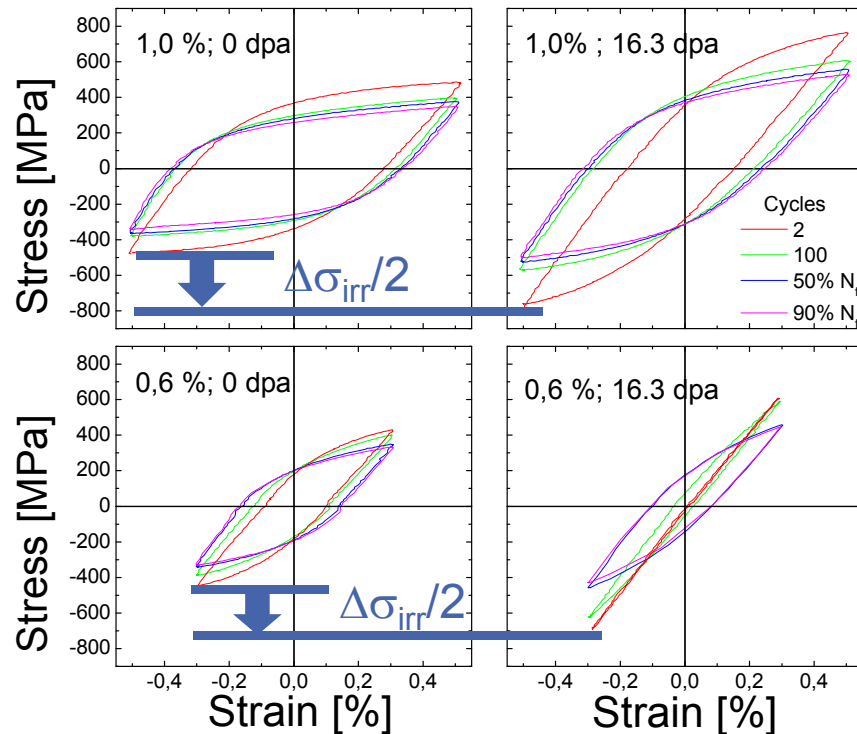
specimen



- high strain regime, shorter lifetime
→ accelerated crack initiation
- low strain regime, prolonged lifetime
→ Micro-crack growth impeded

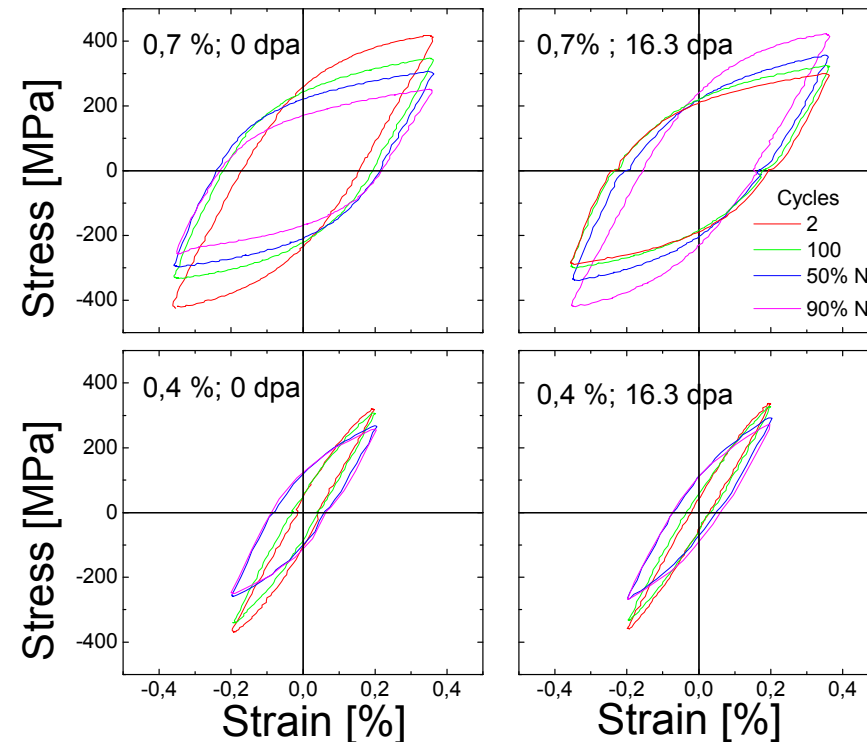
$\Delta\varepsilon$ controlled LCF after 16.3 dpa irradiation

$$T_{\text{irrad.}} = T_{\text{test}} = 250 \text{ }^\circ\text{C}$$



- Significant irradiation induced hardening $\Delta\sigma_{\text{irr}}$ at all $\Delta\varepsilon$
- Cyclic softening somewhat higher after irradiation

$$T_{\text{irrad.}} = T_{\text{test}} = 450 \text{ }^\circ\text{C}$$



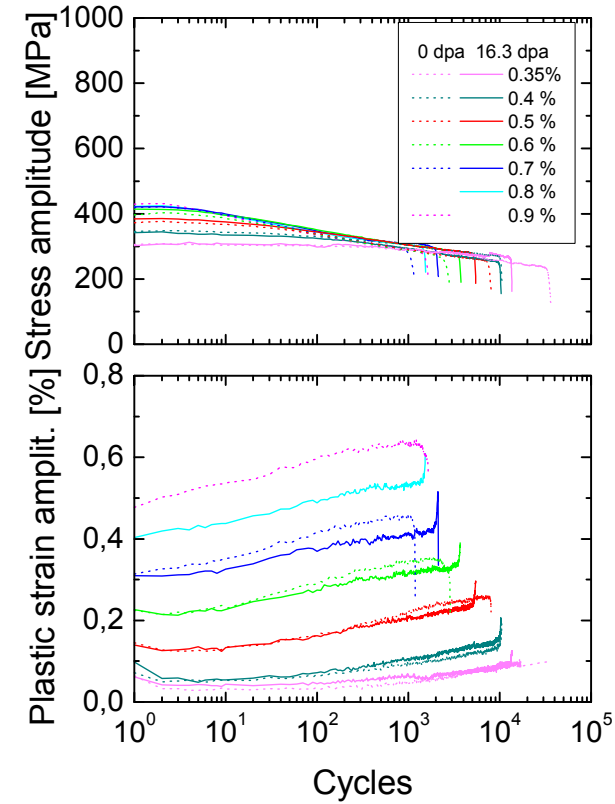
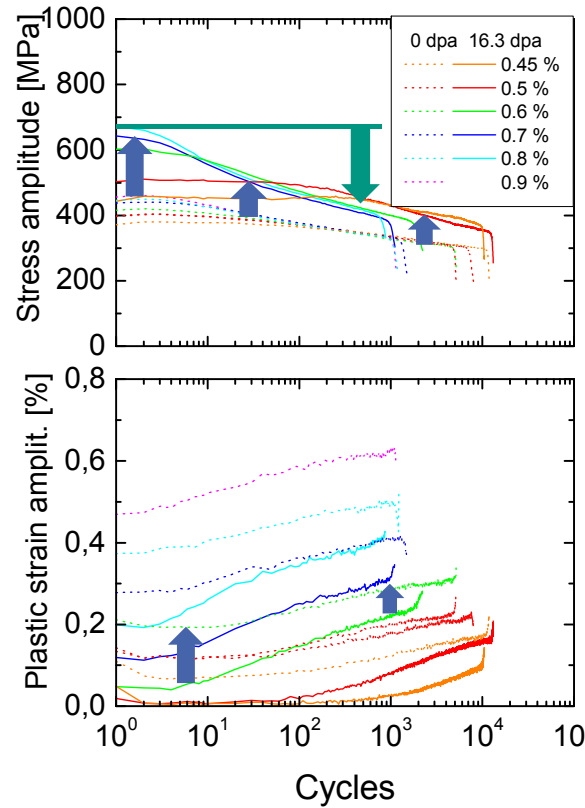
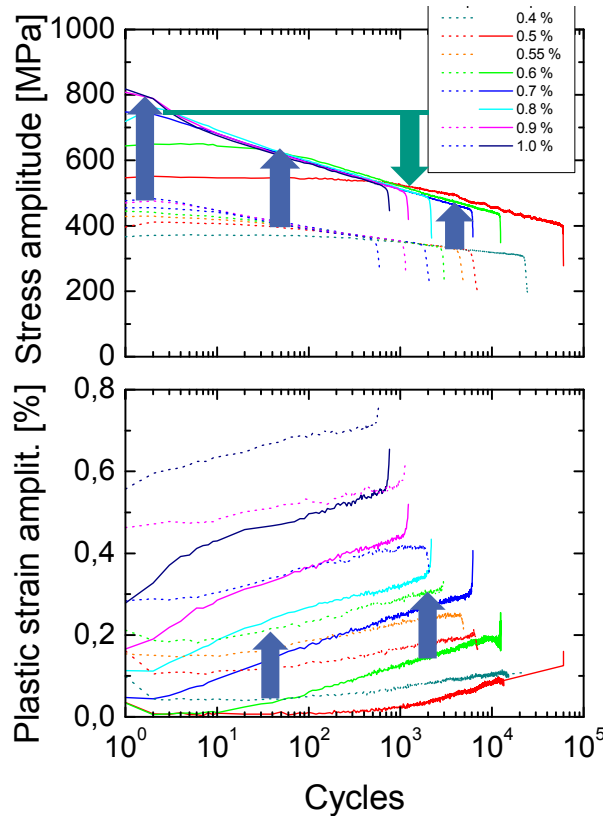
- No irradiation induced hardening $\Delta\sigma_{\text{irr}}$ at all
- Hysteresis loops very similar for irradiated and non-irradiated samples

$\Delta\varepsilon$ controlled LCF after 16.3 dpa irradiation

$T_{\text{irrad.}} = T_{\text{test}} = 250\text{ }^{\circ}\text{C}$

$T_{\text{irrad.}} = T_{\text{test}} = 350\text{ }^{\circ}\text{C}$

$T_{\text{irrad.}} = T_{\text{test}} = 450\text{ }^{\circ}\text{C}$



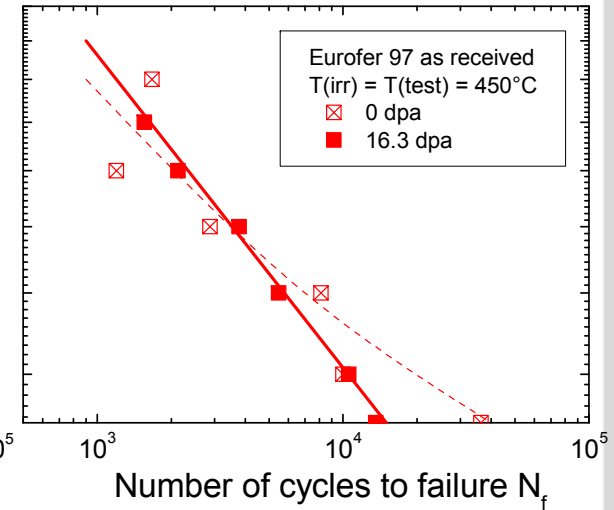
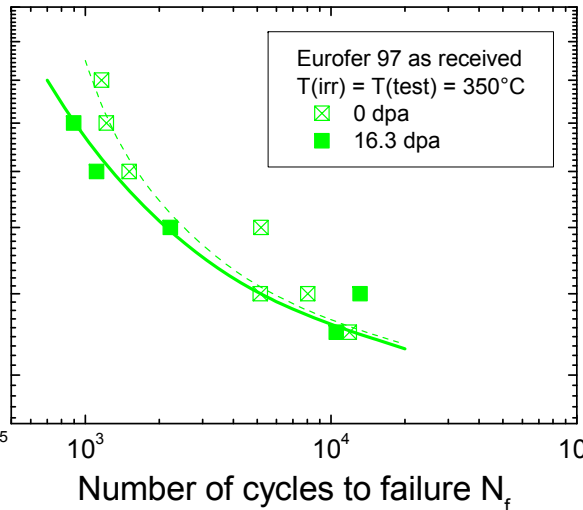
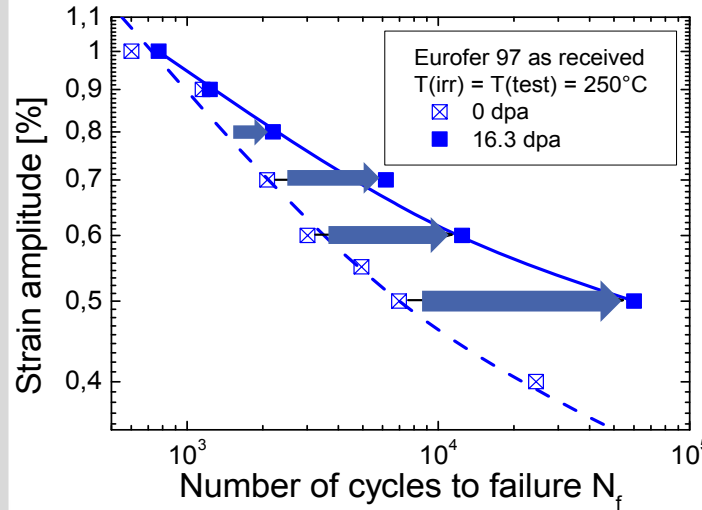
- Below $\sim 400^{\circ}\text{C}$: Significant effect of irradiation on $\Delta\sigma$ and $\Delta\varepsilon_{\text{pl}}$ (\uparrow) as well as on **cyclic softening** (\downarrow)
- Above $\sim 400^{\circ}\text{C}$: Interstitial type defects unstable \rightarrow no irradiation effect

Effect of 16.3 dpa irradiation on lifetime

$T_{\text{irrad.}} = T_{\text{test}} = 250 \text{ }^{\circ}\text{C}$

$T_{\text{irrad.}} = T_{\text{test}} = 350 \text{ }^{\circ}\text{C}$

$T_{\text{irrad.}} = T_{\text{test}} = 450 \text{ }^{\circ}\text{C}$



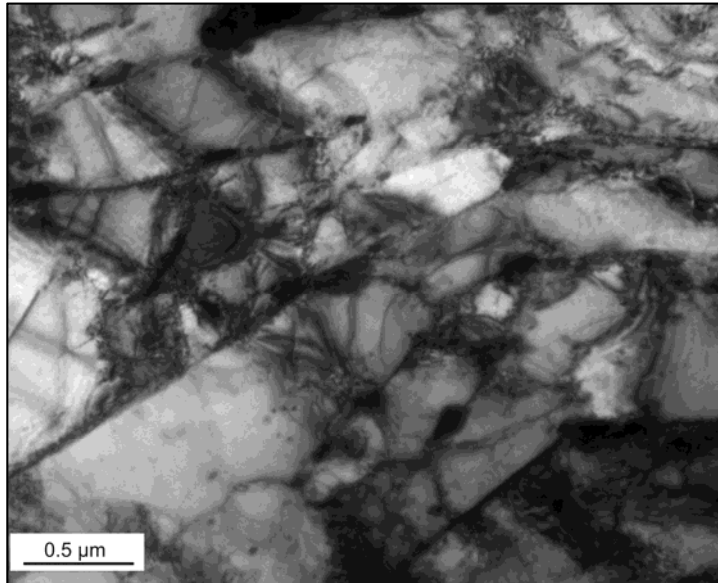
Pronounced increase of lifetime N_f after low temperature irradiation



Lifetime N_f in the range of non-irradiated specimens

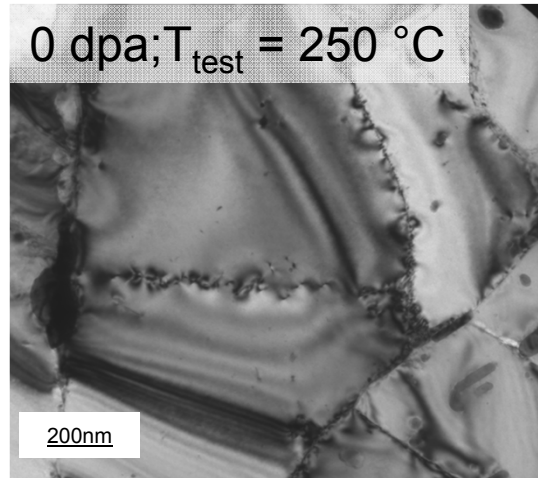
TEM analysis, EUROFER97 (as received)

0 dpa, Before fatigue testing

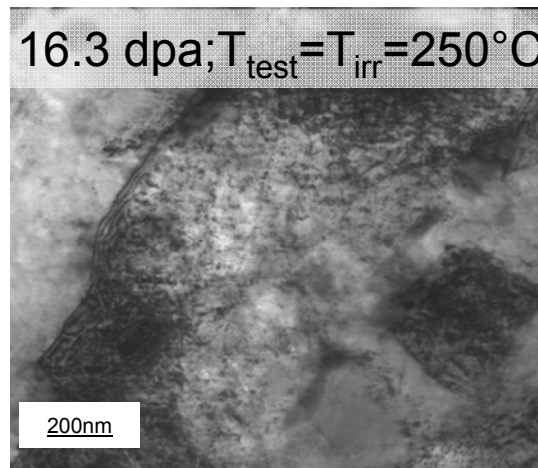


- Microstructure typical for tempered martensite
- High dislocation density

After fatigue testing, $\Delta\varepsilon = 0.5\%$



- Pronounced sub-grain formation
- Low dislocation density within sub-grains
- Dislocation pile-up at sub-grain boundaries

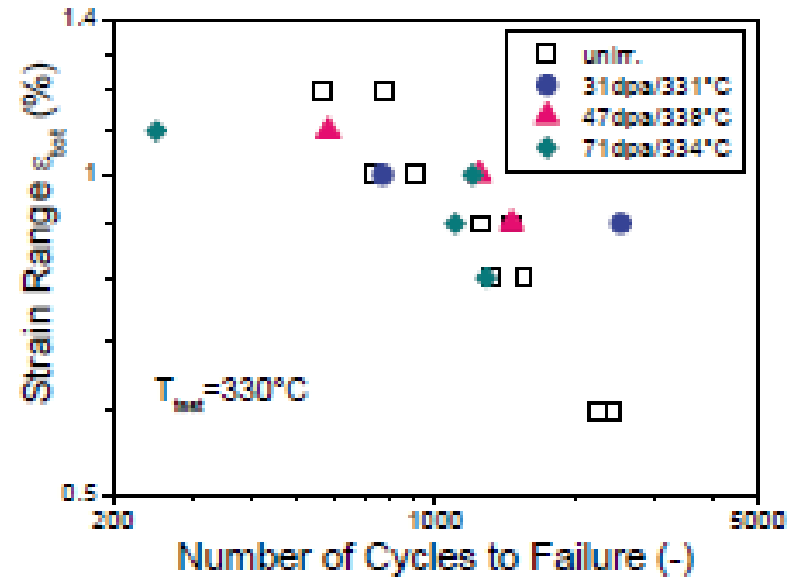
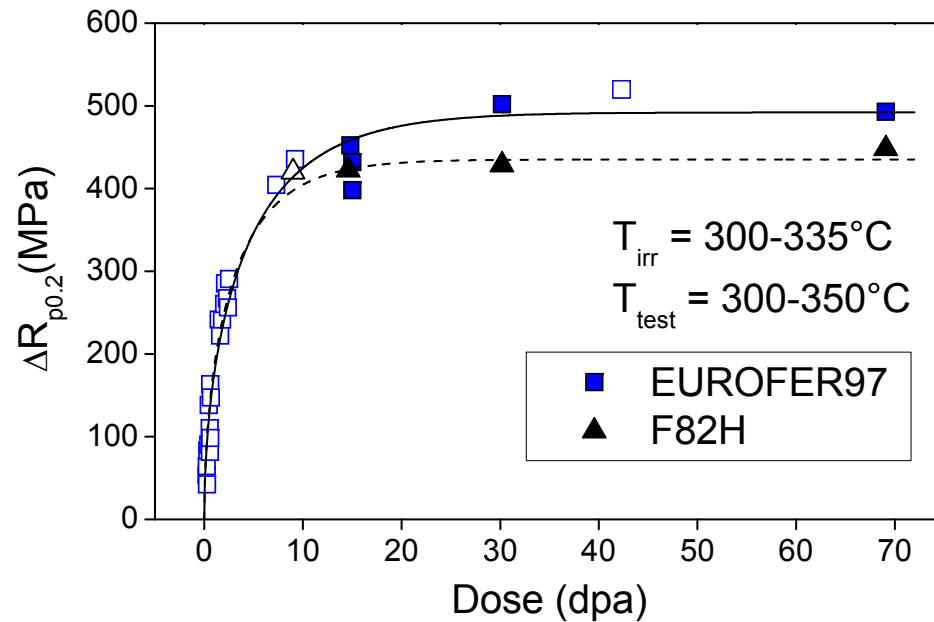


- Also sub-grain formation, but less obvious
- High stability of irradiation induced defects, despite cyclic motion of dislocations

Is there a saturation of properties with increasing displacement damage ?

“no” He, Tensile testing

“no” He, fatigue testing



“Orowan-type fit:

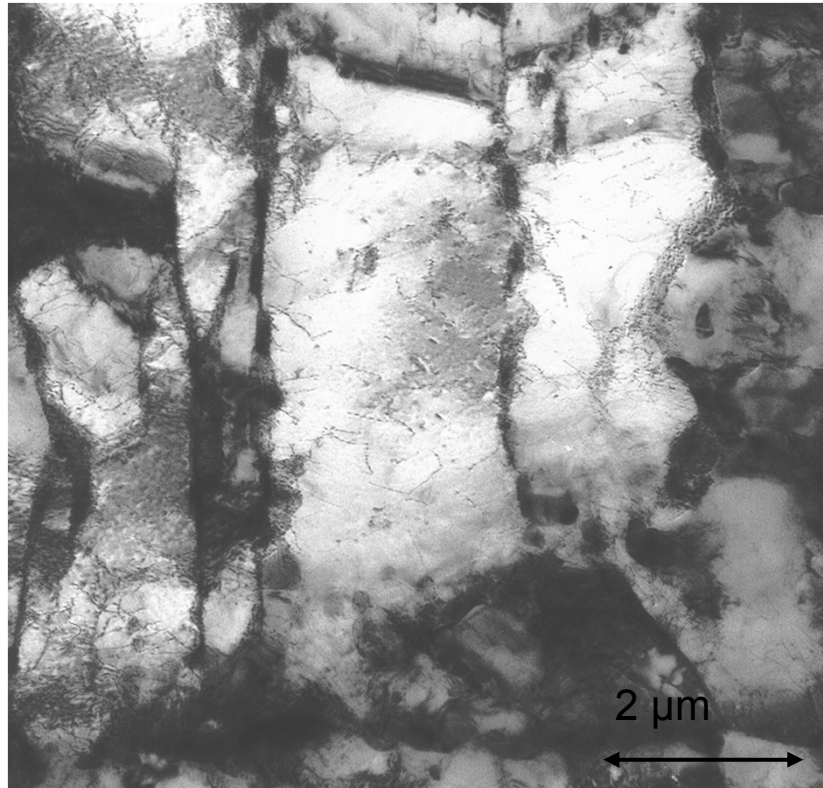
$$\Delta\sigma_{irr} = M\alpha\mu b\sqrt{Nd} \approx \Delta\sigma_{sat} \sqrt{[1 - \exp(-\Theta/\Theta_0)]}$$

➔ Irradiation induced tensile and fatigue properties seem to saturate beyond ~15 dpa

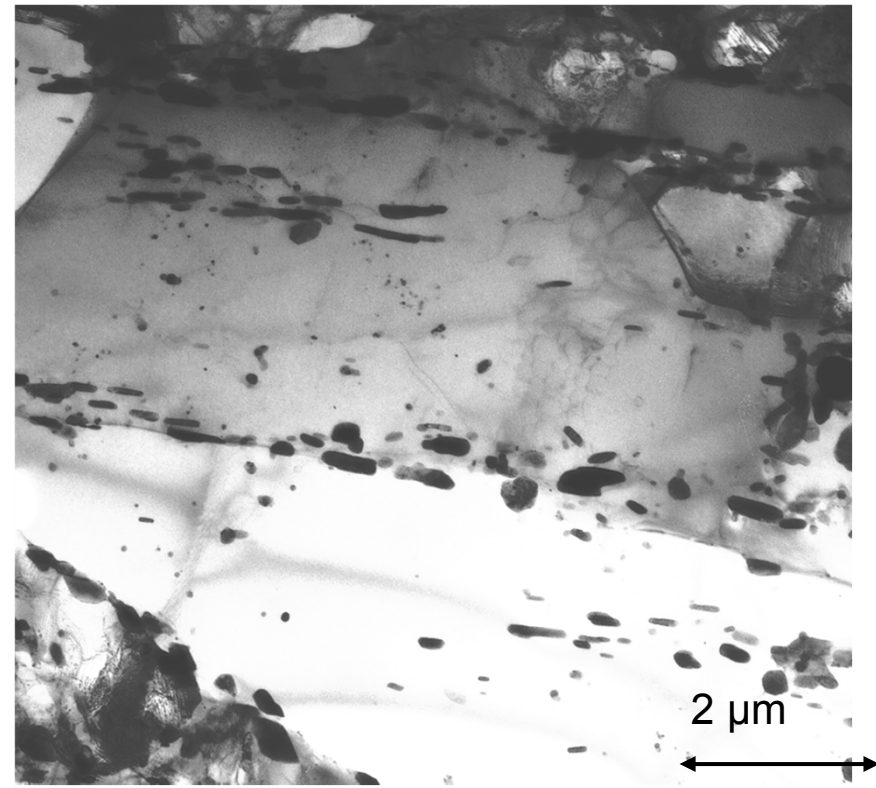
E.Gaganidze, C. Petersen et al., KIT, 2011

TEM Microstructure

Before testing



After Fatigue testing at 550 °C
 $\Delta\varepsilon = 0.45 \%$, $N_f = 60850$

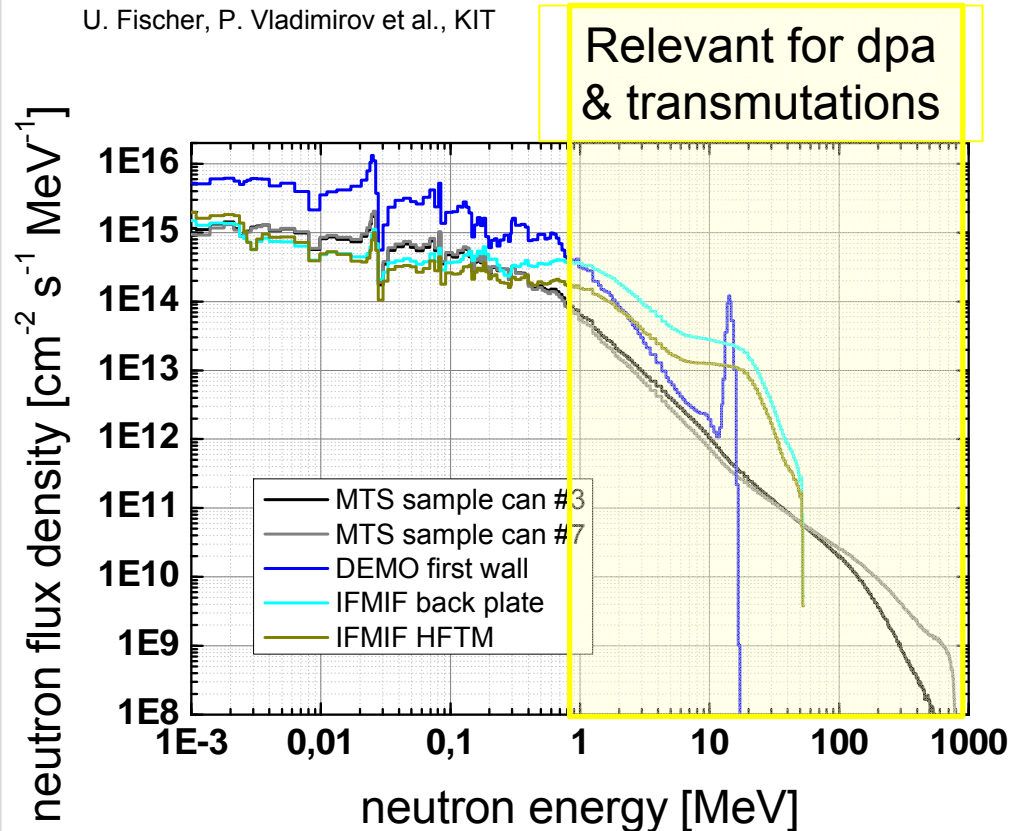


Fatigue testing: Besides sub-cell creation pronounced formation of precipitates (mostly $M_{23}C_6$) → substantial cyclic softening

Neutron spectra effects: Tensile properties

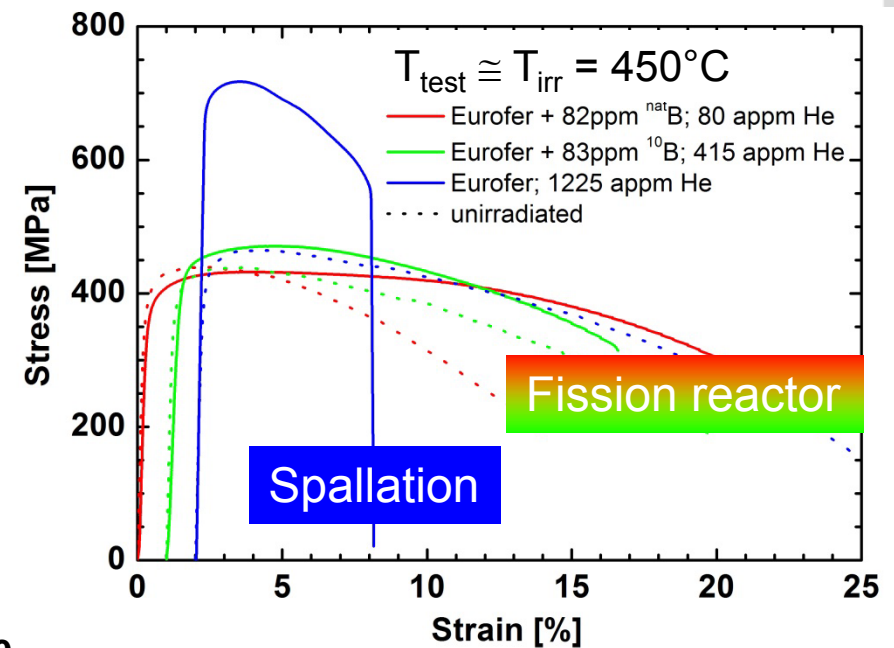
N-spectra: DEMO, IFMIF, Spallation

U. Fischer, P. Vladimirov et al., KIT



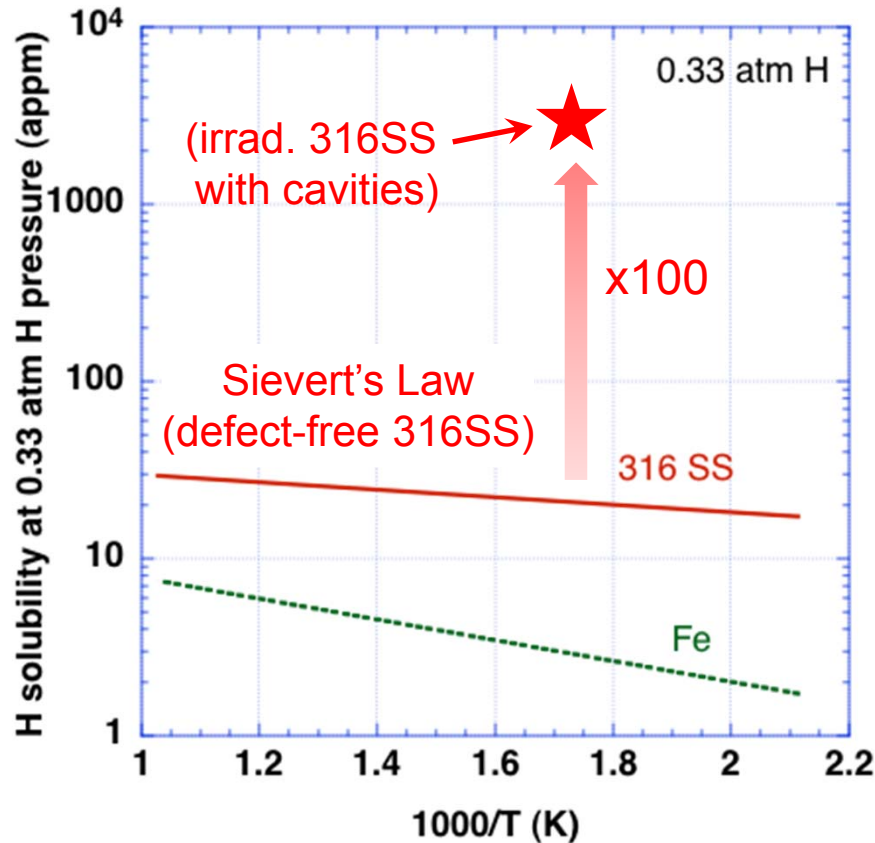
Fission vs. Spallation (PSI)

Y. Dai et al, He-dpa workshop, June 2009, PSI



- Spallation irradiation shows above $T_{\text{irr}} \cong 400^\circ\text{C}$ much higher strength $\Delta\sigma_{\text{irr}}$
- What is the real fusion behavior? Intense N-source would answer this question

Hydrogen effects: Retention of hydrogen in Fe and austenitic Steel 316



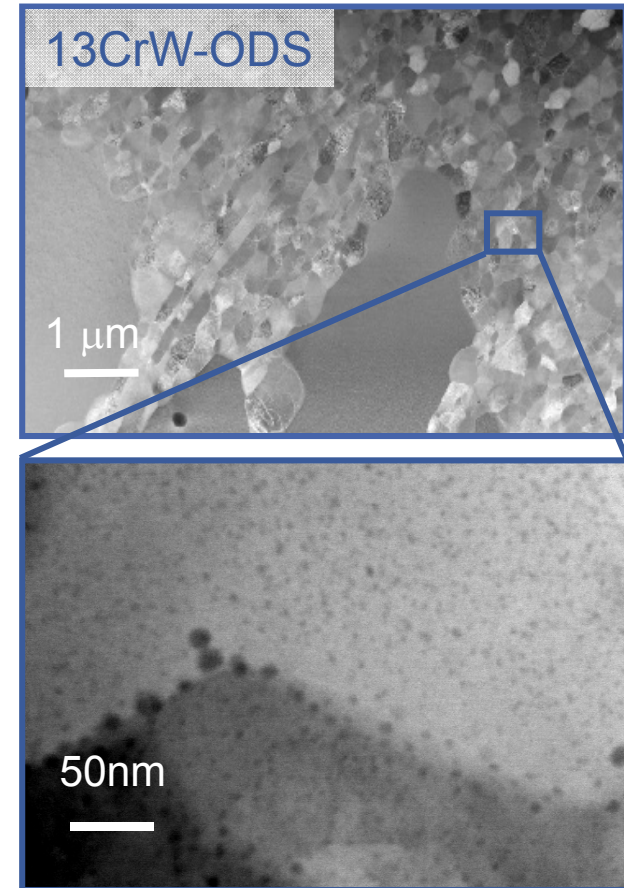
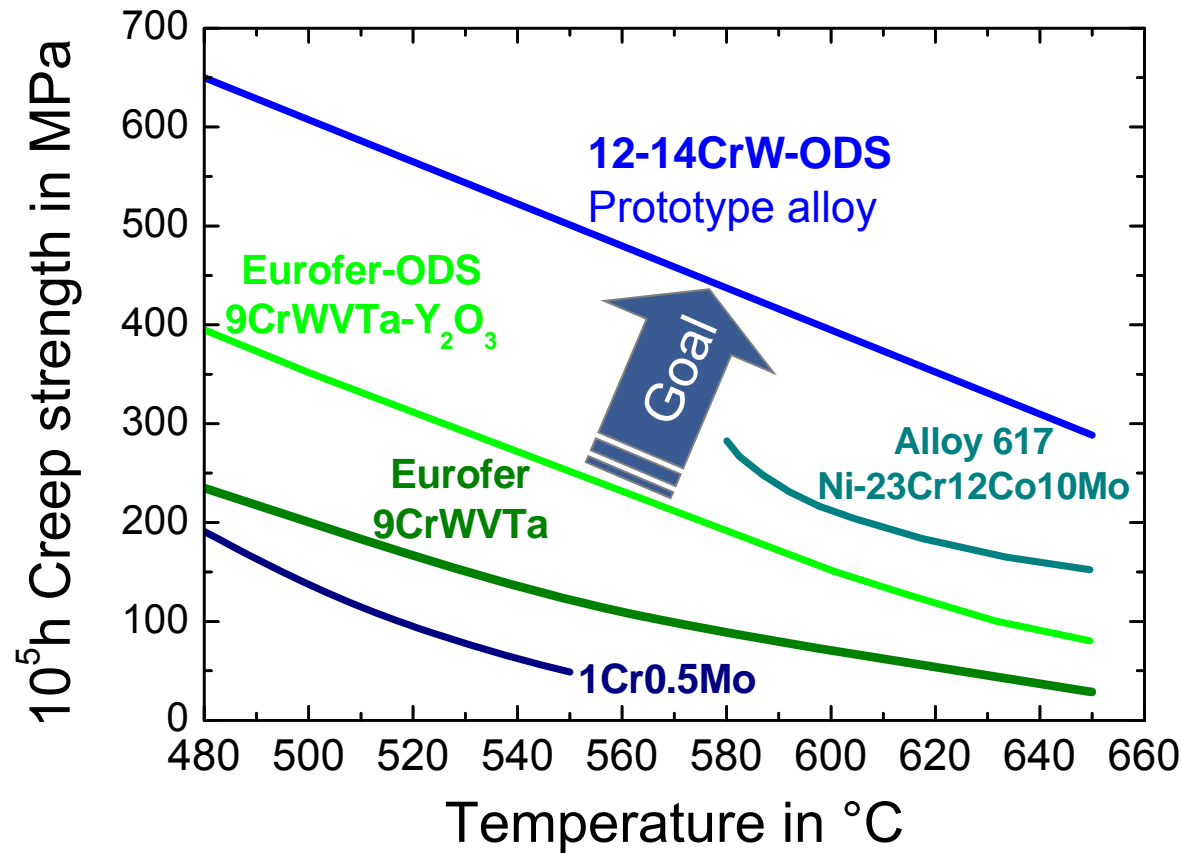
F.A. Garner; J. Nucl. Mater. **356** (2006) 122-135

S.J. Zinkle, A. Möslang, Fus. Engin. and Design
88 (2013) 472– 482

- Hydrogen effects may become a serious issue in fusion environments
- Typical for 14 MeV fusion neutrons is the simultaneous production of dpa, hydrogen and Helium → **Intense fusion neutron source indispensable for realistic validation**

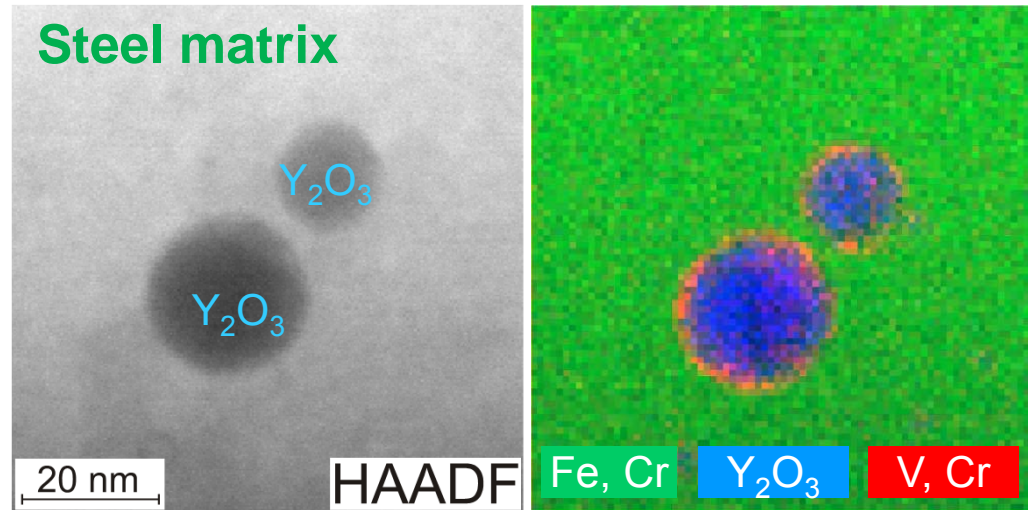
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- Technical Readiness and database maturity
- Role of materials in fusion roadmaps,
Need for fusion specific irradiation source

International challenge: Development of nanoscaled iron based “super alloys” (RAF-ODS)



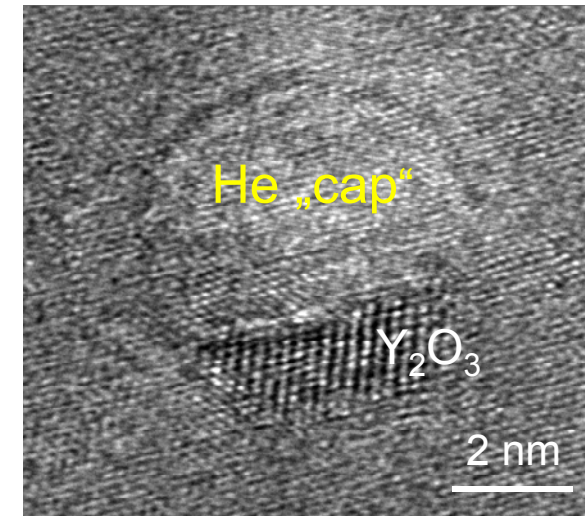
E. Eiselt, M. Klimenkov, R. Lindau, A. Möslang
 J. Nucl. Mater. 2010

Nanoscaled ODS-steels



P. He, M. Klimenkov et al., J. Nucl. Mater. 428 (2012) 131-138

1000 appm He impl. at 500°C

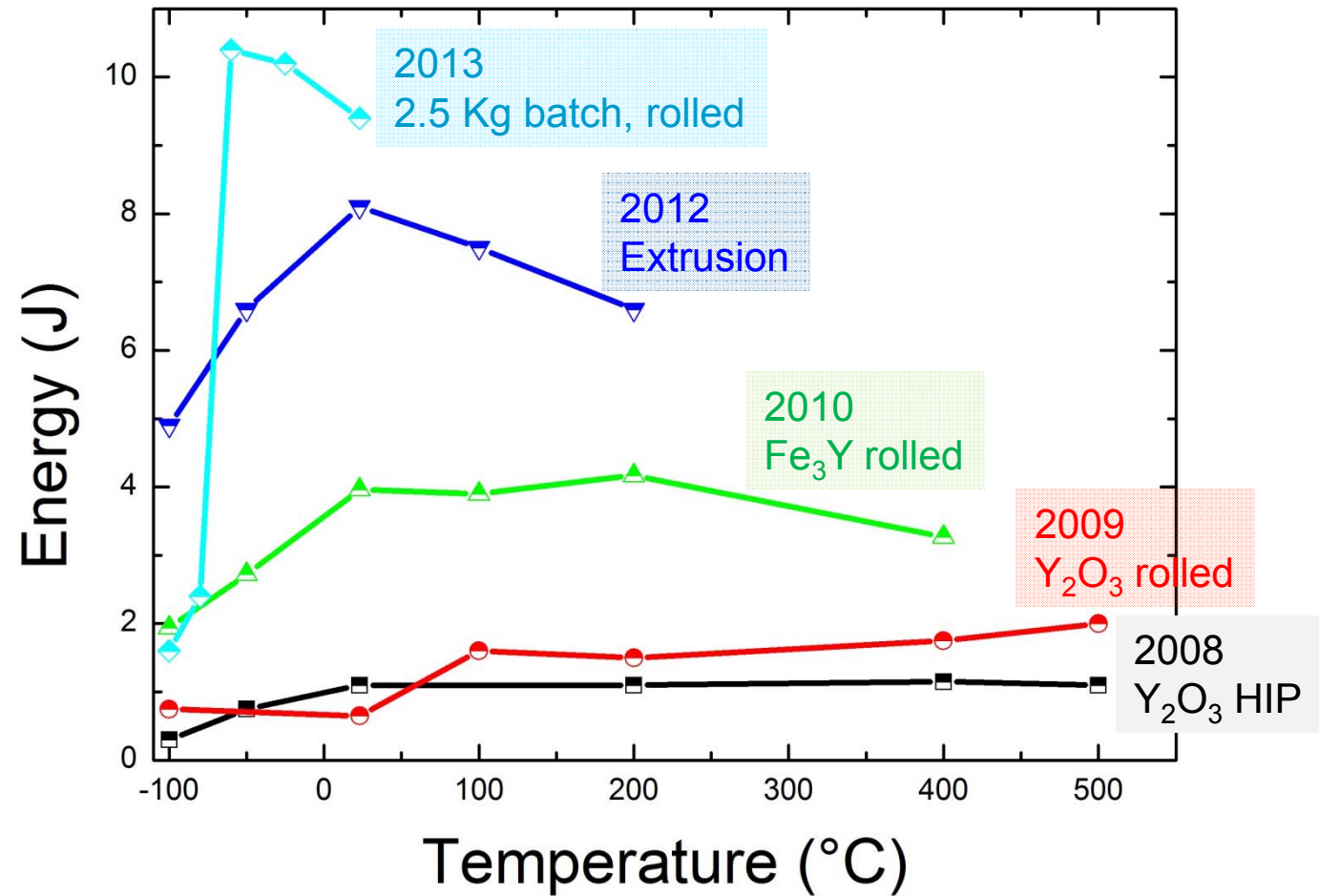


A. Ryazanov et al; JNM (2013) 153-157

- Nano-scaled ODS particles like Y_2O_3 or $Y_2Ti_2O_7$ are efficient trapping centers for diffusing alloying elements (Cr, V) and irradiation induced defects (vacancies, He)
- Therefore, nanoscaled ODS steels have potential for outstanding aging and irradiation resistance

Nanoscaled ODS-steels:

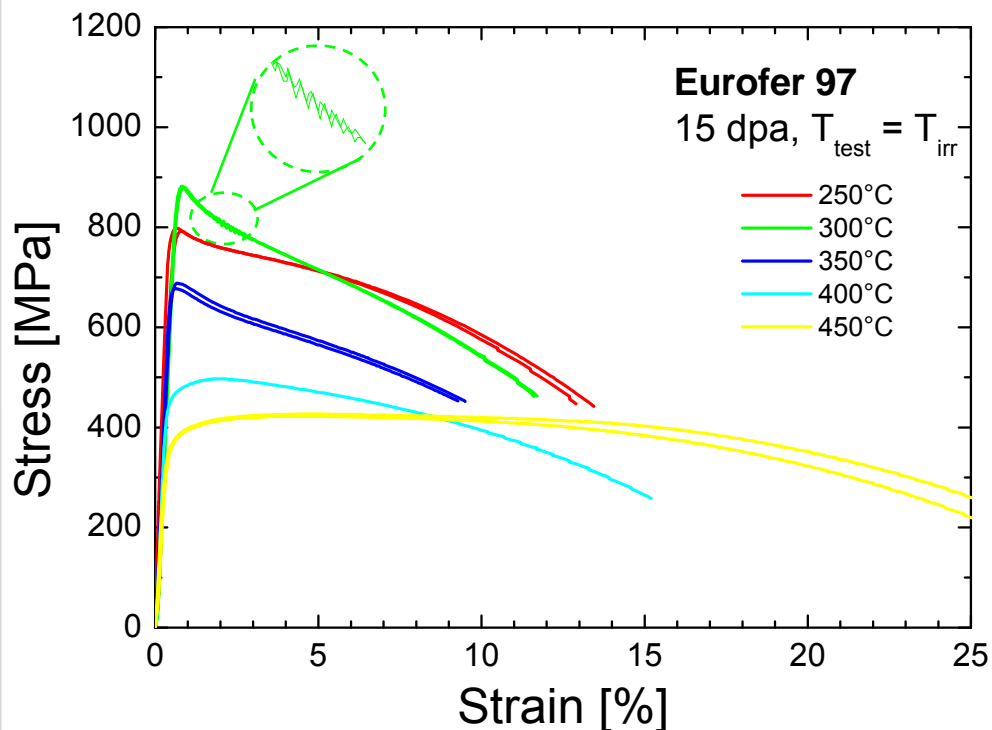
Substantial improvement of dynamic fracture toughness



J. Hoffmann et al, IAM, KIT, 2014

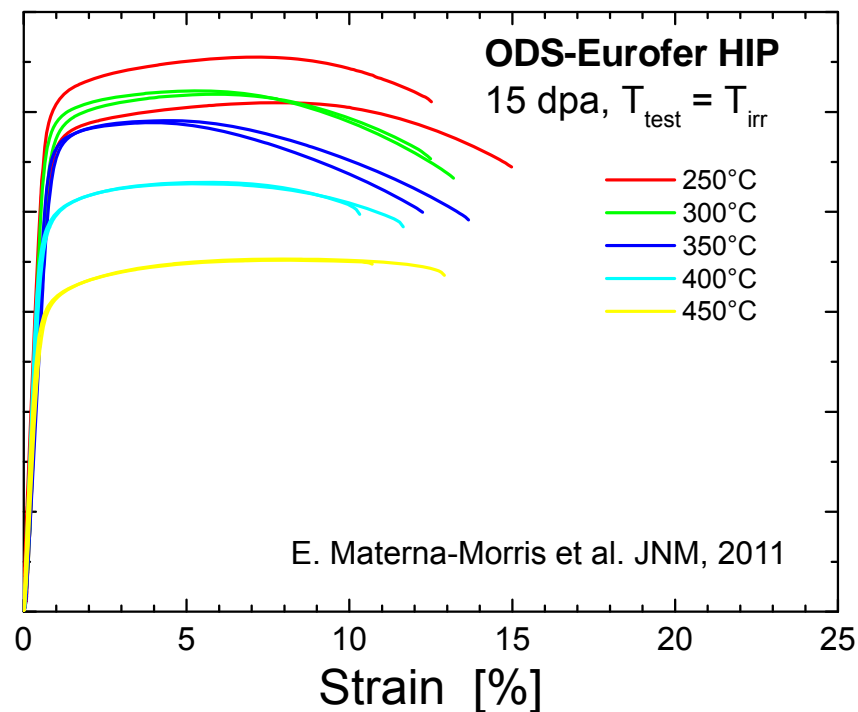
ODS EUROFER after Neutron irradiation: Substantial improvement of tensile properties

RAFM Steel



- Substantial irradiation hardening
- Early strain localization due to dislocation channeling → $A_u \sim 0.3\%$

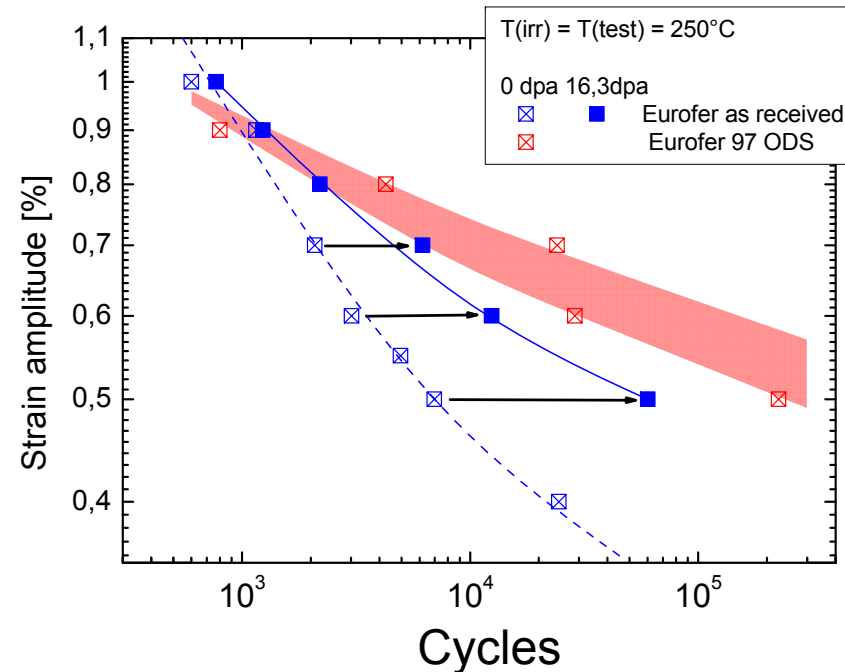
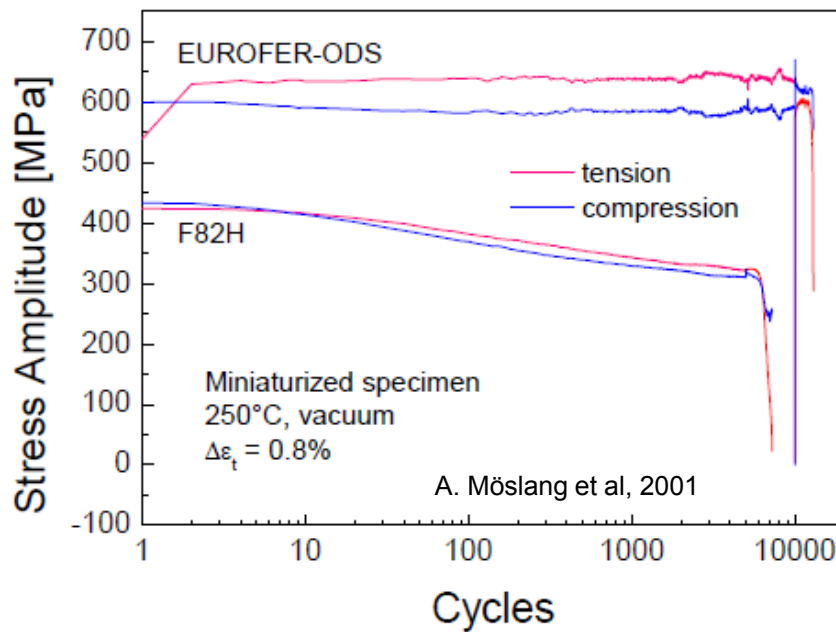
RAFM-ODS Steel



- Still work hardening → almost no loss of uniform elongation ($A_u \sim 7\%$)

Outlook Fatigue:

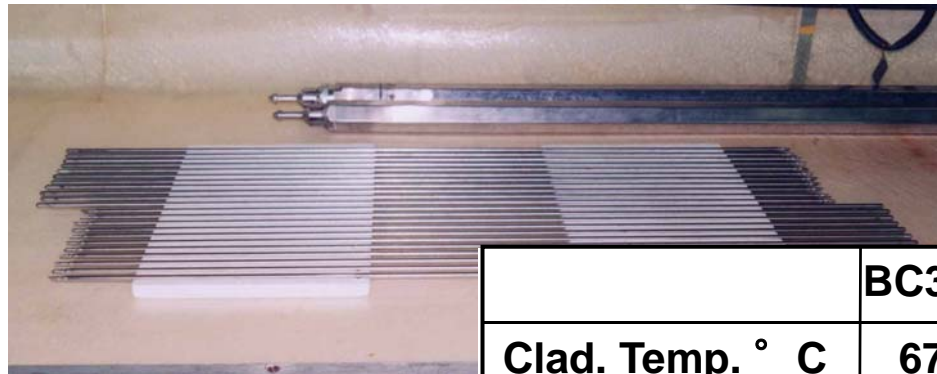
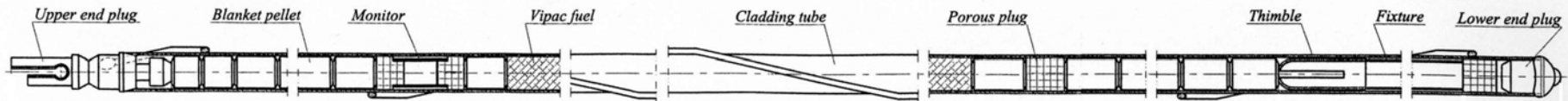
Is it possible to overcome the cyclic softening phenomena of ferritic-martensitic steels?



Nano-scaled ODS particles are extremely stable, that is:

- (almost) no cyclic softening or dislocation channeling
- Suppression of alloy dissolution and aging due to their high sink strength

9Cr ODS steel fuel pins: Production in Japan, irradiation in FBR BOR60, Russia



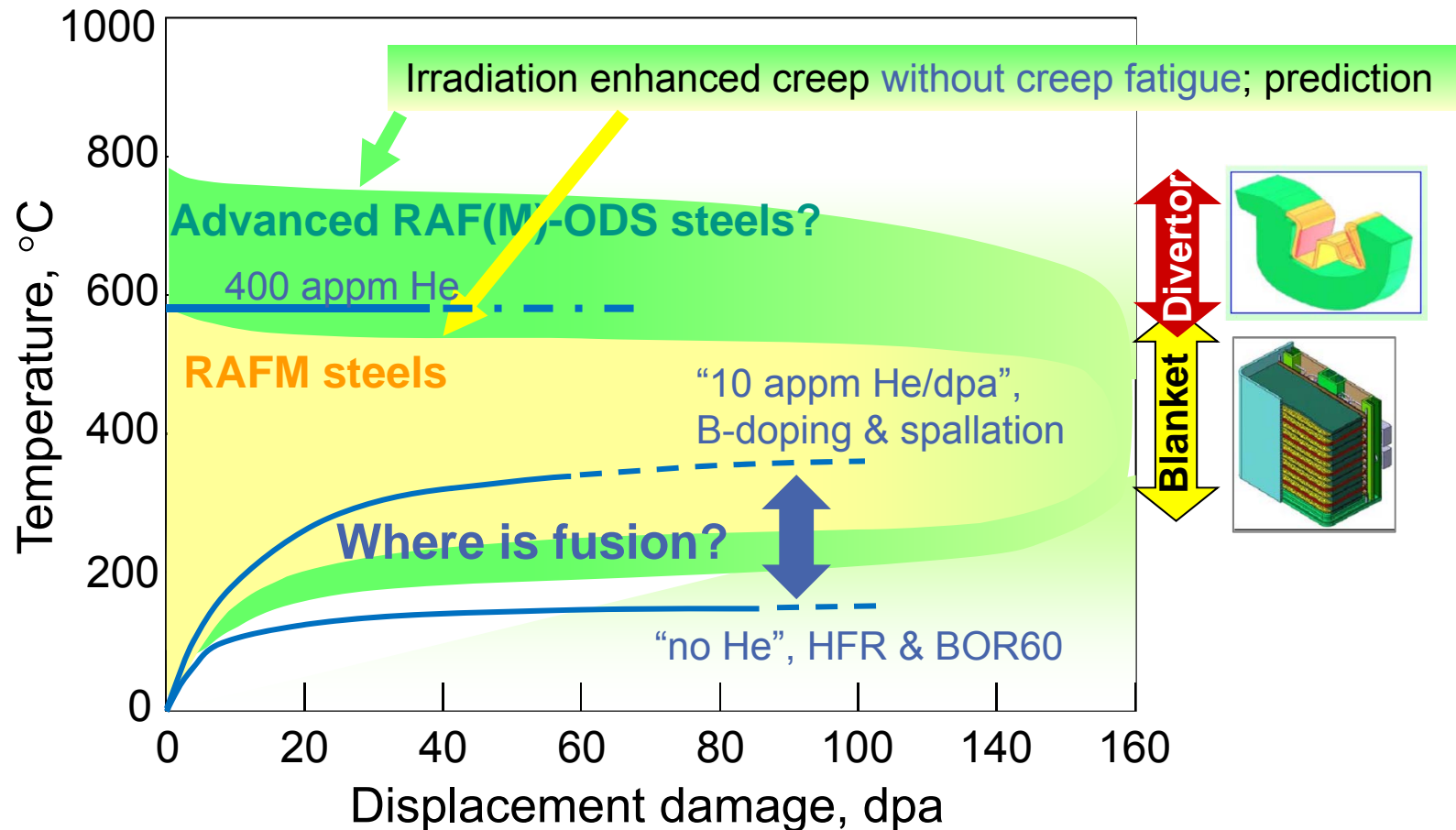
	BC358	BC359
Clad. Temp. ° C	670	720
Burnup at%	15	
Dose dpa	75	

S. Ukai et al., Hokkaido University, Japan

Fig. 12 Appearance of fabricated ODS fuel assemblies (BC358E and BC359E)

- Materials challenges: fusion – fission – spallation
- Neutron irradiation: Examples of progress and issues
 - Reduced activation ferritic/martensitic steels
 - Oxide dispersion strengthened steels
 - W alloys: I-1, I-3, O-1, O-2: Yesterday
I-7, O-5, O-6, I-8: Today
- **Technical Readiness and database maturity**
- **Role of materials in fusion roadmaps,
Need for fusion specific irradiation source**

Application window of RAF(M)-ODS-Steels - schematic -



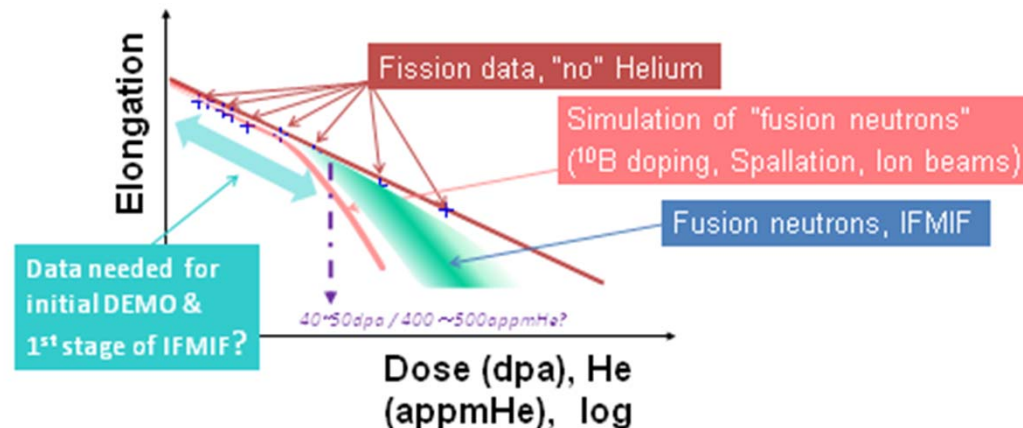
- ❑ Operation window 350-550 °C: Are RAFM steels limited to ~ 50dpa at the FW?
- ❑ Fusion relevant data well beyond 50 dpa urgently needed → fusion n-source

Design code extension by RAFM steels

In order to get the licensing (and to protect the investment) all these issues must be taken into account in the design codes used during the design phase

Possible steps towards a fusion design code:
Tensile properties as example

In the style of H. Tanigawa, E. Wakai

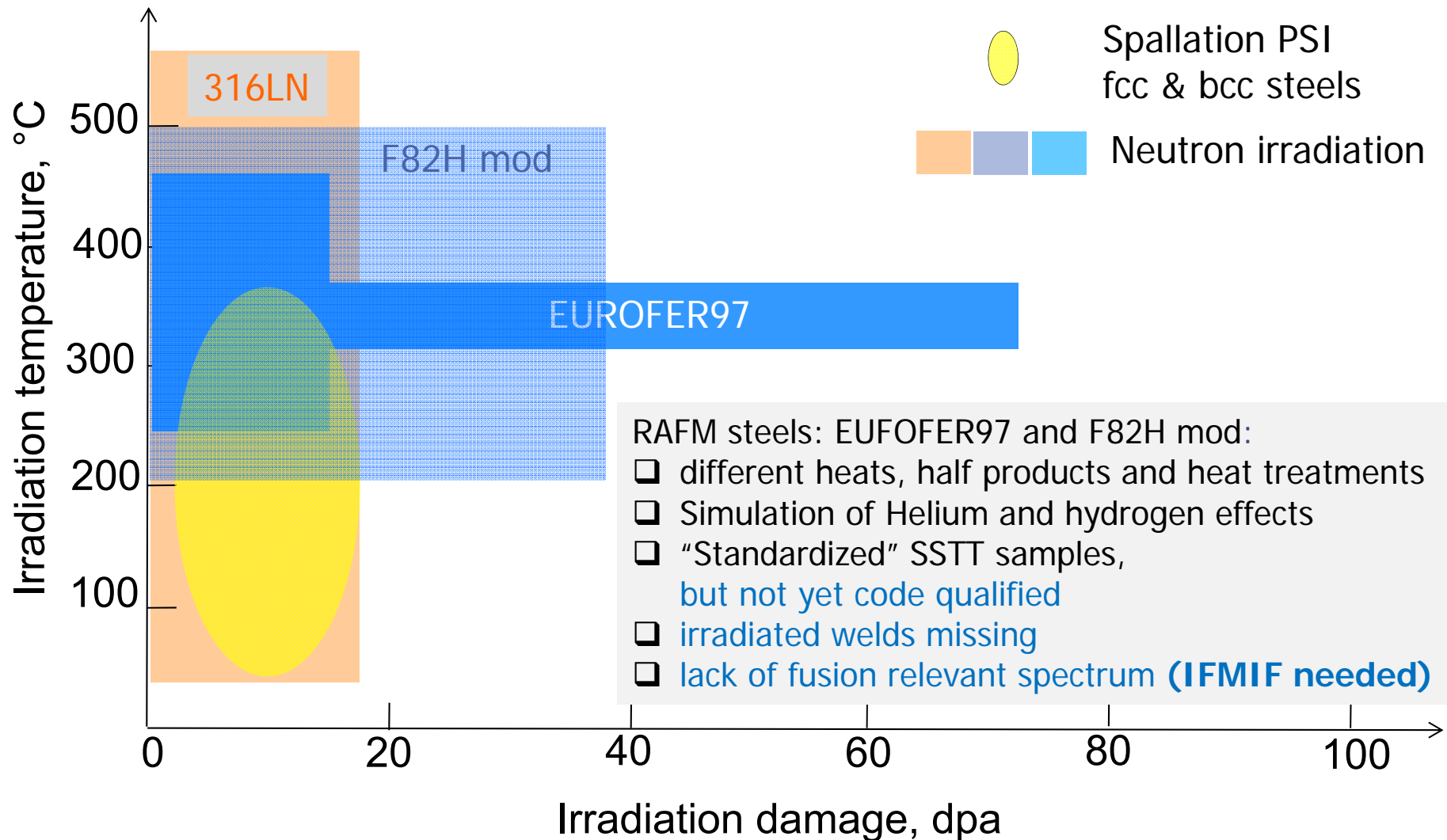


Fusion-like irradiation data are needed

- To provide timely a fusion relevant materials database
- To monitor a few properties at high doses (to see the potential of the material for advanced DEMOs and FPP)

Existing database on neutron irradiated steels

- schematic -



Design code qualification, e.g. for RCC-MRx

RAFM_Fatigue

MAIN LIST SEARCH REPORTS PRODUCT COMPOSITION TENSILE IMPACT FRACTURE CREEP SUMMARY QUIT

PRODUCT

Alloy	Metal	Designation	Manufacturer	Weight_kg
Eurofer	HIP_Powder	Eurofer97 powder	CEA/G	
Heat	Product No	Sub-Product No	CW %	Thick (mm)
E83699	E6			100

HEAT TREATMENTS

HT (C)	HT (min)	Temper (C)	Temper (min)	PWHT (C)	PWHT (min)	Aging (C)	Aging (h)
979	111	739	222	4h 1040C			

IRRADIATION

State	Irr. Facility	Experiment	Position in Rig	Irr. T (C)	Dose (dpa)	He_appm
	HFR	SOSIA-3		500	2.5	

TEST

Source Data	Country	Test No	Environment	Extens.	Norme	Type	Cycle	R	1st Cyc
NRG	Netherlands	315	air	axial	ASTM	Push-Pull	Triangular	-1	Tension

SPECIMEN

Plan of Cutting	Sp. Drawing	Orient	Strain Rate 10 ⁻³	Sp. No	Form	t or d (mm)	Total L.	Gauge (mm)
	RH-MMI-	L	10	H503	C	3	45	7.5

RESULTS

Test T (C)	ΔE_t	ΔE_p	ΔE_e	E	1st 1/4 cycle		at N _i		at S _{max}		
500	.6	0.28	0.32	189000	Sao	f ₉₀	St	Sc	$\Delta S_a/2$	Stmax	N _i Stmax
					373				276	368	2

Cyclic Hdg

N	1	10	20	50	100	10 ³	10 ⁴	10 ⁵	
St	373.0	361.0			330.0	270.0			
Sc	360.0	353.0			306.0	258.0			

Relaxation Cycle

State_I	Hold_T	Srtmax	Srtmin	Srt	State_c	Hold_C	Srmax	Srmin	Src

Observations

References: J. Rensman, private comm., N50 & Dep & Dee added from NRG-21641/09.95503, 20 May 2009

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Collection of broad based materials data

Validation by expert groups

code qualified "Materials Properties Handbook"

Distribution to manufacturers and designers for comments

RCC-MRx Design code implementation of the new material class

Irradiation effects: Materials Database Maturity



Complimentary: R. Kurtz, PNNL

	1 st DEMO Blanket						2 nd DEMO Blanket						Adv. DEMO						
Data base need	<20 dpa/200appm He						~50 dpa/500appm He						>100 dpa/1000appm He						
Materials	RAFM	FM-ODS	W	SiC	Be	Li ceramic	RAFM	FM-ODS	W	SiC	Be	Li ceramic	RAFM	FM-ODS	W	SiC	Be	Li ceramic	RAFM

Irradiation effects

Hardening/Embrittlement	Green	Yellow	Red	Yellow	Yellow	Red	Yellow	Red	Red	Yellow	Red	Red	Red	Red	Red	Red	Red	Red	Red
Phase stabilities	Green	Yellow	Red	Yellow	Yellow	Yellow	Yellow	Red	Red	Yellow	Red	Red	Red	Red	Red	Red	Red	Red	Red
creep & fatigue	Green	Yellow	Red	Yellow	Red	Red	Yellow	Red	Red	Red	Red	Red	Red	Red	Red	Red	Red	Red	Red
Volumetric swelling	Green	Yellow	Red	Yellow	Green	Yellow	Yellow	Red	Red	Yellow	Yellow	Red	Yellow	Red	Red	Red	Red	Red	Red
High Temp He&H effects	Yellow	Red	Red	Red	Yellow	Yellow	Yellow	Red	Red	Red	Red	Red	Red	Red	Red	Red	Red	Red	Red

- Adequate knowledge base exists
- Partial knowledge base exists
- No knowledge base

Note: He levels are only for FM steels

Suggested TRLs for in-vessel materials

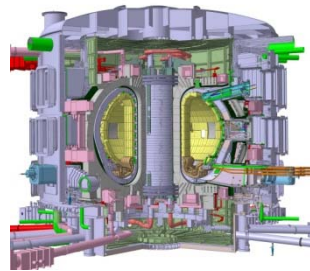
According to M.S. Tillak et al, ICFRM-15, 2012

	Concept development			Proof of Principle			Proof of Performance		
TRLs	1	2	3	4	5	6	7	8	9
Material class									
RAF	█	█	█	█	█				
ODSS 9Cr(12)	█	█	█	█					
ODSS 13-15Cr	█	█	█	█					
W-alloy structure	█	█	█	█					
Functional W	█	█	█	█	█				
SiC/SiC	█	█	█	█	█				
Beryllium	█	█	█	█	█				

TLR 5-6-7 not achievable by "human resources".
Facilities are needed at 10-100-1000 Mio\$ level

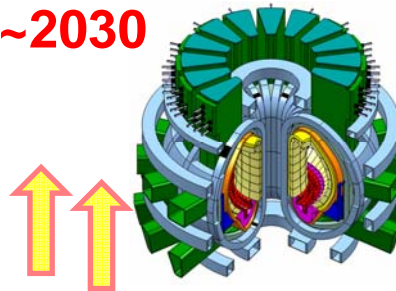
Role of Materials in Fusion Road Maps - simplified -

ITER, DT-phase
beyond 2027

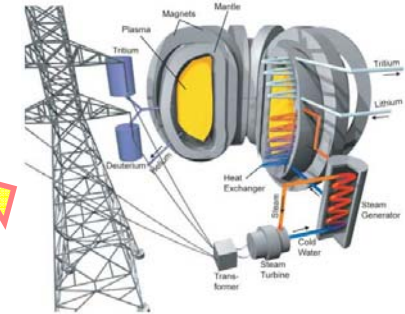


Early DEMOs
EDA end ♦ Start operat.

~2030



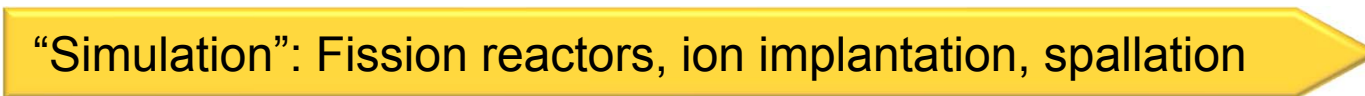
FPP
beyond 2060



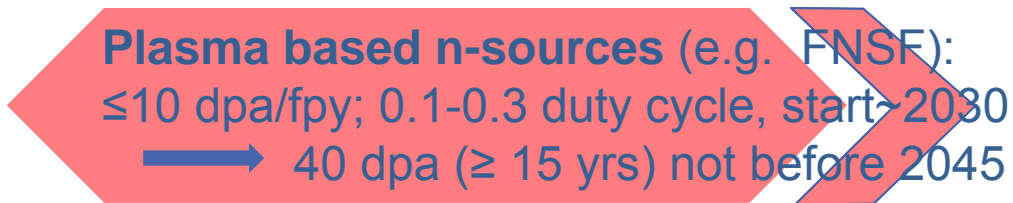
Materials Database



preferable option

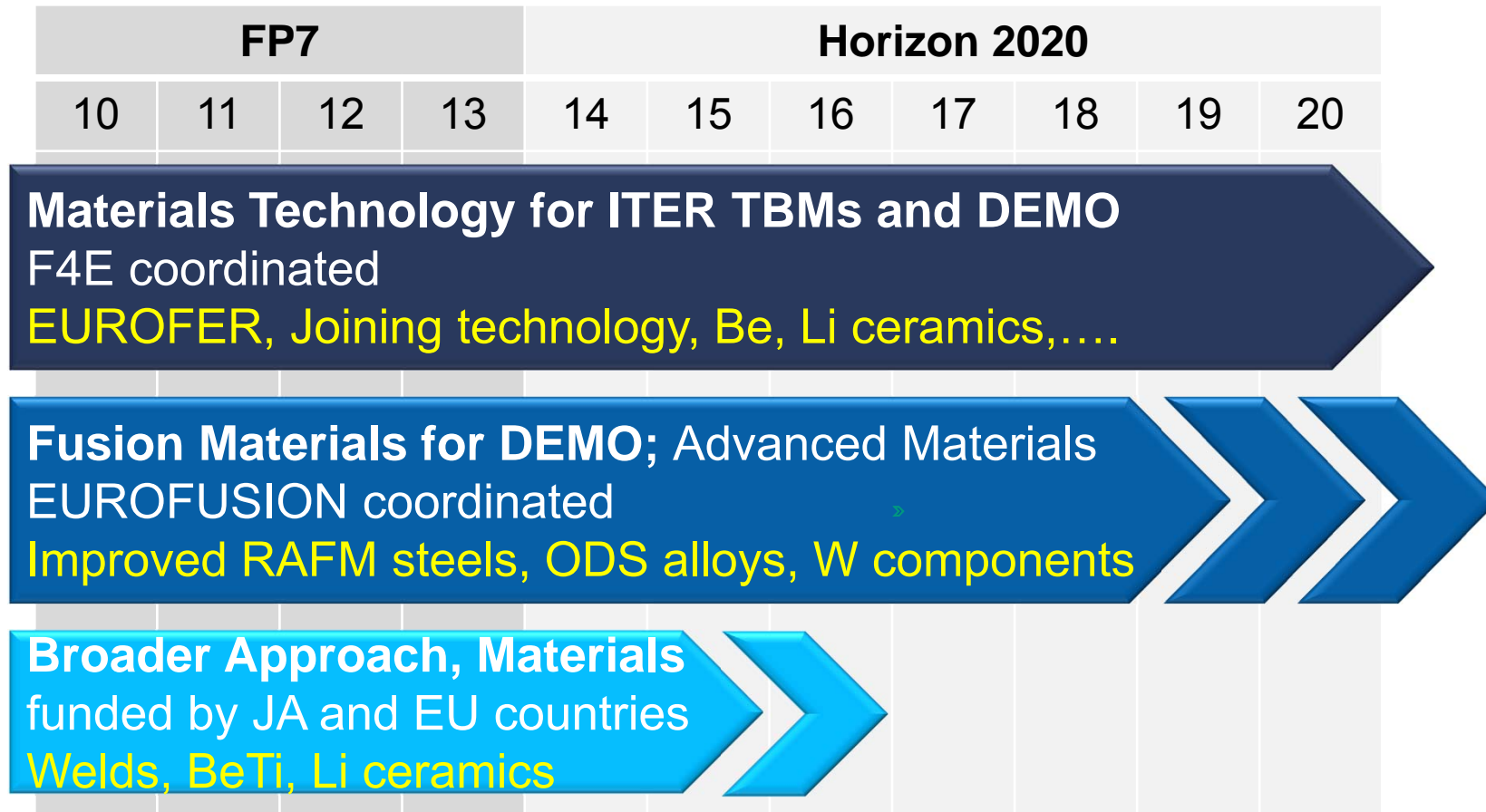


ongoing



For DEMOs too late

Pillars of the EU Fusion Materials Programme



Bottleneck:
DEMO specific irradiation and PIE programmes

International Road Map

High-temperature materials are at a critical path

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