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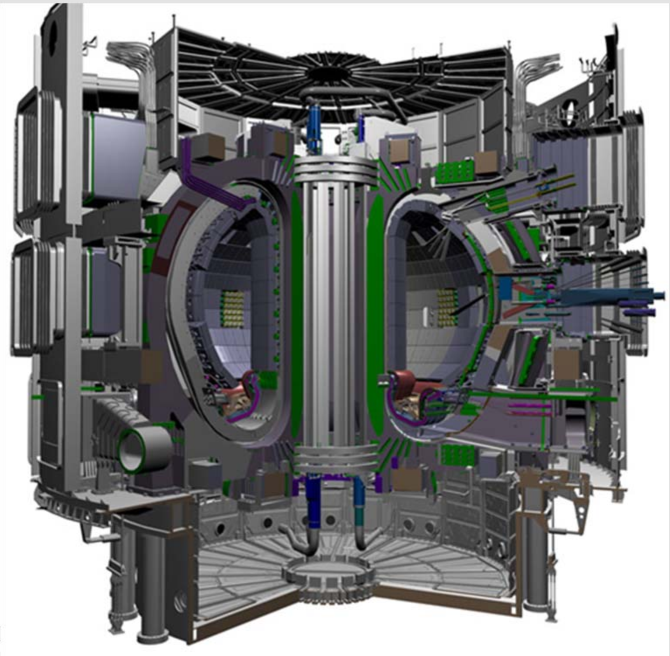
Fatigue-structure correlation of 13.5%Cr ODS steels at 550°C for fusion application

P. He, M. Klimenkov, A. Möslang, R. Lindau



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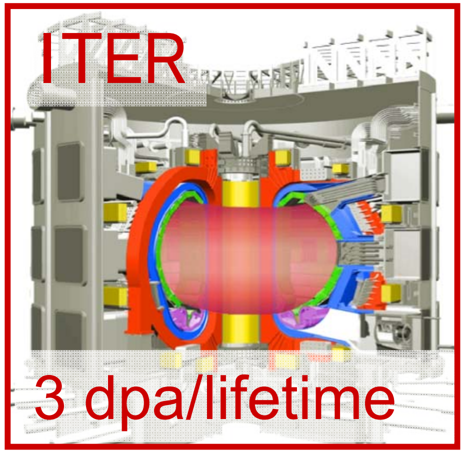
Outline

- Introduction
  - Life-limiting failures of first wall and blanket structures
  - Fatigue issue on Reduced-activation F/M steels and ODS varients
  - Development of ODS RAF steels
- Experimental
  - Fabrication route
  - Sample preparation and monitoring
- Fatigue and microstructural results
  - S-N curve, hysteresis loops
  - Structural results: grain structure, dislocation density, precipitates and ODS particles
- Summary

Materials challenges in fusion reactors

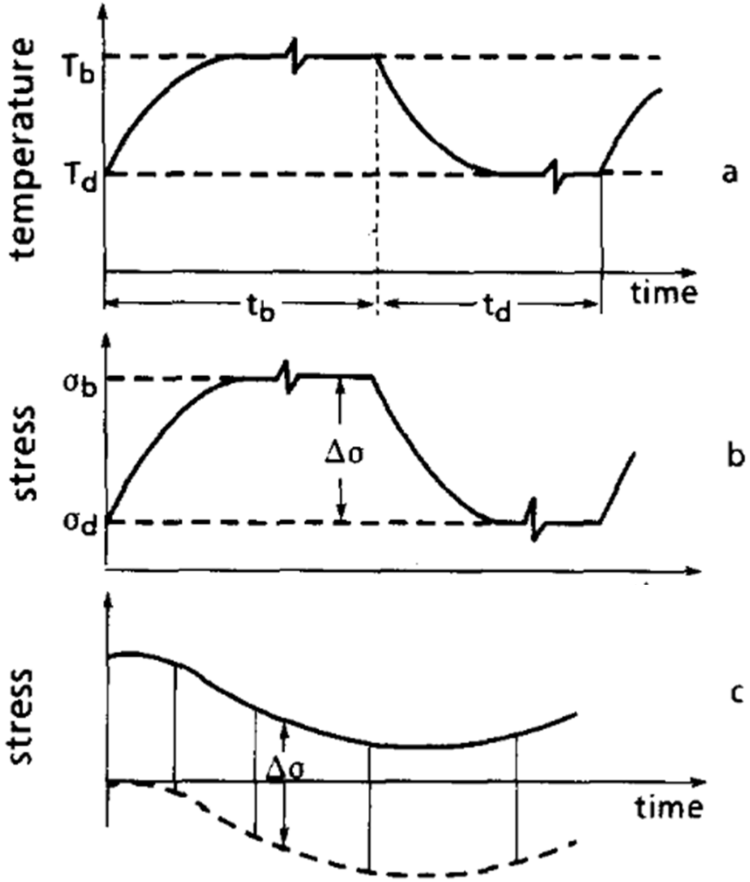


Structural components:

- Fatigue
- Creep
- Creep-fatigue
- Irradiation
- Erosion
- Corrosion

	First wall in ITER	DEMO adv.
Peak surface heat flux [MW/m^2]	0.6	2.5 (blankets)/ 10-15 (divertor)
Number of pulses [×10^4]	2-5	Pulsed or steady-state operation
Total burn time [h]	10^4-3×10^4	Open
Neutron damage [dpa]	12-36	~150

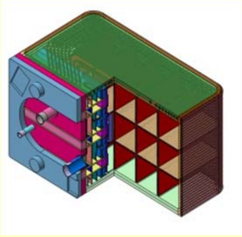
Material challenges in Fusion Reactor



Temperature (a) and stress (b) variation during one burn/dwell cycle and long-term stress variation (c)
D. Munz et al., FUSION ENG DES (1991)

Structural components:

- The lifetime can be limited by different failure modes
- For pulsed tokomaks (ITER), fatigue is the most life-limiting event.
- For steady-state operation, creep-fatigue and irradiation damages are of particular concern.



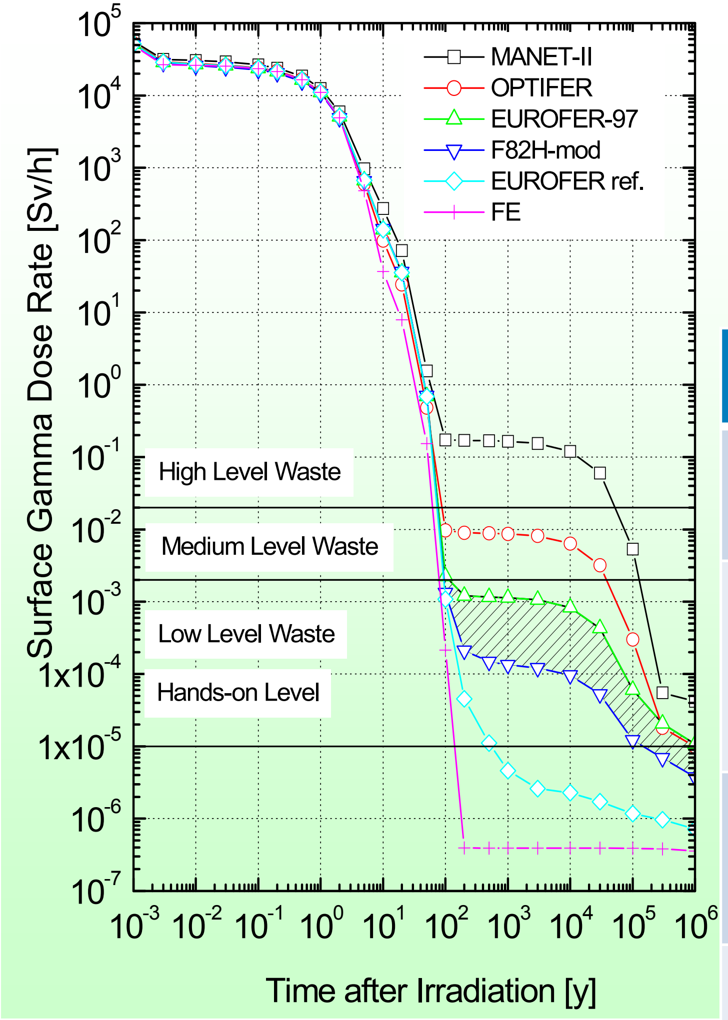
Blanket: ≤30 dpa/year, 2.5MW/m^2

Reduced-activation ferritic-martensitic steels

Nanostructured RAF(M)-ODS-steels

→ ~300 - 800 °C

Reduced Activation F/M steels:



Long term irradiation (12.5 MWa/m^2) of a DEMO reactor first wall
Lindau et al., FUSION ENG DES (2005)

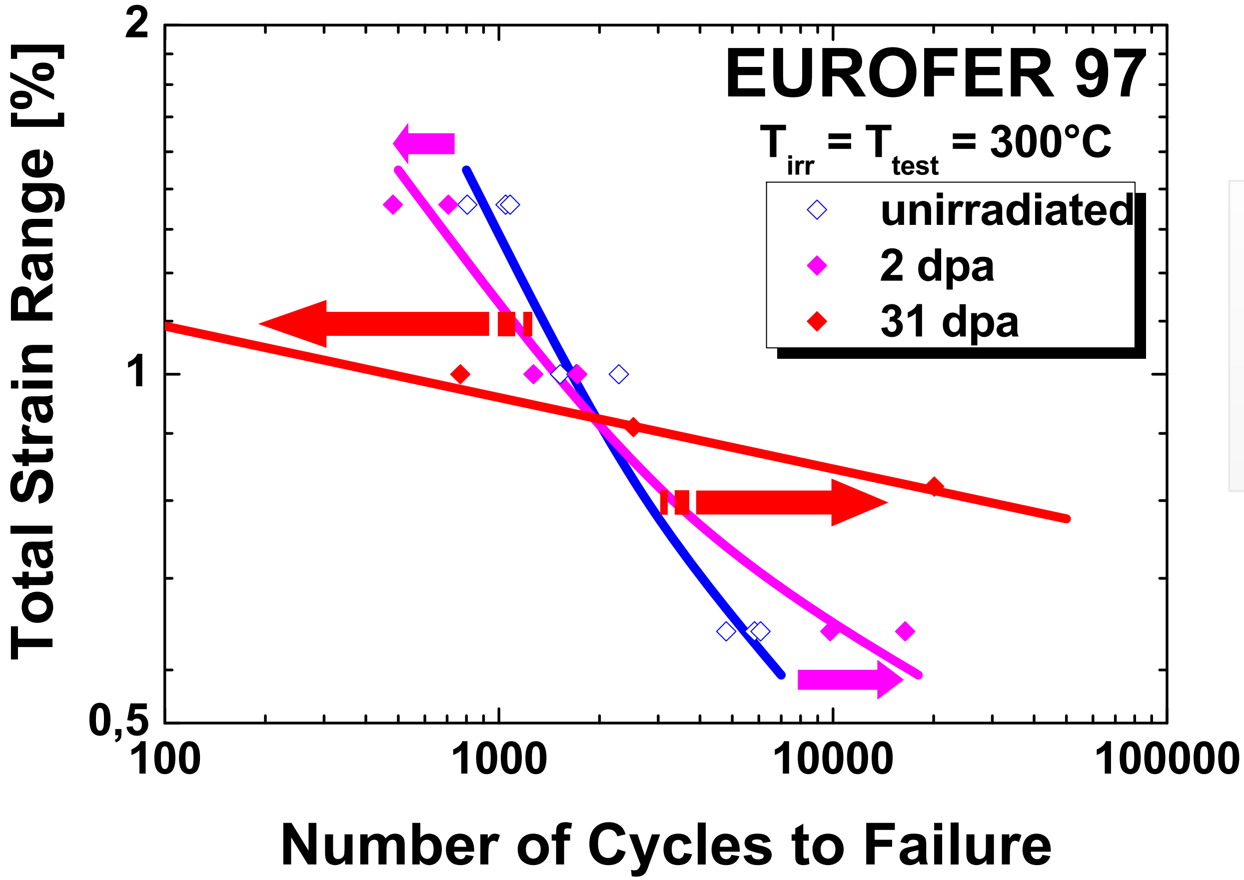
The superiority of RAFM steels:

- Low activation capability
- low swelling, high thermal conductivity, low thermal expansion and better liquid-metal compatibility in comparison to austenitic steels.

Materials	Fatigue	Topic	Author
12Cr steel MANET	Strain-controlled isothermal	Influence of irradiation (T, dose) and He implantination; In-situ and post-irradiation	R. Lindau, J. Bertsch
9Cr F82H mod.	Strain-controlled isothermal	Fatigue behavior and the growth of microcracks after He implantination; Post-neutron irradiation Post-ion implantination	J. Bertsch T. Hirose
F82H mod. OPTIFIER IV MANET II	Thermal fatigue (TF)	Fatigue endurance of RAFM steels under cyclic strains and stresses produced by temperature changes	C. Petersen
EUROFER 97	Isothermal; TF; Multiaxial fatigue; multi-step loading; Fatigue-creep interaction	Cyclic softening; Modelling of high temperature damage under TF loading; Creep-fatigue interaction	C. Petersen J. Aktaa C. Vorpahl

Fatigue behavior of EUROFER 97

Alloy	C	Si	Mn	Cr	V	W	Ta
EUROFER 97	0.12	0.06	0.42	8.87	0.19	1.1	0.14

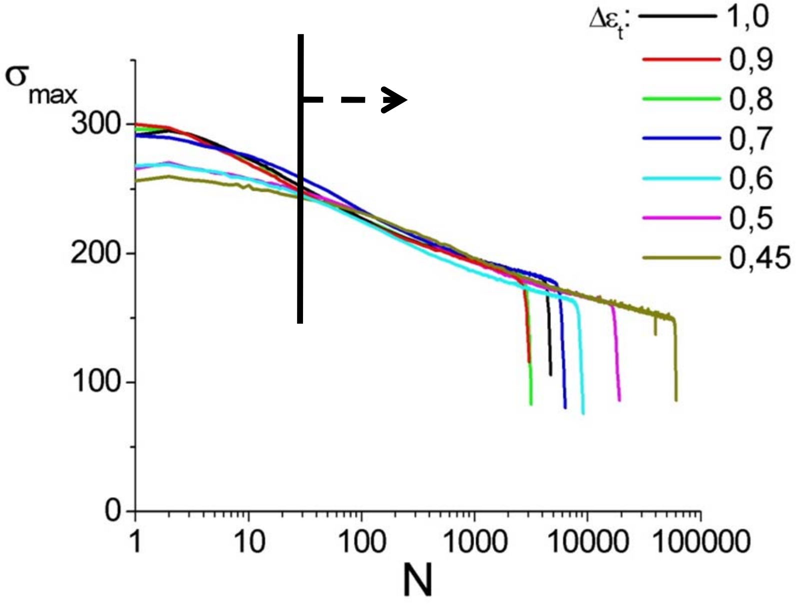


C. Petersen et al., J. Nucl. Mater. (2009)

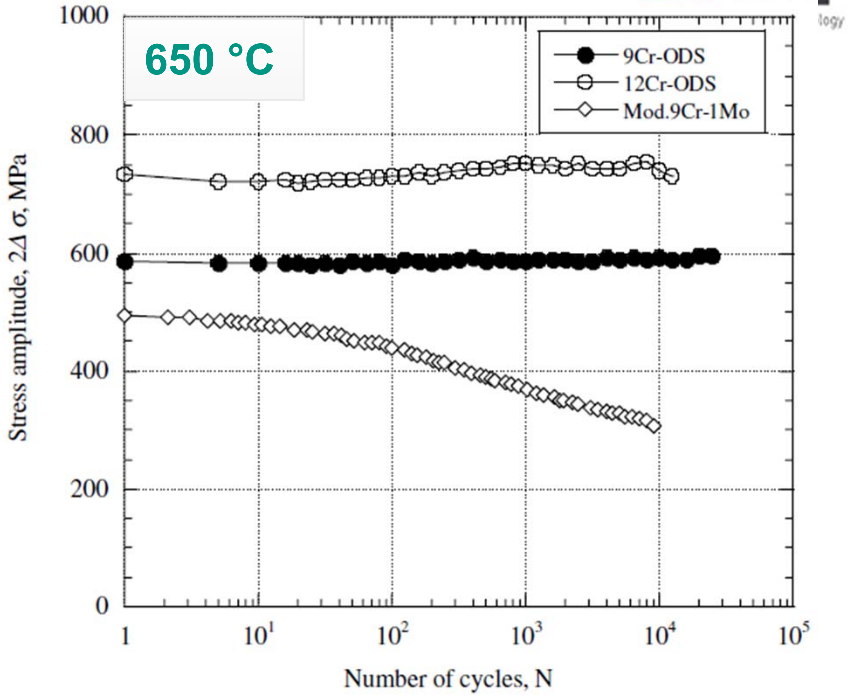
- Above $\Delta\varepsilon_t \approx 0.9\%$: Lifetime reduction
- Below $\Delta\varepsilon_t \approx 0.9\%$: Lifetime extension

Cyclic softening

EUROFER 97, 550 °C



C. Vorpahl et al., J. Nucl. Mater., 2011, p16



S-N curve for 9Cr–ODS and 12Cr–ODS steels and Mod. 9Cr– 1Mo steel at 0.5% total strain at 923 K
Ukai et al., J. Nucl. Mater. (2007)

- Pronounced cyclic softening (> 100 MPa)
- beyond N~50 σ_{max} becomes independent of $\Delta\varepsilon_t$

→ lifetime limiting crack initiation & network formation occurs before 100 cycles

- No evident cyclic softening for 9Cr-ODS and 12Cr ODS steels.
- Ferritic ODS steels enable higher operational temperature.

The objective of this work

- Developing RAF ODS steels by optimization of production route
  - powder metallurgy + thermomechanical processing (TMP)

No.	Cr	W	Ti	Y_2O_3	Remarks
K9	13.5	1.1	0.3	0.3	**1 kg**, HIP + TMP
K14	13.5	1.1	0.3	0.0	**1kg**, HIP + TMP

- Exploring structure-property correlation
  - Strain-controlled low cycle fatigue tests at 550 °C
  - Structure evolution before and after fatigue tests

    -- grain structure

    -- precipitates and oxide particles

    -- dislocation density

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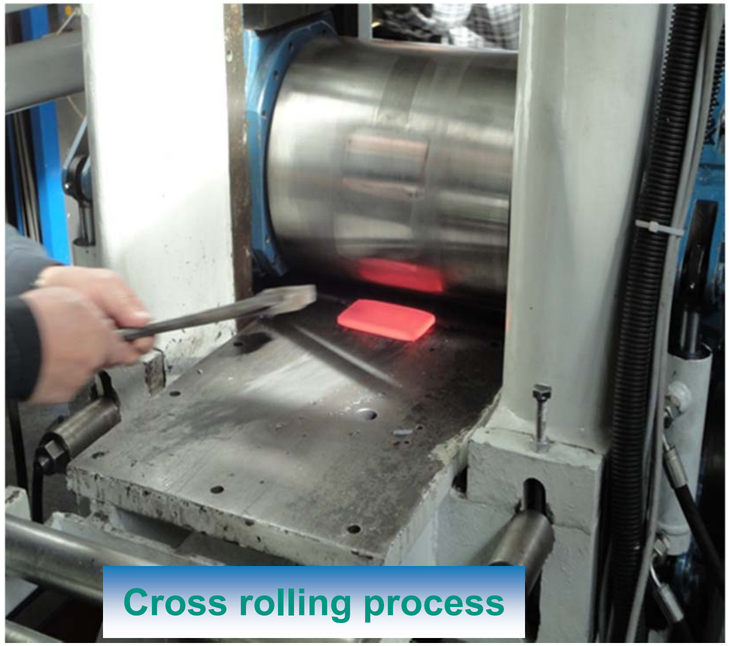
Fabrication process


milling container

ZOZ Simoloyer CM01


Capsules after HIP


Cross rolling process

- MA: under H2, 1000/4'/700/1'/24h, Glove-Box
- HIP: 1150°C, 100 MPa, 2.5h
- TMP: 3 passes cross rolling at 1100°C + Annealing at 1050°C, 2h, Vacuum

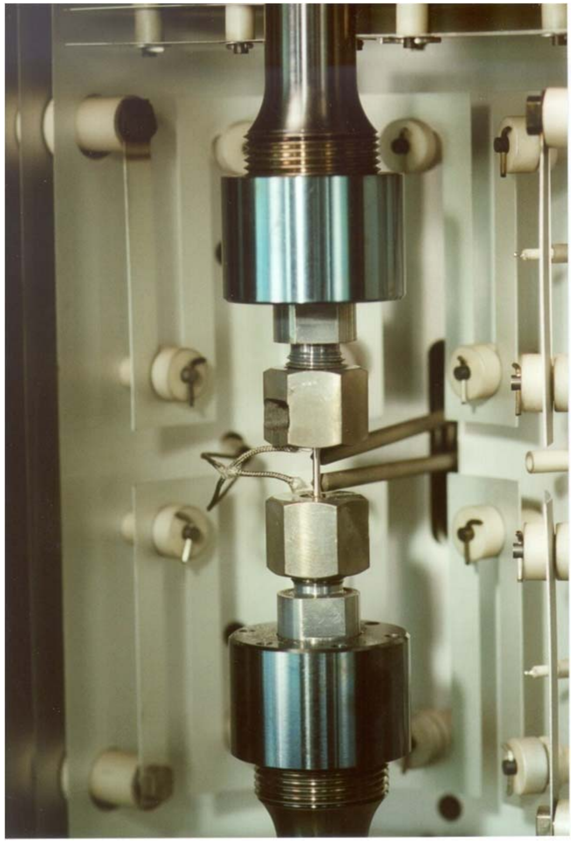
Fatigue sample and Universal testing machine

LCF Specimen

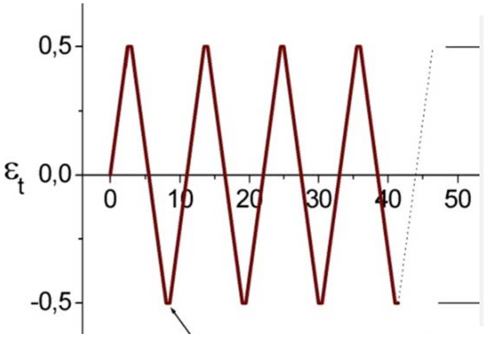
- Deformation volume:
  - Length 7.60 mm
  - Diameter 2.00 mm
- Total specimen length: 27.0 m
- Cylindrical gauge length

UTM with vacuum furnace

T = 550 °C



fatigue



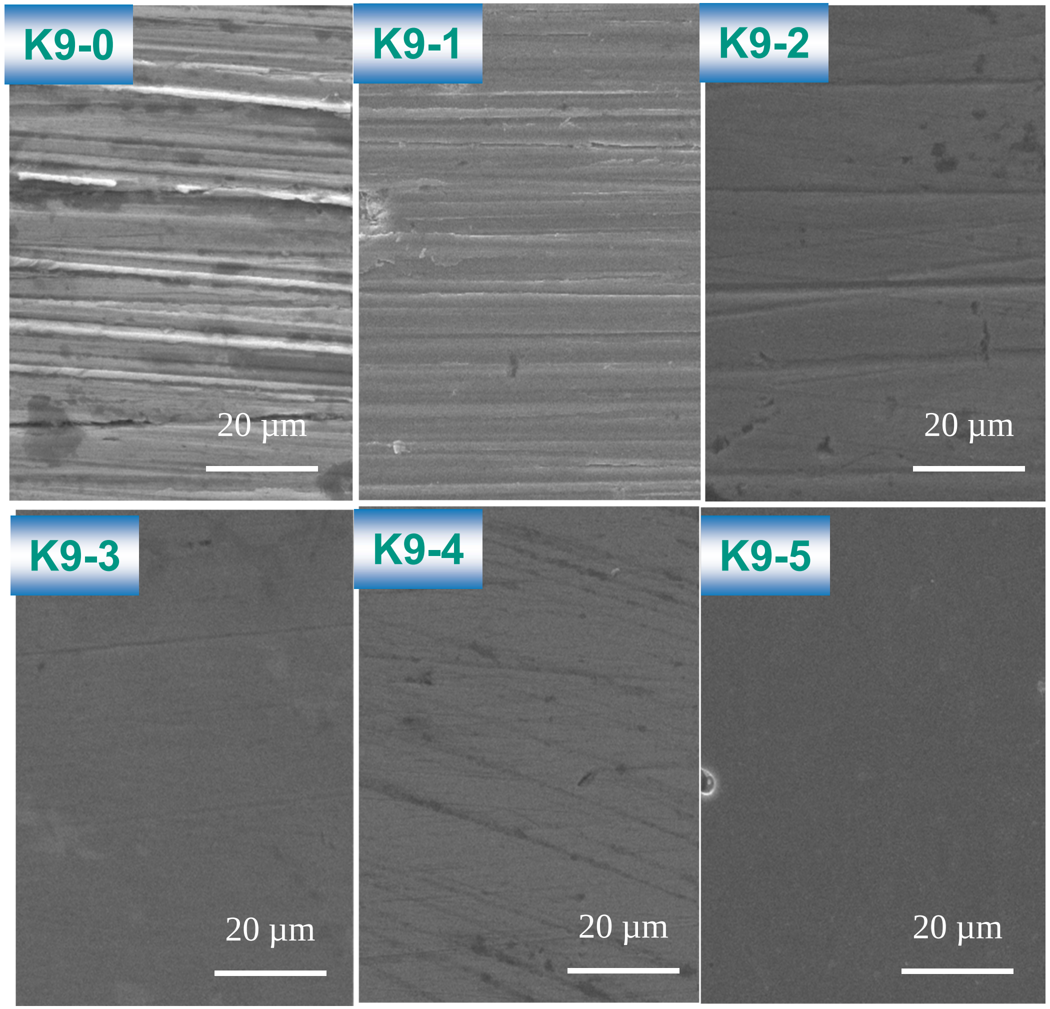
Fatigue sample geometry and preparation

Mechanical polishing procedures

No.	Abrasive size [µm]	Remarks
1	18.3	Grinding paper
2	15	Thread and diamond paste
3	9	
4	6	

Electro polishing procedure

No.	Electrolyte	Voltage
5	20 % H_2SO_4+ 80 % CH_3OH	12 V



- **The surface conditions of fatigue samples are evidently improved.**

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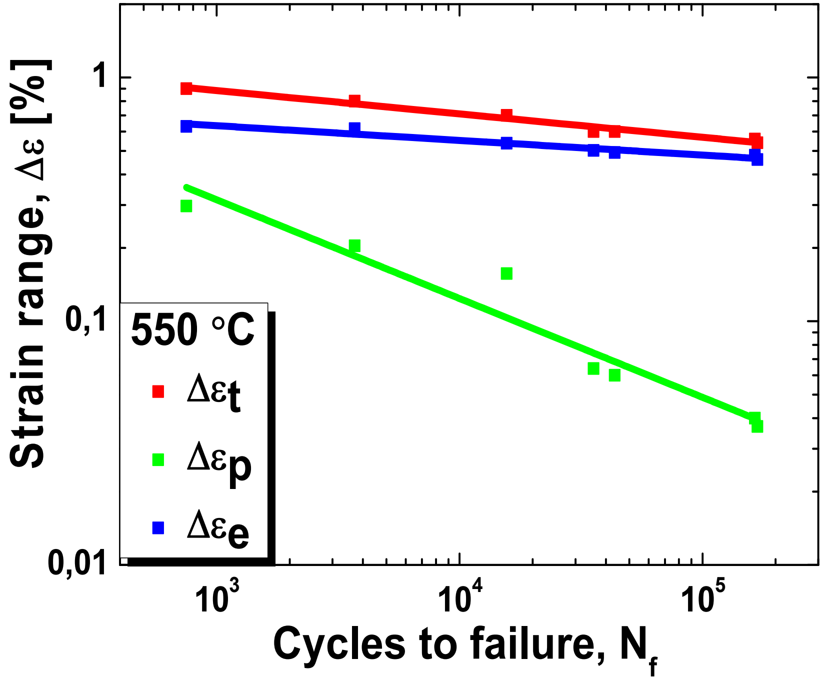
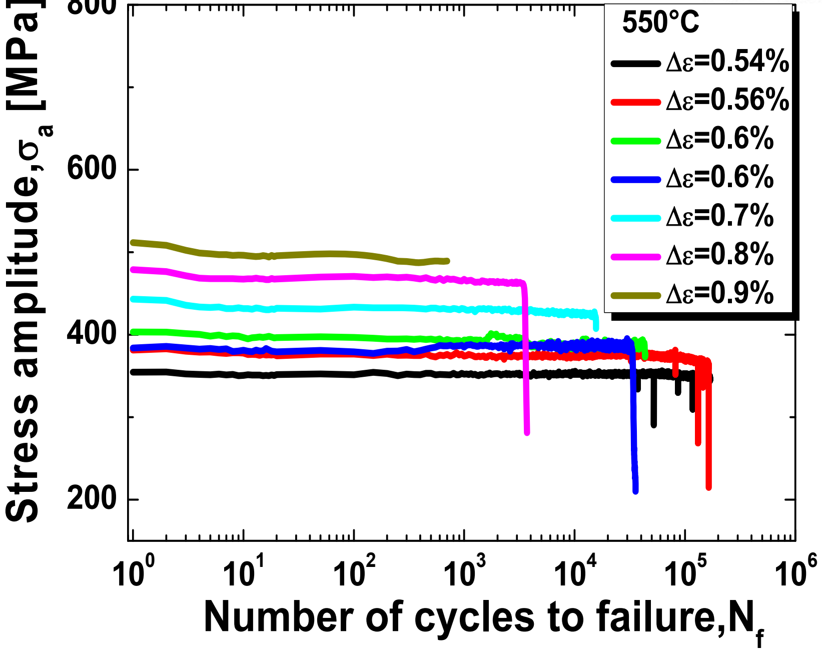
Fatigue results for 13.5Cr1.1W0.3Ti ODS steel (TMP)





- Simple linear relationship for total strain and elastic strain and plastic strain as a function of N_f.

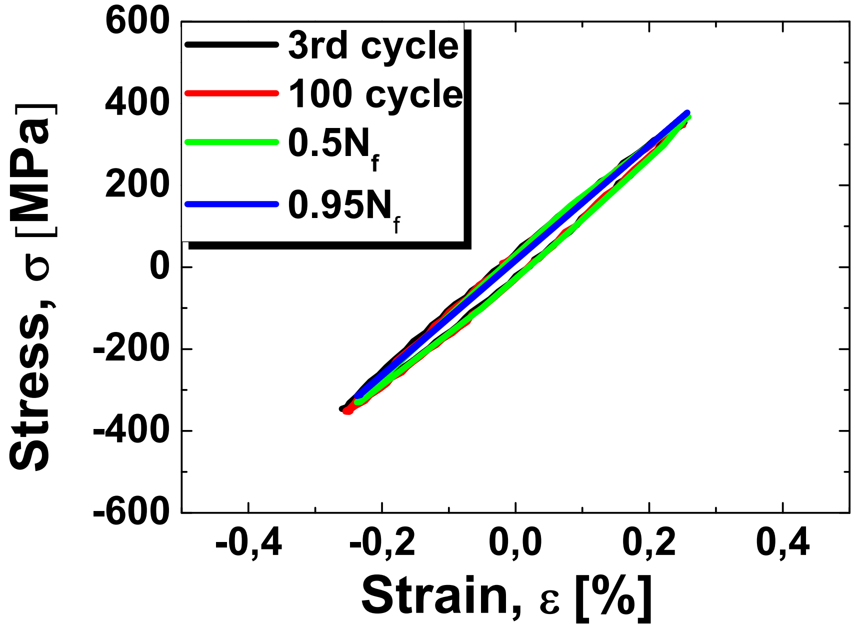
- S-N curves reveal constant stress during the whole test irrespective of the strain range

  (no cyclic softening)

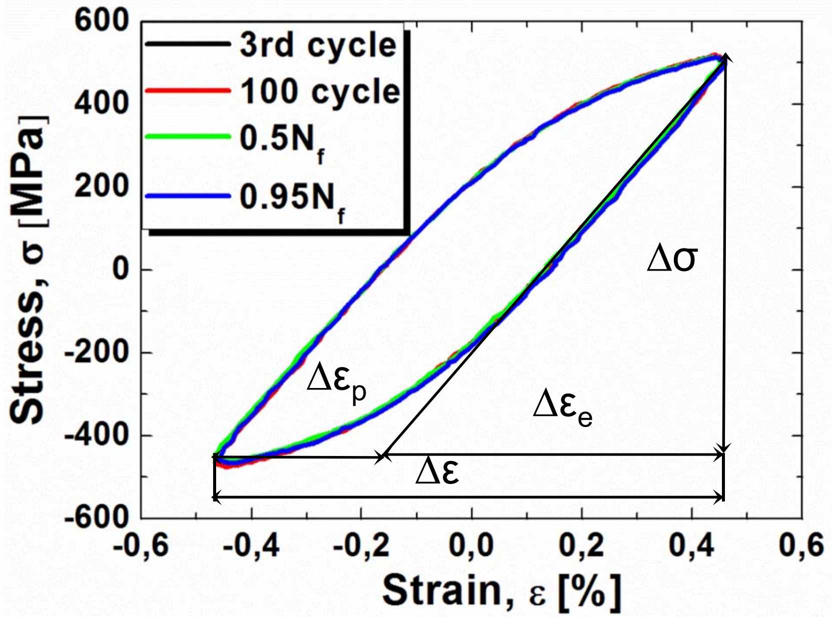
$\Delta\varepsilon_t$	σ_a [MPa]	N_f
0.54%	350	900
0.9%	520	$1.7*10^5$

Fatigue results for 13.5Cr1.1W0.3Ti ODS steel (TMP)

$\Delta\varepsilon_t \approx 0.54$ %, T=550°C



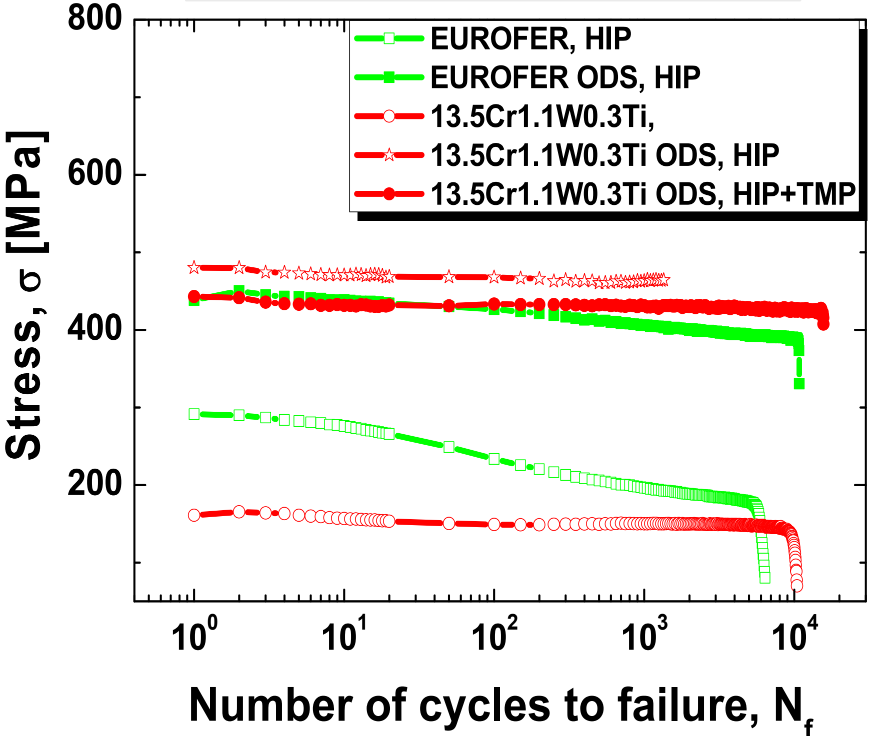
$\Delta\varepsilon_t \approx 0.9$ %, T=550°C



- The stress stability of 13.5Cr1.1W0.3Ti ODS ferritic steel is supported by hysteresis loops.
- Superposition of hysteresis loops suggest no cyclic softening or hardening
- At $\Delta\varepsilon_t \approx 0.54$ %, slight plastic deformation; at $\Delta\varepsilon_t \approx 0.9$ %, plastic deformation ≈ 0.3%

Fatigue results

$\Delta\varepsilon_t \approx 0.7$ %, T=550°C



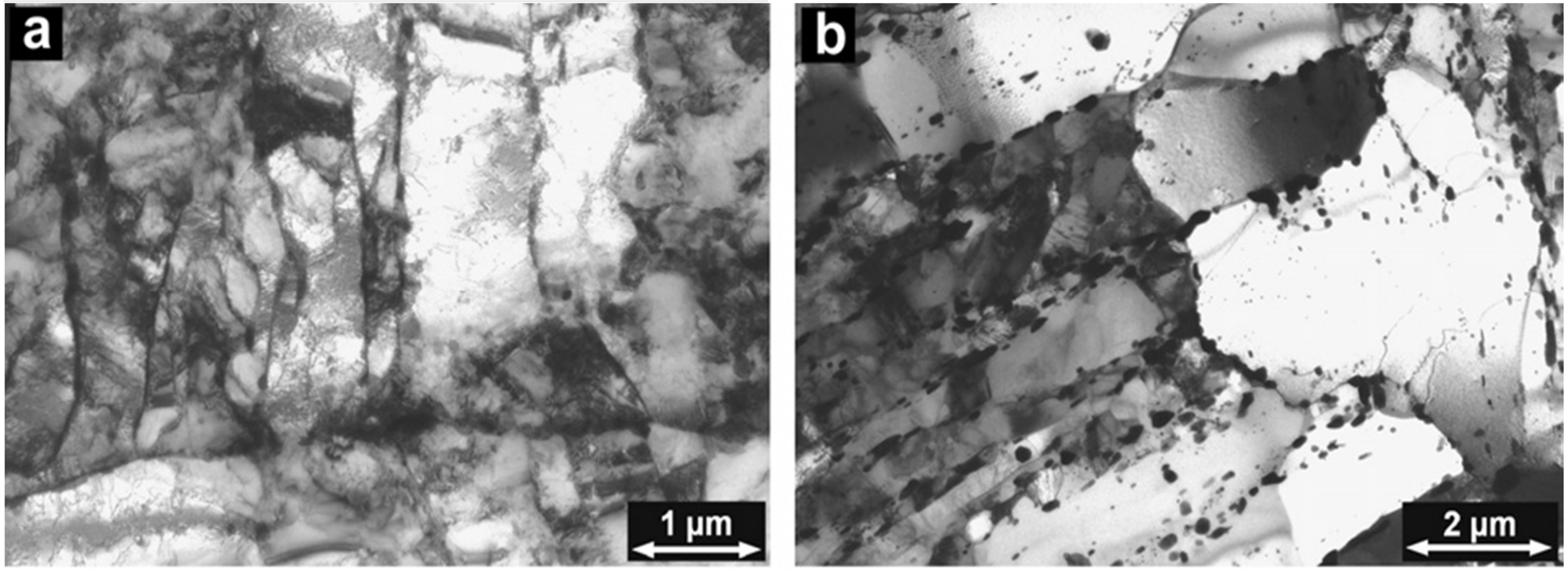
- RAFM steels (EUROFER):

  pronounced cyclic softening
- 13.5Cr ferritic ODS steel (TMP):

  - outstanding fatigue resistance
  - constant stress

Microstructure evolution for EUROFER 97 at 550 °C

As-recieved | **$\Delta\varepsilon_t$ = 0.45% after 60,850 cycles**

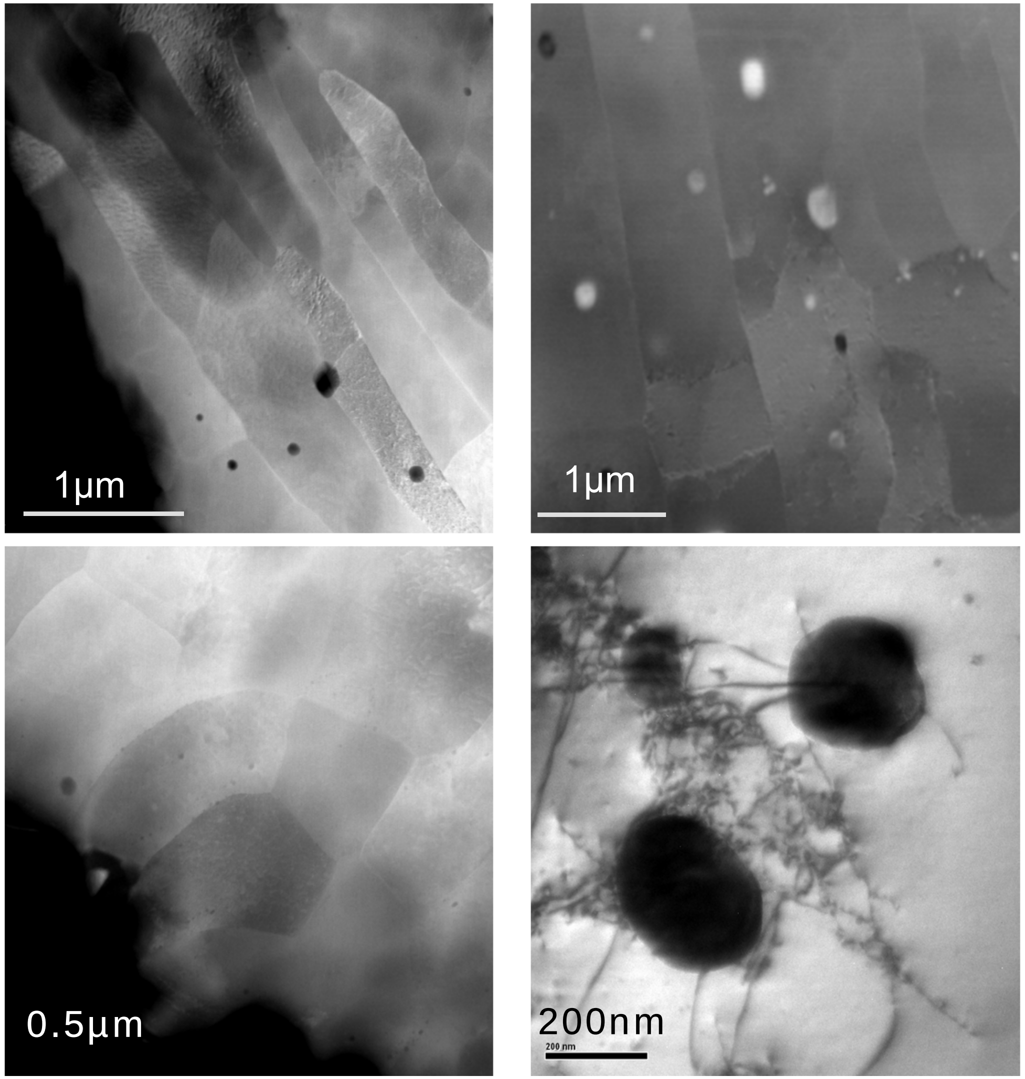


C. Vorpahl et al., J. Nucl. Mater., 2011, p16

Cyclic softening (100 MPa) can attributed to

- Formation of coarse precipitates at GB
- Dramatic reduction of dislocation density

Microstructure evolution for 13.5Cr ODS steel



As recieved

$\Delta\varepsilon_t$=0.9%, after failure

As received

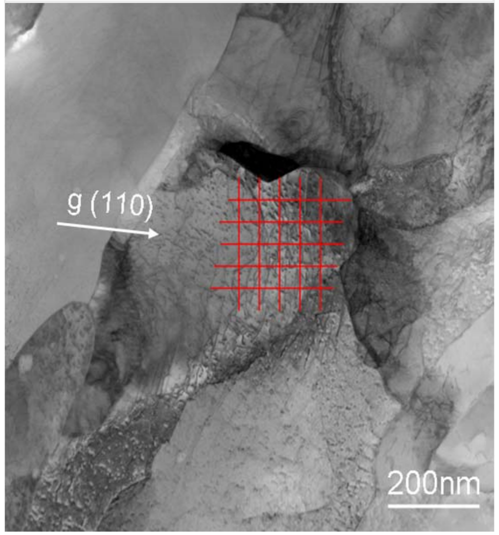
- Elongated grains retained from TMP
- Nanoscale equiaxed grains
- Random distribution of Ti oxides

After LCF test

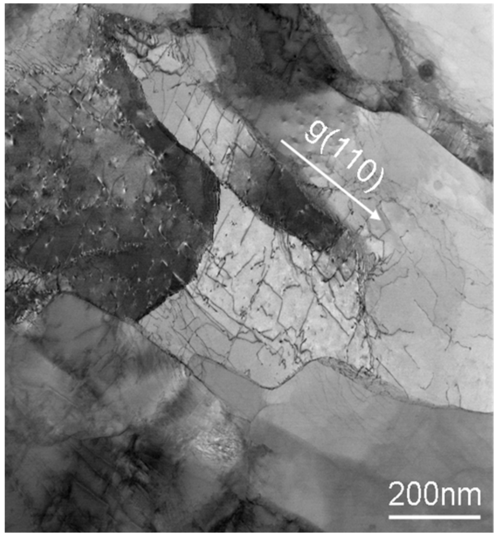
- Stable grain structure
- Multiple dislocation-precipitate (Ti oxide) interaction

Dislocation density

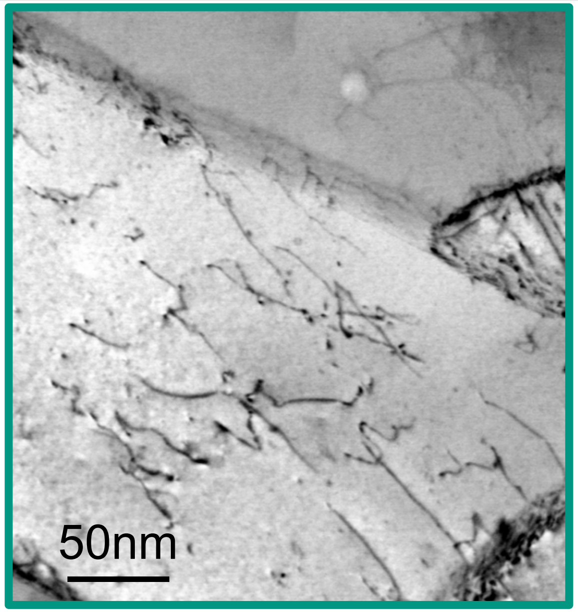
As recieved



$\Delta\varepsilon_t$=0.9% after failure



Particle-dislocation interaction



$$\rho = \frac{2n}{Lt}$$

n: intersection number of the gridlines and dislocations;

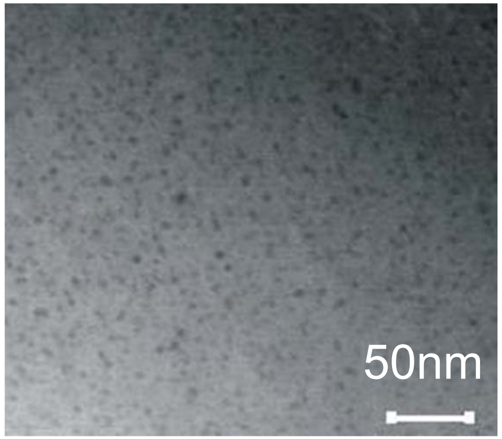
L: length of the gridlines;

t: thickness of the sample

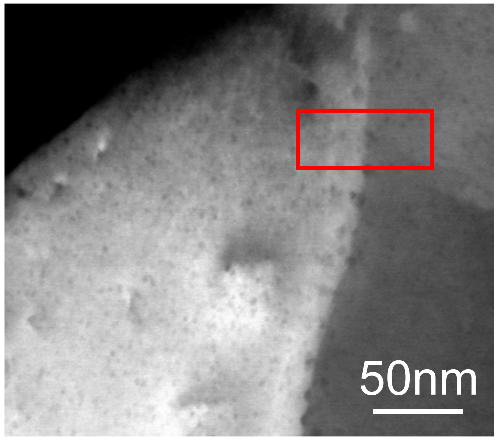
Measurement	**Dislocation density before LCF [m^{-2}]**	**Dislocation density after LCF [m^{-2}]**
1	1.15×10^{15}	7.0×10^{14}
2	4.21×10^{14}	4.77×10^{14}
3	7.56×10^{14}	8.05×10^{14}
Average	**7.75×10^{14}**	**6.6×10^{14}**

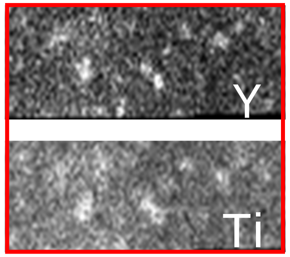
ODS particles

As received



$\Delta\varepsilon_t$=0.9%, after failure



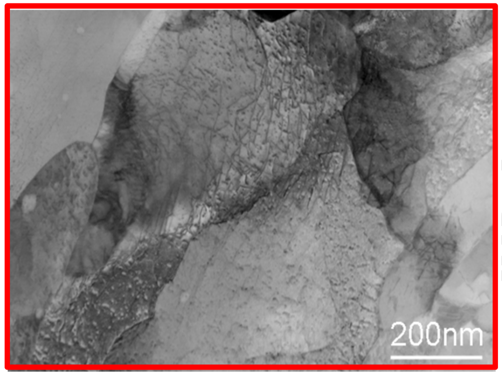


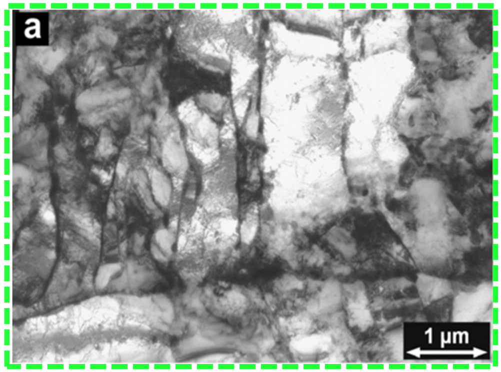
Y-Ti oxides

d ≈ 5 nm

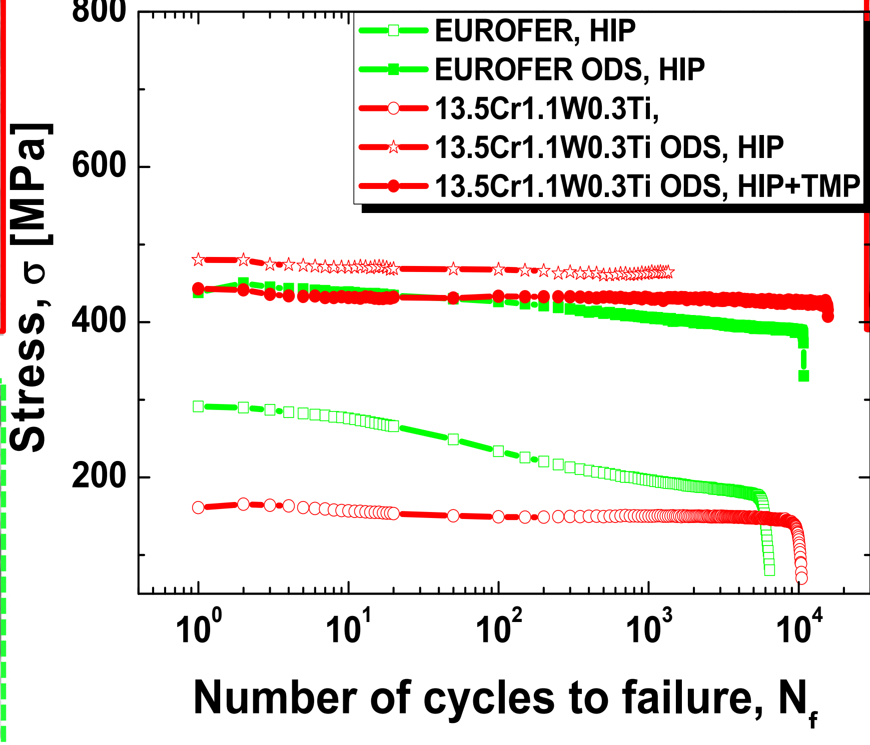
Fatigue-Microstructure correlation

Before LCF test

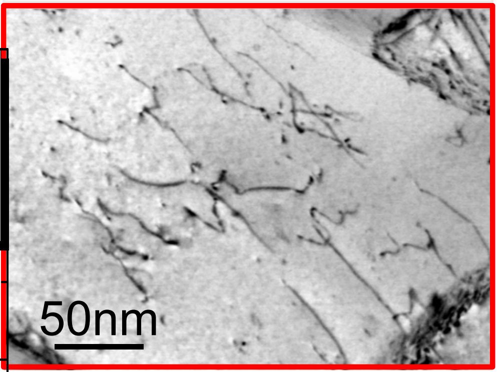


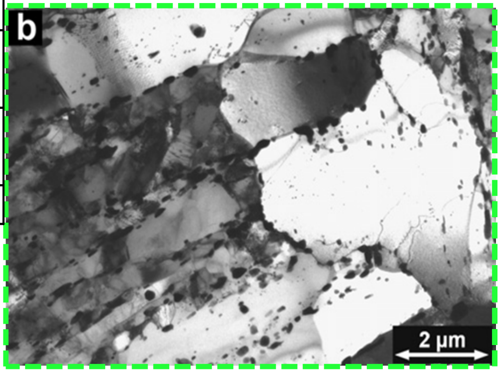


$\Delta\varepsilon_t \approx 0.7$ %, T=550°C



After LCF test





Stablized nanoscale dispersoids

→

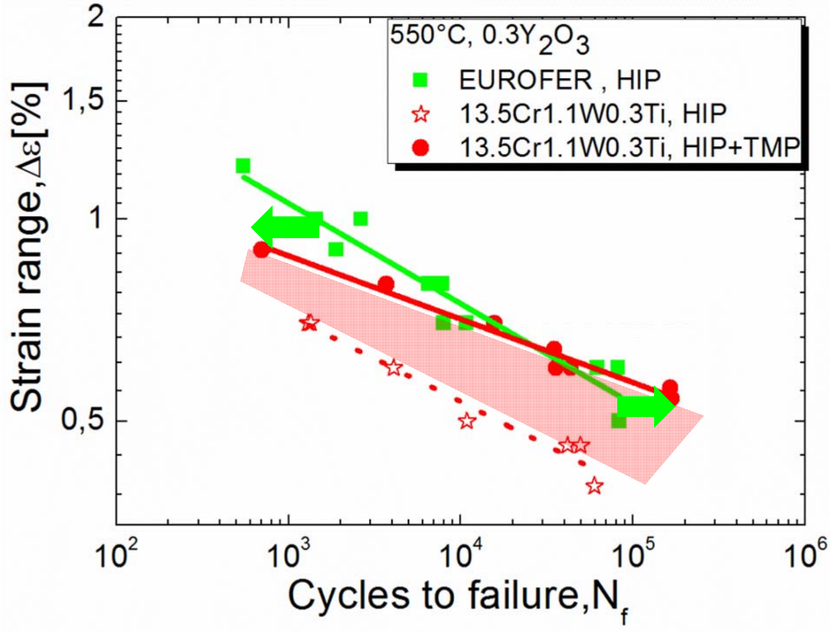
Ultra stable microstucture

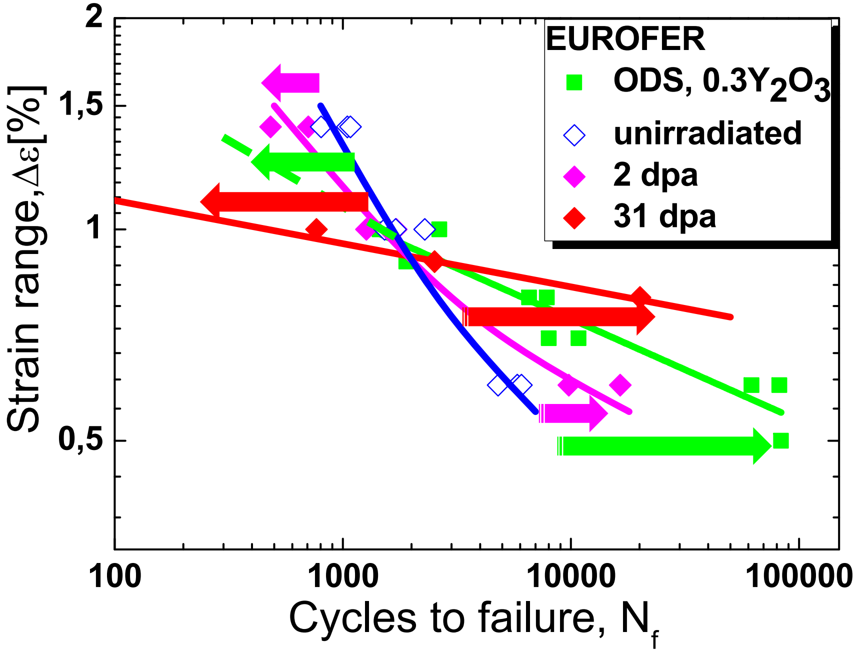
(grain, dislocaiton density)

→

Outstanding fatigue resistance for 13.5Cr ODS steel after TMP

Fatigue-Microstructure correlation





- HIP vs. HIP+TMP

  -10 time lifetime prolongation at $\Delta\varepsilon$=0.7% after TMP

- 13.5Cr1.1W ODS (TMP) vs. EUROFER ODS

- Above $\Delta\varepsilon_t \approx 0.7$ %: Lifetime reduction
- Below $\Delta\varepsilon_t \approx 0.7$ %: Lifetime extension

- Critical strain range is dependent on the materials (0.7% or 0.9%).

- high strain regime, shorter lifetime

  → accelerated crack initiation due to irradiation hardening or oxide dispersion strengthening.

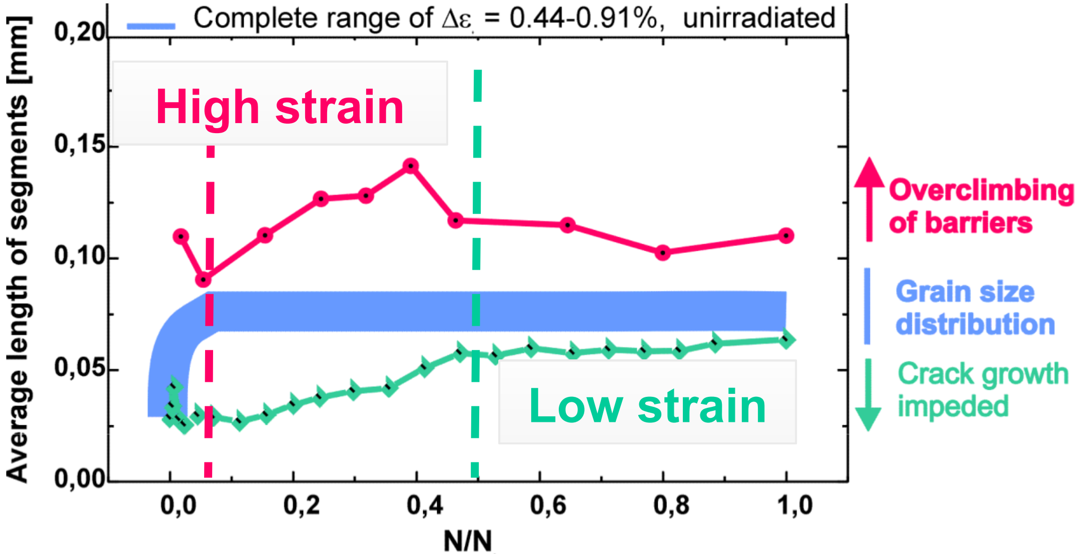
- low strain regime, prolonged lifetime

  → Micro-crack growth impeded by irradiation defects or oxide particles

Fatigue-Microstructure correlation

$$K_{Ic} = \sigma\sqrt{\pi a}$$

Very early crack network formation due to irradiation hardening or oxide strengthening

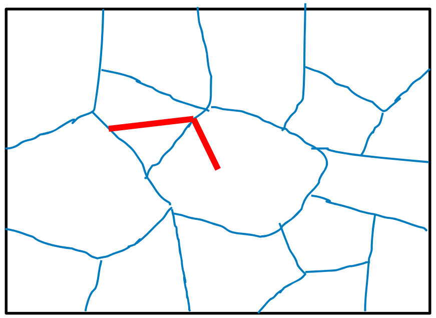


J. Bertsch et al., J.Nucl.Mater. (2000)

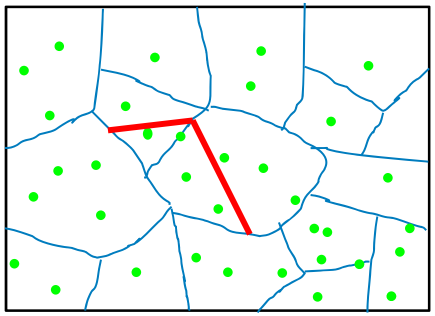
50% life time for crack segment with the dimension of grain size

RAFM steel:
Low strength

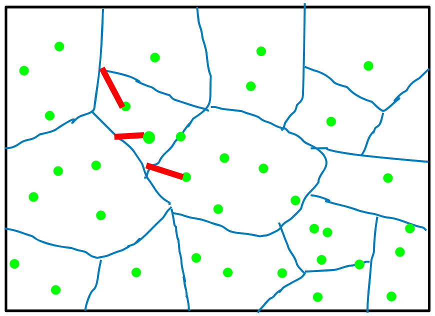
ODS steel or irradiated steels:
high strength



- **Grain boundary**
- **Crack**
- **Irradiation defects or ODS particles**



High strain



Low strain

Summary

- **TMP leads to a remarkable lifetime extension for 13.5Cr1.1W ODS steel.**

  → Lifetime extension with a factor of 10 to 20 when $\Delta\varepsilon_t \leq 0.7\%$.

- **EUROFER ODS vs. 13.5Cr1.W ODS steel after TMP**

  → dependent on the strain range.

  $\Delta\varepsilon_t > 0.7\%$, shorter lifetime; $\Delta\varepsilon_t \leq 0.7\%$, prolonged lifetime.

  → different microstructural barriers (oxide particles, irradiation defects or grain boundaries) and their influence on the crack growth.

- **The constant stress amplitude for 13.5Cr1.1W ODS steel irrespective of strain range**

  → stable grain structure, constant dislocation densities of 10^{14} m^{-2}.

  → highly stabilized nanoscale oxide with an average diameter of 5 nm.

Thanks for your attention!