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Materialentwicklung für Energieanwendungen mit extremen Bedingungen

Christian Linsmeier

J.W. Coenen, J. Riesch*, B. Unterberg, T. Dittmar, B. Jasper, Y. Mao, F. Koch*, A. Litnovsky, J. Brinkmann, T. Wegener

* Max-Planck-Institut für Plasmaphysik, Garching b. München, Germany

Overview

Introduction: the material challenge

Neutron damaged materials

Research towards advanced materials

- Extrinsic toughening: composite materials
- Intrinsic safety: smart alloys
- Hydrogen isotope permeation: barrier layers

Summary and outlook

Extreme loads in different technologies

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PWR fuel element

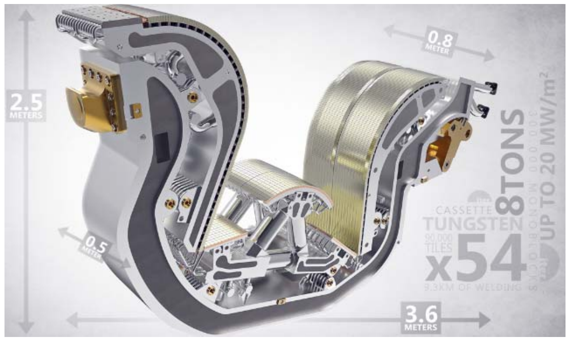
Reentry vehicle

Ariane 5 /Vulcain 2

≈ 1 | ≤ 20 | 85 | 2000

power density MW/m^2

Rolls-Royce Trent 900



ITER Divertor

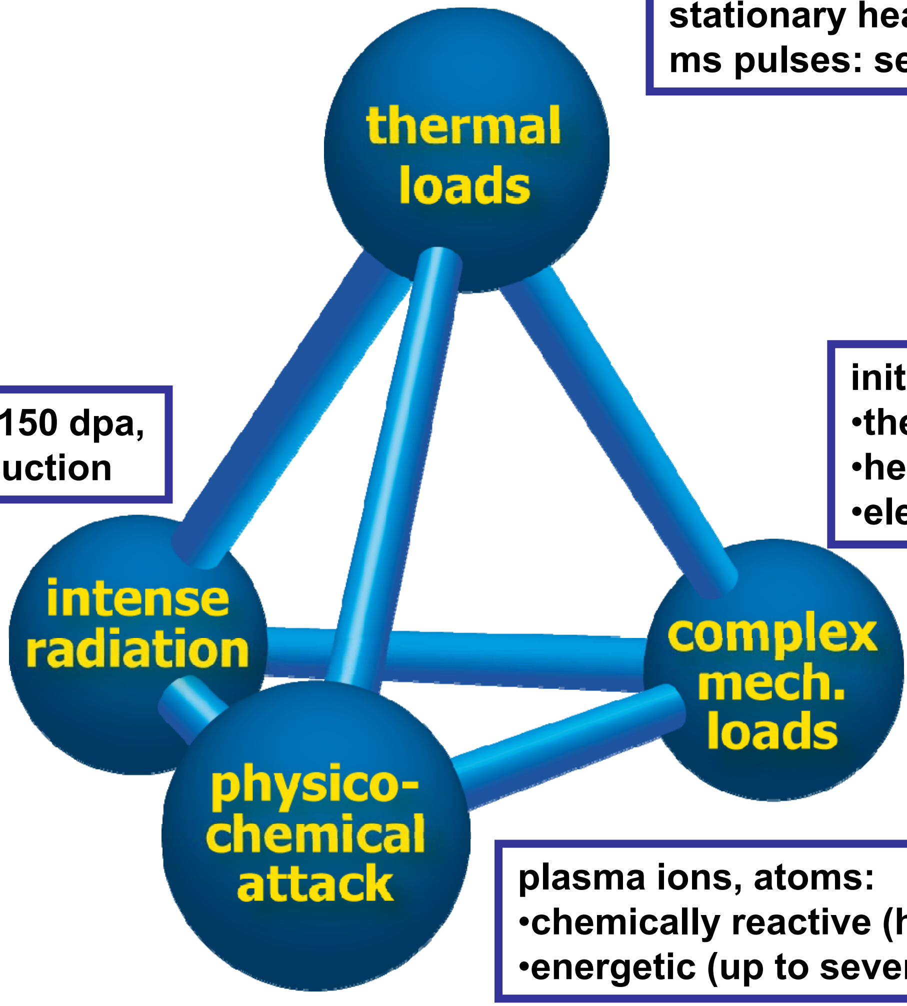
ELMs in ITER

Operational conditions

Plasma-facing materials and components are subject to extreme loads

stationary heat flux: up to 20 MW/m^2
ms pulses: several GW/m^2

reactor: up to 150 dpa,
H and He production

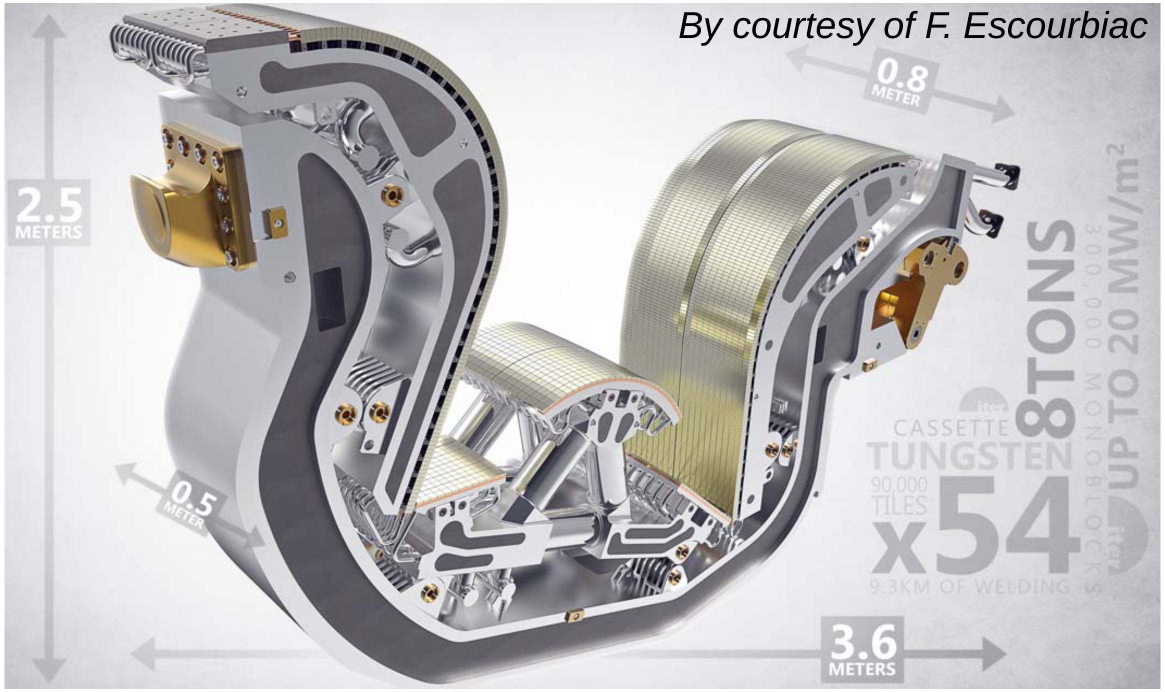


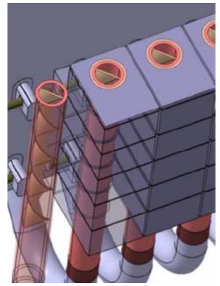
initiated through
- **thermal gradients**
- **heterogeneous material composites**
- **electromagnetic forces**

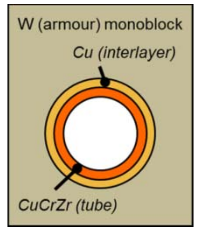
plasma ions, atoms:
- **chemically reactive (hydrogen, oxygen)**
- **energetic (up to several 100 eV)**

ITER Divertor: status

Full-W armor: one divertor until 4 years into DD/DT phase







- fully actively cooled (water, 70 °C)
- dome: flat tiles
- vertical targets (2,000): monoblocks (320,000)
- themal performance up to 20 MW m^{-2} steady-state

Monoblock technology available (JADA)

ITER Divertor: nuclear analysis

R. Villari et al., FED (2013)

Radiation damage after 4 years DD/DT operation (5.26×10^{26} neutrons)

- Steel < 0.3 dpa
- Tungsten armour < 0.1 dpa
- CuCrZr < 0.5 dpa

Helium production

- rewelding criterion: 3 appm (design: 1 appm) reached after 9 years, in critical areas acceptable

Nuclear heating

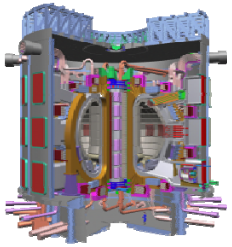
- approx. 1 MW per cassette assembly (roughly 2:1 for steel and W armour), input for thermo-mechanical analysis

Conclusion

- ITER divertor nuclear loads not critical lifetime into DD/DT operation

From ITER to DEMO

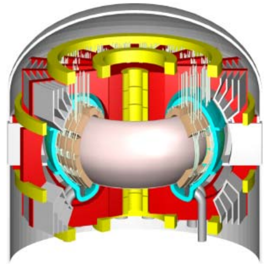
ITER



- operational flexibility (experimental device)
- transient heat flux events
- T-codeposition on „cold“ surfaces
- no energy conversion (70°C water coolant)
- low duty cycle
- low neutron dose (wall: ~1 dpa)

Need to apply available materials and technology

DEMO



- lifetime (erosion, ageing)
- very limited transient heat flux events
- energy conversion (coolant: ≥300°C water, ≥400°C He)
- high duty cycle
- high neutron dose (wall: 80…100 dpa divertor: 2-5 dpa/fpy)
- low activation materials

Need for innovation in non-activating materials and technology

DEMO divertor challenges

Requirements

- High power handling capability: 10-15 MW m^{-2} in steady-state (20 MW m^{-2} in slow transients)
- Erosion resistance: low sputtering

Proven divertor concept: W monoblock design, water cooling at elevated temperatures (e.g. PWR: 300 °C)

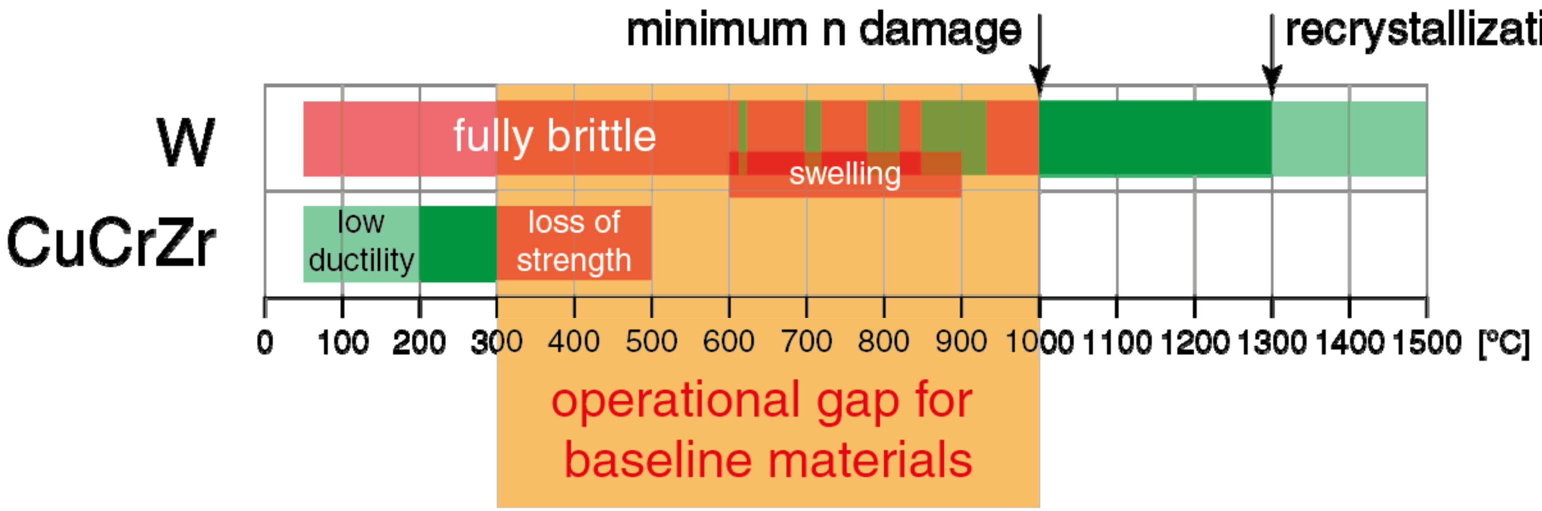
Neutron loads

- Tungsten armour 2 - 4 dpa/fpy
- CuCrZr alloy tube 2.5 - 5 dpa/fpy

Consequences

- Reduction of thermal conductivity
- Embrittlement (defects, He production)

Baseline materials: Operational gap



Tungsten

- Operation mostly within brittle regime
- W→Re transmutation increases brittleness after irradiation

Cu alloy (heat sink, cooling tube)

- narrow optimum regime
- loss of strength above 300 °C

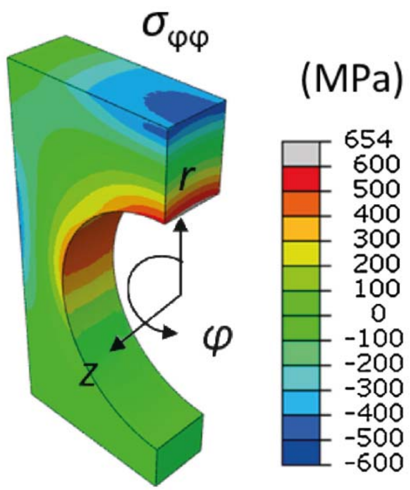
New material solutions: Metal-matrix composites

CuCrZr SiC fiber / Cu matrix
W fiber / Cu matrix

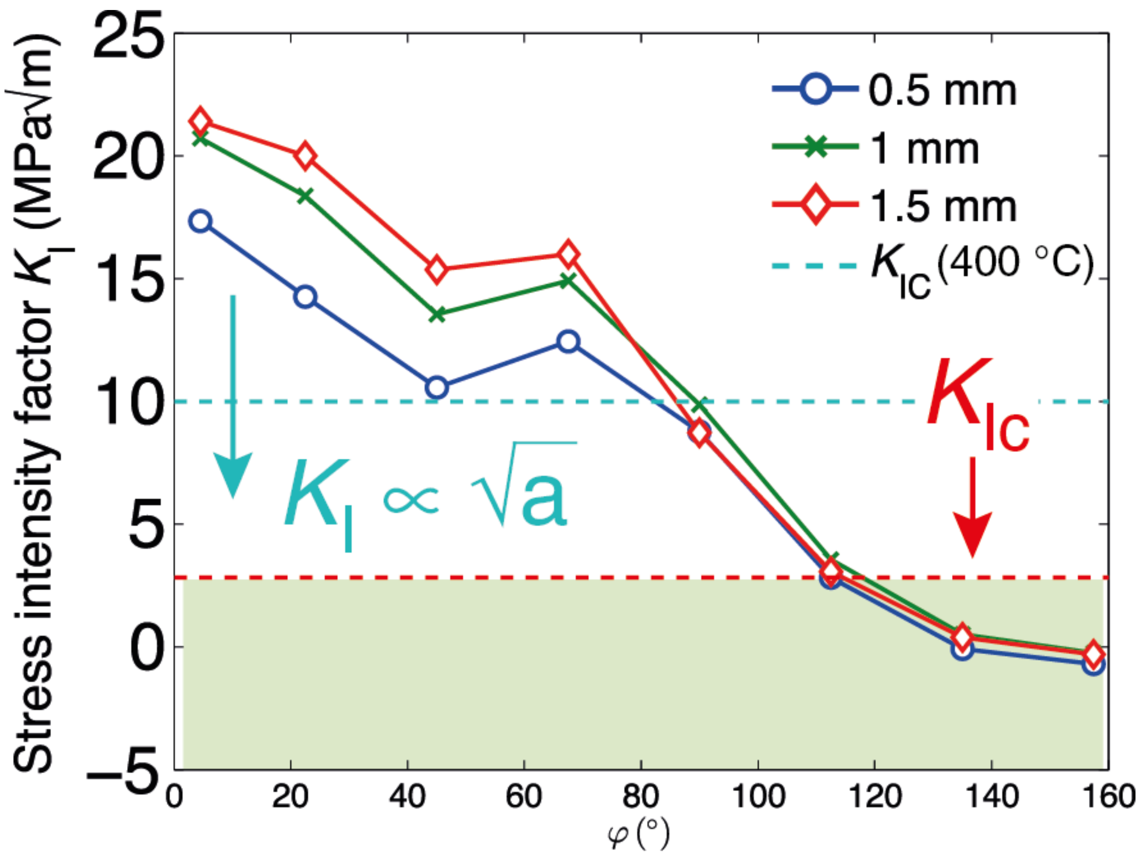
Tungsten: W fiber / W matrix

Quantitative criterion: Fracture toughness

Li & You, FED (2014)



FE Simulation:
15 MW m^{-2}
$T_{coolant}$: 200 °C



Solution for tungsten materials:

Increase fracture toughness by extrinsic energy-dissipating mechanism!

PMI and material aspects in fusion research

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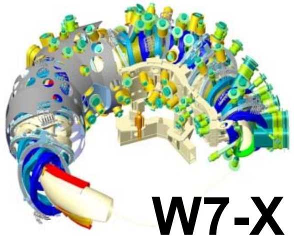
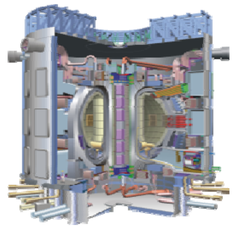
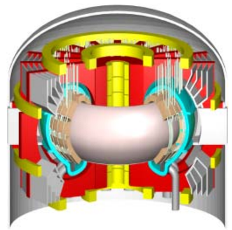
current fusion experiments →



W7-X →



ITER →



DEMO

Wall materials

- tungsten as first wall
- C+Be+W material mix
- chemical erosion
- impurities and transport
- hydrogen inventory

Stationary operation

- actively cooled wall components
- bonding technologies (W, C / Cu, steel)

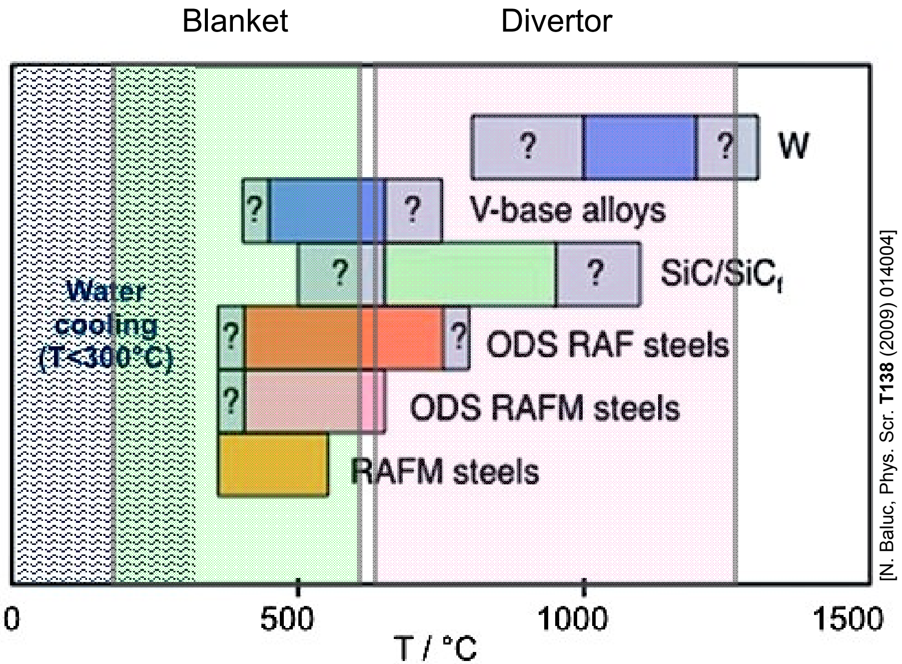
Tritium inventory

- permeation barrier layers
- functional coatings

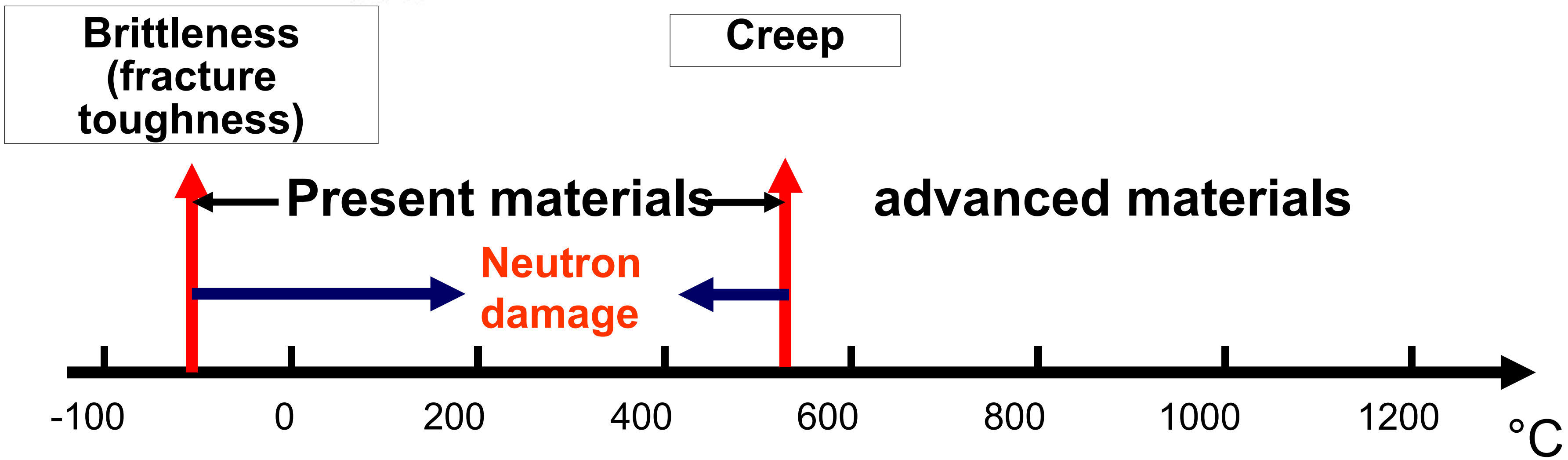
Reactor operation

- high power heat sinks
- neutronen damage
- operational safety

New material approaches needed



- Conventional materials are limited mostly by their parameters in addition to engineering approaches
- New materials should consider limits and mechanisms to improve on limits (composites, FGM) - think component!



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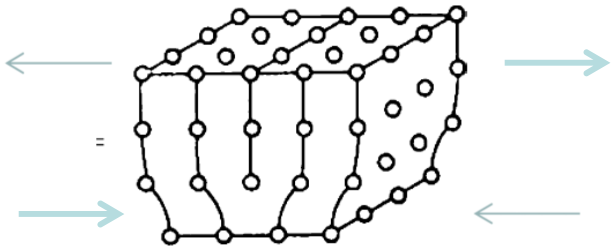
Effects of neutron irradiation

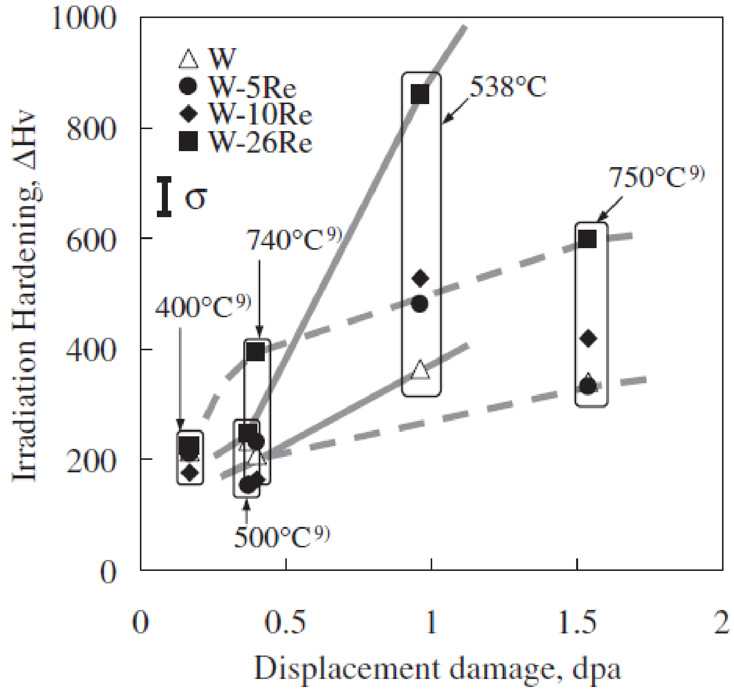
atomistic	thermo-mechanical	component	operational
Defect production and migration Transmutation, He production	Hardening and deformations Fracture and embrittlement Creep, swelling surface modifications Precipitation and segregation He embrittlement	Crack formation/ enhanced erosion, melting Brittle destruction / dust formation Fuel retention	Reduced life time of plasma facing components Issues for operational safety
Radiation damage event *Damage cascade* *Defect formation and diffusion* *Void and bubble formation*	*Physical and mechanical effects of radiation damage*	*Enhancement of PWI processes / material damage under heat and plasma loads*	*Relevance for nuclear fusion reactors*

Irradiation hardening

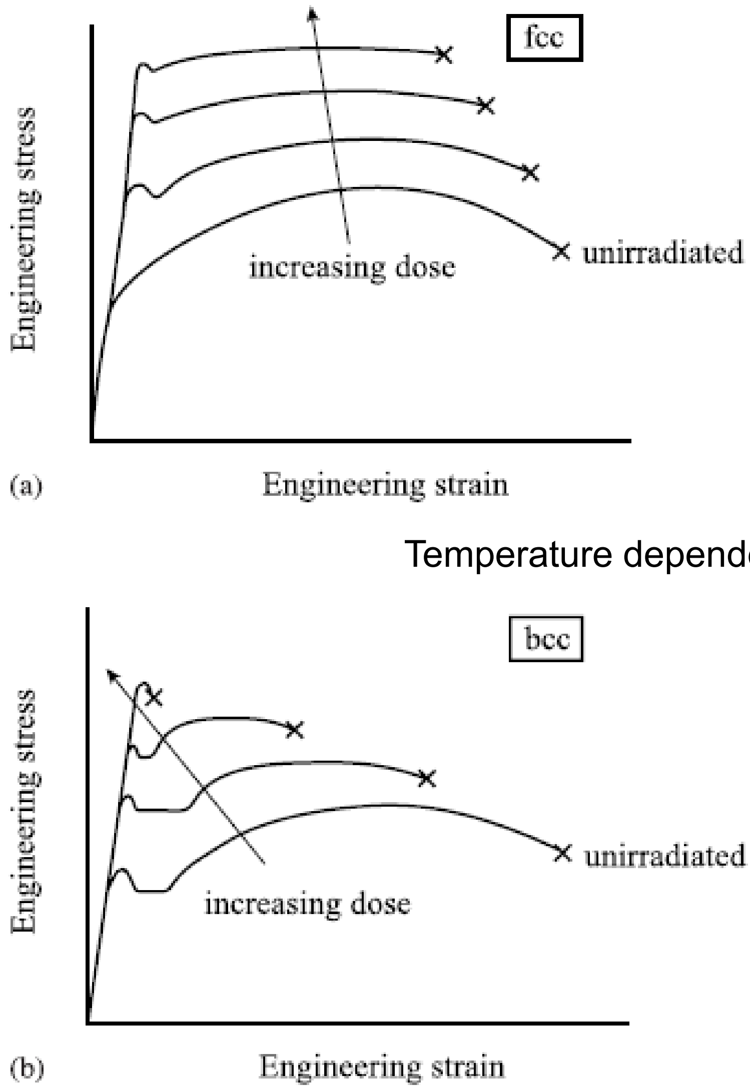
Increase in stress required to start a dislocation moving on its glide plane



Gary S. Was, *Fundamentals of Radiation Material Science, p. 268*



Tanno et al., Materials Transactions, Vol. 52, No. 7 (2011) pp. 1447 to 1451

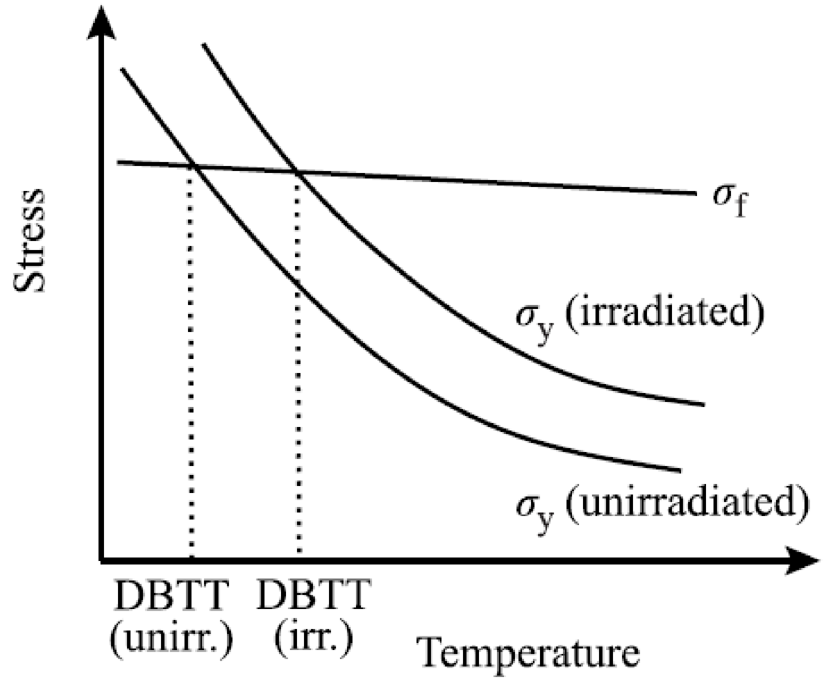


Temperature dependent!

Gary S. Was, *Fundamentals of Radiation Material Science, p. 582*

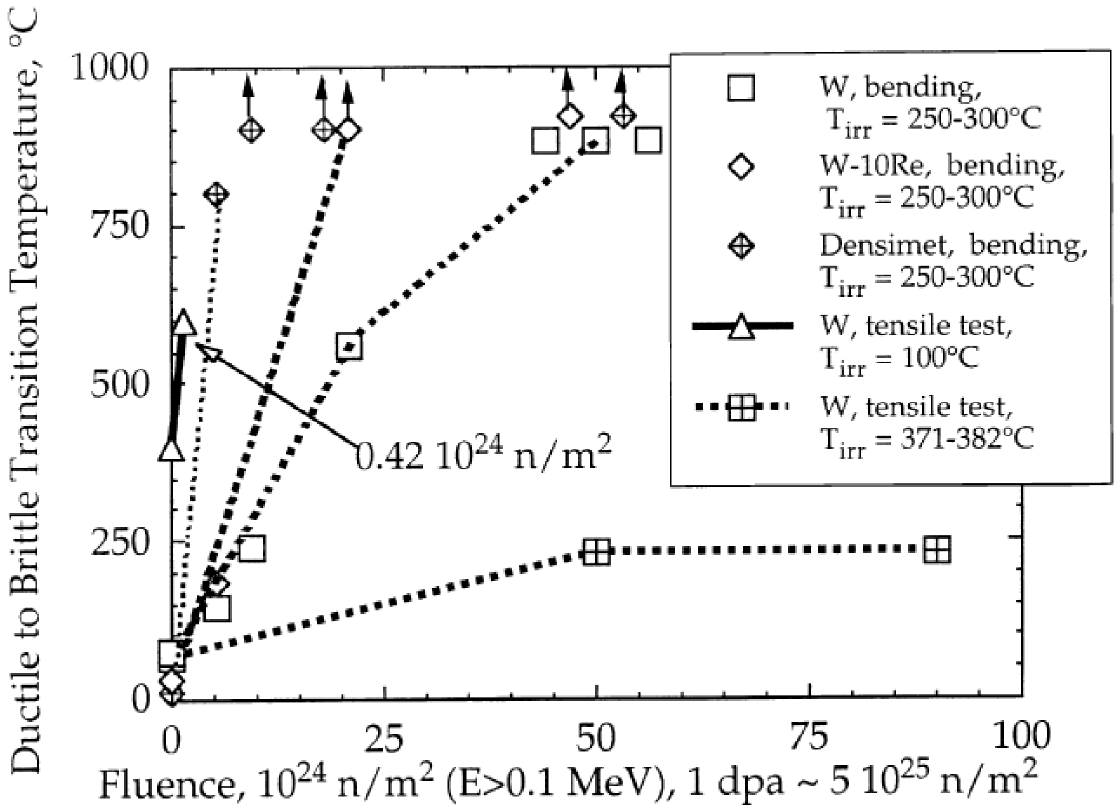
Embrittlement

- Ductile-to-brittle transition temperature (DBTT):
  yield stress σ_y equals fracture strength σ_f
- Irradiation causes DBTT to increase:
  different sensitivities of yield stress and fracture strength to neutron damage



Gary S. Was, *Fundamentals of Radiation Material Science, p. 661*

Embrittlement of tungsten

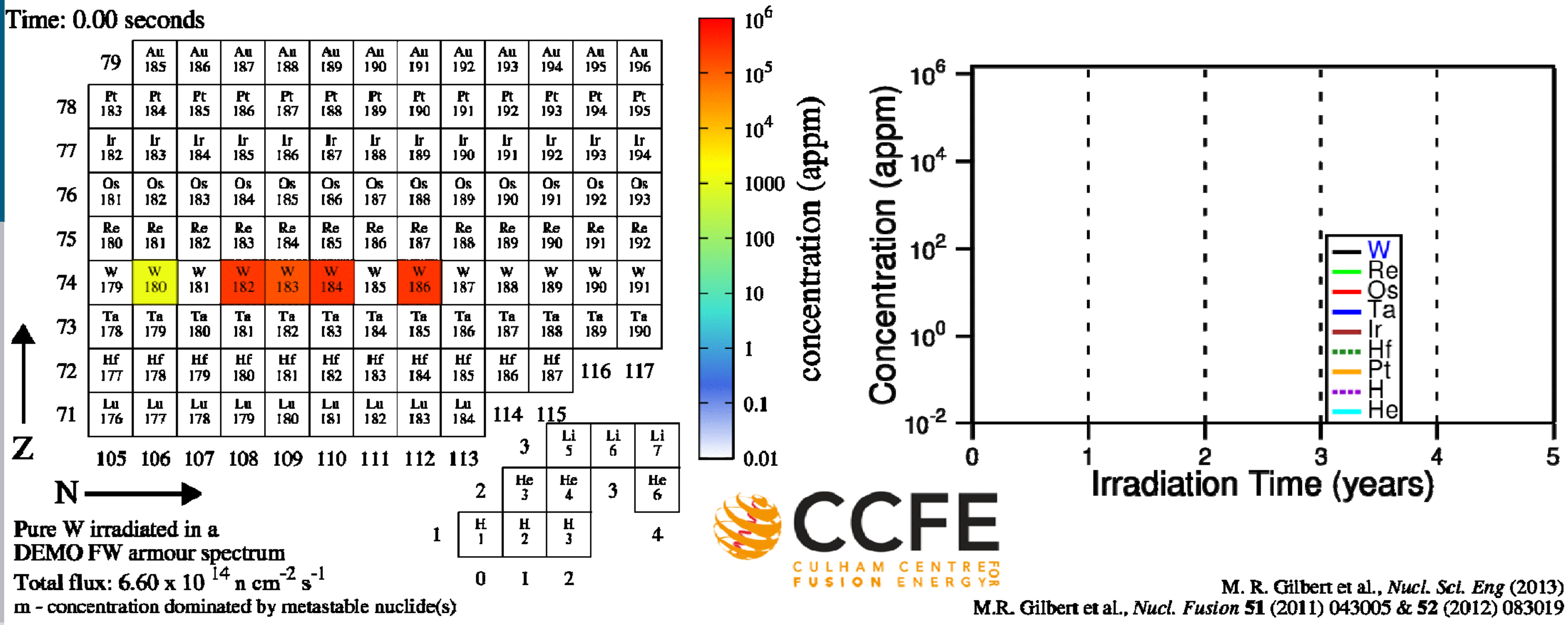


- Severe reduction of operational temperatures
- For PFMs: enhanced probability of crack formation

V. Barabash et al. / Journal of Nuclear Materials 283-287 (2000) 138-146

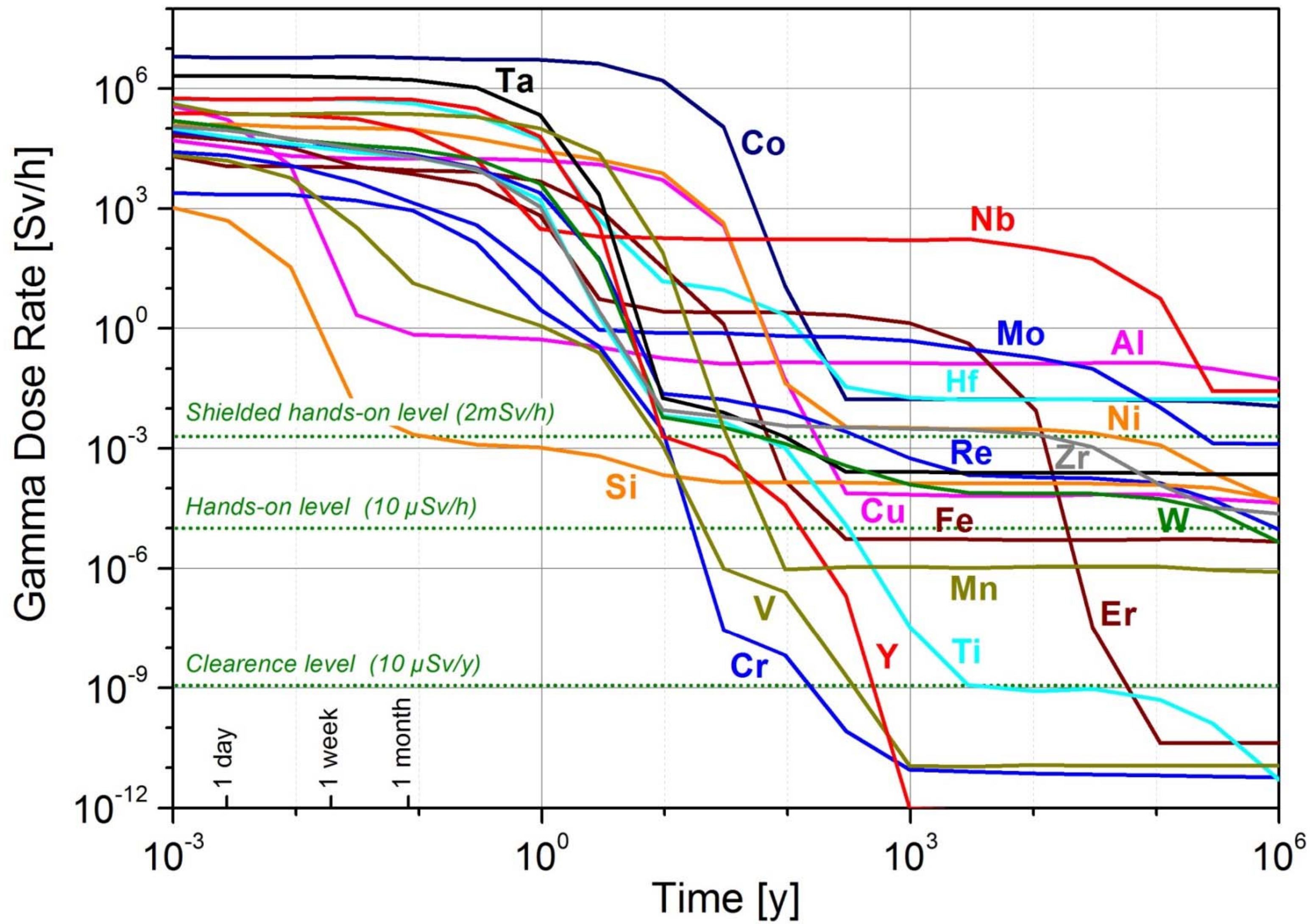
Transmutation of W in a DEMO spectrum

M. Gilbert, CCFE



- Initial material: pure W
- Exposure to neutron spectrum of a DEMO fusion reactor
- Transmutation to other elements, incl. He, Re (3.8 at%), Os (1.8 at%), Pt
- He causes grain boundary embrittlement

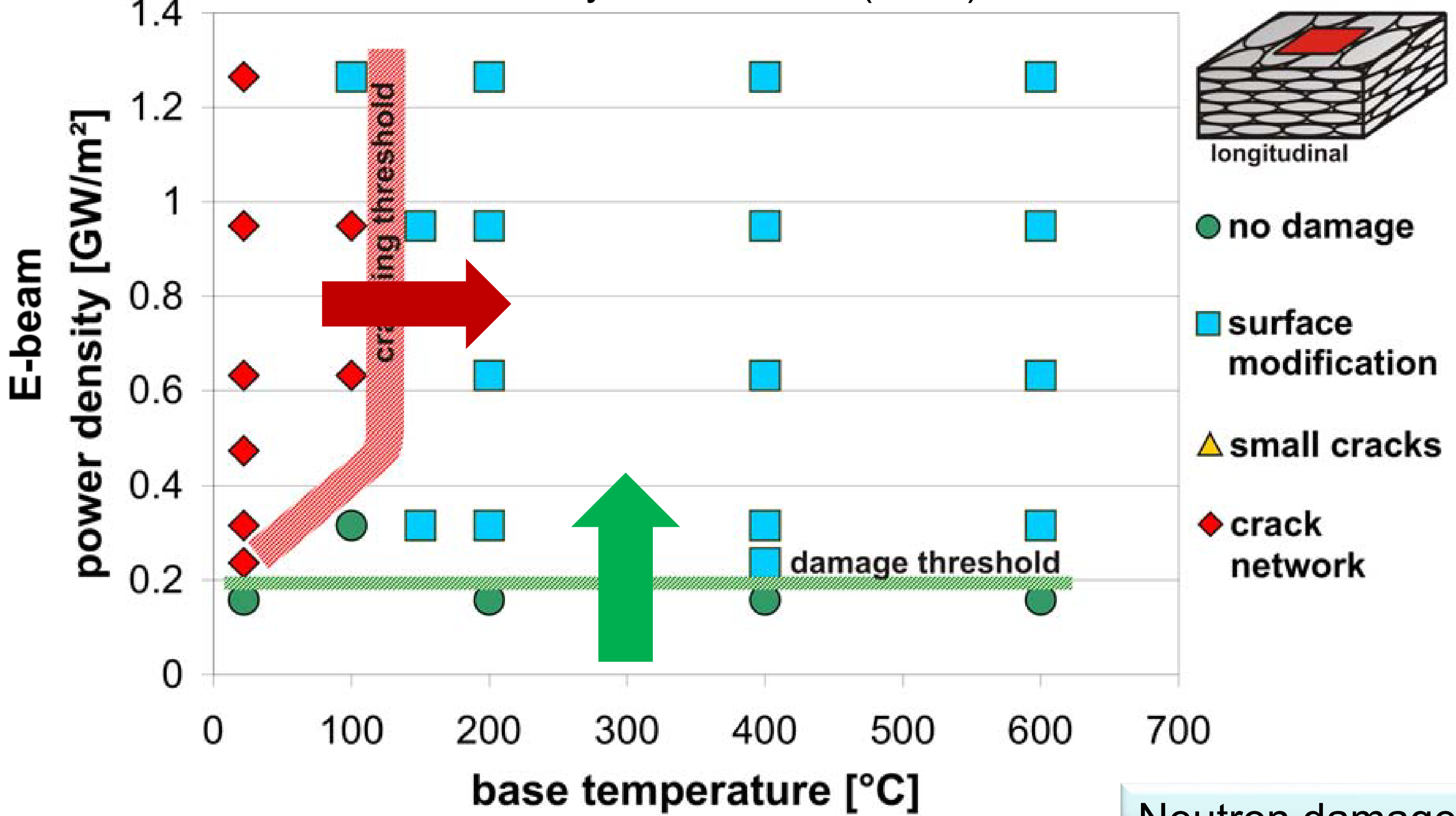
Transmutation: Decay times after n-irradiation



Source: R.A. Forrest et al., Handbook of Activation Data, 2009

Impact of synergistic loads on crack formation

M. Wirtz et al., Phys. Scr. **T145** (2011) 014058

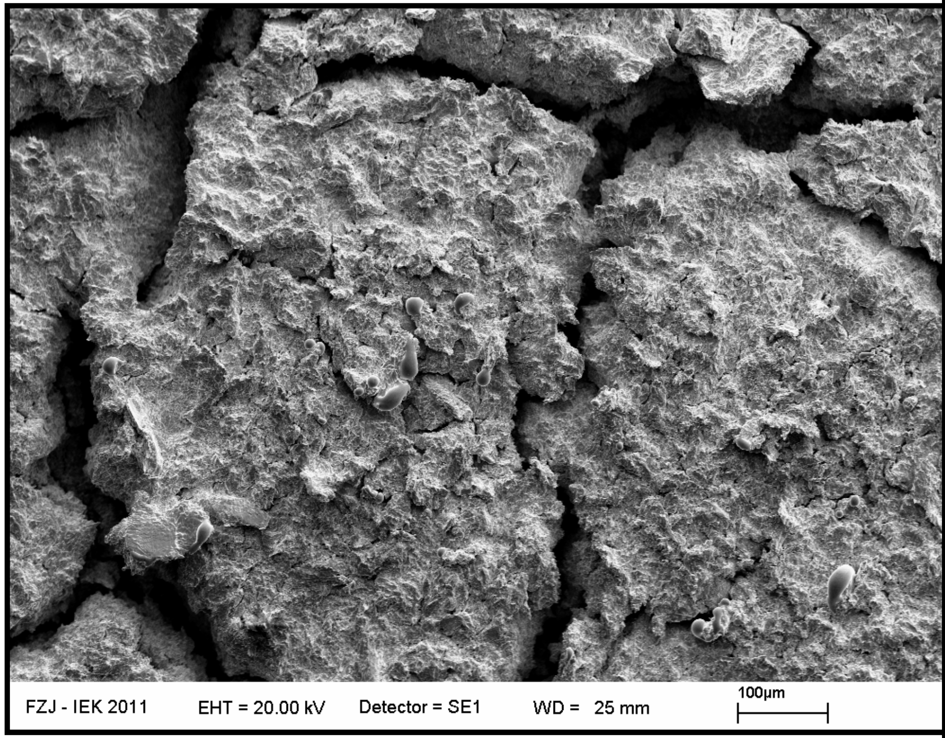


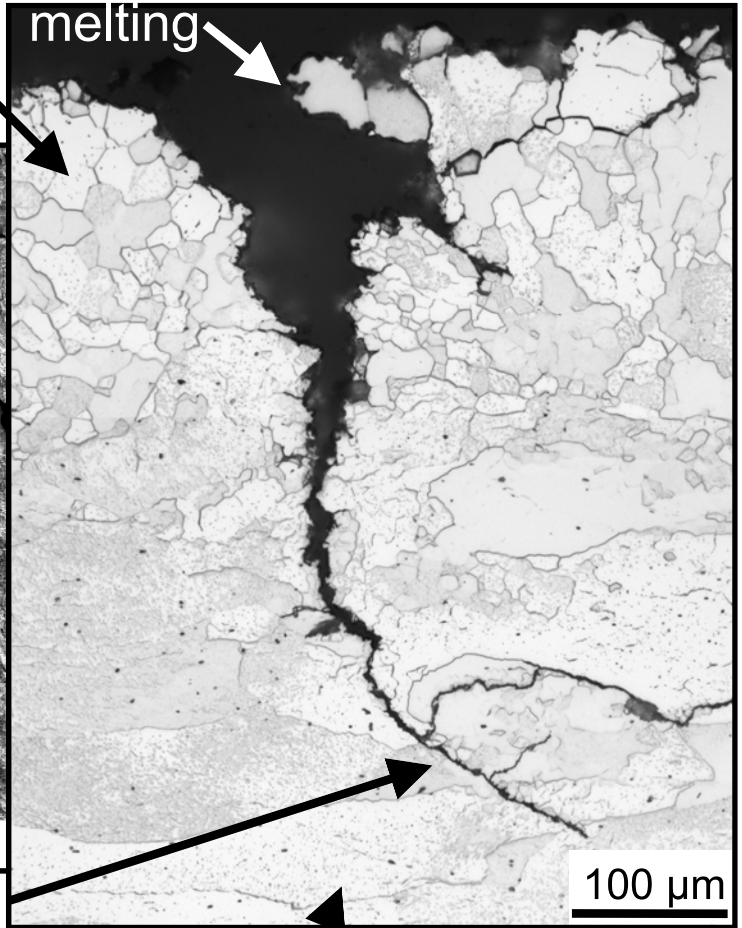
Neutron damage
Plasma load

Enhanced erosion of irradiated tungsten in synergistic loading conditions

ELM simulation with high rep rates

recrystallization





recrystallization around crack edges

original grain structure

Th. Loewenhoff, et al., Fusion Engineering and Design 87 (2012) 1201-1205

Change of material composition

Radiation (plasma) induced:

- RIS: spatial redistribution of solute and impurity elements in a metal → enrichment or depletion of alloying elements near surfaces
- Reason: different coupling of solutes to defects
- Phase instabilities

- Issue for functional surface coatings (e.g. passivating layers or permeation barriers)
- Impact on surface composition as determined by preferential sputtering (e.g. EUROFER)
- *Cr enriches in F-M alloys, leading to grain boundary embrittlement [Gupta et al., J. Nucl. Mater. 351 (1-3) (2006), 162.]*

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Summary

Research tailored towards fusion reactors

Research focus:

- material development
  - definition
  - preparation and characterization
  - optimization

- PWI issues
  - erosion
  - retention
  - lifetime

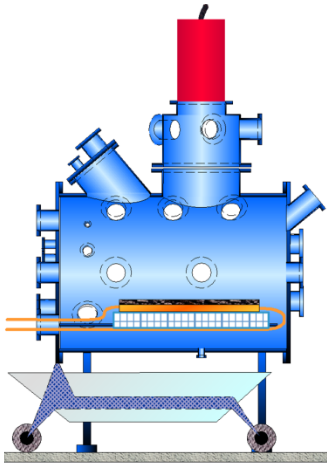
→ Materials tests:

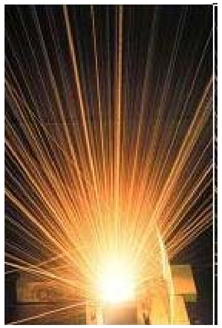
- neutron damage (simulation AND “real” neutrons)
- plasma exposure
- ELM (off-normal events) simulation

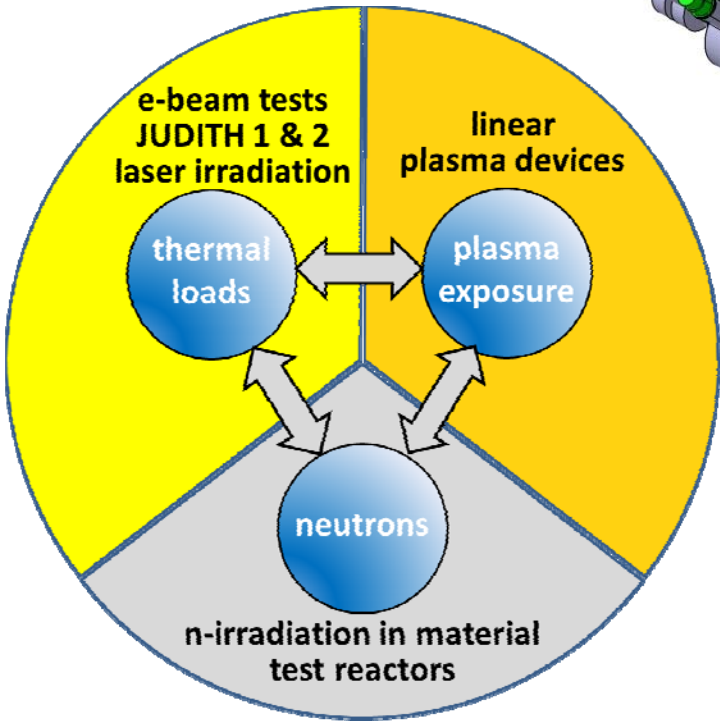
Material testing at FZJ

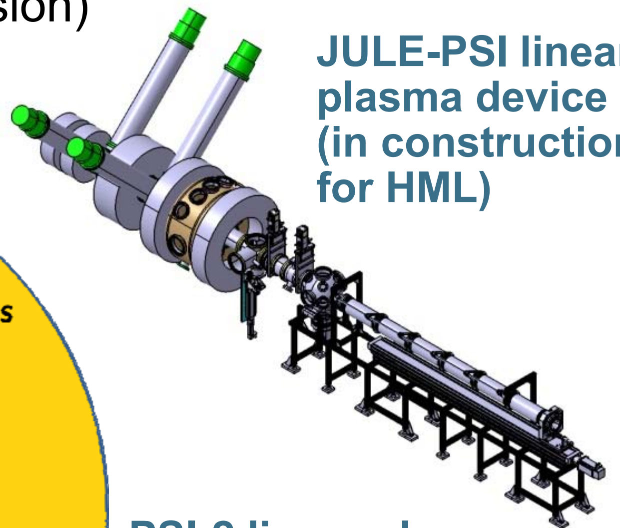
- Integrated characterization of thermo-mechanical and physical-chemical properties of neutron irradiated and toxic plasma-facing materials under high heat loads and plasma exposure
- Selection of plasma-facing materials tested under n-irradiation and optimized for PMI processes (tritium retention, embrittlement, erosion)

JUDITH 1 and JUDITH 2
e-beam facilities



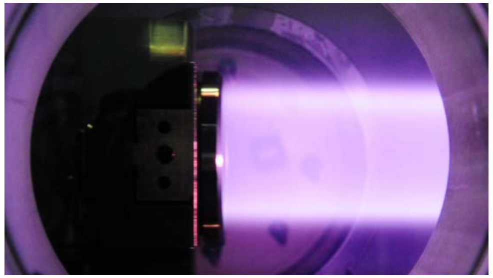






JULE-PSI linear plasma device (in construction for HML)

PSI-2 linear plasma experiment (outside HML)



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Tungsten: Brittleness problem

Limitations of operation temperatures for tungsten:

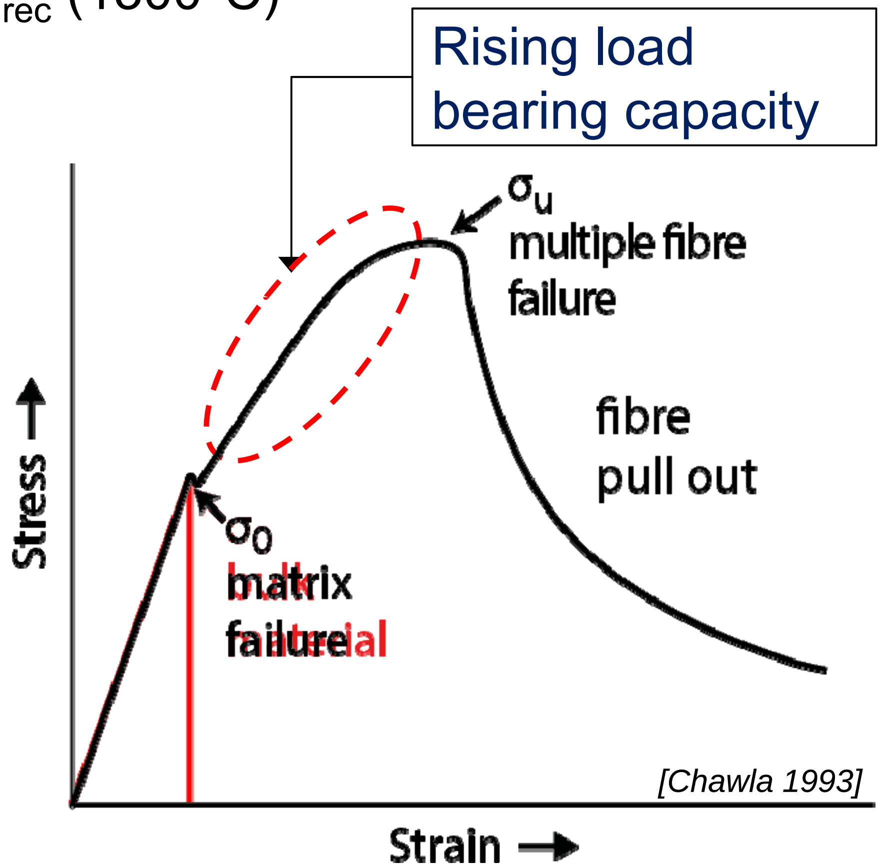
Lower limit: ductile-brittle-transition temp. T_{DBT} (260-650°C)

Upper limit: recrystallization temp. T_{rec} (1300°C)

plus: neutron embrittlement

- scattering in strength (small Weibull modulus)
- no damage tolerance
- uncertainty in lifetime prediction

Solution: extrinsic toughening (ductilization) mechanisms



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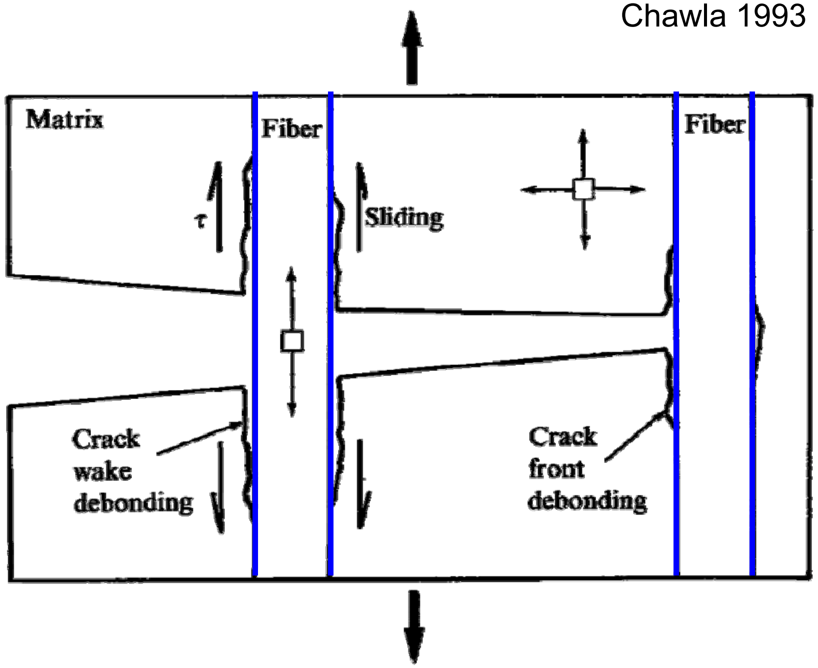
Chawla 1993



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⇒ local energy dissipation
- crack bridging
- fiber oull-out
- crack deflection

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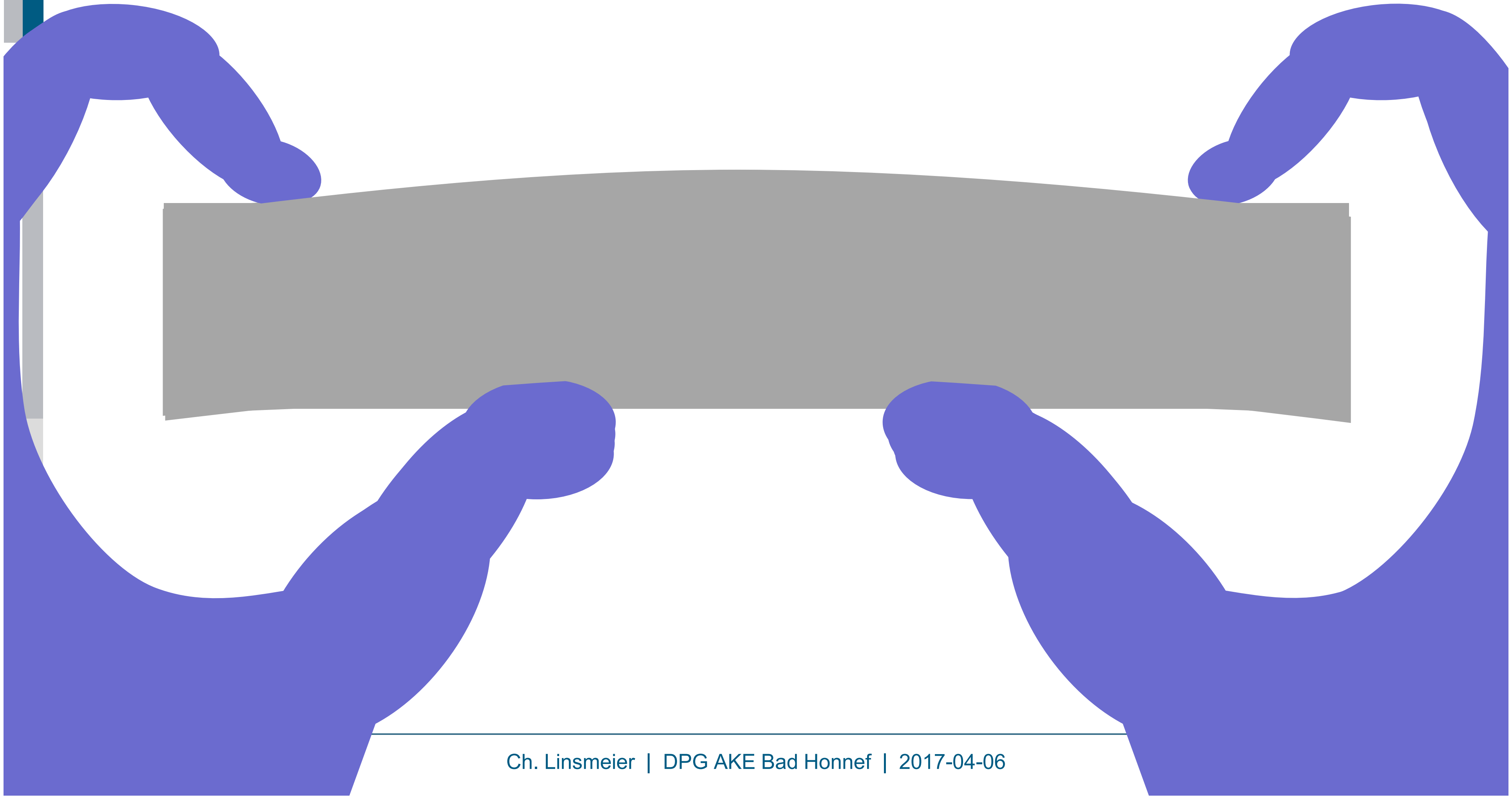
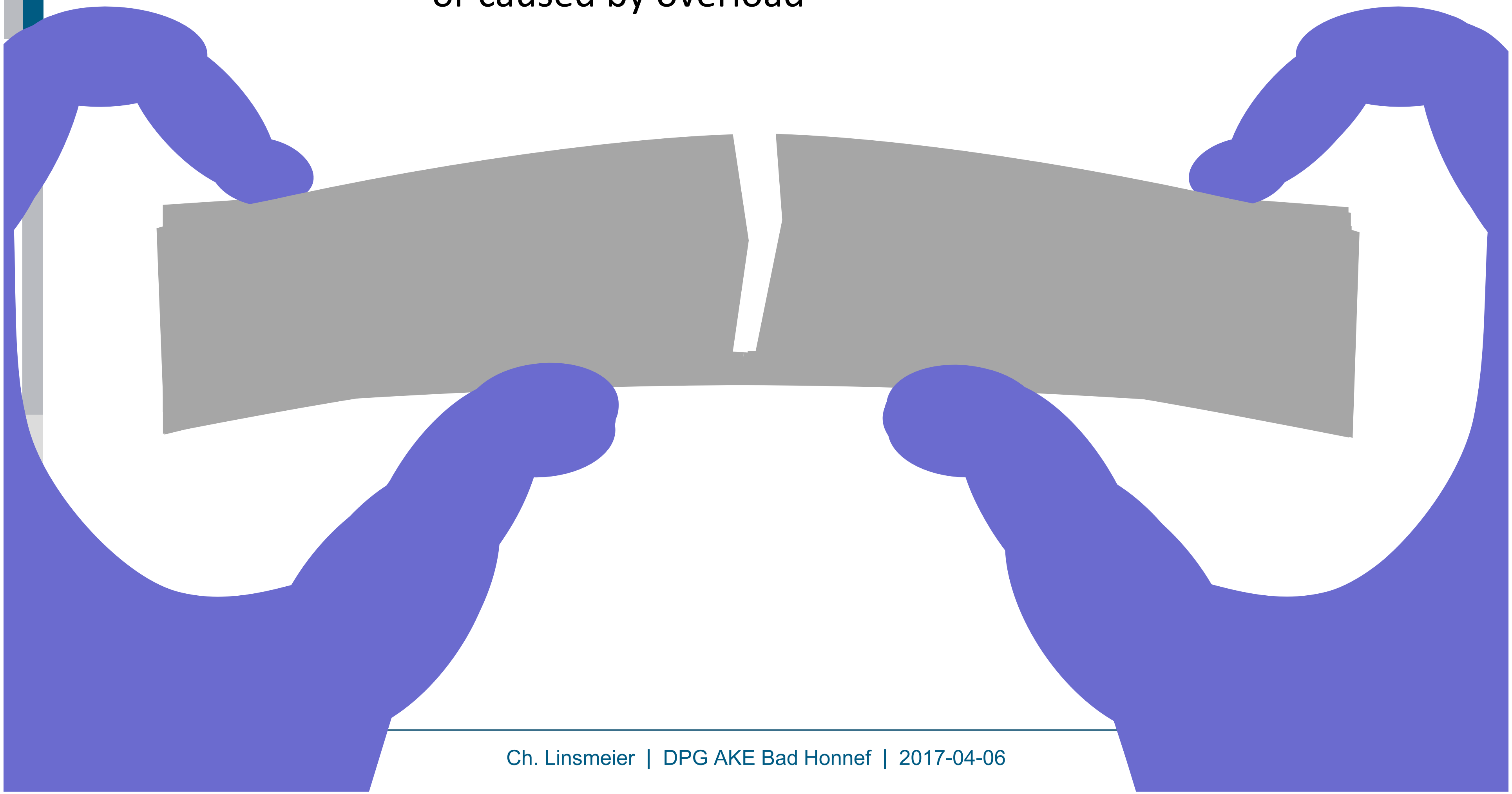
Main advantages for fusion

- damage tolerance
- mechanical effect

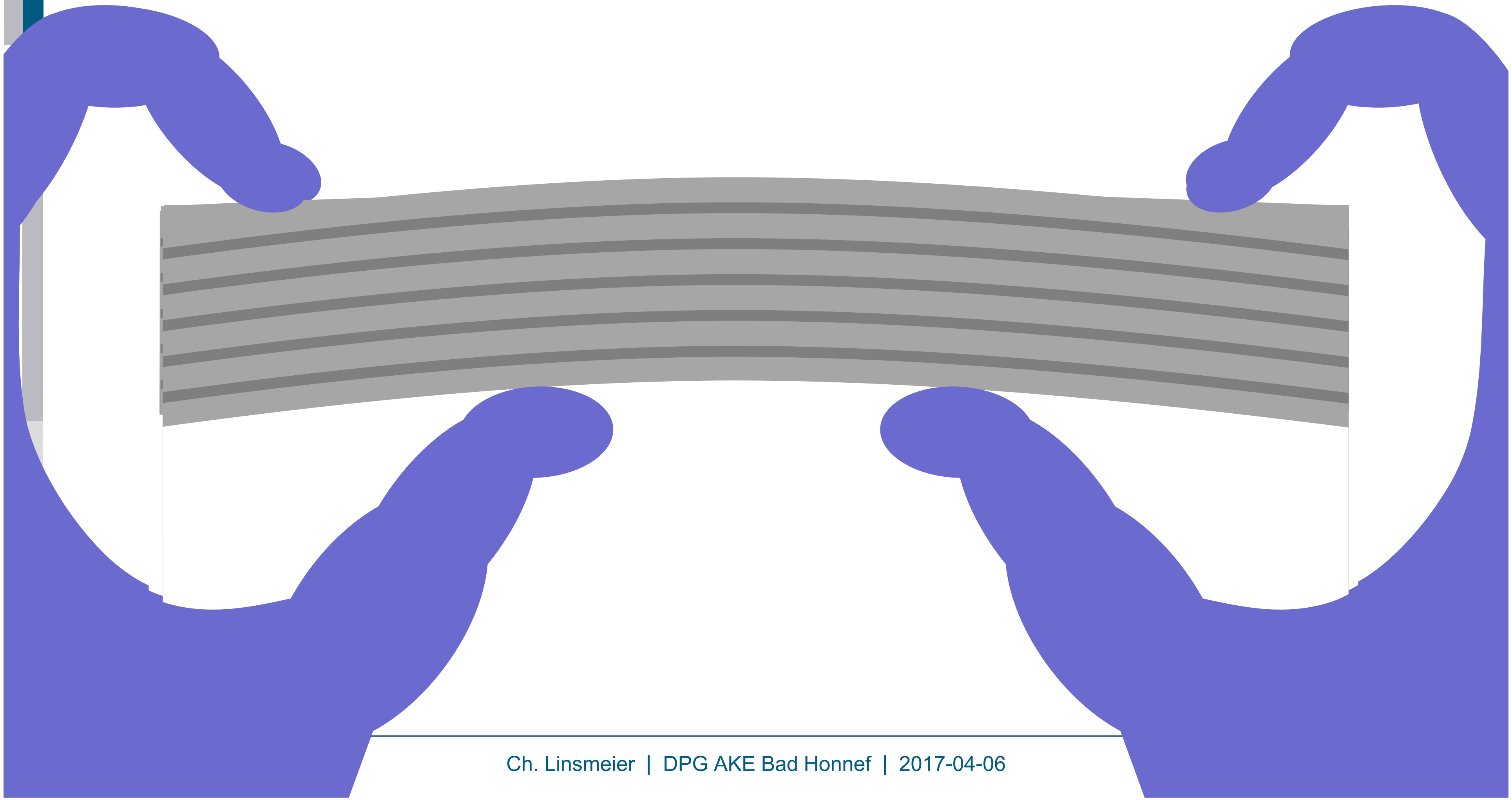
⇨ **less susceptible to operational embrittlement** e.g. full tungsten tile under cyclic loading



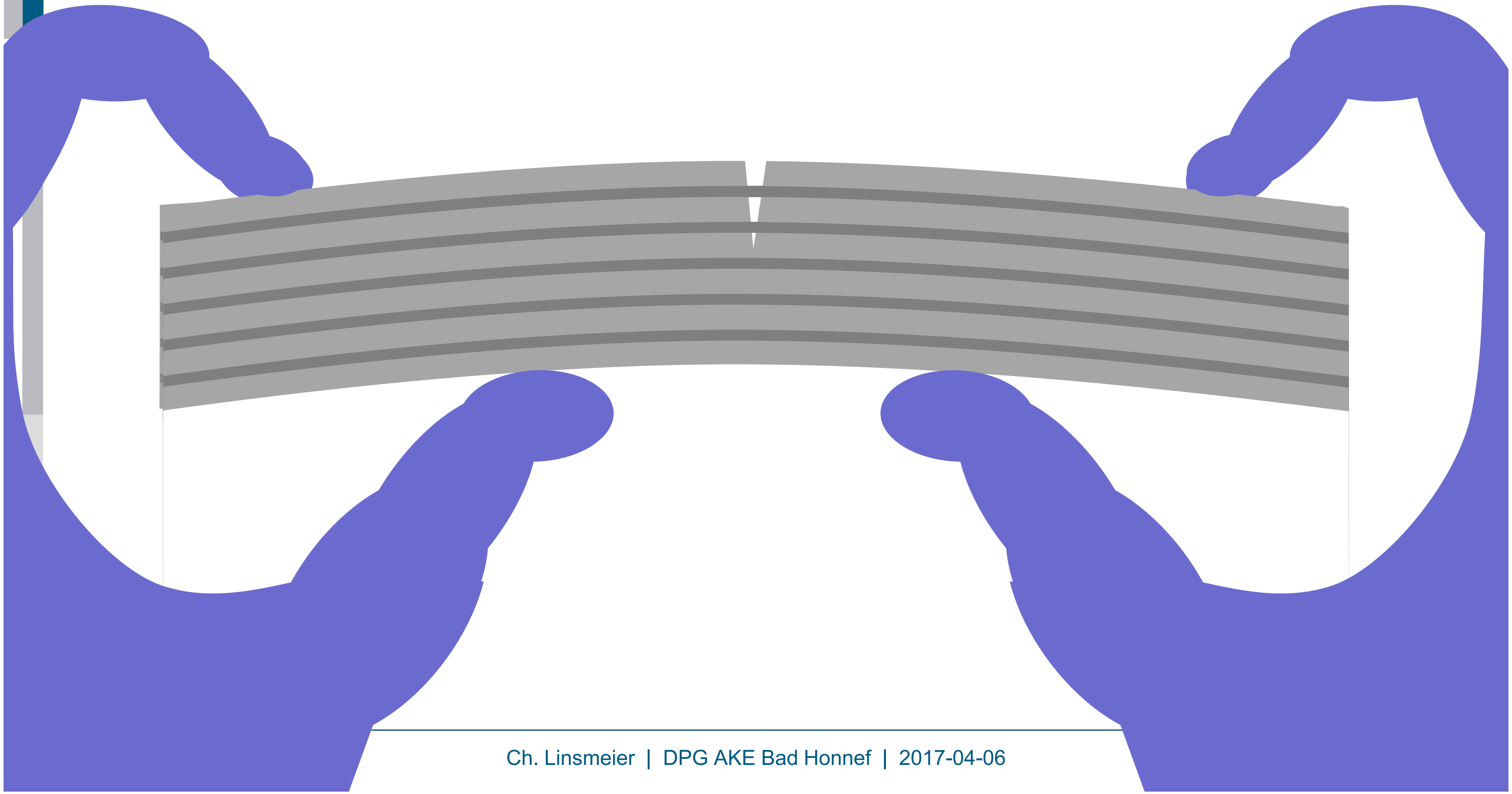
catastrophic failure by brittle fracture
after a random number of cycles
or caused by overload



W_f/W under cyclic loading

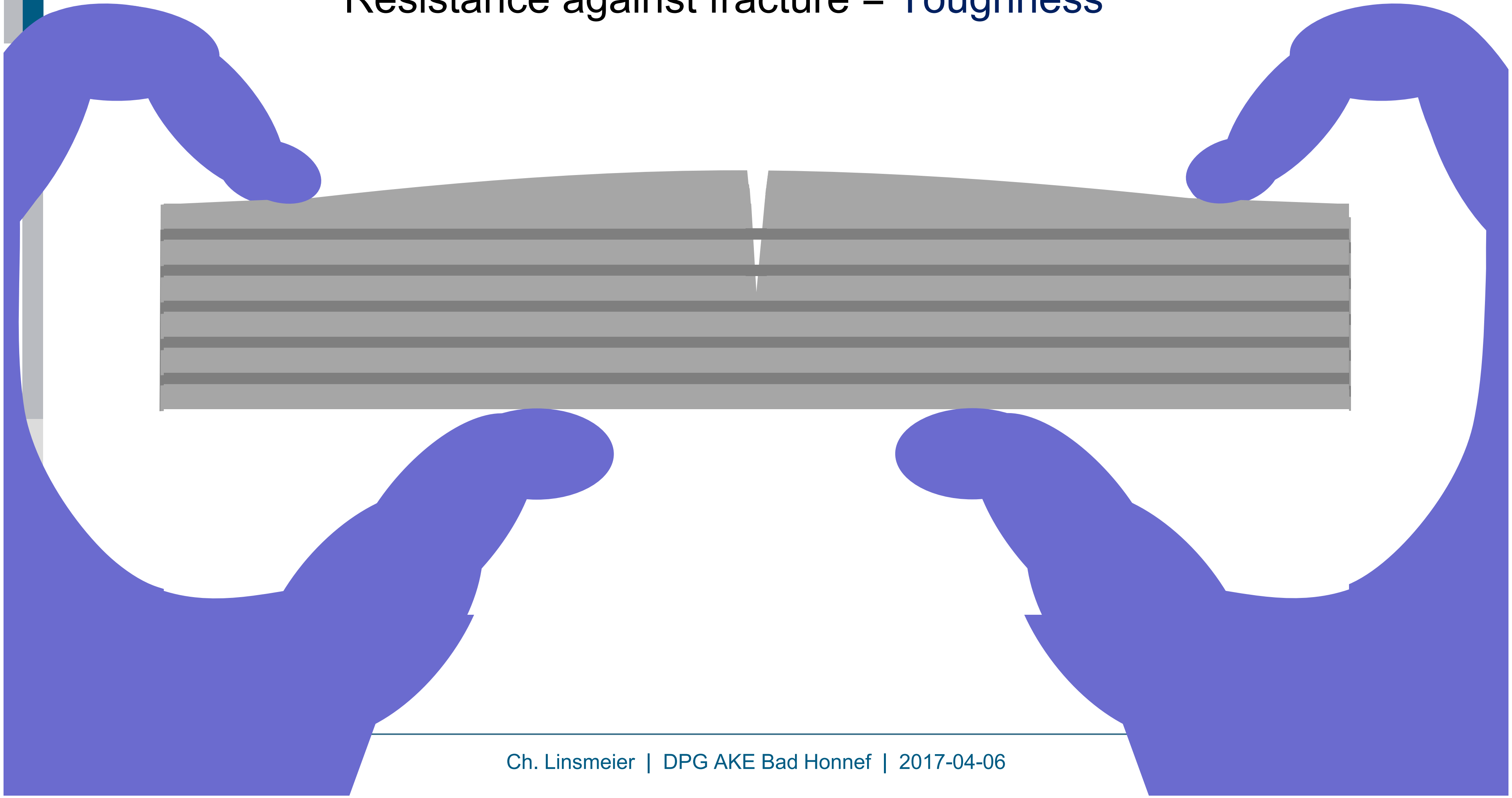


Crack is bridged by fibres



Further loading still possible

Resistance against fracture = Toughness



Architecture of W_f/W

- **Fibre**

  Drawn tungsten wire (d = 150 µm):
  high strength + some ductility

- **Interface**

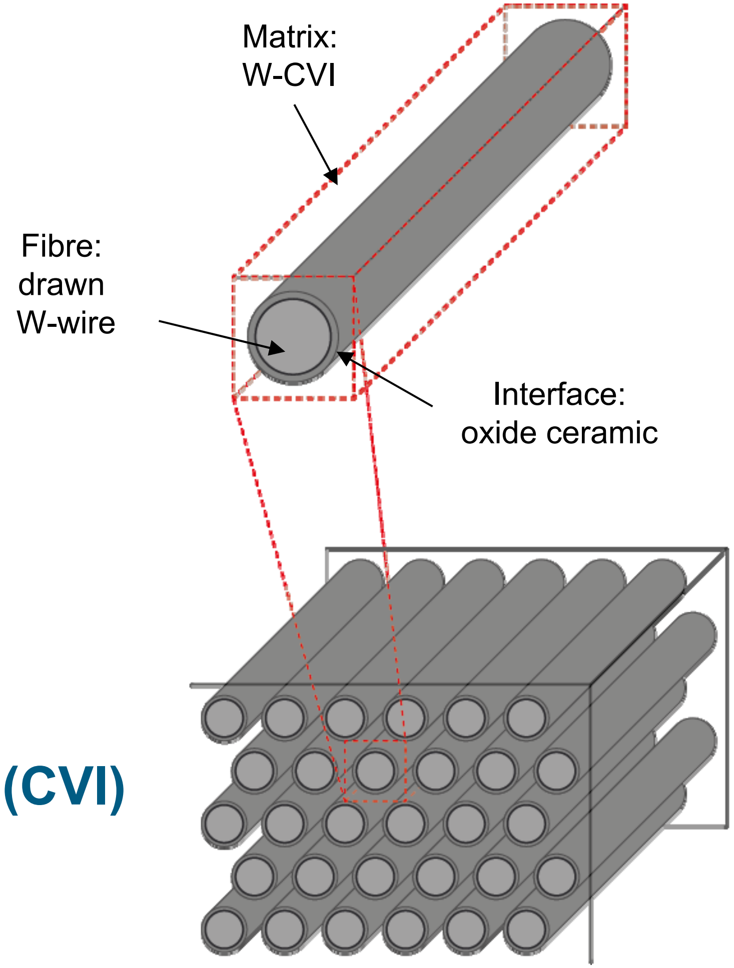
  PVD coating:
  Optimised adhesion + stability

- **Matrix**

  Interface integrity + high density

  Develop **chemical vapour infiltration (CVI)** technique for W_f/W

  Powder metallurgy (PM-W_f/W)



Matrix synthesis

Chemical vapor infiltration (CVI)

+ Low temperature process (600 – 1000 K)
+ No mechanical impact

→ Preservation of interface/ fiber integrity

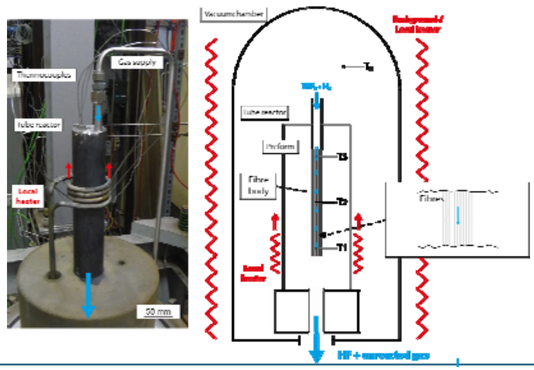
− Low experience in W bulk production
− Residual porosity

Powder metallurgy (PM)

+ High experience in W bulk production and processing e.g. PIM
+ Easier implementation of alloying (e.g. self passivating W)

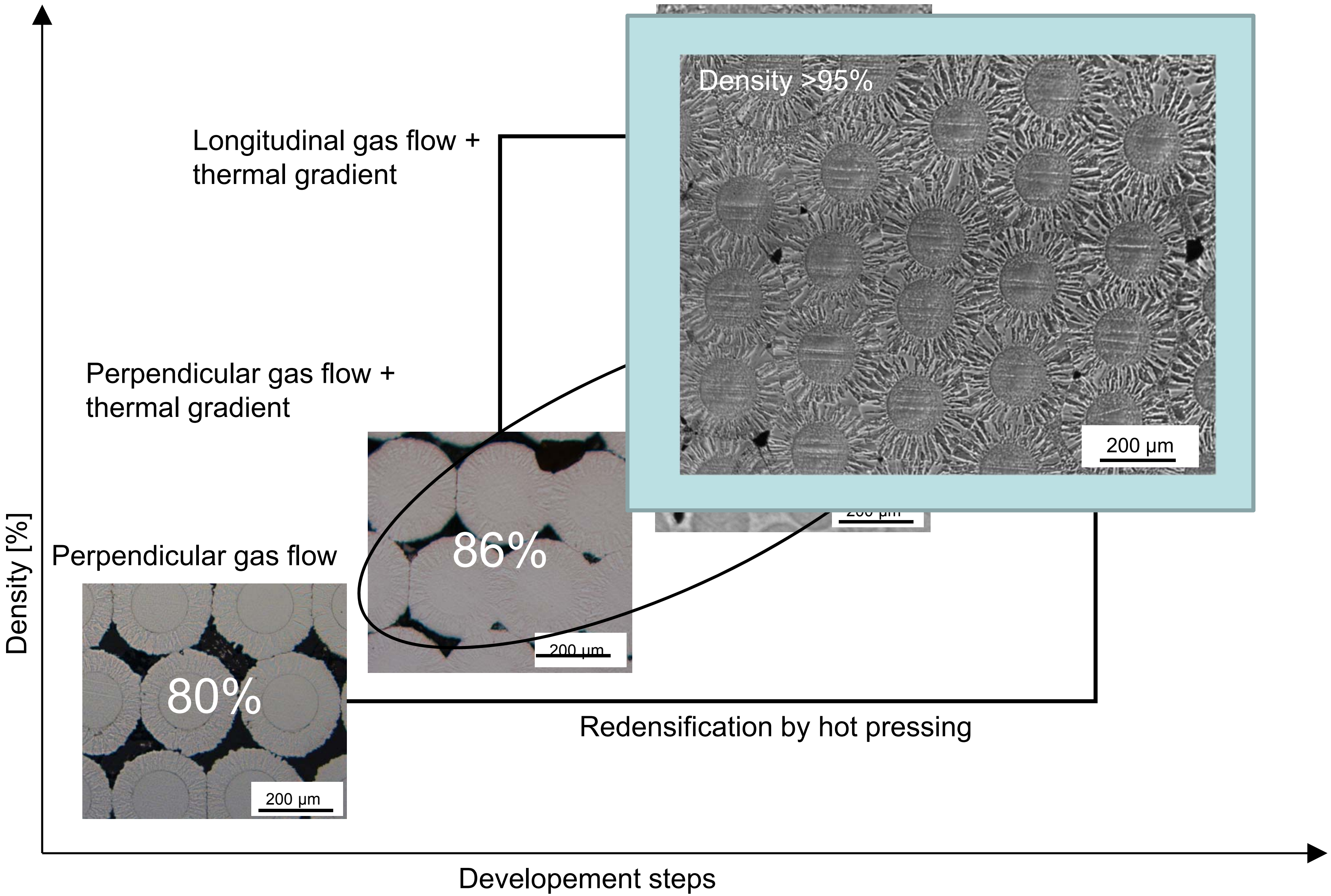
− High temperature
− High pressure

Tungsten infiltration (WILMA)



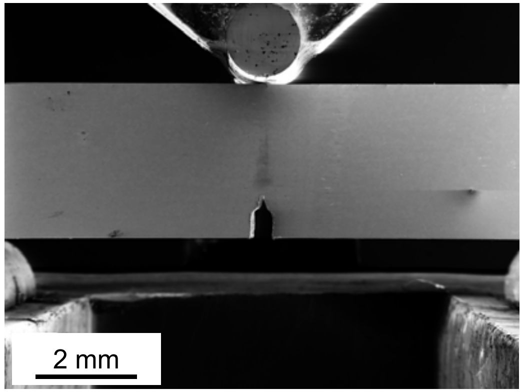
Production of W from the gas phase

Development of CVI-tungsten



3-Point bending test (ESI Leoben)

Stepwise 3-point bending



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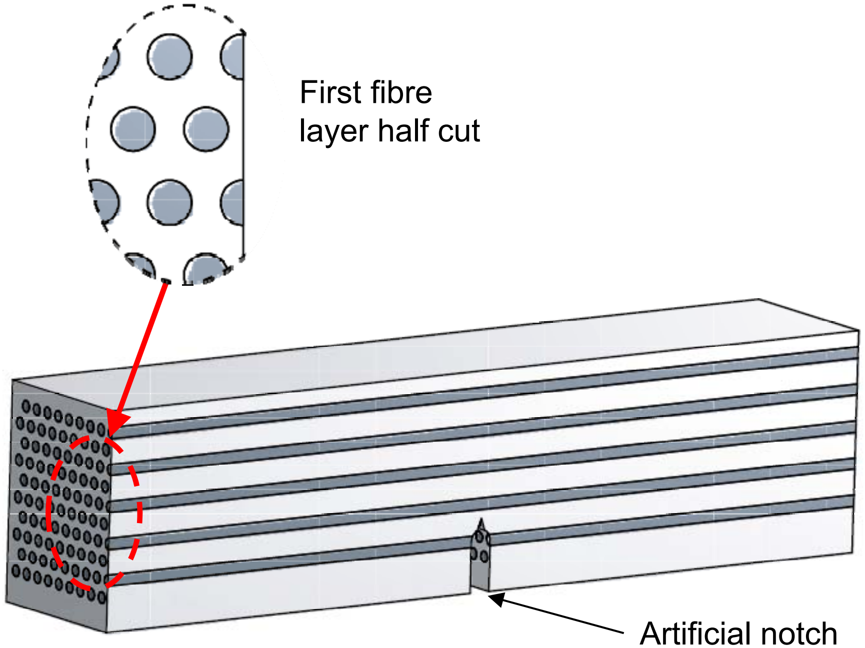
In-situ surface observation in electron microscope

Multi-fibre composite

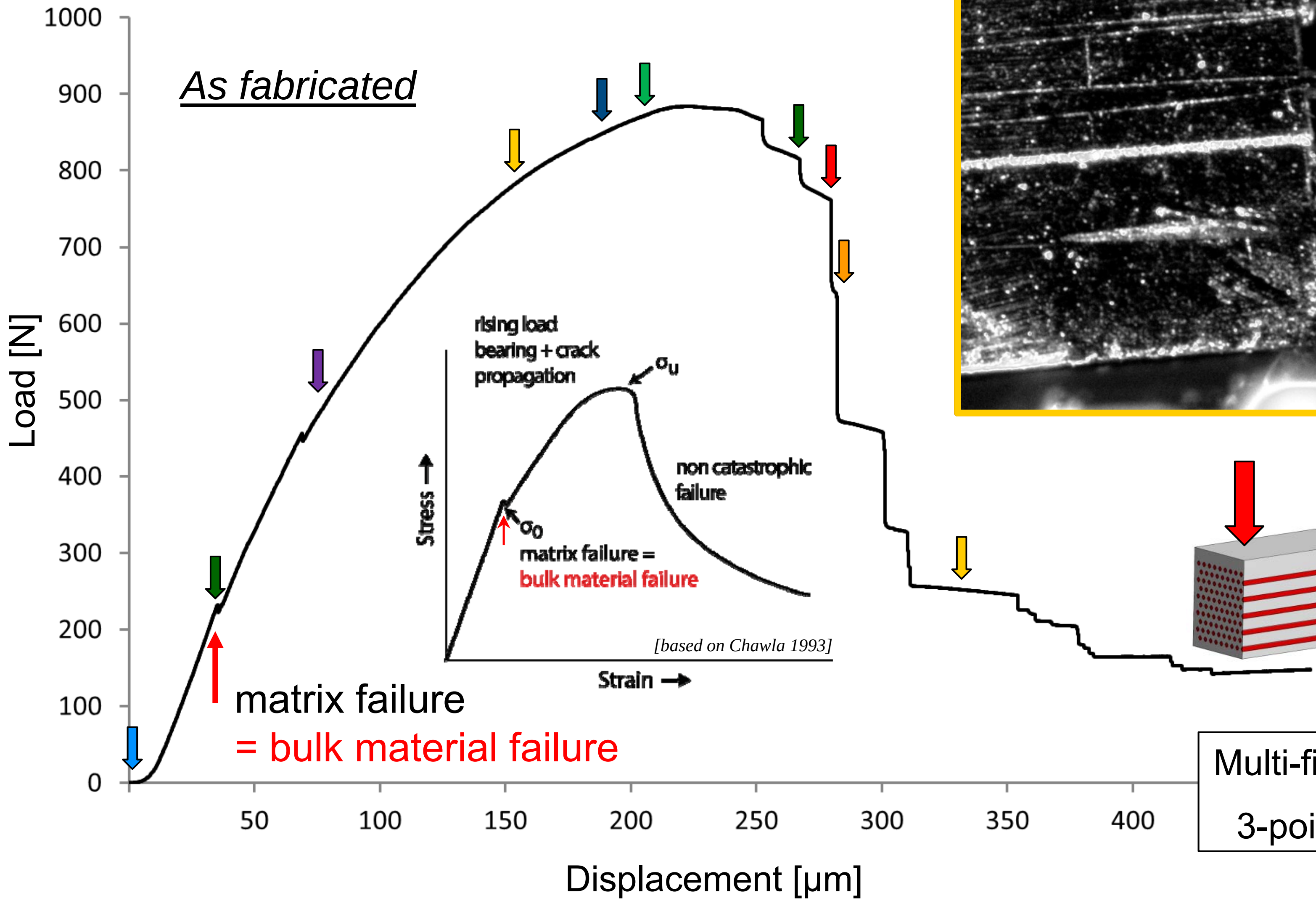
→ W-CVI

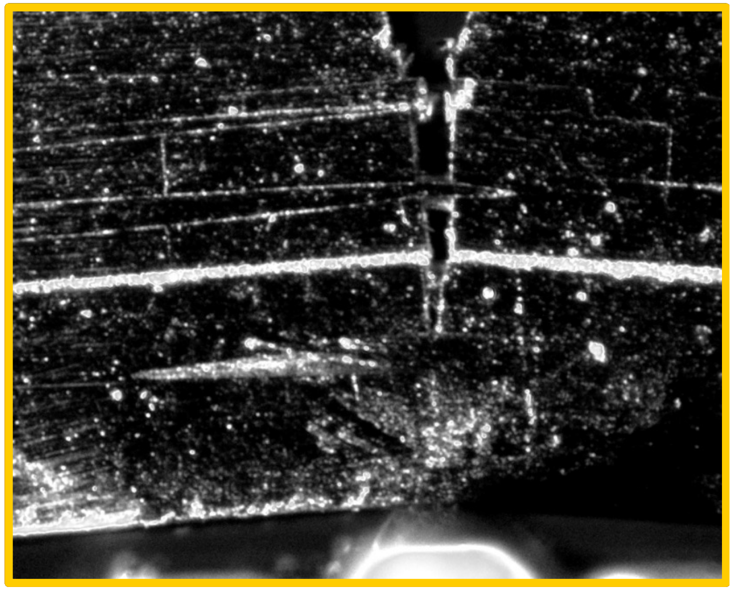
→ 10 layers x 9 fibres

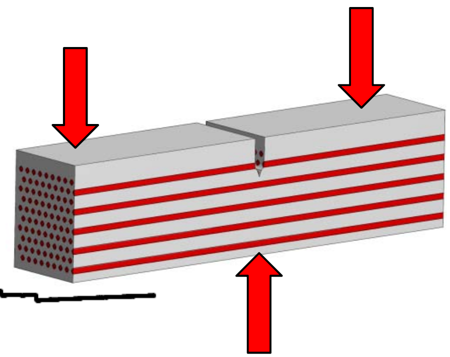
→ 2.2 mm x 3 mm



Demonstration: in situ bending test



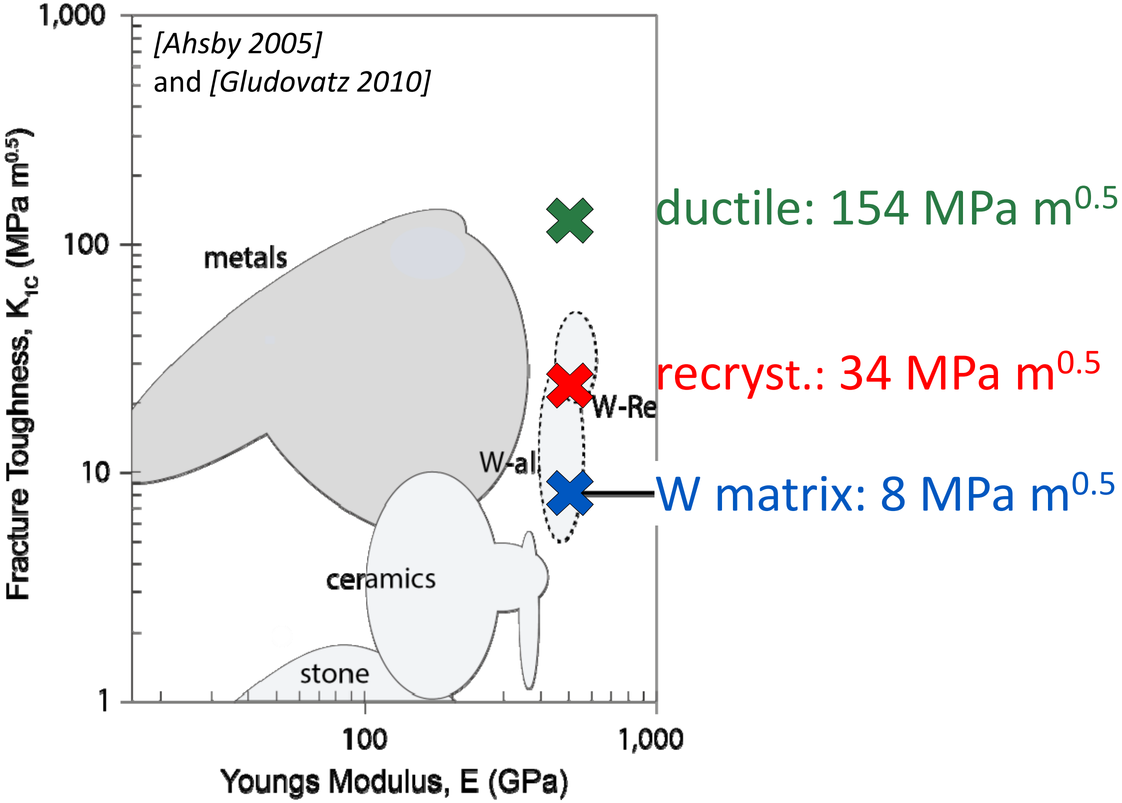


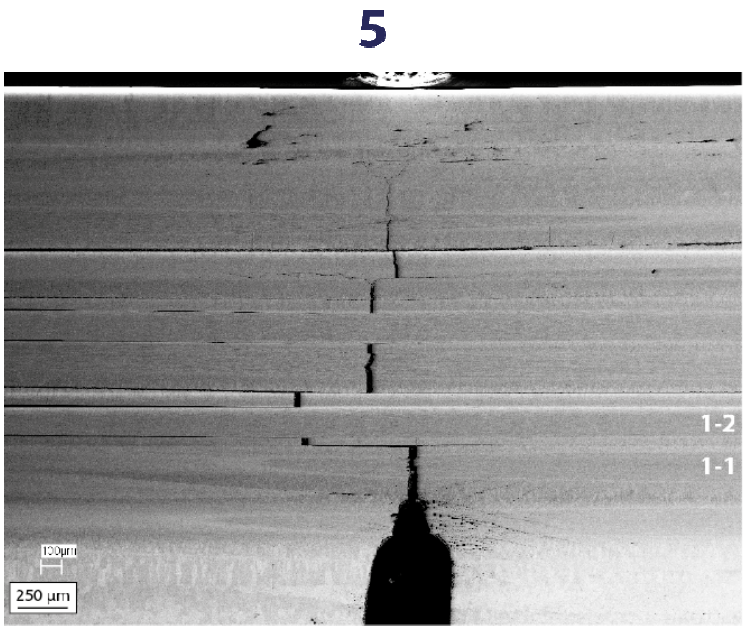


Multi-fibre sample,
3-point bending

Master thesis G. Holzner

Toughness enhancement W_f/W



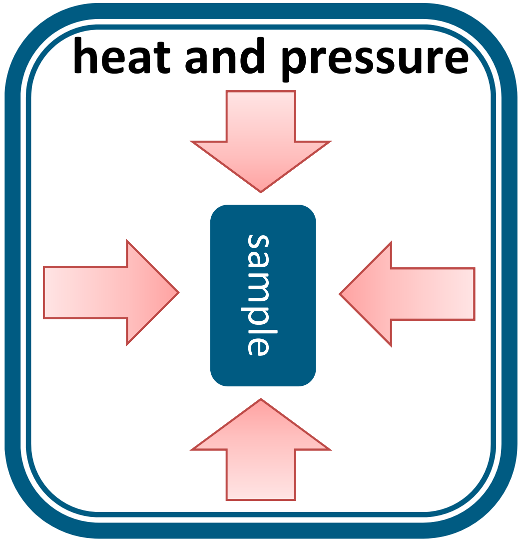


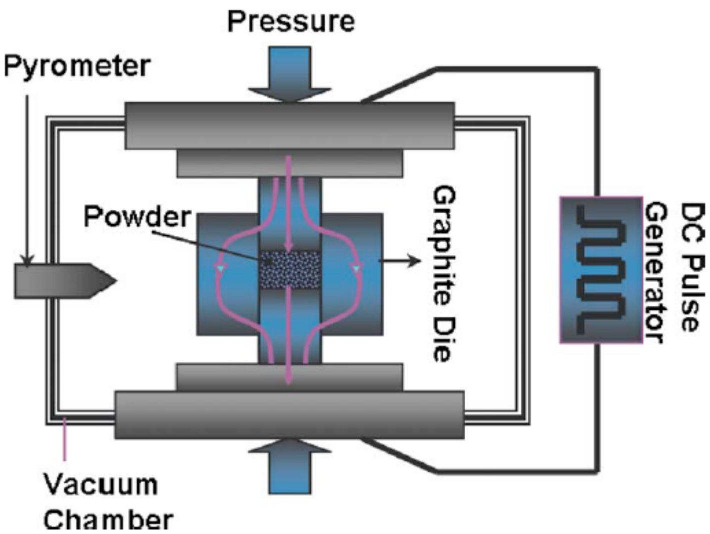
Controlled crack propagation + **rising load bearing capacity**

Alternative production routes

HIP – Hot isostatic pressing

- capsule filled with tungsten powder and fiber inside a pressure vessel
- powder compaction due to high pressure and temperature
- T_{max} 2000 °C
- p_{max} 350 MPa (via Ar)





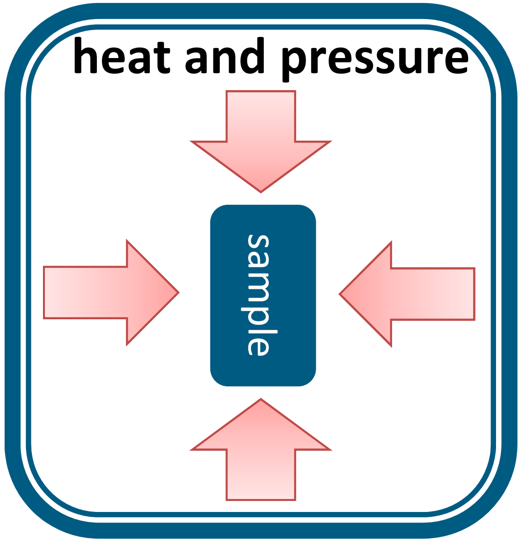
FAST – Field-assisted sintering technology (SPS – Sparc plasma sintering)

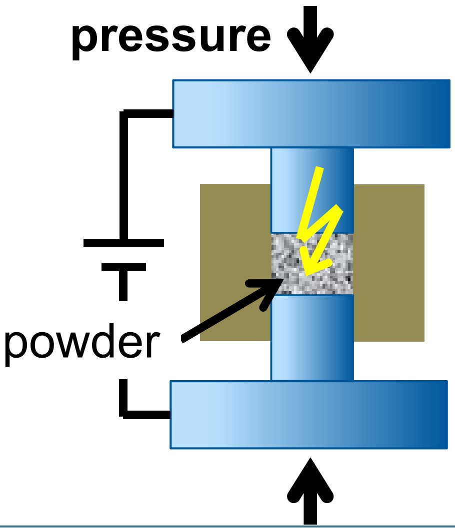
- powder compaction due to Joule heating/melting, enhanced diffusion (electromigration)
- T~1900°C
- uniaxial pressure (p_{max} 60 MPa)
- process time ~4 min, 200 K/min

Alternative production routes

HIP – Hot isostatic pressing

- capsule filled with tungsten powder and fiber inside a pressure vessel
- powder compaction due to high pressure and temperature
- T_{max} 2000 °C
- p_{max} 350 MPa (via Ar)





EDS – Electro discharge sintering

- tungsten powder and fiber are put inside an extrusion die
- powder compaction due to ohmic heating by a high current (500 kA) + uniaxial pressure (p_{max} 350 MPa)
- process time < 1 s, E_{max}=80 kJ

W_f/W: fibers and matrix

HIP

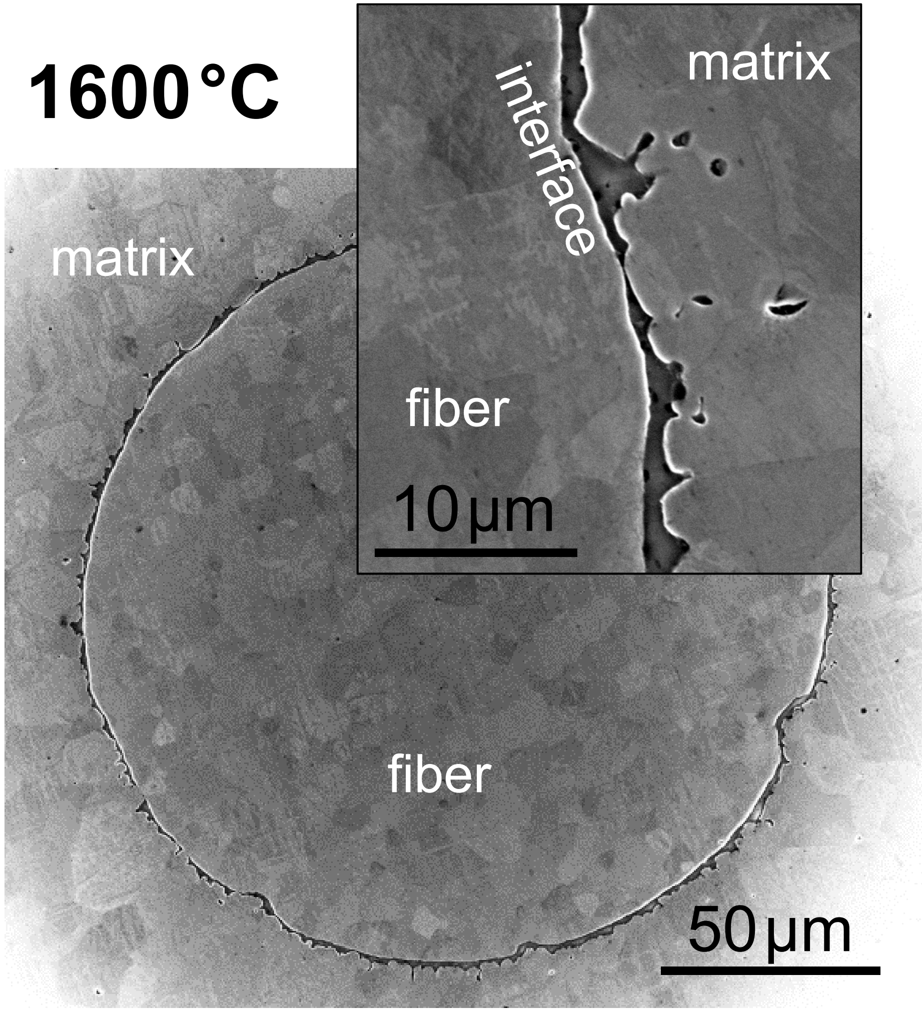
powder: d =10 µm
fibers: l ~ cm
d =150 µm

FAST

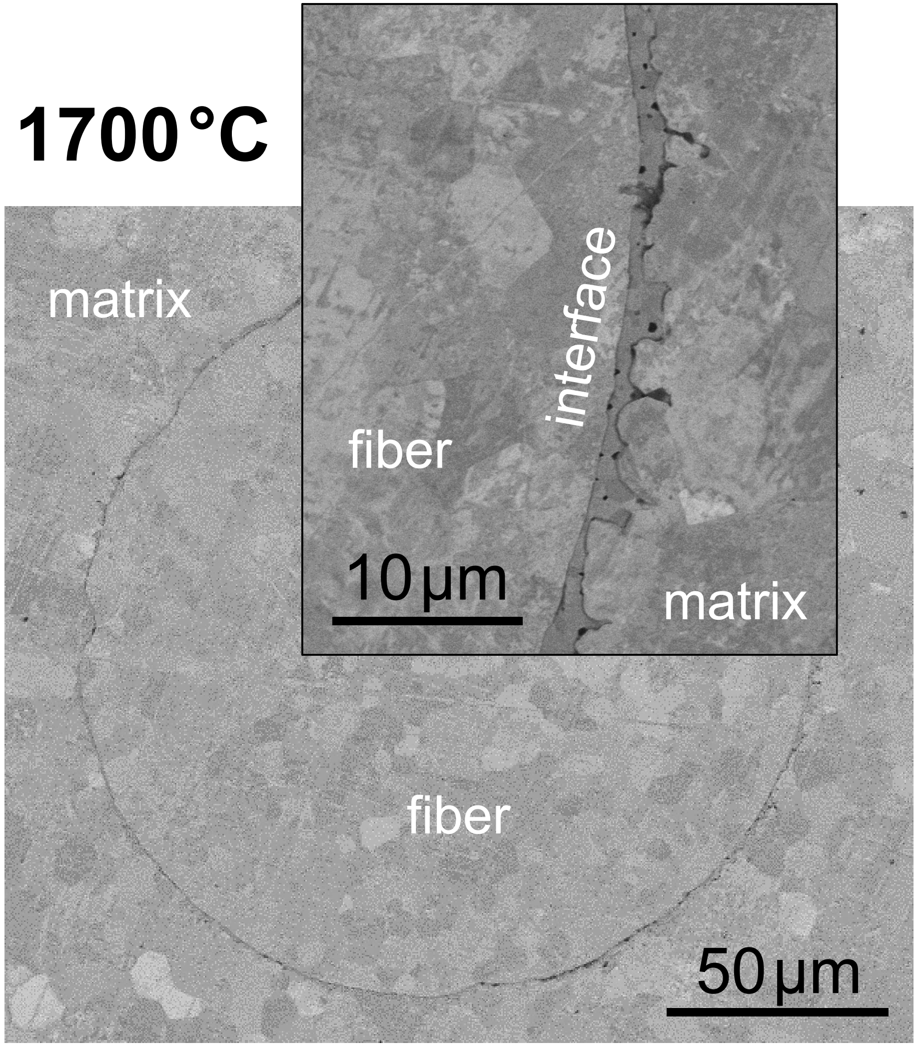
powder: d =5 µm
fibers: l =2.5 mm
d =240 µm

HIP W_f/W composites

1600°C

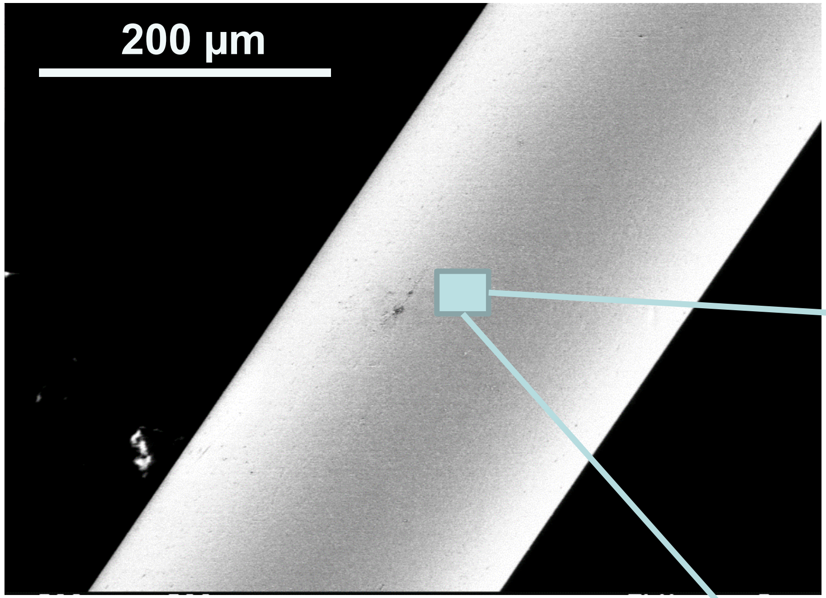


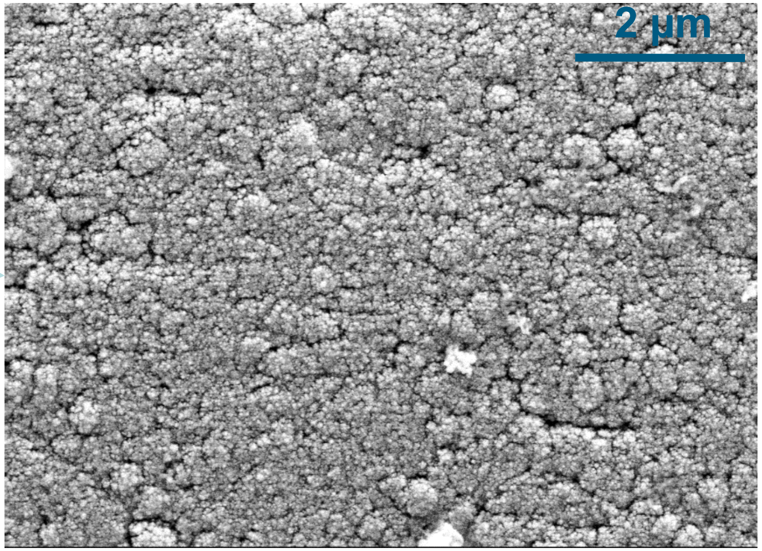
1700°C

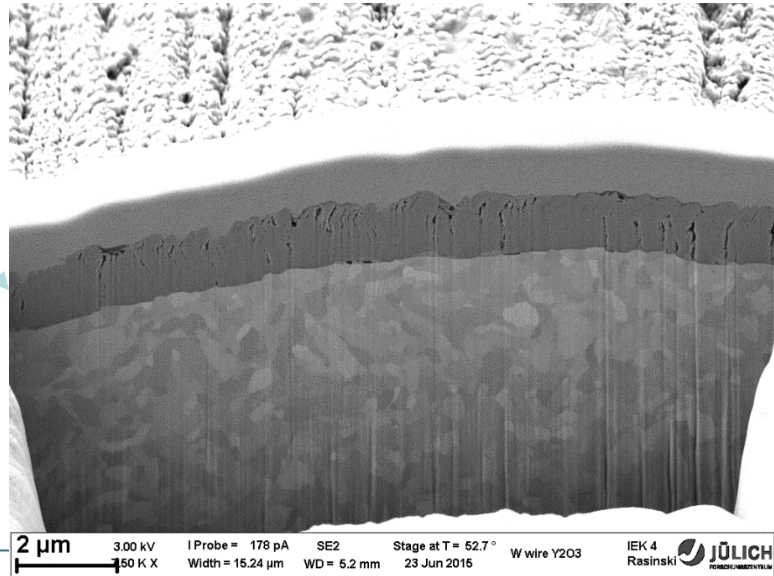


- intact interface after HIPing
- dense matrix achievable

Fiber coating by magnetron sputtering

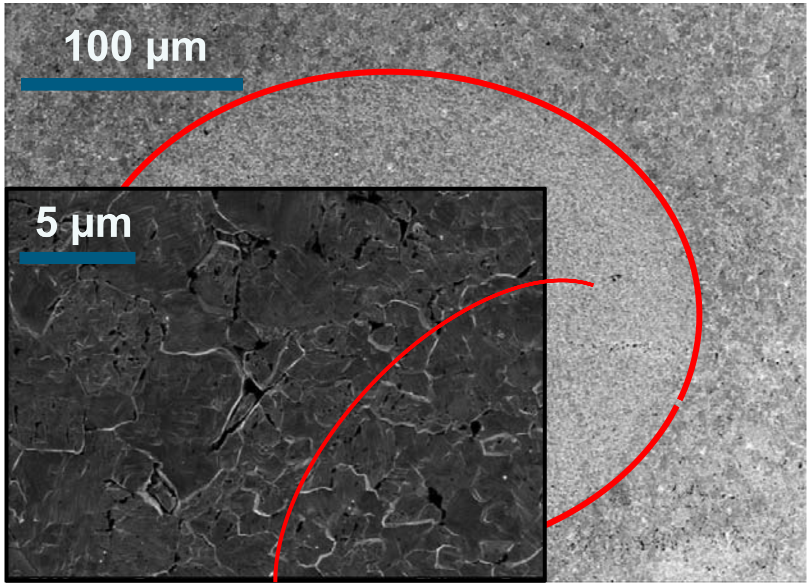




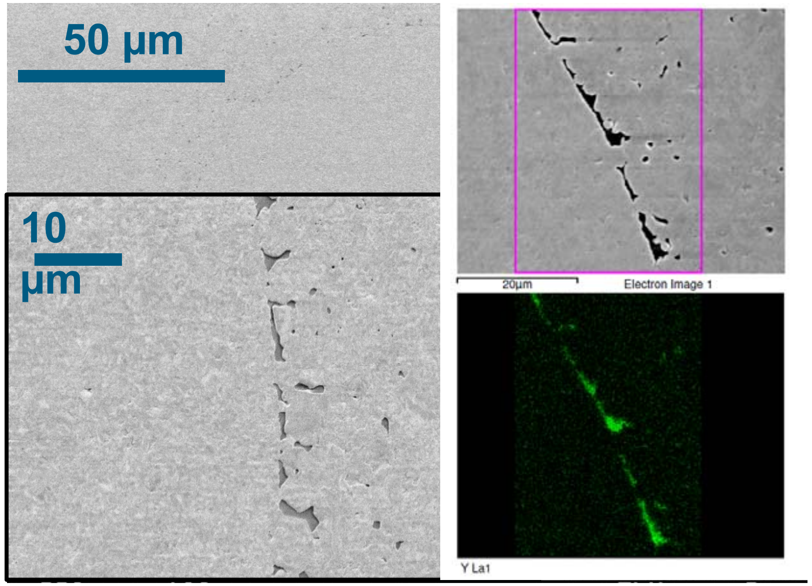


High quality, homogeneous distributed Y_2O_3 coating for the short fibers can be prepared by magnetron sputtering

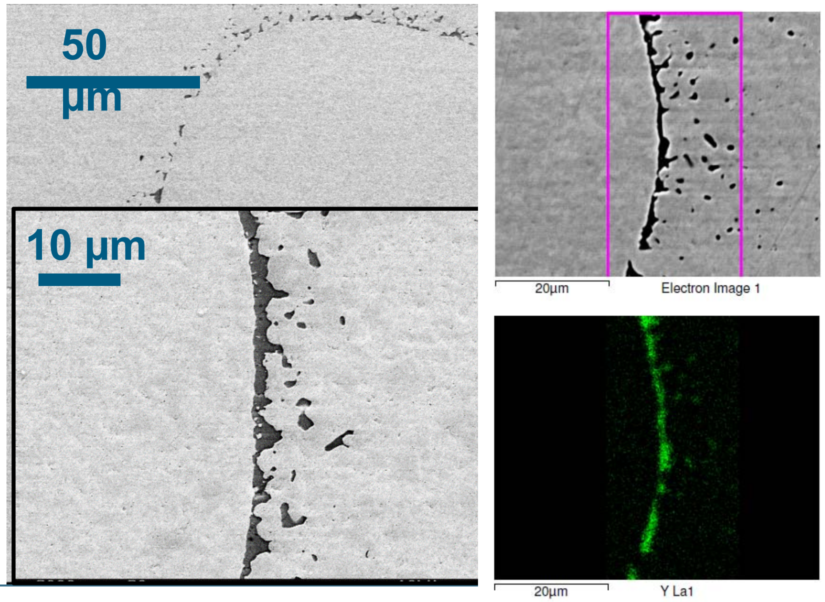
FAST W_f/W: Interface thickness



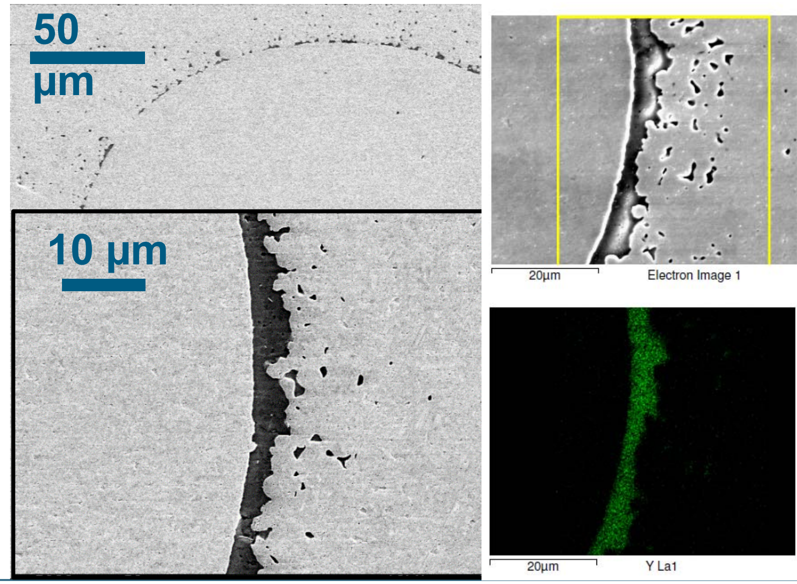
No interface



1 µm Y_2O_3 interface



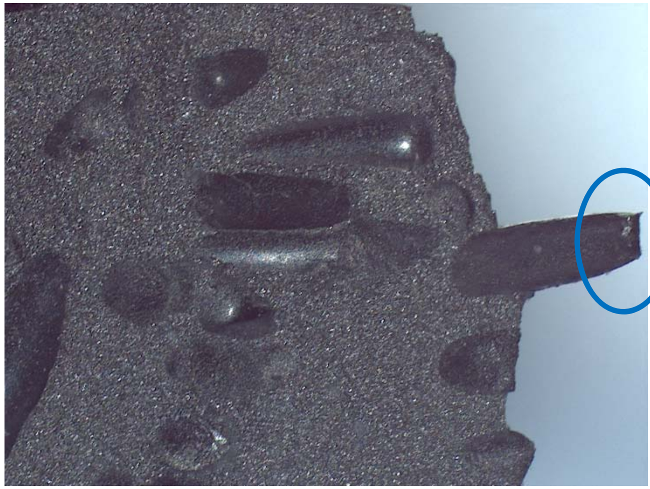
1 µm W/1 µm Y_2O_3 interface

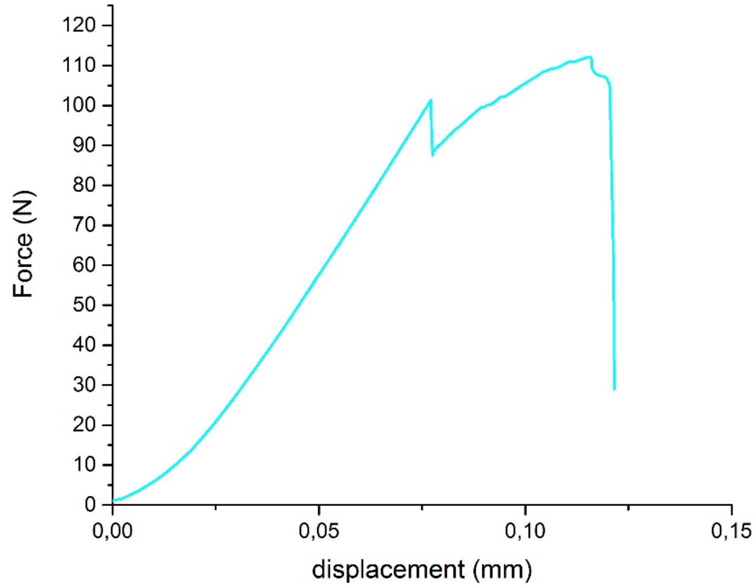


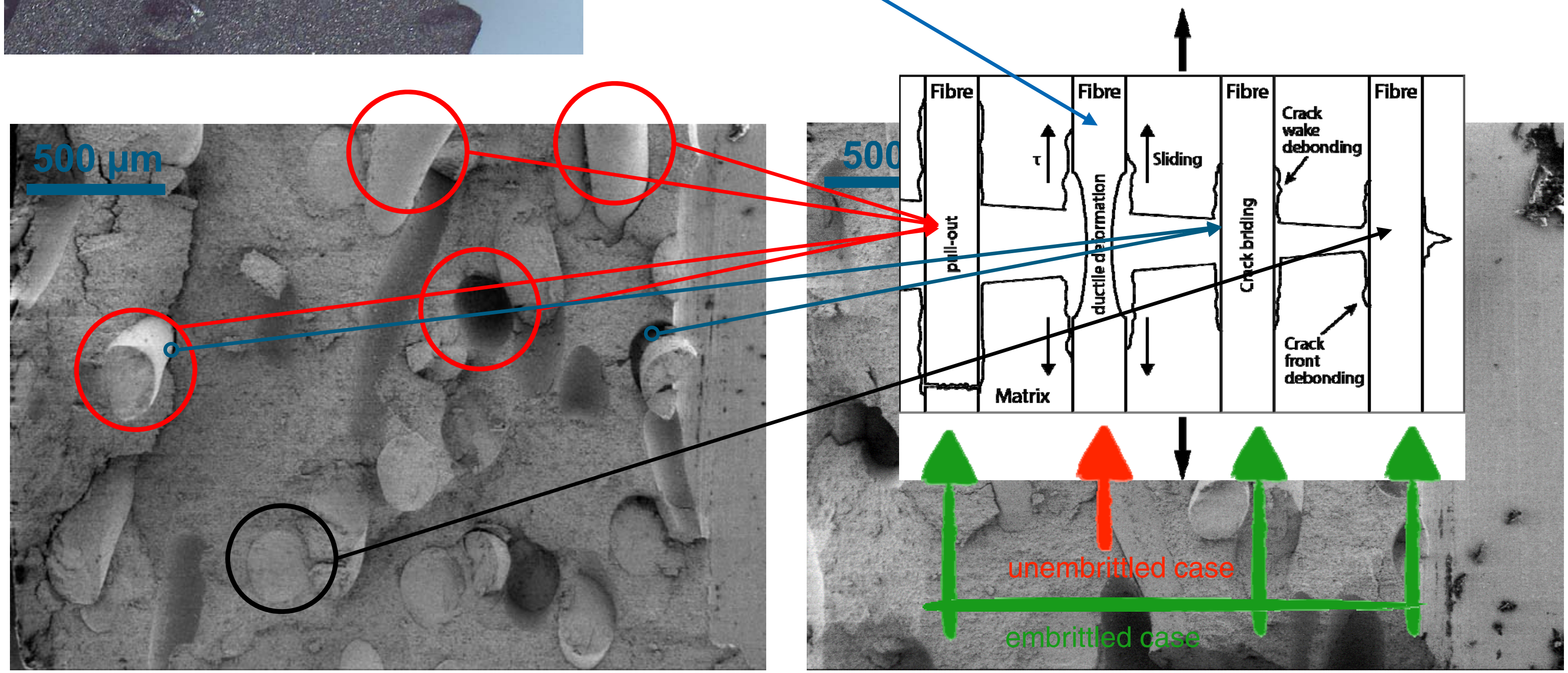
2.5 µm Y_2O_3 interface

3-point bending test crack section

FAST production, 2.5 µm interface





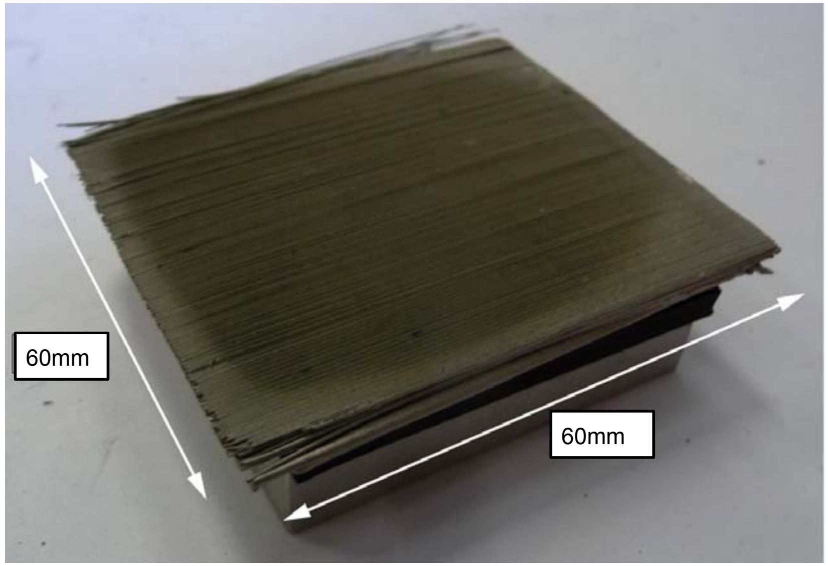


Towards W_f/W bulk production

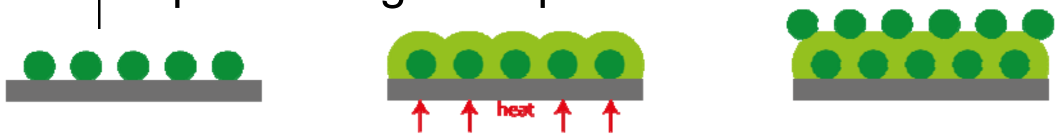
FAST multi-fiber composite

- d = 40 mm, h = 5 mm, 121 g
- 5 µm powder (OSRAM)
  30% fibers (150 µm x 1.5 mm)
  random orientation
- 94-95 % density
- FAST: 60 MPa, 1900 °C (4 min)

CVI/CVD oriented fibers



- 50 x 50 x 3.5-4 mm^3, 194 g
- 10 layers à 220 fibers
- unidirectional long fibers
- density 93-98 % (94.2 % average)
- pore-free growth possible



Overview

Introduction: the material challenge

Neutron damaged materials

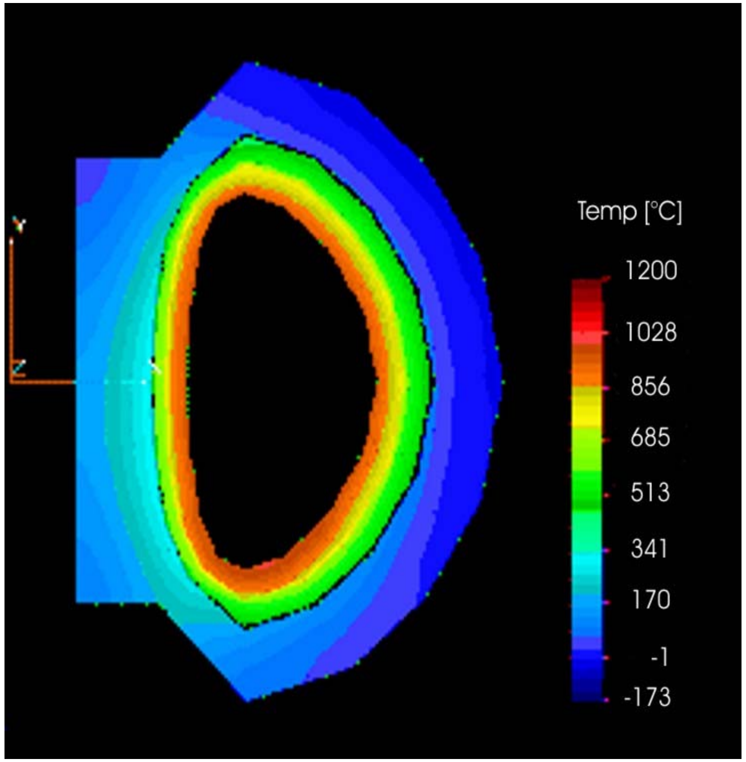
Research towards advanced materials

- Extrinsic toughening: composite materials
- Intrinsic safety: smart alloys
- Hydrogen isotope permeation: barrier layers

Summary

Accidential loss of coolant in reactor

Power plant conceptual study



Temperature profile in PPCS Model A, 10 days after accident with a total loss of all coolant.

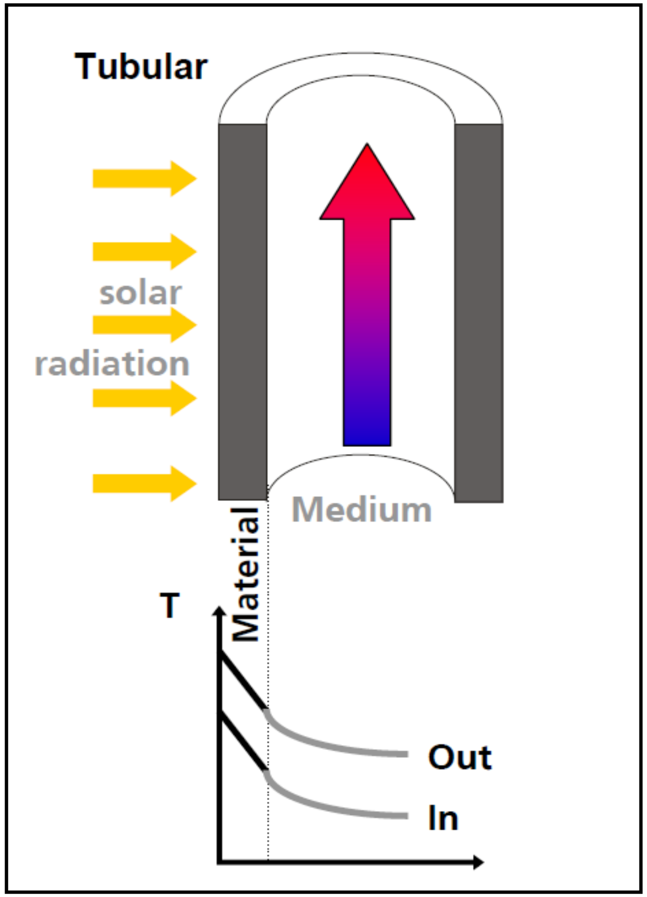
[Final Report of the European Fusion Power Plant Conceptual Study, 2004]

- Accidental loss of coolant: peak temperatures of first wall up to 1200 °C due to nuclear afterheat
- Additional air ingress: formation of highly volatile WO_3 (Re, Os)
- Evaporation rate: order of 10 -100 kg/h at >1000°C in a reactor (1000 m^2 surface)
  → large fraction of radioactive WO_3 may leave hot vessel

→ **Development of self-passivating tungsten alloys**

Concentrated solar power

Increased efficiency with high receiver temperature in air



- Highest potential: solar tower concept
- Status: Solar steam turbines: η <30%
- available receiver temperatures <900 °C
- aim: Solar gas and steam process

  receiver T: >1500°C, efficiency ~50%

Requirements for “smart” W alloys

Fusion

CSP

Normal operation conditions

Fusion

- W-dominated plasma-wall interactions
- Limited and controlled H isotope retention

CSP

- Operation in air
- Fast thermal gradients
- No element restrictions due to nuclear activation

After LOCA event
(loss of coolant accident)

- Strong reduction of oxidation rate
- Stable protective layer

General

- Large-scale bulk material production routes
- No formation of brittle phases

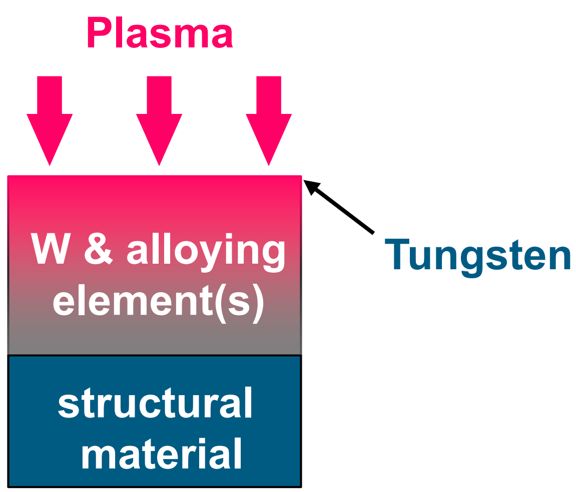
Idea: Tungsten alloys

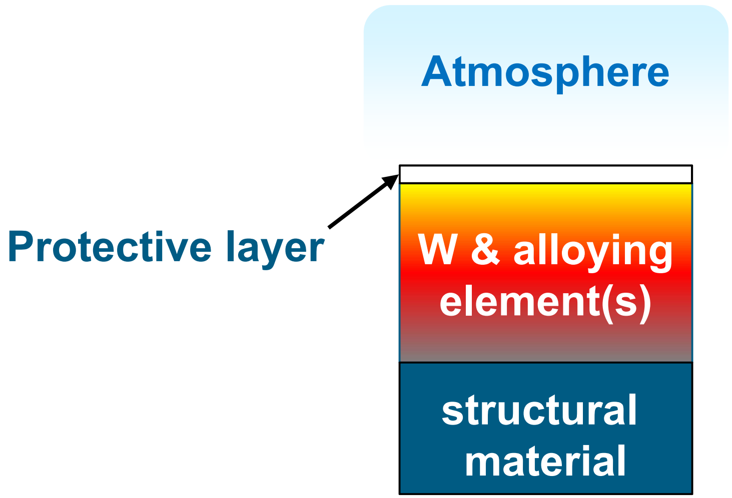
Self passivating tungsten-based alloys:

Surface composition automatically adjusts to the requested property

Normal operation (600°C):
Formation of tungsten surface by depletion of alloying element(s) due to preferential sputtering

Accidental conditions:
(air ingress, up to 1200 °C)
Formation of protective barrier layer

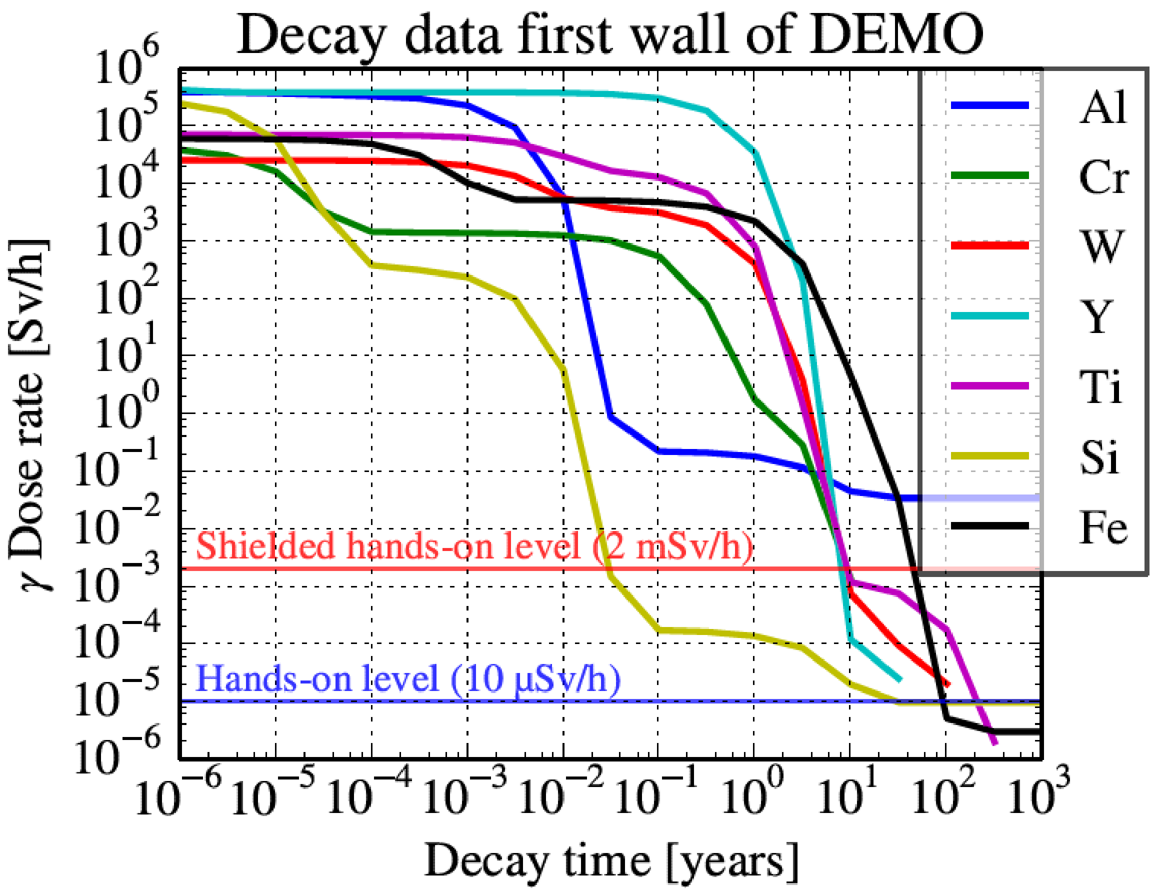




Behave like tungsten during plasma operation

Suppress oxidation during accident

Choice of alloying elements



Requirements

❖ Low neutron activation

→ **Cr, Ti, Si, Y**

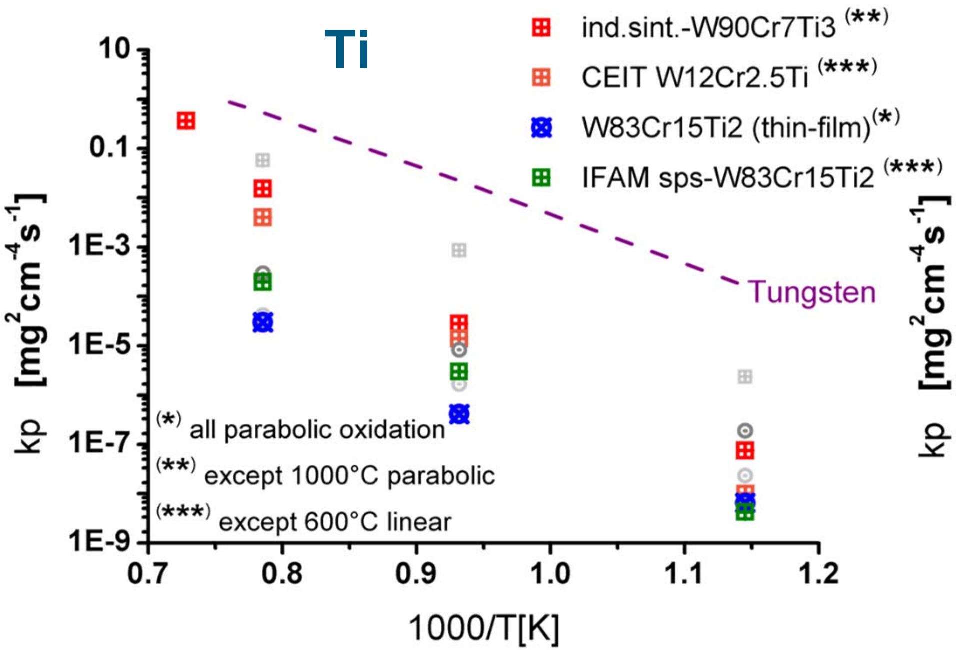
➕

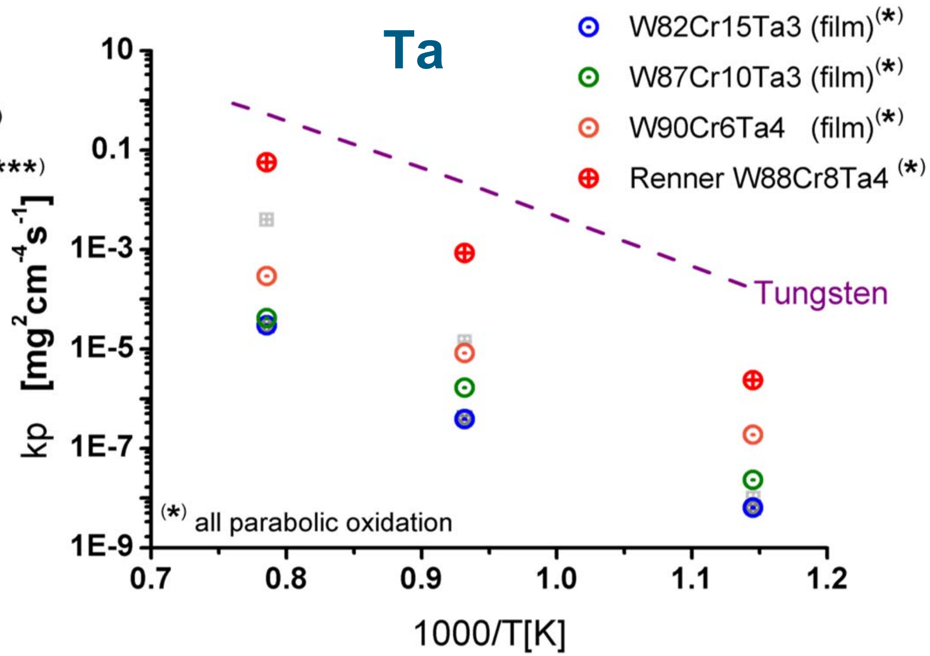
❖ Low volume increase by oxidation

❖ Good adhesion of the oxide to the alloy

❖ High melting point of alloys and oxides

Si-free alloys: W-Cr- (Ti / Ta / Y)





Reduction of oxidation rates

- Model thin films: several orders of magnitude
- Bulk materials: less reduction, different mechanisms?

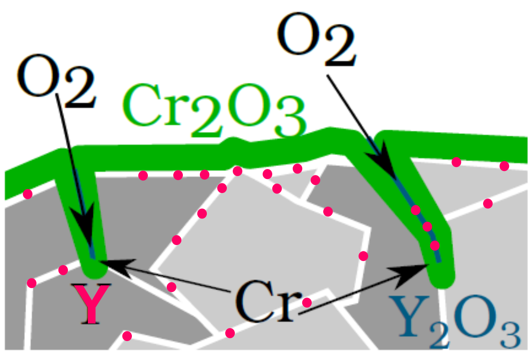
Composition

- Both Ti and Ta alloys successful
- Maximise W fraction:
  W-Cr6-Y0.04: 82 at% W
  Oxdiation rate <$5x10^{-6}$ $mg^2cm^{-4}s^{-1}$

Yttrium as active element

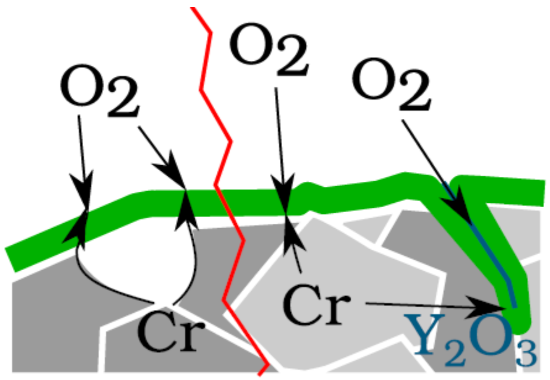
Y at the grain boundaries

- Smaller grains
- Thinner oxide layer



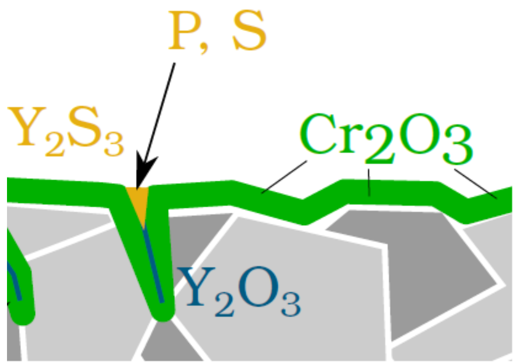
Y at the oxide-alloy interface

- Oxidation pegs, good adhesion
- Oxidation inwards to the surface
- Less pores



Reactivity towards impurities

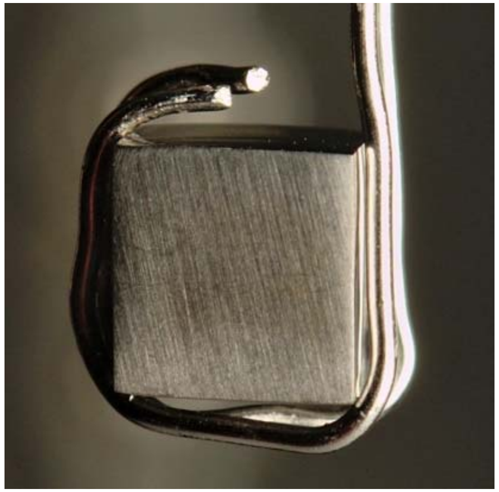
- Very stable oxide vs. impurities



Oxidation suppression: direct observation

10 hours, 1000 °C, dry air, 1 atm

Pure W



Before exposure

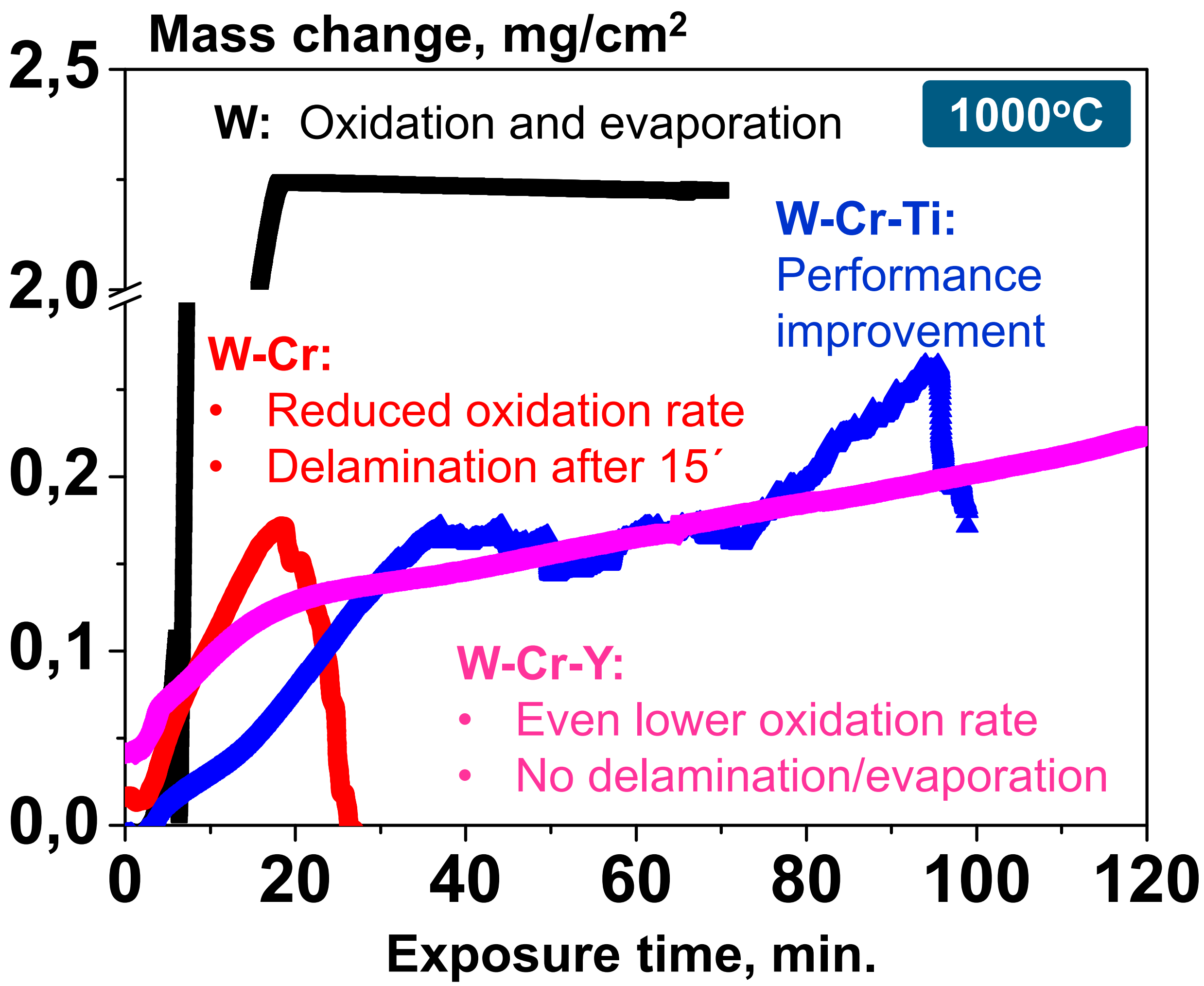
After exposure

W-Cr-Y alloy

Before exposure

After exposure

High temperature oxidation



Oxidation rates:

W: 0.52

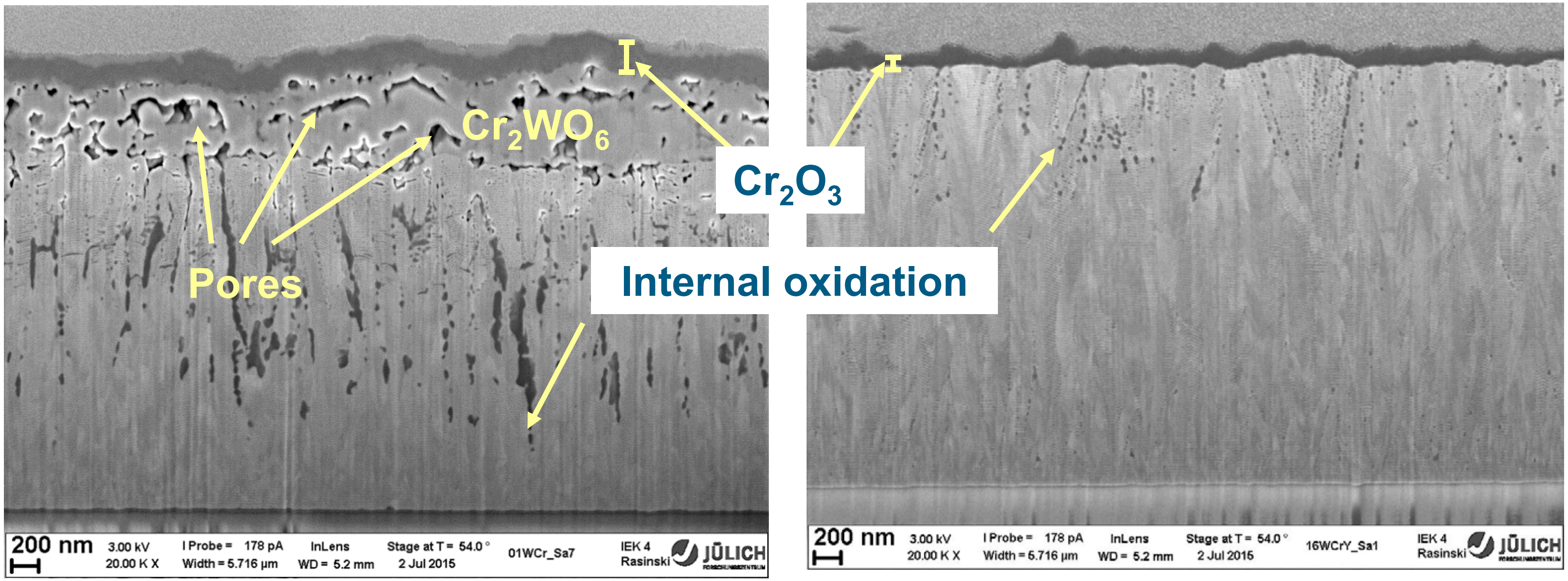
W-Cr-Y: $3*10^{-6}$

→ **Best passivation behavior of W-Cr-Y alloy**

Protective surface layer

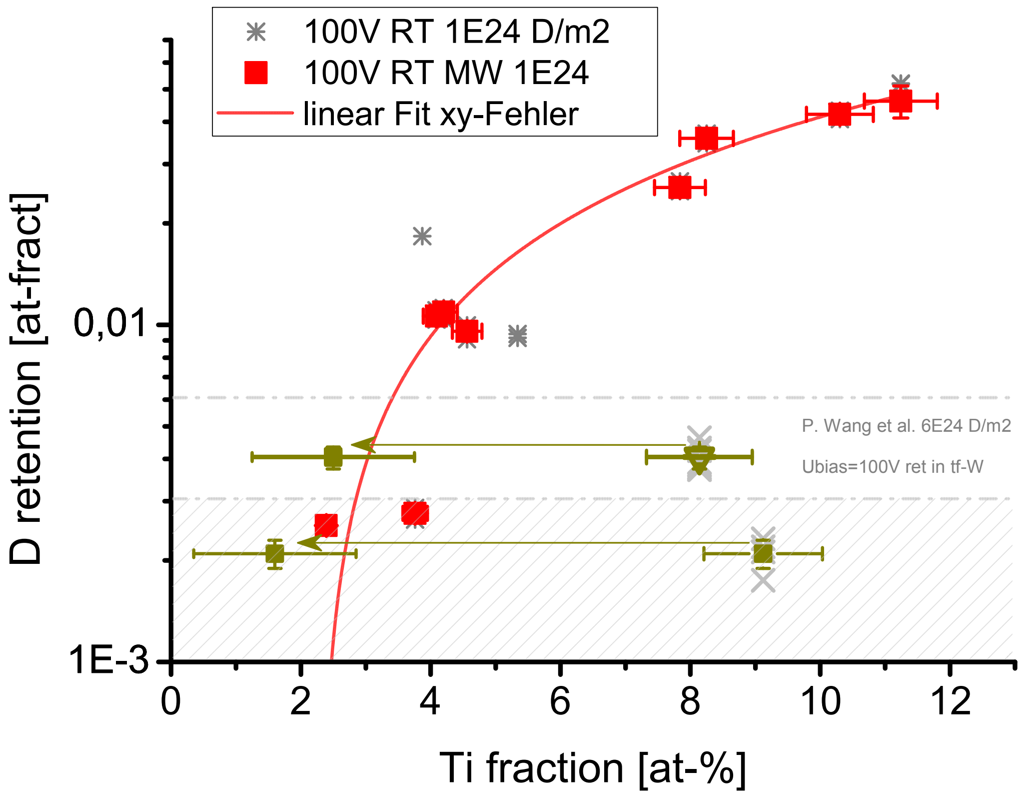
80 vol.% Ar + 20 vol.% O_2 | 1 bar | 1000°C | 15’

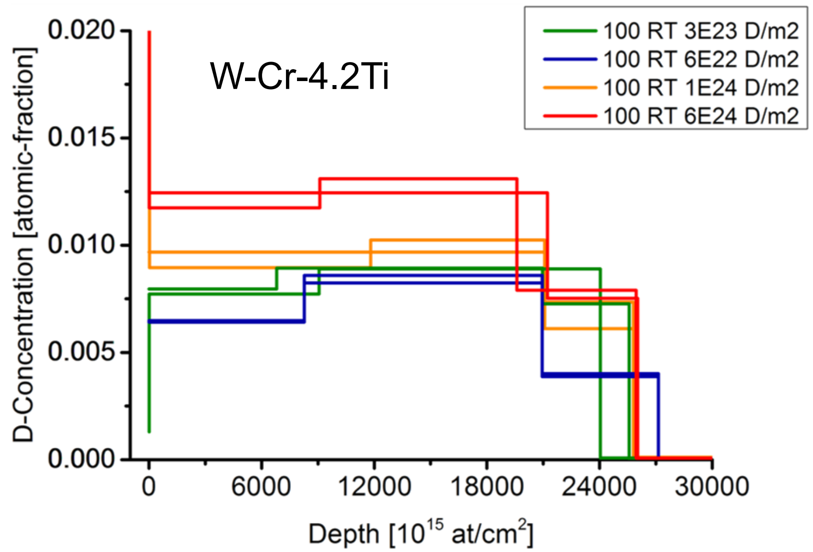
W-Cr | **W-Cr-Y**



- Smooth thin oxide layer in W-Cr-Y
- Suppressed internal oxidation
- No W-containing oxides
- No visible pores

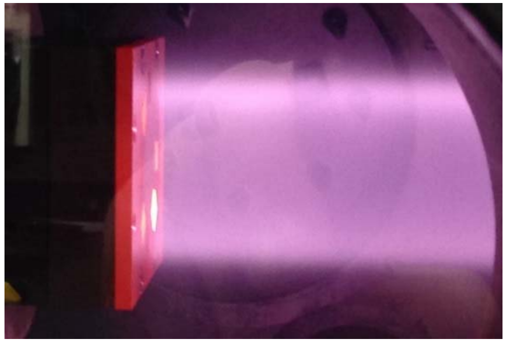
D retention after plasma exposure





- correlation between Ti concentration and D retention
- comparable to PVD tungsten for low Ti fraction
- bulk material: similar after Ti correction for oxide fraction

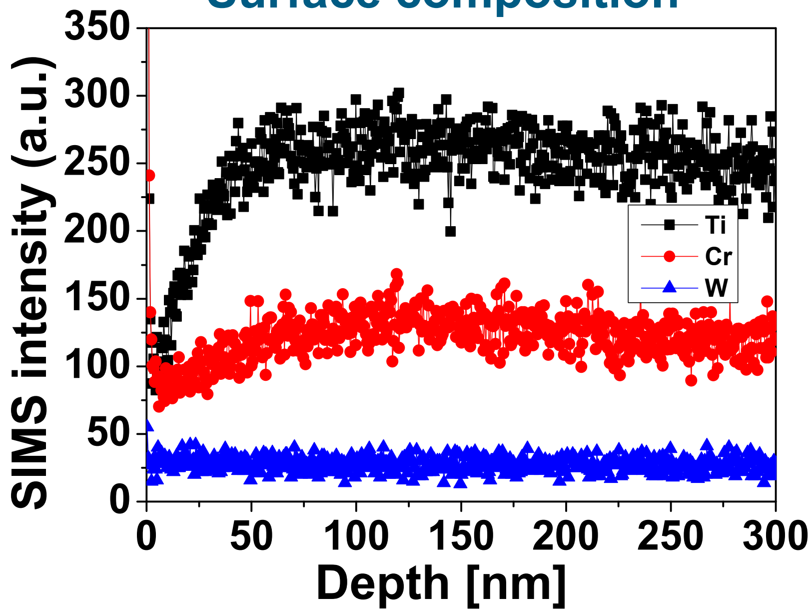
Plasma exposure of W-Cr-Ti alloys



PSI-2 linear plasma device

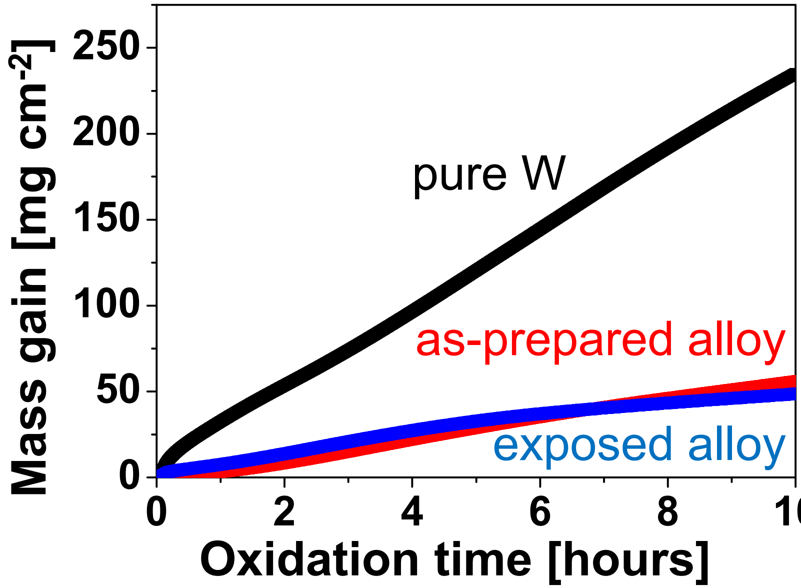
- D plasma, ion energy ~ 200 eV (bias -250 V)
- fluence 1.3 x 10^{22} D cm^{-2}d
- T_{sample}: 600-700 °C (FLIR)

Surface composition



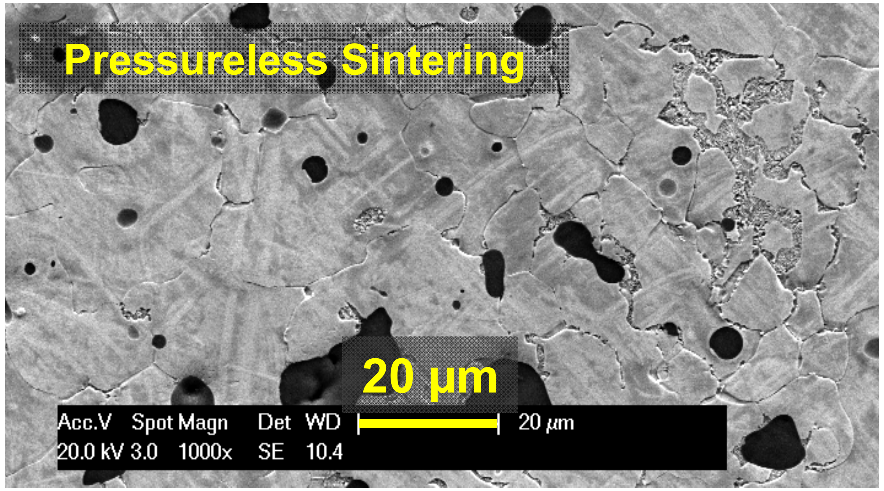
- constant W depth signal
- Cr, Ti depletion until ~60 nm
- W surface enrichment

Oxidation behavior

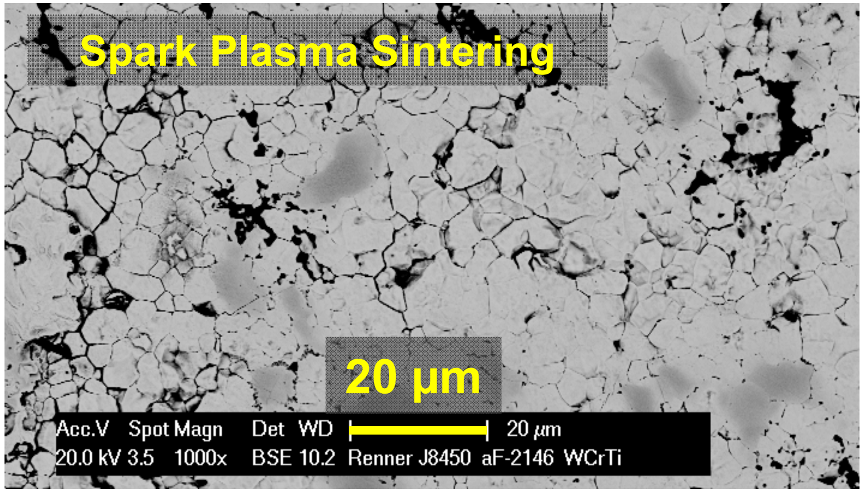


- no plasma influence on oxidation
- alloy: 1/3 evaporation rate
- linear oxidation – improvement required

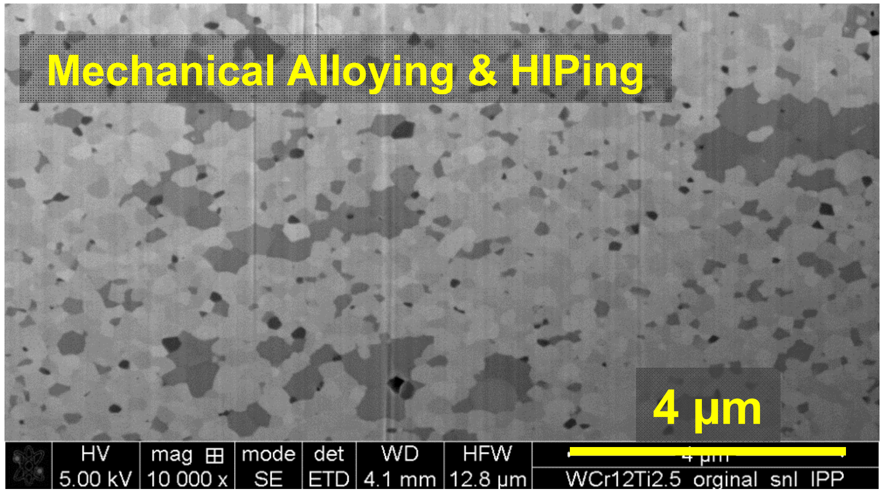
W alloy bulk production methods



T = 2000 °C, t = 30 min



T = 1474 °C, P = 64 MPa, t = 10 min



T = 1300 °C, P = 150 MPa, t = 60 min

MA + HIP:	submicron grains, Ti inclusions
PS:	grain size ~ 20 µm, TiO_x inclusions
SPS:	grain size ~ 10 µm, „cracked“ Ti inclusions
all:	Cr-rich/Cr-poor grains

Overview

Introduction: the material challenge

Neutron damaged materials

Research towards advanced materials

- Extrinsic toughening: composite materials
- Intrinsic safety: smart alloys
- Hydrogen isotope permeation: barrier layers

Summary

Hydrogen isotope permeation

Hydrogen diffuses easily in metals – Important in various application fields

Fusion

- Radioactive inventory and material embrittlement
- Permeation of T_2 into coolant
- Consider impact of Tritium inventory on TBR

⇒ Reduction of permeation by a factor 50…100 necessary

Concentrated solar power

- Hydrogen from thermal decomposition of heat carrier
- Diffusion through glass into insulating vacuum

⇒ Efficiency reduction of plant

Alternative chemical energy carriers, Hydrogen system

- Thermochemical synthesis: syngas, water gas shift reaction (H_2, CO, CO_2, CH_4)
- Loss of H_2: reactors, power plants
- Transport and storage of hydrogen

⇒ Wide ranges of temperature and composition for H_2 permeation

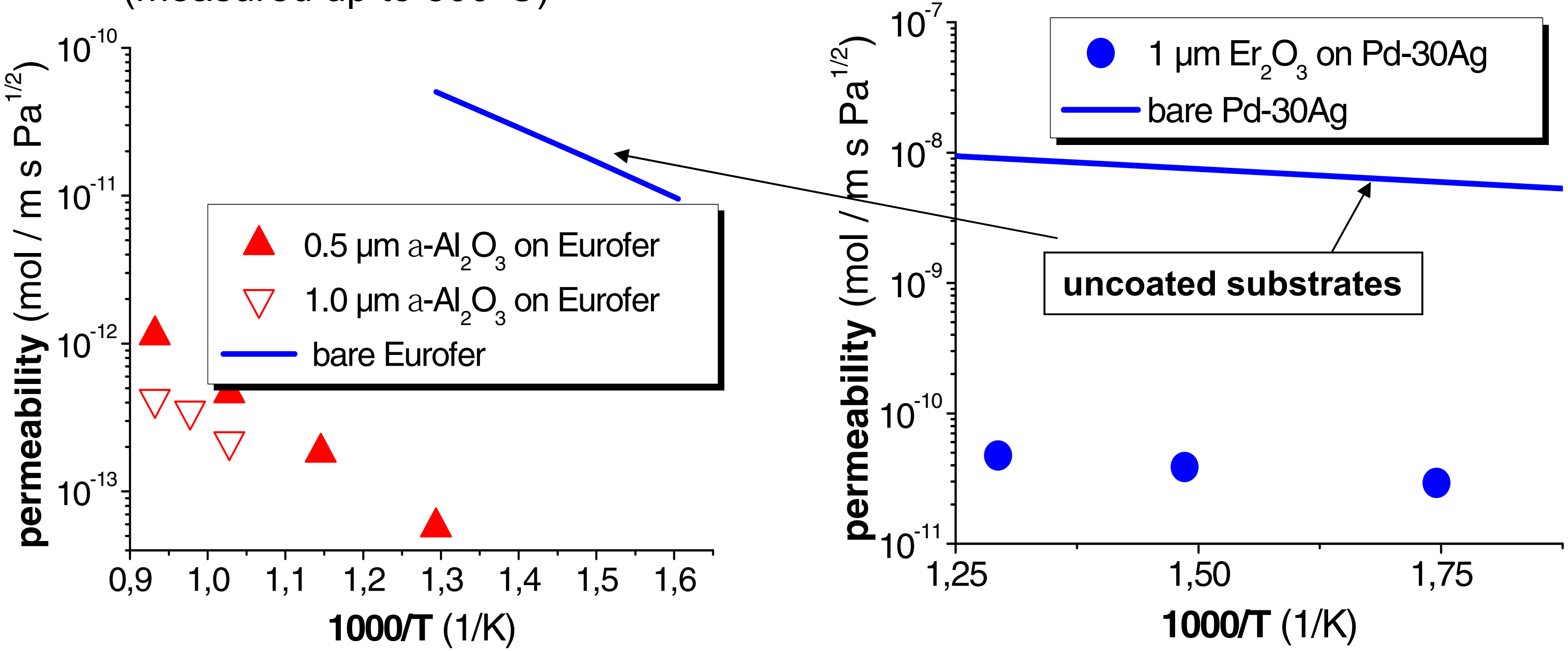
First tests: Al_2O_3 and Er_2O_3

Suppression of hydrogen permeation by **Al_2O_3 films** (α phase, from filtered arc):

permeation reduction ~ 1/1000
(measured up to 800°C)

Corrosion-resistant **Er_2O_3 films** (in liquid metals, e.g. Li-Pb):

permeation reduction ~ 1/200

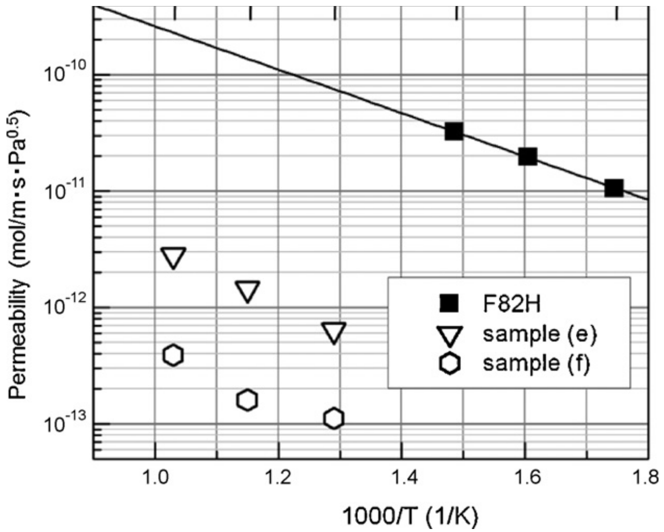


- ✓ Er_2O_3 and α-Al_2O_3: successful permeation reduction
- ✓ Er_2O_3 enables lower deposition temperatures

D. Levchuk, IPP

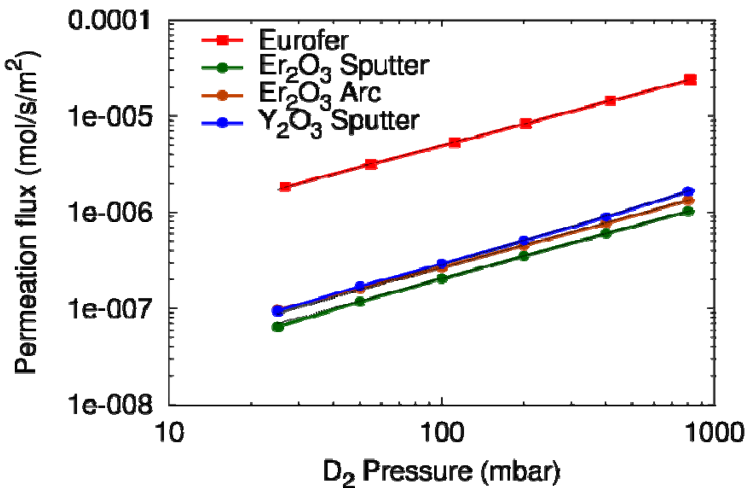
Barriers on 9-Cr steels

Er_2O_3 by sol-gel deposition



T. Chikada et al. / Fusion Engineering and Design 85 (2010) 1537–1541

Y_2O_3 by magnetron deposition



- Various deposition techniques: arc deposition, chemical routes, magnetron sputtering
- Hydrogen permeation is drastically reduced by applying erbia, alumina or yttria
- Thin (< 1µm) layers stable (no cracks) during thermal cycling

Reduction of permeation by a factor 50…100

Summary

Advanced materials for DEMO / a fusion reactor

- Combination of neutron / thermo-mechanical / particle loading
- No operational window for available materials
- Development and testing for new material (composites) required

W fiber / W matrix composites

- Development of W-CVI and powder metallurgical routes
- Verification of toughening effect: Stable crack propagation + rising load bearing capacity: damage tolerance
- Active toughening mechanism for fully brittle samples: resistance against embrittlement

Self-passivating W alloys

- Up to 10^5-10^6 reduction of oxidation rates for ternary alloys
- Transfer from thin films to bulk material successful
- Combine extrinsic toughening concept with new alloys!

Outlook

Multi-component materials

- Combination of materials solutions: brittle alloys with composites
- Hydrogen isotope inventory (PWI processes):
  - Dynamic evolution of composition during operation
  - Composites and alloys: new transport/trapping channels for T
  - n-induced damage: increased T retention?

Applications: DEMO, CSP, Alternative fuels

- Thermomechanical properties after 14 MeV neutron irradiation?
- Neutron damage: large T inventory, erosion behavior?
- Fusion and other energy applications: similar requirements, similar material and failure criteria: synergistic research

end of presentation