

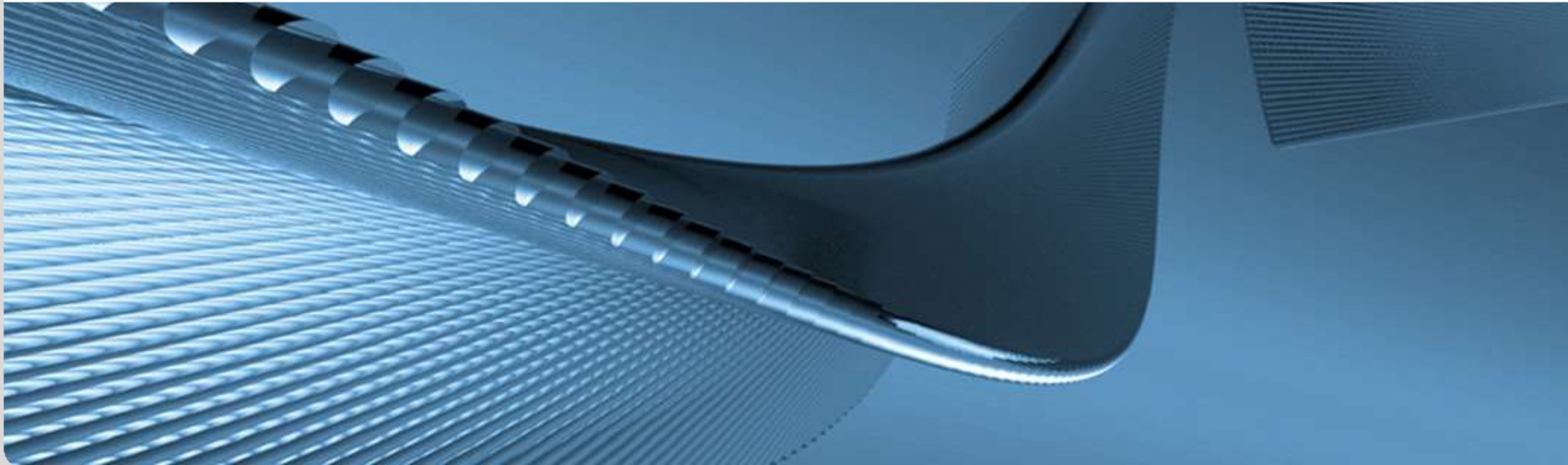
# TUNGSTEN

**An overview of production, basic properties, processing and divertor applications in fusion power plants**

**Michael Rieth**

**D. Armstrong, ... (U. Oxford),  
A. Hoffmann, W. Knabl, W. Schulmeyer, H. Traxler, ... (PLANSEE),  
S. Antusch, L. Commin, W. Krauss, A. Nemeth, J. Reiser, ... (KIT)**

KARLSRUHE INSTITUTE OF TECHNOLOGY – Campus Nord, INSTITUTE FOR APPLIED MATERIALS – Applied Material Physics (KIT, IAM-AWP)



# CONTENTS

## ■ Intro: Tungsten basics & production

- + PIM (Steffen)
- + Oxidation (Lorelei)

Divertor Workshop KIT  
& nächstes Germ. DEMO meeting

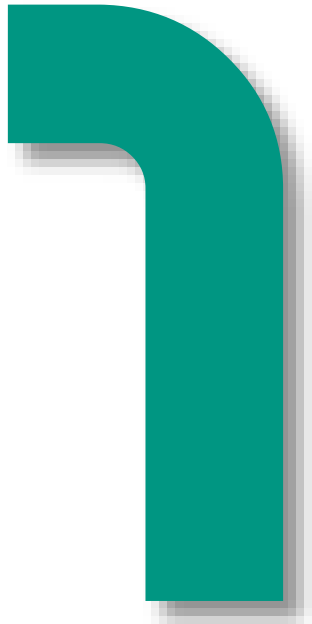
## ■ Machining & Processing

- Mechanical
- Electro-chemical

## ■ Divertor design

- Armor
- Water cooling
- He cooling

## ■ Summary and path forward



# TUNGSTEN BASICS

# REACTIONS



**W**<sup>183.85</sup><sub>74</sub>

## Reactions with Non-metals

C	>800 °C	W <sub>2</sub> C, WC
O	>400 °C	WO <sub>3</sub> → sublim. at 800 °C
S	>400 °C	WS <sub>2</sub>
F	>RT	WF <sub>6</sub> (volatile)
F+O	>RT	WOF <sub>4</sub>
Cl	>250 °C	WCl <sub>6</sub>

## Reactions with Compounds

H <sub>2</sub> O	>RT	WO <sub>2</sub> (OH) <sub>2</sub> (volatile)
H <sub>2</sub> S	>350 °C	WS <sub>2</sub> + 2H <sub>2</sub>
SO <sub>2</sub>	300 °C	WO <sub>3</sub> + S <sub>2</sub>
CO	80-200 °C	tungsten hexacarbonyl
	>1000 °C	W <sub>2</sub> C + CO <sub>2</sub>
SiC	>1100-1900 °C	WC and WSi
SiO <sub>2</sub>	>2000 °C	slight oxidation
Al <sub>2</sub> O <sub>3</sub>	<1900 °C	compatible in vacuum
BeO	<2000 °C	compatible in vacuum
MgO	<1500 °C	compatible in vacuum
ZrO <sub>2</sub>	<1600 °C	compatible in vacuum
Glass	<1400 °C	stable

# ALLOYING ELEMENTS / PHASES

(W RICH SIDE IN THE PHASE DIAGRAM AT ROOM TEMPERATURE)

$M_2W$ <b>Be</b>	<b>Mg</b>	$MW$ <b>B</b>	$MW_2$ <b>C</b> $M_{1-x}W$	$M_4W$ <b>Al</b>		<b>Y</b>	<b>La</b>	
<b>Ti</b> >3wt.% >300°C	<b>V</b>	$MW_3$ <b>Cr</b>	<b>Mn</b>	$MW, M_7W_6$ <b>Fe</b>	$M_7W_6$ <b>Co</b>	$MW$ <b>Ni</b>	<b>Cu</b>	
$MW_2$ <b>Zr</b>	<b>Nb</b>	<b>Mo</b>		<b>Ru</b> < 3 wt. %	<b>Rh</b> < 2 wt. %	$M_3W$ <b>Pd</b>	<b>Ag</b>	<b>Cd</b>
$MW_2$ <b>Hf</b>	<b>Ta</b>		$MW$ <b>Re</b> < 26 %	<b>Os</b> < 5 %	$MW$ <b>Ir</b>	$MW$ <b>Pt</b>	<b>Au</b>	

Insoluble

Intermetallic Phases

Line Compounds

Solid Solution

# POWDER-METALLURGICAL PRODUCTION ROUTES

## Industrial Route

- Blending
- Pressing
- Sintering
- Hot forming

- + Cost effective
- + Large quantities/  
Semi-finished products
- + High density
  
- ± Specific, anisotropic  
micro-structure

## Mass Fabrication

- Blending (+Binder)
- Injection Molding
- Debinding/Sintering
- HIP (optionally)

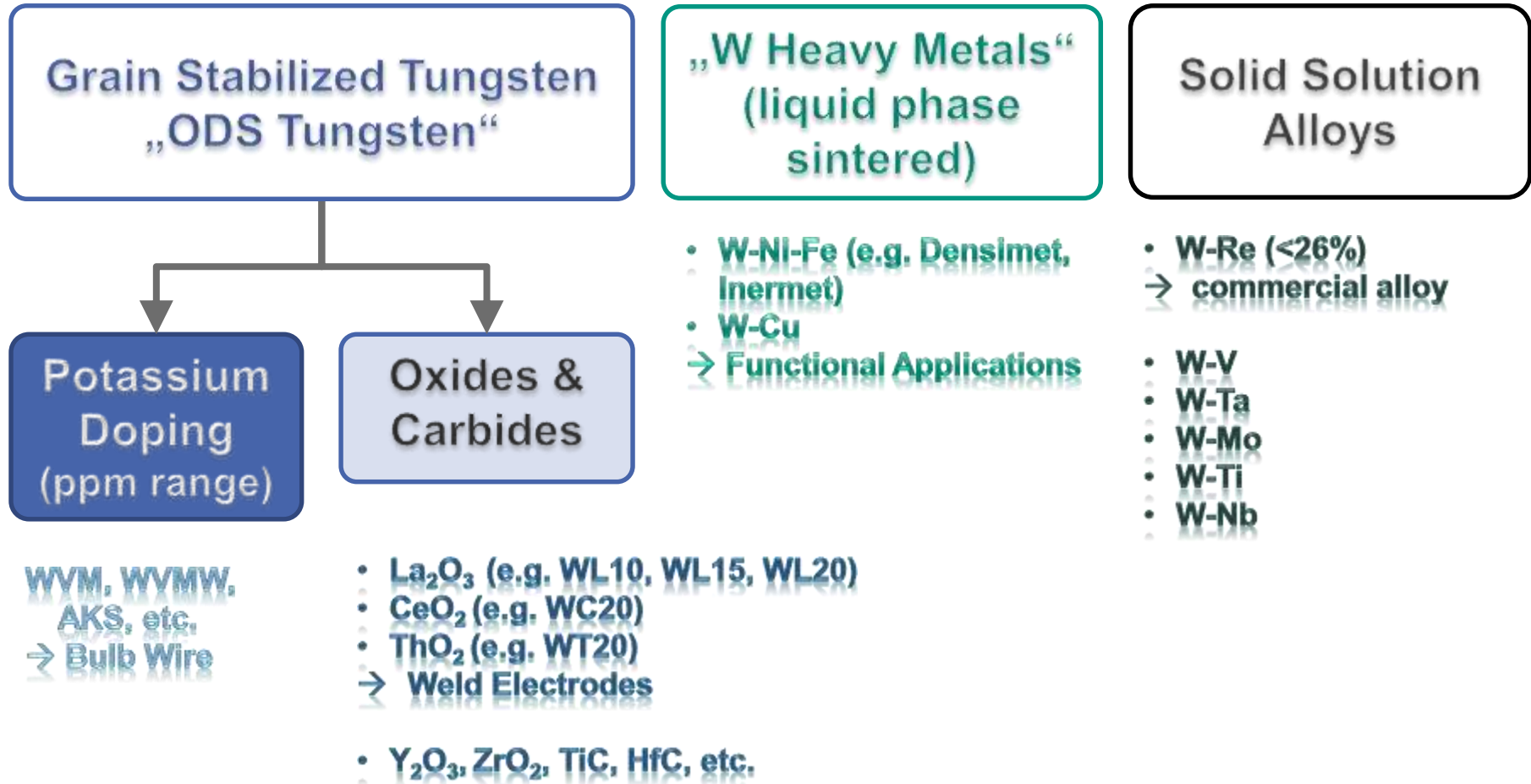
- + Mass production of  
near net-shaped parts
- ± Homogenous  
microstructure
  
- Rest porosity

## Mechanical Alloying

- Blending
- Ball Milling
- Encapsulation
- HIP or hot forming

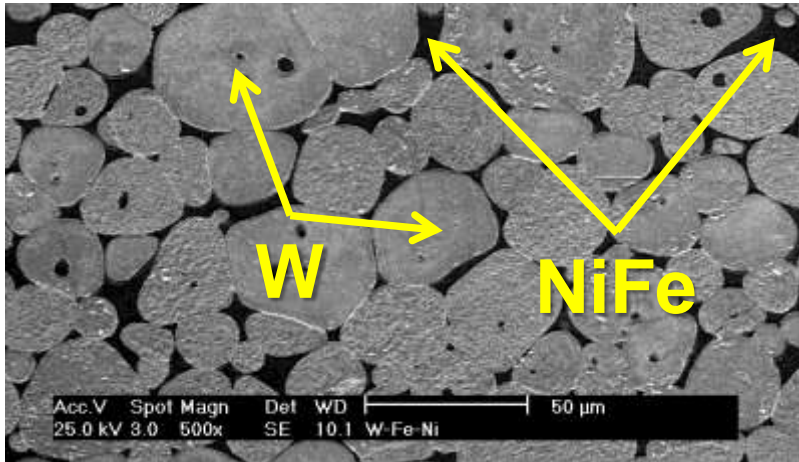
- + Higher ductility  
possible
  
- Small scale production  
route

# TUNGSTEN MATERIALS (BESIDE PURE W & WC)

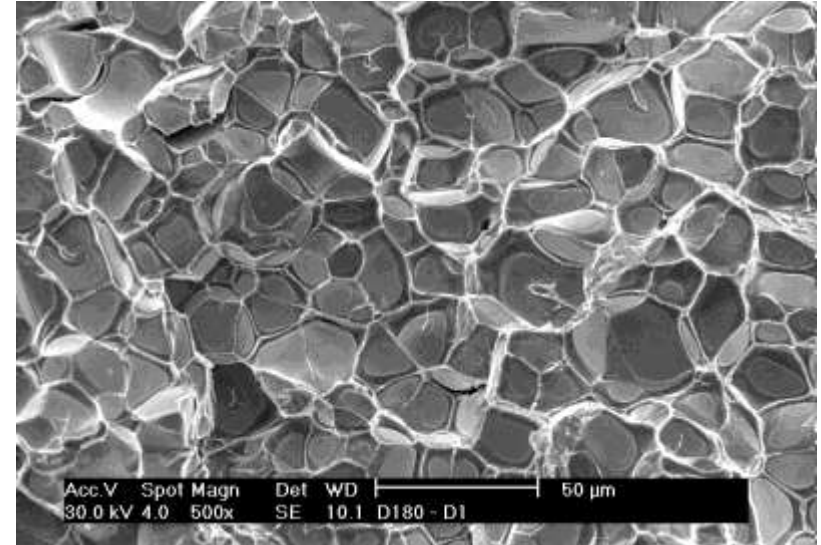


# MICROSTRUCTURE OF “W HEAVY METALS”

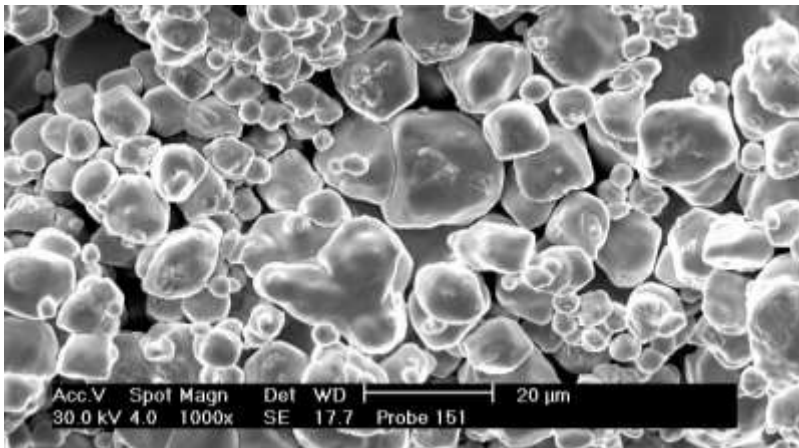
## DENSIMET D180, W-3.5Ni-1.5Fe



## Cold fracture surface



## W-Cu, hot fracture surface



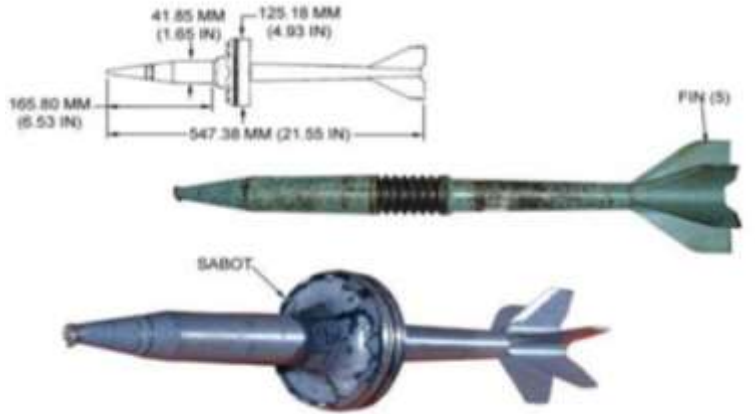
### DENSIMET D180

Density	18 g/cm <sup>3</sup>
Young's mod.	380 GPa
Therm. Cond.	83 W/mK
Therm. Exp.	5.5x10 <sup>-6</sup> /K
ferromagnetic	

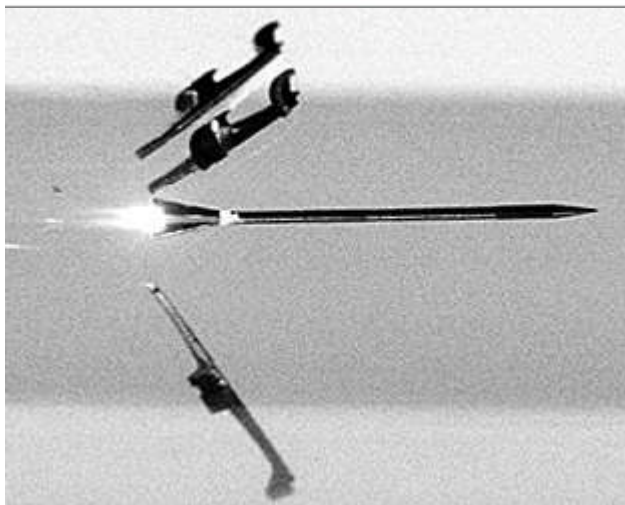


# APPLICATIONS

## Heavy Metal



and ailerons in modern aeroplanes (indicated by the arrows); the weights are made of tungsten up to 850kg of counterweights are used in a Boeing 747



different kinds of "green" ammunition for waterfowl and game hunting



Polymer-tungsten sheet with a density of 11g/cm<sup>3</sup>. It is used as a substitute for lead.

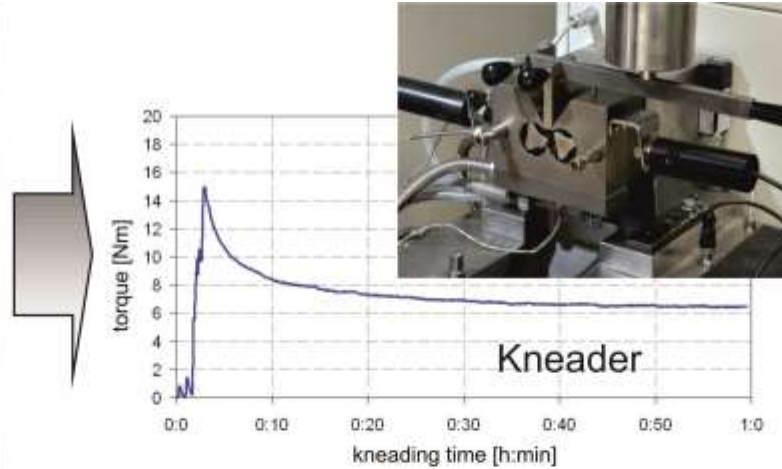
# APPLICATIONS

## Cemented Carbides



Coated inserts indicating the manifold geometries which exist to different cutting operations

# The KIT PIM process for tungsten



Feedstock development



debinding + heat-treatment process

# The KIT PIM process for tungsten



# The KIT PIM process for tungsten



**green parts**



**finished parts**

# Summary (wrt Divertor Applications)

## ■ Preferred Production Routes

- Industrial Sintering & Hot Forming
- Powder Injection Moulding → mass fabrication of near net-shaped parts

## ■ Potential Tungsten Materials

- Pure W and Potassium doped W
- W Solid Solutions → W-V, W-Ti, W-Ta
- Grain Stabilised W →  $\text{La}_2\text{O}_3$ ,  $\text{Y}_2\text{O}_3$ , TiC (& other carbides)
- W Heavy Metals → only W-Cu

## ■ Perfect Tool for W Materials Development

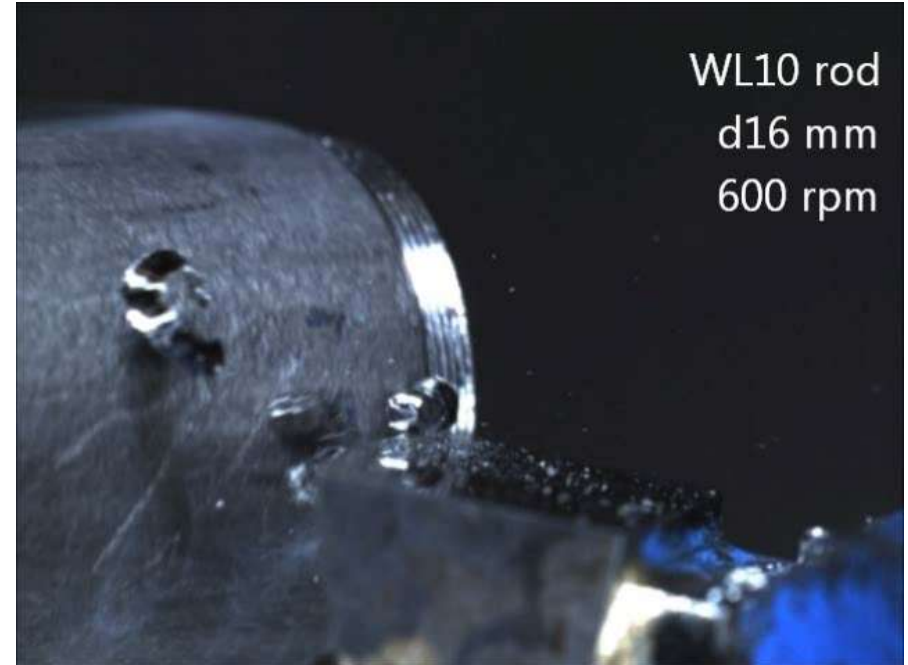
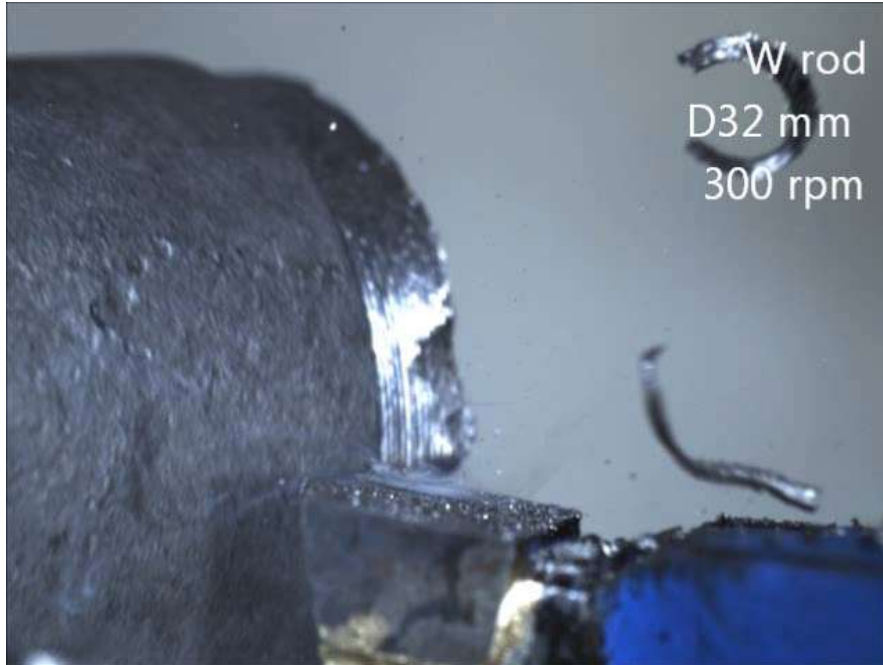
- PIM → “Rapid Prototyping of W Alloys”

2

MACHINING

# Mechanical Machining

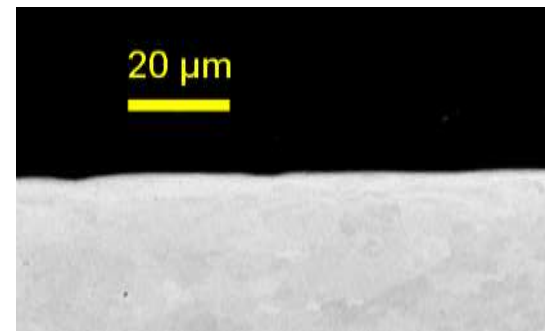
## ■ Turning



## ■ Milling

## ■ Grinding

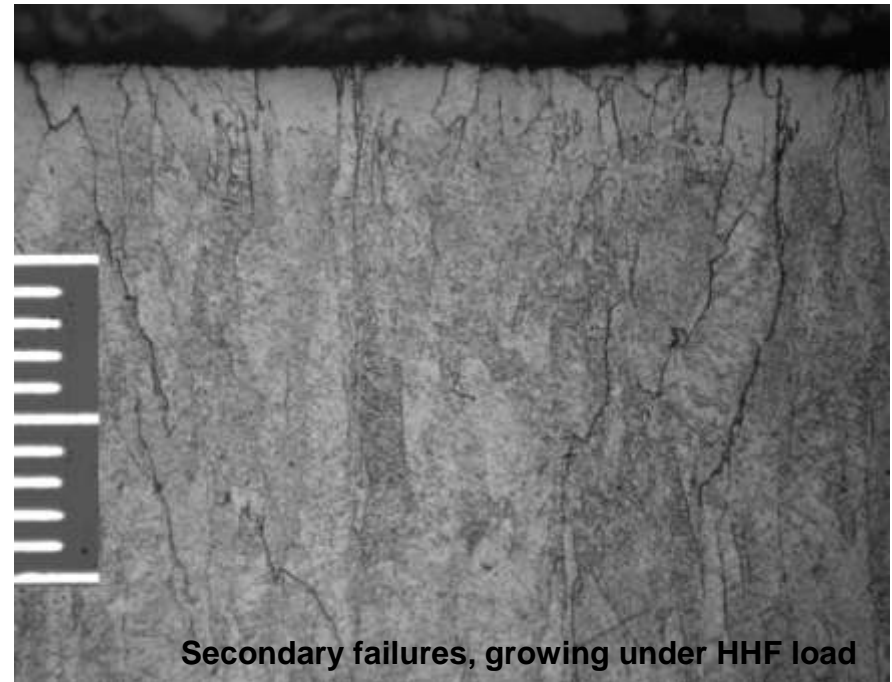
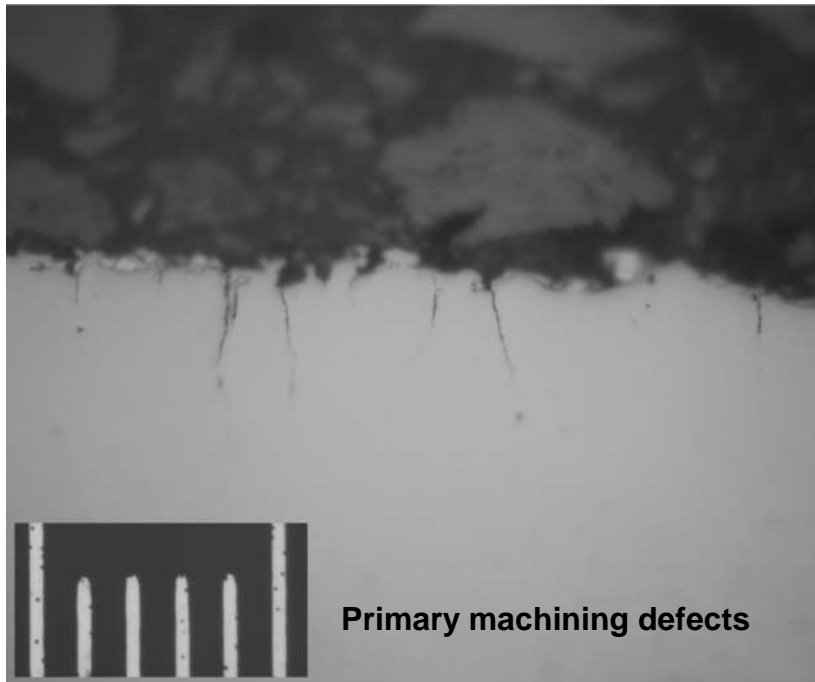
- Possible with acceptable surface quality
- Pure W is harder to machine





# Electro-Discharge Machining

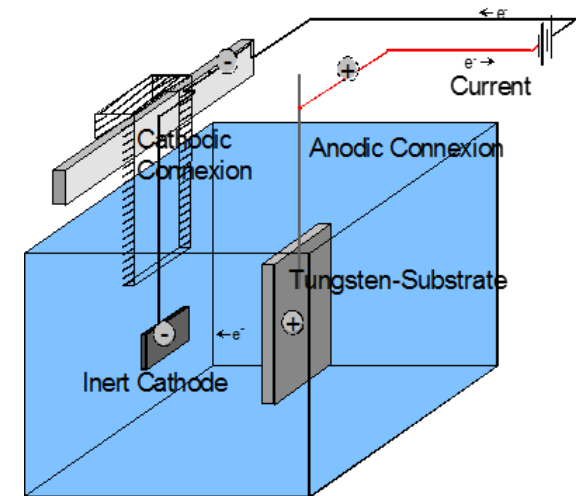
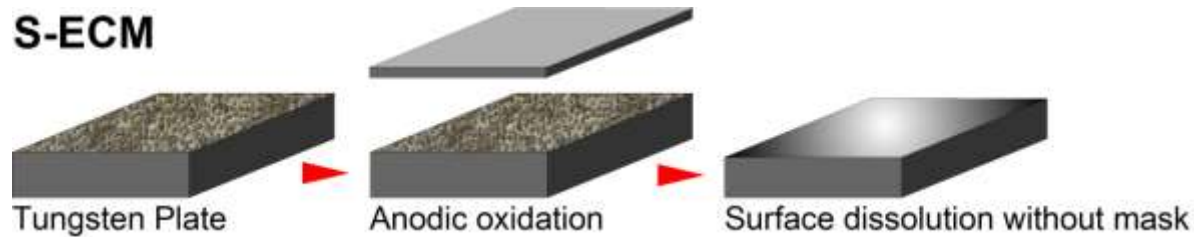
- No problem
- BUT: Surface crack formation



# Electro-Chemical Machining

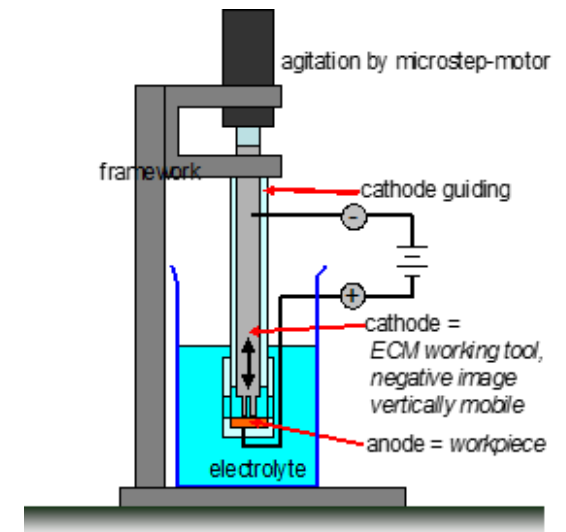
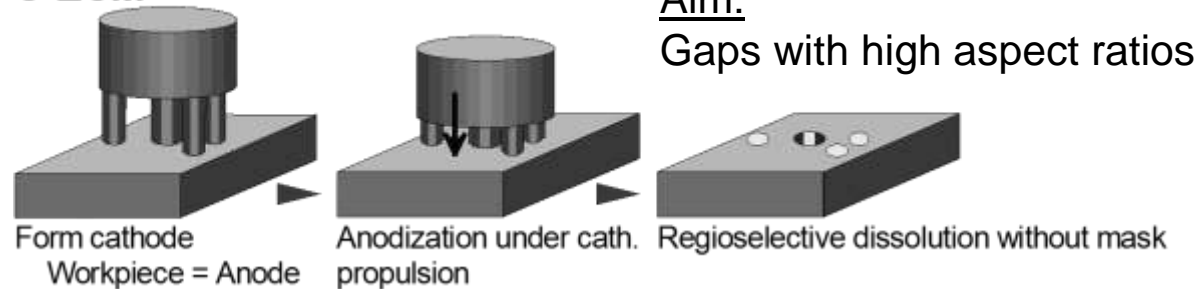
- Surface finishing on the micron scale → S-ECM

## S-ECM

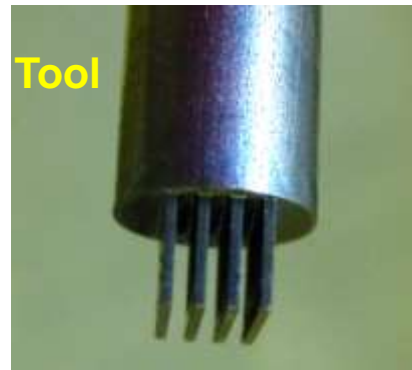


- Bulk structuring → C-ECM

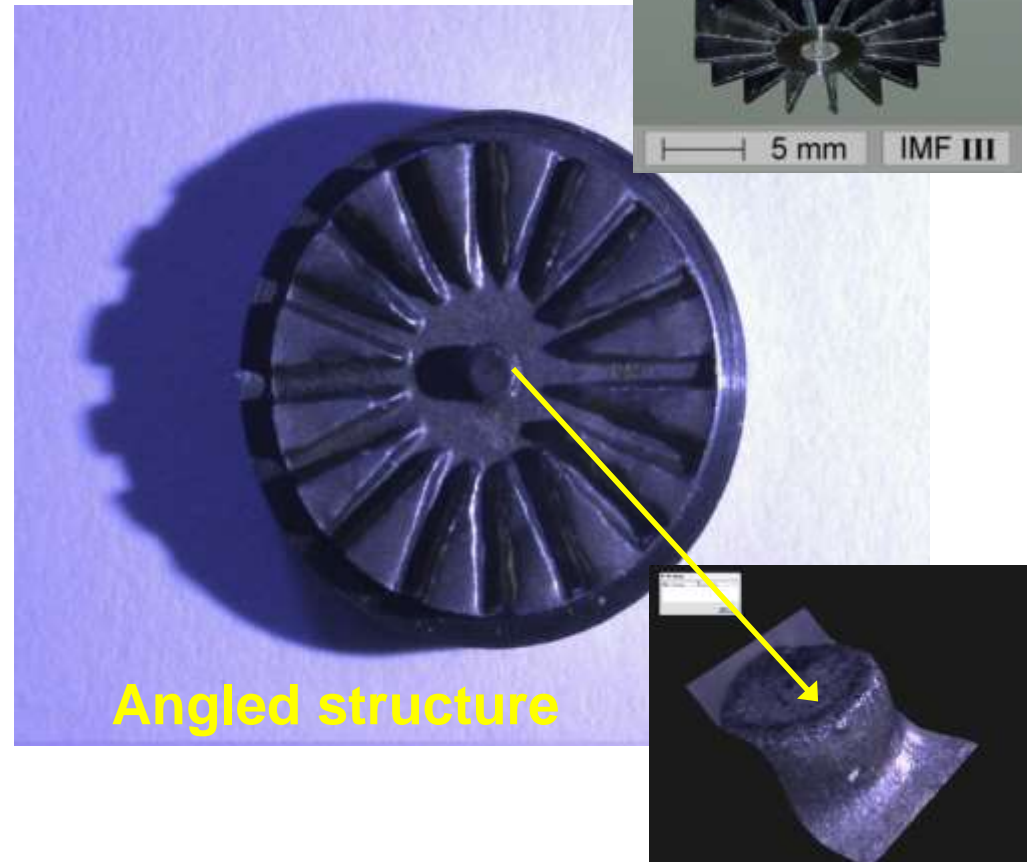
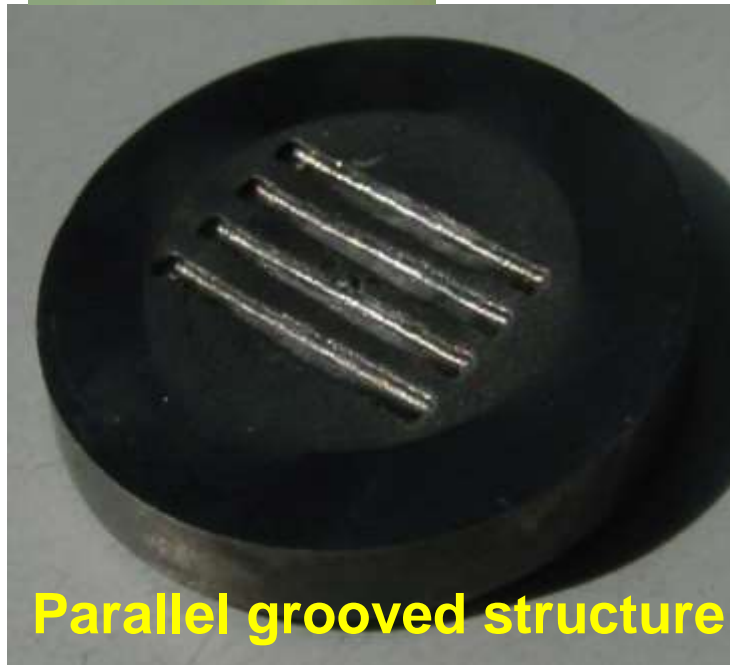
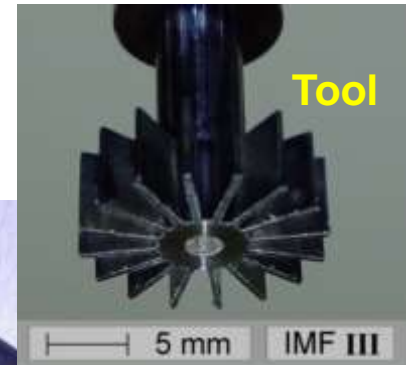
## C-ECM



# Electro-Chemical Machining

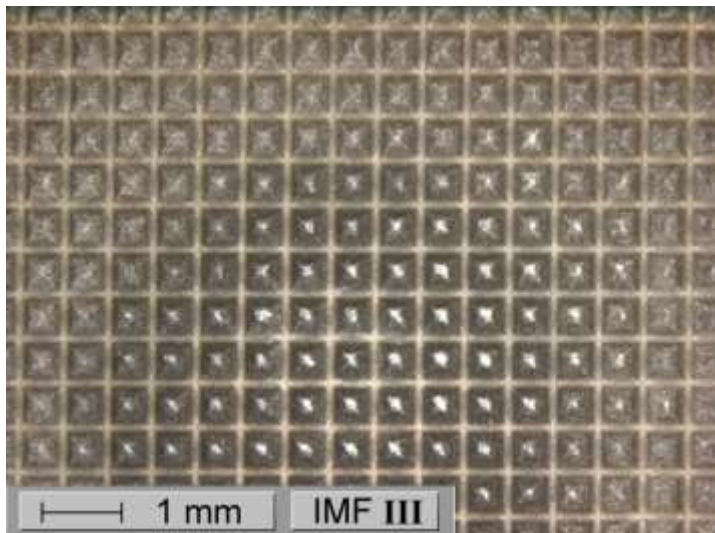


## Some Examples

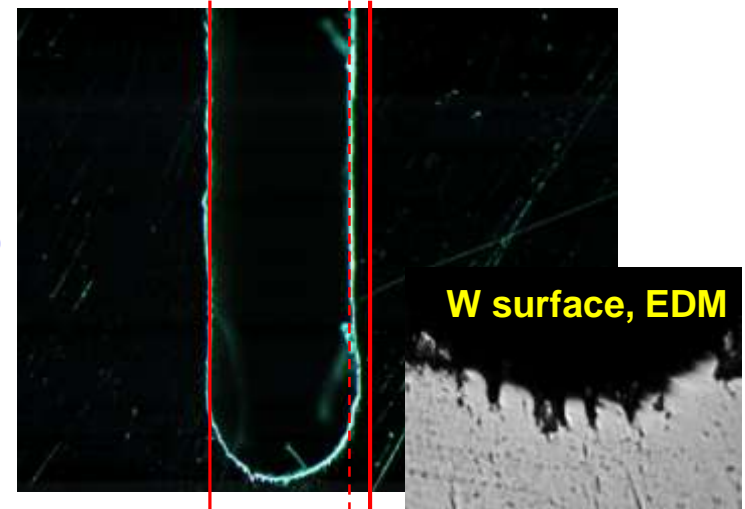


# Electro-Chemical Machining

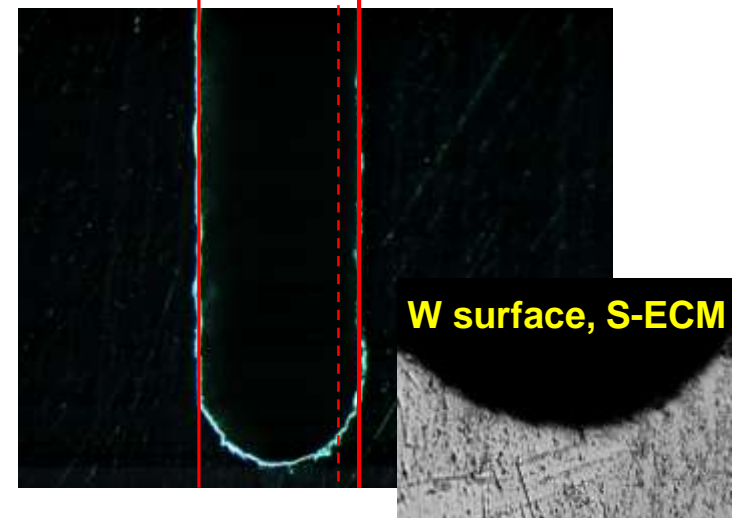
Surface treatment:  
*Electro-polishing*



*Electro-discharge  
machining*



*Electro-chemical  
machining*



# B

# PROCESSING

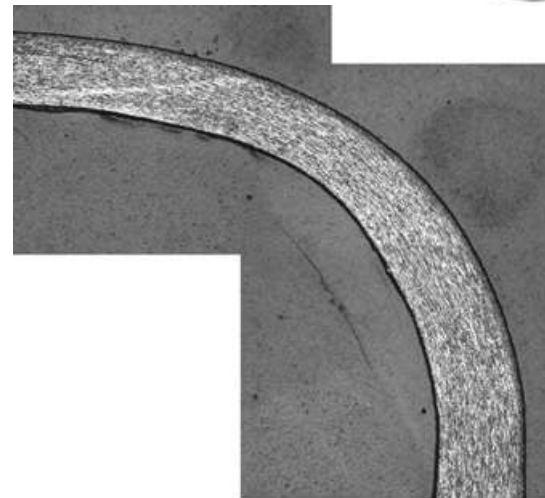
# Deep Drawing & Bending



10 mm



10 mm

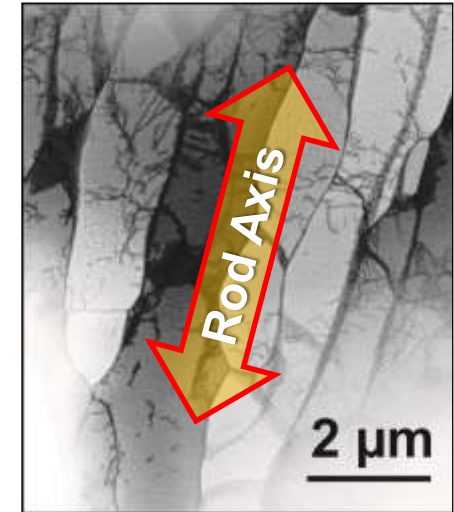
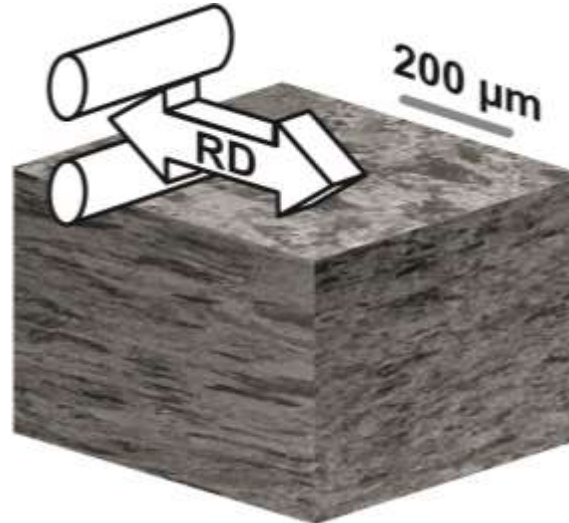
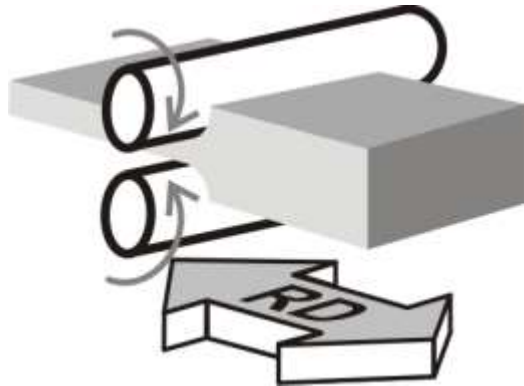


1mm

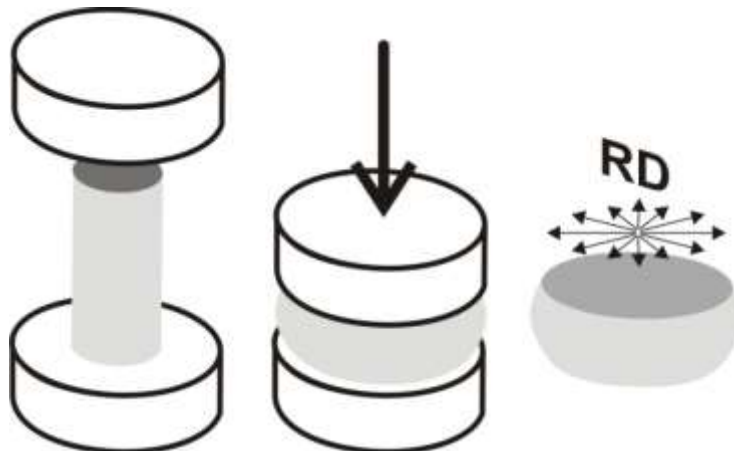
→ Possible at 600 °C with very low strain rate (in vacuum)

# HOT FORMING

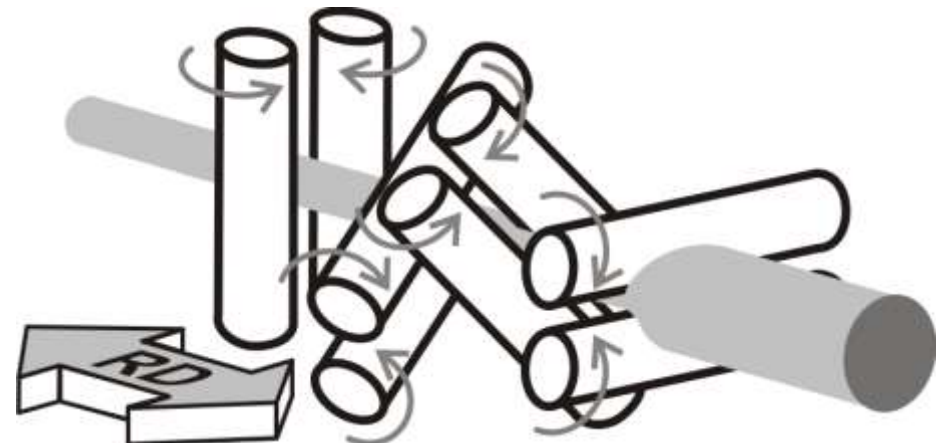
## Rolling Plates



## Forging Round Blanks



## Rolling/Swaging Rods



# Hot Forming







# OXIDATION IN AIR

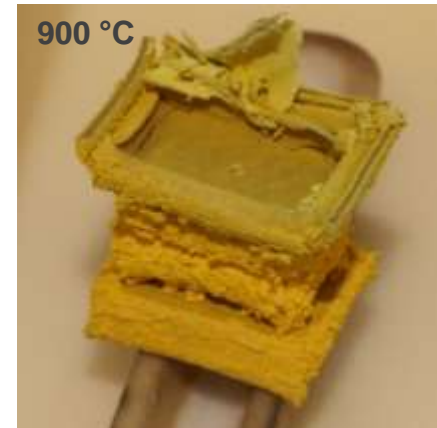
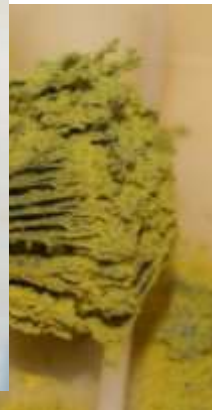
## W plates

24 hours

PIM W

96 hours

48 hours

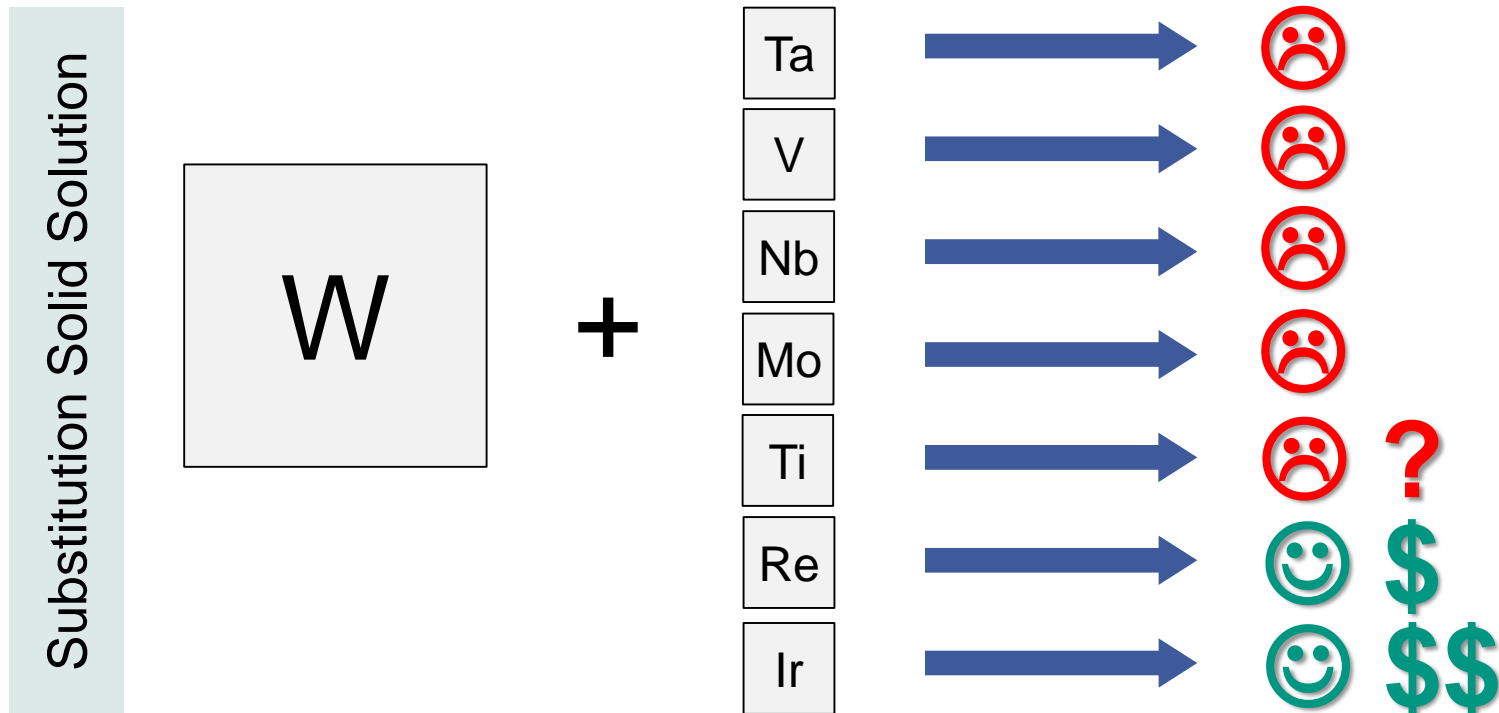


# DUCTILISATION

## Known Strategies

1. Nanostructuring
2. Composites
3. Alloying

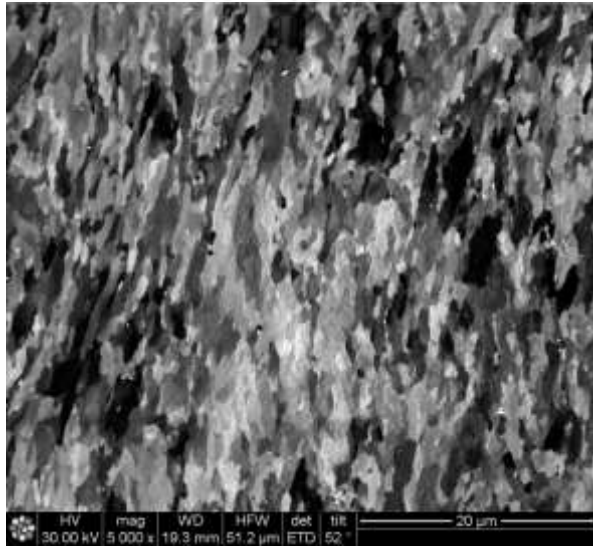
→ **Small quantities only!**  
→ **Not yet commercially available!**



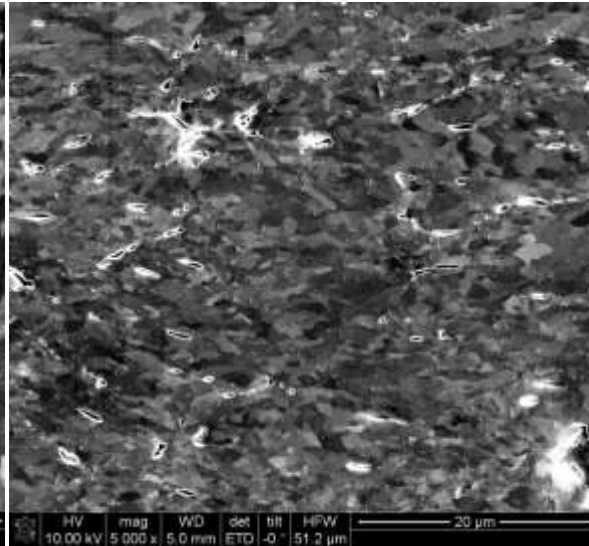
**Conclusion:** For large-scale applications we have to live with the intrinsic brittleness of tungsten materials!

# MICROSTRUCTURE

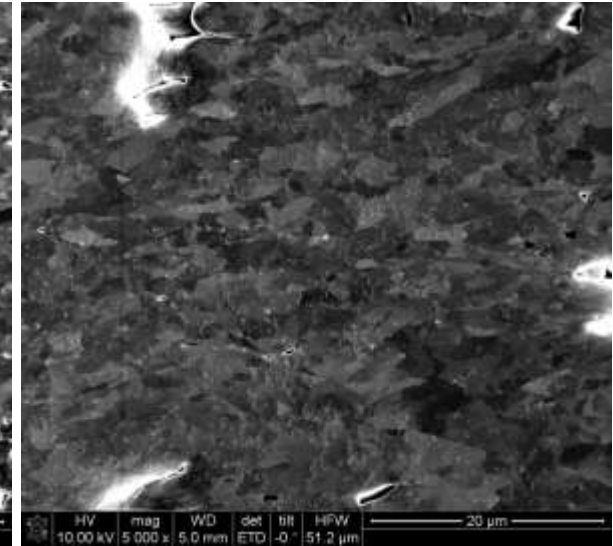
### W-5%Ta



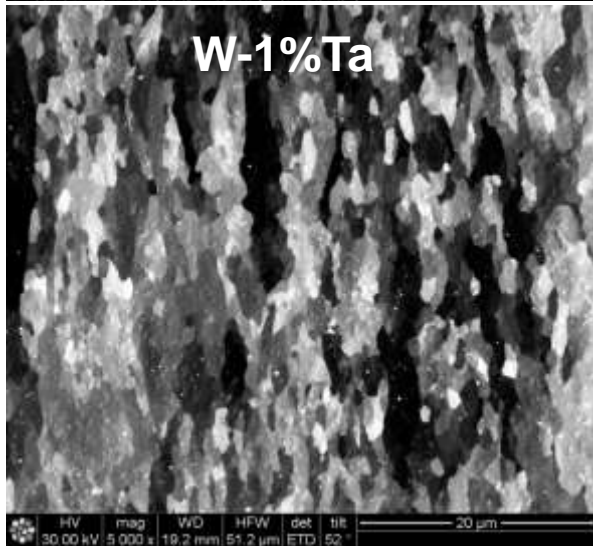
### W-5%V



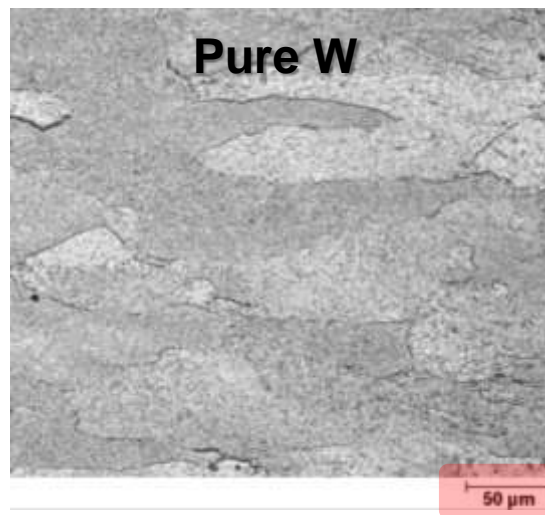
### W-20%Mo



### W-1%Ta

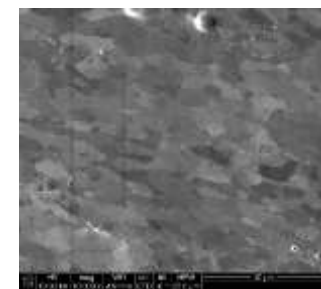


### Pure W

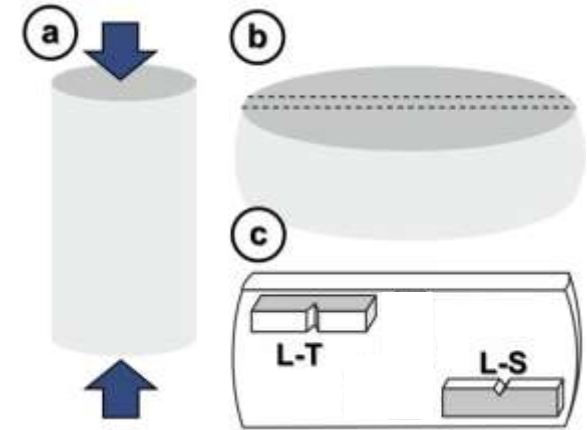
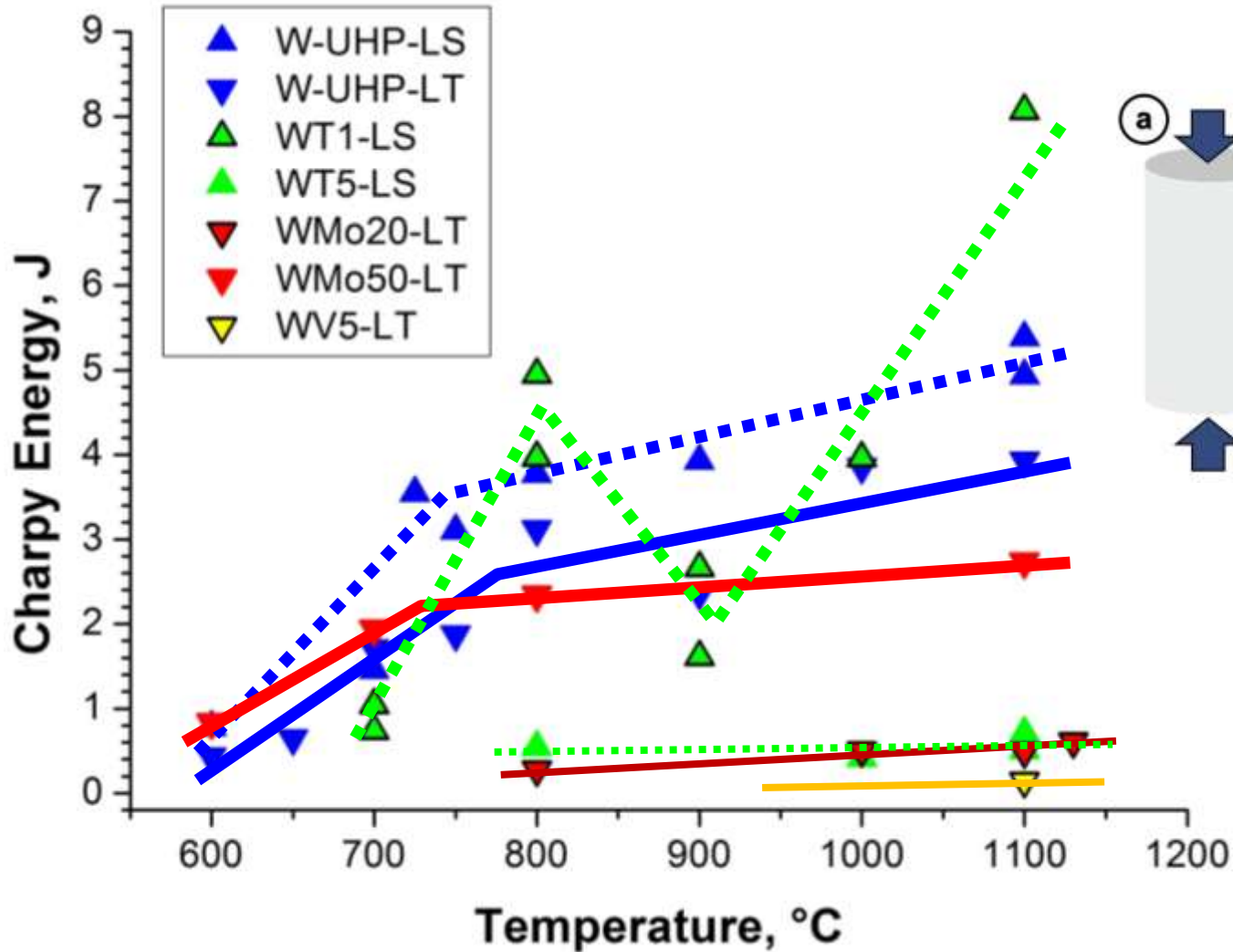


### 20 μm

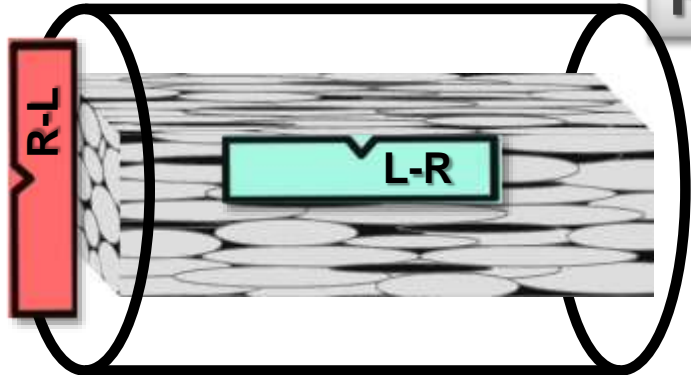
### W-50%Mo



# ALLOYING EFFECT (ON DUCTILITY)



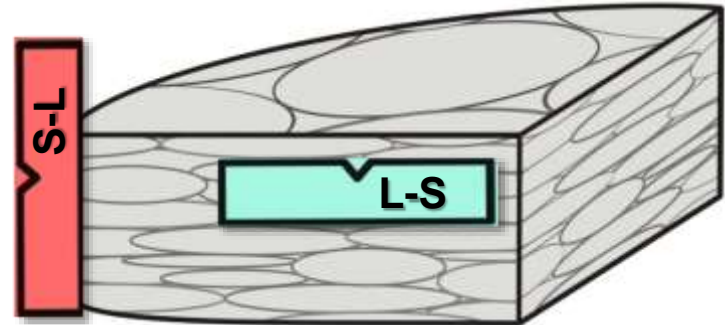
Rods



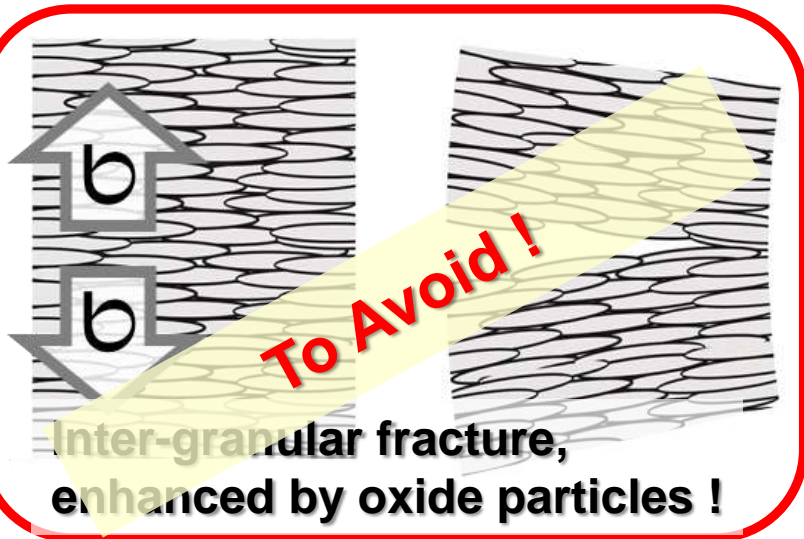
Bundle of „Fibres“

Plates

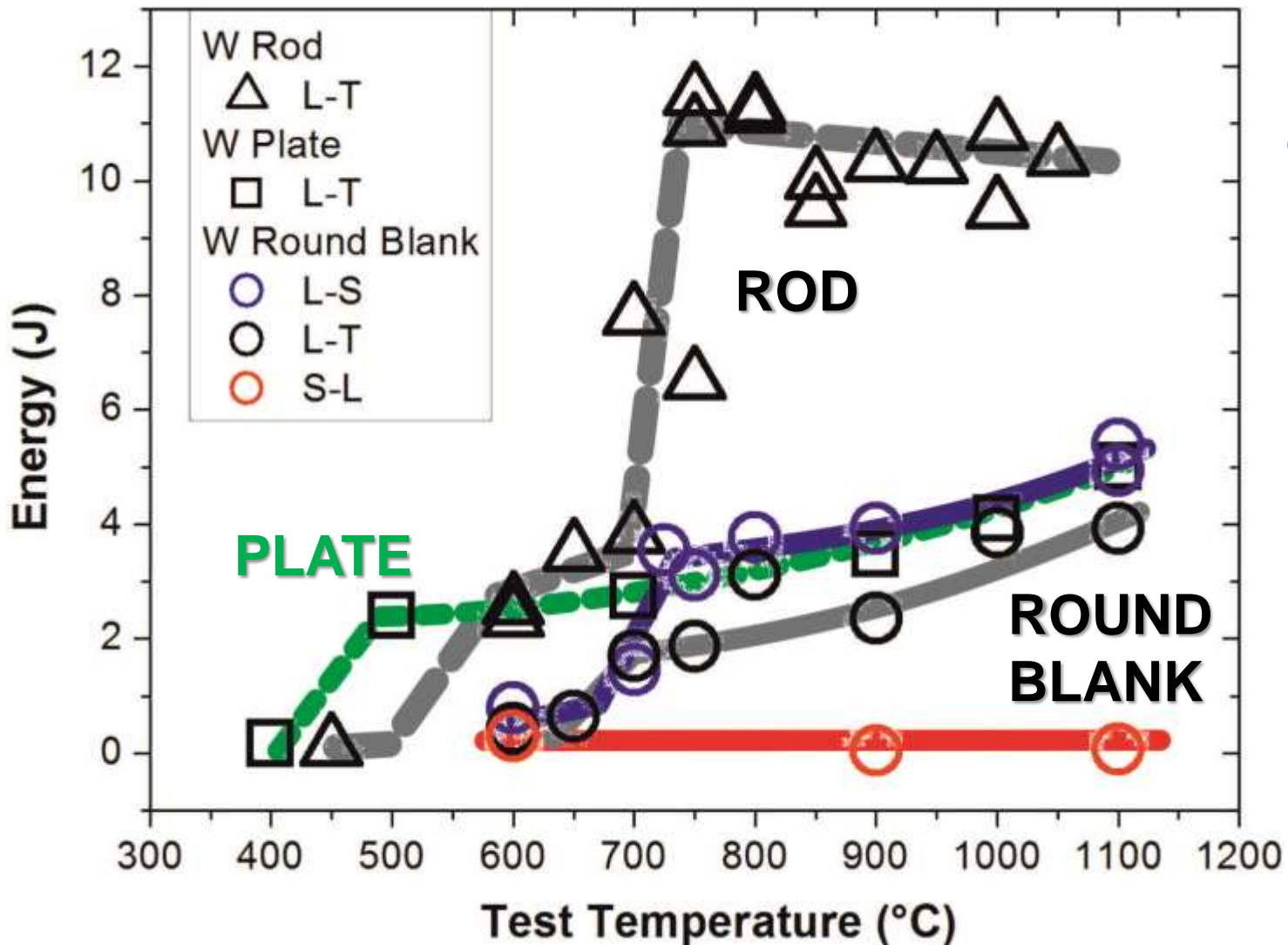
Round Blanks



Stack of „Pancakes“



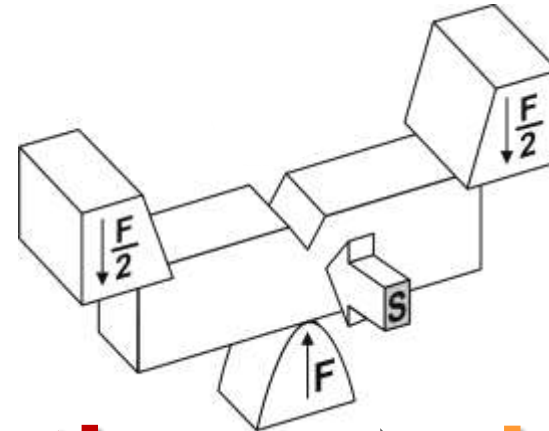
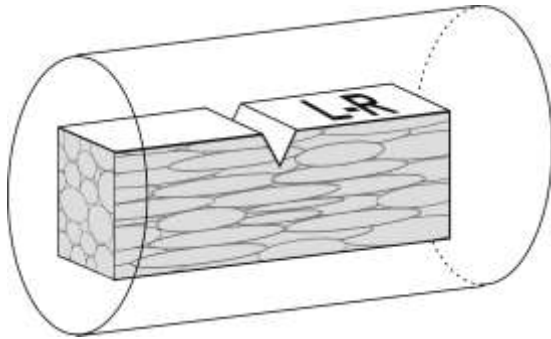
# Impact on Toughness



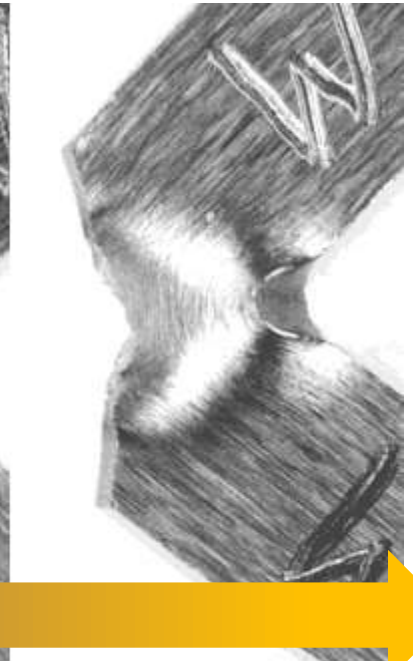
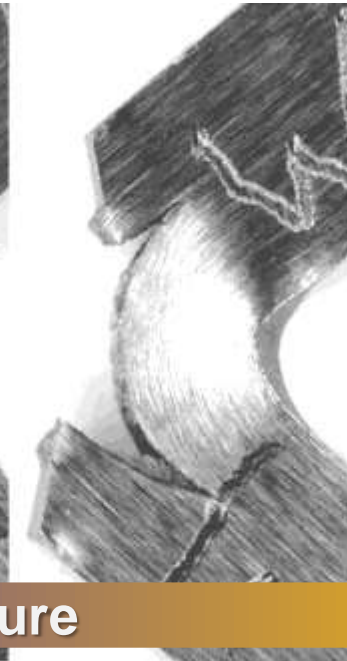
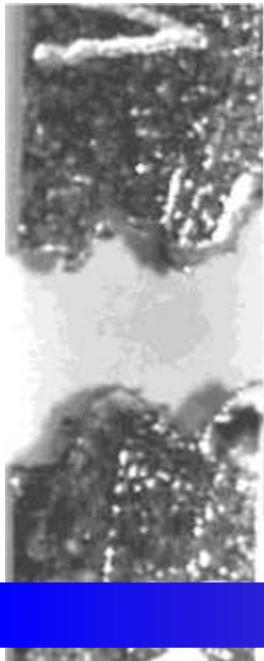
DELAMINATION DUCTILE



# Delamination Fracture



**brittle** → **delamination** → **ductile**

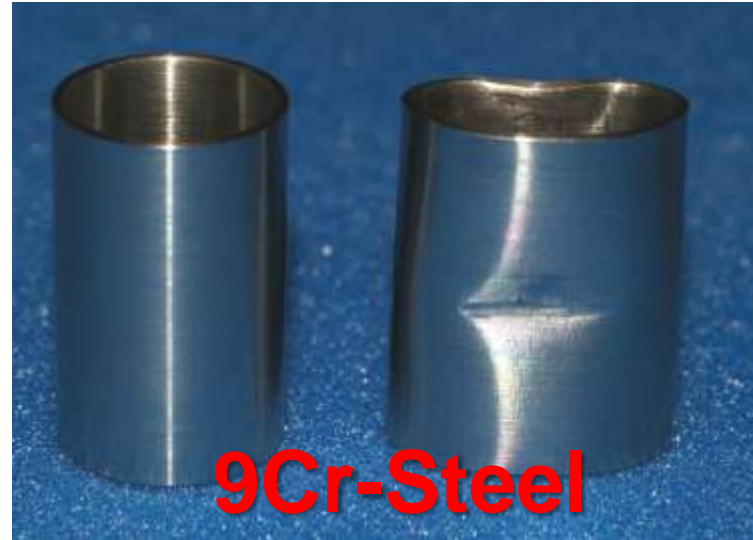


Test Temperature

# Problem of Microstructure Orientation



**Pipe Impact Test**

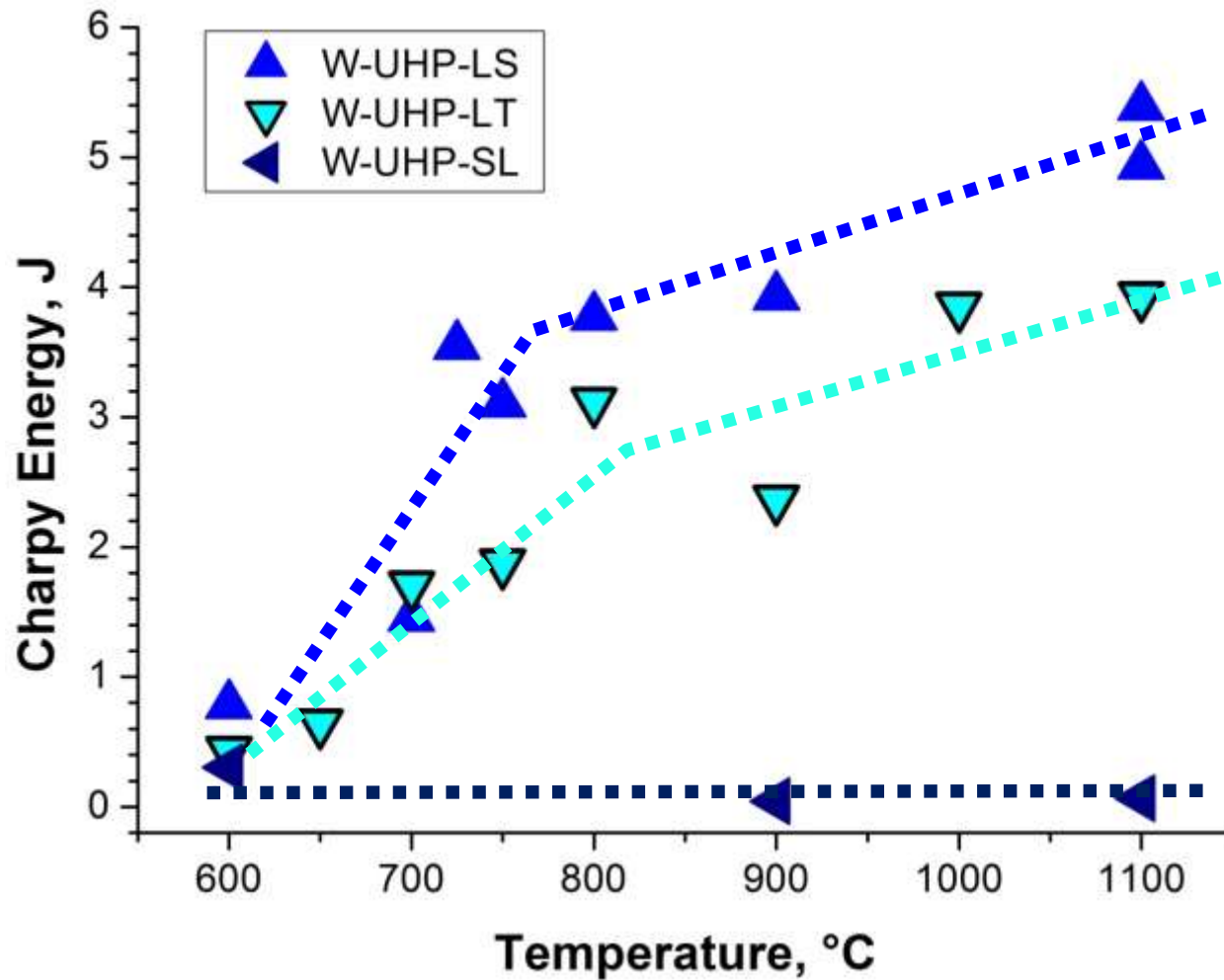


**9Cr-Steel**

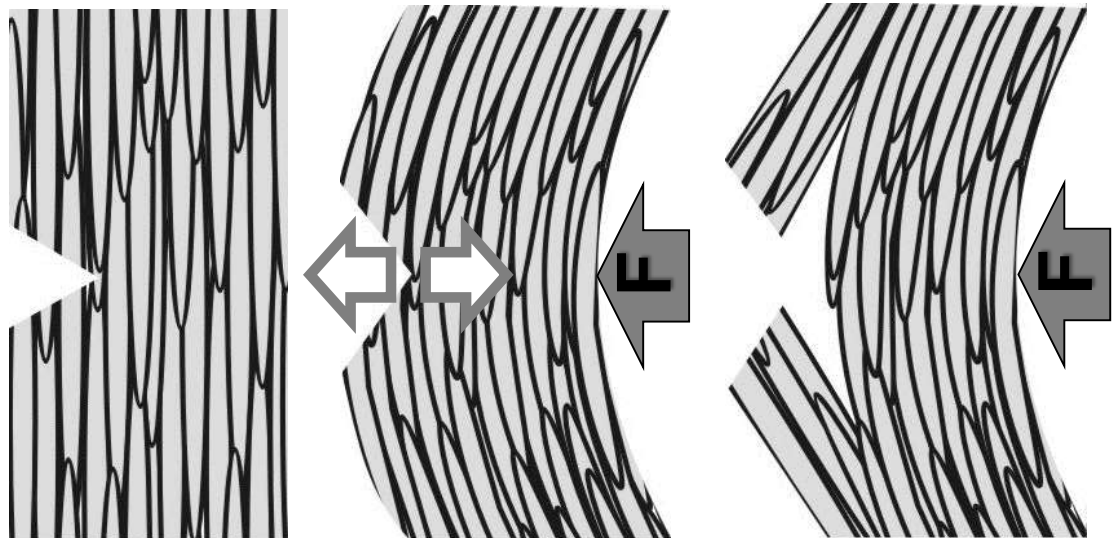
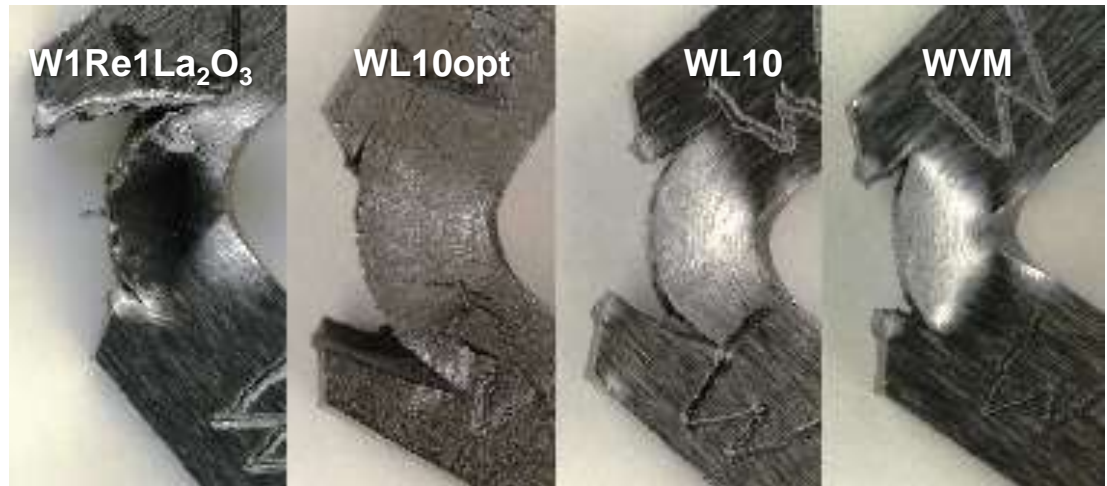
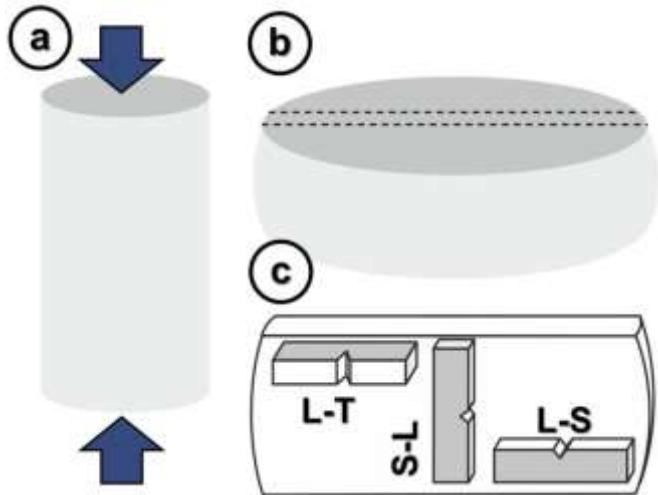


**W**

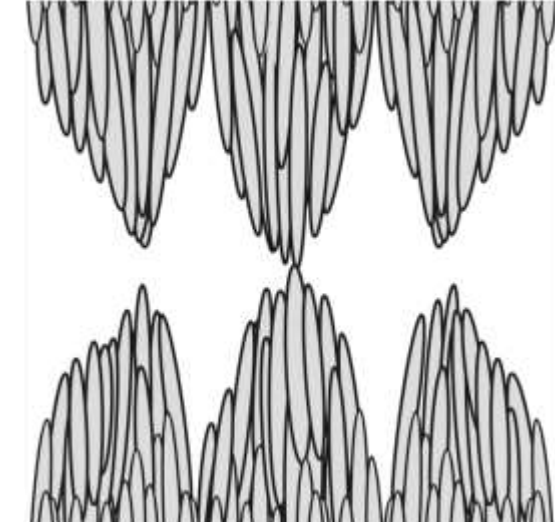
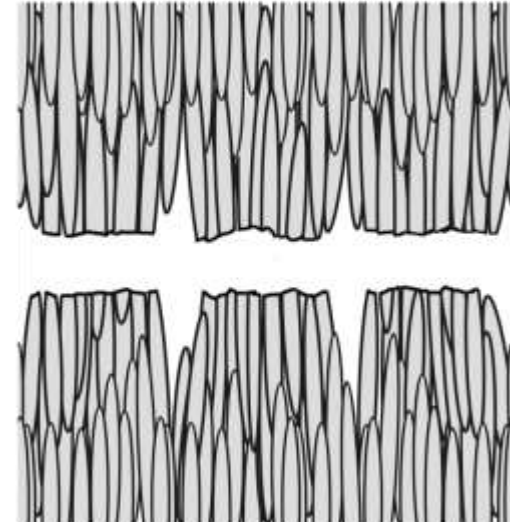
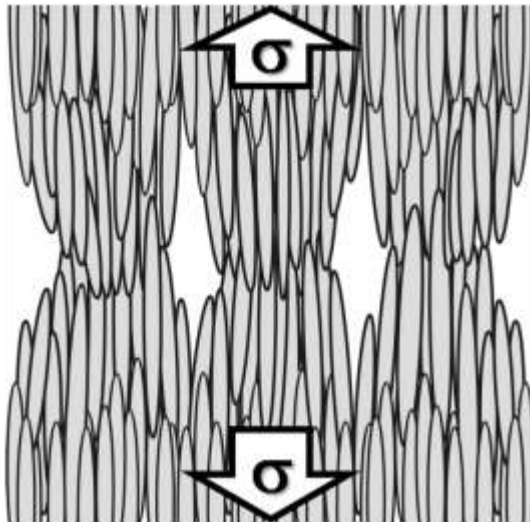
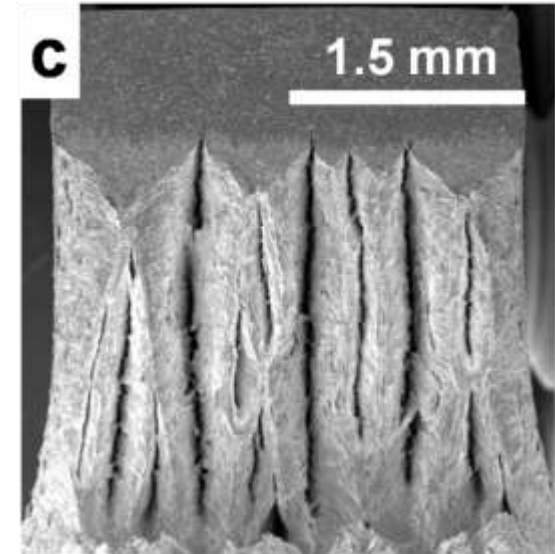
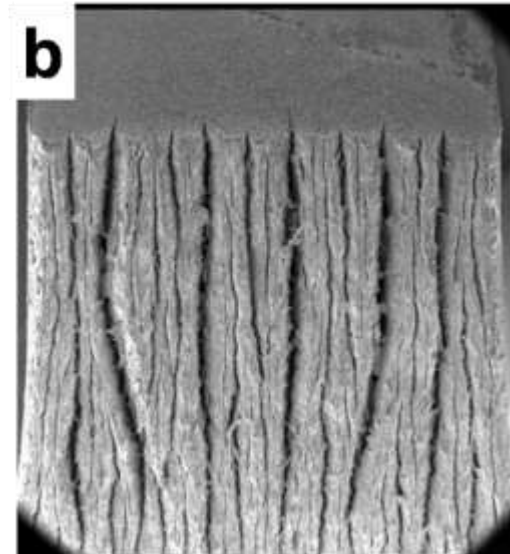
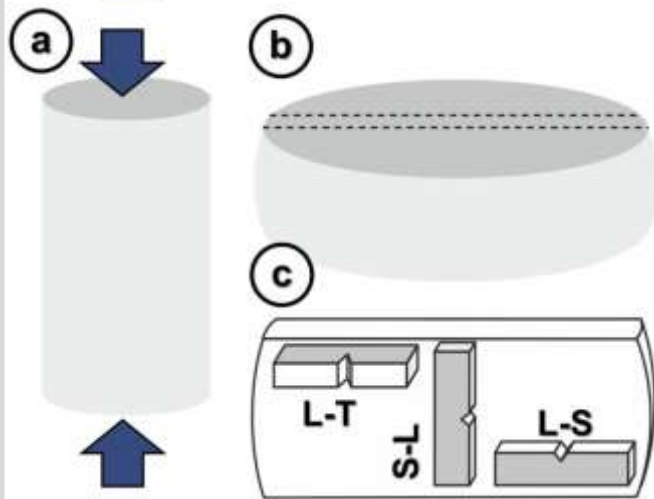
# ORIENTATION EFFECT (ANISOTROPY) IN PLATES



# Delamination Fracture (L-S)



# Delamination Fracture (L-T)



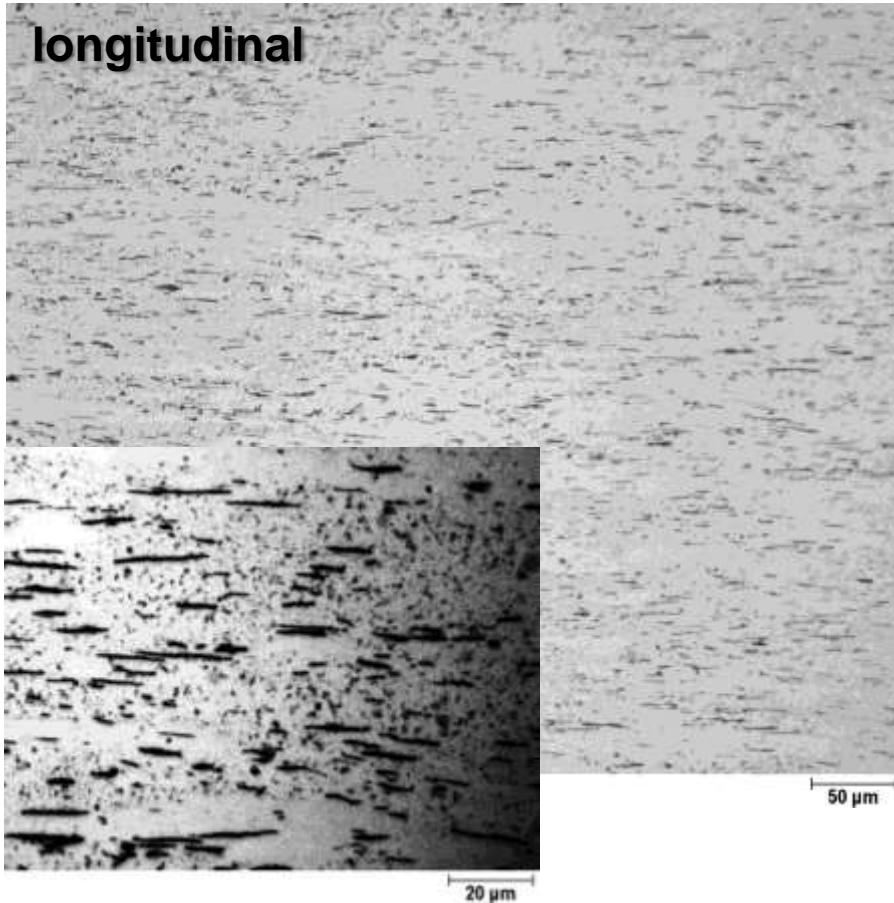
# Effect of ODS particles in W

WL10 Rod, Ø7 mm

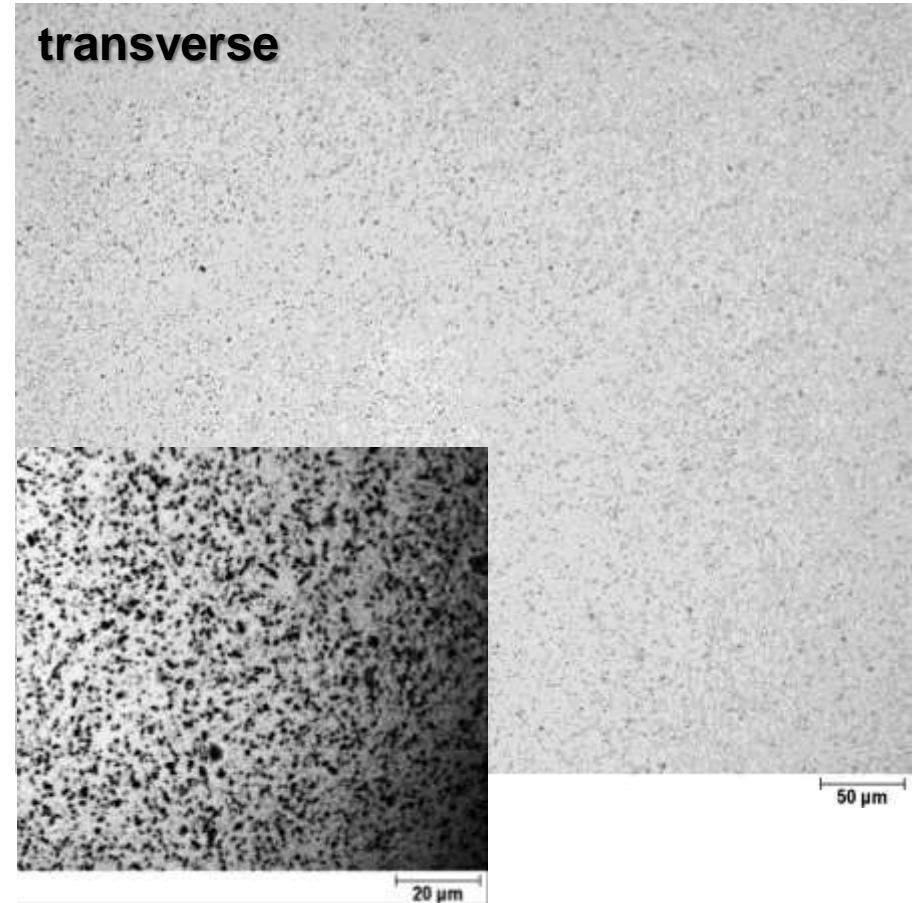


Rods: Metallography

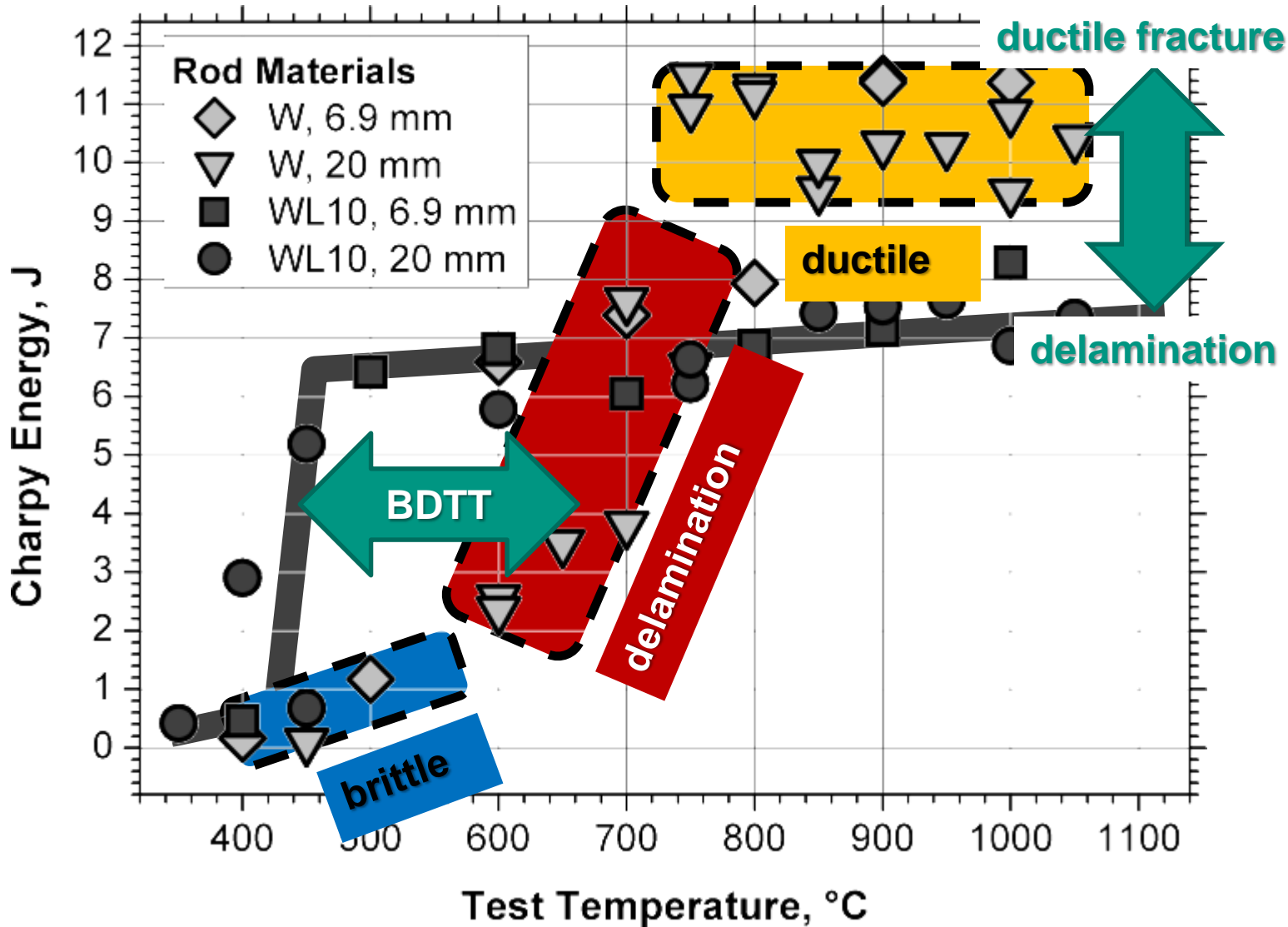
longitudinal



transverse



# Effect of ODS particles in W



So far, the best suitable tungsten materials for structural applications (divertor or other large scale components) are

**Thin Plates, Thickness < 4 mm**

**Produced by Sintering (Hydrogen Atmosphere) and Cross-Rolling**

**Pure Tungsten (maybe small amounts of grain stabilizers, like  $\text{La}_2\text{O}_3$ )**



4

HELIUM COOLED  
DIVERTOR DESIGNS

The expected life time of a power plant divertor is **TWO** years !?

High heat flux

- High operating temperatures (peaks > 1800°C)
- Microstructural stability, aging, ...
- Thermo-shocks, cyclic loading, fatigue, ...

Heavy ion bombardment

- Sputtering, cracking/fracturing
- Surface interactions and modifications
- Microstructural changes

Neutron load  
15 dpa/year (in W)

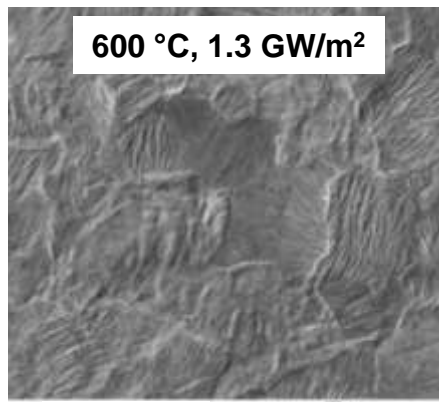
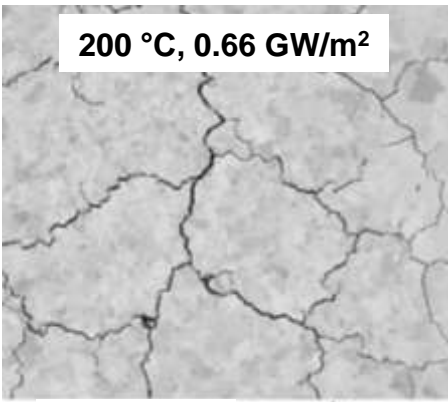
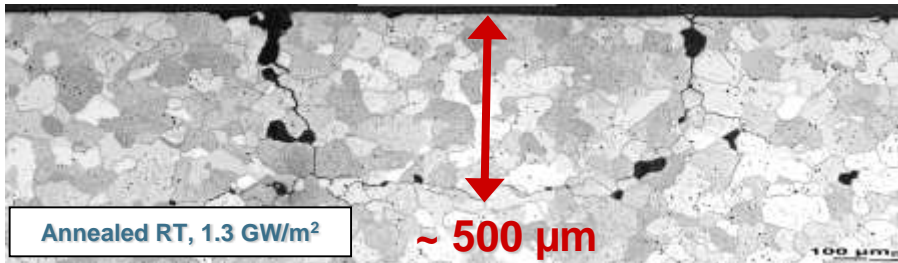
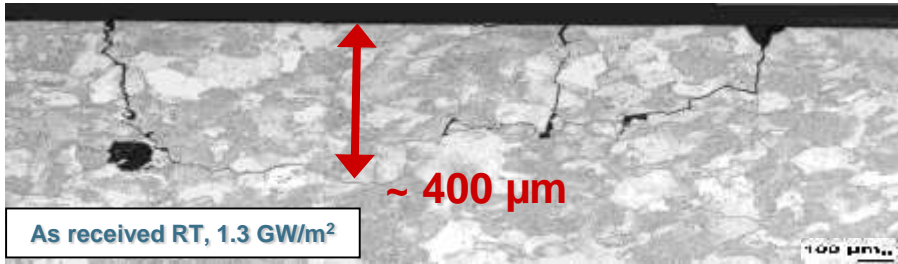
- 30 dpa (in W) until end of service
- Transmutation effects (Re, Os)
- Swelling (order of magnitude: 3% for pure W)

**QUESTION:** What can we do about this? How can we improve **ARMOR** materials?

**ANSWER:** There is not much we can do now! We have to live with most properties and effects. A final assessment would require real in-service conditions.

# HIGH HEAT FLUX EFFECTS

## Electron Beam

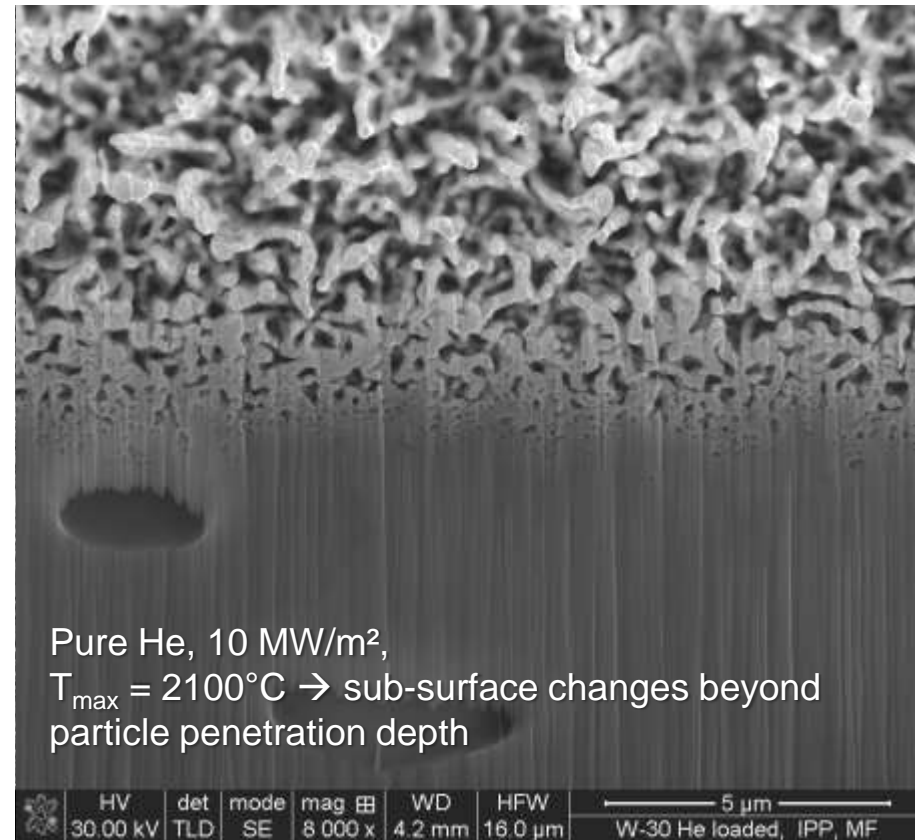


as received

annealed

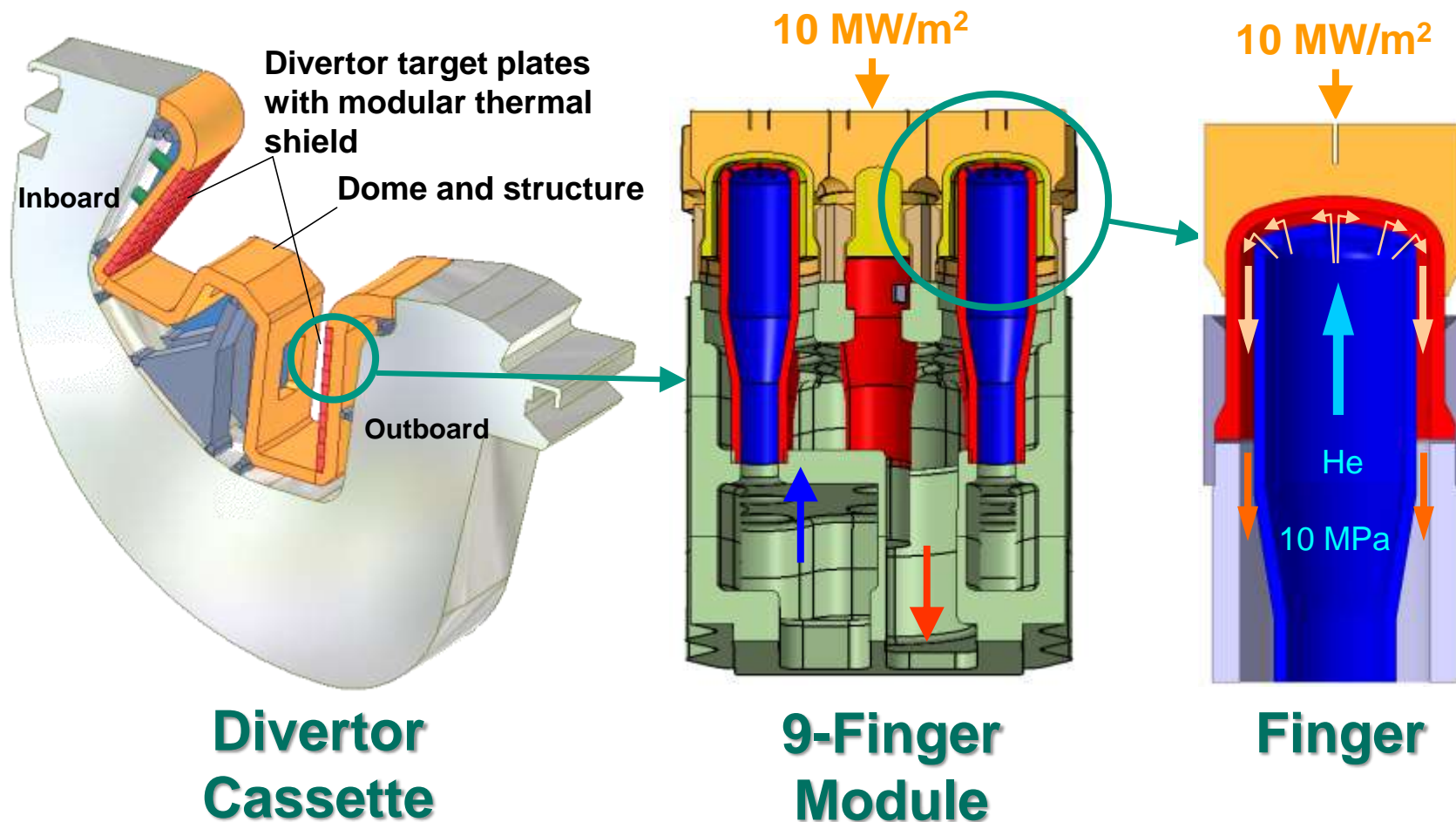
→ G. Pintsuk, J. Linke, et al., FZJ

## Hydrogen/Helium Ion Beam



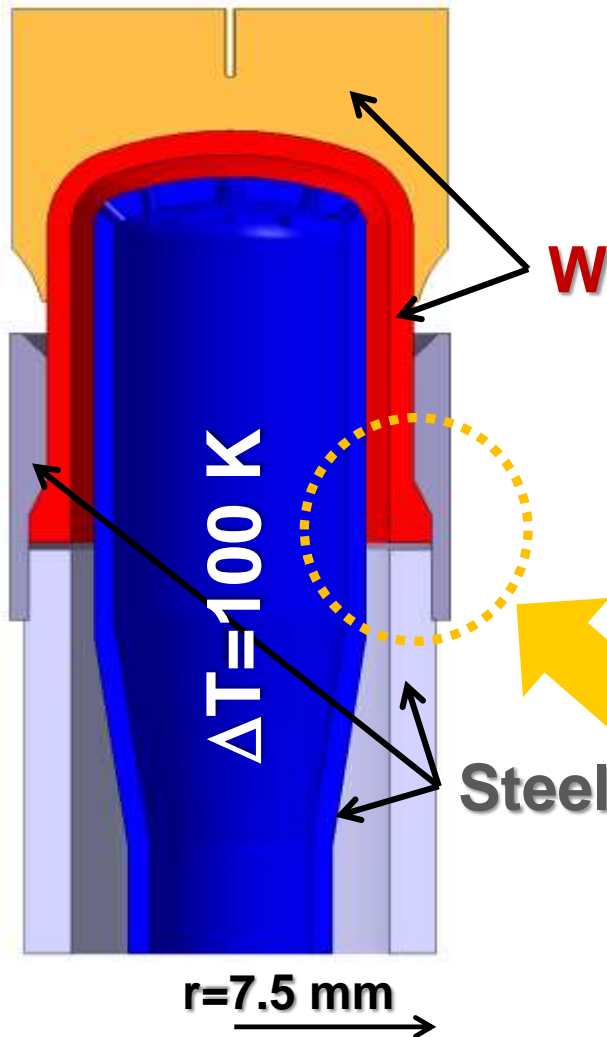
→ H. Maier, H. Greuner, M. Rasinski,  
Ch. Linsmeier, IPP

# FINGER DESIGN, JET COOLING



→ P. Norajitra, T. Ihli *et al.*, 2003-2009

# FINGER DESIGN, JET COOLING



+ High heat flux:  $>10 \text{ MW/m}^2$

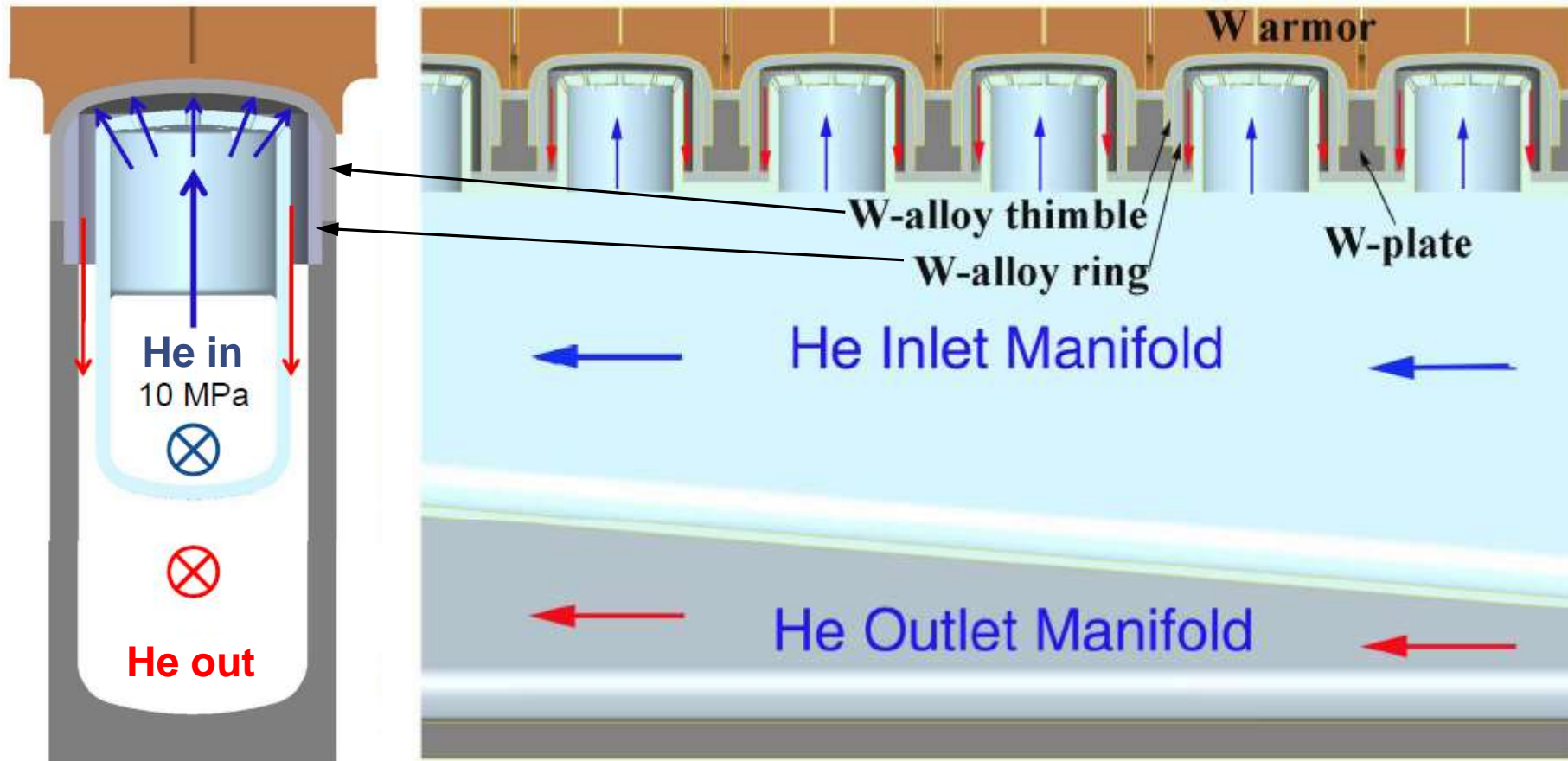
+ Small Size, Thin Walls

- Large Numbers:  $\sim 500\,000$

- Joints between W and Steel

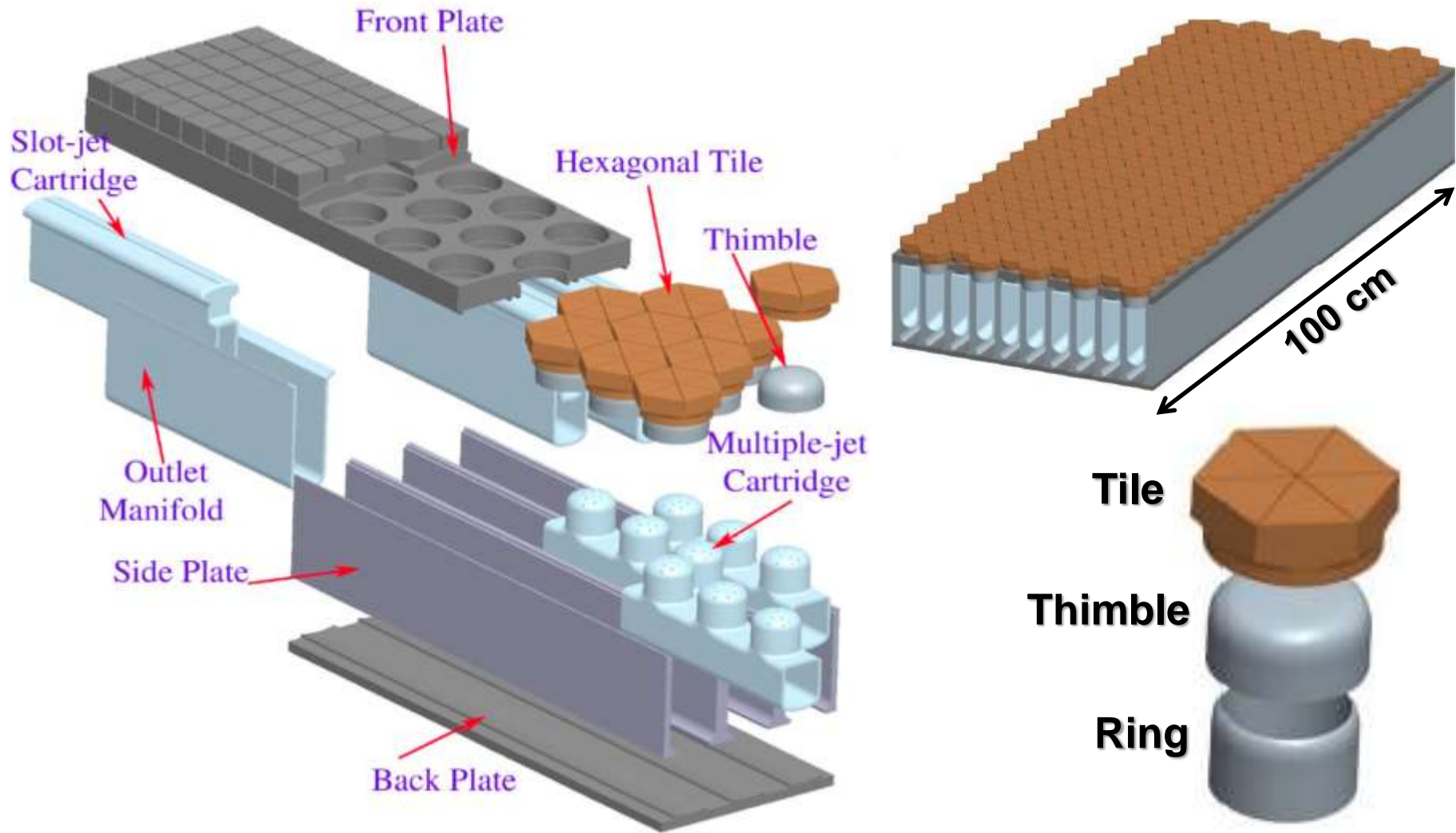
He and W temperature limited by steel  
Ferritic ODS  $\rightarrow 750^\circ\text{C} ???$   
Eurofer ODS, 9Cr ODS  $\rightarrow 650^\circ\text{C}$   
Eurofer 97, F82H  $\rightarrow 550^\circ\text{C}$

# PLATE DESIGN (ARIES), JET COOLING



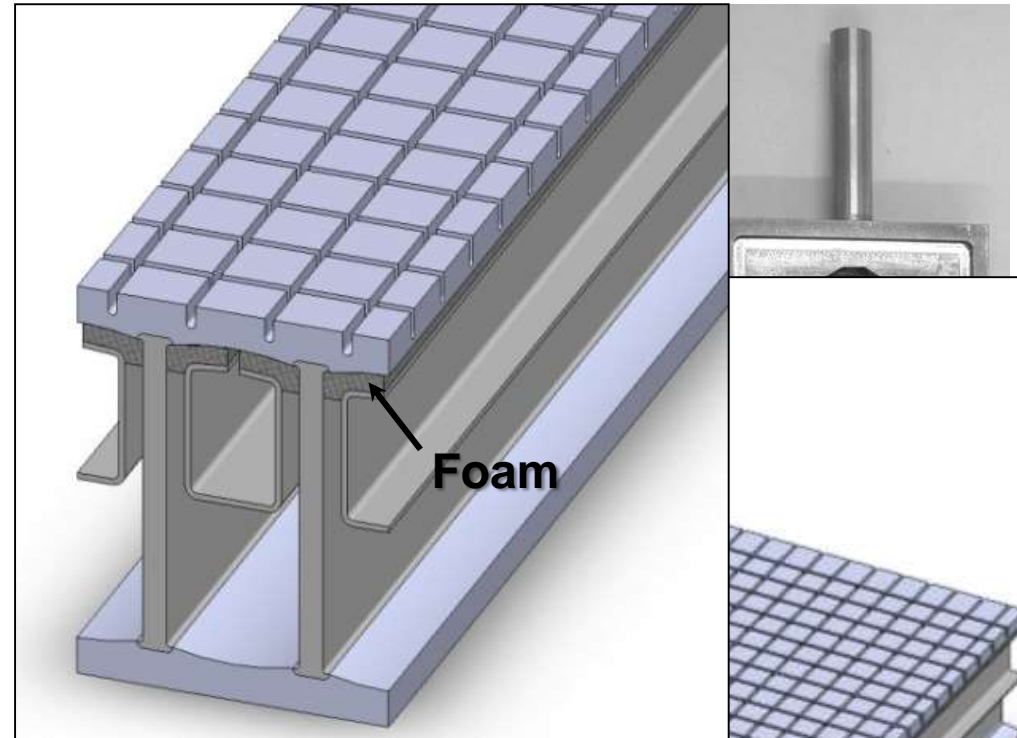
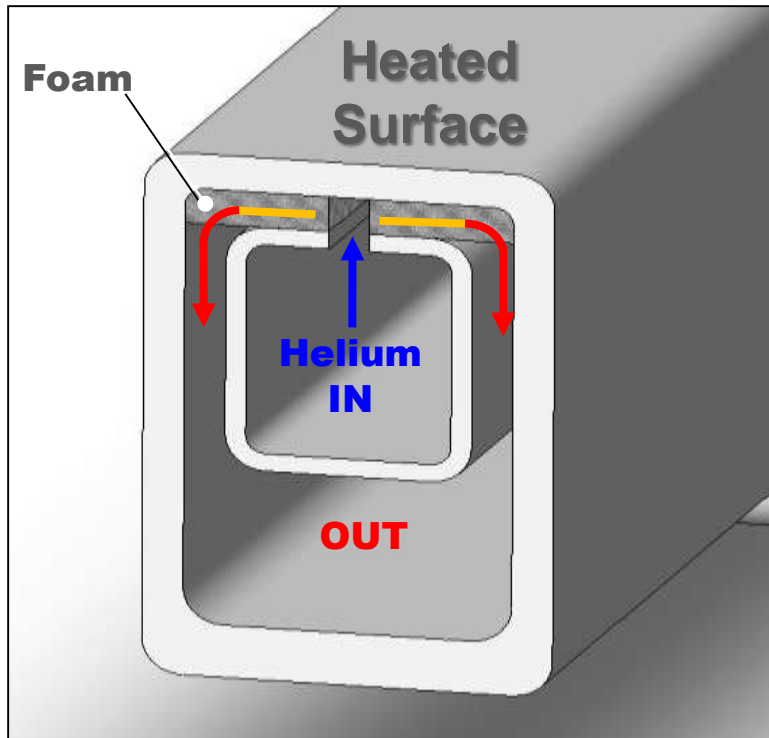
X.R. Wang, S. Malang, M.S. Tillack & ARIES Team, 2008-2011

# PLATE DESIGN (ARIES), JET COOLING



X.R. Wang, S. Malang, M.S. Tillack & ARIES Team, 2008-2011

# PLATE DESIGN, FOAM PROMOTER

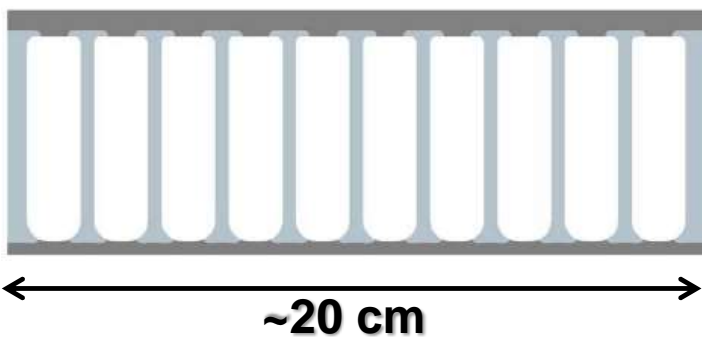
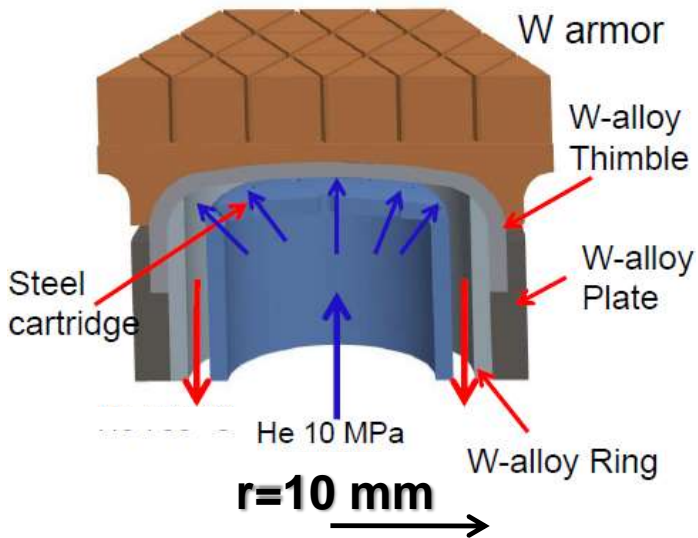


→ S. Sharafat *et al.*, UCLA, 2005-2009

→ Mo, Nb, SiC Foam:  
D. Youchison *et al.*, SNL, 2011



# PLATE DESIGN (ARIES), JET COOLING

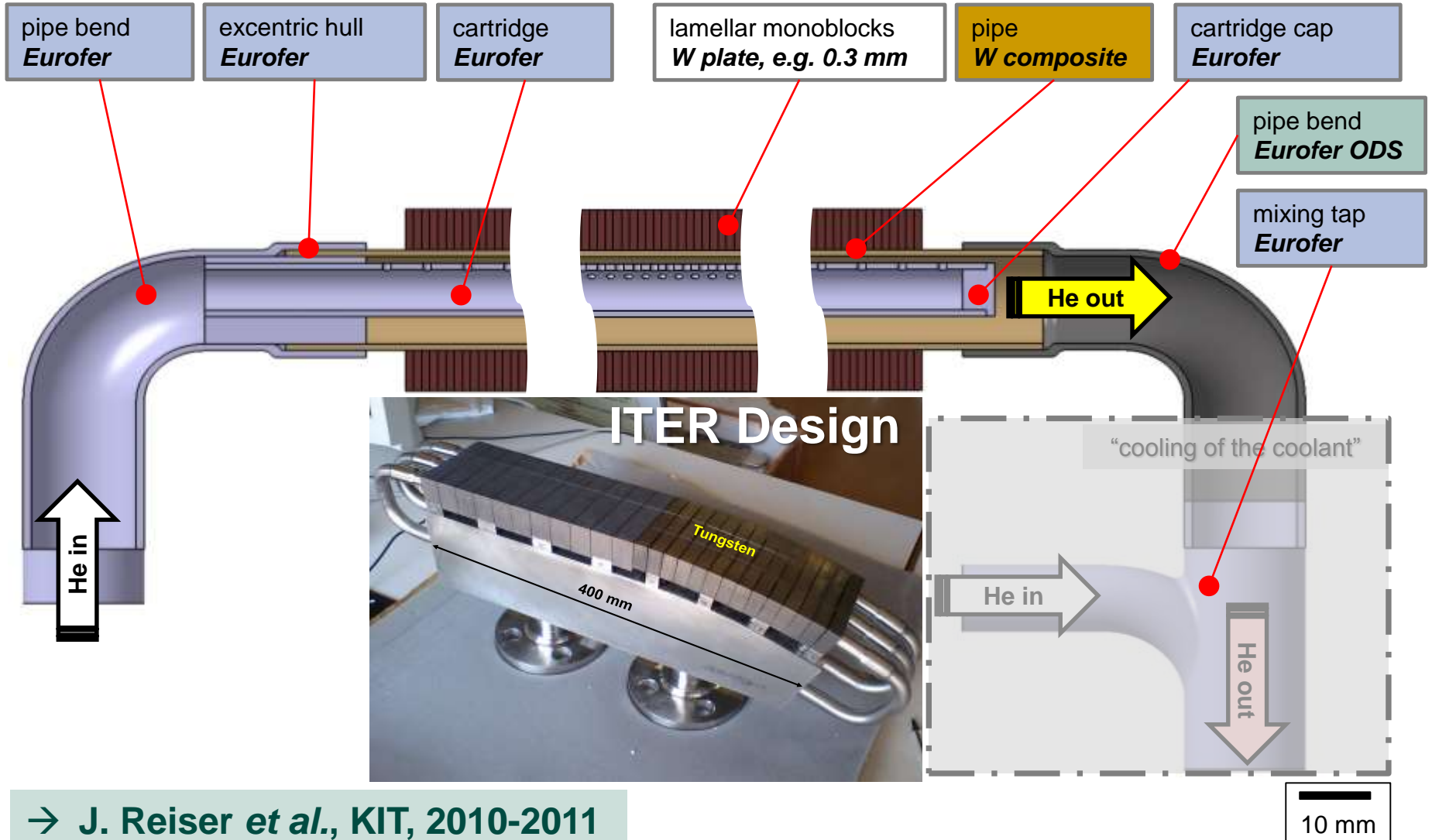


- + High heat flux:  $\sim 15 \text{ MW/m}^2$
- + Small Size, Thin Walls
- + “Double Containment”

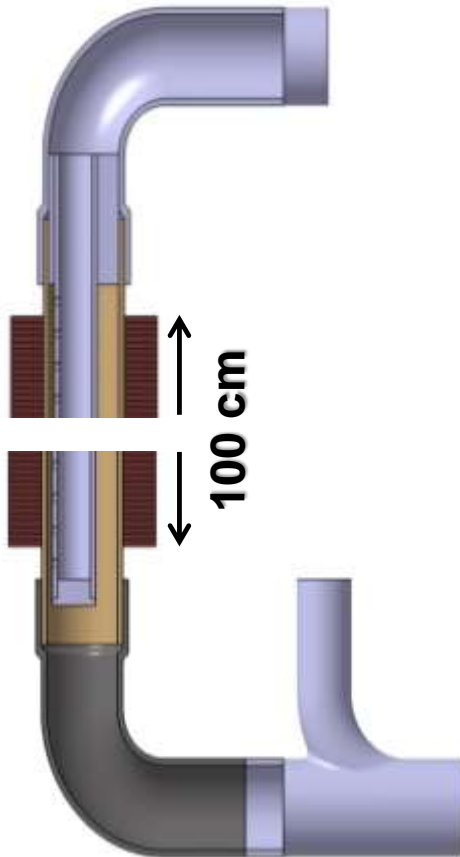
- Large Numbers:  $\sim 300\ 000$
- Many/Long Struct. W-W Joints

He temperature limited by steel  
(multiple jet cartridges)  
Ferritic ODS  $\rightarrow 750^\circ\text{C}$  ???  
Eurofer ODS, 9Cr ODS  $\rightarrow 650^\circ\text{C}$   
Eurofer 97, F82H  $\rightarrow 550^\circ\text{C}$

# PIPE/MONOBLOCK DESIGN, JET COOLING



# PIPE/MONOBLOCK DESIGN, JET COOLING



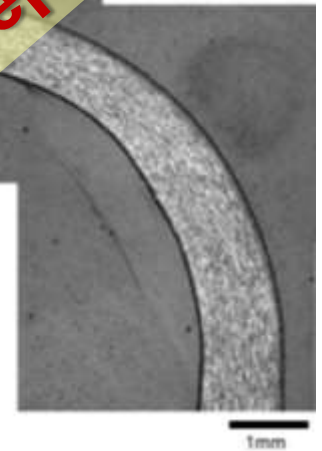
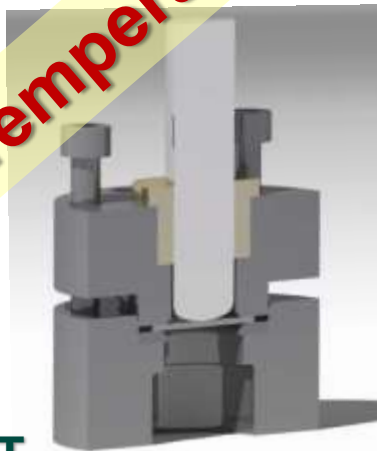
- + Simple Design
- + “Easy Joints”
- + “Small Number of Parts”
- + Inexpensive
- Low Temperatures for W
- Low Performance
- Speculative Pipe Material

He temperature limited by steel  
Eurofer ODS → 650°C (upper)  
Eurofer 97 → 350°C (lower)

# 5

DESIGN  
ANALYSIS

# FABRICATION

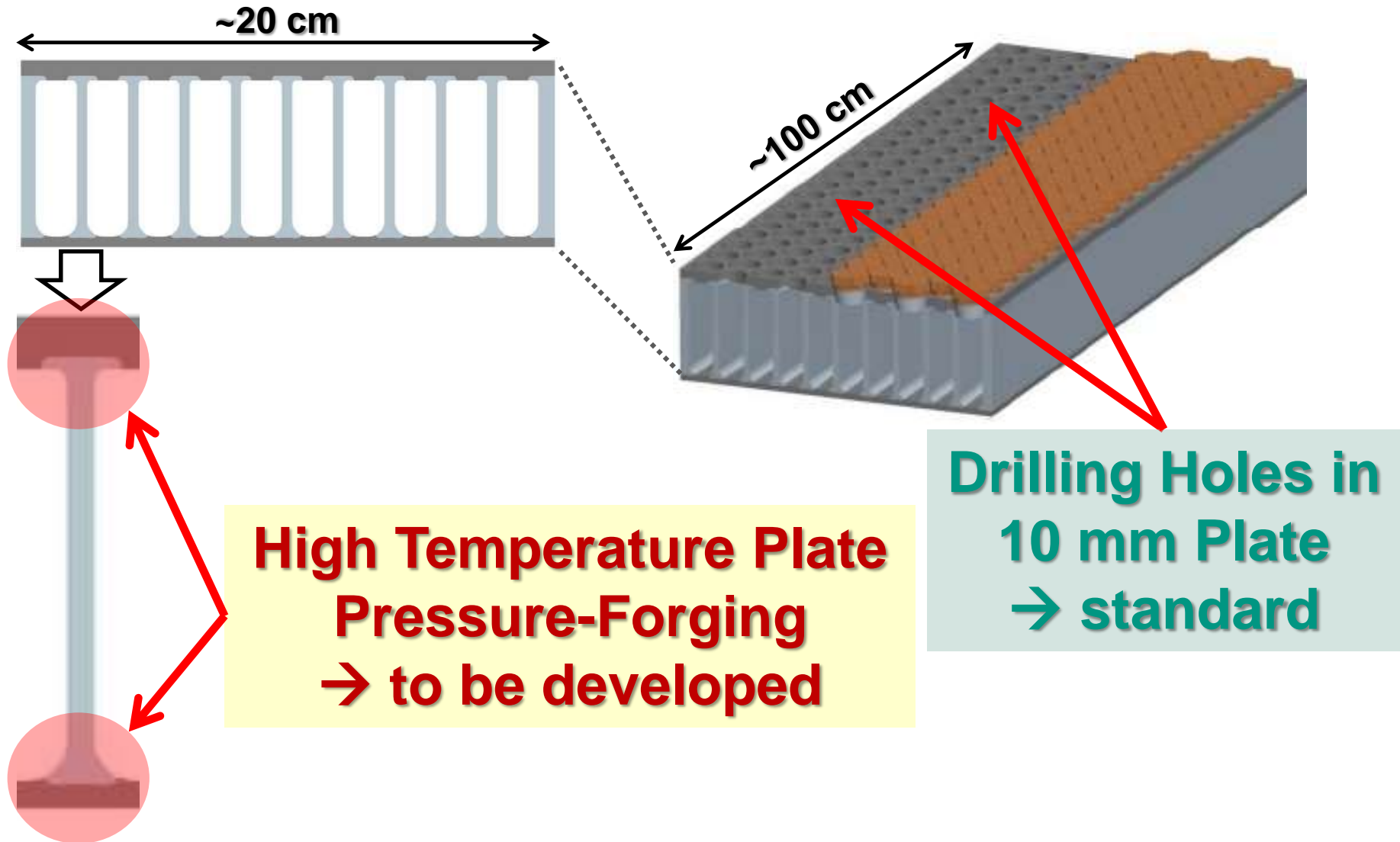


Machining: Turning, Milling, Grinding, ...

High Temperature Deep Drawing

P. Norajitra, J. Reiser, S. Baumgärtner *et al.*, KIT

# FABRICATION



**IMPORTANT: We have to consider joints for STRUCTURAL applications!!!  
Joining W tiles to W timbles is NOT considered as structural application!**

## Brazing Material for W-W Joints

Brazing temperature must be  $\gg 900-1200$  °C (operating temp.)

Brazing temperature must be  $< 1800$  °C → Grain growth

Formation of brittle compounds cannot be tolerated

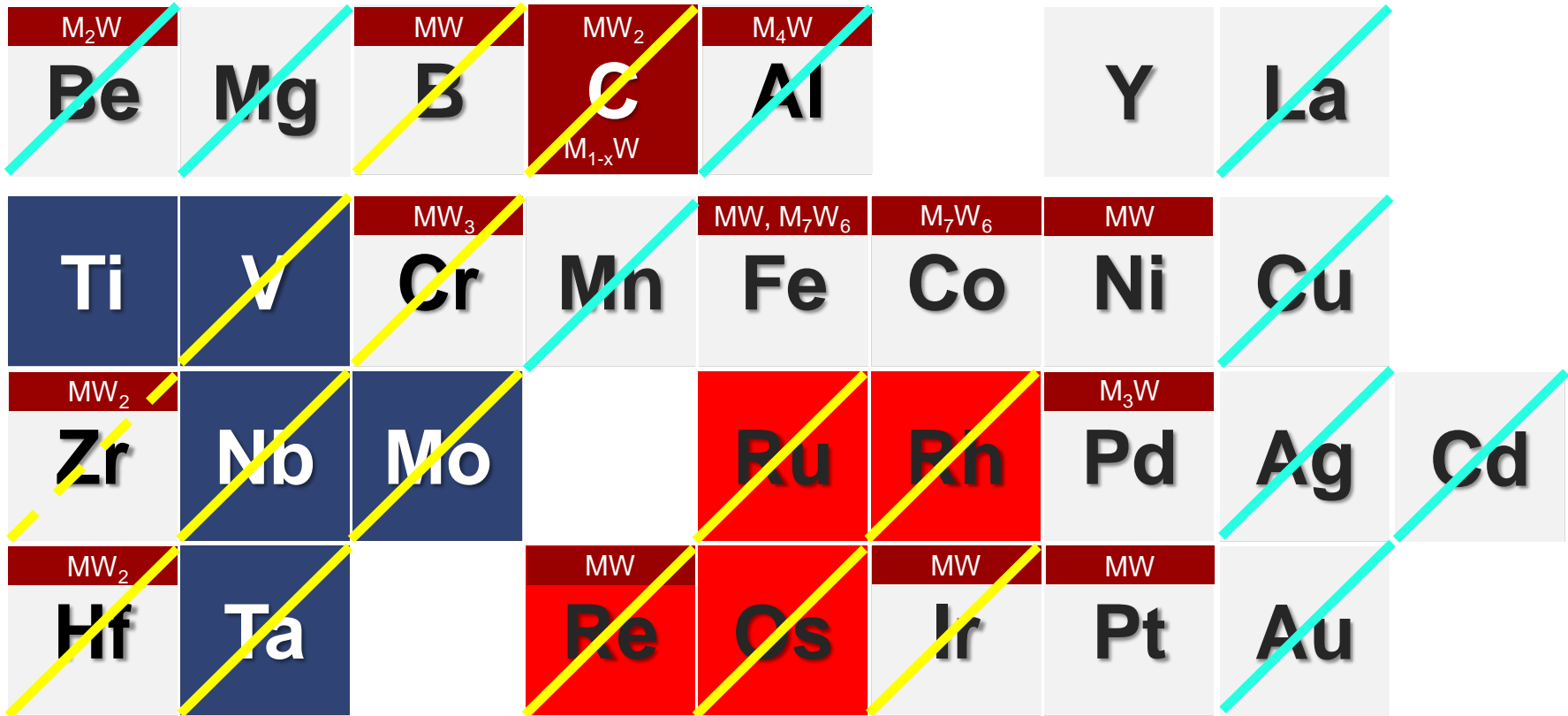
## Brazing Material for W-Steel Joints

Brazing temperature  $< 1100$  °C → Grain growth (in steel)

Brazing temperature must be  $\gg 550-750$  °C (operating temp.)

Formation of brittle compounds cannot be tolerated

# BRAZING W→W



W Insoluble

Intermetallic Phases

W Rich Line Compounds

Solid Solution

$T_m > 1800^\circ$

$T_m < 1400^\circ$



# BRAZING MATERIALS, W → W

1670°C	<b>Ti</b>	<740°C: <b>Difficult, ... ?</b>	
1520°C	<b>Y</b>	Strong <1570°C: <b>Difficult, ... ?</b>	
1850°C	<b>Zr</b>	<2160°C: W → ZrW <1700°C: <b>Brittle Joint</b> <860°C: Zr → ZrW <sub>2</sub>	
1550°C	<b>Pd</b>	<1800°C: W → ZrW <900°C: intermetallic Pd <sub>3</sub> W ???	
1770°C	<b>Pt</b>	<2400°C: μ phase Pt <sub>3</sub> W	
1540°C	<b>Fe</b>	<1700°C: μ phase Fe <sub>7</sub> W <sub>6</sub> (Fe,W) <sub>4</sub> <1000°C: Laves phase Fe <sub>2</sub> W	
1500°C	<b>Co</b>	<1700°C: μ phase Co <sub>7</sub> W <sub>6</sub> (Co,W) <sub>4</sub> <1000°C: Laves phase Co <sub>3</sub> W	
1450°C	<b>Ni</b>	<1000°C: peritectoid intermetallics NiW <950°C: peritectoid intermetallic Ni <sub>4</sub> W	

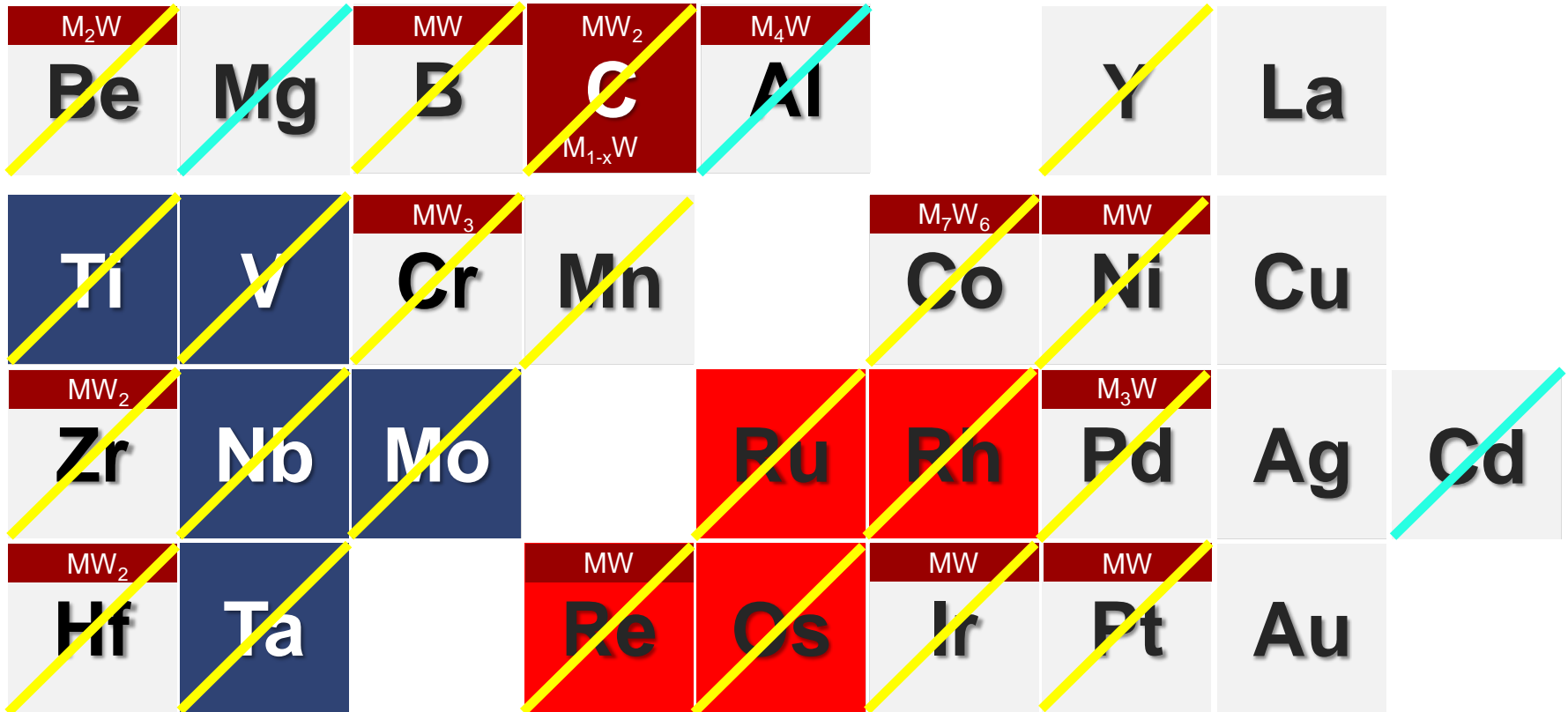
→ T. Hirai, 2008

**Brittle Joints**

W, Ni, Fe, Co  
Intermetallic phases

**WL10**

# BRAZING W → STEEL



W Insoluble

Intermetallic Phases

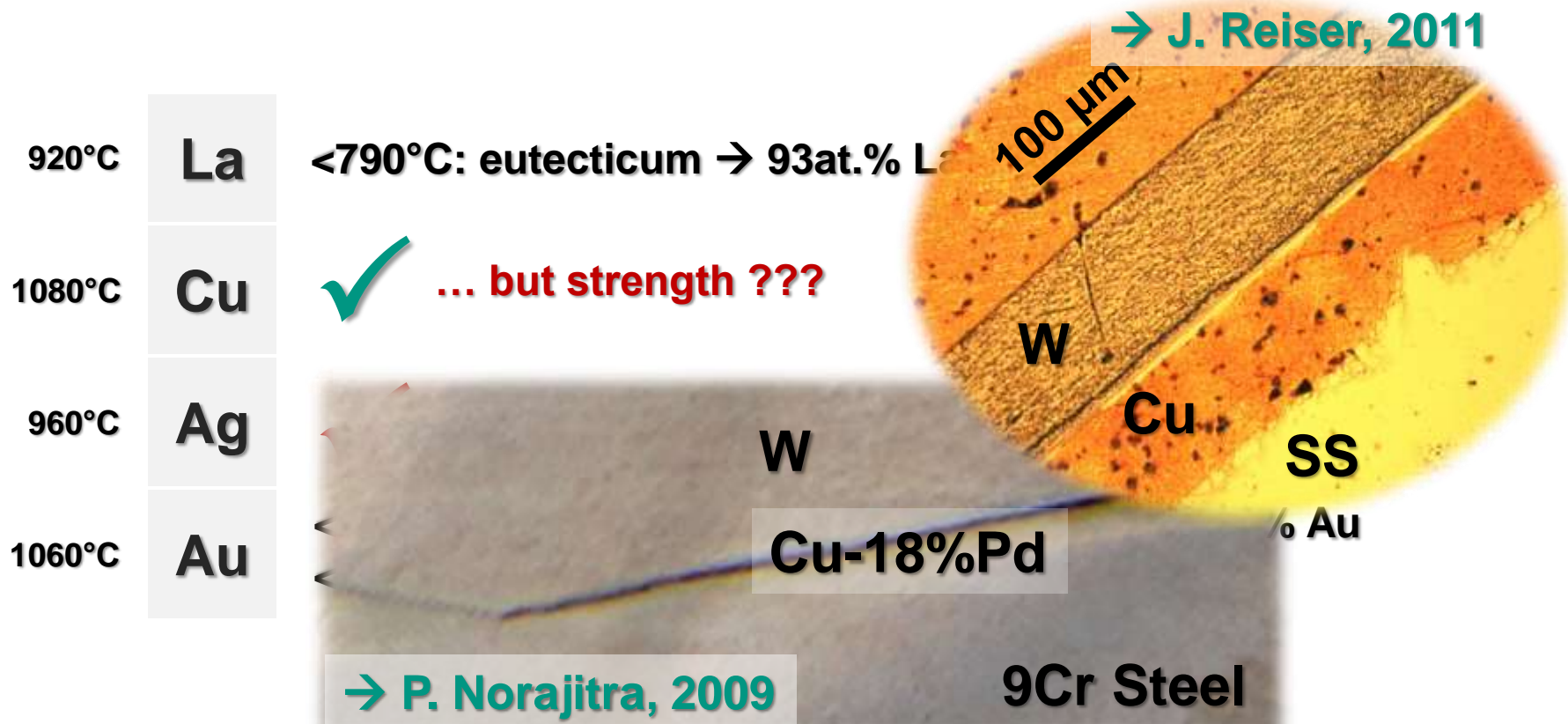
W Rich Line Compounds

Solid Solution

$T_m > 1200^\circ$

$T_m < 900^\circ$

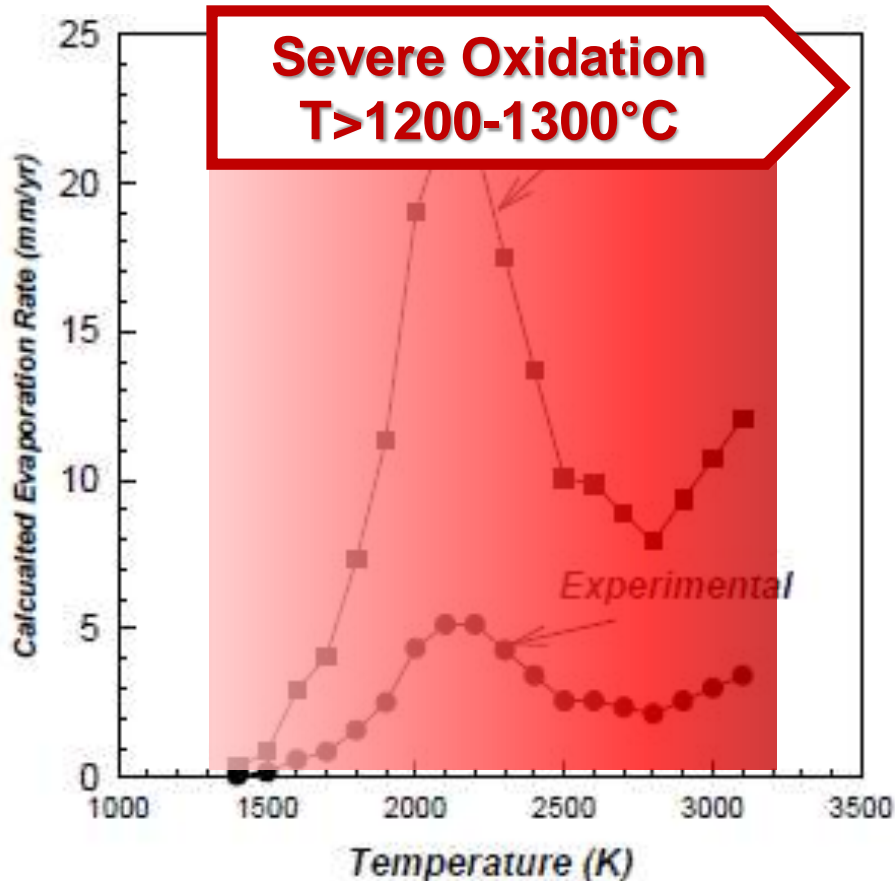
# BRAZING MATERIALS, W→STEEL



**CONCLUSION: Copper has to be used as sealing rather than as a braze material !!!**

# ENVIRONMENT → OXIDATION

$\text{WO}_3$  yellow,  $T_m=1470^\circ\text{C}$ ,  $7200 \text{ kg/m}^3$ , volatile in vacuum



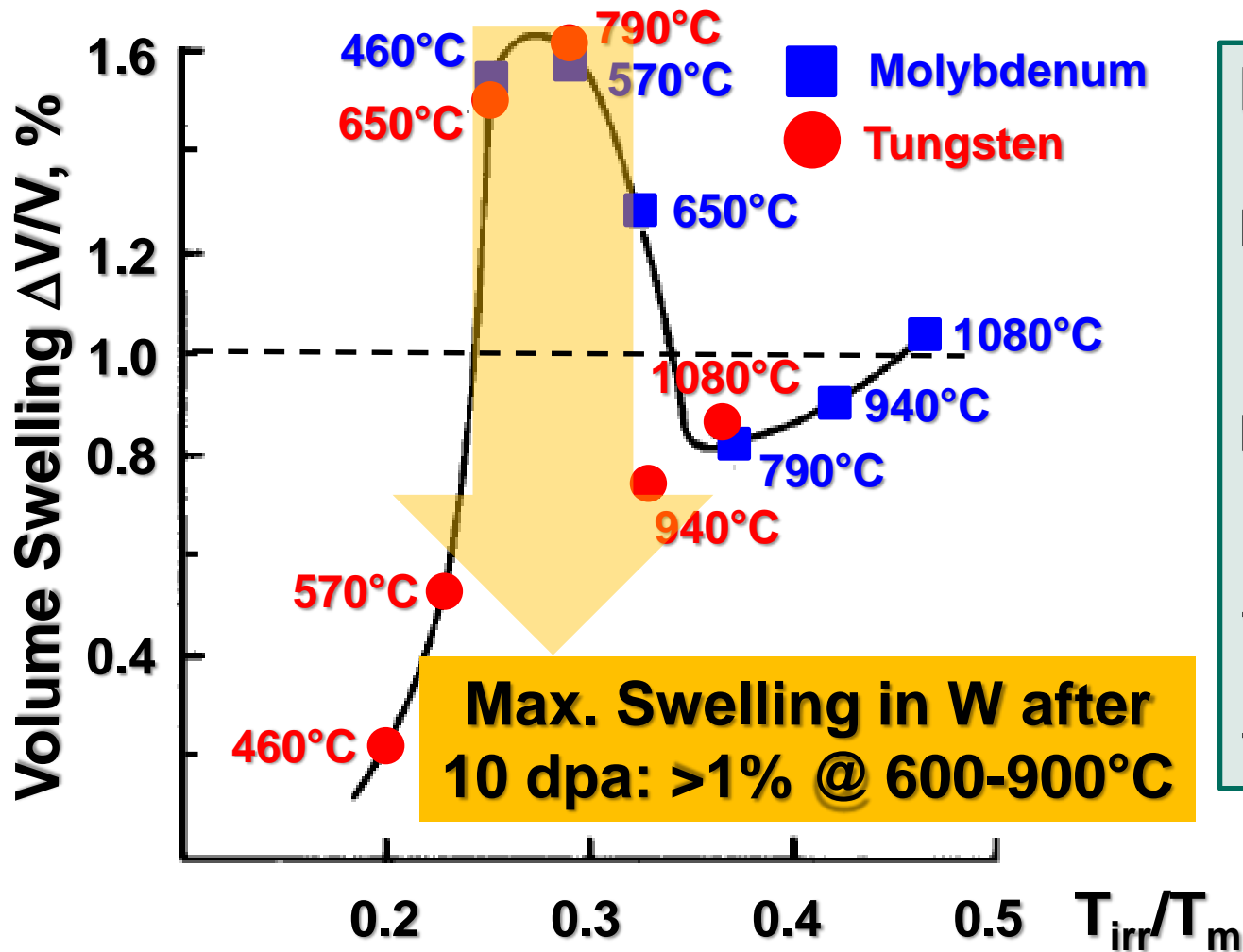
→ N.M. Ghoniem, 1998



## CONCLUSION

For tungsten operating at 50 atm. He coolant, at 0.1 ppm oxygen, the upper temperature is estimated at  $1200-1300^\circ\text{C}$ .

# IRRADIATION EFFECTS → SWELLING



## EBR-II

$E_n > 1 \text{ MeV}$   
 $1 \times 10^{22} \text{ n/cm}^2$

$E_n > 0.1 \text{ MeV}$   
 $1.6 \times 10^{22} \text{ n/cm}^2$

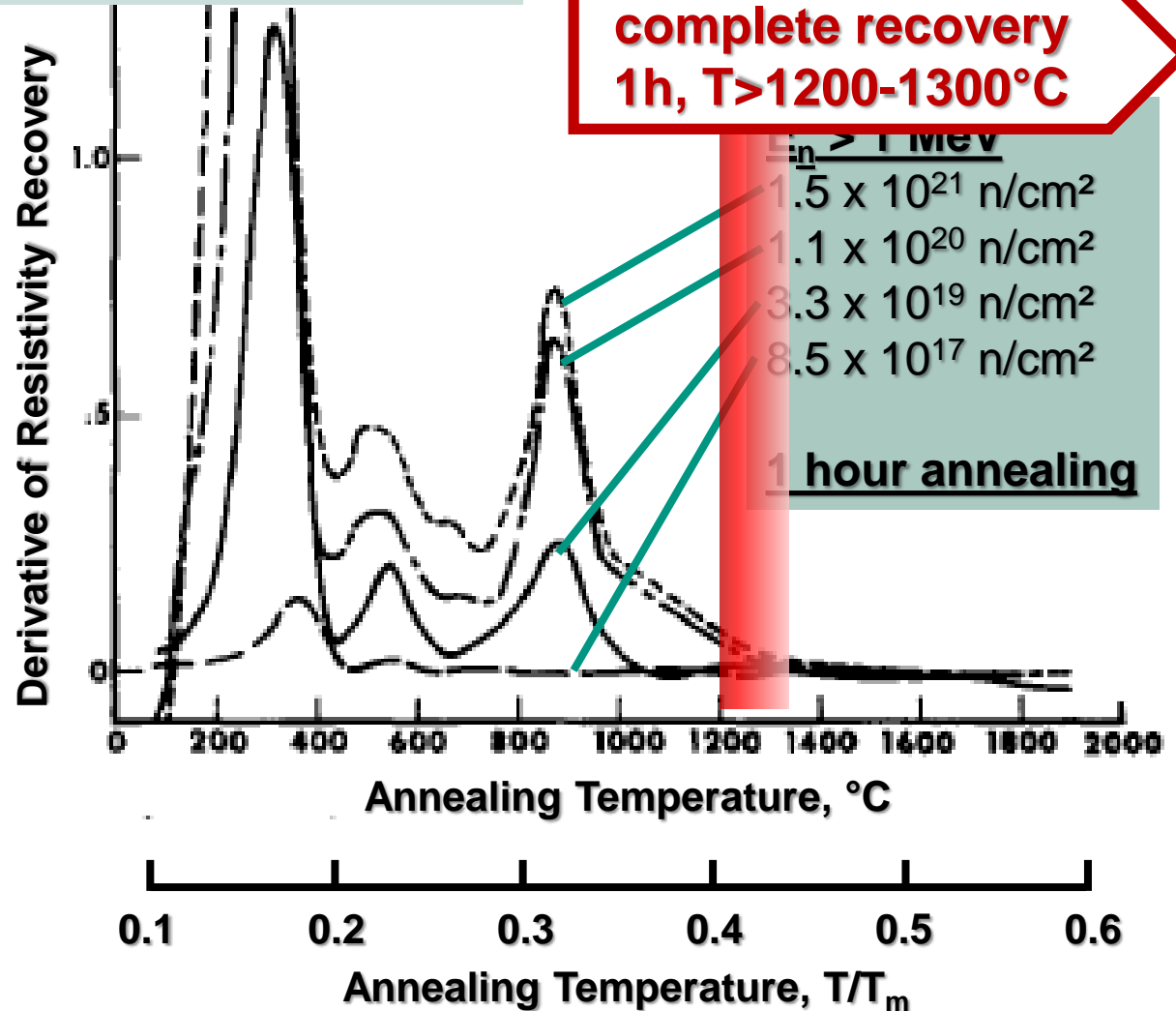
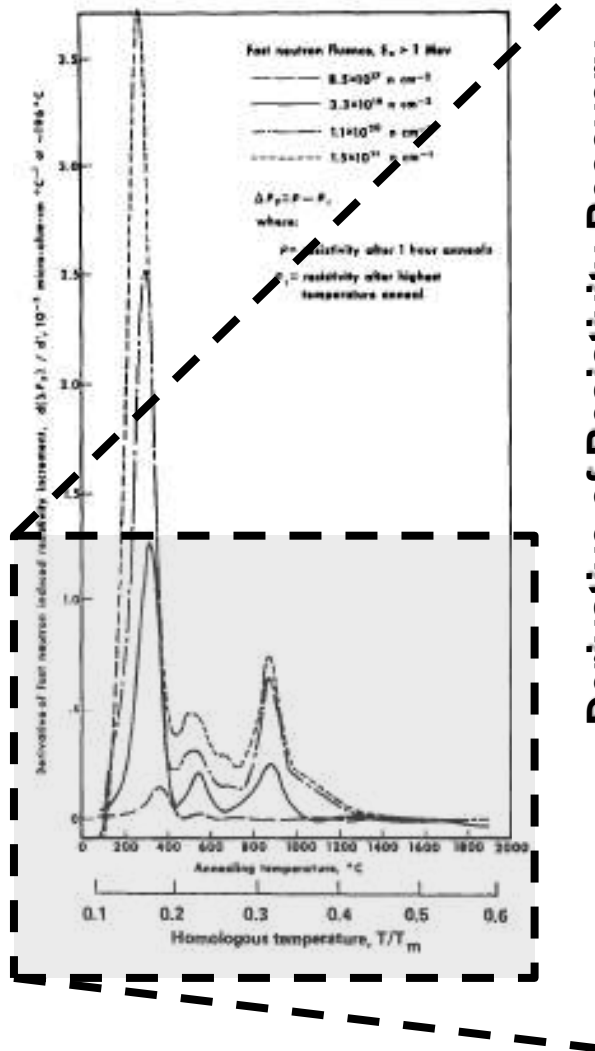
→ 29 dpa in Mo

→ 9.6 dpa in W

→ F. Lee, J. Matolich, J. Moteff, JNM 62 (1976) 115-117

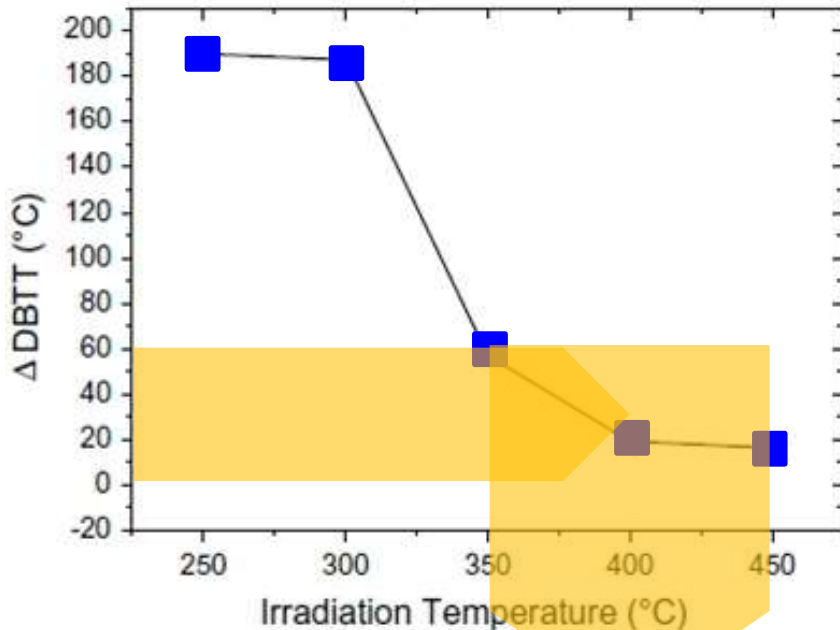
# IRRADIATION EFFECTS → RECOVERY

→ L.K. Keys, J. Moteff, JNM 34 (1970) 260-280



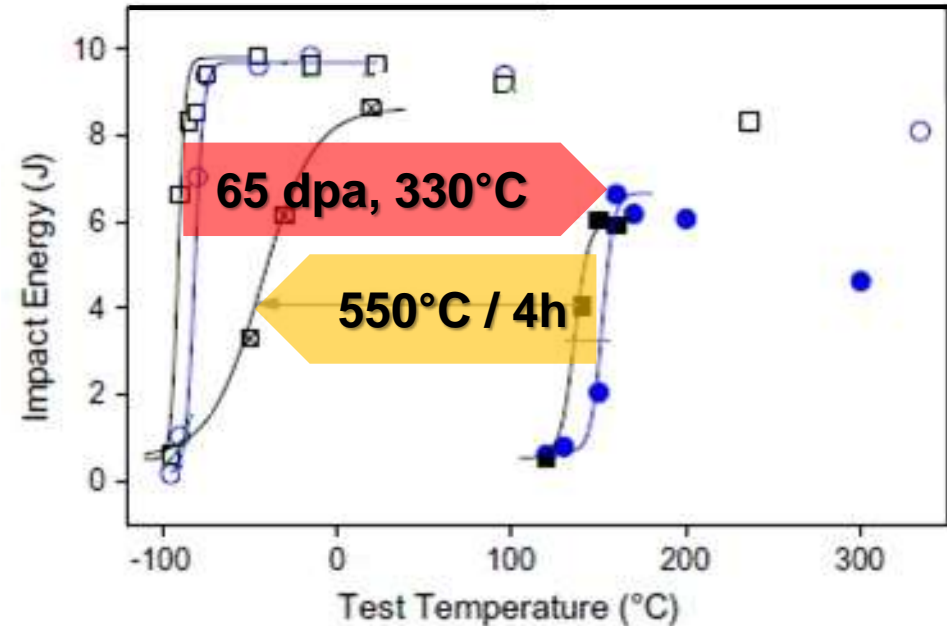
# IRRADIATION EFFECTS → EMBRITTLEMENT

**EUROFER:** In-service irradiation embrittlement after ~10 dpa



**Possible Operating Temperature  $T_{op} > 350^{\circ}\text{C}$**

**EUROFER:** Recovery of 65 dpa irradiation embrittlement

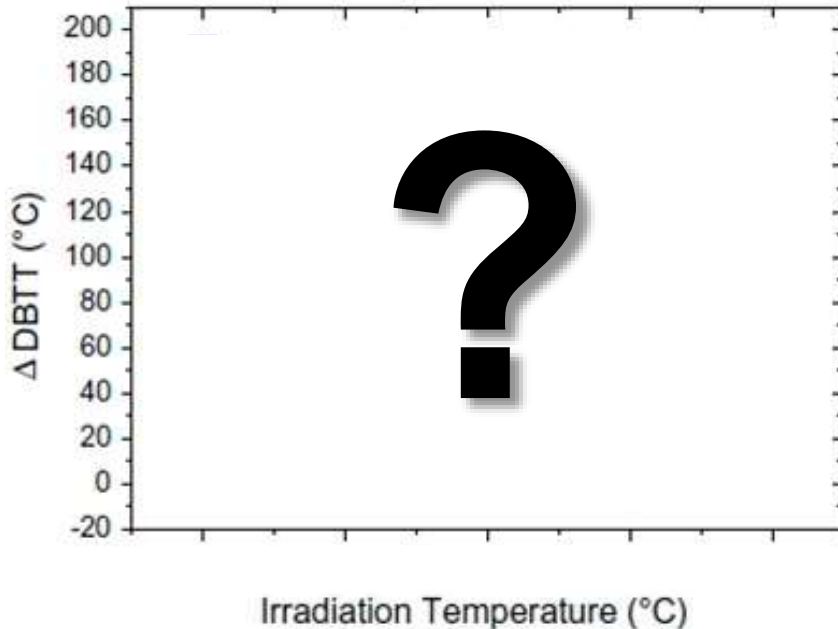


**Possible Recovery Temp.**  
 $T_{rec} > 550^{\circ}\text{C}$

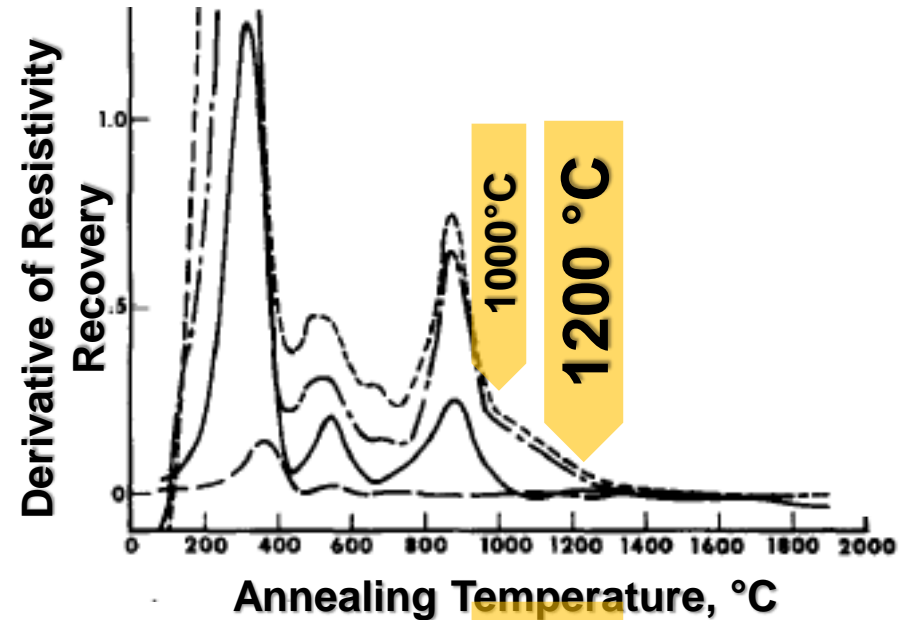
→ E. Gaganidze *et al.*, KIT

# IRRADIATION EFFECTS → EMBRITTLEMENT

**TUNGSTEN:** In-service irradiation embrittlement after 10-20 dpa



**Tungsten:** Recovery of ~2 dpa stage IV irradiation hardening

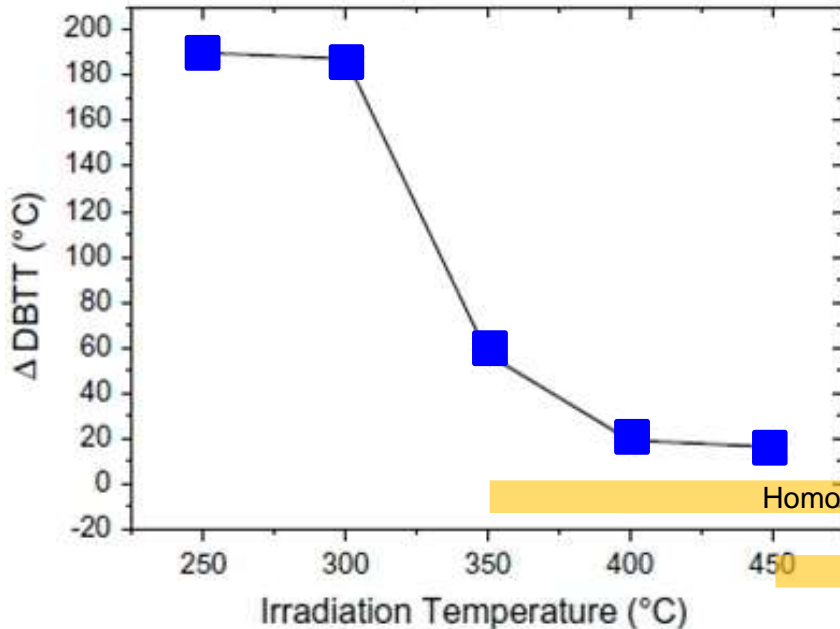


**Possible Operating Temp.**  
 $T_{op} > 800^{\circ}\text{C} \dots 1000^{\circ}\text{C}$

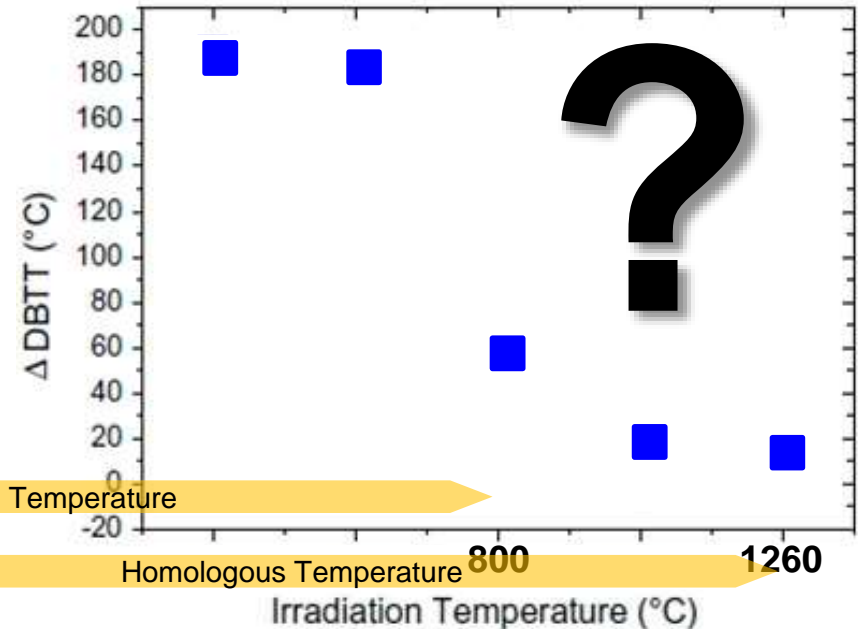


# IRRADIATION EFFECTS → EMBRITTLEMENT

**EUROFER:** In-service irradiation embrittlement after ~10 dpa



**TUNGSTEN:** In-service irradiation embrittlement after ??? dpa



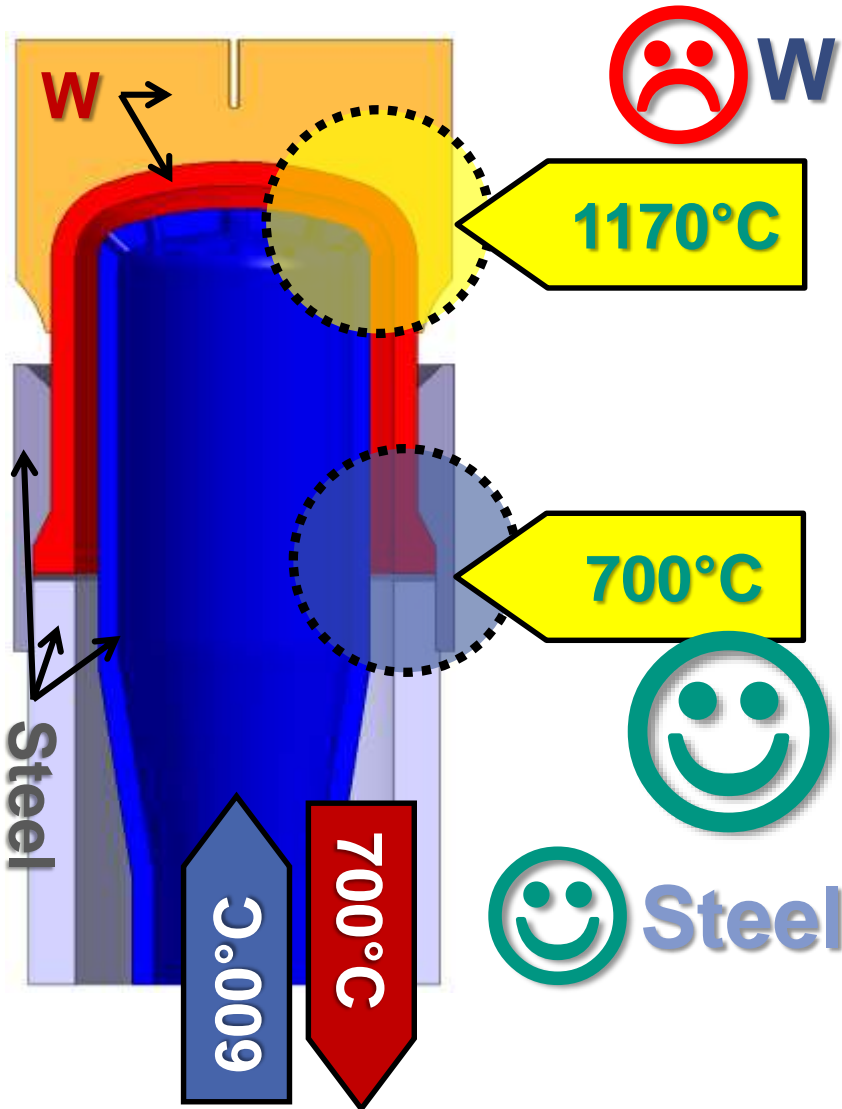
**Fe9Cr: Possible Operating Temperature  $T_{op} > 350^{\circ}\text{C}$  !!!**

**W: Possible Operating Temperature  $T_{op} > 800^{\circ}\text{C}$  ???**



# DESIGN ASSESSMENT

# MATERIALS / DESIGN WINDOW → FINGER



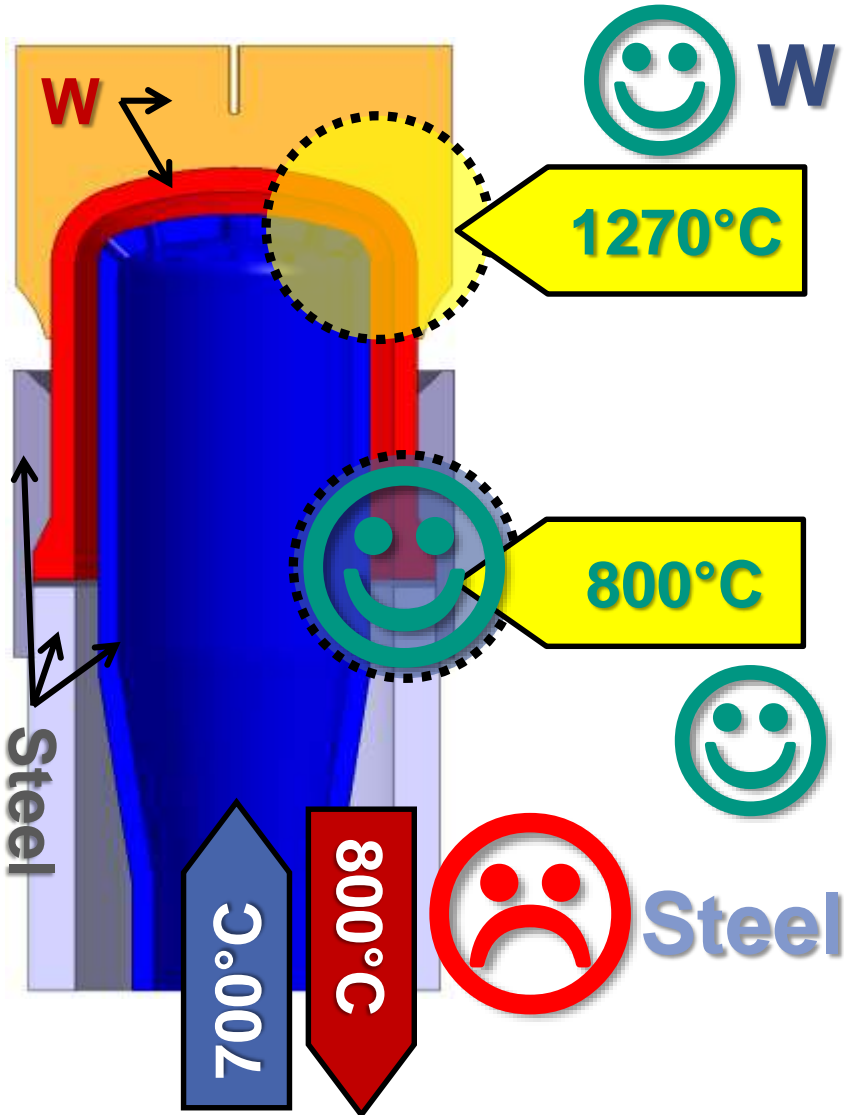
Oxidation	→	OK
Swelling ~3%	→	?
Embrittlement	→	OK
Grain Growth	→	? (ODS)

Swelling ~5%	→	?
Embrittlement	→	<b>NO GO (?)</b>

Brazing (Cu)	→	OK (...)
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Embrittlement	→	OK
Strength, ...	→	? ( <b>ODS</b> )

# MATERIALS / DESIGN WINDOW → FINGER



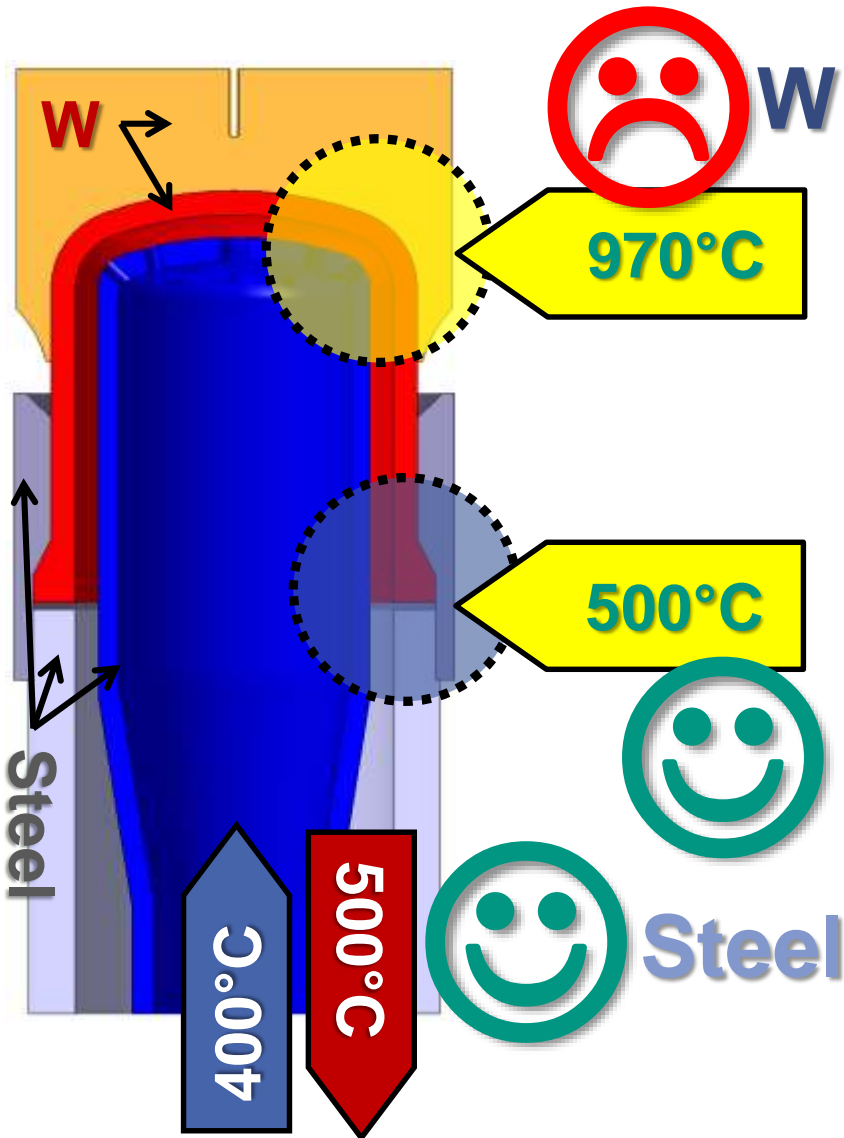
Oxidation	→	OK
Swelling ~3%	→	?
Embrittlement	→	OK
Grain Growth	→	ODS

Swelling ~5%	→	?
Embrittlement	→	OK (tbc)

Brazing (Cu)	→	OK (...)
--------------	---	----------

Embrittlement	→	OK
Strength, ...	→	ODS ???

# MATERIALS / DESIGN WINDOW → FINGER



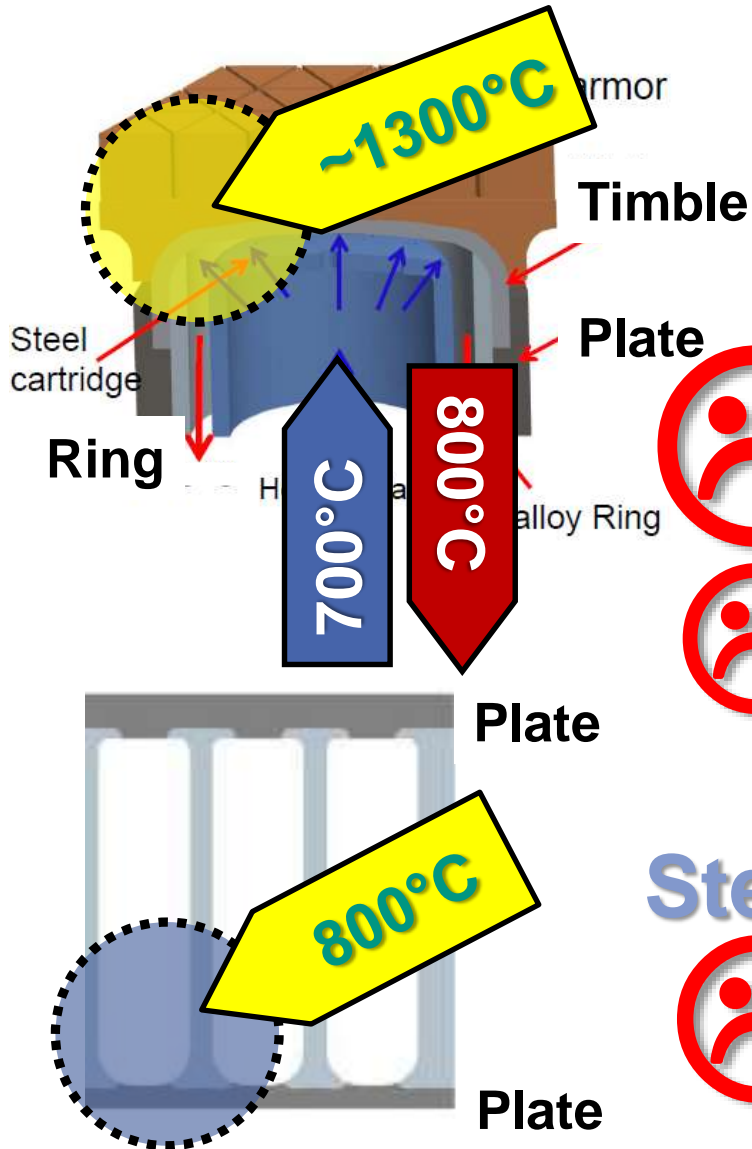
Oxidation	→	OK
Swelling <3%	→	OK (?)
Embrittlement	→	OK
Grain Growth	→	OK

Swelling <2%	→	OK (?)
Embrittlement	→	<b>NO GO</b>

Brazing (Cu)	→	OK (...)
--------------	---	----------

Embrittlement	→	OK
Strength, ...	→	OK

# MATERIALS / DESIGN WINDOW → PLATE



**W**

Oxidation	→	OK
Swelling ~3%	→	???
Embrittlement	→	OK
Grain Growth	→	ODS



Brazing W→W	→	Pd, Pt ???
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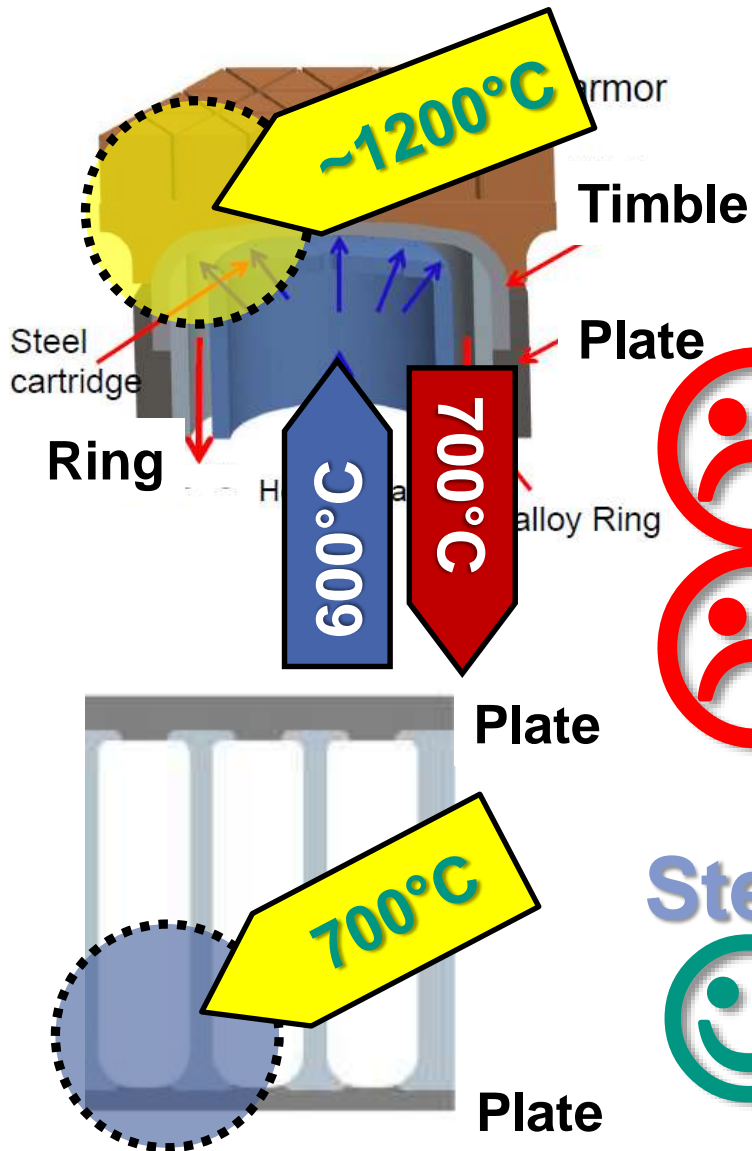
Swelling ~5%	→	?????
Embrittlement	→	OK (tbc)

**Steel**



Embrittlement	→	OK
Strength, ...	→	ODS ???

# MATERIALS / DESIGN WINDOW → PLATE



**W**

Oxidation	→	OK
Swelling ~3%	→	???
Embrittlement	→	OK
Grain Growth	→	? (ODS)

**Brazing W→W** → Pd, Pt ???

Swelling ~5%	→	?????
Embrittlement	→	NO GO (?)

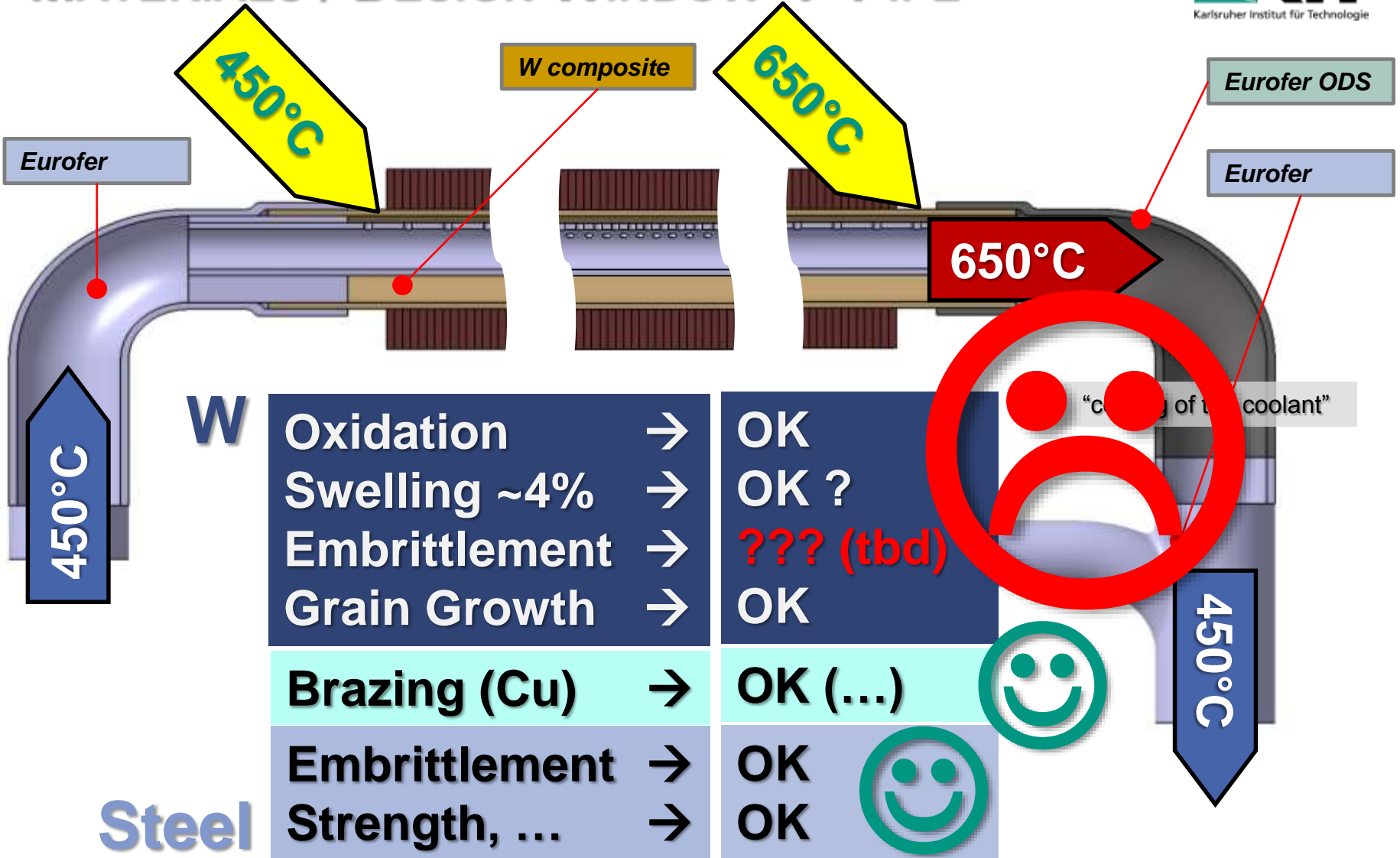


**Steel**



Embrittlement	→	OK
Strength, ...	→	? (ODS)

# MATERIALS / DESIGN WINDOW → PIPE







# SUMMARY AND PATH FORWARD

# SUMMARY

→ Thin plates (<4 mm) of pure W (including small amounts of grain stabilizers, if necessary) are the most suitable semi-finished products for structural applications !

→ The microstructure has to be adapted to the contour of the component (depending on the fabrication process to be developed) !

→ Coatings can be used as brazing material for W-steel joints, but needs additional strengthening by desing !

→ Suitable structural W-W joints are not yet demonstrated and characterized ! → Pt, Pd, ...?

→ W irradiation data needed for design (determination of lowest possible operating temperature) !!!

