

# Tungsten (W) foil laminate materials

## Advances for innovative high temperature energy conversion systems?

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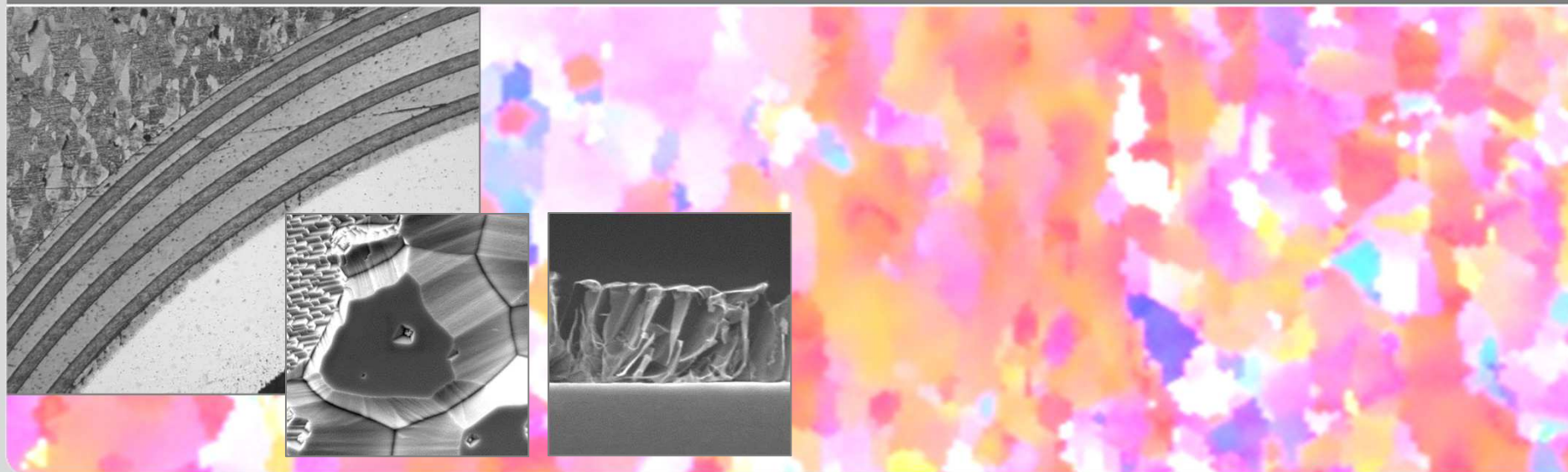
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INSTITUTE FOR APPLIED MATERIALS, APPLIED MATERIALS PHYSICS



# Contents

- W as a structural material
- W foil: ductility
- W foil: toughness
- W foil laminates
- 
- Outlook
- My vision

## What is the problem?

- Advanced energy conversion systems ask for **high temperature structural materials** (pipes)
  - High fracture toughness,  $K_{IC}$
  - Low brittle-to-ductile transition temperature (BDTT)

### Concentrated solar power (CSP)

#### CSP, tower

Molten salts, 565°C – 625°C, Ni-base  
 → corrosion, temp. gradient, liquid metal



photo: ABENGOA

Heller, DLR (2011)

#### CSP, Dish-Stirling

Helium, 900°C, Ni-base



photo: DLR, Markus Steur

#### AMTEC: alcali metal thermal to electric converter

Topping system, 24/7

$\beta''$ -alumina solid electrolyte (BASE)  
 Na (liquid) → electron +  $[Na^+]_{BASE}$

Na, 1000°C  
 → corrosion


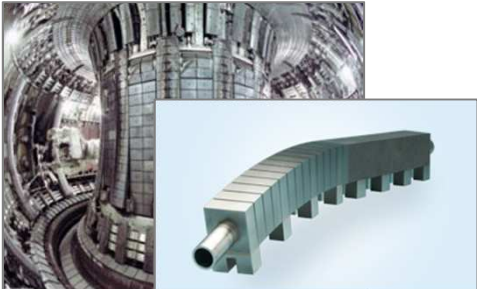



W. Hering, KIT

→ Beyond Ni-base super alloys

## What is the problem?

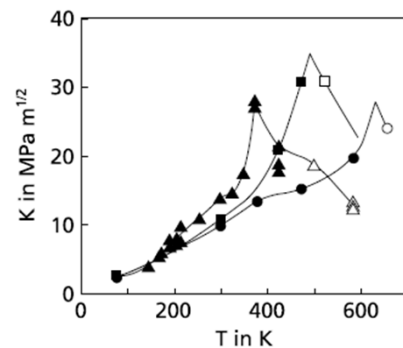
- Advanced energy conversion systems ask for **high temperature structural materials** (pipes)
  - High fracture toughness,  $K_{IC}$
  - Low brittle-to-ductile transition temperature (BDTT)

Solar chemistry	Fusion	Biomass-to-liquid (BTL)
<p><u>Solar reactor</u>            storable fuels like hydrogen (<math>H_2</math>)            metal oxide (MO) redox system:  <math>H_2O + MO_{red} \rightarrow MO_{ox} + H_2</math>  <math>MO_{ox} \rightarrow MO_{red} + \frac{1}{2}O_2</math></p>  <p style="text-align: center;">T. Denk, PSA (2011)</p>	<p><u>Divertor</u>            Helium, <math>400^\circ C - 600^\circ C</math>, <math>20 MW/m^2</math>,            neutrons  <math>D + T \rightarrow He (3.5 MeV) + n (14.1 MeV)</math></p>  <p style="text-align: center;">photo: JET      picture: PLANSEE SE</p>	<p><u>bioliq<sup>®</sup> BTL process</u>            gas cleaning and conditioning  <math>80 bar, 500^\circ C - 800^\circ C</math></p>  <p style="text-align: center;">photo: KIT</p>

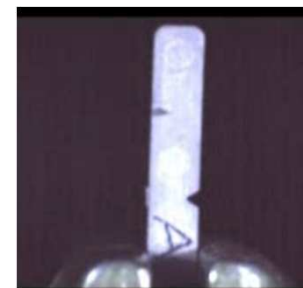
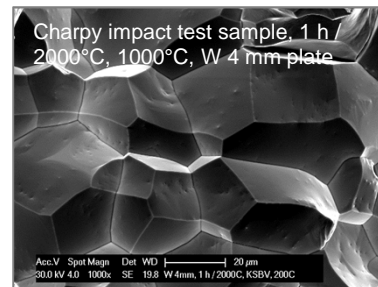
→ Beyond Ni-base super alloys

# Advantages and disadvantages of W

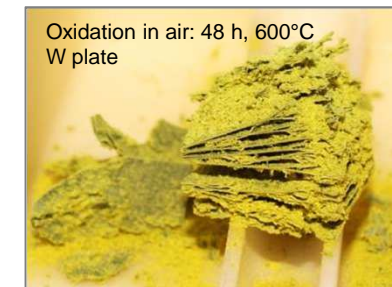
- $T_m = 3422^\circ\text{C}$
- Powder metallurgy: reduction, sintering, hot and cold working
- bcc  $\rightarrow$  BDT, strain rate dependent
- Problems:
  - Low RT fracture toughness  $\rightarrow$  **How to make W ductile?**
  - Low HAGBs cohesion  $\rightarrow$  How to strengthen the GBs?
  - Oxidation above  $600^\circ\text{C}$   $\rightarrow$  Oxidation protection layer?



P. Gumbsch, J. Nucl. Mater, 323 (2003) 304.



M. Rieth (KIT)



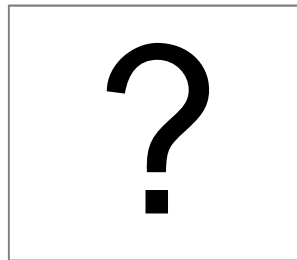
L. Commin (KIT)

## Advantages and disadvantages of W

- The mechanical properties do strongly depend on the hot and cold working process → microstructure

Paradox:

**“For W and Mo: The higher the degree of cold work the lower the BDTT.”**

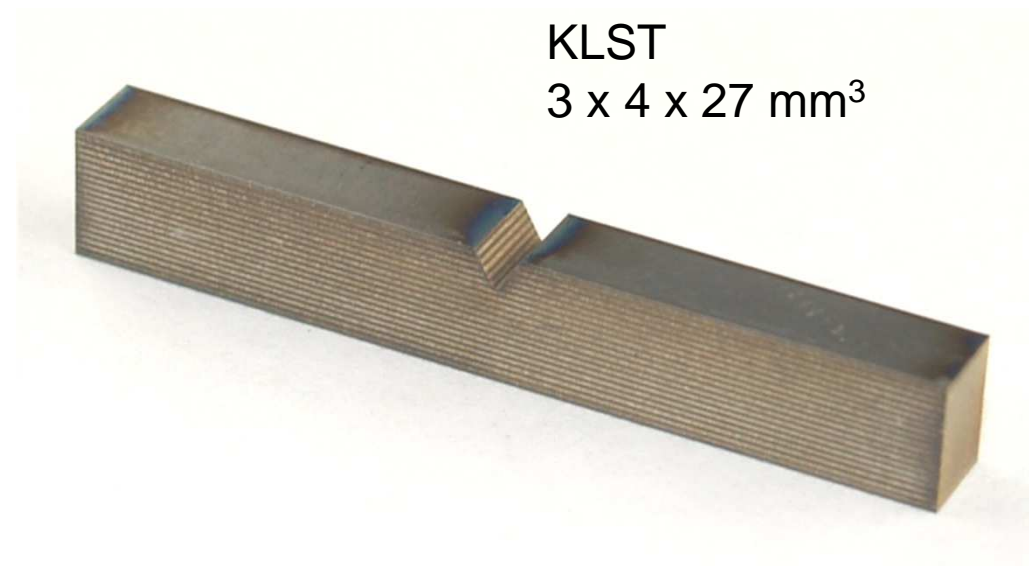
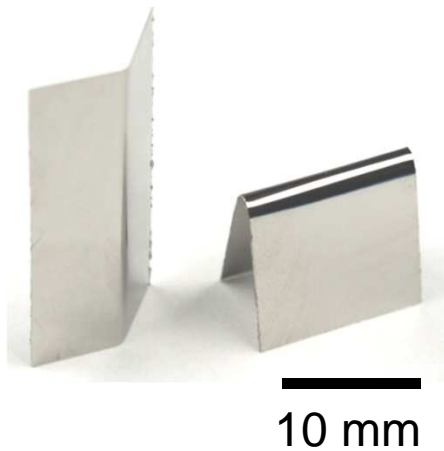


*diagram*

- W foil, 100  $\mu\text{m}$ 
  - 600°C – 800°C, grain size, dislocation density,  $\phi = 7$ ,  $\rho = 99.96\%$

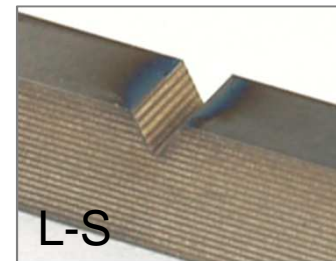
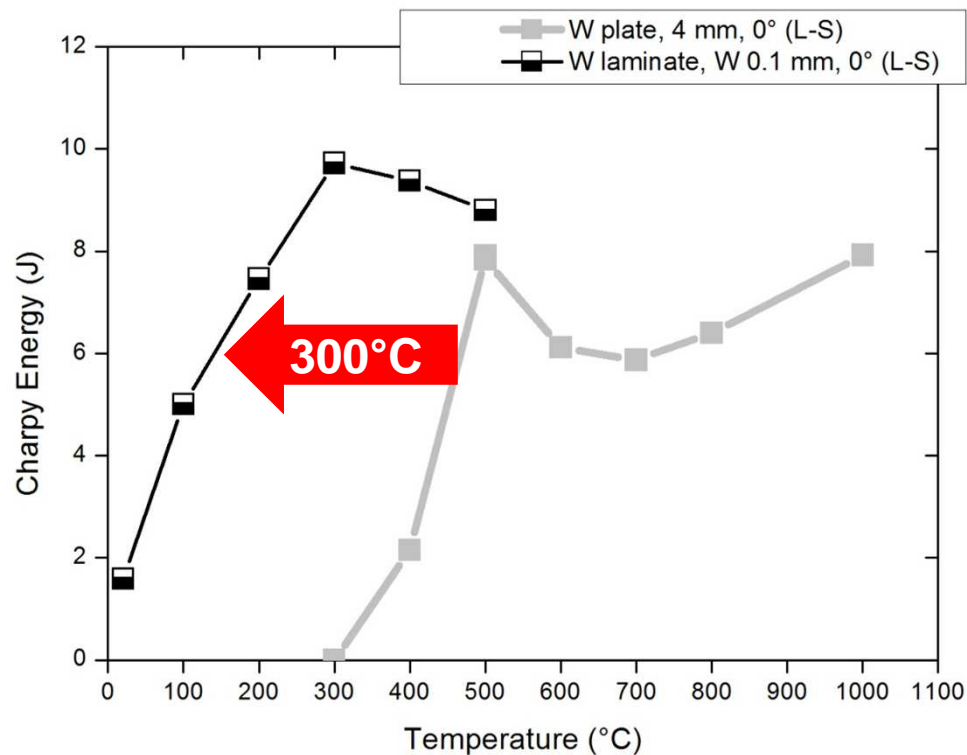
## Our idea: W foil laminate material

- Is it possible to expand the ductile properties of a W-foil to the bulk?



## Our idea: W foil laminate material

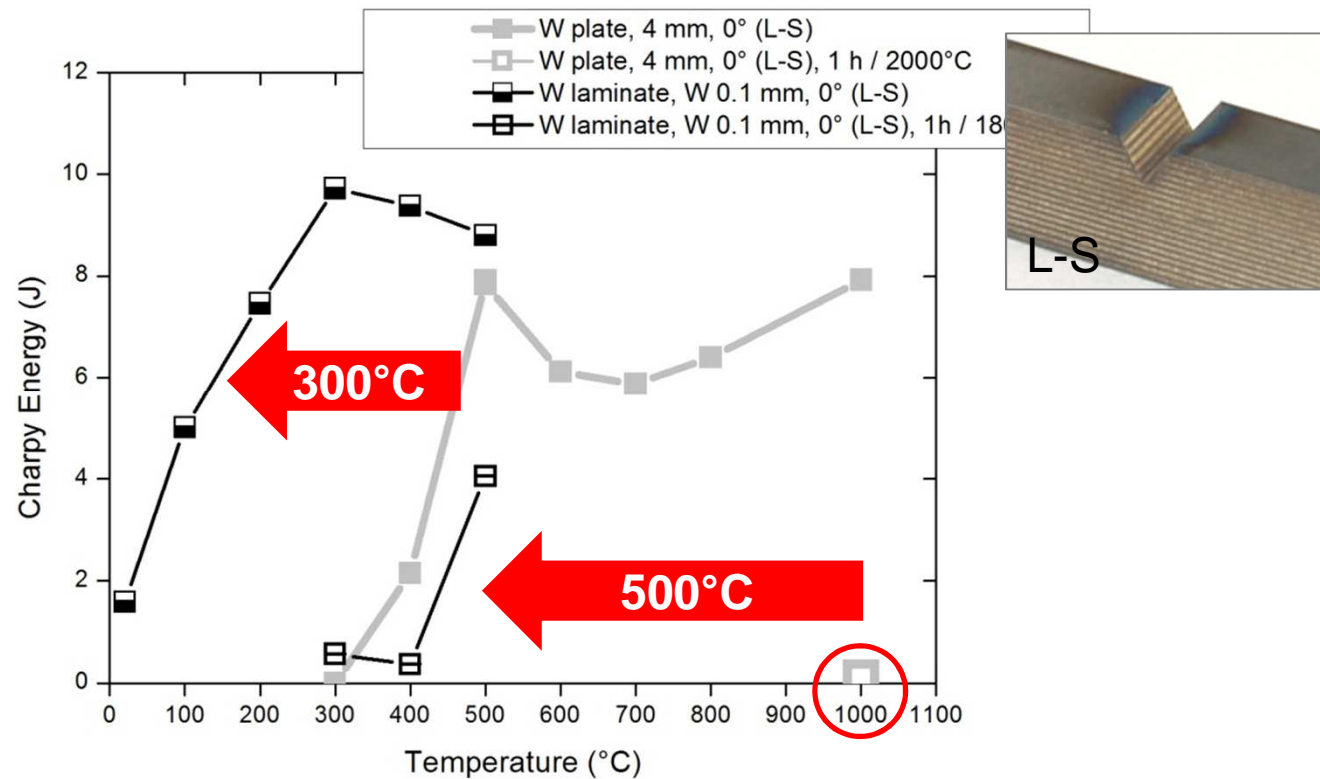
- Is it possible to expand the ductile properties of a W-foil to the bulk?
  - As-received: improvement of 300°C





## Our idea: W foil laminate material

- Is it possible to expand the ductile properties of a W-foil to the bulk?
  - As-received: improvement of 300°C
  - Recrystallized: improvement of 500°C



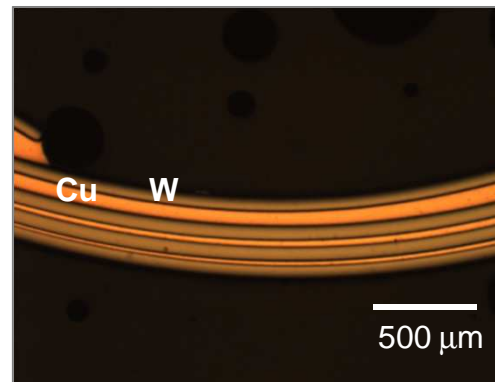
## Our idea: W foil laminate material

- Pipes with internal pressure
  - Low cohesion of HAGBs:
  - L-T direction:

applied on pressure only

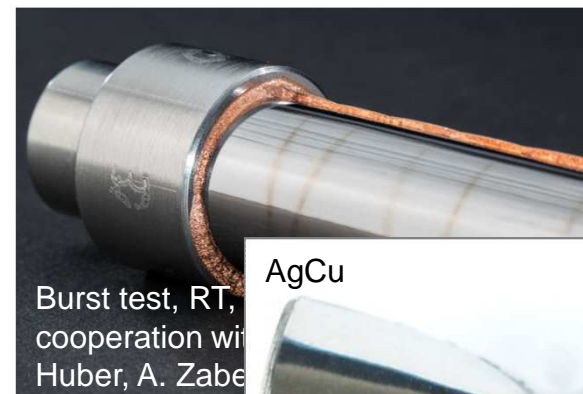
$$K_{IQ} = 70 \text{ MPa(m)}^{1/2}$$

W-laminate pipe

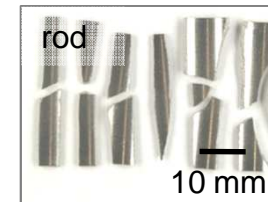


10 mm

Burst test



Charpy, 300°C

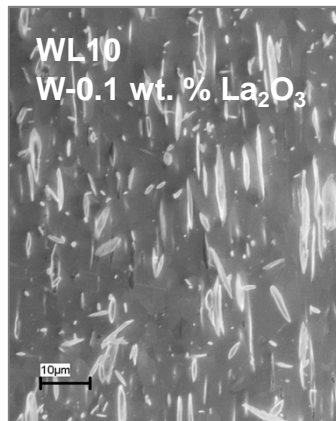


5 mm

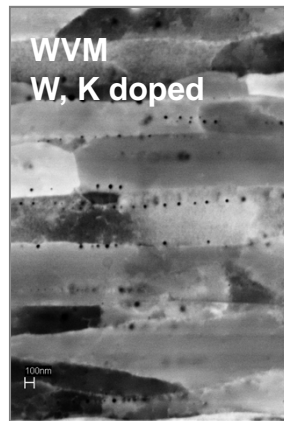
# Our idea: W foil laminate material

- Pipes with internal pressure
  - UFG grain size stabilized by: 30 - 70 ppm K (WVM foil)
  - Creep performance enhanced by: 30 - 70 ppm K

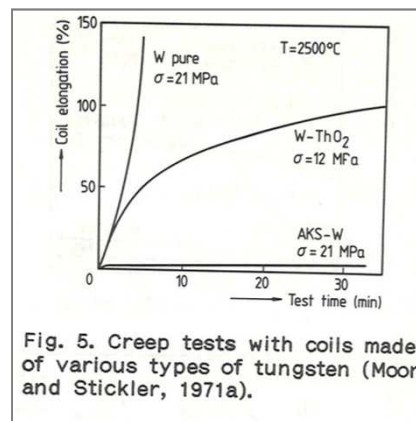
stabilized microstructure



R. Pippan, Leoben

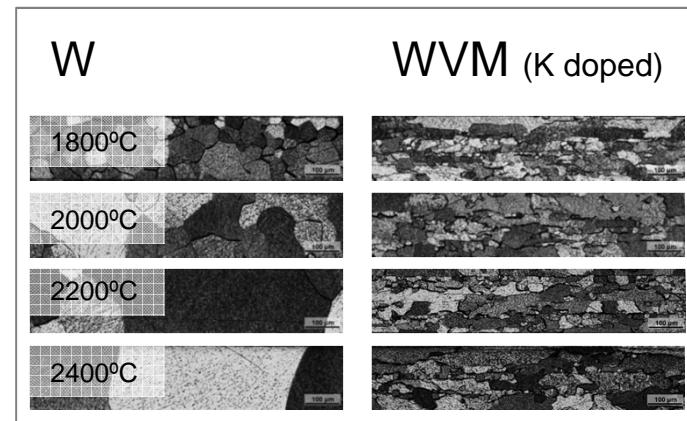


creep



E. Pink and Laszlo Bartha, 1989

recrystallization: foil, 200 µm

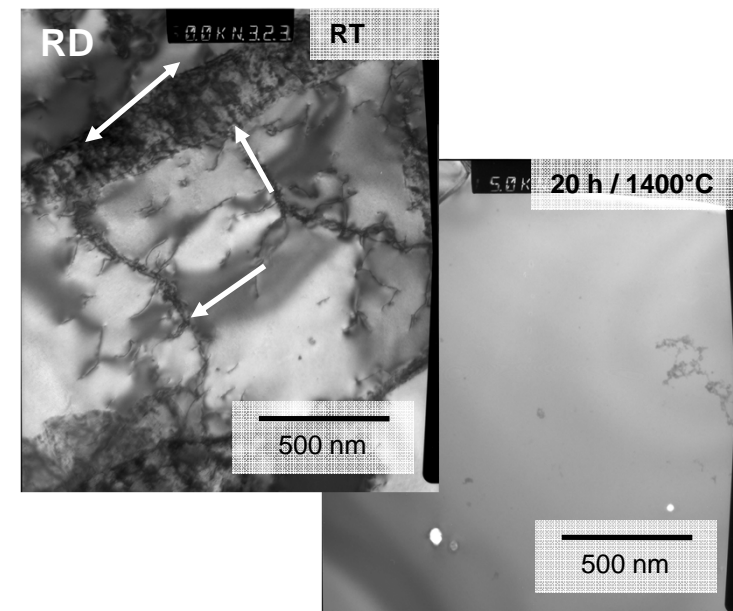
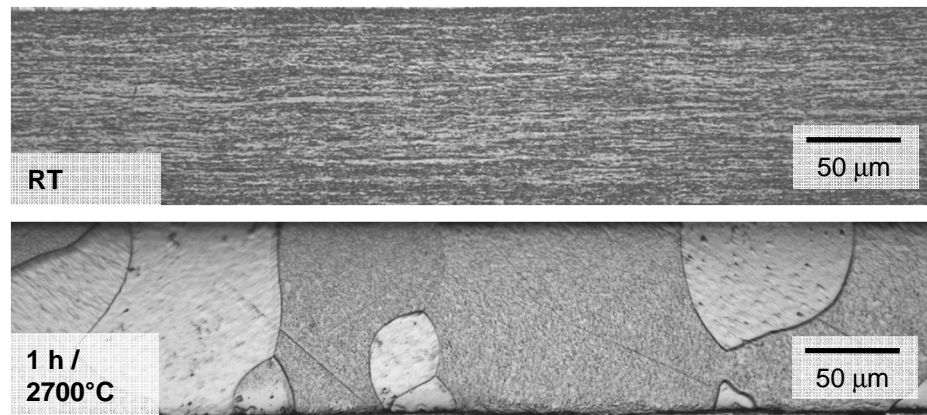
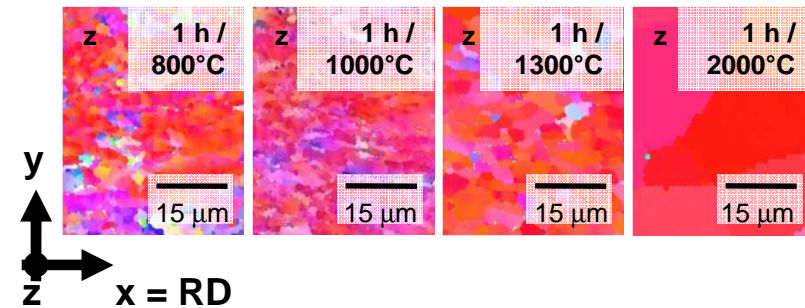




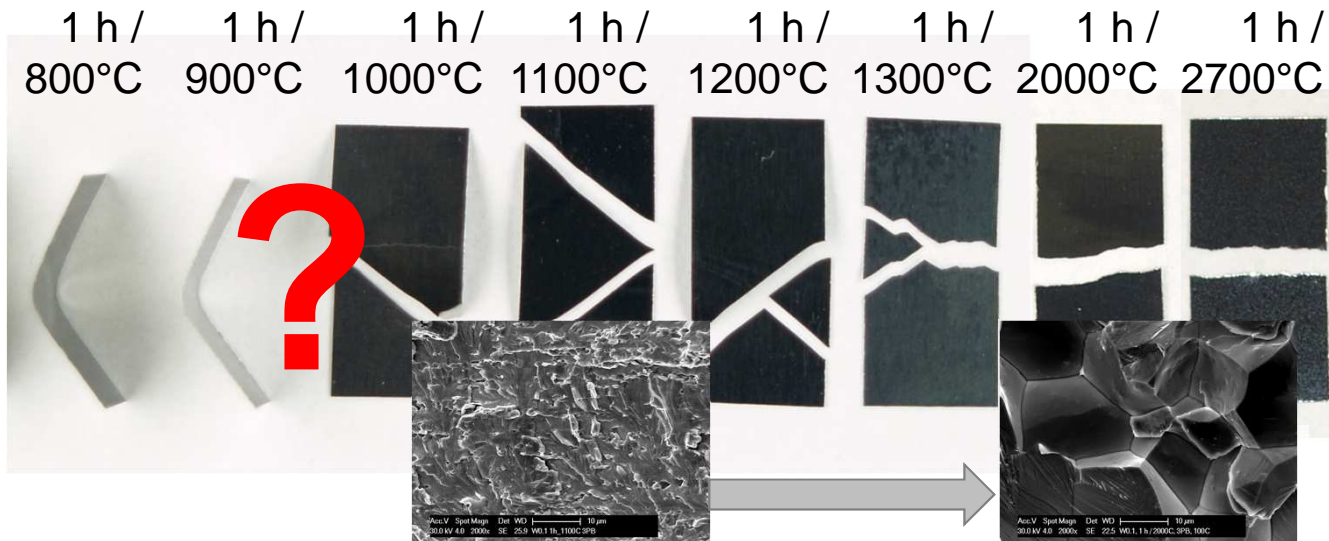
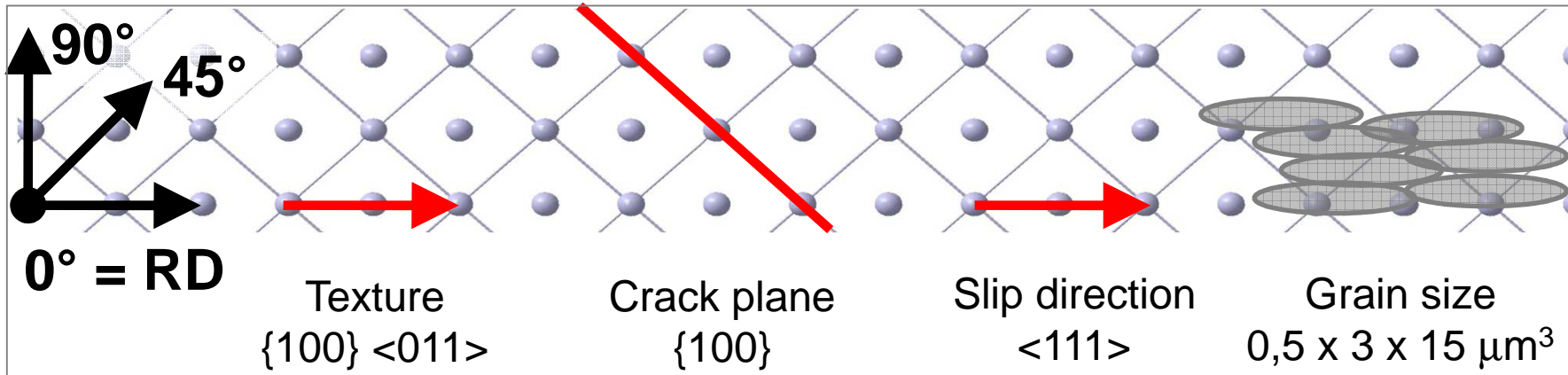
## Microstructure of W-foil, 100 $\mu\text{m}$

- Grain size:
  - As-received:  $0.5 \times 3 \times 15 \mu\text{m}^3$
  - 1 h / 2700°C:  $100 \times 100 \times 100 \mu\text{m}^3$
- Texture:  $\{100\} \langle 011 \rangle$  ; rotated cube
- Sub grains: nearly free from disl.
- Begin rxx: 1200°C

(pictures: J. Reiser, Y. Xiaou, D.E.J. Armstrong)



# 3PB-tests on W-foil, 100 μm

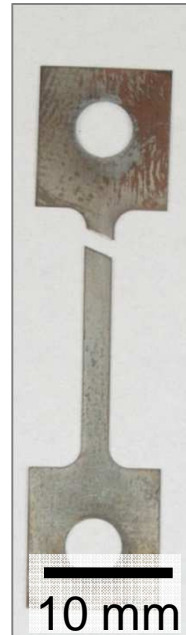
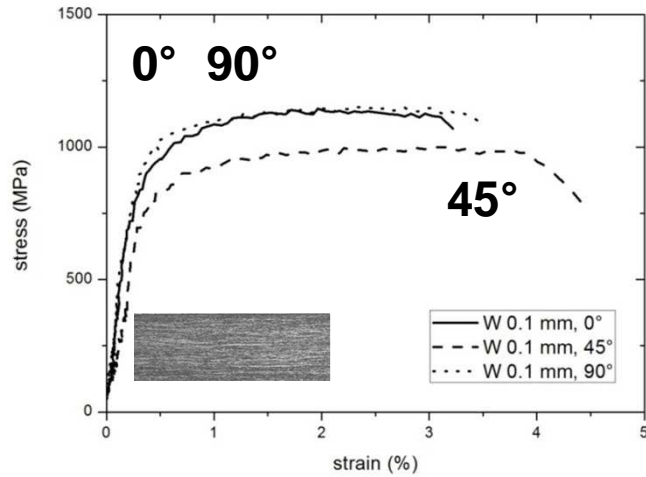


3 point bending test at RT

# Tensile tests on W-foil, 100 $\mu\text{m}$

As-received, 600°C

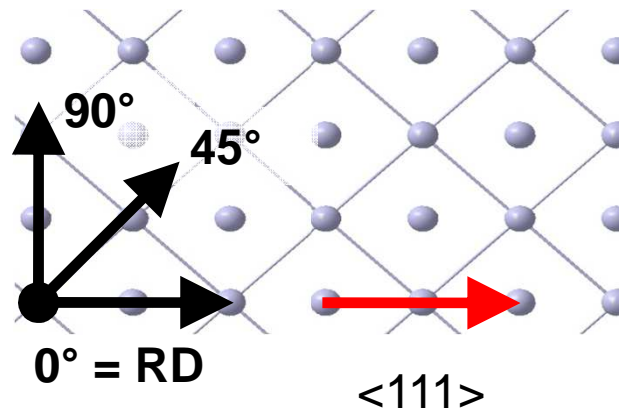
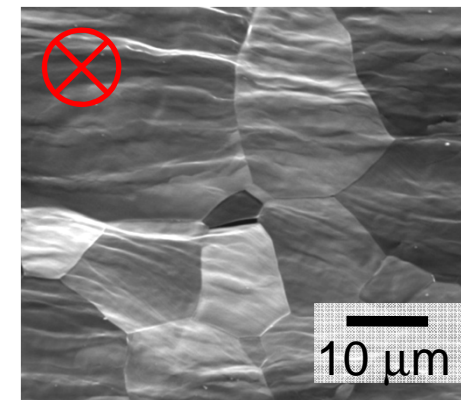
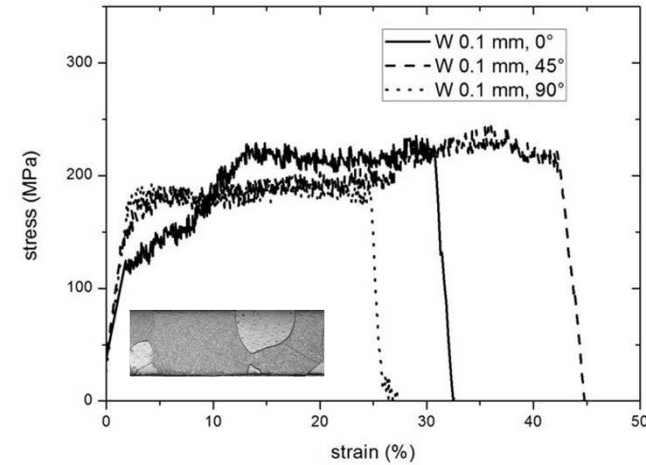
1 h / 2000°C, 600°C



As-received

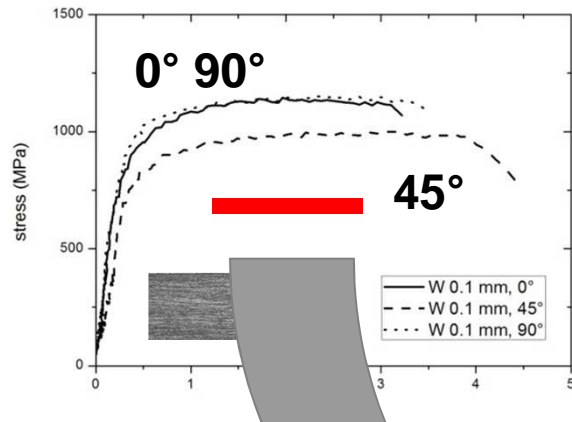


1 h / 2000°C

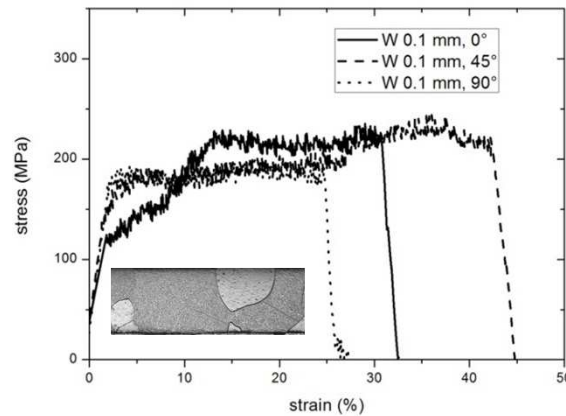


# Tensile tests on W at 600°C

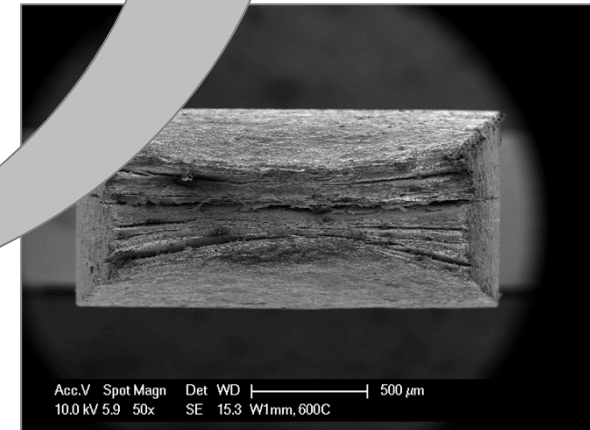
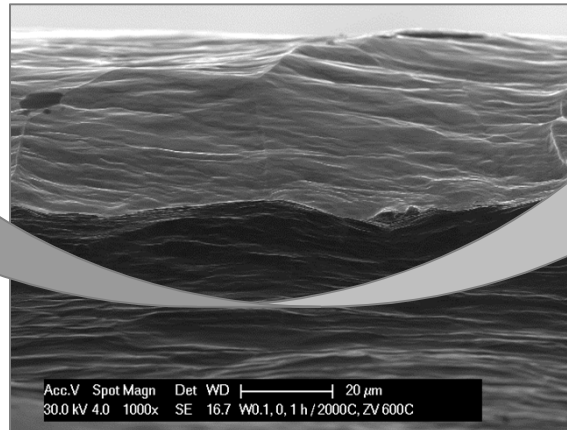
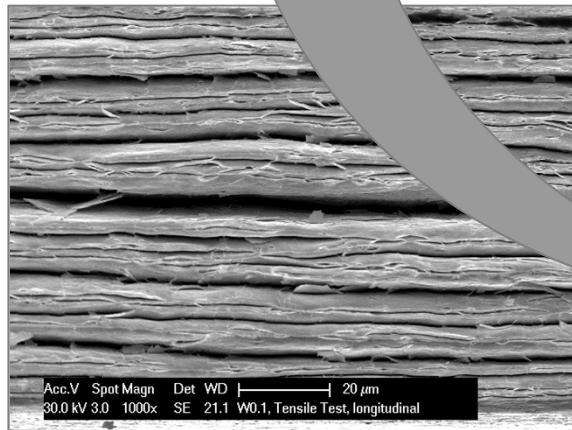
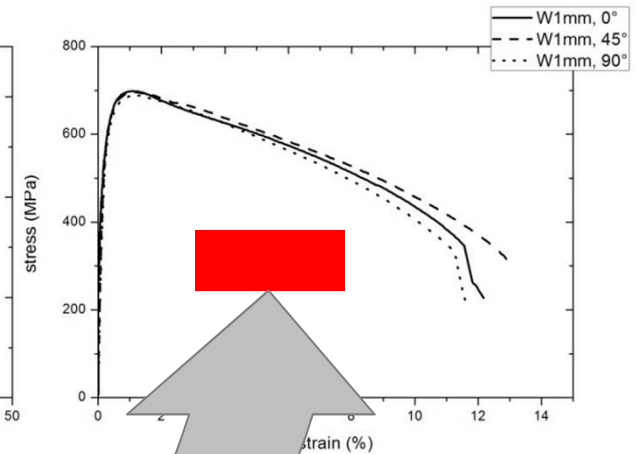
W0.1, as-received



W0.1, 1 h / 2000°C



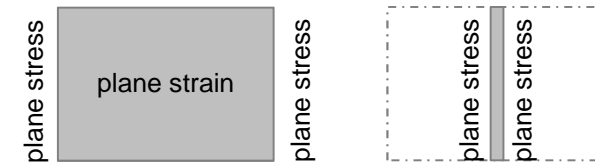
W1, as-received



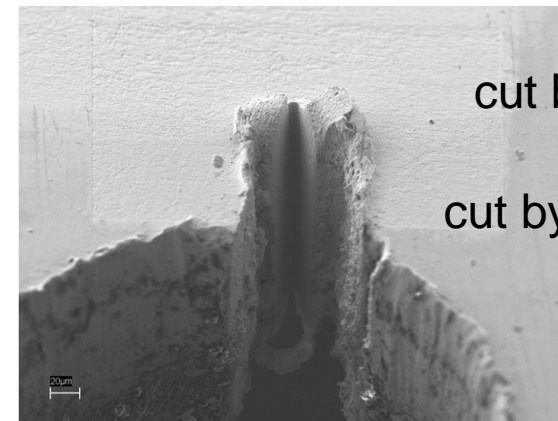
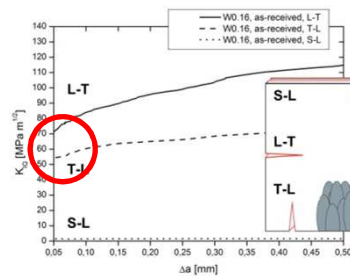
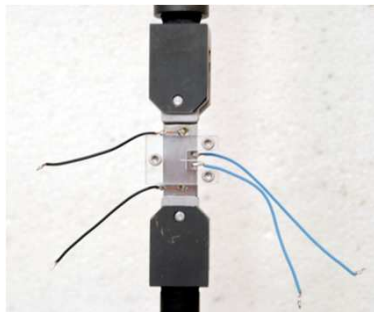


## W foil and toughness: theory

- Definition “toughness”: the ability of a material to withstand crack propagation
- W foil and size effect
  - Statistic
  - Plane strain, plane stress (ASTM E399)



$K_{I,Q}$ , pure W foil, 0.16 mm, rolled, as-received, RT

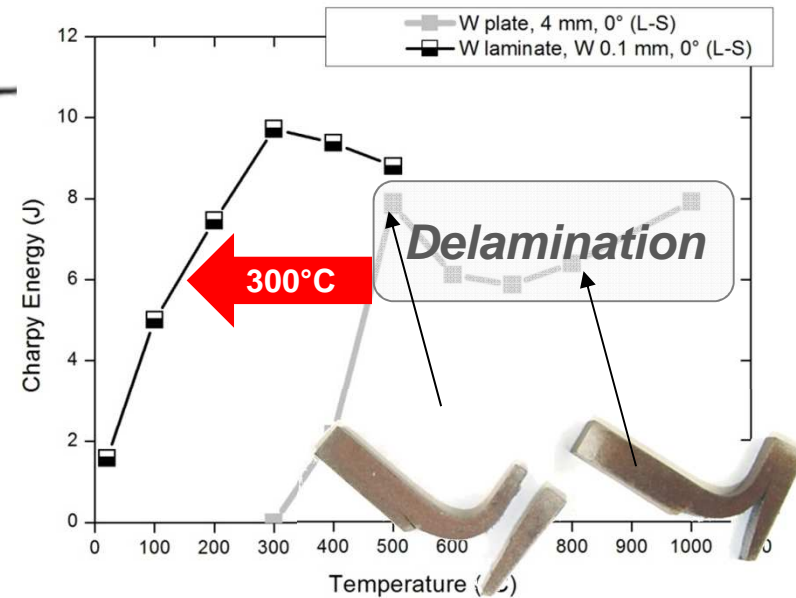
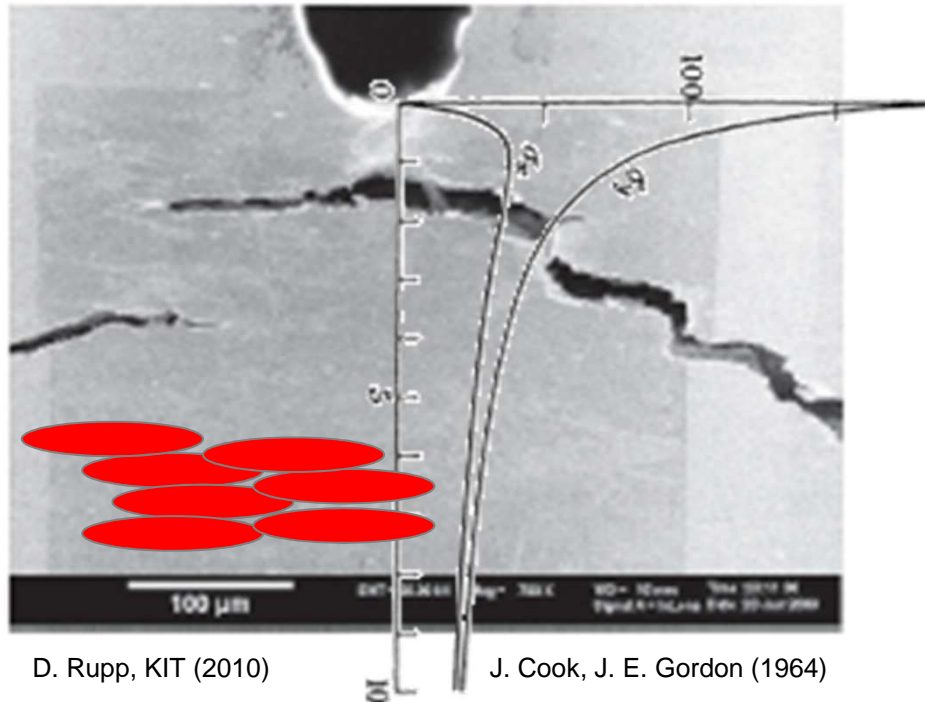


cut by FIB  
cut by razor blade  
cut by wire

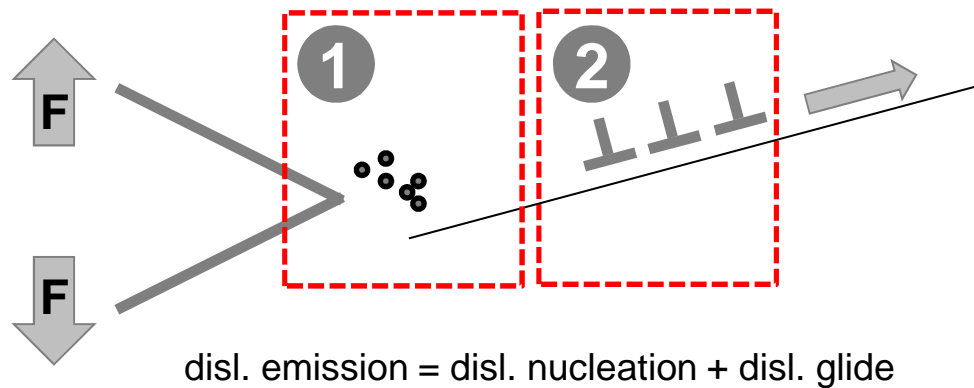
R. Pippan, presentation at W-conference, organized by R. Odette, UCLA, Santa Barbara (2011).

# Polycrystalline W and delamination

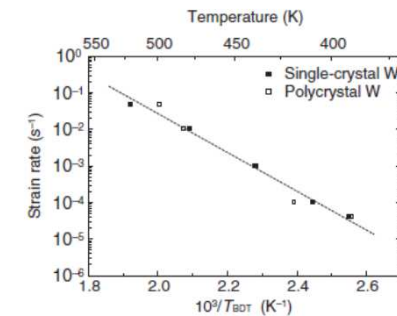
- Weak g.b. → mixed mode: mode I / mode II (Cook-Gordon mechanism)



# The controlling mechanism of the BDT

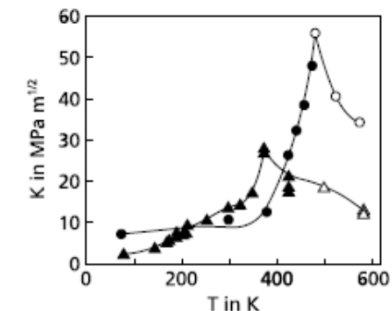


$$\dot{\varepsilon} = A \exp\left(-\frac{\Delta H_{\text{BDT}}}{kT_{\text{BDT}}}\right)$$



Roberts (2007)

- $T_{\text{BDT}}$  is strain rate dependent:  $\Delta H_{\text{BDT}} = 1,05 \pm 0,05 \text{ eV} \sim \Delta H_{\text{kp}} > \Delta H_{\text{edge}}$ 
  - BDT mobility controlled
- pronounced influence of disl. sources on  $K$ 
  - nucleation-controlled



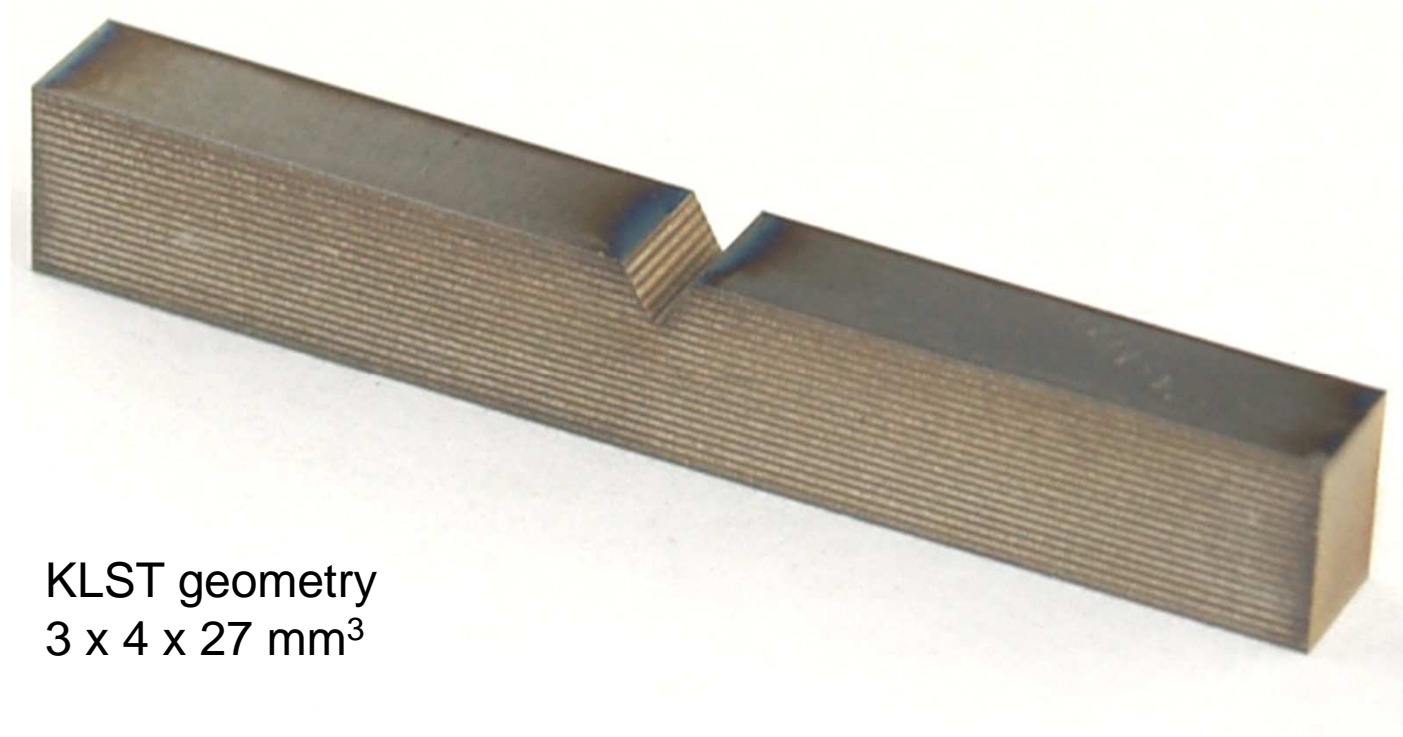
Gumbsch (2003)

## W-laminate: microstructure

The mechanical properties of a W-laminate depend on

- the condition of the W-foil as well as
- the interface

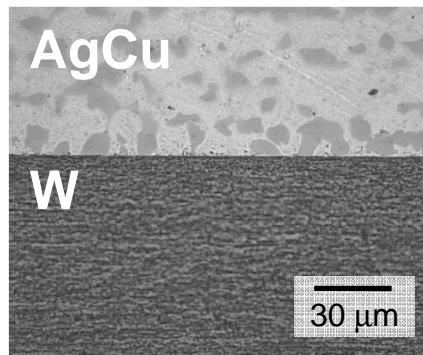
after the joining process.



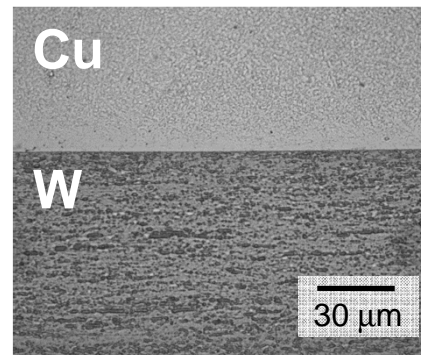
KLST geometry  
3 x 4 x 27 mm<sup>3</sup>

## W-laminate: microstructure

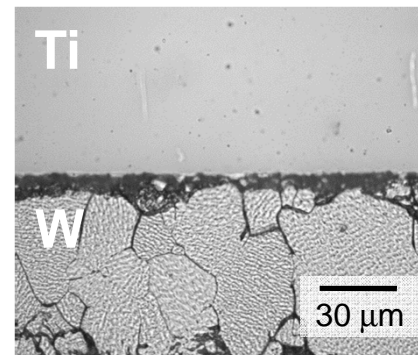
- Condition of the W-foil:
  - As-received
  - Recrystallized
- Condition of the interface:
  - Wettability
  - Solid solution
  - Intermetallic compounds



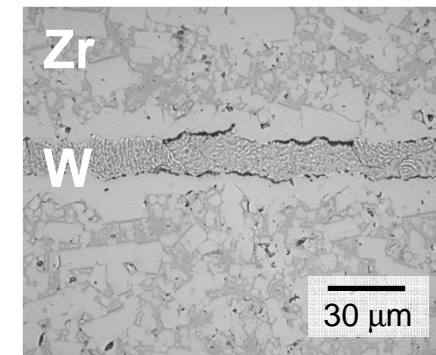
$$T_S^{\text{AgCu}} = 780^\circ\text{C}$$



$$T_S^{\text{Cu}} = 1085^\circ\text{C}$$



$$T_S^{\text{Ti}} = 1670^\circ\text{C}$$



$$T_S^{\text{Zr}} = 1855^\circ\text{C}$$

## W foil laminates: interlayer

Ti	V	Cr	Mn	Fe	Co	Ni	Cu
Zr	Nb	Mo		Ru	Rh	Pd	Ag
Hf	Ta		Re	Os	Ir	Pt	Au

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Ti	V	Cr	Mn	Fe	Co	Ni	Cu
Zr	Nb	Mo		Ru	Rh	Pd	Ag
Hf	Ta		Re	Os	Ir	Pt	Au

Insoluble

## W foil laminates: interlayer

Ti	V	Cr	Mn	Fe	Co	Ni	Cu
Zr	Nb	Mo		Ru	Rh	Pd	Ag
Hf	Ta		Re	Os	Ir	Pt	Au

Insoluble

Intermetallic phases



## W foil laminates: interlayer

Ti	V	$MW_3$ Cr	Mn	$MW, M_7W_6$ Fe	$M_7W_6$ Co	MW Ni	Cu
$MW_2$ Zr	Nb	Mo				$M_3W$ Pd	Ag
$MW_2$ Hf	Ta				MW Ir	MW Pt	Au
			Re	Os			

Insoluble

Intermetallic phases

Line compounds

## W foil laminates: interlayer

<b>Ti</b>	V	$MW_3$ <b>Cr</b>	Mn	$MW, M_7W_6$ <b>Fe</b>	$M_7W_6$ <b>Co</b>	MW <b>Ni</b>	<b>Cu</b>
$MW_2$ <b>Zr</b>	Nb	Mo				$M_3W$ <b>Pd</b>	<b>Ag</b>
$MW_2$ <b>Hf</b>	Ta					MW <b>Pt</b>	Au
			Re	Os	Ir		

Insoluble

Intermetallic phases

Line compounds

**Recommended: brazing**

## W foil laminates: interlayer

<b>Ti</b>	V	$MW_3$ <b>Cr</b>	Mn	$MW, M_7W_6$ <b>Fe</b>	$M_7W_6$ <b>Co</b>	$MW$ <b>Ni</b>	<b>Cu</b>
$MW_2$ <b>Zr</b>	Nb	Mo		<b>Ru</b>	<b>Rh</b>	$M_3W$ <b>Pd</b>	<b>Ag</b>
$MW_2$ <b>Hf</b>	Ta		<b>Re</b>	<b>Os</b>	$MW$ <b>Ir</b>	$MW$ <b>Pt</b>	<b>Au</b>

Insoluble

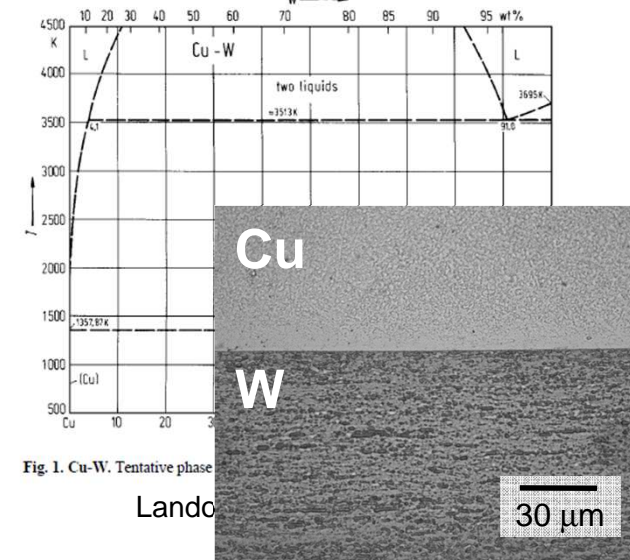
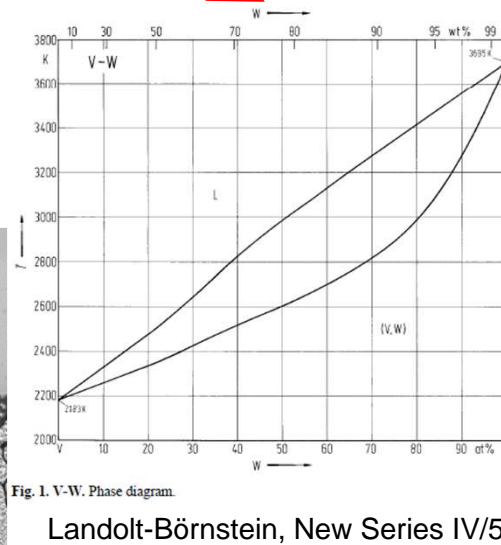
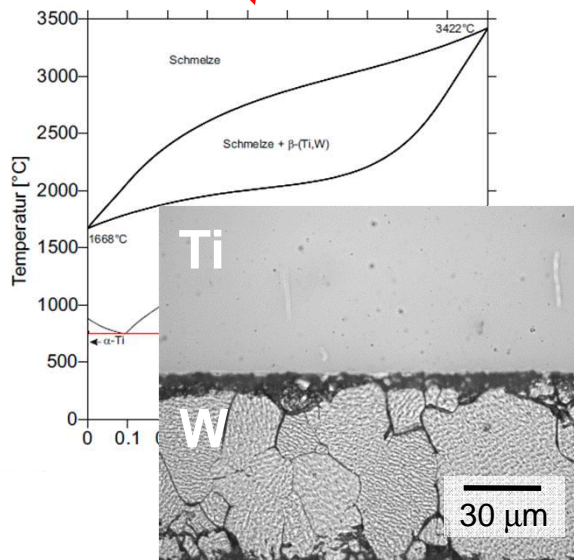
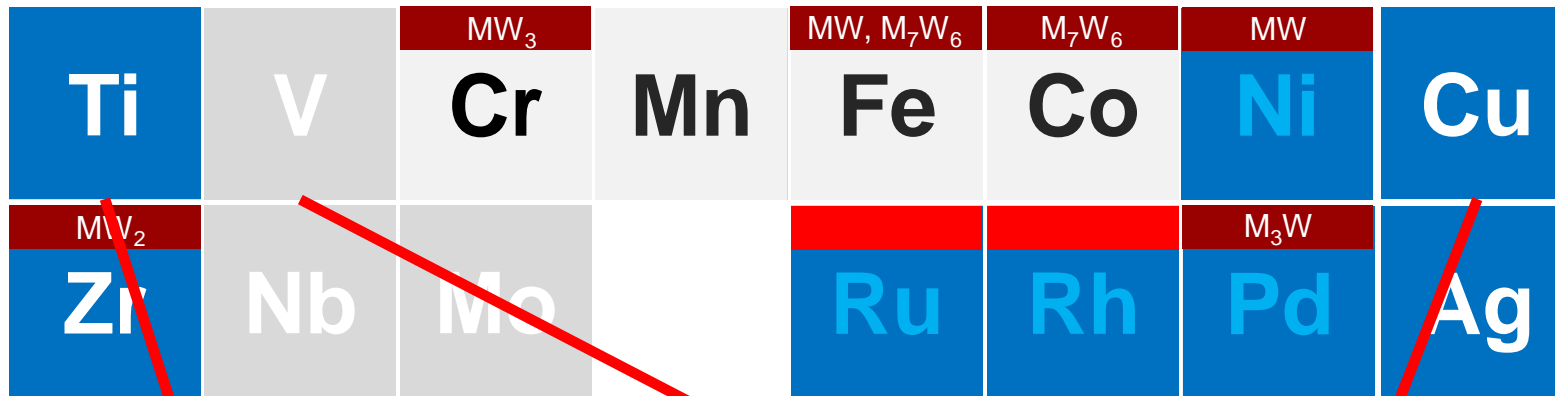
Intermetallic phases

Line compounds

Recommended: brazing

Recommended: diff. bonding

# W foil laminates: interlayer



## W foil laminates: energy dissipation

### How do W laminates dissipate energy?

Example: W-AgCu, brazed, recrystallized foil

1. plastic deformation of the W foil? → YES!
2. plastic deformation of the brazing filler? → NO!
3. crack deflection, creation of surface? → NO!



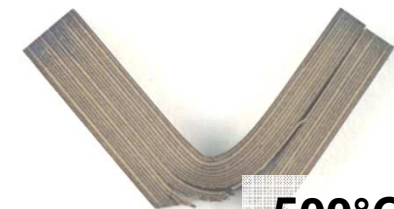
100°C



300°C

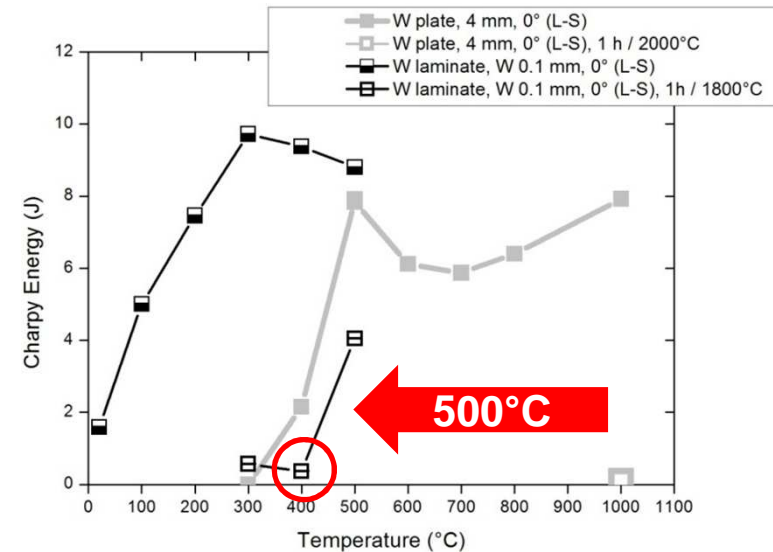


400°C



500°C

Fractured surfaces of Charpy impact test samples made of recrystallized foil.



# My vision



13.11.2013

J. Reiser, IAM-AWP, KIT

# Thank you for your attention

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