

## The brittle-to-ductile transition in cold rolled tungsten: Low-temperature toughness opens a new era in industrial application of tungsten

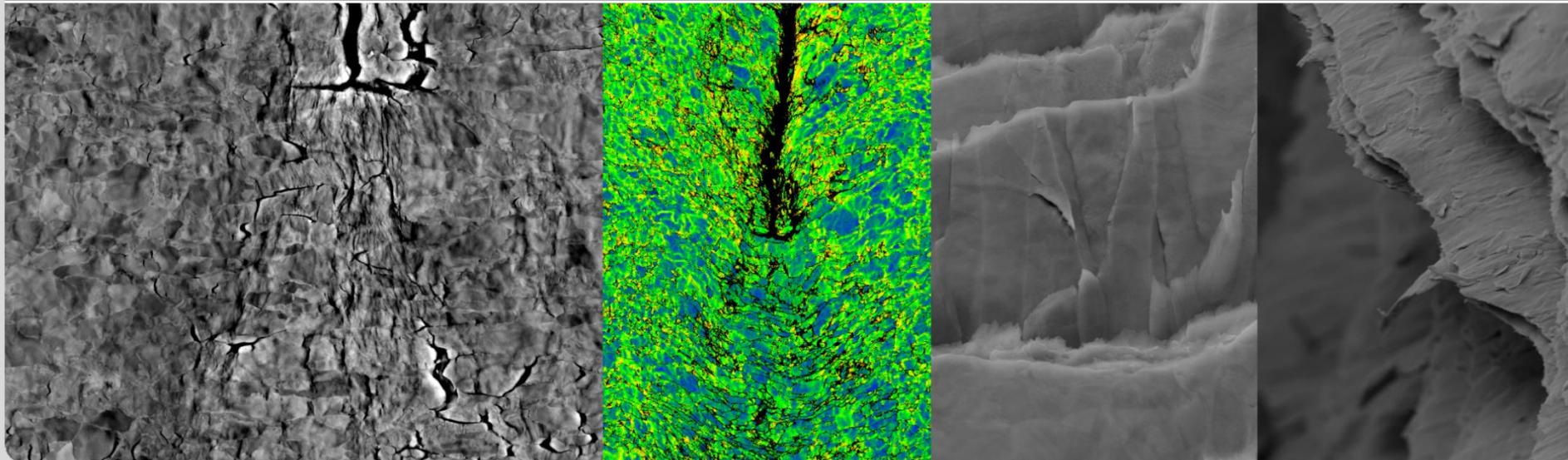
C. Bonnekoh<sup>a</sup>, H. Leiste<sup>a</sup>, J. Hoffmann<sup>a</sup>, U. Jäntschi<sup>a</sup>, A. Hoffmann<sup>b</sup>, J. Reiser<sup>a</sup>

a) Karlsruhe Institute of Technology, Institute for Applied Materials, Eggenstein-Leopoldshafen, Germany

b) Plansee SE, Flat Products, Reutte, Austria

Materials Science and Engineering (MSE) Congress, Darmstadt, 27.09.2018

IAM-AWP, Department Metallic Materials, High-temperature Materials Group



# Outline

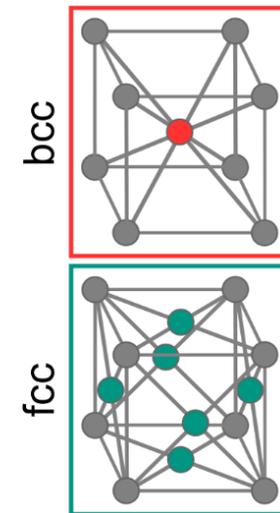
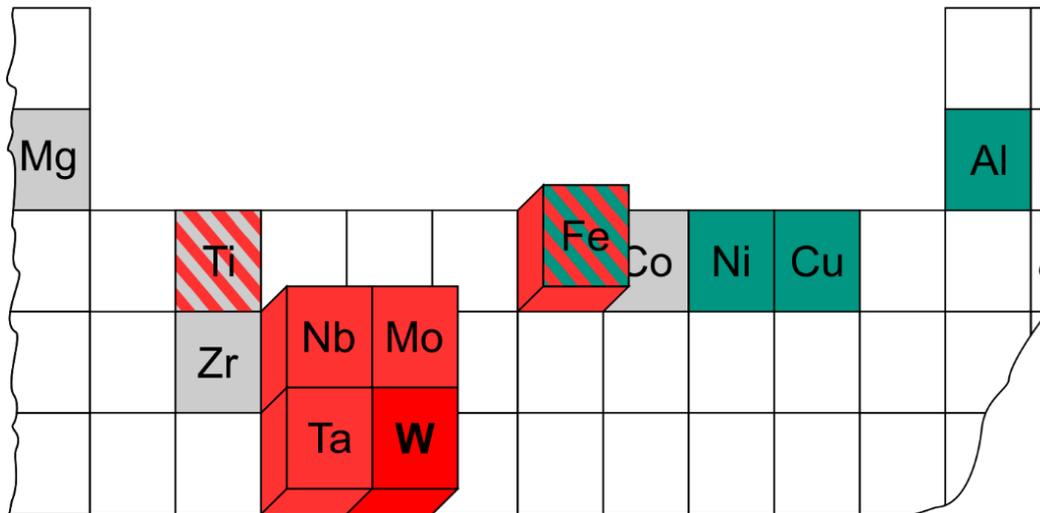
- Motivation
- Methods
- Materials
- Results
- Summary

# Outline

- Motivation
- Methods
- Materials
- Results
- Summary

# Motivation | Brittle-to-ductile transition

- ❖ Brittle-to-ductile transition (BDT) limits the field of application for safe operation of tungsten (W) above its BDT temperature ( $\sim 680 \text{ K} - 880 \text{ K}$ )<sup>1-3</sup>



- ❖ **Pre-deformation improves** mechanical properties of pure W materials<sup>4-6</sup>
- ❖ What mechanism is responsible for this improvement?

[1] Smid, I. et al.: J. Nucl. Mater. 1998;258-263:160-172  
 [2] Faleschini, M. et al.: J. Nucl. Mater. 2007;367-370:800-805  
 [3] Giannattasio, A. et al.: Philos. Mag. 2010;90(30):3947-3959

[4] Reiser, J. et al.: Int. J. Refract. Met. Hard Mater. 2016;54:351-369  
 [5] Németh, A.A.N., et al.: Int. J. Refract. Met. Hard Mater. 2015;50:9-15  
 [6] Nikolić, V. et al.: Int. J. Refract. Met. Hard Mater. 2018;76:214-225

# Outline

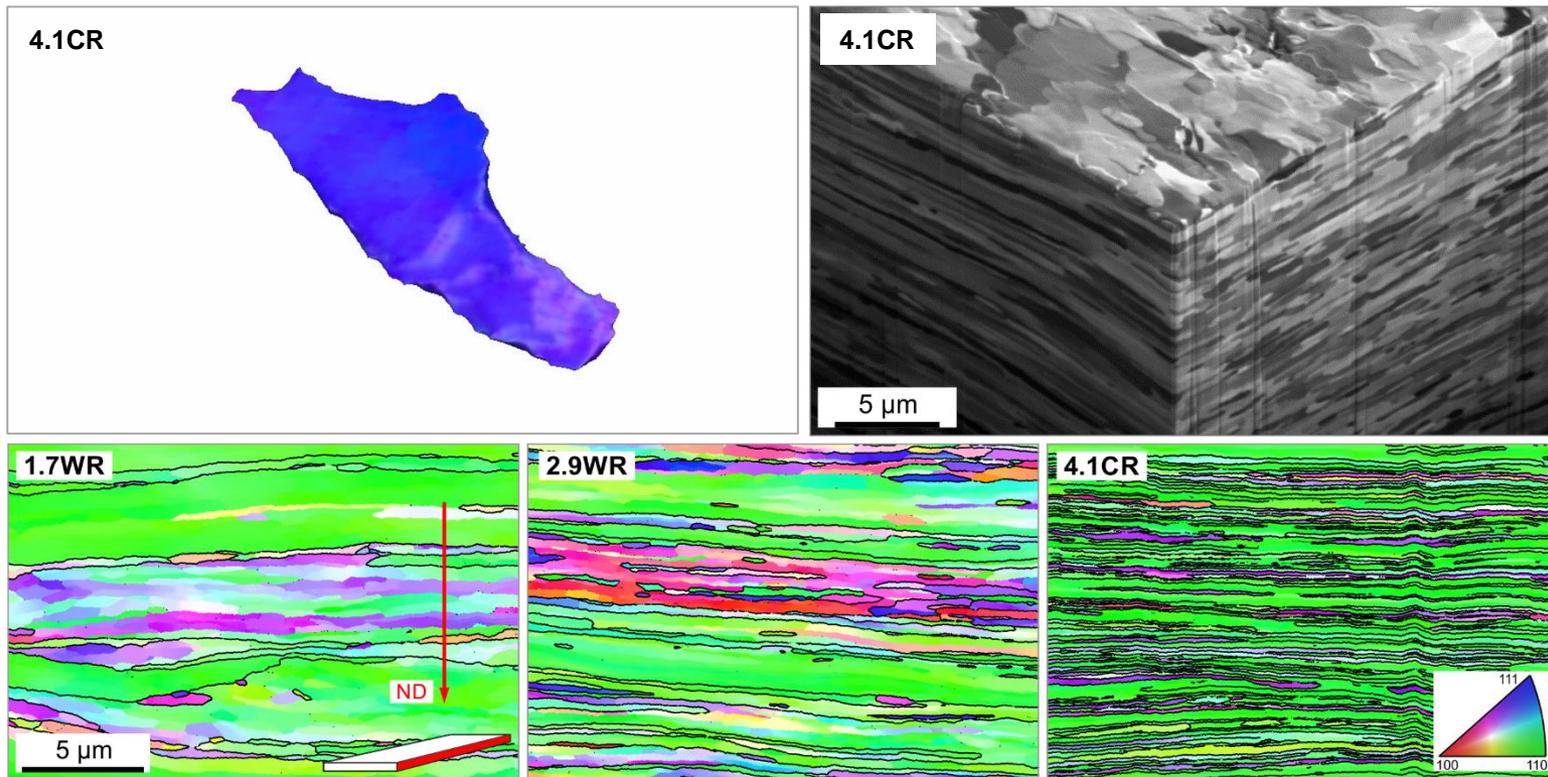
- **Motivation**
- **Materials**
- **Methods**
- **Results**
- **Summary**

# Outline

- Motivation
- **Materials**
- Methods
- Results
- Summary

# Materials | Microstructure

- ❖ Warm- and cold-rolled W sheets (log. 1.7 – 4.1, 1.0 mm – 0.1 mm thick) made of a **single hot-rolled plate** in cooperation with Plansee SE, Reutte



[7] Bonnekoh, C. et al.: Int. J. Refract. Met. Hard Mater. 2018;71(71):181–189  
 [8] Bonnekoh, C. et al.: Int. J. Refract. Met. Hard Mater. 2019;78(78):146–163

# Outline

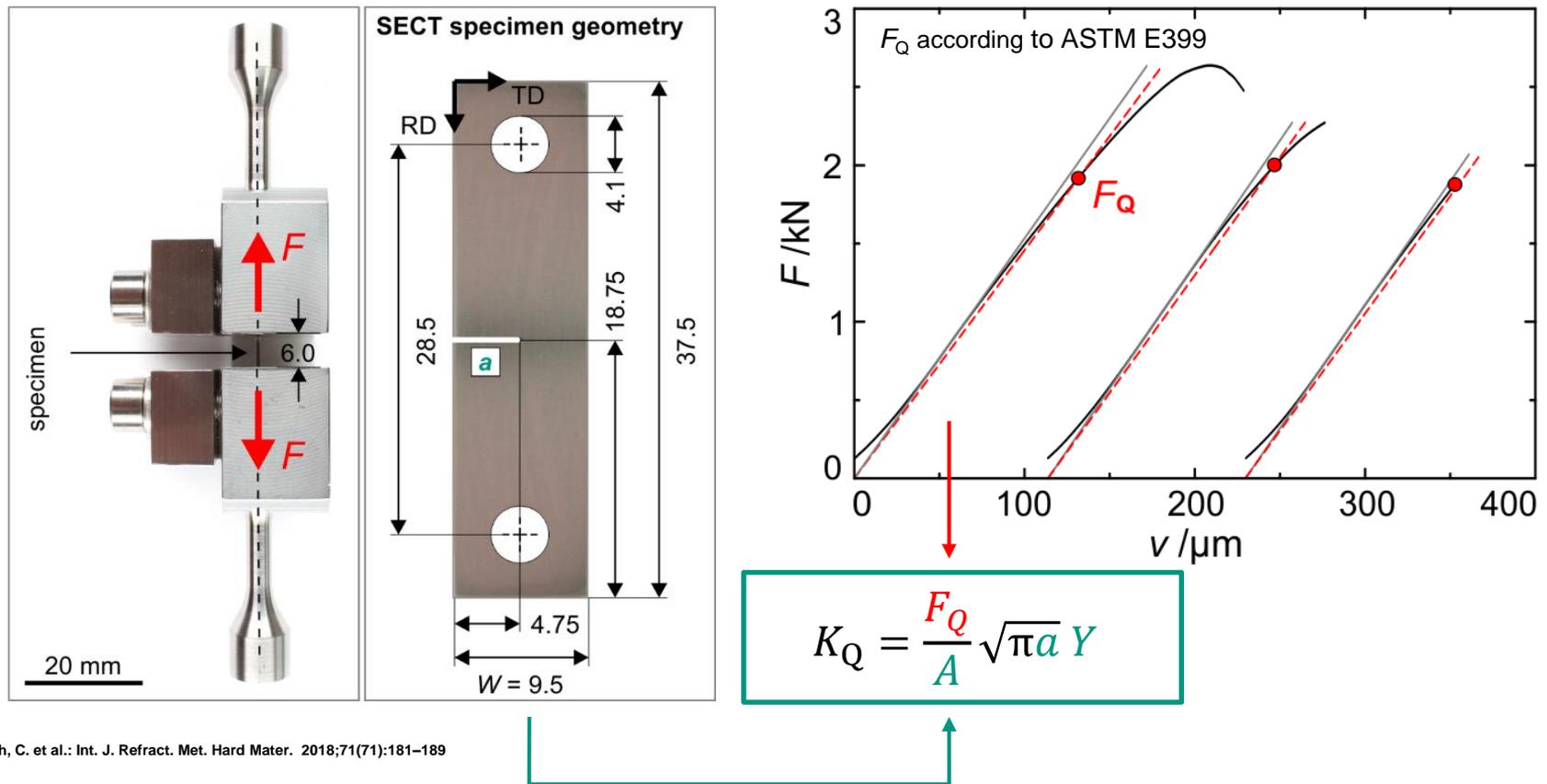
- Motivation
- **Materials**
- Methods
- Results
- Summary

# Outline

- Motivation
- Materials
- **Methods**
- Results
- Summary

# Methods | Fracture toughness tests

- ❖ SECT specimens with L-T crack system stressed by a modulus I load
- ❖ Parameter range:  $120 \leq T \leq 580$  K and  $0.01 \leq dK/dt \leq 100$  MPa m<sup>0.5</sup> s<sup>-1</sup>



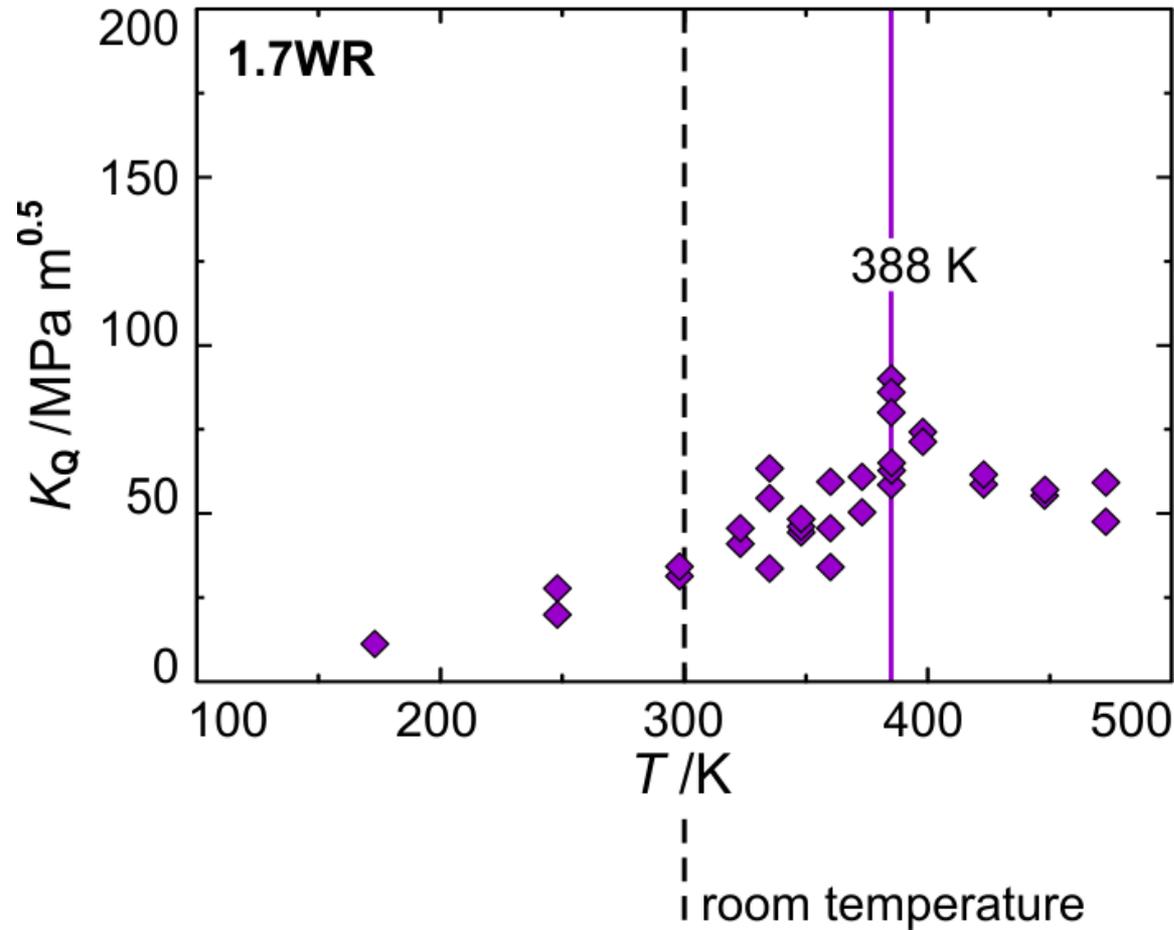
# Outline

- Motivation
- Materials
- **Methods**
- Results
- Summary

# Outline

- Motivation
- Materials
- Methods
- **Results**
  - **BDT temperatures**
  - BDT activation energies
  - BDT / microstructure correlations
- Summary

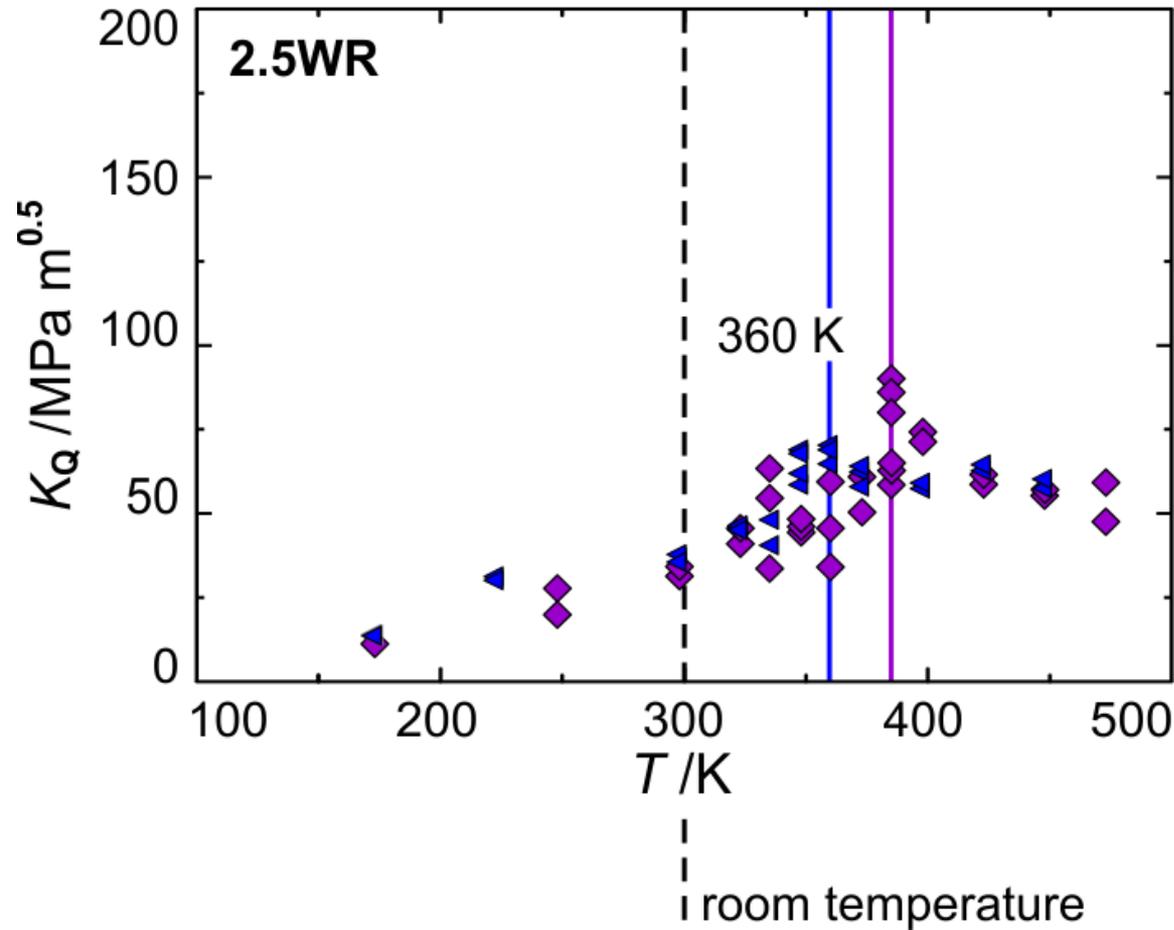
# Results | BDT temperatures



[7] Bonnekoh, C. et al.: Int. J. Refract. Met. Hard Mater. 2018;71(71):181–189

[8] Bonnekoh, C. et al.: Int. J. Refract. Met. Hard Mater. 2019;78(78):146–163

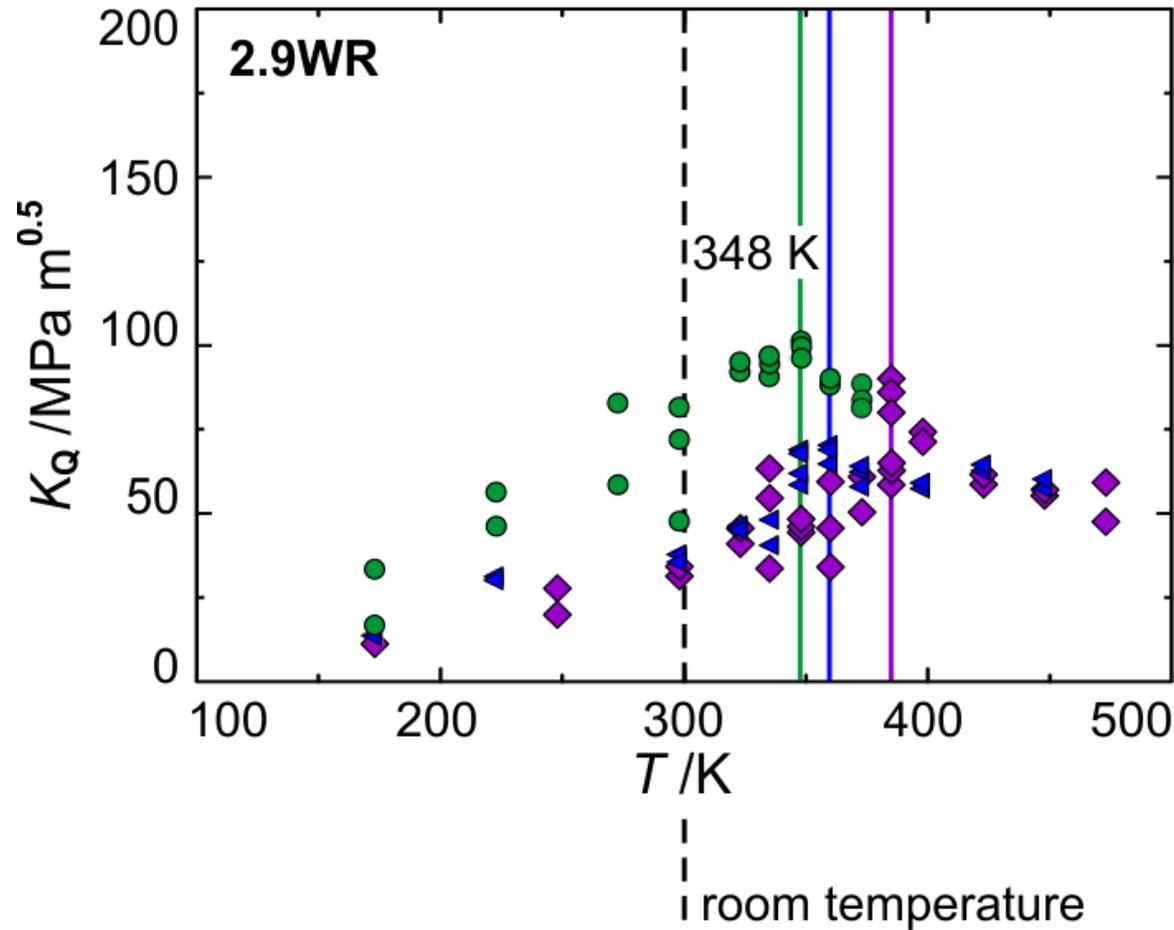
# Results | BDT temperatures



[7] Bonnekoh, C. et al.: Int. J. Refract. Met. Hard Mater. 2018;71(71):181–189

[8] Bonnekoh, C. et al.: Int. J. Refract. Met. Hard Mater. 2019;78(78):146–163

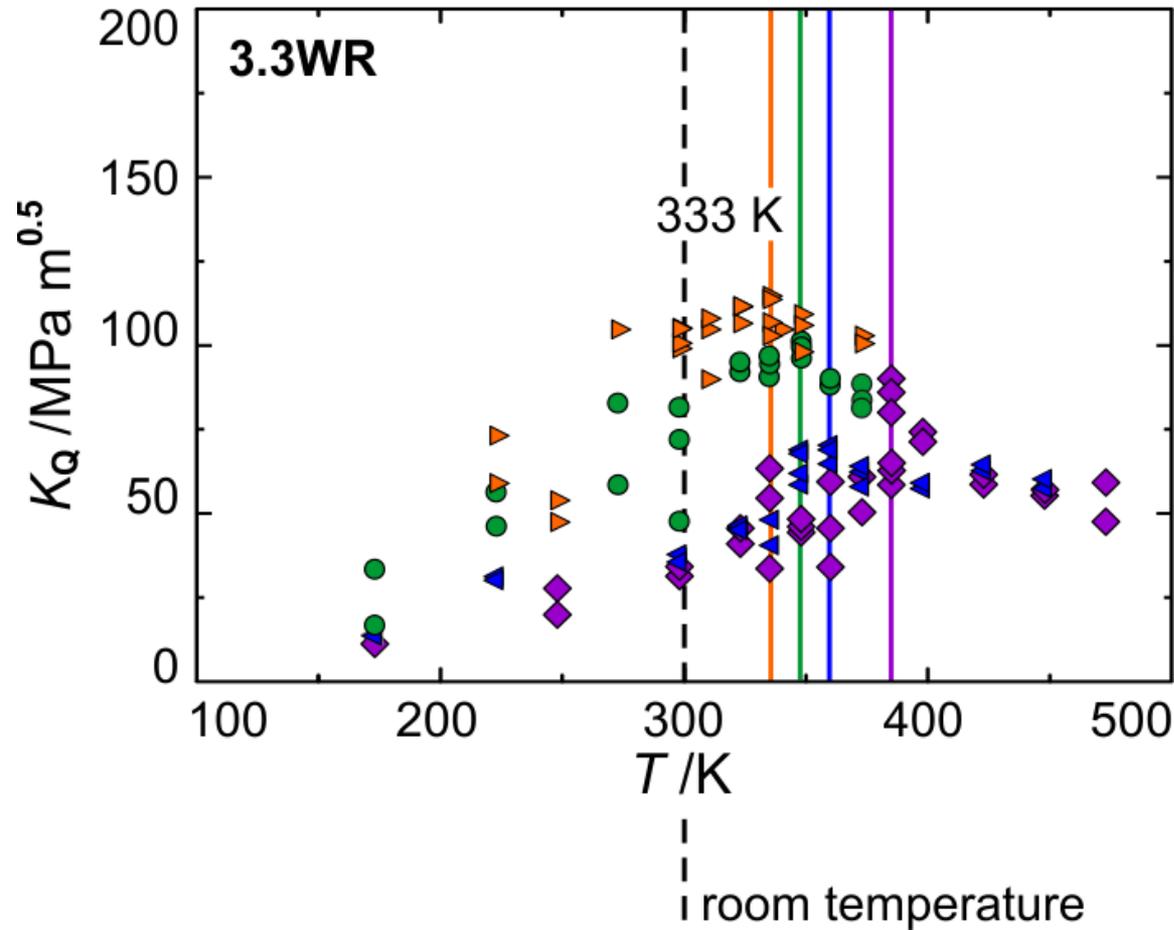
# Results | BDT temperatures



[7] Bonnekoh, C. et al.: Int. J. Refract. Met. Hard Mater. 2018;71(71):181–189

[8] Bonnekoh, C. et al.: Int. J. Refract. Met. Hard Mater. 2019;78(78):146–163

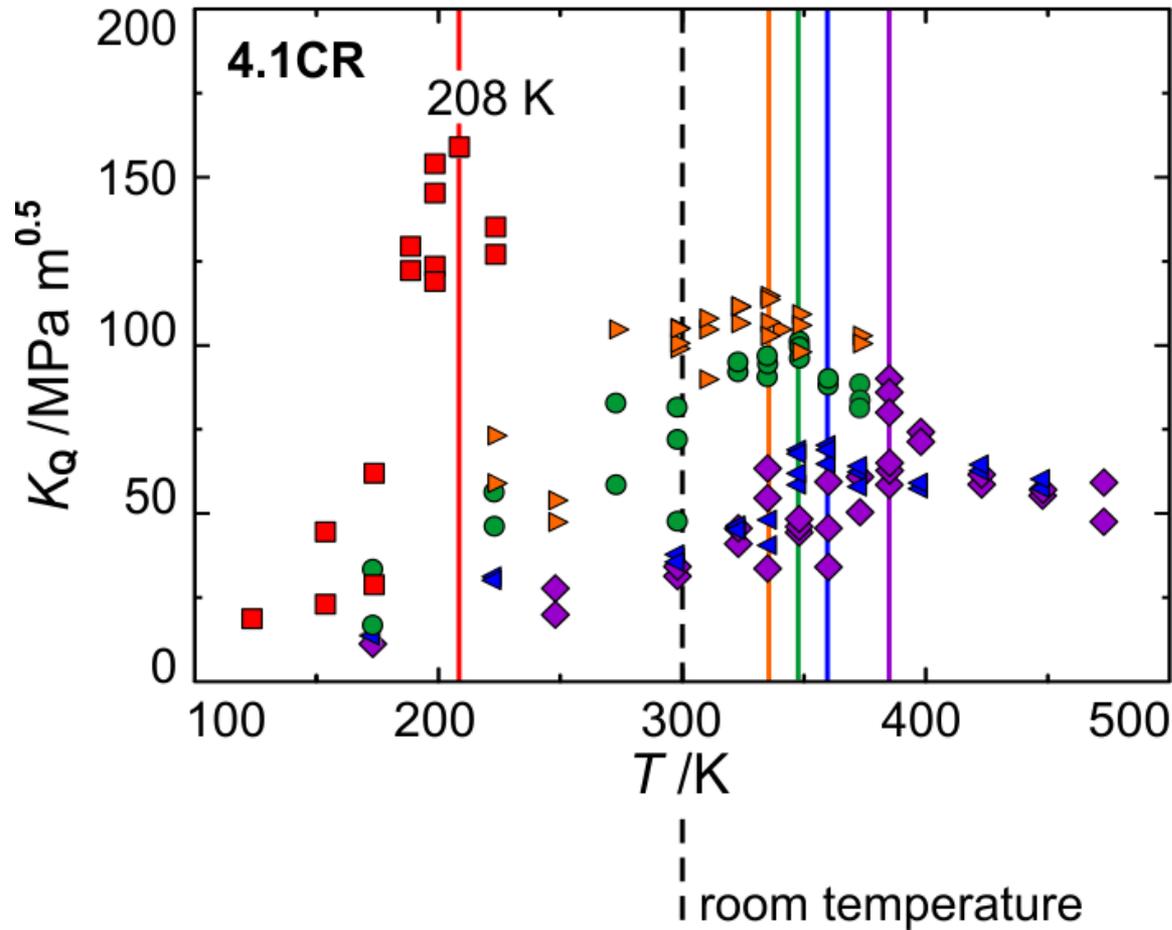
# Results | BDT temperatures



[7] Bonnekoh, C. et al.: Int. J. Refract. Met. Hard Mater. 2018;71(71):181–189

[8] Bonnekoh, C. et al.: Int. J. Refract. Met. Hard Mater. 2019;78(78):146–163

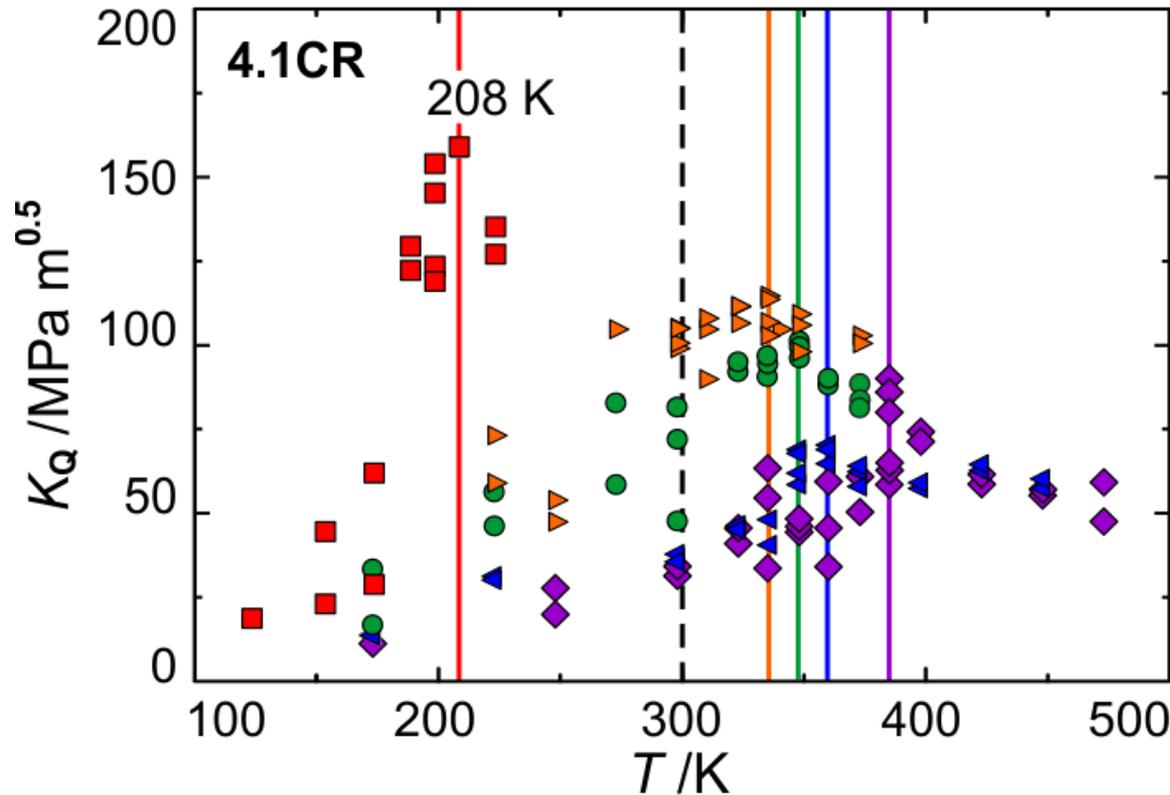
# Results | BDT temperatures



[7] Bonnekoh, C. et al.: Int. J. Refract. Met. Hard Mater. 2018;71(71):181–189

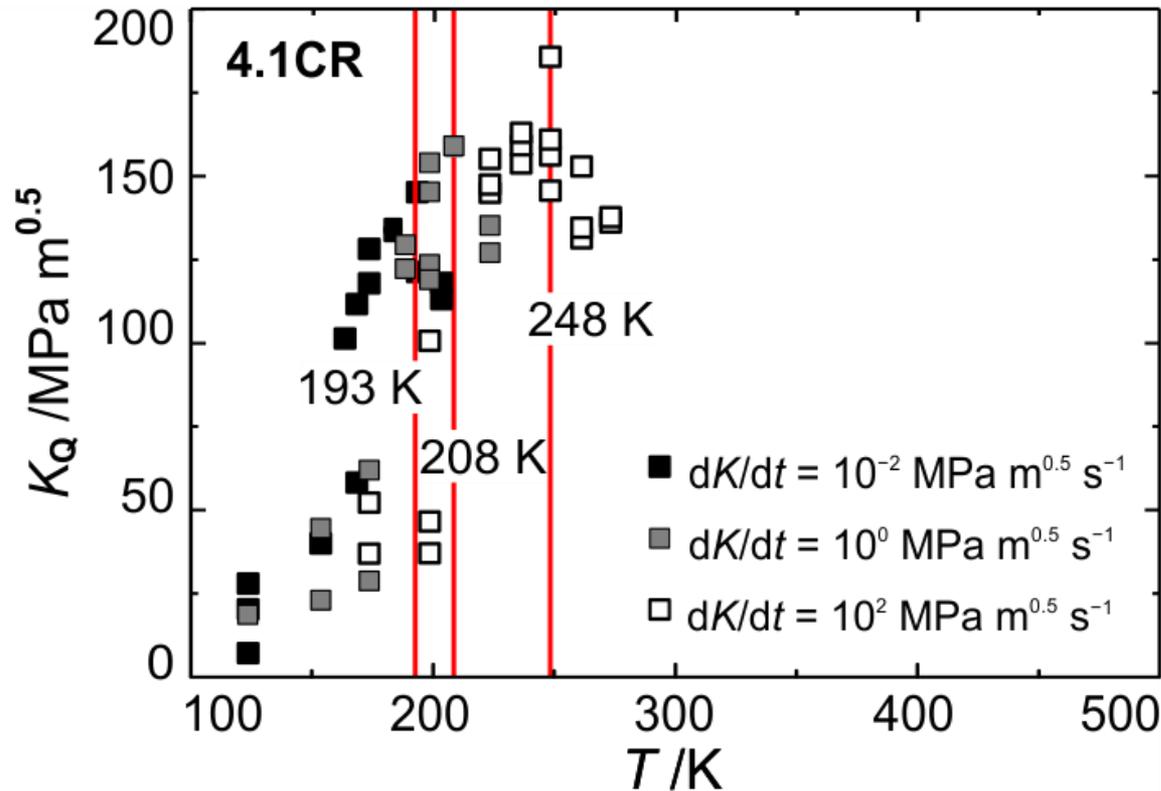
[8] Bonnekoh, C. et al.: Int. J. Refract. Met. Hard Mater. 2019;78(78):146–163

# Results | BDT temperatures



- ❖ **BDT temperature below RT (208 K, -65 °C) achieved by cold-rolling**
- ❖ **Change in BDT controlling mechanism?**

# Results | BDT temperatures



- ❖ All materials exhibit a loading-rate dependence:
  - BDT temperature, i.e.:  $T_{\text{BDT}} = f(dK/dt, \dots)$
- ❖ BDT and crack-tip plasticity have to be **thermal activated**

# Outline

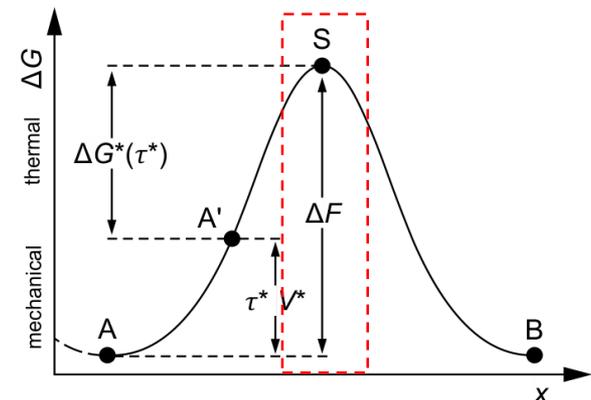
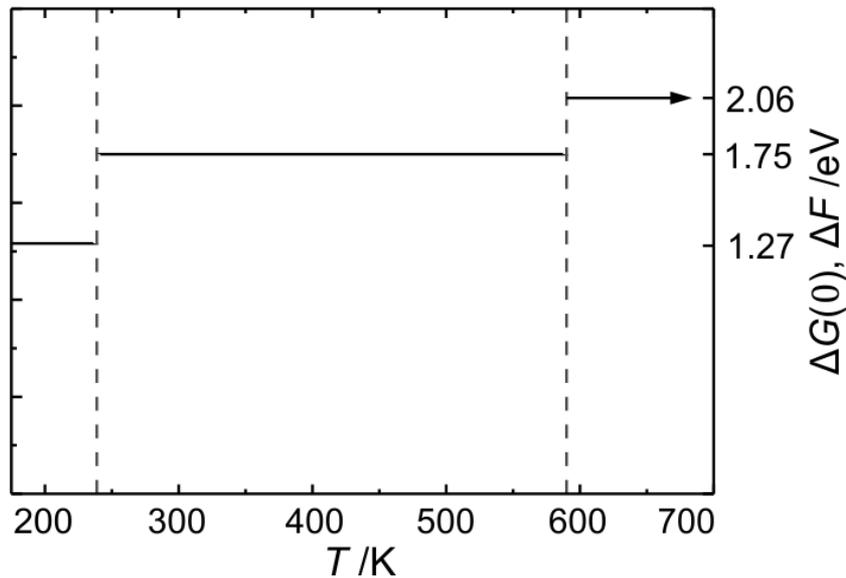
- Motivation
- Methods
- Materials
- **Results**
  - **BDT temperatures**
  - BDT activation energies
  - BDT / microstructure correlations
- Summary

# Outline

- Motivation
- Methods
- Materials
- **Results**
  - BDT temperatures
  - **BDT activation energies**
  - BDT / microstructure correlations
- Summary

# Results | BDT activation energies

- ❖ Helmholtz free energy of activation for **kink-pair formation** ( $\Delta F$ ,  $\Delta G^*(0)$ ) and temperature-dependent critical resolved shear stress ( $\tau^*$ ) available<sup>9–11</sup>
- ❖ Gibb energy of activation  $\Delta G^*(\tau^*)$  mandatory for comparison with  $E_{A(BDT)}$



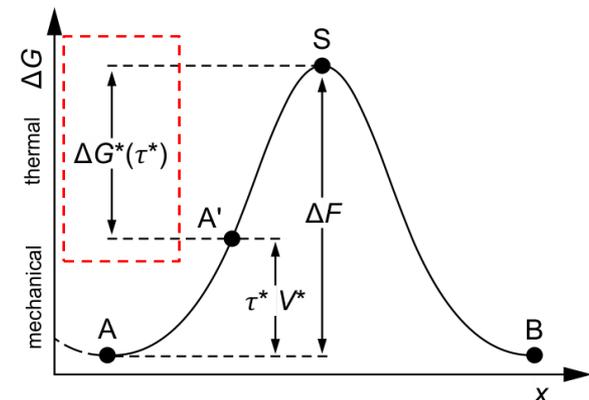
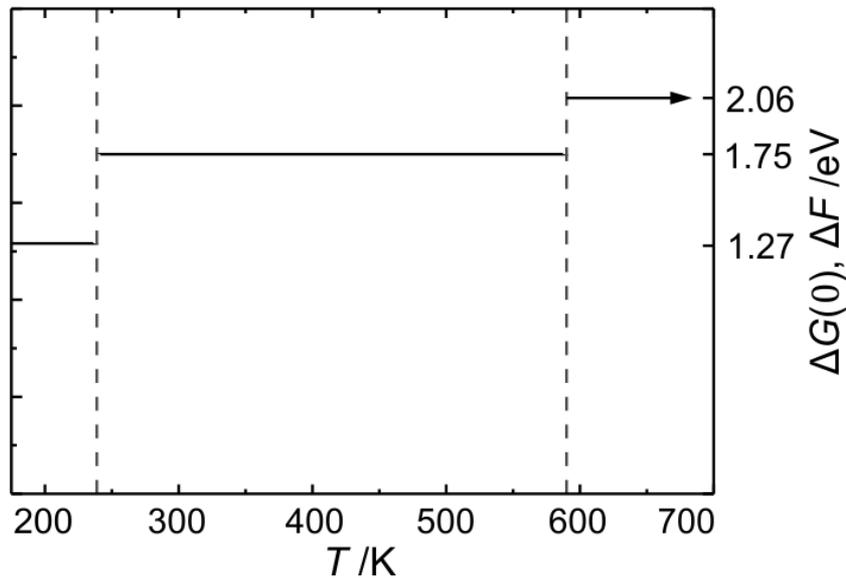
[9] Brunner, D.: Mater. Trans., JIM. 2000;41(1):152–160

[10] Brunner, D. et al.: Mater. Lett. 2000;42(5):290–296

[11] Brunner, D. et al.: Mater. Lett. 2000;44(3–4):144–152

# Results | BDT activation energies

- ❖ Helmholtz free energy of activation for **kink-pair formation** ( $\Delta F$ ,  $\Delta G^*(0)$ ) and temperature-dependent critical resolved shear stress ( $\tau^*$ ) available<sup>9–11</sup>
- ❖ Gibb energy of activation  $\Delta G^*(\tau^*)$  mandatory for comparison with  $E_{A(BDT)}$



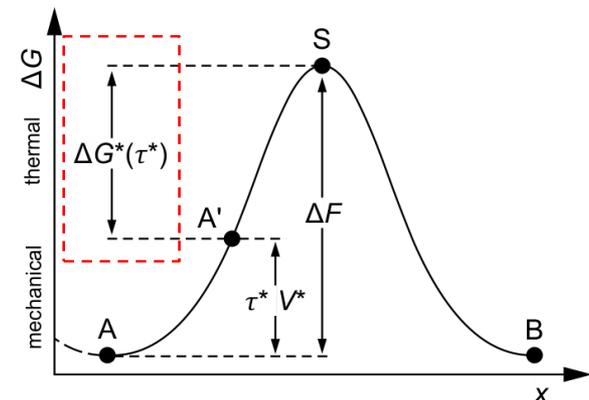
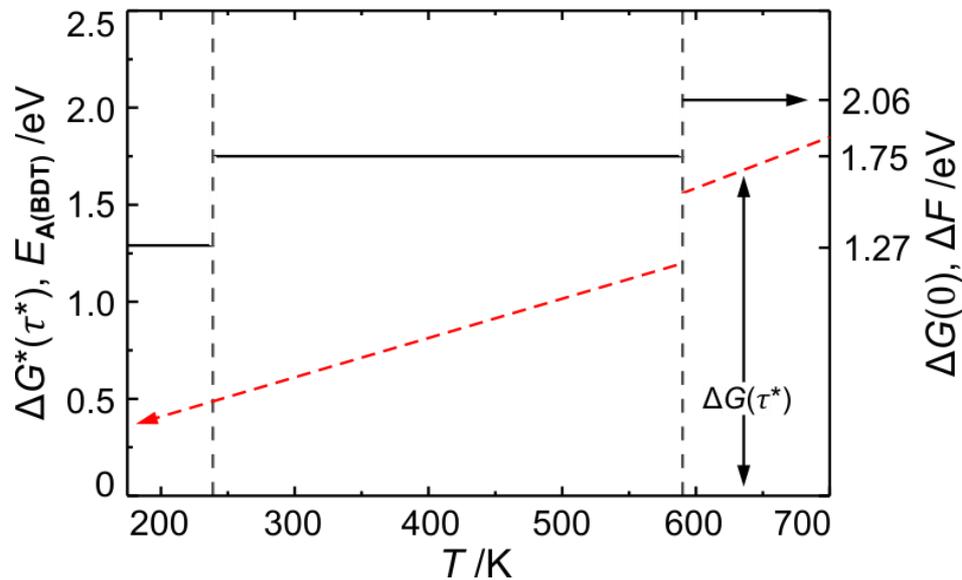
[9] Brunner, D.: Mater. Trans., JIM. 2000;41(1):152–160

[10] Brunner, D. et al.: Mater. Lett. 2000;42(5):290–296

[11] Brunner, D. et al.: Mater. Lett. 2000;44(3–4):144–152

# Results | BDT activation energies

- ❖ Helmholtz free energy of activation for **kink-pair formation** ( $\Delta F$ ,  $\Delta G^*(0)$ ) and temperature-dependent critical resolved shear stress ( $\tau^*$ ) available<sup>9–11</sup>
- ❖ Gibb energy of activation  $\Delta G^*(\tau^*)$  mandatory for comparison with  $E_{A(BDT)}$



[9] Brunner, D.: Mater. Trans., JIM. 2000;41(1):152–160

[10] Brunner, D. et al.: Mater. Lett. 2000;42(5):290–296

[11] Brunner, D. et al.: Mater. Lett. 2000;44(3–4):144–152

# Outline

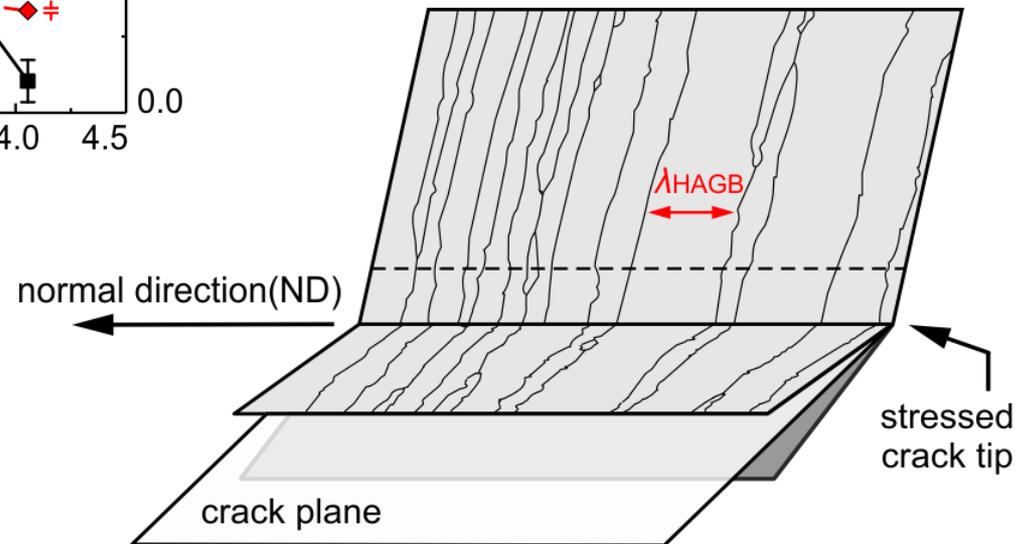
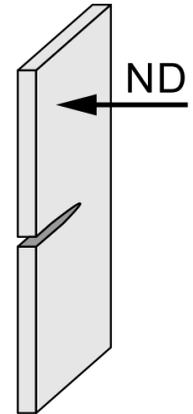
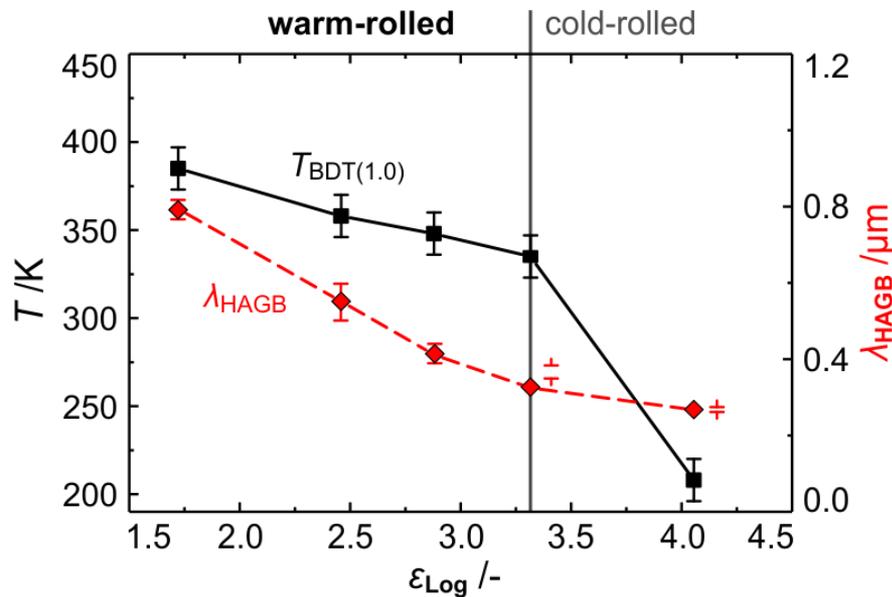
- Motivation
- Methods
- Materials
- **Results**
  - BDT temperatures
  - **BDT activation energies**
  - BDT / microstructure correlations
- Summary

# Outline

- Motivation
- Methods
- Materials
- **Results**
  - BDT temperatures
  - BDT activation energies
  - **BDT / microstructure correlations**
- Summary

# Results | BDT / microstructure correlations

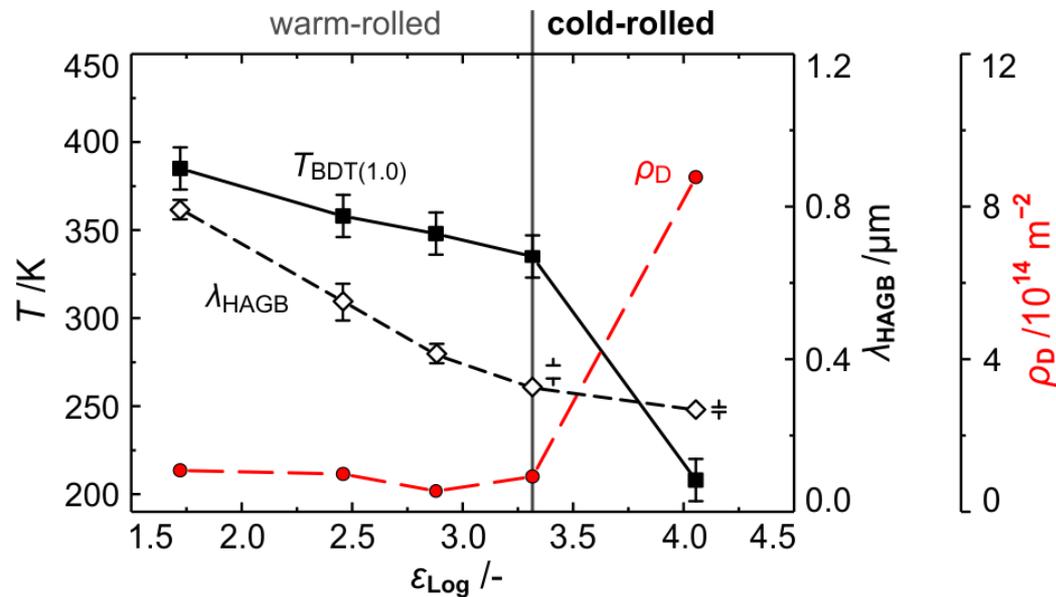
❖ Key properties: grain size in ND ( $\lambda_{HAGB}$ ), dislocation density ( $\rho_D$ )



[7] Bonnekoh, C. et al.: Int. J. Refract. Met. Hard Mater. 2018;71(71):181-189  
 [8] Bonnekoh, C. et al.: Int. J. Refract. Met. Hard Mater. 2019;78(78):146-163

# Results | BDT / microstructure correlations

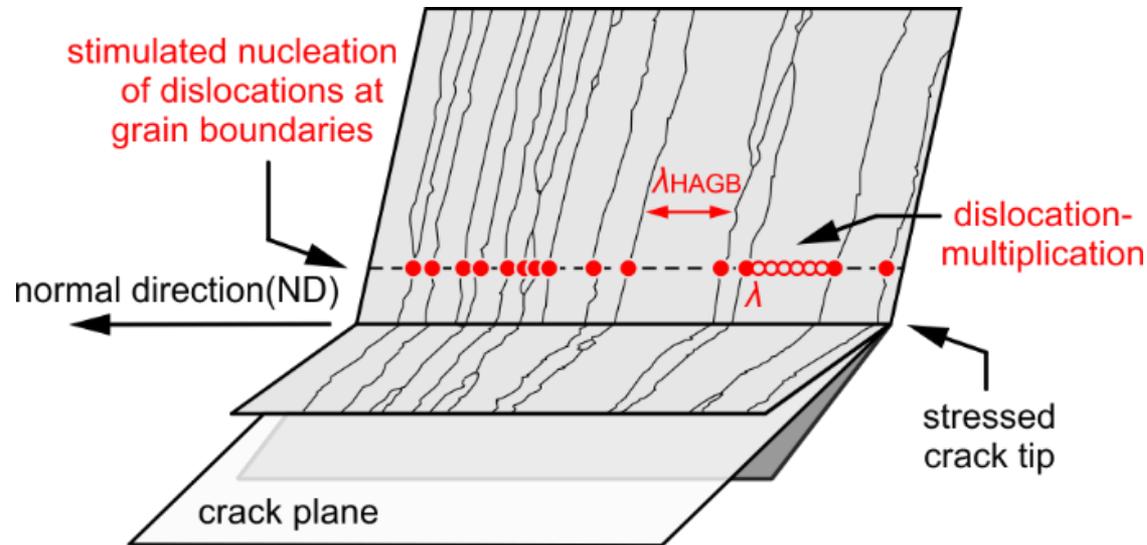
- ❖ Key properties: grain size in ND ( $\lambda_{\text{HAGB}}$ ), dislocation density ( $\rho_{\text{D}}$ )



[8] Bonnekoh, C. et al.: Int. J. Refract. Met. Hard Mater. 2019;78(78):146–163

# Results | BDT / microstructure correlations

- ❖ Key properties: grain size in ND ( $\lambda_{\text{HAGB}}$ ), dislocation density ( $\rho_{\text{D}}$ )



- ❖ Mean spacing between sites of **dislocation nucleation ( $\lambda$ )** controls the **BDT temperature**
  - Spacing of primary nucleation sites: grain size in ND ( $\lambda_{\text{HAGB}}$ )
  - Spacing of secondary nucleation sites: dislocation density ( $\rho_{\text{D}}^{-0.5}$ )

# Outline

- Motivation
- Methods
- Materials
- **Results**
  - BDT temperatures
  - BDT activation energies
  - **BDT / microstructure correlations**
- Summary

# Outline

- Motivation
- Methods
- Materials
- Results
- **Summary**

# Summary

- ❖ Five W sheets have been rolled out from a single hot-rolled plate by a **industrial-scale production** process
- ❖ **Cold-rolling** shifts BDT temperature to 208 K ( $-65\text{ °C}$ ) and **causes room temperature ductility**
- ❖ **Glide of screw dislocations** still **governs crack-tip plasticity** even below room temperature
- ❖ **Spacing of nucleation sites** along the crack front **controls BDT temperature**
- ❖ Room temperature ductility in combination with a easily to scale-up production process opens a new era in the application of W as a powerful structural material

# Thank you for your attention

## The authors are grateful to:

DFG, Grant RE3551/4-1

Plansee SE

Max-Planck-Institut für Eisenforschung GmbH



## Results published in:

Bonnekoh, C. et al.: *The brittle-to-ductile transition in cold rolled tungsten: On the decrease of the brittle-to-ductile transition by 600 K to -65 °C.* Int. J. Refract. Met. Hard Mater. 2018;(71):181–189.

Bonnekoh, C. et al.: *The brittle-to-ductile transition in cold rolled tungsten plates: Impact of crystallographic texture, grain size and dislocation density on the transition temperature.* Int. J. Refract. Met. Hard Mater. 2019;(78C):146–163.