



Manufacturing and Characterization of PIM-W Materials as PFMs

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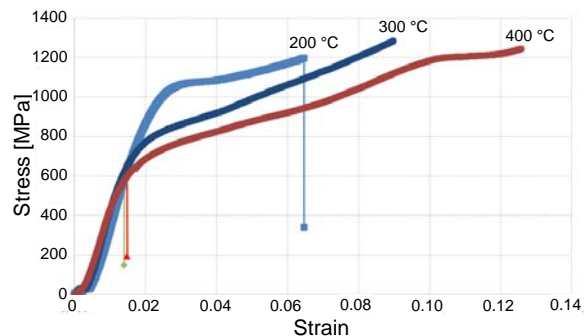
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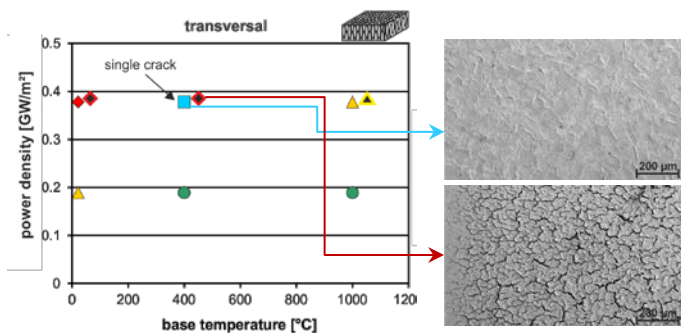
This work has been carried out within the framework of the EUROfusion Consortium and has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement number 633053. The views and opinions expressed herein do not necessarily reflect those of the European Commission.

❖ DEMO divertor development within the EUROfusion program WPMAT



❖ **Pure and particle strengthened materials** interesting characteristics: *strength, fracture toughness, ductility, recrystallization resistance, and sufficiently high thermal conductivity*

❖ **PIM: near net shape technology**, materials with *isotropic microstructure and properties*; fast screening of different compositions



❖ **Qualification via e-beam testing** (standardized method) *vs. a reference W-grade*

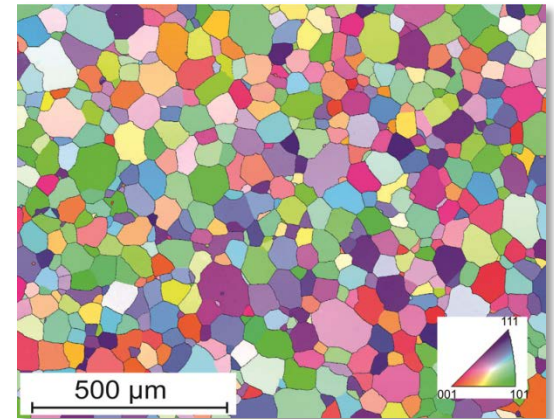
Mass production of components



Time & cost effective
near-net-shape forming process

Shape complexity & high final density

Materials development



Tailoring new materials
&
Investigation of properties

Mass production of tungsten parts

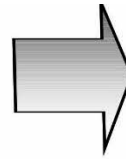


Powder



Binder

Mixing /
Kneading /
Extrusion



Feedstock

Mass production of tungsten parts



Powder

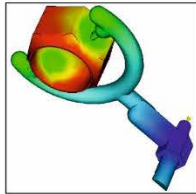


Binder

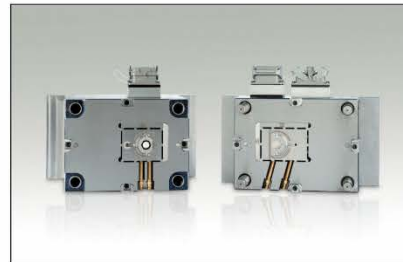
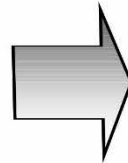
Mixing /
Kneading /
Extrusion



Feedstock



Filling simulation



PIM-tool

Mass production of tungsten parts

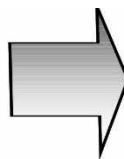


Powder

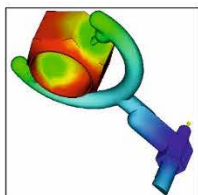


Binder

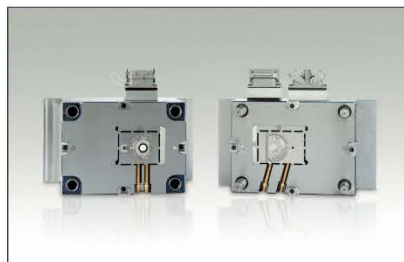
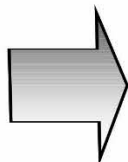
Mixing /
Kneading /
Extrusion



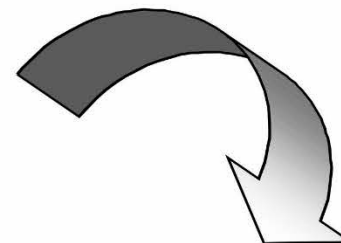
Feedstock



Filling simulation

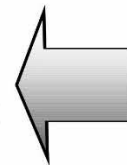
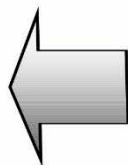


PIM-tool



Green parts (dark), finished parts (bright)

Debinding /
Heat-treatment

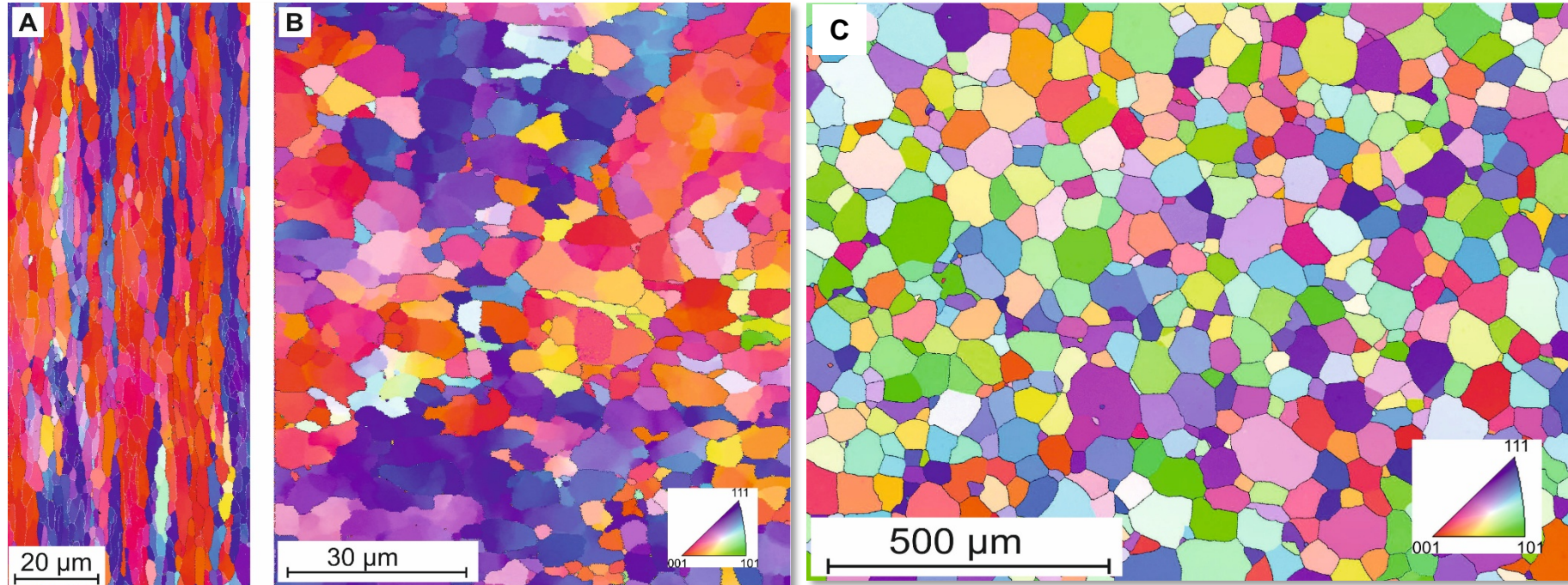


Injection molding of green parts



Texture analysis: EBSD

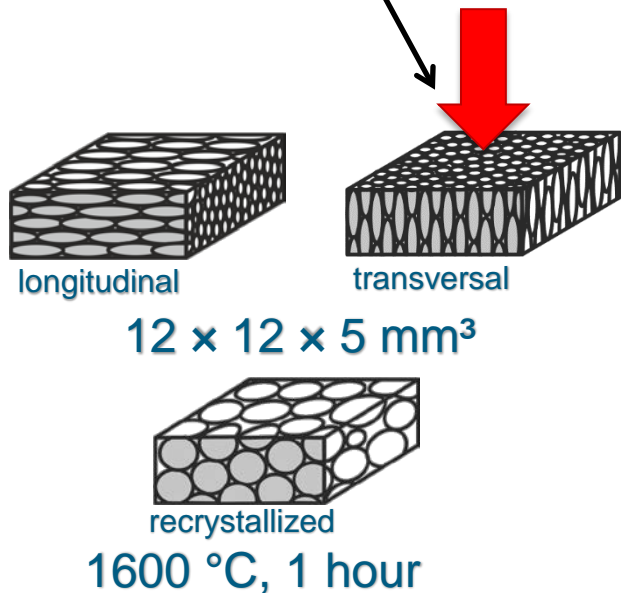
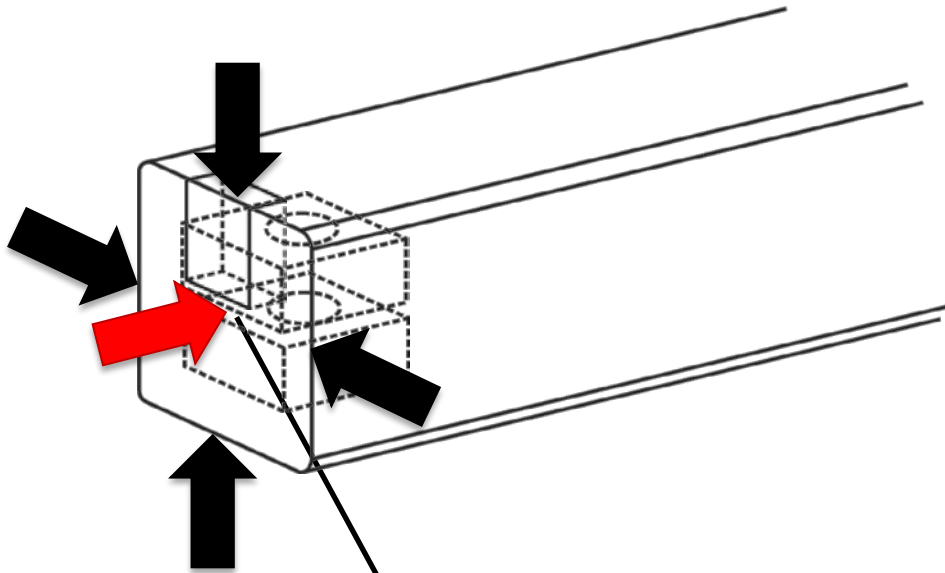
Rolled W-plate (Plansee AG) vs. PIM-W



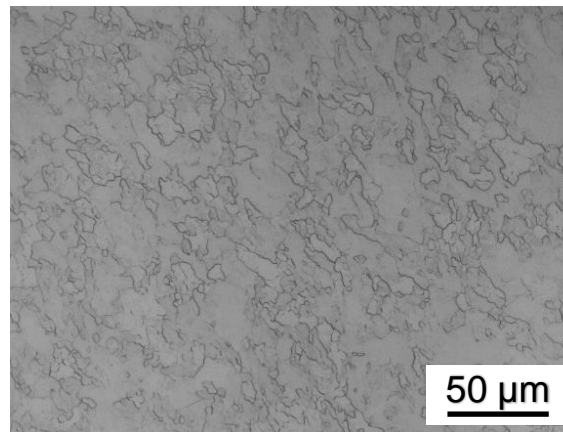
Rolled-W: [A] *in rolling direction – e.g. high strength and bending toughness*
[B] *perpendicular to rolling direction*

PIM-W: [C] *fully isotropic*

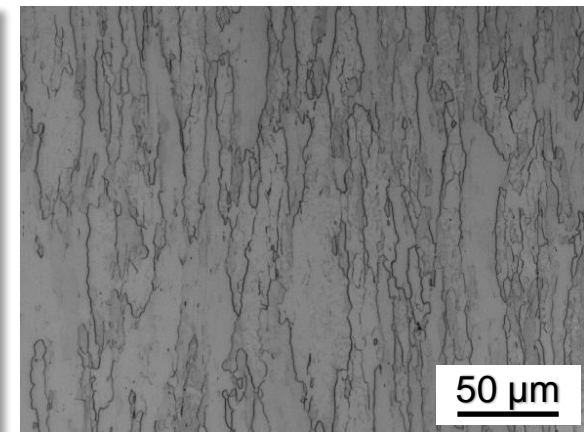
Reference W according to ITER specs („IGP“)



Top view, $\phi \sim 10 \mu\text{m}$



Side view, $5 \times 25 \mu\text{m}^2$





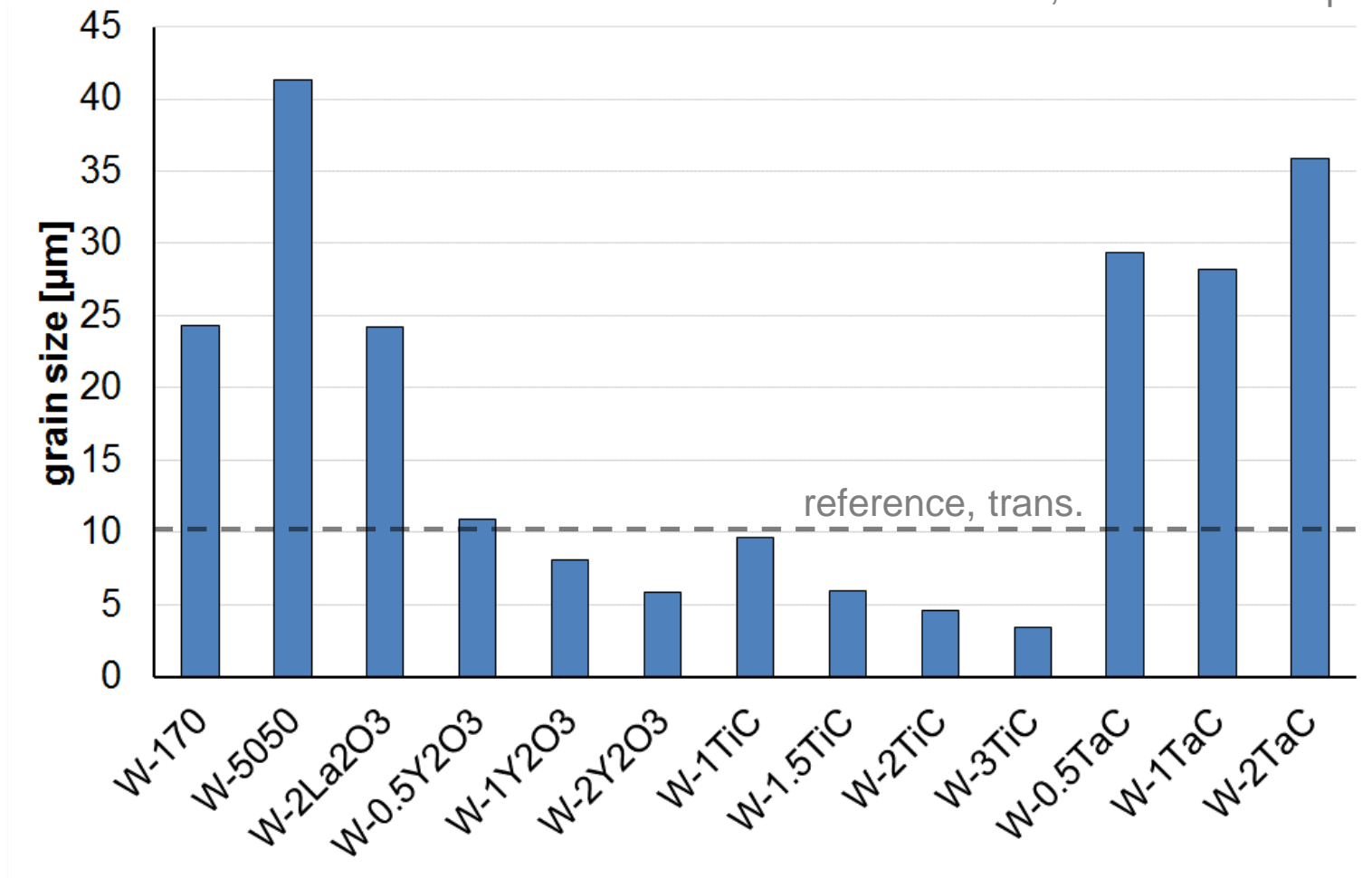
Manufacturing according to shown PIM-process; final sintering at **2400 °C**; **particle sizes < 1 μm**

- ❖ **pure W**: 2 grades with different powder sizes (*W-170*, *W-5050*)
- ❖ **W-2La₂O₃**: initial composition; vaporization of La₂O₃ during sintering ⇒ pure W with “clean” grain boundaries
- ❖ **W-Y₂O₃**: 3 grades with different Y₂O₃ content (*W-0.5Y₂O₃*, *W-1Y₂O₃*, *W-2Y₂O₃*)
- ❖ **W-TiC**: 4 grades with different TiC content (*W-1TiC*, *W-1.5TiC*, *W-2TiC*, *W-3TiC*)
- ❖ **W-TaC**: 3 grades with different TaC content (*W-0.5TaC*, *W-1TaC*, *W-2TaC*)

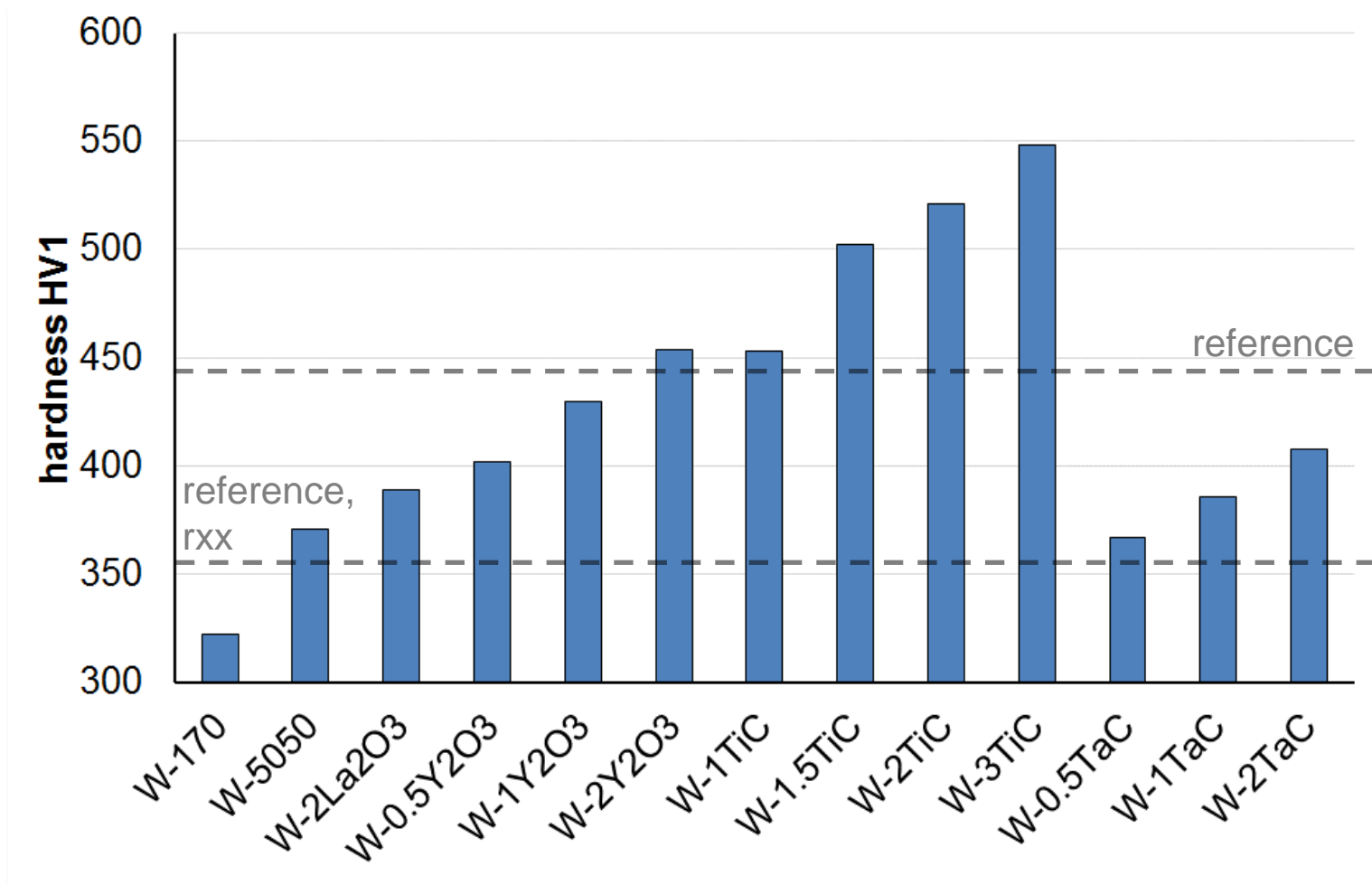
Materials grain size



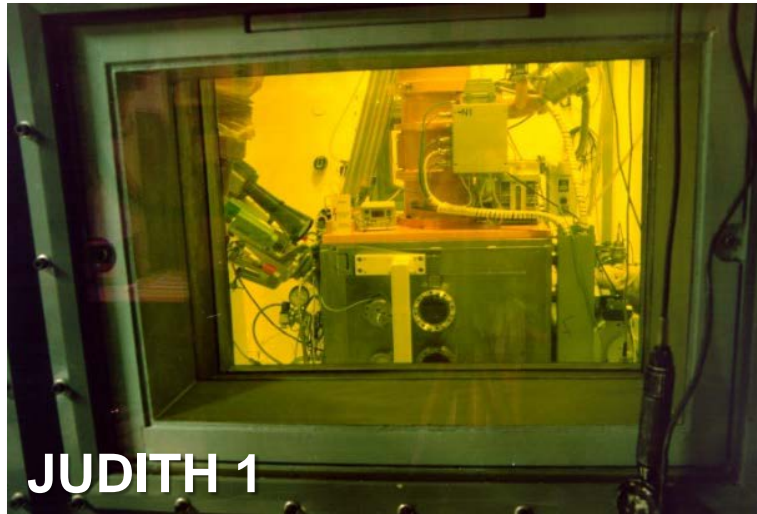
reference, long.: 10 x 25 μm
reference, rxx: 64 x 102 μm



❖ Optimization potential by variation of raw materials (powder sizes)



❖ Pure PIM-W similar to recrystallized W; hardness increase only for W-TiC



JUDITH 1

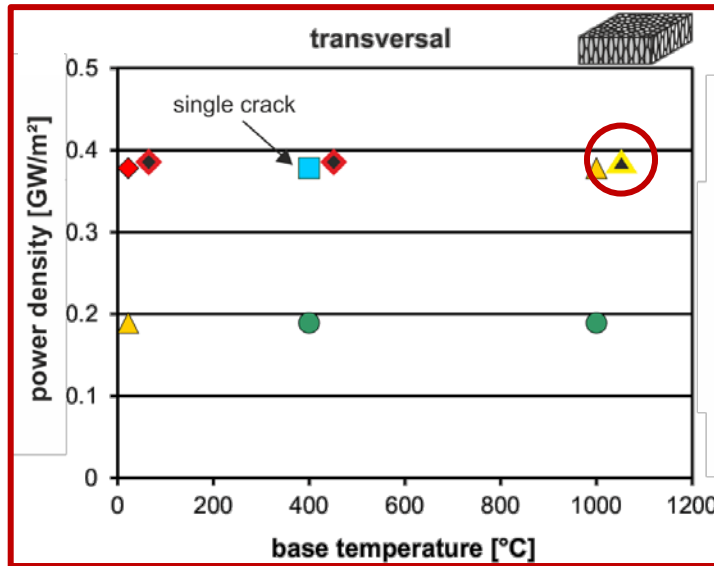
E-beam parameters

<i>acceleration voltage:</i>	120 - 150 kV
<i>beam power:</i>	≤ 60 kW
<i>pulse length:</i>	1 ms – cont.
<i>beam diameter:</i>	1 mm
<i>scanning frequency:</i>	≤ 100 kHz

Test parameters

<i>power density:</i>	0.19-0.38 GW/m²
<i>pulse length:</i>	1 ms
<i>pulse number:</i>	100, 1000
<i>base temperature:</i>	RT, 400 °C, 1000 °C

Reference W according to ITER specs („IGP“)



n = 100

n = 1000

● no damage

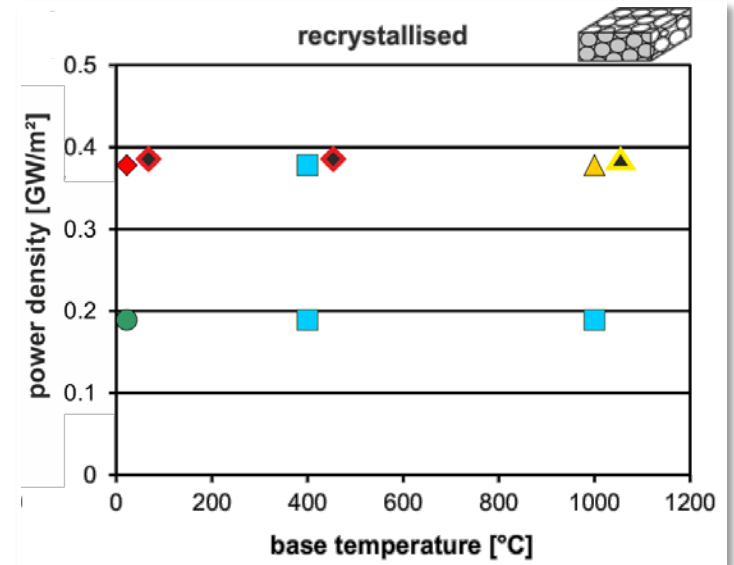
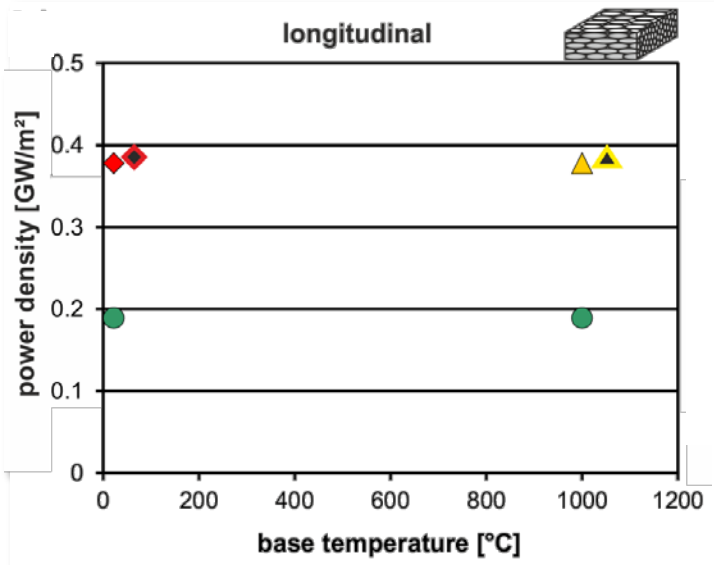
■ surface modification

◆ crack network

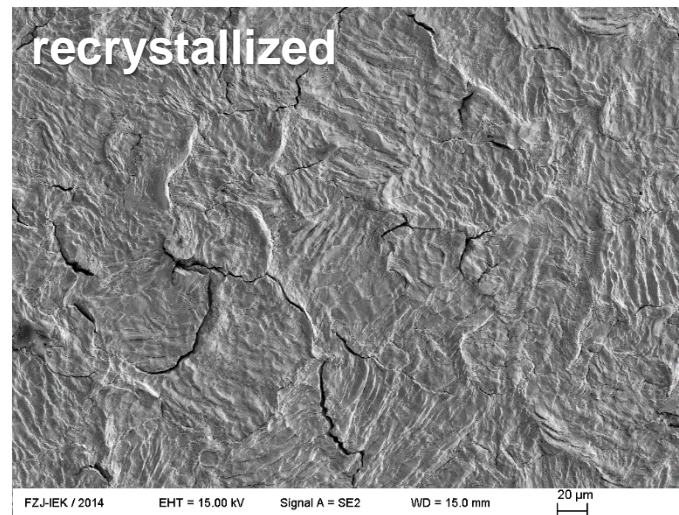
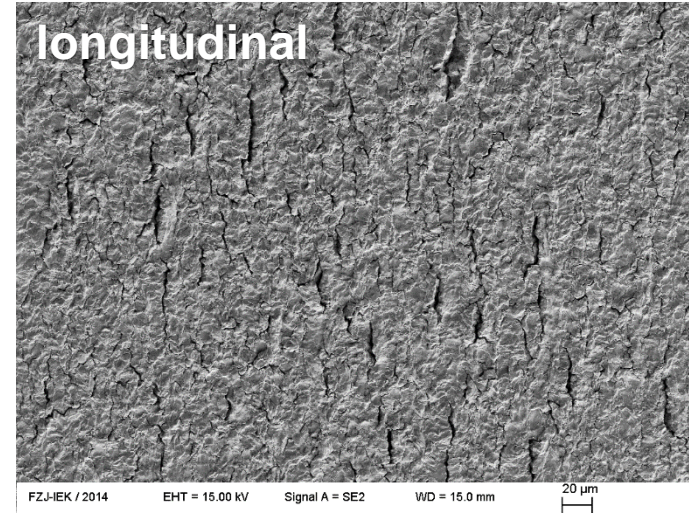
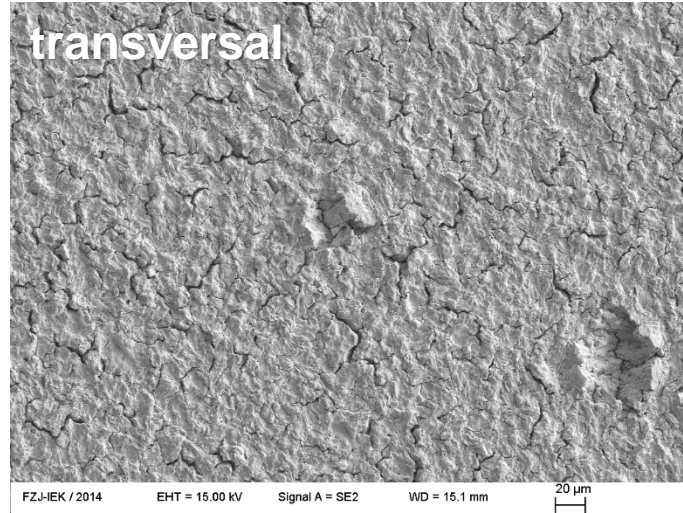
▲ small cracks

◆ crack network

▲ small cracks

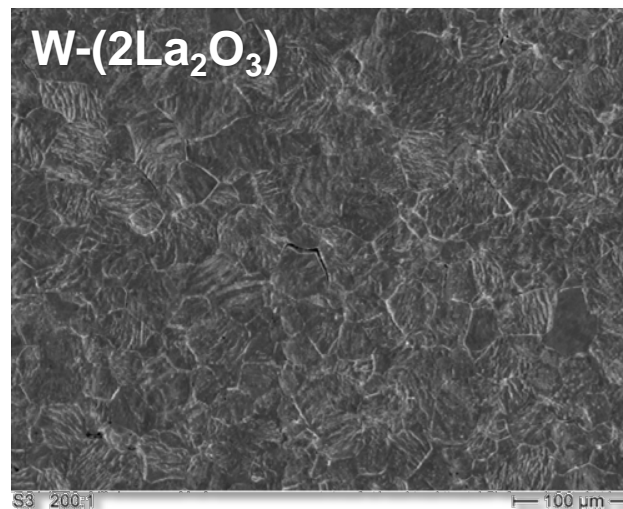
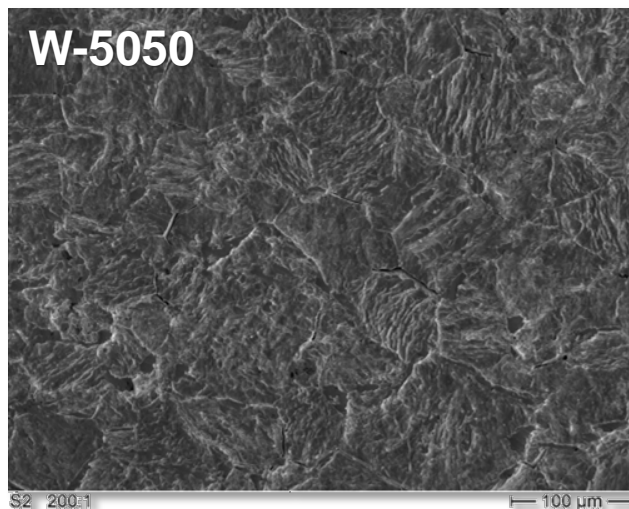
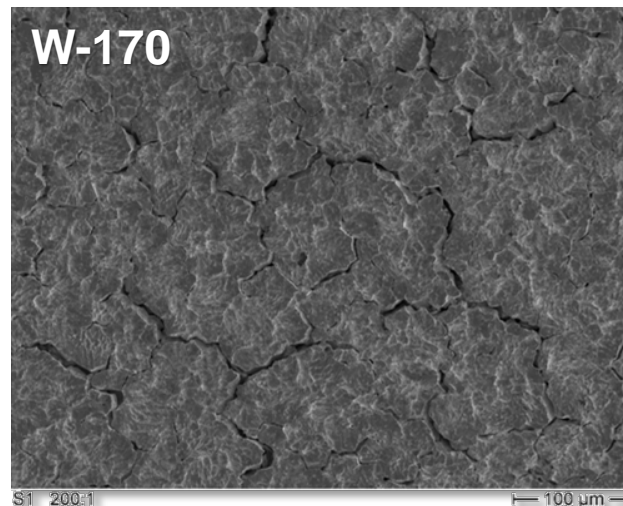
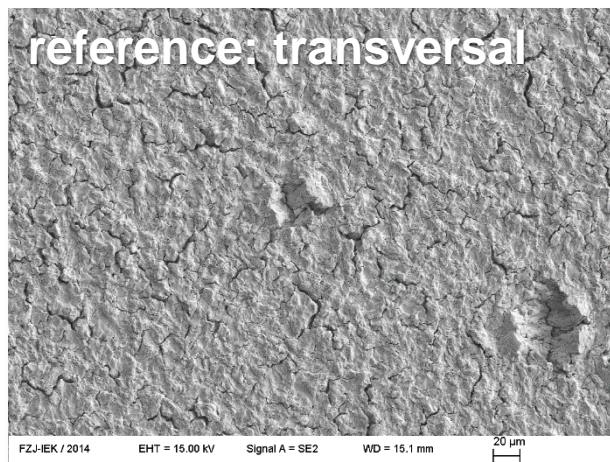


Thermal shock response: reference W



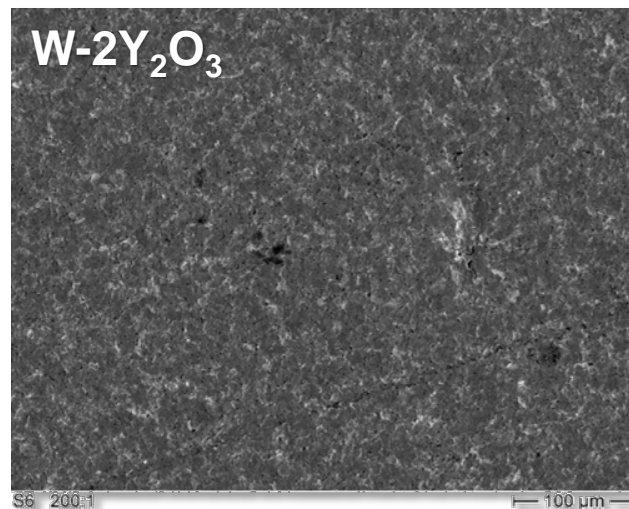
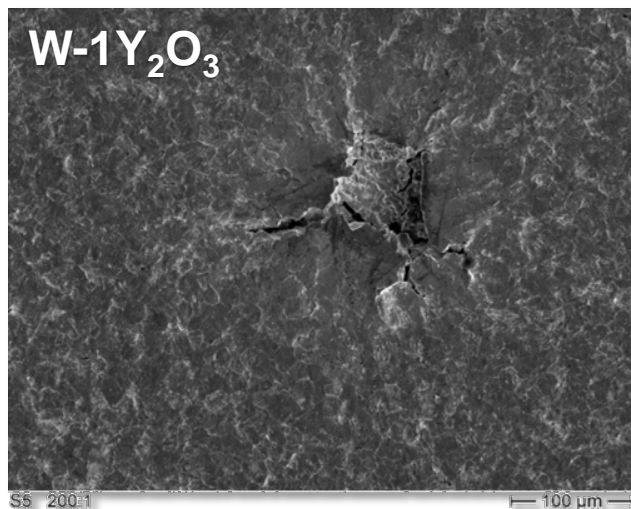
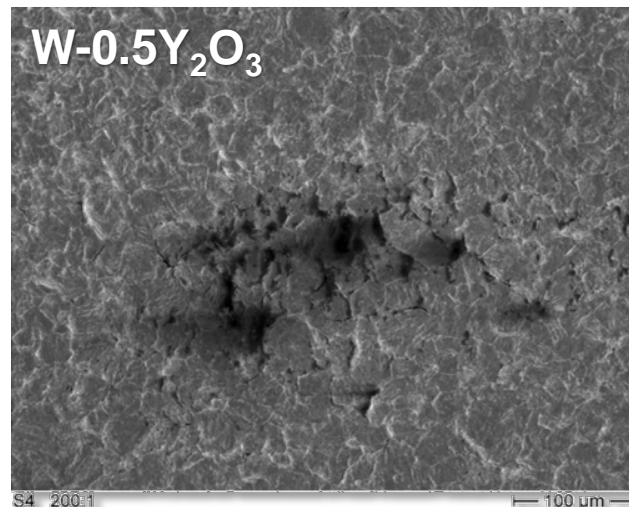
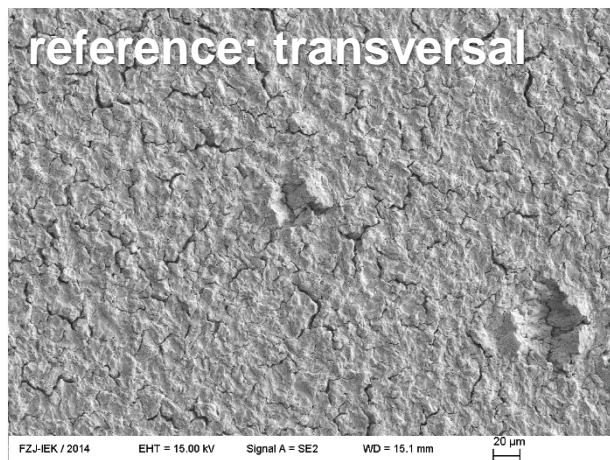
- ❖ small crack formation
- ❖ grain loss for transversal orientation
- ❖ significant surface roughening for recryst. material

ELM-test at elevated temperature



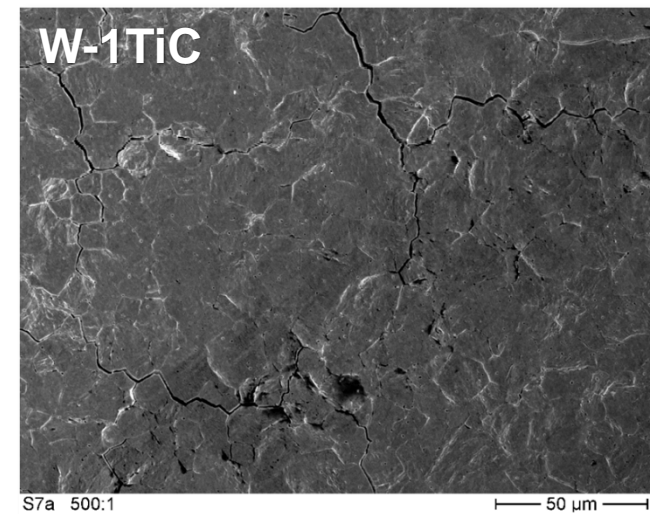
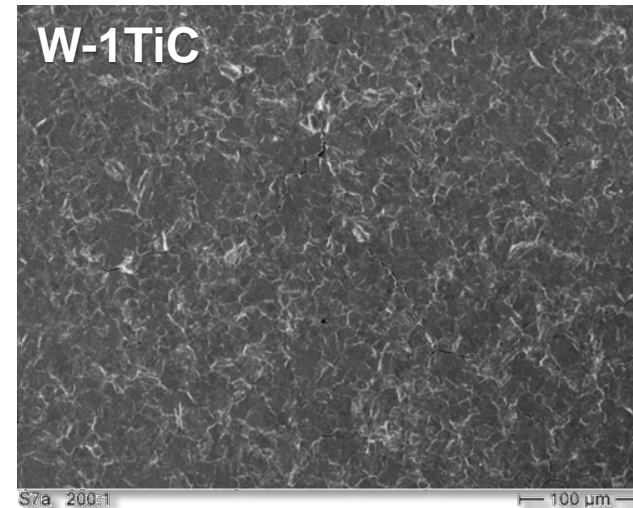
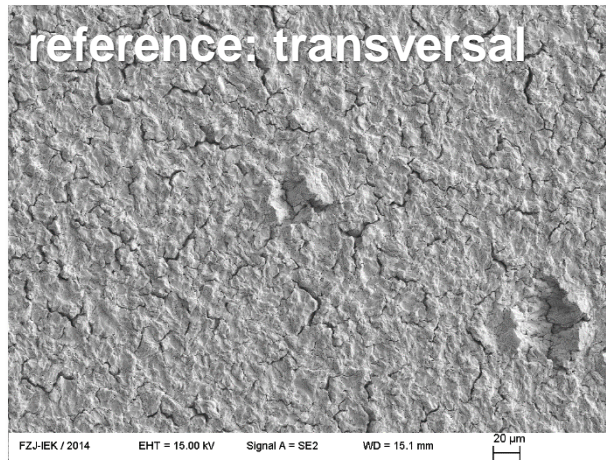
- ❖ Significant surface roughening – similar to recrystallized material!
- ❖ Reduced crack formation compared to reference

ELM-test at elevated temperature



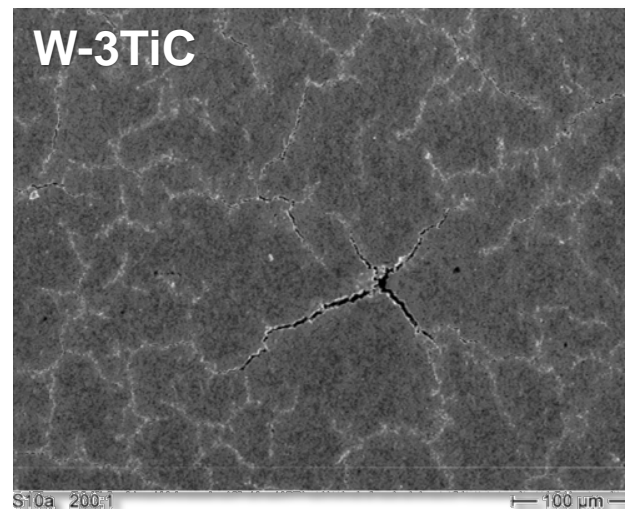
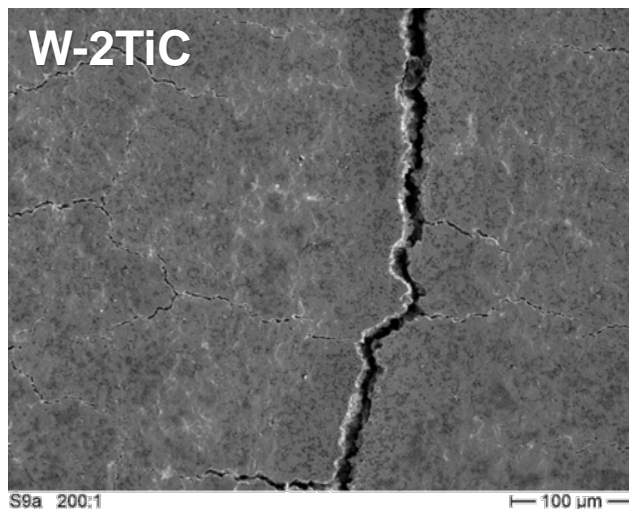
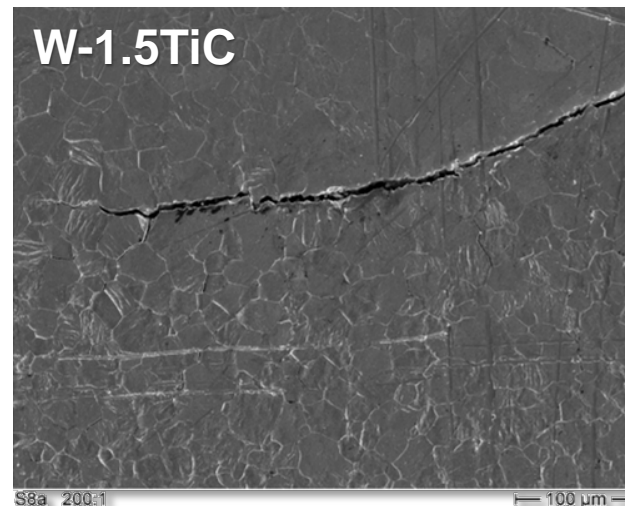
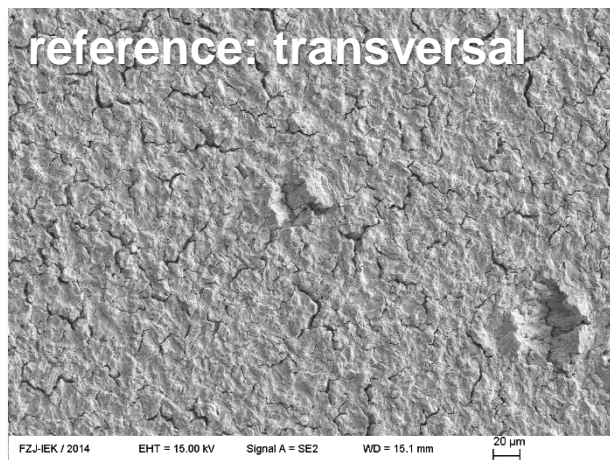
- ❖ Local μ -cracks due to inhomogeneities
- ❖ Reduced crack formation compared to reference

ELM-test at elevated temperature



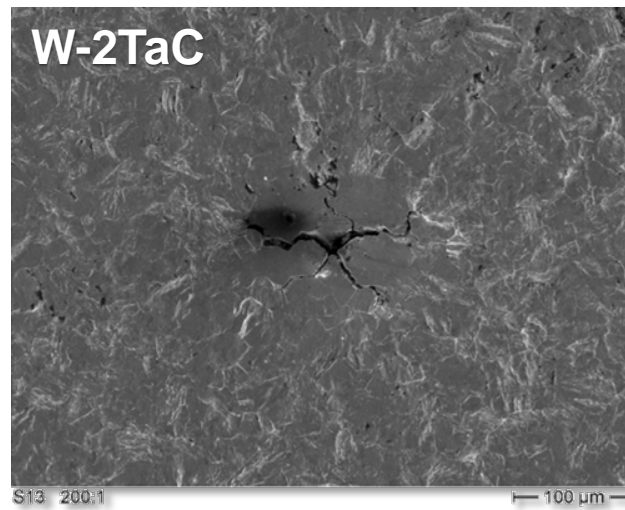
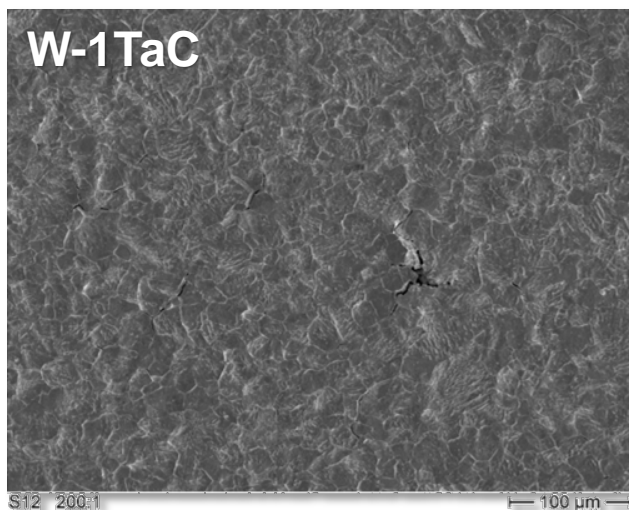
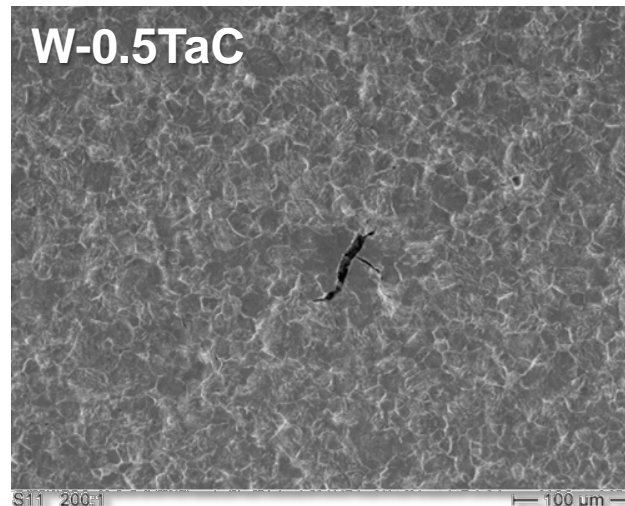
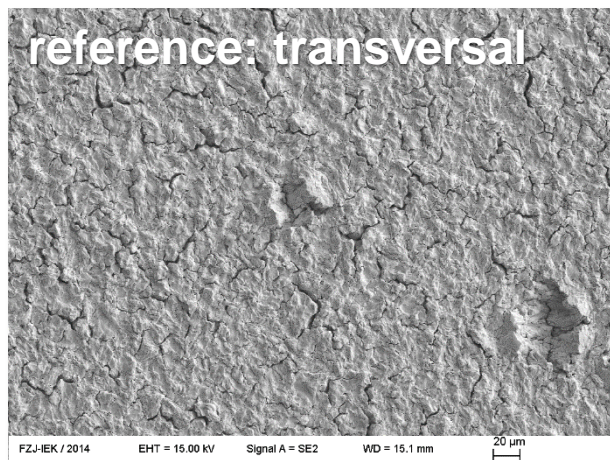
- ❖ Low surface roughening
- ❖ Crack formation similar to reference

ELM-test at elevated temperature



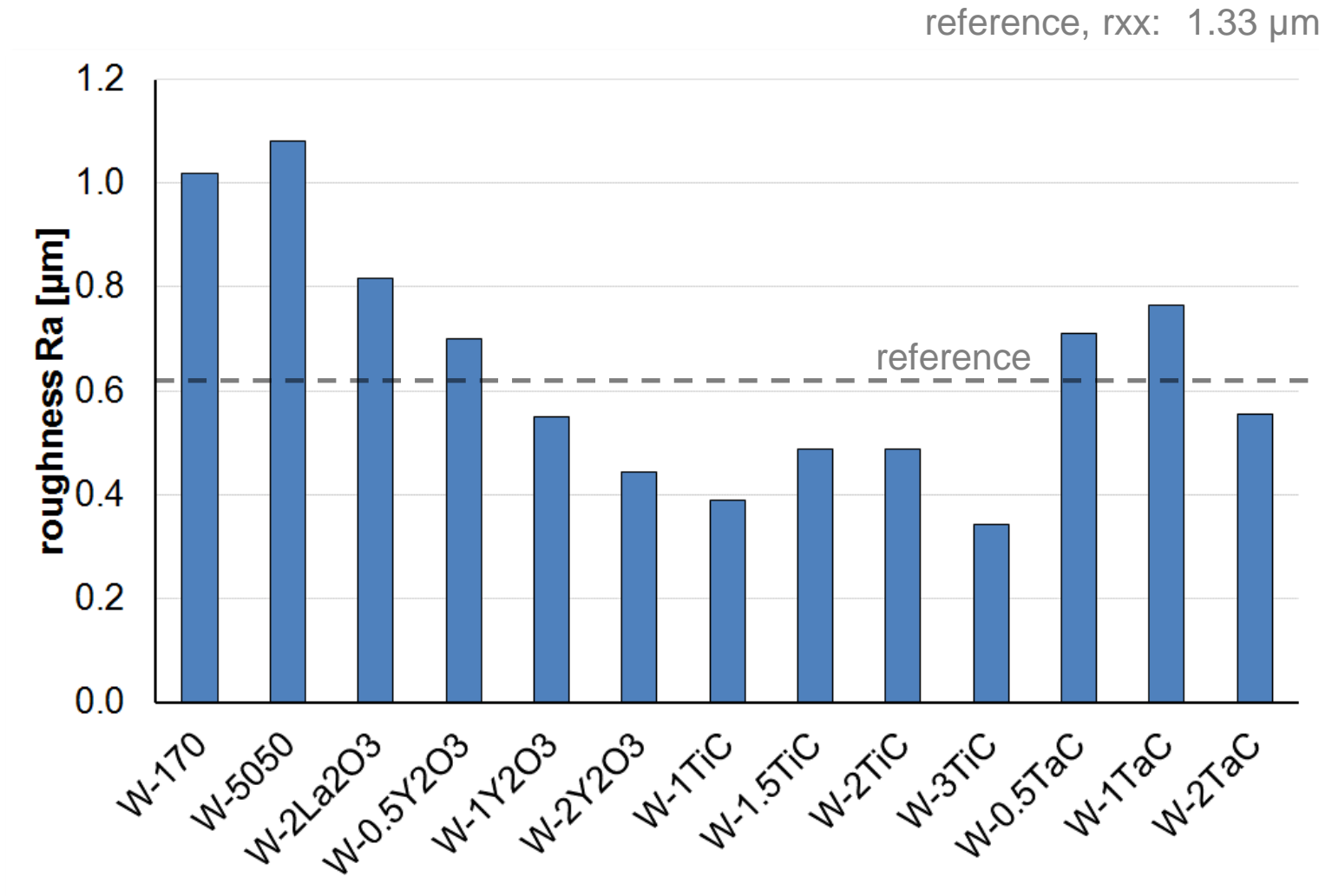
- ❖ Large crack formation
- ❖ No improvement compared to reference

ELM-test at elevated temperature



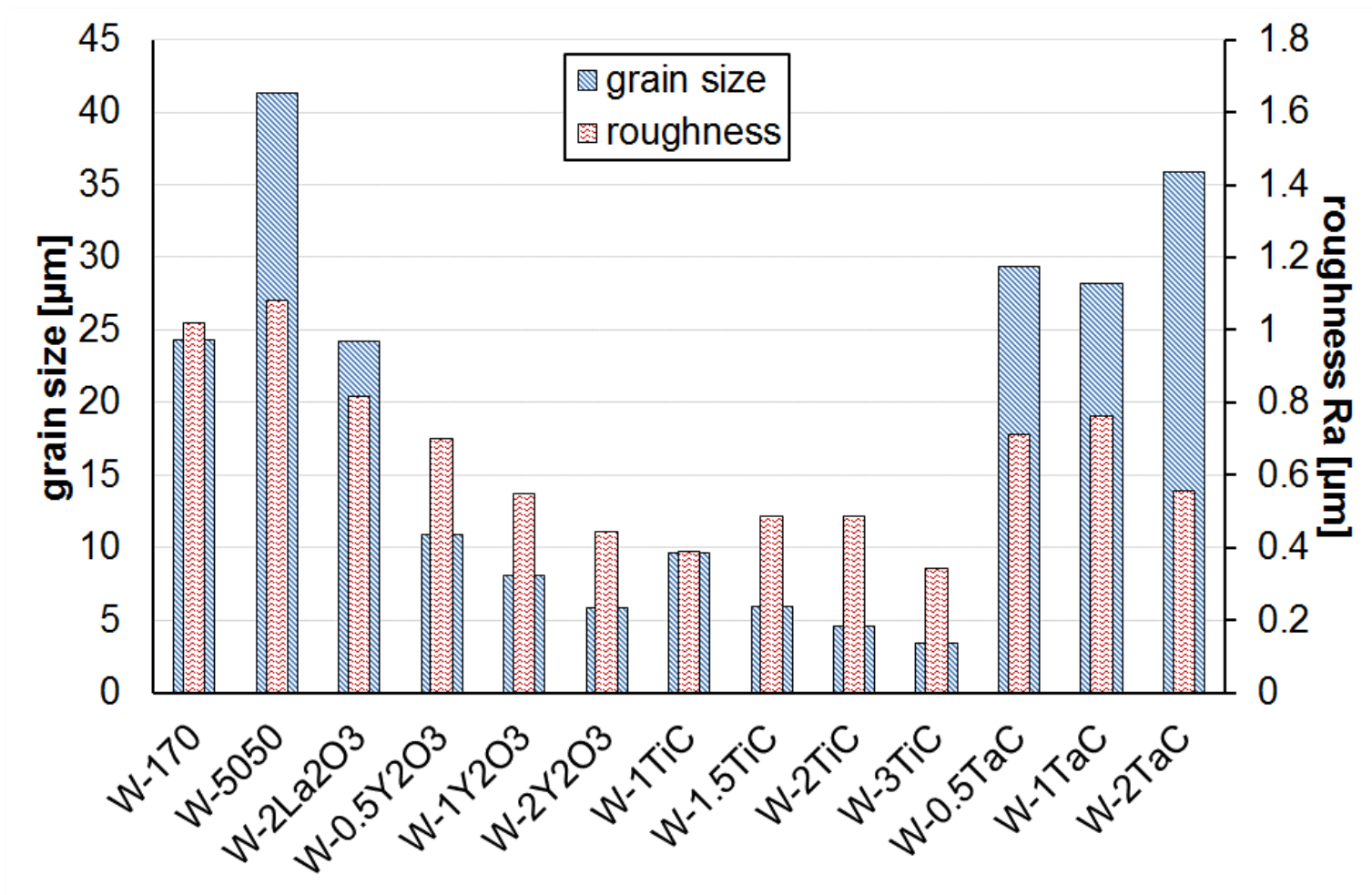
- ❖ Local μ -cracks due to inhomogeneities.
- ❖ Reduced crack formation compared to reference

ELM-test at elevated temperature



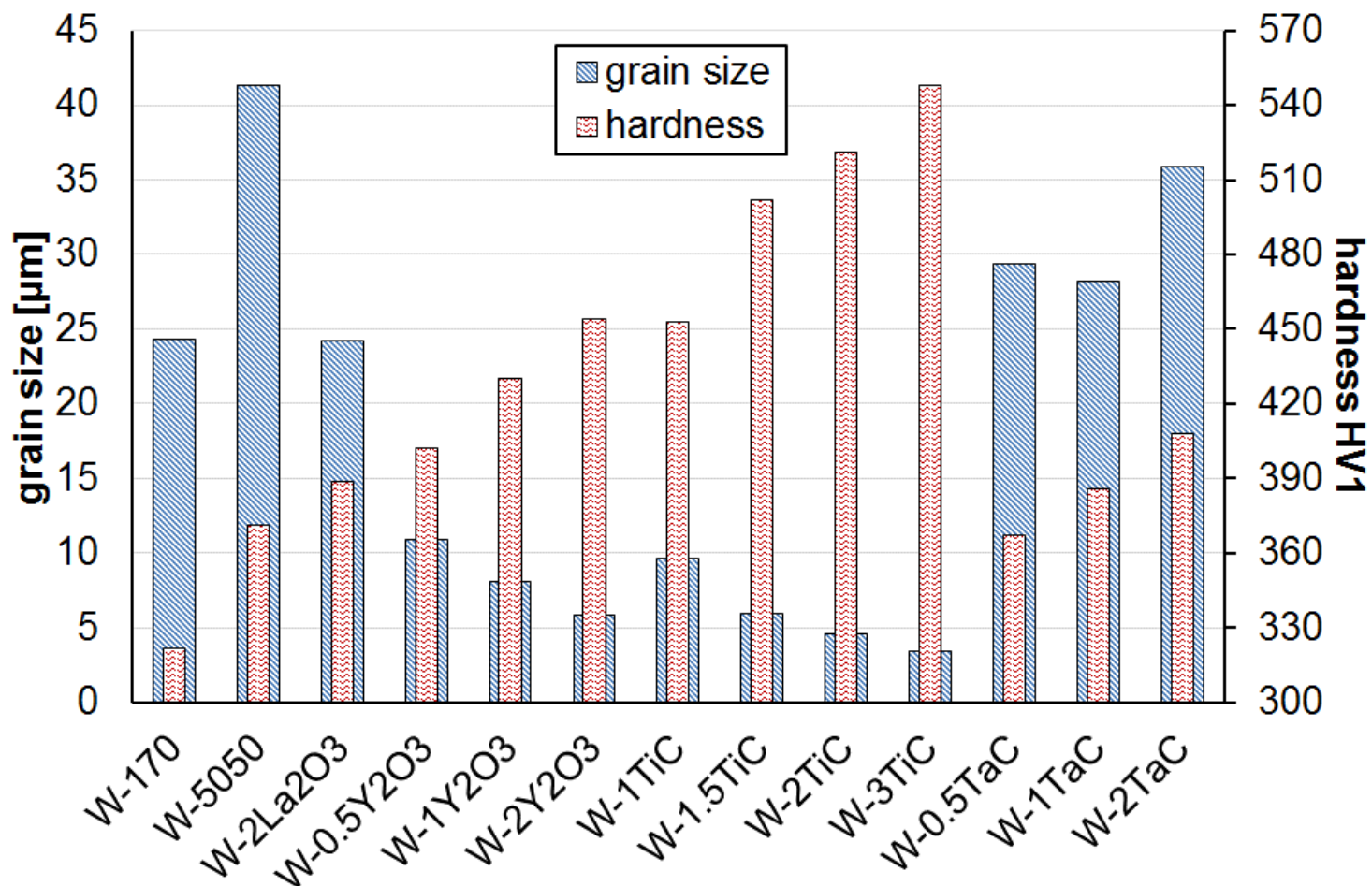
❖ Reduced roughening / plastic deformation for W-Y₂O₃ and W-TiC materials

Grain size vs. roughness



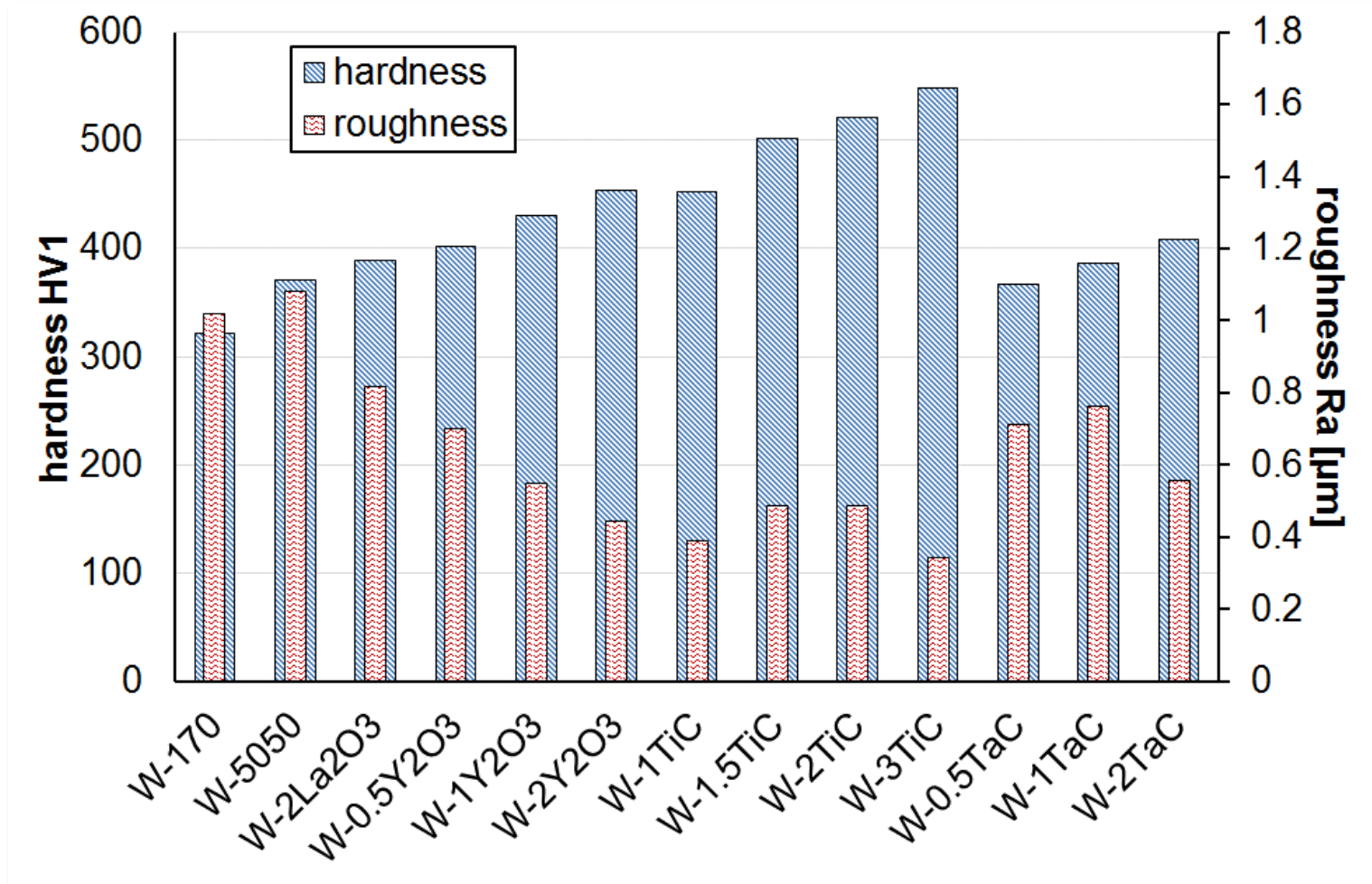
❖ Less roughening with decreasing grain size

Grain size vs. hardness



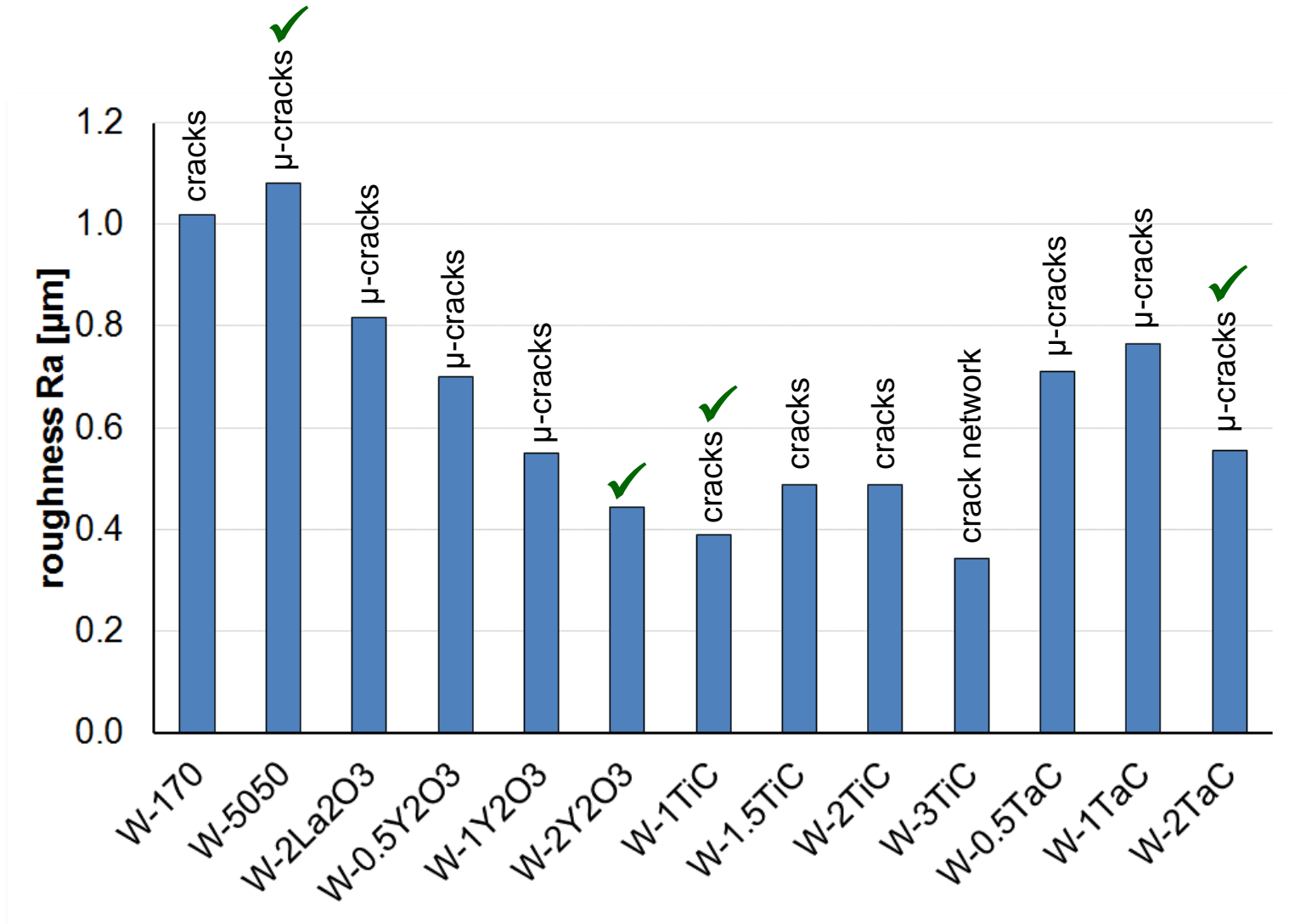
❖ Increased hardness with decreasing grain size

Hardness vs. roughness



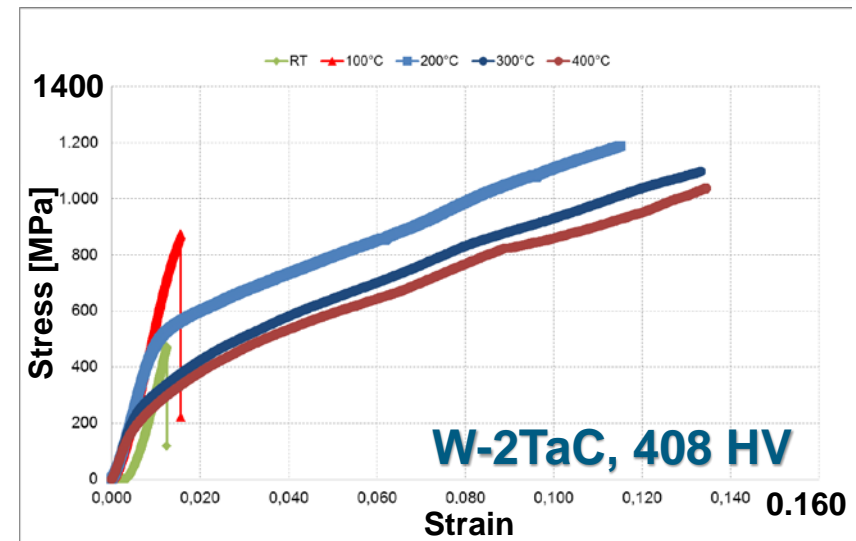
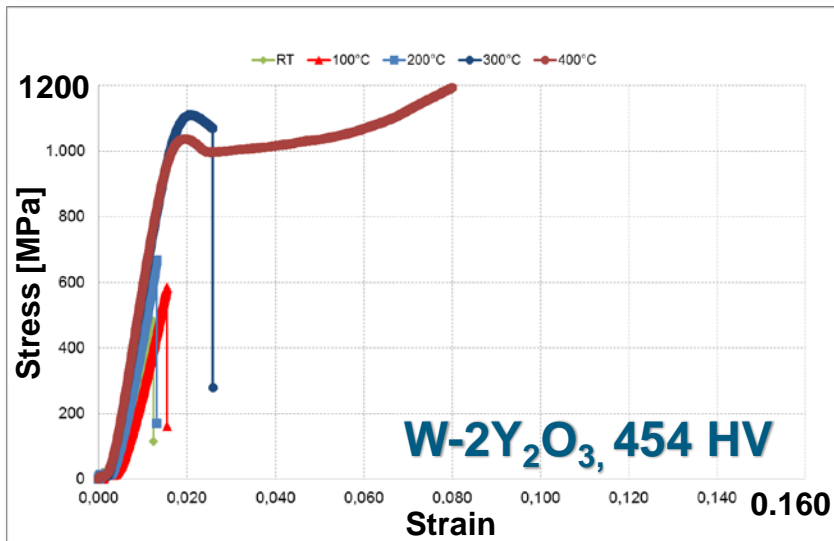
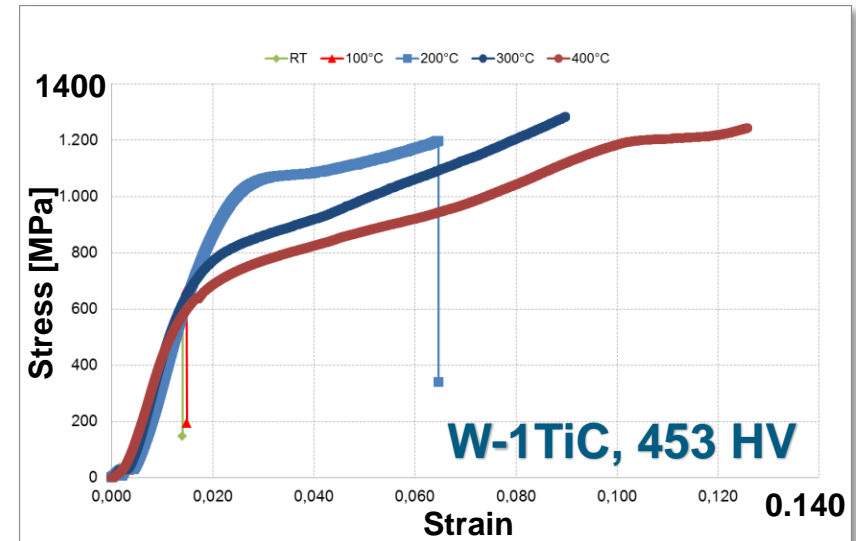
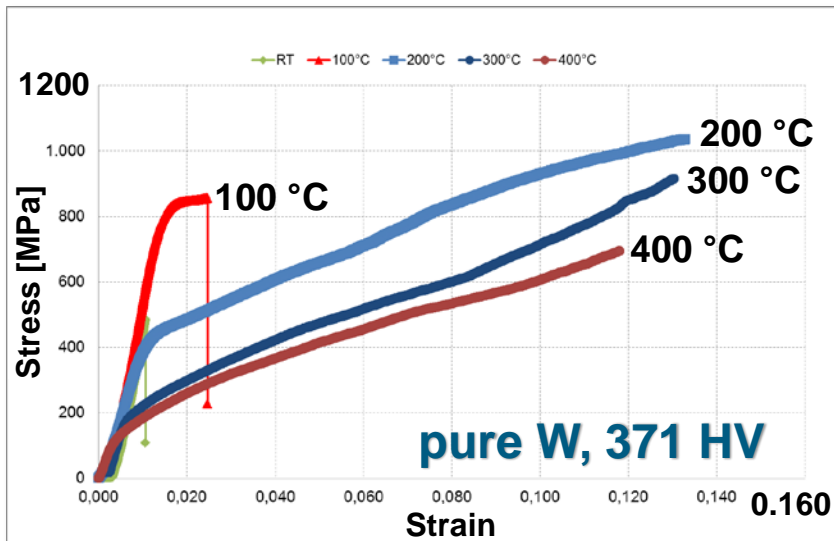
❖ Decreasing roughness with increasing hardness

ELM-test at elevated temperature



✓ Further investigation via 4pt-bending & disruption tests

Mechanical testing via 4pt-bending



Sample geometry: $12 \times 1 \times 1 \text{ mm}^3$
Strain rate: 0.033 mm/min

Disruption test - Rationale

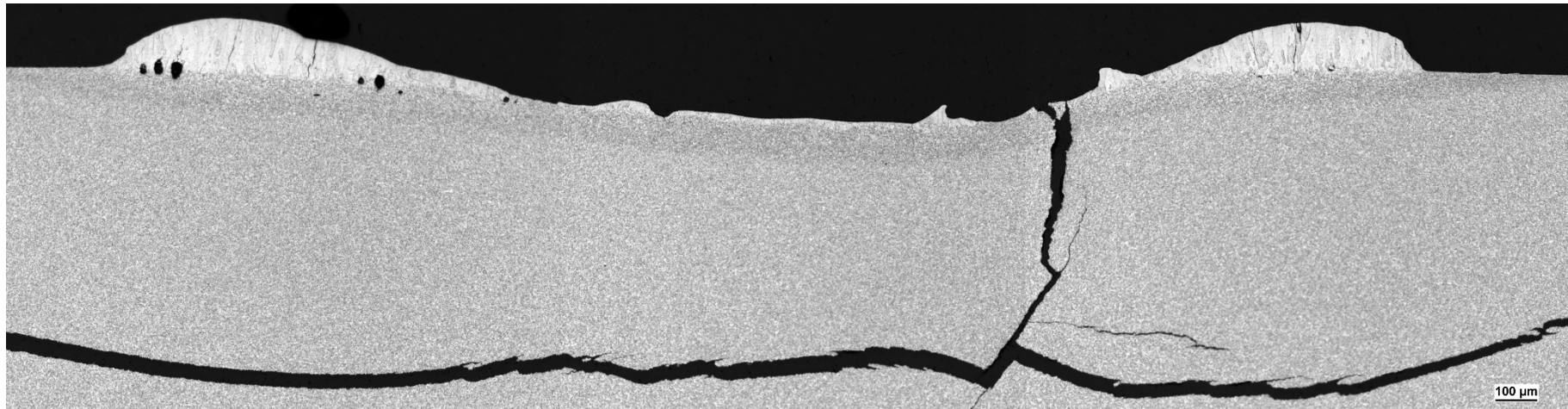


W-1.1TiC (H. Kurishita): $P_{abs} = 0.38 \text{ GW/m}^2$, $t = 1 \text{ ms}$, $n = 1000$, $T = 1000 \text{ }^\circ\text{C}$

\Rightarrow low roughness ($R_a = 0.3 \text{ } \mu\text{m}$)

\Rightarrow only small and localized crack formation

$P_{abs} = 1.13 \text{ GW/m}^2$, $t = 5 \text{ ms}$, $n = 10$, $T = \text{RT}$

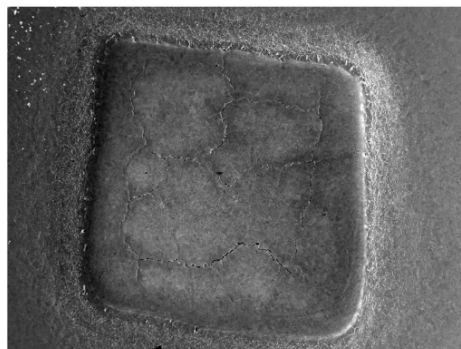


No disruption on cool surface \Rightarrow disruption test will be done at $1000 \text{ }^\circ\text{C}$!

Disruption test

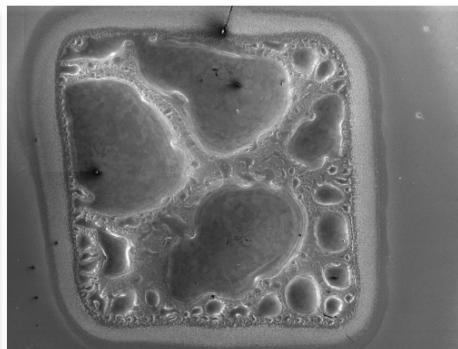


W-5050



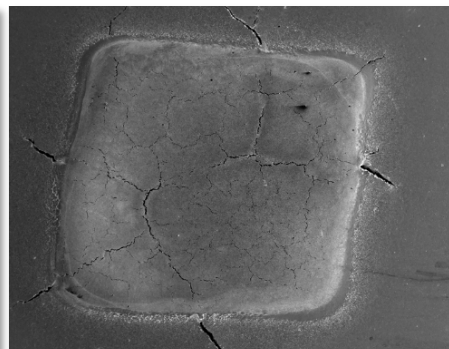
Antusch S 2 15:1

W-2Y₂O₃



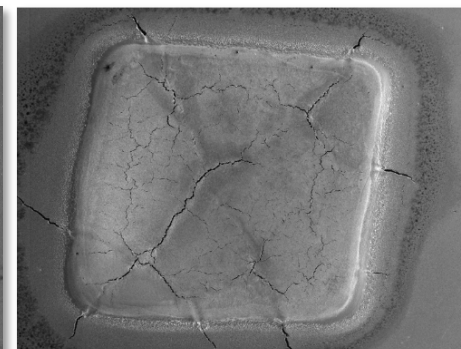
Antusch S 6 15:1

W-1TiC

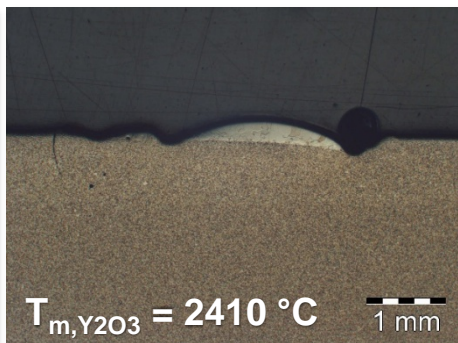
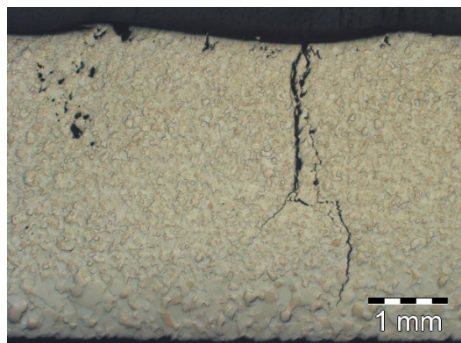


Antusch S 13 15:1

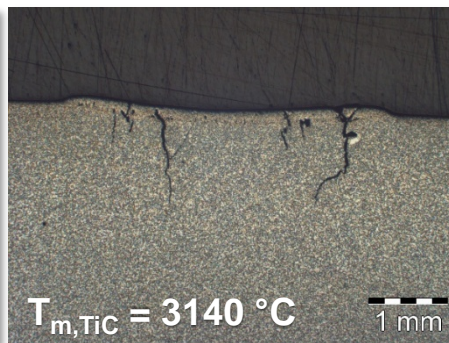
W-2TaC



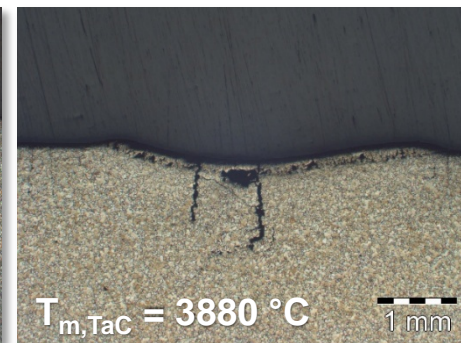
Antusch S 7 15:1



$T_{m,Y2O3} = 2410 \text{ } ^\circ\text{C}$



$T_{m,TiC} = 3140 \text{ } ^\circ\text{C}$



$T_{m,TaC} = 3880 \text{ } ^\circ\text{C}$

Loading conditions: $n \equiv 100$
 $T \equiv 1000 \text{ } ^\circ\text{C}$
 $\Delta t \equiv 5 \text{ ms}$
 $L_{\text{abs}} \equiv 1.13 \text{ GW/m}^2$

**Crack formation
vs.
inhomogeneous melting!**



Fabrication

- ❖ Mass production of near-net-shape parts via PIM @ KIT
- ❖ Brittle to ductile transition for *pure PIM W* at **200 °C (low strain rates)**
- ❖ High density (> **99 % T.D.**)
- ❖ Fully *isotropic* material properties

Qualification

- ❖ Increased hardness and mechanical strength for ***W-Y₂O₃*** and ***W-TiC*** materials ⇒ ***reduced roughening / plastic deformation***
- ❖ Reduced roughening ⇒ ***thermal fatigue*** induced crack formation (in view of > 10⁶ ELM pulses) ***is slowed down***
- ❖ Disruption test: ***crack formation*** (≥ 1 mm) is still an issue even ***at 1000 °C***
- ❖ Disruption test: ***additives*** with relative ***low melting point*** may cause an ***inhomogeneous melt distribution***