



**PWIE-SPA 4-D002**

# **Annealing of chosen tungsten-based materials and quantification of recrystallization kinetics**

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## Task specification




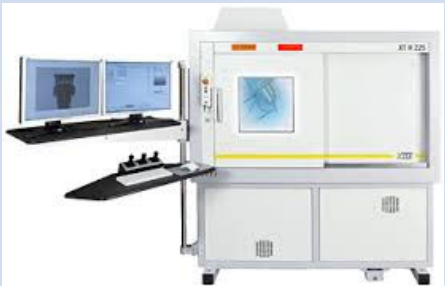

### Investigated Materials

1. Additively manufactured W EBM      Stefan Antusch
2. Powder metallurgical W<sub>f</sub>/W      Yiran Mao
3. Cold-rolled W 80 ppm potassium-doped

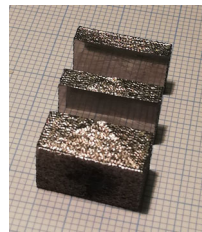
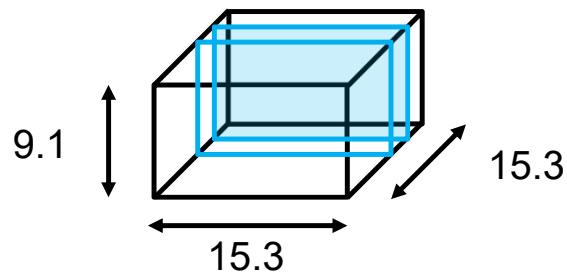


### Characterization techniques

- Before mainly EBSD and HV
- More recently supplemented by XRD and CT

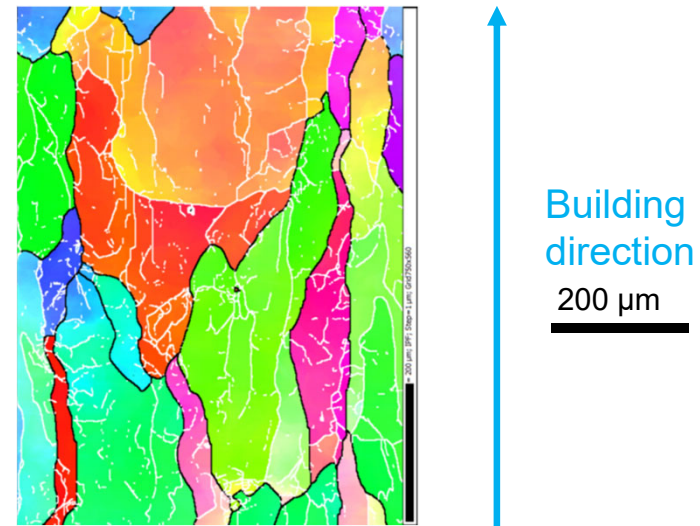
	<b>ZEISS Xradia 410 Versa</b>	<b>Nikon XT H 225</b>	<b>ZEISS nanoXCT-100</b>
Resolution	1 - 50 $\mu\text{m}$	5 - 200 $\mu\text{m}$	Down to 50 nm
Sample size	1 - 50 mm	5 - 50 mm (max FOV 35 mm)	Up to 65 $\mu\text{m}$
Power	10 W	225 W	
X-ray Energy	40 - 150 keV	Up to 225 keV	9.2 keV (Ga K $\alpha$ )
			
	Most suited for purpose	Best transmission, but low resolution	Best resolution, too low energy

- Specimens from MAT-T.03.01-T003
- Cutting



- KIT 99.6% dense
- EBSD investigation on cross section

- Map with HAB (15°) and LAB (1°)

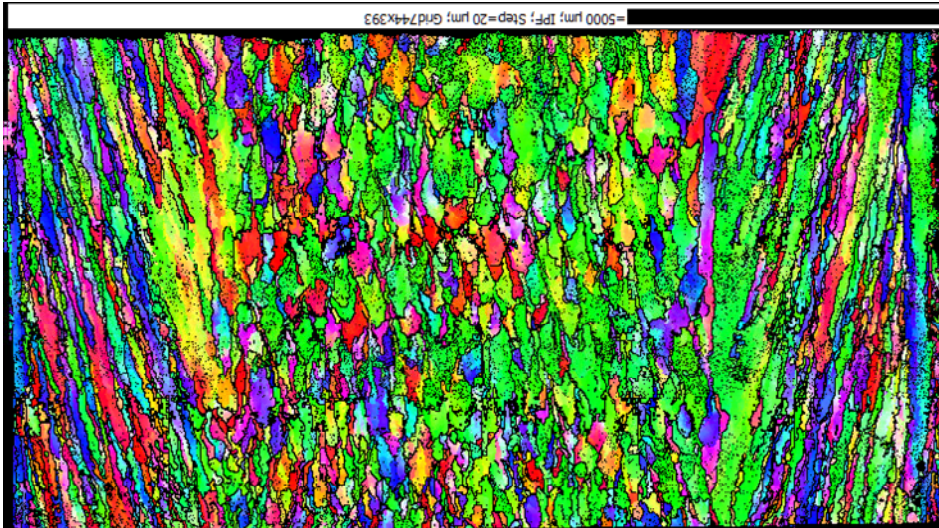


- Huge elongated grains in building direction
- Mainly low disorientations below 2° within grains



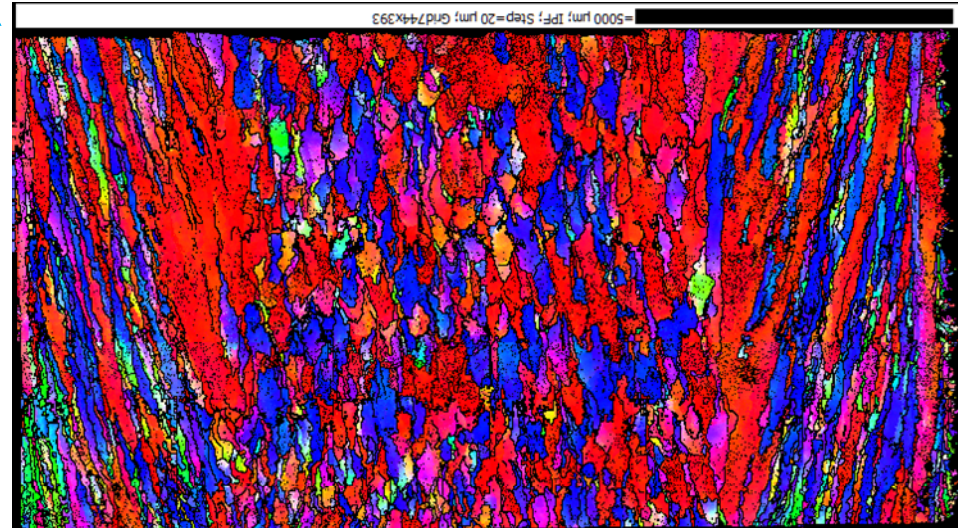
- Large overview map over entire slice

X

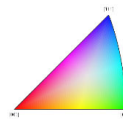


- Rotated cube +  $\langle 111 \rangle$  fiber texture

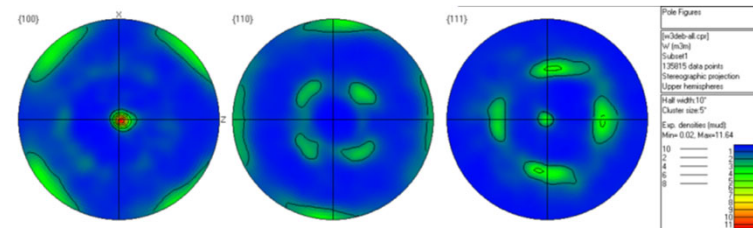
BD



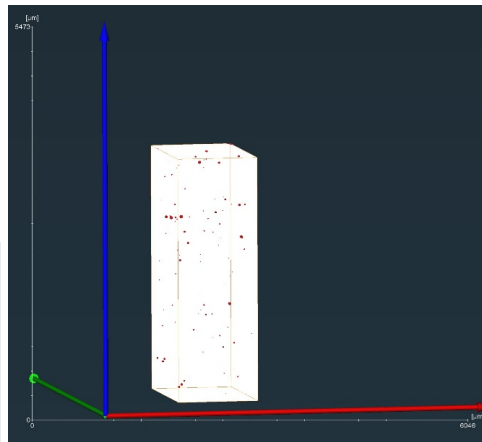
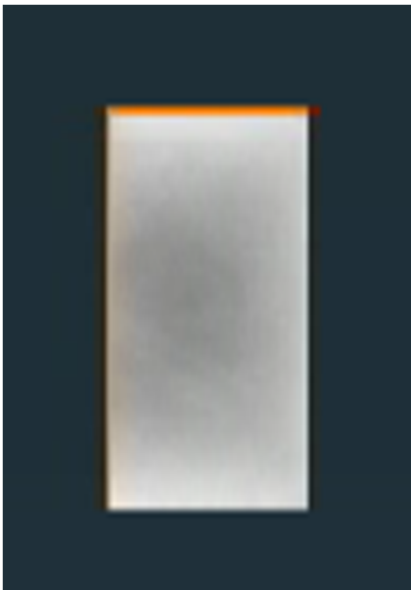
- Map size 14.9 x 7.9 mm<sup>2</sup>
- Morphological heterogeneity from spiral printing strategy
- 13 layers



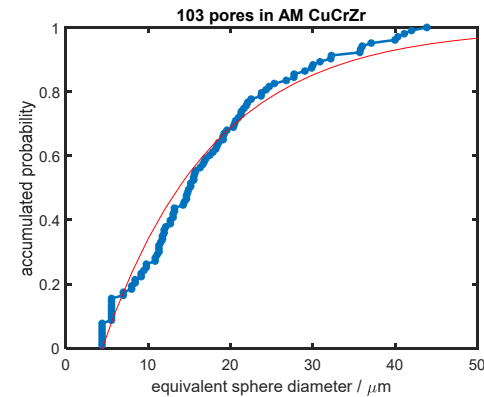
- Pole figures from center part



- Cut parts from cubes  $12 \times 12 \times 12 \text{ mm}^3$
- CT
- Reconstructed pores



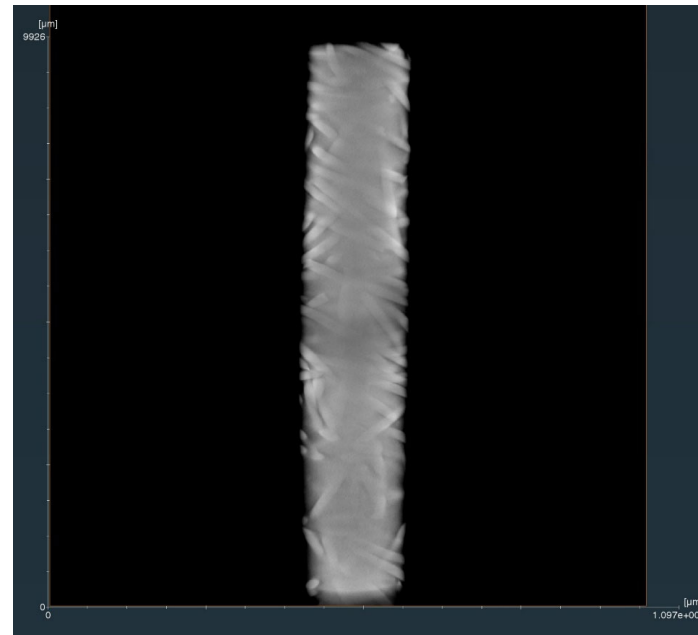
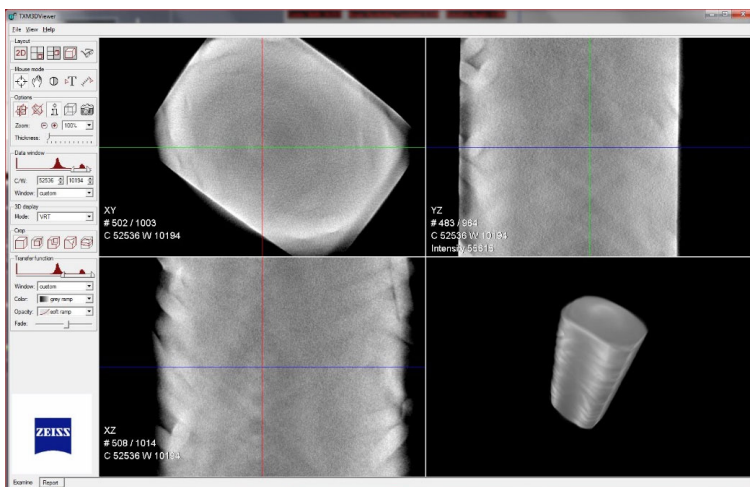
- Quantification of pores
  - Volume fraction 0.35 %
  - High density close to top surface
  - Pore size distribution



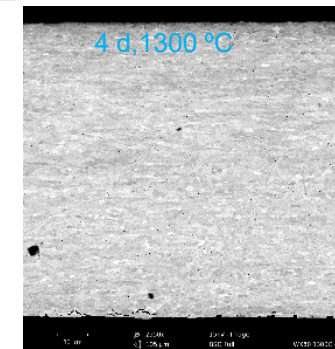
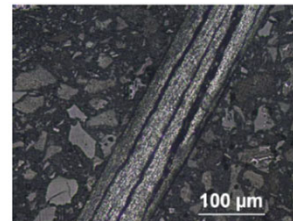
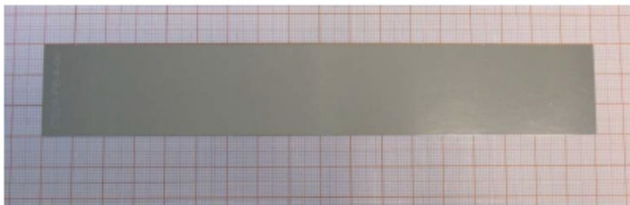
- Note, missing pores below 4  $\mu\text{m}$

- Specimens PRD-5.HFFM.PFM-T011
- Cut 2x1.5x12 mm<sup>3</sup>
- Unfortunately slightly too thick
- Computer tomography

- Quantification of wire fraction (in parts allowing reconstruction)
- 48% (51%)
- depends slightly on chosen resolution



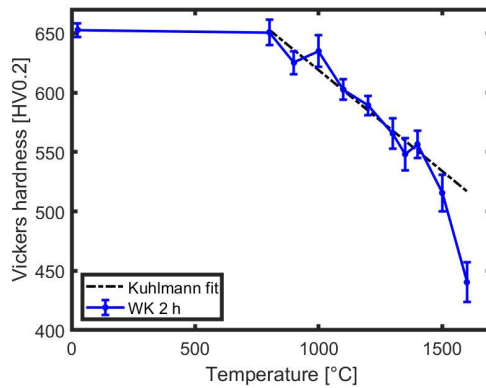
- Supplement to rolled tungsten with 60 ppm K
- Rolled plates 180 x 30 x 0.101 mm<sup>3</sup> supplied by Plansee SE (Andreas Hoffmann)



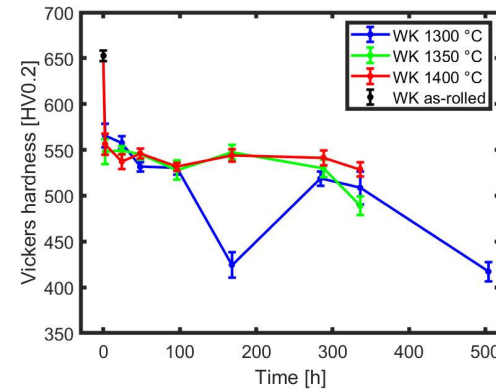
- Difficulties in cutting, preparation and annealing due to delamination ... solved!
- Microhardness on RD/TD with Vickers indent, load 200 g
  - Initial microhardness 653±1 HV0.2
  - After annealing at 1300 °C for 4 days 531±2 HV0.2
- Annealing at 1300 °C, 1350 °C, 1400 °C done
- Continued with 1125 °C



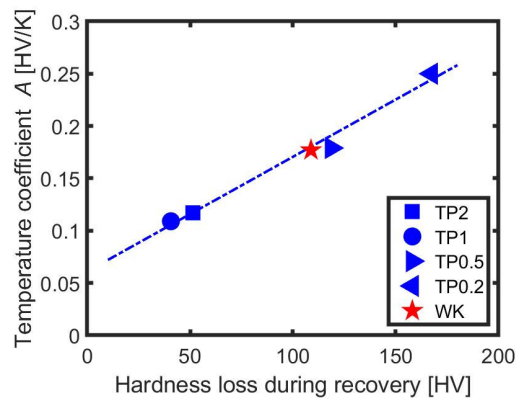
- Isochronal annealing



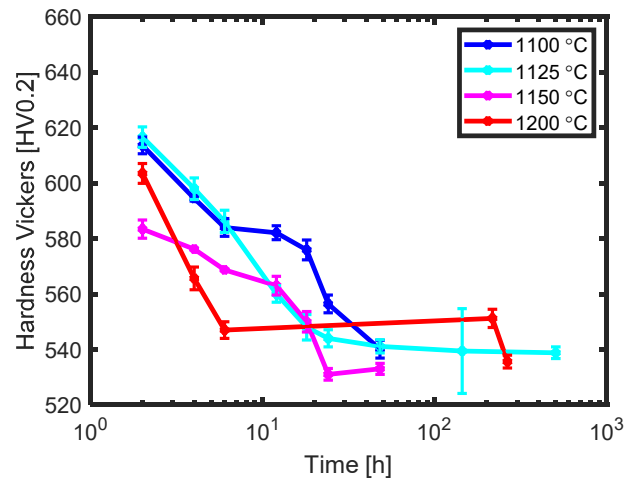
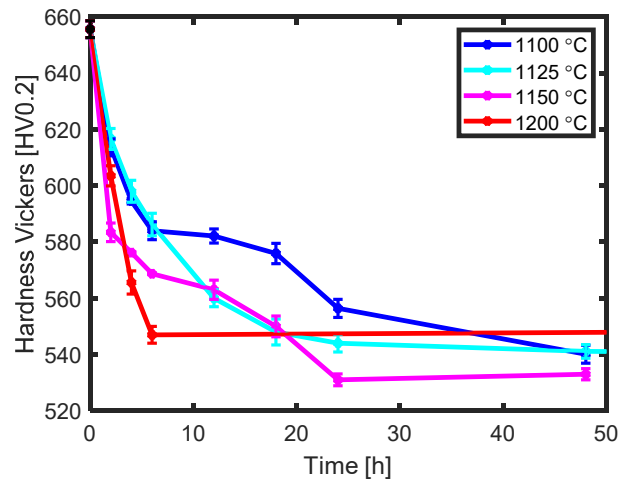
- Isothermal annealing



- Comparison with pure tungsten

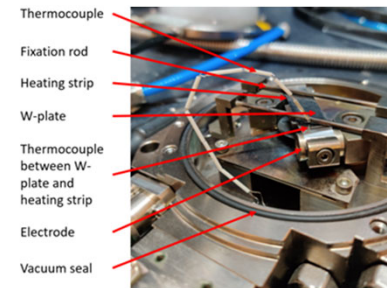
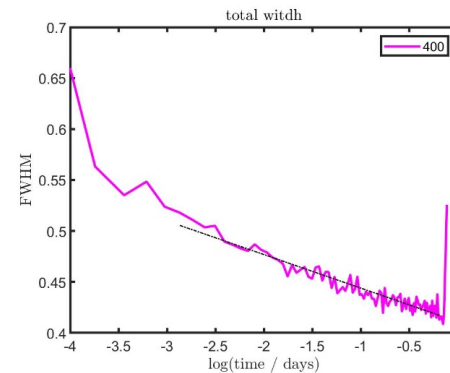
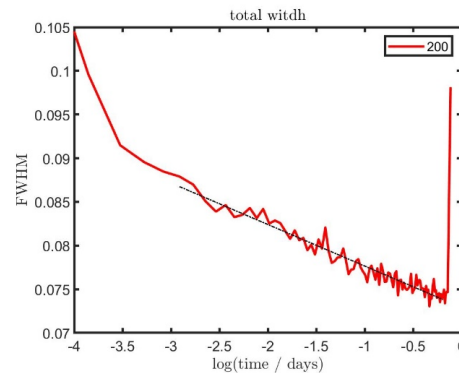
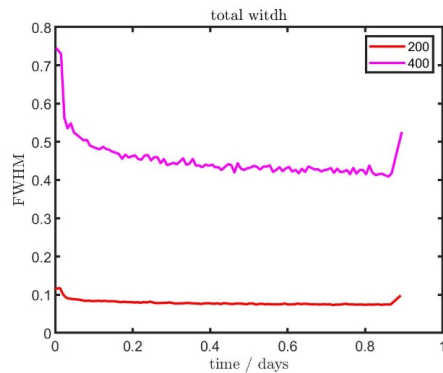


- Annealing at 1300 °C, 1350 °C, 1400 °C done
- Continuation to lower temperatures down to 1100 °C up to 75 days



- Recovery with logarithmic time dependence for very short time
- Apparent stagnation at 540 HV0.2
- Onset of growth not before 1800 h (75 days) at 1125 °C

- XRD in-situ investigations in reflection at three different temperatures
- Strong  $\{100\}\langle 011\rangle$  texture – only 200 and 400 peak assessable
- Annealing at 1050 °C, 1100 °C, 1150 °C under vacuum up to 24 h



- Almost perfectly following logarithmic time dependence

$$FWHM = FWHM_0^* - M \ln(t)$$

- Interpretation in terms of temperature dependence
- Profile analyse to clarify (main) origin for broadening



## Task specification



1. Additively manufactured W EBM                      Stefan Antusch  
*EBSD performed, quantitative analysis pending*
2. Powder metallurgical W<sub>f</sub>/W                      Yiran Mao  
*CT performed, promising, thinning of samples*
3. Cold-rolled W 80 ppm potassium-doped  
*Ex-situ annealing, in-situ annealing with XRD performed, detailed analysis*
  
4. Additively manufactured W LBM                      Alexander von Müller  
*Put on hold*
5. Chemical vapor deposited W<sub>f</sub>/W                      Johann Riesch  
*Samples received, to be annealed*
6. Cross rolled tungsten  
*Awaiting new differently rolled plate*

