Annealing studies of tungsten-based materials

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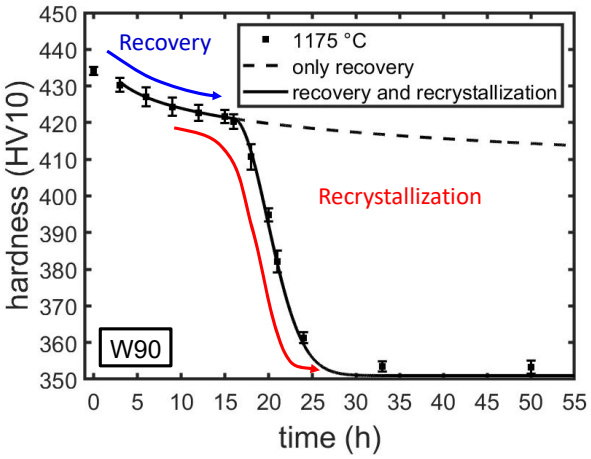
Thermal stability of rolled tungsten plates

Thermal stability of tungsten - degradation

- Annealing of tungsten plate warm-rolled to 90% reduction



- Restoration of properties: removal of defect content
- Recovery: dislocation annihilation and rearrangement
- Recrystallization: dislocation removal by boundary motion
- Competition about driving force

Investigated material

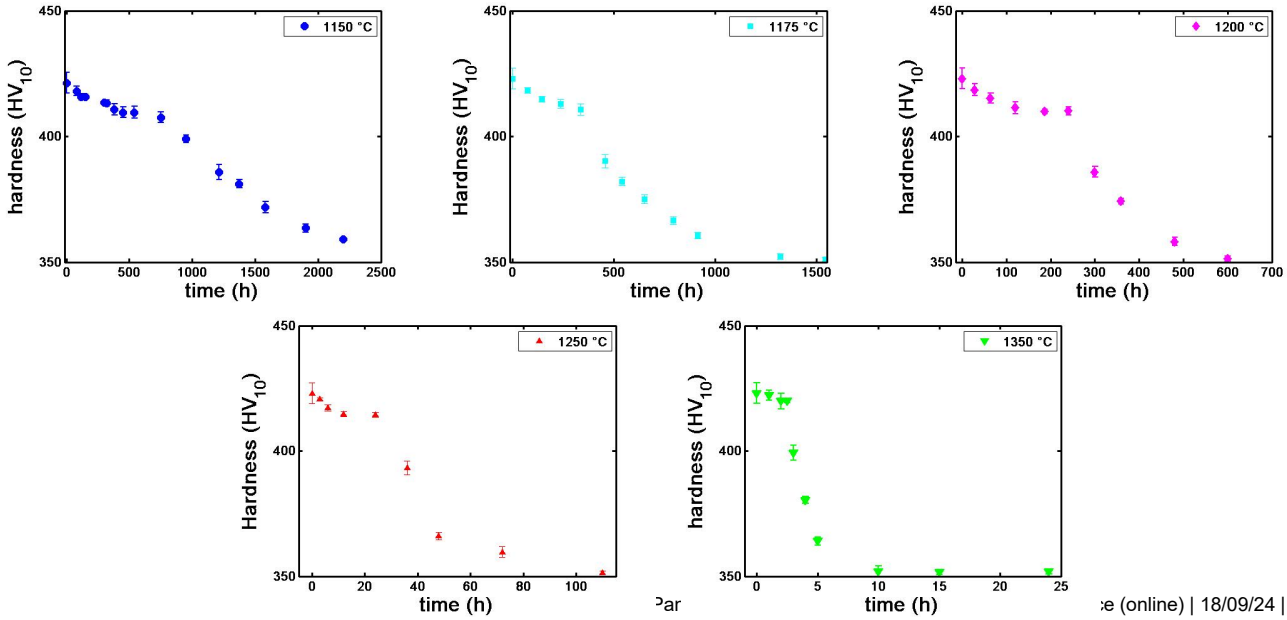
Rolled tungsten plates from different manufacturers					
AT&M, China		Plansee S/E, Austria		A.L.M.T., Japan	
Warm-rolled to different thickness reductions		Warm- and cold-rolled to different thickness		Uni-directionally and cross-rolled	
67 %,	W67	WR 2 mm	TP2	UR	IGW
80 %	W80	WR 1mm	TP1	CR low ratio	CLW
90 %	W90	CR 0.5 mm	TP 0.5	CR high ratio	CHW
		CR 0.2 mm	TP0.2		

IGW baseline material for EUROfusion 2020

Isothermal annealing - overview

	AT&M			Plansee				A.L.M.T.		
	W67	W80	W90	TP1	TP2	TP05	TP02	IGW	CLW	CHW
1100 °C			☑					☑	☑	☑
1125 °C								☑	☑	☑
1150 °C	☑	☑	☑					☑	☑	☑
1175 °C	☑	☑	☑					☑	☑	☑
1200 °C	☑	☑	☑					☑	☑	☑
1225 °C			☑					☑	☑	☑
1250 °C	☑	☑	☑							
1300 °C		☑		☑	☑	☑	☑			
1325 °C				☑	☑	☑	☑			
1350 °C	☑	☑		☑	☑	☑	☑			
1375 °C				☑	☑	☑	☑			
1400 °C		☑		☑	☑	☑	☑			

W67 Vickers hardness after isothermal annealing



W67 Comparison of overall kinetics

- Combined hardness

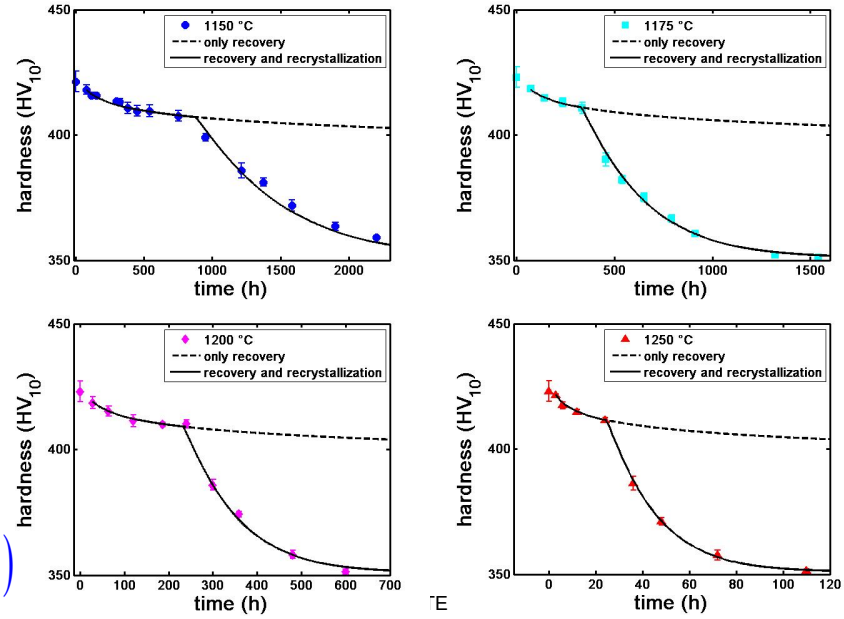
$$HV = X\ HV_{rex} + (1 - X)\, HV_{rec}$$

- Recovered fraction (Kuhlmann kinetics)

$$HV_{rec} = HV_0^* - C \ln t$$

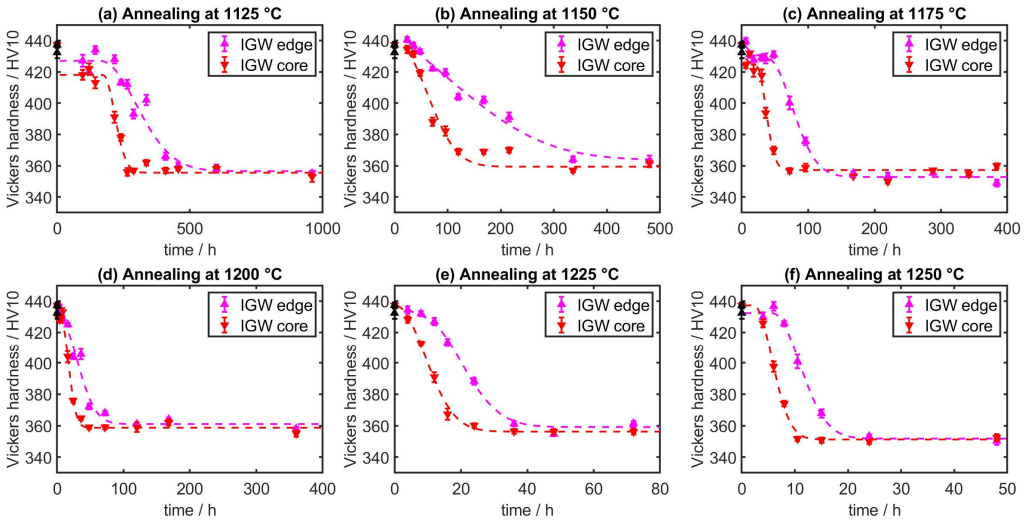
- Recrystallized fraction HV_{rex}
- Recrystallization (JMAK kinetics)

$$X = 1 - \exp\left(-b^n \left(t - t_{inc}\right)^n\right)$$



Hardness evolution IGW 1125 °C to 1250 °C

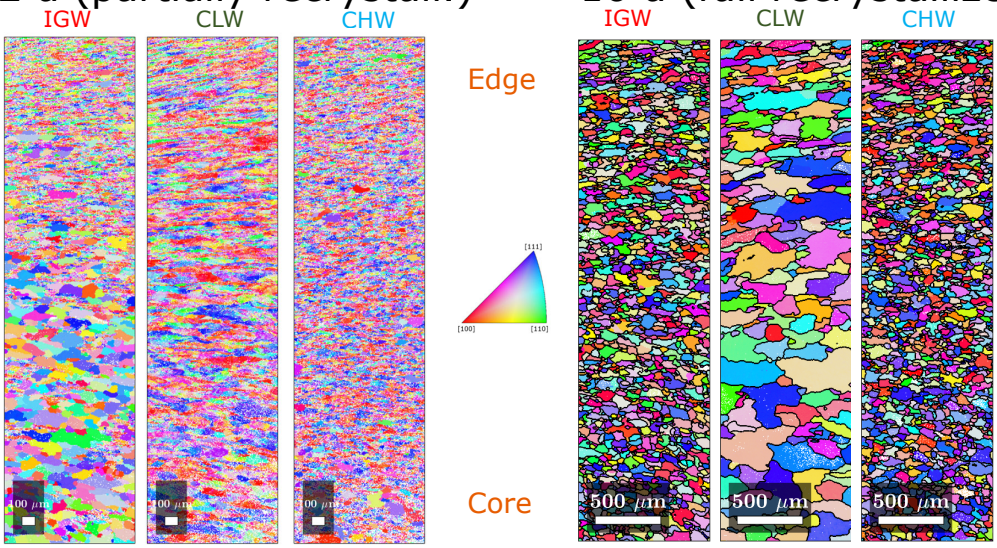
- IGW



Edge slower than core

Microstructural investigation EBSD – annealing at 1175 °C

- 2 d (partially recrystall.)
- 16 d (full recrystallized)

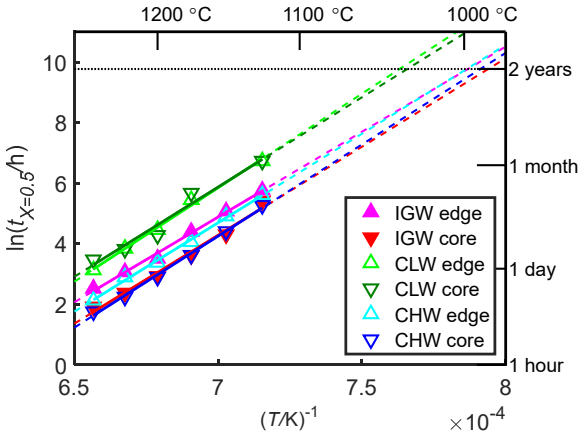


Annealing kinetics – half recrystallization

- Time to half of the entire hardness loss
- Thermal activated process

$$t_{X=0.5} = t^{*}_{X=0.5} \exp\left(Q/RT\right)$$

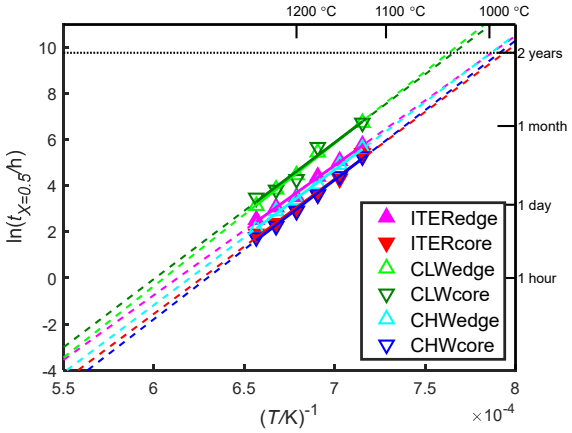
- Activation energy
- Maximal temperature for two years of operation



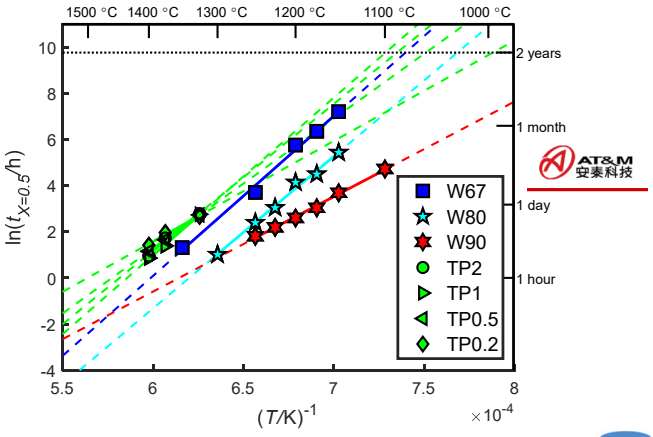
Half recryst.	IGW Edge	IGW Core	CLW Edge	CLW Core	CHW Edge	CHW Core
Q / kJ/mol	468	486	516	492	467	503
T (2 y)	998 °C	987 °C	1037 °C	1033 °C	999 °C	991 °C

Annealing kinetics – comparison of all plates

- Inferior to other plates (W67 AT&M or TP2/TP1 Plansee)



A.L.M.T. Corp.



AT&M 安泰科技

PLANSEE

Tungsten fiber-reinforced tungsten composites

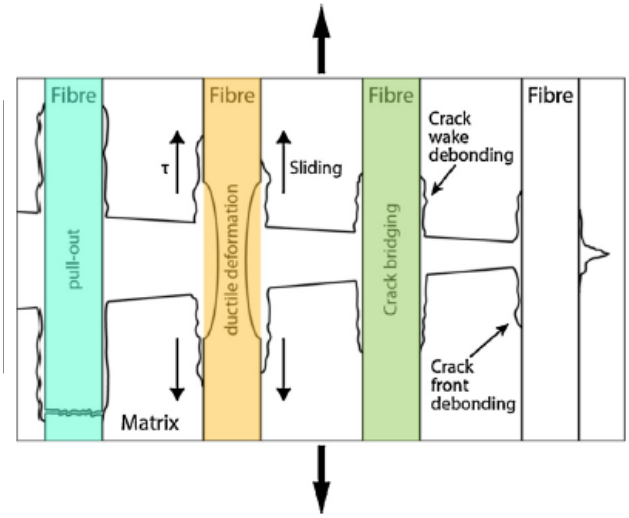
Tungsten fiber-reinforced tungsten composites

- Fiber composite

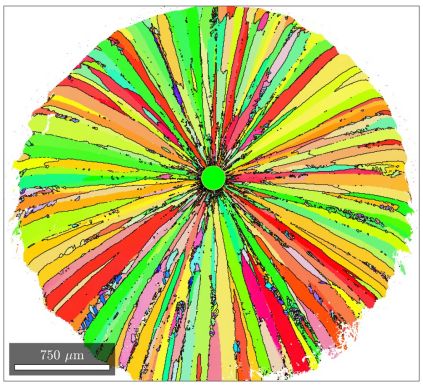
Fiber	Matrix
K-doped tungsten	pure tungsten
Wire	CVD
Ductile	Brittle

- Pseudo-ductile behavior
- Matrix fails, fibers bridge

- Different interlayers none / erbia / yttria



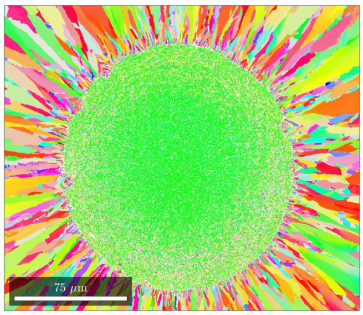
Growth of W on W_f by CVD

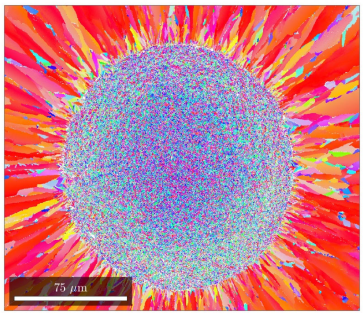


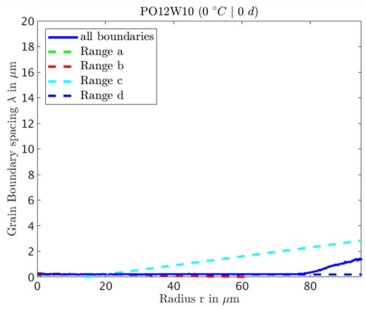
W wire

CVD W
Columnar grains

W_f/W As-received







0 d
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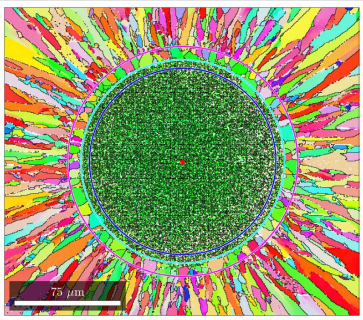
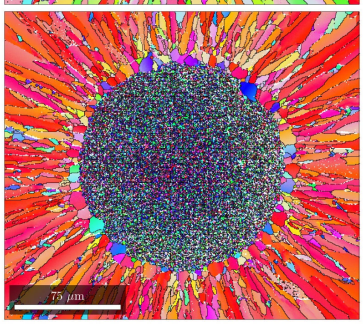
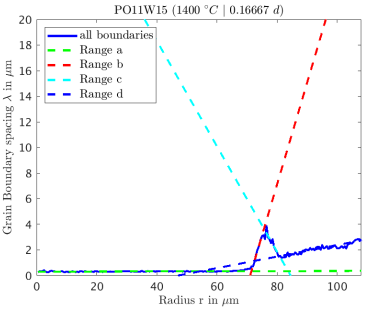
28 d

W_f/W Annealed 1400 ºC, 0.16 d







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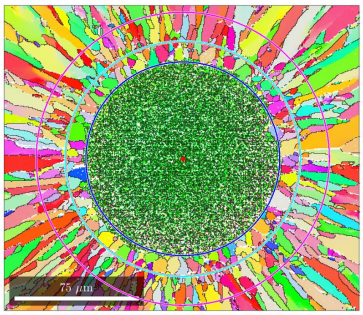
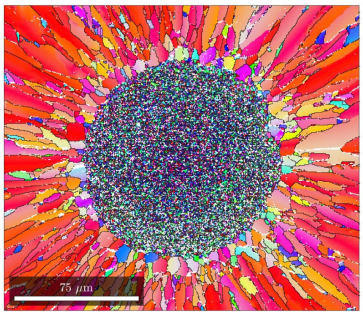
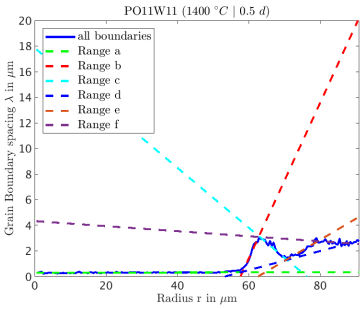
W_f/W Annealed 1400 ºC, 0.5 d







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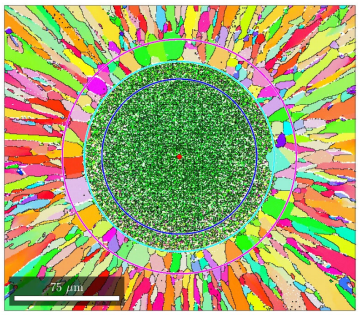
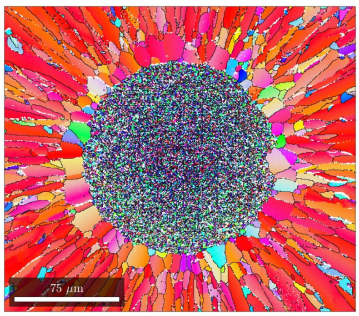
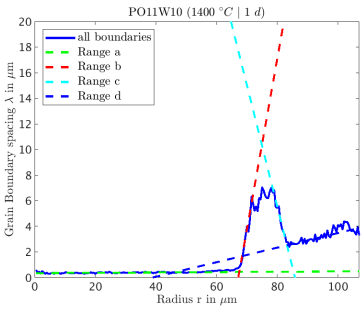
21 d
28 d

W_f/W Annealed 1400 ºC, 1 d







0 d
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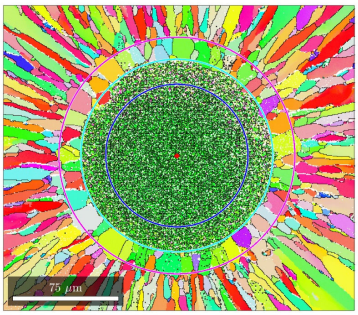
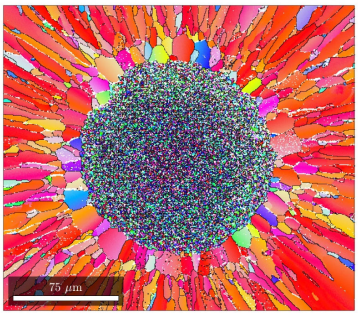
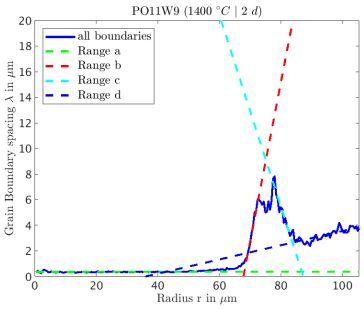
4 d
7 d
14 d
21 d
28 d

W_f/W Annealed 1400 ºC, 2 d







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0.5 d
1 d
2 d
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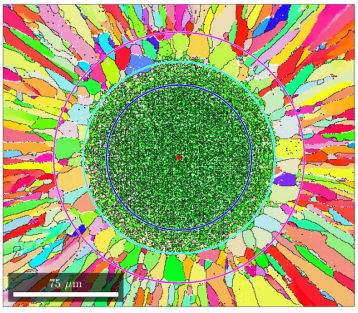
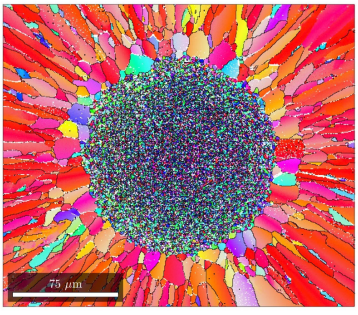
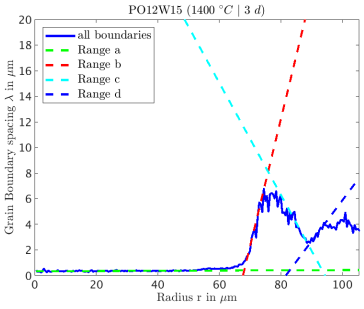
4 d
7 d
14 d
21 d
28 d

W_f/W Annealed 1400 ºC, 3 d







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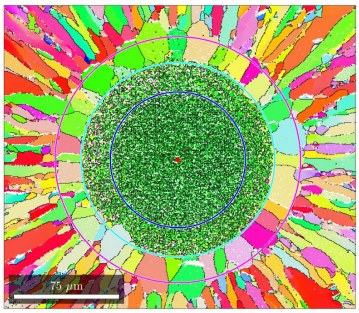
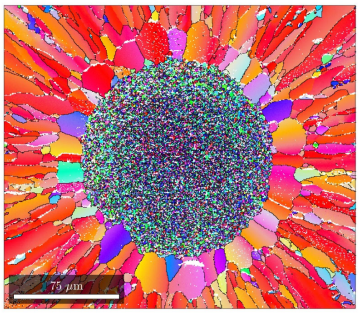
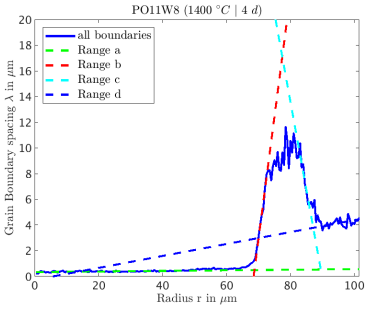
7 d
14 d
21 d
28 d

W_f/W Annealed 1400 ºC, 4 d







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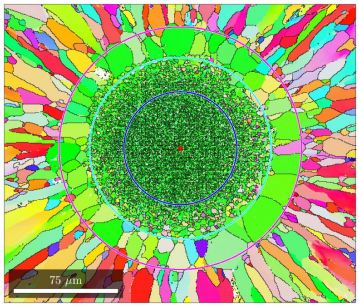
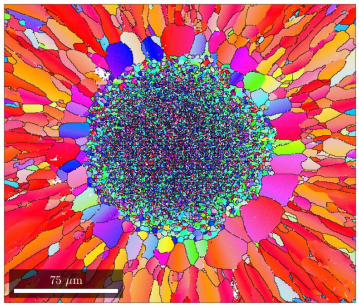
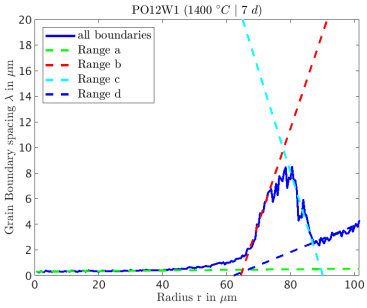
21 d
28 d

W_f/W Annealed 1400 ºC, 7 d







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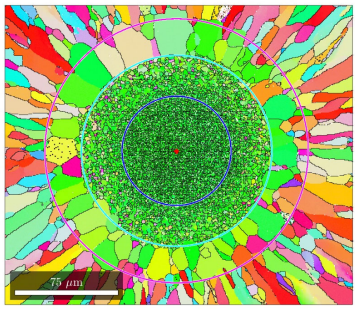
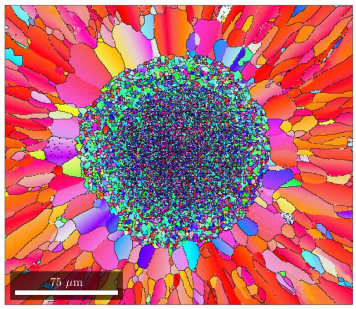
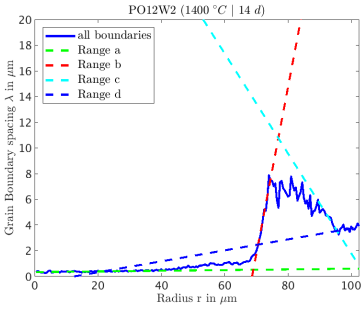
28 d

W_f/W Annealed 1400 ºC, 14 d







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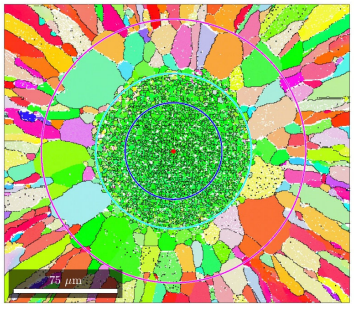
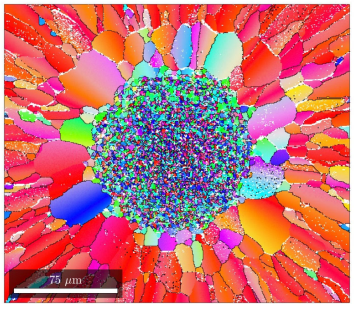
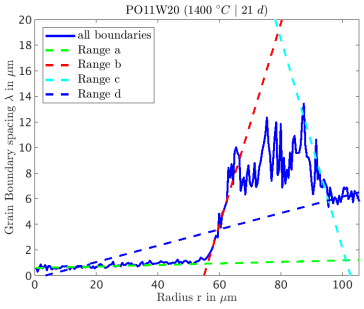
28 d

W_f/W Annealed 1400 ºC, 21 d







0 d
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1 d
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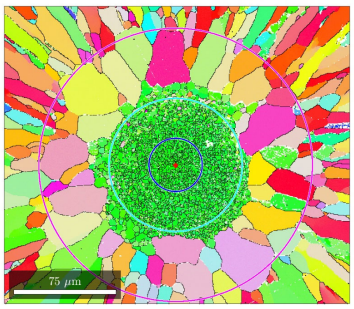
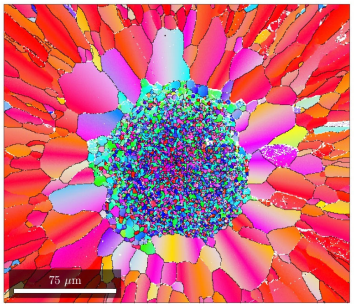
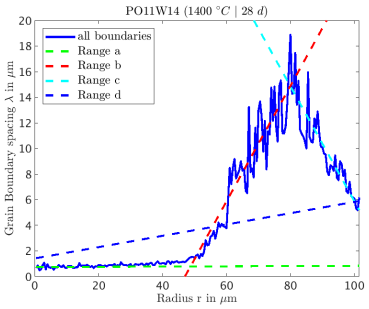
28 d

W_f/W Annealed 1400 ºC, 28 d







0 d
0.16 d
0.5 d
1 d
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3 d
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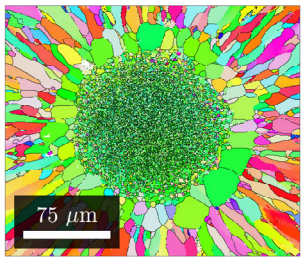
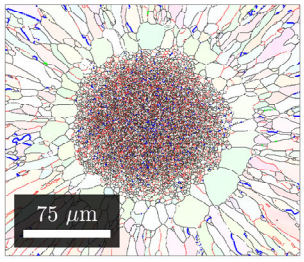
7 d
14 d
21 d
28 d

Comparison W_f/W and W_f/Er_2O_3/W



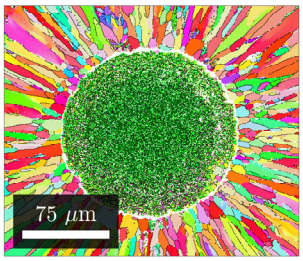


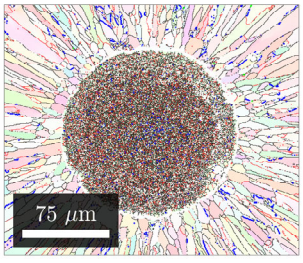
Similarities

- Outward growth
  - »Secondary rex
- Apparent grain growth in outer layers of wire
  - »Primary rex

Differences

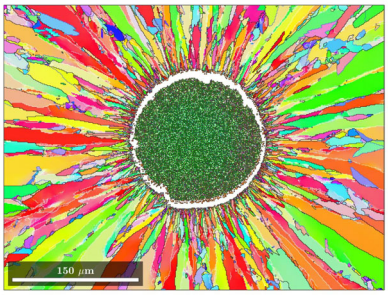
- Inward growth
  - »Secondary rex
- Texture



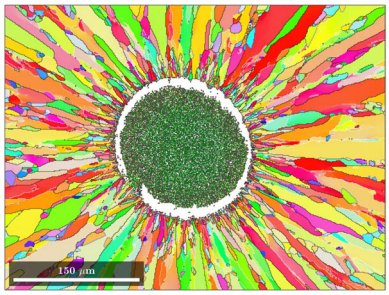


Thermal stability of tungsten fiber-reinforced tungsten composites – higher temperatures

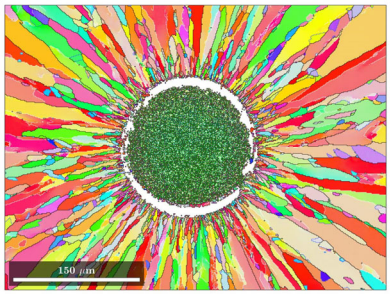
W_f/W 3 µm yttria interlayer — annealing at 1450 °C (Cartesian)



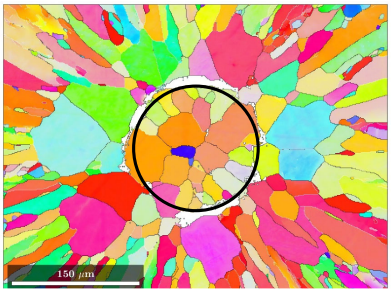
As processed



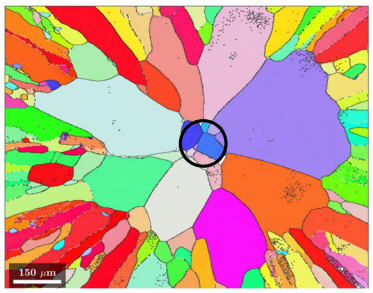
1 day



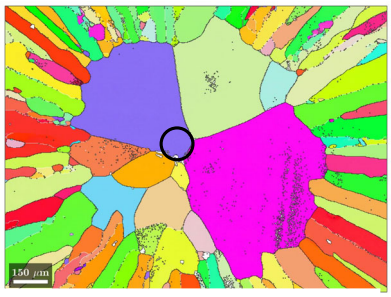
2 days



4 days



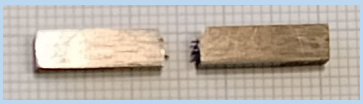
1 week



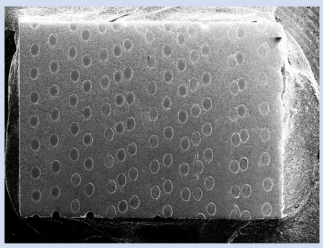
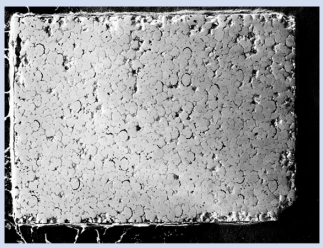
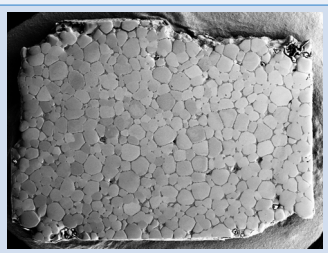
2 weeks

Tungsten fiber-reinforced tungsten W_f/W

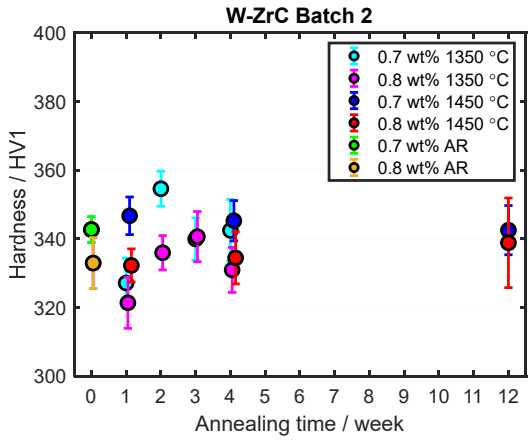
- Multi fiber composites from Yiran Mao
- Powder metallurgical route
- Single edge notch bending specimens 27x4x3 mm^3 (three point bending test specimens)

Spec.	Fibers	Alignment	Matrix	Interlayer	State	
A	Conti-nuous	Parallel in layers	Dense	Yttria	Bend to fracture	

W_f/W continuous fibers – Light optical microscopy

As-received		Annealing for 1 week at 1450 °C	
 notch side	Note fiber elongation from manu-facturing / bending		Grain growth in matrix
Annealing for 3 days at 1450 °C		**Annealing for 2 weeks at 1450 °C**	
	Grain growth in matrix		Grain growth eliminated fibers completely

Tungsten with ZrC dispersoids – hardness evolution



- Hardness almost constant
- 0.7 wt.% ZrC slightly harder than 0.8 wt.% ZrC
- Not from porosity, but from grain size
- Hardness unchanged even after 12 weeks

Summary

- Deformed pure tungsten plates suitable for operations temperatures below 1100 °C (from long term kinetics)
- Tungsten fiber-reinforced tungsten composites
  - slow microstructural changes up to 1400 °C
  - interlayers do not prevent intergrowth at 1450 °C
  - not only for CVD also for PM composites
- Particle-reinforced tungsten composites
  - W_2C: coarsening and ripening at 1400 °C
  - ZrC: dispersoids hinder growth for 12 weeks at 1450 °C

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