

The evolution of ferritic ODS alloys at KIT

From laboratory size to large scale batches

Microstructure, mechanical characterization and nanostructure

Jan Hoffmann

Institute for Applied Materials (IAM-AWP)



Large lab scale production

Challenges – Powder handling



Filling

Larger scale

- Capsule design was adapted to feature 2 nozzles
- Upgraded glove box for handling of larger powder quantities
- Sealing process was optimized



Sealing / Welding

Large lab scale production

Challenges – compacted capsules

Lab scale

- ~ 500 g powder
- max. load 250 MPa



large (lab) scale

- Ø 2 m
- max. load 200 Mpa



Larger lab scale production

First assessment

- 50 kg of base powder were ordered and gas atomized

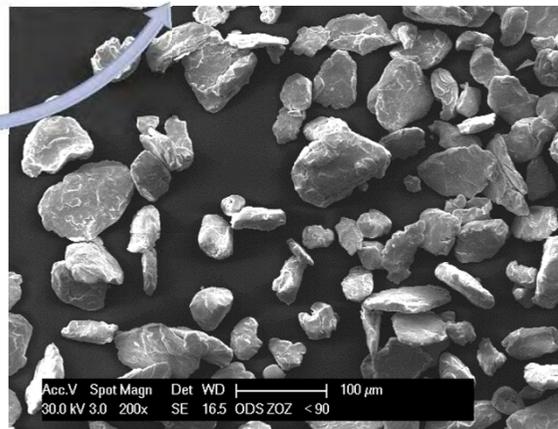
Powder production

Composition:

Fe	bal.
Cr	13%
W	1%
Ti	0.3%
Other elements ALAP	

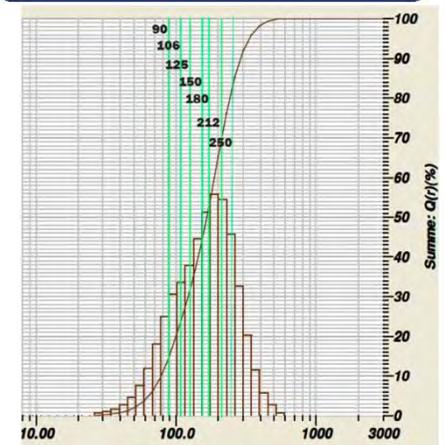
Mechanical alloying

- Mechanical alloying of 10 kg of base powder with **0.3 wt.% FeY** MA at Zoz GmbH

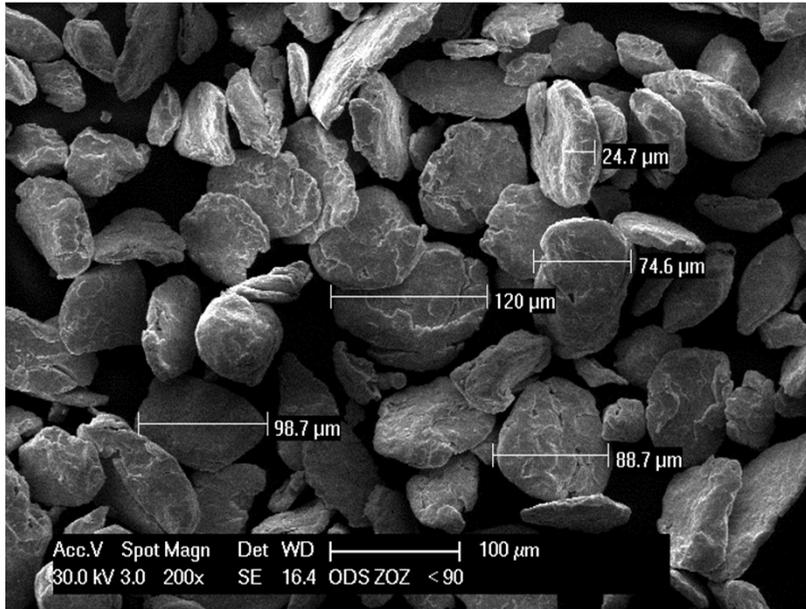


- Powder was sieved into two fractions:
- R** > 90 µm
r < 90 µm

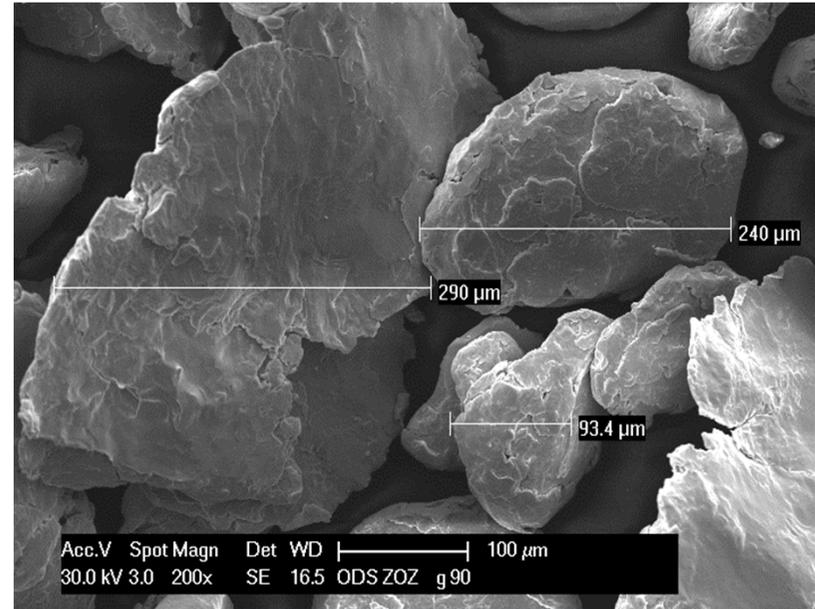
Sieving



Powder after MA - particle sizes



r
d50 < 90 μm
~ 4 kg



R
d50 > 90 μm
~ 5 kg

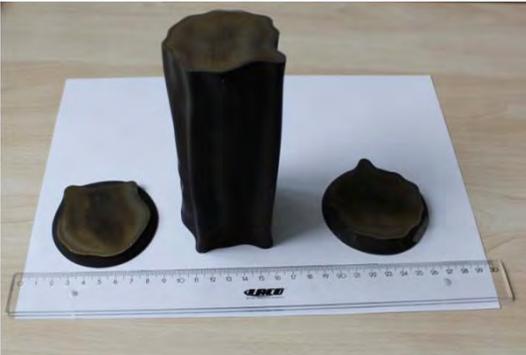
As-milled

- difference in size, but no difference in shape

Powder after MA - particle sizes

Powders
< 90µm + > 90µm

HIP



HIP
Rolling



Extrusion



Powder after MA - particle sizes

Powders
< 90µm + > 90µm

HIP

HIP
Rolling

Extrusion

R ✓

~~r~~ X

R ✓

PSI r ✓
lab-size



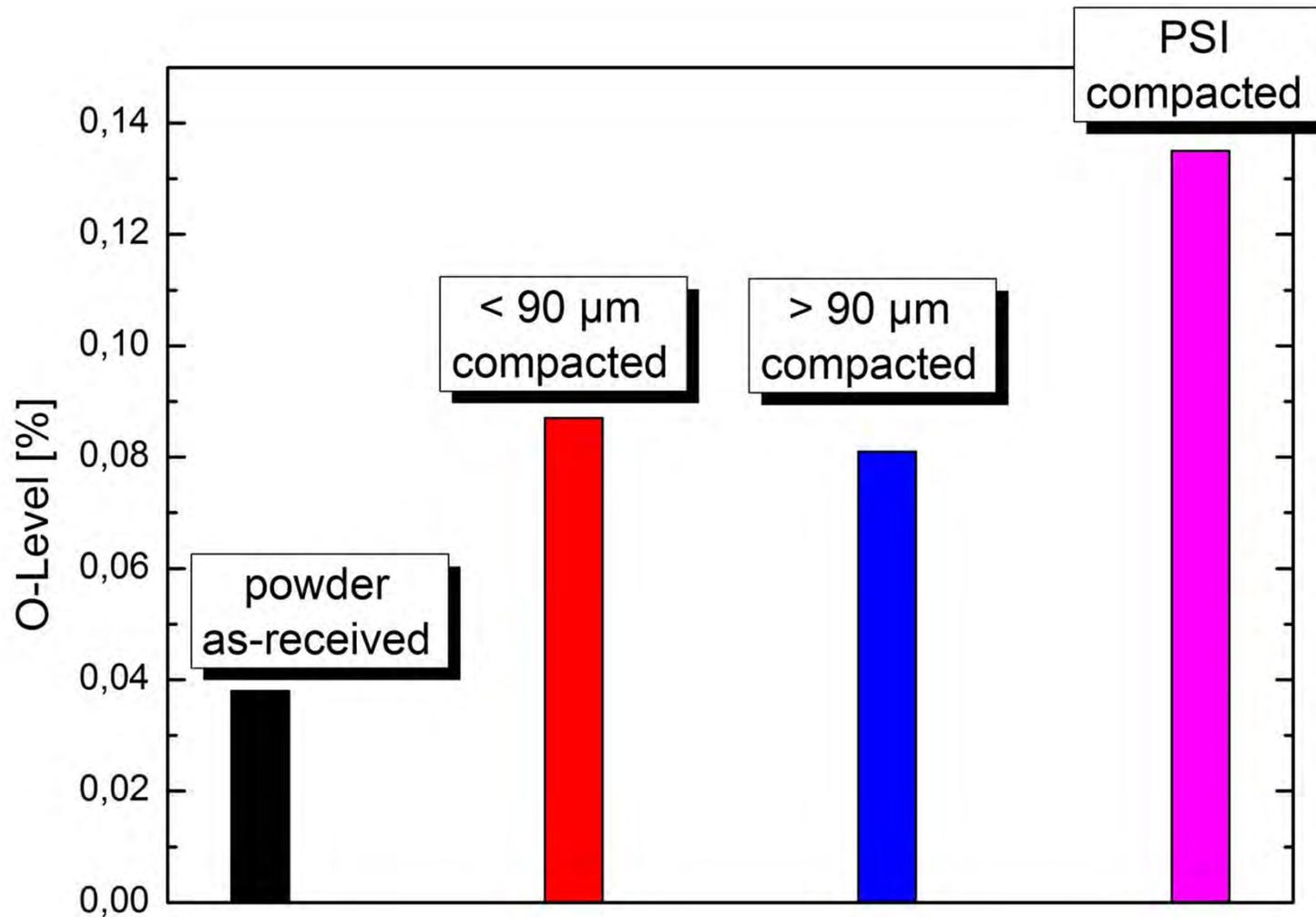
Powder after MA - impurities

Size fraction	< 90 μm		> 90 μm	
element	average value	SD	average value	SD
B	< 0,005	-	< 0,005	-
C	0.0216	0.0004	0.0214	0.0001
N	0.0098	0.0004	0.0101	0.0001
O	0.087	0.002	0.081	0.001
Ti	0.152	0.0001	0.151	0.0002
Cr	13.1	0.01	13.1	0.01
Co	0.0071	0.0001	0.0072	0.0000
Ni	0.0876	0.0009	0.0894	0.0005
Cu	0.0118	0.0001	0.0118	0.0001
Y	0.199	0.02500	0.217	0.00040
Mo	0.0029	0.00010	0.0030	0.00010
W	1.13	0.002	1.11	0.003
S	14.8189		14.8119	

As-milled

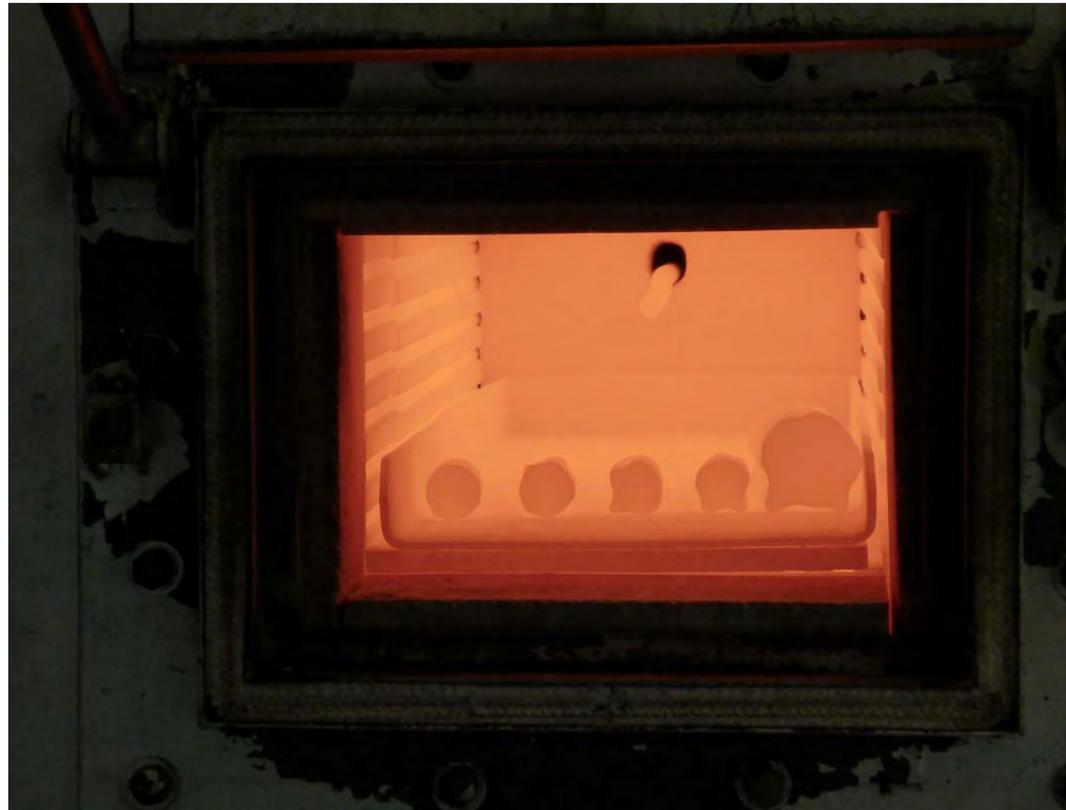
- no significant difference in chemical composition and impurities

Powder after MA - impurities



Large lab scale production

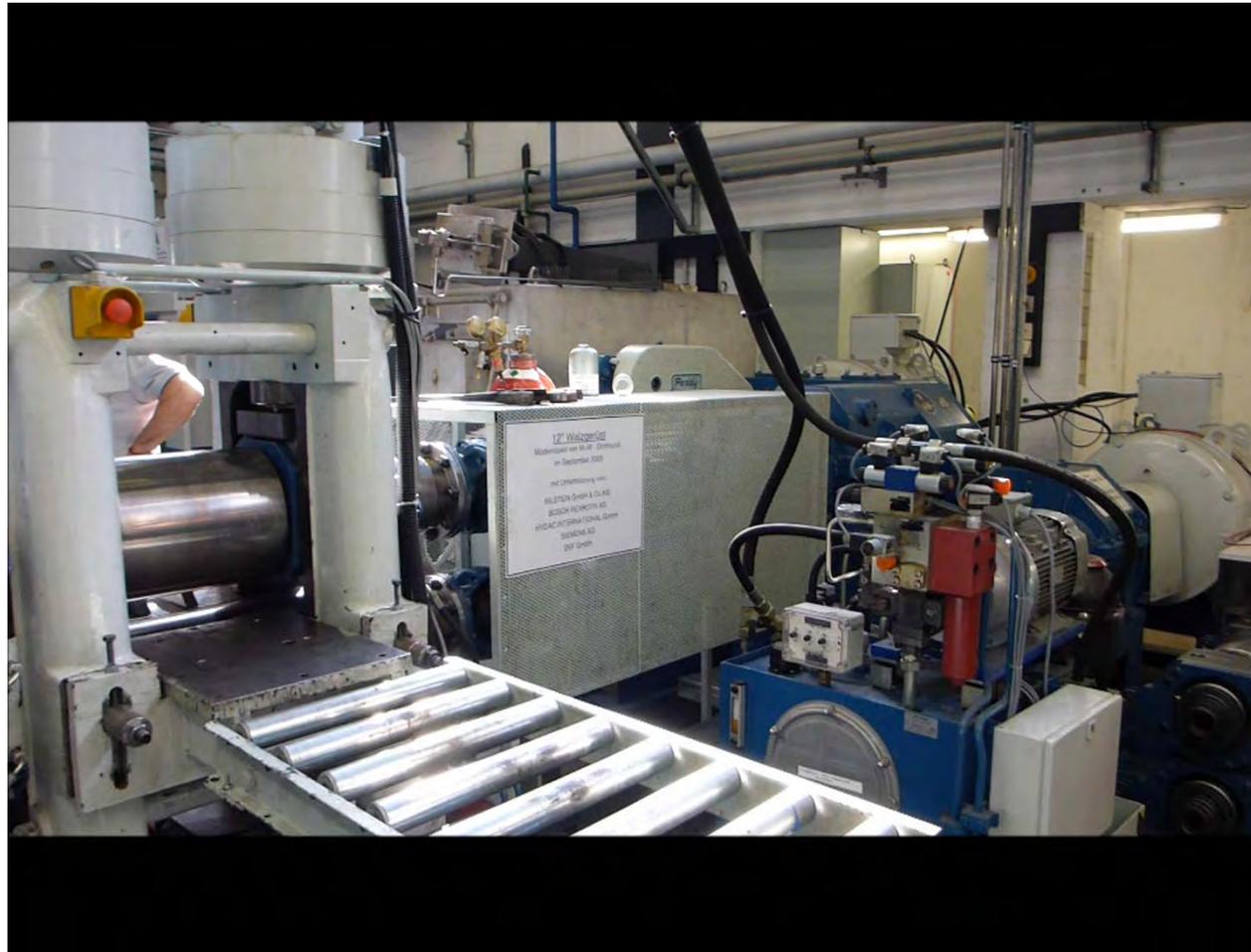
Hot rolling



Before rolling

- Heating of the capsules up to 1100°C

Large lab scale production Hot rolling

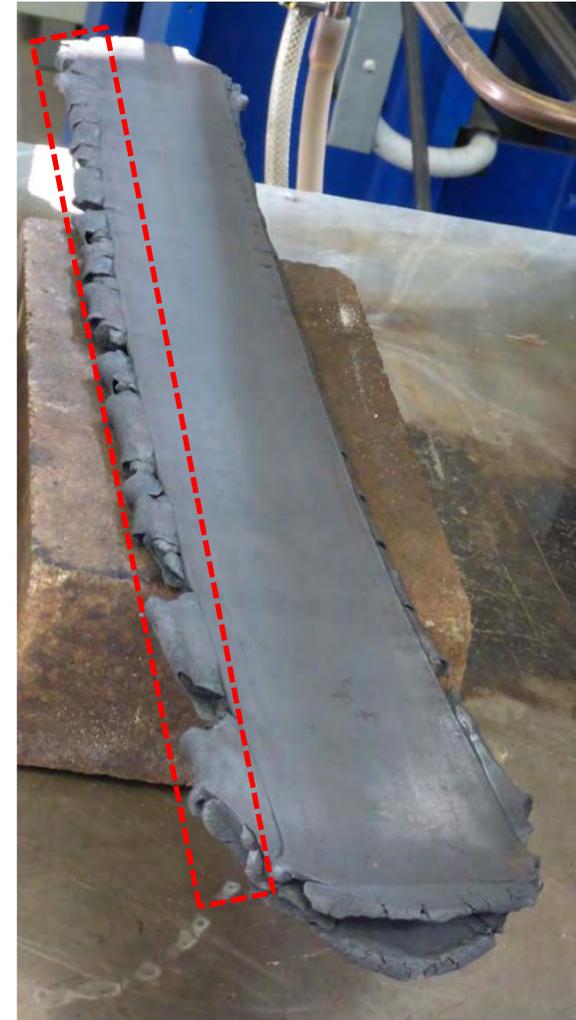


Large lab scale production

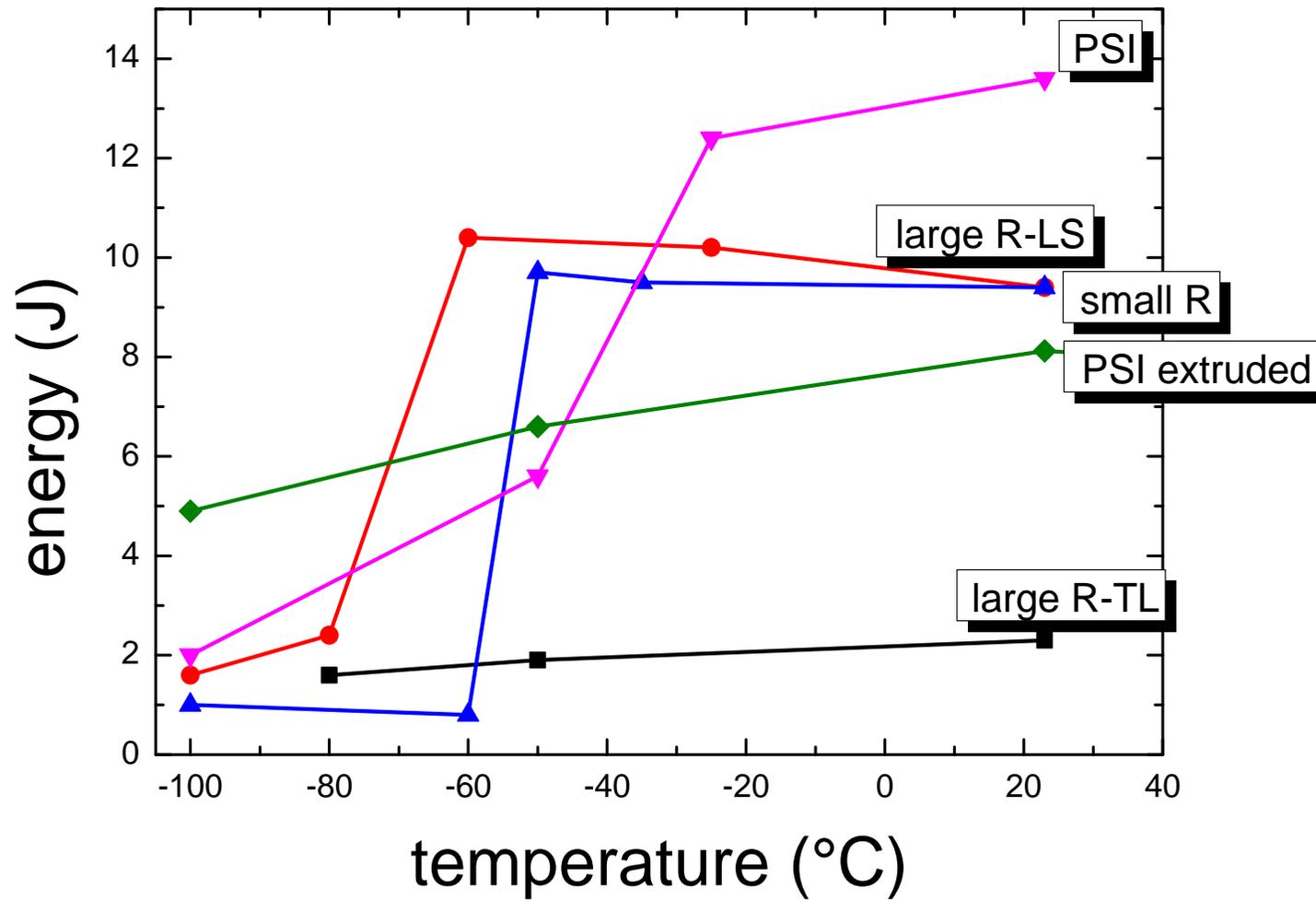
Hot rolling

After rolling

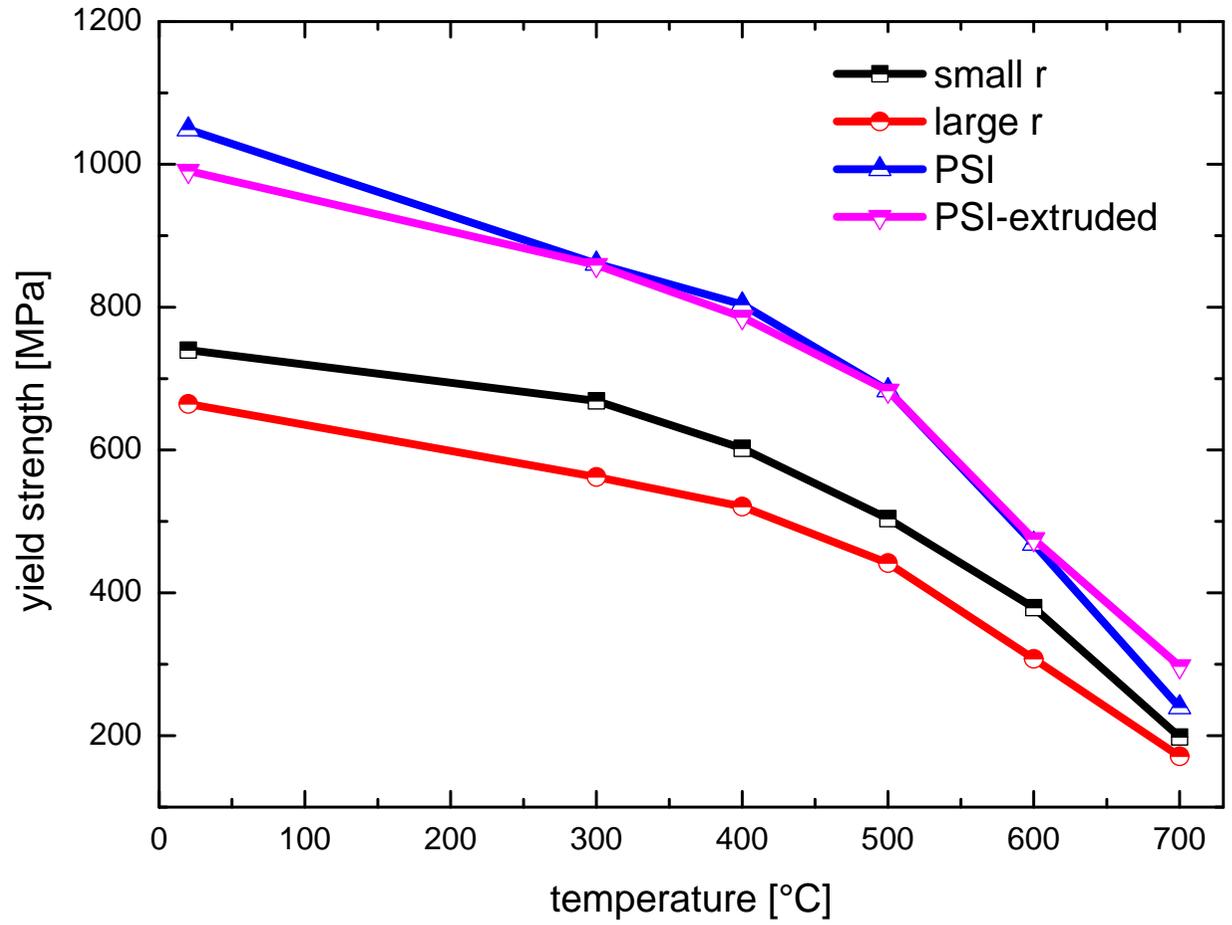
- Final dimensions
 - 600 mm length
 - ~8 mm thickness
- Capsule material still needs to be taken off!
- Fabrication of specimens for mechanical tests



Large lab scale production – Charpy impact

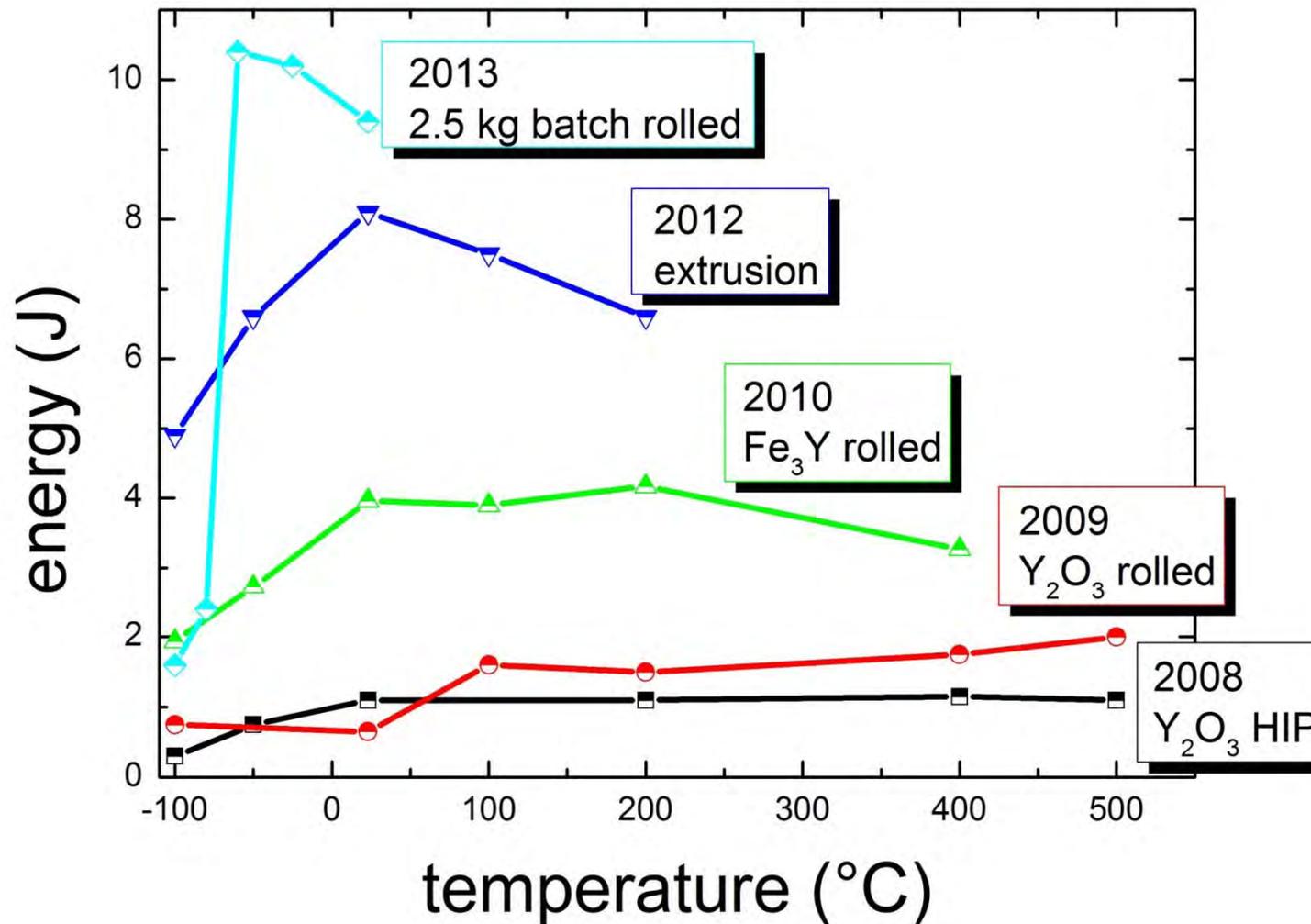


Large lab scale production – Charpy impact

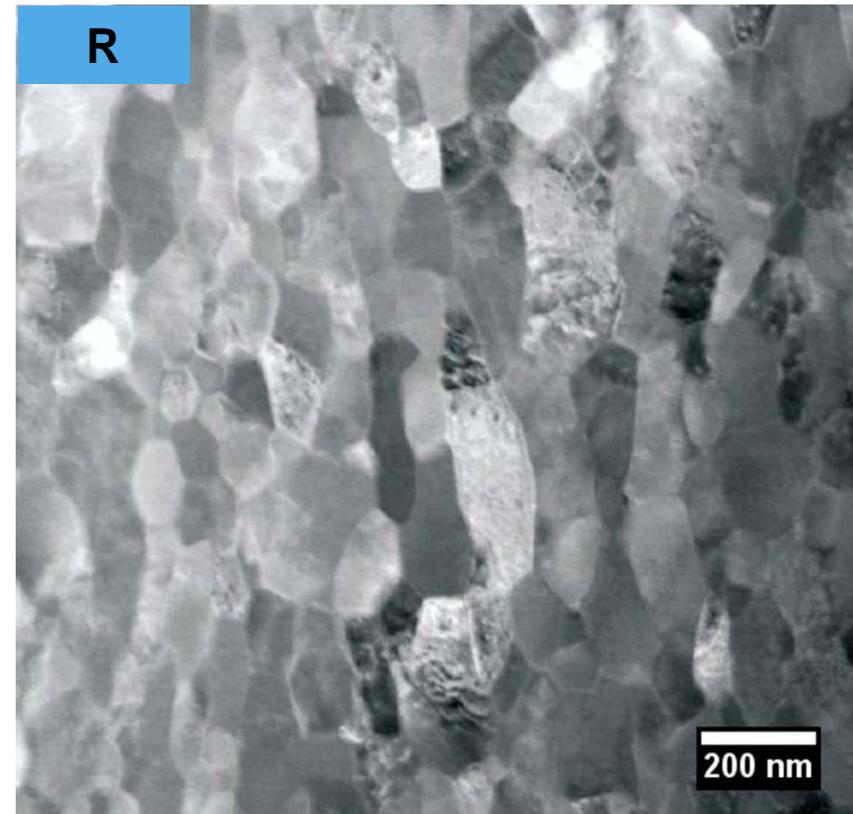
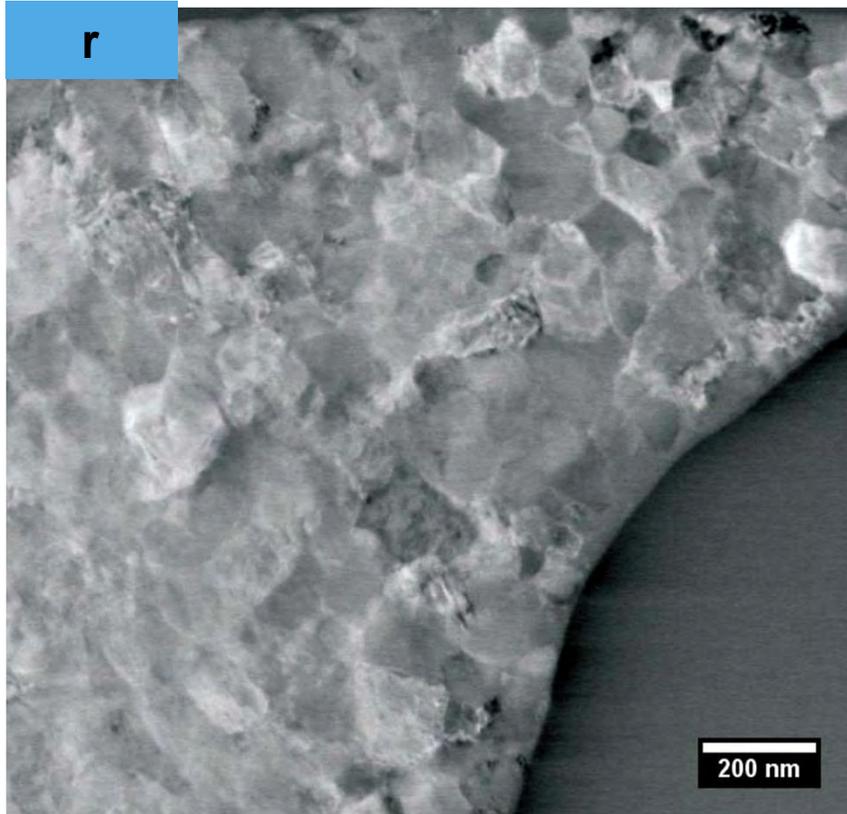


Large lab scale production – Charpy impact

Evolution of 13Cr-ODS steels at KIT



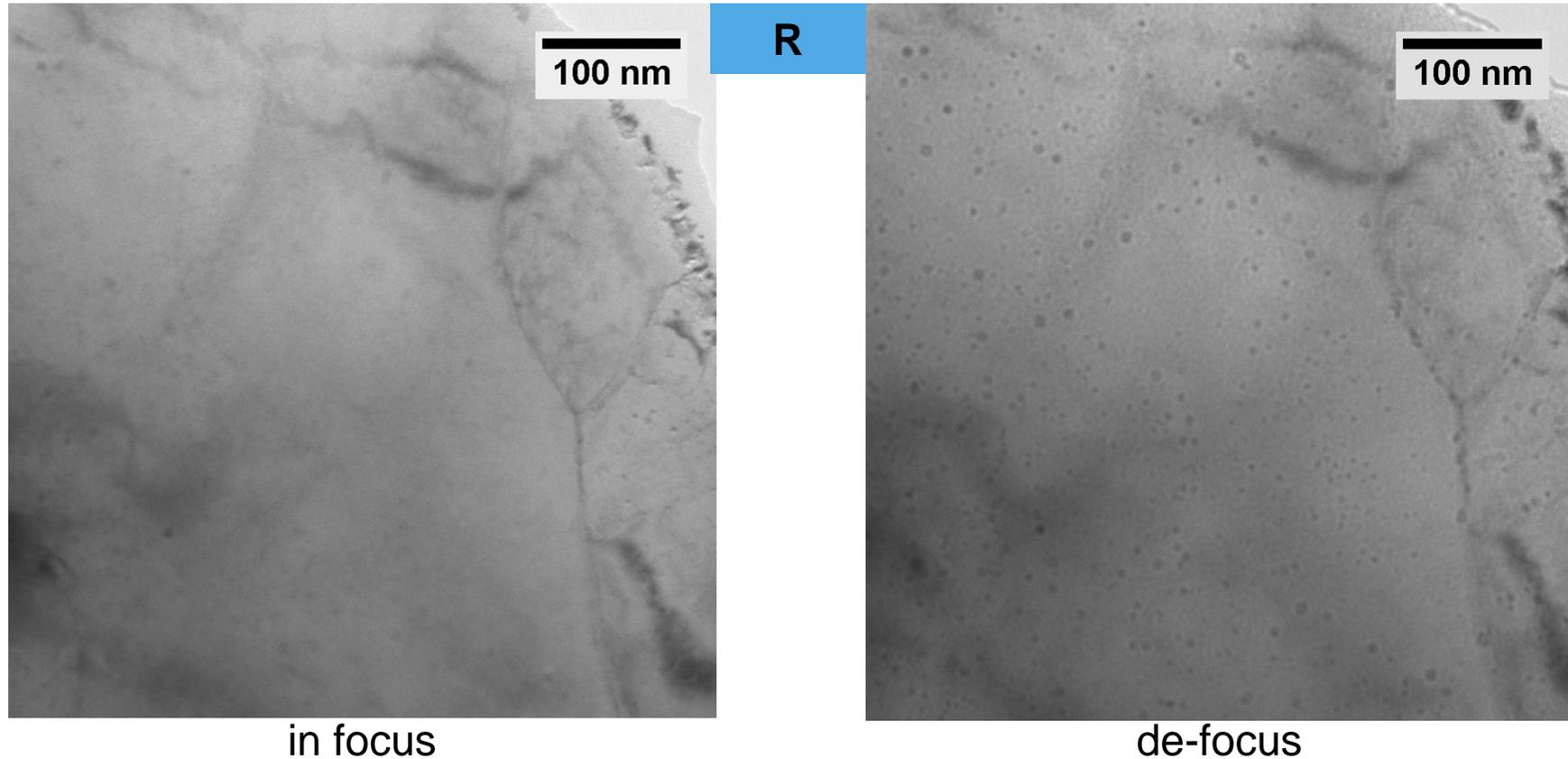
Characterization (as-hipped) TEM-HAADF



As-hipped state

- HAADF images illustrate the grain structure
- grain sizes slightly larger in „R“ material

Characterization (as-hipped) TEM-BF



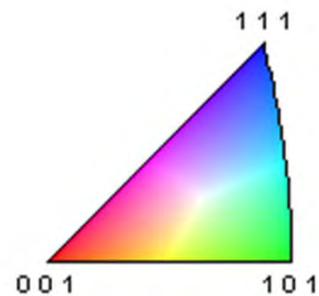
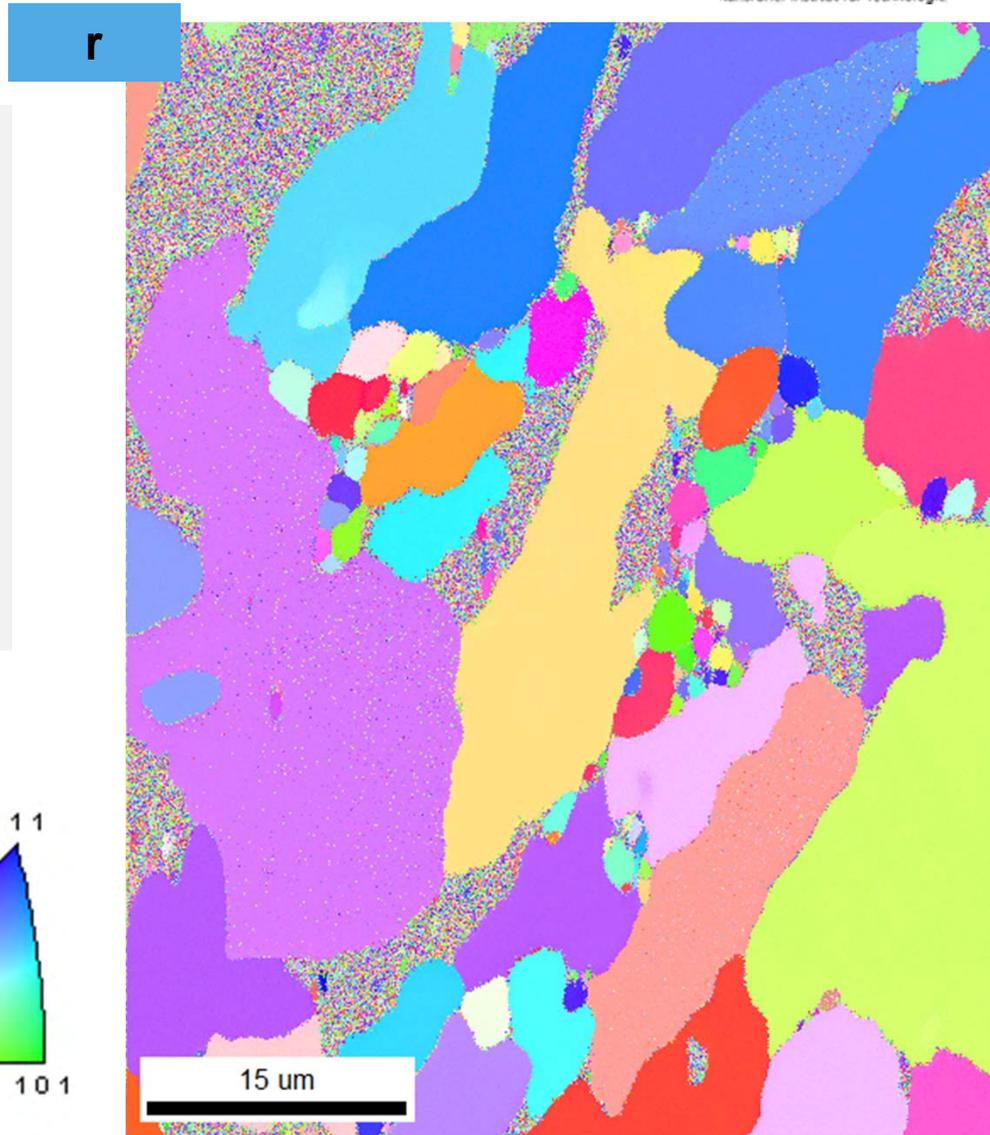
As-hipped state

- BF images show nano-clusters inside the microstructure
- size approx. 10-15 nm

Characterization (as-hipped) EBSD

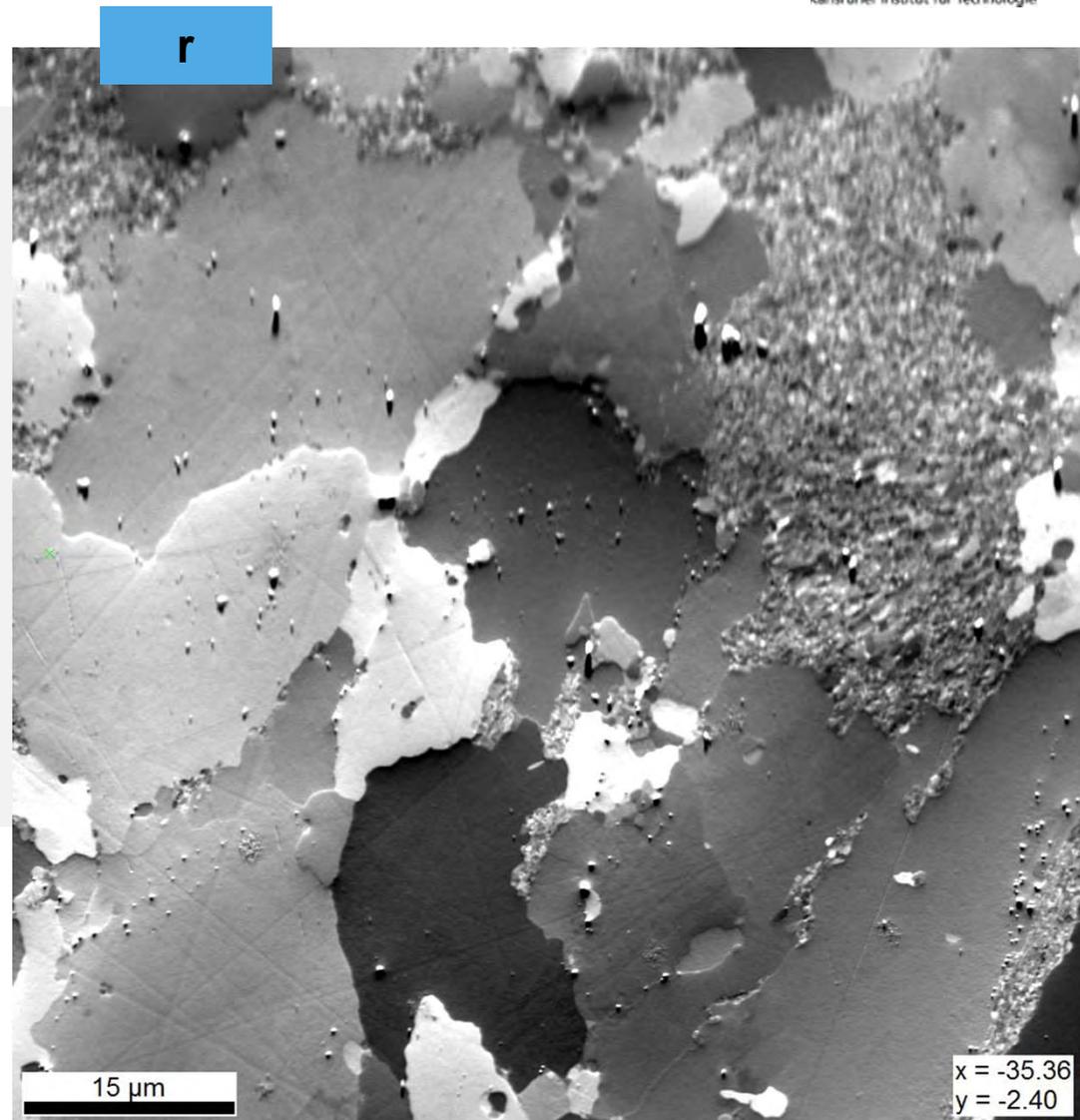
- EBSD Map
 - 50 by 100 μm size
 - 50 nm step-size
 - ~ 2.3 million points

- Maps looks misindexed, but > 99.999% of points are indexed



Characterization (as-hipped) EBSD

- EBSD Map
 - 50 by 100 μm size
 - 50 nm step-size
 - ~ 2.3 million points
- Maps looks misindexed, but > 99.999% of points are indexed
- FSD image also shows small areas



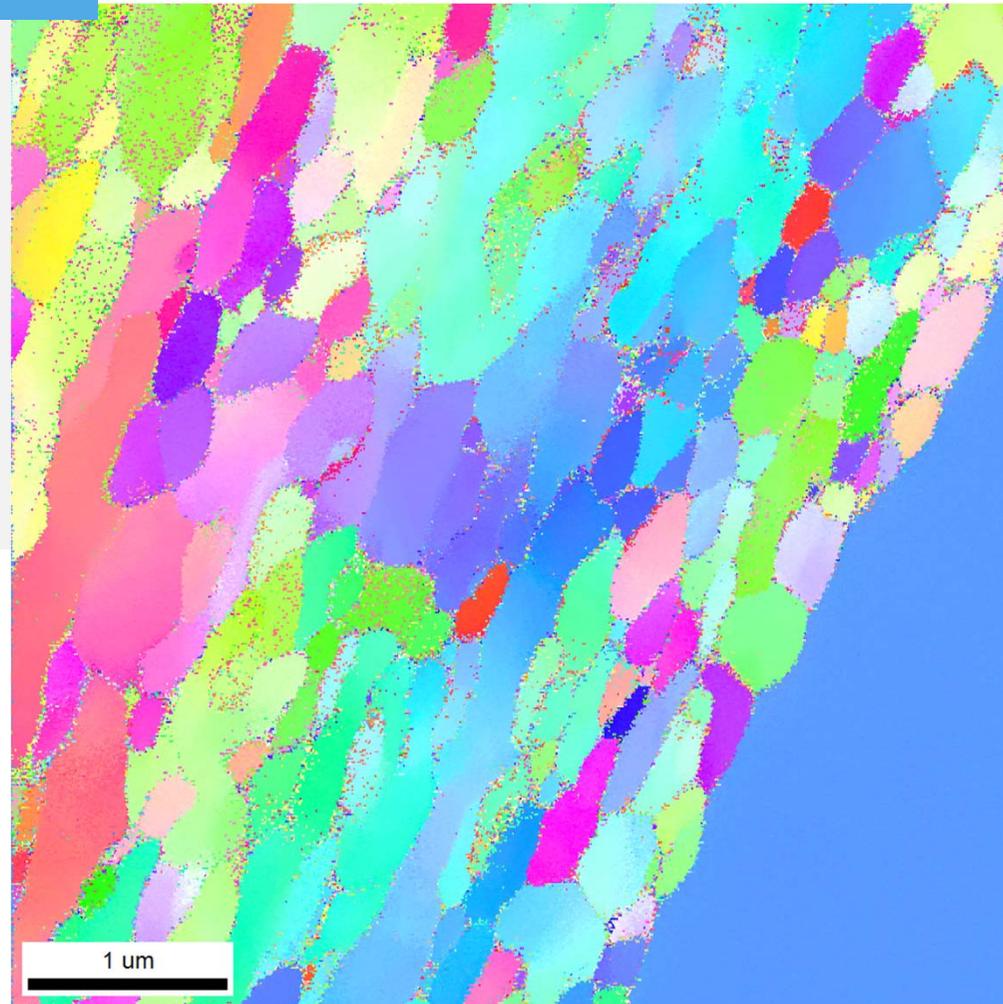
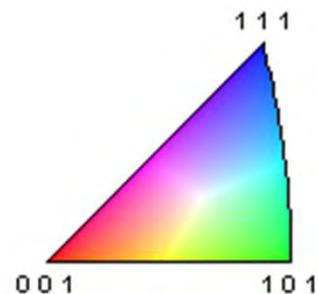
Forward scatter diode on EBSD camera

Characterization (as-hipped) EBSD

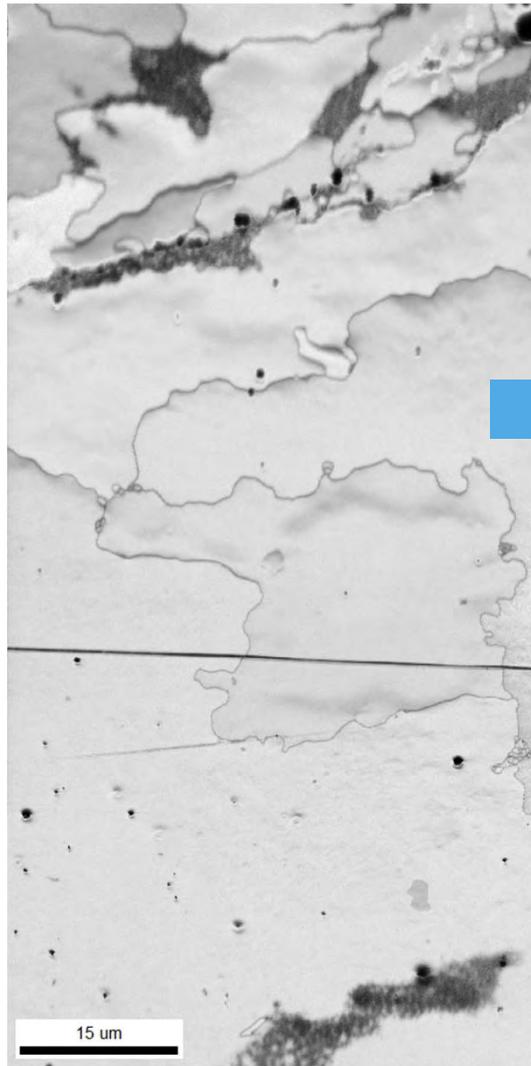
r

- EBSD Map at higher mag.
 - 5 by 5 μm size
 - 10 nm step-size (theoretical)
 - ~ 290.000 points

- Small grained areas become visible

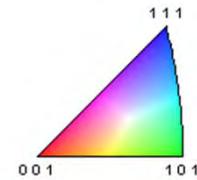


Characterization (as-hipped) EBSD



IQ Map

R



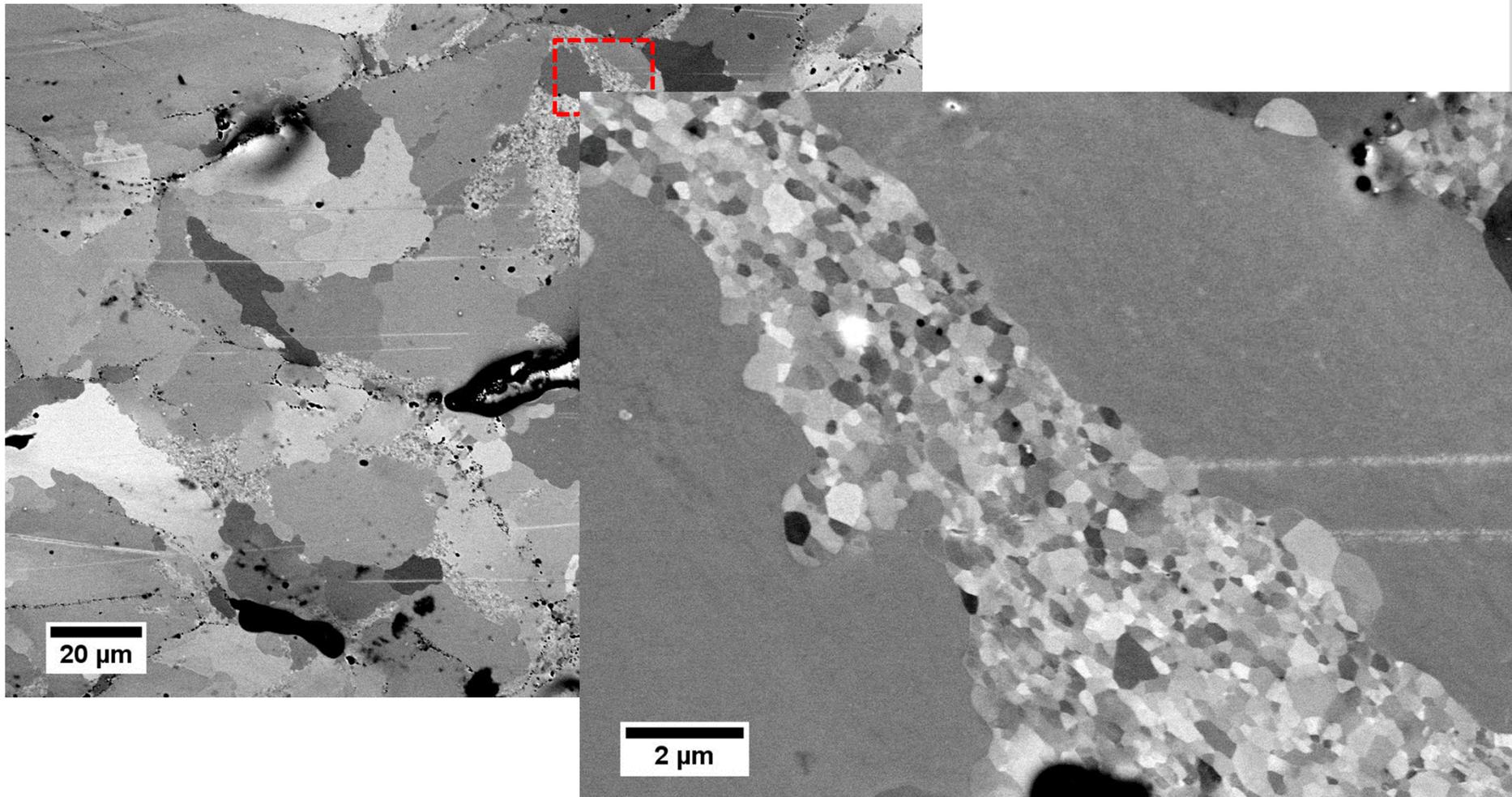
IPF Map



- Map is well indexed
- Grain shapes look odd

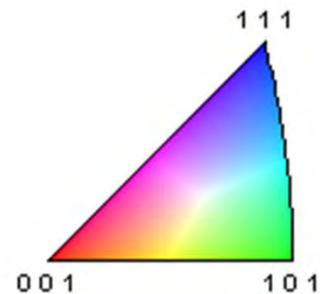
Characterization (as-hipped) EBSD

R

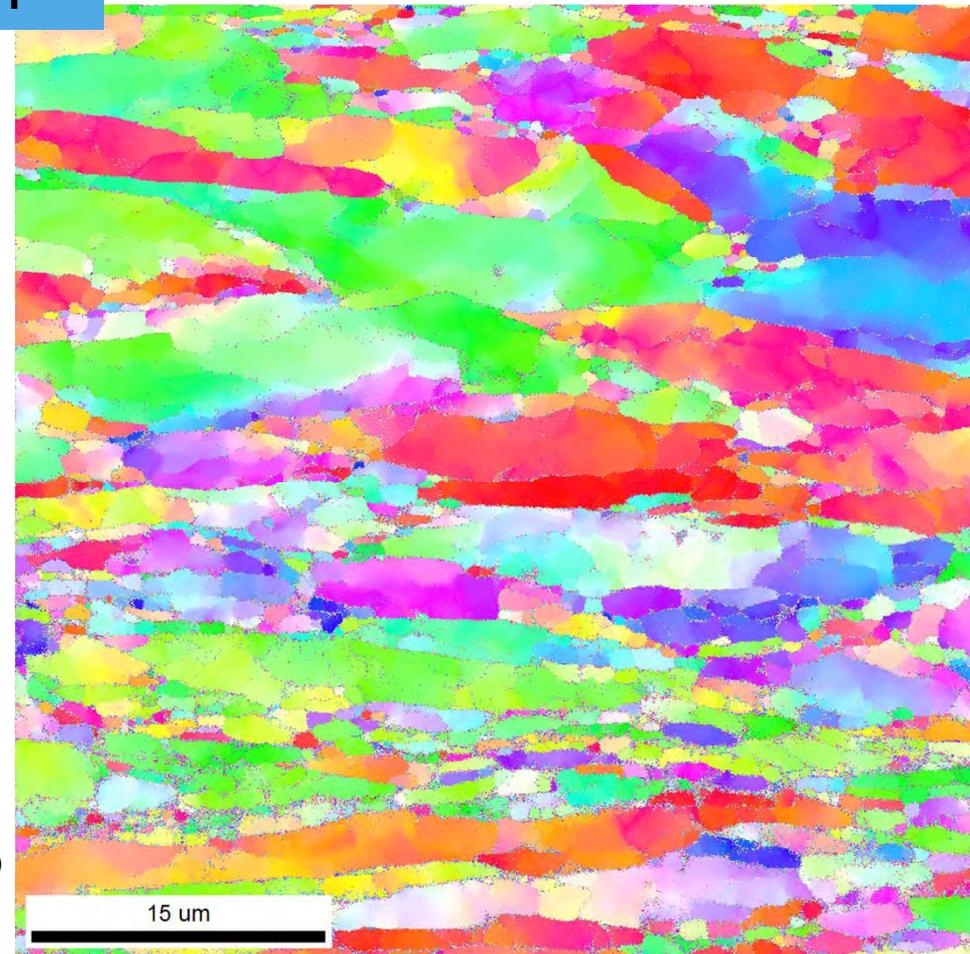


Characterization (as-rolled) EBSD

- Bi-modal grain structure can be observed
- Average grain size might be misleading



r



ND
↑
⊙ RD
TD

Characterization (as-rolled) EBSD

- Production of large batch was successful
- Excellent mechanical properties
- Conclusion from microstructural investigation need to drawn carefully (possible mismatch TEM \leftrightarrow SEM)
- FIB analysis may give more information about grain shapes

Special Thanks

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