Latest insights into additive manufactured materials by a new "Large volume 3D EBSD" (ELAVO 3D) system

Max-Planck-Institut für Eisenforschung GmbH

Stefan Zaefferer

with important contributions of

S.-P. Tsai, Z. Sun (now Singapore)

P. Konijnenberg (now Jülich), T. Griffiths (now Vienna)

Financial support of Nippon Steel and QATM is highly appreciated

NIPPON STEEL

QATM

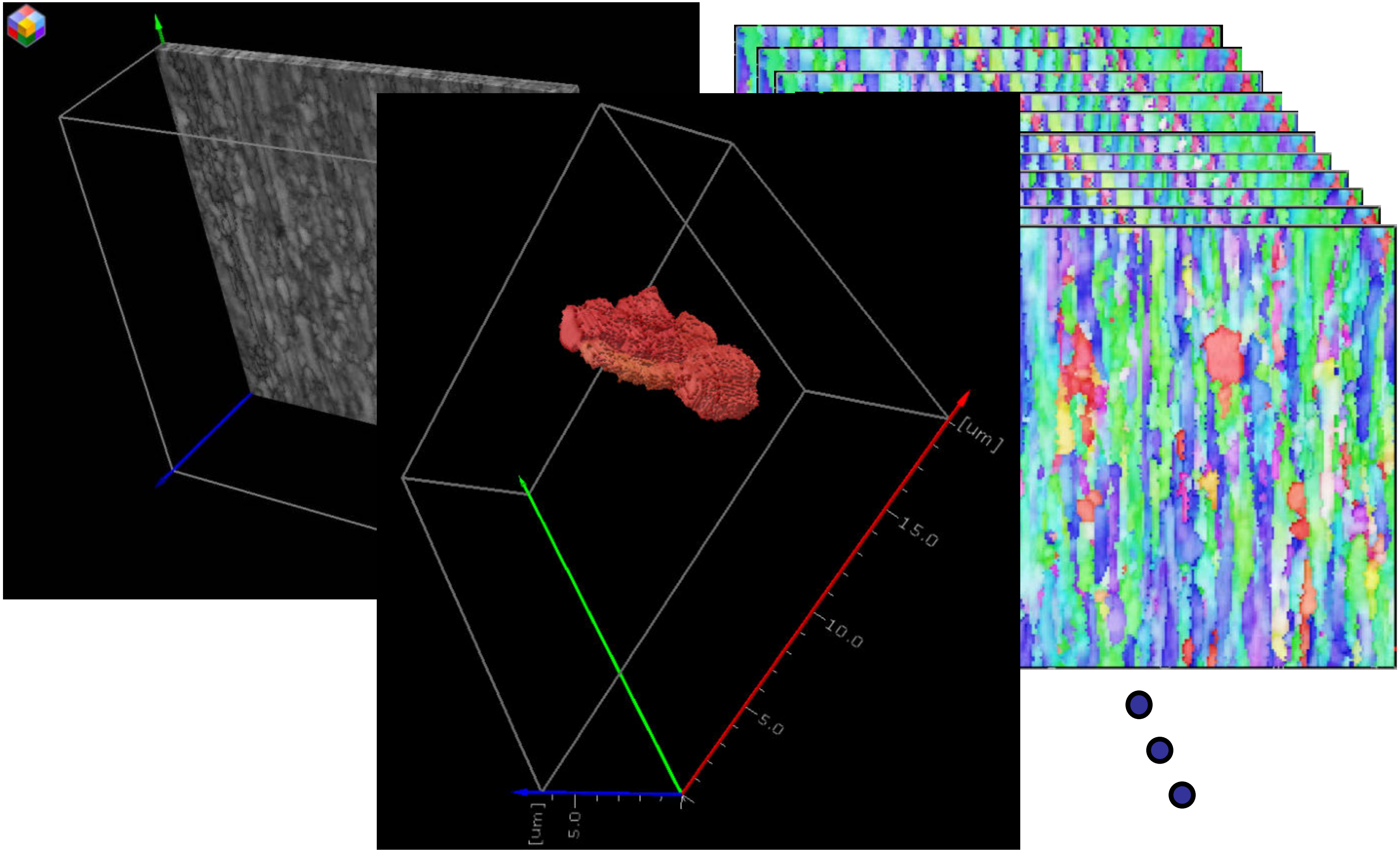
Outline

- Introduction:
  - Why and how doing 3D EBSD?
- Development of a large volume 3D EBSD system
- Application examples:
  - Understanding corrosion of 316 stainless steel
  - Microstructure and texture of AM-produced SS 316
- Conclusions

Outline

- Introduction:
  - Why and how doing 3D EBSD?
- Development of a large volume 3D EBSD system
- Application examples:
  - Understanding corrosion of 316 stainless steel
  - Microstructure and texture of AM-produced SS 316
- Conclusions

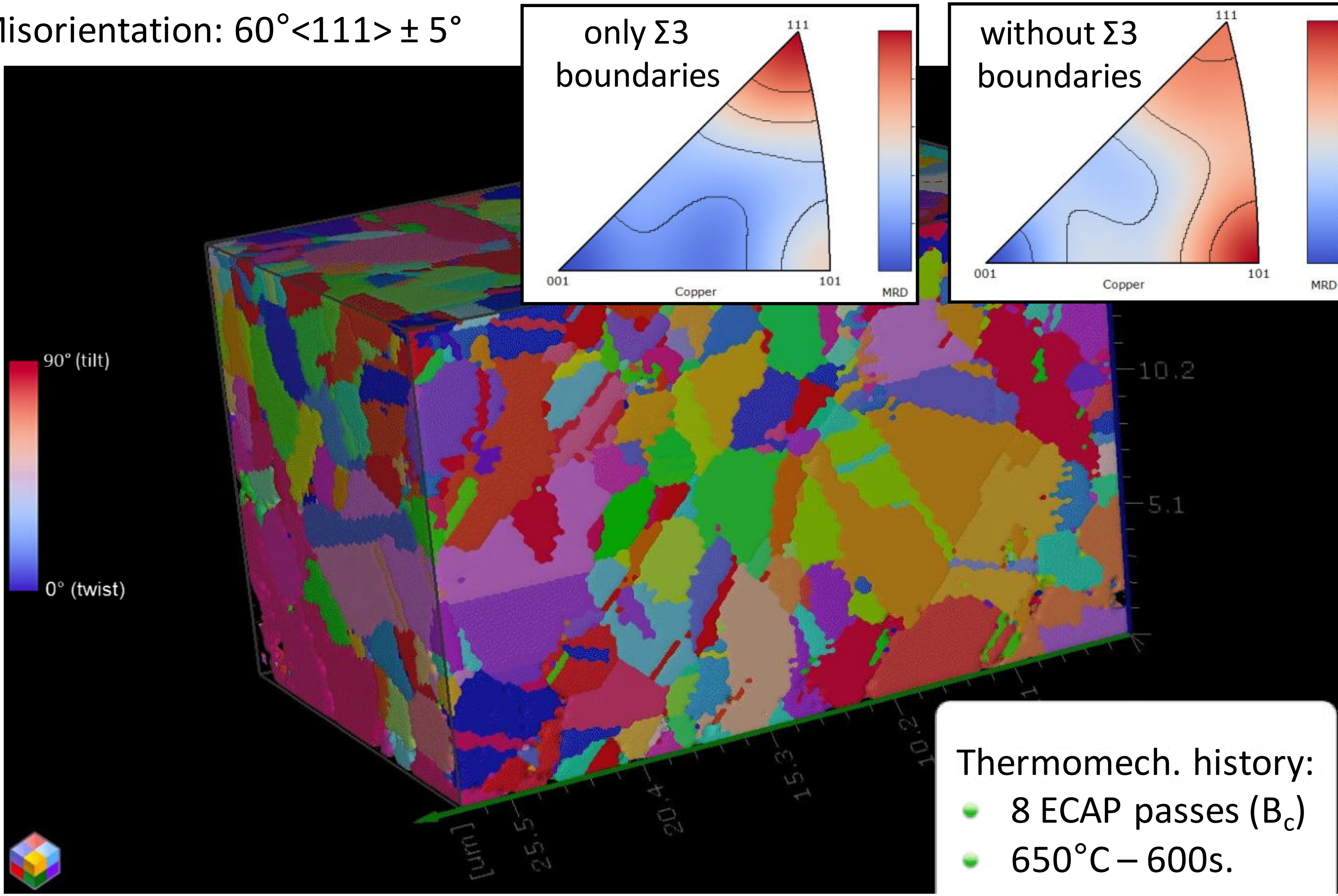
3D orientation microscopy or „3D EBSD“



Serial section by FIB milling, LASER ablation, automated mechanical polishing

Example: grain boundaries in Cu-0.17wt%Zr

Misorientation: 60°<111> ± 5°

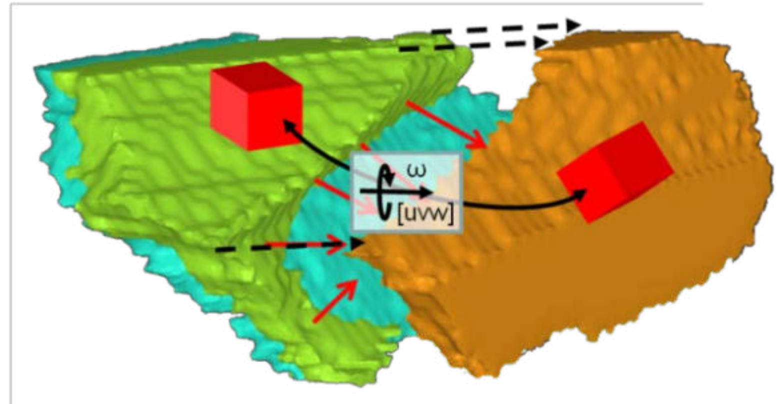


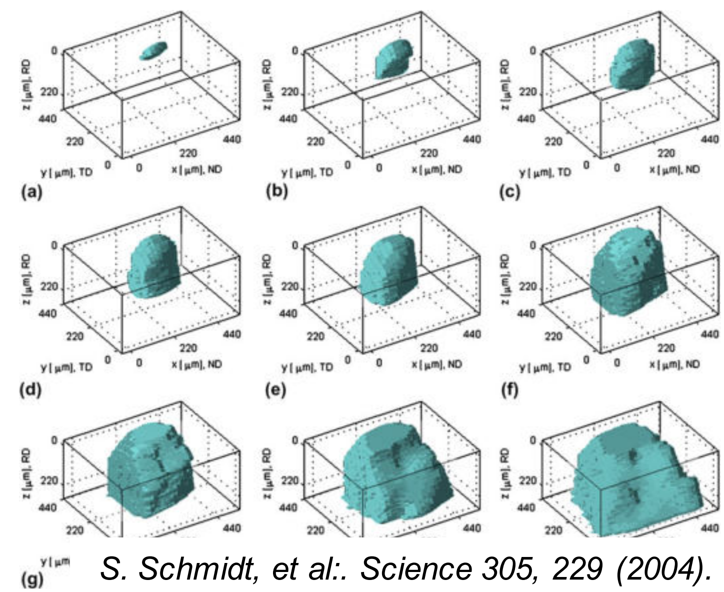
Thermomech. history:

- 8 ECAP passes (B_c)
- 650°C – 600s.

Potential applications of 3D EBSD

- Fundamental questions
  - Character of grain and phase boundaries (morphology, crystallography, and energy)
  - Structure-properties relations of interfaces
  - Microstructure of 3-dimensional deformation fields
  - Location of nucleation and growth morphology of recrystallization and phase transformation products
  - Input for theoretical simulations

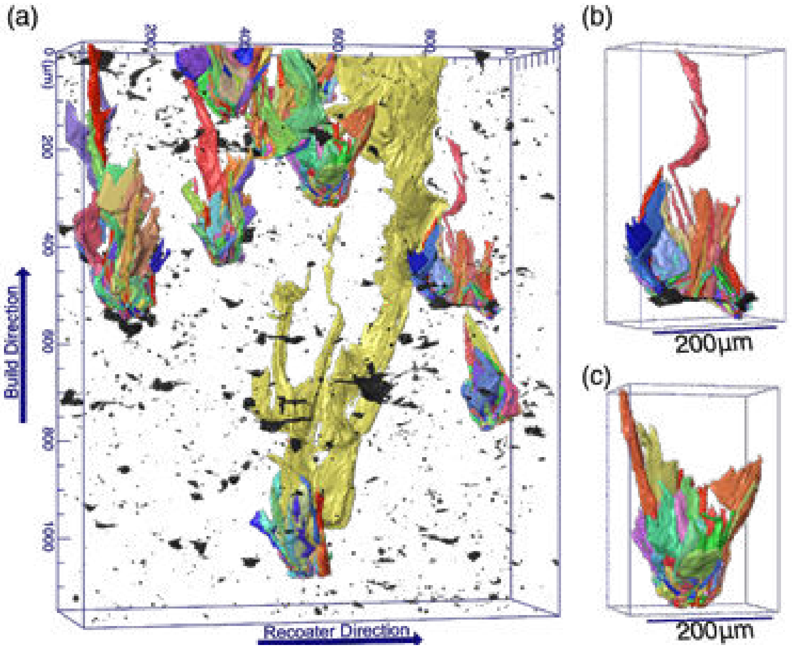




S. Schmidt, et al:. Science 305, 229 (2004).

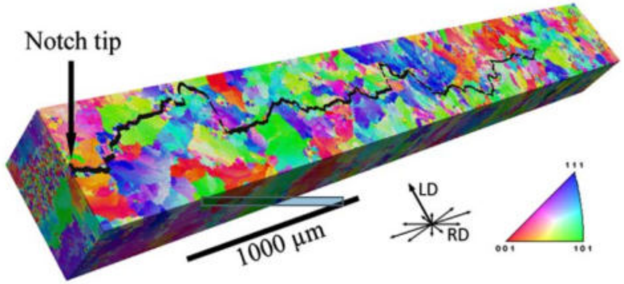
Potential applications of 3D EBSD

- Engineering questions
  - Microstructure and properties of additive manufactured materials
  - Morphology and crystallographic relation of pores (e.g. for H-reduction of iron ore)
  - Morphology and microstructure relation of cracks (fundamental, cause of damage investigations, quality control)
  - Crystallography and morphology of defects in semiconductors



Rowenhorst et al., Cur. Op. Solid State Mat. Sci. (2020)

Pirgazi et al, Mat. Character. 90 (2014), 13



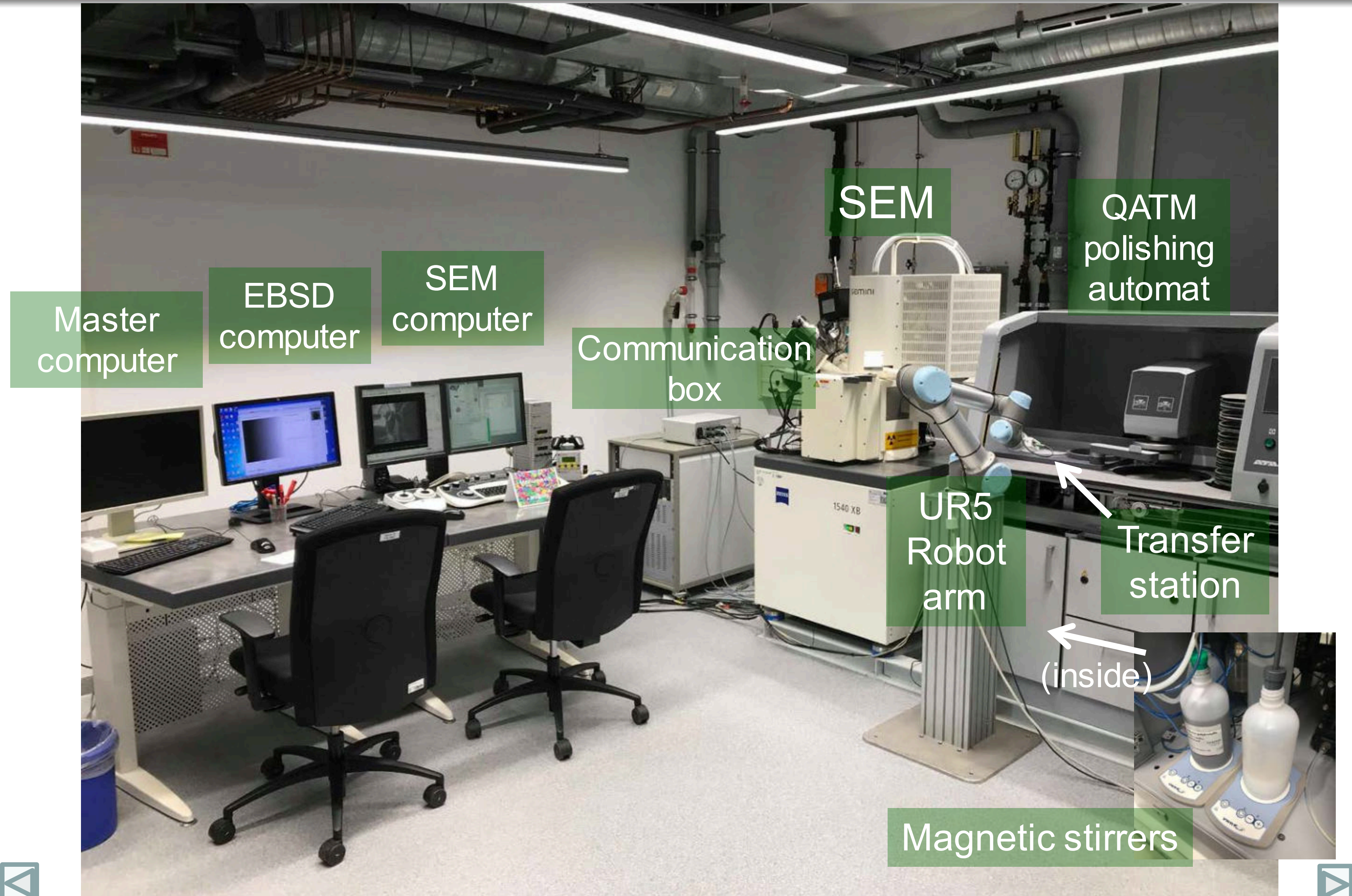
Outline

- Introduction:
  - Why and how doing 3D EBSD?
- Development of a large volume 3D EBSD system
- Application examples:
  - Understanding corrosion of 316 stainless steel
  - Microstructure and texture of AM-produced SS 316
- Conclusions

Alternative sectioning methods

- Automated mechanical or chemical-mechanical polishing *(Uchic in: S. Ghosh and D. Dimiduk (eds.), Computational Methods for Microstructure-Property Relationships, c Springer Science+Business Media, 2011)*
  - large area/volume, no beam damage,
  - difficult to automate, depth accuracy
- Combination of laser ablation and FIB milling *(Echlin et al., A new TriBeam system for three-dimensional multimodal materials analysis, Rev. Sci. Instrum. 83, 023701 (2012))*
  - large area/volume,
  - depth accuracy, beam damage
- Milling with a Plasma-FIB (Tescan Fera) *(Hrnčíř et al., Novel plasma FIB/SEM for high speed failure analysis, 3D tomography and other applications, Proc. EMC 2012, Manchester)*
  - large area/volume, less beam damage than Ga-FIB?
  - surface quality

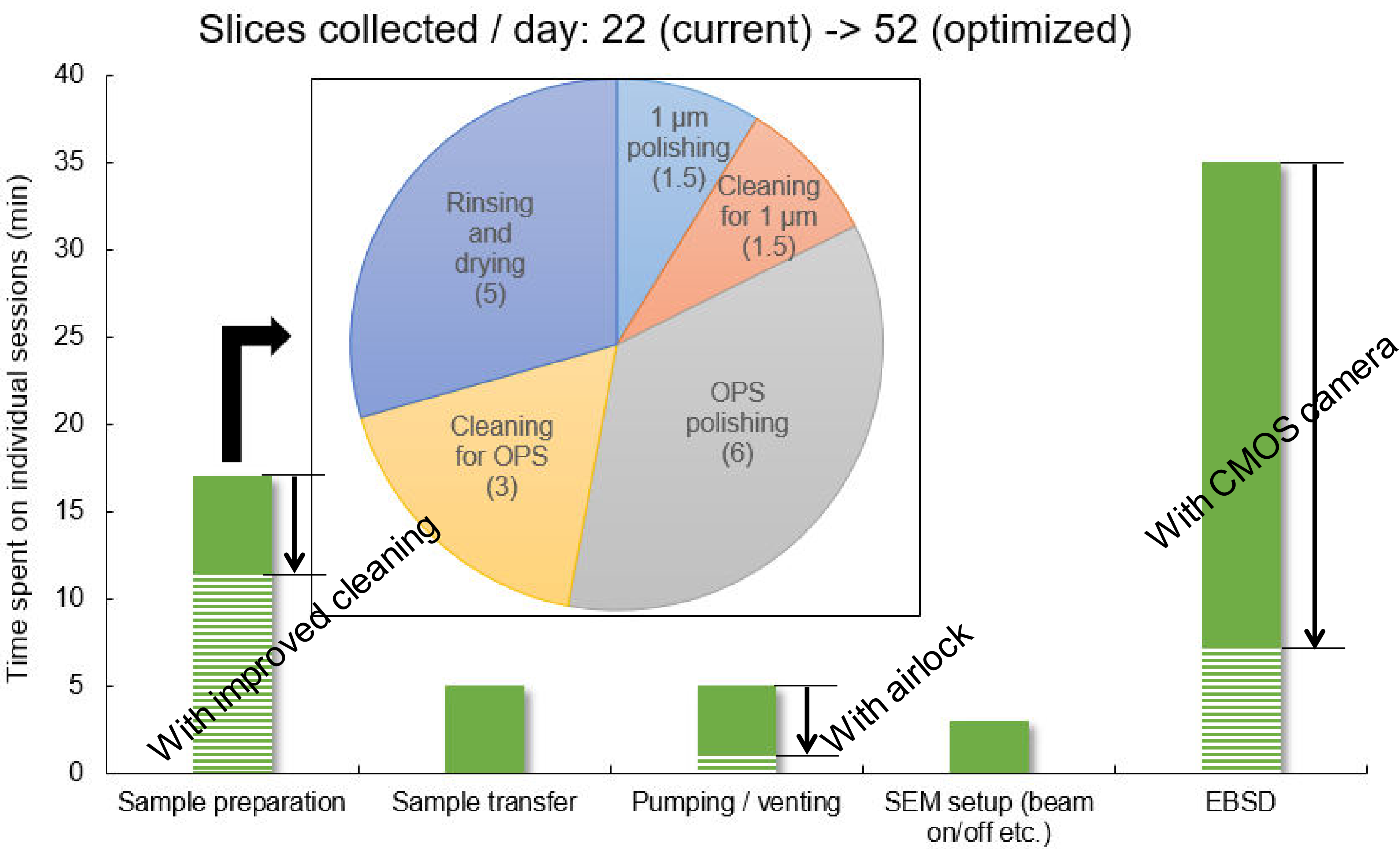
ELAVO 3D: System overview



A few numbers on our system

- Very large (up to ~4 cm^2), perfectly flat and defect free surfaces
- Large volume measurable:
  - 1 mm x 1 mm x 0.3 mm at 1 μm^3 resolution
- About 30 slices per day of 1000 x 1000 pixel at 1 µm step size
- Smallest slice thickness currently ~ 0.5 µm, to be reduced
- Up to 30.000.000 EBSD data points per day, each consisting of up to 10 numbers
- High reliablility:
  - < 1° rotation, < 30 µm shift, rare errors (currently about every 50 slices, to be reduced in near future)

Time consumation per measurement cycle

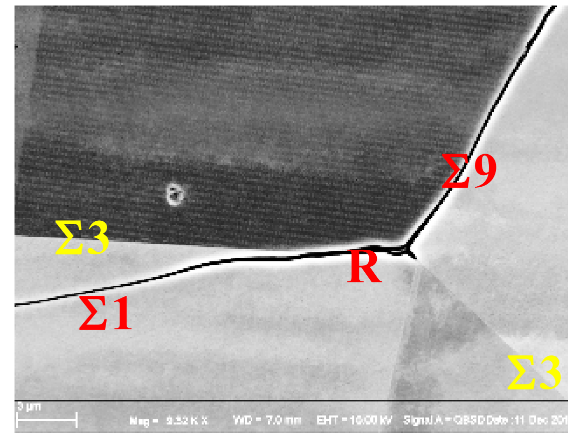


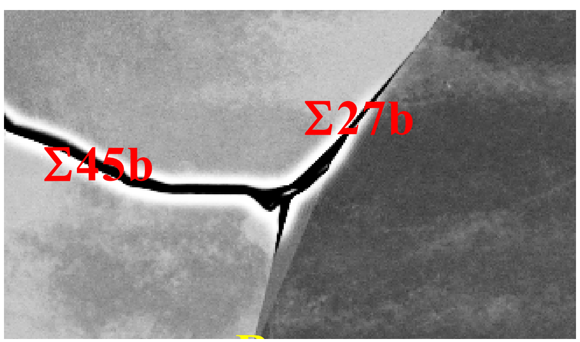
Outline

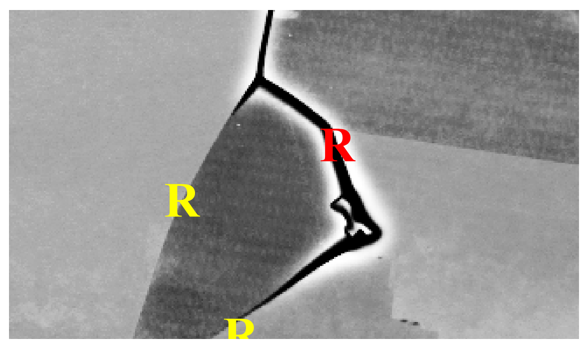
- Introduction:
  - Why and how doing 3D EBSD?
- Development of a large volume 3D EBSD system
- Application examples:
  - Understanding corrosion of 316 stainless steel
  - Microstructure and texture of AM-produced SS 316
- Conclusions

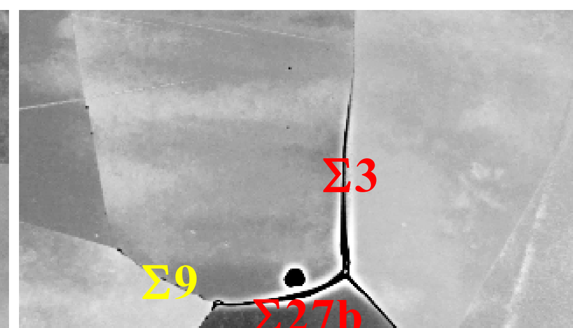
Corrosion of boundaries of stainless steel – why and how?

- Stainless steels (e.g. 304 „V2A“, 316) are made to be corrosion resistant
  - Formation of a protective Cr-oxide layer
- But they are not always
  - in agressive media e.g. H_2O_2-HF
  - after wrong heat treatment: sensitization
- This may lead to significant economic and ecological costs





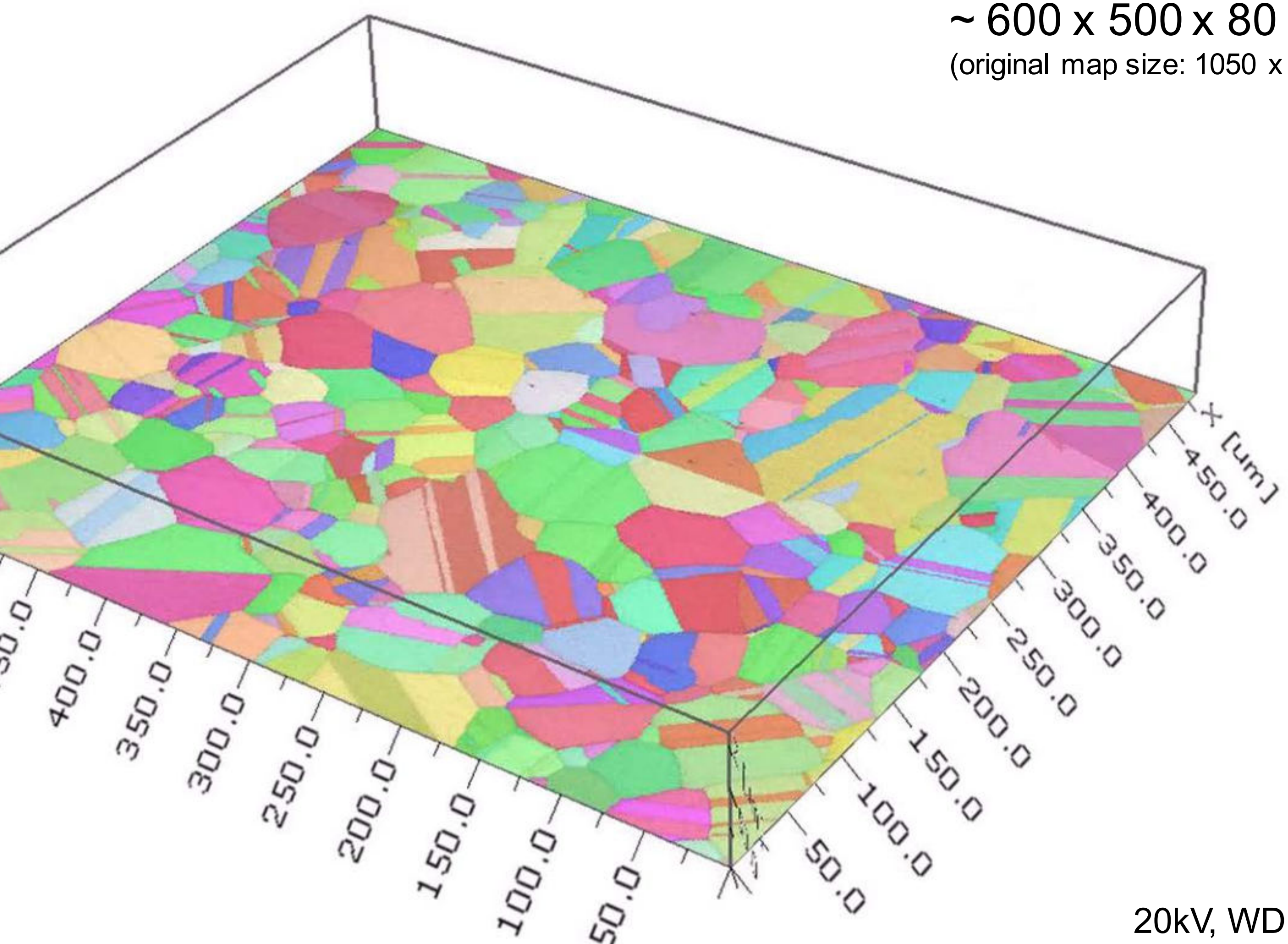


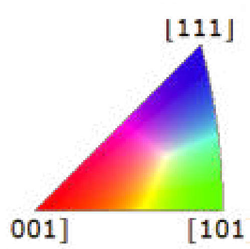


Corrosion behaviour is not (only) depending on misorientation
What is the role of the grain boundary plane?

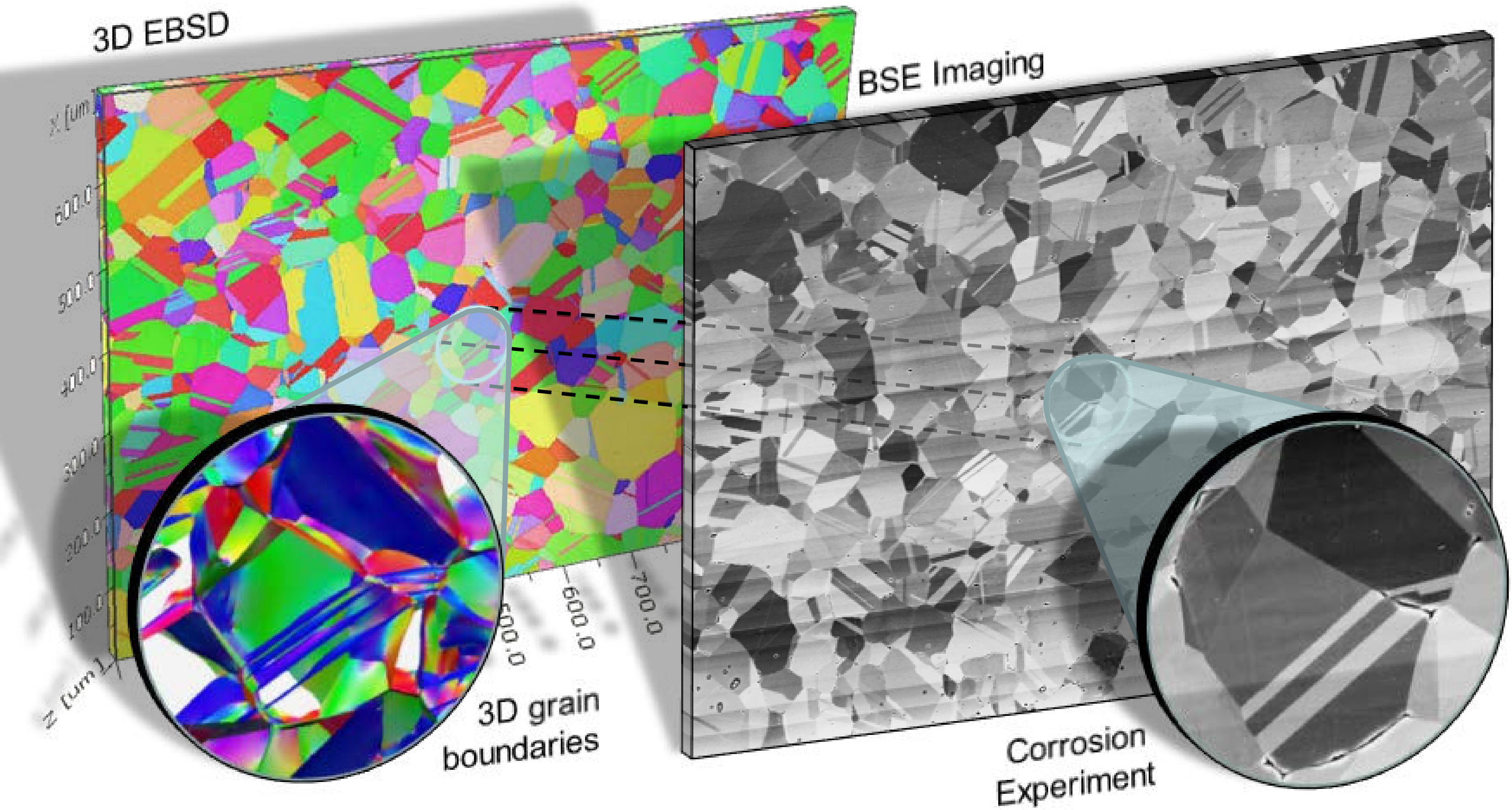
4 x 20 (80) slices of 3D EBSD (IPF + IQ)

~ 600 x 500 x 80 µm^3
(original map size: 1050 x 850 µm^2)





20kV, WD19
Camera bin: 8*8
Gain: 15.68
Exposure: 2.09



3D EBSD
BSE Imaging
X [um]
600.0
500.0
400.0
300.0
200.0
100.0
Z [um]
500.0
600.0
700.0
3D grain boundaries
Corrosion Experiment

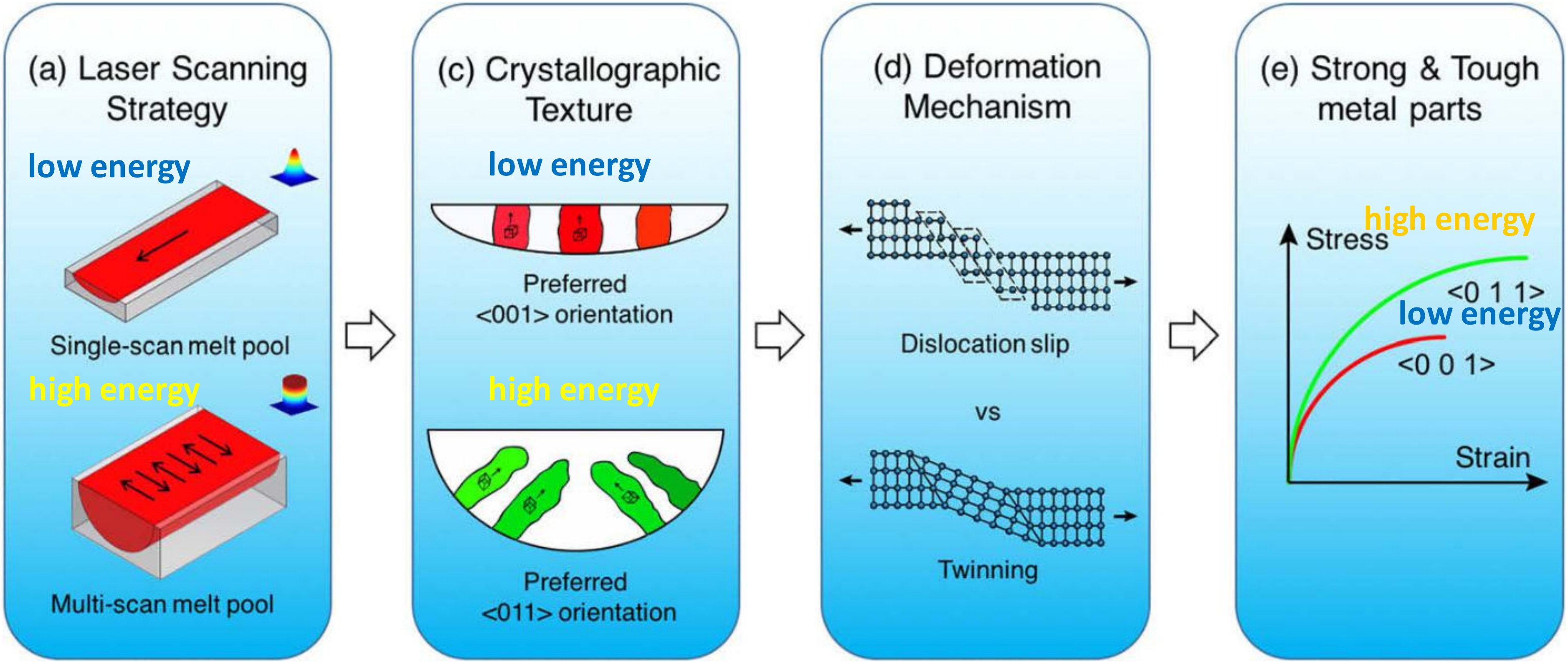
Summary on grain boundary corrosion

- The corrosion of grain boundaries is controlled by the combination of structural and compositional features:
  - Grain boundary structure determines diffusion rate and precipitation nucleation rate
  - Diffusion and nucleation determines rate of growth of precipitations
  - Precipitation growth determines Cr-depletion of grain boundaries
  - Cr-depletion determines corrosion rate
- $\Sigma 5$ and $\Sigma 7$ GBs appear to be prototypes of grain boundaries with particular properties

Outline

- Introduction:
  - Why and how doing 3D EBSD?
- Development of a large volume 3D EBSD system
- Application examples:
  - Understanding corrosion of 316 stainless steel
  - Microstructure and texture of AM-produced SS 316
- Conclusions

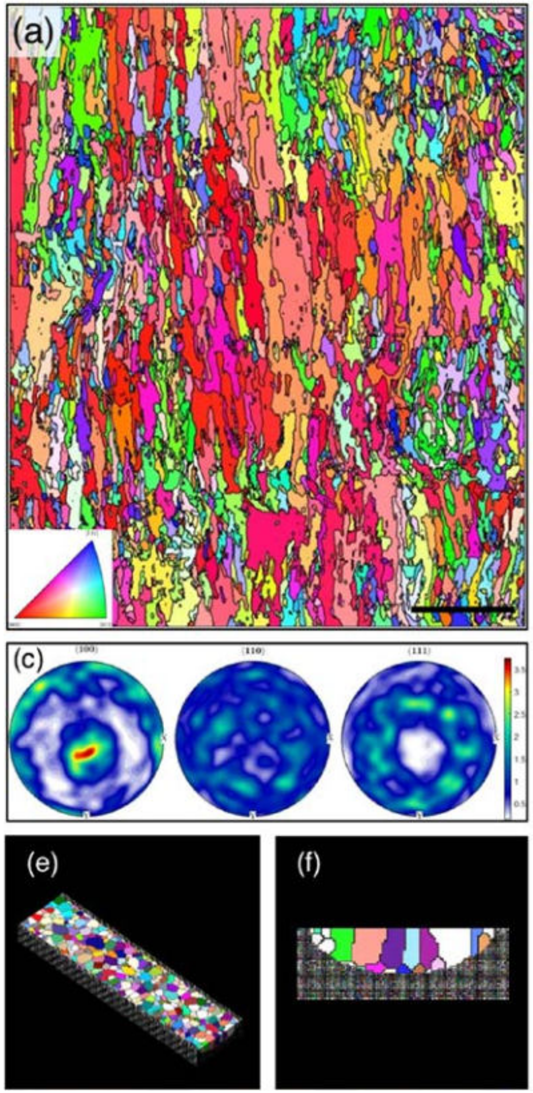
Controlling texture and microstructure in AM parts

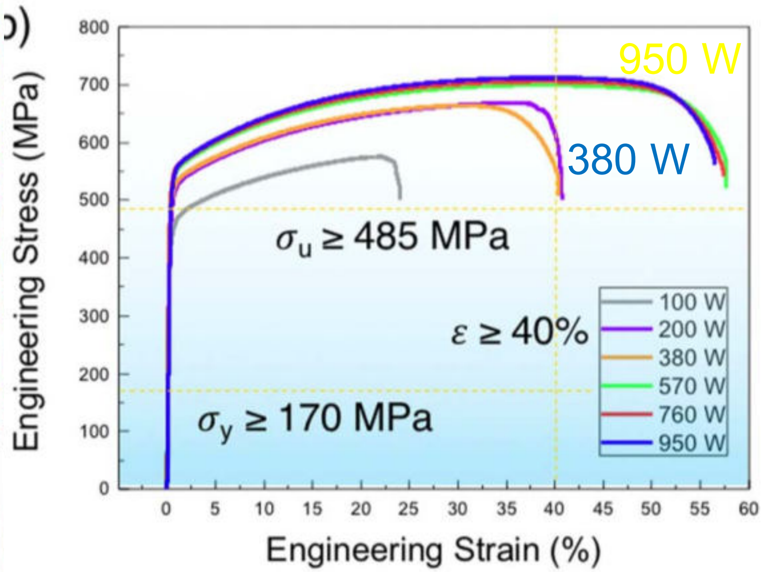


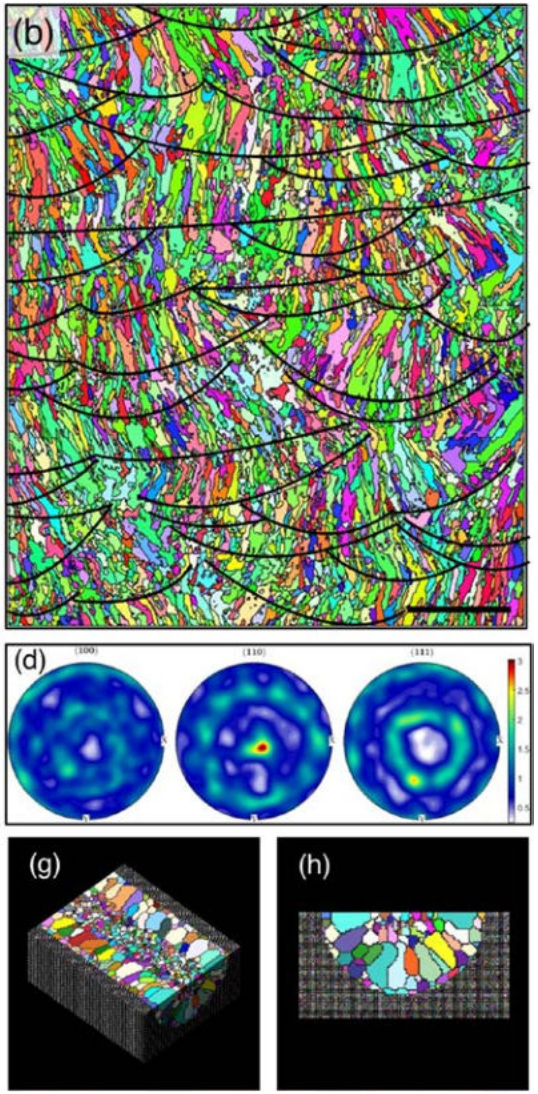
Zhongji Sun et al.: *Simultaneously enhanced strength and ductility for 3D-printed stainless steel 316L by selective laser melting,* NPG Asia Materials (2018) 10: 127–136

Mechanical properties and microstructure

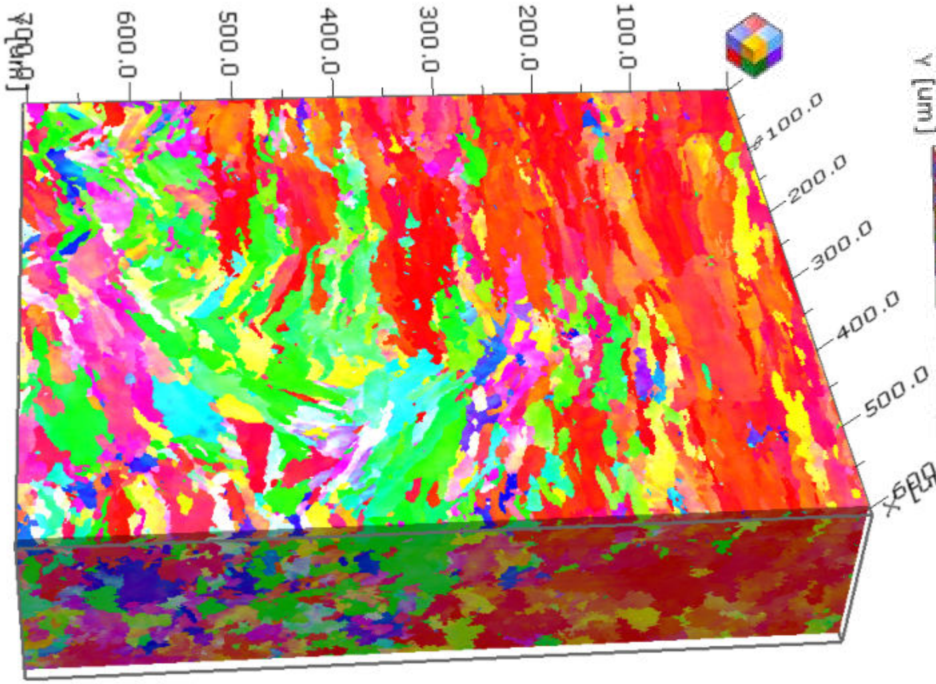
380 W **950 W**

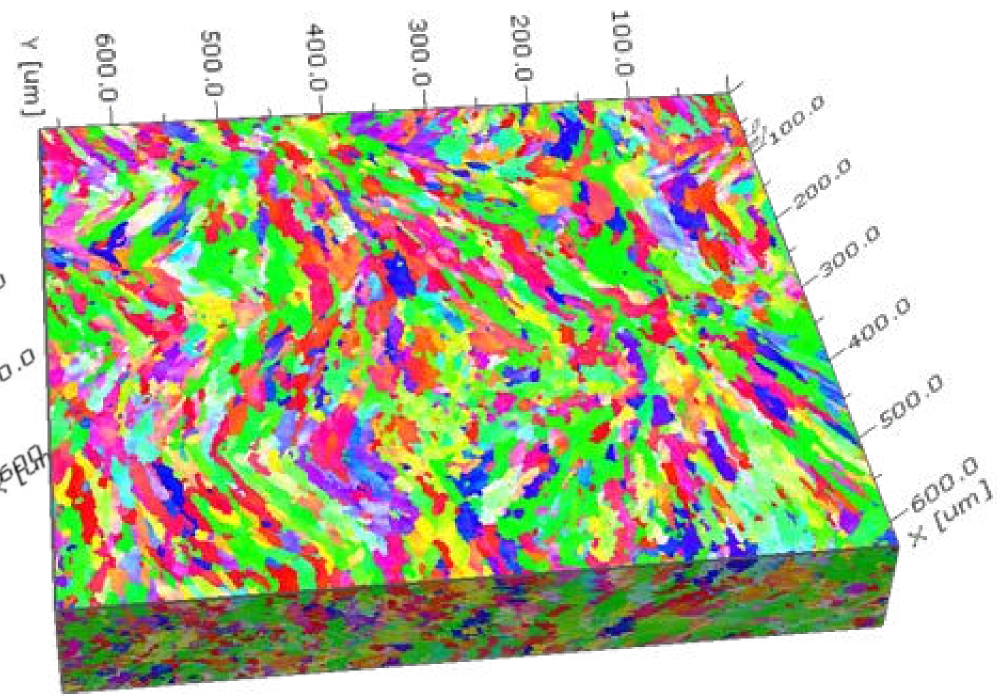




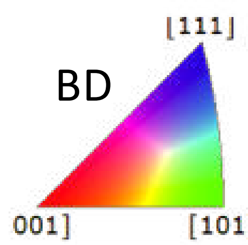


ELAVO 3D results



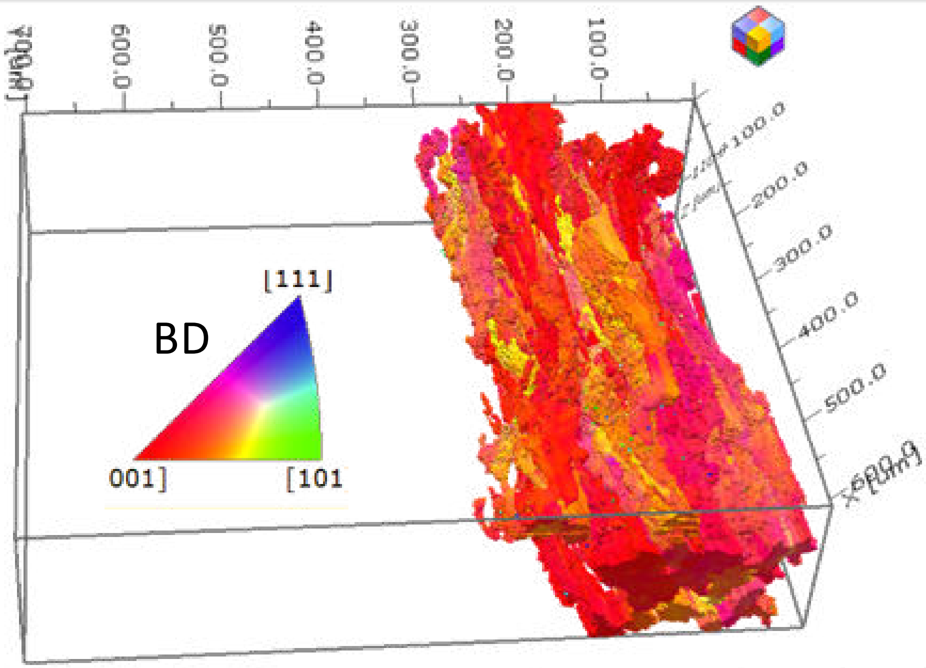


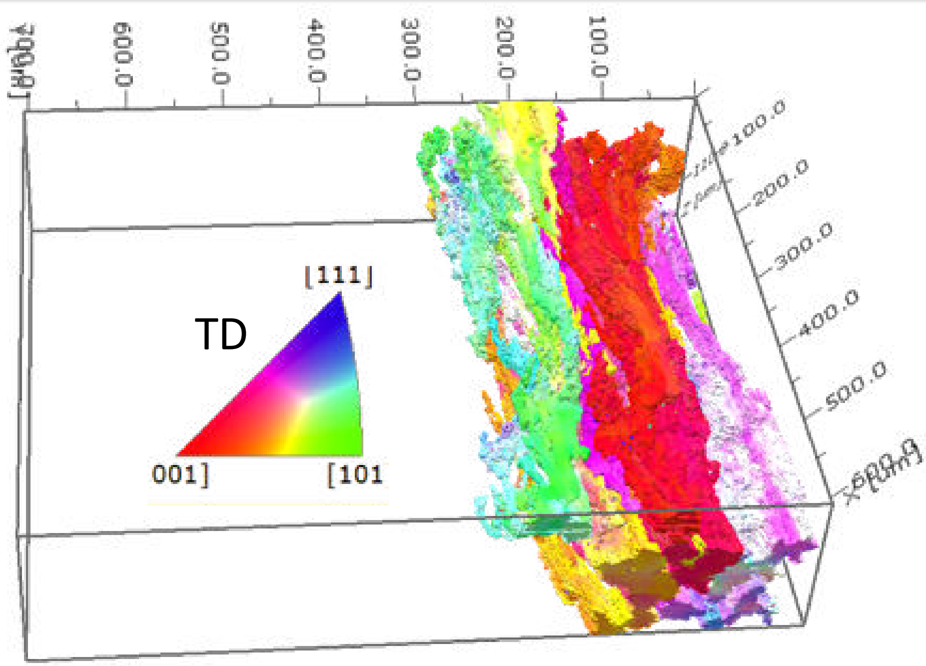
380 W



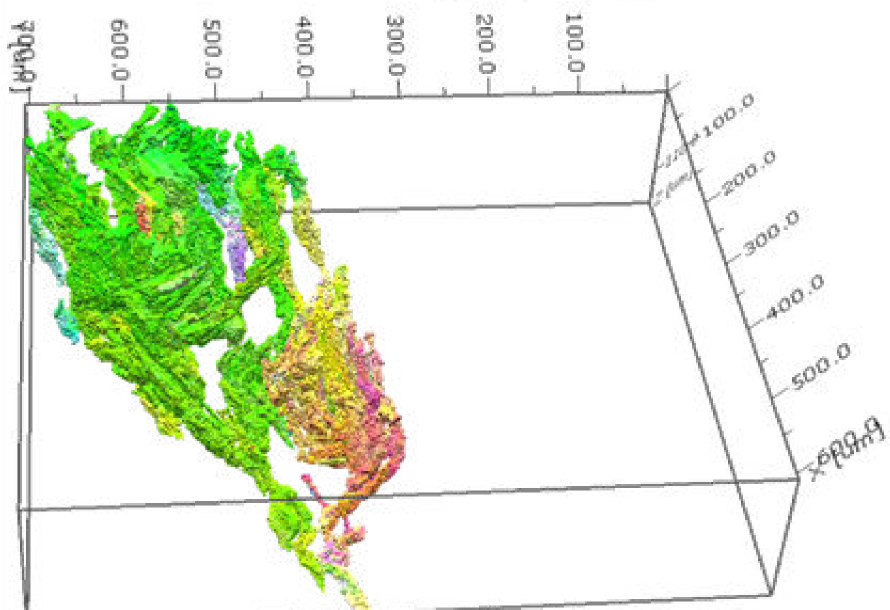
950 W

Some morphological observations on 3D grains





380 W

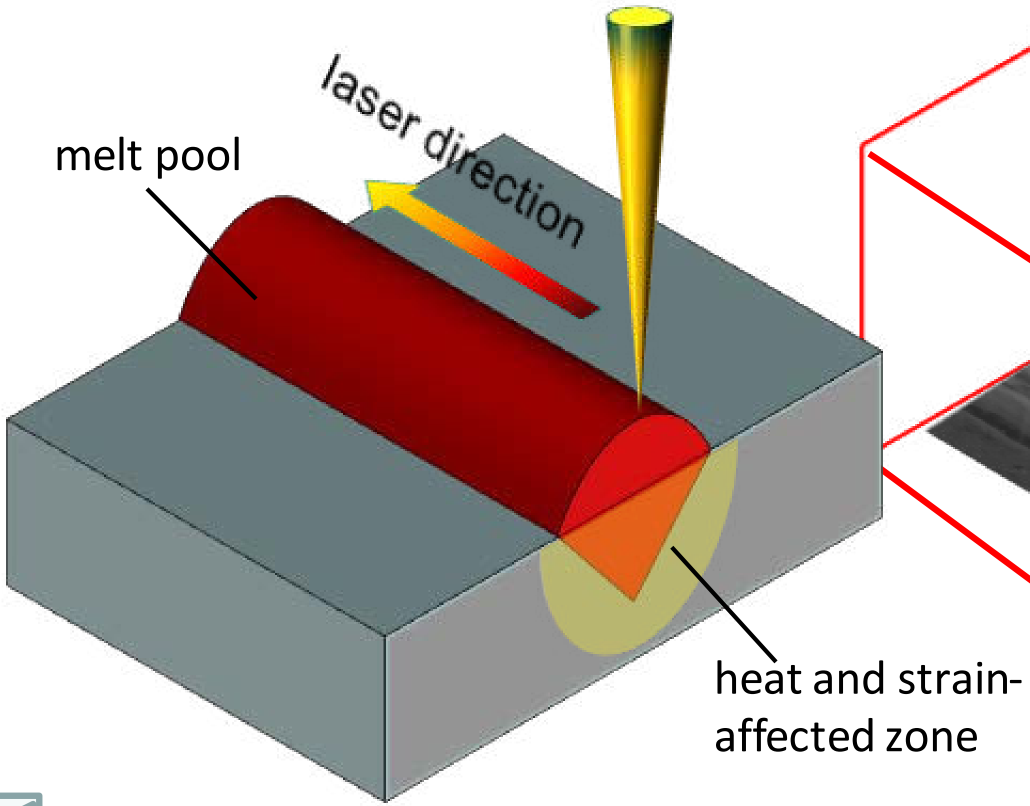


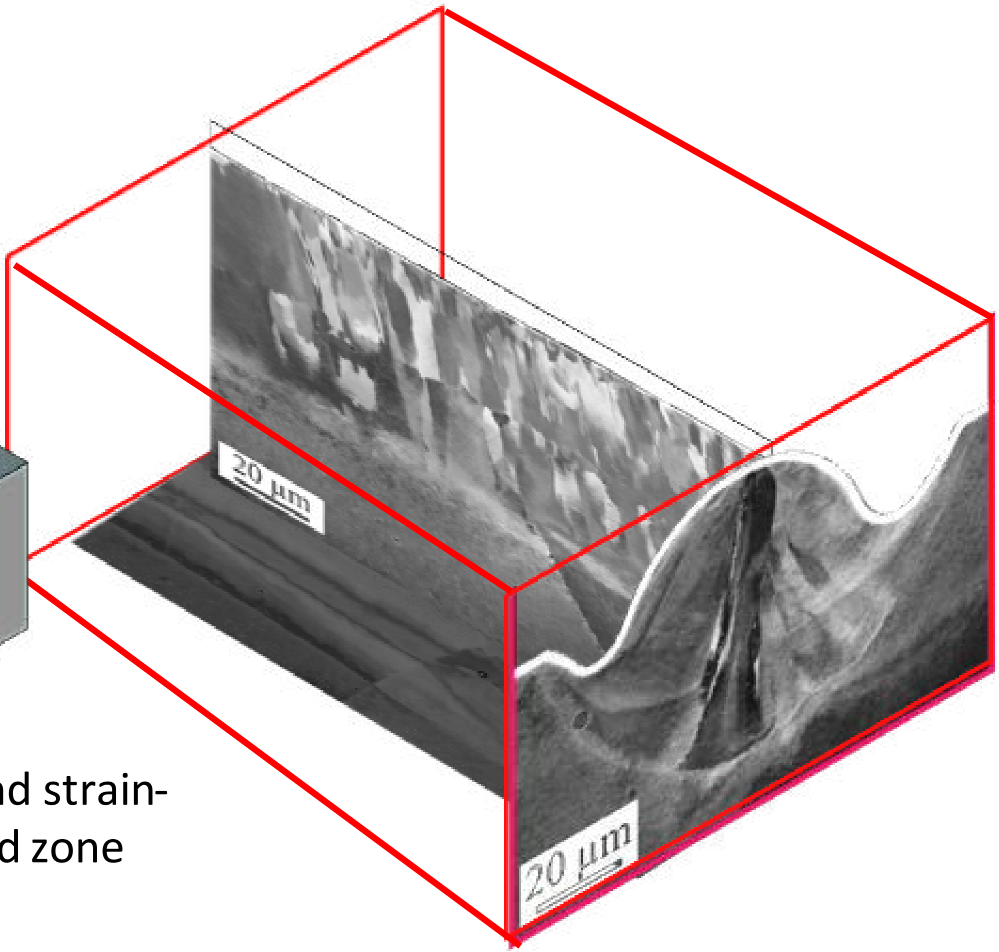
Tree-like structure with large orientation gradients

- Mechanisms of orientation formation (still speculation, but to be published soon):
  - Two orienting effects during solidification:
    - ⇨Solidification along thermal gradient
    - ⇨Tendency to turn towards quickly growing crystal direction
  - Thermal stresses during cooling cause strains which rotate the crystal lattice (crystal plasticity) and creates new grains
  - Epitaxial growth across melt pool preserves orientations and grains

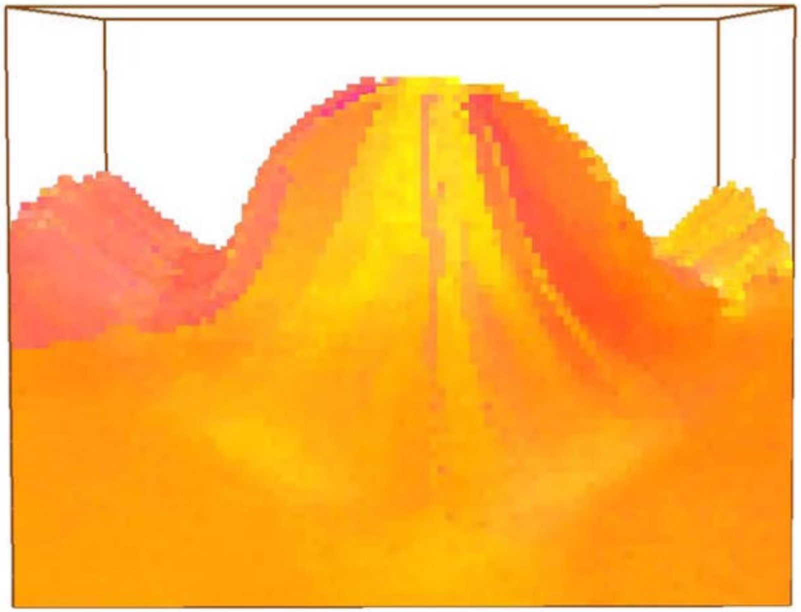
Strain patterns along individual laser lines

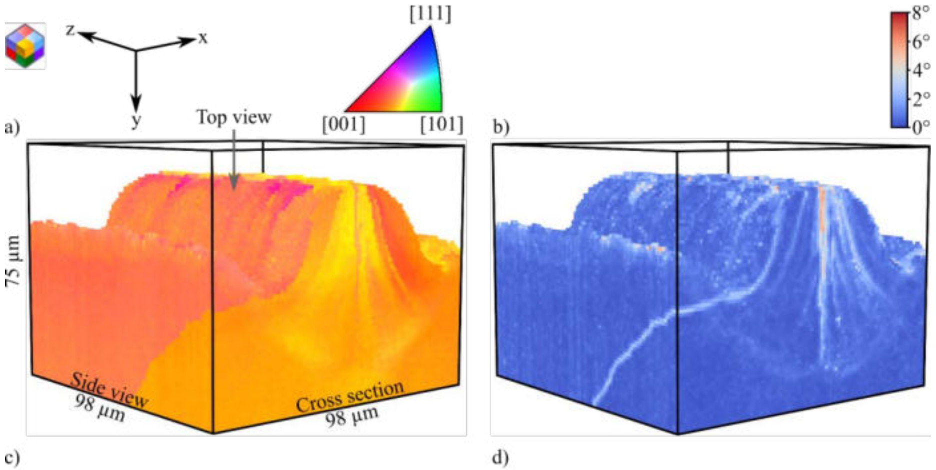
Leonie Gomell (with B. Gault): Laser line melting of Fe_2VAl (bcc)
Shao-Pu Tsai (with S. Zaefferer): 3D characterization
Francisco Gallardo (with F. Roters): Crystal plasticity modelling



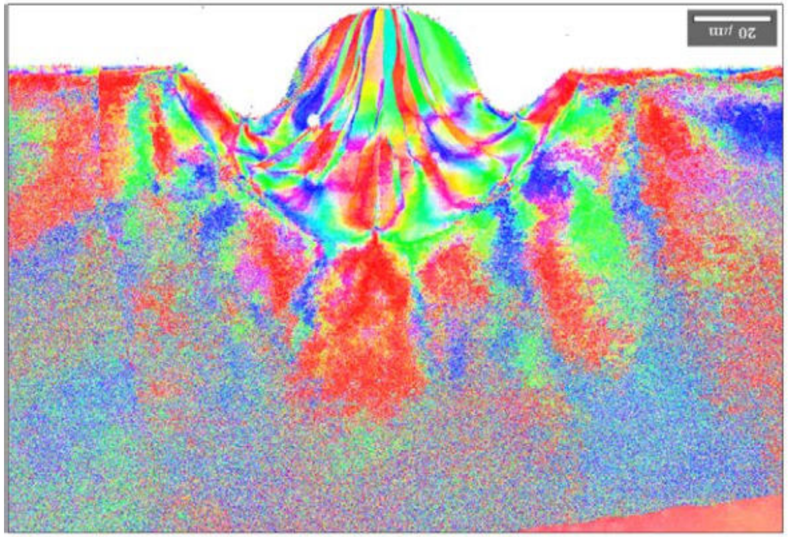


Crystallographic characterization

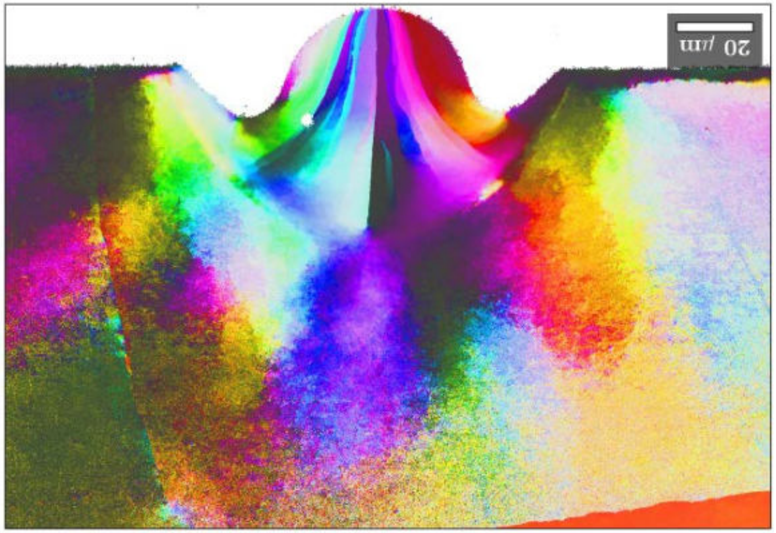




GROD axis – crystal frame



GROD axis – sample frame



Modelling of deformation in the HAZ

Sorry, coming shortly!

Outline

- Introduction:
  - Why and how doing 3D EBSD?
- Development of a large volume 3D EBSD system
- Application examples:
  - Understanding corrosion of 316 stainless steel
  - Microstructure and texture of AM-produced SS 316
- Conclusions

Summary: Large volume 3D EBSD system, ELAVO 3D

- The system works and produces great results!
- Mechanical polishing produces very flat and defect free surfaces
- Polishing can be adapted to many different materials (metals, mineral, semi-conductors)
- Important fundamental and engineering questions on microstructure-property relations can be addressed
- Additive manufacture material is a particular target, due to its complex 3D microstructure