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OPTICS & PHOTONICS
UNIVERSITY OF NOTTINGHAM

Unveiling the Mysteries of Material Recrystallization in situ through Spatially Resolved Acoustic Spectroscopy

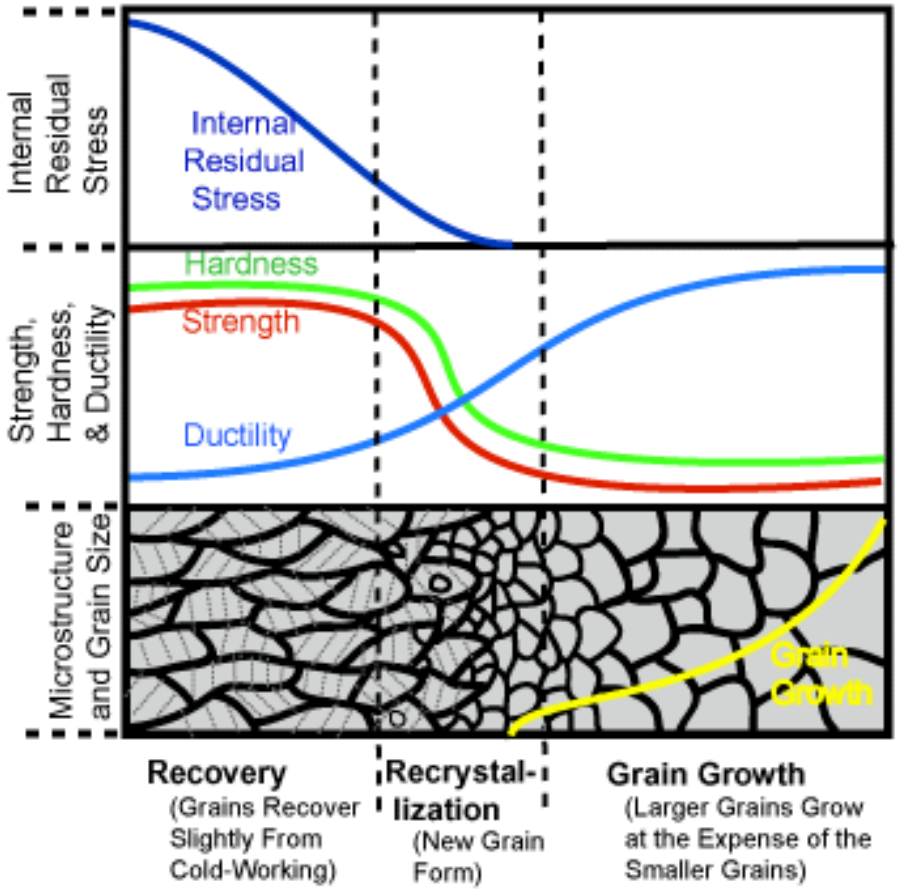
Carolina Guerra, Arthur Ford, Wenqi Li, Rafael Fuentes-Dominguez, Rikesh Patel, Richard J Smith, and Matt Clark

Optics and Photonics Research Group
University of Nottingham

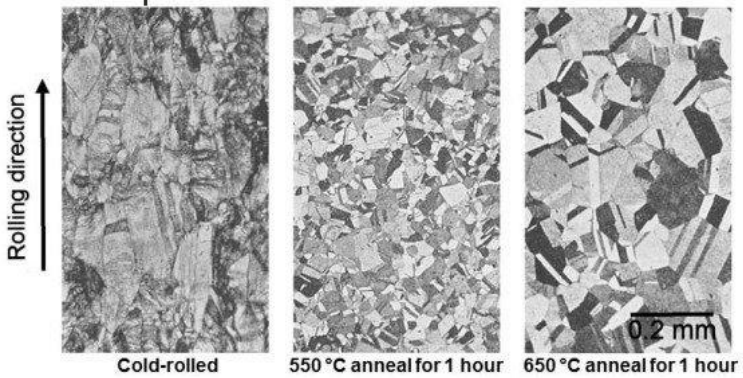
22-23th Jun 2024 – LUS4Metals

Annealing process

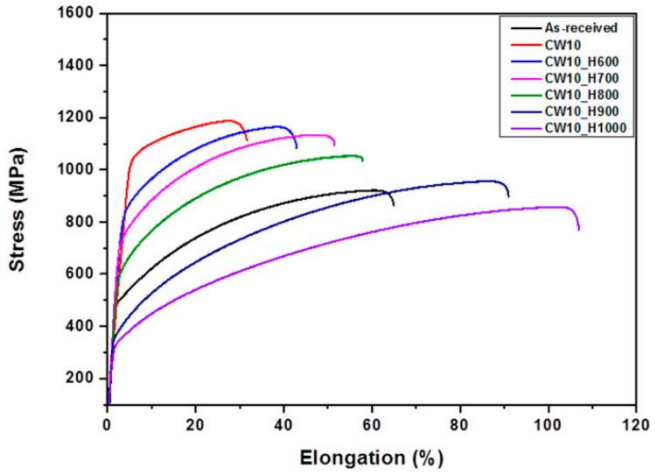
Mechanical properties change at the different annealing stages



Micrographs of brass alloy with 50 %CW and annealed at two different temperatures.



Engineering stress-strain curves of cold-worked high-Mn steels before and after annealing: CW10 (10 %CW)



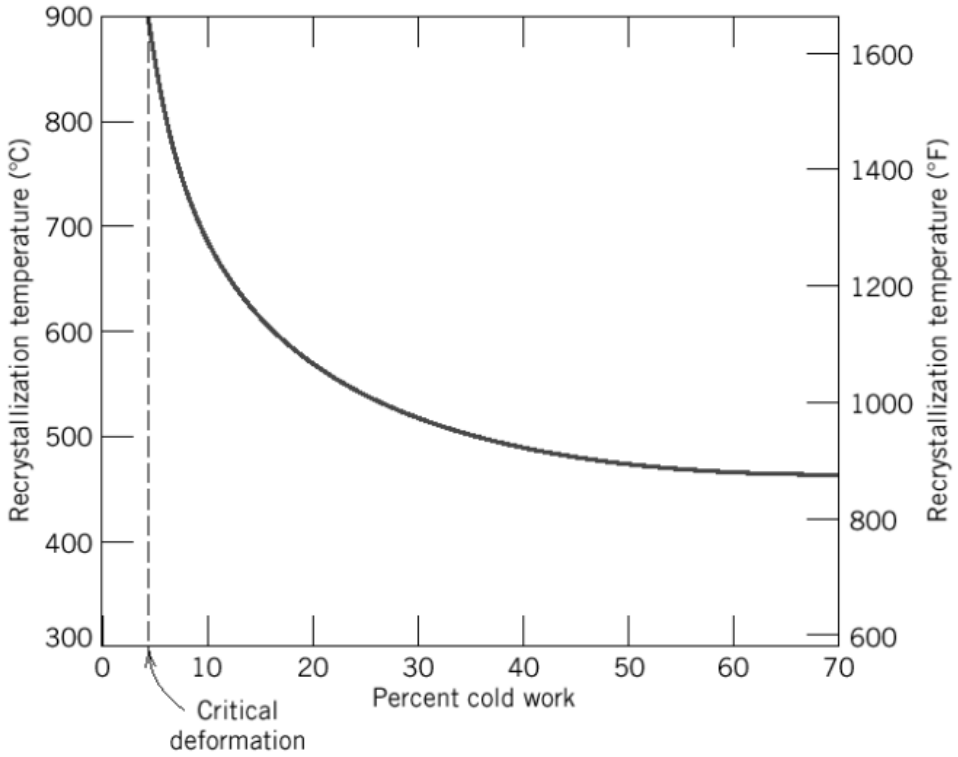
CW: cold –working
H: heat treatment

Carolina Guerra, carolina.guerra@nottingham.ac.uk

Minha Parket al. (2019), Metals

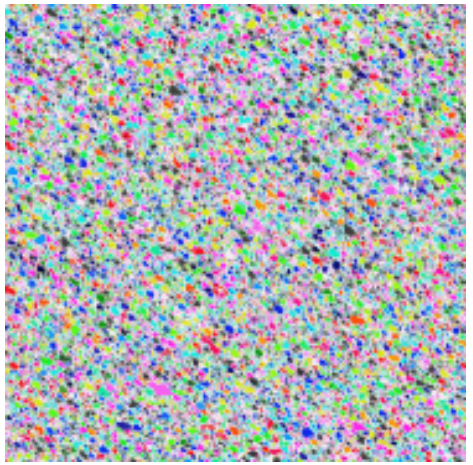
Annealing process

Recrystallisation temperature depending on the cold work



Below a "critical deformation", recrystallisation does not occur.

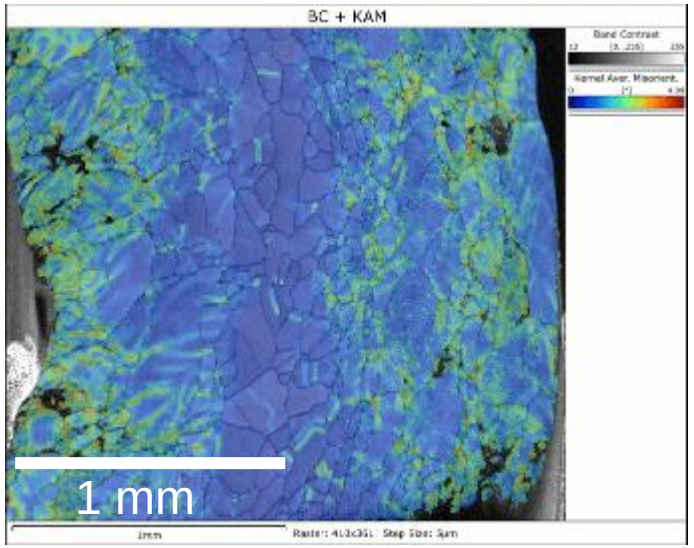
Grain growth simulation using a cellular automaton



To observe recrystallisation Considerations

- Cold work
- Temperature
- Time

Animation of kernel average misorientation (KAM) maps of a bent Al sample, held at a constant 295°C.

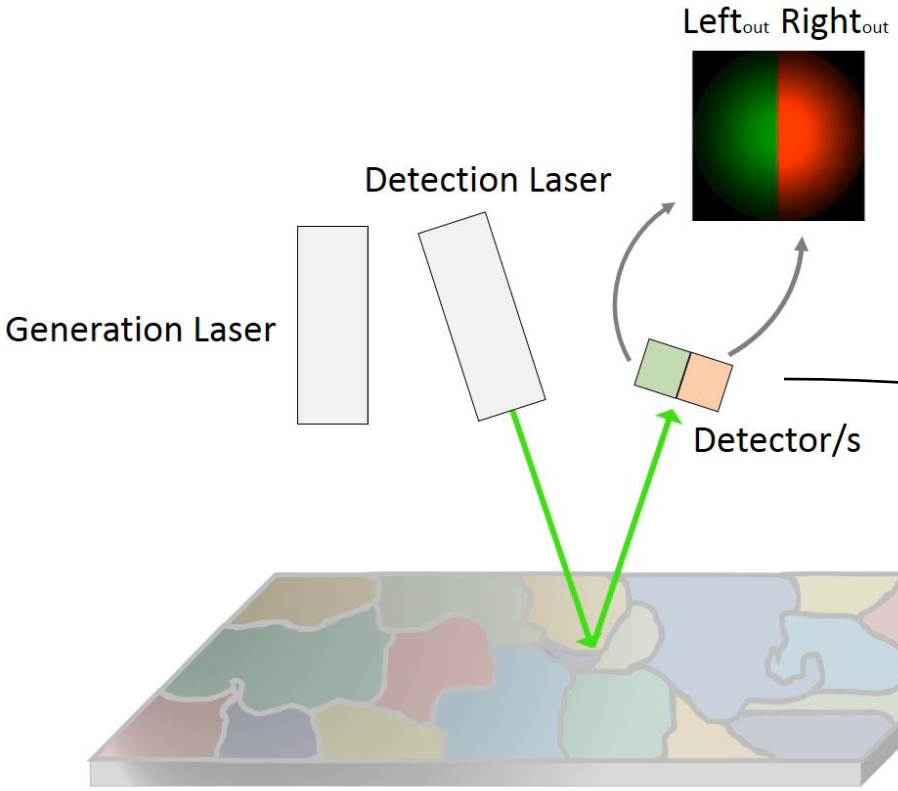


Carolina Guerra, carolina.guerra@nottingham.ac.uk
https://www.dierk-raabe.com/recrystallization-and-grain-growth-simulation/basic-recrystallization-phenomena/

Spatially Resolved Acoustic Spectroscopy (SRAS)

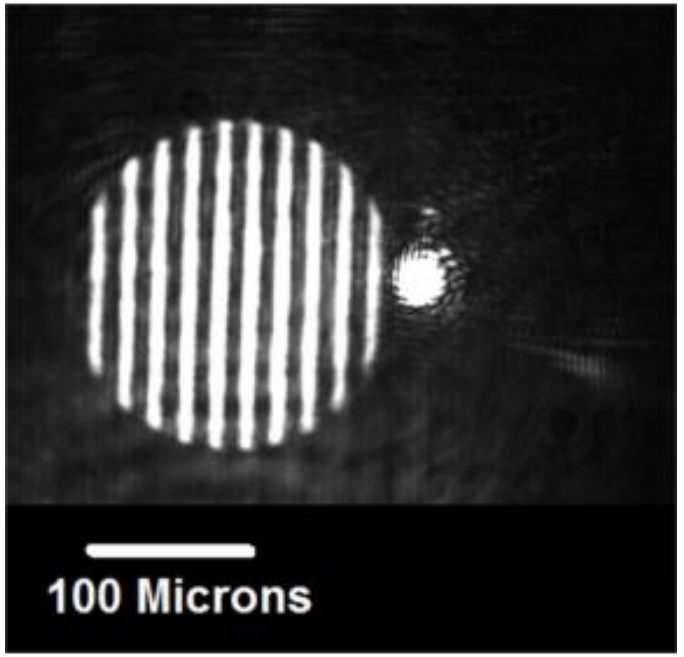
Recovery, recrystallisation and grain growth in – situ monitoring

Spatially resolved acoustic spectroscopy

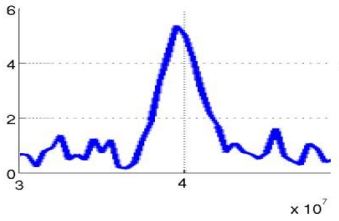


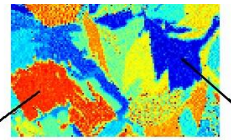
$$V_{saw} = f_{sig} * \lambda_{gen}$$

Frequency
↓
Velocity

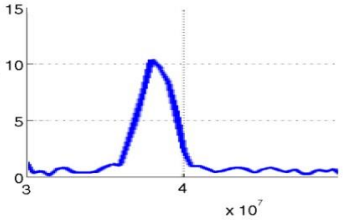


Higher *f* - Faster *v*



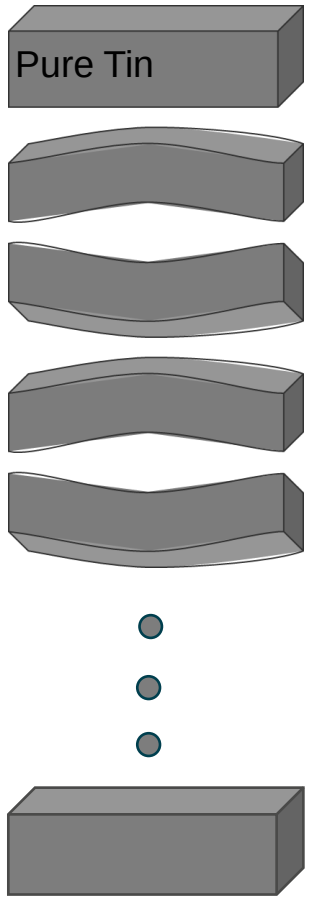


Lower ***f*** - Slower ***v***

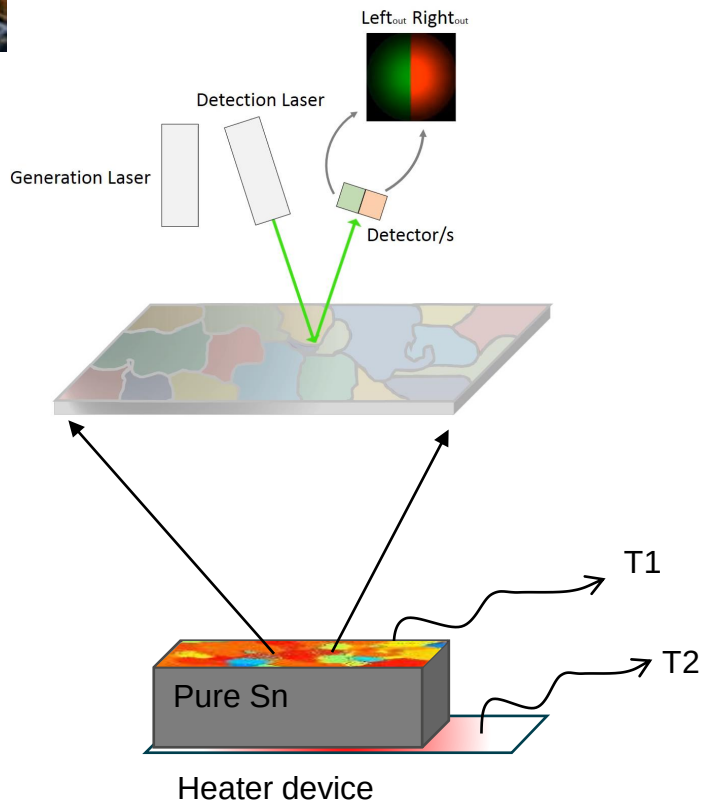


Methodology

Cold work

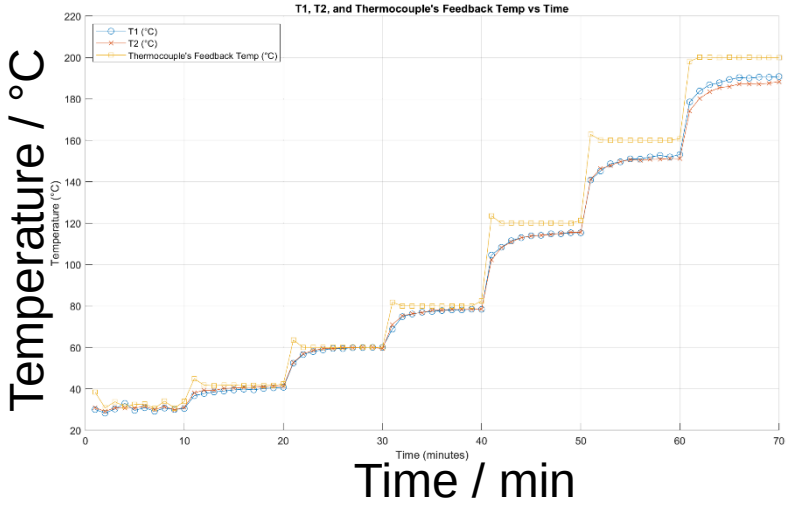


SRAS scanning during annealing



Temperature range: 20 – 200 ° C
Time per scan: 20 min

Representative graph of the temperature control

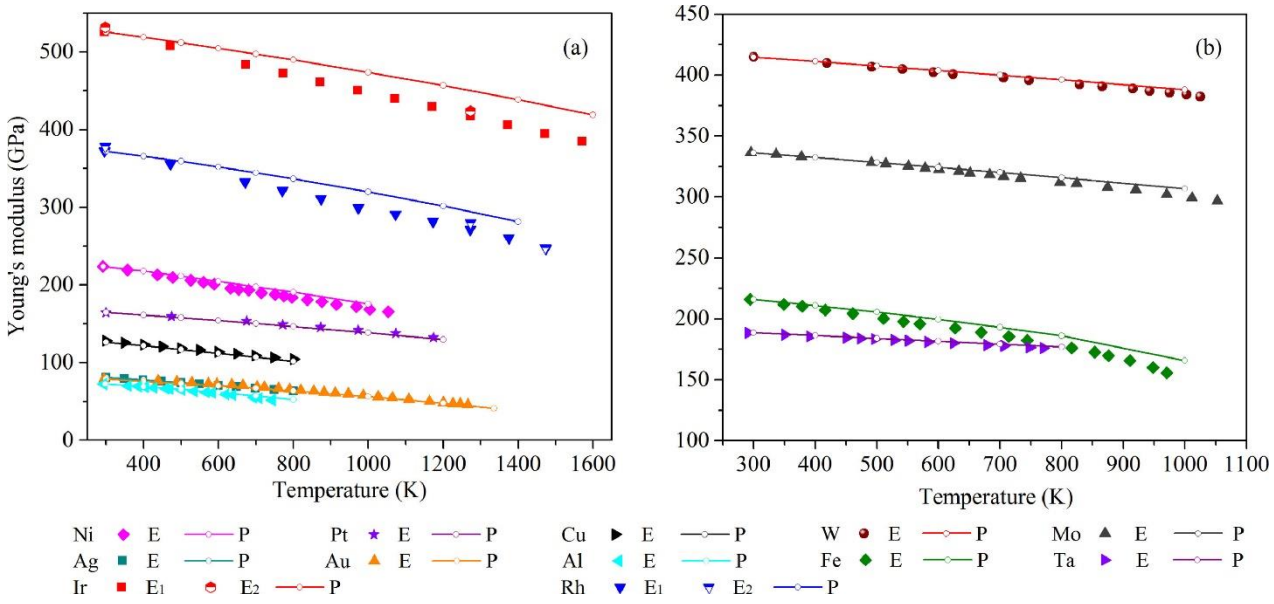


Carolina Guerra, carolina.guerra@nottingham.ac.uk

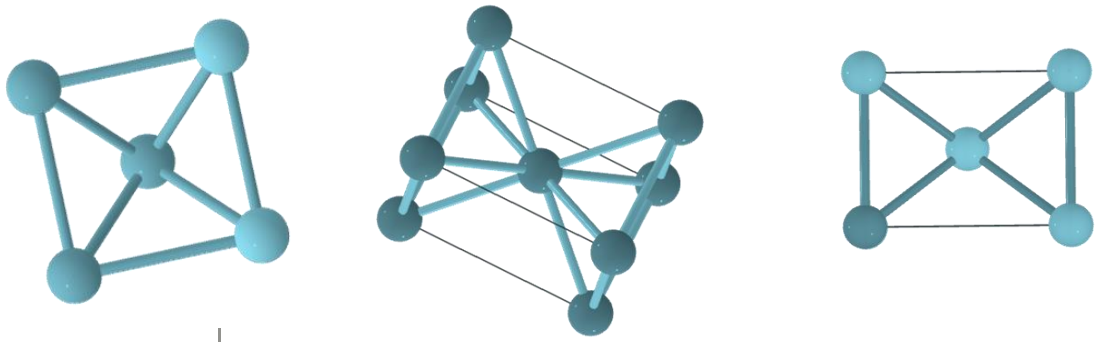
Tin characteristics and E vs T

Tin advantages

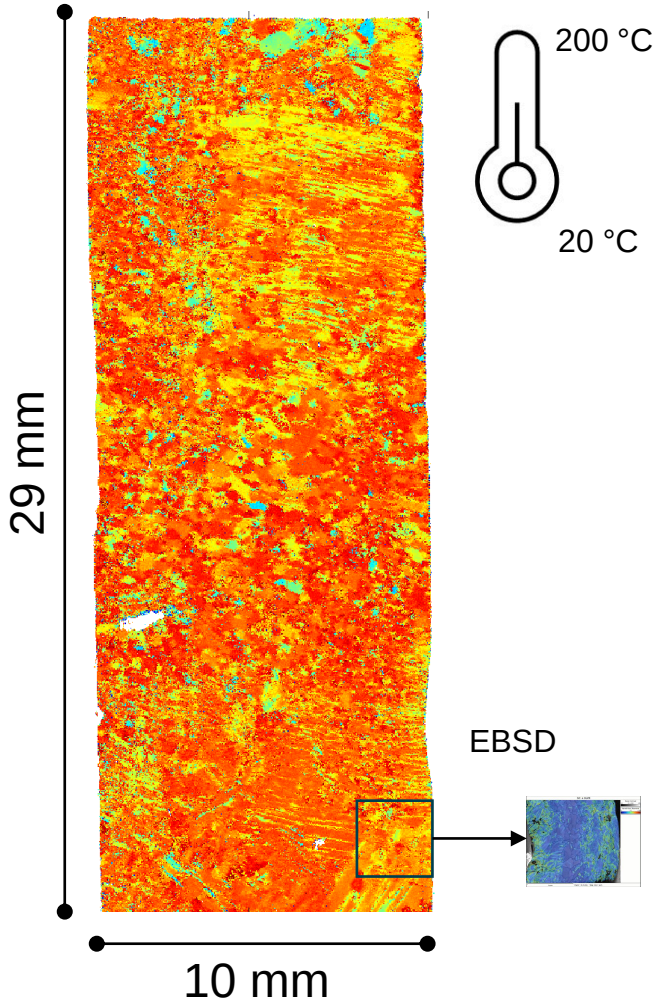
- Low melting point (232 °C)
- Only one phase (β phase – tetragonal)
- Easy to deform plastically
- No phase transition between 20 – 200 °C
- No intermetallic precipitations

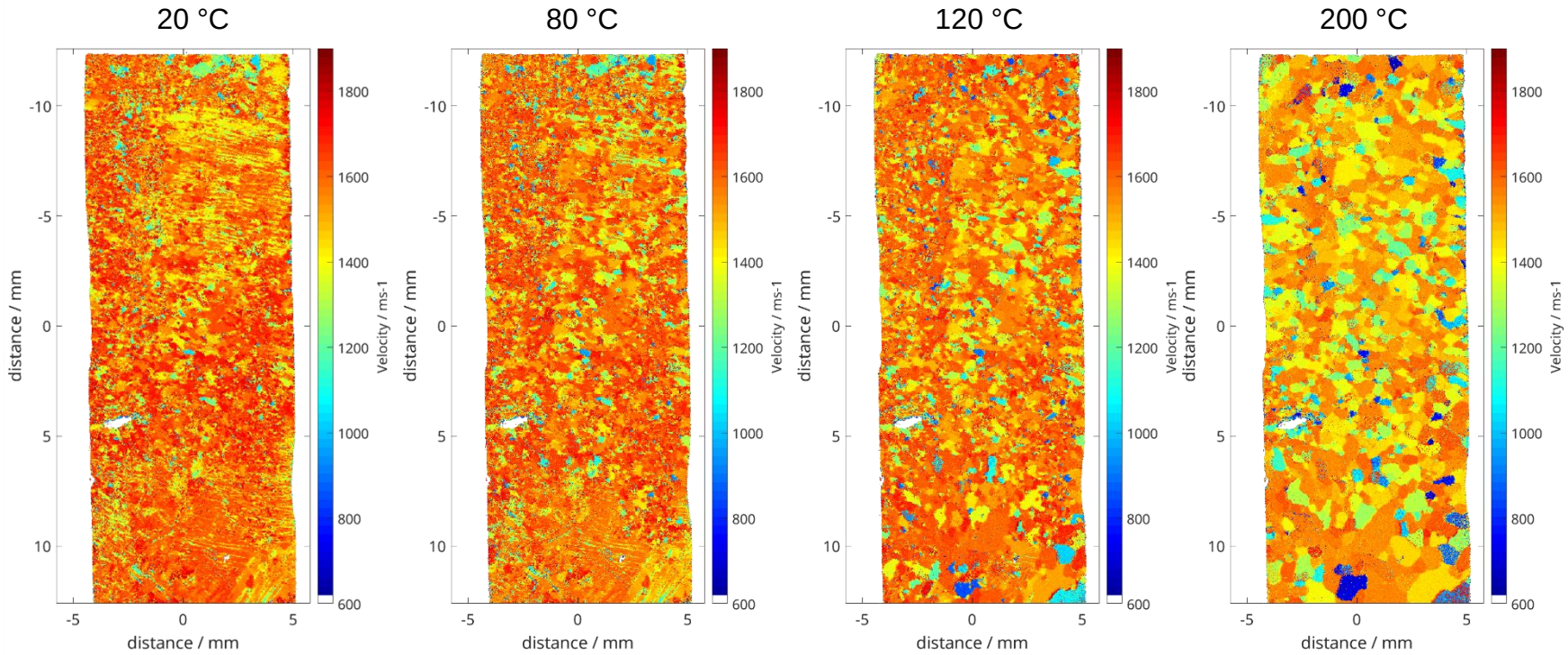


Space group: I4/mmm
Point group: 4/mmm
Lattice parameter: a=5.8 Å, c=3.2 Å



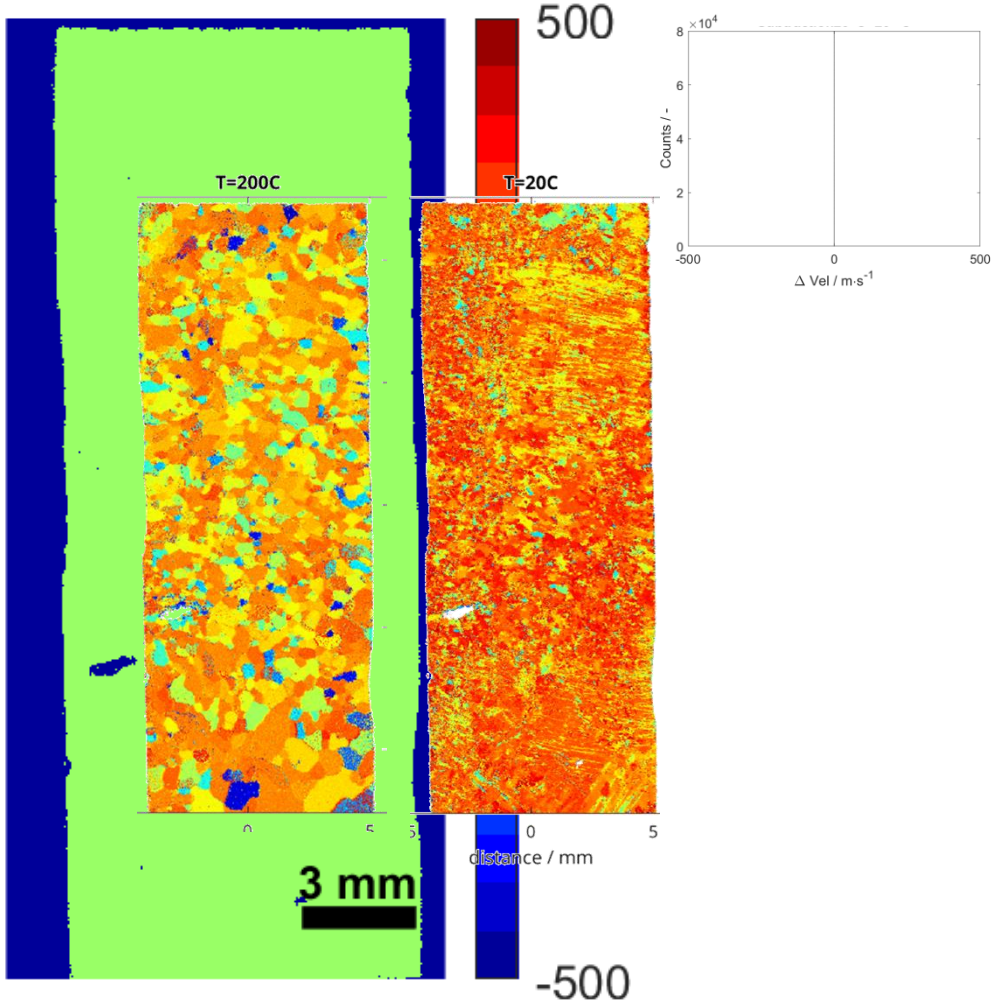
Vel. map tracking by raising the temperature

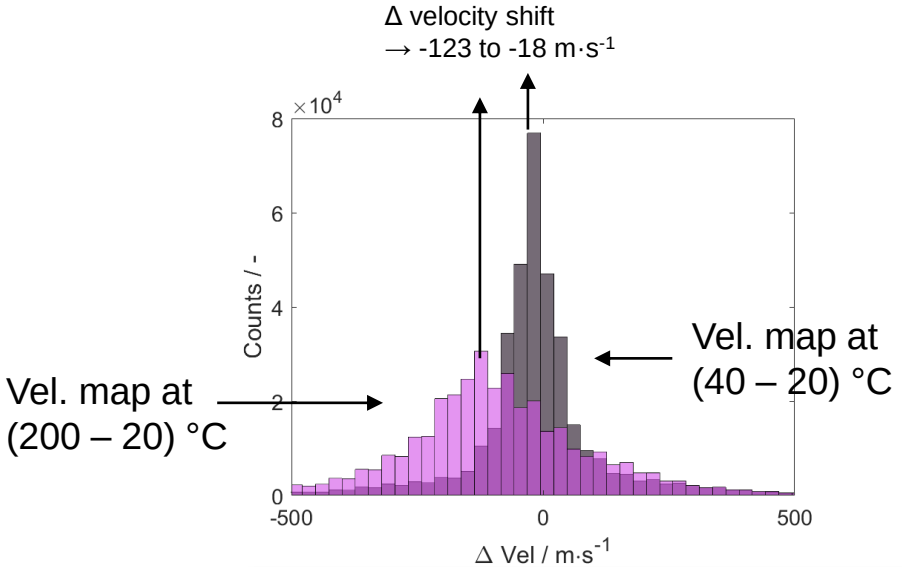




Vel. map tracking by raising the temperature

Case 1. Subtraction velocities x °C – 20 °C (x = 20 40 60 80…200)



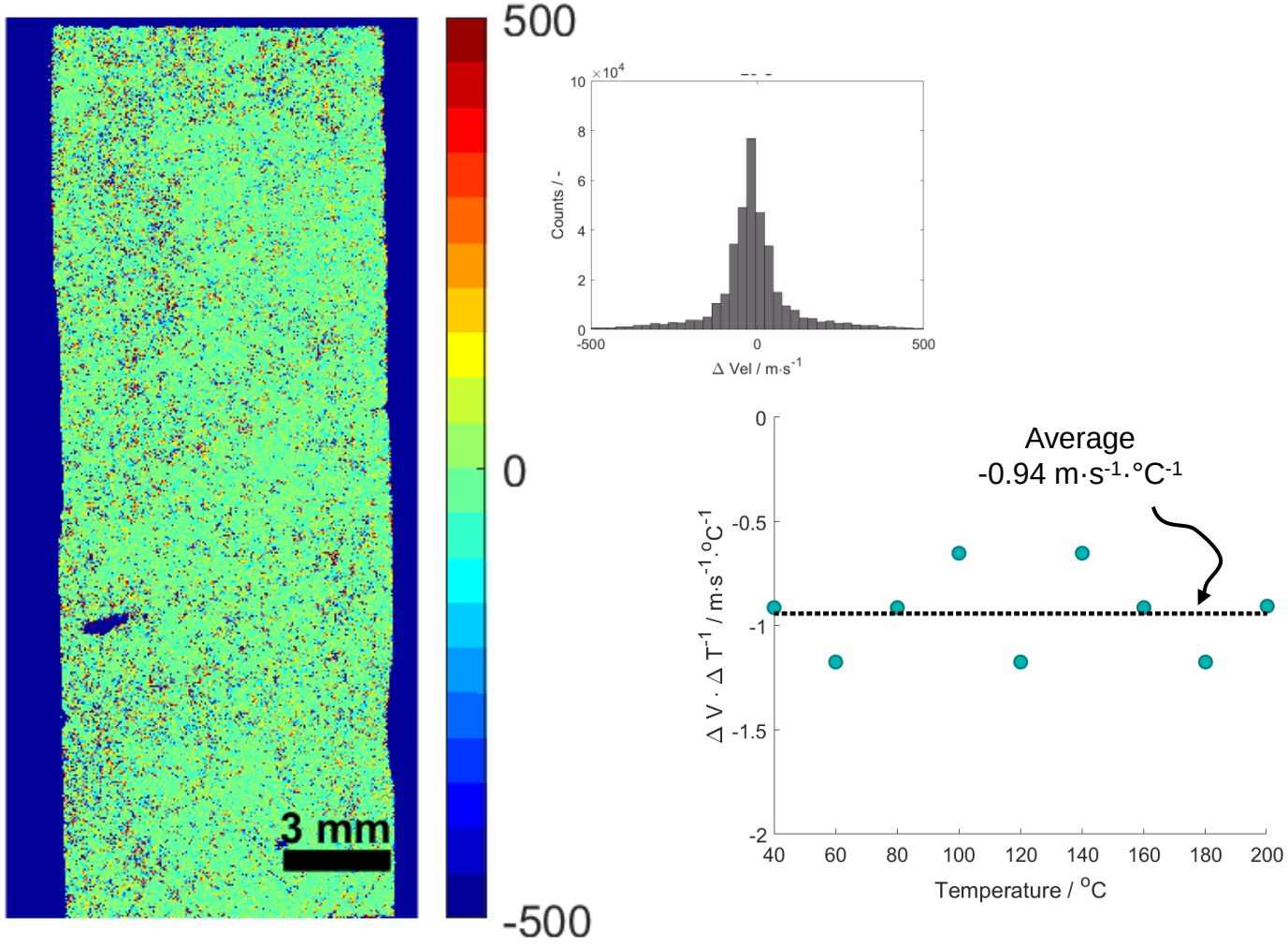


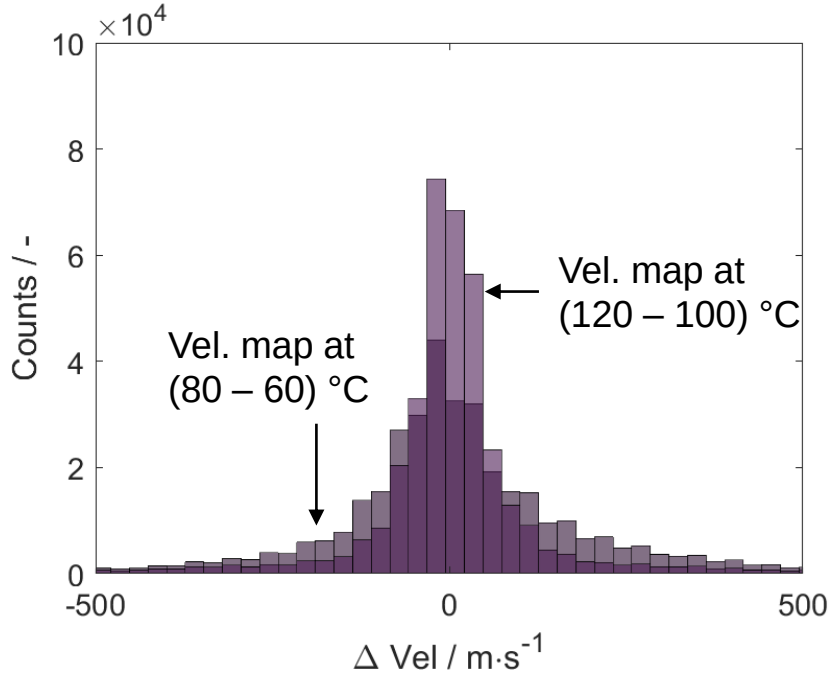
Negative variation (Δ Vel) means that the sample at a higher temperature has a lower velocity.

The sample at 200 °C shows a wider velocity range than at lower temperatures.

Vel. map tracking by raising the temperature

Case 2. Subtraction velocities (x+20) °C – x °C (x = 20 40 60 80…200)

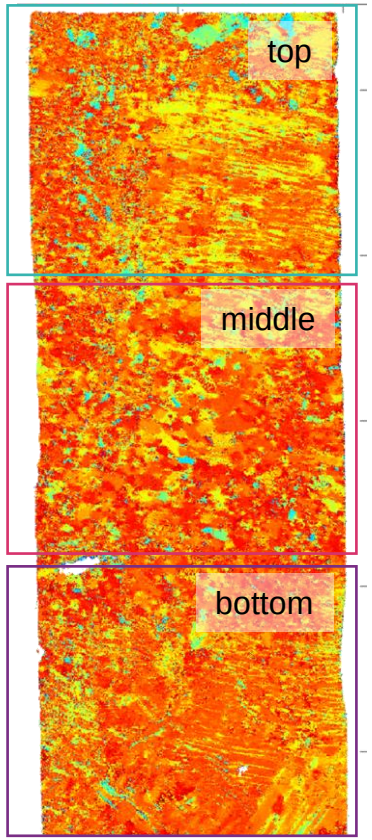


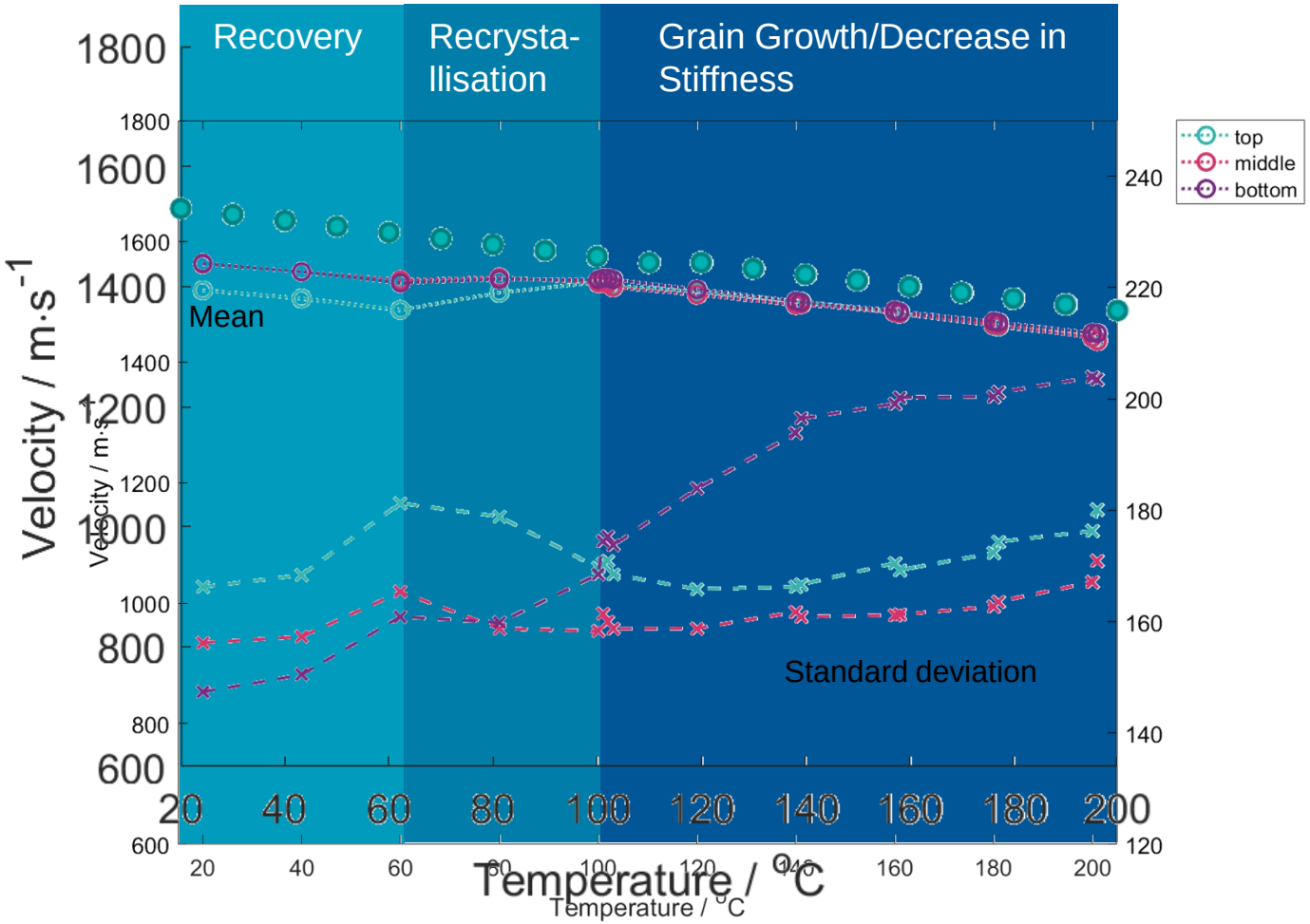


The velocity range is kept narrow, meaning smaller changes occur between temperatures.

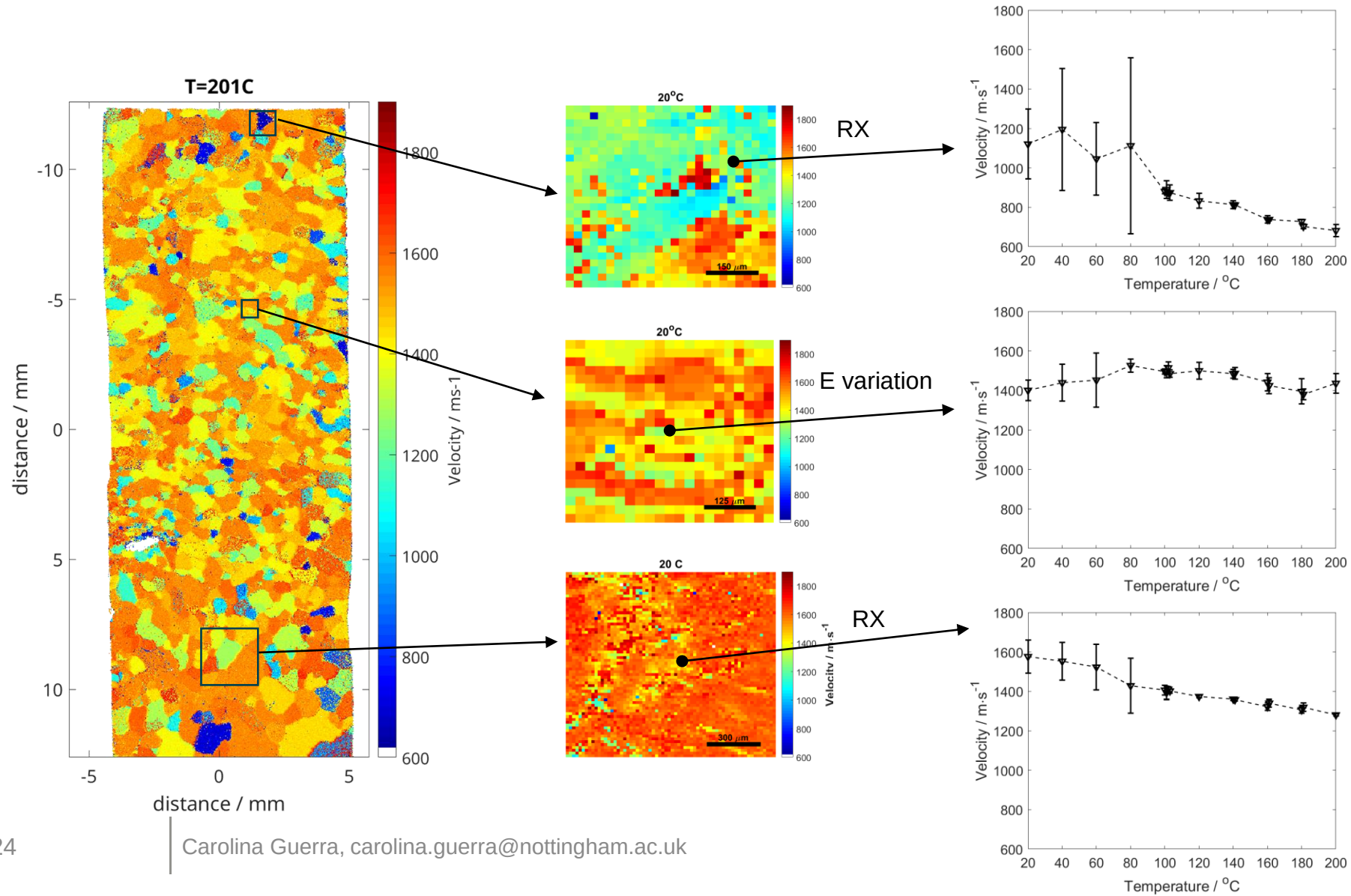
The greatest changes are occurring between 60 and 120 °C

Analysis by regions





Analysis by smaller regions

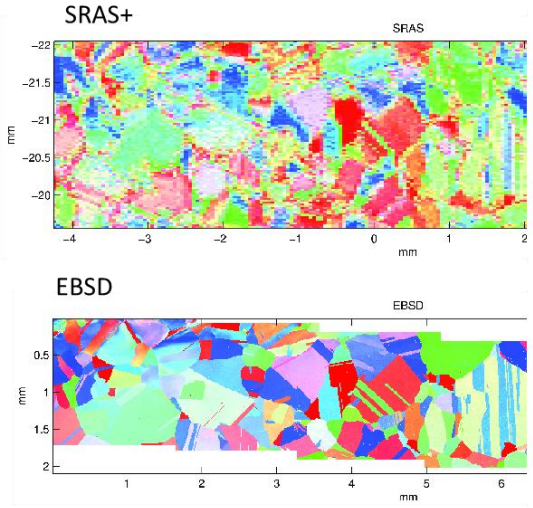


RX: recrystallised grain
E: elastic modulus

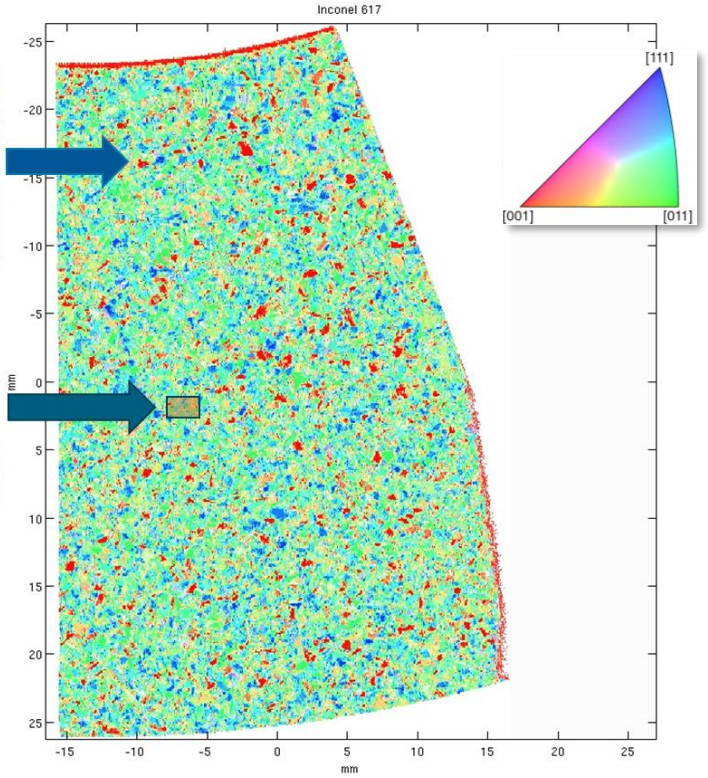
Carolina Guerra, carolina.guerra@nottingham.ac.uk

SRAS - crystallographic orientation

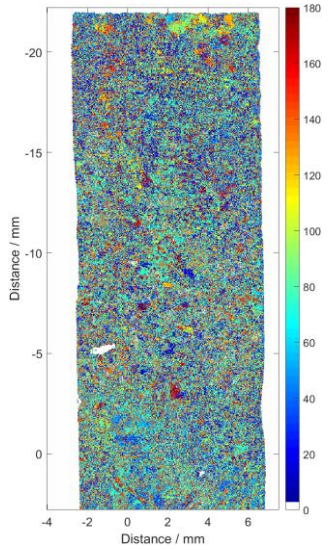
SRAS+ capabilities



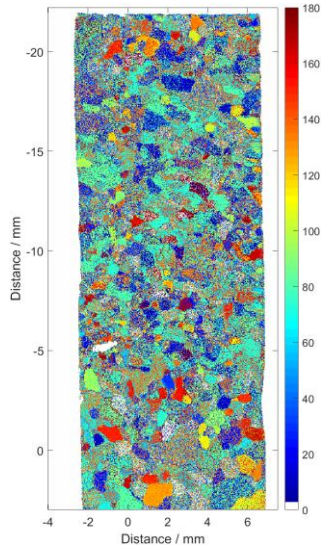
Inconel 617



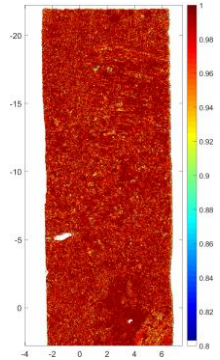
20 °C
Euler1



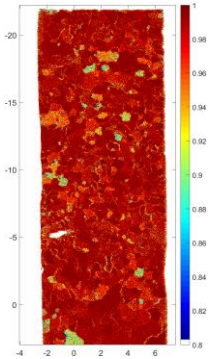
After 200 °C
Euler1



20 °C
Certainty



200 °C
Certainty



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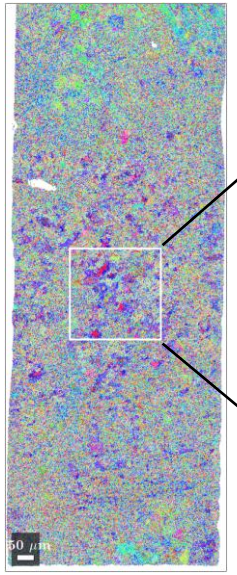
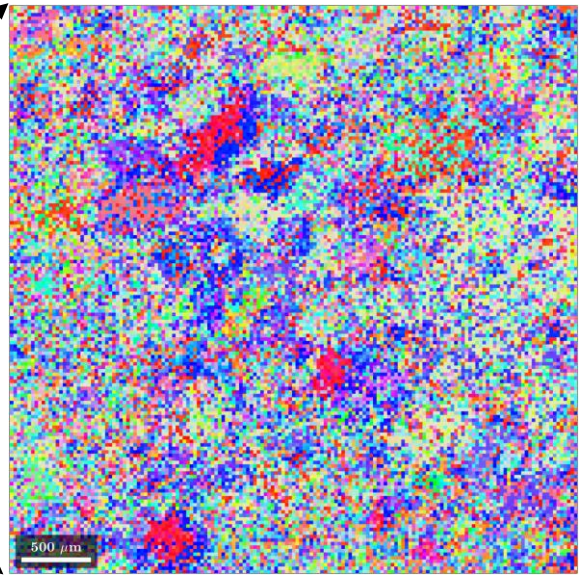
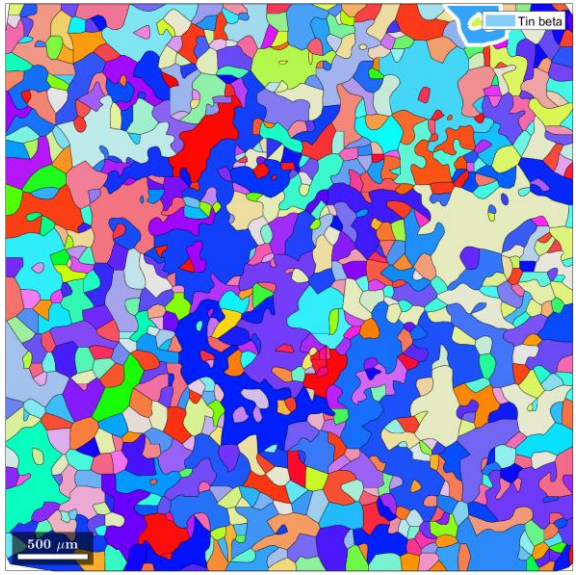
SRAS grains reconstruction by MTex

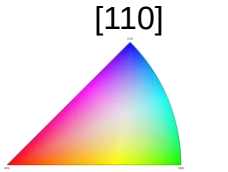
Initial state

Cold-worked Sn
Room temperature



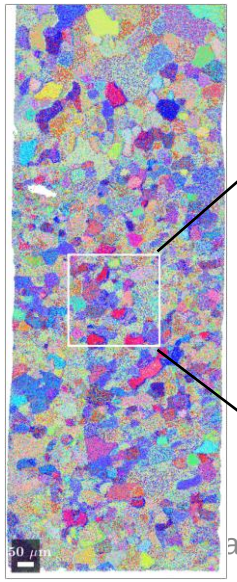


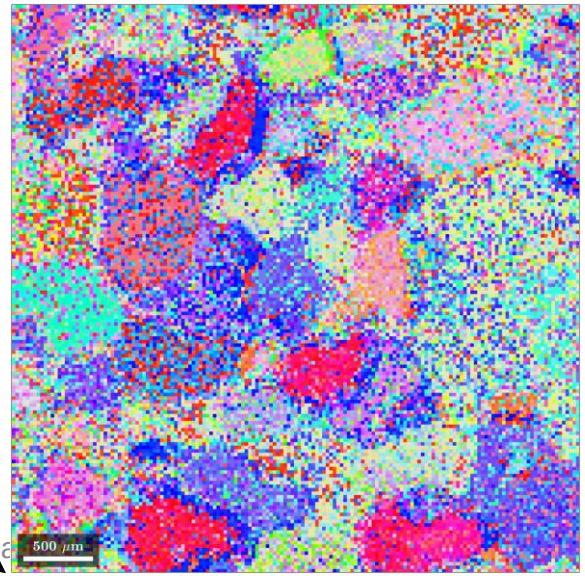


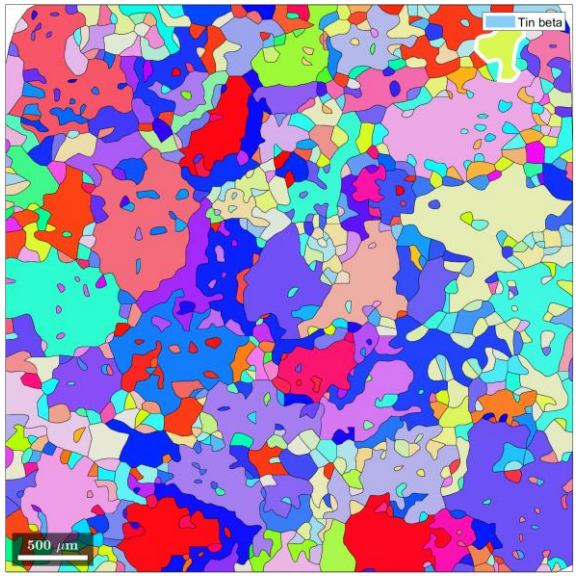


Final state

(after being at 200 °C)
Room temperature



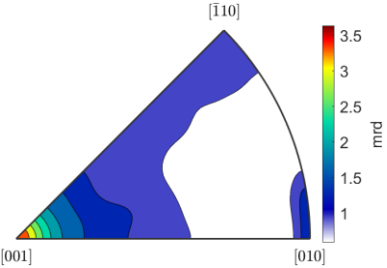




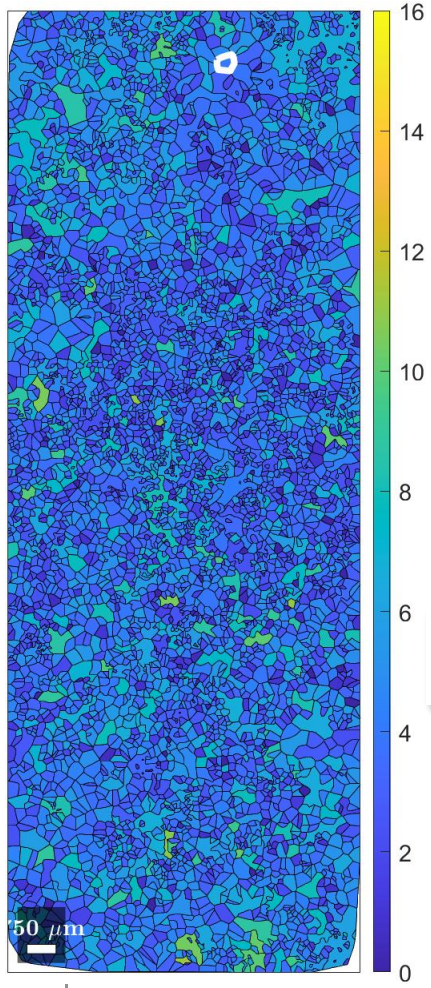
SRAS grains reconstruction by MTex

Initial state

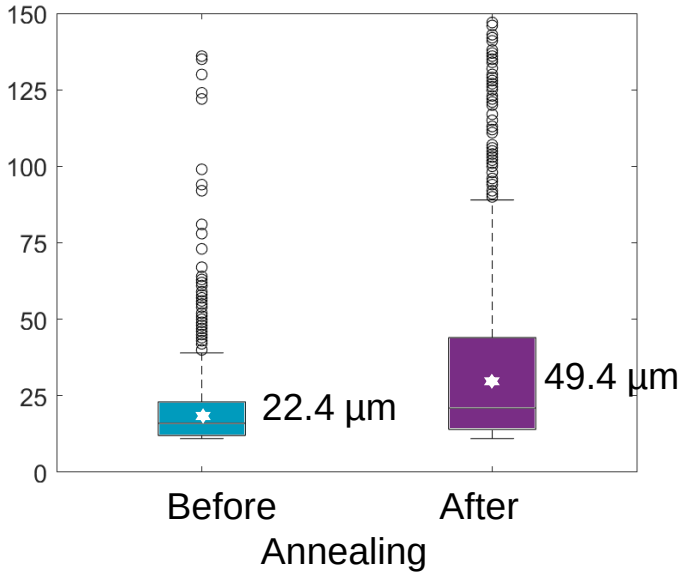
Cold-worked Sn
Room temperature



Grain orientation spread



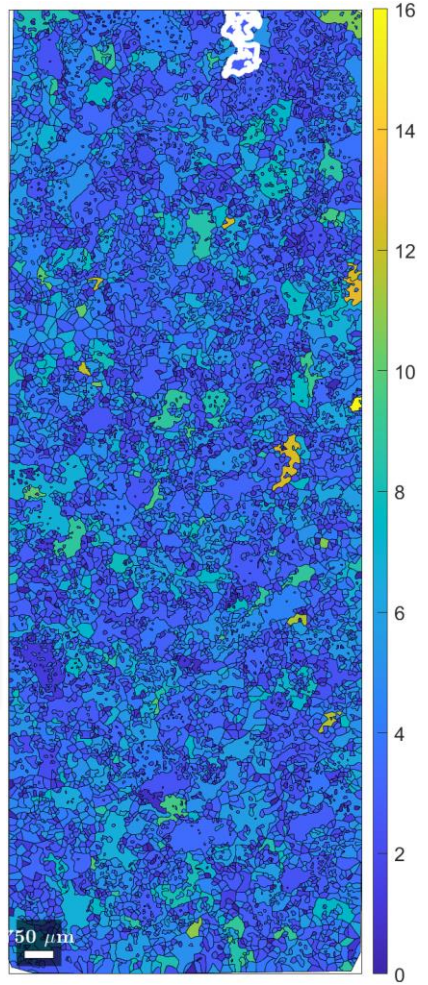
Box plot for grain size



Grains > 10 pixels
500 Units

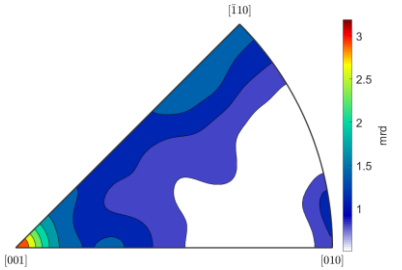
Grains > 10 pixels
1,326 Units

Grain orientation spread



Final state

(after being at 200 °C)
Room temperature



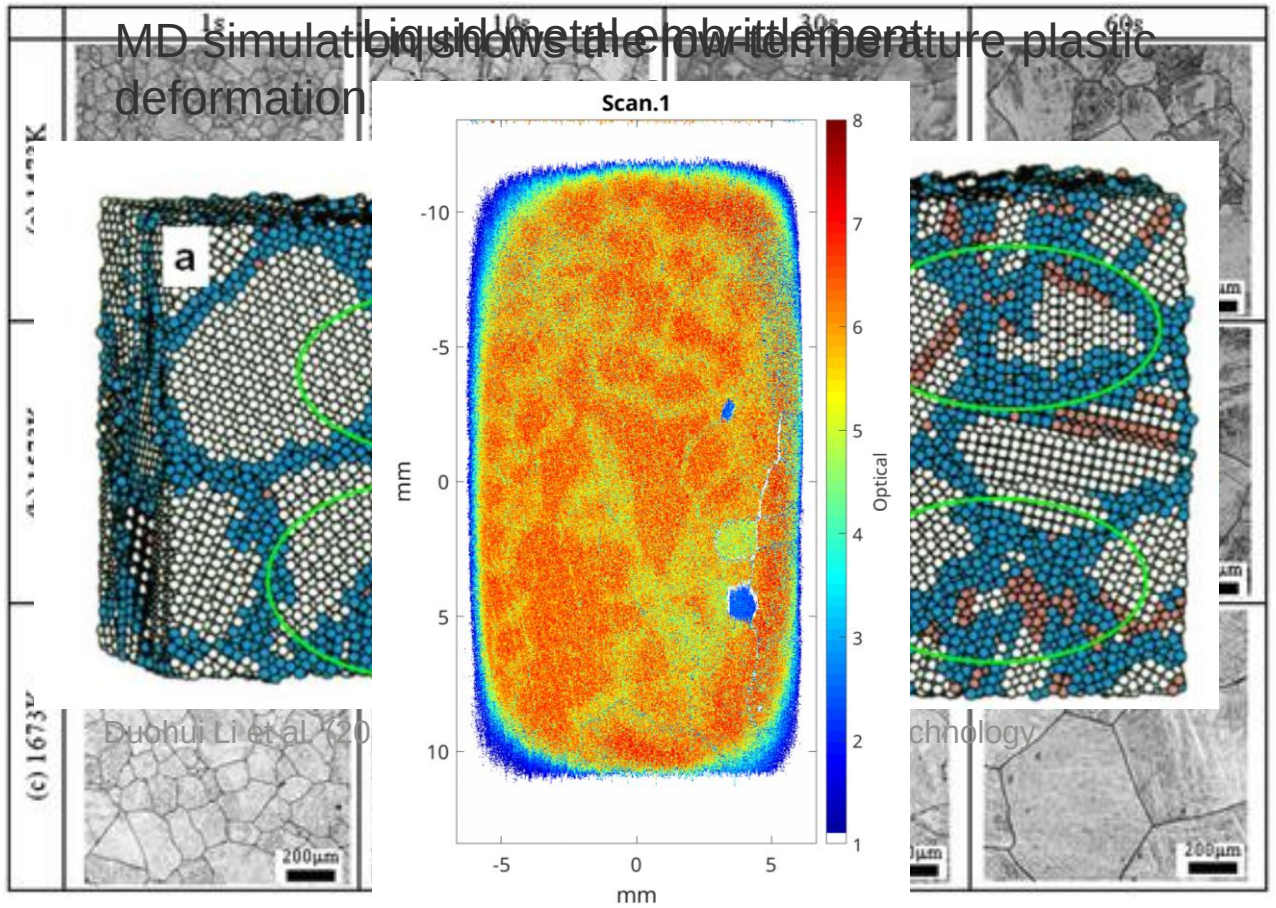
Outlook - In-situ measurements

Measurements of elastic constants/crystallographic orientation under stimulus

- Temperature
  - Phase change in real time, grain growth, precipitation tracking
- Stress application
  - Twin formation, phase transformation, plastic deformation
- Alloy chemical alteration
  - Diffusion process observation, oxidation process, carburation

Microstructure of steel A at isothermal process

MD simulation shows the low-temperature plastic deformation



Duohui Li et al. (20 chnology

Naoto Fujiyana et al. (2015), Science and Technology of Advanced Materials

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en.wikipedia.org/wiki/SRAS

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UNIVERSITY OF NOTTINGHAM


Prof Matt Clark

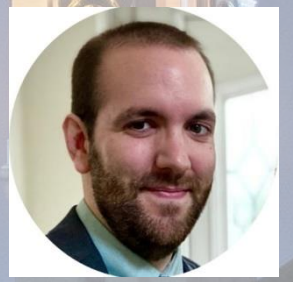

Dr Richard Smith


Dr Rikesh Patel


Dr Wenqi Li


Dr Carolina Guerra


PhD(S) Arthur Ford


Dr Rafael Fuentes-Dominguez

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Questions