Workshop on Dark Field X-ray Microscopy

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M4D erc
Metal Microstructures in 4D
DTU

Local residual stresses in steels

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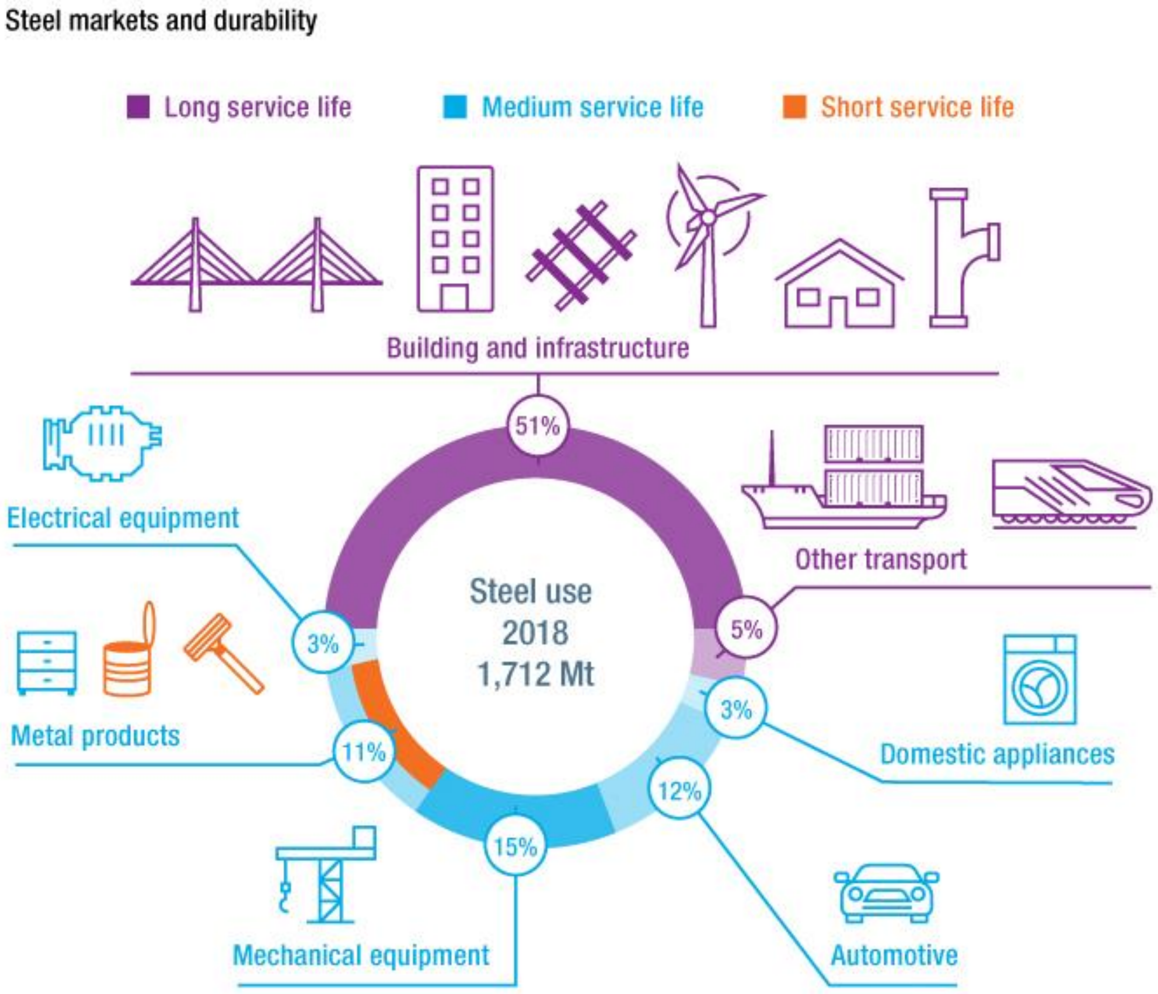
The world is migrating to high performance steels

- Steel is critical simply because no other material has the same unique combination of strength, formability and versatility

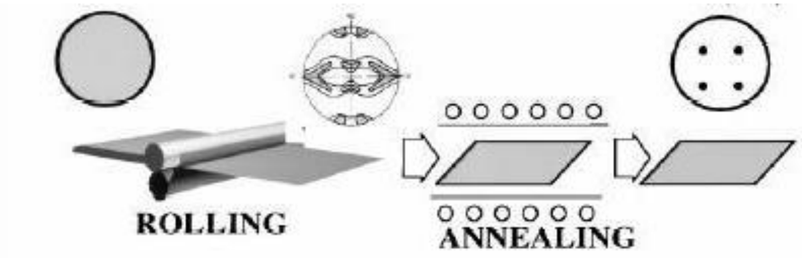
- **Advanced High-Strength Steels (AHSS) are now used for nearly every new vehicle design**
  - New grades of AHSS enable carmakers to reduce vehicle **weight** by **25-39%** compared to conventional steel.
  - When applied to a typical five-passenger family car, the overall weight of the vehicle is reduced by 170 to 270 kg, which corresponds to a lifetime saving of 3 to 4.5 tones of greenhouse gases over the vehicle's total life cycle.
  - This saving in emissions represents more than the total amount of $\mathbf{CO_2}$ emitted during the production of all the steel in the vehicle.

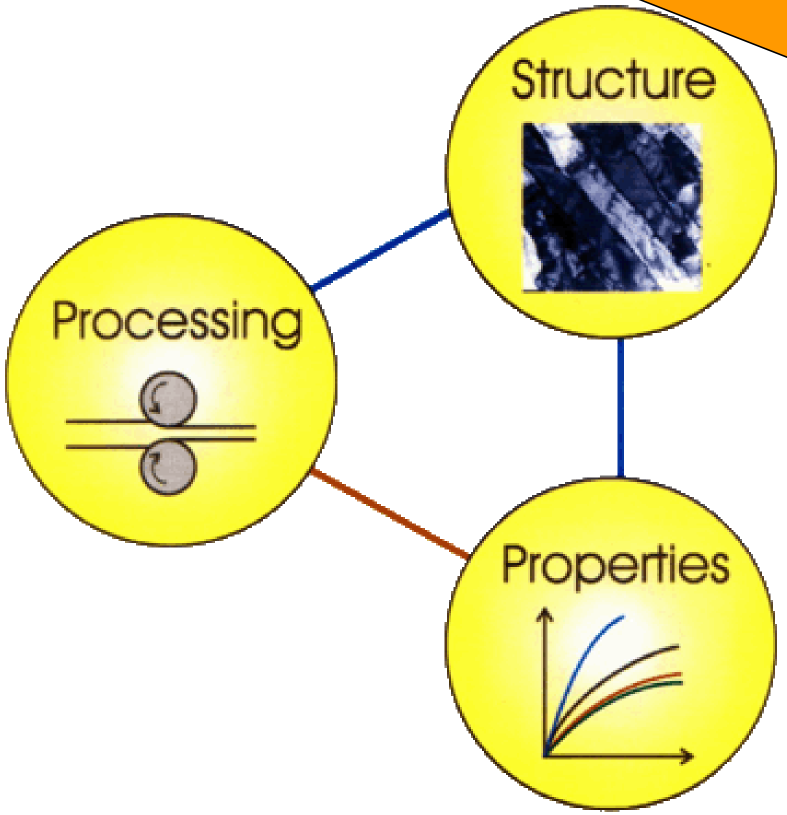


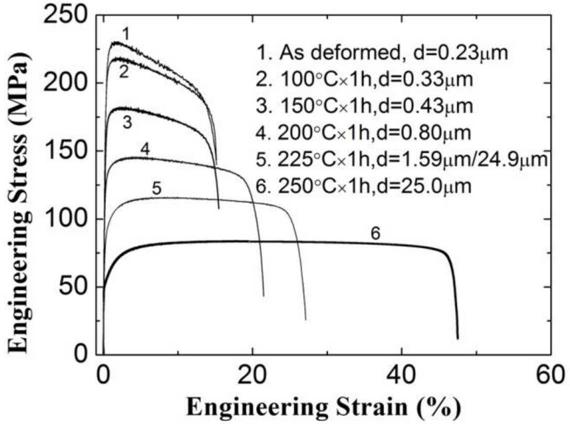
https://www.worldsteel.org

Steel design

- **Strains/stresses are largely missing**



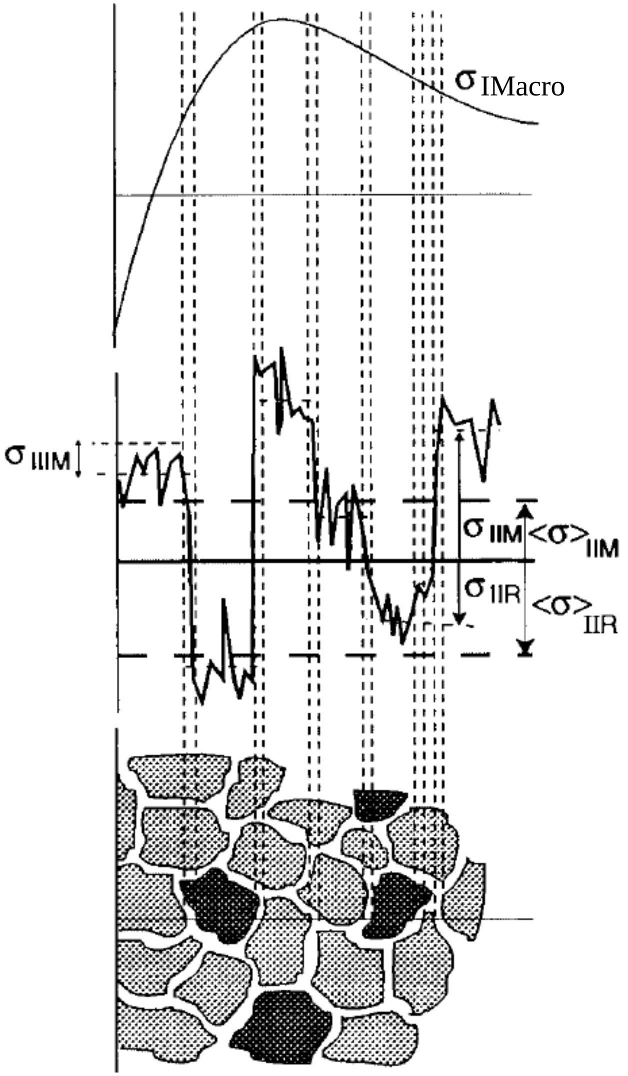




crostructures in 4D

Local residual stresses

- Type II and III stresses
- Develop in nearly all steels
- Hard to predict (depending on many parameters)
- Critical for mechanical properties and materials failure, as it may lead to stress concentration and crack nucleation



P.J. Withers, H.K.D.H. Bhadeshia, Residual stress Part 1 – Measurement techniques, Mater. Sci. Technol. 17 (2001) 355–365.

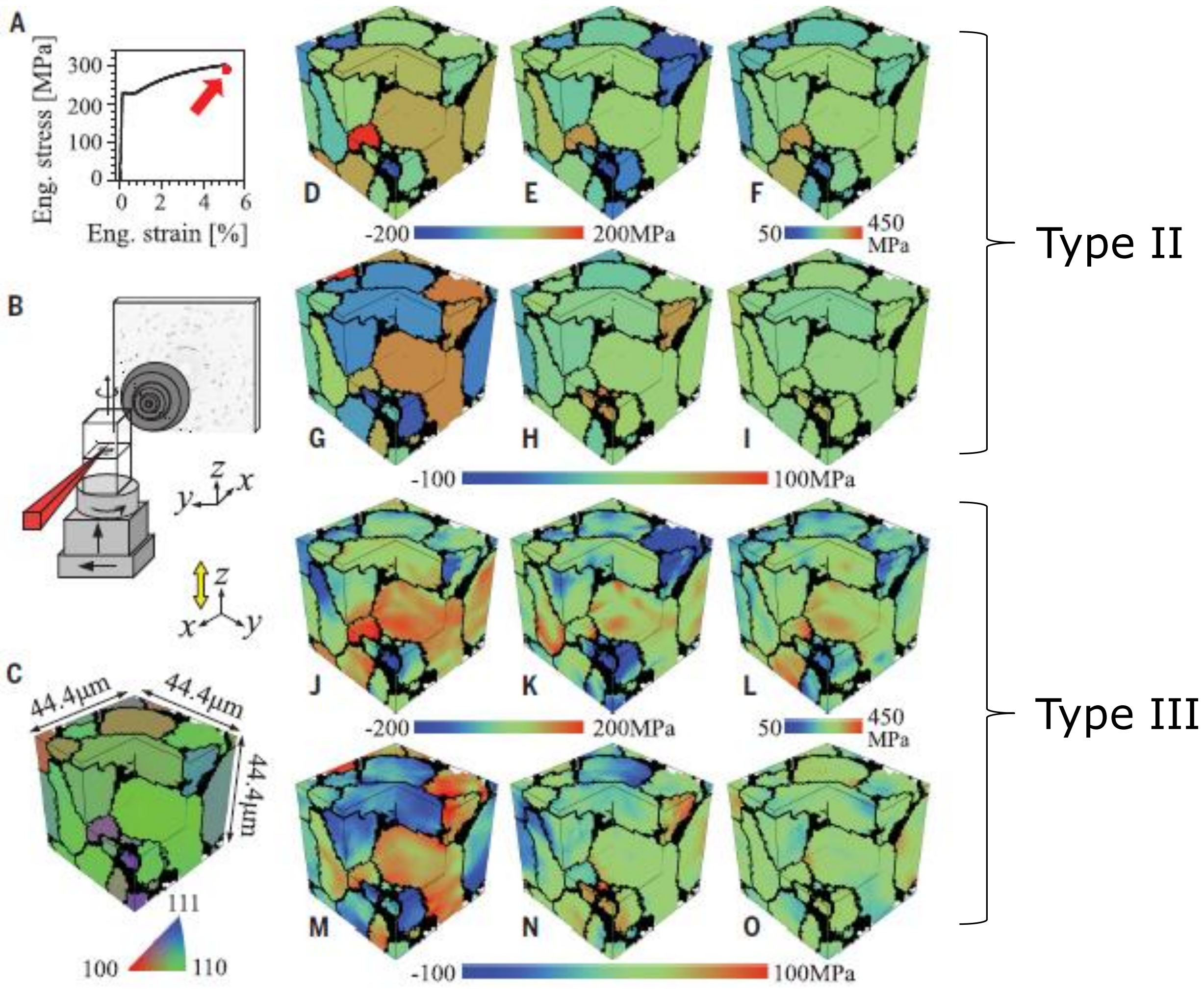
Characterization of local strains/stresses

- Synchrotron X-ray methods
  - 3DXRD, DFXM
  - DCT
  - 3D Laue micro-diffraction
  - Scanning-3DXRD
  - Bragg CDI
  - …

- Bulk measurement

- High spatial and strain resolution

- Critical for developing advanced materials models and lifetime assessment methods with sufficient predictive power



Y. Hayashi, D. Setoyama, Y. Hirose, T. Yoshida, H. Kimura, Science. 366 (2019) 1492–1496.

Aim

- Characterizing local strains/stresses in several steel systems for understanding the formation mechanisms and their correlation with **microstructural parameters** and **processing parameters**
  - DFXM and 3D Laue micro-diffraction

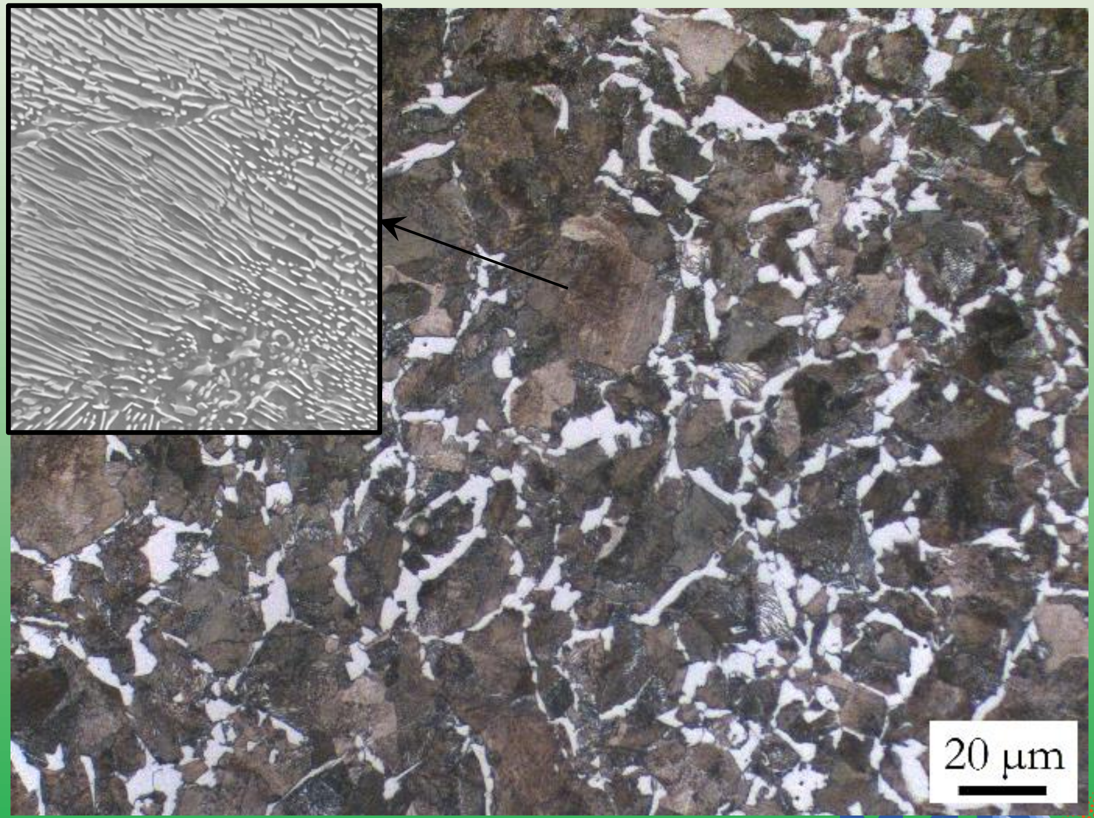
- **Pearlitic steel (phase transformation induced)**
- **Partially-recrystallized pure iron (plastic deformation induced)**

- Concluding remarks

Local residual stresses in pearlitic steel

- Medium-carbon hypo-eutectoid steel
- Wheels and rails in railway industry
- Pro-eutectoid ferrite (soft) + pearlite (hard)
- **Aim of the study:**
  - **Magnitude and distribution of local residual stresses**
  - **Their development upon annealing**

Collaboration with Prof. Johan Ahlström's group from Chalmers University



ER8 grade wheel steel

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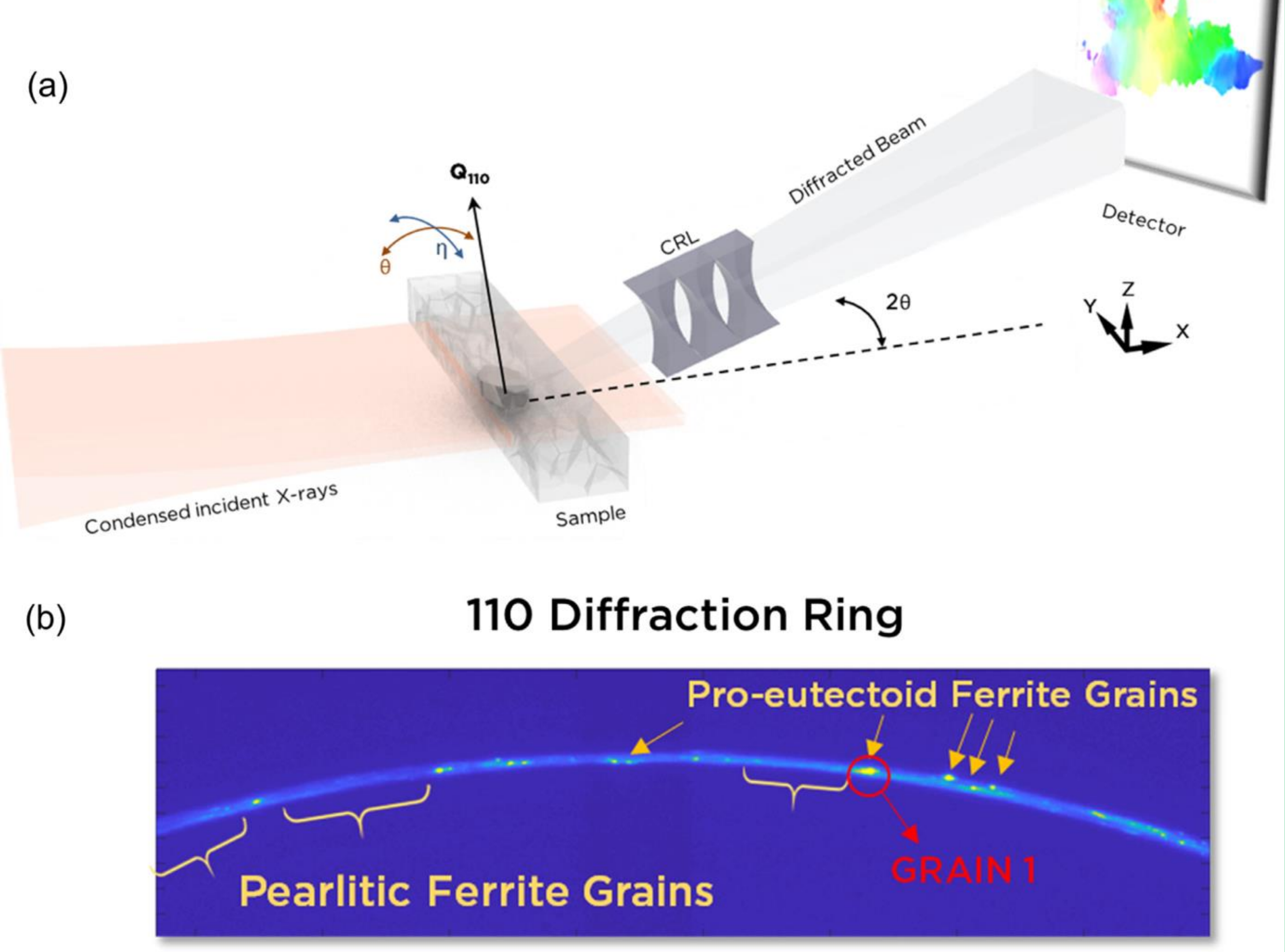
DFXM

- 17keV
- 88 Be lenses
- Magnification factor: 18.67
  - Spatial resolution of **75 nm/pixel** in horizontal and 197nm/pixel in vertical directions

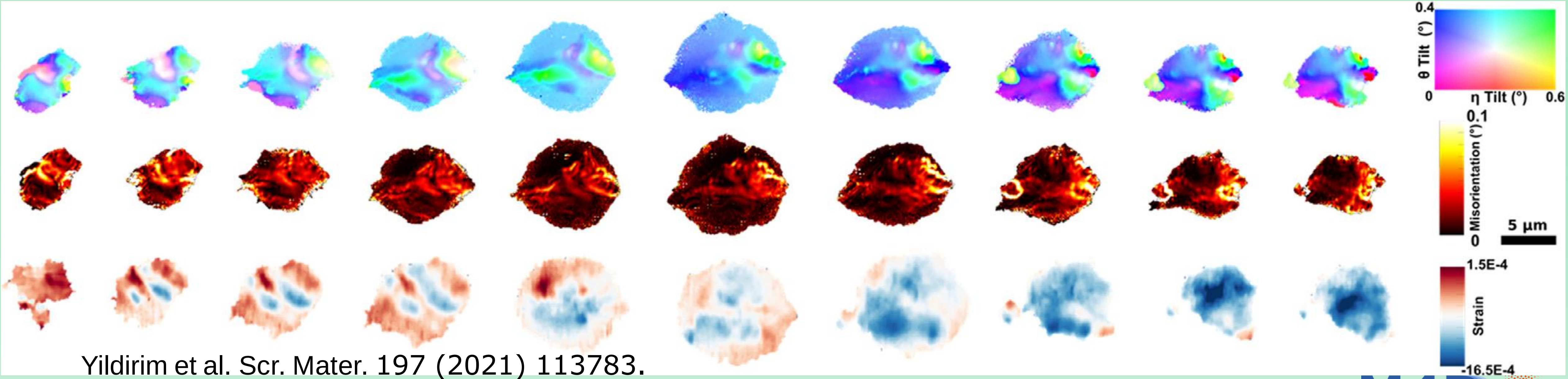
- Lattice parameter determined based on chemical composition
  - a_0 = 2.8673 Å

Together with Can Yildirim and Carsten Detlefs



Microstructure and strain distribution

- Pro-eutectoid ferrite grains are deformed in the as-received condition
  - suggesting that the residual stresses develop already at high temperature, where ferrite are soft, to relax part of the residual stresses
- No clear correlation between the microstructure and the strain distribution
- Systematic strain variation from one side of the grain to the other side
  - Suggesting neighboring grains play more important role

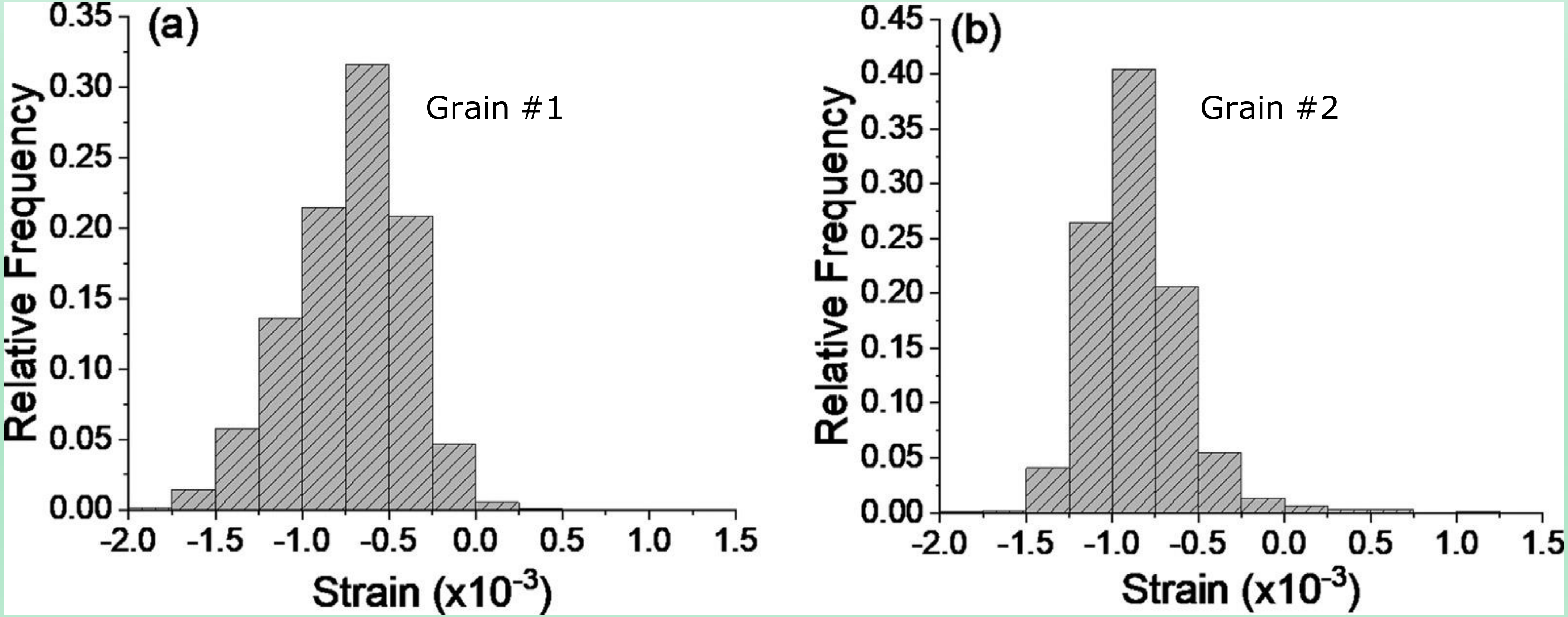


Yildirim et al. Scr. Mater. 197 (2021) 113783.

Metal Microstructures in 4D

Strain variation

- Of the order of $2x10^{-3}$

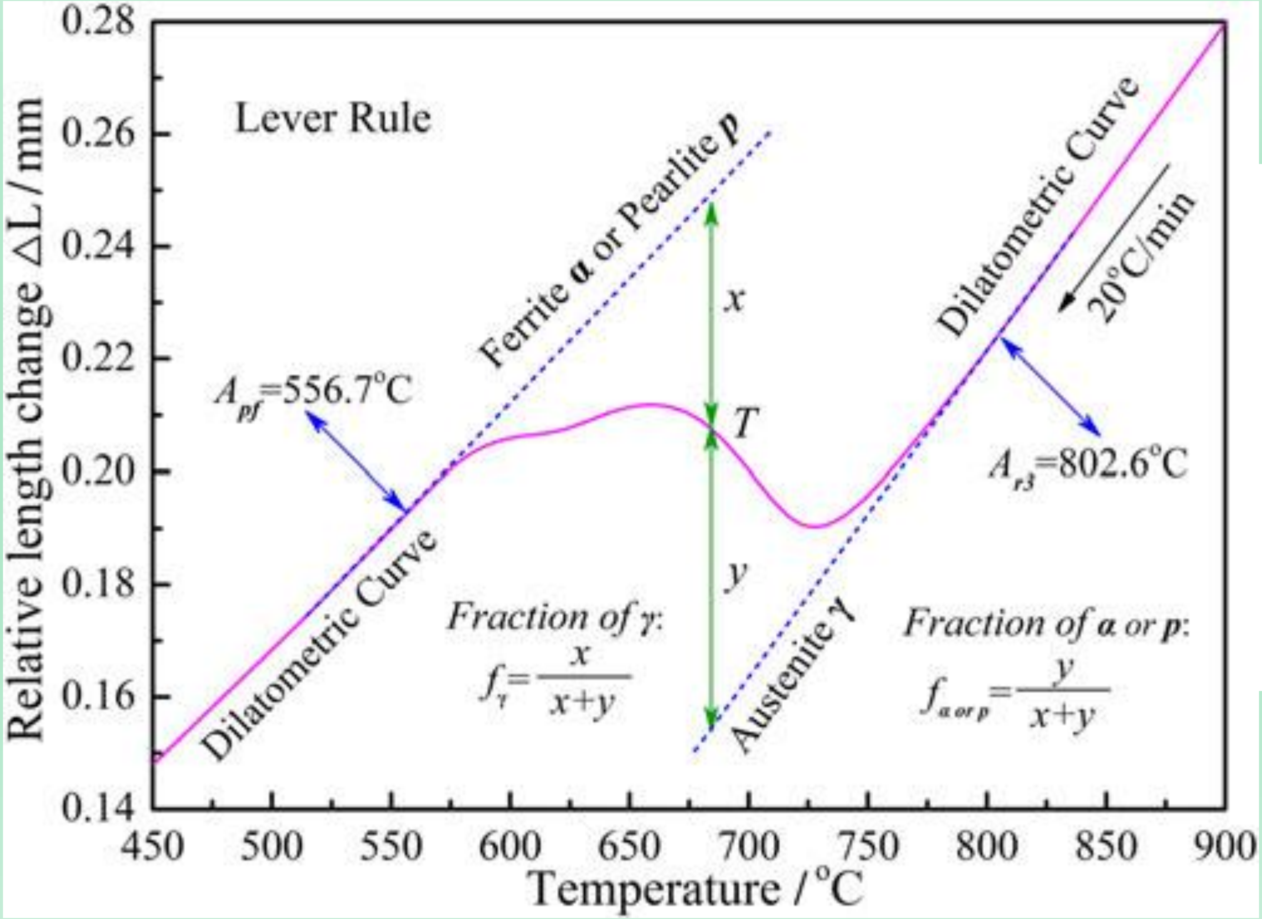


Yildirim et al. Scr. Mater. 197 (2021) 113783.

Metal Microstructures in 4D

Formation mechanisms

- Phase transformation induced local residual stresses
- Thermal stresses should be small



Liu et al. J Mater. Res. 33,2018

Table 3
Thermal expansion coefficient of cementite and ferrite [23].

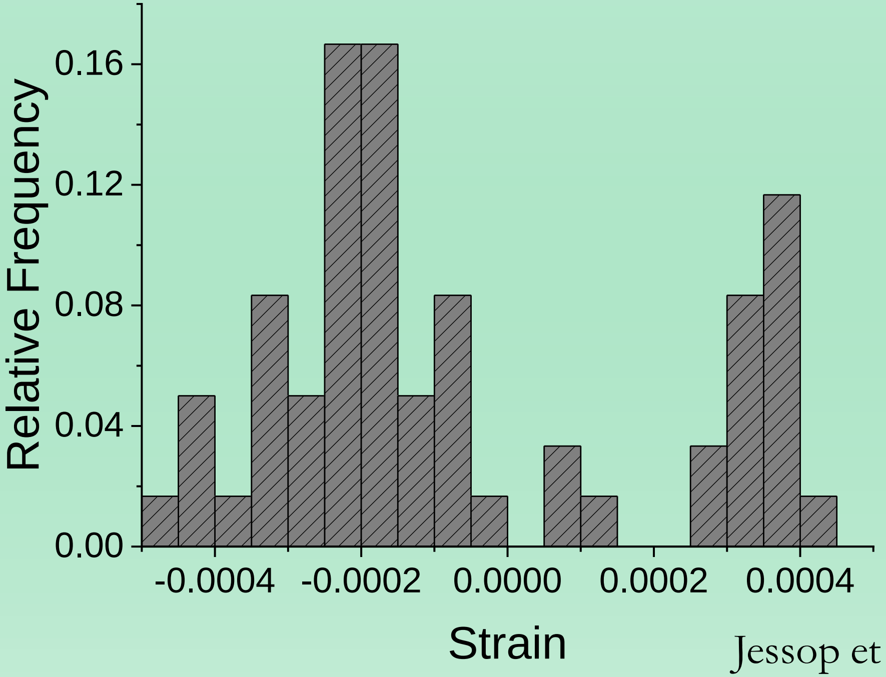
	Thermal expansion coefficient ($10^{-5}K^{-1}$) 300-420°C	500- 640°C
Fe_3C	1.54	1.74
Ferrite	1.45	1.50

Residual strains after annealing

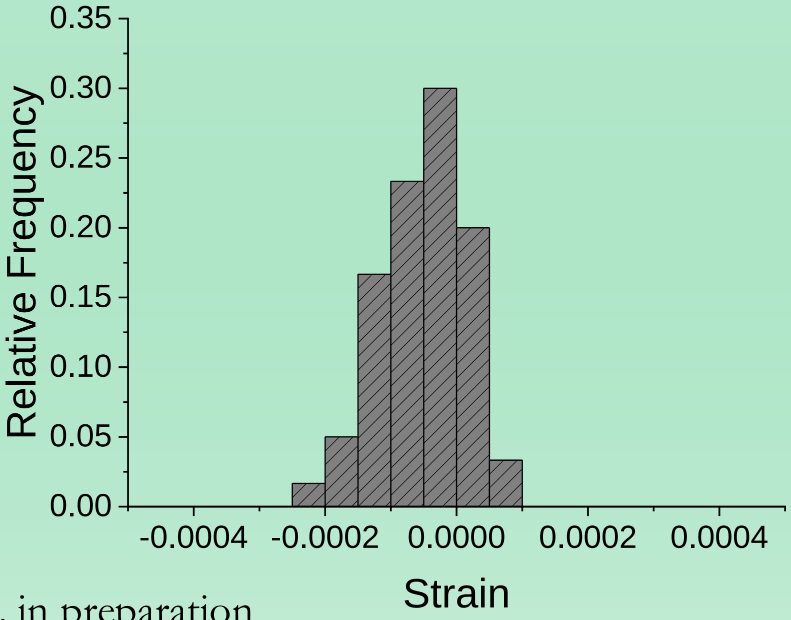
- Measurements on 3 ferrite grains using 3D micro-diffraction
- Range of residual strains reduces
  - Further supporting that the residual stresses developed are not due to CTE differences between ferrite and cementite

As-received: $2x10^{-3}$

300° C, 4h: $8x10^{-4}$



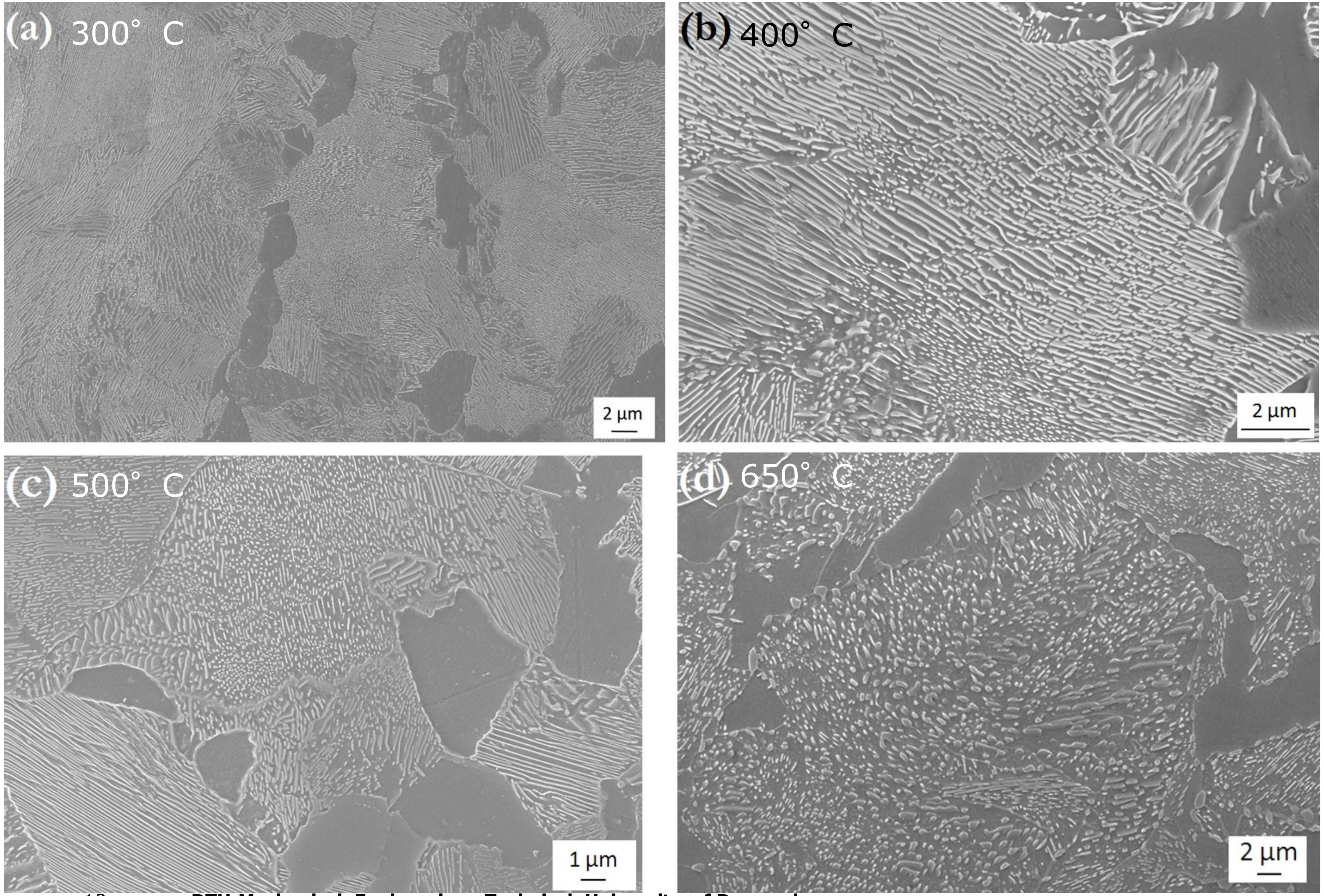
500° C, 4h: $3x10^{-4}$

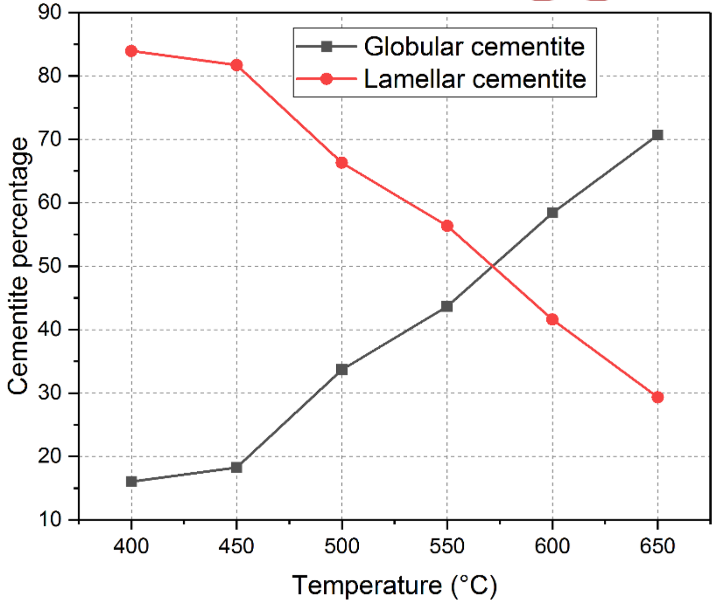


Jessop et al. in preparation

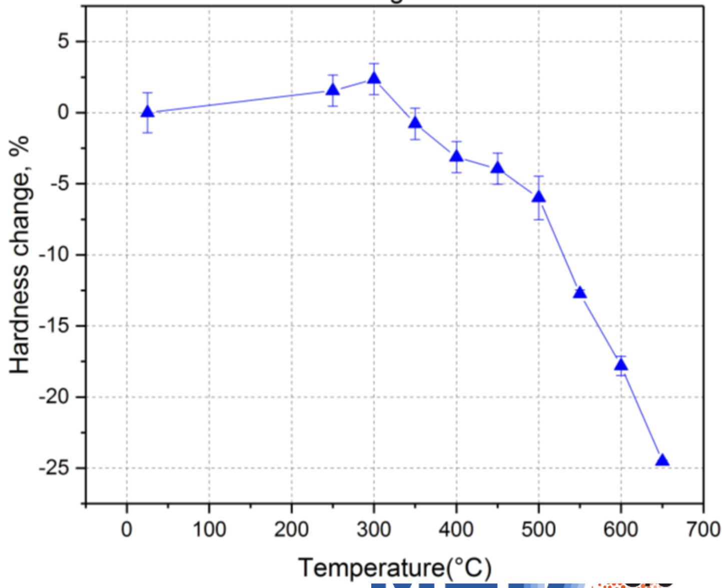
Metal Microstructures in 4D

Microstructure evolution during annealing





Hardness testing-Undeformed R8T



Jessop et al. in preparation

Metal Microstructures in 4D

Summary

- Local residual strains in the order of 2×10^{-3} develop in pro-eutectoid ferrite grains during manufacturing due to **phase transformation**.

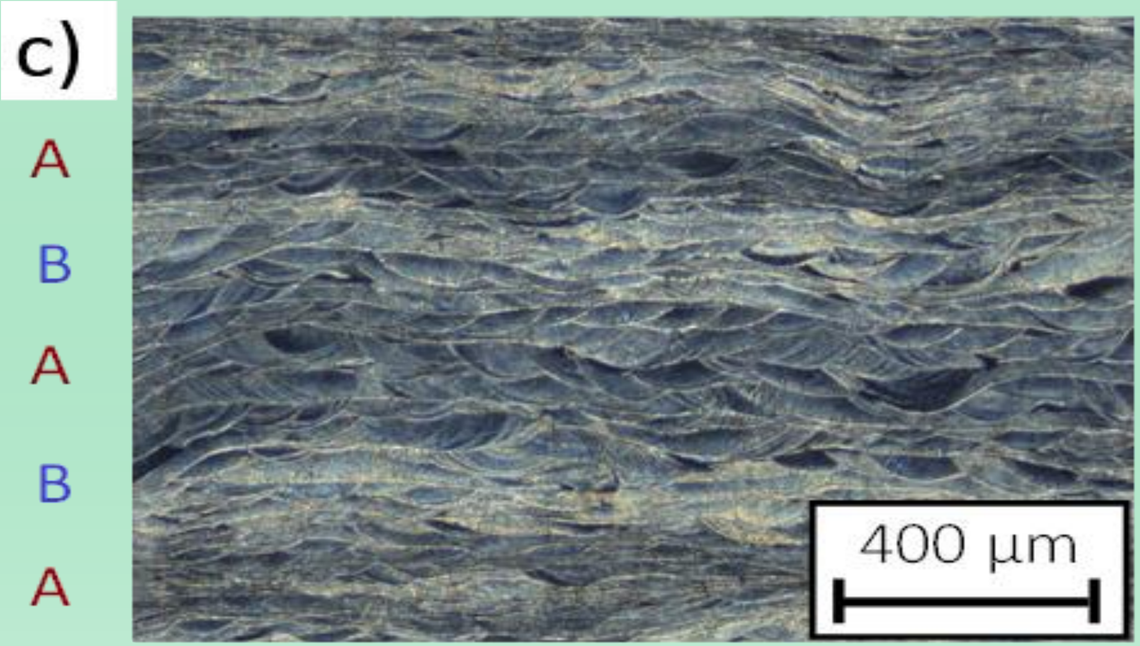
- Residual stresses release after annealing at high temperatures, suggesting that thermal residual stresses is small.

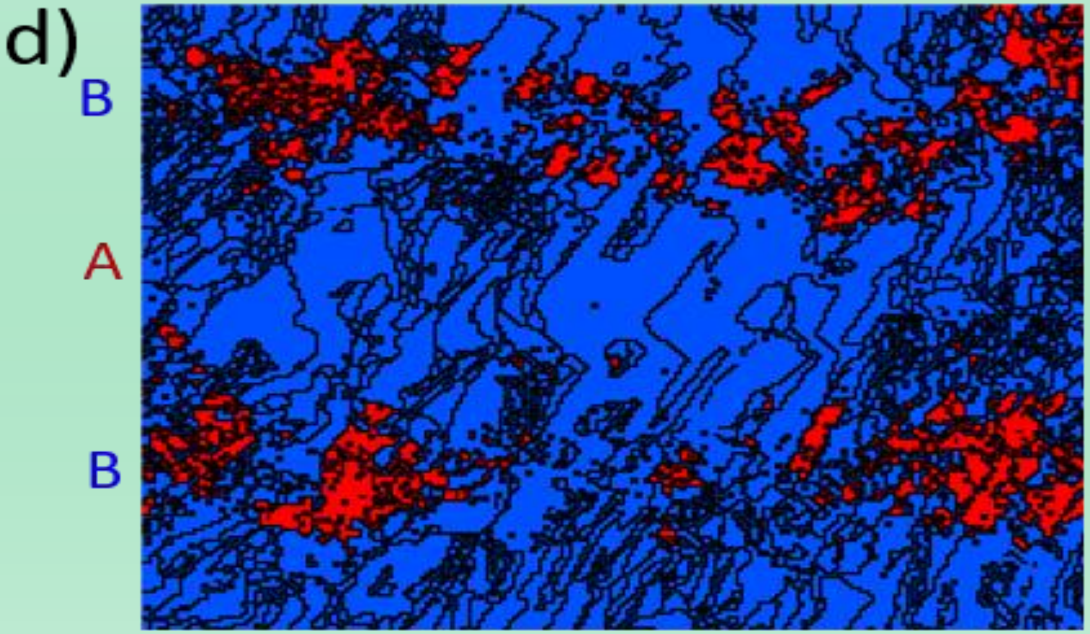
- Local residual stresses should be considered when evaluating the mechanical properties of pearlitic steel.

Future plans

- In situ studies to follow the microstructural and strain evolution of pearlitic steels
- To study effects of manufacturing processing parameters (e.g. cooling rate, and plastic deformation)

- Different manufacturing methods
  - Multilayered steel-steel composite produced using AM



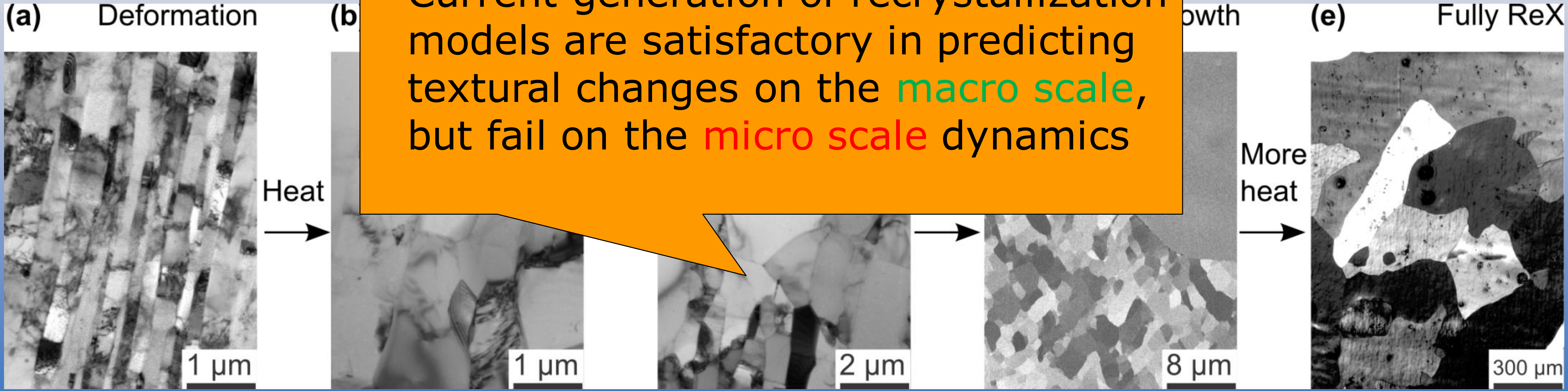


Nadimpalli et. al. Manuf. Lett. 28 (2021) 46-49.

Metal Microstructures in 4D

Local residual stresses in partially-recrystallized pure iron

- Recrystallization is an important industrial processing method, providing large windows for optimizing material texture and properties
- It is generally assumed that recrystallized nuclei/grains are defect-free and strain-free



Current generation of recrystallization models are satisfactory in predicting textural changes on the macro scale, but fail on the micro scale dynamics

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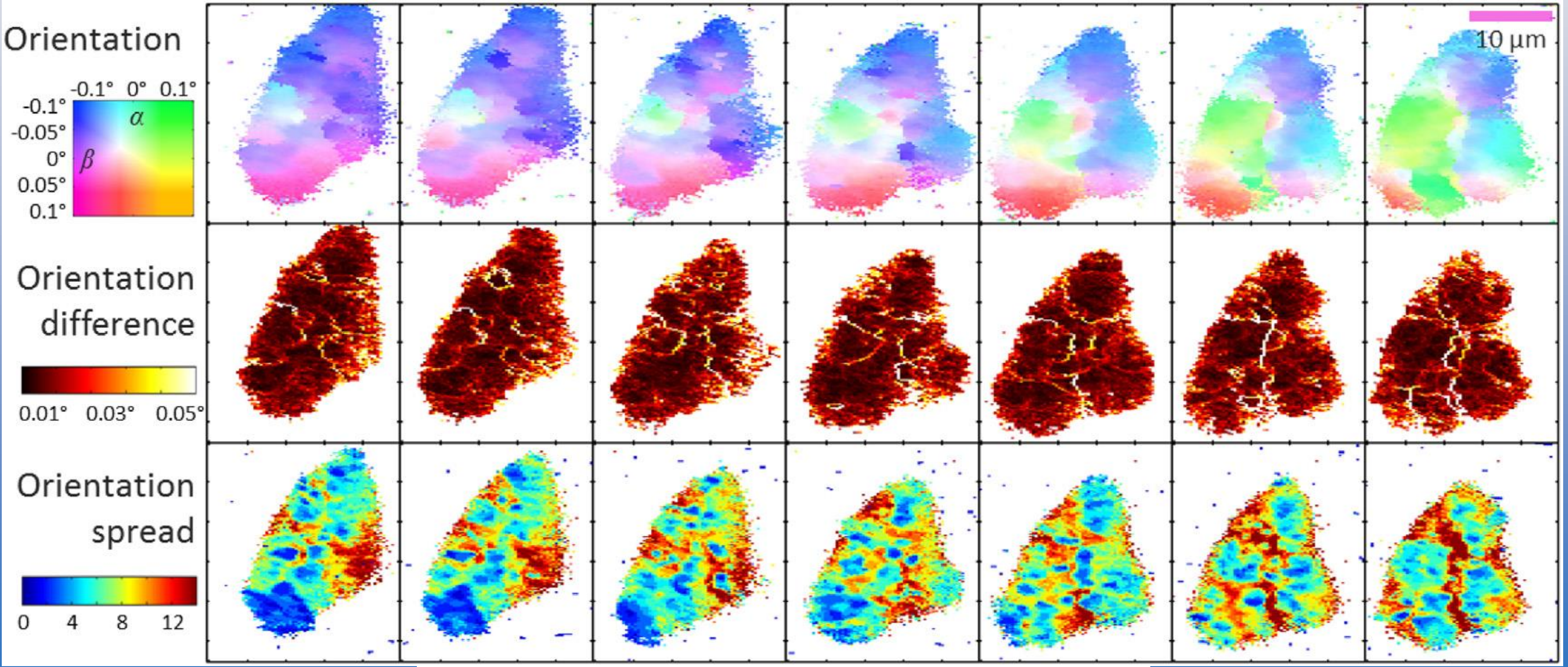
Aim:

- To study whether significant local residual stresses present in recrystallized grains

- Collaboration with Dr. Tianbo Yu, Prof. Dorte Juul Jensen from DTU and Prof. Andy Godfrey from Tsinghua University

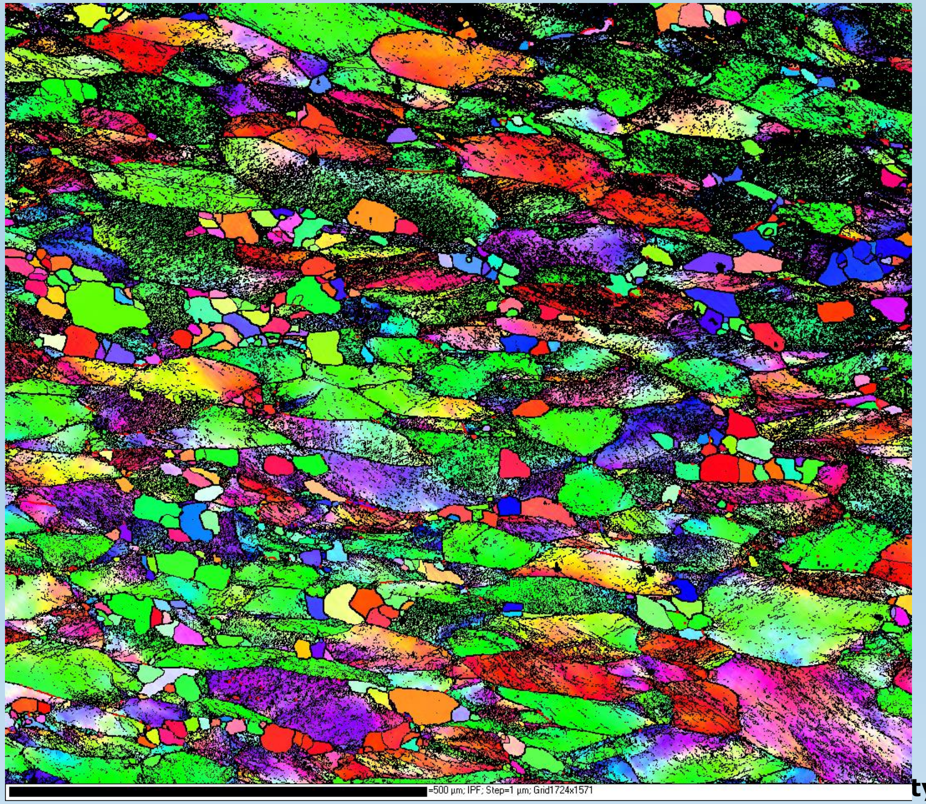


Ahl et al. Scr. Mater. 139 (2017) 87–91

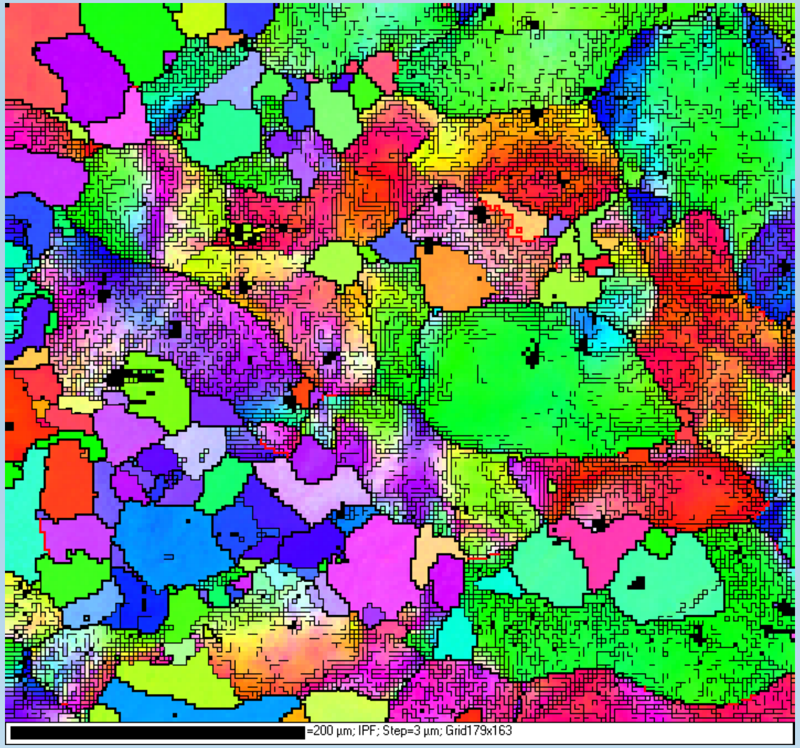
Armco iron

- Cold rolled to 50% and 90% - **plastic strain**
- Annealed at 550ºC and 500ºC for different time – **recrystallization volume fraction**
- Water quench (WQ) and air cooling (AC) – **cooling rate**

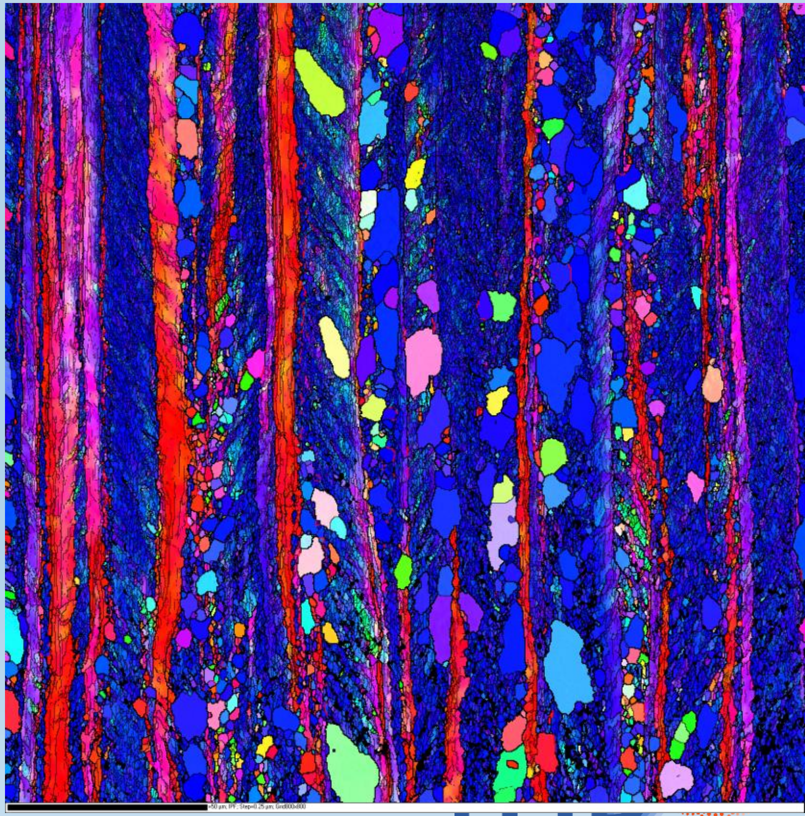
CR50_550_10min – 10% rex



CR50_550_30min – 60% rex

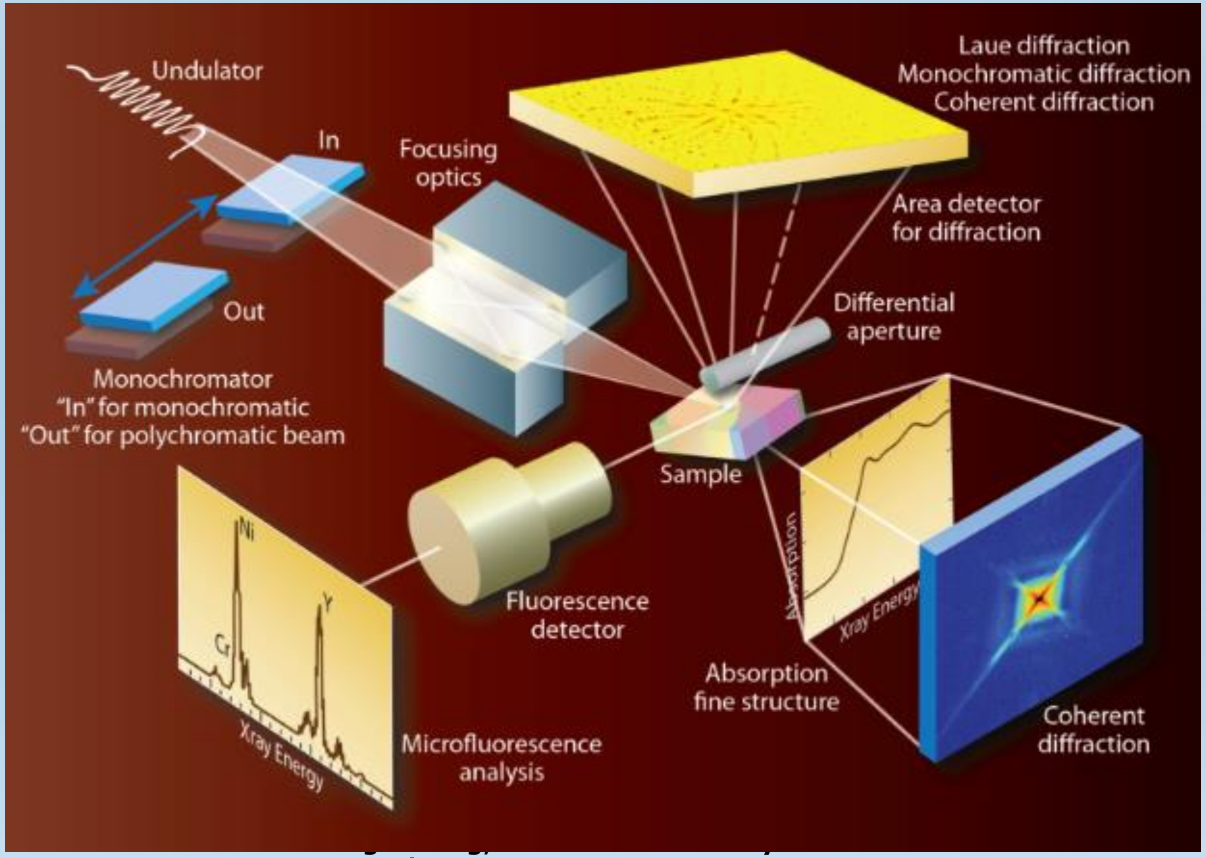


CR90_500_30min – 10% rex

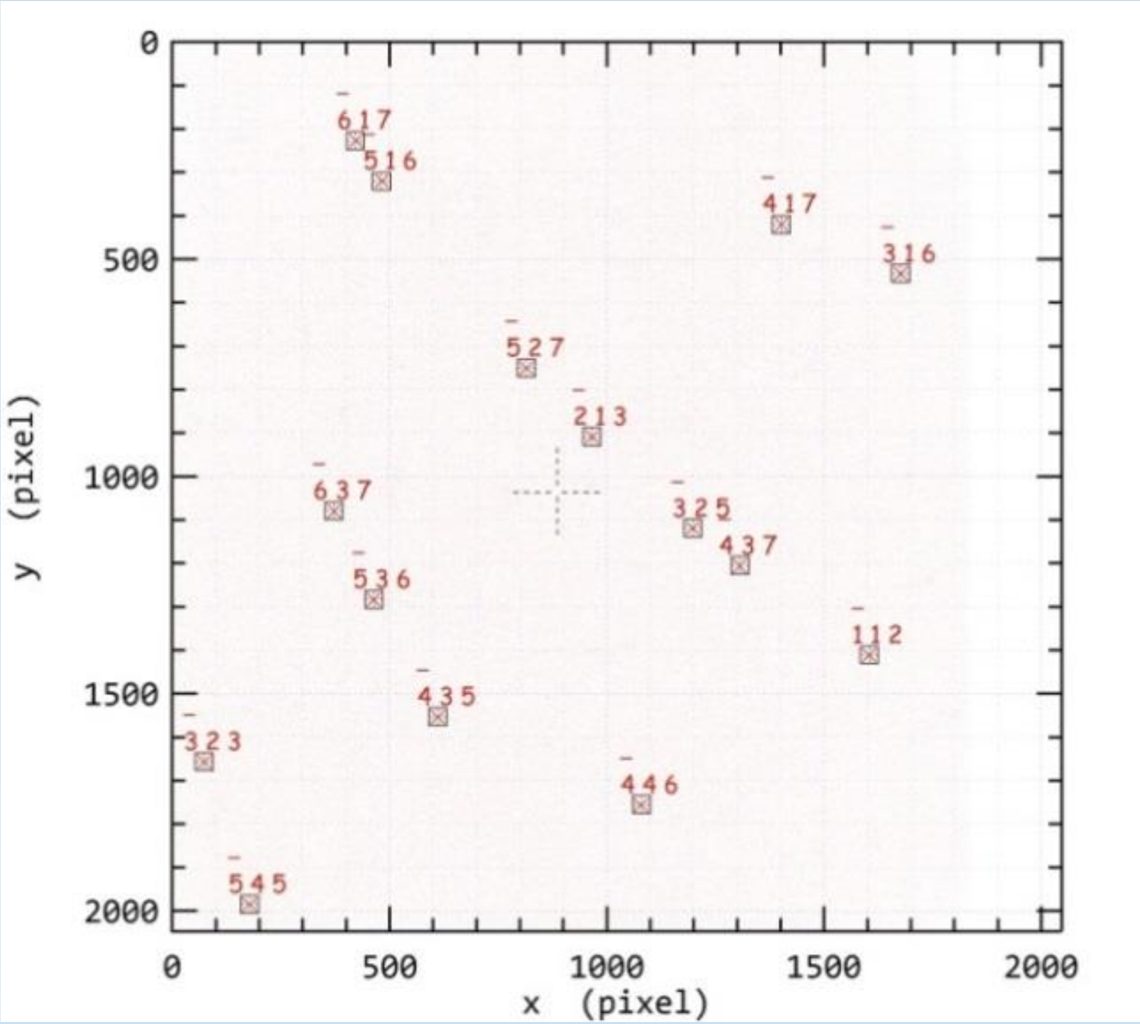


3D micro-diffraction (3DµXRD)

- Crystallographic orientation and deviatoric strain tensor based on Laue pattern
- Combining with differential aperture
  - Local depth-resolved information

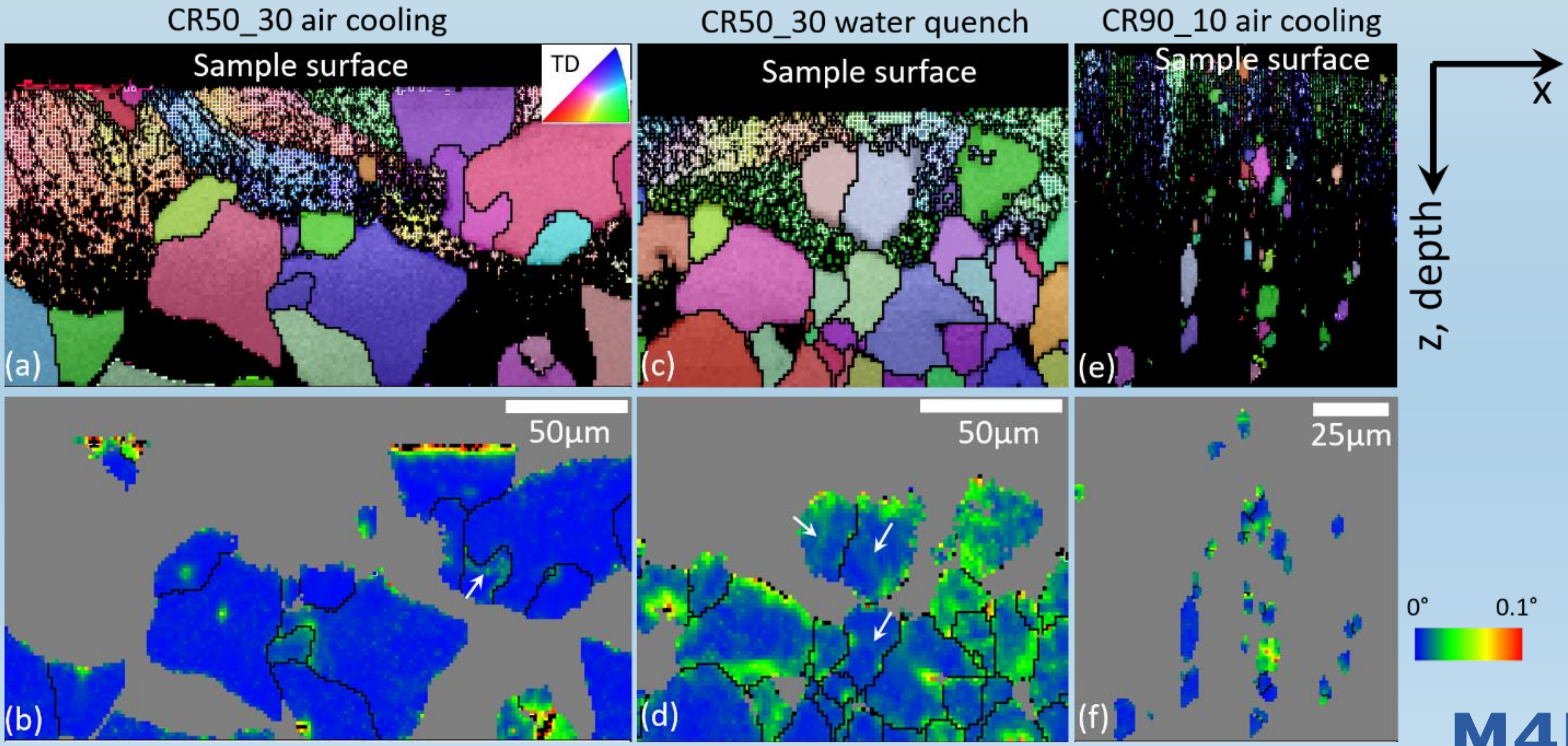


Ice G. et al. Science 334,2011, 1234.



Microstructure (3DμXRD)

- Recrystallized grains are not defect-free
- Water quench introduces more defects



Zhang et el. In preparation

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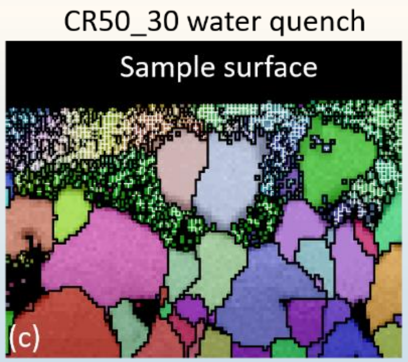
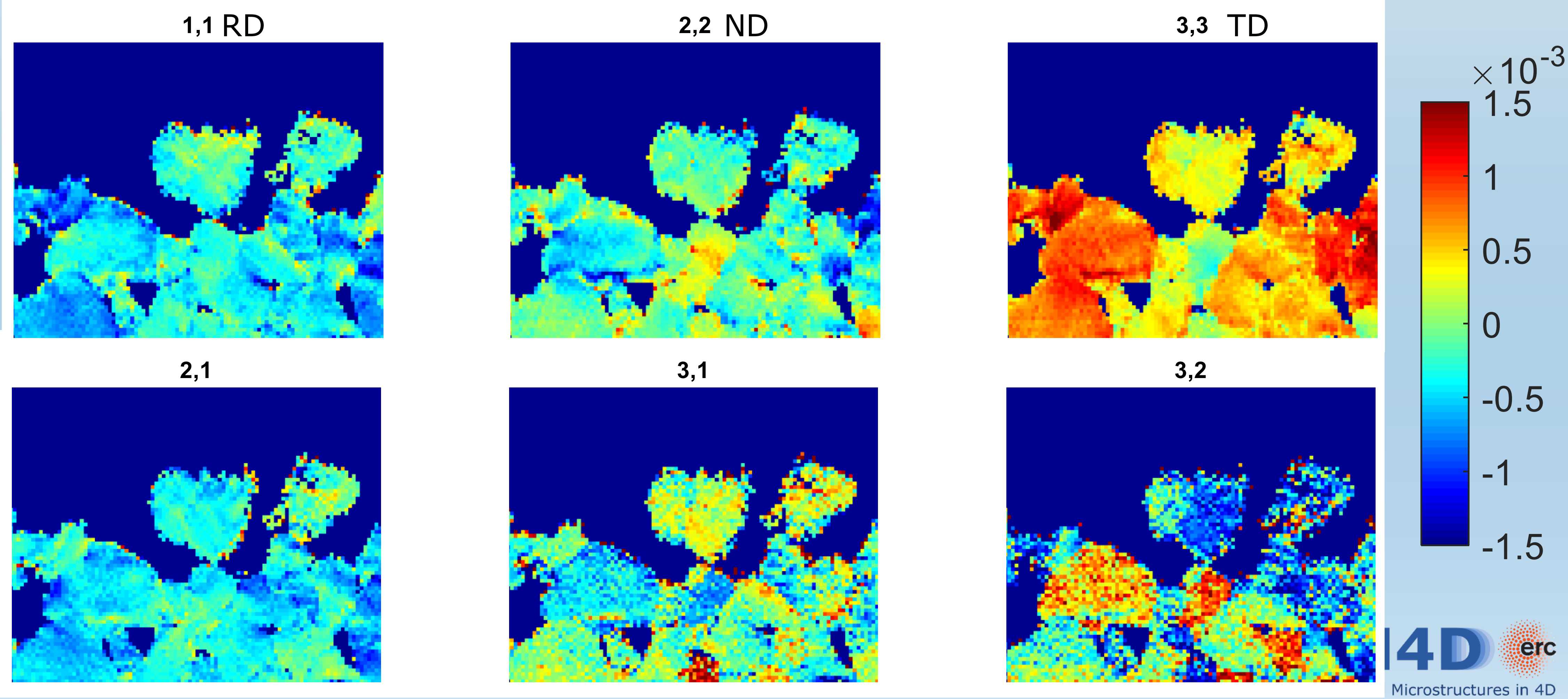
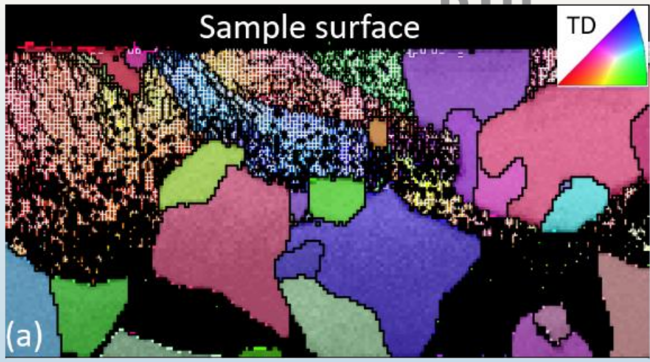
Residual elastic strains (water quenched)

- Strain components along different sample directions are different
  - Macroscopic **rolling deformation** is important
- Variations within grains and between grains
  - Local **microstructure** is important



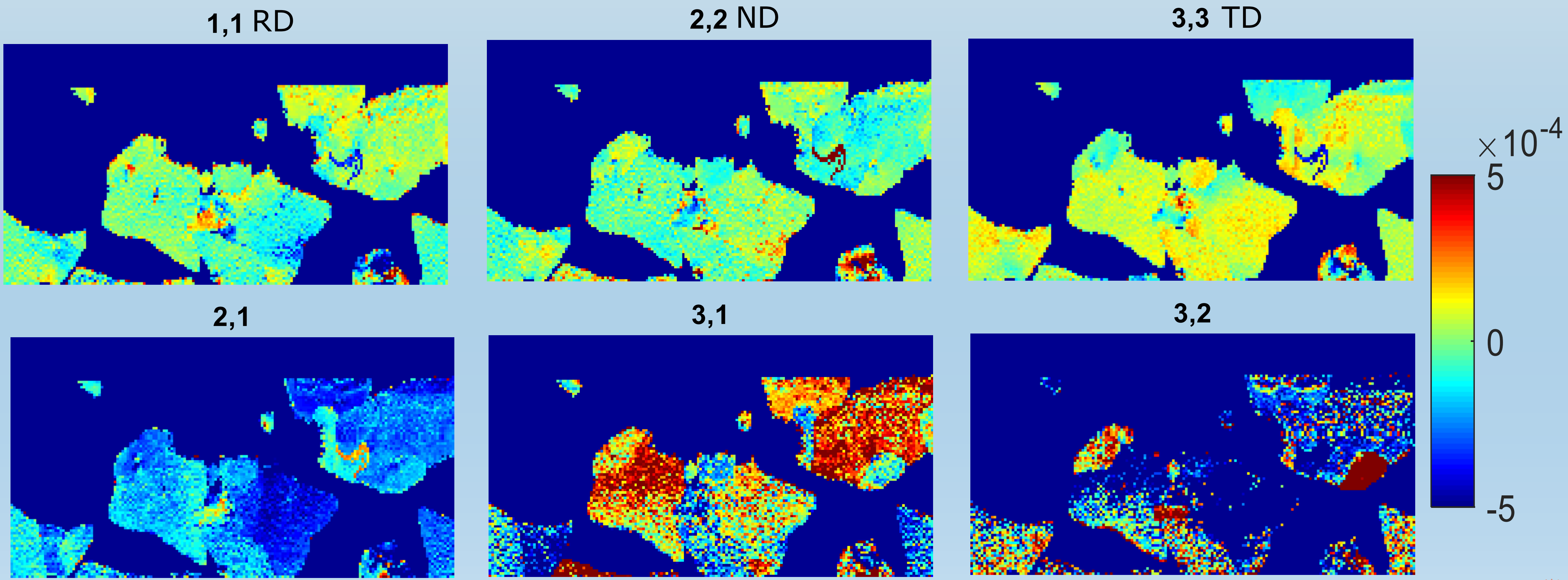


CR50_30 air cooling



Residual elastic strains (air cooled)

- Similar patterns
- Magnitude is lower: **Cooling rate** has strong impact on the residual stresses



Zhang et el. In preparation

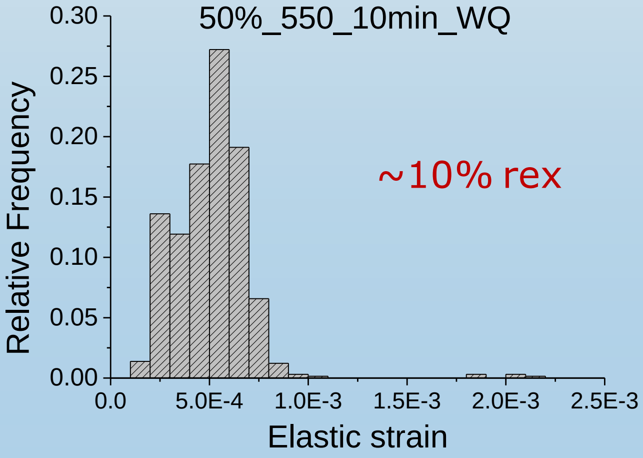
Effects of recrystallization volume fraction and plastic strain

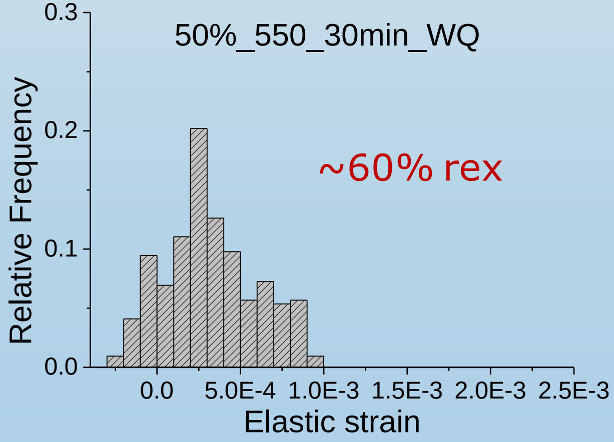
- The residual strains decrease with increase in **fraction** of recrystallization
- The **rolling strain** has a less impact on the residual strains

CR50% CR90%

ε_{33} TD

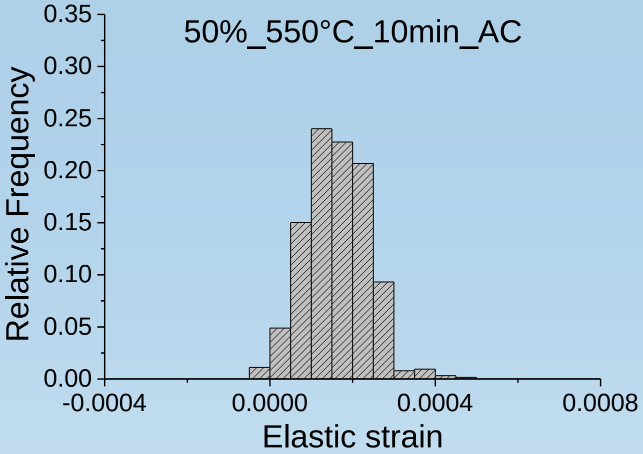
WQ

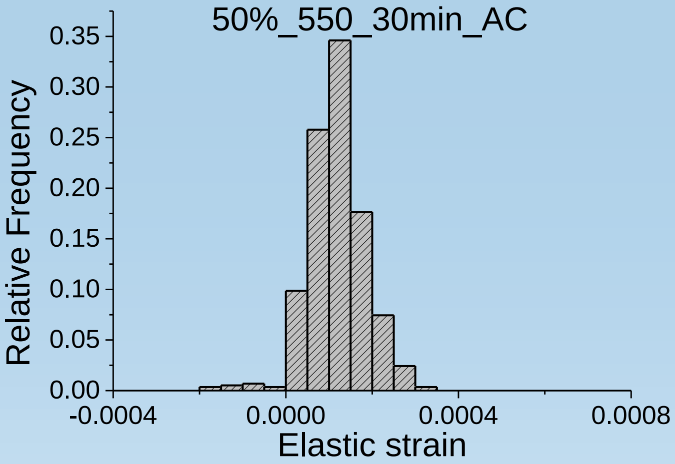


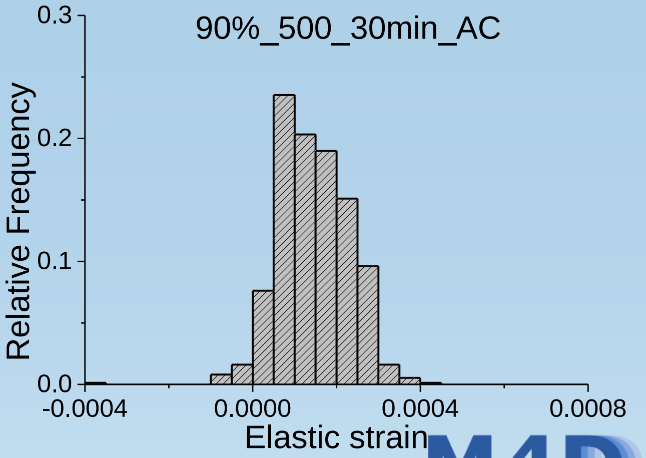


~10% rex

AC

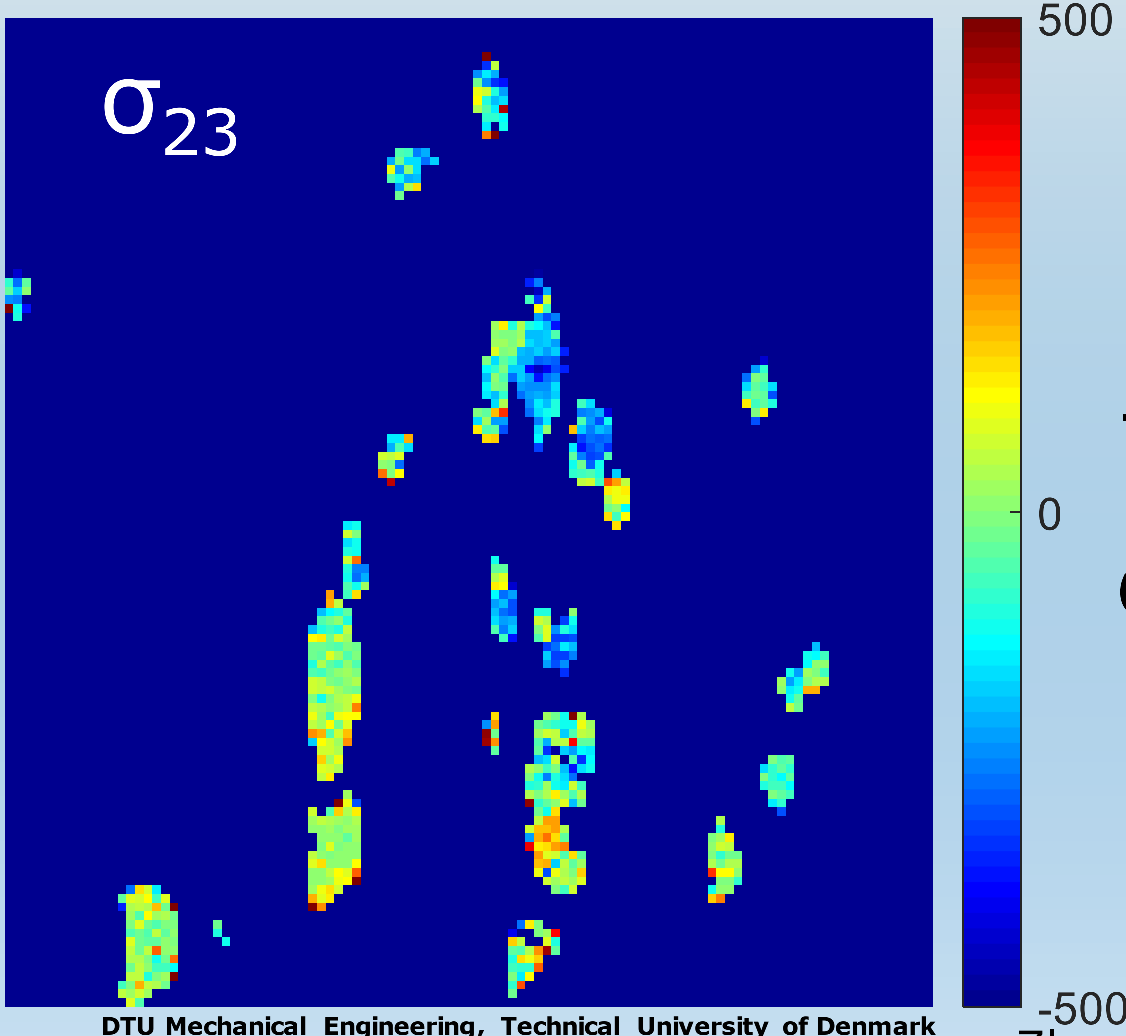


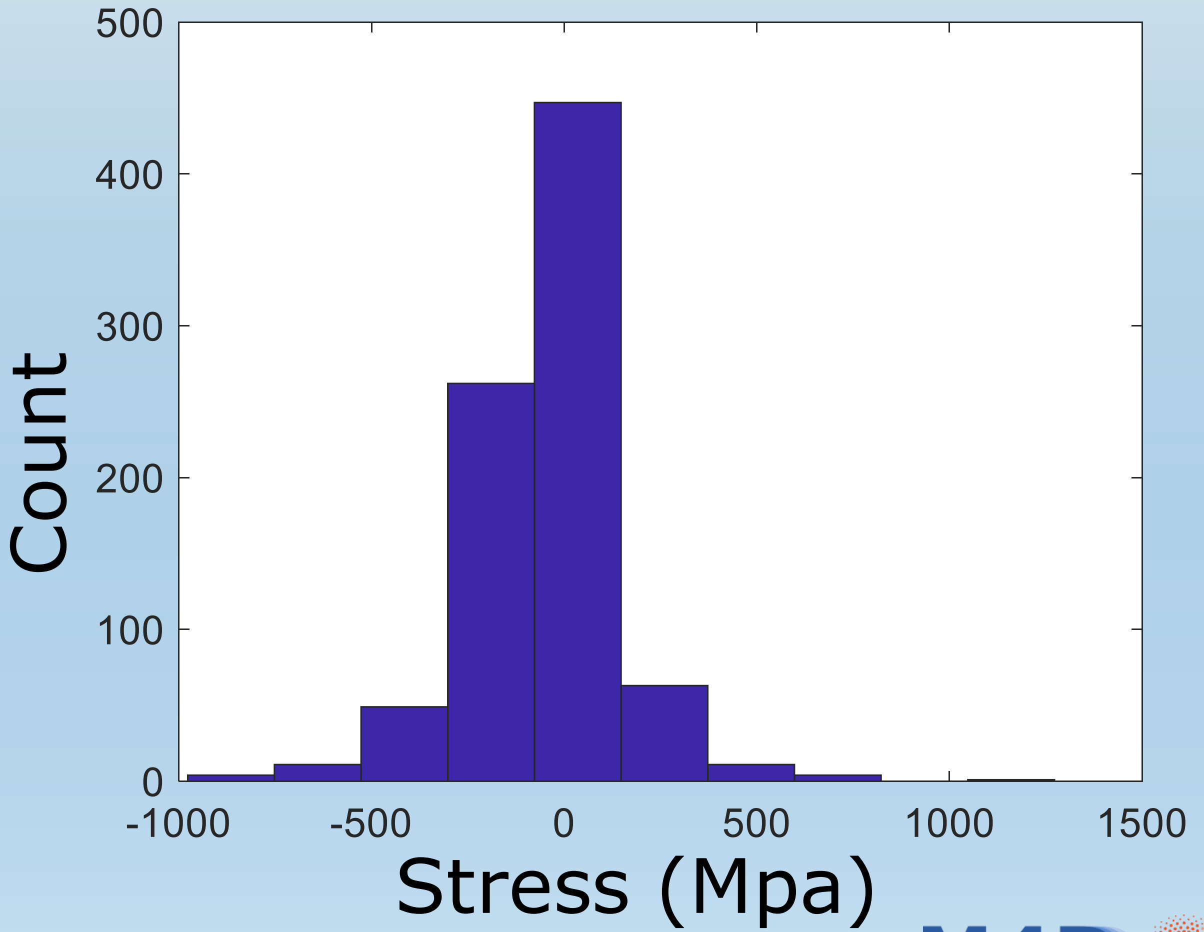




Zhang et el. In preparation

Residual shear stresses (CR90, air cooling)





Zhang et el. In preparation

Summary

- A **non-uniform distribution** of local residual stresses/strains is seen within recrystallized grains in partially recrystallized pure iron.

- The strain components along different sample directions are significantly different, suggesting that **macroscopic deformation** plays an important role for the residual stresses.

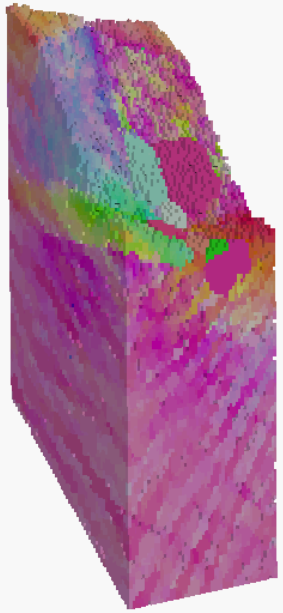
- The strain level is affected significantly by the **cooling rate**, while the volume fraction of recrystallized grains and plastic strain of the deformed sample play a less important role.

- The presence of local residual stresses is suggested to have an influence on the microstructural evolution during thermo-mechanical processing.

Future plans

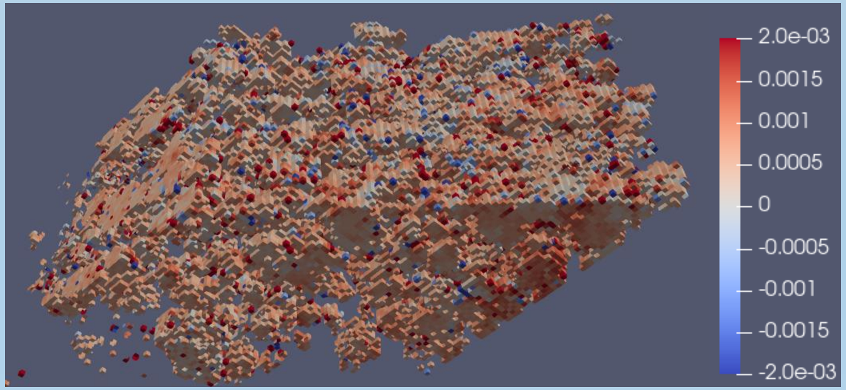
- Nucleation (new PhD project)
  - In-situ study to follow the recovery and nucleation process of **deformed** metals
  - Pure iron and gummetal
- Grain growth (after recrystallization) – strain level 10^{-5}
  - High spatial resolution of DFXM is essential

Nucleation



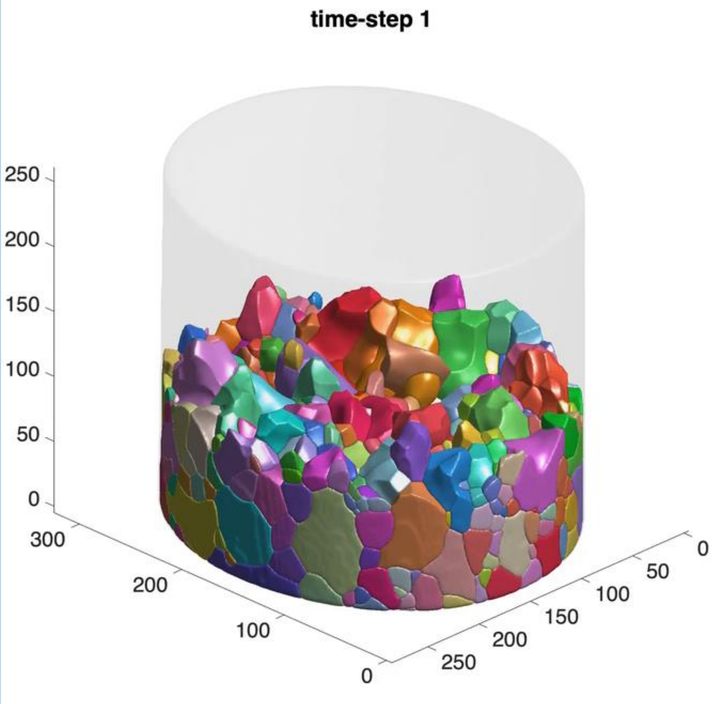
Xu C. et al. Sci Rep. 2017

Gum Metal



Lindkvist et al. in preparation

Grain growth



Zhang J. et al. Acta Mater. 191 (2020) 211-220

Concluding remarks

- Synchrotron 3D X-ray techniques are powerful tools, giving new possibilities for metal research
  - 4D **full-field** characterization

- The results have proven that significant levels of local residual stresses develop even in microstructures which are believed to be stress-free (e.g. single-phase partially recrystallized materials) and play important roles for the microstructural evolution during both manufacturing and post-loading

- Local residual stresses have to be included in the development of new theories and advanced models that can be used for optimization of products (e.g. decrease the magnitude of safety factors) and design of new steels