EBSD measurement of deformation induced dislocation sub-structures

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MTEX workshop 2021

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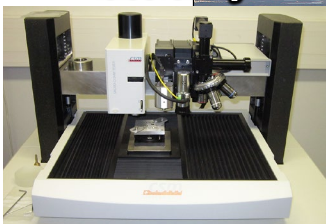
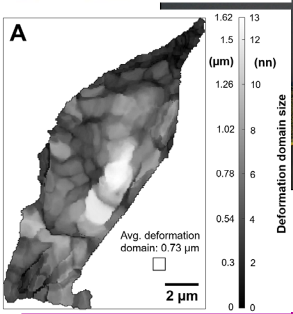
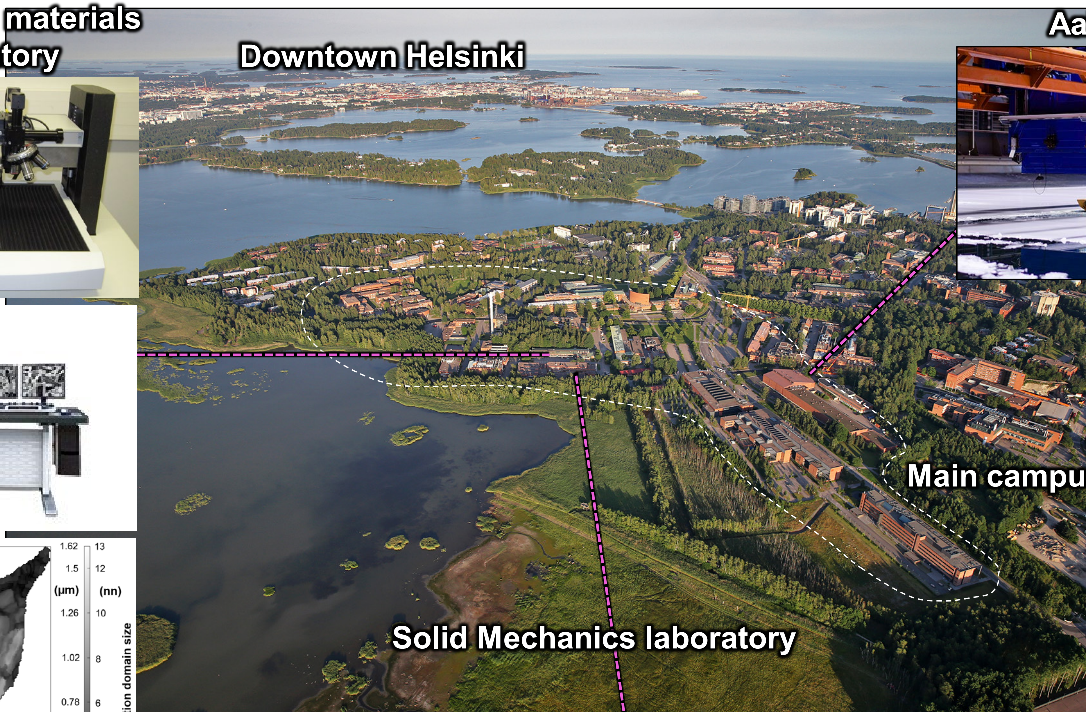
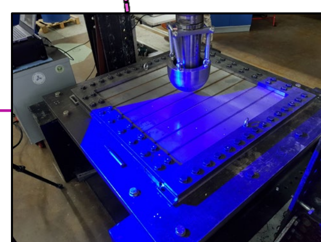
Engineering materials laboratory

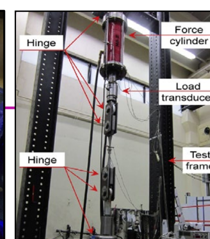


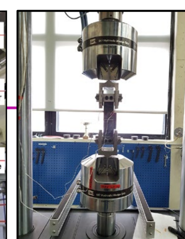




Aalto Ice Tank

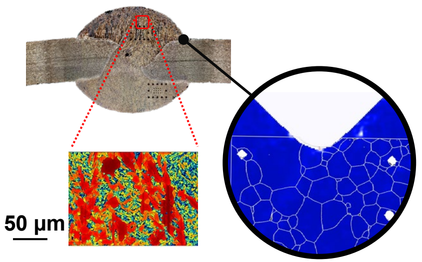






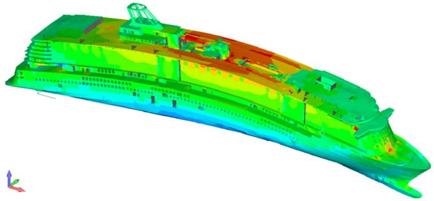
Research focus

Strength of materials



Micro-scale

Mechanics of structures



Macro-scale

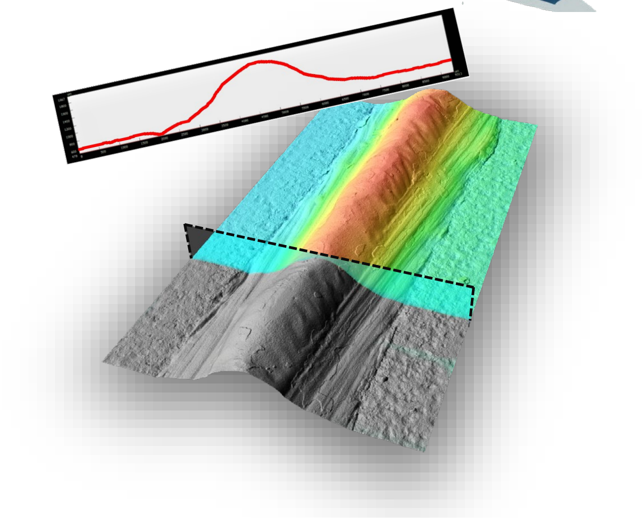
Structural engineering

Full-scale

Background

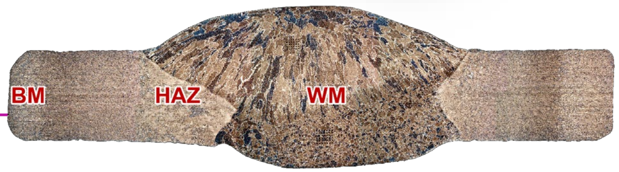
Sustainable growth requires the effective use of welded high strength steel

- Steel's mechanical properties are affected by the manufacturing process



Research in the microstructural length scale reveals deformation mechanisms

- Optimisation of materials and manufacturing processes made possible by understanding the plastic deformation processes in the microstructural scale



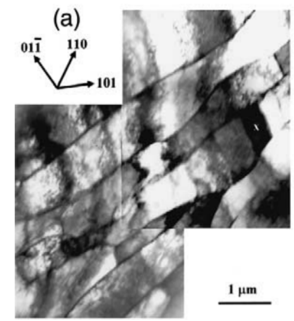
Plastic deformation in polycrystalline materials

Deformation mechanisms need to be characterized in the microstructural scale in order to fully utilize high strength materials in large scale structures

- For polycrystalline materials heterogeneous intragranular deformation patterns are developed during plastic deformation
- Typically TEM is required to resolve the dislocation sub-structure

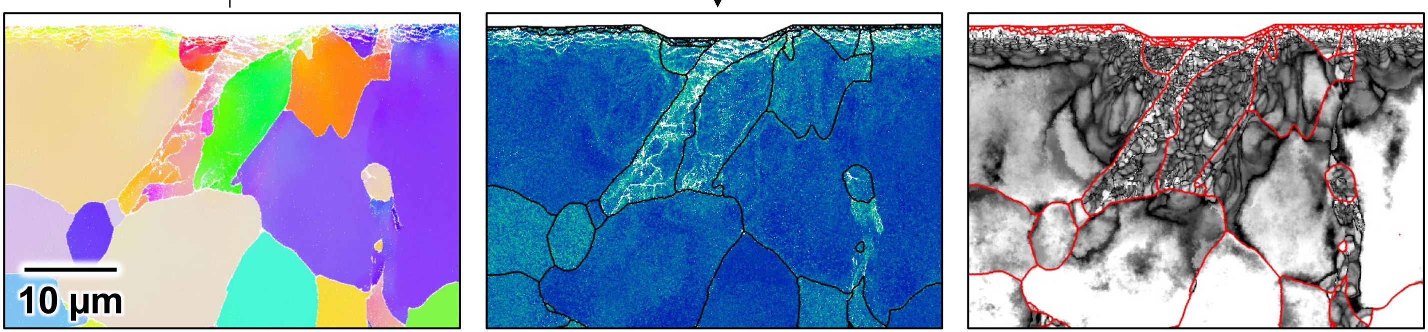
SEM/EBSD is not conventionally able to resolve e.g. the dislocation cells **=> Tailored approach for the cell-forming mechanism**

TEM, Fe sample [1]



SEM/EBSD

Conventional EBSD analysis



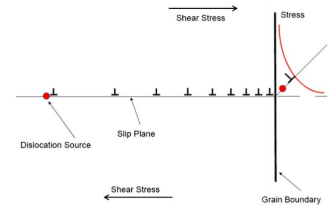
Developed approach

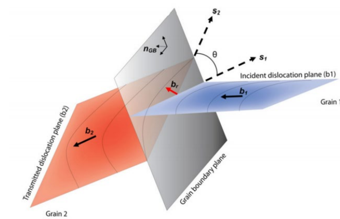
[1] **N.R. Tao, Z.B. Wang, W.P. Tong, M.L. Sui, J. Lu, K. Lu**, *An investigation of surface nanocrystallization mechanism in Fe induced by surface mechanical attrition treatment*, Acta Mater. 50 (2002) 4603–4616. https://doi.org/10.1016/S1359-6454(02)00310-5.

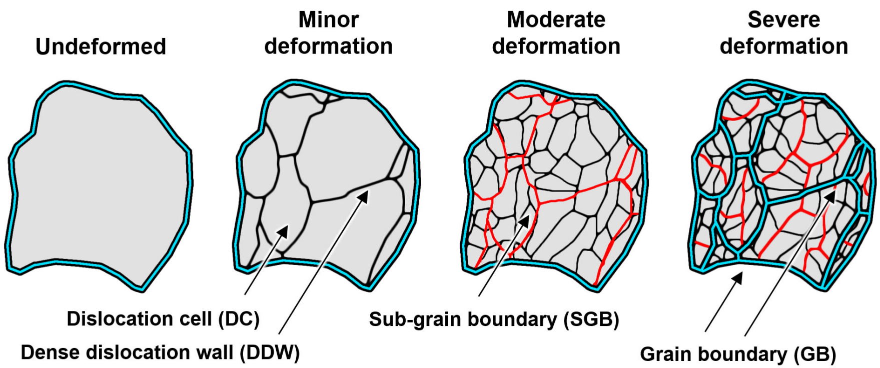
Dislocation-mediated plastic deformation

Cell-forming deformation process in polycrystalline material

- Grain boundaries and other interfaces restrict the rotation of the crystal lattice and inhibit the motion of dislocations
- Heterogeneous intragranular deformation patterns are generated to achieve compatibility and an equilibrium energy state
- The cell-forming deformation process is commonly observed for many materials







Accommodation of strain

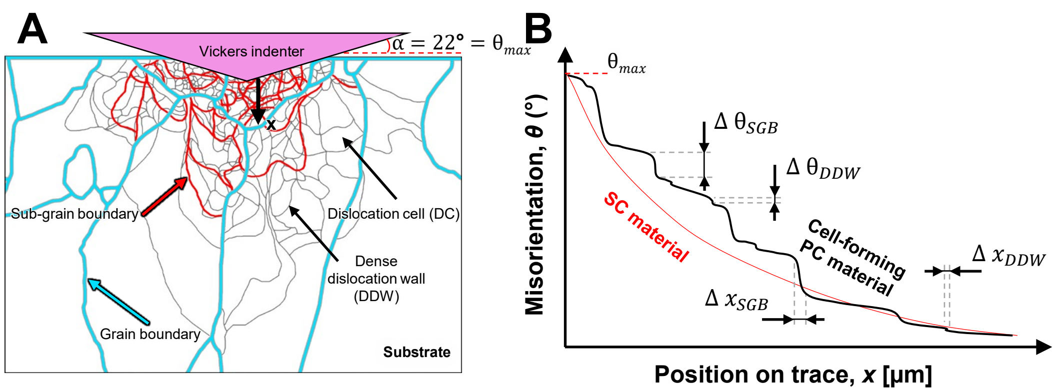
Local plastic deformation (hardness indentation)

- Grain boundaries restrict plastic deformation => dislocation re-arrangement influences lattice curvature

- Lattice curvature, i.e. misorientation gradient, has local variations at the sub-structural boundaries; gradient much higher compared to interiors

- Thickness (Δx) and misorientation ($\Delta \theta$) of the boundary region varies

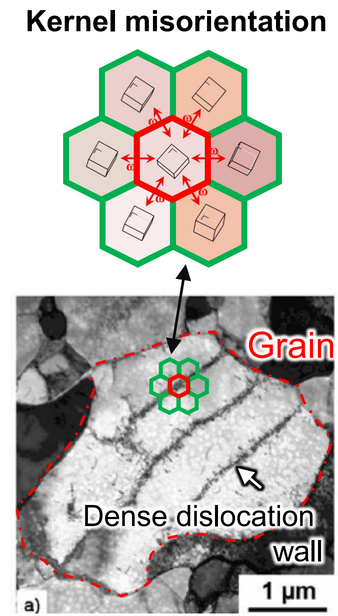
  => *Difficult to assess using conventional EBSD analysis, especially the dense dislocation walls*



Dislocation sub-structures and EBSD

Kernel average misorientation (KAM)

- Often used for qualitative assessment of strain localization with EBSD
  - *Scalar value of KAM represents GND density, i.e. lattice curvature*
  - *Corresponds to applied macroscopic strain when averaged over a large area*



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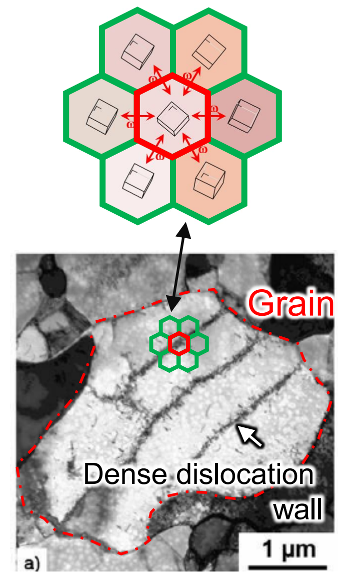
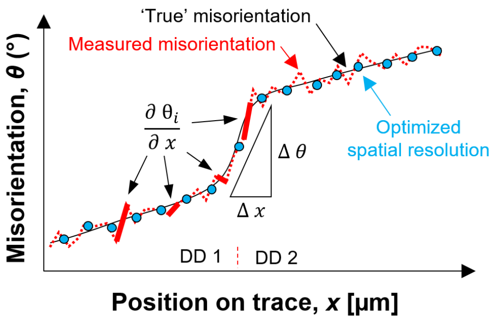
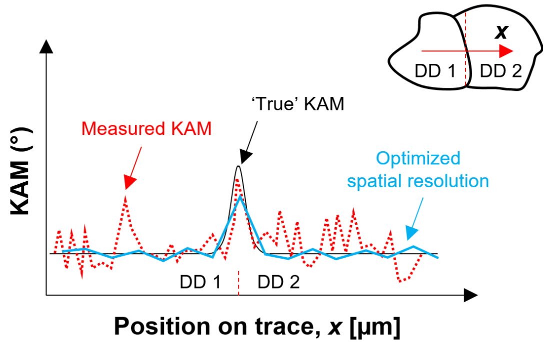
- Sensitive to angular and spatial resolution
  - *Misorientation of DDWs approximately the same as angular resolution of Hough based conventional EBSD (~0.5°)*
  - *Highest scalar value achieved when spatial resolution (Δx) maximizes the measured misorientation ($\Delta\theta$), but thickness of boundary region varies and is unknown prior to the measurement*

=>KAM is not effective for resolving dislocation sub-structures

Kernel misorientation



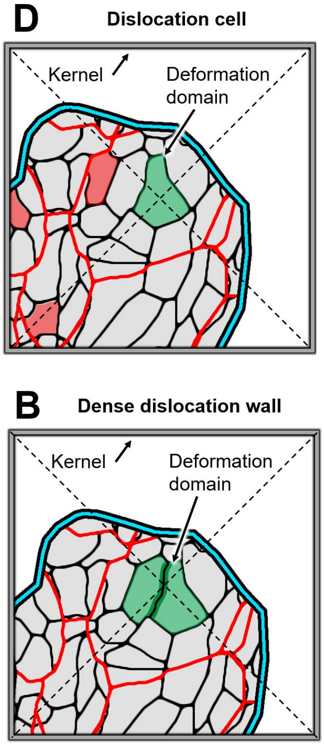




Dislocation sub-structures and EBSD

Adaptive domain misorientation approach (DAM / DMM)

- Developed for characterizing the dislocation sub-structures for cell-forming polycrystalline materials
  - *Measurement area is adapted to the size and shape of the sub-structures, misorientation threshold e.g.* $\Delta\theta_{DDW} = 0.5°$ *and* $\Delta\theta_{SGB} = 2°$ *(Based on [1])*



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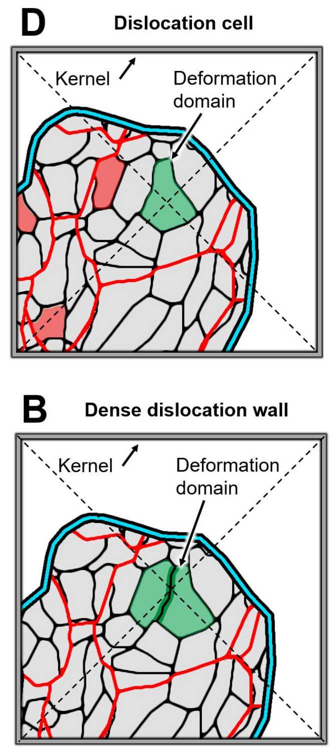
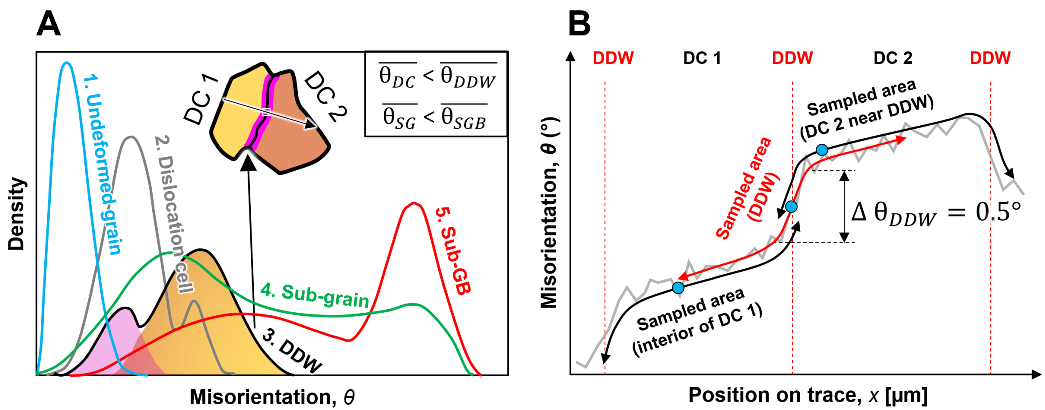
  - *Average / median misorientation of the deformation domain is an effective proxy to changes in lattice curvature*
  - *Enables detection of sub-structural boundaries with varying thickness (*Δx*) and misorientation (*$\Delta\theta$*) using conventional EBSD*





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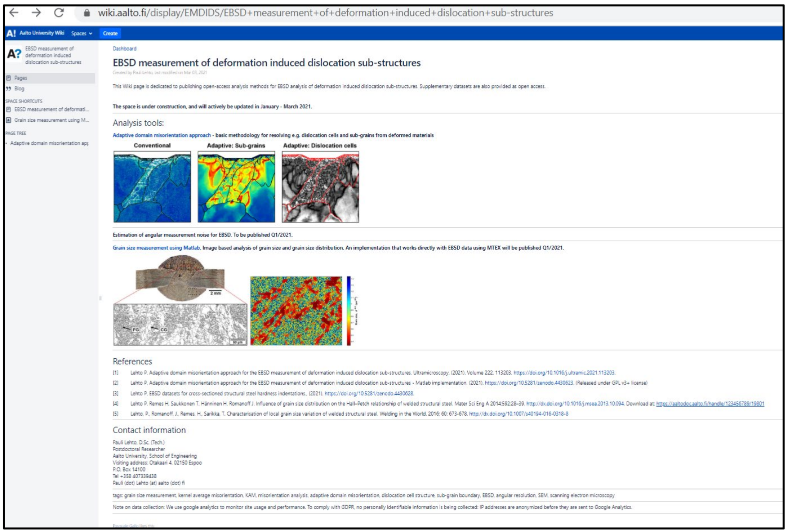
Open Access principle

Adaptive domain misorientation approach (DAM / DMM)

- Methods and example datasets for structural steel are available as open access



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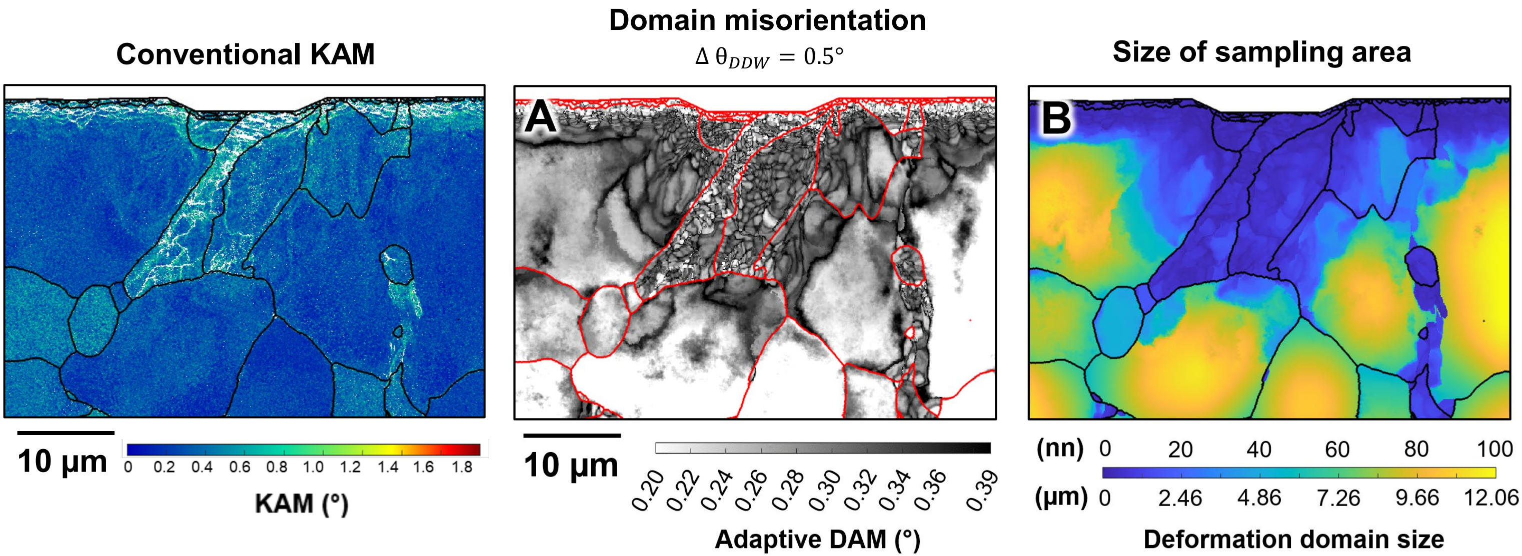
- Aalto University Wiki - EBSD measurement of deformation induced dislocation sub-structures, (2021). https://wiki.aalto.fi/display/EMDIDS
- Adaptive domain misorientation approach for the EBSD measurement of deformation induced dislocation sub-structures - Matlab implementation, (2021). https://doi.org/10.5281/zenodo.4430623
- EBSD datasets for cross-sectioned structural steel hardness indentations., (2021). https://doi.org/10.5281/zenodo.4430628

Analysis example - Dislocation cells 1

Cross-sectioned hardness indentation (Structural steel S355)

- Conventional KAM using raw measurement data not effective for mapping the extent of deformation or the dislocation cell structure
- Domain misorientation approach reveals the sub-structures effectively (Half-Quadratic filtering in MTEX)
- Size of the sampling area (deformation domain) reveals sub-structures and extent of deformation



Analysis example - Dislocation cells 2

Cross-sectioned hardness indentation (Structural steel S355)

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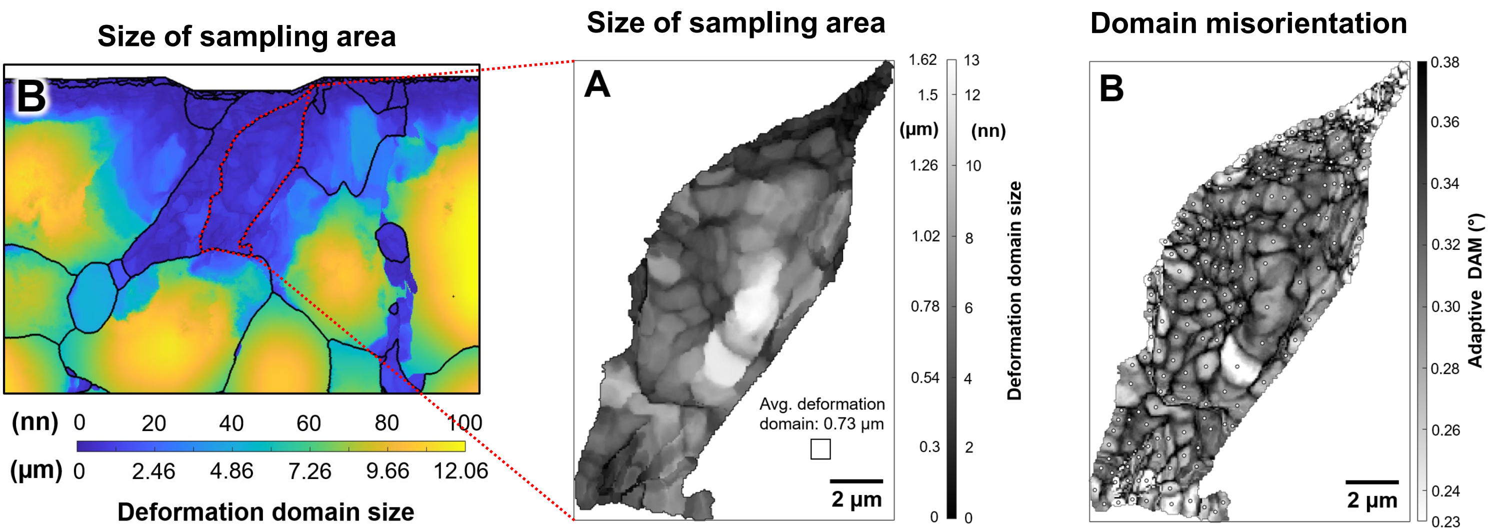


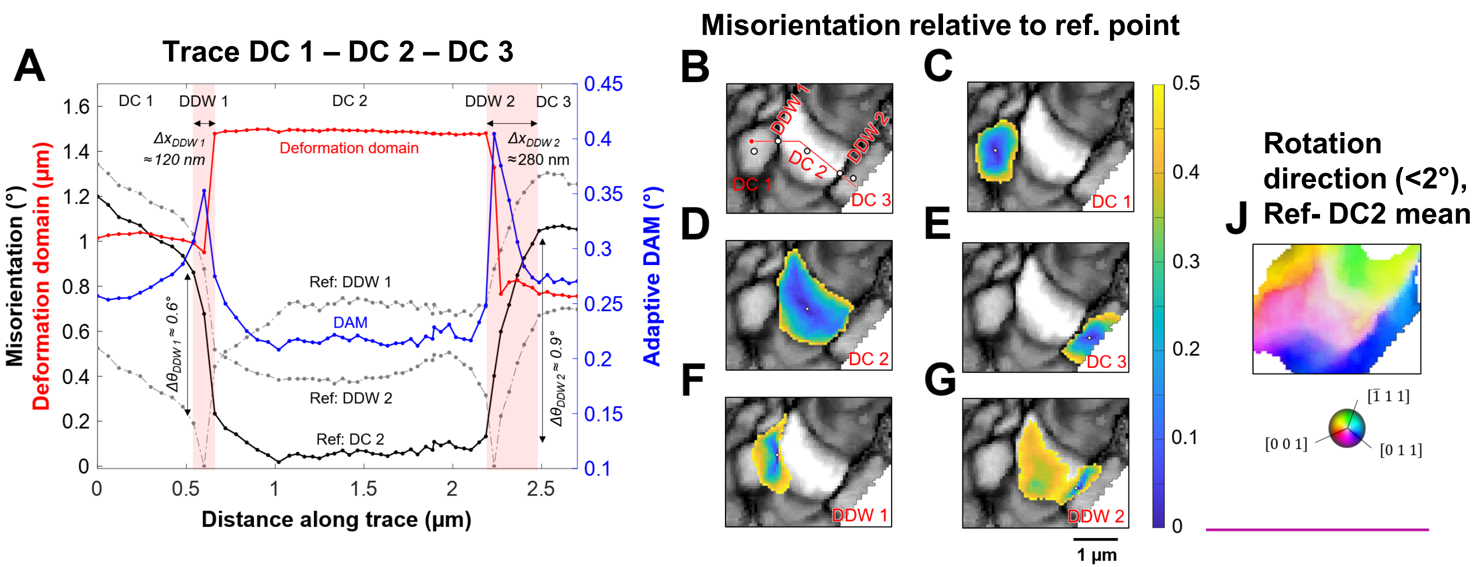
Analysis example - Dislocation cells 3

Does the sampling conform to dislocation cells?

- Good agreement with the boundary locations shown by DAM
- Deformation domain size has sharp transitions on the DDWs

Varying nature of the DDWs for the investigated grain

- Thickness of boundary region (Δx), defined as the area with high gradient compared to surroundings, usually varies between 100 – 300 nm
- DDW misorientation mostly varies between 0.3 – 1.0°

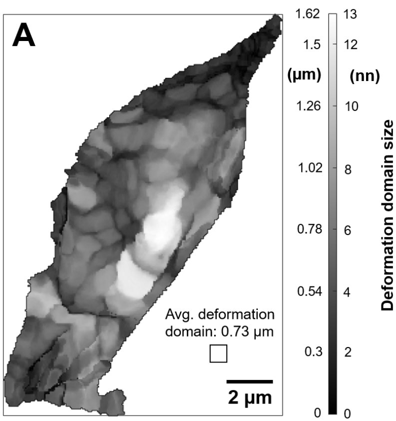


Analysis example - Dislocation cells 4

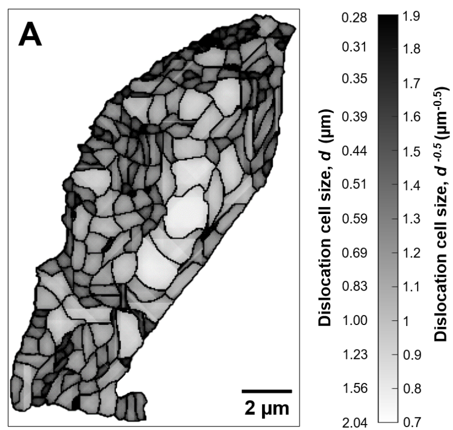
Size of the dislocation cells

- Indication that the size of the sampling area may correspond with the dislocation cell size
- DCs traced from DAM, and measured using the point-sampled intercept length [1, 2]
- Good agreement for average DC size and size dispersion

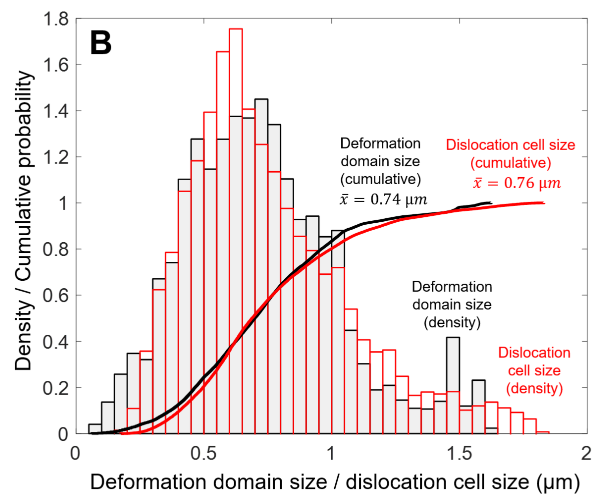
Size of sampling area



Point-sampled intercept length



Dislocation cell size

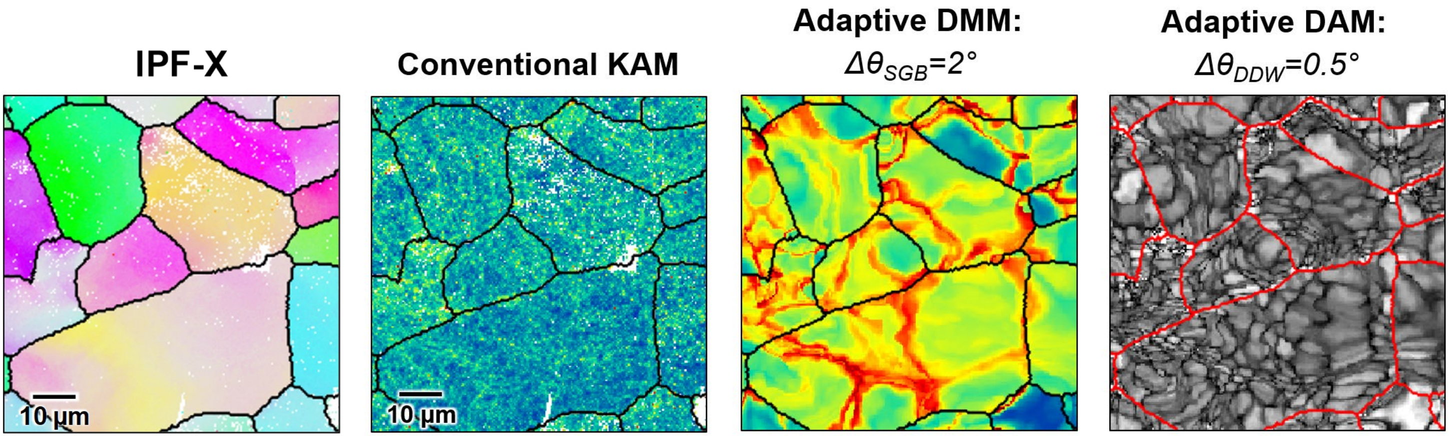


[1] **Lehto, P.; Romanoff, J.; Remes, H.; Sarikka, T.** *Characterisation of local grain size variation of welded structural steel.* Welding in the World. 2016; 60: 673-678. http://dx.doi.org/10.1007/s40194-016-0318-8
[2] **EBSD-based method will be made available Q1/2021 at**: https://wiki.aalto.fi/display/GSMUM

Literature dataset – IF Steel – 1

Uniaxially strained data for interstitial free steel [1]

- Conventional KAM (using raw data) shows some deformation localization, but sub-structural patterns are not resolved
- Using the domain misorientation, we can reasonably resolve sub-grains and DCs



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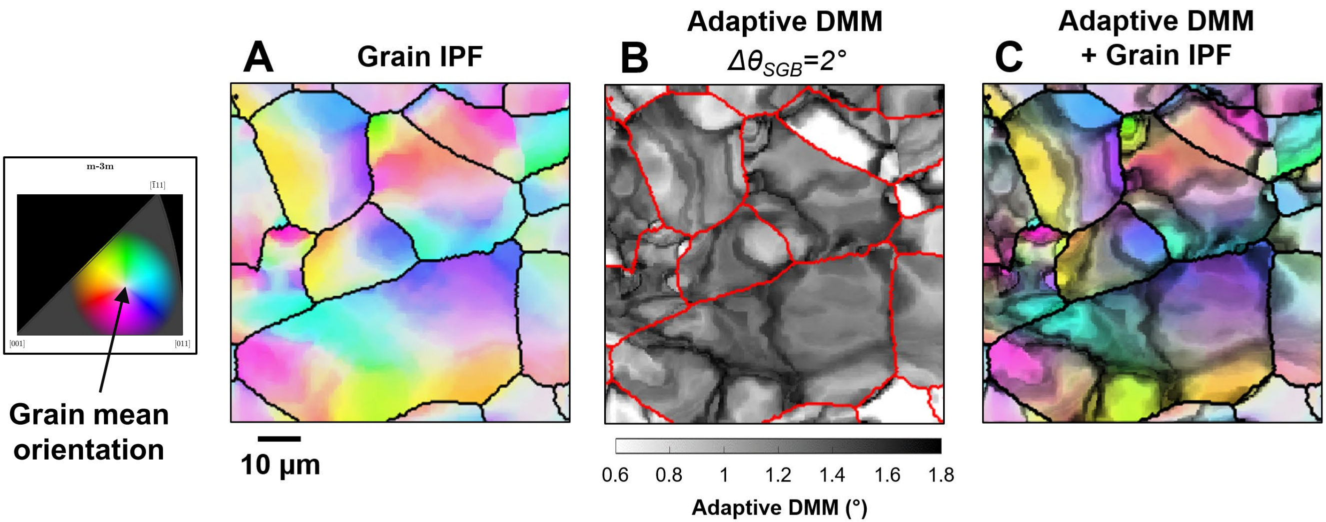
[1] B. Britton, J. Hickey, Example MTEX EBSD data from Iron, (2018). https://doi.org/10.5281/ZENODO.1434868

Literature dataset – IF Steel – 2

A closer look at the sub-grain boundaries

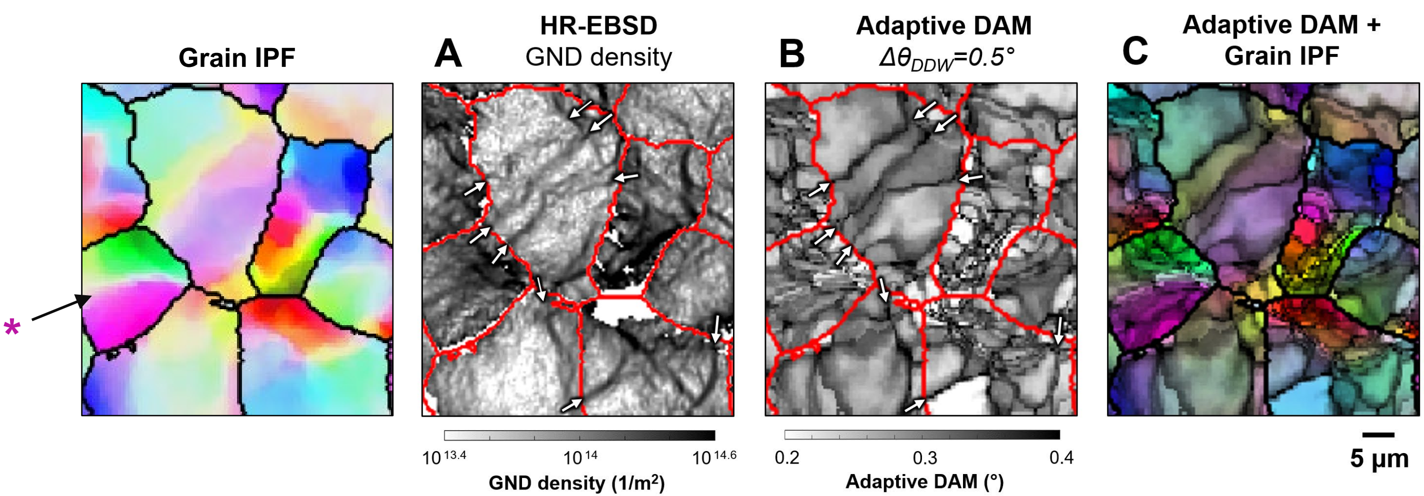
- The Grain IPF is used to visualize the sub-granular rotation patterns: Rotation direction relative to grain's mean orientation within 15°
- The regions outlined by DMM, although many are somewhat blurred, corresponds with rotation patterns



Literature dataset – IF Steel – 3

Dislocation cell structure vs HR-EBSD

- Several locations of the DDWs can be identified from the GND density (white arrows)
- Rotation directions are uniform within the DCs (e.g. central grain)
- Grains with high dislocation density have sub-grains (e.g. *), the global misorientation gradient hides the rotation directions of the individual dislocation cells



Conclusions

A novel domain misorientation approach was developed for the measurement of dislocation sub-structures

Approach found suitable for steel and various other metals that have different processing histories

- Any material with a change in lattice curvature at the sub-structural boundaries should be possible to investigate

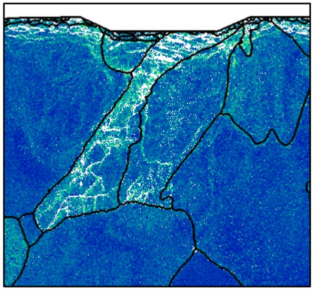
Characterisation of dislocation sub-structures

- Misorientation and thickness of the boundary region can be measured
- Simulateneous dislocation cell size measurement
- Potential for characterization of plastic deformation

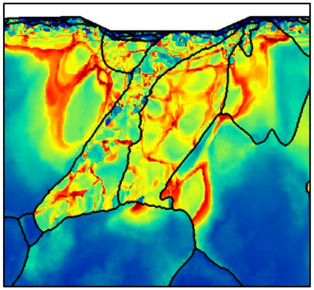
All analysis methods available as open access

Future work: comparison with direct observation (TEM)

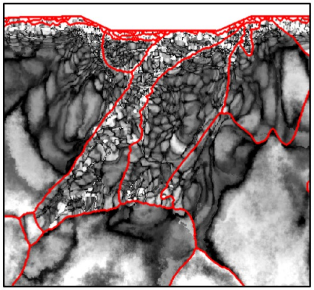
Conventional



Adaptive: Sub-grains



Adaptive: Dislocation cells



References

Reference list

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