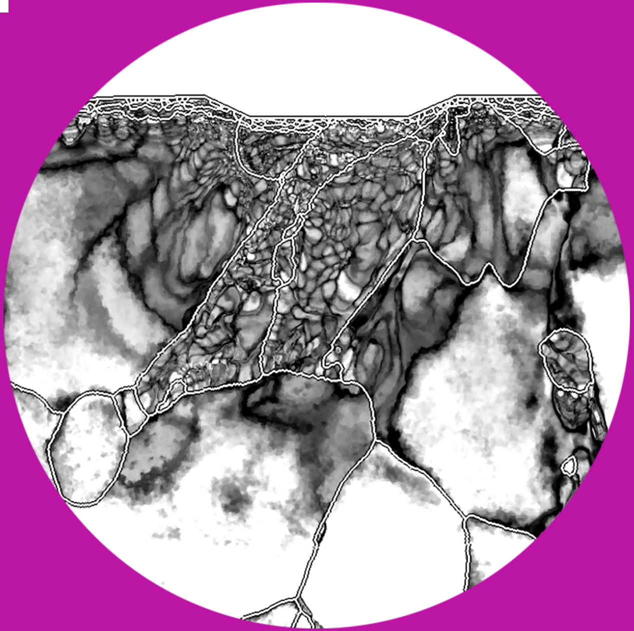
Characterisation of local plastic deformation

MTEX 2020 workshop



Aalto University
School of Engineering
Marine Technology

D.Sc. Pauli Lehto

10.3.2020

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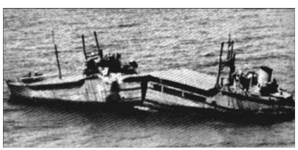

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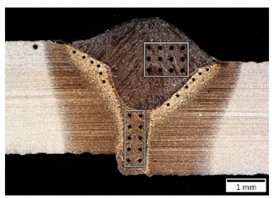
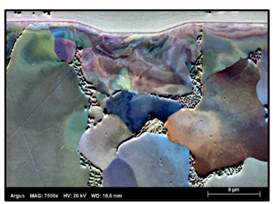
Scope of work





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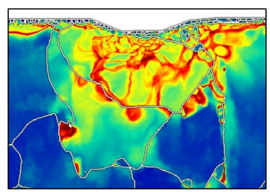
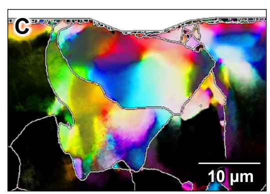
Misorientation analysis in MTEX





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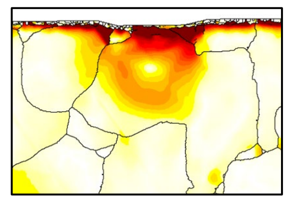
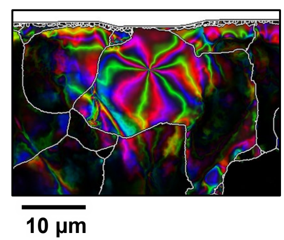
Analysis examples





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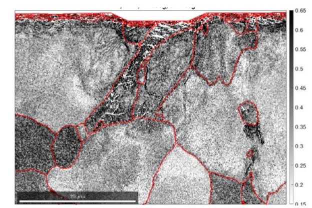
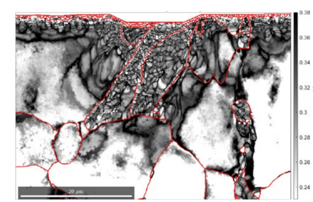
Conclusions





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Introduction

Aalto University

Unique merger of three leading Finnish universities

to cover cross-discipline teaching and research

The Aalto University community:
80 000 alumni,
20 000 students,
5 000 staff members,
370 professors

1849 Helsinki University of Technology

1871 University of Industrial Art Helsinki

1911 Helsinki School of Economics

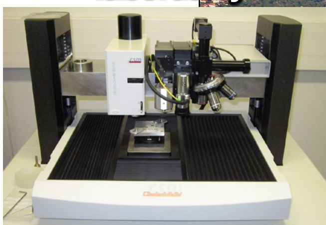
2010 Aalto University

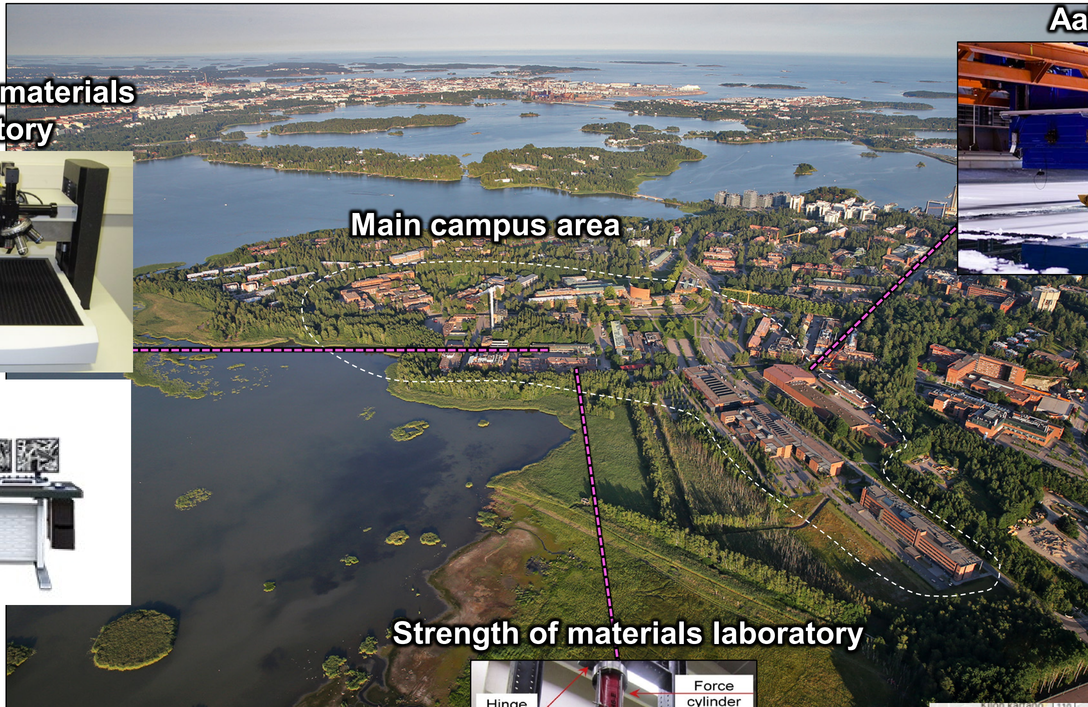
where science and art meet technology and business

Aalto University
School of Engineering

Aalto University – Otaniemi Campus

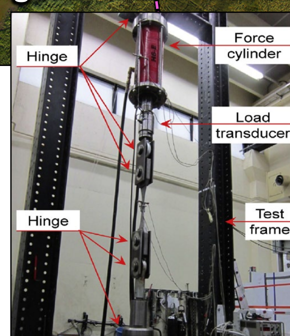
Engineering materials laboratory

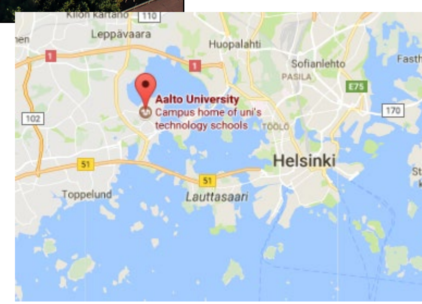




Aalto Ice Tank

Strength of materials laboratory





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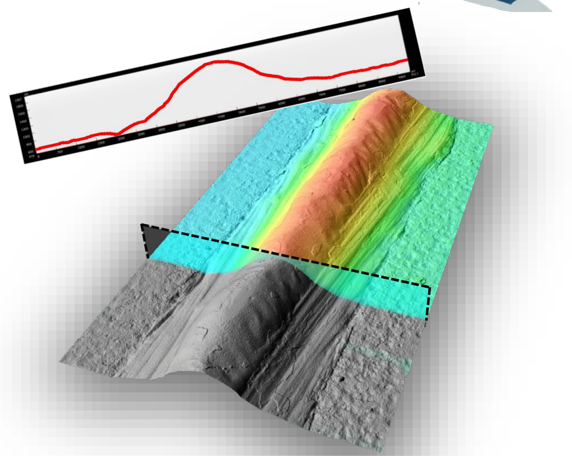
Background

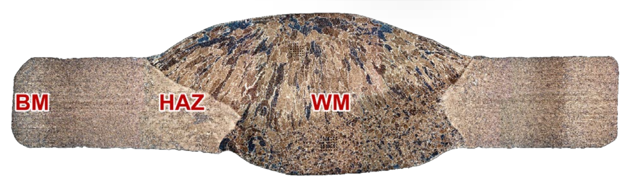
Sustainable growth requires the effective use of high strength steels

- Steel's mechanical properties are affected by the manufacturing process
- Statistical analysis is required across several length scales for engineering purposes

Research in the microstructural length scale reveals deformation mechanisms

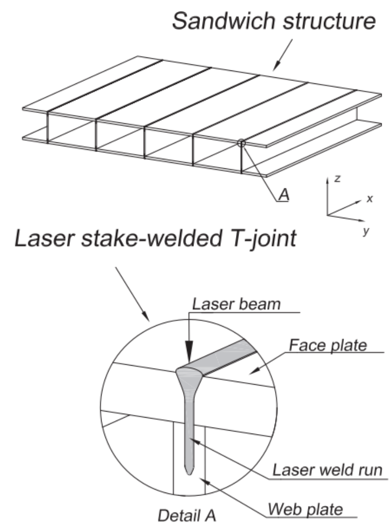
- Optimisation of materials and manufacturing processes made possible by understanding the fundamental microstructure – strength relationships

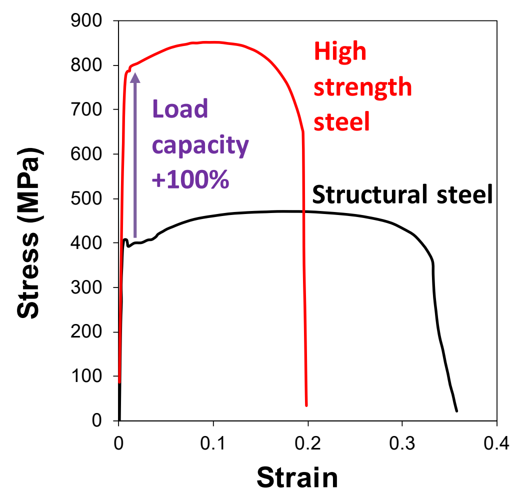




Steel in shipbuilding

- **Energy efficiency of ships calls for alternative structural topologies and stronger steel grades (relative density)**
- New manufacturing methods need to be implemented to fully utilize the higher strength and to guarantee the safety of the structure (e.g. fatigue)
- Welds are often structural weak points prone to failure (microstructural effects)





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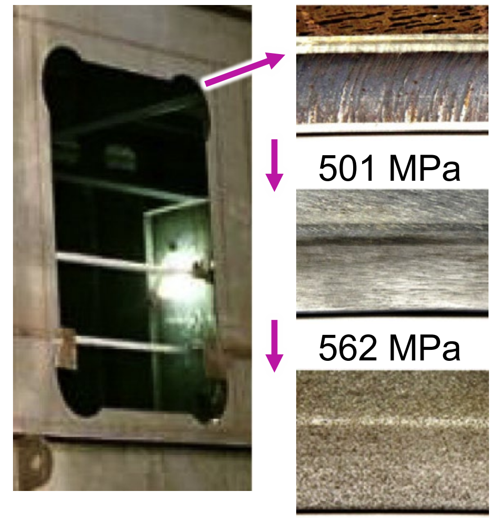

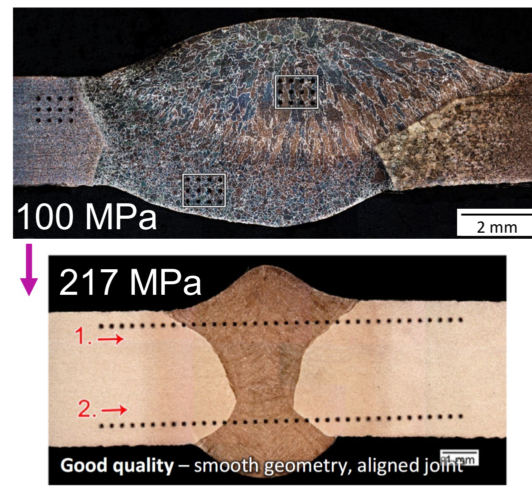

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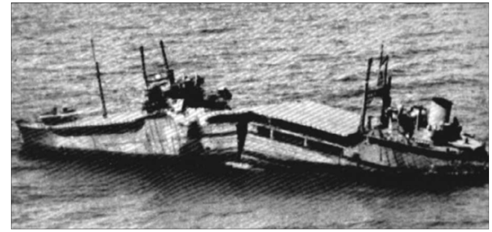
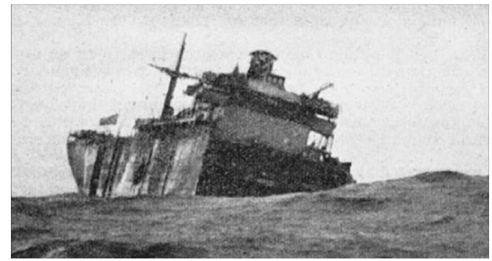
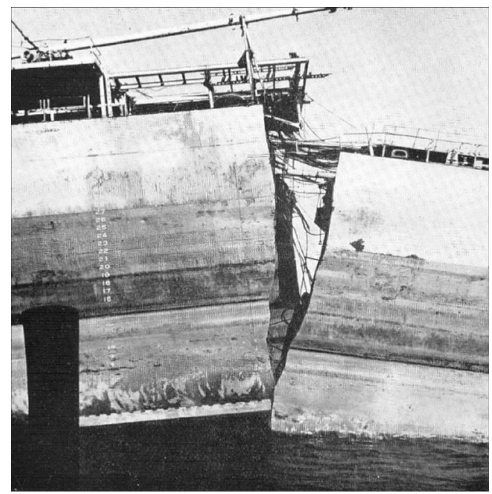






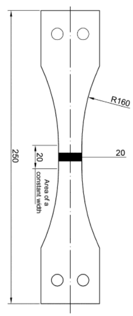
- Energy efficiency of ships calls for alternative structural topologies and stronger steel grades (relative density)
- New manufacturing methods need to be implemented to fully utilize the higher strength and guarantee the safety of the structure (e.g. fatigue)
- **Welds are often structural weak points prone to failure (microstructural effects)**

Length scales of research

- To prevent catastrophic failures, we need to understand the load carrying mechanisms in different length scales
- Fundamental question: how does the manufacturing process affect the strength properties of steel?
- How to approach the problem, top-down or bottom-up?

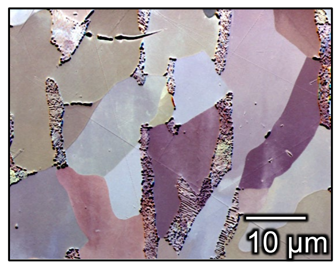
300m +

Ship structure



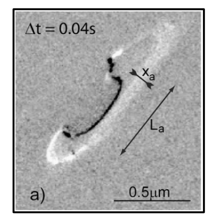
1 m … 1 cm

Weld detail



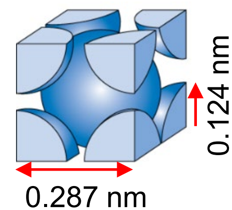
1000…1 µm

Microstructure



1 µm...100 nm

Dislocations



1 Å = 0.1 nm

Atoms

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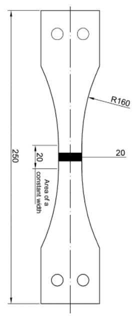

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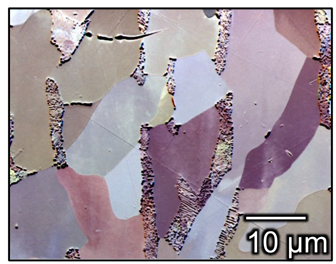
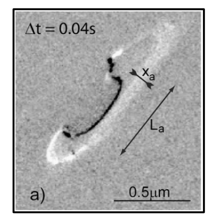


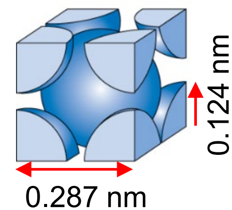
1000…1 µm

Microstructure



1 µm...100 nm

Dislocations



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Atoms

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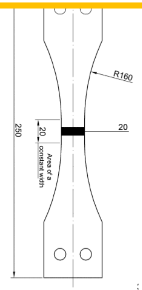
Statistical variation

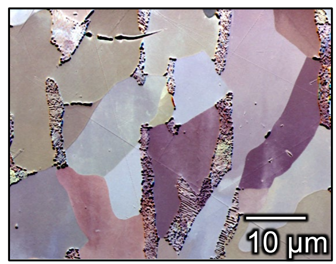
300m +

Ship structure



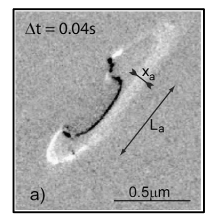
1 m … 1 cm

Weld detail



1000…1 µm

Microstructure



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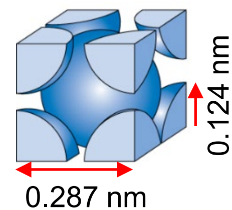

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Atoms

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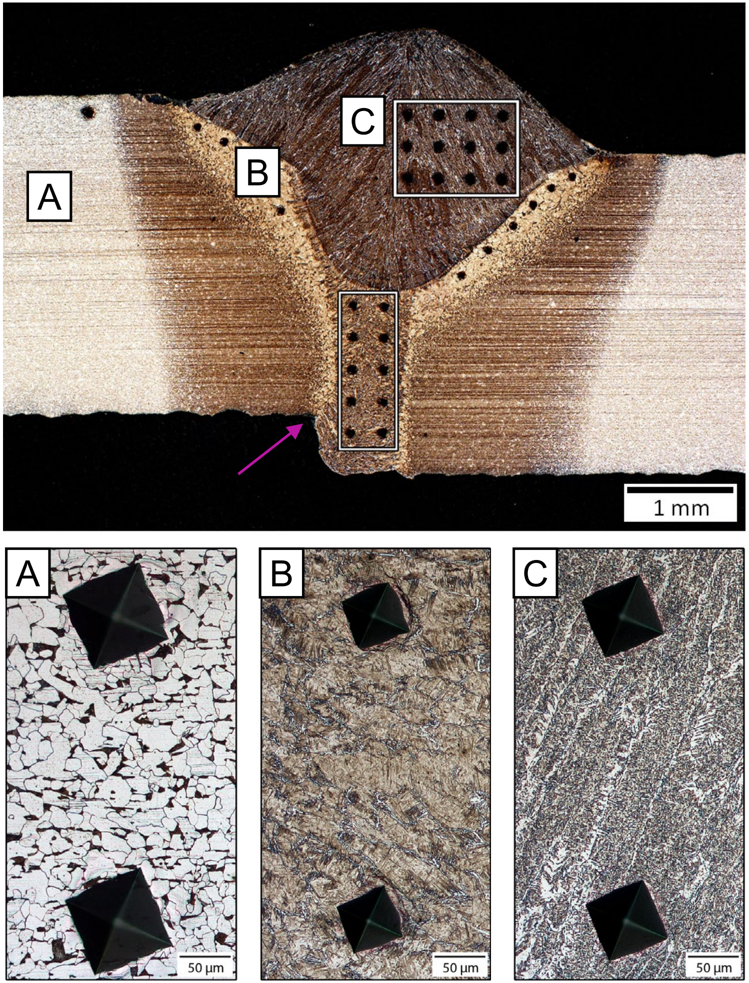
Scope of work

Scope of work

How to predict average strength properties of welded steel from statistical analysis of the microstructure?

Research questions:

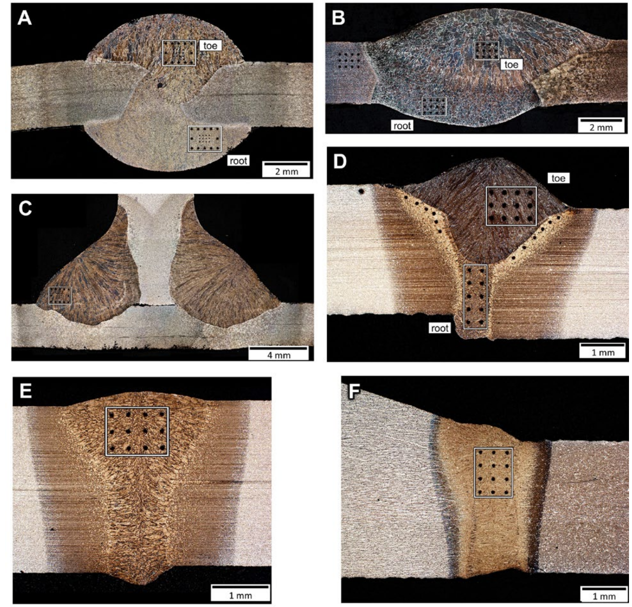
- **1) How to characterize the microstructure**
- **2) How to measure local strength variation**
- **3) Define dependencies between microstructure and strength**

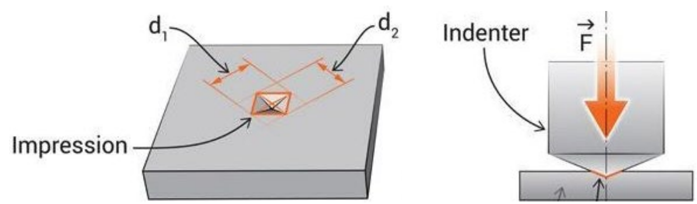


Experiments

Microstructure and strength characterised for several weld metals:

- **Instrumented indentation testing to measure local strength properties**
- Scanning electron microscopy used for the characterization of microstructure and plastic deformation





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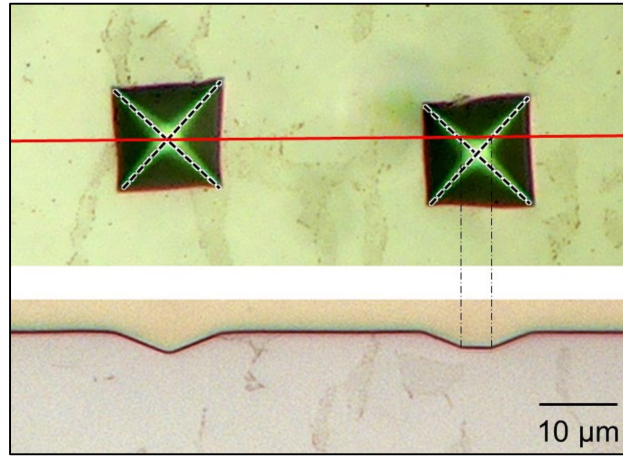
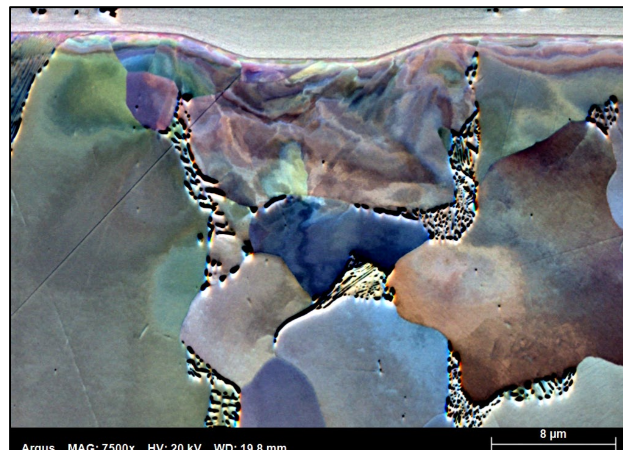


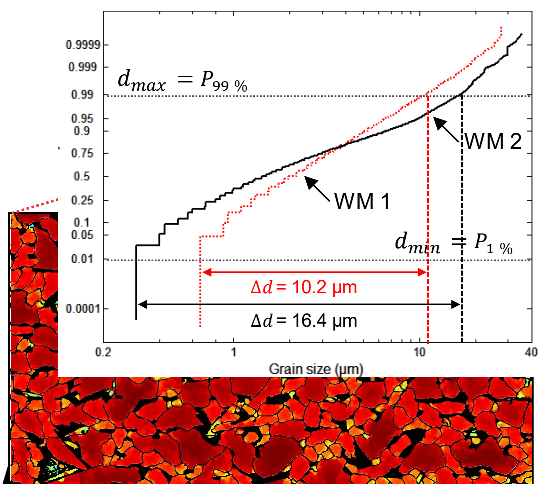


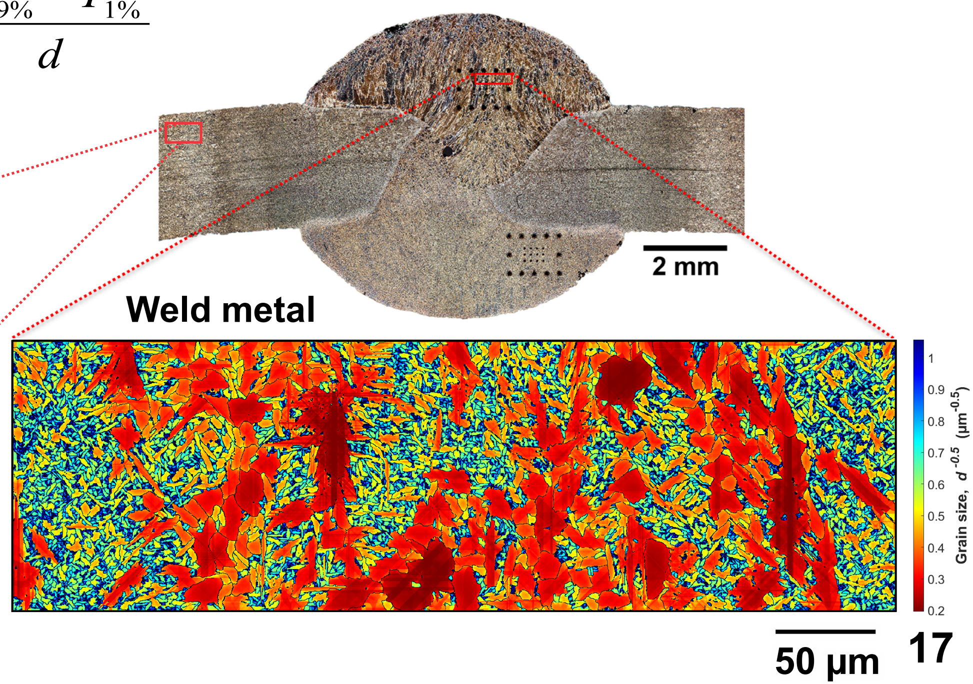
Grain size of steel

- Solidification of a weld creates a broad dispersion of different grain sizes in comparison to base metal
- Methods developed for the characterisation of the heterogeneous grain structure

$$\frac{\Delta d}{d} = \frac{d_{\max} - d_{\min}}{d} = \frac{P_{99\%} - P_{1\%}}{d}$$

Log-normal probability plot





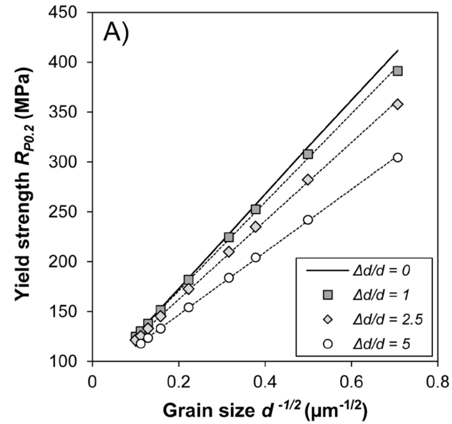
Prediction of strength

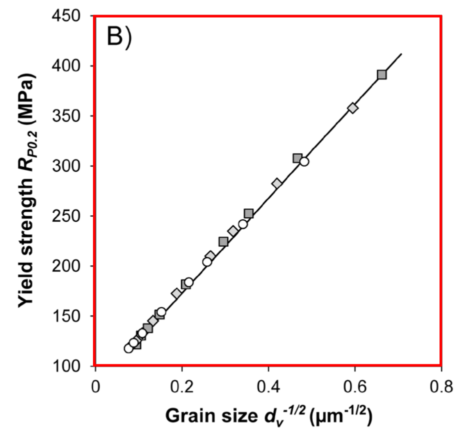
- Average strength properties can be predicted using average grain size
- However, the grain size dispersion must be considered
- **Based on the observations of this dissertation, the Hall-Petch equation was modified:**

$$\sigma = \sigma_0 + kd^{-1/2}\left(1 + f\,\frac{\Delta d}{d}\right)$$

Relative grain size dispersion

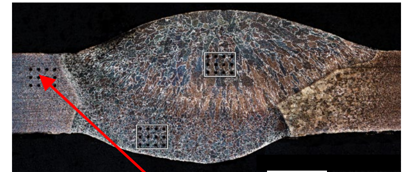
Literature: simulated base metal

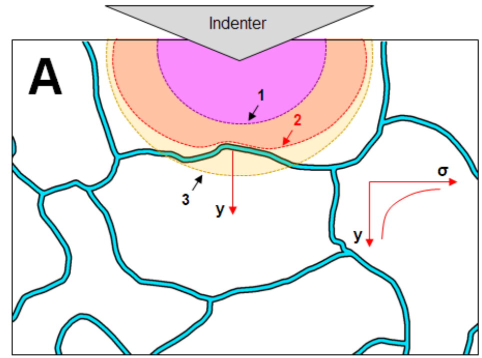


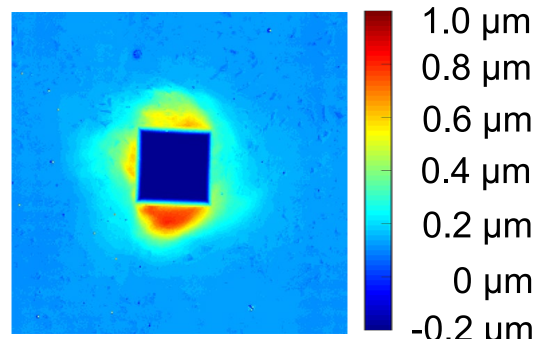


Local plastic deformation

- **In a hardness test the material must permanently change it’s shape to accommodate the indenter**
- The mechanism for the shape change is material rotation
- At microstructural level the grains in the material are subdivided into smaller units
- Dislocations are moving on specific slip systems







Indentation depth 4.9 μm

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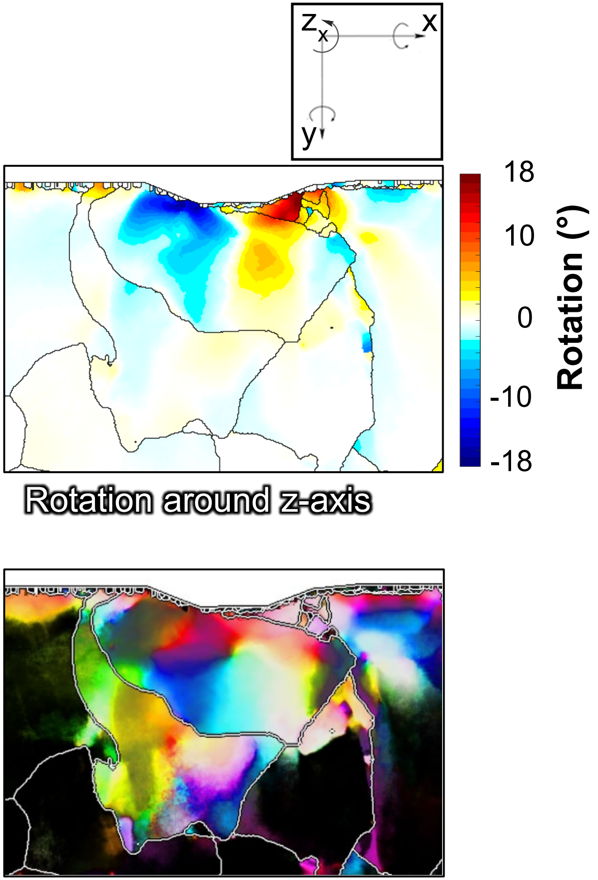

Rotation around z-axis

Rotation axis

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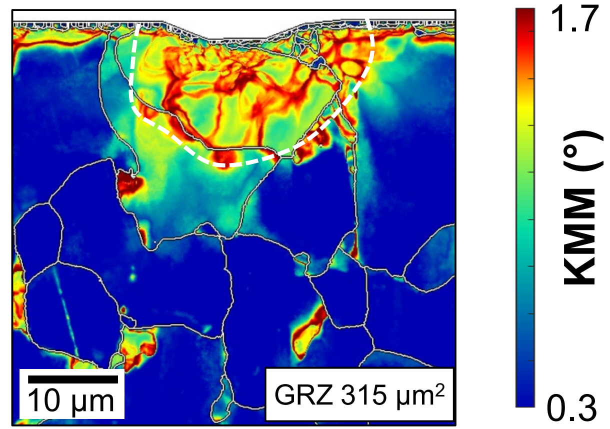

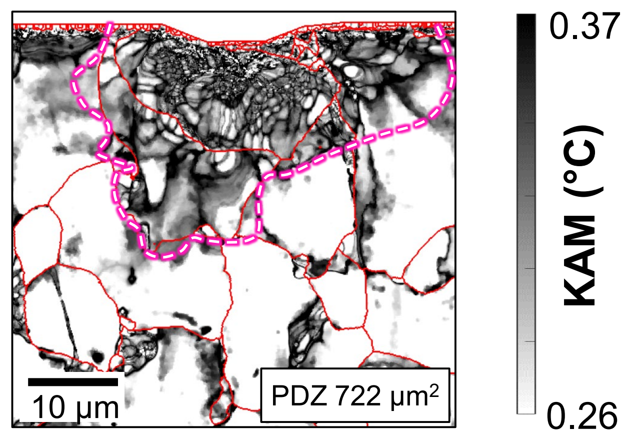

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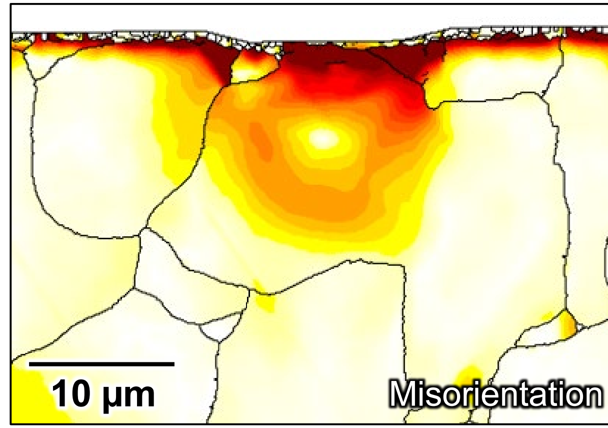

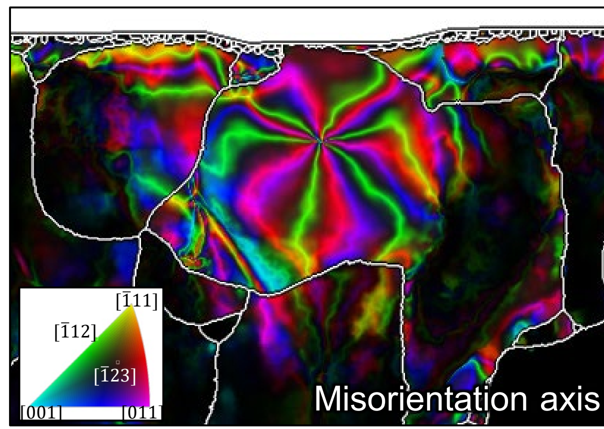

10 µm

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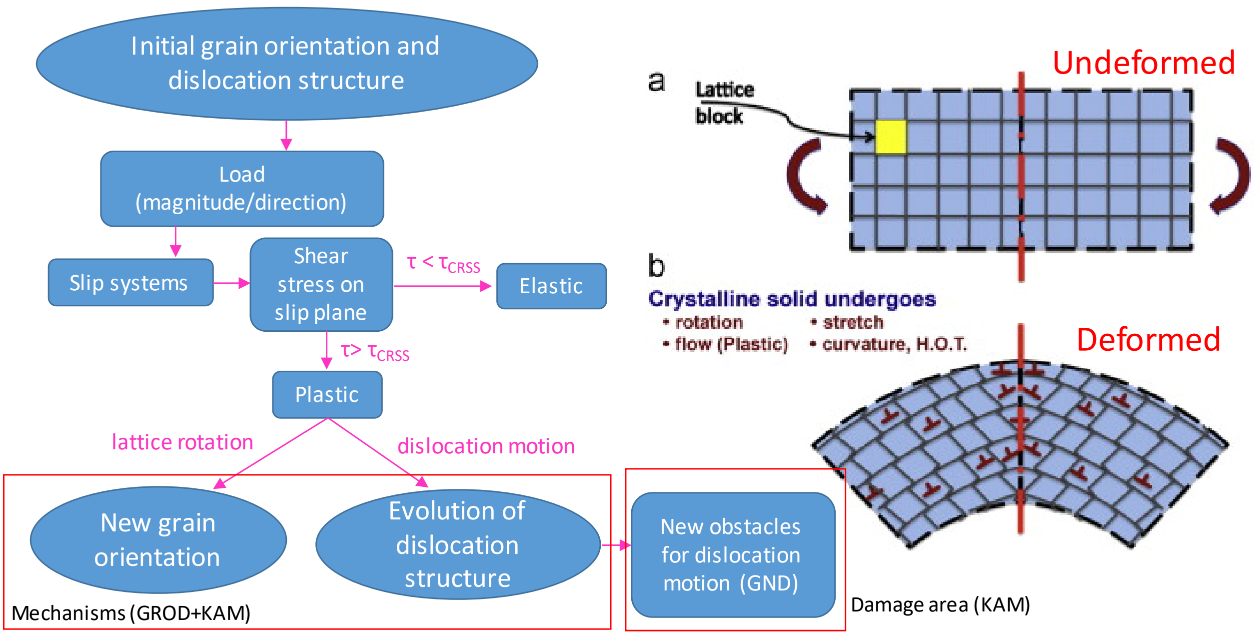
Local misorientation analysis in MTEX

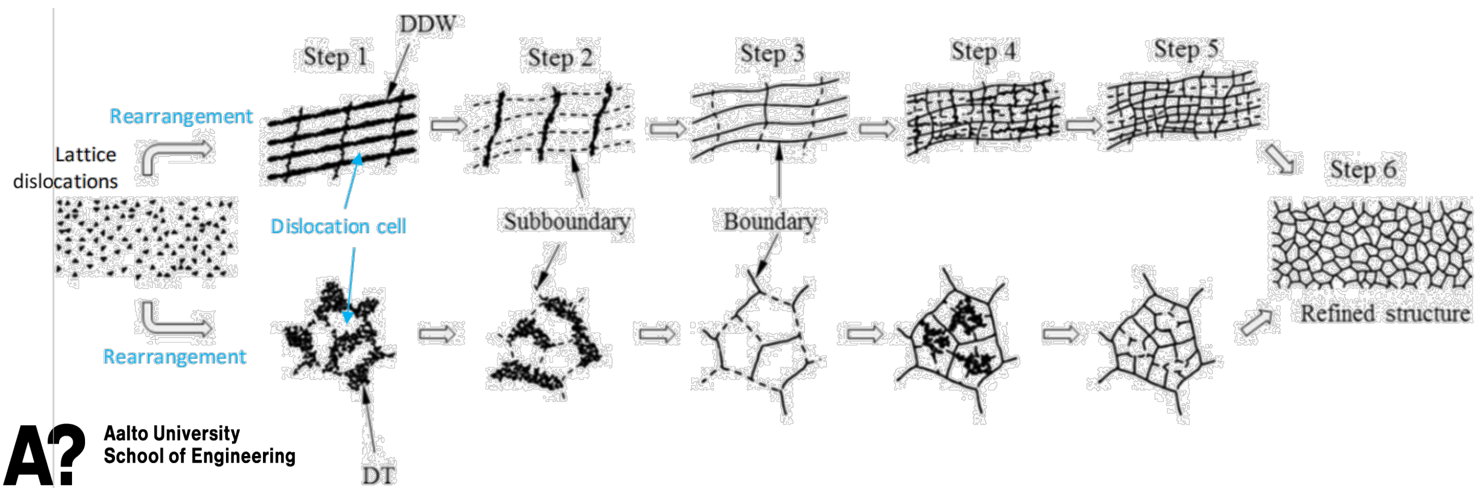
Deformation mechanism of steel

- Two aspects need to be analyzed for local plastic deformation:
  - 1) Changes in grain orientation
  - 2) Evolution of dislocation structure



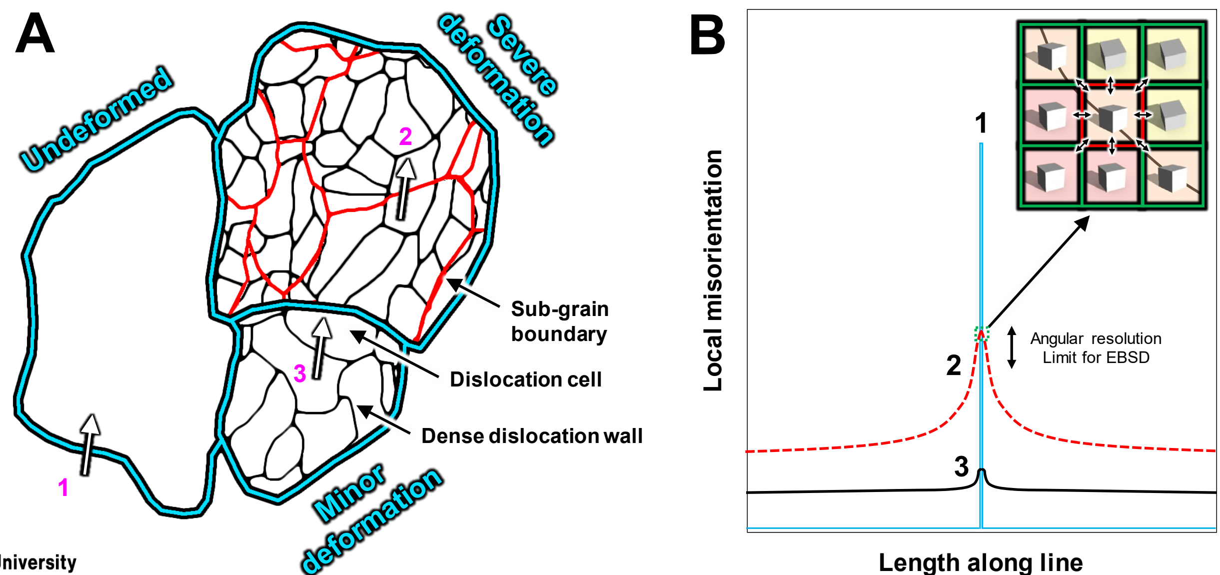
Deformation mechanism of steel

- Grain subdivision process, where lattice dislocations rearrange to form dislocation tangles (DT) and dense dislocation walls (DDW).
- As deformation continues, dislocation density increases, and new sub-grain and grain boundaries are formed.
- With sufficient plastic deformation the process is repeated in the newly formed grains, further refining the grain size.



Deformation mechanism of steel

- Sub-grain boundaries are harder to detect than grain boundaries from EBSD data for two reasons:
  - 1. Boundaries are gradual, not sharp
  - 2. Misorientation is small in relation to angular resolution



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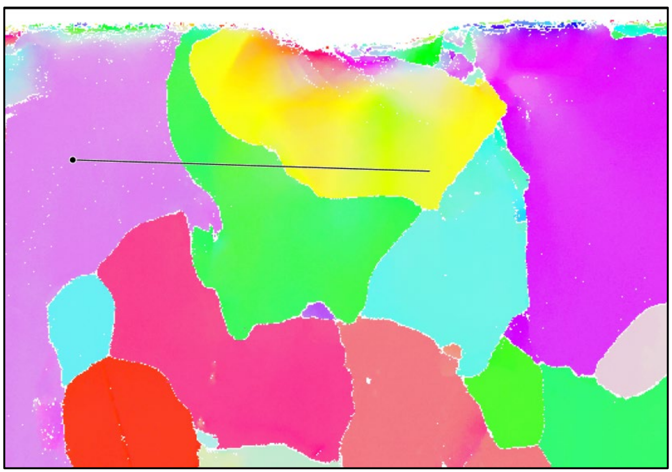
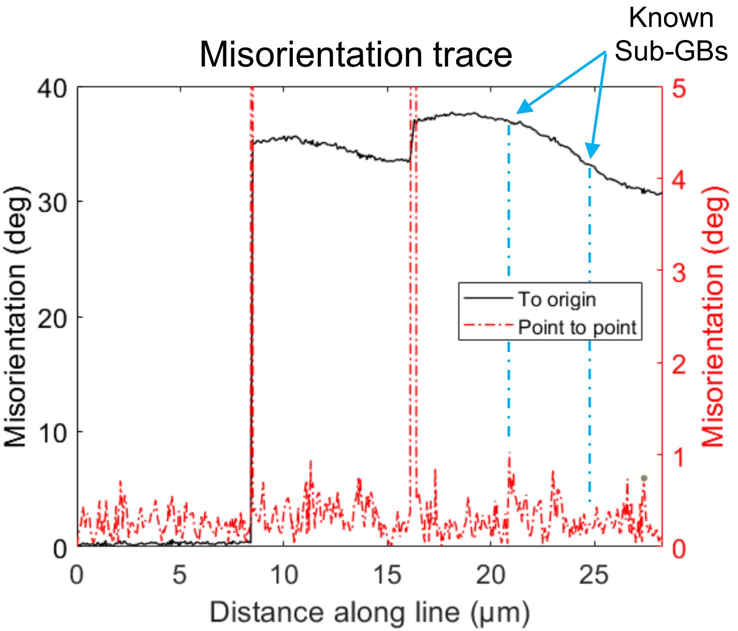
IPF-Z



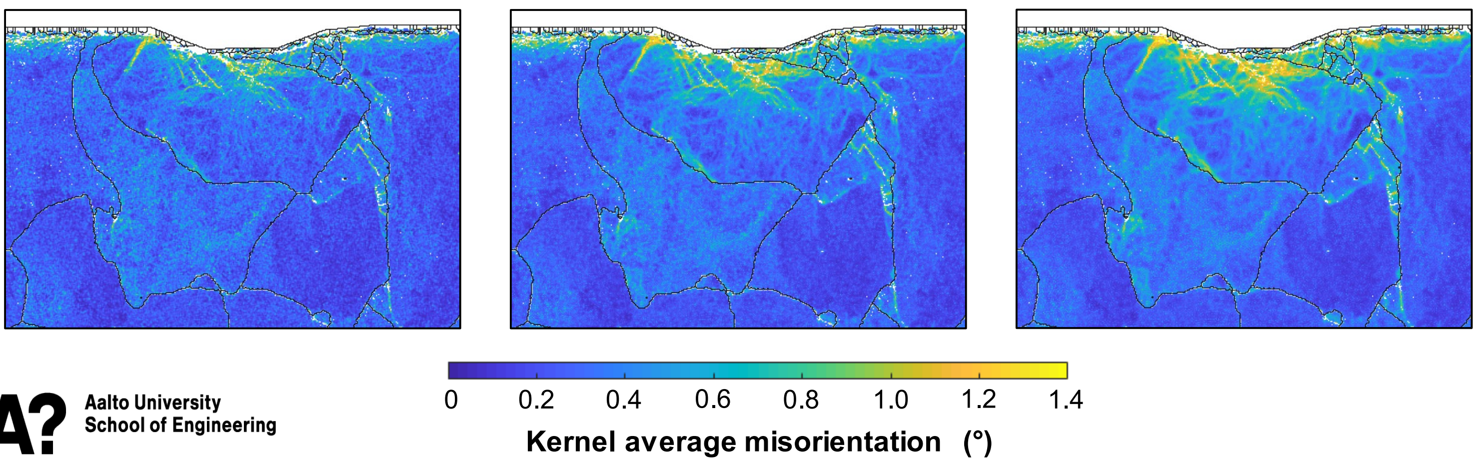
Misorientation trace



Kernel misorientation

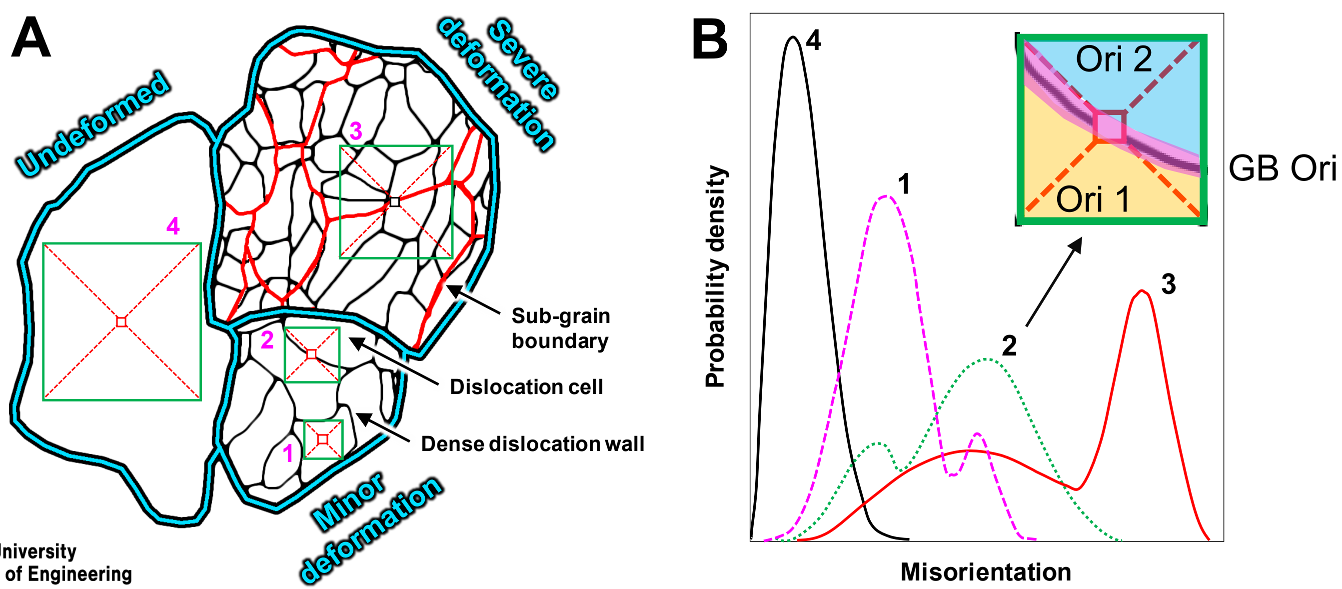
- Kernel misorientation measures the average misorientation between a central point and its nearest neighbours
  - Small kernels affected by the global orientation gradient
  - Some boundaries visible, but the measurement noise inhibits reliable detection of sub-granular features
  - **=> A new measurement strategy is needed**

3x3 kernel (nn=1) **5x5 kernel (nn=2)** **7x7 kernel (nn=3)**



Adaptive kernel size analysis

- Instead of sampling the immediate vicinity of the gradual border, the misorientation between neighbouring sub-grains should be measured
    - Kernel size should be in proportion to the measured sub-granular units
    - Different sub-GB types have characteristic misorientation ranges
    - Large kernels (20..100 nn) effective in averaging EBSD measurement noise



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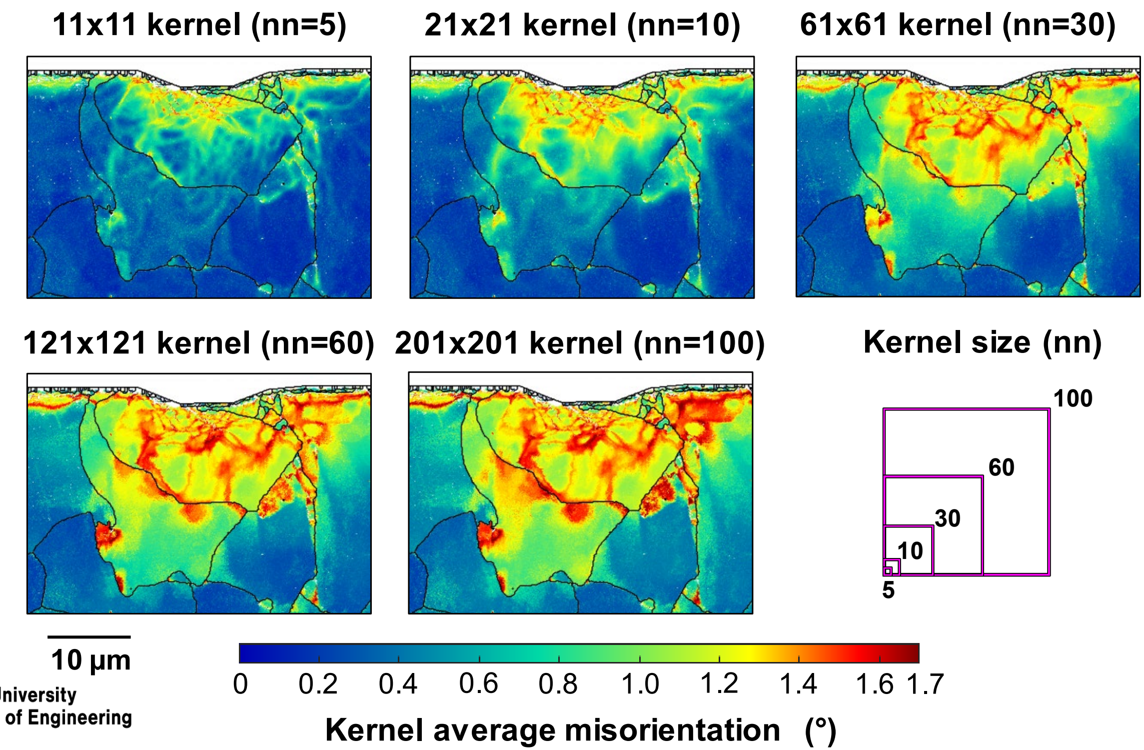
Adaptive kernel size analysis

- Kernel size up to 100 nearest neighbours (0.1 µm step size)
- Misorientation analysis range 0 – 2° reveals sub-grain boundaries
- Large kernels minimise the effect of global orientation gradient



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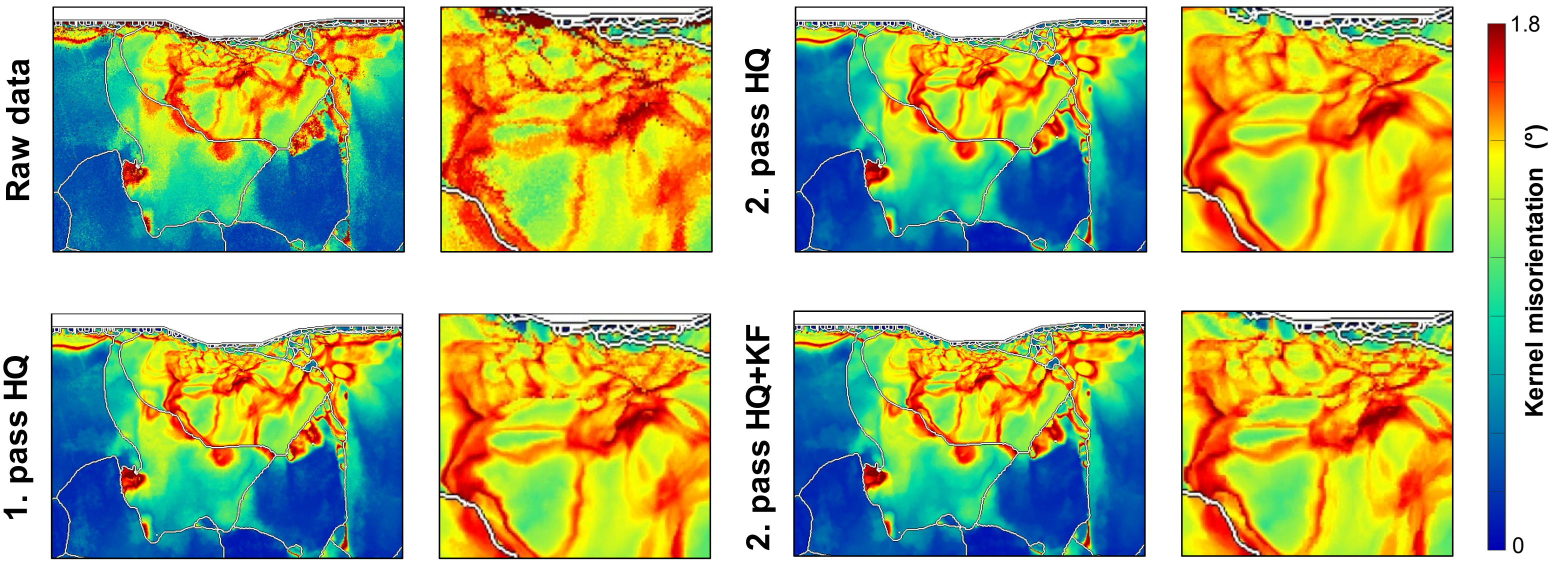
Post-processing of EBSD data

- Noise of EBSD data can be effectively reduced with MTEX to enhance visibility of sub-granular features
- Combination of Half-Quadratic filter (α=0.15) and Kuwahara Filter (1st NN) suitable for reducing measurement noise while retaining sub-granular detail (MTEX 5.0.1)



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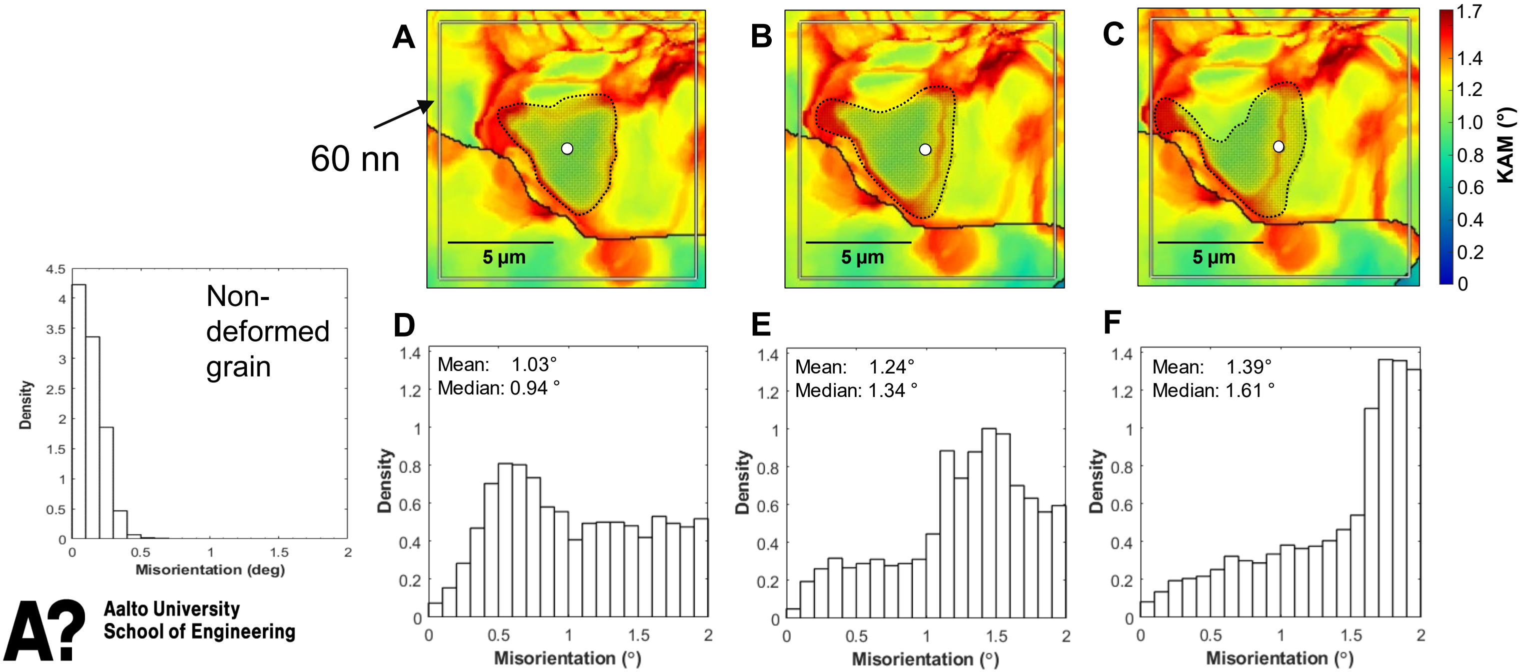
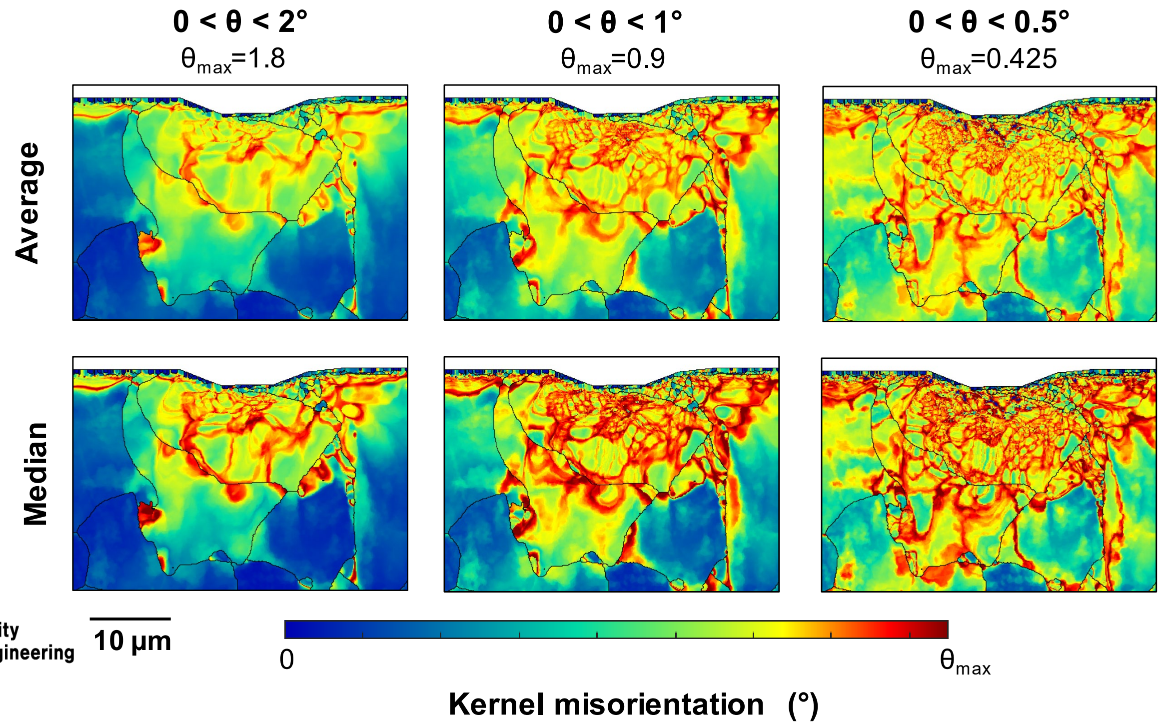
Effective kernel size

- The effective kernel size is dependent on size of features and location of measurement point
- In the example below, 60 nn kernel is moved inside a sub-grain



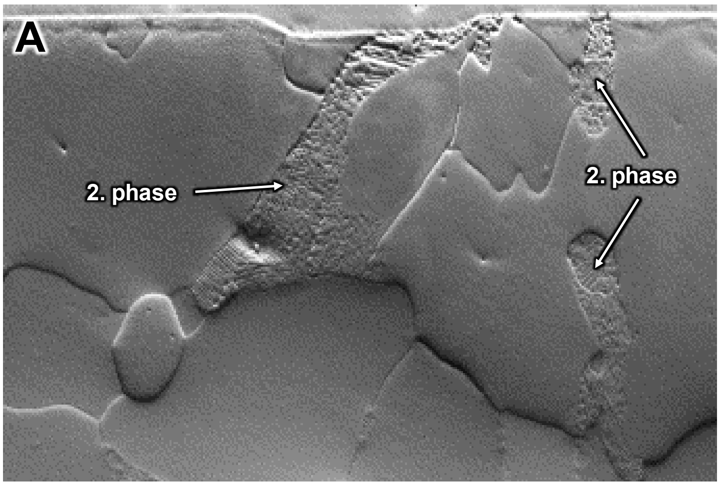
Sub-grains and dense dislocation walls

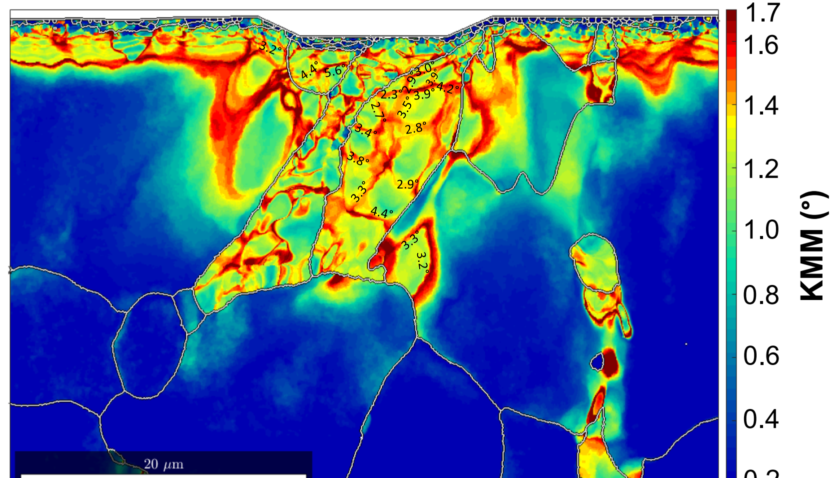
- By analyzing smaller misorientation ranges, the dense dislocation walls become visible
- Median value enhances contrast between non-deformed and deformed areas for 0-2° range



Sub-grains and dense dislocation walls

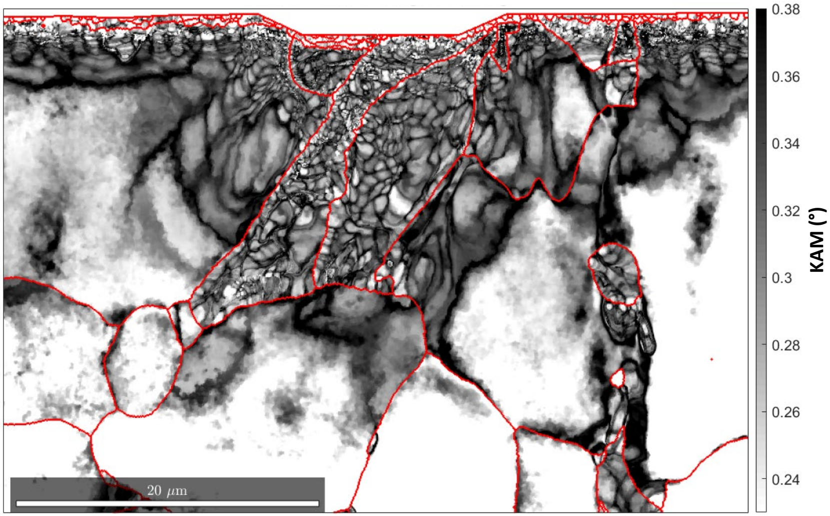
- Misorientation between neighbouring sub-grains 2.3 – 5.6°, average 3.5°

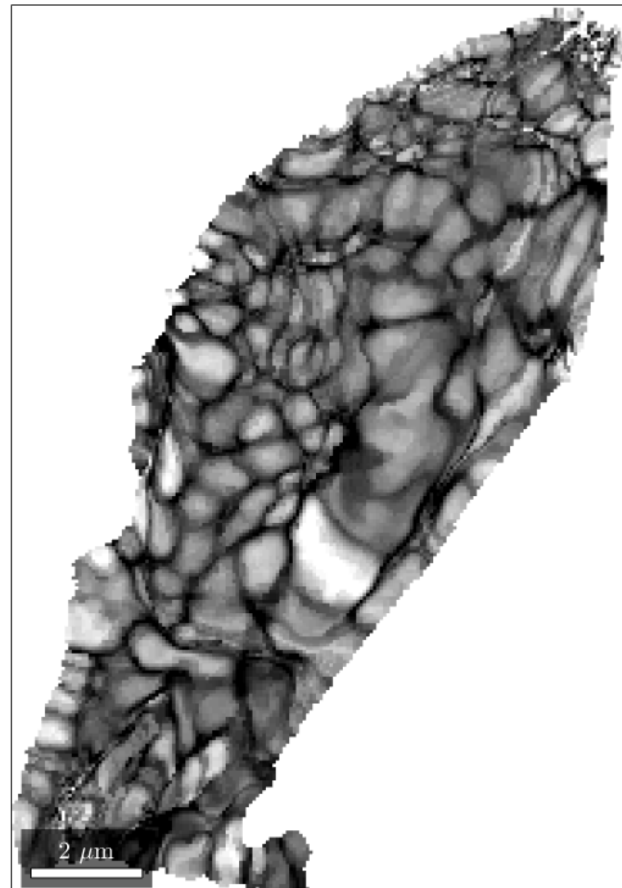




Sub-grains and dense dislocation walls

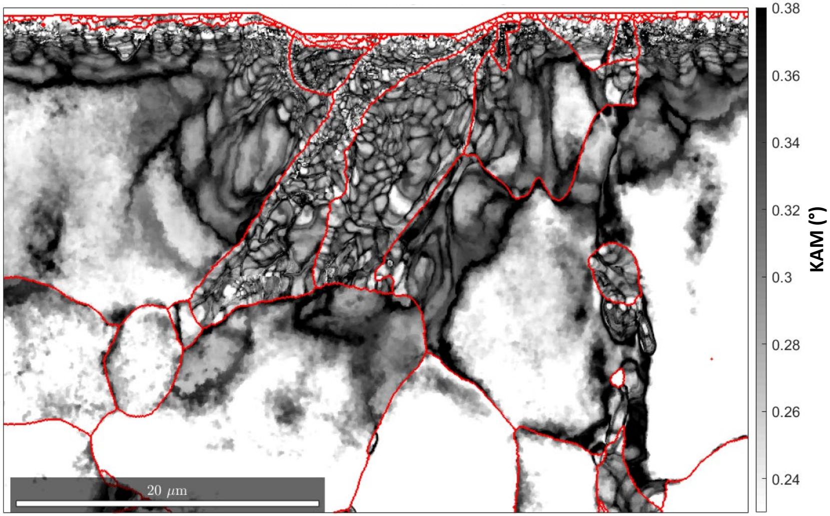
- Misorientation between neighbouring dislocation cells 0.5 – 2.1°, average 1.0°

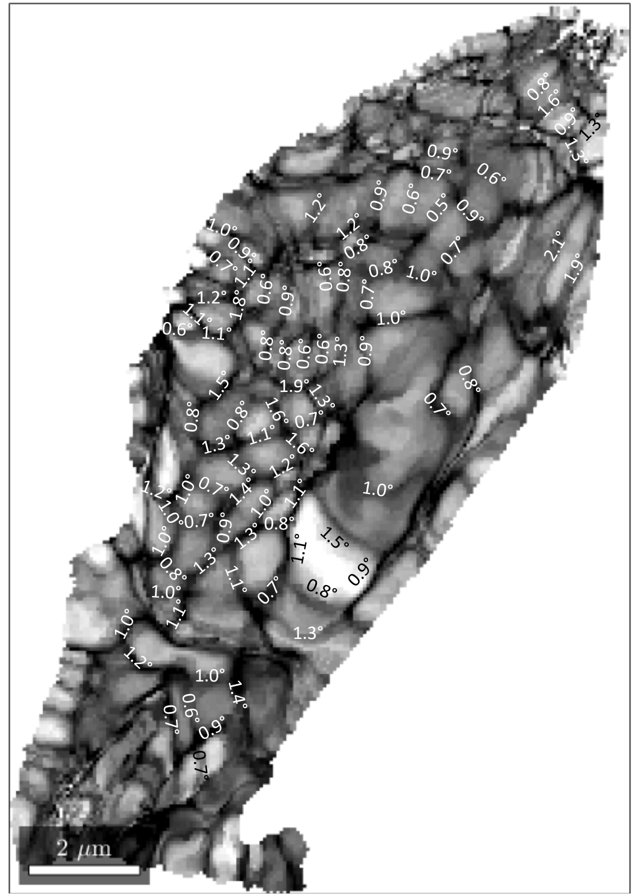




Sub-grains and dense dislocation walls

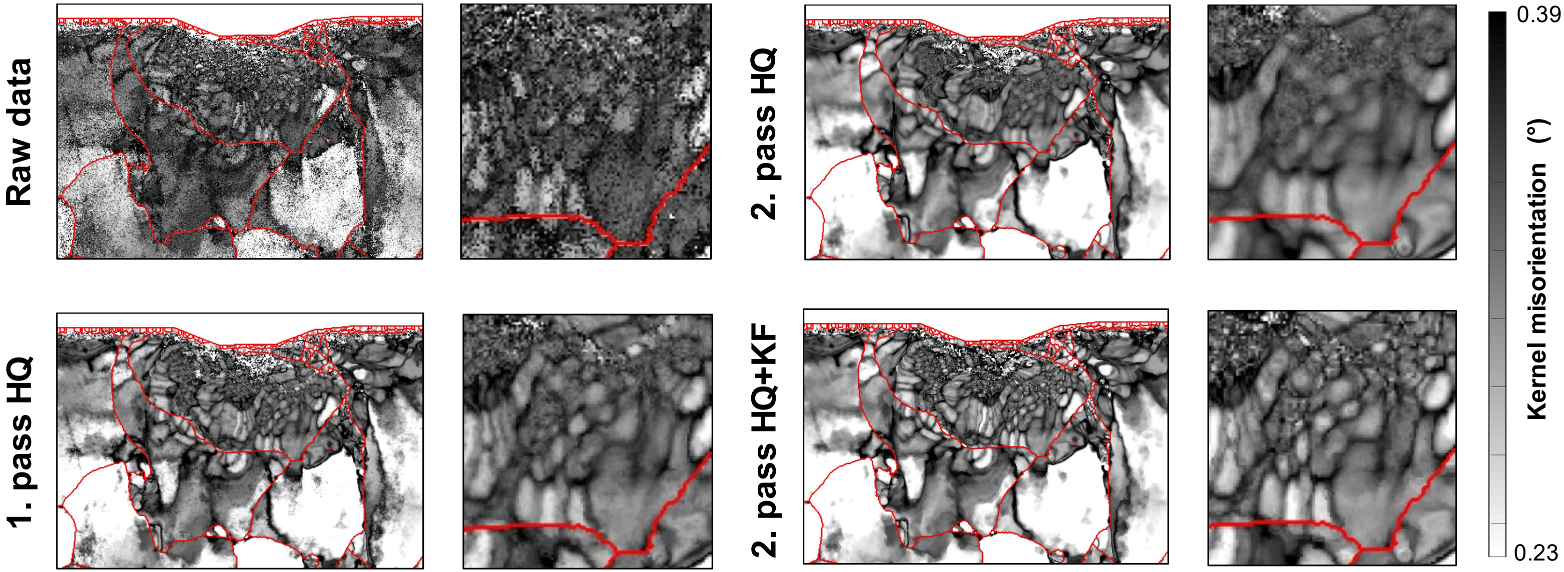
- Misorientation between neighbouring dislocation cells 0.5 – 2.1°, average 1.0°





Post-processing of EBSD data

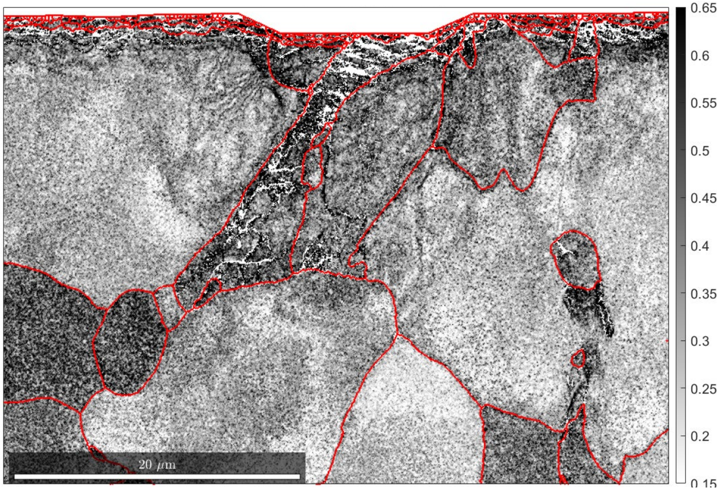
- Combination of Half-Quadratic filter (α=0.15) and Kuwahara Filter (1st NN) suitable for reducing measurement noise while retaining dislocation cell detail (MTEX 5.0.1)



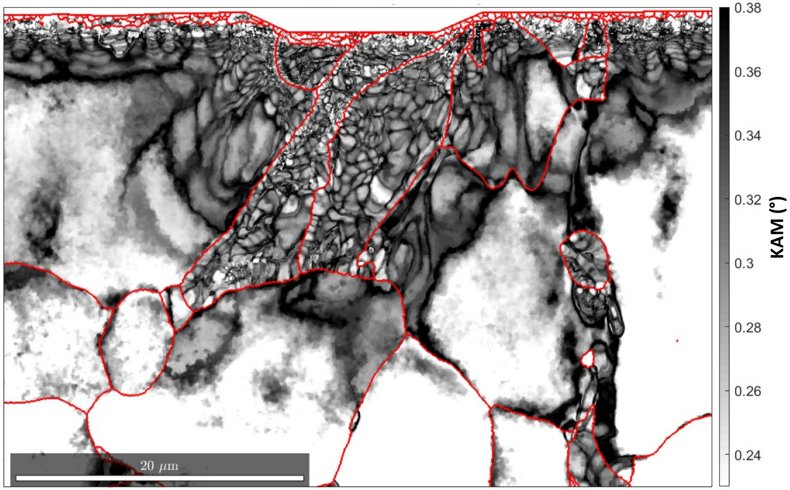
Post-processing of EBSD data

- Comparison of detail resolved with 0.06 µm step size:
  - Raw data and conventional KAM
  - Post-processed data and adaptive kernel size approach

Conventional KAM 1 nn & Raw data

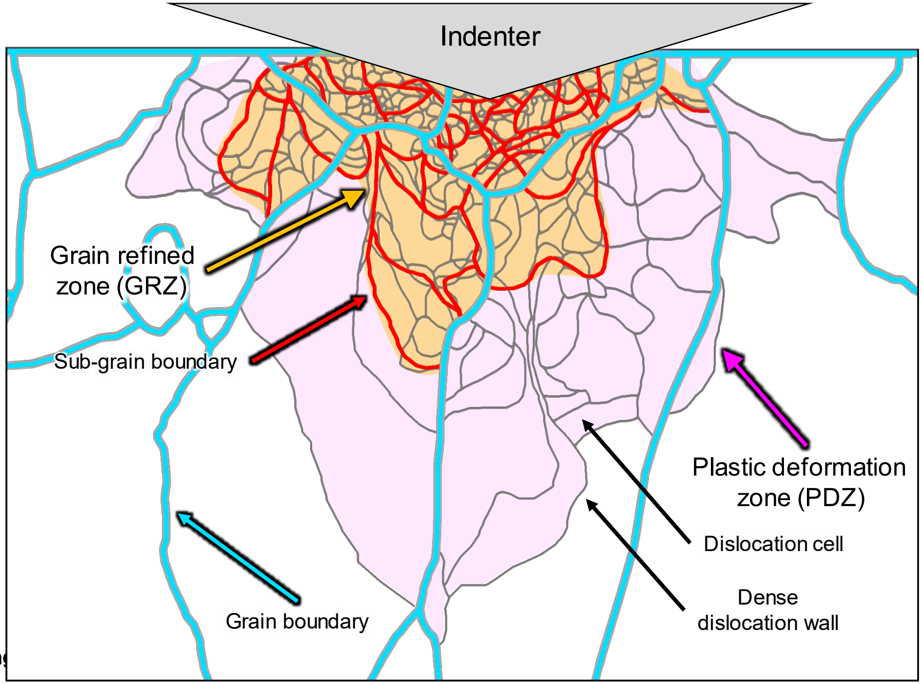


Kernel size: 100 nn & 2. Pass HQ + KF 1



Plastic deformation zone

- **Two deformation zones can be defined:**
  - 1) Grain refined zone with sub-grain boundaries (KMM 0-2°)
  - 2) Plastic deformation zone with dense dislocation walls (KAM 0-0.5°)



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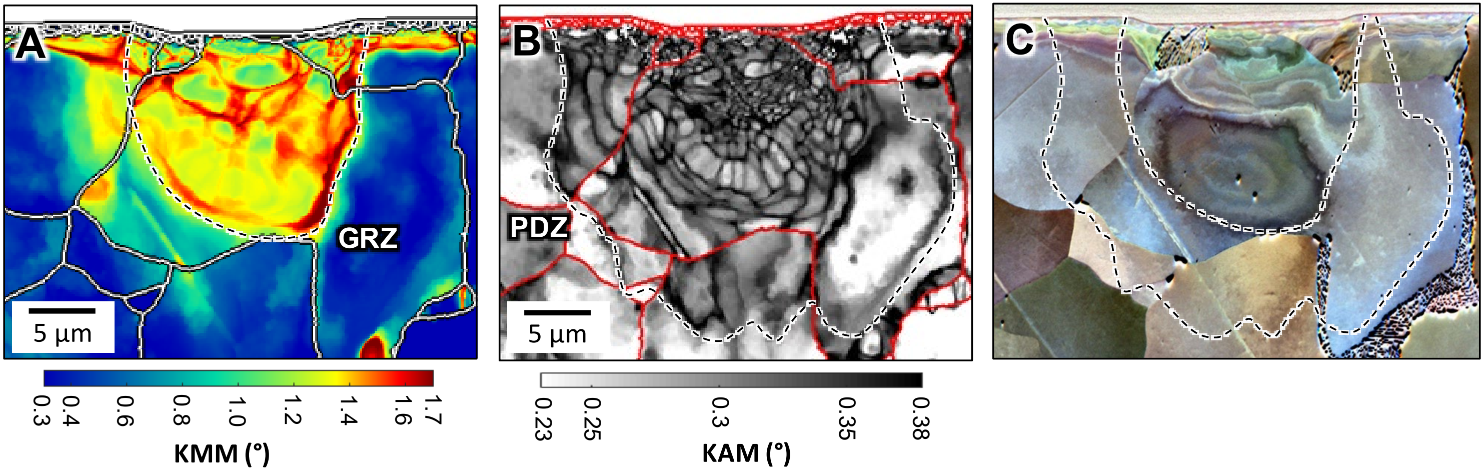
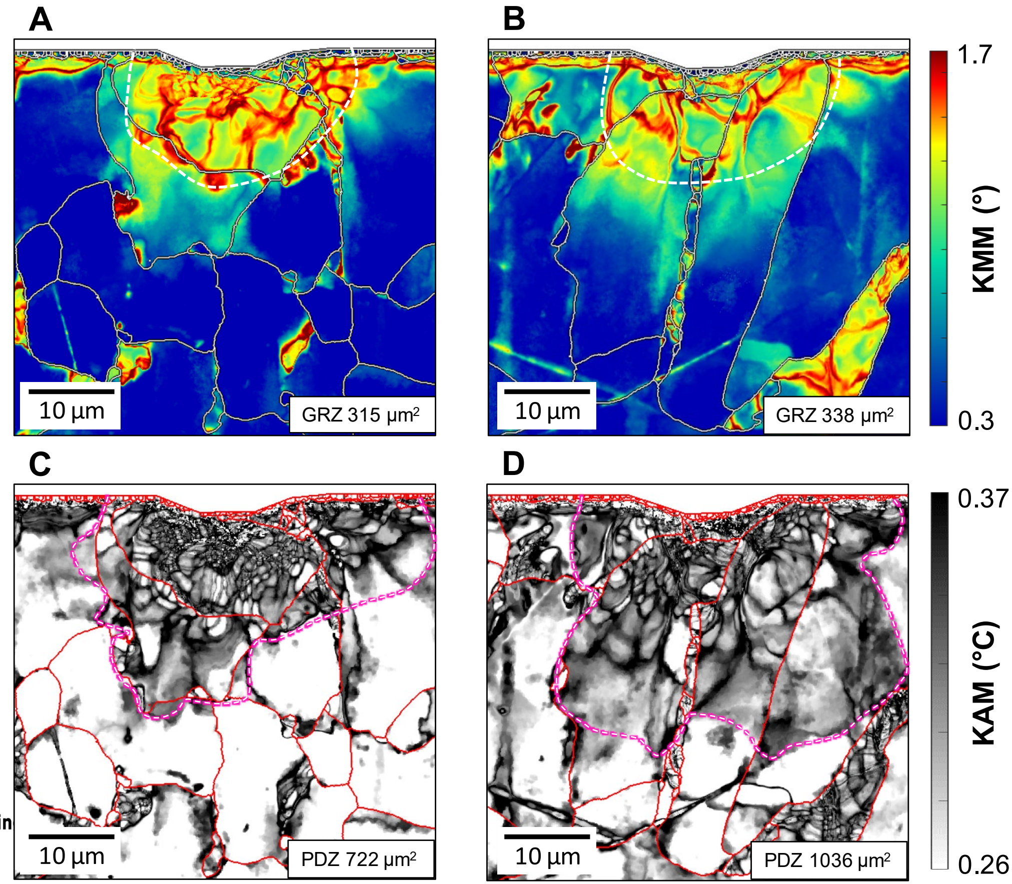


Plastic deformation zone

- **Influence of grain boundaries and grain size on deformation can be analysed**



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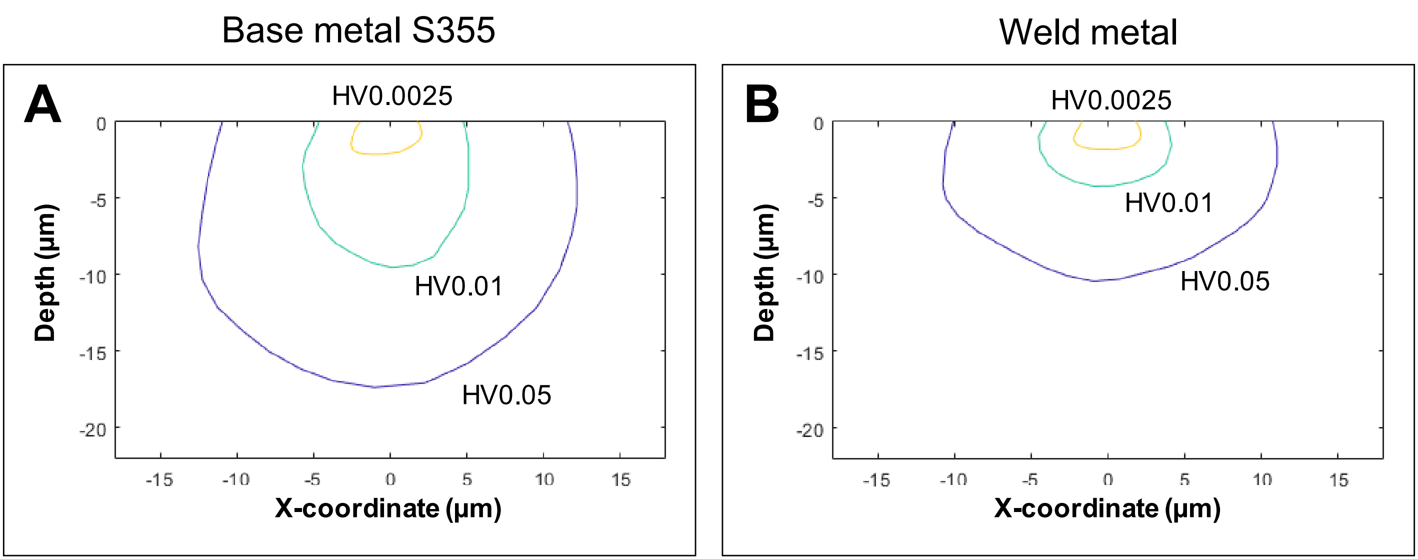
Plastic deformation zone

- **Average shape of grain refined zone for two materials at different indentation load levels**



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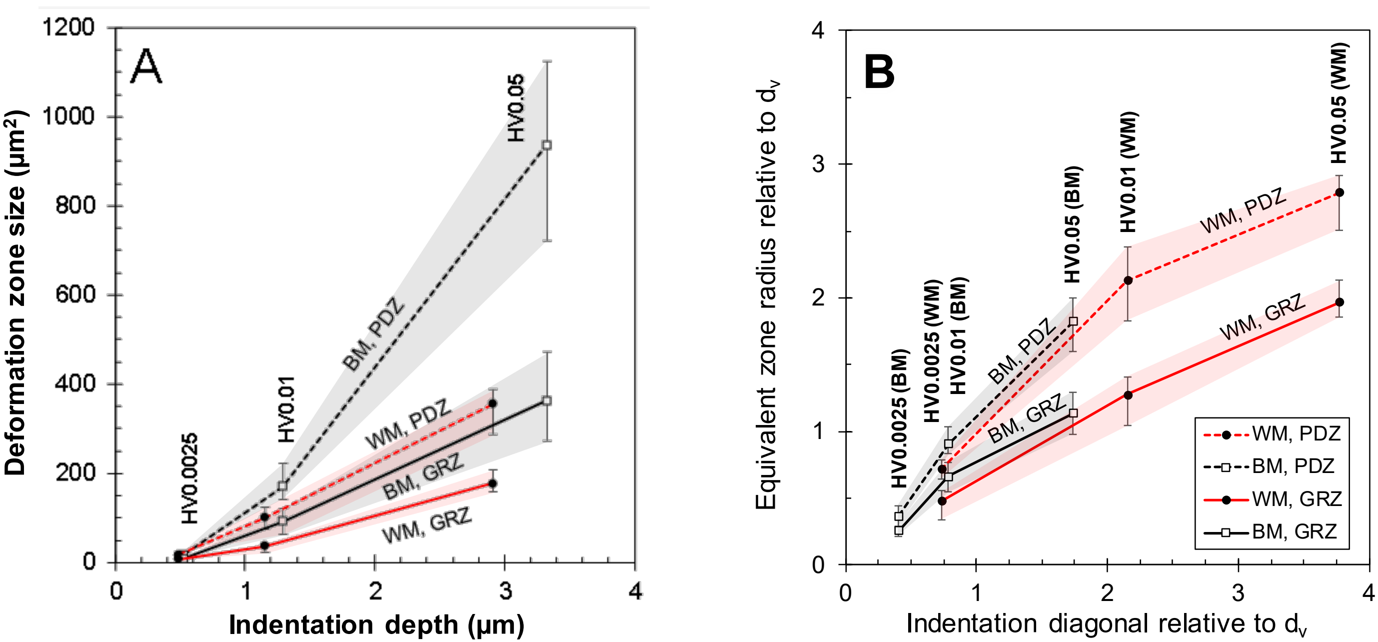
Plastic deformation zone

- **Average plastic deformation zone can be related to grain size**
- **Normalisation of deformation zone radius and indentation diagonal with volume-weighted average grain size**



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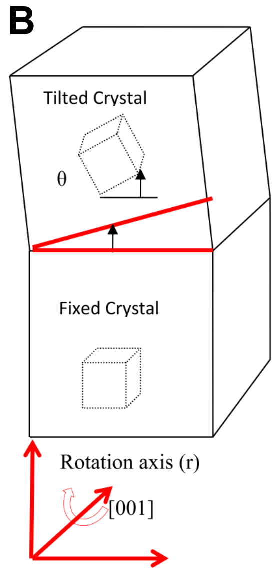
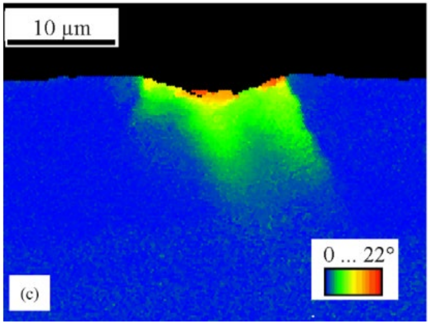
Lattice rotation analysis in MTEX

Lattice rotation analysis

- **Deformation inside a grain can be measured by comparing the orientations inside a grain to a pre-defined reference orientation**
  - Grain reference orientation deviation (GROD) or in MTEX mis2mean
- **Angle – axis pair**
- **Accurate definition of reference important, but challenging for polycrystalline materials**





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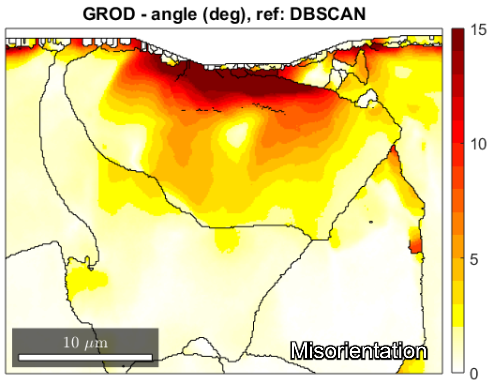
Lattice rotation analysis

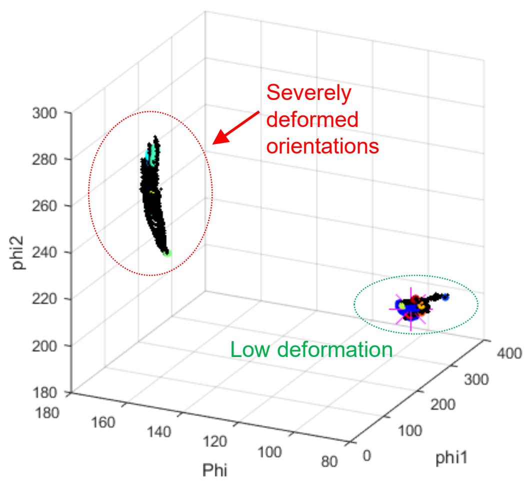
From single crystal to polycrystalline material:

- Grain orientation not known before testing

Level of deformation inside the grain can be severe: how to determine reference orientation?

- Average orientation of the grain
  - Suitable only when level of deformation is low and limited to a small area inside the grain
- Point with the highest pattern quality
  - Assumption that pattern quality correlates with plastic deformation.
  - However, pattern quality is affected by a number of other factors, making this approach unreliable.
- Point with the lowest local misorientation
  - Local misorientation also correlates with plastic deformation.
  - Reference determination unreliable, as the single orientation can deviate significantly from other points with low local misorientation

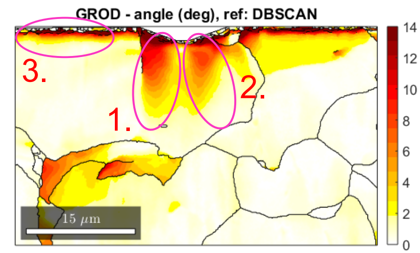


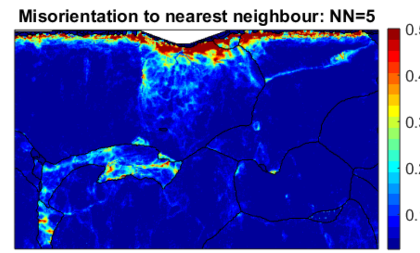


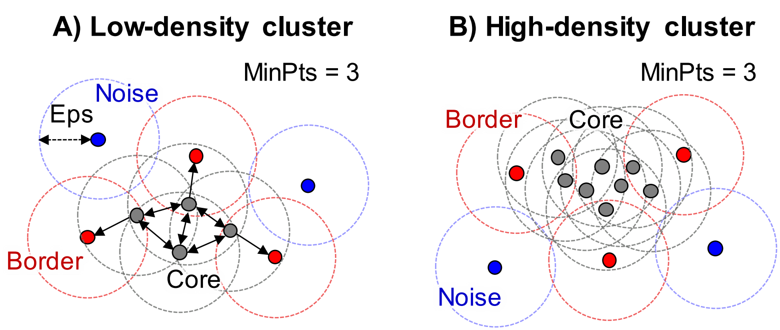
Clustering based reference orientation

- **Deformation causes orientation gradients: large misorientation to the closest neighbour inside the grain**
- **Undeformed areas have similar orientation, causing the misorientation to the nearest neighbour to be very small; In the example case ~45% of all orientation in the grain are in close proximity to each other**

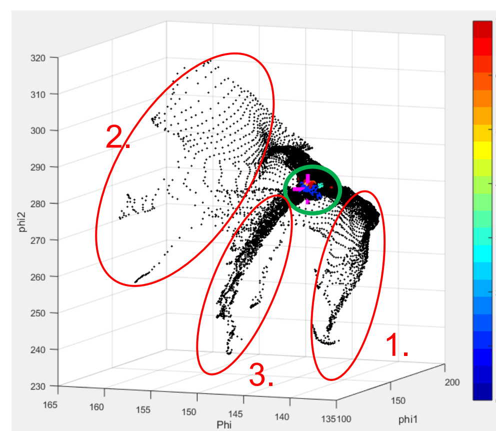
=> Orientations can be grouped with a density based clustering algorithm DBSCAN





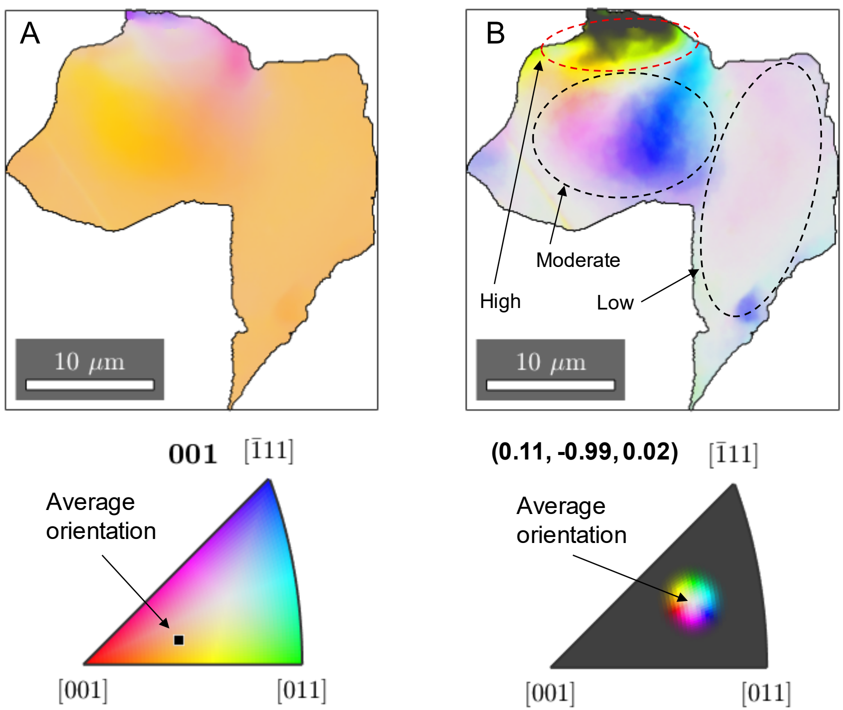


Orientation distribution inside the grain



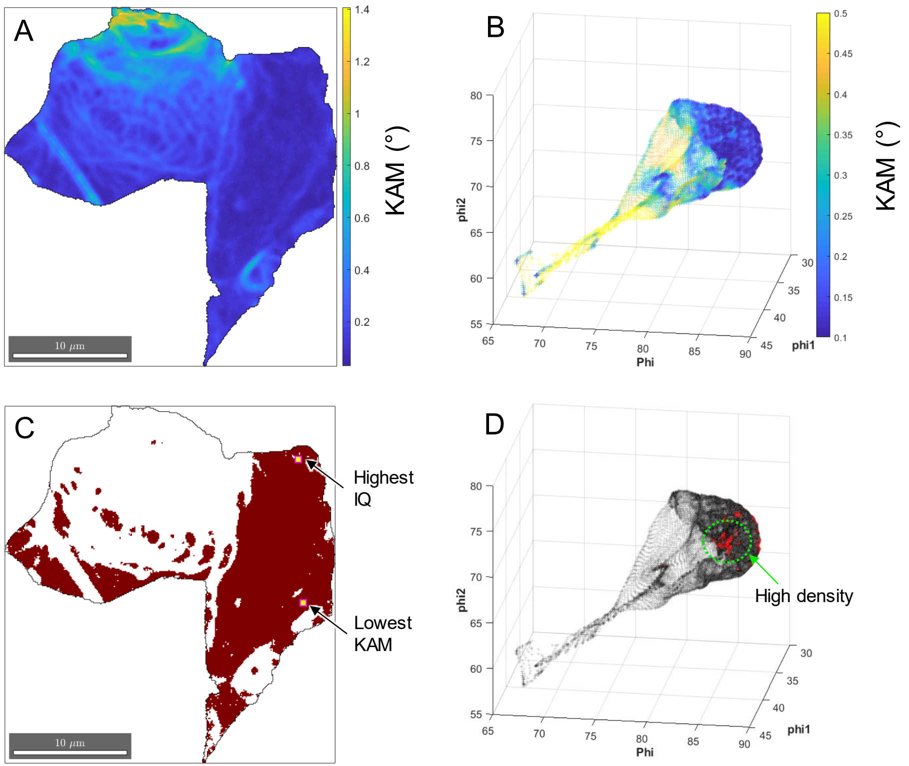
Clustering based reference orientation

- **Instead of using all grain orientations, the areas with low deformation should be used**



Clustering based reference orientation

- **A-B) Kernel misorientation used to determine plastic deformation inside the grain (5th nn)**
- **C-D) 50% of Lowest KAM are used for clustering, and this already leaves out most of the deformation**
- **While using a fixed percentage of low KAM areas is feasible, automating the correct value is difficult (grain size, level of deformation)**
- **=> Clustering used to group similar orientations**



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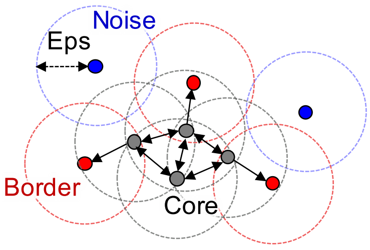
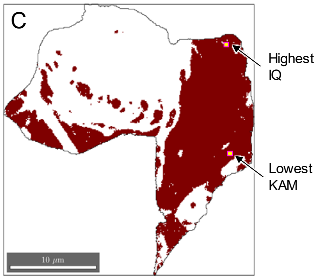
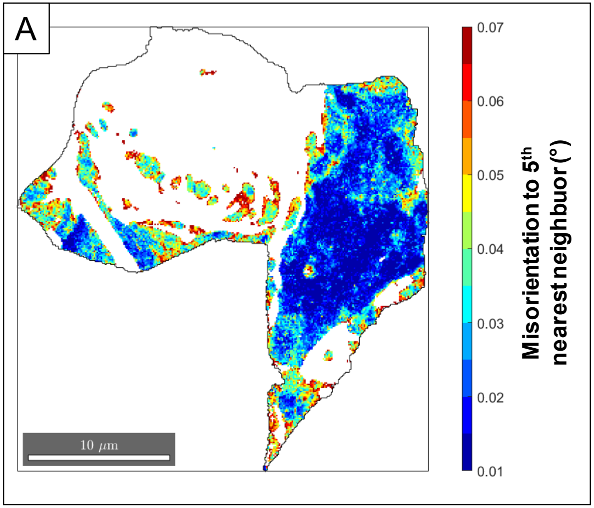
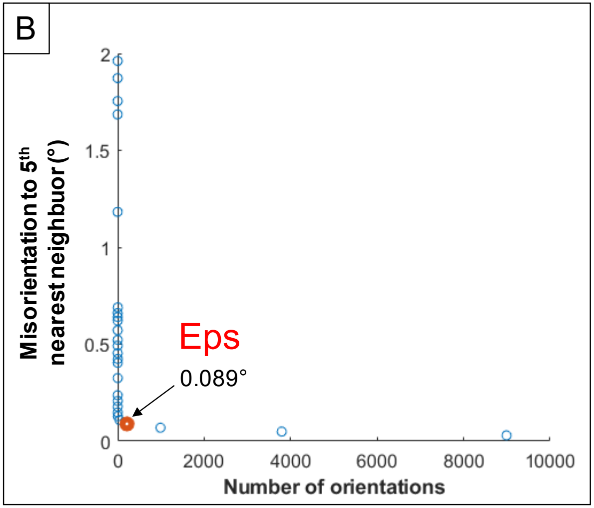
Clustering based reference orientation

- An algorithm is required to determine the clustering parameter ‘Epsilon’
- Done individually for each grain, (A) shows the misorientation to 5th nearest neighbour (no spatial information)
- In (B) the knee-point defined with bi-linear fitting defines the clustering parameter



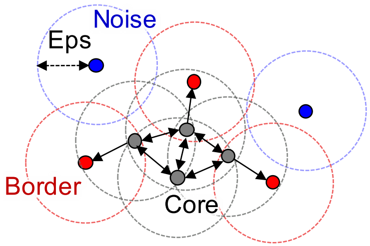


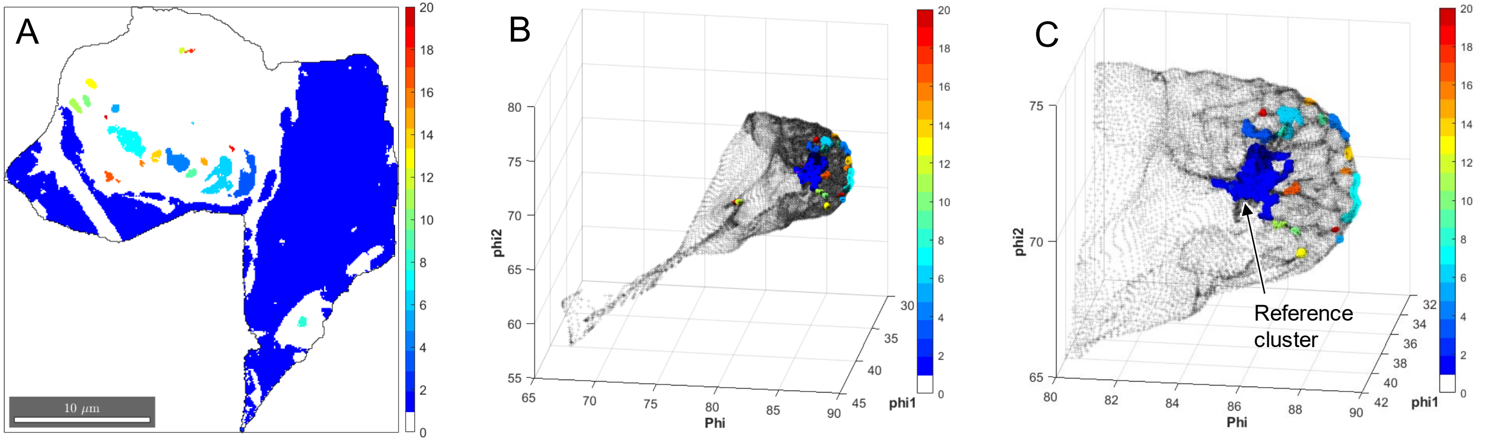




Clustering based reference orientation

- **Clustering parameters: Eps= 0.089°, MinPTS = 5**
- **One large cluster with large density, 19 small clusters**
- **Reference orientation is the median of the largest cluster**
    - Flexibility to manually choose other clusters, or to re-run clustering with alternative parameters for selected grains





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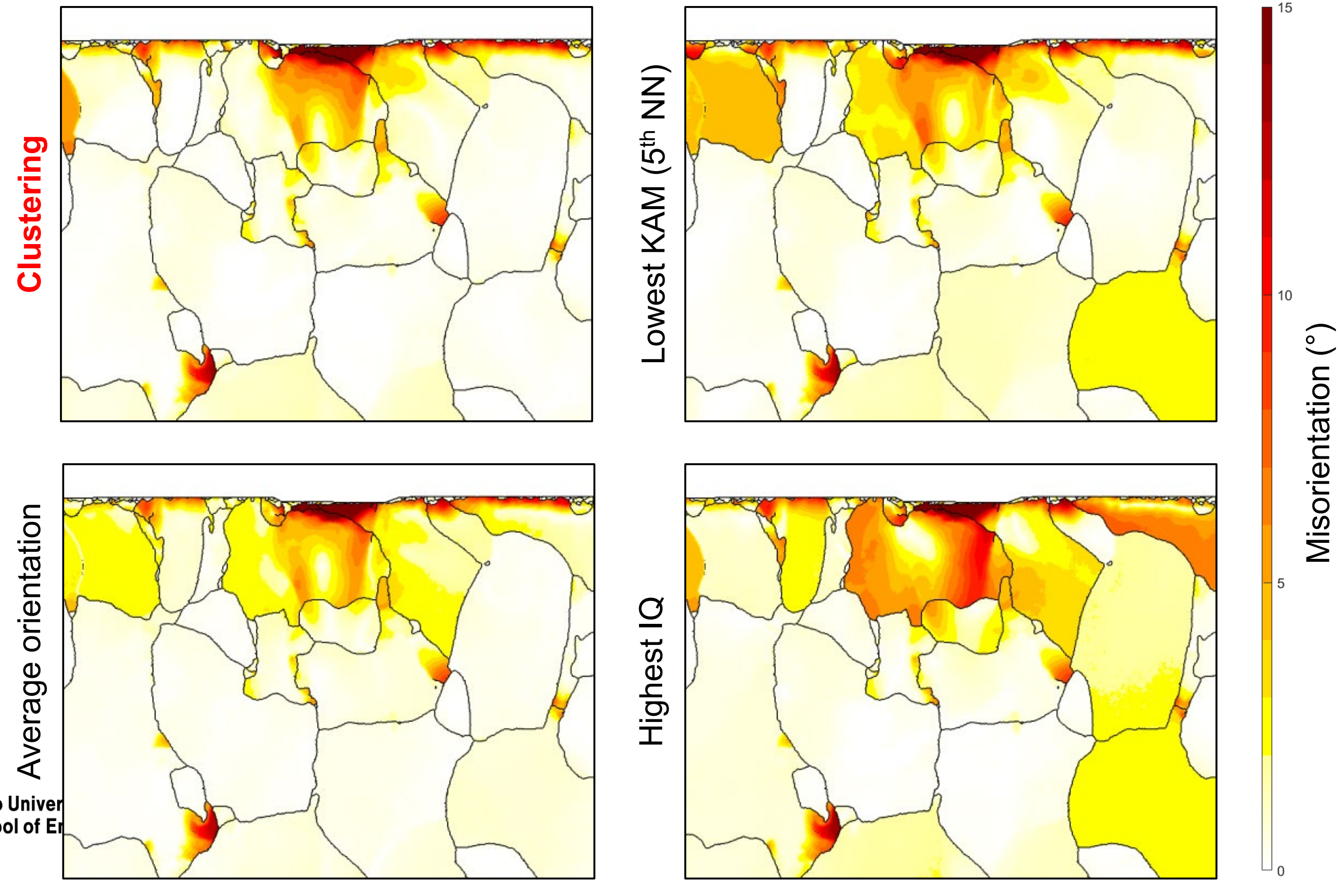
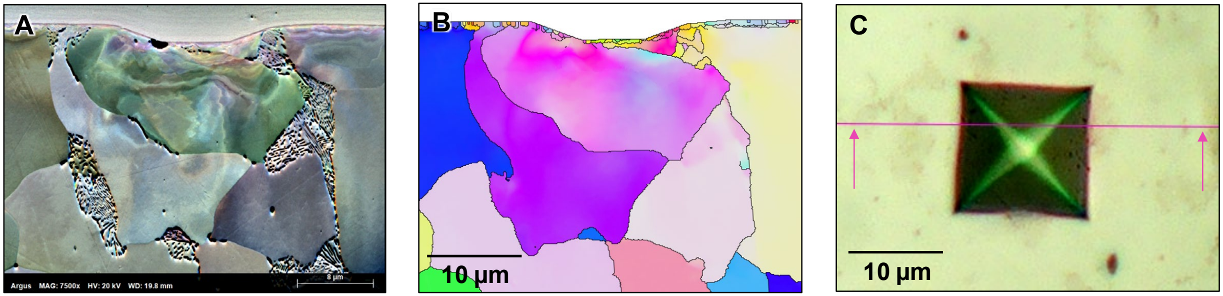
Clustering based reference orientation

- **Example of misorientation with different reference orientations**



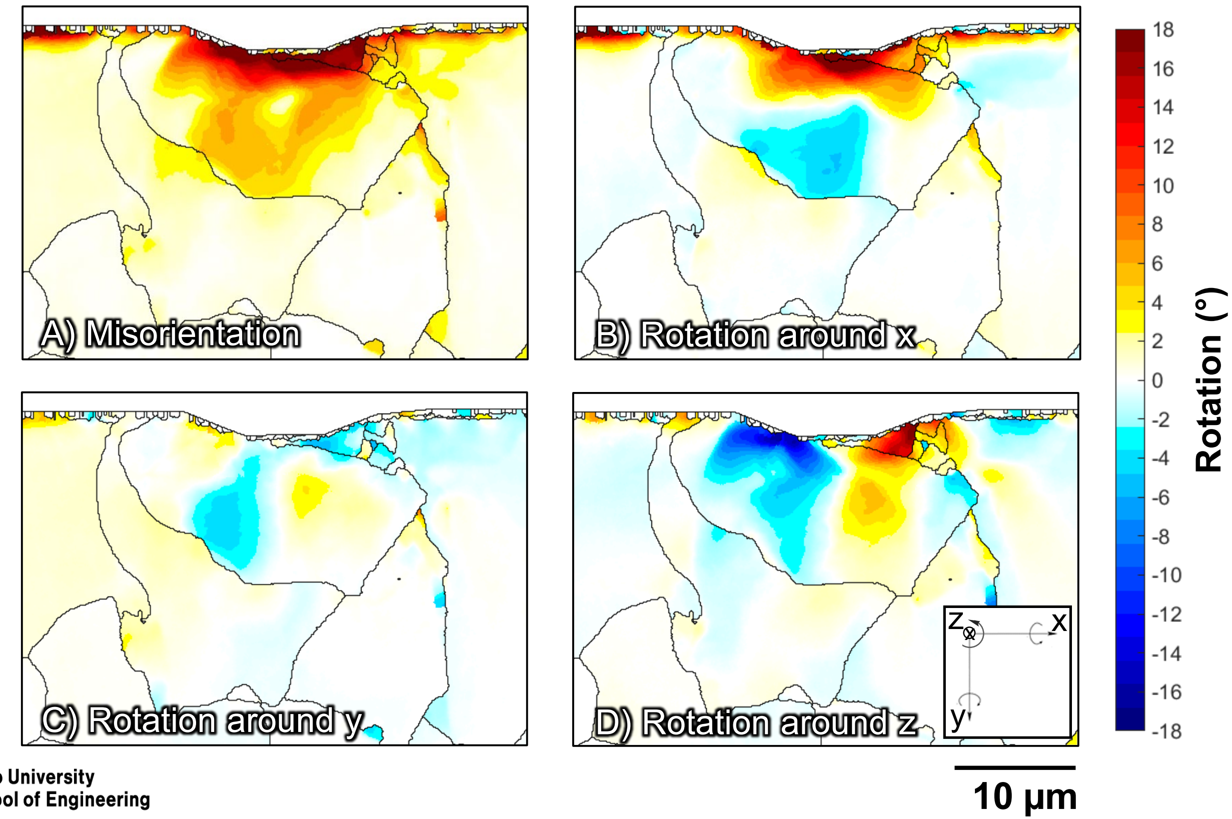
Misorientation analysis

- Example case with S355 structural steel and HV0.05 indentation to demonstrate misorientation analysis with clustering based reference orientation
- For misorientation analysis the Kuwahara Filter is not recommended, often 1. pass of Half-Quadratic filter is enough (in some cases 2. passes)



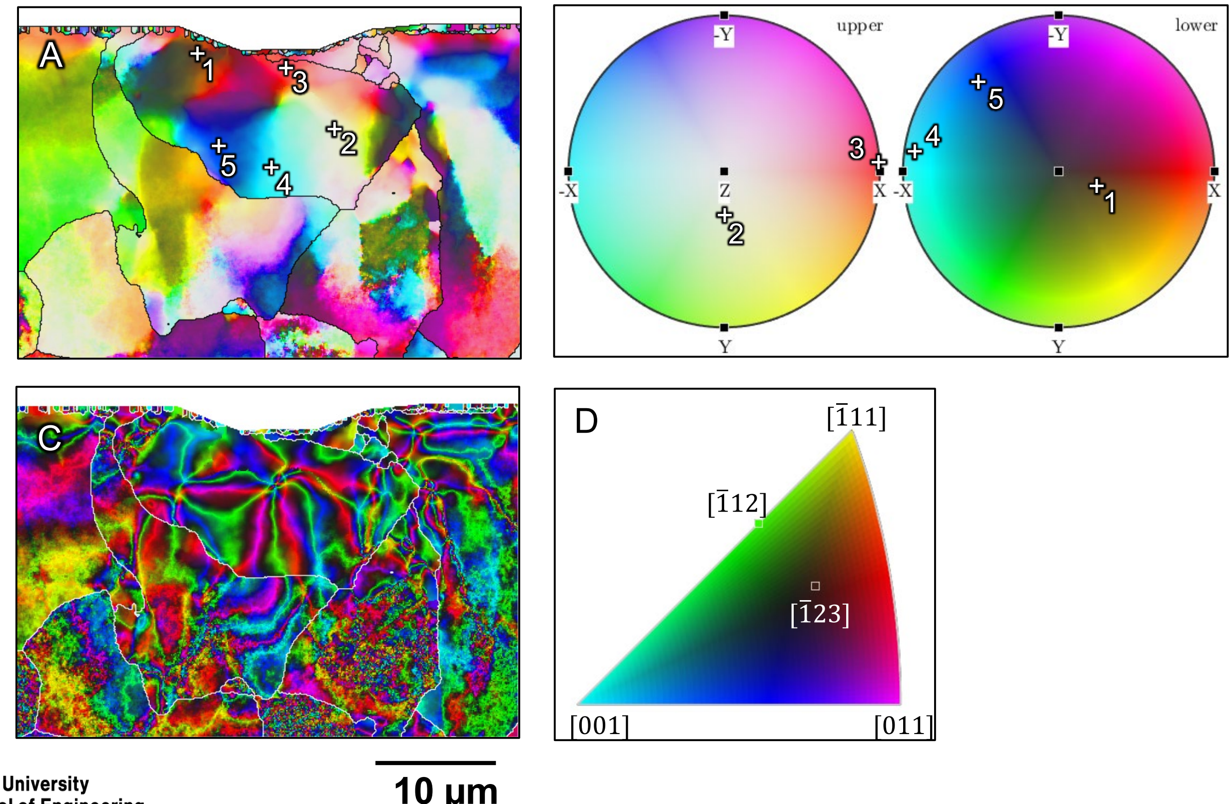
Misorientation analysis

- **Misorientation angle, and rotation relative to the specimen coordinate system**



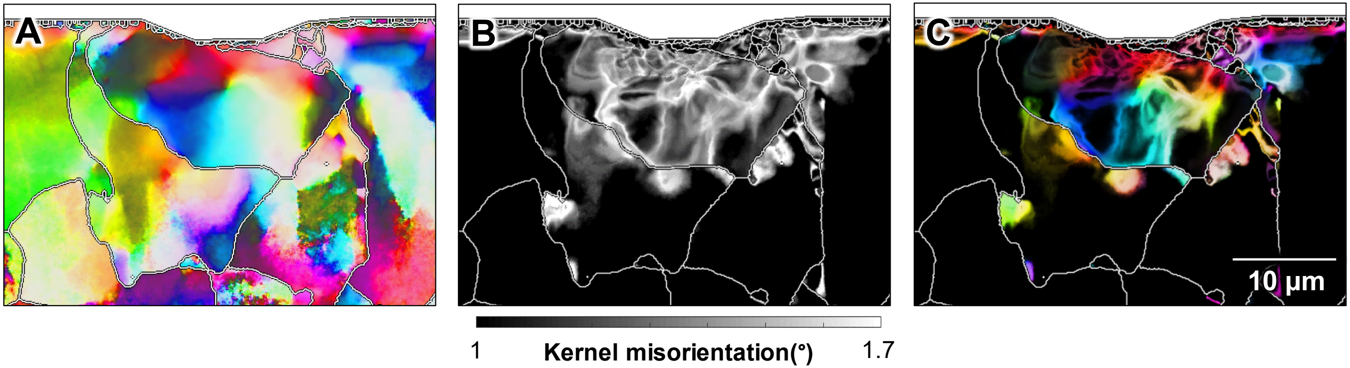
Misorientation analysis

- **Misorientation axis in specimen and crystal coordinates**

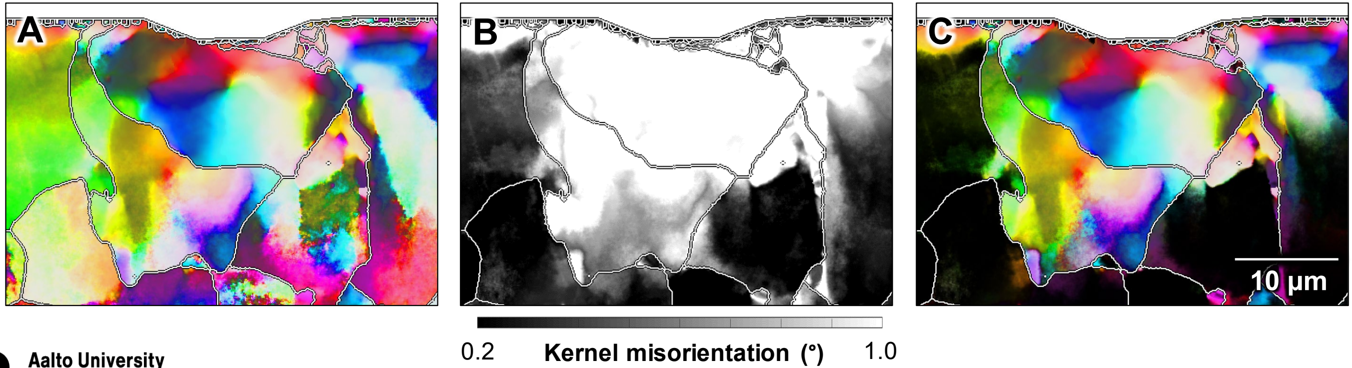


Combination of analysis approaches

- **Misorientation axis (SS) + Sub-grain boundaries**

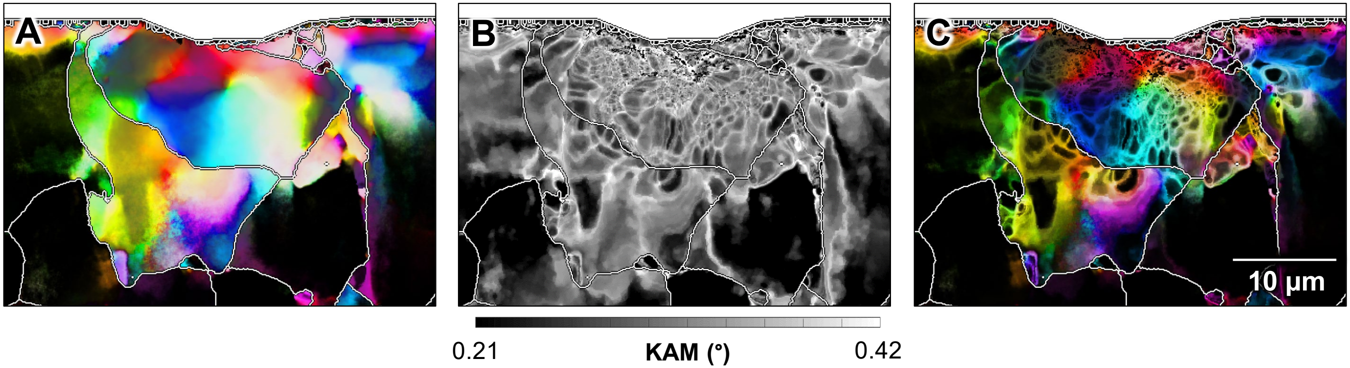


- **Misorientation axis (SS) + Extent of plastic deformation**

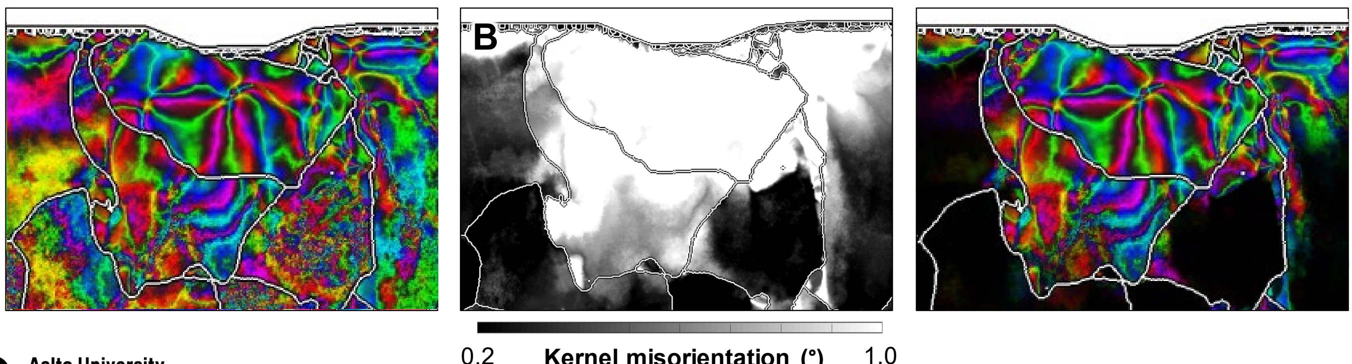


Combination of analysis approaches

- Misorientation axis (SS) including plastic deformation zone + Dense dislocation walls



- Misorientation axis (CS) + Extent of plastic deformation



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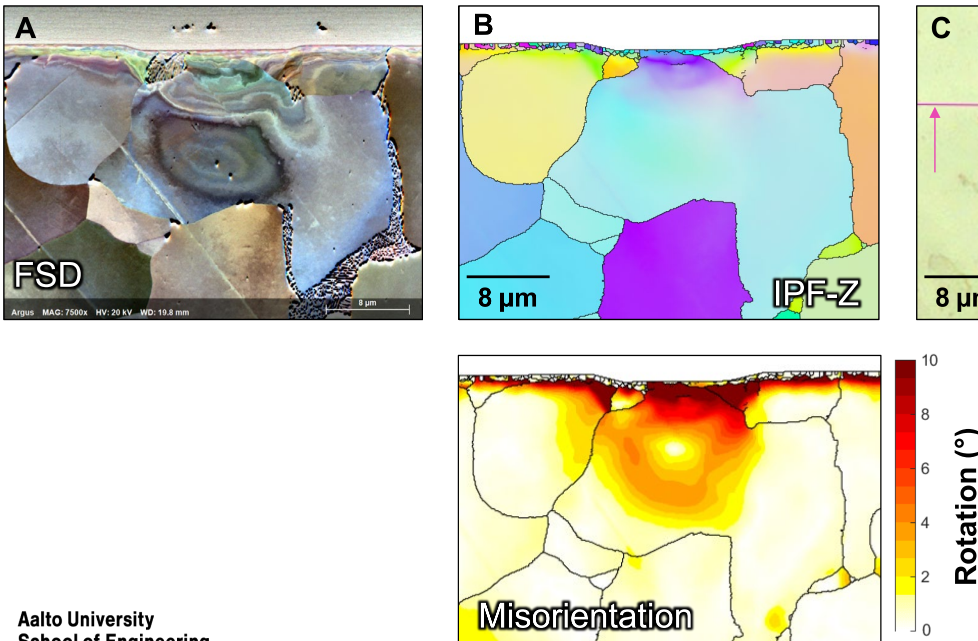
Analysis example #1

Dislocation motion

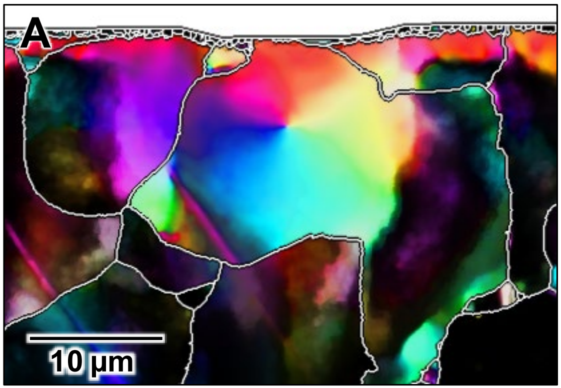
Dislocation motion

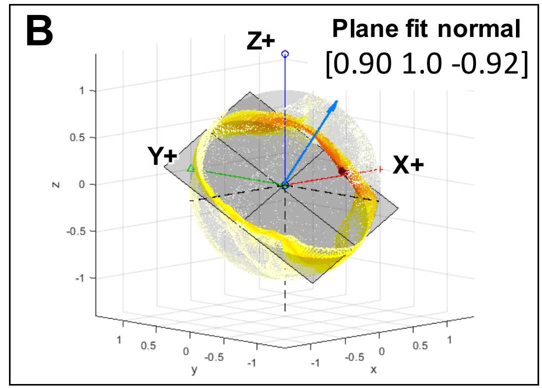
- **Clustering enables more accurate deformation analysis**
- **Investigation to resolve what causes local minima in the misorientation field**
- **Attempt to link dislocation motion with the rotation patterns**

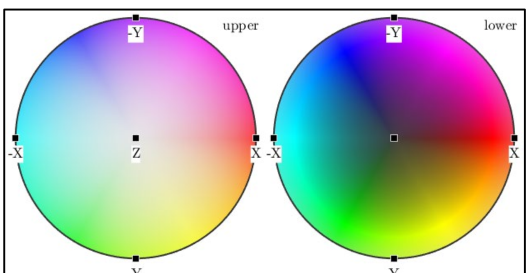


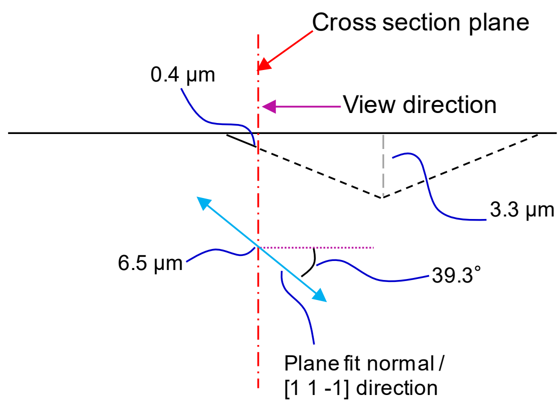
Dislocation motion

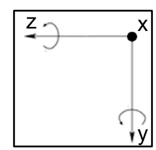
- **Misorientation axis distribution (specimen symmetry) shows strong symmetry**
- **A plane fit to the data reveals a normal vector close to [1 1 -1], indication of dislocation slip direction**







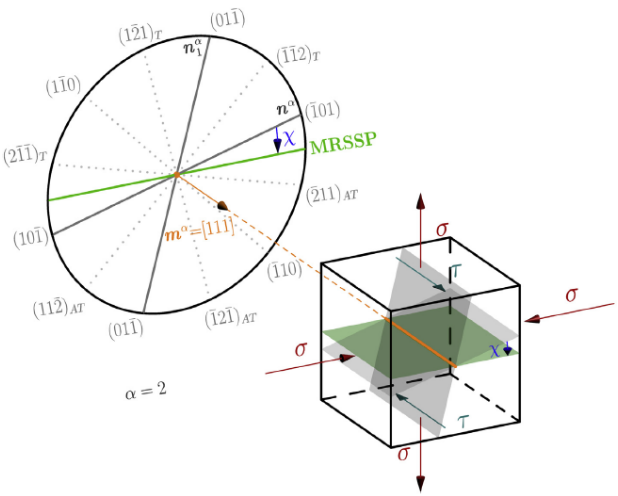




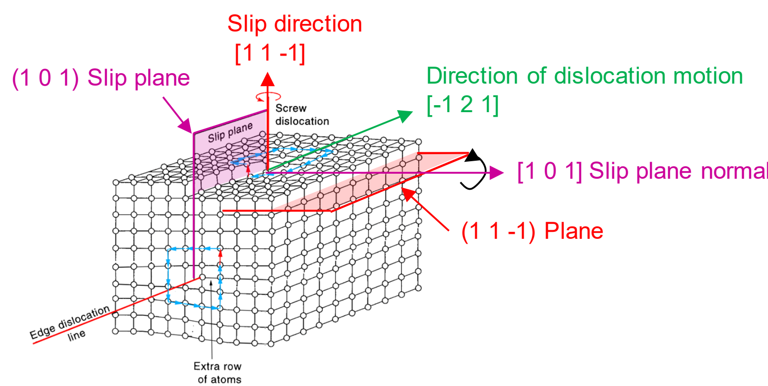
Dislocation motion

- **For BCC materials such as the structural steel, the [11-1] direction is a zone axis associated with three {110}<11-1> and three {211}<11-1> slip systems**
- **Screw dislocations determine the plastic deformation process (below Tc)**
- **For a screw dislocation with [11-1] slip direction, the dislocations on a (1 0 1) plane move to the [-1 2 1] direction**

[111] zone axis



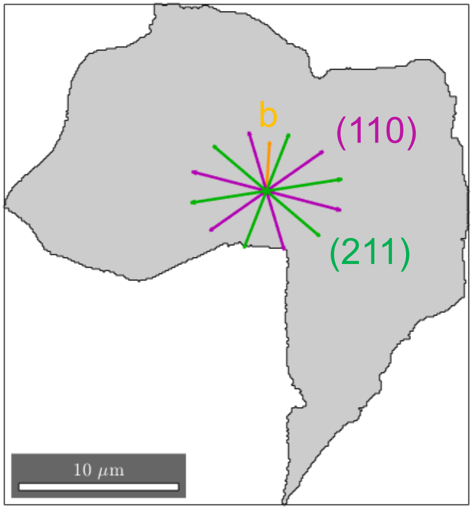
Geometry of dislocation motion for screw dislocation



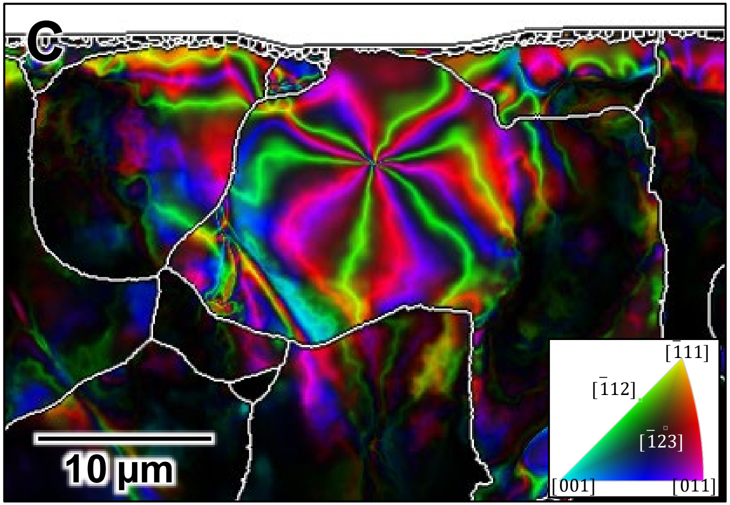
Dislocation motion

- **Based on the clustered reference orientation, direction of dislocation motion can be estimated for the {110} and {211} slip planes**
- **Experimental result shows traces radiating outward from the misorientation minimum at the centre of the grain**

Estimated dislocation motion directions



Experimental patterns



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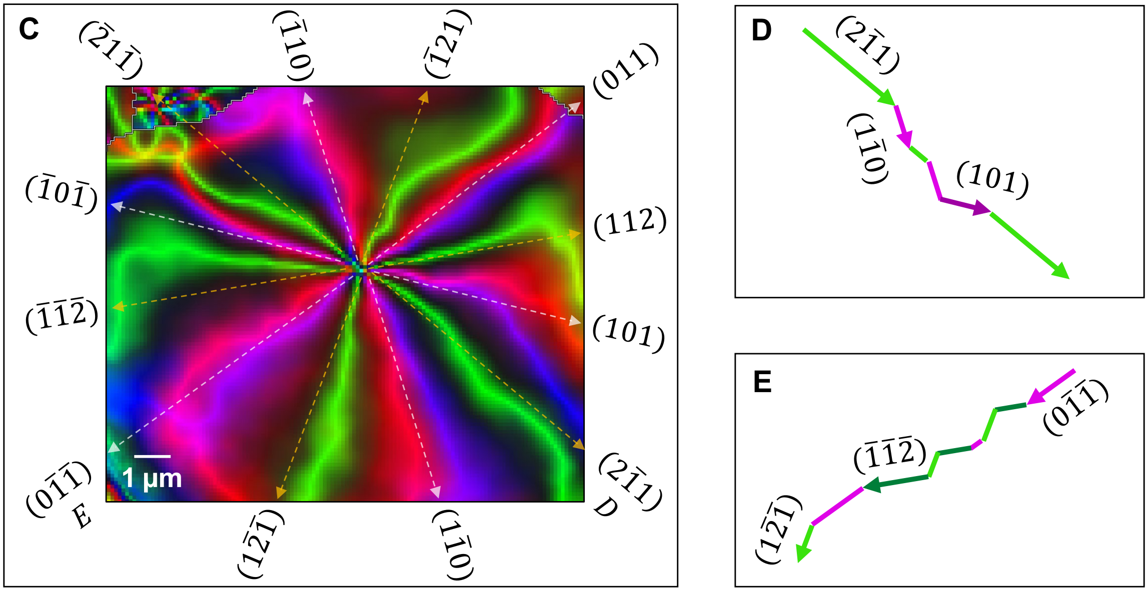
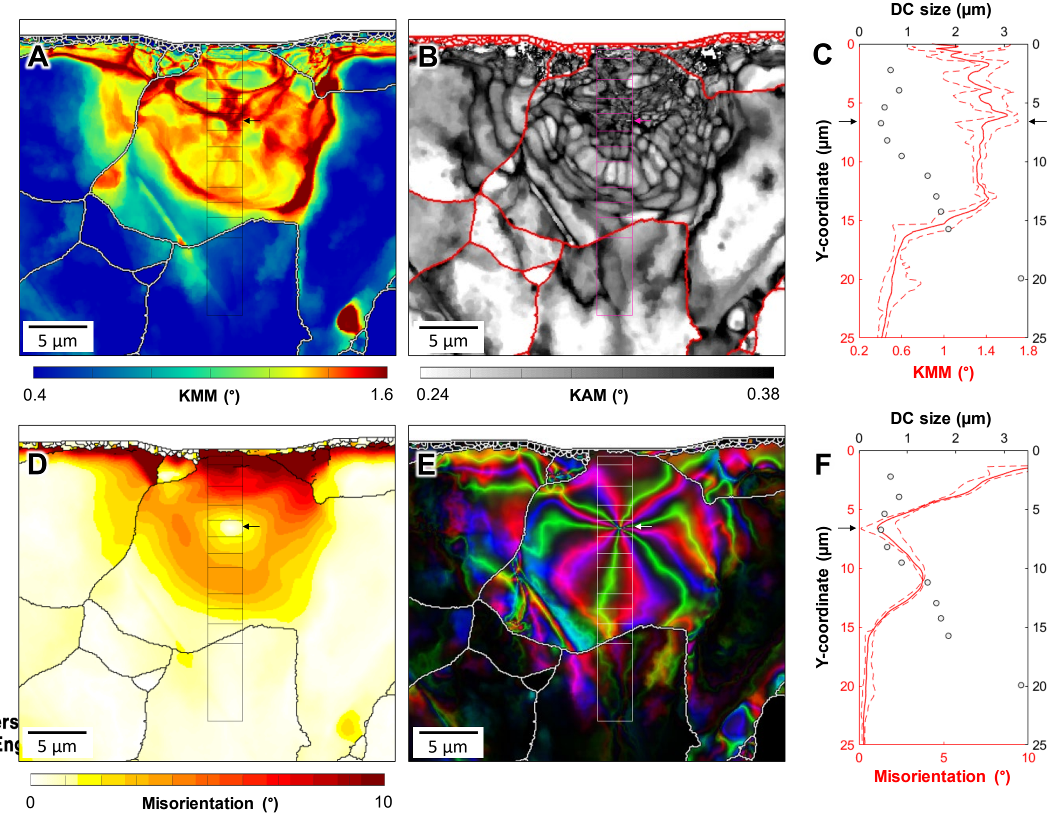
Dislocation motion

- **Global direction of the traces is consistent with the estimated directions**
- **Jagged appearance of the traces indicates cross-slip processes on neighbouring (110) and (211) slip planes, producing macroscopic slip in (211) and (110) slip planes directions**



Dislocation motion

- **Dislocation cell size is the smallest at the location of the misorientation minimum**
- **Small dislocation cell size indicates large local stresses and strains**



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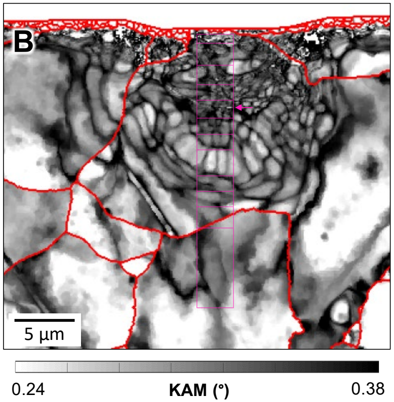
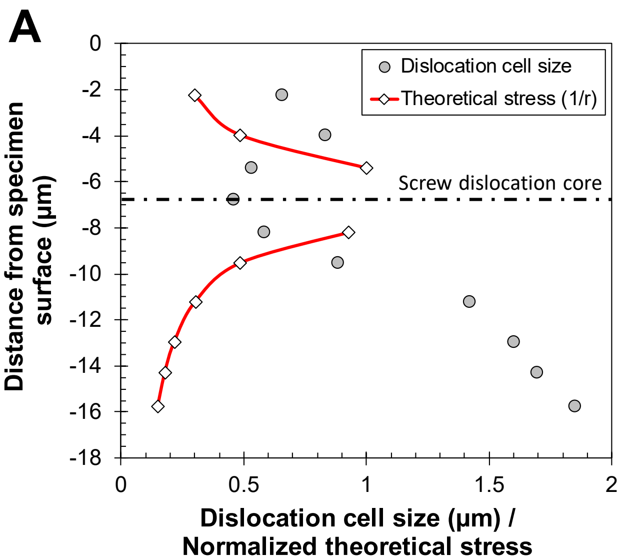
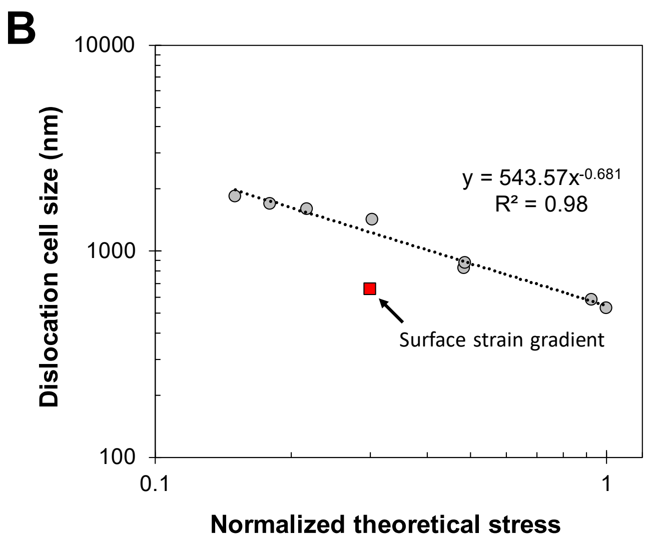
Dislocation motion

- **Assuming we are dealing with a screw dislocation, it’s stress distribution is inversely proportional to the distance from its core**
- **With this assumption, the dislocation cell size follows the theoretical stress**

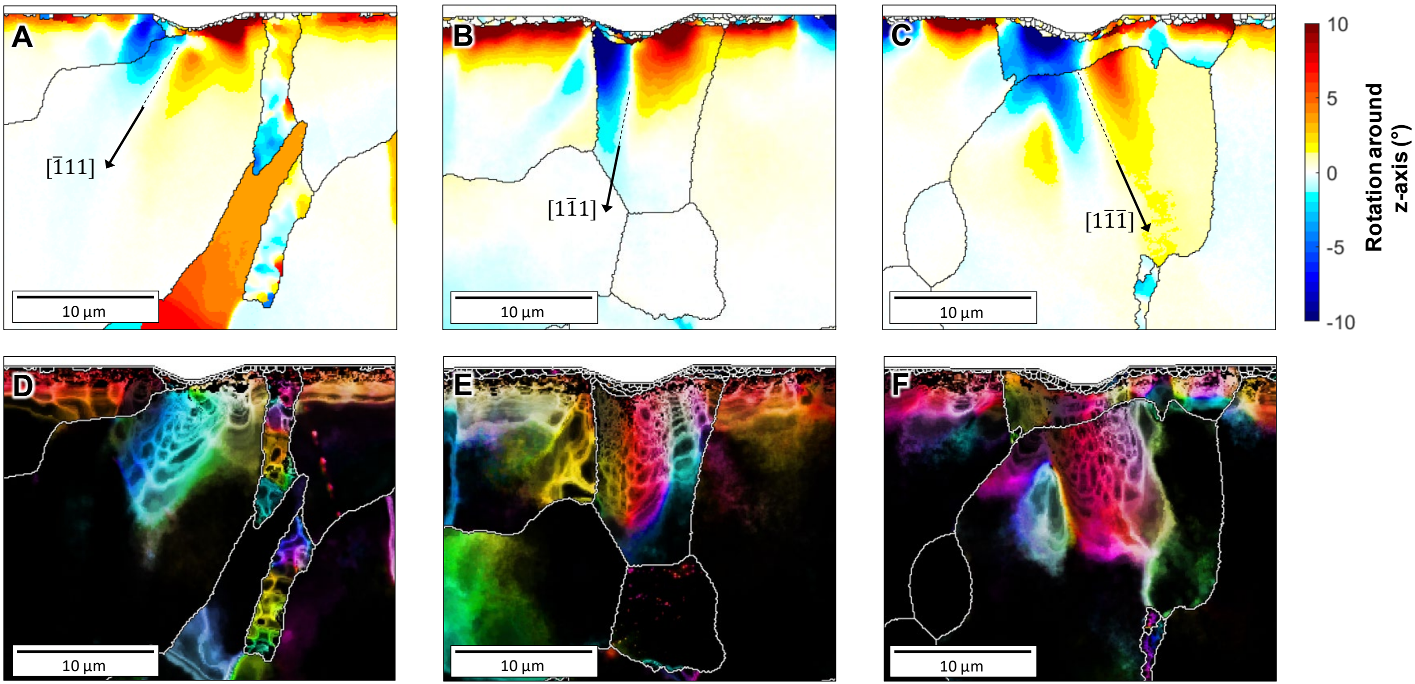






Dislocation motion

- Deformation progresses in <11-1> directions for the studied BCC steels
- Influence of grain boundaries and grain size on slip transmission can be studied



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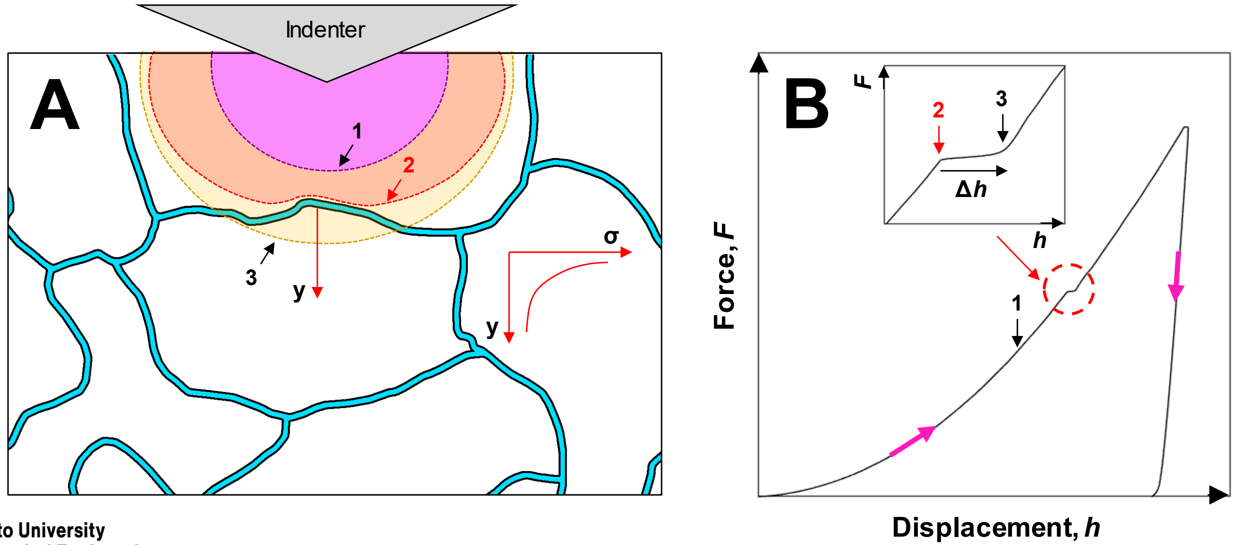
Analysis example #2

Grain interaction

Grain interaction

- As an indenter is pushed deeper into the material, the plastic deformation zone will start to influence multiple grains
- Strain bursts (Δh) during an instrumented indentation test indicate significant dislocation pile-up – slip transmission events at grain boundaries
- As transmission takes place, energy of the pile-up is released, resulting in a temporary increase of indenter's speed of displacement (in a force controlled test)



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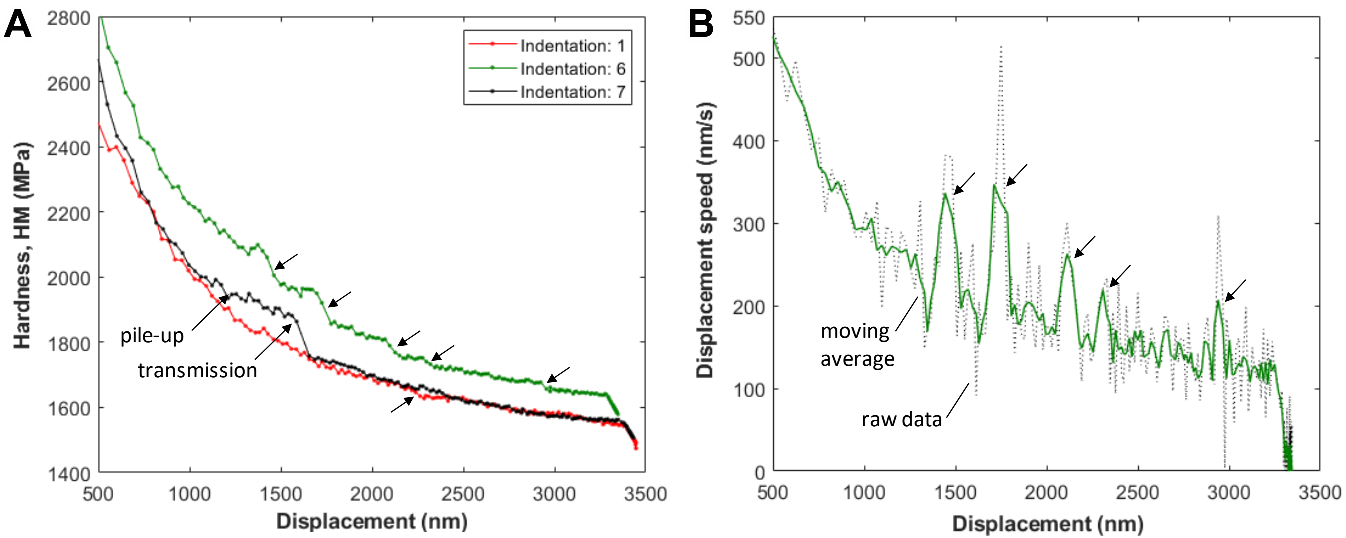
Grain interaction

- **When hardness is measured as a function if indentation depth, the changes in displacement speed (B) are visible as drops in hardness (A)**
- **Three cases:**
  - 1) No significant strain bursts (1)
  - 2) One significant strain burst (7)
  - 3) Multiple strain bursts (6)



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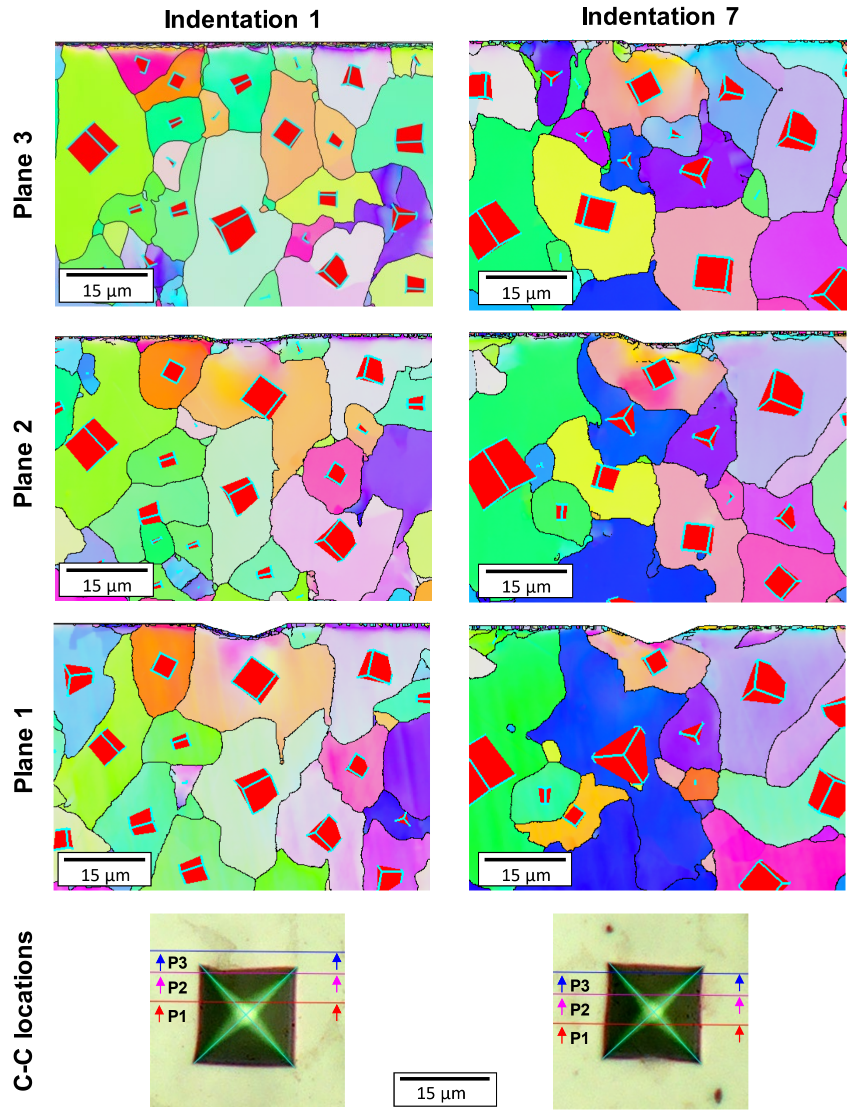
Grain interaction

- Comparison between no bursts (1) and one significant burst (7)
- Similarity of orientation for the large grain under the indenter, with a significantly larger grain size for indentation 1



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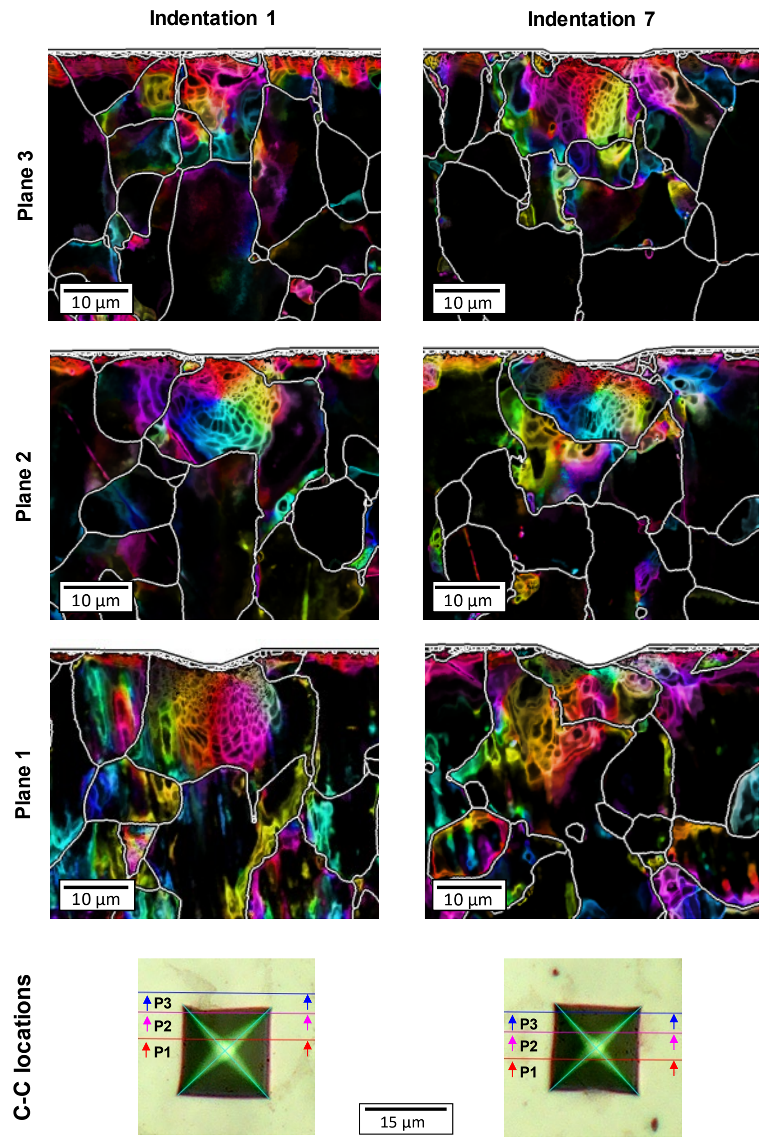
Grain interaction

- **For indentation 1 the majority of the deformation is contained in the large grain, without transmission taking place from this grain (plane 1 and 2)**
- **For indentation 7 there is clear transmission from the grain under the indenter visible on planes 1 and 2**
    - Most likely slip transmission has taken place near plane 1, as there's continuity of rotation axes (ss) across the GB, and the grain has a sharp corner (stress concentrator)



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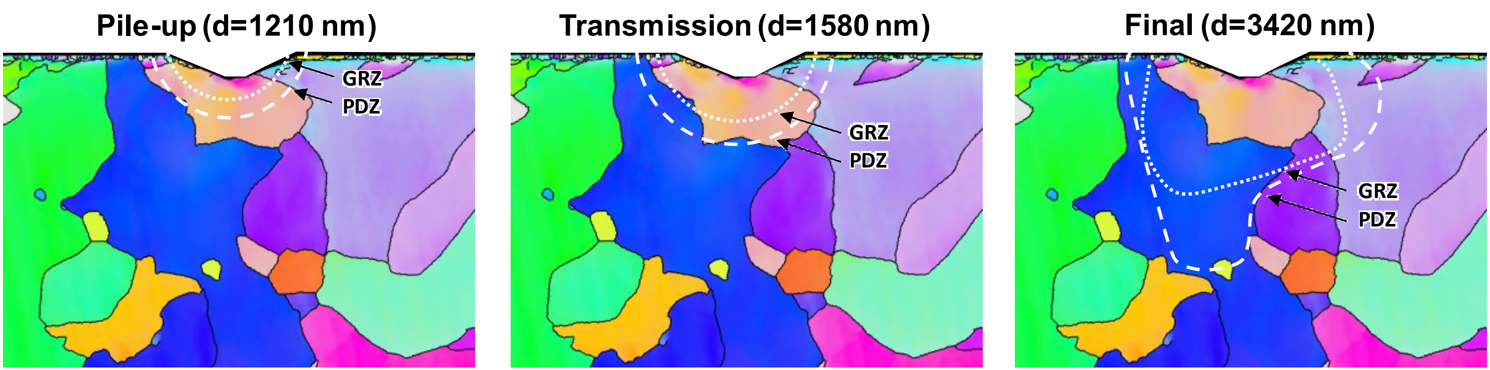
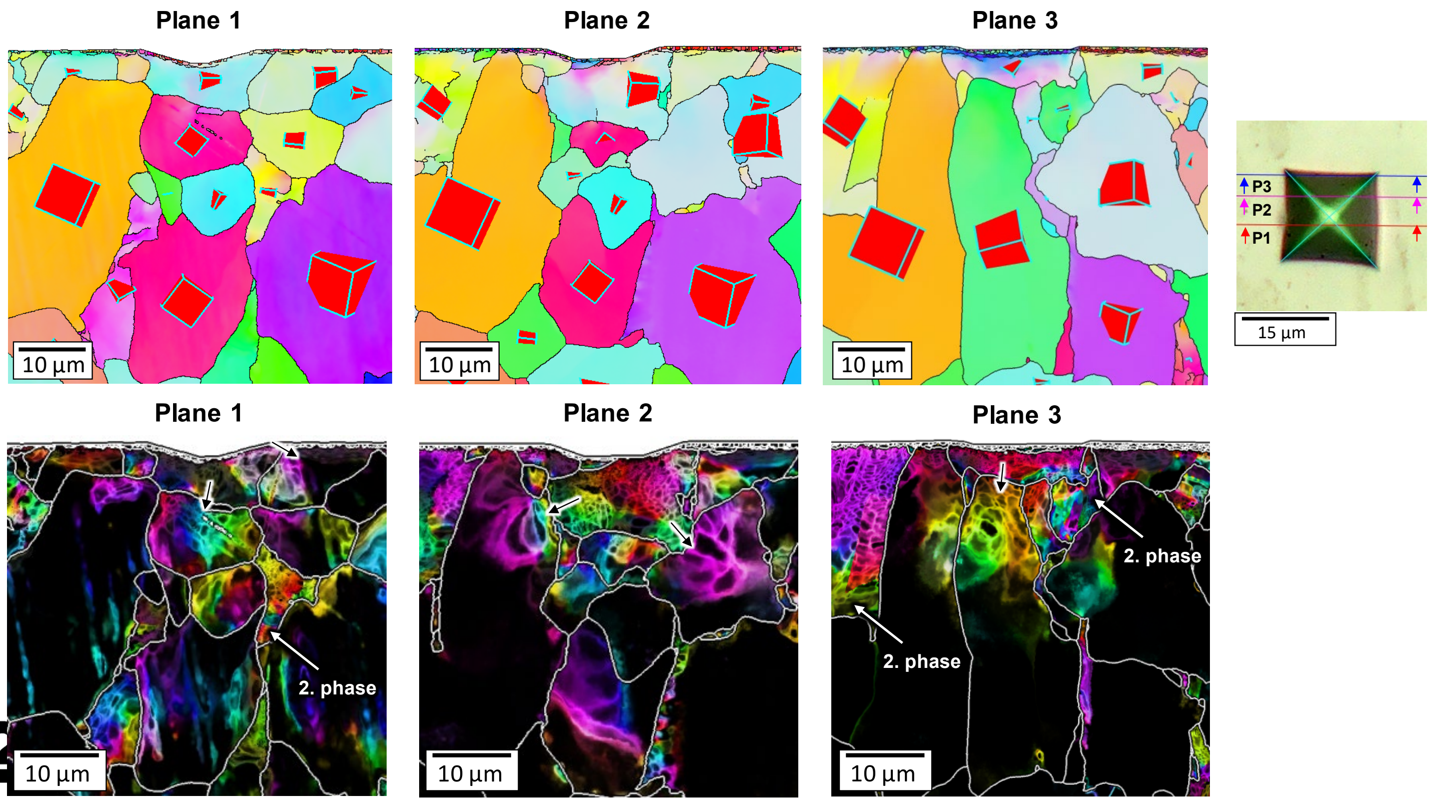
Grain interaction

- **The size of the grain refined and plastic deformation zones is estimated for the onset of the pile-up and slip transmission for indentation 7**
    - Hemispheric shape assumed for simplicity
- **Based on the estimation, pile-up effects are visible when the PDZ reaches the GB.**
- **Transmission took place approximately when the GRZ reached the GB.**



Grain interaction

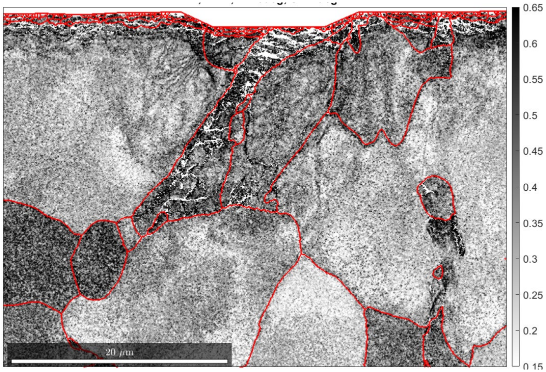
- Indentation 6 has multiple small grains under the indenter
- Misorientation analysis reveals that deformation has been transmitted to multiple grains, consistent with the strain bursts in indentation data

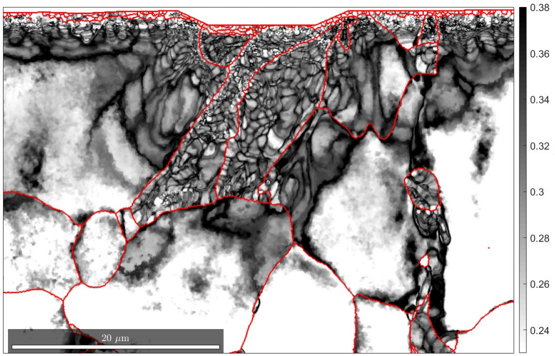


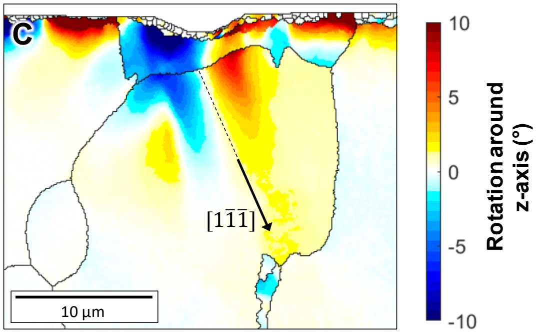
Conclusions

Conclusions

- MTEX is extremely versatile for orientation data post-processing and analysis!
- For plastic deformation analysis even Hough-based EBSD data contains a lot of information when processed and analyses in an efficient manner
- Deformation patterns can be linked with deformation mechanisms







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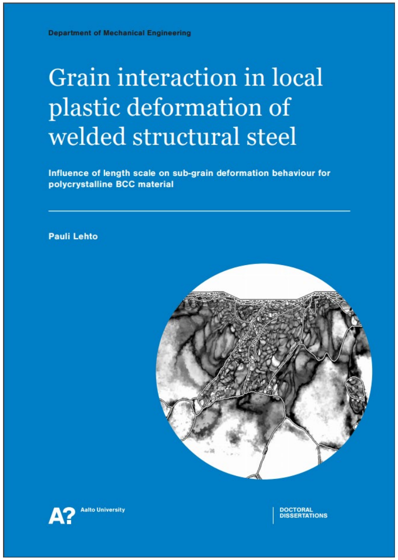
References

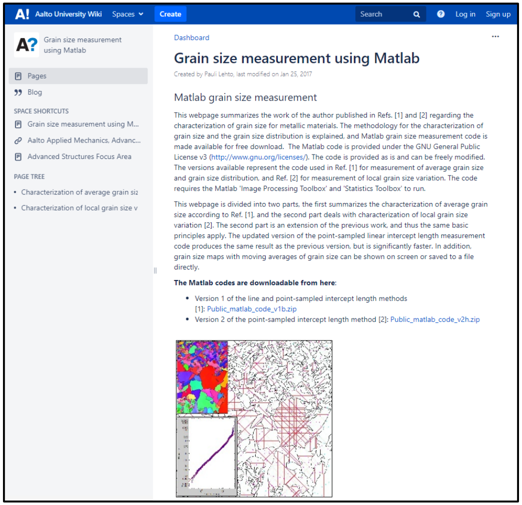
- More detailed information on the methods can be found in my doctoral dissertation: http://urn.fi/URN:ISBN:978-952-60-8807-5

- The grain size measurement methods are available at: http://wiki.aalto.fi/display/GSMUM/
  - MTEX implementation is future work

- The adaptive kernel misorientation method and clustering based misorientation analysis will be made available as an open source code in due course with example datasets

- **For any questions don't hesitate to contact me at pauli.lehto (at) aalto.fi**





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