Aalto University
School of Engineering

Materials testing & characterization

in the age of big data

MEC-E1070 Seminar 1
September 4, 2023

Sven Bossuyt

Materials testing & Characterization

Test *[verb, with object]*

- take measures to check the quality, performance, or reliability of (something), especially before putting it into widespread use or practice
- reveal the strengths or capabilities of (someone or something) by putting them under strain
  - *destructive vs. non-destructive testing, proof testing*

Characterize *[verb, with object]*

- describe the distinctive nature or features of
- characterization includes analyzing the result of testing, as well as analyzing or describing the results of observations

Often used for mechanical *testing and* microstructure *characterization*

Three reasons for mechanical testing

Exploration

- when the material behaviour is a priori unknown
- experiments to learn what variables are important and what constitutive equations relate the response to these variables
- analysis should not pre-suppose anything

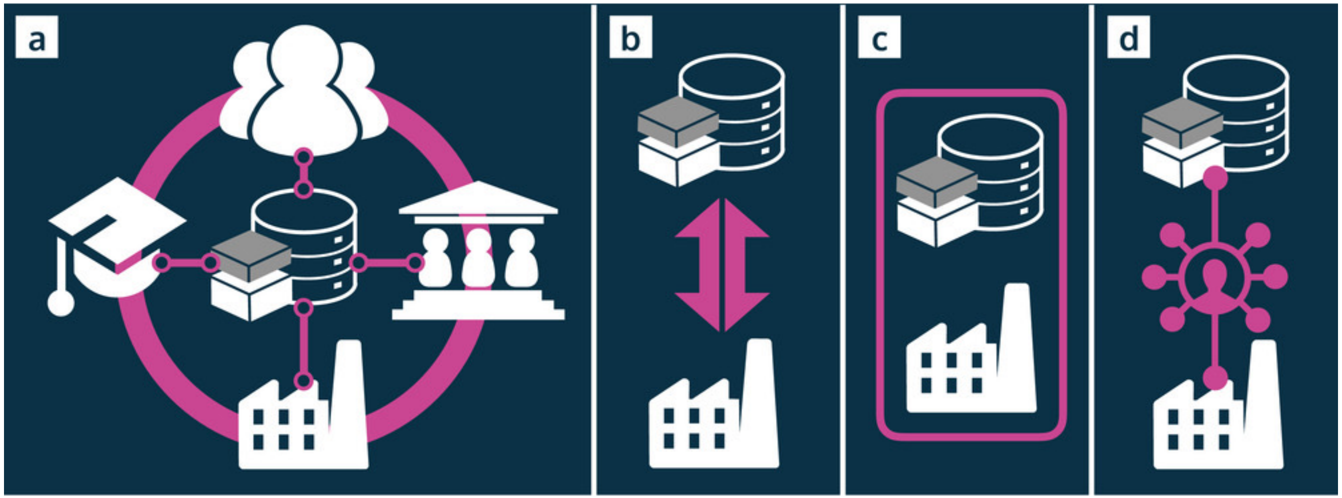
Calibration

- to identify the parameters in the constitutive equations
- experiments maximise sensitivity to these parameters, and minimise sensitivity to others

Validation

- to show that the modelling properly combines the influence of all the important variables

Review article by Patrik Rinke



a) Schematic of an ecosystem in data-driven materials science with materials data platforms at the center. In this ecosystem, different stakeholders from universities, the public, industry, and government facilitate the development of a technology. b–d) Possible relationships between data platforms and industry, discussed in the text.

Himanen, L., Geurts, A., Foster, A. S., Rinke, P., Data-Driven Materials Science: Status, Challenges, and Perspectives. *Adv. Sci.* 2019, 6, 1900808. https://doi.org/10.1002/advs.201900808

Data-driven computational mechanics

e.g., Mike Ortiz (Caltech), Julien Rhétoré (EC Nantes)

Principle

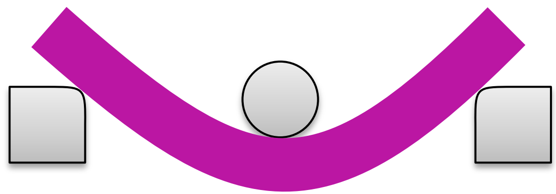
- Replace constitutive equations with additional stress state: ”mechanical stress” and ”material stress”
- Instead of minimizing virtual work of constitutive equations, minimize ”distance” between calculated stress field and a data set

Practice

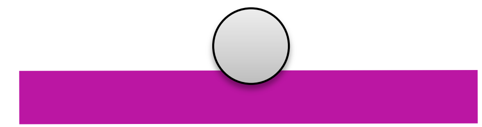
- Needs data set spanning stress states of simulation
  - *Full-field measurements help!*
- Needs internal variables for non-elastic behaviour
  - *Some theoretical work, not shown with experimental data*

Origin of Mechanical Properties

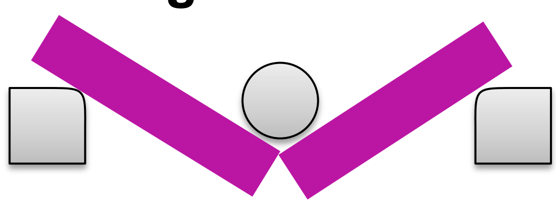
Stiffness



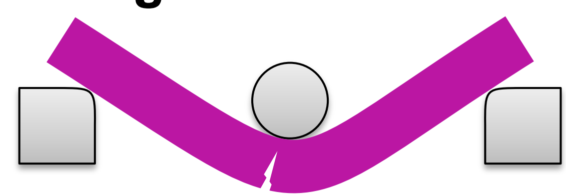
Hardness



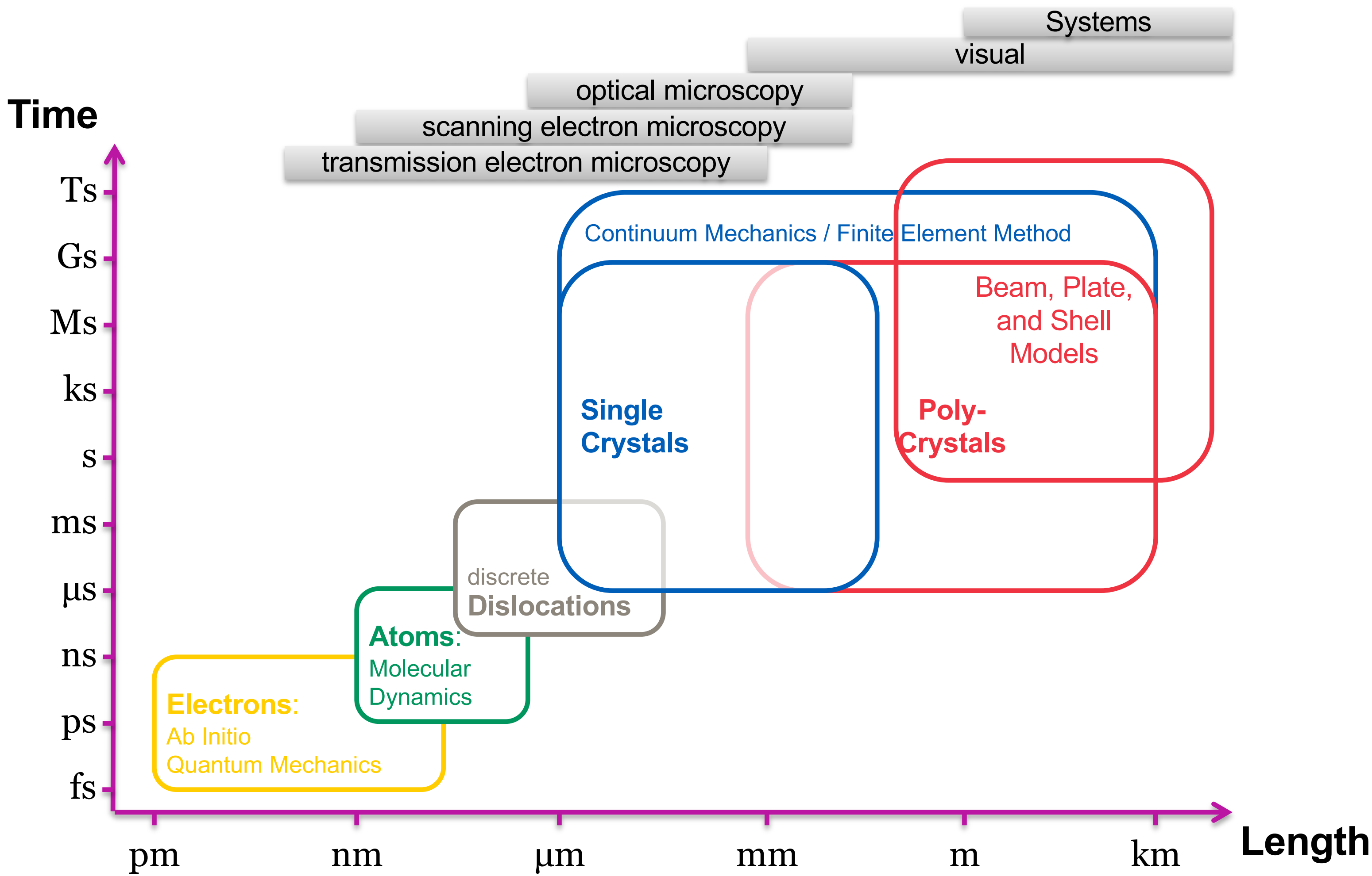
Strength



Toughness



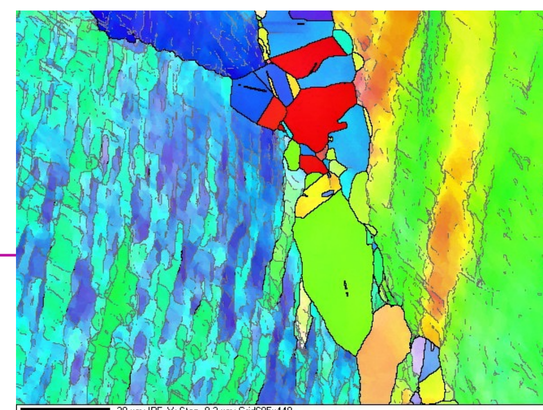
Multi-Scale Modeling

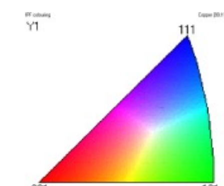


Materials characterization

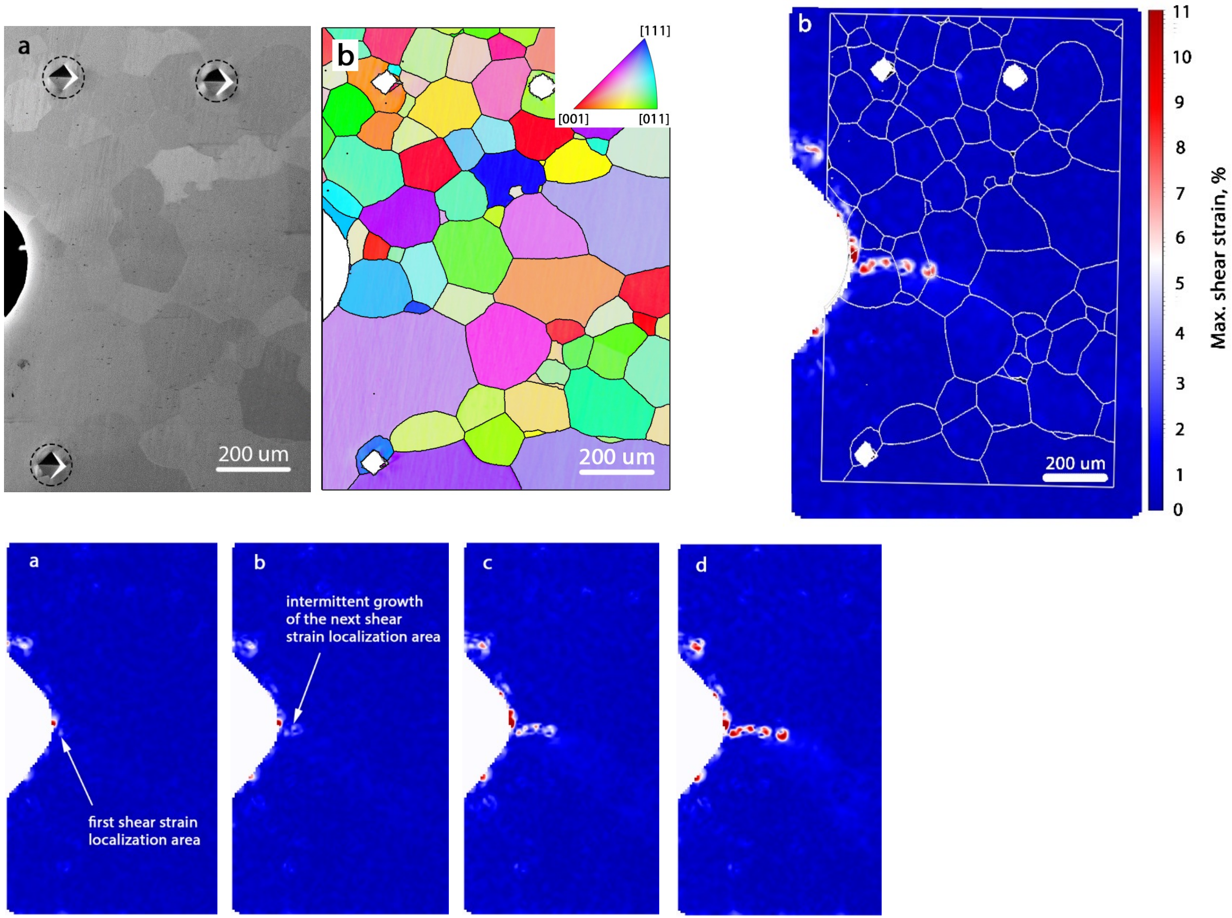
at Aalto Engineering Materials Lab

- FEGSEM+EBSD/EDS
- 6 mechanical testing systems
- Optical strain measurement
- Metallography and microhardness testing
- Nanoindentation
- X-ray diffraction and ring-core residual stress measurement
- Unique thermal fatigue testing system
- High-temperature DSC
- Electrochemical measurements
- Thermal desorption spectrometry of hydrogen
- Non-destructive testing facilities
  - *(ultrasound, acoustic emission, eddy current, Barkhausen noise)*

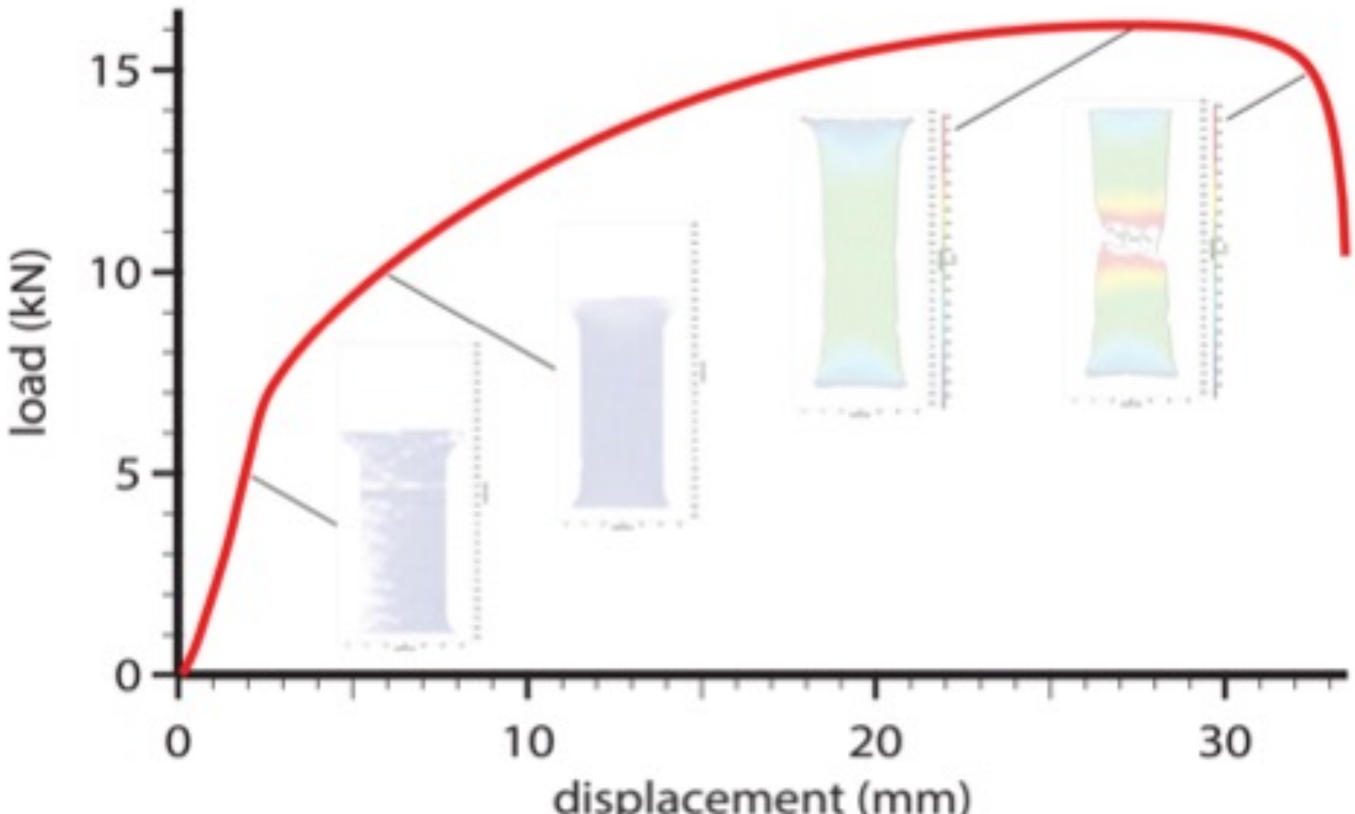




Micromechanical Strain Measurement



Localized deformation



Digital Image Correlation
principle

Find displacement fields that map sequence of images of un-deformed material onto observed sequence of images of deformed material

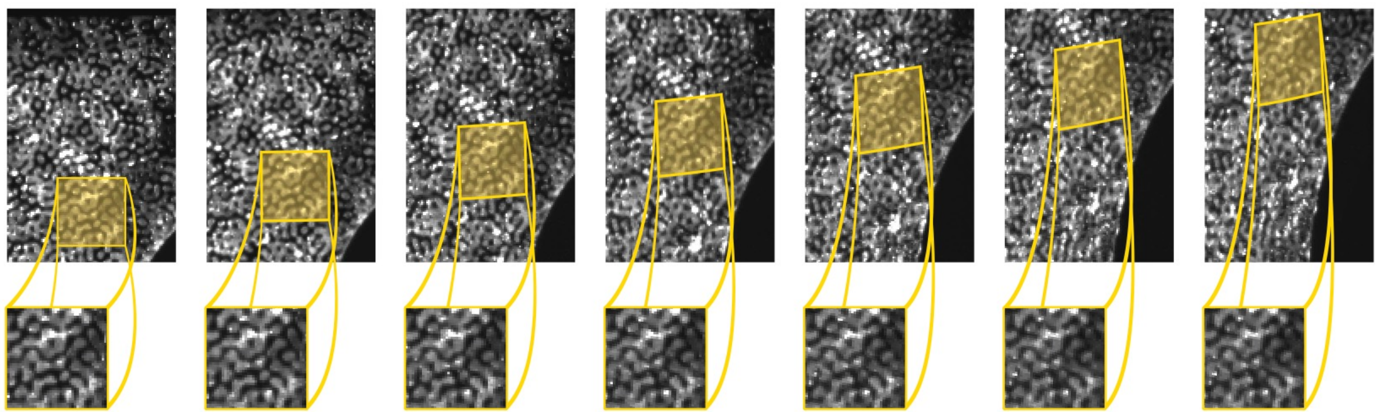


Image Requirements

for feasibility of digital image correlation

any image in digital format

- data must be converted to pixel array
- optical, electron, scanning probe, tomography…

predictable image of deformed object

- distinguishable from original
- uniform (or known) imaging geometry and illumination
- no new or missing features

Image Requirements

for quality of digital image correlation results

large pixel count, low noise

- maximize potential information content

small feature spacing

- allow small correlation window for best spatial resolution

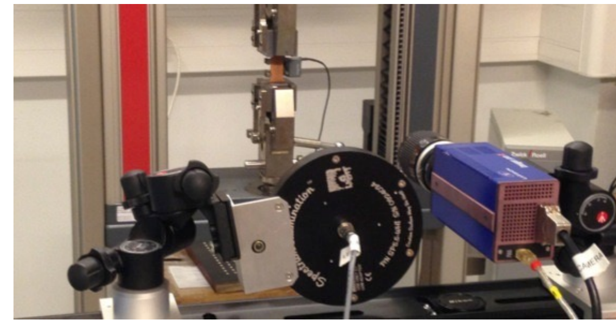
high contrast, irregular, well-resolved features

- give significant changes in correlation for best accuracy and precision

Digital Image Correlation
in brief

match images of deformed object to reference image of that object

- cross-correlation via FFT
  - *peak amplitude indicates how well it matches*
- Lucas-Kanade
  - *deform reference image with hypothetical displacement fields, then interpolate and calculate sum of squared differences*
- find the displacement field that gives the best match with observed image

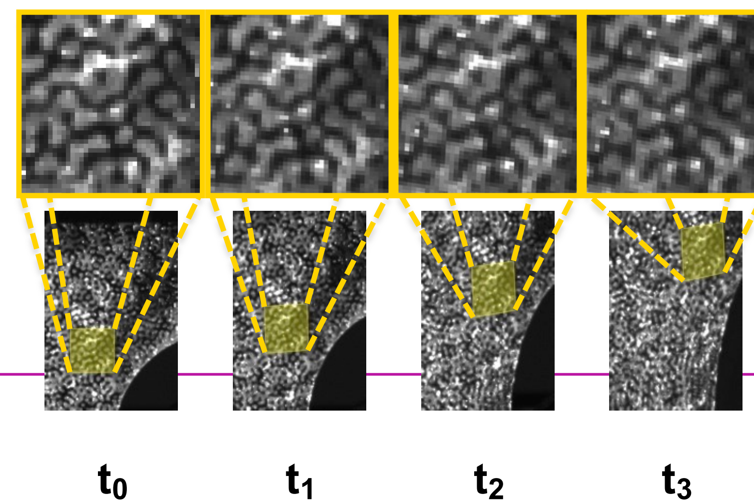


advantages:

- instantaneous non-contact optical full-field measurement
- leverage advances in digital cameras and computers
- sub-pixel resolution (due to peak fitting or interpolation)
- 3D displacements from stereo image pairs

issues:

- calibrating camera geometry and distortions
- contrast and feature spacing in image
- implicit assumptions in algorithm and in discretization method of fields
  - *e.g., cracks and shear bands replaced by unrealistically high but smooth localized strain*



Summary of Challenges and Opportunities

Applying optimised patterns

- at different length scales on different materials
- convenience versus accuracy and reliability

Theoretical analysis for patterns with sharp edges

- level sets for location of edges
- additional information

Full understanding and characterisation of measurement accuracy

- calibration and standardisation
- "experimodelment"

Closer integration with numerical simulation

- finite element shape functions to make results more comparable
- initial guesses for displacement field

Numerical methods for highly localised deformations

- physics-based regularisation
- calculation speed and numerical stability
- pattern changes with very large deformations

Industrial internet

- monitor real-world performance (e.g. vibrations)
- strategies for archiving video data to be used for a posteriori analysis

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Characterisation of Dynamic Strain Ageing Using Full-Field Measurements

BUSINESS
FINLAND
MaNuMiES project

Tuomas Pihlajamäki’s M.Sc. Thesis

<https://tinyurl.com/y76tfphg>

<https://aaltodoc.aalto.fi/handle/123456789/24749>

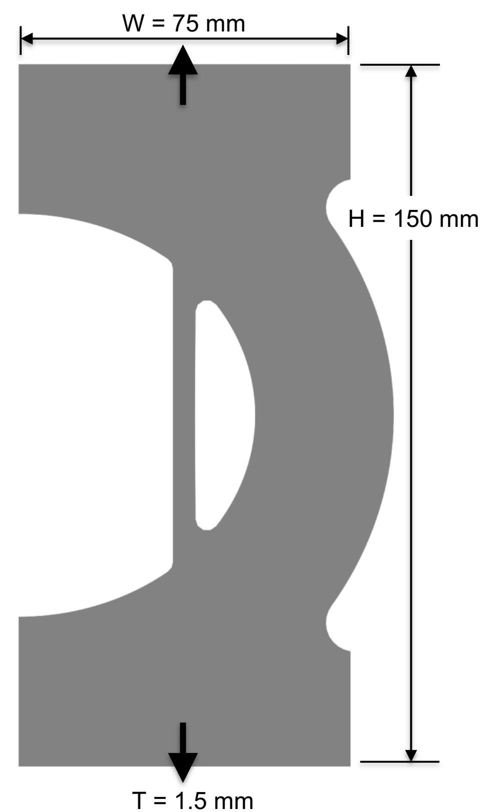
Complex specimen geometry *Elizabeth Jones (Sandia)*

Complex specimen geometry is available when using full-field diagnostics.

Sandia National Laboratories

Design Criteria

- Objective:
  - Maximize strain/stress heterogeneity
  - Maximize range of strain rates
- Constraints:
  - Minimize large gradients near sample edges
  - Uniaxial loading
  - Planar sample

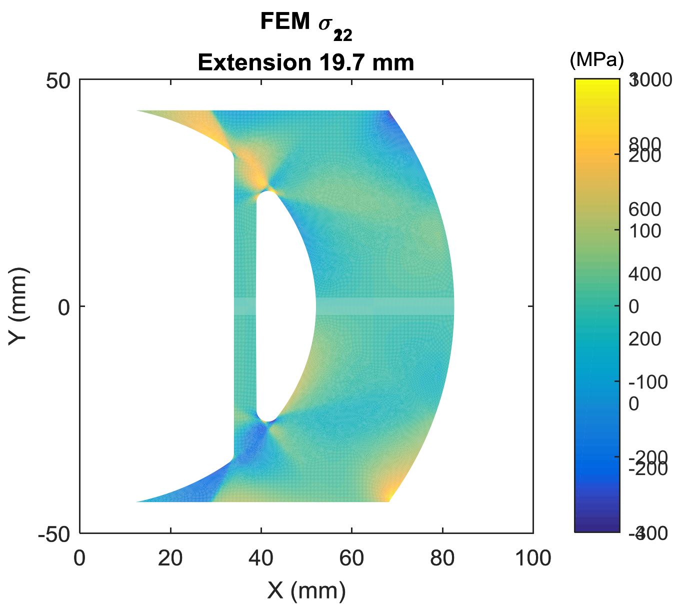


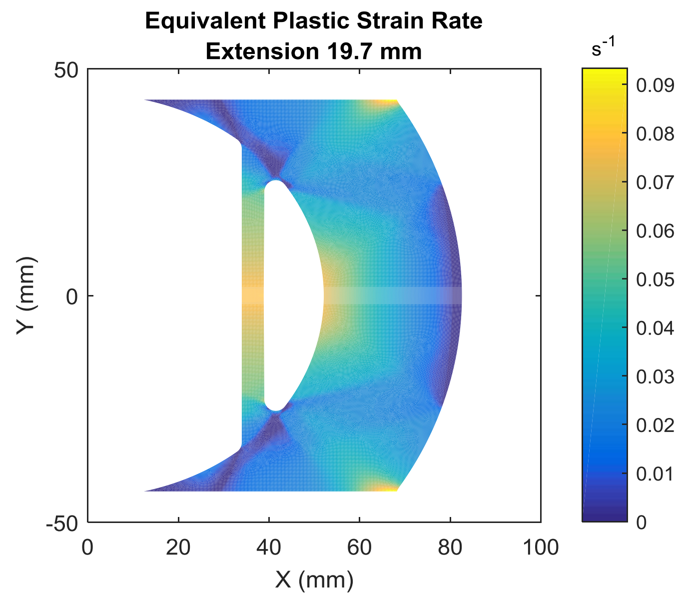
Complex specimen geometry *Elizabeth Jones (Sandia)*

Sandia National Laboratories

Complex specimen geometry induces stress and strain rate **heterogeneity** in sample.

Predicted Results from FEM Simulation





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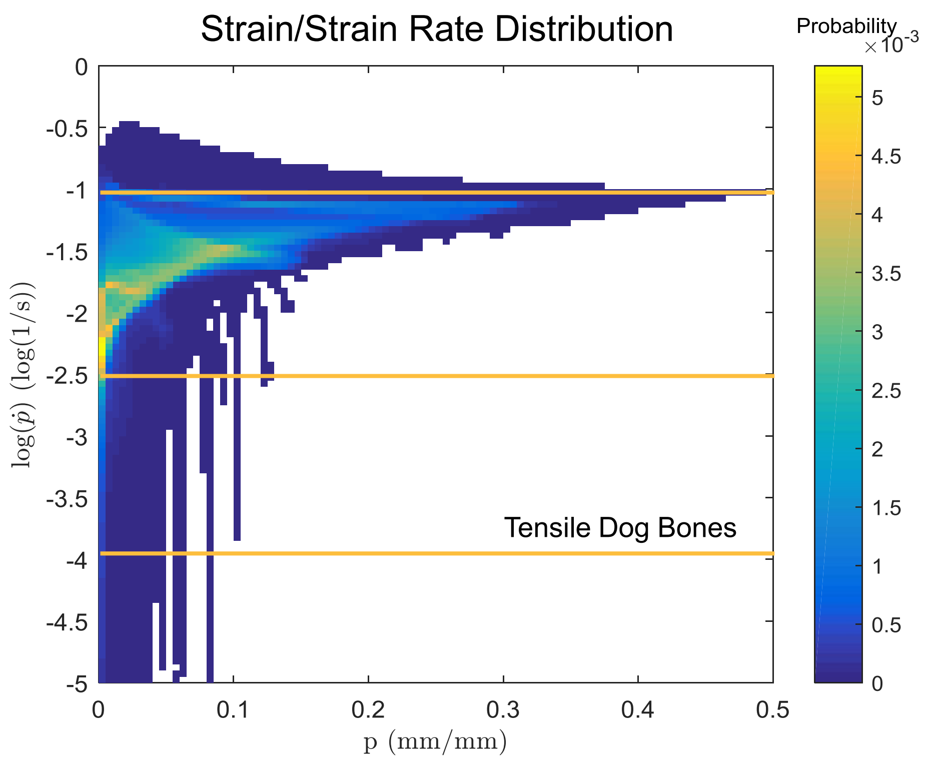
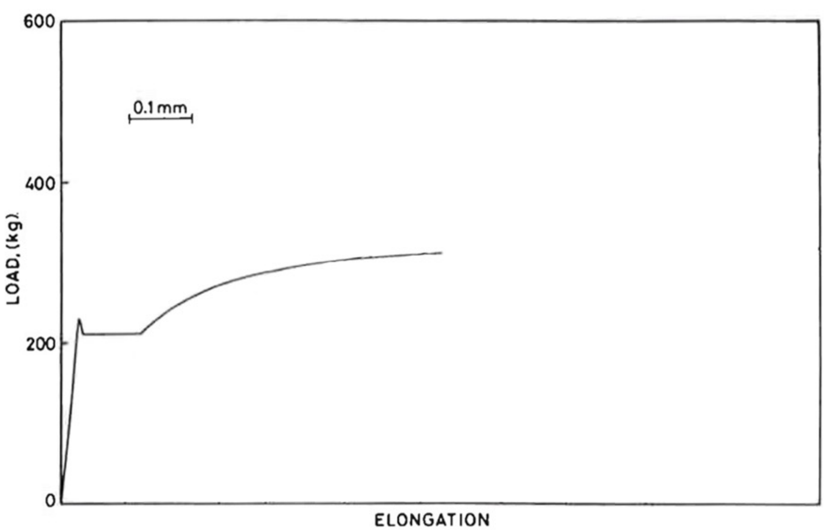
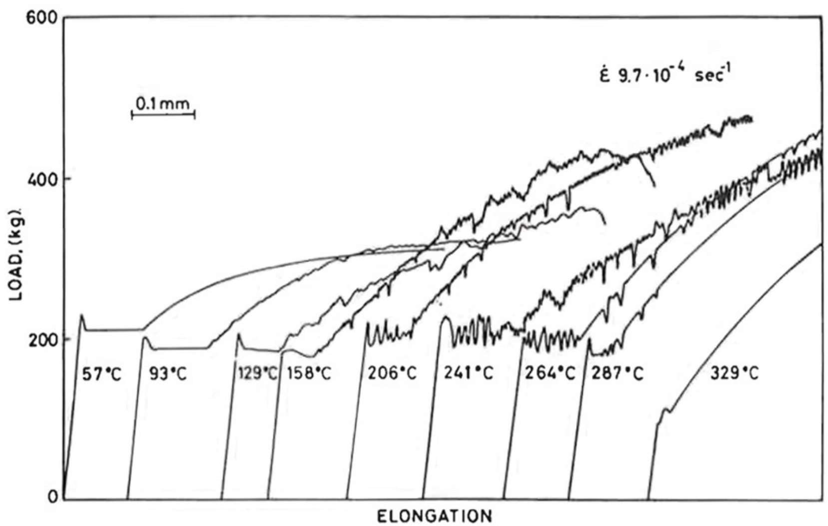


Yield point
in plastic deformation of (some) metals



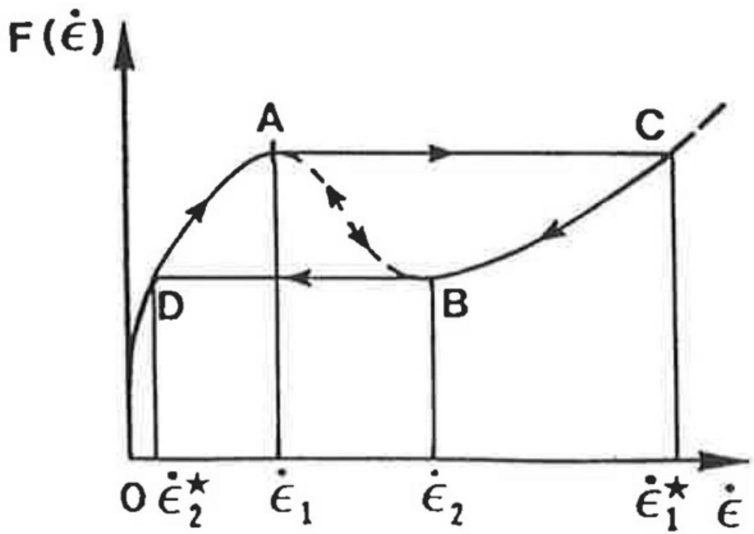
- Interaction of solute atoms with the dislocations responsible for plastic flow
- Lower flow stress after initial yielding
- Yield point reappears after ageing

Dynamic strain ageing
in plastic deformation of (some) metals



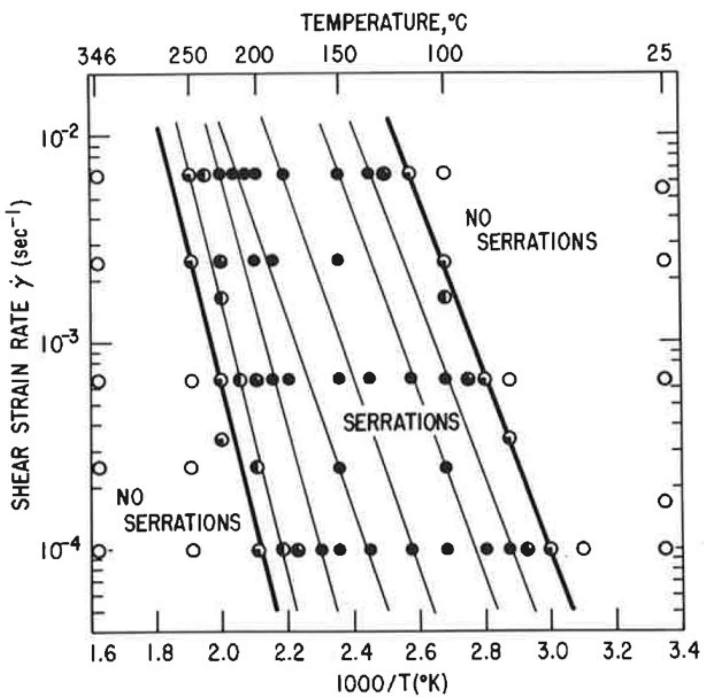
- Microstructural effects happen on time scale of deformation
- Increased strain hardening
- Lüders bands
- Serrated Flow (Portevin – Le Chatelier effect)

Strain rate sensitivity
related to dynamic strain ageing



- Negative strain rate sensitivity causes localization of flow
- Different effects contribute to instantaneous and steady-state strain rate sensitivity

Strain rate dependence
of serrated flow in dynamic strain ageing



- Arrhenius activation energy
  - *Consistent with activation energy for diffusion of interstitial solutes*
  - *Both for onset and end of serrations*
- Spatial and temporal variations of local strain rate

Full-field measurements
for characterizing dynamic strain ageing

Resolve spatial and temporal variations in strain rate

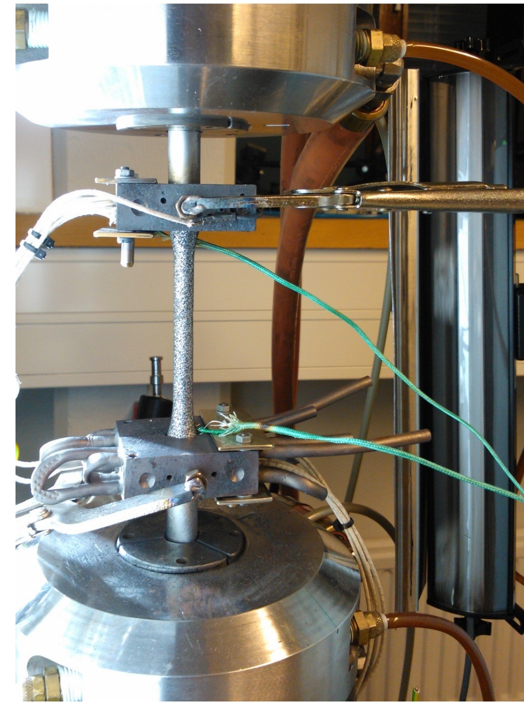
Allow to introduce additional variations in relevant parameters

Sample more of the relevant parameter space

Reduce the number of specimens to be tested

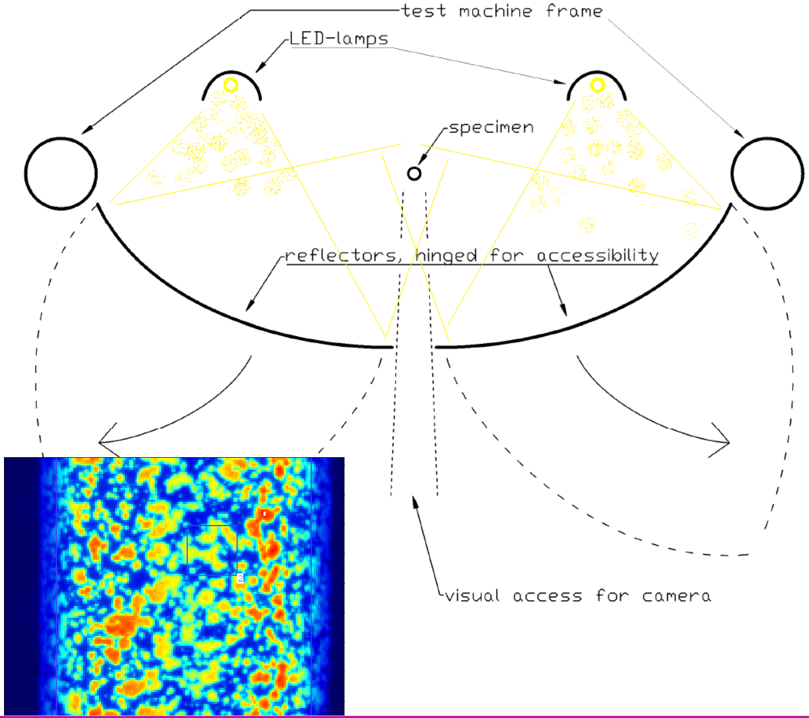
Mechanical setup
for mechanical testing with a temperature gradient

- Servo-hydraulic universal testing machine
  - *Cross-head displacement control*
  - *Water-cooled grips*
- Cylindrical dog-bone specimens
  - *⌀ 10 mm, 60 mm gauge length*
- Digital Image Correlation for strain profile measurement
  - *Spray-painted pattern with silicone-based high-temperature paint*



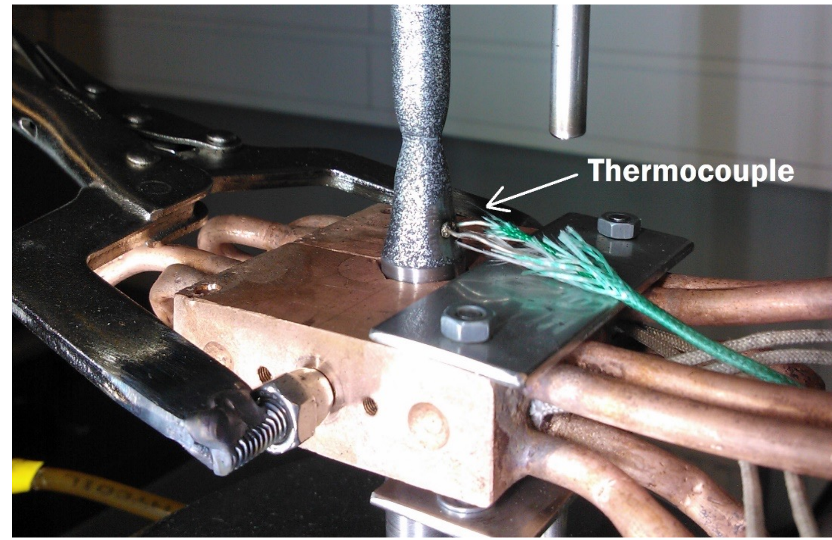
Digital Image Correlation
for mechanical testing with a temperature gradient

- Spray-painted pattern
  - *Silicone-based high-temperature paint*
- Indirect illumination
  - *Avoiding specular reflections*
- LaVision Imager X2 cameras
  - *100 mm telephoto lenses*
- DaVis 8.2 software
  - *25 × 25 px subsets (~1.7 mm)*



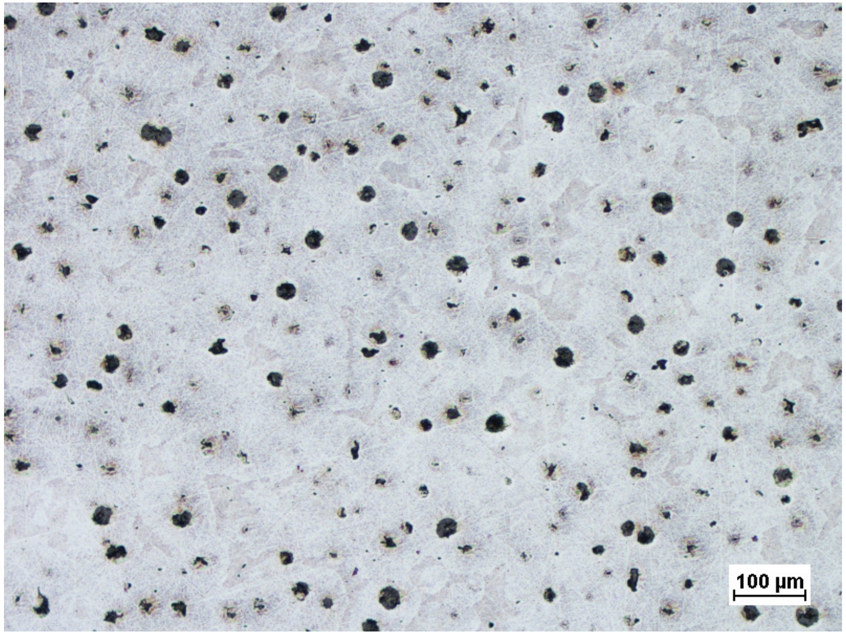
Temperature control
for mechanical testing with a temperature gradient

- Cartridge heaters in copper blocks
  - *Clamped using modified locking pliers*
- Infrared imaging for temperature profile
  - *Painted matte black, measured emissivity is 0.7*
- Thermocouples at each end
  - *spot-welded to the specimen just outside the gauge section*
- Independent PID controllers for each heater block
- Additional cooling unnecessary



Material
for characterizing dynamic strain ageing

- Ductile cast iron
  - *⌀ 70 mm GGG50 / GJS500-7 bar*
  - *Ferritic-pearlitic microstructure as-received*
  - *Sawn and turned to ⌀ 20 mm rods*
- Heat-treated to obtain fully ferritic microstructure
  - *900 ° C and 2 hours at 700 ° C*
  - *Final machining and grinding to ⌀ 15 mm grips and ⌀ 10 mm gauge section*

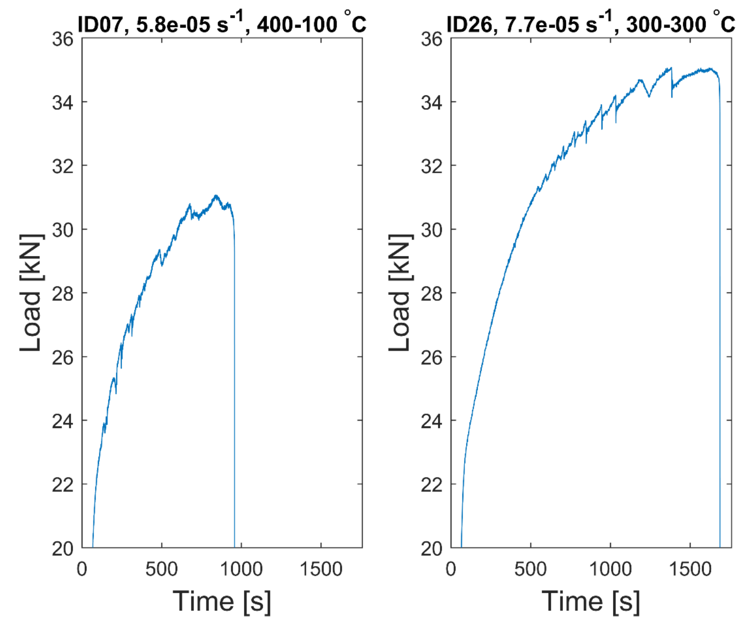


Results

comparing temperature gradient test to uniform temperature test

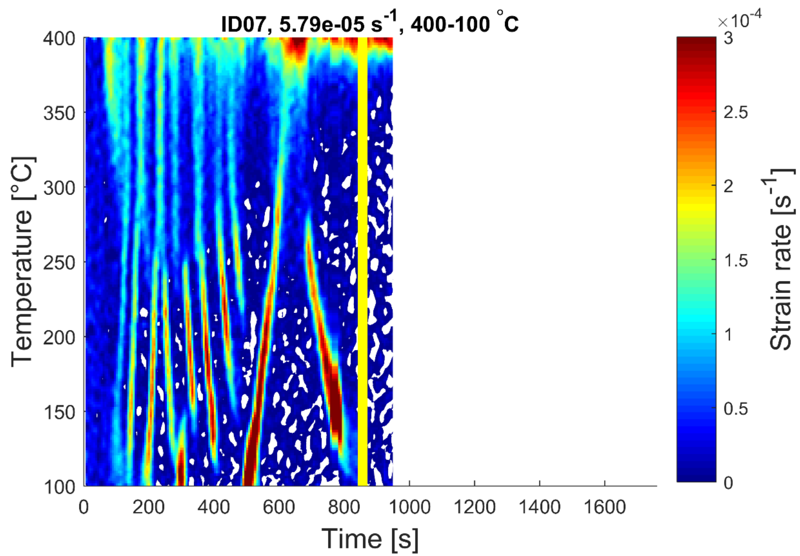
Global load-displacement plot

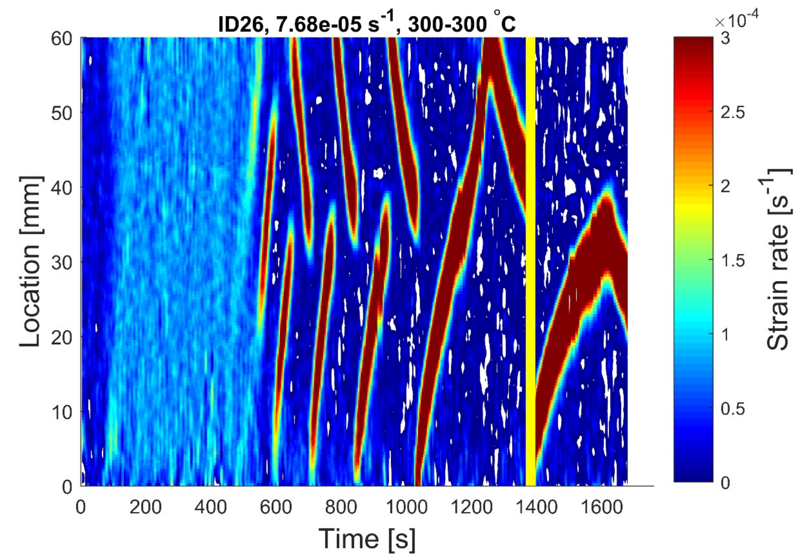
- Earlier onset of serrations
- Shorter time before rupture



Results

comparing spatial and temporal variations in strain rate



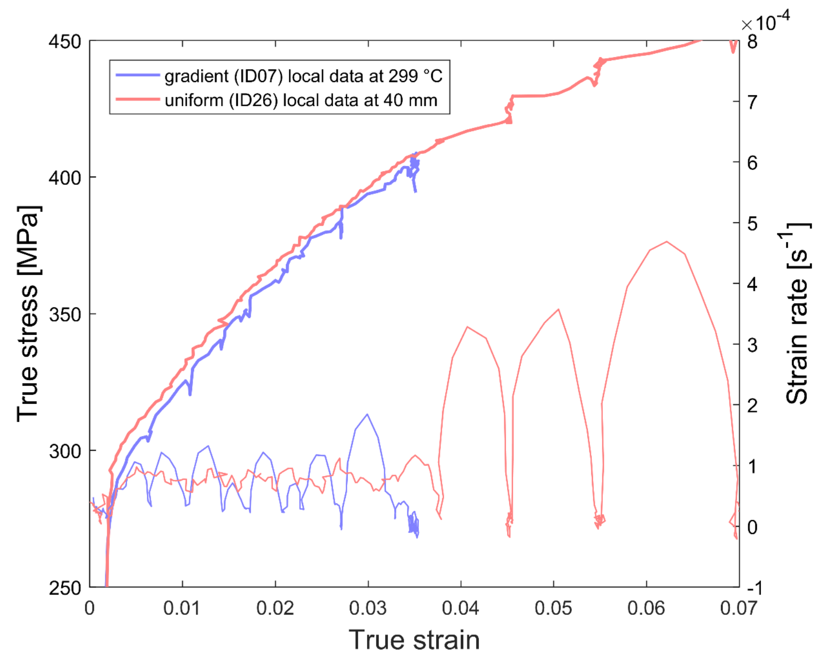


Results

comparing temperature gradient test to uniform temperature test

Local stress-strain plot

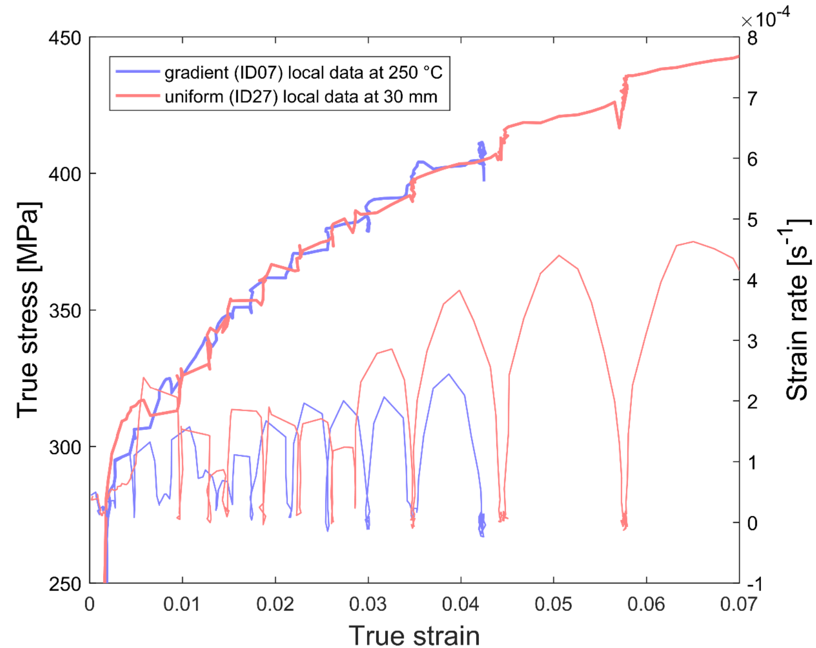
- Earlier onset of serrations
- Shorter time before rupture
  - *Specimen ruptured elsewhere even before onset of serrations at this uniform temperature*



Results
comparing temperature gradient test to uniform temperature test

Local stress-strain plot

- Behaviour is more similar at some other temperatures

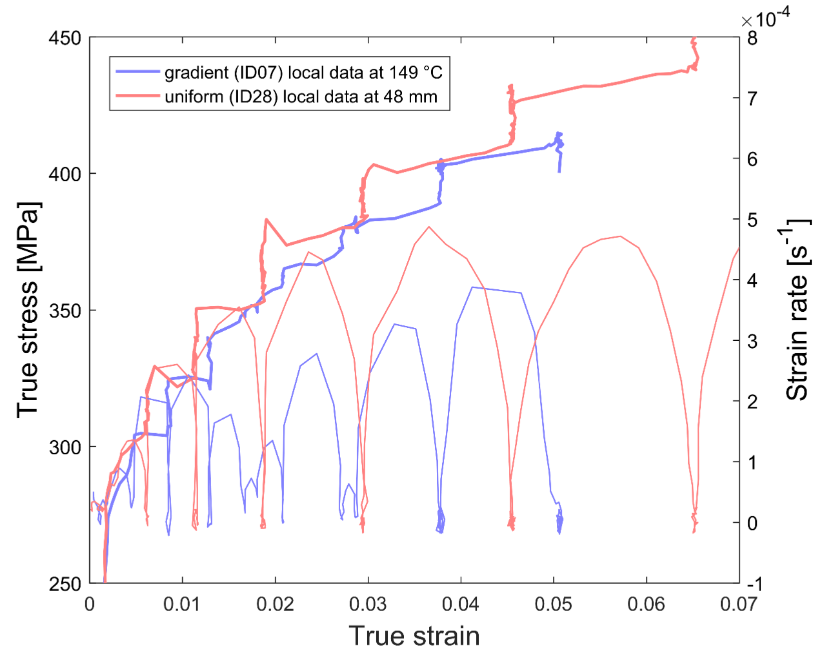


Results

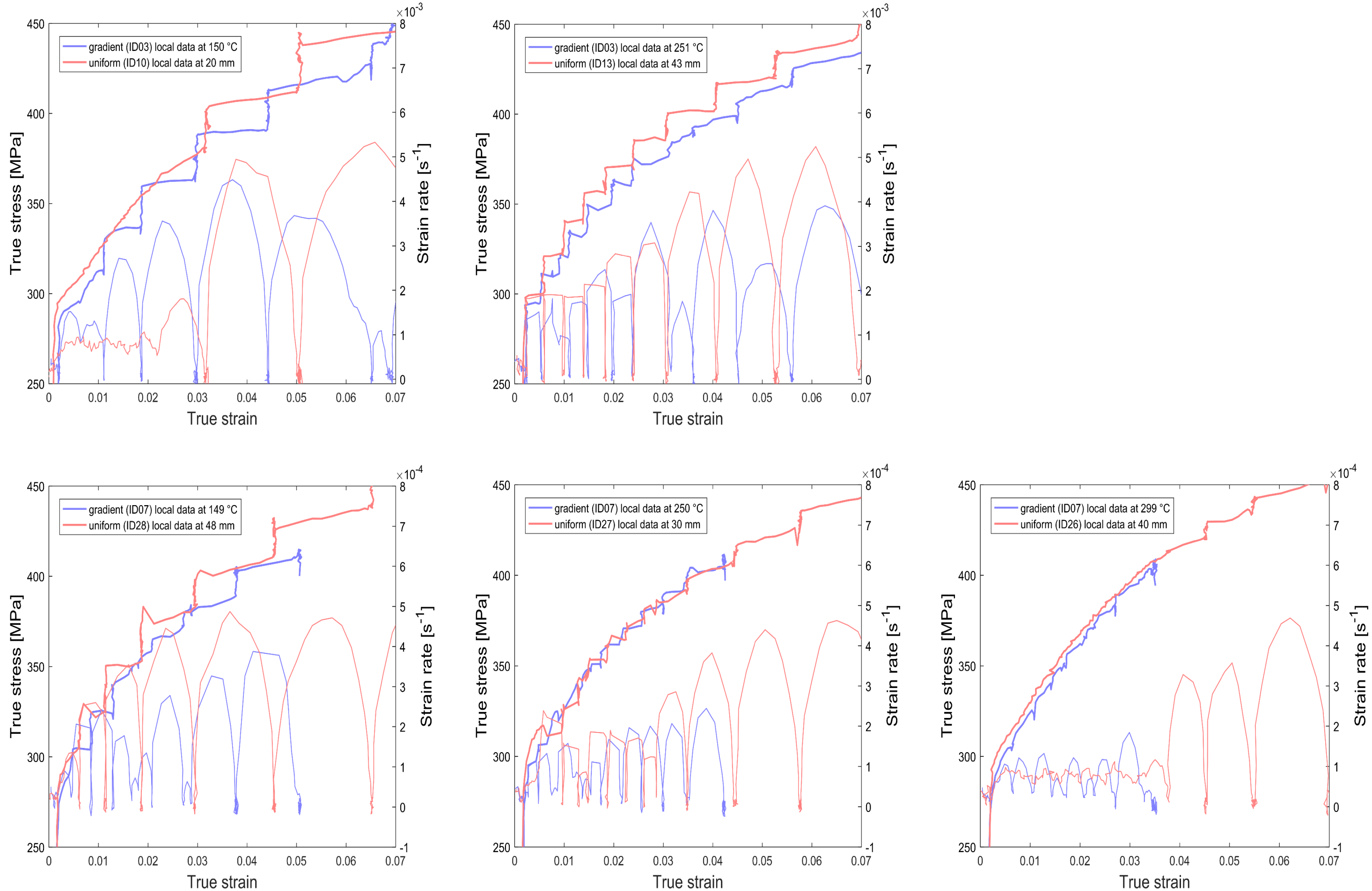
comparing temperature gradient test to uniform temperature test

Local stress-strain plot

- Behaviour is more similar at some other temperatures

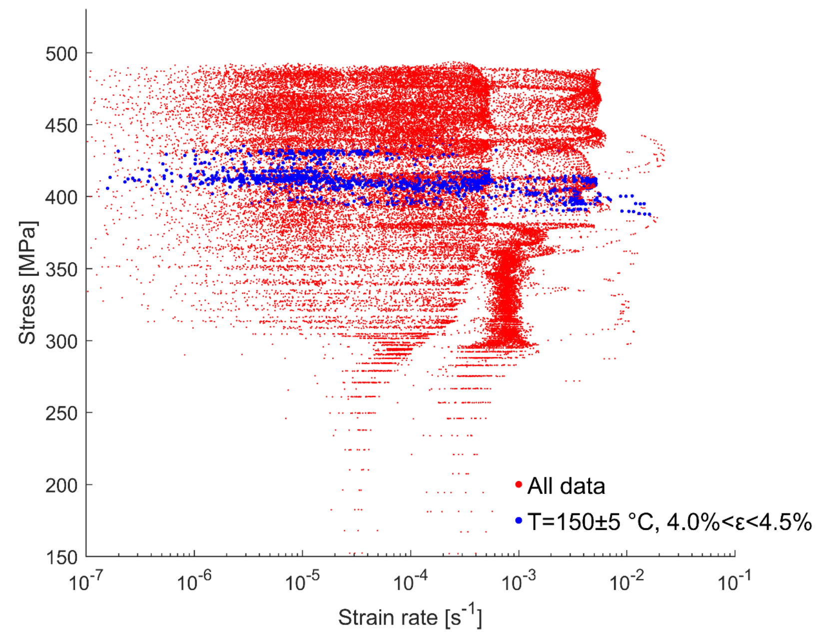


Higher strain rate



It works!

- More data from each experiment
- Sampling more of the parameter space
- More coupling with rest of parameter space
  - *Loading history seen by each material segment depends on what happens in rest of sample*
- Few limitations on testing
  - *Tension, compression, fatigue…*



Open questions

- Do the gradients affect the material behaviour?
- Is it a problem that deformation bands propagate into material at a different temperature from where they initiated?
- How much spatial and temporal resolution is needed?
- Inverse problem of identifying material models from this
- Modulating specimen diameter to avoid the necking and fracture at one temperature to prematurely end experiment for other temperatures.
- Link with modelling

Tuomas Pihlajamäki's M.Sc. Thesis

<https://tinyurl.com/y76tfphg>

<https://aaltodoc.aalto.fi/handle/123456789/24749>

Aalto University
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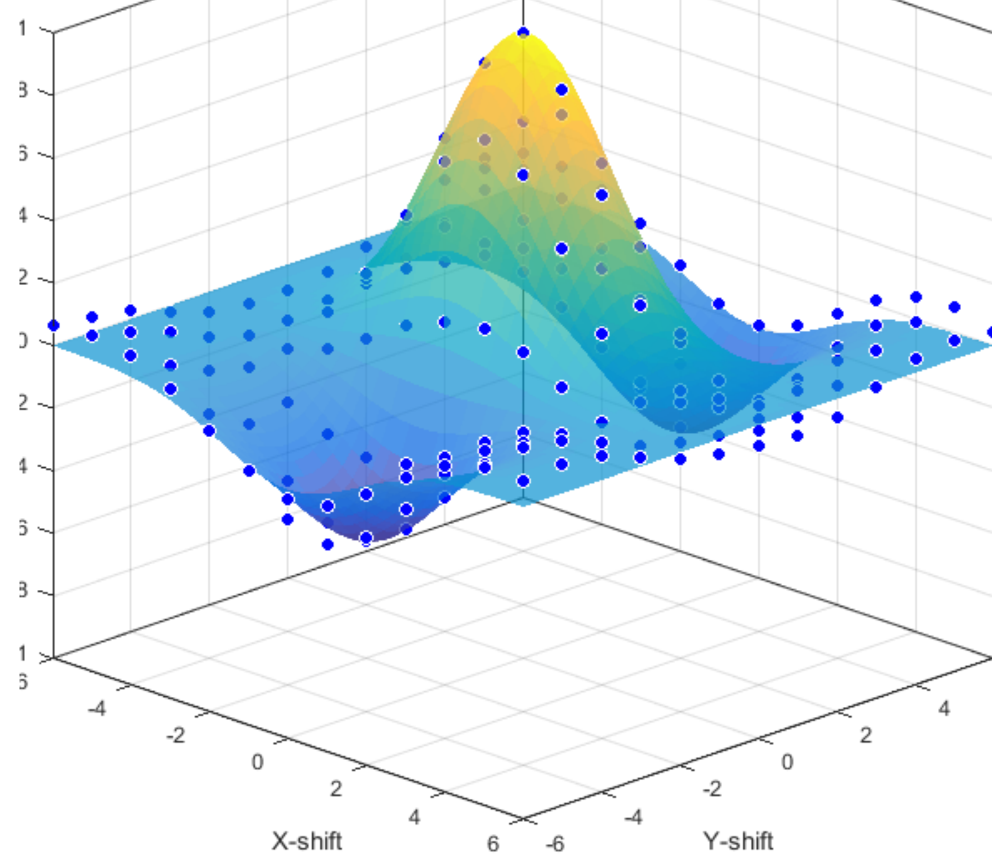
INTERNATIONAL DIGITAL IMAGE CORRELATION SOCIETY

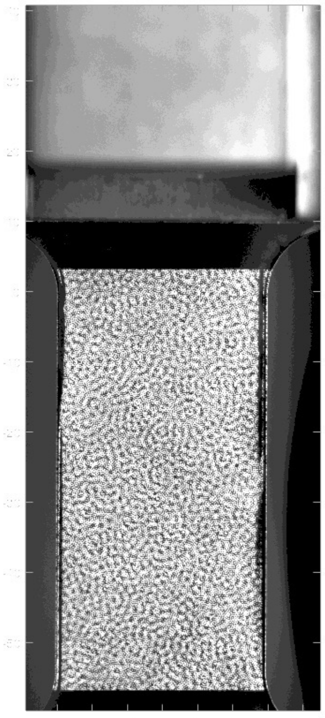
Quantifying the effectiveness of different sample preparation methods and DIC parameters for characterization of plastic strain localization in copper

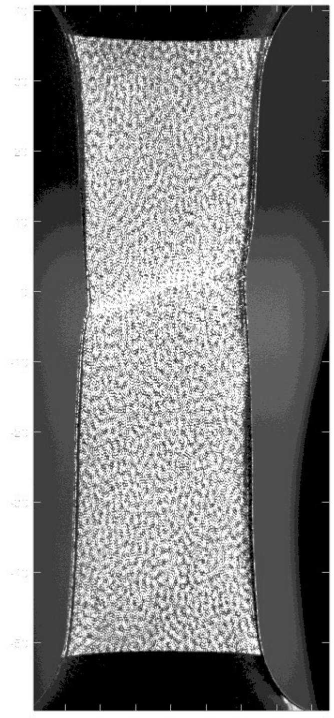
Antti Forsström, Sven Bossuyt, Gianmario Scotti, Hannu Hänninen
November 8, 2017
Annual iDICs conference, Barcelona

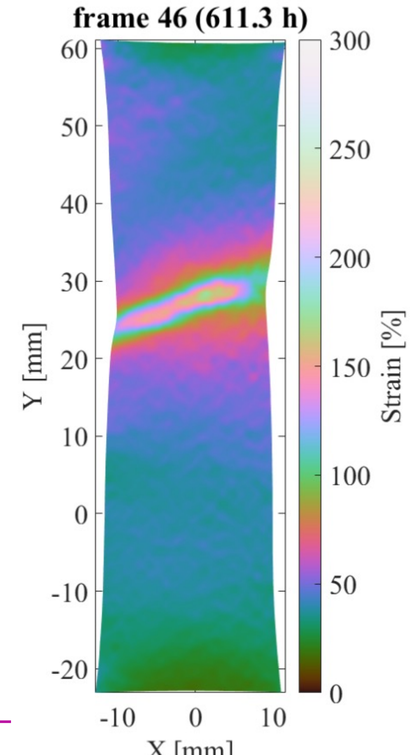


Antti Forsström, Sven Bossuyt, Gianmario Scotti, and Hannu Hänninen:
Quantifying the effectiveness of patterning, test conditions, and DIC parameters for characterization of plastic strain localization
Exp. Mech. **60**, 3–12 (2020) DOI: 10.1007/s11340-019-00510-6

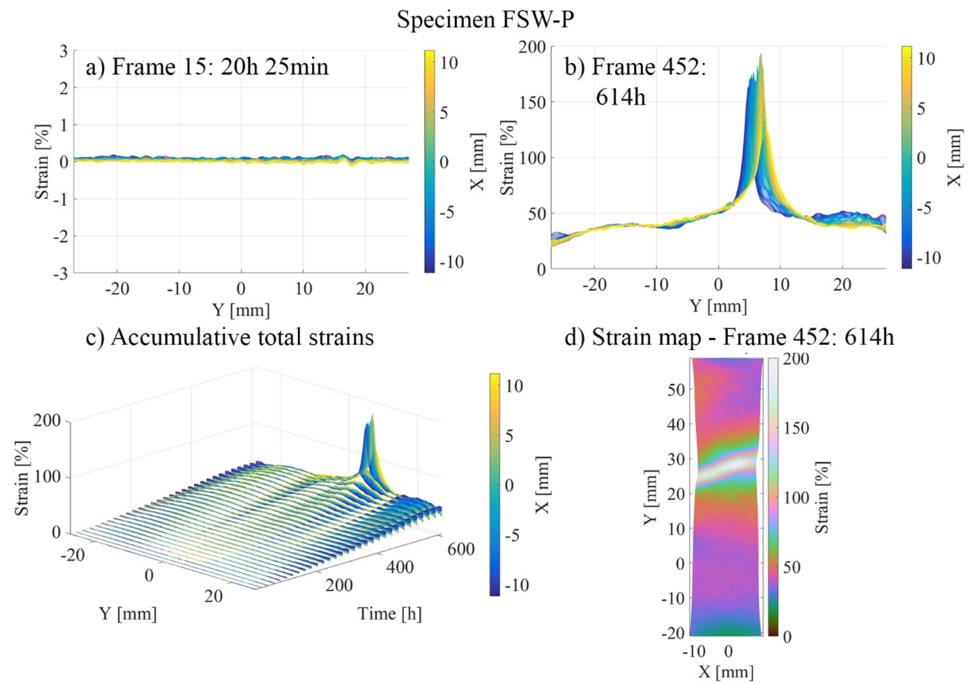
DIC
to visualize and quantify strain localization



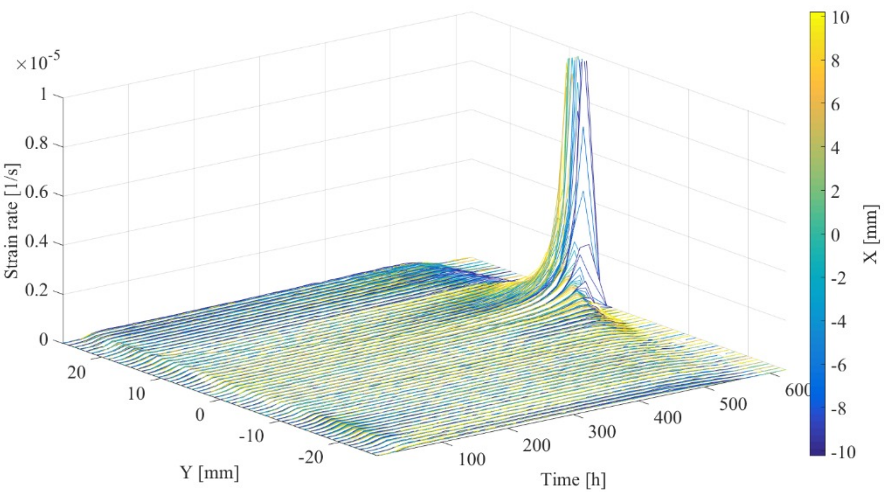


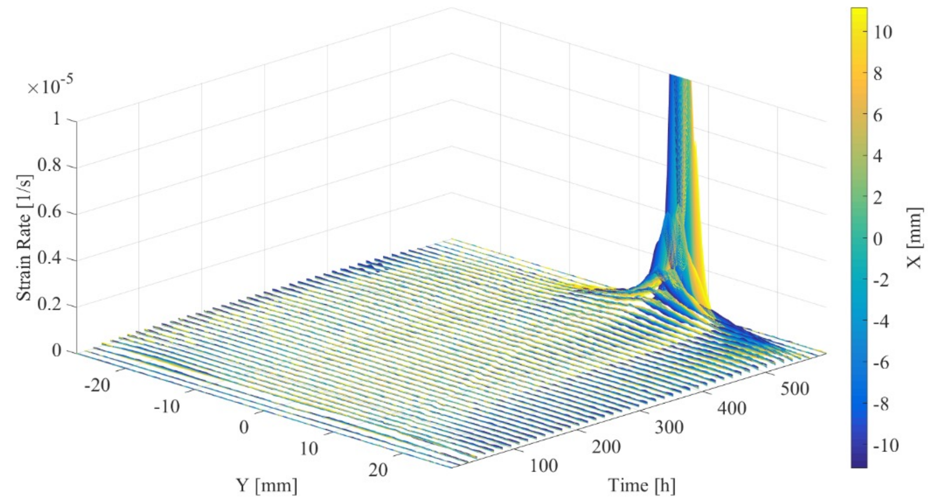


DIC
to visualize and quantify strain localization

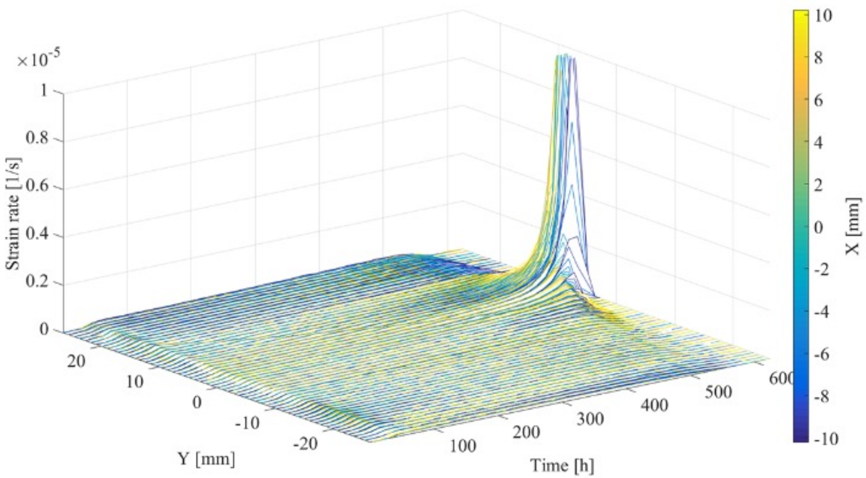


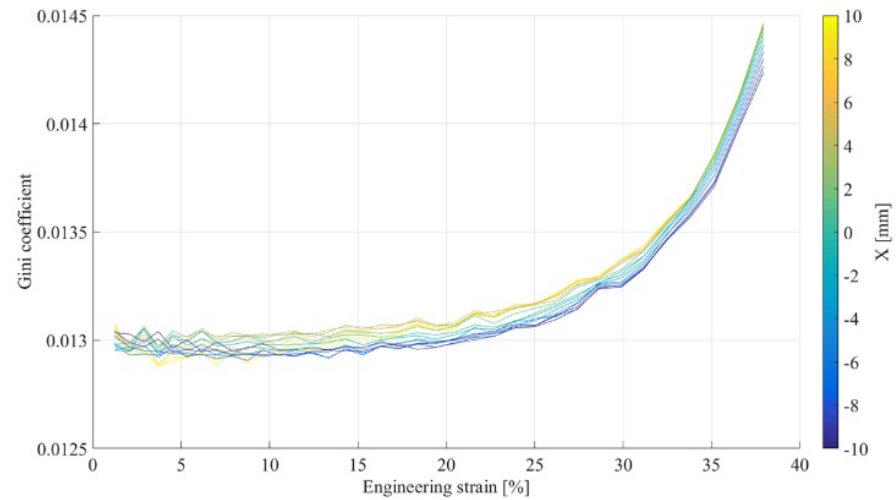
DIC results
Strain rate along the gauge length plotted in time



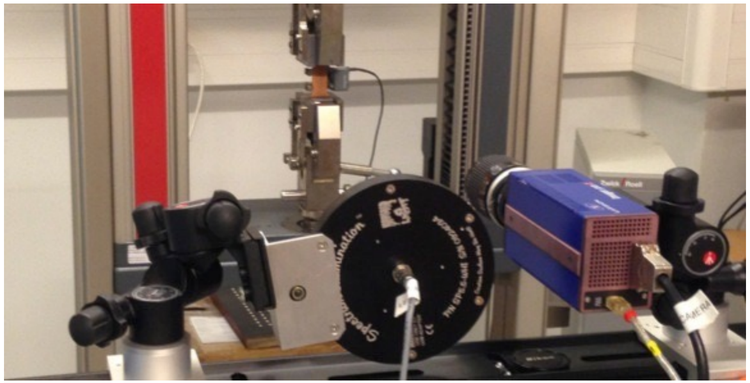


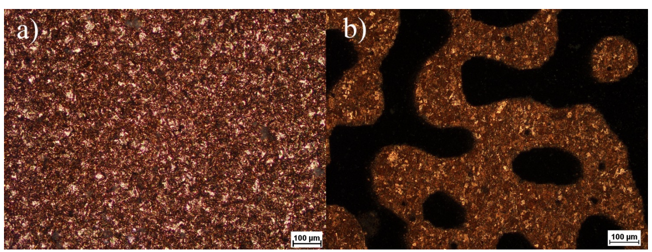
DIC results
Strain localization presented with Gini coefficient

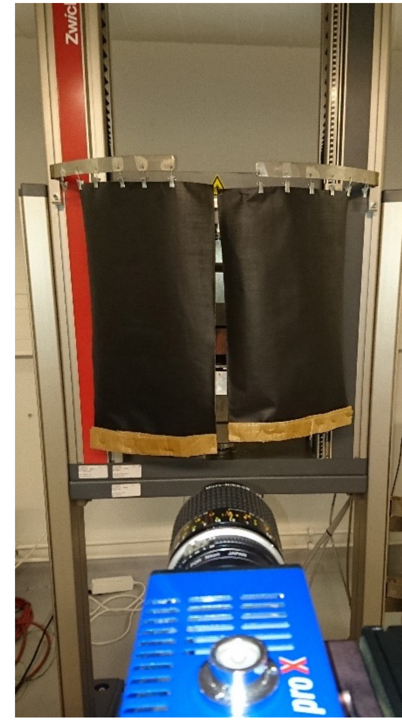




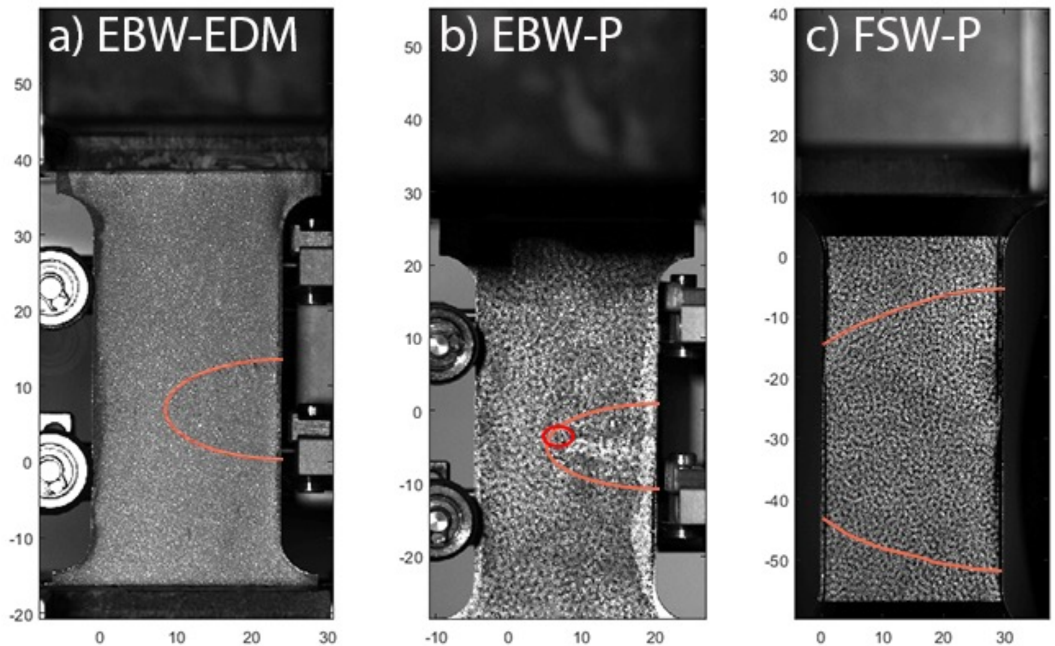
Tensile testing setup



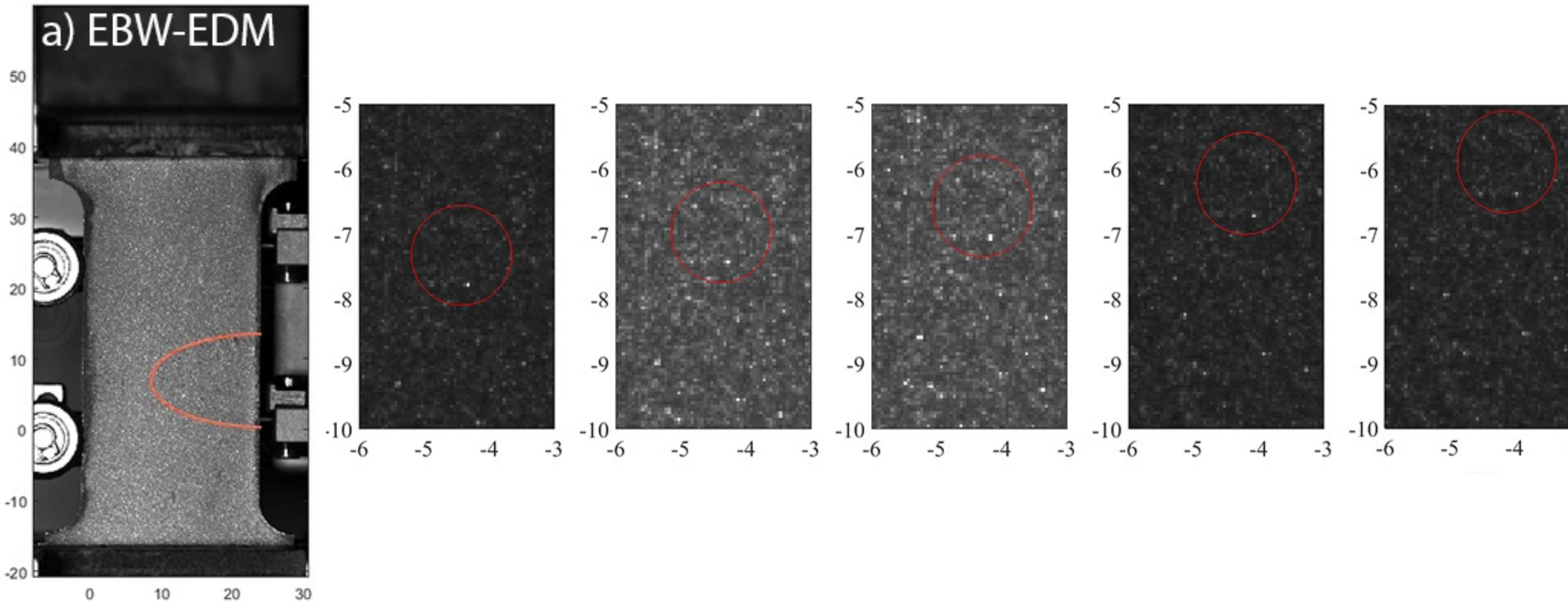




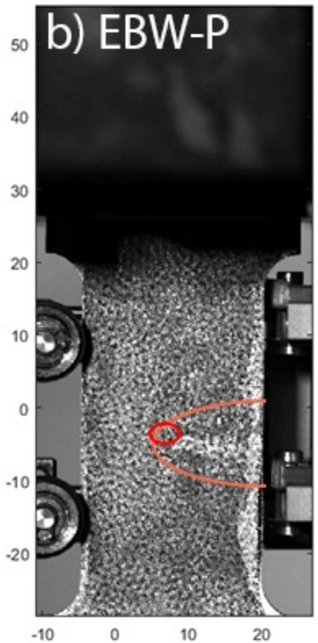
Three specimen types

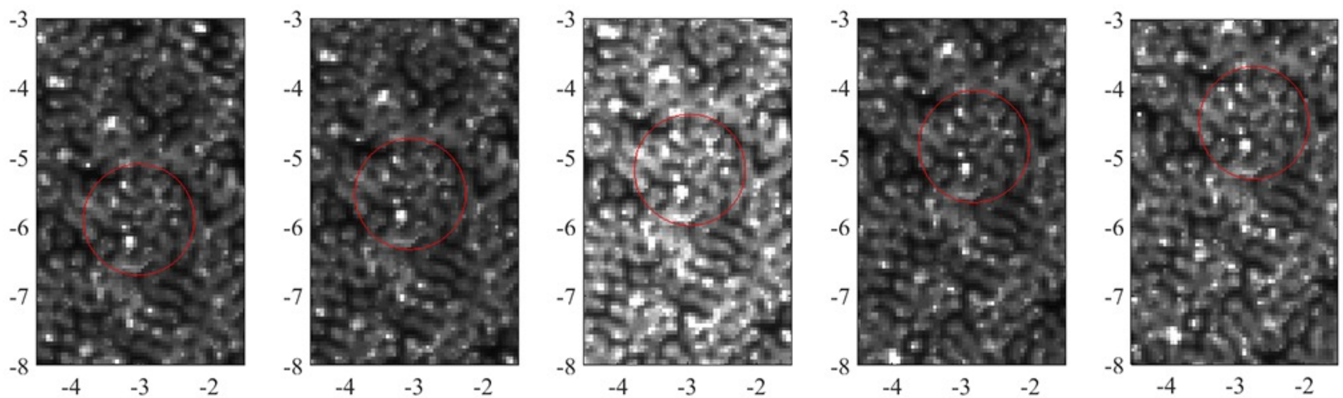


Three specimen types
use surface roughness

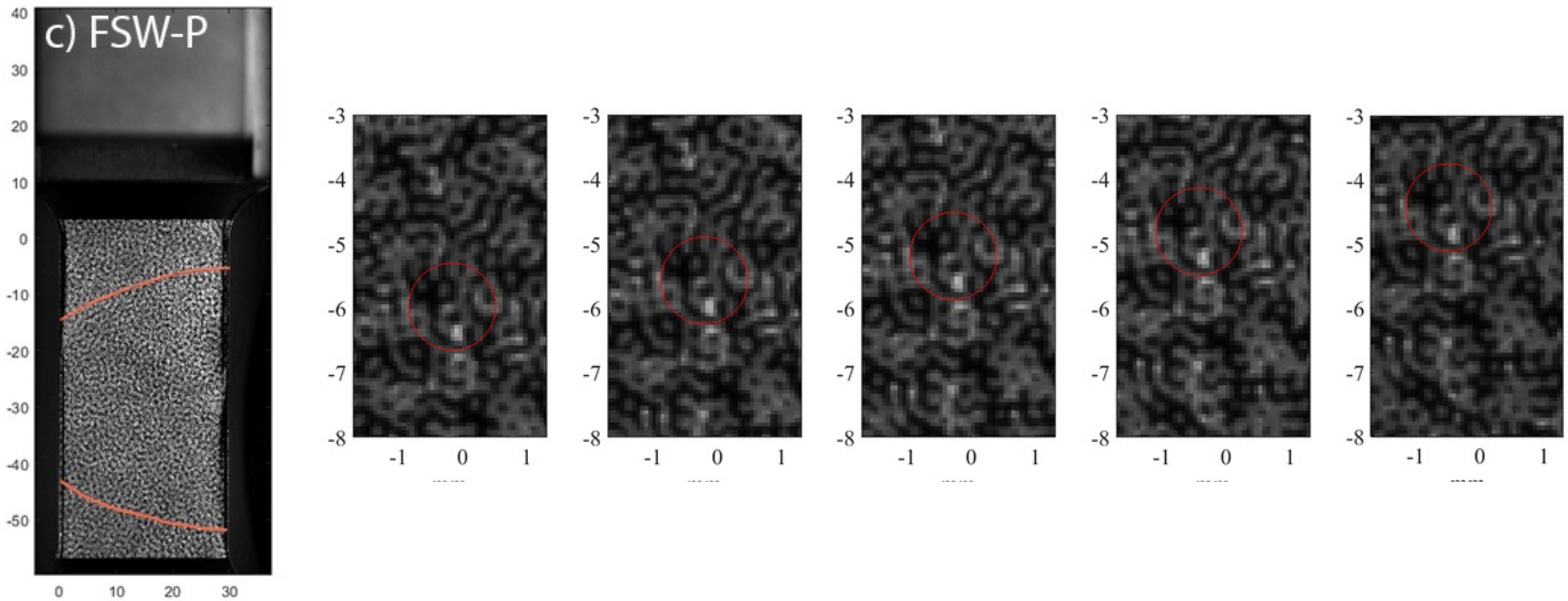


Three specimen types
black copper oxide patterned by photolithography

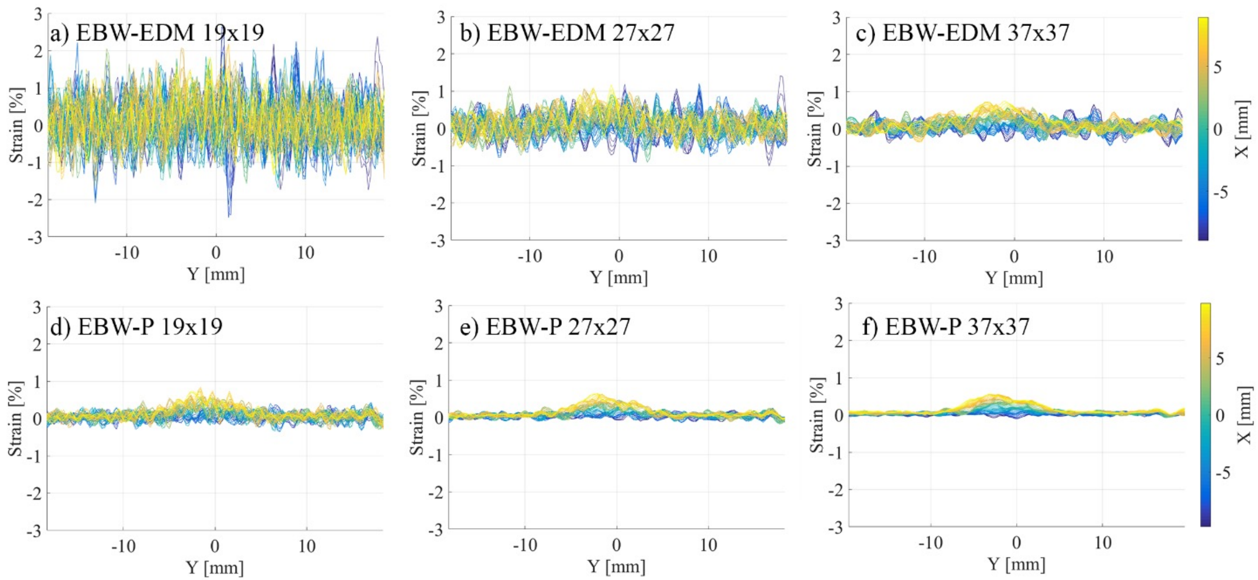




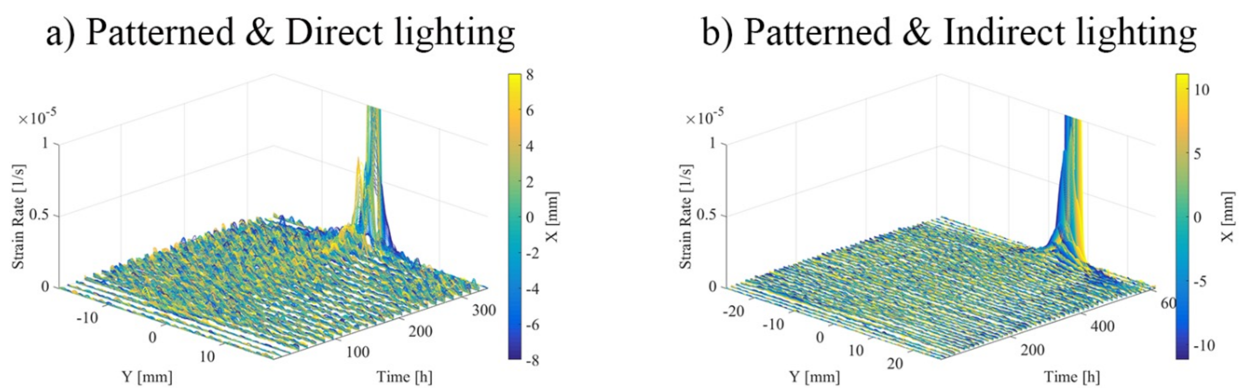
Three specimen types
pattern with indirect illumination



Effect of pattern on displacement noise



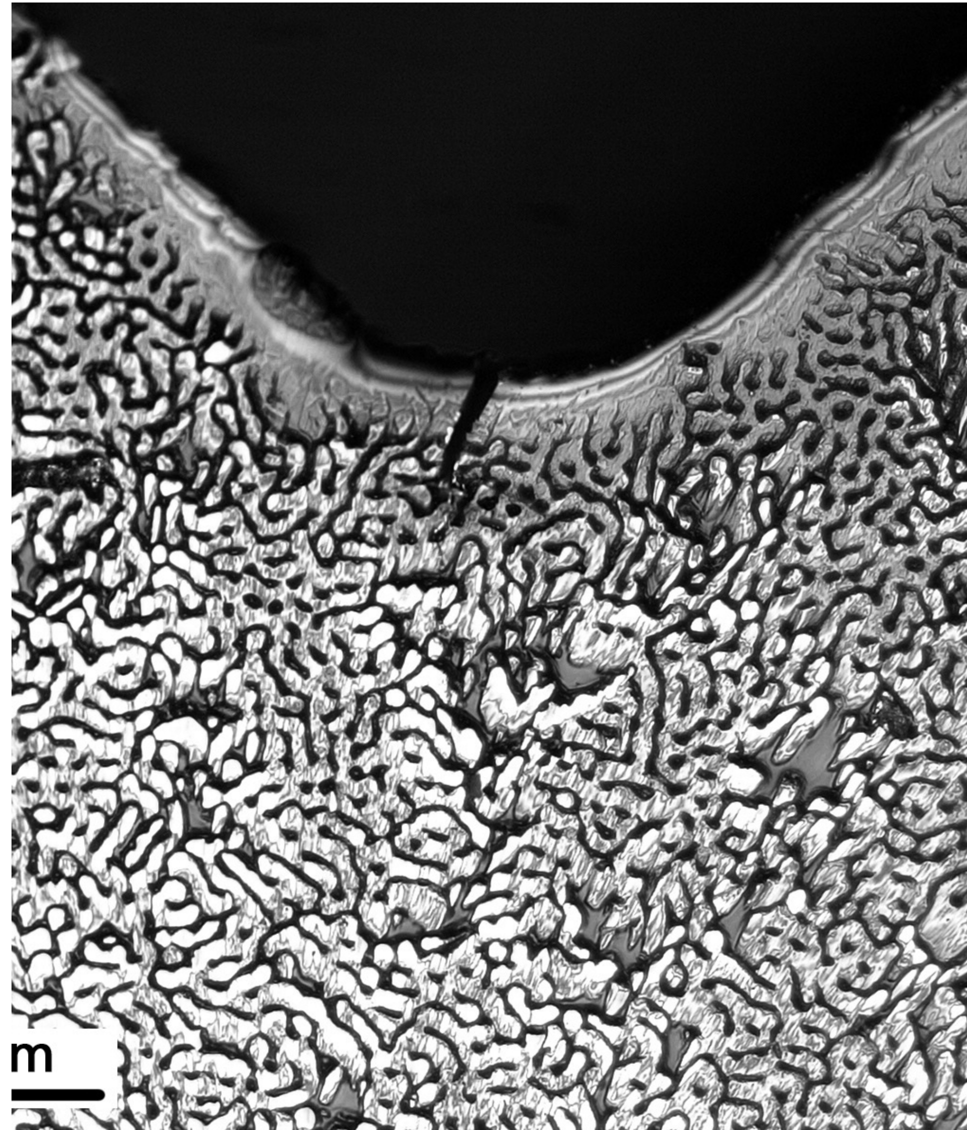
Effect of lighting on displacement noise

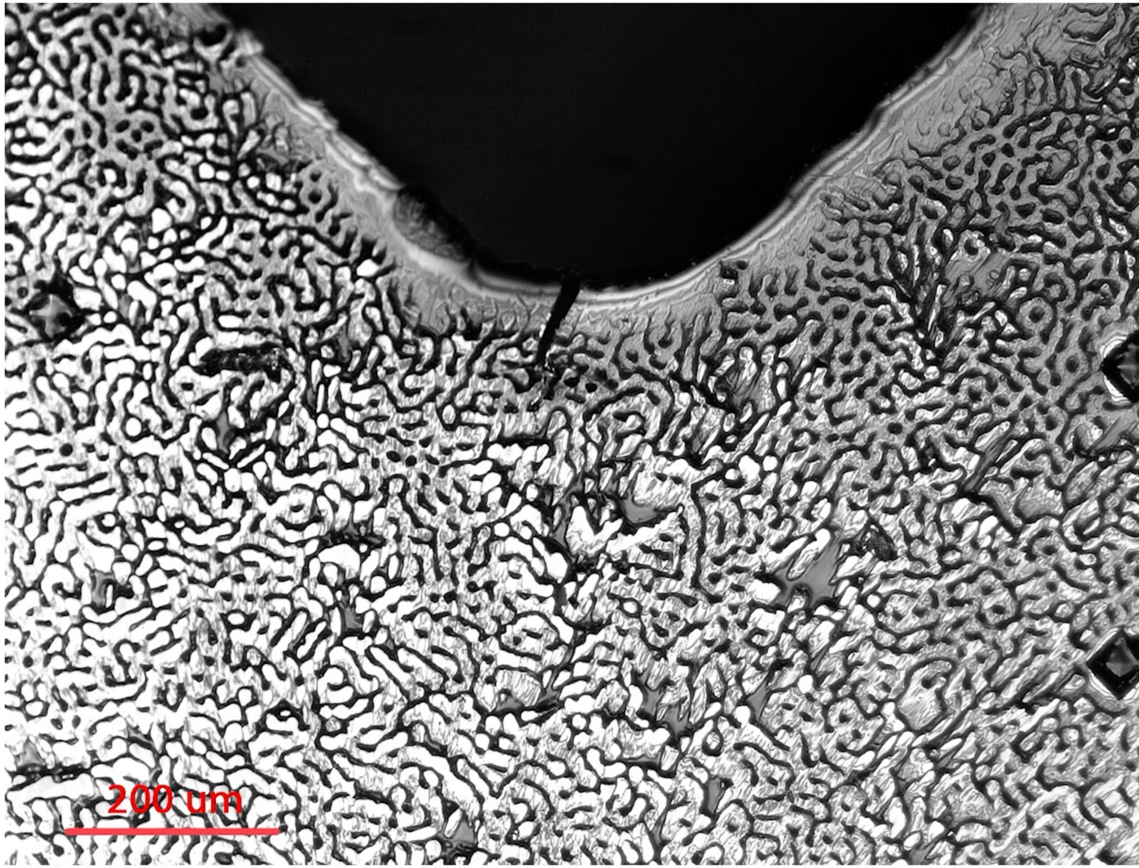


Aalto University
School of Engineering

Full-field Strain Measurements for Microstructurally Small Fatigue Crack Propagation using Digital Image Correlation Method

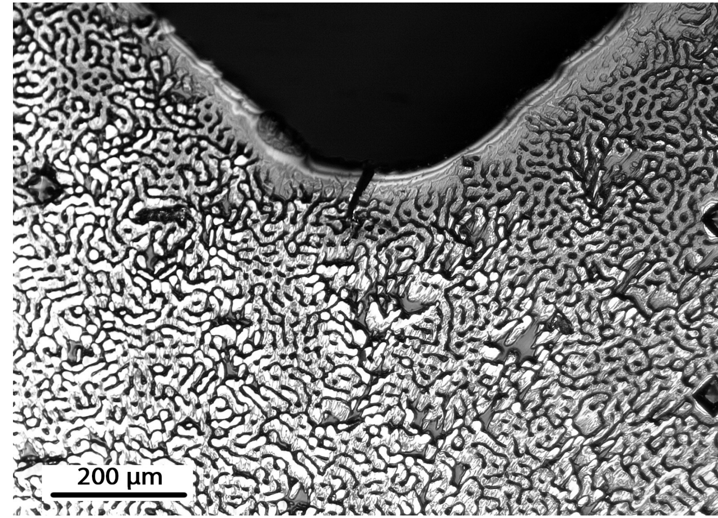
Evgeny Malitckii, Heikki Remes, Pauli Lehto, Sven Bossuyt





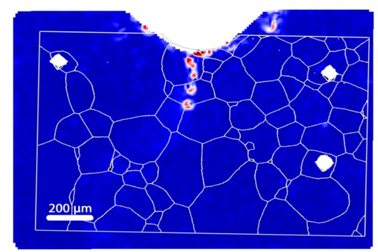
Malitckii, E., Remes, H., Lehto, P., Bossuyt, S.: *J. Vis. Exp.* (), e59134, doi:10.3791/59134 (2018)

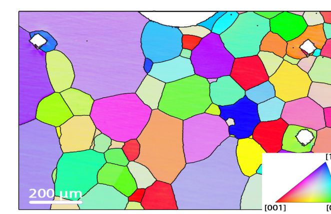
Micro-mechanics of fatigue in polycrystalline metal



Full-field measurements

- Resolve displacement, strain, and strain rate inside grains
- Electron BackScatter Diffraction characterizes grain structure
- Non-contact measurement during fatigue test at typical rates (few Hz)





Limitations

- Surface measurements
- Optical imaging ≳ 1µm
- Sub-µm resolution much slower

E. Malitckii, H. Remes, & al. *Acta Mater.*, 144, p. 51-59, 2018. DOI 10.1016/j.actamat.2017.10.038

Conclusions

Materials Testing and Characterization in the Age of Big Data

Challenges

- Paradigm change comes with uncertainty
  - *Methods are still evolving*
  - *Standardization and industry acceptance lags state of the art*
- Multidisciplinary expertise needed

Opportunities

- Progress in computer hardware and machine learning algorithms
- Measurements closer to the actual use cases
  - *More reliable materials information enables more efficient material use*
- True multiphysics characterisation
- New discoveries to be made!