FULL-FIELD MEASUREMENTS FOR PROPERTY AND PROCESS CHARACTERISATION (IN MECHANICS)

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50 000 grains of sand imaged by x-ray μ-tomography…

(14 μm voxel size, sample dimensions: height = 22 mm, diameter = 11 mm, ID15A ESRF)

This is very pretty… but so what!

Challenge: to extract pertinent, quantified information to elucidate mechanics
Objectives

• Presentation of the concept of full-field measurements
• Demonstration of how useful information can be extracted from “pretty images”

• Examples:
  • Analysis of localised deformation in sandstone
  • Quantification of non-homogeneous fluid flow in a deformed sandstone
  • Analysis of localised deformation, structure and fabric evolution and grain scale kinematics in sand
A quick experiment to start....
A quick experiment to start….
A quick experiment to start….

We can measure some things at the boundary
  Applied load (heating)
  Sound of water boiling
  Observation of steam

But we do not know what the processes are going on inside - how does water become coffee?
  This might be inferred from the external observations or by destructive characterisation (taking the machine apart) but not measured directly

Would like measurement approaches permitting a full field view of the processes, i.e., inside the device and, even better, during operation…
Time for coffee…
Full-field measurements - why?

• Materials are **not homogeneous** - heterogeneity (at different scales) is the rule rather than the exception
• Laboratory test **boundary conditions** are never perfect
• Processes are generally not homogeneous, e.g., localisation of deformation (shear/compaction bands, tensile/shear cracks/fractures), viscous fingering of flow
Full-field measurements - why?

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• Processes are generally not homogeneous, e.g., localisation of deformation (shear/compaction bands, tensile/shear cracks/fractures), viscous fingering of flow
  ⇒ In the presence of heterogeneity, point-wise measurements at a boundary do not well characterise a system

⇒ **“full-field” measurement techniques**
  (field record of quantities as opposed to point-wise data)
Why might we want to measure fields of properties?

allow qualitative and quantitative characterisation of heterogeneities in both material properties and processes during a test

• material characterisation and specimen inspection
• assessment of actual test boundary conditions
• tracking of heterogeneous response during a test
• validation and identification of models
Fields of a range of physical variables can be measured,
  • scalars (e.g., temperature), vectors (e.g., displacement) or even
tensors (e.g., strain)

Many different techniques exist (each having its advantages
and its limitations)

  • Optical Methods for Kinematics (speckle, speckle interferometry,
    geometric moiré, moiré interferometry, holographic
    interferometry, image correlation, grid method, ...)
  • X-ray Tomography
  • Ultrasonic Tomography
  • Magnetic Resonance Imaging (MRI)
  • Electrical Resistivity Tomography
  • Neutron Tomography
  • ...

Full-field measurements - what?
Selection of a method

• The selection of a technique is based on:
  – the target of the testing and the analysis method (mechanics)
  – the performances of the techniques (resolution, sensitivity to material/phenomena, data treatment...)
  - the feasibility of using the method for the given experimental set-up

• Some basic criteria to select a method:
  – the measurand (displacements, strains...) and experimental conditions
  – the range of the measurement
  – the sensitivity, resolution, spatial resolution
  – the dynamic of the phenomena
  – the intrusivity (non contact methods)
  – the cost, complexity, maintenance, training...
Two examples:

• Strain localisation in a sandstone
  • Pre- and post-mortem x-ray tomography
  • Full-field analysis, including 3D-DIC
  • Neutron radiography for flow mapping

• Grain-scale analysis of strain localisation in a sand
  • In-situ micro-tomography
  • Continuum and discrete DIC
  • 4D Contact analysis
  • Grain-strains from diffraction
**Cone Beam Variable magnification:**

- $\varnothing 4 \text{ mm} \Rightarrow \approx 5 \mu \text{m voxel width}$
- $\varnothing 210 \text{ mm} \Rightarrow \approx 220 \mu \text{m voxel width}$

Adaptability to access the physics of materials at the pertinent scale(s)

**Grès de Vosges** (grain diameter $\approx 300 \mu \text{m}$)

- Voxel width: 90 $\mu \text{m}$  \hspace{1cm} 30 $\mu \text{m}$  \hspace{1cm} 7 $\mu \text{m}$

E. Charalampidou, 2011, PhD Thesis, Heriot-Watt University and Université de Grenoble
Cone Beam
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X-RAY TOMOGRAPHY @ 3S-R

Pre- / post-mortem scanning

Data acquisition:
- radiographs at different angles

Reconstruction

Image volume

Visualisation

3D geometries

Pre-mortem scan

Triaxial loading

Post-mortem scan

Raw data (CT number)
- density information

Standard deviation
- heterogeneity

(Image voxel size ≈ 30µm)

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X-ray tomography results:

- **VE2** (50 MPa conf. pres.)
- High resolution scans ~30 µm voxel size

- Localised deformation appears as higher density zones (dark = higher density)
- Two bands meeting in middle of sample

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3D-volumetric DIC
- strain fields

(Image voxel size ≈ 30µm)

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3D-volumetric digital image correlation

(code “TomoWarp”)

X-ray tomography image volume **before** loading

X-ray tomography image volume **after** loading

3D search 3D for displacement vector based on best image correlation (integer voxel shifts)

Sub-voxel refinement of displacement vectors

Vector displacement field

dX  dY  dZ

Full 3D strain tensor field

\[ \varepsilon_{ij} = \begin{pmatrix} \varepsilon_{xx} & \varepsilon_{xy} & \varepsilon_{xz} \\ \varepsilon_{yx} & \varepsilon_{yy} & \varepsilon_{yz} \\ \varepsilon_{zx} & \varepsilon_{zy} & \varepsilon_{zz} \end{pmatrix} \]

Continuum hypothesis

Strain invariants
- volumetric and shear strains

\[ \varepsilon_v = \varepsilon_1 + \varepsilon_2 + \varepsilon_3 \]

\[ \varepsilon_s = \sqrt{\left(\frac{\varepsilon_1 - \varepsilon_2}{2}\right)^2 + \left(\frac{\varepsilon_1 - \varepsilon_3}{2}\right)^2 + \left(\frac{\varepsilon_2 - \varepsilon_3}{2}\right)^2} \]

(See Hall (2005, Geophysics) and Hall et al., (2009, ComGeo))
3D-volumetric DIC Results:
Shear vs volumetric

- **VE2** (50 MPa conf. pres.)
- High resolution scans ~30 µm voxel size

DIC grid spacing = 10 voxels
Correlation window = 5 voxels

E. Charalampidou, 2011, PhD Thesis, Heriot-Watt University and Université de Grenoble
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E. Charalamidou, 2011, PhD Thesis, Heriot-Watt University and Université de Grenoble
3D-volumetric DIC Results:
Shear vs volumetric

- VE2 (50 MPa conf. pres.)
- High resolution scans ~30 µm voxel size

- Highlights features of localisation and coalescence zone
- Greater shear strain than volumetric
- Volumetric strain varies between compression and dilation

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X-ray tomography results:

- **VE6** (130 MPa conf. pres.)
- High resolution scans ~30 µm voxel size

No evidence (to the naked eye) of localised deformation

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3D-volumetric DIC Results:
Shear and volume strains

- **VE6** (130 MPa conf. pres.)
- High resolution scans ~30 µm voxel size

Maximum shear strain

Volumetric strain

**DIC grid spacing = 10 voxels, correlation window = 5 voxels**

E. Charalampidou, 2011, PhD Thesis, Heriot-Watt University and Université de Grenoble
Local characterisation of fluid flow in sandstone with localised deformation features through fast neutron imaging


Acknowledgements: Darren Hughes and Steve Rowe, ILL And Andrew Harrison (Director, ILL) for his support of this work and for providing access to the Neutrograph facility at the ILL.
**Challenge:**
- Quantification of effect of localised deformation on fluid-flow in sandstone
- Connect deformation/strain to changes in local flow
  - Requires techniques that can
    1. see inside a test specimen
    2. see the fluid distinctly from the solid part
   => Neutron imaging

**Methods:**
- Triaxial loading of specimens under confining pressure
- Post-test imbibition monitored by neutron radiography (ILL, Grenoble, France) and “front-tracking” to measure local flow velocities
- Pre- and post-test x-ray tomography + 3D-volume DIC for tensor strain field
Experimental set-up

Radiography/tomography setup similar to that of Dierick et al (2005):

- Sandstone core (Ø 40 mm, h 40 mm) (supported by three aluminium feet - not visible due to the low absorption of aluminium - implanted in “blutac” in the reservoir)
- Water injected into reservoir through pipe (to the right of the image)
- Image acquisition every 5-15 s (first example)
- Image pixel size ≈ 200 µm first example and ≈ 124 µm for 2nd example

Fig. 1. The setup: a, fluid reservoir, valve and pipe; b, motor stage; c, scintillator; d, mirror; e, camera + lens; f, lead shielding.
Neutron radiography of water imbibition into previously deformed cylindrical samples of Vosges sandstone

(a) Intact  (b) open shear-band  (c) closed shear-band

(b) and (c) deformed previously under triaxial compression under 20 MPa and 50 MPa
Water imbibition into a sandstone (neutron radiography)

Radiographs after subtraction of first image in sequence
Observations

• **Localised deformation** features have higher water saturation than matrix.

• **Intact sample**: advancing fluid front is only deformed near boundaries and flow-rate only varies due to the distance from reservoir

• “**Open**” shear-band sample: water enters rapidly and advances in shear-band with diffusion from the fracture into the surrounding rock. Propagation is faster in the shear-band

• “**Closed**” shear-band sample: water advances much less rapidly, but the flow is still faster in shear-band

• But not quantified...
2nd example

“2D” specimens and multiple fluid phases

Slice through x-ray CT image after deformation

Shear and volumetric strain fields (thresholded) from Digital Image Correlation of pre- and post-deformation images - Note “2D” nature of band
“2D” specimens and multiple fluid phases

Oil

Dry

Water
Time-lapse images

start →

<table>
<thead>
<tr>
<th>images</th>
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<tbody>
<tr>
<td>Rise of curved front - “homogeneous” material</td>
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<tr>
<td>Acceleration up lower branch of shear-band (faster velocities in band)</td>
</tr>
<tr>
<td>Acceleration up main shear band, increased saturation in band</td>
</tr>
<tr>
<td>Water front pushes into oil-zone and appears to “push back” oil</td>
</tr>
<tr>
<td>Layer of lower neutron image intensity - Pooling of water in layer or mixed oil-water effect?</td>
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</tbody>
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→ end

Every 4th image during imbibition
Quantification

• Automatically picked fluid fronts at time N
• Track each point on the front at time N through each image for all time steps
  • Challenging due to concavities and convexities of front and need to follow points on the front through time
  • Deformable contour tracking and front propagation
  • Flow fronts at each step
  • Flow lines from start to finish
• Flow velocities from distances along flow lines and image times
Quantification

Flow-fronts

Flow-velocities

Pixel width = 0.124 mm
Conclusions and Perspectives (Part 2)

• Neutron imaging allows clear mapping of fluid flow through (deformed) rock specimens as water is more absorbing than rock
  - measurement of local flow throughout a specimen
  - assessment of local modifications to the flow due to (localised) deformation

• In this case it appears that the shear-bands represent conduits of increased water flow and storage

• Next steps:
  • Connection of flow and strain
  • Fast 3D (tomographic) imaging;
  • Pressure-controlled flow;
  • Multi-phase flow;
  • In-situ loading at “high” confining pressures with fluid flow monitoring (neutrons allow to see through metal pressure cells)
Summary (Sandstone):

- **Full-field methods** essential in the study of localised phenomena
  - Capture heterogeneity of the processes
  - Allow measurement of geometries e.g., localisation widths/orientations

- **X-ray tomography** provides insight into 3D density distributions
  - High spatial resolution - Geometrical features and dimensions
  - Low sensitivity to damage (only sees larger density changes)

- **Local 3D image analyses** reveals features in x-ray images

- **3D-DIC**
  - Clearer view of localisation structures
  - Quantification of strain and decomposition into shear and volumetric (compaction or dilation) components

- **Neutron imaging**
  - Direct observation of fluid flow through samples with little processing required (better sensitivity than x-rays)
  - Image analysis required for quantification of flow

- **Next steps** - in-situ experiments (imaging during loading/flow linking imaging, loading and flow)
  - Integration of methods
Second example:

- Grain-scale analysis of strain localisation in a sand
  - In-situ micro-tomography
  - Continuum and discrete DIC
  - 4D Contact analysis
  - Grain-strains from diffraction

Objectives

Imaging

material evolution and deformation processes with grain-scale resolution for a sand undergoing triaxial compression

Combining

• 3D in-situ synchrotron x-ray microtomography
• 3D-volumetric digital image correlation (DIC)
  • Continuum approach (strain localisation)
  • Discrete approach (full 3D grain kinematics)
• 3D-volumetric digital image analysis
  • Porosity
  • Grain and contact morphology
Objectives

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*material evolution and deformation processes with grain-scale resolution* for a sand undergoing triaxial compression

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In-situ triaxial testing and x-ray μ-tomography

- Fine-grained, angular siliceous sand
  - $d_{50} \approx 280 \, \mu m$

- Sample dimensions: Ø11 x h22 mm
- Total number of grains $\approx 50,000$

⇒ Small but remains mechanically pertinent
⇒ About 5500 voxels per grain

See Lenoir (2006) and Viggiani et al. (2004) for experimental set-up

Data were acquired at ID15A, ESRF (but many more tests since run using the lab facility at 3SR in Grenoble)
In-situ x-ray $\mu$-tomography

(See Hall et al., 2010)
Porosity evolution

- Porosity calculated throughout the volume based on binarised x-ray tomography images
- Localisation appears as band of dilatation

(See Hall et al., 2010)
**3D-DIC: Displacement fields**

3D-DIC over entire specimen for each load level

- spacing of DIC grid = 20 voxels
- correlation domain = 20 voxels$^3 \approx$ mean grain size

(See Hall et al., 2010)
3D-DIC: Strain fields

(See Hall et al., 2010)
Summary so far

- **In-situ synchrotron x-ray micro-tomography** for triaxial compression tests on sand
  - Following of full mechanical evolution in 4D with grain-scale detail
  - Porosity calculations show a localised dilation evolves

- **3D-volumetric DIC**
  - DIC shows localisation of strain well before stress-peak and organisation (condensation?) of the deformation into a localised shear-band
  - Structures in shear band (consistent with postulated “force-chain” structures)

⇒ Structures indicate grain-scale mechanisms, so what are the grains doing?

Oda et al. [2004]
Objectives

Imaging

*material evolution and deformation processes with grain-scale resolution* for a sand undergoing triaxial compression

Combining

• 3D *in-situ* synchrotron x-ray microtomography
• 3D-volumetric digital image correlation (DIC)
  • Continuum approach (strain localisation)
  • Discrete approach (full 3D grain kinematics)
• 3D-volumetric digital image analysis
  • Porosity
  • Grain and contact morphology
Image segmentation $\Rightarrow$ grain identification + contacts

Tomography image $\Rightarrow$ Individually identified and labelled grains

Tomography image $\Rightarrow$ Binarised volume $\Rightarrow$ Watershed

Binarised volume $\Rightarrow$ Watershed lines $\Rightarrow$ Contacts

Contacts $\Rightarrow$ Grouping and labelling
Contact analysis:

- Tomo
- Contacts
- Contact density
- Coordination number

Contact orientations

- Contact normal azimuth
- Contact normal declination

Work is ongoing (PhD E. Andò, Grenoble)
Objectives

Imaging material evolution and deformation processes with grain-scale resolution for a sand undergoing triaxial compression

Combining

• 3D in-situ synchrotron x-ray microtomography
• 3D-volumetric digital image correlation (DIC)
  • Continuum approach (strain localisation)
  • Discrete approach (full 3D grain kinematics)
• 3D-volumetric digital image analysis
  • Porosity
  • Grain and contact morphology
“Discrete” 3D DIC - Collab. M. Bornert LMS-Paliseau

1. “Classic” DIC
   ⇒ initial estimate of 
   \([dx, dy, dz]\) for each grain

2. Image segmentation
   → grain-mask to define
   grain-shape correlation
   domains (3 voxels expansion
   to capture grain boundary)

**Discrete DIC**

- “Grain shape” correlation domain centred on each grain (from 2)
- Initial estimate of displacements from classic DIC results (from 1)
  ↓
  sub-voxel refinement
  (6 parameters: 3 displ. + 3 rotations)
  ↓
  full grain kinematics for each grain

\[
\Phi(X) = X + T(X_0) + R(X - X_0)
\]

(Hall et al. 2010, Géotechnique)
3D grain kinematics: displ. + rot.

Incremental grain displacements

3-4  6-7

Incremental grain rotations

3-4  4-5  5-6  6-7

- large grain rotations after peak localised with shear strain

(See Hall et al., 2010)
Summary: continuous - discrete analyses

Loading Steps:

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Slices through 3D Volume:

(c) X-Ray Attenuation

(d) Porosity Map

(e) Continuum DIC: Shear Strain

(f) Discrete DIC: Grain Rotations

(Vertical slices through middle of volume roughly perpendicular to localisation)

(See Hall et al., [2010])
Summary (sand)

• **Discrete-DIC / grain ID-tracking**
  • Following of individual grain kinematics for 10’s of 1000 grains
  • Observe significant grain rotations in shear-band as it develops around the stress-peak
  • Tests with different sands indicate that rounder grains rotate more freely - “cog” effect for angular grains

• **4D characterisation of grain/grain-contact structure:**
  • Reduced contact density and grain coordination numbers with elevated shear strain
  • Bridges of higher contact density across shear zone
  • Contacts (possibly) aligned with the shear band orientation but analysis is on-going
  • Next: Full 4D analysis of contacts and structural evolution, contact kinematics