# Experimental report ESRF

## Background

Observing ongoing internal deformations under constant external load in a diverse range of particulate materials from natural origin. Coarse grained: (dry Hostun sand and initially dry Bocahut sand) and fine grained (saturated quick clay from Sweden)

## Experimental Methodology

All materials and mechanical test equipment brought (and removed) by experimenters

*Test Cell*
-Cylindrical Peek cell with 10.25 mm wall thickness and sample dimensions of 10.5 diameter and up to 25 mm tall (samples used up to 10 mm tall).
-wet samples (quickclay) contained between two sintered glass porous discs
-additional aluminium jacket with thin section at x-ray spot for mechanical stability of actuator
-large rotation stage used

*Load application*
-Low stress < 200 kPa on sample boundary: deadweight loading
-High stress < 20 MPa on sample boundary: Electromechancial actuator

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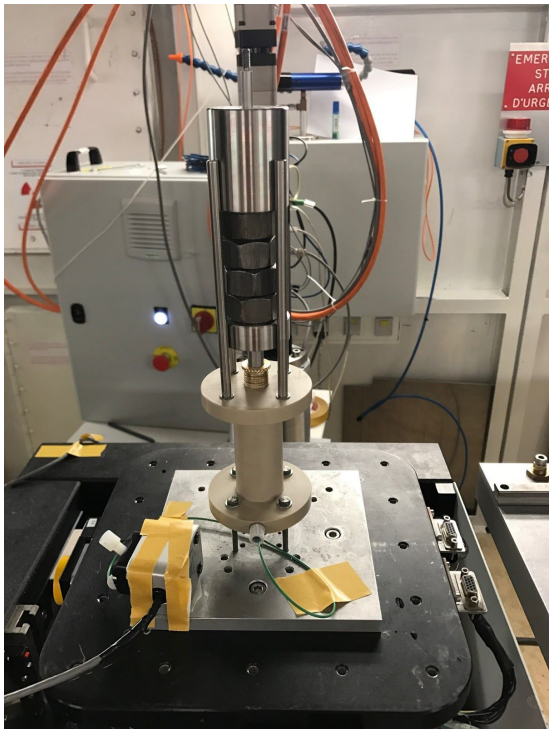
|  |  |
| Oedometric test setup for low stress clay | high stress loading of sand |

*Preparation method*
-Sand: Pouring in dry aim for dense packing
-Clay: cut from master sample in separate kapton ring (5-7 mm tall).

*Materials*
-Quickclay samples: natural quick clay (saturated, illite, montmorillonite, platelet size around 2 um)
-Sand samples: Hostun sand (average particle size ??), Bocahut (average particle size; carbonate material prone to dissolving in water)

*Acquisition settings*
Dual camera for multiresolution:

- PCO Edge with 1X optics for ~6.5µm/px
- PCO Dimax with 10X optics for ~0.65µm/px

For all tests: Low resolution scans at start and end of each loading increment, subsequent high resolution scans to monitor internal 'strain gauge' at set time intervals inbetween (5, 10 or 30 mins). 54 (low res clay) - 93 keV (high res sand)

*Reconstruction*
Paganin method required to obtain sufficient contrast in materials (especially the clay), only this is retained.

## Brief testing programme and testing notes

| Experiment | Test type | #acquisitions | Notes |
|---|---|---|---|
| Quickclay 1 | 1D oedometric loading; 2 load steps (70 & 140 kPa) | 6x 6.5 µm<br>10x 0.65 µm | Cracks appear in sample after prolonged exposure to x-ray during beamline configuration. |
| Quickclay 2 | 1D oedometric loading; 3 load steps (20,50 & 140) | 5x 6.5 µm<br>10x at 0.65 µm | Test terminated after beam line shutter not closed (and cracks appear) |
| Sand 1 (hostun) | 1D oedometric relaxation tests of 30-60 mins each for subsequent stress levels of 600, 1200, 2500, 0 (unload) and 600 kPa (reload) | before/after load step 6.5 µm; 0.65 µm at increasing time intervals (5,10 & 15 minutes) | More relaxation in cell observed than anticipated |

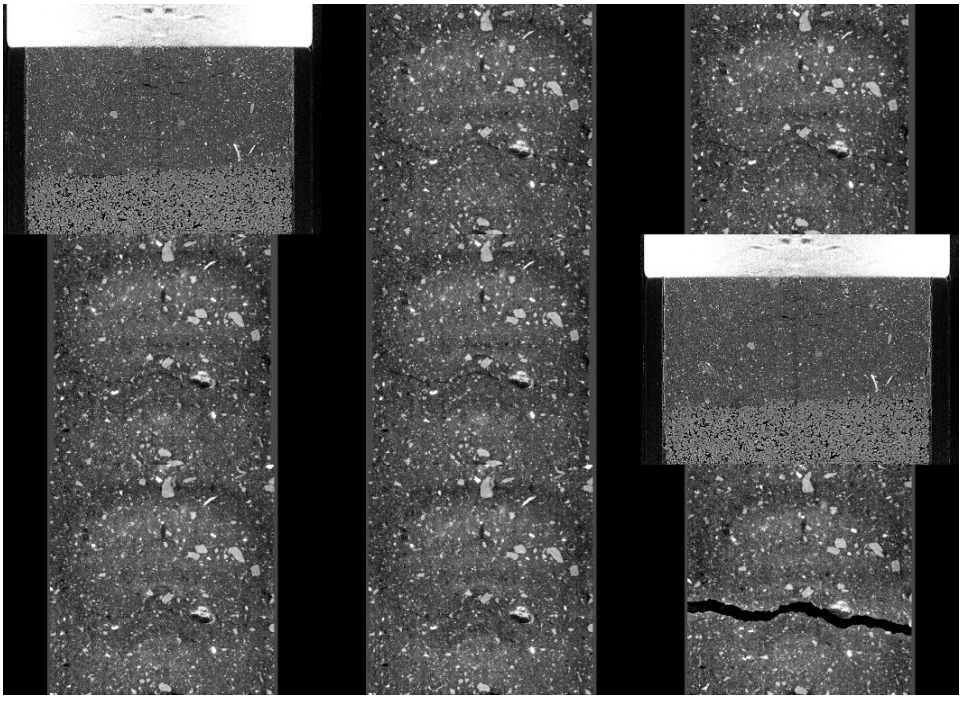
| | | | |
|---|---|---|---|
| Sand Calibration Sample | Rigid-body displacement | 2x 6.5 µm<br>2x 0.65 µm | Hostun grains fixed with epoxy |
| Sand 2 (Hostun) | 1D oedometric creep tests at 2500 kPa (2 hours) | before/after load step 6.5 µm; 0.65 µm at increasing time intervals (5,10 & 15 minutes) | Manual load control +/- 1% |
| Sand 3 (Bocahut) | 1D oedometric creep (1h15m duration)<br>250 kPa - 1000 kPa - water imbibition after 45 mins - 250 (unloading) | before/after load step 6.5 µm; 6.5 µm at increasing time intervals (5,10 & 15 minutes) | Difficult to maintain load due to collapse of material in sample |

## Experimental difficulties

-Temperature logger in test hutch seemed uncalibrated
-Cracks created in high water content clay samples (>50% by mass) after long exposures (> 10 min). Possibly x-ray water interaction photodissociation(?)
-Relaxation in Peek test cell (not detected in pre-testing at 3SR)

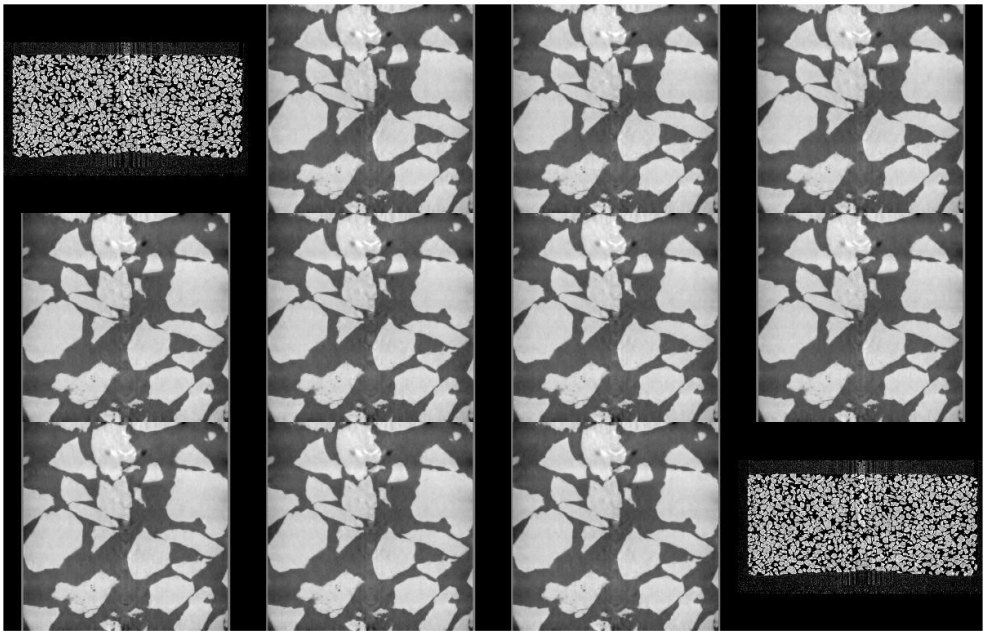
## Prelim experimental results

*Quick clay*
Cracks after long exposure to full beam

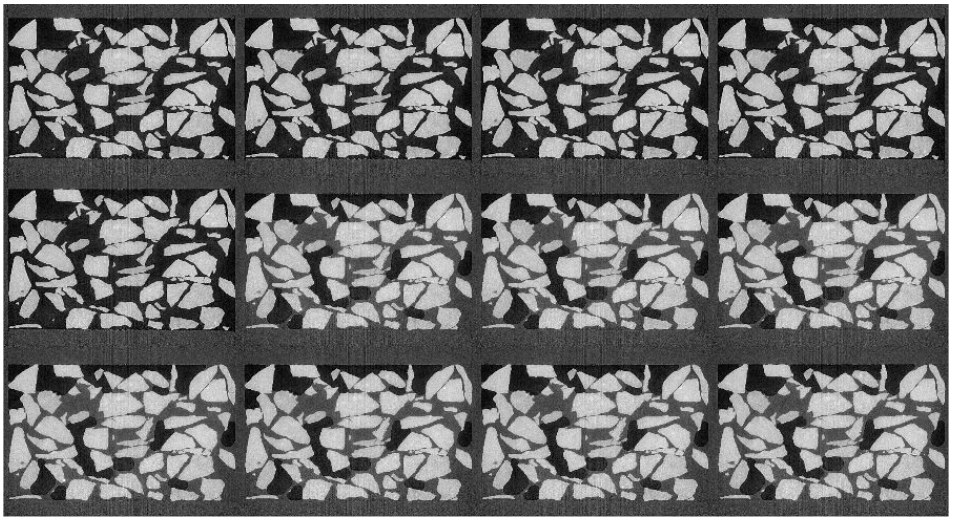


Unexpected self similar material structure at 0.65 μm and 6.5 μm resolution.

*Hostun*
Relaxation and creep in dry granular media are indeed particle re-arrangement processes



*Bocahut*
Water imbibition has profound effect on material response both on the overall strain response with constant external load and on visible grain breakage.



## Planned Analysis

We plan to do DVC (Digital Volume Correlation) on all of these data sets in order to quantitatively extract kinematical quantities. In the case of the sands an in-house particle tracking code will be used, and good results are expected since some rearrangement is visible to the naked eye.