ESRF	**Experiment title:** 3DXRD to characterise stress-distributions in granular geomaterials	**Experiment number**: Ma828
Beamline: ID11	**Date of experiment**: from: 14/04/2010 to: 20/04/2010	**Date of report**: 12/10/2010
Shifts: 18	**Local contact(s)**: Jonathon Wright	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Stephen Hall*

Not on original proposal, but present at experiment:
Edward Andò
Cino Viggiani

Report:

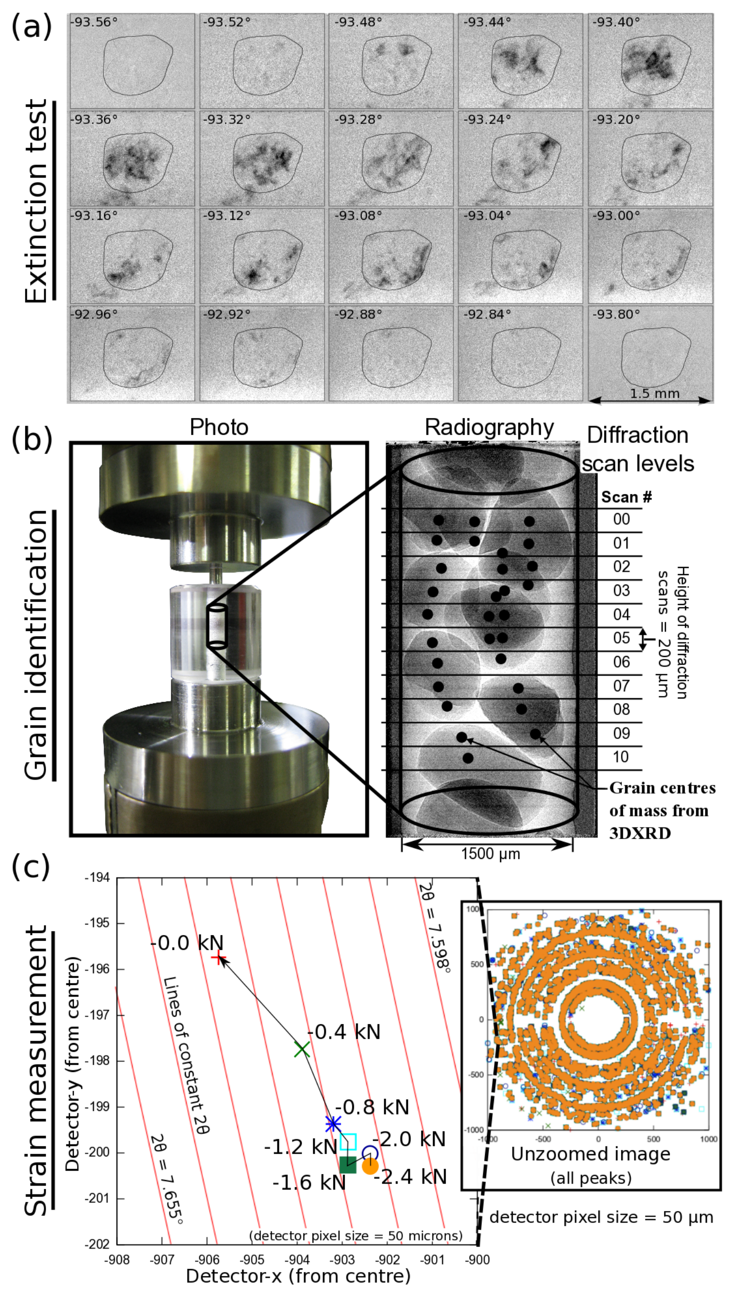
This experiment aimed at applying 3DXRD to follow the evolution of granular structures in sand at the scale of the individual grains. In particular we aimed to use 3DXRD to detect the internal strains of the grains, due to loading, which, by assuming elastic deformation of the grains, can be associated with the force transmission though the granular medium. Such analysis will allow a better understanding of the fundamental mechanics at the grain-scale and the driving forces behind localized deformation and failure.

1D compression was carried out *in-situ* in the 3DXRD set-up on ID11 using quartz-glass "oedometers" (quartz-glass is non-crystalline, *i.e.*, amorphous, and so scattering from the container that could interfere with the analysis is avoided). Displacement-controlled axial loading (using the ID11 5 kN loading rig) from both ends maintained the sample centred throughout the tests. Two oedometers were used, one of 1.5 mm internal diameter, to test the principle in simplified conditions, and the other having 10 mm internal diameter (external diameters were 10 mm and 50 mm, respectively). The Ottawa 20-30 sand (average grain size about 720 μm) was used; this sand consists non-bonded grains of crystalline quartz ($Si0_2$).

In applying 3DXRD to sand, an assumption is made that the individual grains are themselves single crystals. This assumption has been tested using "extinction analysis" for a single grain and single diffraction angle. This involves identifying a single diffraction angle associated with a particular diffraction peak and acquiring radiographs for small increments in angle traversing the peak position. At the diffraction angle of the crystal all the energy should be diffracted away from the direct path and so the grain should "extinguish" (*i.e.*, go dark on the radiogram). If the grain being imaged is in fact a single crystal then the whole grain should show extinction at the same angle. Figure 1a shows the result of such a test. In fact the grain does not quite extinguish completely at a single discrete diffraction angle, but the difference in extinction angles is very small (about 0.6° from first to last extinction). The variations in scattering angle across the grain are likely due to small variations in crystal growth, or, more likely, plastic deformation through the long life-history of the sand grain.

Clear, isolated diffraction peaks can be seen in the diffraction scans, which were succesfully over a range of ω can be indexed and associated with individual crystals using the ID11 3DXRD algorithms. Figure 1b shows a comparison of the centres-of-mass of the grains identified in 3DXRD scans for the 1.5 mm oedometer with a radiograph (x-ray absorption) image of the specimen. A good correspondence of the positions of the grain centres (in each slice) is seen.

So, can grain-strains be measured? Using the 10 mm diameter oedometer, a loading-unloading cycle was carried out, under displacement control, with diffraction measurements made on unloading (to avoid large grain rotations/displacements expected upon loading). 3DXRD measurements were made, with the piston blocked, at 7 positions over the sample width and at 4 heights about the centre of the sample (the beam was about 1.5 mm wide and 0.5 mm high); this total set of 28 scans took about 11 hours. Initial results indicate that strains within grains and very small grain rotations can be measured. Figure 1c shows the movement, over the 7 levels of reducing axial force, of a single identified peak position (one of very many peaks) for a grain within the central part of the specimen. This diffraction peak moves as the load decreases, showing a rotation in η and, more importantly, increasing 2θ, which indicates decreasing d-spacing, *i.e.*, shortening of this grain in this direction (roughly horizontal) corresponding to about $-2.4e^{-3}$. This is a key result as it indicates that the internal grains-strain can be measured using 3DXRD.



(a) Extinction test: radiographs after subtraction of the average of the first and last in sequence (to remove approximately direct beam image) at 0.04° steps from -93.56° to -92.80°. (b) Comparison of centres-of-mass from 3DXRD and grain positions from radiography (1.5 mm oedometer), (c) Movement of a selected Bragg peak, for a grain at the centre of the 10 mm oedometer sample, as a function of decreasing load.

Conclusion

Individual grains-strains have been measured using 3DXRD and such measures could lead to direct observation of force-chain structures in 3D. In addition individual grain (and sub-grain) kinematics can be observed using this technique. Analysis of the data is continuing including automated tracking of all the indexed diffraction spots over all the load steps, which will yield the full set of grain strains across a thick layer through the sample. These first results provide tantalising indications of the potential, but futher experiments are needed and in particular 3DXRD linked with tomography imaging could provide datasets of full grain kinematics (from analysis of the tomography images at differen load steps) and force transfer (from the 3DXRD grain strains) will be possible.