ESRF	**Experiment title:** Force chains and intra-granular strain fields in sand	**Experiment number**: Ma1913
Beamline: ID11	**Date of experiment**: from: 01/12/2013 to: 04/12/2013	**Date of report**: 01/09/2014
Shifts: 9	**Local contact(s)**: Jonathon Wright	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Stephen Hall*

Division of Solid Mechanics, Lund University, Lund, Sweden and European Spallation Source AB, Lund, Sweden

Report:

This experiment, at ID11, involved the study of the evolution of granular structures and grain-strain distributions at the scale of the individual grains as part of a granular assembly. The techniques used were 3DXRD and x-ray tomography, which provided, respectively, the strains in the individual grains and the structural juxtaposition of the grains in the assembly. The experiment built on previous developments of experiments ma828 and ma1216. The experimental set-up and, in particular, the advances from the previous work are outlined in the following before describing the key results. The results of the work have been presented at 3 international conferences and a paper on the work is currently being finalised for submission in the coming weeks.

The material considered was an assembly of “synthetic sand grains”. Each of the grains was made from a 1 mm^3 single quartz crystal cube eroded in an abrasive drum within which the grains were circulated at high velocity by a jet of compressed air. The results of this milling were near “perfect” grains, i.e., single crystal grains of quasi-spherical form. The grains were between about 100 and 300 µm (smaller than the previous experiment, which yielded a much improved packing of the grains in the studied sample).

As with the previous experiments, the “perfect” sand grains were loaded in 1D (oedeometric) compression in a quartz-glass oedeometer (a rigid cylindrical tube), in-situ in the beamline setup, with 3DXRD and x-ray tomography measurements performed at different stages of compression. 77 grains were contained in the oedeometer (internal diameter 1.5 mm) to form a specimen of about 1.5 mm initial height. This sample size provided sufficient grains to minimise the locking effects seen in a previous experiment and also permitted imaging of the full sample in one acquisition (leading to improved measurements as well as more data along the loading sequence).

The loading system used in the previous work was replaced for this experiment with a custom built compression loading device that permitted 360° viewing of the sample, thus avoiding artefacts in the reconstructed tomography images. (In previous experiments the force-return bars on the loading system produced blind angles leading to artefacts in the 3D reconstruction of the sample, which hampered correct grain-contact analyses.)

The camera arrangement was also improved in this experiment such that both the diffraction and imaging detectors were placed in-line with the beam (as opposed to having the diffraction detector at an angle to the beam in the previous experiments). This provided better quality 3DXRD data with less risk of bias to certain directions of strain measurement. Unfortunately, the imaging camera mirror was attached with metal screws that cast a shadow on the diffraction detector and so simultaneous tomography and 3DXRD measurements

were not possible. Thus, the imaging detector was moved out during the diffraction acquisition and moved back in again for tomography acquisition, which resulted in longer acqusiition times at each load step.

During the experiment the incoming wavelength was periodically measured (before and after each mesurement) to monitor variations. In addition the ID11 beamline staff had worked on the wavelength stability over the preceeding year. The result of these improvements was that the wavelength drift was much reduced compared to previous experiments and that there was a measurement that could be used to correct any variations. The result was that the volumetric strain for each grain was much more correctly and precisely determined throughout the in-situ testing than was previously possible.

The main experiment involved a loading experiments with 77 grains of about 100-200 μm diameter with loading upto 65 N axial compression force, unload back to 0 N, reload to about 100 N (past a failure event of the sample) and unload to 0 N. 3DXRD and tomography acqusitions were made every 5 N up to 65 N during the initial loading then, during unloading to 0 N, every 10 N. 5 further acqusitions were made during the reload to 90 N and 2 on the final unloading. Acquisition times were about 25 minutes for 3DXRD and 40 minutes for the tomography, during which time the piston displacement was held fixed (some relaxation was observed in the force).

During the experiment the 3DXRD data processing was performed to yield the cell parameters for each grain at each loading step. These cell parameters were subsequently interpreted in terms of strain relative to the first load step (i.e., 1 N; a small initial load was applied to avoid movement of the grains during the first measurement). As an example of the output from the analysis, Figure 1 shows the strain tensor values for each grain (note that these are the average strain tensors for each grain at each load level) during the first loading; the strain tensors have been rendered on to the surface of spheres positioned at the centres of mass of the grains (the actual grain surfaces can be extracted from the tomography, but due to the large number of grains a better visulaisation can be made using spheres). This sequence shows the development of force chains through the granular assembly and their evolution with changes in the grain configuration. It was noted that the unloading showed similar patterns of strains to the iniital loading, but with increased assembly-average levels; this corresponeds to a higher sample stiffness (based on the axial force-displacment response). On reloading the strain distributions patterns were again similar, and the assembly-average strain level was close to the initial loading, however the sample stiffness increased in the final stages, which appears to be contradictory to the grain-strains being similar. In fact, the distribution of the strains across the granular assembly was seen to be more equal in the reload, thus a link between the equality of the strain distribution and the macroscopic properties could be observed.

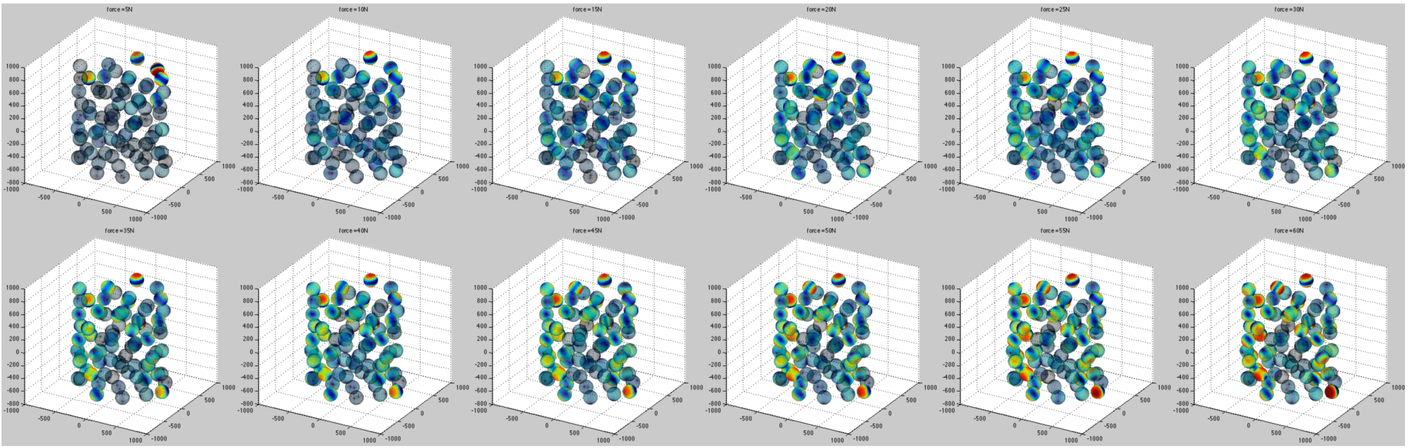

Fig. 1: Average strain tensors for each of the individual grains in the granular assembly during the initial loading of the sample to 65 N. The oriented strain tensors are rendered on to the surfaces of spheres positioned around the centre-of-mass of each grain: red indicates compression and blue extension (range: $-1.5e^{-3}$ (compression) to $+0.5e^{-3}$ (extension).

The grain surfaces can also be extracted from the tomography data such that the contact points between the grains can be identified. In the coming months the strain data plus the contact positions will be expolited to provide contact forces for each grain-grain contact in the assembly (collaboration with J. Andrade, CalTech).

During the expriment a number of other interesting observations were made, including the collapse of the specimen, though failure of some grains, whilist the sample was left over night under a fixed axial displacement. This suggests some sort of slow relaxation-creep phenomena that should be further investigated.