ESRF	**Experiment title:** Investigation of spatio-temporal evolutions of force chains in granular geomaterials using 3DXRD	**Experiment number**: Ma1216
Beamline: ID11	**Date of experiment**: from: 13/06/2012 to: 19/06/2012	**Date of report**: 11/01/2013
Shifts: 18	**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 extended the previous work with 3DXRD to follow the evolution of granular structures and grain-strains at the scale of the individual grains (ma828). Building on the previous experience that indicated that the internal crystal structure of natural sand grains was not completely uniform (due to sub-grains and intra-granular cracks), an "artificial sand" was used to avoid many of the complexities of the natural material. The artificial material was made from 1 mm^3 single quartz crystal cubes eroded in a "ball-mill" (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.

Two loading experiments were performed: one with about 100 grains of about 400 μm diameter and another with more grains ranging from about 200-400 μm diameter. The experiments both involved loading the "perfect" sand grains in 1D (oedeometric) compression in a quartz-glass oedeometer, in-situ in the beamline setup, with 3DXRD and x-ray tomography measurements performed at different stages of compression. The grains were contained in a cylindrical tube, of internal diameter 1.5 mm, to form a specimen of 5.21 mm initial height. Scans were made at 4 different heights to image the whole sample (beam height was1.5 mm). Initial results of the data analysis are presented in the following for the first experiment, focussing on the first height scan.

The first experiment involved 100 grains of about 300 μm diameter (see Fig. 1) undergoing a 1D compression load-unload cycle from 0 to 70 N to 0 axial force. At loading increments of about 7 N during loading and -14 N during unloading, the loading was paused, with displacement held. 3DXRD and tomography scans were performed. simultaneously at these moments using different detectors over angular ranges of -76.4°-75.6° and 103.6°-255.6° at 2° increments; the gaps in the range being due to the loading frame tie-bars obscuring the line-of-sight to the sample. The tomography detector was placed in-line with the beam and the 2D diffraction detector was offset to one side. From the transmission data it was possible to reconstruct the 3D image of the specimen (using the ASTRA toolbox; Palenstijn et al., 2011); although missing angle artefacts do exist, the individual grains can be clearly identified and separated. From these tomography data details on the grain shapes, contacts and movements are being derived. The 3DXRD data consist of full 2D diffraction patterns from all the illuminated grains at each angle. These patterns hav been processed to extract all the diffraction spots corresponding to each of individual grains, from which the grains' crystal orientations and cell parameters have been determined. The quality of the "perfect" grains meant that this process was very successful, for example, the centre-of-mass positions of the grains correspond well to those from the tomography images. Furthermore, consistency in the data across load steps

allow the grain rotations and displacements to be followed through the load cycle and changes in the diffraction patterns have been used to determine the individual grains strains.

Fig. 1 presents a tomography image of the upper part of the sample in the initial load step, the evolution of the volumetric grain-strain of each of the illuminated grains over the first 15 load levels and the grain-averaged principal grain-strain vectors for the same load levels. From the tomography image the quality of the grain shapes can be seen as well as the structure of the sample. The force-strain curves show a clear evolution of the volumetric grain-strains consistent with the sample being under compression. The curves show a generally higher initial gradient of compression as a function of applied force, which flattens gradually after about 25 N (indicating a stiffening of the sample response). On sample unloading, the grains in general appear to unload also, but not significantly and some grains continue to exhibit further compression. The grains show a higher strain level on unloading at the final load level plotted (about 30 N) than in the loading leg, which indicates a locking of the grains in their confined positions. It is noted that the grain-strain curves have a certain degree of scatter, which might be reduced through improvements to the experimental method and data analysis (also note that the strains are relative to an "ideal" crystal, hence the initial strain at zero load; currently the values should be taken as indicative, as opposed to absolute). The global strain curve, when compared to the grain strain curves, also indicates that the initial strain in the whole sample involves a lot of non-grain contributions; this is to be expected as the sample will have densified through porosity reduction and closure of contacts before significant force could be transferred through the grains. The strain tensor plot, indicating the evolution of the strain tensor orientations, shows some consistency in the strain orientations over different load steps, but with some abrupt "flips" in direction. The different loading histories of the grains and these principal orientation variations are being compared to the grain contact distribution and evolution in on-going work.

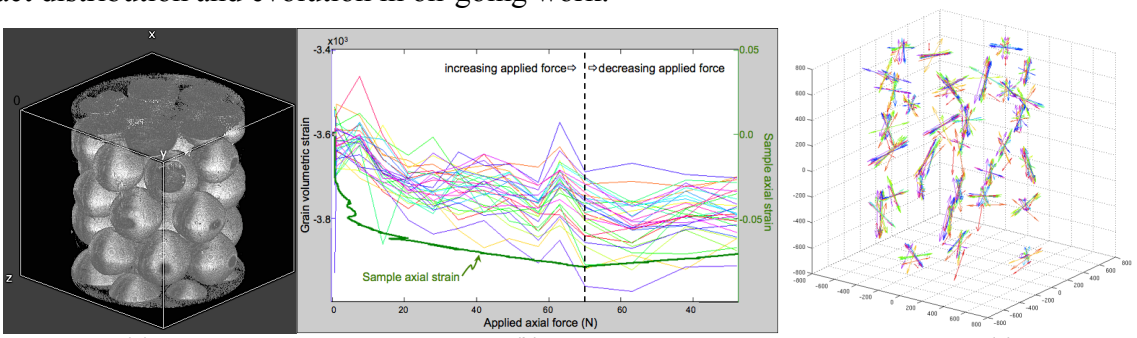

Fig. 1: (a) rendering of tomography image of upper part of the sample; (b) volumetric strains in each individual grain from 3DXRD plus global (axial) strain as functions of applied axial force; (c) Principal strain vectors for the grains at each load level in (b).

Additional measurements performed during the experiments, involving "extinction" analysis in transmission (enhanced attenuation due to diffraction), demonstrated that the manufactured grains are perfect single crystals with uniform diffraction, and thus uniform crystal structure, over their volume in their unloaded state. However, as load was applied, the transmission of forces across grain contacts led (as would be expected) to inhomogeneous strain fields in the grains, which could be observed as a change in the crystal lattice spacings, and thus of diffraction angles, through the grains; see Fig. 2. In particular, these results show that the variations in diffraction angle are focussed, unsurprisingly, on the contact points between the grains. Such data are very rich and could potentially provide very detailed insight into force distributions, although this will require extensive experimental and analysis developments.

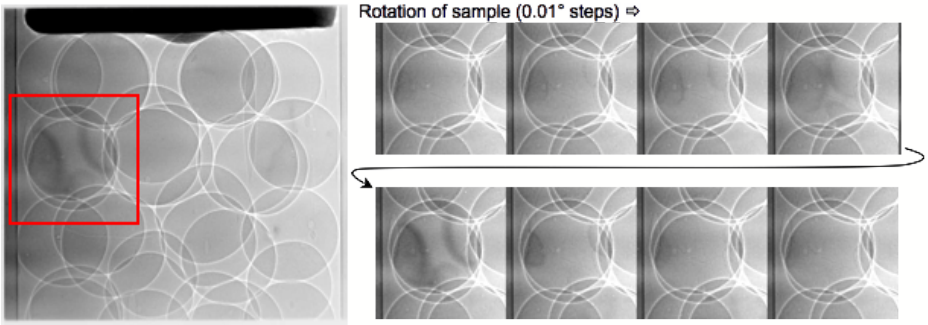

Fig. 2 Radiography image of the upper part of the sample under 14 N compressive load and a zoom for different sample rotations indicating varations in transmission due to "extinction" that reflects the local strain distribution in the grains.