ESRF	**Experiment title:** **Linking structure, kinetics and ultrasound propagation in deforming granular matter**	**Experiment number**: Ma4200
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Shifts: 12	**Local contact(s)**: M. Makjut & J. Wright	

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Report:

The aim of this experment was to study relationships between structure (particle morphology, contact topology), kinetics (inter- particle forces, particle stresses) and ultrasonic wave propagation in granular materials under multi-axial stress conditions. The experiment extended previous developments using 3DXRD and x-ray computed tomography at ID11 combined with mathematical inversion techniques (ma1913, ma2665, ma3373) that enabled simultaneous measurements of structure and kinetics *in-situ* during uniaxial and triaxial compaction of granular systems of up to 1100 grains. Exploratory work during ma3373 demonstrated the possibility of acquiring ultrasonic wave propagation data through granular packings *in-situ* during loading and simultaneously with x-ray measurements.

The orignial experiment plan had two main goals:

1. To peform triaxial compression on a large assembly of grains to investigate the evolution of force transmission, in relation to granular structure change, during the evolution of localised deformation. In experiment ma3373 and attempt at triaxial compression with 3DXRD and tomography was made and yielded new results, but the 3DXRD analysis revealed that the expected stress state was not achieved indicating that the membrane surrounding the sample, which should transmit the confining pressure, was too stiff. Consequently the experiment was redesigned with more flexible membranes allowing the transmission of the confining pressure and the freedom for the sample to develop shear.
2. Perform ultrasonic measurement in conjunction with the in-situ loading, 3DXRD and x-ray tomography in an extension to initial tests in experiment ma3373.

In terms of the original objectives outlined above, the first objective was realised, as described below, whilst issues with the ultrasonic measurement system experienced during the beamtime prevented meeting the second objective. The consequence of the latter was that the experiment plan was revised during the beamtime to investigate new avenues, namely, investigation of intragranular strain by scanning-3DXRD and DCT. The experiment outcomes and the current state of analysis are described in more detail below.

Triaxial testing

A triaxial compression test on a sample of about 1.4 mm diameter and 2.4 mm high of >1000 ruby grains (average diameter about 150 microns) was prepared in a pre-moulded elastomer membrane and mounted in the triaxial device. Tomography data (pixel size 1.54 microns) were acquired in two vertical positions (1600 images over 180° with 1s exposure; about 32 minutes per acquisition) to cover the full sample height.

3DXRD data were acquired at 3 vertical positions (1440 images over 180° with 0.14s exposure time; approximately 7 mins per acqusition). The cell pressure was raised to 1.5 MPa then to 3 MPa and the acquisition repeated at each pressure level. Deviatoric loading was applied, with the confining pressure constant at 3 MPa, over 7 load steps of 40 pixels (61.6 microns) displacement at each step.

The triaxial coompression, with the new experimental design, was succesful and the data are currently being analysed. Initial analysis suggests that with 3 MPa confining pressure the crystalographic strains within the ruby grains are perhaps not significant enough to be measured in the 3DXRD data, but further analysis is underway. However, the analysis of the tomography images provides data on the structural evolution of the sample and the 3DXRD data allows even small grain rotations (that can not be measured in the tomograph images) to be detected and analysed.

Intragranular strain fields

In recent work (described in Hektor et al., 2019), a scanning 3DXRD method was utilised at ID11 to study intragranular strains in a polycrystalline tin-plated copper sample. Based on the positive outcomes of this work and the issues with the ultrasonic measurement system, it was decided to try the same approach for granular samples. Furthermore, it was decided to acquire DCT data for the same sample such that the possibilities with the different approaches might be assessed. As such a sample of 18 rounded, single-crystal quartz grains was mounted in a PEEK tube the in-situ loading device and a small load was applied to hold the grains in place. Three data sets were acquired in this state: (i) full-field transmission tomography; (ii) full-field, far-field 3DXRD; (iii) DCT. A 20 N axial load was then applied to the grains and the three measurements were repeated plus a scanning 3DXRD acqusiition, which used a 20 x 20 μm^2 beam scanned laterally across the sample in 71 steps of 20 µm with a 180° rotation at each position with diffraction acquisition over 1° sweeps and 0.14 s exposure time. 15 such layers, with a spacing of 20 microns, were acquired taking about 70 minutes per layer.

The data analysis is on-going based on a new analysis approach for scanning-3DXRD developed by Henningsson et al. (2020) with the aforementioned tin-plated copper sample data. The application of these algorithms is underway for the quartz data. Figure 1 shows initial results from the analysis in the form of the voxel-by-voxel measurement of the strains in the grains and the rotation of the unit cell, with repect to each grain average.

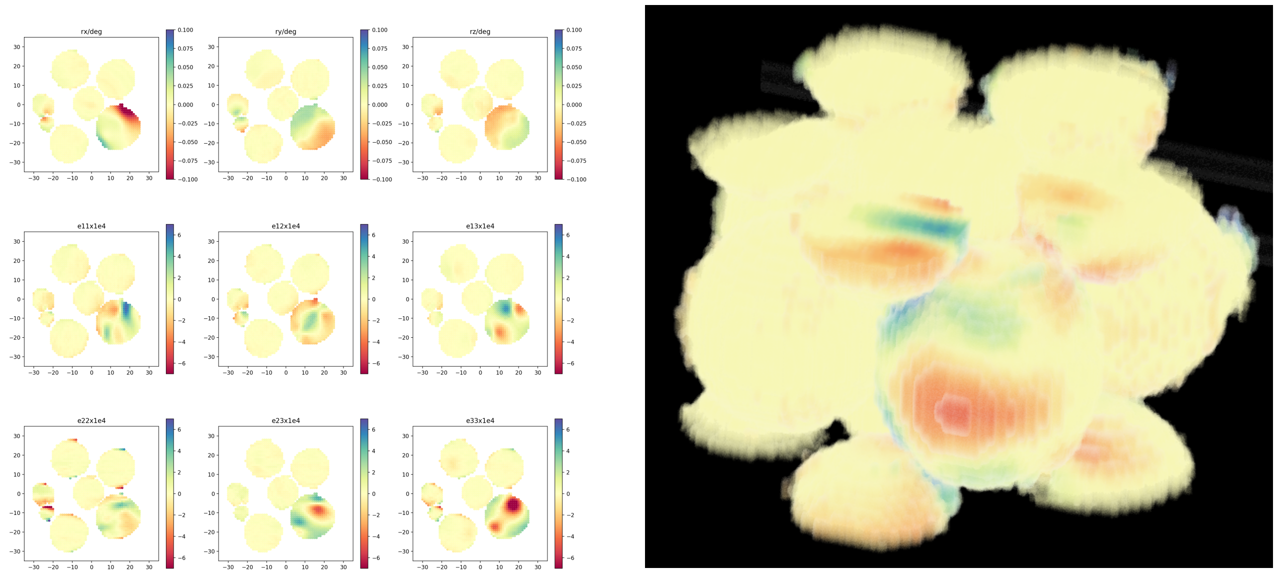

Figure 1. Initial results from the scanning-3DXRD analysis of 18 single-crystal quartz grains under 20 N axial load and confined in a PEEK tube (sample width is about 1 mm). The left image shows a single slice from the anlaysis with the crystal lattice rotations in x, y, and z in the top row and the 6 components of the strain tensor in the bottom two rows. The image to the right shows a 3D rendering of the e_{11} component of strain for the 15 acquired layers.

Hektor, J., Hall, S.A., Henningsson, N.A., Engqvist, J., Ristinmaa, M., Lenrick, F. and Wright, J.P., 2019, Scanning 3DXRD Measurement of Grain Growth, Stress, and Formation of Cu_6Sn_5 around a Tin Whisker during Heat Treatment, Materials, 12, 446

Henningsson, N.A., Hall, S.A., Wright, J.P. and Hektor, J., 2020, Reconstructing intragranular strain fields in polycrystalline materials from scanning 3DXRD data, J. Appl. Cryst., 53