ESRF	**Experiment title:** Intra-granular strain, force transmission and fracture in granular matter under load	**Experiment number**: MA 4493
Beamline: ID11	**Date of experiment**: from: 01 December 2020 to: 06 December 2020	**Date of report**: 24/08/22 *Received at ESRF:*
Shifts: 12	**Local contact(s)**: Jon Wright, Wolfgang Ludwig	

Names and affiliations of applicants (* indicates experimentalists):

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Report:

The aim of this experiment was to employ recent advances in scanning-3DXRD (s3DXRD) to measure intra-granular strain fields in granular materials under load and investigate the evolution of internal grain stresses and inter-granular force transfer up to and beyond grain fracture. Two types of grains were to be studied, a "model" system of synthetic, single crystal grains of quartz and an assembly of natural quartz sand grains; the latter to explore the influence of pre-existing internal grain heterogeneity.

Due to Covid-19 restrictions, the experiment was performed as a "mail-in" experiment with the local contacts, J. Wright and W. Ludwig, on site and the PI connected remotely for discussion via zoom. The aim was to employ a new light-weight uniaxial loading frame with an actuator to run in-situ constrained 1D compression tests on the granular materials. The system was designed to be mounted on the nano-station hexapod of ID11 and permit 360° sample visibility for 3DXRD measurements and tomography. To adapt to the mail-in nature of the experiment, the loading device was simplified to involve manual loading by a screw with monitoring of the applied, axial force by a load cell connected to a single NI-DAQ measurement card and laptop computer (see Figure 1). This implied less-fine control on the loading increments, but worked sufficiently well. In addition, the samples were assembled in "cassettes" before shiping so that they could be easily placed in the load rig, minimising issues with sample preparation. The samples were scanned with x-ray tomography in Lund prior to shipping.

Two experiments were performed, one on natural Ottawa sand, with grain diameters of about 300 µm, and the other on quasi-spherical single crystal quartz grains with grain diameters of about 400-500 µm. The grains were mounted in cylinders of about 1-1.2 mm high and 1.4 mm diameter at the start of the test. The two experiments were run over a series of load steps until grain breakage occurred. The aim was to run full-field (ff)3DXRD, scanning (s)3DXRD and phase-contrast tomography (PCT) at each load step.

For the first test on 12 grains of synthetic quartz, for 3 N and 10 N axial load, tomography and ff3DXRD plus a "box scan" 3DXRD involving scanning a wide+short box beam then a tall+narrow box beam horizontally or vertically over the sample, respectively, with a continuous sweep scan over 360° at each position. The aim of this approach was to speed up the acquisition in the early stages, given that the s3DXRD would take about 14 hours with a "pencil" beam of 20 x 20 µm^2 cross-section scanned across the sample over 43 lateral y-positions and a continuous sweep scan over 360° at each y-position integrating with 1° intervals repeated over 59 vertical

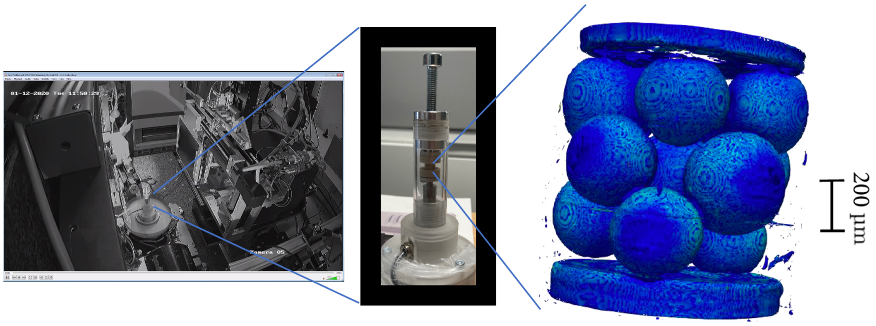

Figure 1: photographs of the experimental set-up, sompified loading device and a 3D rendering of the reconstructed PCT data for the synthetic quartz sample at 20N load.

positions (layers). At 20 N axial load ff3DXRD, s3DXRD and PCT were performed, with a 20 x 20 μm^2 beam corss-section for the s3DXRD, after which the sample was loaded to 40 N then 60 N. On loading to 90 N grain breakage occurred and the final measurements were made at this load level. For 40, 60 and 90 N loads the s3DXRD beam cross-section was changed to 25 x 25 μm^2 to speed up the measurements (less measurements required to cover same sample volume), resulting in 9-hour scans. A second experiment was run following the same procedures as the first, but using natural Ottawa quartz sand grains. In this second experiment, ff3DXRD, s3DXRD and PCT were performed for all load steps using a 25 x 25 μm^2 beam cross-section for the s3DXRD. Measurements were made at 3, 20, 40, 60 N.

Data analysis has been performed using the ASR-s3DXRD approach of Henningson et al. (2019) for the synthetic quartz sample (Vestin, 2022) and analysis is on-going for the sand sample. Unfortunately, a solution to the analysis of the acquired box-scan data has not yet been found, therefore only the 20, 40, 60 and 90 N load levels could be fully analysed in terms of intra-granular strain fields, but PCT data for 3 and 10 N are also being utilised in the analysis. Example results are shown in Figure 2, showing the 6 strain tensor components from s3DXRD at 20 N axial load and the volume stress field evolution over load-steps 20, 40, 60 and 90 N. In the final step (90N) broken grains can be observed.

A paper is currently being prepared for publication on the synthetic quartz experiment.

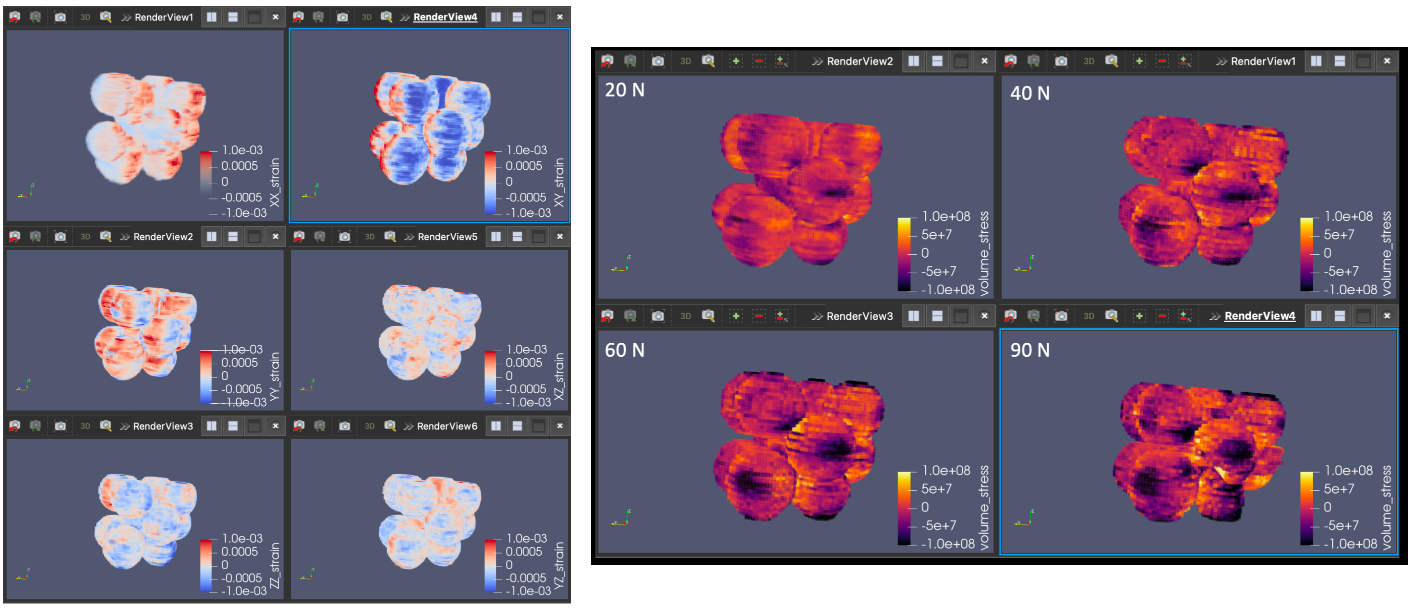

Figure 2. Left: 3D renderings of the 6 strain tensor components for the synthetic quartz derived from s3DXRD acquired using a 20 x 20 x20 μm^3 voxel size. Right: 3D renderings of the volume stress derived from the s3DXRD tensor strain fields for load steps 20, 40, 60 and 90 N. In the final step (90N) broken grains can be observed.

1. A. Henningsson et al., 2019, *Reconstructing intragranular strain fields in polycrystalline materials from scanning 3DXRD data*, Journal of Applied Crystallography.
2. P. Vestin, 2022, *Estimation and interpretation of the intra-granular stress and strain evolution in a uniaxially loaded silica sample using scanning x-ray diffraction*, MSc thesis, Div. Solid Mechanics, Lund University