ESRF	**Experiment title:** Force chain structure, evolution and failure in granular materials	**Experiment number:** Ma2665
Beamline: ID11	**Date of experiment:** from: 18/02/2016 to: 23/02/2016	**Date of report:** 02/08/2016
Shifts: 12	**Local contact(s):** Jonathan Wright	*Received at ESRF:*

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Report:

Experiment ma2665 at ID11 involved studying a granular material, composed of over 1,000 ruby spheres, subjected to quasi-static 1D (oedeometric) compression cycles in-situ in the beamline. The goal of the experiment was to investigate force transfer and heterogeneity in a sample with a representative number of grains, and their influence on failure mechanisms such as force chain reorganization and grain fracture. The experimental methods included 3DXRD, x-ray tomography and interparticle contact force inversion techniques. These techniques have been developed for granular materials in previous experiments at ID11 (ma828, ma1216, ma1913; see [1, 2]). The results represent the first known case in which a large assembly (over 1,000 grains) has been subjected to loading in-situ with simultaneous imaging in a manner allowing interparticle force network quantification.

Experiment and measurements. – The granular sample was composed of 1,098 single-crystal ruby spheres with diameters between 80-120μm, as shown in the tomographic reconstruction in Fig. 1a. The sample filled a 1.5 mm tall by 1.5 mm diameter volume inside of an aluminum (Al) cylinder. The cylinder was placed in a custom built uniaxial loading device with 360° transparency, as shown in Fig. 1b. The sample was loaded with an upward acting piston while displacement and load were monitored with a displacement transducer and top-mounted load cell. The sample was loaded and unloaded three times, to three different axial force levels, and finally loaded to a crushing point before the experiment concluded, as illustrated in the load cell data in Fig. 1c. At regular increments of approximately 10N in loading and 30N in unloading, sample strain was held constant for approximately 40 minutes while 3DXRD and tomography scans were performed. Sample strain was also held constant for six scans during the final loading stage in order to investigate stress-relaxation effects close to sample failure. 3DXRD scans involved recording 2D diffraction patterns over sample rotation of 180° in increments of 0.25°. Tomography scans involved recording 2D transmission data as the sample was rotated 180° in increments of 0.1°.

Results and Analysis. – We performed tomographic reconstruction and morphological grain segmentation (e.g., Fig. 1a) for all load steps and processed diffraction data (using methods from [3]) to obtain average tensor stresses for each grain in the sample. The homogenized sample stress from the diffraction data, similar to the average of the grain stresses, is illustrated Fig. 2a and demonstrates trends agreeing closely with macroscopic load cell readings in Fig. 1c. The vertical components of individual grain stresses demonstrate increasing homogeneity with load, as recently suggested by [1] and [2] using data from ma1216 and ma1913 and visualized using the Gini coefficient for the current sample in Fig. 2b. Deviatoric stresses within each

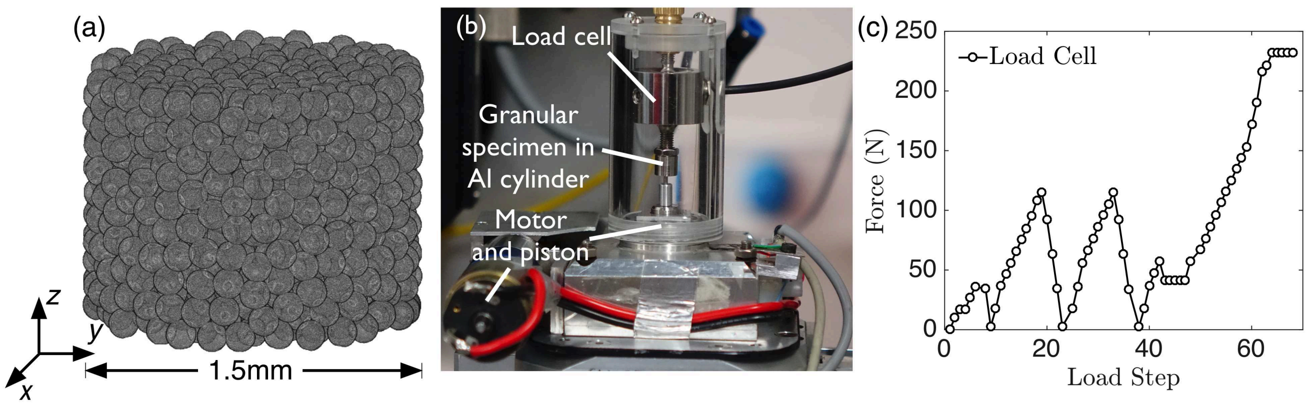

Figure 1: (a) Tomographic reconstruction of 1,098 single-crystal ruby spheres as imaged on ID11 in load step 4 of the experiment. (b) Custom uniaxial loading device featuring aluminum cylinder housing a granular sample illuminated by the beamline. (c) Loading protocol of granular sample during the experiment.

grain are being analyzed and compared with known fracture criteria to locate imminent fracture events. Analysis of radiographic and tomographic images suggests that the sample degradation and failure was by means of grain fracture and strain localization. This hypothesis is being studied by analyzing center-of-mass (CoM) particle motion from 3DXRD data. Fig. 2d illustrates CoM particle motion, visualized by linking CoM positions for each grain with straight lines between adjacent load steps. As expected, more motion occurs at the bottom of the sample, where the loading piston is contacting the material. We also see more motion toward one side of the sample, highlighted with a dashed red line. This motion may correspond to a region with more frequent grain fractures and force chain rearrangements. We are currently exploring quantitative metrics for investigating nonaffine grain motion and employing force network reconstruction [2], both of which will aid in interpreting these results. The findings of these studies will have significant implications for interpreting the bulk response of a granular material in terms of localized failure events.

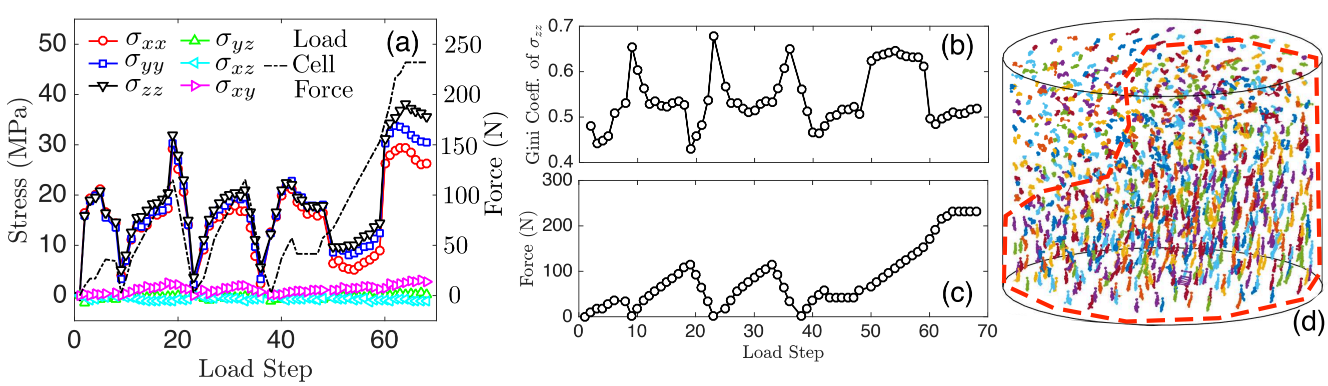

Figure 2: (a) Homogenized (averaged) grain stresses during the experiment. (b) Gini coefficient illustrates increasing stress homogeneity with load. (c) Load cell data for reference to (b). (d) Grain trajectories indicating localized deformation, suggesting that grain fracture induces force chain rearrangement preferentially on one side of the sample.

[1] SA Hall and J Wright. *Géotechnique Letters*, 5(4):236–242, 2015.

[2] R.C. Hurley, S.A. Hall, J.E. Andrade, and J. Wright. *Physics Review Letters (In Press)*, 2016.

[3] Jette Oddershede, Søren Schmidt, Henning Friis Poulsen, Henning Osholm Sørensen, Jonathan Wright, and Walter Reimers. *Journal of Applied Crystallography*, 43(3):539–549, 2010.