X-ray Tomography and Diffraction Measurements to Study Elasticity and Fracture in Concrete

Ryan C. Hurley *[1] and Darren C. Pagan[2]

[1]Johns Hopkins University, Baltimore, MD, USA
[2]Cornell High Energy Synchrotron Source, Cornell University, Ithaca, NY, USA

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Summary: We combine *in-situ* X-ray computed tomography (XRCT) and 3D X-ray diffraction (3DXRD) to we measure cement paste strain fields, quantify the nucleation and growth of fractures, and measure full stress tensors in ∼40 sand particles embedded in the cement paste of a concrete sample compressed to failure. The measurements provide new insight into stress heterogeneity before and after sample fracture.

1. INTRODUCTION

Extensive experimental research in the concrete community has employed X-ray computed tomography (XRCT) to observe the nucleation and growth of fractures through sample microstructures [1, 2]. Ample theoretical research has also examined the stresses occurring in aggregate particles and the influence that they, and the interfacial transition zones (ITZ) separating them from the cement paste, have on macroscopic concrete properties [3]. However, past experimental work has not provided local stress measurements needed to validate theoretical models. Here, we will present a novel use of *in-situ* 3D X-ray diffraction (3DXRD) and XRCT measurements during compression of a concrete sample to failure. At each macroscopic load step of the experiment, XRCT measurements provide details of the concrete's microstructure, including the location of cement paste constituents, voids, sand particles, and fractures. Also at each step, 3DXRD measurements provide the orientation and full, individual strain and stress tensors in ∼40 sand particles embedded in the sample [4]. The intra-particle stresses are observed to be highly heterogeneous, even prior to microscopic fracture nucleation and macroscopic sample failure, but to follow mean-field predictions, on average. Results highlight needed amendments to mean-field theories and provide important data for validating meso-scale models.

2. EXPERIMENTAL METHOD

Experiments were performed at beamline F2 of the Cornell High Energy Synchrotron Source (CHESS). The concrete sample was made by mixing portland cement, 177 μm - 250 μm angular single-crystal quartz particles, and water in an approximate 1:2:6 ratio to form a thick mixture. The mixture was vibrated and cured in a silicone mold, submerged in a hydrated lime solution for 28 days, and finally cut to 1 mm x 1 mm x 1 mm for experiments. The sample was placed between stainless steel platens in the rotation and axial motion system (RAMS2) at CHESS. In small increments, the stainless steel platen above the sample was lowered to impose sample strain. After each small increment, sample strain was held constant while the sample was rotated first through 180°, then through 360°, while illuminated with a 41.991 keV X-ray beam for 1800 radiography and 1440 diffraction pattern measurements, respectively. The experiments are described in detail elsewhere [5]. XRCT data was processed in Matlab and Python to segment constituents of the microstructure at each load step (cement paste, voids, sand particles, and fractures) and analyze their location and strain. 3DXRD data was processed in heXRD to yield intra-particle strain tensors with 10^{-4} resolution per component. This data was then processed in Matlab to yield intra-particle stress tensors and to combine it with XRCT data to study stress heterogeneities and the evolution of stress relative to fractures.

*e-mail: rhurley6@jhu.edu

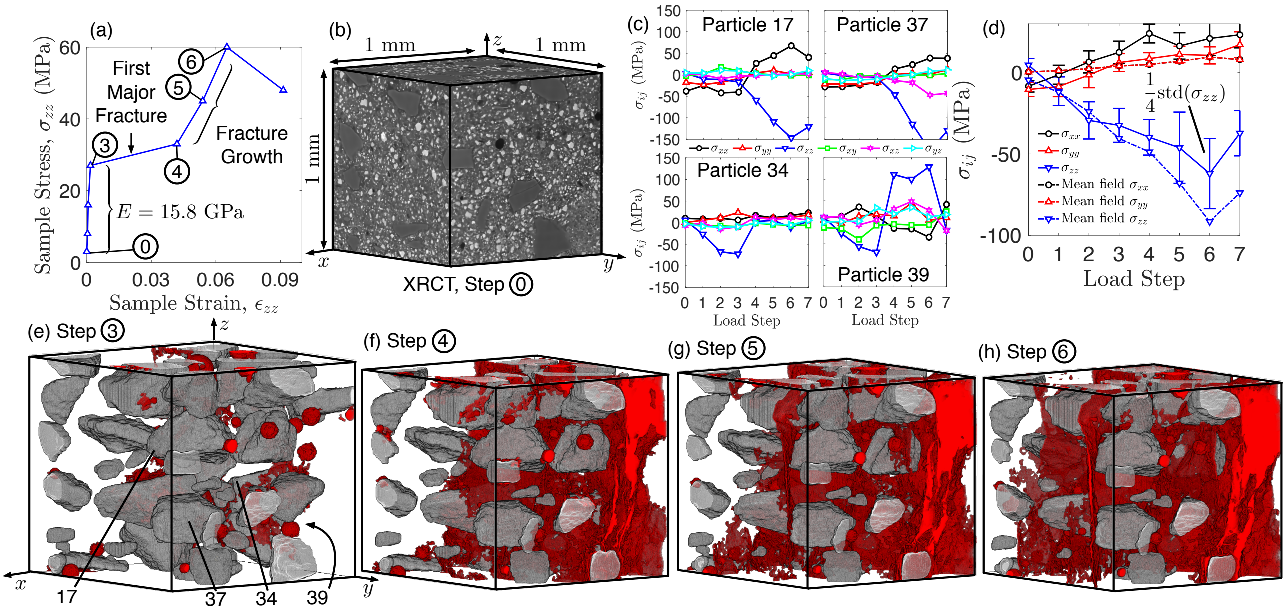

Figure 1: (a) Stress-strain curve for the sample subjected to unconfined uniaxial compression. (b) XRCT image at load step 0 obtained prior to imposing sample strain. (c) Stress tensor components, σ_{ij}, within four particles (identified within the sample in (e)), found from 3DXRD. (d) Average stresses (solid lines) in all aggregates for which stress measurements are available from 3DXRD. Error bars are $\frac{1}{4}$ of the standard deviation of σ_{ij}. Dashed lines are a mean-field estimate of particle stresses, assuming no aggregate interactions [3]. (e) through (h) show XRCT images in which aggregates (grey) and fractures or void space (red) have been segregated, illustrating the nucleation and growth of fractures through the sample.

3. RESULTS

Figure 1(a) shows the stress-strain response of the concrete sample throughout the experiment. The sample was subjected to strain in seven increments, after each of which XRCT and 3DXRD measurements were made. Salient features of the sample response include an initially elastic response, the development of a major fracture, and steady fracture growth. Figure 1(b) shows an XRCT image of the sample at load step 0, illustrating the presence of voids, aggregates, and cement paste (containing hydrated and unhydrated cement particles). Figure 1(c) shows the evolution of intra-particle stress tensors in four aggregate particles whose location within the sample is shown in Fig. 1(e). Significant stress fluctuations occur in these aggregates, particularly after the first major fracture emerges in step 4. Figure 1(d) shows stress components, σ_{ij}, averaged across ~40 sand particles, which are seen to agree on average with a mean-field estimate [3, 5], but demonstrate dramatic heterogeneity across aggregates. Figures 1(e) through 1(h) illustrate the growing fracture network (in red) as it develops in step 4 and spreads throughout the sample as it is strained.

References

[1] E.N. Landis, T. Zhang, E.N. Nagy, G. Nagy, & W.R. Franklin. Cracking, damage and fracture in four dimensions. *Materials and Structures*, 40, 2007.

[2] C. Poinard, W. Piotrowska, Y. Malecot, L. Daudeville, & E.N. Landis. Compression triaxial behavior of concrete: the role of mesostructure by analysis of X-ray tomographic images. *European Journal of Environmental and Civil Engineering*, 16, 2012.

[3] M. Königsberger, B. Pichler, & C. Hellmich. Micromechanics of ITZ-aggregate interaction in concrete part I: stress concentration. *Journal of the American Ceramic Society*, 97(2), 2014.

[4] J. Oddershede, S. Schmidt, H.F. Poulsen, H.O. Sorensen, J. Wright, & W. Reimers. Determining grain resolved stresses in polycrystalline materials using three-dimensional X-ray diffraction. *Journal of Applied Crystallography*, 43(3), 539-549, 2010.

[5] R.C. Hurley, & D.C. Pagan. An *in-situ* study of stress evolution and fracture growth during compression of concrete. *International Journal of Solids and Structures*, In Review, 2019.