ESRF	**Experiment title:** Quantifying Microscale Stress and Strain Fields in Cementitious Composites	**Experiment number:** Ma4978
Beamline:	**Date of experiment:** from: 23/2/2022 to: 28/2/2022	**Date of report:** 2/27/2023
Shifts:	**Local contact(s):** Pierre-Olivier Autran & Jon Wright	*Received at ESRF:*

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Report:

Objective – The aim of this experiment was to use a combination of *in-situ* x-ray tomography (XRT), 3D x-ray diffraction (3DXRD), and scanning 3DXRD (s3DXRD) at the ID11 beamline of the ESRF to study the microscale stress and strain fields generated during elastic and inelastic deformation of cement samples containing single-crystal sand. The goals of studying such microscale fields were to (1) examine stress variability within aggregates which would imply deviation from well-established Eshelby inclusion theories, (2) examine strain variability within cement paste surrounding aggregates to quantify stress/strain concentrations which nucleate macroscopic sample failure, and (3) compare experimental measurements with mesoscale simulations for model validation and development of a robust virtual testing environment. The experiments build on progress by the applicants in employing and integrating XRT, 3DXRD, and s3DXRD measurements at the ESRF (ma3373, ma4200, ma4493) [1]–[4]. The experiments also build on mesoscale modeling capabilities developed in Hurley's lab at JHU [5], [6].

Experiment Summary – Three experiments combining XRT, 3DXRD, and s3DXRD were successfully executed on distinct samples of cement containing single-crystal quartz aggregate. The quartz aggregate volume fraction varied in the three samples from 20 to 30%. The samples were compressed in strain increments in a custom load frame mounted on the rotation stage at ID11. Figure 1(a) shows the load frame, Figure 1(b) shows a cross section through an XRT image of one sample, and Figure 1(c) shows the load frame mounted at ID11. For each of the three samples, between 5 – 8 strain increments were executed and XRT and 3DXRD were performed between each strain increment. s3DXRD was performed before any strain increment in each sample and at two or three subsequent strain increments.

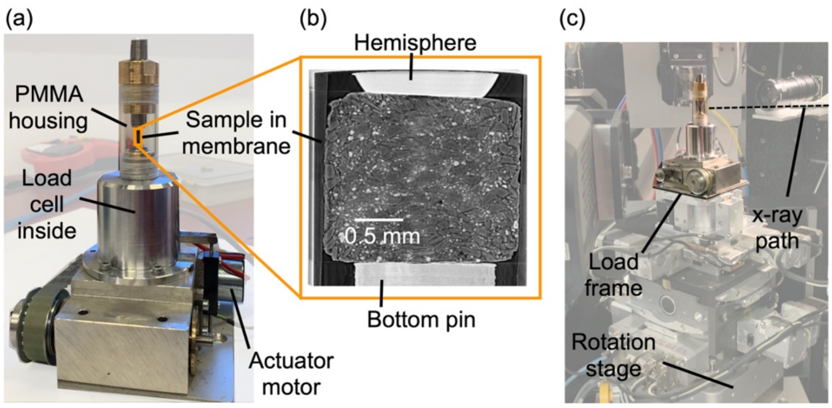

Figure 1 – (a) Load frame with sample. (b) Vertical slice through XRT image of one sample. (c) Load frame at ID11.

Results – Significant progress has been made toward goal (3). A paper has been published in *Cement and Concrete Research* which employed the XRT and 3DXRD data from ma4978 to validate a mesoscale model of one of the cement samples and subsequently use the mesoscale model to understand the effect of microstructural property heterogeneity on the modeled sample response. This integration of XRT and 3DXRD data and mesoscale modeling has employed the highest fidelity data available to-date on cementitious composites and has provided new insight into robust modeling approaches for such materials. Figure 2(a) provides a comparison of the experimental stress-strain curve and mesoscale simluation results with various levels of microstructural property heterogeneity for one sample. Figure 2(b) provides an illustration of the XRT processing pipeline for the same sample. Figure 2(c) provides an illustration of the finite element mesh of the sample. More details are provided in the paper published in *Cement and Concrete Research* [6].

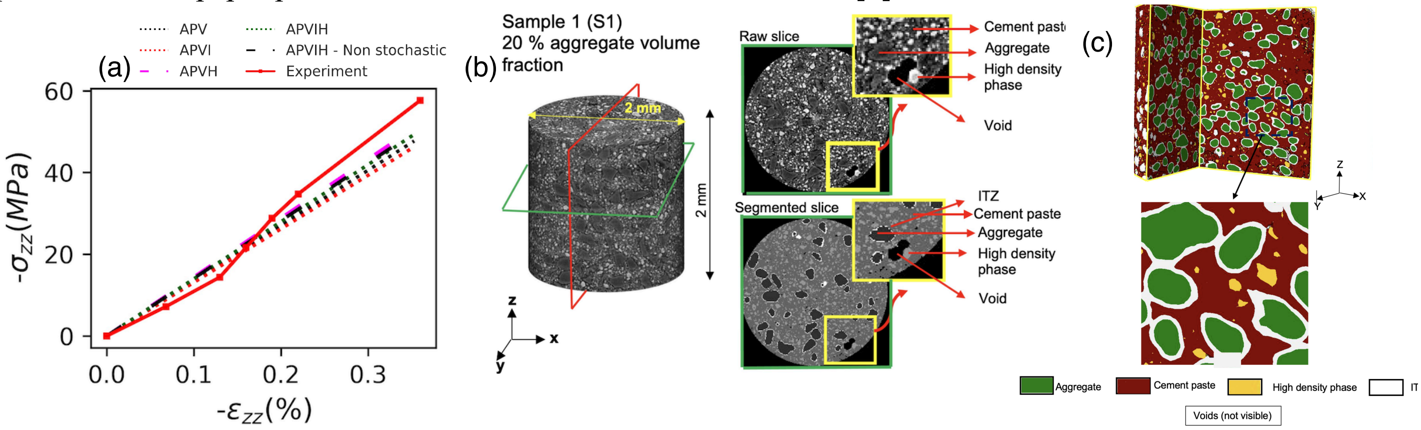

Figure 2 – (a) Experiment vs. mesoscale modeling response for one sample. (b) XRT image, slice through the image, and segmentation of the image for one sample. (c) Finite element mesh of a the sample.

Progress towards research goals (1) and (2) is progressing well. We have reconstructed strain and stress fields within aggregates examined using s3DXRD and are in the process of registering this dataset with 3DXRD and XRT datasets. Figure 3 shows an example of the reconstructed vertical strain (zz, or in the loading direction) in a central region of aggregates in the same sample showed in Figure 2. The s3DXRD measurements were made with a resolution of 20 microns – i.e., the size of the x-ray beam was 20x20 microns and the voxels in Figure 3 represent cuboid regions of aggregates 20x20x20 microns in size. The data provided by this reconstruction is the highest fidelity to-date on intra-aggregate strains and ongoing analysis is expected to provide important insights into micromechanics theories for concrete and cementitous composites. We anticipate finishing work with this dataset by summer 2023.

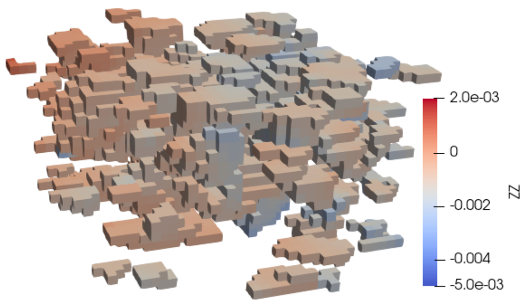

Figure 3 – Reconstructed vertical strain fields measured using s3DXRD.

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