ESRF	**Experiment title:** **In situ study of dynamic mechanical behavior and fatigue damage evolution of Shale rocks under cyclic loading and unloading**	**Experiment number**: ES 992
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Report:

For the advancement of civil engineering, it is essential to comprehend the mechanical behavior of natural soils when blended with cement for stabilization. Despite the critical necessity of comprehending fatigue in these widely used construction materials, the behavior of cement-soil admixtures when subjected to cyclic stresses is a subject that has received little research. A potent method for studying geomaterials in 4D—that is, 3D and time—is time-resolved micro-computed X-ray tomography (CT). We describe triaxial testing studies with cyclic loads on Portland cement mixed with broken shale fragments under synchrotron-based CT control. We obtained the temporal evolution of the displacement vector field by digital volume correlation (DVC), from which we further deduced the volumetric and von Mises equivalent strain fields. We monitored the evolution of fatigue damage under cyclic loading in unprecedented detail and discovered that the ultimate failure of the samples took place within the cement matrix after high-strain compression of the considerably softer shale particles.

To execute this experiment we used three samples made up of Portland cement and Draupne shale(obtained from the North). Two samples namely A1 1-2 mm diameter shale particles and A2 with >2 mm diameter shale particles mixed with Portland G cement. The third sample B was prepared without cement, thus with only Draupne shale fragments of diameter 1-3 mm, which were compacted inside the rubber sleeve used in the experiment. Brine solution (3.7 wt%) prepared using table salt (NaCl) was used as a pore fluid for all the experiments. The samples were saturated after installation in the sample cell, to be tested under realistic geotechnical conditions.

The measurements were carried out using a polychromatic "white" X-ray beam with a representative photon energy of 70 keV, collimated to a rectangular cross-section covering the whole sample. During each CT scan, the sample stage with the entire triaxial cell was rotated from 0 to 180 degree while the applied axial and confining stresses were maintained constant. Each scan lasted around 90 s and series of 2500 radiographic transmission exposures of the three-dimensional sample were recorded. The voxel size of the reconstructed tomograms was 6.5 micrometres, with a spatial resolution between two and three times the voxel size.

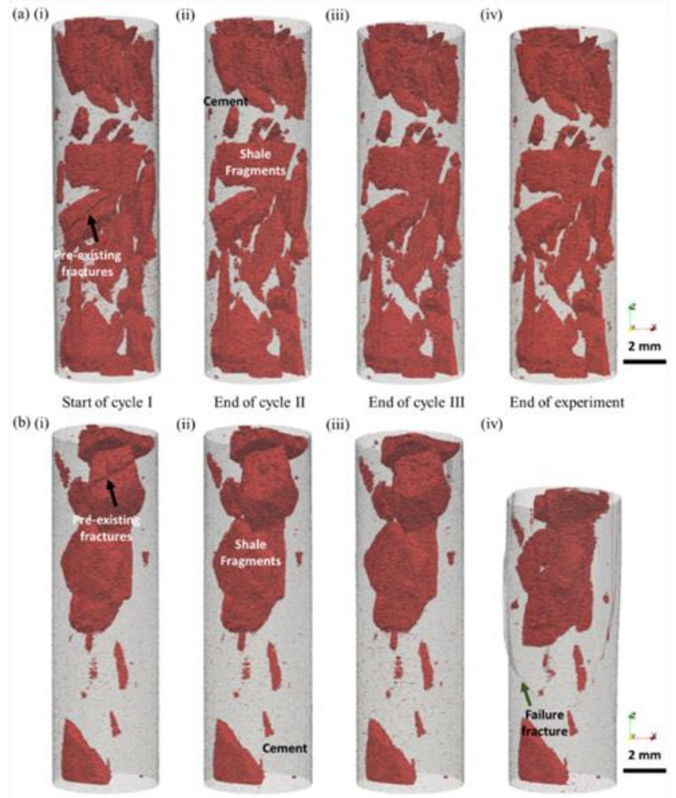

FIG. 1.: Perspective view of segmented shale fragments within samples a) A1 and b) A2. The segmented shale particles are represented in red, and the cement is represented in grey. The black arrows indicate a pre-existing fracture present in the samples prior to the experiment. The green arrow in (b-iv) points to the final fracture at the end of the experiment.[1]

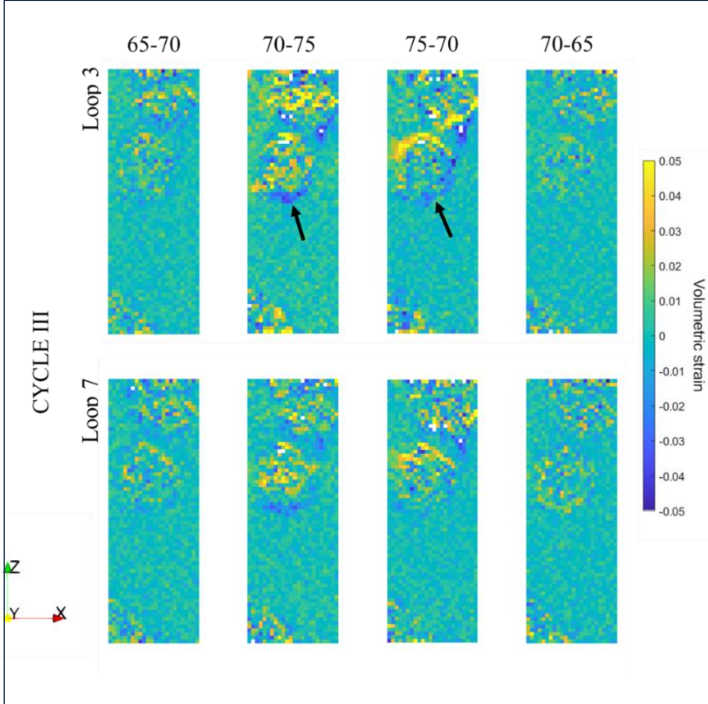

FIG. 2. Evolution of volumetric strain for cycle III, loops 3 and 7, for sample A2. The black arrows point to dilation bands in the shale regions of the sample.[1]

Our experiment aimed to explore the mechanical properties of shale particles often found in construction sites when mixed with cement, a common practice in large infrastructure projects. We conducted cyclic triaxial loading compression tests on Portland G cement-shale composites under geotechnically relevant in situ conditions. Using X-ray micro computed tomography and digital volume correlation (DVC), we analyzed damage and fatigue evolution in the samples. Our findings provide detailed insights into fatigue progression and failure mechanisms. The fatigue investigation methods and analysis techniques we employed reveal local in-situ mechanics within the shale-cement blend. These insights have potential applications in assessing the long-term stability of these materials in civil engineering projects like buildings, roads, dams, and even CO2 storage. We have compiled and submitted these results for publication in a prestigious journal [1], with hopes of a prompt acceptance. We also express our gratitude to ESRF for their beamtime allocation and technical support.

[1] Aldritt Scaria Madathiparambil, Fazel Mirzaei, Kim Robert Tekseth, et. al. Mechanical response of cement and shale admixtures under cyclic triaxial loading monitored by in-situ synchrotron micro-computed tomography, 2023, **(Manuscript Submitted)**

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