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Summary: Salt weathering drastically reduces the durability of building stones. A thorough understanding of the coupled transport-crystallization-mechanics phenomena is therefore indispensable. Building on previous work in which neutron radiography was employed, we now focus on a more profound characterization of the micromechanics of crystallization-induced fracturing by DVC analysis of a 4D X-ray tomographic dataset.

1. INTRODUCTION

Salt crystallization is a major cause of weathering of building stones. When saline solutions are present inside the stone, changes in climatic conditions (temperature, humidity), may induce the precipitation of salts. If this occurs within the pore space, the salt that crystallizes may cause a build-up of crystallization-induced stresses in the solid matrix, which can lead to cracking and spalling, thereby drastically reducing the stone's durability.

In order to prevent or remediate salt weathering of stone, it is essential to comprehend the coupling between transport, precipitation and mechanics in the stone. Derluyn et al. [1] designed an experimental campaign elucidating on this coupling, graphically illustrated in Figure 1a. They quantified the drying process and the crystallization-induced deformations simultaneously in a porous limestone using high resolution neutron radiography. Samples were prepared to create one-dimensional liquid transport and to induce a localized crystallization zone within the sample. NaCl was found to be particularly damaging, already after one wetting-drying cycle, cracking occurs. Although the quantification of fluid flow and precipitation is sufficiently detailed with neutron radiography, the resulting deformations can only be assessed globally as the change of the total area occupied by the sample in the neutron radiograph (Figure 1a bottom). X-ray scanning of a damaged sample reveals however a complex crack pattern (Figure 1a top) and to advance our understanding of crystallization-induced fracture dynamics, the current study addresses its micromechanical analysis.

2. EXPERIMENTAL METHOD

A cylindrical sample of 8.4 mm in diameter and 9 mm in height was cored from a Savonnières limestone plate, perpendicular to its bedding plane. A hydrophobic treatment (SILRES® BS 280, Wacker) was applied on the top surface and the sample was sealed circumferentially with aluminum tape to avoid lateral drying-out. The sample was saturated by capillarity with a 6.1 molal NaCl solution, after which the bottom surface was sealed with tape as well.

The saturated sample was then dried in a conditioned cell at a temperature of 26 ± 1°C and a relative humidity of 5 ± 1%, placed in the EMCT X-ray scanner of the Centre for X-ray tomography at the Ghent University [2]. The first 19.5 hours of drying were imaged at a time interval of 30 minutes and the further 24 hours at a time interval of 1 hour. For each scan, 1501 projections were acquired over an angle of 360° at a tube voltage of 80 kV, with an exposure time of 200 ms per projection, and averaging over 2 projections at each angle position. The projections were binned by a factor 2x2 in order to increase the signal-to-noise ratio. The raw data were reconstructed with the 4D suite from Tescan XRE, resulting in volumes with a voxel size of 9.4 µm.

The build-up of local deformations during the drying process was quantified by means of digital volume

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correlation (DVC). The local 3D structural changes between the initial scan and the consecutive scans were calculated with the open-source DVC analysis software TomoWarp2 [3]. A correlation window size of 10 voxels and a node spacing of 10 voxels ensured an optimal volume correlation for the dataset at hand.

3. RESULTS

The local deformations obtained through DVC are represented in Figure 1b for one vertical cross section of the sample, overlaid on the microstructure of the stone as obtained from the X-ray visualization. The deformations are represented here as local volumetric changes, i.e. calculated as volumetric strains. Dilation results in positive values (red color), compression in negative ones (blue color). In the selected cross section, cracking initiates around a hollow ooid and then further propagates mainly around the ooidic structures, and from time to time also crossing an ooid grain. These crack paths are congruent with the preferential salt precipitation sites in the pore space of Savonnières limestone, as previously identified by synchrotron X-ray tomography [4]. The DVC analysis identifies an initial development of one primary crack (first 7 hours in Figure 1b), whereas later in the drying process, secondary crack branches develop from this initial main crack.

Further processing of this dataset is ongoing, with the aim of advancing the micromechanical description of crystallization-induced damage. The complete set of results will be presented at ICTMS.

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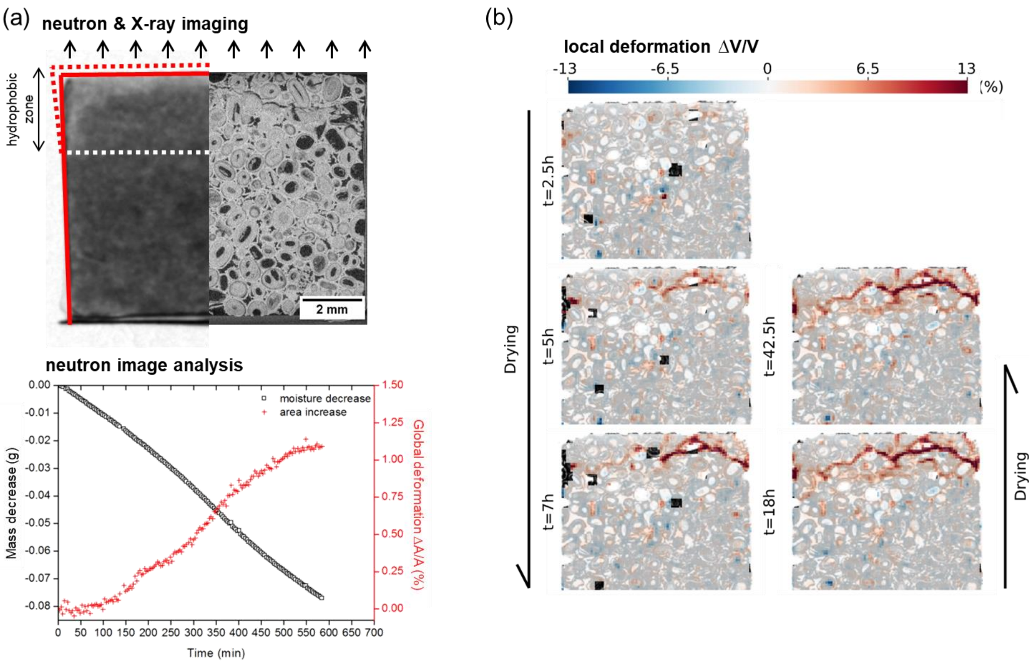

Figure 1: (a) Top: Drying of a Savonnières limestone sample, hydrophobically treated on the top and initially saturated with a highly concentrated sodium chloride solution, visualized by time-lapse neutron radiography. Drying could only occur via the upper surface, all other sides were sealed. The sample deformed during the test, as indicated by the red lines, due to NaCl precipitation in the hydrophobic zone. After being-dried out, X-ray micro-tomography revealed the presence of a crack in the upper volume. Bottom: From the change in attenuation values of the neutron radiographs, the mass decrease could be quantified, and from the 2D dimensional changes, the global area increase due to the deformations [1]. The deformations are caused by the displacements induced by crack formation due to salt crystallization. (b) Local deformations quantified with digital volume correlation from a 4D X-ray dataset acquired during cracking of a Savonnières limestone sample induced by localized precipitation of NaCl close to the upper surface (red zones represent high volume increase, i.e. cracking).