

## Time-resolved and Multi-modal Evaluation of Building Stone Weathering – New Advances in 4D Imaging and Analysis

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With the arrival of faster image acquisition and the ability to acquire time-resolved or 4D images (Bultreys et al., 2015) using X-ray microtomography, the need for new visualization and analysis arises. In this work, we show a detailed workflow for the imaging and analysis of gypsum crust formation and growth on a Belgian limestone (Lede stone; De Kock et al., 2017). The workflow uses a novel approach to gather information from hundreds of 3D dataset, and quickly evaluate and show results in an intuitive way.

The degradation of limestone, and formation of a superficial gypsum crust, is an aesthetic and structural form of weathering of building stones, that often occurs in urban environments. Although a lot of work has been done on the study and imaging of this gypsum formation (Charola et al., 2007; Dewanckele et al., 2013), we present a true dynamic workflow that follows the formation of gypsum crystals and the degradation of the calcite in real time. Over the course of almost 3 days (68 hours), 138 full tomograms of 30 minutes each, at a voxel size of 5  $\mu\text{m}$ , were acquired of a small Lede stone plug, while exposed to an acidic environment. Combining the information of all those datasets, a 3-day movie of the formation of the gypsum crust can be generated (Figure 1).

As each dataset of the experiment represents a few gigabytes of data, a traditional image analysis approach, including registration, differential imaging, image segmentation and analysis would be very time-consuming and computationally intensive. Furthermore, creating an intuitive visual representation, that enables to instantly understand and convey the results, is an additional challenge. Therefore, a new tool was developed to determine the exact time point where each individual voxel of the dataset flips over from one phase to another (i.e. the flip-point). This allows to pin-point the exact time step where each voxel changes from air to crust. Additionally, it allows one to efficiently detect where and when the original rock-material is dissolved, and new (micro-)porosity is formed. By combining all the flip-point information, a single, comprehensive image can be formed, that shows the time of change for any point on the 3D volume, in a single glance (Figure 2).

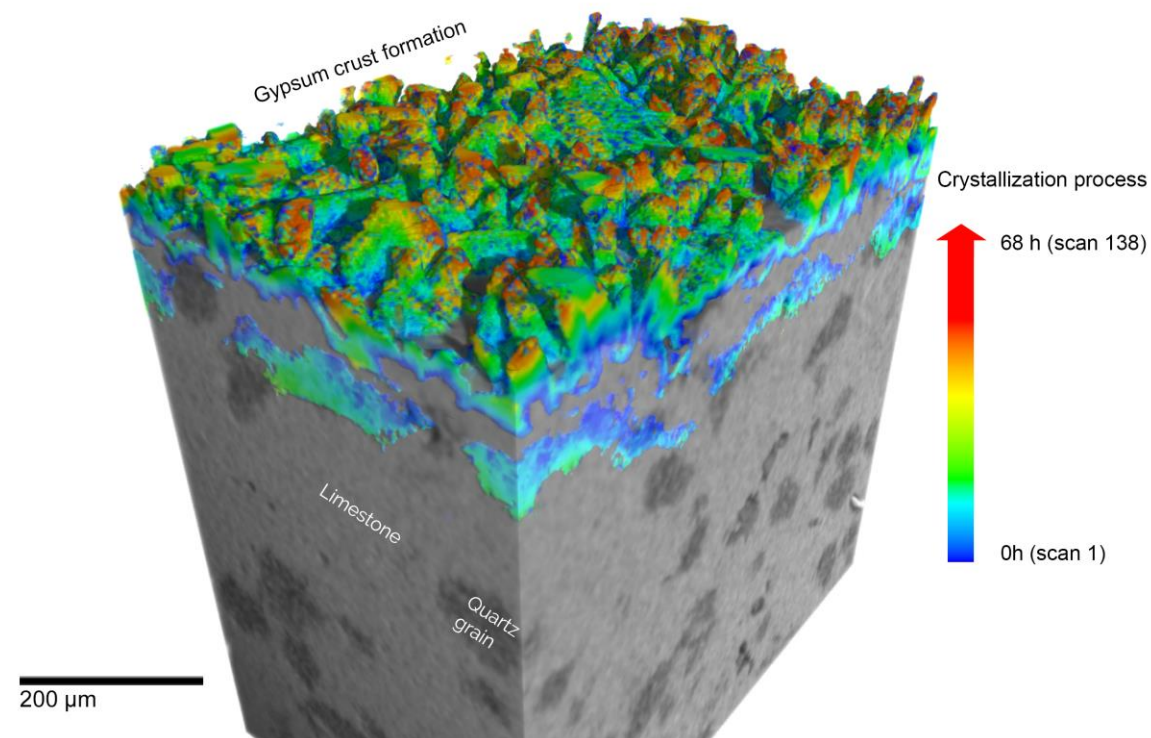
Although the formation of a gypsum crust on building stones is an aesthetical problem, the main structural degradation of the rock is the formation of a weathered, porous zone below the surface of the material and below the gypsum crust. Computed tomography provides a useful overview on which zones are affected by the porosity formation and loss of material. In order to proceed with a detailed quantification of the microporosity of the Lede stone before and after weathering, Focused Ion Beam tomography (FIB-SEM tomography) was employed to complement the micro-CT data. FIB-SEM datasets were acquired on the surface of the rock prior to the weathering experiment. After the completion of gypsum formation, the new, weathered surface of the sample was re-exposed, by dissolving the gypsum in water. This enabled the acquisition of a new FIB-SEM dataset, right next to the original one, in order to visualize the newly formed micropores and overall degradation of the rock.

The presented work shows a unique real-time pore-scale insight in the origin of a well-known problem on historic building materials. By combining dynamic X-ray tomography with state-of the art FIB-SEM, the

influence of local microstructure on the weathering behavior of the Lede stone can be imaged and analyzed.



**Figure 1.** 3 time steps out of 138 full X-ray microtomography datasets that show the formation of gypsum crystals on the Lede stone.



**Figure 2.** Comprehensive representation of the flip-point analysis of the 4D experiment. Each altered voxel is color-coded with respect to the time of change.

#### References

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