

## Plasma focused ion beam microscopy: A novel tool to characterize 3D structure and porosity of OPC mortar

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### ABSTRACT:

Ordinary Portland cement (OPC) and its concretes have been used in construction industry for more than 100 years. The permeability and corresponding transport properties of OPC mortar or concrete are largely determined by pore characteristics, including porosity, connectivity, and pore throat size. Many techniques such as scanning electron microscopy (SEM), or atom force microscopy (AFM) can provide information even at nanometer range. But they are limited to 2D range, which is usually misleading. Transmission electron microscopy (TEM) tomography and atom probe tomography allow 3D analysis of structure at even higher resolutions. The sizes of volume of interest (VOI) in above techniques are not representative though, and any meaningful measurement would require a large amount of repetitive experiments. The X-ray computed tomography (CT) is applicable to bulk samples, while the resolution is limited to micrometric scale and relies on absorption contrast which gives little information about chemistry. This is not enough to analyze nanoscale pores distribution and materials chemistry. Finally, mercury intrusion porosimetry (MIP), a traditional technique used in cementitious materials, enables the analysis of pore size distribution. However, the results possess an overestimation of small pores due to the ink-bottle effect [1].

Focused ion beam nanotomography (FIB-nt) allows insights into 3D structure with relatively large volume and a nanoscale resolution, which is suitable to characterize pore system in different scale. By alternating imaging a block face of material, following by slicing that material using an ion beam, a serial sectioning tomography method is possible. New plasma sources that use Xe<sup>+</sup> ions produce a higher milling rate than traditional Ga liquid metal ion sources make 3D tomography with volumes up to hundreds of microns in higher milling rate with better surface finish. [2, 3]. Using

a Xe<sup>+</sup> Plasma FIB at the Canadian Center of Electron Microscopy (CCEM) at McMaster University, we will conduct 3D tomography on an OPC mortar specimen using Plasma FIB and show the feasibility of acquiring large volume, high resolution tomograms of cementitious materials. The 3D data will be aligned and segmented using software. The pore characteristics of pore size distribution, pore connectivity and pore throat will be shown based on segmentation model.

#### REFERENCES

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