

## Investigation of reactive transport processes in porous mineral systems by means of Focused Ion Beam

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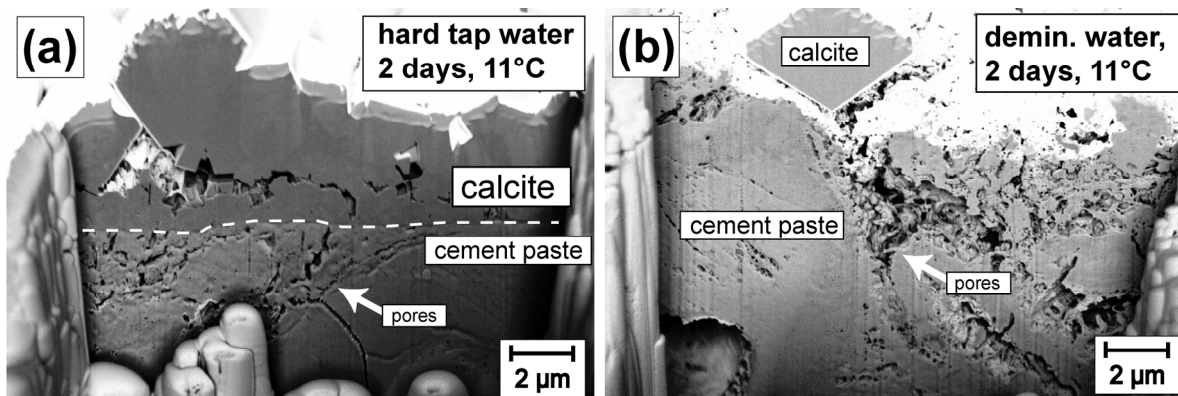
Porous mineral materials such as cement or gypsum based systems are used in construction and in various technical applications, e.g. molding processes. In practice material failure is not only caused by mechanical load but also by exposure to aggressive environments. These materials often are multi component systems with a pore system characterized by pore sizes ranging from the nanometer up to the millimeter scale. Under common climatic conditions, the pore system contains an aqueous solution with a complex composition. The chemical stability of these porous mineral systems is generally determined by chemical equilibria between the pore solution and the solid phases. It's inevitable that these equilibria will be disturbed during the use of the materials. Due to concentration gradients between the pore solution of these highly porous materials and a surrounding aqueous media reactive transport processes are initiated. The consequence is that the chemical equilibrium between the pore solution and the solid phases is disturbed and thus dissolution and crystallization reactions are triggered. This holds especially for reactive systems such as cement based materials.

In order to investigate the material behavior of cementitious systems usually the long term behavior, (weeks and months), of these materials is studied. These processes occur at the material/water interface starting immediately after coming in contact with the aggressive environment. A detailed knowledge of the reaction mechanisms of the reactive transport processes is a necessary precondition to develop reliable prognostic models with respect to the durability of materials. In order to investigate the relevance of chemical and micro structural changes in the material/water interface short term experiments were carried out in this study.

The focused ion beam preparation technique enables detailed investigations of surface reactions by means of electron microscopic methods. A scanning beam of Gallium ions is applied in order to remove material from the sample surface with nanometric spatial precision. Utilizing this method profile cuts are performed in order to investigate depth dependant chemical and structural properties in the areas nearest to the surface, (~20  $\mu\text{m}$ ).

In this study results of laboratory experiments are presented. Freshly cut surfaces of cement paste samples prepared with white Portland cement with a water/cement ration of 0.4 were exposed to aqueous solutions with different chemical compositions. Cross-section-cuts with a depth of several tens of microns were performed (Figure 1). The pore system and the chemical composition of the cross-section profile were studied. The results show that formation of crystalline phases on materials surface, e.g. calcium carbonate layers, on the alkaline surfaces of the cement based materials do not always act as a protection against chemical attack (Figure 1a). In certain cases crystal growth on the surfaces causes a further degradation of the materials (Figure 1b).

In summary the properties of the material/water interface are crucial when considering the durability of cement based systems in contact with aggressive aqueous solutions. Focused Ion Beam is a versatile tool for the elucidation of the interrelationship between changes in chemical composition and structural properties.



**Figure 1.** Electron microscope investigation of Focused Ion Beam cuts in the cement paste/water interface of a sample exposed for two days at 11°C to (a) hard tap water and (b) demineralized water.