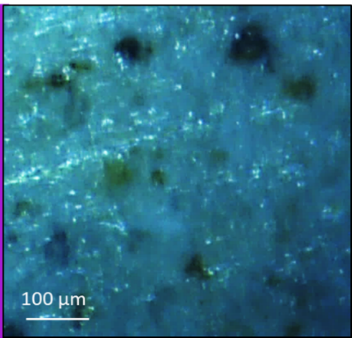
Application Note

Tracking Temperature-induced Nano-structural Changes of Concrete by High-temperature Nanoindentation



Introduction

Cement-based materials are composed of different phases with largely varied compositional changes throughout the microstructure that reflect the mechanical properties of cements in both the nano- and micro-scales. Macroscopic mechanical performance of cements is therefore highly dependent on the variation in mechanical properties within the microstructure[1]. Nanoindentation is a very powerful tool in measuring the microstructural mechanical properties of cement-based materials and in quantitatively evaluating microstructural modification in the mechanical properties of newly developed cement-based materials. The growing application of nanoindentation techniques to the analysis of cement-based multi-phase materials increasingly attracts researchers in this field to develop new analysis methods for understanding length-scale effects and how micromechanical properties dominate deformation behavior at the macro-scale. Valuable microstructural mechanical information (e.g. Young's modulus, hardness and creep compliance) of cement paste has been reported during the last decade[2-5]. Another important area of research in cement paste materials is the effect of high temperature on mechanical properties due to multiple phenomena occurring during the heating of cement. Complex physical and chemical changes take place in concrete during the heating process, due to the gradual evaporation of water and the binding changes among other solid phases. In addition, the mass liquid or gas movements generate internal stresses that result in creating cracks that can propagate throughout the microstructure and ultimately damage the concrete[6].

This application note focuses on the nanomechanical properties of Ordinary Portland Cement (OPC) paste at elevated temperatures. The Express Test method of the KLA Nano Indenter® G200 with the laser-heated tip and stage option was utilized to measure the in-situ elastic properties of microscopic phases present in the paste while exposed to elevated temperatures up to 250°C. This in-situ characterization of the high-temperature mechanical properties of OPC paste enabled the use of statistical analysis to deconvolute the mechanical parameters of the individual active phases derived from a large number of nanoindentation tests.

Experimental Method

Cement samples were produced by the Department of Civil and Environmental Engineering at the University of Maine. Essential ingredients for OPC paste were mixed and then compacted into 1" cubes. The curing process took place at ambient temperature over three months. The measurement samples were thin flat slices (1-2mm thick) that were cut from the cubes with a diamond saw, and measurement surface quality was ensured during the cutting process.

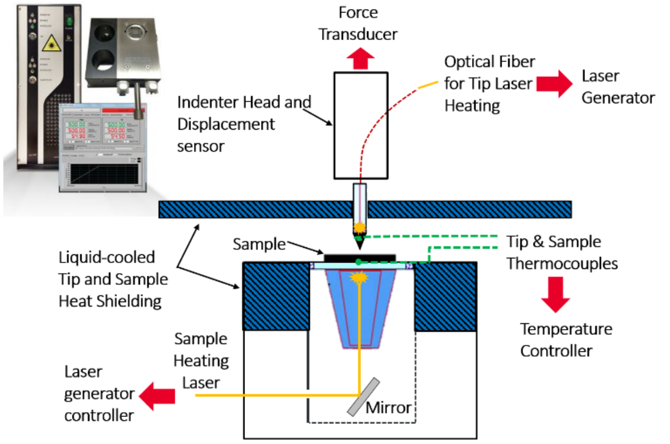


Figure 1. G200 laser heater system and a schematic of the high temperature nanoindentation setup.

The sample slices were mounted on the laser heater sample tray for nanoindentation tests. The G200 Express Test option with the laser-heated tip and stage was used to evaluate the elastic modulus and hardness of cement paste at elevated temperatures. Express Test rapidly acquired a large amount of data at each test temperature. Arrays of indents to a specified depth of ~200nm were performed for each individual temperature using a sapphire laser-heated tip installed on an XP head. Both sample and tip were independently heated to the testing temperature to ensure maximum thermal stability.

Both tip and sample temperatures were monitored during the test using the integrated thermocouples with the temperature control software. Figure 1 shows the G200 laser heater system and a schematic of the high temperature nanoindentation setup. The selected test temperatures were room temperature (23°C), 90°C, 150°C, and 250°C. At least 400 indentations were made on each OPC paste sample at each specified temperature. With the benefits of Express Test, fast sample/tip heating and temperature stabilization, each set of 400 indentations required only about 2 hours.

Express Test was also used to map the mechanical properties of cement paste at room temperature by making shallow indents (~250nm depth) with a small interspace (2µm) between indentations in the 100µm by 100µm area. Modulus maps align well with compositional changes in the microstructure while hardness maps track changes in material plastic response caused by processing methods. The statistical nanoindentation technique (SNT) was used to deconvolute the mechanical parameters of the individual active phases from the large amount of data[7].

Results and Discussion

Figure 2 shows the surface topography (left), Young's modulus map (center) and hardness map (right) of the OPC paste at room temperature. The inset at the top left is an optical image of the sample and shows the different phases. These high resolution maps reveal the details of compositional and/or structural variations within the microstructure, and the combination of soft and hard phases in the cement is clearly visible.

The nanomechanical properties of OPC paste, as obtained by Express Test measurements at different temperatures, are displayed in Figure 3. Using the statistical deconvolution process, five microscopic phases were identified. Each elastic modulus can be correlated to an individual phase, and corresponding chemical composition can be verified using SEM analysis[8].

From the express nanoindentation results of OPC paste and matching the modulus values with literature[8], typical hydration products were identified, including the porous phase, IP C-S-H, OP C-S-H, calcium hydroxide and calcium carbonate. At 90°C, the relative proportion of the phase where E = 39GPa increased, indicating stiffening of C-S-H. However, at 150°C, the proportions of the phase where E = 15GPa decreased, indicating that after the removal of physisorbed water, the C-S-H gel, in this case, loses some stiffness. At the temperature of ~250°C, it is expected that the physisorbed water of the C-S-H phase will be completely removed and only partial chemisorbed water will be removed.

Conclusions

In-situ nanomechanical measurements at elevated temperature revealed the effect of the moisture loss on intrinsic properties of both the microscopic phases and the overall OPC paste matrix. The application of nanoindentation provided a deep insight into the microstructural mechanical properties and their effects to improve the macroscopic mechanical performances of cement materials. Detailed

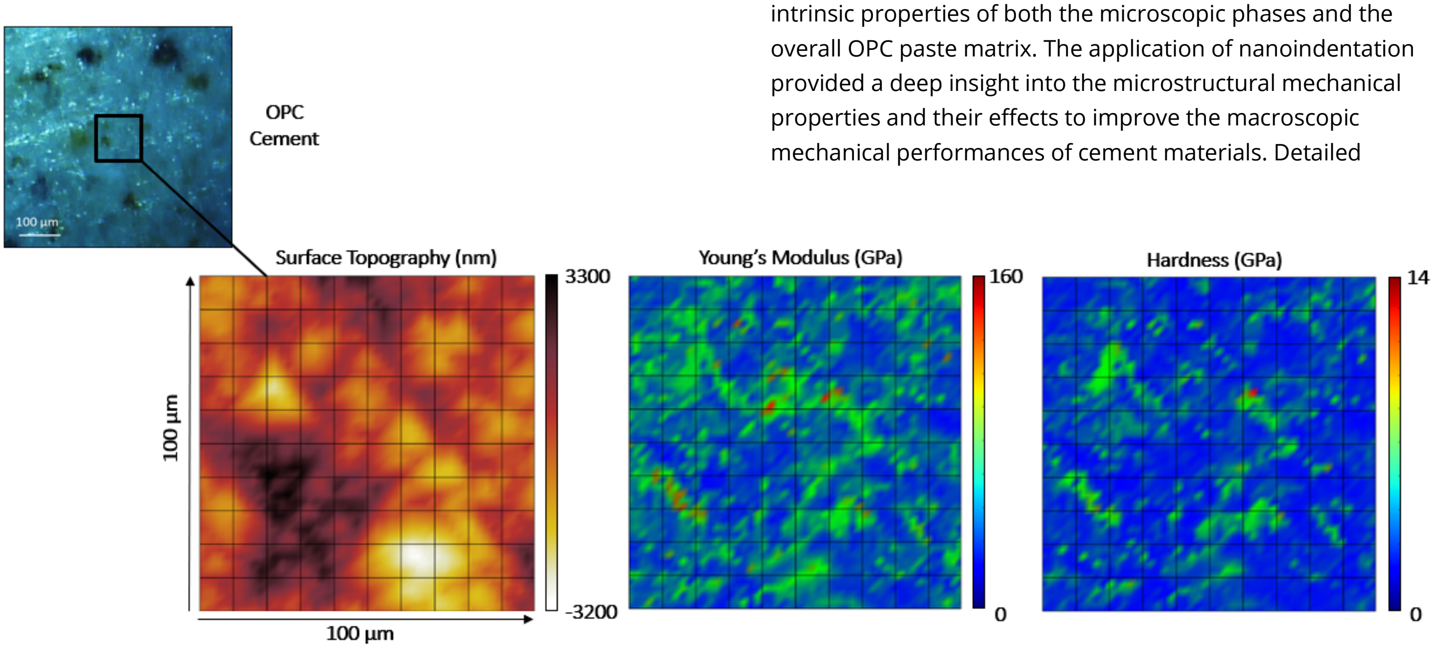


Figure 2. Surface topography (left) and distribution of Young's modulus (center) and Hardness (right) mapped for OPC paste at ambient temperature. The inset at the top left shows an optical microscope image of the polished OPC paste before nanoindentation.

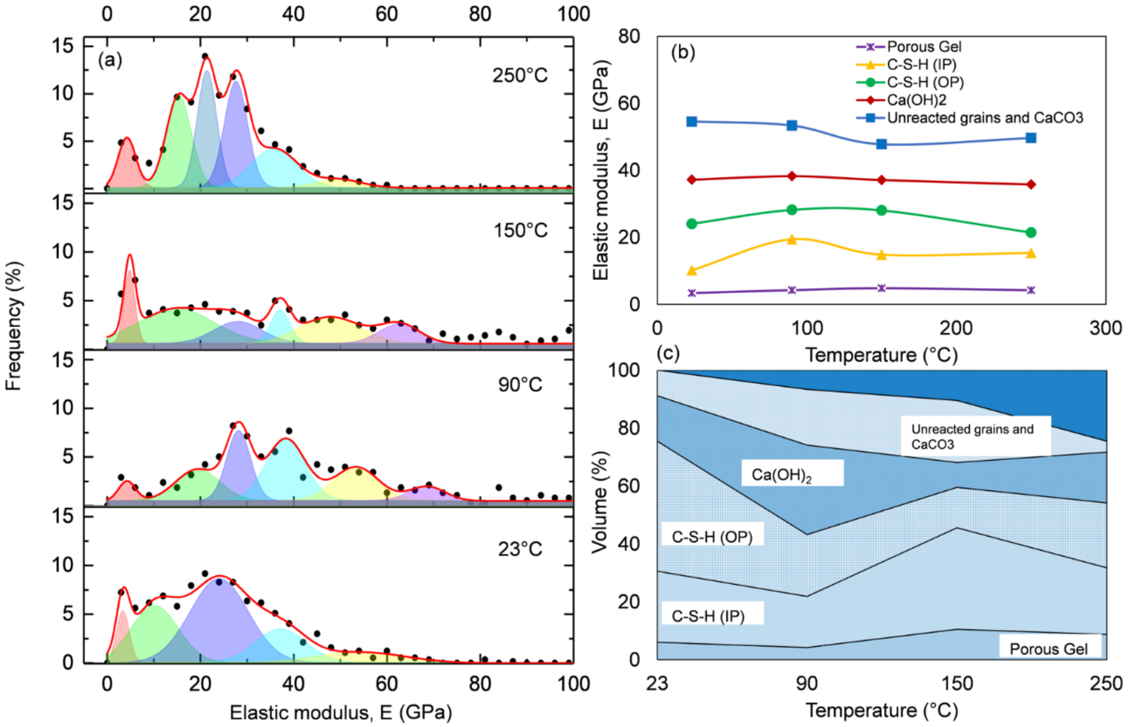


Figure 3. (a) Frequency distribution of elastic modulus for OPC paste at different temperatures; (b) variation of elastic moduli of the microscopic phases with temperature; and (c) volume fractions of the microscopic phases as obtained from statistical nanoindentation analysis.

characterization obtained from micro-scale analysis could also provide valuable data for multi-scale analytical and numerical modeling of concrete cement materials.

Acknowledgments

KLA gratefully acknowledges Dr. Warda Ashraf, University of Maine, Department of Civil and Environmental Engineering, for collaboration with the KLA Instruments group and providing cement paste samples for this research.

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Milpitas, CA 95035
Printed in the USA
Rev 4 2020-09-11