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MEASUREMENT OF DELAYED DEFORMATION AFTER NANOINDENTATION

MĚŘENÍ ZPOŽDĚNÉ DEFORMACE PO NANOINDENTACI

Abstract

In this study, elastic and inelastic properties of cement paste at microlevel were investigated using nanoindentation technique. Special attention was paid to delayed deformation processes after full unloading. Comparison with different types of materials (fused silica, copper and polyethylene) was also performed. The aim of the work was to determine how the material deforms after the load release as a stepping stone for separation of elastic, viscous and plastic parts of the deformation in the material. Such knowledge is very important for further modeling of nanoindentation and for accessing of inelastic material properties at cement microlevel.

Abstrakt

V tomto příspěvku jsou zkoumány elastické a neelastické vlastnosti cementové pasty na mikroúrovni s použitím nanoindentace. Speciální pozornost byla věnována zpožděné deformaci po úplném odtížení. Bylo provedeno srovnání s různými typy materiálů (silica, měď, polyethylene). Cílem práce bylo stanovit, jak se materiál deformuje po uvolnění zatěžovací síly jako první krok k oddělení viskózní a plastické části deformace. Znalost těchto deformací je velmi důležitá pro další modelování nanoindentace a pro stanovení neelastických vlastností cementu na mikroúrovni.

1 INTRODUCTION

Hardened cement paste is a heterogeneous material. Several phases can be distinguished at its microlevel. Namely, we can find products of hydration (C-S-H gels, Portlandite and others), unhydrated clinkers and pores. All of them occur at micron and submicron length scale.

Generally, we can distinguish between two types of deformations – reversible (e.g. elastic deformation or temperature change) and irreversible (cracking, chemical shrinkage, other inelastic deformations). All these deformations can be observed and also measured at macrolevel but for all phases’ compound. Slightly different situation is at microscale where some deformations, for example cracking, can be easily detected (e.g. by ESEM). Also different material phases can behave very differently. For instance, viscous deformation can be attributed almost purely to hydrated phases (mainly C-S-H gels) while clinkers are mostly elastoplastic. Avoiding some discontinuities (cracking) we can split microlevel deformations to elastic part that is fully reversible, plastic part that is irreversible and causes permanent deformation in time and viscous deformation that is time dependent. The question is how to separate them from microscopic measurements.

Overall microlevel deformations can be successfully measured by nanoindenter at its tip up to full load removal whereas deformations after unloading can be scanned with atomic force microscope

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(AFM) or other type of scanning probe microscope (SPM). Nanoindentation is the only experimental technique that enables direct measurement of mechanical response of individual material phases at submicron scale even for such heterogeneous system like cement paste [1]. In this process a very small tip is brought to the sample surface producing an imprint. Load versus depth of penetration diagram (Fig. 1) is measured through the whole loading, holding and unloading process. Loading and holding parts of the diagram contain elastic, plastic and viscous deformations whereas the unloading part is usually supposed to be elastic and elastic constants are extracted from this part using semi-analytical elastic solutions [2].

On the other hand, SPM provides exact three-dimensional morphological information of the material surface, (i.e. after indentation, possible fracture surfaces, cracks, etc.) and can be used to construct delayed recovery of the deformation.

Separation of individual types of the deformation is a difficult task even at microlevel due to complicated stress states under indenter probe during loading. However one can utilize simplified stress state after full unloading. At this stage, the delayed deformation recovery (caused mainly by viscoelastic effects) takes place.

2 SAMPLES

Cement paste sample was prepared for this study from ordinary Portland cement CEM I – 42.5 with water/cement ratio 0.4. The sample was cured in water for 34 days. Before nanoindentation the sample surface was polished to achieve a flat surface with maximum roughness 10-20 nm. For the sake of comparison, additional measurements were performed on fused silica copper and polyethylene samples.

3 METHODS

Nanoindentation was used for performing a single imprint and the final penetration depth (at load release) was taken as a base (time 0) for subsequent considerations. SPM called ‘in-situ imaging’ was used for surface scanning after the load removal. ‘In-situ imaging ‘is a patented technology of Hysitron® that uses the same tip (Berkowich in our case) for indentation as well as for surface scanning. Indents were scanned several tens of minutes after the load removal. The evolution of maximum depth was monitored and evaluated in time. Results were normalized with respect to the base depth (at time 0) for comparison purposes. Also final penetration depth was kept similar for all materials (close to 100 nm) for comparisons except for polyethylene (around 500 nm) where a smaller penetration depth was difficult to achieve.

4 RESULTS

Measurements proved the existence of the delayed recovery of the deformation on all samples. As expected, the smallest recovery (almost negligible) was observed on fused silica (Fig. 3). Cement sample evolved strongly in time and the deformation had descending (approximately logarithmic) trend. Similar behavior was found on copper, although the descent was less rapid and deformations stabilized relatively soon (Fig. 3). This could be attributed to the smaller viscous deformation of copper compared to cement paste. It can be supposed that the deformation approaches to the final plastic deformation that is irreversible and that was accumulated in the sample during the whole loading process. Polyethylene was found to have the largest delayed deformation recovery. It might be caused (i) by the viscous nature of the polyethylene itself and (ii) by the higher force (and corresponding stress) used for its loading.

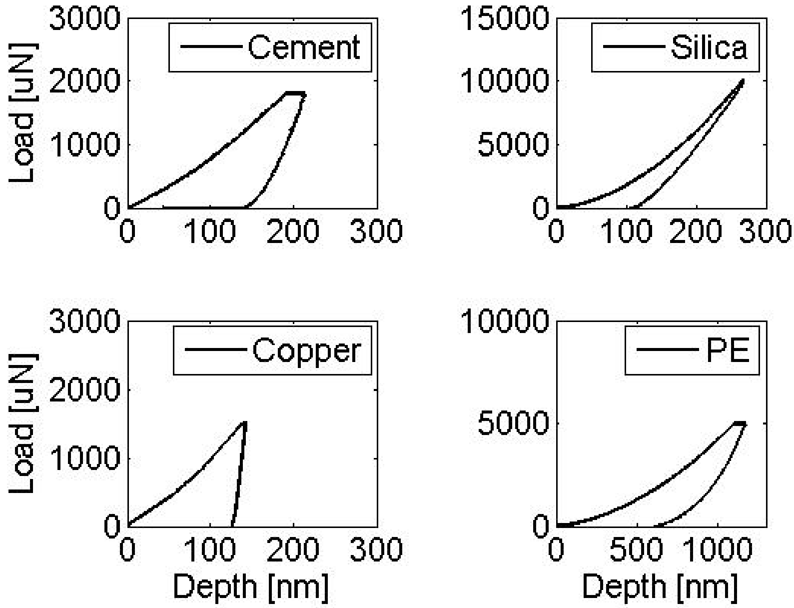

Fig. 1 Load vs. depth diagram from nanoindentation on cement paste, fused silica, copper and polyethylene.

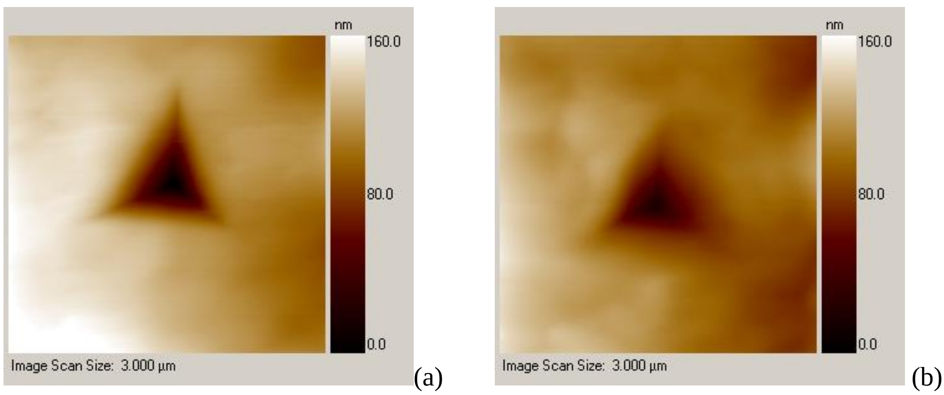

Fig. 2 SPM image of the imprint in time= 146 s (a) and in time= 4772 s after load removal (b) measured on cement sample

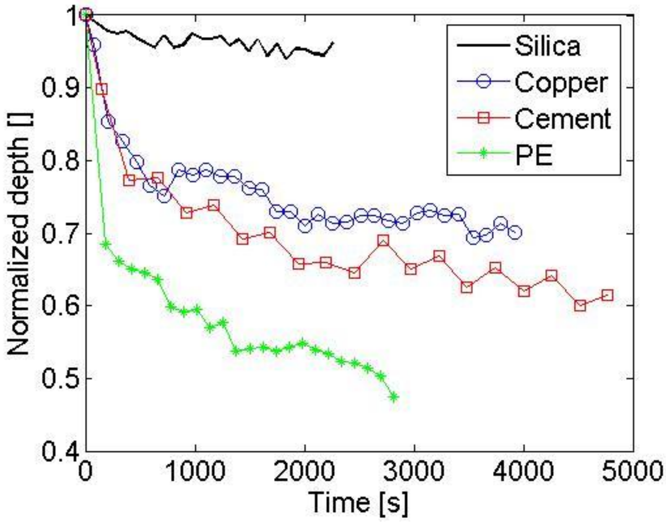

Fig. 3 Comparison of the delayed deformation recovery for fused silica, copper, cement paste and polyethylene after load removal.

5 CONCLUSIONS

The delayed recovery of the deformation after load removal was studied on four materials. It appears that fused silica shows a very little change in deformation and can be considered as almost non-viscous material. In contrast, cement and copper exhibit viscous behavior. For copper, the recovery was smaller and is in the order of minutes to an hour for the given load level. However, for cement sample the evolution of the deformation does not approach to the final value at this time period. This finding points out the higher viscosity of this material. Polyethylene sample was also studied and its behavior was found to have similar trend like for cement paste but the deformations were more pronounced. This study is a first step to the further research on cement samples that will lead towards separation of the viscous and plastic deformations and extraction of relevant parameters of constitutive models.

ACKNOWLEDGEMENTS

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