EXPERIMENTÁLNÍ VÝZKUM DOTVAROVÁNÍ CEMENTOVÉ PASTY NA MIKROÚROVNI

EXPERIMENTAL INVESTIGATION OF CREEP OF CEMENT PASTE ON MICROSCALE

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Abstrakt

Příspěvek pojednává o nanoindentaci na cementové pastě. Zvláštní pozornost je věnována efektu dotvarování a studiu vhodné metody pro vyhodnocení jejích mikromechanických vlastností. Nanoindentace je použita pro stanovení elastických i neelastických parametrů cementové pasty. Limity tradičního elastického řešení naznačují provedené experimenty s různými historiemi zatížení. Lepšího popisu indentačního procesu lze dosáhnout při použití viskoelastického řešení nebo s obecným viskoelastoplastickým modelem implementovaným do MKP modelu. Tyto modely byly použity pro odhad materiálových parametrů na mikroúrovni.

Klíčová slova: Nanoindentace, cementová pasta, dotvarování.

Abstract

This paper deals with nanoindentation of cement paste. In particular, creep effects are studied together with the appropriateness of methods used for evaluation of its micromechanical properties. Nanoindentation experiments are used for assessment of elastic as well as inelastic parameters of cement paste. Limitation of traditional elastic solution is indicated by experiments with different loading histories. Better descriptions of indentation process based on analytical viscoelastic solution and finite element model with general viscoelastoplastic constitutive relation are proposed. These models are used for simulation of indentation and for estimation of material parameters at micrometer scale.

Keywords: Nanoindentation, cement paste, creep.

INTRODUCTION

Experimental investigation and numerical modeling of cementitious materials is of prior importance in the building industry. According to the world trend the description of any material cannot be established without proper knowledge of the material microstructure and their properties at microscale. Several experimental techniques can be used for such investigation. Nanoindentation plays an important role among them [1]. This technique is based on the direct measurement of the load-displacement relationship using a very sharp diamond tip pressed into the material. The depth of penetration starts from the level of nanometers. Although, nanoindentation was originally developed and used mainly for studying homogeneous materials like metals, coatings, films, glass, and crystal materials, the evolution of this method allows us to use it also for materials like concrete and cement. The major studies can be found e.g. in [2], [3], [4]. However, the interpretation of measured data is more complicated due to the large heterogeneity of concrete

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and cement as well. In contrary to classical macroscopic tests, numerous microscale phenomena can occur during indentation tests. Thus, evaluation of nanoindentation results must be done with care since different interpretation based on the used model can be obtained. Creep of the material was found to be the main factor contributing to usual misinterpretations. Ignoring creep in the evaluation of results can lead to spurious size effect on elastic properties. Simulation of indentation process and comparison with experimental data can answer the question on the appropriateness of different constitutive relations and the underlying material behavior.

INSTRUMENTATION

For all measurements we used Nanotest nanoindenter (Micro Materials, UK). The indenter itself is a machine consisting of anti-vibration table, very stiff frame with a pendulum hanged on a frictionless pivot. The pendulum is equipped with a diamond tip on one side. A coil attracts the other side of the pendulum. The nanoindentation technique is based on the measurement of penetration of the diamond tip into the material. The diamond tip may have different shapes (pyramidal, spherical, flat...). For the present measurements we used so-called Berkowich indenter, which has a three-sided pyramidal shape with the side to edge inner angle $\approx 124^{o}$ and with a very sharp tip with the radius around 40 nm. The result of the measurement is a load versus depth of penetration diagram.

For microstructural analysis an environmental scanning electron microscope XL30 ESEM-TMP, Philips Ltd. was used. This microscope is able to work in high and low vacuum as well as in an environmental mode and it is suitable for testing of non-conductive samples like cement.

NANOINDENTATION OF CEMENT PASTES

Cement paste is a heterogeneous material. In this work, separation of material phases and micromechanical testing was done using nanoindentation and ESEM. Several tens of indents need to be done to capture heterogeneity of the material. Usually, indents produce a matrix as shown in Fig.1. Experimental results from nanoindentation cover the load vs. depth of penetration diagram (the P-h curve). This diagram contains loading and unloading branch and may contain also holding (dwelling) period at the peak of the diagram (Fig.2). Several types of experiments have been conducted using different loading histories including long dwell periods and cyclic loading. It was found that traditional methods of evaluation of elastic properties [6] are appropriate only in special cases of loading. In the case of time-dependent material, this model fails even in prediction of elastic properties. Therefore, more general model should be used for data analysis and for simulation of the indentation process.

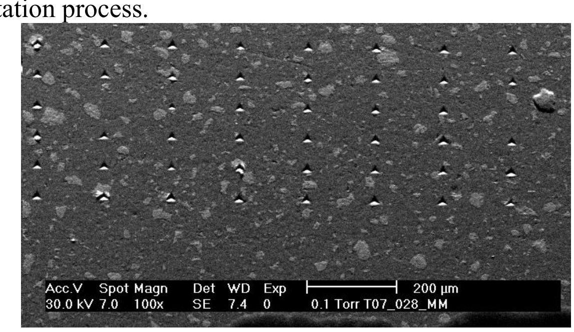


Fig.1 ESEM image of indented cement paste- matrix with large indents (5500 nm in depth)

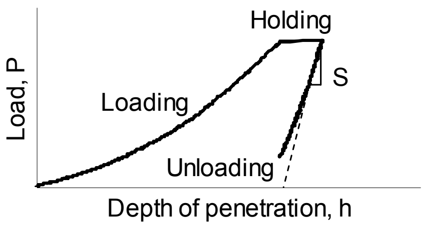
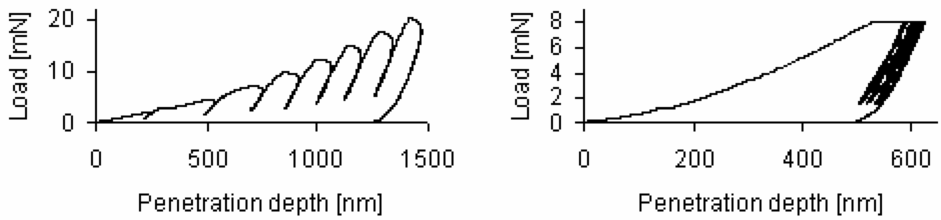


Fig.2 Nanoindentation load vs. depth of penetration diagram (P-h curve)

EXPERIMENTAL RESULTS

White cement paste samples (CEM-I 52,5 White, Holcim, SK) mixed in water/cement ratio w/c=0.5 were prepared and stored in water for 28 days. Before testing, a 2 mm thick slice from the bulk material was cut and polished on coarse to very fine emery papers to achieve very smooth and flat surface, e.g. [5]. Specimens were washed in ultrasonic bath to remove all the dust. The resultant surface had the roughness about several tens of nm as checked by AFM.

To study creep effects two different series of specimens were tested. The first series “O” (Fig.3a) was tested without holding period at the peak. Long holding period was applied for the second series “C” (Fig.3b). Both series were tested in several load levels in the range of 2-300 mN. It can be seen in Fig.3a that the P-h curve contains a “bulge” at the beginning of unloading in each cycle. It shows the role of creep that is present even on the unloading branch. As a consequence of this finding the assumptions of elastic solution are not fulfilled and evaluation of results leads to spurious size effect on elastic properties.



a) b)

Fig.3 Experimental results- examples of indentation load-depth diagrams:
a) Series “O”- multiple loading cycles with increasing load with no dwell period at the peak.
b) Series “C”- 5 loading/unloading cycles to the same load with dwell period at the peak

Elastic modulus and hardness are overestimated in the first cycles of “O” series as a consequence of using elastic solution for the unloading part of P-h curve [6]. This effect can be significantly decreased in case of “C” series and such a loading history can be utilized for good estimate of elastic constants. Indentation creep can also be measured during the holding phase of the P-h curve (Fig.4). It is clear from Fig.4 that most of the creep is present at the first cycle. Then, the creep significantly decreases in next cycles.

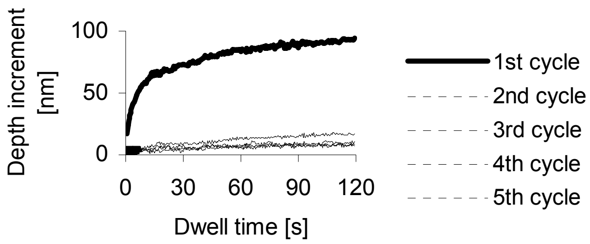
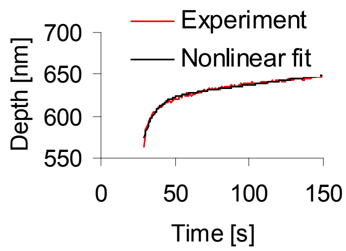
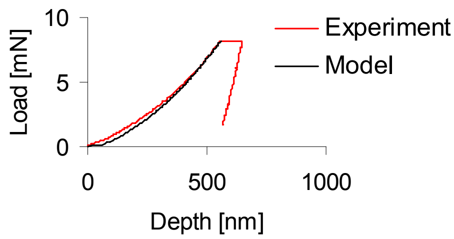


Fig.4 Experimental results. Example of indentation creep during the dwell period at the peak of loading diagram (series "C")

ANALYSIS OF NANOINDENTATION DATA

Three different models were constructed. The first model served for the estimation of elastic properties of cement paste and it is widely used for interpretation of nanoindentation results [6]. The second model was based on the viscoelastic solution [7] and it was used for simulation of the loading and holding part of experimental P-h curve. This model performed quite well for one cyclic loading and it was possible to identify elastic as well as viscosity parameters of cement paste. However in a general case of cyclic loading it appears that neither the elastic, nor the viscoelastic model fully represent the behavior of cement paste. Thus, the third more general FE model was constructed. The implemented constitutive law included the evolution of elastic, creep and plastic strains. Such general model was capable to describe qualitatively all the major features of the experimental response curve: the creep during load-dwell periods as well as variation of tangential stiffness during the loading phases. However, the number of parameters was too high to be determined by a simple trial and error approach. The main difficulty consisted in the fact, that even during the dwell periods when the loading force was constant, creeping of the material under the indenter resulted in changing contact area and consequently in changing stress field. Thus, it was not possible to separate the response due to plastic yielding and creeping.





a) b)

Fig.5 Comparison of experimental results with viscoelastic solution. a) Fitting of material parameters from creep in the holding period, b) Simulation of loading curve

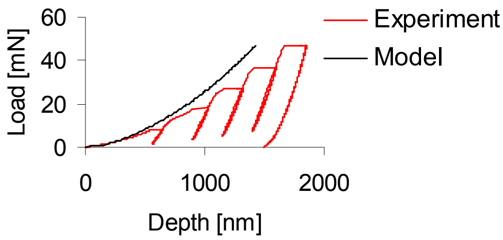
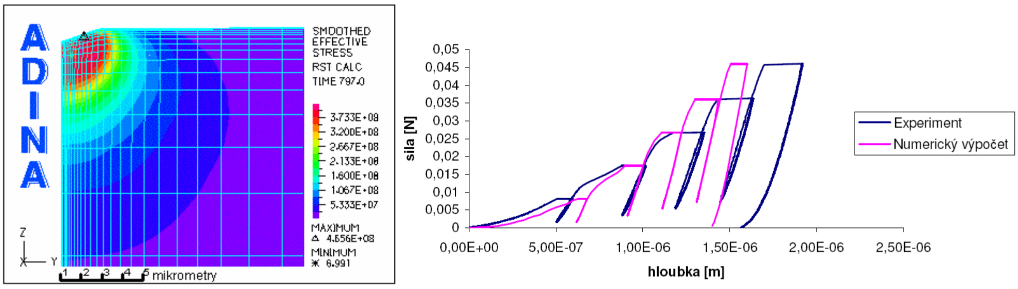


Fig.6 Simulation of loading curve by viscoelastic model with cyclic loading.



a) b)

Fig.7 FE model. a) Effective stress under indenter probe. b) P-h response curve for cyclic loading.

CONCLUSIONS

The presented work showed the im portance of creep in the evaluation of nanoindentation experiments measured on cement pastes. Since the traditional approaches based on elastic solution are not adequate for the simulation of cement paste more complex models are needed. The comparison of several approaches showed that classical elastic solution [6] can be used for the estimate of elastic parameters only in connection with a special loading path (long dwell periods and cyclic loading). A simple viscoelastic solution [7] can capture the loading and holding periods of the P-h curve for one cycle experiment. However, using of the same material parameters does not lead to satisfactory results for the case of cyclic loading. Thus, a more general FE model was proposed. The FE analyses showed that in description of the micromechanical behavior of cement paste, both time-independent plastic strains and time-dependent creep stains appear to play an important role. However, parameters of the qualitatively most suitable elastic-plastic-creep model are difficult to obtain. Presently, the possibility of using a more sophisticated method of parameter identification based on genetic algorithms is being researched.

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