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MICRO-SCALE CREEP OF CEMENTITIOUS MATERIALS

Němeček J.*, **Němečková J.****, **Němeček J.*****

Abstract: *Nowadays, micro-scale creep properties are accessible via nanoindentation tests. The paper aims to investigate various analytical expressions used to estimate micro-scale visco-elastic properties of cement paste. Five analytical methods based on various assumptions were tested. The necessity of taking plastic deformations into account was shown. Creep compliance evaluated for a short-term load duration (tens of seconds) was found in the order of usual uniaxial creep tests of cement pastes or concretes that take place on a much longer time scale (tens to hundreds of days). However, the creep response has the same logarithmic nature. Using different evaluation methods exhibited high scatter and direct comparison to uniaxial data does not give a clue how to select the best fitting method.*

Keywords: Cement, creep, visco-elasticity, nanoindentation.

1. Introduction

Creep of cementitious materials is an important phenomenon influencing the long-term performance of concrete structures. For many decades, creep has been studied on a macroscale using large-scale specimens. With the development of techniques such as electron microscopy or nanoindentation, the creep of cementitious matrix has also been accessed at micro-scale. Various theories on the creep mechanisms have been published. The main mechanisms employ sliding of C-S-H globules or C-S-H sheets, their compaction and water movements under conditions of sustained loading (Kai et al., 2021).

The logarithmic kinetics of creep is generally observed after days on concrete samples in the lab or even after years on concrete bridges. Interestingly, creep experiment conducted in a nanoindenter exhibits logarithmic kinetics already in the scale of seconds (Baronet et al., 2022). But, significantly higher stresses are applied by an indenter compared to usual macroscopic testing. Routinely used shapes of sharp indenters such as Berkovich, cube corner or Vickers cause both high stresses and volumetric compaction under the tip. This paper makes a comparison of various analytical approaches used for evaluation of creep parameters from nanoindentation performed on main hydration products of cement pastes (C-S-H gels) at the sub-micrometer level.

2. Theory and methods

Nanoindentation is a technique which uses a sharp (usually diamond) tip that is pressed into the material while accurately recording the force, P, and the tip displacement, h. There are four principal deformations caused by an indenter: elastic, viscous, plastic and fracture. The amount of individual deformation types depends strongly on the acuity of the indentation tip. While flat or spherical indenters tend to produce only elastic or visco-elastic deformations, sharper indenters cause further elasto-plastic or visco-elasto-plastic deformations. Indentation load-displacement ($P-h$) curves of cementitious materials usually contain all

* Prof. Ing. Jiří Němeček, PhD., DSc.: Czech Technical University in Prague, Faculty of Civil Engineering, Thákurova 7; 166 29, Prague 6; CZ, jiri.nemecek@fsv.cvut.cz

** Ing. Jitka Němečková, PhD.: Czech Technical University in Prague, Faculty of Civil Engineering, Thákurova 7; 166 29, Prague 6; CZ, jitka.nemeckova@fsv.cvut.cz

*** Ing. Jiří Němeček, PhD.: Czech Technical University in Prague, Faculty of Civil Engineering, Thákurova 7; 166 29, Prague 6; CZ, jiri.nemecek.1@fsv.cvut.cz

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kinds of viscous-elastic and plastic deformations. For higher stresses, fracture deformation may be observed as sudden burst in $P-h$ curves. The experimental $P-h$ curve usually contains three segments (see Fig. 1a). The first is the loading phase, during which all kinds of deformations occur. This is followed by the holding phase, in which the load is kept constant and the unloading part is final. Purely elastic behavior of the material can be assumed in the unloading part of the $P-h$ curve form which the indentation (reduced) modulus is derived (Oliver and Pharr, 1992) as $E_r = \frac{S\sqrt{\pi}}{2\sqrt{A_c}}$ where S is the contact stiffness and A_c is a contact area. A general relationship between the time-dependent indentation load, $P(t)$, and displacement, $h(t)$, for a step loading of a visco-elastic solid can be expressed as

$$h^m(t) = \kappa P_{max} J(t) \tag{1}$$

where $J(t)$ is the creep compliance function, m and κ are geometric constants (e.g. $m = 2$ and $\kappa = \frac{\pi}{2\tan\alpha}$ for cone or pyramid), P_{max} is the maximum load. Using Kelvin chain model, $J(t)$ can be approximated as

$$J(t) = \frac{1}{E_{r,0}} + \sum_{i=1}^{N} \frac{1}{E_{r,i}} \left(1 - e^{-\frac{t}{\tau_i}}\right) RCF_i \tag{2}$$

where $E_{r,0}$ is the instantaneous reduced modulus, E_i and τ_i are elastic moduli and retardation times of respective Kelvin units and RCF_i are ramp correction factors defined as $RCF_i = \frac{\tau_i}{t_R}\left(e^{t_r/\tau_i} - 1\right)$ with t_R being the ramping time (Oyen and Cook, 2009). If the load is instantaneously applied the $RCF_i = 1$, otherwise it accounts for a finite load ramping.

2.1. Effect of plastic deformation

In many materials including cement paste, nanoindenters equipped with sharp tips (especially the cube corner tip) cause non-negligible plastic strains that evolve mainly over loading period. Ignoring the strains and related add-on deflections to Eq. (1), respectively, result in overestimation of creep compliance values as confirmed both experimentally and numerically (Vandamme et al., 2012). Based on the assumption that plastic deformations appear only at the loading period, a relationship between contact compliance and the testing parameters can be established (Vandamme and Ulm, 2013) as

$$J(t) = \frac{1}{E_r} + \frac{2a_c \Delta h(t)}{P_{max}} = \frac{1}{E_r} + \frac{ln(1 + t/\tau)}{C} \tag{3}$$

where $\Delta h(t)$ is the increment of indenter's penetration depth during holding phase, $a_c = \sqrt{\frac{A_c}{\pi}}$ is the radius of contact. A logarithmic function fitting $\Delta h(t)$ from experiments was proposed by Vandamme and Ulm (2013) leading to the second expression in Eq. (3), where C is the creep modulus and τ is the characteristic time. Němeček et al. (2023) used a simple correction of plastic deformations by adding an additional fitting parameter, h_0, directly to Eq. (1) such that

$$(h(t) - h_0)^m = \kappa P_{max} J(t). \tag{4}$$

This formula leads to a successful fitting of the short-term experimental data. One of the advantages of this simple method is that it allows to estimate the amount of plastic deformation appearing in the loading phase depending on the type of an indenter.

3. Experiments and methods

Nanoindentation (using Hysitron TI-700) was performed on hydrated cement paste CEM I-42,5R (water/binder = 0.40) aged for 1.5 year in lime water. Two types of sharp indenter tips (Berkovich and cube corner) were used. The loading protocol was prescribed as follows: linear loading for 1 s; 40 s holding at 1.5 mN maximum force; linear unloading for 1 s. Always, a representative $P-h$ curve received from well hydrated phases of the cementitious matrix (C-S-H) was selected for each tip (Fig. 1a). Then, the analytical approaches described in Sect. 2. were applied. This included non-linear least square regression of the depth-time data by: (I) standard visco-elastic model assuming the step load (i.e. neglecting finite ramping, Eq. (2) with $RCF_i = 1$); (II) visco-elastic model with ramp correction (Eq. (2); (III) visco-elastic model with h_0 correction (Eq. (4) with RCF_i=1); (IV) visco-elastic model with h_0 and RCF_i correction (Eq. (4); (V) logarithmic fit (Eq. (3). Where applicable, using of two Kelvin units was sufficient in fitting the short-term data. Since the fitting procedure is very sensitive to the data in the beginning of the curve, the first 1 s of the holding (fitted) period was ommited and the data from 1–40 s interval were taken into consideration. Then, creep functions $J(t)$ were constructed for each of the model variant.

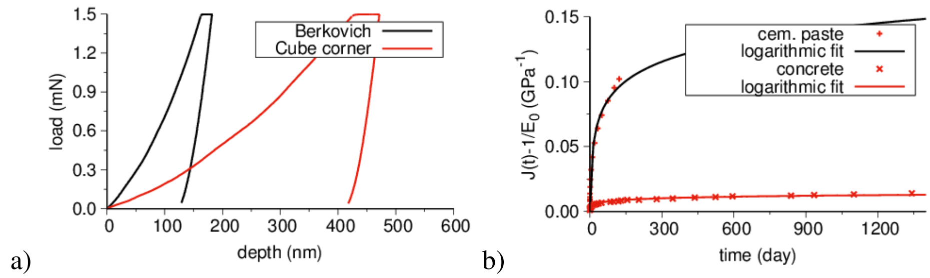

Fig. 1: a) Nanoindentation $P - h$ curves. b) Macroscopic uniaxial tests after Zhang et al. (2014).

4. Results and discussion

Despite receiving of relatively good fits of the experimental data (depth-time data) for every model, very large differences in the $J(t)$ predictions were obtained. It can be seen from Fig. 2 that results from the cube corner tip gave systematically higher estimates, leading to the conclusion that this tip causes non-negligible plastic strains that can hardly be filtered out by any of the methods. The stresses caused by the tip are also higher compared to less acute Berkovich tip and may already lie in the region of non-linear creep. Regardless of the tip used, the highest estimate of compliance was given by standard visco-elastic model assuming the step load and neglecting the plastic deformation. This was especially significant if the sharper cube corner tip was used. The ramp correction decreased the compliance prediction to one half in the case of Berkovich tip. When the h_0 correction was used, the lowest compliance prediction was received, regardless of the RCF_i correction. The logarithmic creep model provides intermediate predictions of $J(t)$. Parameters of different model variants are summarized in Tabs. 1 and 2.

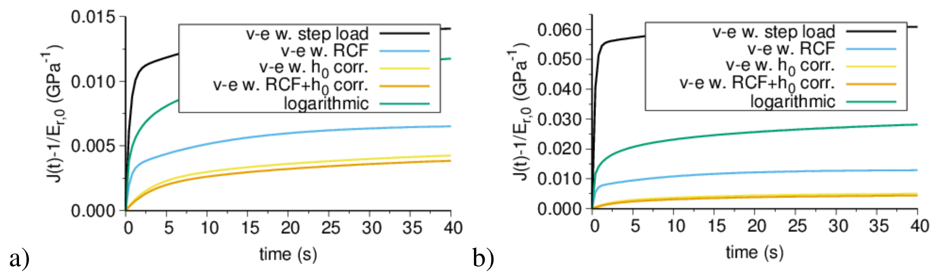

Fig. 2: J(t) functions for a) Berkovich tip, b) cube corner tip.

Deciding on what short-term compliance prediction is correct is a non-trivial task. One can compare the short-term functions with the standard macroscopic uniaxial tests done either on cement paste samples with millimeter dimensions or even on concrete specimens already containing large aggregates Zhang et al. (2014). Examples of such functions are given in Fig. 1b for a comparable cement paste and concrete having a comparable cementitious matrix. It is evident from Fig. 1b and also Tabs. 1 and 2 that the functions predict the creep in a similar order like from nanoindentation but on a completely different time scale.

5. Conclusions

From this study, it can be concluded that utilizing sharp nanoindentation is a feasible technique for quantifying the micro-scale creep of cementitious materials. So far, the methodology for estimation of the short-term

	Berkovich					Cube corner			
Method	$E_{r,0}$ (GPa)	τ_1 (s)	E_1 (GPa)	τ_2 (s)	E_2 (GPa)	τ_1 (s)	E_1 (GPa)	τ_2 (s)	E_2 (GPa)
1	40	0.5	93.9	12.3	281.1	0.1	18.2	3.6	165.4
2	40	0.5	309.2	12.3	292.8	0.1	138.4	3.5	173.6
3	40	2.9	444.9	26.4	389.3	0.6	451.3	8.0	338.7
4	40	2.9	531.4	26.4	396.8	0.6	590.0	8.0	350.5

Tab. 1: Parameters of Kelvin chain based creep compliance functions.

Parameter	**Berkovich**	**Cube corner**	**Uniaxial creep of paste**	**Uniaxial creep of concrete**
C (GPa)	564.5	274.9	46.5	565.8
τ	0.05 (s)	0.02 (s)	1.4 (day)	0.98 (day)

Tab. 2: Parameters of logarithmic creep compliance functions.

creep compliance is not standardized. It follows from the study that the available analytical estimates perform very differently based on their assumptions and the exact experimental methodology applied (e.g. tip or loading protocol selection). It seems that using the most acute tips (cube corner) leads to overestimation of the creep compliance due to significant stresses imposed to the material. Less acute tips (Berkovich) seem to give more reasonable predictions. Plastic strains developed during loading under the indenter cannot be ignored. But, even if the plastic deformations are taken into account, the analytical methods differ in their prediction by more than 100 %. Short-term creep data from nanoindentation provide reasonable creep compliance predictions and can be successfully used for comparisons of local visco-elastic behavior. However, direct linking of the short-term creep data to the uniaxial creep on a higher scale can hardly be achieved and further research in this direction is necessary.

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