MICROINDENTATION INTO A VISCOELASTIC MATERIAL VERSUS ITS LONG-TERM MECHANICAL PROPERTIES

Jiří Minster[1], Olga Bláhová[2], Jaroslav Lukeš[3], Jiří Němeček[3], Martin Šperl[1]

Institute of Theoretical and Applied Mechanics ASCR, v. v. i., Prosecka 76, 19000 Prague, Czech Republic[1]
University of West Bohemia in Plzen, Czech Republic[2]
Faculty of Civil Engineering, Czech Technical University in Prague[3]

Introduction

The nano/microindentation technique has been demonstrated to be effective also for time- or rate-dependent materials. The indentation process depends significantly on the ambient temperature and on the penetration rate. An assessment of the influence of degradation factors like temperature, humidity, ageing, irradiation and others on time-dependent mechanical properties can reduce risks connected with applying these materials. It has been confirmed that a quantitative analysis of time-dependent load-depth curves, based on the linear viscoelasticity theory, successfully yields rheological functions such as creep compliance $D(t)$ or relaxation modulus $E(t)$ [1], [2]. This paper aims to review the correctness of the microindentation technique for assessing long-term mechanical characteristics, namely viscoelastic compliance $D(t)$. The Poisson ratio v is assumed to be constant for direct measurements of the remaining independent material characteristics of a quasi-homogeneous material.

Experimental

Material

The selected characteristic representative of the viscoelastic material mentioned above is an epoxy resin mix consisting of solvent-free low-viscosity bicomponent pigmented systems on the basis of a low-molecular epoxy resin with a content of non-toxic reactive diluents, additives, pigments, fillers and auxiliary admixtures, hardened by a cycloaliphatic polyamide. The quasi-homogeneous and quasi-isotropic material is used for surfacing a range of building substrates, such as concrete, plaster, steel and stone. It is well suited for the manufacture of self-levelling flooring top layers and can be blended with fillers to form towelled polymer mortar or polymer concrete mixes. The samples were made by mold casting from one mixing, cured at room temperature and postcured at 90°C for 4 h. This type of postcure followed by slow cooling to laboratory temperature is indicated as rejuvenation (REJ). To assess the effect of ageing (A), the physical ageing time – five years storage in a black box under laboratory conditions (LA) or five years weathering (climatic ageing (CA)) - were identical for corresponding series of measurements.

Apparatus and Procedures

Two nano/microindenters, each equipped with a Berkovich indenter of effective cone angle α, were used at two different laboratories - Hysitron Triboscan at CTU in Prague and the Nano XP Indenter at UWB in Plzen. The tests were performed in laboratory conditions, with constant relative humidity and temperature control (T≈22-23°C).

The indentation proceeded under a step load (an indentation creep test with a constant load) $P(t) = P_0 H(t)$ ($H(t)$ is the Heaviside unit step function) with series of 5x5 or 6x8 indents. The creep compliance $D(t)$ can then be directly deduced [2] from

$$D(t) = \frac{2h^2(t)}{\pi(1-\nu^2)P_0 \tan\alpha} \qquad (1)$$

Equation (1) implies zero instantaneous compliance at time t=0 because the displacement into the surface $h(t)$ is also zero at the time. An ideal step load history (1) cannot ordinarily be generated in laboratory tests. Instead, ramp loading is used with a short rise time t_0 and a constant load thereafter. The constant loads P_o and loading rates dP/dt were 10 mN and 10 mN/s with t_0 ≈1s and the indentation creep duration t_c ≈ 50 s at CTU and 200 mN, 34 mN/s (t_0 ≤6s) and t_c ≈ 300 s at UWB. Due to these conditions, we reject from the analysis five times interval t_0 after the constant load is reached. It is assumed, however, that after passing this initial loading period, the creep compliance approaches values representing the viscoelastic behaviour.

Two types of standard creep experiments with the loading σ_c were performed on samples in series comprising at least three samples: (i) medium-term creep experiments (duration 6 hours) in laboratory conditions at the higher temperature T≈27-28°C, (ii) long-term creep experiments at a stable temperature T=20°C (duration in general 150 days active loading) to measure viscoelastic compliance in compression. The compressive stress σ_c amounted to 0.2 of strength in

compression σ_p ($\sigma_c \leq 0.2 * \sigma_p$) was taken as a limit for linear viscoelastic behaviour in creep.

Results

Fig.1 shows the long-term histories of a part of the viscoelastic compliance $D_c(t)=D(t)-D(0)$ ($D(0)$ is the instantaneous creep compliance) measured standardly [3] for rejuvenated (REJ), young (control) and aged samples after five-year climatic(CA) or five-year laboratory(LA) ageing.

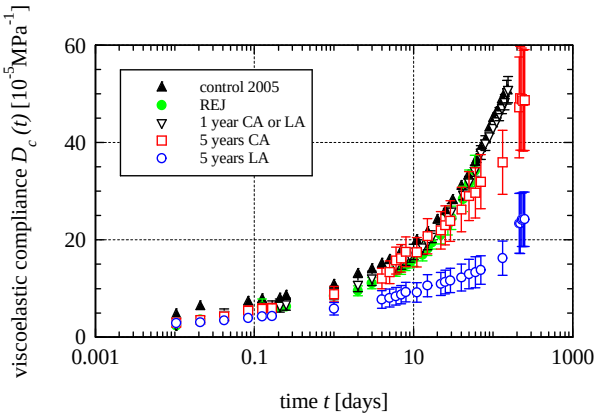

Fig. 1 Long-term histories of the standardly measured viscoelastic compliance $D_c(t)$.

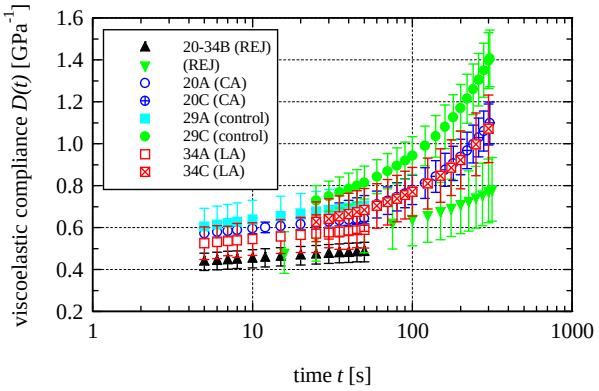

Fig.2 Short-term creep compliance histories $D(t)$ measured by instrumented indentation tests.

Fig. 2 shows creep compliance histories measured by instrumented indentation tests according to Eq. (1). The empty circular marks hold for five-year CA, the empty square marks indicate five-year LA, the full triangle marks represent REJ material, and the others are control samples. The values derived in two different laboratories under dissimilar conditions (loads, loading rates) correlate well. The positive influence of rejuvenation is due to the postcure realized after sample surface polishing which is accompanied by a higher moisture content and higher compliance. Fig. 3 demonstrates that direct microindentation measurements of the viscoelastic properties of time-dependent materials can provide results which are completely outside of the standard data (see the comparable data for rejuvenated samples).

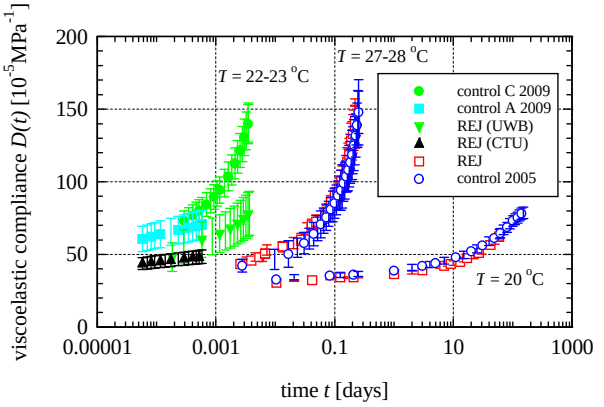

Fig. 3 Histories of viscoelastic compliance $D(t)$ measured by standard macro creep tests (empty marks) at two different temperatures with data derived from instrumented indentation tests (full marks) according to Equation (1) with a different rate of loadings.

Conclusion

Qualitative assessment of the influence of degradation factors on mechanical properties can be handled using short-term creep microindentation data, but the real time-delayed behaviour needs more time to manifest itself. The application of superposition principles [2] or the use of simulation techniques seem to provide a better assessment of the long-term mechanical characteristics than direct microindentation measurements.

Acknowledgements

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