DIRECT MICROINDENTATION MEASUREMENTS OF VISCOELASTIC PROPERTIES OF A QUASI-HOMOGENEOUS EPOXY MATRIX

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Introduction

The nano/microindentation technique developed over the past two decades has been demonstrated to be effective for time- or rate-independent materials. When time- or rate-dependence is the matter of concern, two important simplifications are commonly accepted – the problem is treated as linearly viscoelastic and due to the relatively short time measurements used, the Poisson's ratio is assumed to be constant. Due to these conditions, direct measurements of the remaining independent material characteristics of a quasi-homogeneous material are possible, using monotonic loadings in the course of penetration.
The paper aims, under the stated simplifications, to compare viscoelastic functions, e.g. viscoelastic compliance and relaxation modulus, measured by microindentation with data derived from standard macro measurements.

Experimental

Material
The selected characteristic representative of the materials 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 (R). To assess the effect of ageing (A), the physical ageing time – five years storage in a black box under laboratory conditions - was identical for each series of measurements.

Apparatus and Procedures
Two nano/microindenters, each equipped with a Berkovich indenter, 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.
Four monotonic time-dependent loading histories were applied:
(i) Indentation under a step load (indentation creep test with a constant load) $P(t)$[mN] = $P_0\, H(t)$, where $H(t)$ is the Heaviside unit step function. The creep compliance $D(t)$ can be directly deduced [1] from

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

Equation (1) implies zero instantaneous compliances at time t=0 because the displacement into the surface is also zero at the time. Viscoelastic materials normally have nonzero instantaneous creep compliances. The error is the result of the application of linear viscoelastic analysis. It is assumed, however, that after passing the initial loading period, the creep compliance approaches values representing the viscoelastic behaviour.
(ii) Indentation under a constant load rate, $P(t)=\dot{P}_0\, H(t)$, which leads to

$$D(t)=\frac{4h(t)}{\pi(1-\nu^2)\dot{P}_0\tan\alpha}\left(\frac{dh}{dP}(t)\right) \qquad (2)$$

(iii) Indentation with a fixed depth of penetration, $h(t)$ = $h_0\, H(t)$ with the relation to relaxation modulus $E(t)$

$$E(t)=\frac{\pi(1-\nu^2)\tan\alpha}{2h_0^2}P(t) \qquad (3)$$

Typically $h_0 \geq 600$ nm. An ideal step depth of penetration (or a step load) history cannot ordinarily be generated in laboratory tests. Instead, ramp loading is used with a short rise time t_0 and a constant depth or load thereafter. Due to these conditions, we reject from the analysis a certain time interval after the constant load is reached. This period of time is in general chosen as five to ten times the rise time t_0. To minimize the loss of data, the viscoelastic functions at the beginning of the test can be defined using approach (ii) for the creep compliance or the followed type of indentation for the relaxation modulus: (iv) Indentation under constant rate of penetration, $h(t) = v_0 t$, which leads to

$$E(t) = \frac{\pi(1-\nu^2)\tan\alpha}{4v_0^2}\frac{d^2P(t)}{dt^2} \qquad (4)$$

Results

Creep compliance

Fig.1 shows the results of application of the monotonic loadings (i) and (ii). All marks represent average values of *D(t)*. For reasons of clarity, only some of them are supplemented with characteristic error bars. Circular marks indicate the rejuvenated material with less dispersion of data and lower compliances. The full circular and square marks are derived using Eq. (2).

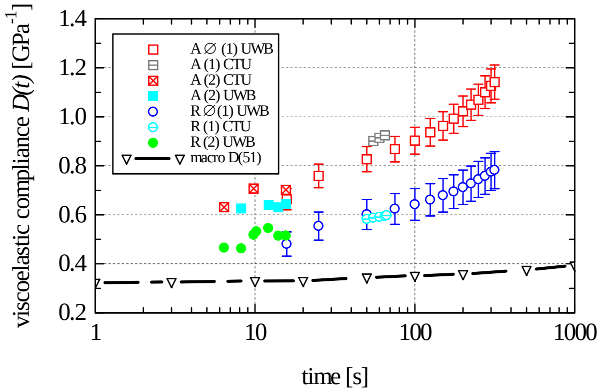

Fig.1 Comparison of creep compliance histories measured by a standard macro test (*D*(51)) with average data derived from instrumented indentation tests with the loading rate 0.66 mN/s according to Eq. (1) or (2)

Relaxation modulus

A computation of the relaxation modulus (or the viscoelastic compliance), in the case when the complementary viscoelastic function is known, can be realized by numerical solution of a linear Volterra integral equation of the second kind for unknown function *u(t)* [2]

$$u(t) + \int_0^t K(t,s)u(s)ds = f(t) \qquad (5)$$

while forcing function *f(t)* and kernel *K(t,s)* are known. Fig. 2 documents that the application the indentation (iv) can extend the time interval for receiving reasonable relaxation modulus data to shorter times. Halving and crossed marks in the figure present results of a prudent use of Eq. (4). Crossed marks supplemented with error bars indicate data derived for a set of measurements from penetration into an unpolished rough surface. Difficult scanning and selection of penetration positions by means of piezo automation is necessary in this case.

It is clear from Fig. 1 and Fig. 2 that the Boltzmann inequality $0.7 \le E(t)D(t) \le 1$ holds for the product of the viscoelastic functions for both material conditions. The relaxation modulus is weakly dependent on loading rates during the rise time which indicates that a nonlinear response is involved.

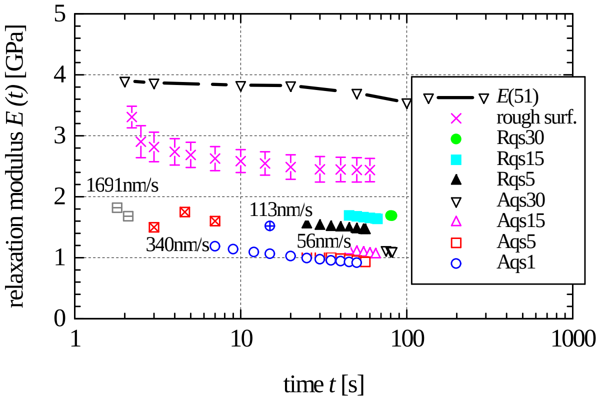

Fig. 2 Comparison of histories of the relaxation modulus measured by a standard macro test (*E*(51)) with data derived from instrumented indentation tests according to Equations (3) or (4) with different rise times t_o ($1s \le t_o \le 30s$) and different rates of penetration v_0 ($56nm/s \le v_0 \le 1691nm/s$)

Conclusion

A cautious use of direct microindentation measurements enables us to make a qualitative assessment of the short-term histories of viscoelastic material functions including the influence of ageing. However, for a common time dependent material the derived data of the relaxation modulus is at least twice as low as the standard macro values (an opposite holds for viscoelastic compliance). Surface modification through polishing before penetration is still an open problem.

Acknowledgements

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References

[1] Knauss, W.G., Emri, I., Lu, H. Mechanics of Polymers: Viscoelasticity. Springer Handbook of Experimental Solid Mechanics, Ed. W.N. Sharpe, Springer, Chapter 3, (2008) 49-96

[2] Wineman, A.S., Rajagopal, K.R. Mechanical Response of Polymers , Cambridge University Press (2000)