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# GEOMETRICAL FACTORS OF A SHARP MICROINDENTATION INTO VISCOELASTIC MATERIALS

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**Summary**: *Two approaches are applied to define short-term histories of creep compliance of a common epoxy composition loaded by a step load with the aim to compare the results with previously received data from standard macro measurements and to examine limitations of the method.*

## 1. Introduction and theoretical considerations

Microindentation with sharp pointed indenters leads to relatively large deformations under the indenter tip. Viscoelastic materials often exhibit nonlinear behaviour at strains larger than ≈0,5%, so it can be expected that nonlinear effects appear during indentation. Linearly viscoelastic analysis should thus be considered a first-order approximation for measuring linearly viscoelastic functions (Knauss et al., 2008). Geometrical factors enter into the analysis of instrumented indentation tests by means of relations between the applied indentation load $P$ to the stresses as well as the induced penetration depth $h$ to the strains.

In the simplest case, forcing a rigid indenter into a homogenous half-space of time-dependent material is treated as a quasistatic boundary value problem for linearly viscoelastic material with a moving boundary between the indenter and the half space. Because of the moving boundary condition, not a classical correspondence principle but a hereditary integral operator based on the associated solution for a linearly elastic material has to be used.

The linearly elastic indentation solution of the load displacement relation for the indentation of a rigid, axisymmetric conical indenter with the effective face angle $\alpha$ - which has been taken as an ideal model of similarly shaped sharp indenters - into a homogeneous, linearly elastic and isotropic half-space defined by the shear modulus $G$ and Poisson's ratio $\nu$ leads to the relation (1) (Sneddon, 1965)

$$P = \frac{4Gh^2}{\pi(1-\nu)\tan\alpha} \tag{1}$$

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Shimizu et al. (1999) deduced more general quadratic *P-h* relation for elastic indentation with a geometrically similar sharp indenter in the form (2)

$$P = \frac{gk}{2\gamma^2}\frac{E}{1-\nu^2}h^2 \tag{2}$$

where $\gamma$ is the geometrical factor combining the total penetration depth $h$ to the contact depth $h_c$ ($\gamma = h/h_c$) and $g$ is the geometrical factor of indenter used and defined by the relation of the projected area of indentation $A_c$ to the contact depth ($A_c = gh_c^2$). Eq. (2) leads to the Sneddon's elastic solution (1) for $\gamma = \pi/2$ ($g = \pi \cot^2\alpha$ and $k = \tan\alpha$).

The analogy of Eq.(1) for a linearly viscoelastic material has the form

$$h^2(t) = \frac{\pi(1-\nu)\tan\alpha}{4}\int_0^t J(t-\tau)\left(\frac{dP(\tau)}{d\tau}\right)d\tau \tag{3}$$

where $J(t)$ is the shear creep compliance at time $t$. Poisson's ratio can be assumed to be constant for short-time loading histories, so the shear creep compliance $J(t)$ or creep compliance $D(t)$ for one of monotonic load histories - a step load

$$P(t) = P_o H(t) \tag{4}$$

where $H(t)$ is the Heaviside unit step function) - can be directly deduced from

$$J(t) = \frac{4h^2(t)}{\pi(1-\nu)P_0\tan\alpha} \qquad resp. \qquad D(t) = \frac{gkh^2(t)}{2\gamma^2(1-\nu^2)P_0} \tag{5}$$

Considering that $1 \le \gamma \le \pi/2$, Eq.(5) enables us to apply two different geometrical factors and thus to define two different histories of viscoelastic material functions. Both direct methods were applied to measure the viscoelastic compliance of a common epoxy composition with the aim to compare the results with previously received data from standard macro measurements (Minster and Hristova (2005)) and to examine limitations of the approach.

## 2. Tested material, specimen preparation and test conditions

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 hardener. It is used for surfacing a range of building substrates, such as concrete, cement screed, plaster, asbestos cement, cement-and-chipboard, steel and stone. It is well suited for the manufacture of self-levelling flooring top layers and can be blended with fillers to form trowelled polymer mortar or polymer concrete mixes.

Specimens need to be prepared carefully prior to measurements. To remove any residual stress, some of them were annealed at temperature 90°C for 4 ours and cooled slowly to room, i.e. test temperature. These samples are mentioned as rejuvenated (REJ). To assess the effect of ageing, the physical ageing time was maintained at the same value for individual series of measurements. The tests were realised in laboratory conditions with constant relative humidity and temperature control.

## 3. Experiments and results

Two nano/microindenters, each equipped with a Berkovich indenter, were used at two different laboratories - Hysitron Triboscan at CTU in Prague and Nano XP Indenter at UWB in Plzen.

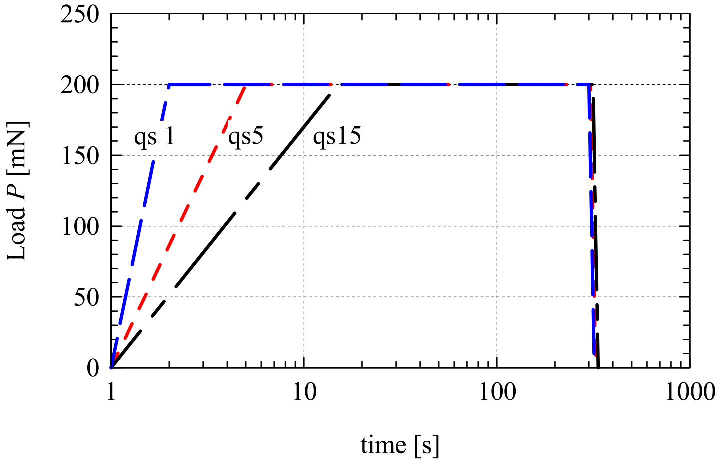

Fig.1 Ramp loading diagrams

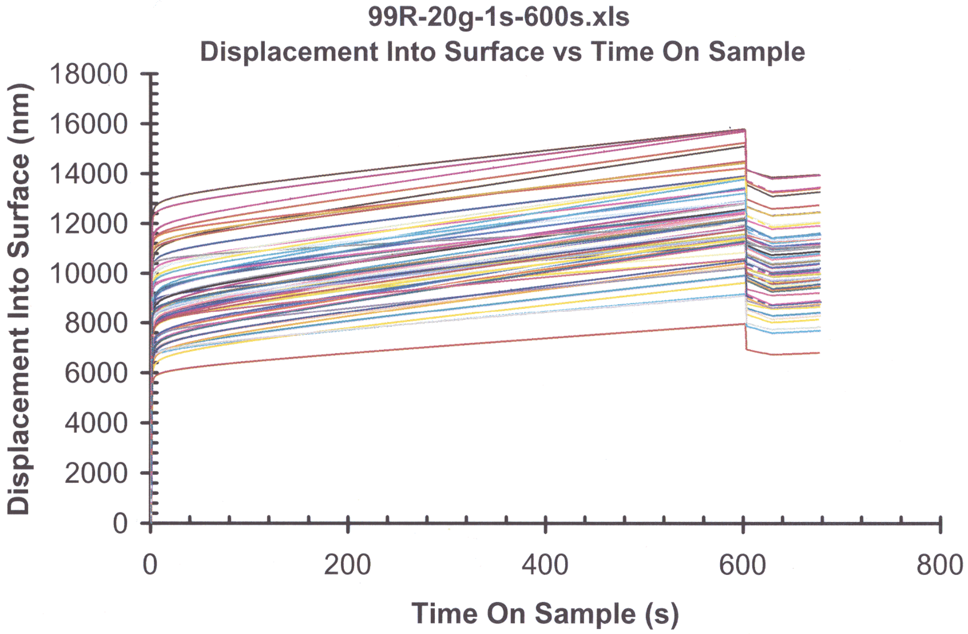

Fig. 2 A record (UWB) of a set displacement versus time histories measured during a test on the 6x8 dot matrix (4 histories were edged out)

An ideal step load history (4) cannot ordinarily be generated in laboratory tests. Instead, ramp loading is used with a short rise time $t_0$ and a constant load thereafter. These conditions give rise to 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 $t_0$. Fig.1 shows an example of used load-time diagrams used for loading with the rise times 1, 5 and 15 seconds.

The micro-inhomogeneity of the tested material leads to sizeable experimental data dispersion (see Fig.2), so the average values and standard deviations have to be considered in the analysis.

Eqation (5) 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.

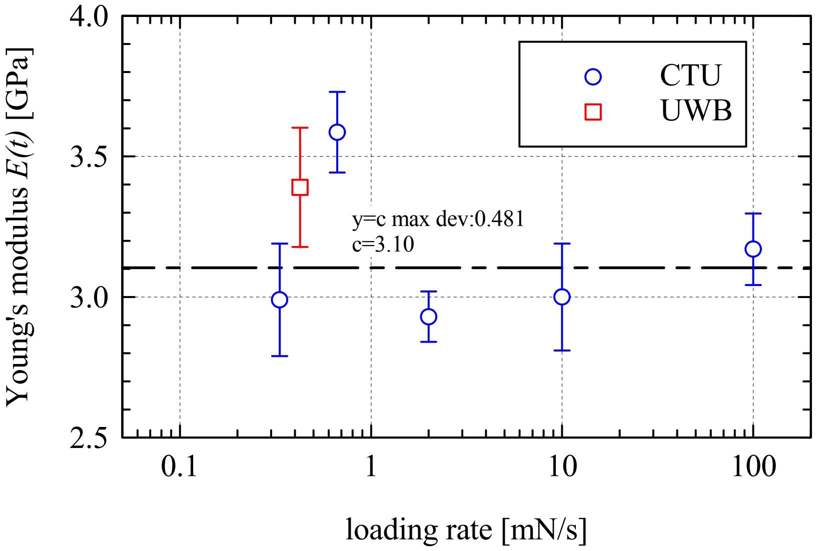

Fig.3 Dependence of the Young’s modulus values measured by the indenters on loading rates up to a constant load

Fig. 3 shows the dependence of the Young’s modulus values measured by the indenters on loading rates up to a constant load. All tests were performed on the same sample. The average value of the modulus $E(0) = 3.1$ [GPa] leads to the corresponding instantaneous viscoelastic creep compliance value $D(0) = 0.323$ [GPa$^{-1}$], and can be very well compared with previous macro data (see Minster and Hristova (2005)).

Fig. 4 presents a comparison of short-term histories of viscoelastic creep compliance $D(t)$, measured by a standard macro test (empty black triangular marks indicated as $D(51)$) with data derived from microindentation according to Eq.(5) for various rise times from 1 till 30 s. Circular and square empty marks with error bars represent average values of the characteristics of rejuvenated (circular) and laboratory aged (square) material of the same sample. In this case, laboratory aging means five years storage in black box under laboratory conditions. The full black triangular marks indicate the average history of $D$(t) of rejuvenated material with rise time $t_0$=1s. The history is in agreement with the course of the other history of rejuvenated material measured with rise time 15 s. These tests were carried out at UWB. The other empty coloured marks present the average viscoelastic compliance data derived from tests with different rise times from 1 s (qs1) till 30 s (qs30). This data came from CTU. All empty coloured marks correspond to the Shimizu approach with geometrical factor $\gamma = h/h_c$, and full marks correspond to the Sneddon solution with $\gamma = \pi/2$. For all tests, the data from the opening time interval with the constant load, influenced by the ramp process of loading, were omitted.

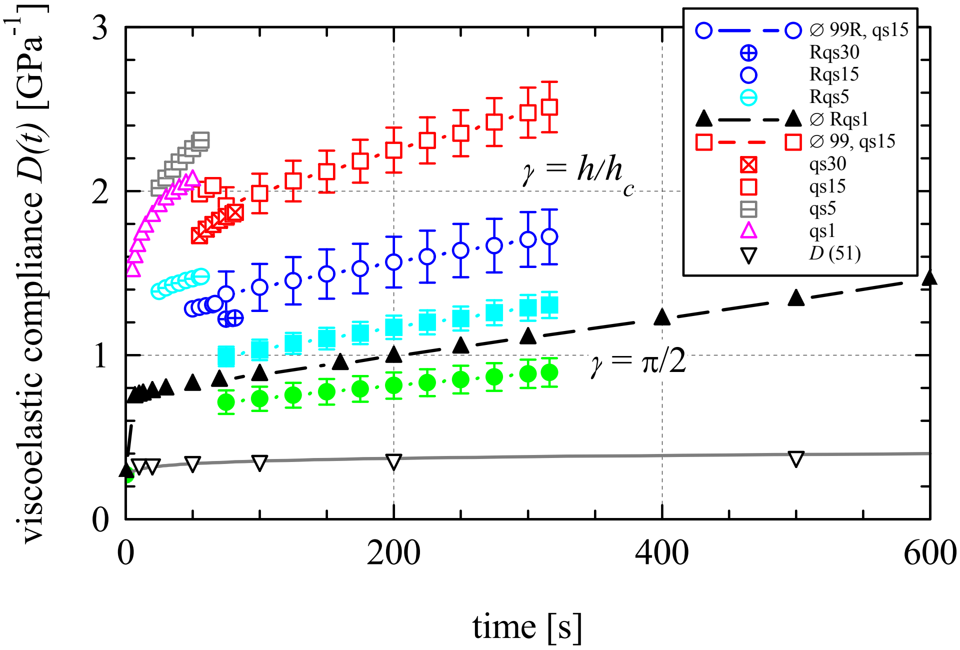

Fig.4 Comparison of short-term histories of the viscoelastic creep compliance *D(t)* measured by a standard macro test (empty triangular marks) with data derived from microindentation according to Eq.(5) for different rise times from 1 till 30 s.

The results proved for the tested material:

(i) The Sneddon solution is closer to the standard macro data. Nevertheles, all viscoelastic compliance values *D(t)* are at least twice as high as the standard values from macro measurements.

(ii) Rejuvenation has a positive influence viscoelastic functions. The viscoelastic compliance of the rejuvenated material is lower and the corresponding viscoelastic creep modulus is higher.

(iii) The viscoelastic functions are weakly dependent on test conditions (on the loading rate) which indicates that a nonlinear response is involved. This fact is documented by the rising course of the viscoelastic compliance histories in accordance with the rising rate of loading.

(iv) Up to now, the measurements certify that the data from the two laboratories correlates well.

## 4. Conclusion

To obtain highly reliable linearly viscoelastic functions of time dependent materials or at least to understand their possible history from instrumented microindentation tests, the application of more monotonic load histories and measurements of quite large test series is highly recommended.

## 5. Acknowledgement

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## 6. References

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

Minster, J., Hristova, Ju. (2005): Time-dependent response of an epoxy system to compressive loading. *10th Jubilee National Congress on Theoretical and Applied Mechanics*, 13-16 September, Varna, Book of Abstracts, 58-59; Proceedings, Volume I, Section 2 “Solid Mechanics”, Eds. Ivanov Ya., Manoach, E., Kazandijev, R., Prof. M. Drinov Publishing House, ISBN-10: 954-322-123-5, Sofia 2005, pp. 293-299

Shimizu, S., Yanagimoto, T., Sakai, M. (1999): Pyramidal indentation load-depth curve of viscoelastic materials, *J. of Materials Research*, Vol.14. No.10, Oct. 1999, 4075-4086

Sneddon, I.,N. (1965): The relation between load and penetration in the axisymmetric boussinesq problem for a punch of arbitrary profile, *Int. J. Eng. Sci*., 3, 47-57