DETERMINATION OF PARAMETERS OF VISCOELASTIC MATERIALS BY INSTRUMENTED INDENTATION. PART 2: VISCOELASTIC-PLASTIC RESPONSE

JAROSLAV MENČÍK

University of Pardubice, Studentská 95, 53210 Pardubice, Czech Republic
jaroslav.mencik@upce.cz

Keywords: mechanical properties, instrumented indentation, nanoindentation, viscoelasticity, plastic deformations

1. Introduction

This paper is a continuation of the article „Determination of parameters of viscoelastic materials by instrumented indentation“[1]. That article has explained the principles of instrumented indentation and brought basic formulae for indentation testing of elastic-plastic materials (with instantaneous response to load), as well as for the description of load response of viscoelastic materials, where the deformations depend not only on the load magnitude, but also on its duration. In Practical part, recommendations were given for the preparation of indentation tests into viscoelastic materials and for the evaluation of results. The article was limited to reversible deformations, which disappear fully during some time after unloading. Often, however, also irreversible deformations occur, e.g. under concentrated load or in materials whose response has viscous character. In this paper, the formulae for indentation response will be extended to irreversible deformations. Advice will be given for tests arrangement and data evaluation, and the proposed procedure will be illustrated on a practical example.

2. Theoretical part

Viscoelastic materials are typical by deformations depending not only on the load magnitude, but also on its duration and time course. As shown in[1], the relationship between indenter load P and depth h of its penetration into a specimen from such material under monotonic loading can generally be expressed as

$$f[h(t)] = K \psi(P, J, t) \tag{1}$$

where f is some function of the indenter shape and penetration, K is a constant characterizing the indenter geometry, and $\psi(P, J, t)$ is a function depending on the load magnitude and history, on material parameters, and on time; J is so-called creep compliance function. For pointed indenters (conical, Vickers or Berkovich)

$$f = h^2 \ ; \ K = \pi / (4 \tan\alpha) \tag{2}$$

α is the semi-angle of indenter tip or of equivalent cone (for Berkovich and Vickers indenter, $\alpha = 70.4°$). The load response of viscoelastic materials is usually described by models assembled from springs and dashpots. Rheological parameters for a viscoelastic model-body consisting of a spring in series with several Kelvin-Voigt units (a spring in parallel with a dashpot), can be obtained relatively easily from the time course of indenter penetration under constant load. For this case[1,2],

$$\psi(t) = P \{ C_0 + \Sigma C_j [1 - \rho_j \exp(-t/\tau_j)]\} \tag{3}$$

C_0 represent the instantaneous compliance, while the constants $C_1, \dots C_j$ pertain to the individual Kelvin-Voigt bodies, and, together with the retardation times τ_j characterize the time-dependent processes. ρ_j is so-called ramp correction factor, which takes into account the fact that the load increase was not infinitely short, but lasted t_R. According to Oyen[2],

$$\rho_j = (\tau_j / t_R) [\exp(t_R / \tau_j) - 1] \tag{4}$$

The irreversible deformations can be time-independent or time-dependent. The time-independent (plastic) behaviour can be modelled by a plastic element (a slider) in the rheological model, and the time-dependent irreversible deformations can be described by a dashpot arranged in series with other elements. The plastic element is characterised by hardness H_0 (or yield strength Y), which is – together with the Young modulus E_0 for instantaneous elastic response – contained in the constant C_0. Time-dependent irreversible deformation is characterised by a dashpot of viscosity η_v, with the creep compliance function

$$J(t) = c_v t \tag{5}$$

where the viscous compliance c_v is related to the dynamic viscosity as $c_v = 1/\eta_v$.

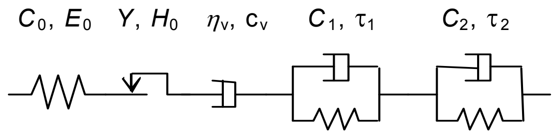

Fig. 1. **Viscoelastic-plastic model "spring + plastic element + dashpot + 2 Kelvin-Voigt bodies"**

The response function for a universal viscoelastic-plastic body, consisting of a spring, a dashpot and several Kelvin-Voigt units in series (Fig. 1), loaded by a constant force P, is

$$\psi(t) = P\{C_0 + c_v(t - t_R/2) + \Sigma C_j [1 - \rho_j \exp(-t/\tau_j)]\} \tag{6}$$

the term $t_R/2$ expresses the fact that the irreversible viscous deformations during the load increase from 0 to P are smaller than they would be if the full force P acted for the time t_R (they correspond to the average force $P/2$).

3. Procedure for the determination of viscoelastic-plastic properties

Parameters, characterizing viscoelastic properties, can be estimated from a simple five-step procedure (Fig. 2). In the first step (I, duration t_R), the indenter is quickly loaded to the nominal load P. Then, a long dwell under this load follows (step II), then rapid unloading to a very low load P_u (step III), followed by a long time under this load (IV), and unloading to zero (V). The response during dwell II provides the base for the determination of viscoelastic parameters, while the back-creep in the low-load dwell IV serves for the verification of the duration of reversible viscoelastic processes and of the necessary number of Kelvin-Voigt elements (determined in step II). The parameters of instantaneous elastic and plastic deforming are best obtained from a separate load-unload test.

The loading in step I should be as quick as possible in order to reduce the time-dependent processes during this period. A constant load rate (dP/dh = const) is recommended here, because the ramp correction factor ρ, Eq. (*4*), corresponds to this case. However, if the duration of load increase is short, the viscoelastic deformations during this time are small, and the details of the load increase $P(t)$ are not very important. The dwell II under constant load should be sufficiently long so that all reversible viscoelastic processes have died away and their duration can be assessed. The unloading III should be fast and the constant load P_u in the following period IV should be very low, only such that the indenter remains in contact with the specimen and can serve as a sensor to measure the deformation recovery (back-creep). The period IV should last about the same time as period II.

The processing of measured $h(t)$ data starts with the determination of constants in the creep compliance function from the dwell II. The practical procedure will be illustrated on a test with a pointed indenter and response function (*6*). Inserting this function together with equation (*2*) into Eq. (*1*) gives

$$h^2(t) = PK\{C_0 + c_v\,(t - t_R/2) + \Sigma C_j\,[1 - \rho_j \exp(-\,t/\tau_j)]\} \qquad (7)$$

where P is the nominal load and $K = \pi/(4 \tan\alpha)$, with the indenter tip semiangle α.

The constants C_0, c_v, C_1, τ_1, ρ_1, etc. can be obtained by minimizing the sum of the squared differences between the measured and calculated $h^2(t)$ values. However, the actual procedure must be modified. In equation (*7*), several terms appear that do not depend on time: C_0, $c_v t_R/2$ and C_j. Regression analysis cannot determine them individually, but only as a whole; otherwise incorrect values could be obtained. Moreover, the terms $C_j\rho_j$ also occur here, as well as the ramp correction factors ρ_j, which depend on the retardation times τ_j that are also unknown. From a curve-fitting point of view, equation (*7*) corresponds to the expression

$$h^2(t) = P\,K\,[B + c_v t - \Sigma\, D_j \exp(-\,t/\tau_j)] \qquad (8)$$

where

$$B = C_0 - c_v\,t_R/2 + \Sigma C_j\,; \quad D_j = C_j\rho_j\,; \; j = 1, 2, 3\ldots \qquad (9)$$

The determination of material parameters proceeds in three steps:

<u>Step 1.</u> Calculation of regression constants B, c_v and D_j, as well as the retardation times τ_j by fitting the $h^2(t)$ data from period II by the regression function (*8*).

<u>Step 2.</u> Calculation of the ramp correction factors ρ_j from Eq. (*4*) for the known duration t_R of load increase and the retardation times τ_j. Then, the constants C_j are found as $C_j = D_j/\rho_j$.

<u>Step 3.</u> Determination of C_0 from Eq. (*9*) as

$$C_0 = B + c_v\,t_R/2 - \Sigma C_j \qquad (10)$$

Similar procedures can be used for other models and indenters. It is recommended to try and evaluate several models; analysis of the back-creep during the low-load dwell (step IV) can be helpful here.

The above data-processing has separated the time-dependent deformations from the instantaneous ones, characterized by the compliance C_0. Now it is necessary to decompose C_0 into the reversible elastic component and irreversible plastic component. These components may be characterized by instantaneous elastic modulus E_0 and hardness H_0, which are determined in a similar way like in elastic-plastic materials, i.e. from the loading and unloading curves, as described in[1]. If the unloading in step III is sufficiently quick, the "fast" contact stiffness S could be obtained. However, this stiffness corresponds to the larger depth at the end of dwell, and the pertinent hardness will be lower than the "instantaneous" one. Therefore, it is better to determine S, E_0 and H_0 in a separate test, with fast loading followed immediately by fast unloading.

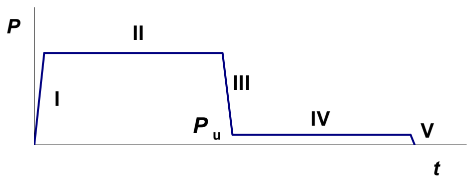

Fig. 2. **Five-step loading-unloading scheme for obtaining the viscoelastic-plastic parameters;** P – indenter load, t – time

4. Experimental part

The above method was used for the determination of viscoelastic-plastic parameters of tooth enamel[3]. A human tooth was embedded into epoxy resin and cut and polished with a diamond paste. The tests were done with a UMIS-2000 nanoindenter and a diamond Berkovich indenter. The experiments were divided into two groups; one served for obtaining viscoelastic parameters from the creep data, and the other was for the determination of the instantaneous elastic modulus and hardness.

In the first group, six tests were made using the five-step loading scheme (Fig. 2). The indenter was loaded to the nominal load P = 250 mN, which was then held constant for 975–1145 s. Then it was unloaded to P_u = 5 mN, kept constant 938–1110 s and then unloaded to zero. The load increase (step I) and decrease (step III) lasted about 20 s each. The characteristic depths varied between 1.83–1.96 µm under nominal load 250 mN, and between 1.40–1.33 µm after unloading to 5 mN; the details are given in[3].

The load-displacement curve during dwell II was approximated by four models:

Spring + one Kelvin-Voigt body,
Spring + two Kelvin-Voigt bodies,
Spring + dashpot + one Kelvin-Voigt body,
Spring + dashpot + two Kelvin-Voigt bodies.

Model d), depicted in Fig. 1, was described by the equation (*7*) or (*8*) with j = 2, while the model c) was only with j = 1, and the models a) and b) were without the dashpot (c_v). The constants B, c_v, D_1, D_2, τ_1 and τ_2 were found by minimizing the sum of the squared differences between the squares of the measured and calculated depth, $\Sigma[h_m^2(t_j) - h_c^2(t_j)]^2 = \min$. The solver in Microsoft Excel was used for the minimization. Then, the ramp correction factors ρ_1, ρ_2 and the constants C_0, C_1, C_2 were found. For better judgement of individual approximations, the relative differences between measured and calculated values, $\Delta_{rel,j} = [h_m(t_j) - h_c(t_j)]/h_m(t_j)$, were also calculated and plotted.

The best approximation was obtained by the model (d) "spring + dashpot + 2 Kelvin-Voigt bodies" (Fig. 3). A very good fit was also obtained by the model (b) "S + 2 KV". The model (c) "S + D + KV" was worse, and the model (a) "S + KV" has not corresponded to the measured curve. The back-creep after unloading has confirmed suitability of the model (d) and the retardation times, which were approximately $\tau_1 \approx 20$ s, $\tau_2 \approx 200$ s.

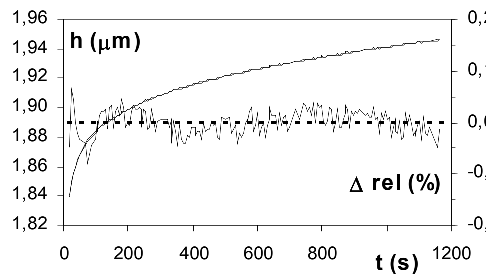

Fig. 3. **Indenter penetration into tooth enamel under constant load during dwell II[3].** Measured values are plotted together with those calculated for model "S + D + 2 KV"; the differences can be seen only in the diagram of relative residuals Δ_{rel}. h – depth, t – time

Elastic modulus E and hardness H, determined in six separate load-unload tests using the formulae numbered (*1*)–(*4*) in[1], were in the range: 96–102 GPa for E, and 4.0–4.6 GPa for H. All measurements are described in paper[3], which also contains all important values.

Hardness and elastic modulus characterize the instantaneous response. As such, they are related to the compliance C_0. Hainsworth et al.[4] and Malzbender et al.[5] have derived the following relationships for the loading curve under a pointed indenter:

$$P = kh^2\ ; \quad k = E\left(\Phi\sqrt{E/H} + \Psi\sqrt{H/E}\right)^{-2} \qquad (11)$$

with constants $\Phi = 0.202$ and $\Psi = 0.638$. The combination with the instantaneous part of Eq. (7), $h^2 = PKC_0$, gives[3]:

$$C_0 = \frac{4\tan\alpha}{\pi}\frac{1}{E_r}\left(\Phi\sqrt{E_r/H} + \Psi\sqrt{H/E_r}\right)^2 \qquad (12)$$

where E_r is the reduced modulus, related to the elastic modulus of the specimen (E) and the indenter (E_i) as $1/E_r = (1 - \nu^2)/E + (1 - \nu_i^2)/E_i$; ν is Poisson´s ratio. The value of C_0 for the tooth enamel, calculated from the measured values H and E (resp. E_r), differed from the instantaneous compliance C_0, obtained by fitting the creep data via Eq. (*12*), by less than 4 % (ref.[3]). This demonstrates the possibility of using the formula (*12*) for verification of parameters in viscoelastic-plastic models.

5. Conclusions

Mechanical properties can be determined by instrumented indentation, where the indenter load and displacement are measured continuously during loading and unloading. The load response of viscoelastic-plastic materials can be described by means of rheologic models, consisting of springs and dashpots. In this paper, following the first part[1], formulae were given for the description of indentation response of these materials. Also a five-step test procedure, combined with a fast load-unload test, was proposed for experimental determination of viscoelastic-plastic parameters. The use of the method has been illustrated on indentation testing of human-tooth enamel.

The work was supported by the Grant Agency of Czech Republic, project No. GAČR 103/08/1340.

REFERENCES

1. Menčík J.: Chem. Listy, in print.
2. Oyen M. L.: Philosophical Magazine *86*, 5625 (2006).
3. Menčík J., He L. H., Swain M. V.: J. Mech. Behav. Biomed. *2*, 318 (2009).
4. Hainsworth S. V., Chandler H. W., Page T. F.: J. Mater. Res. *11*, 1987 (1996).
5. Malzbender J., With de G., Toonder den J.: J. Mater. Res. *15*, 1209 (2000).

J. Menčík *(Department of Mechanics, Materials and Machine Parts, Jan Perner Transport Fakulty, University of Pardubice, CZ-53210 Pardubice, Czech Republic):* **Determination of Parameters of Viscoelastic Materials by Instrumented Indentation. Part 2: Viscoelastic-Plastic Response**

The load response of viscoelastic-plastic materials can be described by rheologic models, consisting of springs and dashpots. This paper, which follows the first part, published in Chemické listy earlier[1], gives formulae for description of indentation response of these materials. Also a five-step test procedure, combined with a fast load-unload test, was proposed for experimental determination of viscoelastic-plastic parameters by instrumented indentation. The use of this method has been illustrated on testing of human-tooth enamel.