# DETERMINATION OF PARAMETERS OF VISCOELASTIC MATERIALS BY INSTRUMENTED INDENTATION

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## 1. Introduction

Instrumented indentation, called also nanoindentation if very low loads are used, provides information about mechanical properties from indenter load and depth measured during loading and unloading (Fig. 1). Today, these methods are well established for elastic-plastic materials like metals or ceramics. They can also be used for characterisation of polymeric and other materials whose response to load depends also on time. However, specific features of their behaviour must be taken into account in the test preparation and data processing. The pertinent methods are still under development. This paper gives a brief review of basic formulae for instrumented indentation into elastic-plastic materials, then it explains the load response of viscoelastic materials and presents the pertinent formulae, with emphasis on indentation testing. Also, a practical test procedure is described.

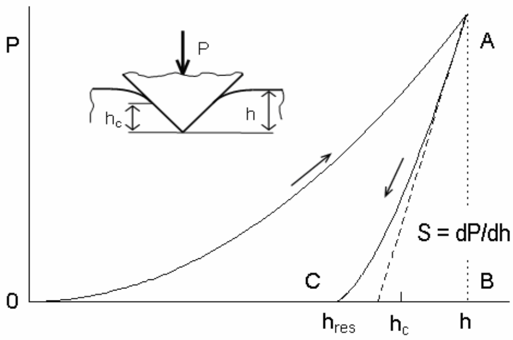


Fig. 1. **Load-depth curves of an indentation test – a schematic.** P – indenter load, h – displacement (penetration), $h_c$ – contact depth

## 2. Basic formulae for indentation testing of elastic-plastic materials

In instrumented indentation, hardness $H$ is defined as the mean contact pressure, and calculated by dividing the indenter load $P$ by the projected contact area $A$:

$$H = \frac{P}{A} \tag{1}$$

The contact area is calculated from contact depth $h_c$, which is obtained from the total indenter displacement $h$, indenter load $P$ and contact stiffness $S = dP/dh$ at the beginning of unloading (Fig. 1), usually by the formula proposed by Oliver and Pharr[1]:

$$h_c = h - \varepsilon \frac{P}{S} \tag{2}$$

where ε is a constant ($\varepsilon \approx 0.75$). The stiffness $S$ is determined from the regression function, mostly of power-law type,

$$P = c\,(h - h_{res})^{m} \tag{3}$$

fitted to the upper part of unloading curve; $c$, $m$ and $h_{res}$ are regression constants ($h_{res}$ corresponds to the residual depth of imprint after unloading). For Berkovich indenter (a three-sided pyramid often used in nanoindentation), the contact area is approximately $A = 24.5\,h_c^2$; more accurate results $A(h_c)$ are obtained by indenter tip calibration.

Equation (*2*) has proved good for elastic materials, where the ratio of the depth of permanent imprint after unloading, $h_{res}$, and the total depth under load, $h$, is less than 0.7. For more ductile materials, with $h_{res}/h > 0.7$, however, Equation (*2*) gives lower values $h_c$ than actual. For these cases, Bec et al.[2] have recommended the relationship

$$h_c = 1{,}2\left(h - \frac{P}{S}\right) \tag{4}$$

a discussion to both approaches can be found in paper[3].

In addition to hardness, also the elastic modulus can be calculated from the contact stiffness and contact area:

$$E_r = \frac{\sqrt{\pi}}{2\beta}\frac{S}{\sqrt{A}} \tag{5}$$

$E_r$ is the reduced modulus, related with the elastic modulus $E$ and Poisson ratio $\nu$ of the specimen (no subscript) a indenter (subscript i) as

$$\frac{1}{E_r} = \frac{1-\nu^2}{E} + \frac{1-\nu_i^{\,2}}{E_i} \tag{6}$$

β is a correction for the indenter tip shape (for Berkovich indenter, $\beta \approx 1.05$).

## 3. Theoretical background for testing of viscoelastic materials

The response of some materials, such as polymers, is more complex, as it depends not only on the load magnitude, but also on its duration and time course. The indenter continues penetrating into the specimen even under constant load (part D of the curve in Fig. 2). Such materials are called viscoelastic or viscoelastic-plastic. In this case, hardness (*1*) is no more a constant, but decreases with the time under load, $H = H(t)$.

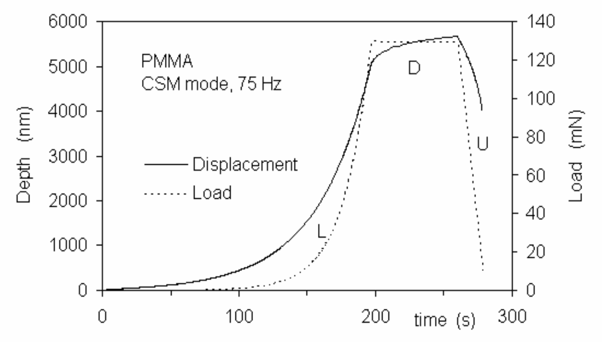


Fig. 2. **Time course of a nanoindentation test into PMMA [4];** L – loading, D – dwell under constant load, U – unloading

Moreover, due to delayed deforming, also the unloading part of the $P - h$ curve is sometimes distorted (Fig. 3); it is more convex than in elastic materials, sometimes with a bulge or even a "nose" at the beginning of unloading. The determination of contact stiffness by a conventional manner from the slope of the unloading curve thus could lead to an error in the determination of contact stiffness $S$, and thus to an error in the contact depth and area, as well as in the elastic modulus and hardness. If the time-dependent effects are not negligible, a special approach is needed. This paper will deal with procedures for viscoelastic materials and monotonic loading and unloading. The determination of viscoelastic properties under harmonic loading can be found in Menčík et al.[4]. The characterisation of viscoelastic-plastic properties, typical by the occurrence of time-independent permanent deformations, will be the topic of another paper.

The delayed deforming in indentation tests can be accounted for in various ways. In order to avoid the distorsion of unloading curve (Fig. 3), it is recommended to insert a dwell (with constant load) between the loading and unloading period. According to Chudoba and Richter[5], the influence of delayed deforming on the unloading curve may be neglected if the creep velocity has decreased so that the penetration depth at the end of dwell grows not faster than 1% per minute. Some disadvantage of this approach is that the indenter depth at the beginning of unloading (after the dwell) is larger than at the end of loading, and this results in larger contact area and lower apparent hardness.

Therefore, some authors recommend to use relatively fast loading followed immediately by fast unloading, and to calculate the contact depth and elastic modulus using the effective contact stiffness S, defined by the following expression proposed by Ngan and Tang[6]:

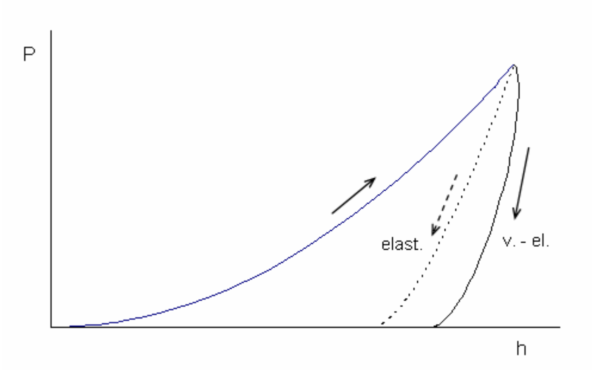


Fig. 3. **Load-depth diagram for elastic and viscoelastic (v.- el.) material;** note the differences between unloading curves

$$\frac{1}{S} = \frac{1}{S_{\mathrm{app}}} + \frac{\dot{h}_{\mathrm{d}}}{\left|\dot{P}_{\mathrm{u}}\right|} \tag{7}$$

$S_{\mathrm{app}}$ is the apparent stiffness, obtained by the common Oliver and Pharr procedure[1] from the unloading curve, $\dot{h}_{\mathrm{d}}$ is the indenter velocity at the end of dwell, and $\dot{P}_{\mathrm{u}}$ is the load decrease rate at the beginning of unloading (Fig. 3); the units are N/m, m/s and N/s, respectively.

However, the results can also be influenced by viscoelastic deforming during the load increase phase. Moreover, it is generally insufficient to characterise materials that flow permanently under load, only by means of a single value of hardness or elastic modulus. The time-dependent properties must be described in a more appropriate way. Usually, rheological models consisting of springs and dashpots are used, such as the Kelvin or Maxwell model and their combinations (Fig. 4). The parameters in these models, suitable as material characteristics, can be obtained by fitting the time course of penetration depth by a suitable creep function, depending on the material, indenter shape and loading history.

The commonly used formulae are based on the approach proposed by Lee and Radok[7], which uses the elastic solution, but replaces the elastic constants by a viscoelastic hereditary integral operator; cf. Johnson[8] or Oyen[9].

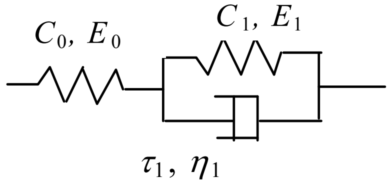


Fig. 4. **Standard Linear Solid**

The relationship between indenter load and depth of penetration into an elastic or viscoelastic material under monotonic loading can be expressed generally as

$$f[h(t)] = K\,\psi\,(P, J, t) \tag{8}$$

where $f$ is some function of the indenter shape and penetration, $K$ is a constant characterising the indenter geometry, and $\psi(P, J, t)$ is a function depending on the load magnitude and history, on material parameters, and on time. For spherical indenter (subscript s),

$$f_S = h^{3/2}\ ;\ K_S = 3\,/\,(8\sqrt{r}) \tag{9}$$

$r$ is the indenter tip radius. For pointed indenters (conical, Berkovich or Vickers, subscript c),

$$f_C = h^2\ ;\ K_C = \pi\,/\,(4\tan\alpha) \tag{10}$$

$\alpha$ is the semiangle of indenter tip or of equivalent cone (for Berkovich and Vickers indenter, $\alpha = 70.4°$). The function $\psi$ for penetration of a rigid indenter into ideally elastic materials is

$$\psi = P\frac{1-\nu}{G} \tag{11}$$

$G$ is the shear modulus and $\nu$ is Poisson ratio. The general formula for linearly-viscoelastic materials (and a rigid indenter) is

$$\psi(t) = \int_0^t J(t-u)[dP/du]\,du \tag{12}$$

where $J(t)$ is the so-called creep compliance function, depending on the material model used, $t$ is time, and $u$ is a dummy variable for integration. For constant load after step change from 0 to $P$, the function $\psi$ is simply the product of load and creep compliance function $J(t)$,

$$\psi\,(t) = P\,J(t) \tag{13}$$

However, step load is impossible to realise; there is always some period of load increase. For ramp loading with constant load rate, $R = \mathrm{d}P/\mathrm{d}t = const$, it holds

$$\psi(t) = R\int_0^t J(t-u)\,du \tag{14}$$

The penetration under constant load $P$ following the ramp load lasting $t_R$ can be described by the function

$$\psi(t) = R\int_0^{t_R} J(t-u)\,du \tag{15}$$

where $t_R$ is the duration of load increase. Formula (*15*), valid for $t > t_R$, was obtained as the sum of two loads growing with the constant rate $R$: the first load starts at $t = 0$, while the other, acting in the opposite direction, starts at time $t_R$. Thus, for $t > t_R$, the load is constant, $P = Rt_R$.

The application of the above formulae can be illustrated on a relatively universal model, consisting of a spring connected in series with a Kelvin-Voigt unit, which is a spring in parallel with a dashpot. For this model, called Standard Linear Solid (Fig. 4), the creep compliance function is

$$J(t) = C_0 + C_1\,[1 - \exp(-\,t\,/\tau_1)] \tag{16}$$

where $C_0$ and $C_1$ are compliance constants, and $\tau_1$ is so-called retardation time. This is a time constant of the system, related to the compliance $C_1$ of the spring and viscosity $\eta_1$ of the dashpot in the Kelvin-Voigt body as $\tau_1 = \eta_1 C_1$. A more complex response can be approximated using more Kelvin-Voigt units in series,

$$J(t) = C_0 + \Sigma\,C_j\,[1 - \exp(-\,t\,/\tau_j)] \tag{17}$$

($j = 1, 2,\ldots n$). The instantaneous compliances $C_j$ (and $C_0$) are related to the shear moduli $G_j$ and Poisson ratios $\nu_j$ by

$$C_j = (1 - \nu_j)/G_j \tag{18}$$

The springs can also be characterised by means of tensile moduli $E$, related to the shear moduli as

$$E_j = 2(1 + \nu_j)\,G_j \tag{19}$$

However, only $G_0$ and $E_0$ correspond to the actual (instantaneous) elastic modulus of the material. The other constants $C_1$, $C_2\ldots$ just characterise delayed deforming. The "moduli" $G_1$, $G_2\ldots$ or $E_1$, $E_2\ldots$ do not represent additional stiffnesses, but reciprocals of additional, time- dependent compliances.

Indentation creep under constant load $P$ following ramp loading (with constant load rate) lasting $t_R = P/R$, can be described by the function derived by Oyen[9]:

$$\psi\,(t) = P\,\{\,C_0 + C_1\,[1 - \rho_1\exp(-\,t\,/\tau_1)]\} \tag{20}$$

or, for a generalised standard linear solid (*15*),

$$\psi\,(t) = P\,\{\,C_0 + \Sigma\,C_j\,[1 - \rho_j\exp(-\,t\,/\tau_j)]\} \tag{21}$$

In expressions (*20*) and (*21*), $\rho_j$ is so-called ramp correction factor, calculated as[9]

$$\rho_j = (\tau_j\,/t_R)\,[\exp(t_R\,/\tau_j) - 1] \tag{22}$$

Note that the formulae (*20*) and (*21*), valid for $t \geq t_R$, differ from those for step loading, based on Eqs. (*16*) and (*17*), only by the factors $\rho_j$ at the exponential terms. For fast loading, with the load increase very short compared to the retardation time, $t_R \ll \tau_j$, the ramp correction factor is close to 1; it attains 1.025 for $t_R/\tau_j = 0.05$ and 1.05 for $t_R/\tau_j = 0.1$, and grows rapidly for higher ratios $t_R/\tau_j$.

REMARK. Creep compliance function (*17*) can also be written in another form[9]:

$$J(t) = C_0' - \Sigma\,C_j\exp(-\,t\,/\tau_j) \tag{23}$$

where

$$C_0' = C_0 + \Sigma\,C_j \tag{24}$$

$C_0'$ expresses the asymptotic compliance, corresponding to very long loading ($t \rightarrow \infty$), in contrast to $C_0$, which characterises the instantaneous compliance, i.e. the immediate reaction to sudden loading. [The form (*23*) is usual in computer programs for the finite element analysis of viscoelastic response.] Similarly, Eq. (*21*) can be written as follows

$$\psi\,(t) = P\,[C_0' - \Sigma C_j\,\rho_j\exp(-\,t\,/\tau_j)] \tag{25}$$

In the limit case with $j = 1$, formulae (*23*) and (*25*) reduce to equations (*16*) and (*20*) for Standard Linear Solid.

## 4. Practical part

A suitable procedure for the determination of material parameters is as follows. The indenter is loaded quickly (with the constant load rate $R$) to the nominal load $P$, then kept under this load for a relatively long time, and unloaded quickly. From the unloading curve, the contact stiffness can be determined, either directly as $S = \mathrm{d}P/\mathrm{d}t$ if the indenter velocity at the end of dwell was negligible, or using correction (*7*). The contact area $A$, necessary for the determination of instantaneous elastic modulus $E_0$ from the unloading curve via Eqs. (*5*) and (*6*), is calculated from the contact depth $h_c$, determined by Eq. (*2*) from the depth $h$ at the beginning of unloading.

The dwell under constant load is used for the determination of constants in rheological (spring and dashpot) models. Depending on the model, the function $\psi(t)$, defined by Eq. (*20*) or (*21*), is inserted into (*8*), together with the function $f$ and constant $K$, chosen from Eq. (*9*) or (*10*) with respect to the indenter shape. This function $f[h(t)]$ is then used to fit the measured $h(t)$ data.

The constants $C_0$, $C_1$, $\tau_1$, $\rho_1$, etc. can be obtained by minimising the sum of squared differences between the measured and calculated $h(t)$ values. A suitable tool for this purpose is Solver, available in some universal computer programs, such as Excel. However, the actual procedure must be modified for the following reason. The constants $C_j$ appear in Eqs. (*20*), (*21*) and (*25*) only together with the constant $\rho_j$ (as product $C_j\rho_j$), or together with the constant $C_0$ (as $C_0'$, cf. Eq. (*24*)). Thus, the regression fitting of experimental data can yield the correct values of $C_j\rho_j$ and $C_j'$, while the individual values of constants $C_j$ can be wrong. The constants $C_j$, however, are the genuine material parameters, independent of the loading history, and must be determined accurately. Their correct values can be obtained using a simple three-step data processing, based on the verified fact that the retardation times $\tau_j$ can be determined correctly for any mathematical form of the model (*25*).

In the first step of the procedure, the $h(t)$ data obtained by indentation during the constant-load part of the test are fitted by the function (*25*), with all constants $C_0'$, $C_1$, $\tau_1$, $\rho_1$, etc. considered as "free". In this way, the retardation times $\tau_j$ are obtained. Then, the ramp correction factors $\rho_j$ are calculated from Eq. (*22*) for these times $\tau_j$ and the duration $t_R$ of the load increase. These values $\rho_j$ are then inserted as fixed constants into Eq. (*25*) or (*21*), and the curve fitting, now searching for the remaining constants $C_0$, $C_1$, $\tau_1$, etc. is done again. Computer modelling has shown that this procedure yields correct results.

REMARK. Due to viscoelastic deformations, the indenter depth at the end of dwell is larger than at the end of load increase, so that also the contact area is larger. This can influence the calculated value of the constant $C_0$. For this reason (and for a check), it is recommended to determine this constant (and elastic modulus $E_0$) using also a separate test with fast loading followed immediately by unloading.

From the compliances $C_0$, $C_1$, etc., it is also possible to calculate the values of $G_0$, $G_1$, etc. for the (chosen) value of Poisson ratio $\nu$. In an ideal case, the instantaneous shear modulus $G_0$ is related to the (instantaneous) tensile modulus $E_0$ from the unloading curve as $G_0 = E_0/[2(1+\nu)]$. If the calculated parameters do not fulfil this condition, it is an indication that a correction is necessary, e.g. for the delayed deforming during the load increase period.

When looking for a suitable model, one should be aware that very complex models, with more than about 6–7 regression constants, can sometimes cause problems in the search for their accurate values, and a compromise between the model complexity, accuracy and "robustness" may be necessary. A similarly good fit is sometimes obtained for various arrangements of springs and dashpots. The model complexity should also respect the amount of experimental data available, especially regarding the test duration. If the measurement lasts only several tens of seconds, it is impossible to obtain accurate information about the behaviour under a load lasting several hours or more.

Finally, it should by reminded that Eqs. (*16*), (*17*), (*20*) and (*21*) are only valid for reversible viscoelastic deformations, which occur under a spherical indenter (sometimes even under pointed indenter[10]) if sufficiently low load is used. For higher loads, also plastic deformations appear. The characterisation of viscoelastic-plastic materials with permanent deformations exceeds the scope of this paper and will be the topic of another work[11].

## 5. Summary

Mechanical properties of viscoelastic materials with time-dependent response can be determined by instrumented indentation, which continuously measures indenter load and displacement. The paper has explained the principle of the method as used for elastic-plastic materials, gave the basic formulae for expressing the load response of visco-elastic materials by means of spring-and-dashpot models, and described a procedure for the indentation testing and the determination of material parameters.

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**J. Menčík** *(University of Pardubice, Jan Perner Transport Faculty, Pardubice, Czech Republic):* **Determination of Parameters of Viscoelastic Materials by Instrumented Indentation**

Deformation of viscoelastic materials depends on the load magnitude and history. Hardness is not constant, but decreases with the time under load. Universal description of the response is possible by means of rheological models consisting of springs and dashpots. The constants in these models can be obtained from the time course of penetration of an indenter loaded by constant force. In real tests it is also necessary to respect the period of the load increase to the nominal value, and to make a correction of the contact stiffness and area, determined from the unloading curve. The paper explains the principles of instrumented indentation, brings the formulae for indenter penetration into viscoelastic material under increasing and constant load, and describes a practical procedure for measurement and data processing.