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# Analysis of nanoindentation curves in the case of bulk amorphous polymers

Load versus displacement curves obtained from nanoindentation experiments on polymers are particularly difficult to analyze due to their complex mechanical and especially time-dependent behavior. The indentation curves obtained on two amorphous polymers (polycarbonate (PC) and polymethyl methacrylate (PMMA)) show an increase in the indentation depth during load hold time. A non-linear time-dependent model shows that this phenomenon could be decomposed in a first part due to a viscoelastic response and a second part assigned both to a viscoelastic and a viscoplastic response. If the first part is not achieved, the values of the elastic modulus computed using the Oliver and Pharr method are abnormally high. If it is achieved, the beginning of the unloading curve presents an abnormality that is linked to the increase in indentation depth during hold load time. Nevertheless, if the load hold time is high enough and if the contact stiffness is calculated from the initial slope of the power fit of the unloading curve, the values of the elastic modulus computed using the Oliver and Pharr method are close to those obtained by tensile tests.

**Keywords:** Nanoindentation; Polymer; Elastic modulus; Hardness; Viscosity

## 1. Introduction

Nanoindentation methods are used routinely to measure the mechanical properties of bulk materials or thin films. This instrument allows measuring the load applied on a tip as a function of indentation depth. Hardness and elastic modulus may be obtained from the loading and unloading curves [1–4].

Nanoindentation gives satisfactory results for isotropic elastoplastic work hardening materials. Extension of the method to anisotropic [4], non-work hardening [5] or viscous materials [6, 7] is more limited. In the case of polymeric and biological materials, the time-dependent response leads to ambiguity in the measurement of the mechanical properties. Whatever the method, values of the elastic modulus obtained by nanoindentation experiments are higher than the values obtained by tensile tests [7]. This difference could be due to several reasons. Deformation mechanisms are sometimes different in traction and compression, and materials could be heterogeneous, especially in the vicinity of the surface. Some authors explain the high values of the elastic modulus obtained by nanoindentation

by an underestimation of the contact area due to the presence of pile-up or the effect of viscoelasticity [6, 7]. It is, for example, well-known that there is a nose in the unloading curve at low unloading rate or for a short hold time [6]. To avoid this problem, it is advisable to maintain the load at its maximum value during a long enough time or to unload at a high loading rate [6]. Whatever, it is important to ensure that the viscoelastic response to load is achieved before unloading.

In this work, the nanoindentation curves of two amorphous polymers have been analyzed. It shows that the beginning of the unloading curve is marked by the history effect of the loading. Nevertheless, if the peak hold time is large enough and if the contact stiffness is calculated from the slope of the power fit of the unloading curve, the elastic moduli obtained by nanoindentation are close to the elastic moduli calculated from tensile tests.

## 2. Materials and methods

Two bulk amorphous polymers (polycarbonate (PC) and polymethyl methacrylate (PMMA)) were tested: Amorphous polymers have been chosen rather than semi-crystalline polymers to prevent any heterogeneity of the crystallinity and of the mechanical properties of the material. PC was from Axxis<sup>TM</sup>, it has a ductile behavior. The nominal mechanical properties are tensile elastic modulus ( $E_{\text{Tensile}}$ ) 2.3 GPa and elastic limit ( $R_e$ ) 50 MPa. Poisson's ratio ( $\nu$ ) is equal to 0.4. PMMA was from Altuglas<sup>TM</sup>, it has a brittle behavior. The nominal mechanical properties are  $E_{\text{Tensile}} = 3.3$  GPa,  $R_e = 74$  MPa and  $\nu = 0.39$ .

Tensile tests were performed on a Zwick<sup>TM</sup> Z010 tensile test machine at a strain rate of  $0.011 \text{ s}^{-1}$ . The elastic moduli determined by tensile test were 2.24 and 3.10 GPa for PC and PMMA, respectively. All nanoindentation experiments were performed using a Nanoindenter XP from MTS<sup>TM</sup> using the continuous stiffness measurement (CSM) method with a frequency of 45 Hz and displacement amplitude of 2 nm. A Berkovich indenter was used. The coefficient ( $C$ ) of the indenter area function that gives the contact area ( $A$ ):

$$A = Ch_c^2 \quad (1)$$

was calibrated using fused silica ( $E = 72$  GPa,  $\nu = 0.17$ ) using the method proposed by Hochstetter et al. [6]. The coefficient  $C$  was calculated to be 24.44.

The mechanical properties were extracted from the load–displacement curves using the Oliver and Pharr meth-

od [4]. Briefly, the contact depth  $h_c$  was computed from the indentation depth  $h_t$  using the following equation:

$$h_c = h_t - \varepsilon \frac{F}{S} \quad (2)$$

where  $\varepsilon$  is a geometrical parameter,  $F$  the load and  $S$  the contact stiffness. According to the work of Pharr and Bolshakov [8],  $\varepsilon$  was calculated from the following equation

$$\varepsilon = m \left[ 1 - \frac{2(m-1)}{\sqrt{\pi}} \frac{\Gamma\left(\frac{m}{2(m-1)}\right)}{\Gamma\left(\frac{1}{2(m-1)}\right)} \right] \quad (3)$$

where  $m$  is the exponent of the power law of the unloading curve and  $\Gamma$  is the gamma function. Typically, in our experiments,  $\varepsilon$  was in the range 0.71–0.75.

The contact area was computed using Eq. (1). The hardness ( $H$ ) is defined as the ratio of the load on the contact area

$$H = \frac{F}{A} \quad (4)$$

and the elastic modulus was computed using the Sneddon's Eq. [9]

$$S = 2\beta E^* \cdot \sqrt{\frac{A}{\pi}} \quad (5)$$

where  $\beta$  is a correcting factor to take into account the non-axisymmetry of the indenter and is equal to 1.034 [10], and  $E^*$  is the reduced elastic modulus:

$$\frac{1}{E^*} = \frac{1 - \nu_1^2}{E_s} + \frac{1 - \nu_i^2}{E_i} \quad (6)$$

where  $E_i$ ,  $E_s$ ,  $\nu_i$ ,  $\nu_s$  are the elastic moduli and Poisson's ratios of the indenter and sample, respectively. Because polymers are time-dependant materials, the unloading curve is sensitive to history effects and strain rate.

In order to reduce the influence of the viscosity the following indentation procedure has been chosen. All experiments were performed using the following load time sequence. The indenter was first loaded at a constant loading rate/load ratio [11] ( $\dot{L}/L = C$ ) to an indentation depth equal to 3000 nm. The value of the constant loading rate/load ratio varies from  $10^{-3}$  to  $0.3 \text{ s}^{-1}$ . For the lower loading rate/load ratio, the loading time is important and the thermal drift is no longer negligible. For the higher loading rate/load ratio, the experimental curves are noisy. For these two ratios, the results are poorly reproducible.

A constant loading rate/load ratio allows obtaining a constant strain rate if the material has a constant hardness. The principle of geometric similarity is then valid even if the material exhibits viscous behavior [12]. The load was then held constant at peak load for a period of time that varies from 0 to 600 s. The indenter was then unloading at a constant unloading rate/load ratio ( $\dot{L}/L = C$ ) until the load reached a value that varied from 1 to 50 % of the maximum load value. The principle of geometric similarity is not valid for the unloading stage because of the non-reversibility of plastic deformation. Nevertheless, the Oliver and Pharr method proposes to compute the contact stiffness using the

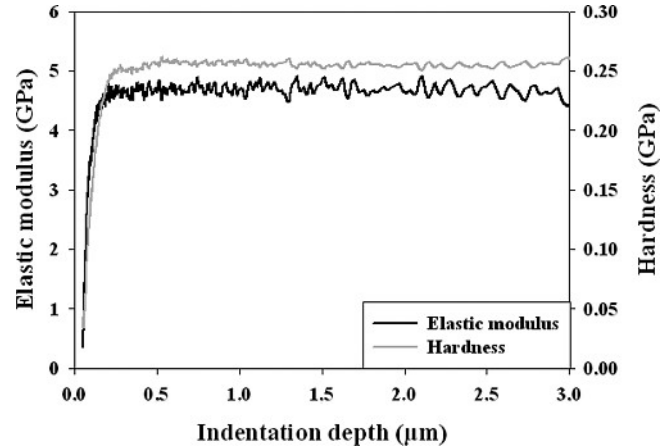


Fig. 1. Elastic modulus and hardness obtained using the CSM method as a function of indentation depth (PMMA  $\dot{L}/L = 0.03 \text{ s}^{-1}$ ).

initial slope of the power fit of the unloading curve. Because polymers are time-dependent materials, it is important that the unloading curves are performed at a strain rate that is as constant as possible. We propose to perform the unloading curve at a constant unloading rate/load ratio, hypothesizing that these experimental conditions give a more constant strain rate than unloading at constant unloading rate. After unloading, the load was then held constant for a period of 600 s.

The two hold load periods (after loading and unloading) are performed to analyze the time dependent behavior of the material and to distinguish the reversibility (viscoelastic) and irreversibility (viscoplastic) of the increase in indentation depth during the peak time. The indentation depth is computed by including the tip defect (the missing portion of the indenter from a perfect Berkovich indenter) according to the method proposed by Hochstetter et al. [6]. The contact stiffness by means of the CSM method is plotted as a function of indentation depth. The origin of the linear relation between contact stiffness and indentation depth gives the virtual first contact point (the first contact point if the indenter has a perfect geometry) and allows one to obtain a constant elastic modulus and hardness (by means of the CSM method) as a function of indentation depth (Fig. 1).

The contact area computed from the Oliver and Pharr method will be compared to the residual imprint measured by means of atomic force microscopy (AFM) imaging. For this purpose, a topographic image is realized using a Dimension 3100 instrument (Nanoscope IIIa from Veeco<sup>TM</sup>), the tapping mode<sup>TM</sup> operating system and a silicon cantilever (OTESPA from Veeco<sup>TM</sup>). A higher slope image is computed from the topographic image according to the following procedure: The topographic image is processed by a Kernel filter in order to obtain 4 slope images according to the X, Y, XY and  $-XY$  directions. The higher slope image is built by taking for each pixel of the image the higher absolute value of the 4 slope images. The higher slope image clearly shows the residual imprint of the indenter (Fig. 2). The borders of the residual imprint are fixed by 3 curved lines and its area is computed by integration. The measurement of the area of the residual imprint is not very accurate due to the inaccuracy of the AFM calibration. The inaccuracy of the measurement was estimated to be approximately 5 %.

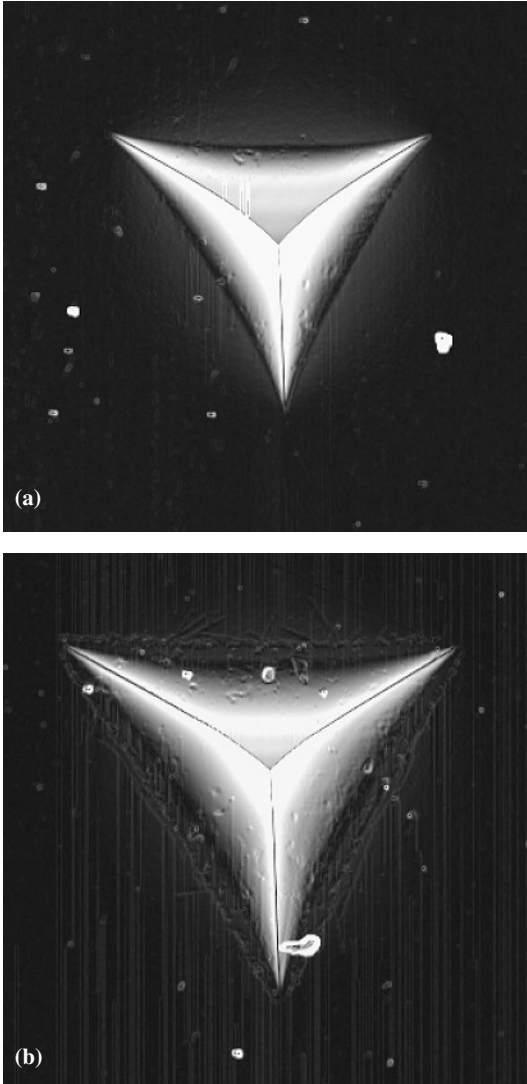


Fig. 2. AFM images ( $40\ \mu\text{m} \times 40\ \mu\text{m}$ ) of the residual imprint for (a) PC and (b) PMMA sample. Two imprints are clearly visible for PMMA.

### 3. Analysis of the load–displacements curves

During the peak hold time, the indentation depth continues to increase even after a very long time (more than one hour). It is not obvious that this increase is due to a viscoelastic or viscoplastic behavior. Figure 3 shows an indentation curve performed on the PC sample. The peak holding time is equal to 600 s, and the indenter is unloaded at 50 % of the maximum load.

To confirm the nature of the increase in indentation depth, a non-linear model based on the work of Oyen and Cook [13] has been developed. The model is built from a series of independent quadratic mechanical elements with elastic (one spring), viscoelastic (two Kelvin–Voigt elements), plastic (one slider) and viscoplastic (one dashpot) responses (Fig. 4). This model is able to fit perfectly the load–displacement curve (Fig. 3). It shows that the increase in the indentation depth during the peak hold time could be decomposed into two parts. The first part is important and reversible as shown during the unload hold time. The indentation depth velocity follows an exponentially decreasing function with a time constant of a few seconds. It is the re-

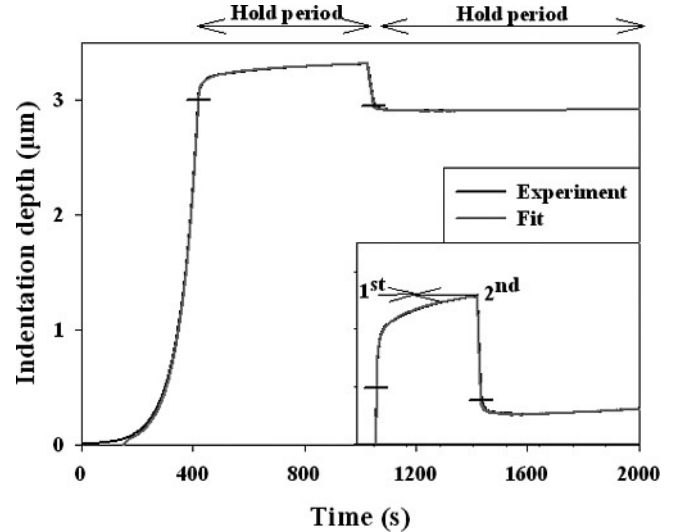


Fig. 3. Indentation depth as a function of time. During hold time, the indentation depth continues to increase. This increase could be decomposed into two parts (inset). The horizontal lines mark the beginning of the hold load periods.

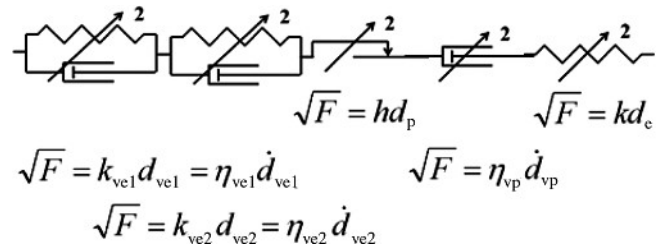


Fig. 4. Viscoelastic viscoplastic model used to fit the indentation curves.

sult of a viscoelastic response. The second part of the increase in the indentation depth is less important, irreversible and seems to follow a constant velocity. It is modeled by the sum of a viscoplastic response and a viscoelastic response with a long time constant.

The elastic modulus is computed according to Sneddon's equation using the contact stiffness by means of the initial slope of the unloading curve. If the first part of the increase of the indentation depth during peak load hold is not achieved, the initial slope of the unloading curve will be influenced both by the elastic-viscous response to unload and by the viscous response to load. As a consequence, the value of the slope of the unloading curve would not be a perfect elastic response. The contact stiffness is computed directly from the initial slope of the unloading curve,  $S_{\text{Tan}}$  (between 90 and 95 % of the peak load value), or from the initial slope of the power law fit of the unloading curve,  $S_{\text{Fit}}$ , leading to two different values of the elastic modulus,  $E_{\text{Tan}}$  and  $E_{\text{Fit}}$ . Figure 5 shows the value of the elastic modulus computed using the Oliver and Pharr method in the case of the PMMA sample as a function of hold time. It confirms clearly that the value of the elastic modulus becomes constant only after a peak hold of approximately 100 s. This time is approximately the same time to achieve the first part of the increase of indentation depth. For an enough long hold time,  $E_{\text{Fit}}$  is close to 3.2 GPa in good agreement with the value obtained from the tensile test (3.1 GPa) whereas  $E_{\text{Tan}}$  is typically 15 % too high.



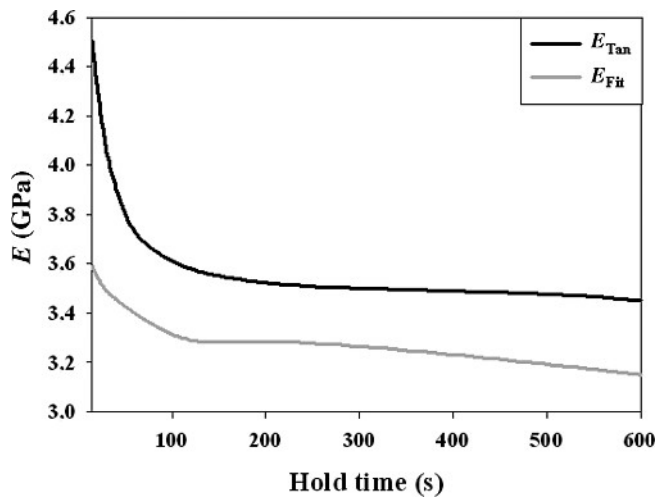


Fig. 5. Elastic modulus as a function of hold time (PMMA,  $\dot{L}/L = 0.05 \text{ s}^{-1}$ ).

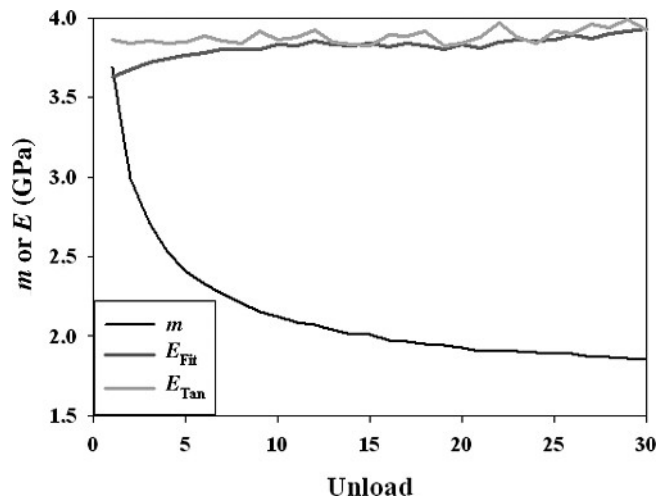
During loading at a constant strain rate, the response of the material is similar to an elastoplastic behavior. In contrast, during load hold time, the response of the material is mainly due to a viscous response. Then, the beginning of unload curve corresponds mainly to the unloading of the increase in indentation depth during load hold time, which means a viscoelastic penetration rather than an elastoplastic penetration.

Analysis of the unloading curves gives important information. The exponent of the power law fit of the unloading curve is very high as compared to the value obtained for a non-viscous material. Typically, the power law exponent derived from the entire unloading curve is close to 3.7 and 2.2 for PMMA and PC, respectively. According to the work of Pharr and Bolshakov [8], the power law of the unloading curve ( $m$ ) can be related to the effective indenter shape. The effective indenter shape is the shape that results in the same unloading curve on a perfectly elastic material. The effective indenter shape can be described by the following equation

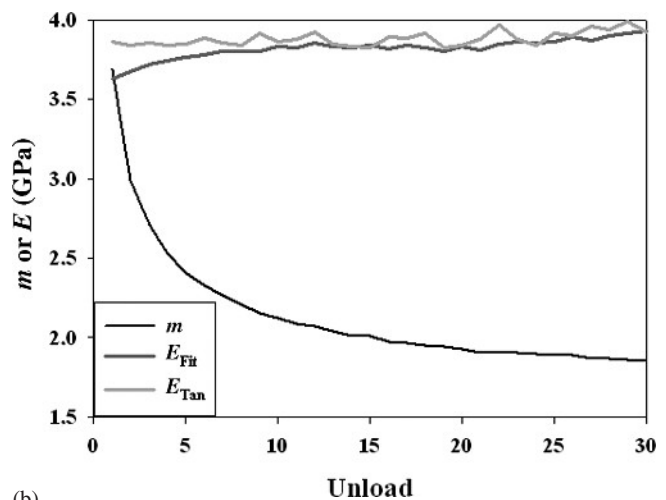
$$Z = Br^{\frac{1}{m-1}} \quad (7)$$

where  $B$  is a coefficient,  $z$  is the vertical distance and  $r$  is the radial distance. According to the experimental values of  $m$ , the effective indenter geometry would be hyperbolic. This means that the contact pressure should be very high in the center of the contact and low at the periphery of the contact. This result is surprising in the case of indentation experiments where a plastic behavior and then a constant pressure distribution is assumed.

This phenomenon may be a consequence of the history effect due to the viscous behavior of the polymer. To check this hypothesis the material is loaded and unloaded a large number of times. The following procedure is repeated 30 times: (1) Loading at a constant loading rate/load ratio ( $0.03 \text{ s}^{-1}$ ), (2) load maintained at maximum value for 600 s, and (3) unload at a constant loading rate/load ratio to 1 % of the maximum load in order to obtain a complete unload. We observe that the exponent of the power law fit decreases rapidly and tends to values close to 1.8 and 1.5 for PMMA and PC, respectively (Fig. 6). The value of 1.5 corresponds to the value obtained on an elastoplastic material [4] whereas the value of 1.8 is intermediate between



(a)



(b)

Fig. 6. Exponent of the power law fit of the unloading curve ( $m$ ) as a function of number of the load/unload cycles ( $\dot{L}/L = 0.03 \text{ s}^{-1}$ ) (a) PMMA, (b) PC.

the elastic ( $m = 2$ ) and the elastoplastic behavior ( $m = 1.5$ ). The elastic modulus computed using the Oliver and Pharr method is approximately constant whatever the number of previous unloads, meaning that the first unloading curve could be used for the analysis. The exponent of the power law fit of the unloading curve is computed as a function of fractions of the unloading curves ( $\Delta F/F_{\text{Max}}$ ) used for the calculus (Fig. 7). We observe that for the first unload the beginning of the unloading curve is best fitted by a power law for which the exponent is close to 7 for a fraction of the unloading curve close to 20 % in the case of PMMA and 7 % in the case of PC. The exponent is not constant, meaning that the unloading curve is poorly fitted by a power law function.

For the 30<sup>th</sup> unloading curve, the beginning of the unloading curve still follows a very high exponent of the power law, but tends to a reasonable value if a larger fraction of the curve is used. After many load–unload cycles, the unloading curves could be well fitted by a power law function, except for the beginning of the curve, with exponent values close to those found for elastoplastic materials ( $m = 1.5$ ). Whatever the number of previous load–unload cycles, the beginning of the curve is abnormal and should be considered with caution.

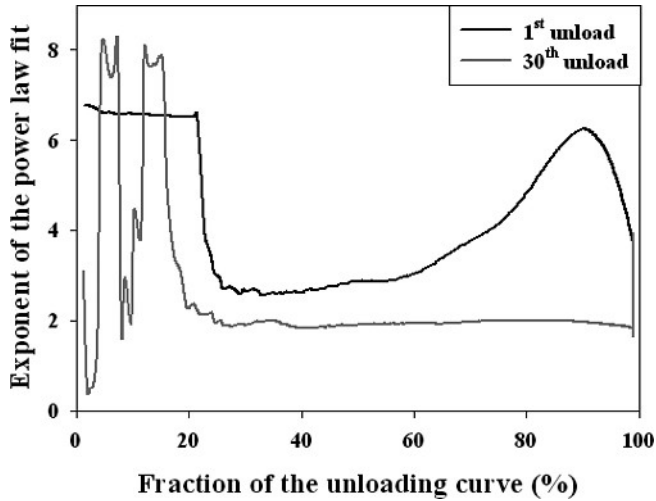


Fig. 7. Exponent of the power fit of the unloading curve as a function of fraction of the unloading curve (PMMA,  $\dot{L}/L = 0.03 \text{ s}^{-1}$ ).

#### 4. Measurement of the elastic modulus

From the analysis of the unloading curve, we have observed the following:

- The elastic modulus computed by means of the Oliver and Pharr method is constant if the peak hold time is higher than a few minutes.
- The elastic modulus is constant whatever the number of previous unloads.
- The beginning of the unloading curve is abnormal as compared to the rest of the unloading curve.

This suggests that the elastic modulus can be computed from the first unloading curve if the peak hold time value is enough high and if the contact stiffness is computed from the power law fit of the unloading curve rather than from the initial slope of the unloading curve.

The following procedure was then employed: The indenter was first loaded at a constant loading rate/load ratio ( $\dot{L}/L = C$ ) [11] until an indentation depth equal to 3000 nm. The loading rate/load ratio varied from  $10^{-3}$  to  $0.3 \text{ s}^{-1}$ . The load was then held constant at maximum load for 600 s. The indenter was then unloaded at a constant unloading rate/load ratio ( $\dot{L}/L = C$ ) until a value equal to 1% of maximum load. Each experiment was repeated 3 times.

The contact stiffness is computed from the initial slope of the power law fit of the unloading curve  $S_{\text{Fit}}$  and from the initial slope of the unloading curve  $S_{\text{Tan}}$  as previously described. The contact area, elastic modulus and hardness are computed according to the Oliver and Pharr method and the two values of the contact stiffness. These values are compared to the experimental values: The area of the residual imprint by means of AFM measurement, the elastic modulus by means of tensile testing and the hardness by means of Eq. (4) (load/area of the residual imprint).

The values of the elastic modulus are plotted as a function of loading rate/load ratio. Figure 8 shows that the elastic modulus is approximately constant for PC:  $E_{\text{Fit}}$  and  $E_{\text{Tan}}$  are computed to be 2.3 and 2.7 GPa, respectively, values to be compared to the value obtained by tensile tests (2.24 GPa). Table 1 presents the values of elastic modulus, hardness and area of the residual imprint measured for a

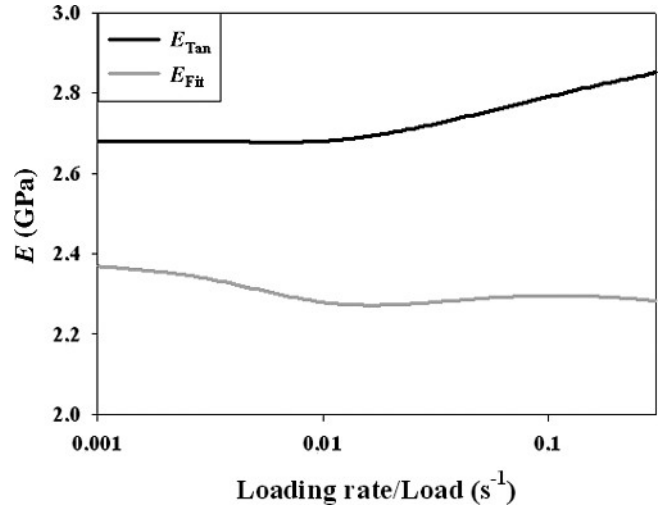


Fig. 8. Elastic modulus of the PC sample as a function of loading rate/load ratio.

loading rate/load ratio equal to  $0.01 \text{ s}^{-1}$ . There is a good agreement between the values measured from the Oliver and Pharr method using  $S_{\text{Fit}}$  and the values measured independently. Figure 9 shows the unloading curve and the best power law fit of the unloading curve. The different slopes at the beginning of two curves confirm that the beginning of the experimental unloading curve presents an abnormality.

PMMA has a much more marked viscous behavior (significant increase in indentation depth during load hold time) than PC. At low loading rate/load ratios, we observe a good agreement between the values of the elastic modulus mea-

Table 1. Mechanical properties obtained on the PC/PMMA sample ( $\dot{L}/L = 0.01 \text{ s}^{-1}$ ). The reference values for  $E$ ,  $H$  and  $A$  are obtained from tensile tests, [Eq. (4)], and AFM residual imprints, respectively.

| Method           | $E$ (GPa) | $H$ (MPa) | $A$ ( $\mu\text{m}^2$ ) |
|------------------|-----------|-----------|-------------------------|
| $S_{\text{Tan}}$ | 2.61/3.67 | 151/192   | 208/238                 |
| $S_{\text{Fit}}$ | 2.22/3.34 | 157/192   | 200/237                 |
| Reference values | 2.24/3.10 | 167/226   | 195/195                 |

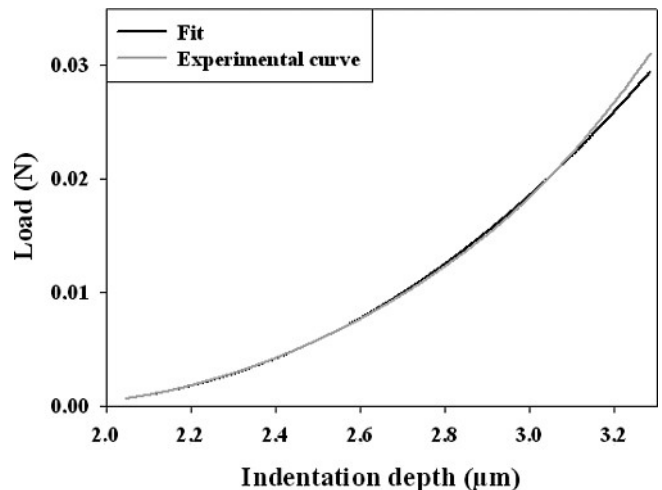


Fig. 9. Experimental unloading curve and power law fit of the unloading curve (PC, 1<sup>st</sup> unload,  $\dot{L}/L = 0.03 \text{ s}^{-1}$ ).

sured by nanoindentation using  $S_{Fit}$  and the elastic modulus measured by tensile testing (Fig. 10). The agreement is poor for the contact area: The difference is about 20 % (Table 1). Nevertheless, if one observes the higher slope image, one can see that there are two residual imprints (Fig. 2b). The first one, in white, has a constant slope and could be considered as a plastic imprint. The second imprint is larger and has an outline for which a slope similar to the surface plane indicating elastic recovery of the border. One noticeable fact is that the area of this second imprint is equal to the area

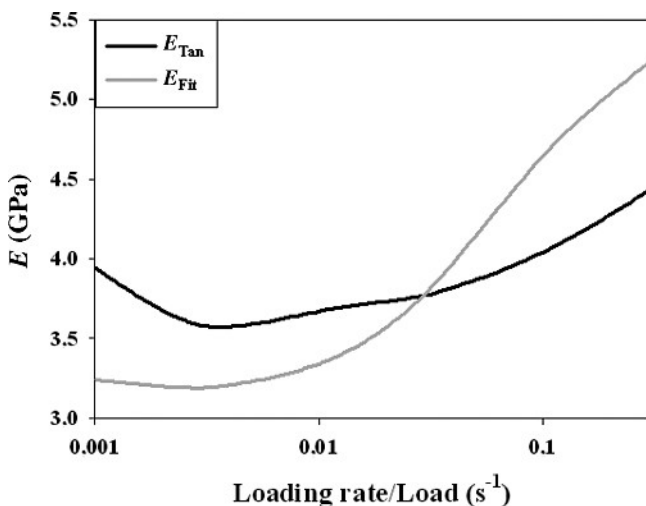


Fig. 10. Elastic modulus of the PMMA sample as a function of loading rate/load ratio.

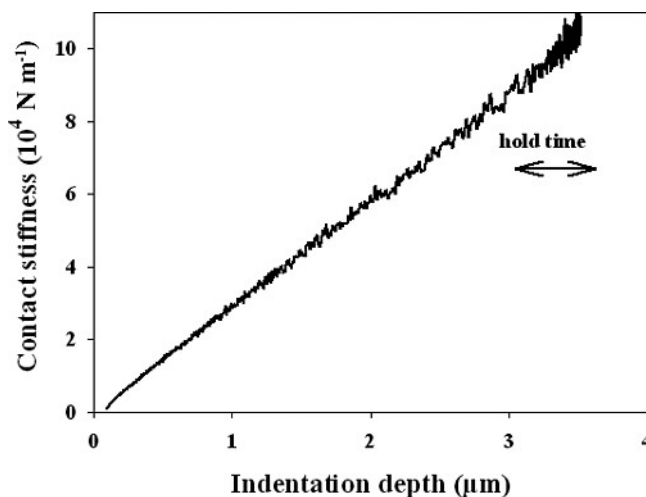


Fig. 11. Contact stiffness as a function of indentation depth. The contact stiffness is proportional to the indentation depth.

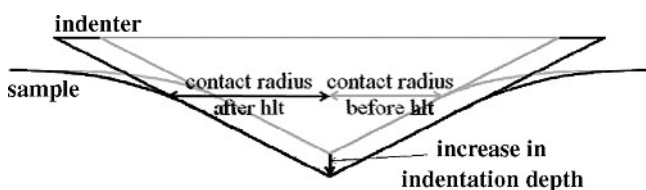


Fig. 12. Increase in indentation depth and contact area during hold load time (hlt). During hold time, due to viscoelastic behavior, the indentation depth increases. As a consequence, the contact area after hlt (black) is larger than before hlt (light gray).

computed by the Oliver and Pharr method. Furthermore, we can see that the contact stiffness by means of CSM measurement is approximately proportional to the indentation depth (Fig. 11). According to the Sneddon equation, the contact stiffness is proportional to the root square of the contact area. This means that during the first part (viscoelastic behavior) and the second part (viscoelastic and/or viscoplastic behavior) of the increase in the indentation depth during hold time, the contact area continues to increase.

This phenomenon is quite surprising because the contact area is supposed to be fixed by hardness and is not supposed to increase for a viscoelastic response. For loading at a constant strain rate, the response of the material was comparable to elastoplastic behavior. Nevertheless, during hold time, due to viscoelastic behavior, the material continues to strain and the indenter depth still increases. An increase indentation depth without an increase contact area seems to be difficult because, at the end of loading, the connection of the surfaces of the indenter and of the sample is a tangent (Fig. 12). During hold time, the viscoelastic response of the material leads to an increase in the contact area, (and then a decrease in the contact pressure), without new plastic flow (elastic contact). Then, the contact evolves from a plastic contact to a elastoplastic contact. The pressure distribution is no longer a plastic pressure distribution but tends to an elasto-plastic distribution. This assumption is confirmed experimentally by the residual imprint of the contact area and the experimental value of the power fit value of the unloading curve.

## 5. Conclusions

Nanoindentation of two amorphous polymers leads to an increase in the indentation depth and contact area during hold load time. This increase could be decomposed into two parts. The first part of this increase is due to viscoelastic behavior whereas the second part is due to viscoplastic and viscoelastic behavior. It is not obvious that the increase in the contact area is followed by an increase in the plastic flow, especially in the case of PMMA where an elastoplastic contact is assumed after hold time.

This viscous behavior is responsible for the abnormal exponent of the power fit of the unloading curve and for the abnormally high slope of the beginning of the unloading curve. Nevertheless, if the peak hold time is long enough, and if the contact stiffness is computed from the initial slope of the power law fit of the unloading curve, the elastic modulus computed using the Oliver and Pharr method is close to the values obtained using tensile tests.

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(Received November 4, 2008; accepted February 6, 2009)

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DOI 10.3139/146.110137  
Int. J. Mat. Res. (formerly Z. Metallkd.)  
100 (2009) 7; page ■ – ■  
© Carl Hanser Verlag GmbH & Co. KG  
ISSN 1862-5282

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