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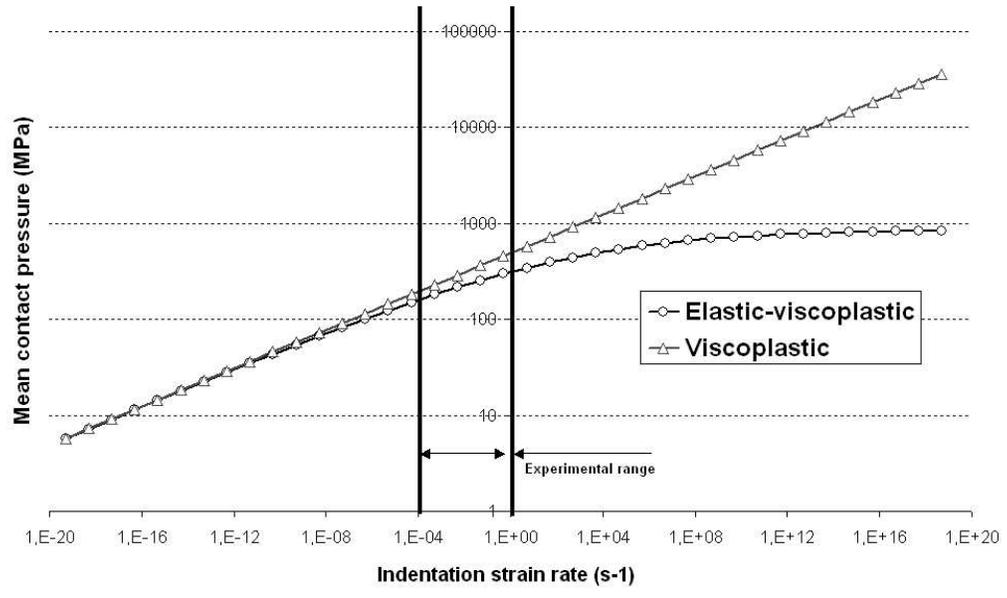


A new index to estimate the strain rate sensitivity of glassy polymers using conical/pyramidal indentation

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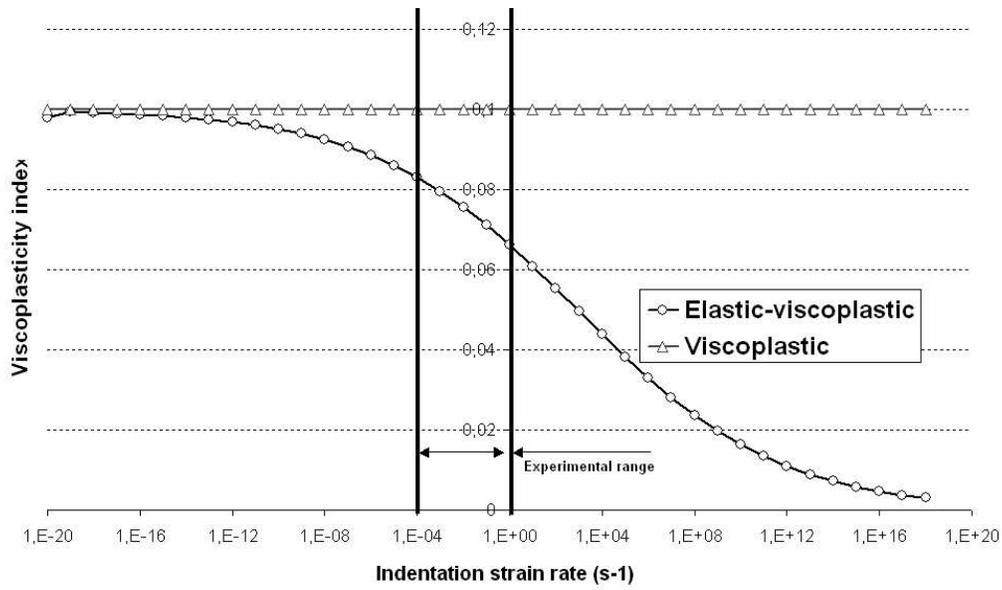


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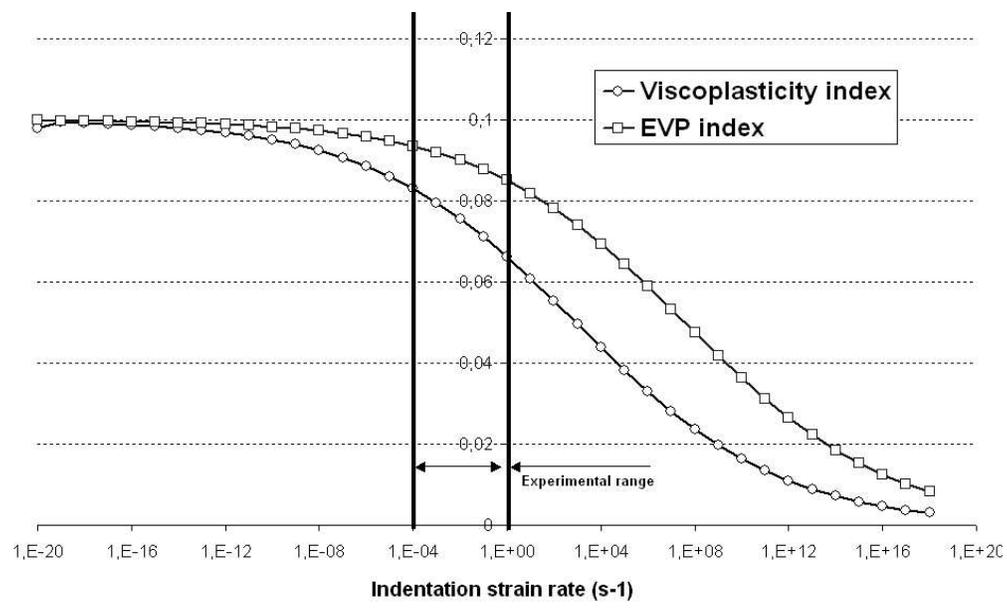


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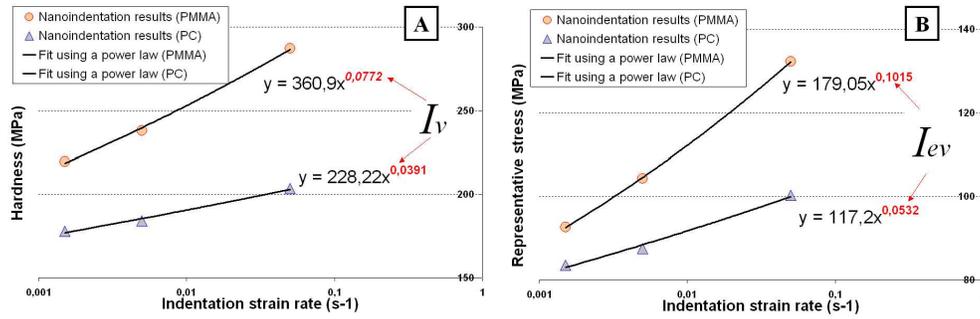
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A new index to estimate the strain rate sensitivity of glassy polymers using conical/pyramidal indentation

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Abstract

The increasing use of polymeric materials as thin coatings requires appropriate mechanical characterization methods taking account of time-dependent effects. A well-known result is that a constant indentation strain rate allows to determine the mechanical properties of such solids from indentation tests. To do that, most people assume a power law creep behavior and use the viscoplasticity index to determine the strain rate sensitivity. In the first part of this paper, we investigate the influence of the indentation strain rate on the hardness of power law creep solids and elastic-viscoplastic solids. It is shown that the assumption of power law creep solid is not sufficient to describe the behavior of polymeric materials in indentation. In the second part, we propose a new index, the EVP index, which gives a better approximation of the strain rate sensitivity than the viscoplasticity index. In the last part, we apply this method to nanoindentation tests on PMMA and polycarbonate samples.

Keywords: Cone indentation, Polymeric materials, Viscoplasticity index, Strain rate sensitivity

1 Introduction

The increasing use of polymeric materials as thin films deposited on substrates requires appropriate techniques of mechanical characterization. Obviously the conventional tensile test cannot

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7 be used to identify their mechanical properties in such conditions and, in recent years, the
8 indentation technique has been developed for that purpose.
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10 The indentation of isotropic elastoplastic material has been widely investigated over the past
11 few years {Tabor [1], Loubet et al [2], Hill et al [3], Dao et al [4], Cheng and Cheng [5]} and
12 many laws from experimental studies and simplified models have been proposed. Recently,
13 the development of nanoindentation techniques has made it possible to finely characterize the
14 mechanical properties of surfaces {Oliver and Pharr [6], Loubet et al [7], Bec et al [8]}. Tabor
15 [1] and Hill [9] have pointed out that the real definition of hardness is the mean pressure under
16 load and that the value of the hardness of ductile solids is essentially a measure of their plastic
17 properties. They showed that the mean pressure can be related to the yield stress of the material
18 by a simple factor which depends on the properties of the indented material and on the shape
19 of the indenter. It was also shown experimentally that, **for self-similar indenter {conical-**
20 **pyramidal tips}**, the indentation of elastoplastic materials follows the principle of geometric
21 similarity. This principle gives a solid theoretical framework which has led to the development
22 of many approximate solutions of elastoplastic indentation problems [10][11]. It is now possible
23 to extract the yield stress and the strain hardening exponent of such solids.
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36 However, currently available theories developed for time-independent materials cannot suit-
37 ably be used to measure the mechanical properties of time-dependent materials such as polymeric
38 materials. Indeed, the principle of geometric similarity is not always satisfied. For power law
39 creep solids, Lucas [12] has observed that the mean pressure is constant during the indentation
40 test when indentation strain rate \dot{h}/h is constant, where h is the penetration depth and, \dot{h} the
41 penetration rate. The same result has been obtained by Cheng and Cheng [5] with the help of
42 a dimensional analysis. **In a previous paper [13], we have shown** that the principle of ge-
43 ometric similarity is satisfied during cone indentation experiments of time dependent materials
44 {viscoelastic solids, elastic-viscoplastic solids}, only if the indentation strain rate is constant.
45 This result is of primary importance for glassy polymers for which the assumption of power law
46 creep solids is not acceptable. Indeed, it is much appropriate to consider an elastic-viscoplastic
47 behavior [14].
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58 In the first part of this paper, we compare the effects of the indentation strain rate on the
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mean indentation pressure of power law creep solids and elastic-viscoplastic solids that follow the Bingham-Norton law [15]. Then, we explain that the viscoplasticity index [16], defined only for power law creep solids, does not equal to the strain rate sensitivity in the case of elastic-viscoplastic solids {such as glassy polymer}. We thus propose a new index, which allows to obtain a better estimation of the strain rate sensitivity than the viscoplasticity index. In the last part, we apply this method to nanoindentation experiments performed on PMMA and PC {polycarbonate}.

2 The effects of the indentation strain rate on hardness values

2.1 Power law creep solids

The assumption of power law creep behavior is very often well adapted to the modeling of anelastic time-dependent materials. The strain rate sensitivity of such materials can be described by a simple power law relation :

$$\sigma = K\dot{\epsilon}^m \quad (1)$$

where σ is the uniaxial stress and $\dot{\epsilon}$ is the uniaxial strain rate. Consistency K and strain rate sensitivity m are materials constants. Cheng and Cheng [5] have shown using dimensional analysis that the mean indentation pressure is given by :

$$p_m = \Pi_\delta K \left(\frac{\dot{h}}{h} \right)^m \quad (2)$$

where Π_δ is a dimensionless function of strain rate sensitivity m and of cone angle β . The same kind of results has been suggested by Storakers [17]. Equation (2) shows that the mean pressure is constant if and only if ratio \dot{h}/h is constant which agrees with the results of Lucas [12]. An approximation of Π_δ is given in reference [13].

2.2 Elastic-viscoplastic solids

Glassy polymers can be modelled as elastic-viscoplastic solids following a Bingham-Norton law [15]. This assumption seems to be well suited to model the loading part of instrumented indentation experiments [14] on such materials. The parameters of the Bingham-Norton law without strain hardening are

- Young's modulus : E ,
- Poisson's ratio : ν ,
- Yield stress at infinitely low strain rate : Y ,
- Consistency : K ,
- Strain rate sensitivity : m .

For glassy polymers, Y is assumed to be equal to zero. The uniaxial constitutive equations of this model are:

$$\dot{\varepsilon}^p = \left(\frac{\sigma}{K} \right)^{\frac{1}{m}} \quad (3)$$

$$\sigma = E\varepsilon^e \quad (4)$$

$$\varepsilon = \varepsilon^e + \varepsilon^p \quad (5)$$

The cone indentation of elastic-viscoplastic solids at constant indentation strain rate has been investigated **in a previous paper [13]**. They showed that the mean pressure, or indentation hardness, can be expressed starting from a purely elastic state for high indentation strain rates and a purely elastoplastic state for low values. The transition between the two states depends on mechanical properties such as consistency, strain rate sensitivity and Young's modulus and also on cone angle β . For a Berkovich tip $\{\beta = 19.7^\circ\}$ and glassy polymers, we proposed the following approximate expression:

$$p_m = \frac{\Pi_\delta K \left(\frac{\dot{h}}{h} \right)^m}{1 + \frac{5.586}{E^*} \Pi_\delta K \left(\frac{\dot{h}}{h} \right)^m} \quad (6)$$

where $E^* = E/(1 - \nu^2)$. Equation above shows that in the limit as $\dot{h}/h \rightarrow 0$, p_m reduces to the mean contact pressure of power law creep solids and in the limit $\dot{h}/h \rightarrow +\infty$, p_m reduces to the mean contact pressure of elastic solids, $p_m = E^* / 5.586$ [18].

2.3 Comparison of these two models

We consider the mechanical properties of PMMA at ambient temperature which has been previously identified by Bucaille [19]. Bucaille showed that for PMMA and Berkovich tips, there

is no influence of the strain hardening on hardness values. Hence, we do not consider the strain hardening of this material. The corresponding mechanical properties are given in table 1.

[INSERT TABLE 1 ABOUT HERE]

The curves $p_m-\dot{h}/h$ have been computed using equation 2 and 6 and are plotted in figure 1. It is clearly shown that the viscoplastic assumption can not be used to model the behavior of PMMA in indentation for the common experimental indentation strain rate range. Moreover, we can remark that the error of hardness determination using the viscoplastic assumption increases with the indentation strain rate. When $\dot{h}/h = 0.0001s^{-1}$ the error is about 23 % and when $\dot{h}/h = 1s^{-1}$, it is up to 50 %. Hence, elastic properties of glassy polymers have to be taken into account to determine time-dependent mechanical properties such as the consistency and the strain rate sensitivity.

[INSERT FIGURE 1 ABOUT HERE]

3 Identification of the strain rate sensitivity

3.1 The viscoplasticity index

The viscoplasticity index [16] is defined as the slope of graph $\ln(p_m)$ to $\ln(\dot{h}/h)$. It is written

$$I_v = \frac{\partial(\ln(p_m))}{\partial\left(\ln\left(\frac{\dot{h}}{h}\right)\right)} \quad (7)$$

As suggested by Lucas et al [12] and Cheng and Cheng [5], this index is a very good mean to determine the strain rate sensitivity for power law creep solids. Unfortunately, when the indented solid exhibits an elastic-viscoplastic behavior, much attention has to be paid to the interpretation of the viscoplasticity index, because I_v varies with \dot{h}/h . **According to equations (6) and (7), we can write:**

$$I_v = m \left(1 - \frac{5.586p_m}{E^*}\right) \quad (8)$$

Equation (8) shows that when $\dot{h}/h \rightarrow 0$, I_v reduces to the strain rate sensitivity m . In return, when $\dot{h}/h \rightarrow +\infty$, I_v is quasi-null. Consequently, the viscoplasticity index under-estimates the strain rate sensitivity of the material. To illustrate this, the

curves $I_v-\dot{h}/h$ are plotted in figure 2 in the case of a power law creep solid and in the case of an elastic-viscoplastic solid.

[INSERT FIGURE 2 ABOUT HERE]

3.2 A new index to estimate the strain rate sensitivity

The **principle of geometric similarity** being satisfied during a cone indentation at constant indentation strain rate, a representative stress σ_r and a representative strain ε_r , characterizing the stress and strain distributions, and corresponding to each other on the uniaxial stress-strain curve can be defined [1][13]. A representative strain rate $\dot{\varepsilon}_r$ corresponding to the strain rate of a uniaxial test can thus also be defined. For glassy polymers, the following relation can be applied: $\sigma \propto \dot{\varepsilon}^m$ [20]. Assuming that the representative strain rate of the indentation test is given by $\dot{\varepsilon}_r \propto \dot{h}/h$ [19], it comes:

$$\sigma_r \propto \left(\frac{\dot{h}}{h} \right)^m \quad (9)$$

where σ_r is the representative stress of the indentation test. Let us now introduce a new index I_{ev} {called EVP index} :

$$I_{ev} = \frac{\partial(\ln(\sigma_r))}{\partial \left(\ln \left(\frac{\dot{h}}{h} \right) \right)} \quad (10)$$

It is now necessary to estimate representative stress σ_r of indentation test. Tabor [1] suggested to relate σ_r to the hardness of the materials:

$$\sigma_r = \frac{p_m}{\gamma_p} \quad (11)$$

where γ_p is a function of the cone angle. For a berkovich indenter, γ_p is about 2.8. The Tabor's representative stress is well adapted to the indentation of rigid plastic solids. Unfortunately, when elastic deformations are not negligible, this definition does not work. Moreover, we can note that equation (11) leads to $I_{ev} = I_v$.

A more general approximate solution to berkovich indentation of elastoplastic solids has been given in a previous paper [11]. It is written:

$$\sigma_r = \frac{0,087p_m}{0,243 - 0,783 \frac{p_m}{E}} \quad (12)$$

For rigid viscoplastic solids $\{p_m/E \rightarrow 0\}$, the equation (12) reduces to equation 11 with $\gamma_p = 2.8$. Hence, I_{ev} equals to I_v in the case of power law creep solids. **For elastic-viscoplastic solids, the definition of representative stress σ_r given in equation (12) yields to:**

$$I_{ev} = I_v \left(1 + 0,783 \frac{\sigma_r}{0,087E} \right) \quad (13)$$

Equation (13) shows that the value of I_{ev} cannot be lower than I_v . Moreover, combining equations (13), (12), (7) and (6), it is easy to show that I_{ev} cannot be greater than strain rate sensitivity m . Hence, we have:

$$m > I_{ev} > I_v \quad (14)$$

Consequently, the EVP Index is always a better approximation of strain rate sensitivity m than the viscoplasticity index in the case of Bingham-Northon elastic-viscoplastic solids. In figure 3, EVP index I_{ev} and viscoplasticity index I_v are plotted according to indentation strain rate \dot{h}/h . I_v and I_{ev} are computed using equations 6 and 12 and using mechanical properties of PMMA {table 1}.

[INSERT FIGURE 3 ABOUT HERE]

4 Application to nanoindentation experiments

Nano-indentation tests on PMMA and PC samples have been performed with the help of a Nano-indenter XP {MTS®}. The aim of this experimental work is to show that the EVP index is easy to use on nanoindentation results and that it gives relevant results for these two polymers. No polishing procedure is used on these materials. The roughness of samples, as taken, is below $0.1 \mu\text{m}$ peak to peak. The tests conditions are:

- Continuous Stiffness $\{\Delta h=1,5 \text{ nm} ; \omega=32\text{Hz}\}$
- Max. Load: 15 mN
- $\dot{h}/h : 0.0015 \text{ s}^{-1}, 0.005 \text{ s}^{-1}, 0.05 \text{ s}^{-1}$
- Berkovich tip
- 5 tests for each \dot{h}/h

Oliver and Pharr model [6] is used to estimate the contact area between the indenter and the solid:

$$h_c = h - 0.75 \frac{F}{K_c} - h_0 \quad (15)$$

where h_c is the contact depth, h is the penetration depth, h_0 is the indenter tip defect, F is the load applied on the indenter and K_c is the contact stiffness. The contact area is given by $A_c = 24.5h_c^2$ and the hardness is computed as $\frac{F}{A_c}$. The calibration method of Loubet [7] is employed to avoid thermal drift errors and to estimate h_0 . For such solids, it has been shown that the Oliver and Pharr model gives acceptable values of hardness [21]. The nanoindentation results are summarized in table 2. σ_r is computed using equation (12). For glassy polymers such as PMMA, the Young's modulus measured by nanoindentation experiments depends on the continuous stiffness frequency [22]. Hence, it does not always correspond to the one which has to be used in equation (12). **For this reason, the value proposed by Bucaille [19], $E=4200$ MPa for PMMA and $E=2400$ MPa for PC, are used to compute σ_r .**

[INSERT TABLE 2 ABOUT HERE]

the curves $p_m-\dot{h}/h$ and $\sigma_r-\dot{h}/h$ are plotted in graph 4. The viscoplasticity index and the EVP index are determined by a power law fit of these curves. **For PMMA, I_v is about 0.077 and I_{ev} is about 0.1. The reference value of PMMA strain rate sensitivity is about 0.1 {see table 1} which agrees well with I_{ev} . For PC, it gives 0.039 for I_v and 0.053 for I_{ev} . According to reference [14], the strain rate sensitivity of PC is about 0.05.**

[INSERT FIGURE 4 ABOUT HERE]

5 Discussion

The experimental results are in good agreement with equation (13) which shows that the value of I_{ev} is greater than the value of I_v for elastic-viscoplastic solids. Moreover, we can observe that for PC and PMMA, the value of I_{ev} determined using nanoindentation experiments is very close to the reference values of the strain rate sensitivity. For PMMA, we can remark that the viscoplasticity index leads to an error up to 25 %. For these two materials, the EVP index gives thus a better approximation of the strain rate sensitivity than the viscoplasticity index.

The use of I_{ev} is based on the assumption that the indented material follows a Bingham-Norton elastic-viscoplastic behavior with $Y = 0$. If Y does not equal to 0, the development

of section 3 are no more valid and thus the use of I_{ev} may lead to wrong estimations of strain rate sensitivity. However, it can be shown analytically that equation (14) is still valid for $Y > 0$. Hence, we recommend to use I_{ev} rather than I_v to estimate strain rate sensitivity of elastic-viscoplastic solids.

Nevertheless, it is important to keep in mind that the EVP index is not always a good approximation of strain rate sensitivity of materials. It highly depends on the experimental indentation strain rate range and on the mechanical behavior of the indented solid. If the assumption of $Y = 0$ is correct for glassy polymers like PC or PMMA, it is not the case for metallic materials. Consequently, care has to be taken concerning the use of this index.

6 Conclusions

In this paper, it has been shown that the assumption of power law creep solids is not always correct to describe the indentation behavior of time-dependent materials such as glassy polymers. In fact, it is necessary to take into account the influence of elastic properties on instrumented indentation hardness measures. Thus an elastic-viscoplastic formalism seems to be much adapted. Consequently, it points out that viscoplasticity index I_v , defined only for power law creep solids, may lead to wrong estimations of the strain rate sensitivity.

For this reason, we propose a new index, EVP index I_{ev} , which can be used instead of the viscoplasticity index to estimate the strain rate sensitivity. This index is based on a power law fit of the representative stress - indentation strain rate curve. For that, we use a previous work to estimate the representative stress from instrumented indentation results {hardness and Young's modulus}. Obviously, the EVP index will only give an approximation of the strain rate sensitivity. Nevertheless, it is shown theoretically that the estimation of the strain rate sensitivity will be better with I_{ev} than with I_v . PMMA and PC nanoindentation results are in good agreement with these conclusions.

The use of EVP index is as easy as the use of the viscoplasticity index. It can be employed directly after nanoindentation experiments at different indentation strain rates and it does not require any inverse methods or complex solving operation to obtain a first approximation of the strain rate sensitivity.

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E {MPa}	K {MPa.s ^m }	m
4200.0	205.0	0.1

Table 1: Mechanical properties of PMMA [19]

\dot{h}/h	PMMA		PC	
	p_m	σ_r	p_m	σ_r
0.05	287 ± 5	132 ± 3.5	204 ± 1.5	100 ± 1
0.005	238 ± 1	105 ± 0.4	184 ± 0.5	87 ± 0.4
0.0015	220 ± 1.2	93 ± 0.6	178 ± 1	83 ± 0.5

Table 2: Results of nanoindentation on PMMA and PC samples. σ_r is computed using equation (12)

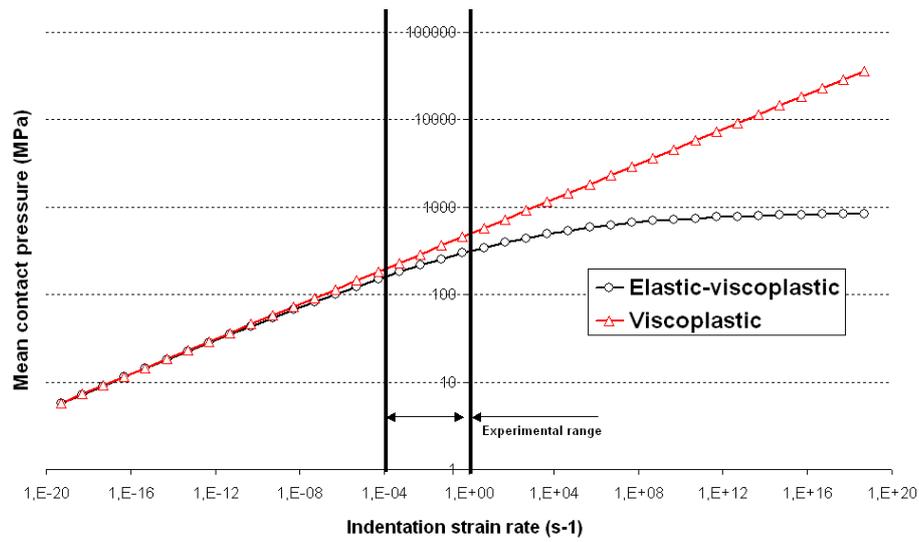


Figure 1: Comparison of PMMA hardness using a viscoplastic model {power law creep} and an elastic-viscoplastic model

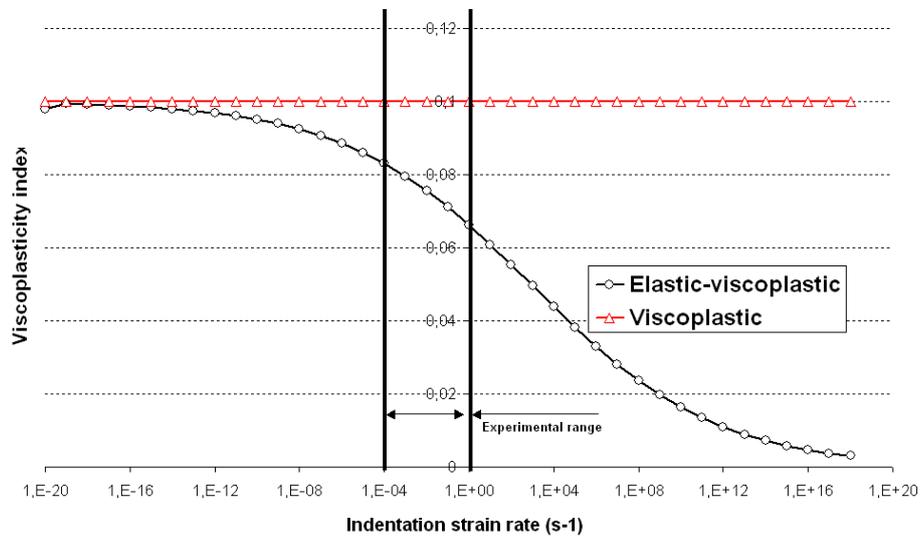


Figure 2: Viscoplasticity index I_v versus the indentation strain rate \dot{h}/h for a power law creep solid $\{K=205 \text{ MPa}\cdot\text{s}^{-1}, m=0.1\}$ and for an elastic-viscoplastic solid $\{K=205 \text{ MPa}\cdot\text{s}^{-1}, m=0.1, E=4200 \text{ MPa}\}$

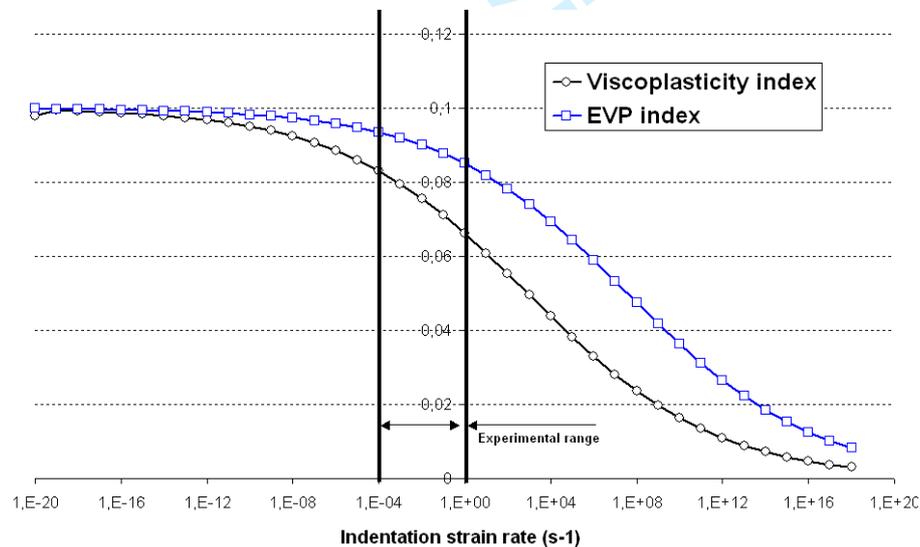


Figure 3: Viscoplasticity index I_v and EVP index I_{ev} vs \dot{h}/h for an elastic-viscoplastic solid representative of the behavior of PMMA $\{K=205 \text{ MPa}\cdot\text{s}^{-1}, m=0.1, E=4200 \text{ MPa}\}$

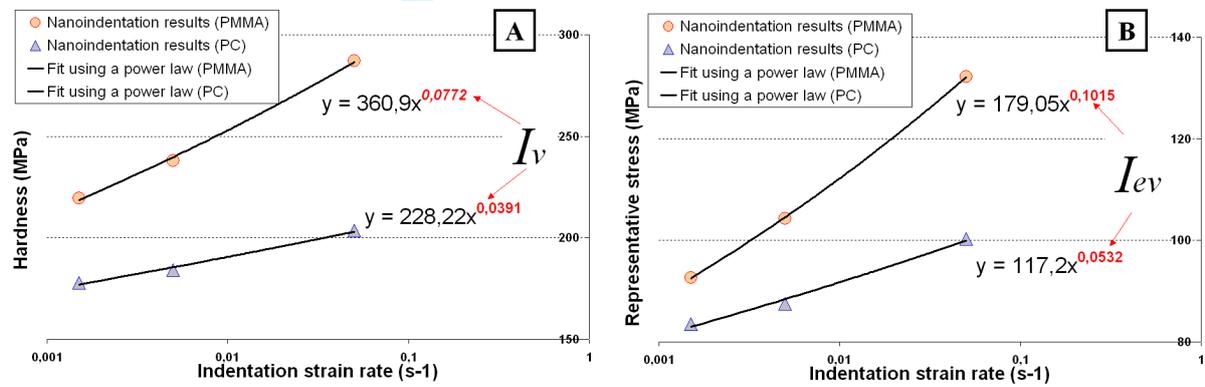


Figure 4: Comparison between I_v , I_{ev} and strain rate sensitivity m for PMMA and PC, A : determination of I_v using a power law fit of the $p_m-\dot{h}/h$ curve, B : determination of I_{ev} using a power law fit of the $\sigma_r-\dot{h}/h$ curve