NUMERICAL-EXPERIMENTAL METHOD FOR THE IDENTIFICATION OF PLASTIC PROPERTIES OF POLYMERS FROM MICROHARDNESS TESTS

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ABSTRACT

A numerical-experimental method for the determination of the plastic properties of polymers from ultramicrohardness tests is developed. Experiments have been performed on five different polymer materials. The yield stress and the tangential modulus are the parameters to be identified. The method is based on the finite element simulation of the indentation contact problem. By using the method of experiment design and the data calculated by the finite element solution, simple models containing the parameters to be identified have been obtained. These simple models together with the experimental data obtained in an indentation experiment are used to derive the yield stress and tangential modulus. Good agreement between the experimental and the finite element results derived from the identified parameters has been observed.

1. INTRODUCTION

In a previous paper (Rikards *et al.*, 1997) the elastic properties of polymers from indentation experiments were obtained. Experimental data of five different polymers were employed to determine the plane strain elastic modulus $E^* = E/(1-\nu^2)$, where E is the Young's modulus and ν is the Poisson's ratio.

In the present investigation the mixed numerical-experimental technique is used to determine the plastic properties of polymers. The same experimental data have been employed to identify the yield stress, σ_y , and the tangential modulus, E_T , of five different polymers.

The numerical-experimental method proposed in the present study consists of the following stages: 1) the experimental data are obtained; the parameters to be identified are selected; the domain of search and the identification functional containing the experimental data have also been selected. 2) The finite element method is used in order to model the microindentation test. The finite element calculations are performed at the reference points of the variables (σ_y and E_T) to be identified. The reference points are determined by using the method of experiment design. 3) The results of the finite element solution at the reference points are used to derive a simple function relating the load with the indentation depth, the yield stress σ_y and the tangential modulus E_T . 4) On the basis of a simple model, σ_y and E_T are determined. For this purpose, the corresponding functional has been minimized by using the method of non-linear programming. The functional characterizes the differences between the predicted loads calculated by the numerical model and the experimentally measured values of the load at different indentation depths.

In the present investigation, the finite element solution has been obtained by using a finite element program (ANSYS, 1994). The pyramidal Vickers indenter is approximated by an axisymmetric cone (Bhattacharya and Nix, 1988a; 1988b; 1991). The contact problem has been solved taking into account the plasticity of the material and the friction along the surface of the indenter.

The method of experiment design for solving the problem of identification is similar to the optimal design procedure. An optimization algorithm for structures using the method of experiment design has been discussed earlier (Rikards, 1993; Rikards *et al.* 1995; Rikards and Chate, 1995).

2. EXPERIMENTAL

2.1 Materials and techniques

Five different polymers were tested: amorphous and semicrystalline poly(ethylene terephthalate) (PET), semicrystalline poly(vinylidene fluoride) (PVF₂), the 60/40 copolymer of poly(vinylidene fluoride) and trifluoroethylene P(VF₂/F₃E) and high pressure crystallized polyethylene (PE). Further details are given in Table 1. The experiments were performed using a Shimadzu dynamic ultramicrohardness tester provided with a Vickers indenter. Indentations were separated by at least 50 μm . The minimum load that can be applied is 0.1 mN with an accuracy of 1%. The displacement of the indenter is recorded with an accuracy of 10^{-3} μm . Cycles of the type load-hold-unload were performed. In all cases, the maximum load applied was of 151 mN. A typical load-unloading curve for semi-crystalline PET is shown in Fig. 1. For further experimental details see reference (Rikards *et al.*, 1997).

2.2 Analysis of indentation

From the experimental loading and unloading curves, the elastic, viscoelastic and plastic contributions to the indentation depth, h , can be separated. Fig. 2 shows an experimental plot of h versus time t for amorphous PET. The total elastic h_e , plastic h_p and viscoelastic h_v depths can be separated according to the expression

$$h = h_e + h_p + h_v \quad (1)$$

For an amorphous PET specimen the plastic part of the indentation depth is about 55% of the total depth, while the elastic deformation is about 36% and the viscoelastic part is only 9%. Therefore, in the analysis of the stiffness properties of the material, we neglect the viscoelastic response.

To determine the hardness of a plastic material, the experimental loading curve can be fitted to a function of the type (Balta Calleja, 1980)

$$P = K H (h - h_0)^n \quad (2)$$

where P is the load, K is a geometrical constant, h is the displacement, H is the hardness of the material under study and n is a constant; h_0 represents the zero correction for the vertical displacement. The n value is known to increase with increasing displacement, h . Ultramicrohardness experiments on PET (Ion *et al.*, 1990) reveal a n value close to 2 when the maximum penetration depth is larger than $5 \mu\text{m}$. Consequently, in the present paper the experimental data reduction was performed with $n=2$. The hardness calculated from the experiment is defined as $H = P/F_V'$, where $F_V' = 24.5h_c^2$ is the analytically determined projected contact area for the Vickers indenter and $h_c = h - h_0$ is the corrected indentation depth.

The actual contact area for the Vickers indenter with an apical angle of 136° is $F_V = F_V' / \cos^2 22^\circ = 26.43h_c^2$. The actual contact area is used to calculate the hardness of the materials (Balta Calleja, 1994). Johnson (1970, 1985) and Tabor (1951) have defined the hardness of a material as applied load divided by the projected contact area. In this case, the hardness is equal to the mean contact pressure under the indenter. Since in the present investigation the emphasis is laid on the stress analysis of the indentation problem, hardness values are calculated through the projected contact area.

3. FINITE ELEMENT SOLUTION

Contact problems were analyzed by many investigators (see, for example, Johnson, 1985). Friction at the contact surfaces, elastoplastic behavior of the material and other non-linearities can be taken into account by using numerical methods: the finite element method or the boundary element one. Finite element solutions of indentation problems have been obtained by Bhattacharrya and Nix (1988a; 1988b; 1991), Giannakopoulou *et al.* (1994) and other investigators.

In the present paper for the finite element model the Vickers indenter is approximated by an axisymmetric cone with an apical angle of 136° . An axisymmetric elastoplastic problem of a solid deformed by a rigid indenter is considered. The finite element model is shown in Fig. 3. The finite element modeling has been performed by using 1600 eight-node axisymmetric finite elements for the solid and 19 contact elements at the interface between the indenter and the solid. The size of the finite elements at the surface of indentation is $0.78 \mu\text{m}$. The indenter is represented as a perfectly rigid body. For this mesh there are 9760 degrees of freedom of the finite element problem. A friction contact has been modeled by the Coulomb's friction law with a friction coefficient $\mu=0.2$.

Time independent elastoplastic behavior of the material is assumed. Elastoplastic behavior of the material is described by the Prandtl-Reuss flow theory with the Mises yield criterion and with the classical bilinear kinematic hardening model (Chen and Zhang, 1991; Skrzypek, 1993). A model which represents the behavior of an elastoplastic material is shown in Fig. 4. In this model, the total strain increment tensor $d\epsilon_{ij}$ can be divided into an elastic $d\epsilon_{ij}^e$ and a plastic $d\epsilon_{ij}^p$ part

$$d\varepsilon_{ij}^p = d\varepsilon_{ij} - d\varepsilon_{ij}^e \quad (3)$$

The stress increment $d\sigma_{ij}$ is related to the total strain increment $d\varepsilon_{ij}$ by the well known equation of flow theory (Chen and Zhang, 1991; Skrzypek, 1993)

$$d\sigma_{ij} = C_{ijkl}^{ep} d\varepsilon_{kl} \quad (4)$$

Where $d\sigma_{ij}$ represents the increment of the stress tensor and C_{ijkl}^{ep} is the elastic-plastic tangential modulus tensor $C_{ijkl}^{ep} = C_{ijkl} - C_{ijkl}^p$, where C_{ijkl} is the elastic modulus tensor. The components of the tensor C_{ijkl}^p are related (see Chen and Zhang, 1991; Skrzypek, 1993) to the Young's modulus E , Poisson's ratio ν , tangential modulus E_T and to the effective Mises stress σ_e . The effective stress for the Mises yield criterion is calculated by $\sigma_e = (3/2 \sigma'_{ij} \sigma'_{ij})^{1/2}$, where $\sigma'_{ij} = \sigma_{ij} - \sigma_{kk} \delta_{ij} / 3$ is the deviatoric stress.

Elastic strains $d\varepsilon_{ij}^e$ are calculated using Hooke's law $d\sigma_{ij} = C_{ijkl} d\varepsilon_{kl}^e$. The elastic properties of the five different polymers investigated here were previously determined (Rikards *et al.*, 1997). In the finite element solution, the Poisson's ratio for all the polymers investigated is assumed to be $\nu=0.4$ (Ania *et al.*, 1991; Birley *et al.*, 1992). The Young's modulus E was calculated from the plane strain elastic modulus $E^*=E/(1-\nu^2)$ obtained from the unloading curve (Rikards *et al.*, 1997). The elastic properties which are used for the finite element solution are presented in Table 2. The plastic properties are to be the parameters of identification. These values are prescribed in the reference points according experiment design. Thus in the finite element solution, which is part of the identification procedure, the elastic properties are given but plastic properties are different in each reference point.

Finite element analysis calculates the applied load as a sum of the vertical nodal forces at the contact surface for a given indentation depth. For different values of the indentation depth, the corresponding load levels were calculated. To illustrate the finite element solution in Fig. 5 the Mises stress distribution in the specimen for amorphous PET is shown. In this example, the material properties were taken as follows: $E=2705 \text{ MPa}$, $\nu=0.4$, $\sigma_y=78.7 \text{ MPa}$ and $E_T=93.9 \text{ MPa}$. The values of the yield stress and tangential modulus are taken from the results of identification (see section 4). Fig. 5 shows that the plastic zone at the Mises stress level 78.7 MPa is between the hemispherical and cylindrical shapes. This result is similar to the one obtained by Bhattacharrya and Nix (1991) for silicon, which has a low E/σ_y ratio. Amorphous PET also shows a rather low E/σ_y value ($=34.4$).

It is of interest to calculate the ratio c/a , where c is the depth of the plastic zone and a is the radius of contact under the indenter. For amorphous PET the ratio $c/a=2.22$ obtained by the finite element solution is practically independent of the indentation depth. For the spherical cavity model developed by Johnson (1970; 1985) the extent of the plastic zone is described as

$$\frac{c}{a} = \left\{ \frac{1}{6(1-\nu)} \left[\frac{E}{\sigma_y} \tan \beta + 4(1-2\nu) \right] \right\}^{1/3} \quad (5)$$

Where $\beta=22^\circ$ is the complementary angle of half the indenter's apical angle ($90^\circ-136^\circ/2$). For amorphous PET a ratio c/a of 1.6 is obtained using equation (5). The ratio c/a in Johnson's model is lower because it assumes a hemispherical shape for the plastic zone.

The development of the plastic zone with indentation depth obtained using the finite element solutions is presented in Fig. 6. A linear relation between the depth of the plastic zone, c , and the indentation depth, h , is observed. A fitting of the calculated results yields $c=5.7h$ for amorphous PET and $c=4.7h$ for semicrystalline PET.

Figure 7 shows the σ_z stress distribution along the indenter surface for amorphous and semicrystalline PET at a given indentation depth. Semicrystalline PET properties were taken as follows: $E=3326 \text{ MPa}$, $\nu=0.4$, $\sigma_y=97 \text{ MPa}$ and $E_T=283 \text{ MPa}$. The values of the yield stress and tangential modulus for semicrystalline PET are taken from the results of identification (see section 4). Compressive normal stresses are observed for both materials. For amorphous PET, which is a more plastic material than semi-crystalline PET, the stresses are lower and slightly increasing towards the center of the indenter. For semi-crystalline PET, which has about three times higher tangential modulus than amorphous PET, the contact pressure is also higher and rapidly increasing towards the center of the indenter. It should be noted that in Johnson's model, where an ideal elastoplastic behaviour of the material is assumed, the pressure distribution under the indenter is uniform.

For the polymers investigated here, the boundary of the plastic zone is just inside the contact zone (see Fig. 5). This is totally different from the materials with high E/σ_y ratios (for example, Aluminum or Silver), where the boundary of the plastic zone is far beyond the end of the contact point (Bhattacharya and Nix, 1991). Figures 8a and 8b represent the stress distribution along the indentation axis in the axial and radial direction respectively. It is seen that all the stress components are compressive and that the stress level for amorphous PET is lower than for the semi-crystalline PET.

4. METHOD OF EXPERIMENT DESIGN FOR IDENTIFICATION PROBLEMS

For the direct determination of material yield stress and tangential modulus it is necessary to perform a multi-iterated finite element solutions. Such direct procedure has been employed to identify the elastic properties of laminated composite plates from the experimentally measured eigenfrequencies (Mota Soares *et al.*, 1993). On each iterative stage the eigenvalue problem for the linear system has been solved by the finite element method and the non-linear programming algorithm was applied in order to minimize the identification functional (Mota Soares *et al.*, 1993). Such direct procedure, even for elastic problems, takes very large computational efforts.

The elastoplastic indentation problem is non-linear and even by using a very powerful computer it is nearly impossible to apply the direct identification procedure. For the identification problems based on the non-linear solutions, it is necessary to apply other procedure, which can considerably reduce the number of direct finite element calculations. Such identification procedure have been developed on the basis of the method of experiment design. The method of experiment design has been previously used to solve the optimization problems of sandwich and laminated composite plates (Rikards, 1993; Rikards *et al.* 1995; Rikards and Chate, 1995). The identification and the optimal design problems are similar. In both cases the solution is obtained by minimization of the functional considered. The method of experiment design to solve the identification problems is briefly outlined below.

For elaboration of the matrices of experiment design, the number of variables n and the number of experiments l are taken as initial information. The main conditions in the proposed approach are the following ones:

- 1) The number of levels in the experiment domain for each variable is equal to the number of experiments. One experiment is performed per level.
- 2) The reference points (points of the experiments) in the experiment domain are distributed as regularly as possible.

To implement the second principle, the following criterion is suggested

$$\Phi = \sum_{i=1}^l \sum_{j=i+1}^l \frac{1}{L_{ij}^2} \Rightarrow \min \quad (5)$$

where L_{ij} is the distance between the reference points having numbers i and j ($i \neq j$). The problem to minimize criterion (5) together with the first condition leads to the necessity to solve a non-linear integer programming problem. This problem has been solved by the program PLANEX (Rikards, 1993). For each combination of n variables and l experiments, the matrices B_{ij} of experiment design were determined. For example, the matrix B_{ij} for the nine reference points ($l = 9$) and for the two variables ($n = 2$) is as follows

$$\mathbf{B} = \begin{vmatrix} 7 & 1 & 2 & 5 & 4 & 9 & 6 & 8 & 3 \\ 2 & 6 & 3 & 5 & 1 & 4 & 9 & 7 & 8 \end{vmatrix} \quad (6)$$

The reference points for this matrix are presented in Fig. 9. The domain of the experiments is determined as $x_j \in [x_j^{\min}; x_j^{\max}]$. Thus, in this domain the reference points are calculated by the expression

$$x_j^{(i)} = x_j^{\min} + \frac{1}{l-1} (x_j^{\max} - x_j^{\min}) (B_{ij} - 1) \quad (7)$$

Where $i = 1, 2, \dots, l$ and $j = 1, 2, \dots, n$. The matrices B_{ij} may be used to solve different optimal design and identification problems.

5. DETERMINATION OF A SIMPLE MODEL

Information about the behavior of the material can be experimentally obtained or computer solved for the reference points. This information can be represented as a table of data, where the response function $y(\mathbf{x})$ of the material must be in related to the variables $x_1, x_2, \dots, x_n$. In our case there are two variables to be identified $x_1 = \sigma_y$ (yield stress) and $x_2 = E_T$ (tangential modulus) and one parameter of the loading curve $x_3 = h$ (indentation depth). The goal is to obtain the mathematical form of the relation $y(\mathbf{x})$ (the so called regression equation) by using the numerical experimental data at the reference points. This regression equation will be a simple mathematical model of the elastoplastic indentation problem. The existing methods of regression analysis assume a form for $y(\mathbf{x})$ and find the corresponding unknown coefficients. However, in most cases, the form of the equation must also be determined. Such a method was proposed earlier (Rikards, 1993; Rikards *et al.* 1995; Rikards and Chate, 1995) to determine a simple mathematical models for the structural optimization problems. In what follows, this method is briefly outlined and applied for identification problems.

Let us consider a method in which the form of the regression equation is unknown. There are two requirements for the regression equation: accuracy and reliability. Accuracy is characterized by a minimum of standard deviation. By increasing the number of terms in $y(\mathbf{x})$, it is possible to obtain a complete agreement between the experimental data and the values given

by the regression equation. Moreover, in this case it may be any form of the regression equation and, consequently, the prediction at the intervals between the reference points can be very poor. Reliability of the regression equation can be characterized by considering that the standard deviations for the reference points and any other point should be approximately the same. Obviously, if the number of terms of the regression equation is decreasing the reliability of the model is increasing. A compromise between the accuracy and reliability of the model must be found.

Let us consider that the regression equation has the following form

$$y(\mathbf{x}) = \sum_{i=1}^m A_i f_i(\mathbf{x}) \quad (8)$$

where A_i are the coefficients and $f_i(\mathbf{x})$ are the functions from a series of simple functions $\varphi_1, \varphi_2, \dots, \varphi_k$. These functions are assumed to have the form

$$\varphi_k(\mathbf{x}) = \prod_{i=1}^n x_i^{\alpha_{ki}} \quad (9)$$

where n is the number of variables and the coefficients α_{ki} take any integer value (positive or negative). It should be noted that equation (8) represents a linear regression model, since even though the equation is not linear in the independent variables, $\mathbf{x}$, it is linear in the parameters A_i . The form of the regression equation (8) is determined in two steps. In the first step selection of the perspective functions $\varphi_k(\mathbf{x})$ is carried out. Then, step by step, an elimination procedure is applied for the further reduction of the selected functions. The details of this procedure and corresponding program RESINT were described in ref. (Rikards, 1993; Rikards *et al.* 1995; Rikards and Chate, 1995). The elimination procedure will be shown below for the case of modeling the curve of load versus indentation depth.

6. IDENTIFICATION

The finite element solutions for the different input data which correspond to the reference points of the experiment design have been obtained. The results of the finite element solution are used to determine a simple model of the load versus indentation depth behavior for the given limits of the yield stress σ_y and tangential modulus E_T of the material. These limits can be chosen from the preliminary information about the material. For example, for semicrystalline PET these limits have been selected as follows

$$50 \text{ MPa} \leq \sigma_y \leq 100 \text{ MPa}; \quad 200 \text{ MPa} \leq E_T \leq 500 \text{ MPa} \quad (10)$$

The model to be determined can be written in the form $P = P(\mathbf{x})$, where $x_1 = \sigma_y$ and $x_2 = E_T$ are the identification variables and $x_3 = h$ is the parameter of the loading curve. The experiment design with 10 reference points ($l=10$) and with three variables ($n=3$) is selected. The matrix of this design is as follows

$$\mathbf{B} = \begin{vmatrix} 7 & 1 & 6 & 4 & 2 & 10 & 5 & 9 & 3 & 8 \\ 6 & 7 & 4 & 10 & 2 & 3 & 1 & 9 & 5 & 8 \\ 6 & 8 & 10 & 5 & 7 & 4 & 3 & 9 & 2 & 1 \end{vmatrix} \quad (11)$$

Using eq. (7) with matrix (11), the limits indicated in (10) for σ_y and E_T and the limits $1 \mu\text{m} \leq h \leq 5.5 \mu\text{m}$ for the indentation depth, the values of σ_y and E_T at the reference points were calculated. Modulus of elasticity is taken from Table 2 and Poisson's ratio is $\nu=0.4$.

Using these input data, the finite element solutions in each of the 10 reference points have been performed and the curves of load, P , versus indentation depth, h , have been obtained. It should be noted that CPU time for the finite element solution of the contact problem in one reference point was 550 sec. on the workstation Silicon Graphics.

The load has been calculated using the finite element solution for a conical indenter. To obtain the load for a Vickers indenter, the correction coefficient k must be used. The theoretical value of this coefficient is $k=F_V'/F_C'=4/\pi=1.27$, where $F_V'=24.5h^2$ is the projected contact area of a Vickers indenter and $F_C'=19.24h^2$ is the projected contact area of a conical indenter. The program RESINT is used to determine a simple model from these numerical data. Finally, the load indentation depth model for semi-crystalline PET has the form

$$P(\mathbf{x}) = 4.32h^2 + 109\sigma_y h + 7.09E_T h - 8.83h \quad (12)$$

In the following, the load is given in mN and the indentation depth unit in μm . Figure 10 is a plot of the correlation coefficient, k_c , as a function of the number of terms, p , included in model (12). Figure 10 shows a significant decrease of k_c with p values smaller as 4. The most significant term in the model (12) is the first one. This term is related to the elastic response of the material. The coefficient of the first term is approximately equal to the plane strain Young's modulus E^* (see Table 2). The last three terms of model (12) characterize the plastic response of the material. The second term is directly related to the yield stress of the material σ_y . The influence of the tangential modulus E_T (the third term) is smaller.

Similar modeling has also been performed for the other polymers:

$$P(\mathbf{x}) = 3.22h^2 + 99.1\sigma_y h + 7.24E_T h - 7.02h \quad \text{for amorphous PET} \quad (13)$$

$$P(\mathbf{x}) = 2.77h^2 + 98.3\sigma_y h + 11E_T h - 6.28h \quad \text{for PVF}_2 \quad (14)$$

$$P(\mathbf{x}) = 1.71h^2 + 134\sigma_y h + 16.5E_T h - 5.6h \quad \text{for P(VF}_2/\text{F}_3\text{E) 60/40} \quad (15)$$

$$P(\mathbf{x}) = 3.74h^2 + 53\sigma_y h + 3.2E_T h - 3.5h \quad \text{for PE} \quad (16)$$

For these models ((13)-(16)), the coefficient of the first term is also approximately equal to the plane strain Young's modulus E^* (see Table 2). A linear dependence of the load on the yield stress of the material σ_y and the tangential modulus E_T is observed. It should be noted that the simple models (12)-(16) have not been used to calculate the hardness of the material, but only as an intermediate result in the identification procedure. These expressions are valid only in the limits, which were preliminary selected for the identification variables σ_y and E_T (eqs. (10) or similar limits for the other polymers), and the limit for the parameter of the loading curve $h \geq 1 \mu m$ and $h \leq h_{max}$. Here h_{max} is the upper limit of the indentation depth, which is different for the different polymers. For example, $h_{max}=5.5 \mu m$ for the semicrystalline PET.

From the above models ((12)-(16)) and using the experimental data, the yield stress σ_y and the tangential modulus E_t have been determined. A conventional least square technique is used which minimizes the deviation between the experimental and calculated loads

$$\Psi(\mathbf{x}) = \sum_{i=1}^I \left[\frac{P_i - P(\mathbf{x}; h_i)}{P_i} \right]^2 \quad (17)$$

Where $\mathbf{x} = \{\sigma_y, E_T\}$ is the vector of the identification variables, P_i are the experimental values of the load (see Fig. 1), h_i are the experimental indentation depth values, $P(\mathbf{x}; h)$ is calculated through (12)-(16) and I is the number of experimental points. In our case, I is about 220.

By minimizing the functional (17) with constraints on the domain of search (for semi-crystalline PET these constraints are given by the eqs. (10)) the plastic properties of all five polymers have been obtained. The results of identification are presented in Table 3. It should be noted that the minimization of the functional (17) must be carried out with the same constraints (upper and lower limits of the parameters of identification) which were used in the selection of the domain of experiment design for the finite element simulation.

Using the data presented in the Table 3, an experimental load-indentation depth curve was simulated. Comparison of the results obtained by the finite element simulation with the experimental results is shown in Fig. 11a-e for semi-crystalline PET, amorphous PET, PVF₂, P(VF₂/F₃E)60/40 and PE respectively. The agreement between the finite element solution and the experimental results is very good in the loading curve and the initial part of the unloading curve. In the second part of the unloading curve the differences between numerical and experimental results increase. These differences may be due to the time dependence, i. e., to the viscoelastic behaviour. It should be noted that Young's modulus values were independently identified using the experimental data from the initial part of the unloading curve in our previous paper (Rikards *et al.*, 1997), where a simple analytical model of a square punch deforming an elastic solid was used to identify the elastic properties of the material from the unloading curve. Figures 11a-11e show that using the previously derived elastic properties, the more exact finite element model is in good agreement with the experimental data in the initial part of the unloading curve.

7. HARDNESS

In the present paper, the hardness of the material is calculated using four different methods:

- The first method is based on the spherical cavity expansion model. According to Johnson's model (see Johnson, 1970; 1985), the hardness of an indented material can be calculated according to the following expression

$$H = \sigma_y \frac{2}{3} \left(1 + 3 \ln \frac{c}{a} \right) \quad (18)$$

Where the c/a ratio is derived from eq. (5). Equation (18) assumes that the core in the spherical cavity model is incompressible. Thus, the input data to calculate the hardness by Johnson's model are E , ν , σ_y and $\beta=22^\circ$ (inclination angle of the indenter).

- In the second method, the hardness is directly calculated by fitting the experimental loading data to equation (2).

- The third method uses the load, indentation depth and the contact area results obtained by the finite element solution to the indentation problem. Hardness values at a given indentation depth are calculated dividing the load by the projected numerical contact area. This area is calculated using $F'_{C-FEM} = K' h^2$, where K' was determined by the numerical approximation. Such smoothing procedure is used to exclude the influence of the stepwise changing of the contact area, where a new interface element is added. Figure 12 shows a plot of the increase of the contact area with indentation depth for semicrystalline PET. In this case, K' is equal to 13.24.

Figure 13 presents the hardness values versus indentation depth obtained for semicrystalline and amorphous PET. From these results it is seen that hardness values are independent of the indentation depth.

- The fourth method calculates the hardness values using the load values provided by the finite element solution and the analytical contact area of a conical indenter.

Table 4 collects the hardness values calculated using these four different methods. Figure 14 is a plot of the data presented in Table 4. Johnson's model seems to give hardness values which are underestimated. On the other hand, hardness values obtained using the finite element solution are higher than those determined from the power-law fitting to the experimental loading curve (second and third row in Table 4 respectively). This discrepancy is a consequence of a different contact area considered: the radius of the contact area a_{FEM} obtained by the finite element solution (see Figures 5 and 15) is smaller than the radius a_{an} used in the analytical approach. In this latter method, the indenter is assumed to be in contact with the material till point *A* (see figure 15). However, the actual indenter contact ends at point *B*. Thus, as an example, the hardness value of semicrystalline PET calculated using a_{an} is 45% lower than that obtained through a_{FEM} . The analytical contact area of a conical indenter has an intermediate value between that of a Vickers indenter and the one derived using numerical methods; thus, the corresponding hardness lie in between those derived using the two other methods (see last row in Table 4). Figure 12 compares the projected contact area for semicrystalline PET calculated using the analytical contact area $F'_C = 19.24h^2$ of a conical indenter (dashed line) and that determined using the finite element solution (solid line) $F'_{C-FEM} = 13.24h^2$. Similar numerical approximations can be also obtained for the other polymers. For example, the projected numerical contact area for amorphous PET is $F'_{C-FEM} = 15.52h^2$, which is only 24% lower than the analytical contact area. The more plastic the material is, the closer the value of the numerical contact area will be to the analytical one. For materials with low tangential modulus and high E/σ_y ratio (for example, Aluminum or Silver), the contact area obtained by the numerical solution is rather close to the analytical one. Since polymeric materials have rather low E/σ_y ratios, reliable hardness calculations must be performed using the numerically derived projected contact area.

Fig. 16 illustrates the plot of H versus σ_y where H has been derived using the second and third method described previously. When H was numerically derived, the slope of the H - σ_y plot is equal to 3.02, which is very close to Tabor's relation $H=3\sigma_y$ (Tabor, 1979). A linear approximation of the experimental hardness values gives $H=1.88\sigma_y$ (dashed line in Fig. 16).

8. CONCLUSIONS

The results obtained using the numerical experimental method show that it is an appropriate procedure to derive the elastoplastic properties of a material. The yield stress and the tangential modulus of polymers have been identified from the microhardness tests. Comparison of experimentally obtained hardness values with those derived from the finite element solution shows that the latter method gives higher hardness values. A plot of the numerically obtained H value versus the yield stress gives a linear correlation very close to Tabor's relationship.

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Table 1 Description of the investigated samples

Sample	Processing	Reference
PET	Quenched from the melt	Santa Cruz <i>et al.</i> , 1991
Glassy PET	Annealed at 150°C	Santa Cruz <i>et al.</i> , 1991
PVF ₂	Quenched form the melt	Balta Calleja <i>et al.</i> , 1992
P(VF ₂ /F ₃ E) 60/40	Quenched from the melt	Balta Calleja <i>et al.</i> , 1992
High pressure crystallized PE	500 MPa at 260°C	Basset and Carder, 1973

Table 2 Plane strain elastic modulus E^* for the investigated polymers calculated from the unloading curve (Rikards *et al.*, 1997).

Polymer	amorphous PET	semicrystalline PET	PVF ₂	P(VF ₂ /F ₃ E) 60/40	PE
E^* , GPa	3.22	3.96	2.77	1.69	4.31

Table 3 Results of identification of the yield stress and tangential modulus for the investigated polymers.

Mechanical property	amorphous PET	semicrystalline PET	PVF ₂	P(VF ₂ /F ₃ E) 60/40	PE
σ_y , MPa	78.7	97	51	28.2	44
E_T , MPa	93.9	283	320	152	198

Table 4 Hardness H (in MPa) of the investigated polymers evaluated by different methods.

Method	amorphous PET	semicrystalline PET	PVF ₂	P(VF ₂ /F ₃ E) 60/40	PE
Johnson's model	126	154	91	52	95
Experiment*	135	181	113	57	92
FEM**	193	292	187	109	189
FEM***	155	201	132	76	140

*Load is divided by the projected analytical contact area of the Vickers indenter $F'_V = 24.5h^2$.

**Load is divided by the projected numerical contact area of the conical indenter

$F'_{C-FEM} = K'h^2$, where $K'=13.24$ for semicrystalline PET, $K'=15.52$ for amorphous PET, $K'=13.63$ for PVF₂, $K'=13.41$ for P(VF₂/F₃E) 60/40 and $K'=14.22$ for PE.

***Load is divided by the projected analytical contact area of the conical indenter

$F'_C = 19.24h^2$.