Indentation creep of heterogeneous blends of poly(ethylene terephthalate)/impact modifier

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Abstract

The indentation of a sphere into a flat support under a constant load provides information on the resistance of polymeric materials to pressure, which may be of basic importance in various applications. The existing theoretical background for indentation creep in an elastic–plastic regime is reviewed briefly. The indentation creep was measured over the interval 0.1–100 min; in addition, five auxiliary measurements lasting 0.4, 1, 4, 10 and 40 min were performed with the same load. After a recovery period 10 times longer than the creep time, the irreversible part of the indentation depth was read off. The acquired experimental data were fitted with a suitable function in order to calculate irreversible deformation for any time in the interval 0.1–100 min. An essential part (up to 50%) of the indentation depth was found to be irreversible; plastic deformation grew with the creep time somewhat faster than total deformation. Indentation mainly proceeded in short periods after the initiation of loading, while a further increase in penetration depth was much slower; however, no “break” was observed in the time dependencies of measured properties. The acting pressure was linearly decreasing with the logarithm of time due to the rising penetration depth and contact area in the course of individual measurements. The penetration depth/sphere radius ratio was in all cases smaller than 0.10. The logarithm of compliance, which was proportional to the fraction of impact modifiers, was for all samples a linear function of the logarithm of creep time. Although the slope of these dependencies was rather small, it increased with the modifier content, thus indicating the rising creep rate. The effects of two types of impact modifiers on the indentation creep of PET were virtually identical.

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1. Introduction

As is generally known, the suitability of a polymer for an application frequently depends on its mechanical properties. Since various end-products made of polymers are frequently exposed to a long-lasting dead load, their resistance to creep becomes a significant characteristic. Thus, the knowledge of creep behaviour over appropriate intervals of time, stress and temperature is of great practical interest [1,2]. Although creep of many thermoplastics has been described in literature, relatively little is known about the creep behaviour of polymer blends, which have been studied rather sporadically [3–9]. However, heterogeneous blends have been traditionally treated in analogous ways as individual polymers (simple materials) and no models were attempted to correlate the creep of blends with that of their constituents. In our previous papers on heterogeneous two- or three-component polymer blends, we have proposed a versatile predictive scheme for tensile modulus $E_b$ [10–15], yield strength $S_{yb}$ [10,12,14], tensile strength $S_{ub}$ [11,12,14]...
and permeability to gases $P_b$ [11,12,16]. Recently, we have modified our predictive format for the time-dependent tensile compliance $D_i(t)$ of heterogeneous binary blends in the regions of linear [17,18] and non-linear [18,19] stress–strain relationships.

We have also applied the latter approach [20] to the tensile creep of blends of poly(ethylene terephthalate) (PET) with commercial impact modifiers (IM) Lucalen A3110 MQ 244 and Paraloid EXL. Using microphotographs of fractured surfaces of the blends, we have shown that the interfacial adhesion between PET and IM is sufficiently high so that the fracture surfaces (produced in liquid nitrogen) do not follow the interface. Microphotographs have also revealed that (i) the concentration and size of IM particles increase in proportion to the IM fraction in blends and (ii) IMs incorporated in fractions up to 15% do not form a partially continuous component. Ultra-thin stained sections displayed Lucalen particles on the micrometer scale (their size distribution covers approximately one order of magnitude); the particles were oblong due to their orientation in the direction of injection. The structure of PET/Paraloid blends was more complex, with relatively large and irregular particles of Paraloid. The propensity of blends to tensile creep grew with the IM fraction and the time dependence of the compliance became more visible. The effects of the two types of IM seemed to be almost identical from the viewpoint of creep of the blends.

In many practical applications, the resistance of a polymeric material to acting pressure may be a basic characteristic. As well-known examples we can give UHMWPE parts in hip joints [21,22] and tribological composites with thermoplastic matrix exposed to high pressures in various connecting and sliding elements [23,24]. Also, this mode of testing is of great practical importance if the tested materials are not available in the form of prescribed test pieces (e.g. dumb-bells) for tensile, flexural or shear tests, if a material cannot be firmly clamped to prevent slippage (e.g. gels), or if the measurements should be carried out with immersion in a liquid, etc. A suitable method for studying the resistance to pressure is the indentation of a sphere into a flat surface. The indentation method has been widely used for measuring the modulus of hard substrates (metals and glasses) which follow Hooke’s law [25–27]. Dynamic methods [25–27] have frequently been used which operate with extremely small indentations (say 0.1–1 µm), so that the modulus of thin layers (films) can be determined [22,28]. Indentation hardness measurements are commonly used for the characterisation of rubbers and rubber-like materials [29–32].

Although increasing interest in the applications of indentation methods in the field of polymers has been observed in recent years [31–38], relatively little is known about the indentation creep of thermoplastics showing viscoelastic behaviour [21,28,33,36]. The reason is that the common practice consists of measuring indentation depth at one defined (constant) time or over a relatively short interval. Moreover, indentation of polymeric glasses or crystalline polymers has been found to follow an elastic–plastic regime, which makes indentation creep very different from tensile creep. For instance, the plastic strain of poly(ethylene naphthalate) [38] undergoing indentation was approximately equal to 50% of the produced strain. A significant percentage of plastic deformation was also observed for PE [21,33] and PET [28]. Thus, it is practically impossible to anticipate the indentation creep from other types of creep measurements. To our knowledge, no predictive model exists for the indentation creep of multiphase materials. After completing a study of the tensile creep of PET/IM blends [20], we have attempted to detect and analyse their creep behaviour by using a simple indentation method. The objectives of this communication are (i) to determine the compliance of recycled PET and its blends (with two types of impact modifiers incorporated in amounts up to 15 wt%) by measuring the penetration depth of a spherical indenter under a constant load, (ii) to separate reversible and irreversible parts of the compliance and (iii) to describe the compliance as a function of time.

2. Format for the indentation creep of thermoplastics

2.1. Indentation model for a sphere

The depth $d$ of penetration of a sphere of radius $R$ into an elastic sheet (half space) is given as [26,34,36,37]

$$d = \left\{\frac{3}{4}PD^*R^{2/3}\right\}/R^{1/3}$$

(1)

where $P$ is the load and $D^*$ is the reduced tensile compliance. The assumption of stress-free boundaries is believed [29] to hold provided the indentation is less than 10% of the sheet thickness. Deviations from ideal behaviour may be caused by (i) the strains outside the Hookean region or (ii) distortion of the stress patterns owing to the finite (insufficient) thickness of the sheet. The reduced compliance combines the compliance $D_m$ and the Poisson ratio $\nu_m$ of the tested material and the compliance $D_i$ and the Poisson ratio $\nu_i$ of the indenter [25,26,28,34,37]:

$$D^* = \left(1-\nu^2\right)D_m + \left(1-\nu^2\right)D_i$$

(2)

If a steel indenter is used for thermoplastics, then $D_i \ll D_m$, so that the second term in Eq. (2) can be neglected. Combining Eqs. (1) and (2), we obtain a relationship between the time-dependent compliance and the time-dependent penetration depth of a tested material:

$$D_m(t) = K_mR^{1/2}d(t)^{3/2}/P$$

(3)

where $K_m = \left\{\frac{3}{4}(1-\nu^2)\right\}^{-1}$. For typical thermoplastics
with \( v_m = 0.4 \) we obtain \( K_m = 1.5873 \). The variable \( d(t) \ll R \) can be related to the radius \( a(t) \) of the circle of contact of indenter/polymer. Simple geometrical consideration shows that

\[
a(t)^2 = 2Rd(t) - d(t)^2 \approx 2Rd(t)
\]

(4a)

However, the area of contact in the elastic–plastic indentation is smaller so that the following approximation is considered [26,36] as more appropriate:

\[
a(t)^2 = Rd(t)
\]

(4b)

As long as Eq. (4b) holds, the mean pressure over the circle of contact is [26,36]

\[
S(t) = P/\pi a(t)^2 = P/\pi Rd(t)
\]

(5)

which indicates that \( S(t) \) decreases with advancing penetration \( d(t) \) under a constant load. The mean strain \( e(t) \) formally corresponds to the product of stress and compliance:

\[
e(t) = S(t)D(t) = K_n d(t)^{1/2}/(\pi R^{1/2})
\]

(6)

2.2. Empirical functions for the creepoplastics

Creep deformation \( e(t,S,T) \) of polymers depending on time \( t \), stress \( S \) and temperature \( T \) is customarily viewed as consisting of three components [1,2]: (i) elastic (reversible, instantaneous) deformation \( e_o(S,T) \); (ii) viscoelastic (reversible, time-dependent) deformation \( e_v(t,S,T) \); (iii) plastic (irreversible) deformation \( e_p(t,S,T) \):

\[
e(t,S,T) = e_o(S,T) + e_v(t,S,T) + e_p(t,S,T)
\]

(7)

The corresponding tensile compliance \( D(t,S,T) = e(t,S,T)/S \) reads

\[
D(t,S,T) = D_o(S,T) + D_v(t,S,T) + D_p(t,S,T)
\]

(8)

Storage of experimental creep data in a graphical form is rather impractical. If they can be fitted by an equation, then evaluation of creep rate, interpolation or extrapolation of creep deformation and quantititative description of the effects of external variables are facilitated. Many attempts have been made [1,2,39–41] to describe the creep as the product of independent functions of time or stress or temperature, i.e. \( D(t,S,T) = C_o g_o(t) g_o(S) g_o(T) \). The parameters of such empirical equations are customarily determined a posteriori by fitting experimental data. While \( g_o(T) \) is usually identified with the Williams–Landel–Ferry (WLF) or Arrhenius equation, of numerous empirical functions proposed for \( g_o(t) \) and \( g_o(S) \) that we have found [18–20], the following equation is suitable for tensile creep of thermoplastics [42]:

\[
D(t,S) = W(S)(t/\tau_m)^\alpha
\]

(9a)

where \( W(S) \) is a function of the stress, \( \tau_m \) is the mean retardation time and \( 0 \leq \alpha \leq 1 \) is the creep curve shape parameter reflecting the distribution of retardation times. We will use Eq. (9a) in the form

\[
\log D(t,S) = \log W(S) - n \log \tau_m(t) + n \log(t)
\]

(9b)

\[= \log C(t,S) + n \log t\]

also in this paper, to fit the total compliance \( D(t) \) as well as its plastic component \( D_p(t) \) calculated from experimental results for indentation creep. Their difference \( D(t,S) - D_p(t) = D_v(t,S) + D_p(t,S) = D(t,S) - D(t,S) \) corresponds to the reversible component of the compliance.

Plastic deformation \( e_p = SD_p \) can be in an idealized case related to the viscosity \( \nu_D \) of the creeping material (cf. Ref. [33]) defined by the following relation:

\[
\nu_D = S/(de_p/dt)
\]

(10)

Hence

\[
e_p(t) = \eta dt/\eta D_p(t) = \Delta t/\Delta D_p(t) = \nu (t_{n+1} - t_n)/[D_p(t_{n+1}) - D(t_n)]
\]

(11)

Thus, a material function \( \nu_D(t) \), the unit of which is [Pa s], can tentatively be evaluated as the ratio of the increments of time and of compliance (calculated values \( \nu_D \) are plotted against \( t = (t_{n+1} + t_n)/2 \)).

3. Experimental

3.1. Materials

Poly(ethylene terephthalate) was produced by Eco Seleka Italia Srl (Salorno, Italy) as a recycled product obtained from bottles used for beverages (density ISO 1183:A): 1.328 g/cm³; MVR ISO 1133 = 115 ml/10 min; intrinsic viscosity ISO 1628 – 5 = 0.70 dl/g. To improve its impact properties required in intended applications, blends of PET with polymeric impact modifiers were prepared. Two types of IM were used: (1) Luaclean A3110 MQ 244, which is a commercial ethylene/acrylic acid/acrylate terpolymer; (2) Paraloid EXL, which is a commercial core/shell acrylic impact modifier. Thus, two series of blends were prepared with 7, 10 and 15 wt% of IM.

Polymers were mixed in a single-screw extruder (model EEGT/35/1-D36/ESI) working at 160 rpm and at about 280 °C. The produced pellets were used for feeding a Sandretto injection moulding machine model 310/95 (average barrel temperature 270 °C; injection pressure 13.5 MPa) to produce specimens for the measurements of mechanical properties. The mould was permanently cooled with circulating water at 12 °C, but temperature changes of the mould in the course of injection were not registered. Test pieces 28 mm × 22 mm × 3.3 mm for indentation creep were machined from
marginal parts (used for clamping) of the ASTM D638 dumb-bell specimens (length 210 mm; width 20 mm; thickness 3.3 mm; gauge length 80 mm; gauge width 12.8 mm). Specimens used for creep studies were stored for more than six months at room temperature to avoid any interfering effect of physical aging during measurements.

3.2. Indentation creep measurements

The indentation creep test consisted of monitoring the penetration depth $d(t)$ as a function of time under the action of a constant load. Indentation measurements were performed utilising a simple apparatus equipped with a mechanical stress amplifier (lever) 20:1. To prevent any buckling of the indenter and to alleviate the problems of its counterbalancing (to avoid the indentation caused by the weight of the operating column), a tensile load acted on the lower surface of the test specimen (via a “reverser”); thus, the upper surface of the specimen was pulled against the fixed indenter. The indentation depth $d(t)$ was measured with a mechanical displacement gauge with an accuracy of about 2 µm, which corresponds to about 0.5% of the final indentation depth. (The produced $d(t)$ was of the same size as that commonly attained for rubbers [29,30].) An electrically operated vibrator was rigidly attached to the gauge holder in order to minimise the friction in the gauge mechanism. As the indenting sphere should be free to slide, the measured surface was lubricated with low viscosity oil. Indentations were located 6–8 mm from the edge of the specimen; the distance between two indentations was about 6 mm. Indentation creep measurements in the interval 0.1–100 min were performed under a constant load utilising a spherical indenter with a radius $R = 4.725$ mm. The applied load of 3 kg gave rise to pressures between 100 and 150 MPa. Corrections were made for instrument deformation [26], which was obtained with the same indenting sphere and load, but with a steel test specimen. No mechanical conditioning of specimens before creep measurements was performed. All creep tests were implemented at room temperature, i.e. at 21–23 °C.

In this study, we have tried to imitate the regime used in previous tensile creep experiments [20] in order to obtain analogous material time functions. The creep time was registered from the beginning of the loading. The loading interval extended over about 1 s; the reading of the displacement was started after 6 s (0.1 min). This kind of test is not a “true” creep test because the acting stress diminishes with time in proportion to the rising contact area $\pi a(t)^2 = \pi Rd(t)$. It is a specific feature of the indentation method that the lowest contact area and, consequently, the highest pressure are attained at the start of the experiment. Although an infinitesimal initial deformation is viewed as an elastic process, the pressure exceeding the yield stress initiates plastic deformation, which generally develops at a high pressure [36]. Thus, a substantial part of plastic deformation is produced in the initial stages of the indentation because the high initial pressure rapidly drops with the penetration depth.

Owing to the elastic–plastic character of the deformation, it is inevitable that data on plastic deformation are acquired along with the total deformation from the start of the creep experiment. To this end, auxiliary creep experiments were performed with the same specimen (but at different positions). As the basic measurement of the creep extended over the interval 0.1–100 min, five auxiliary measurements lasting 0.4, 1, 4, 10 and 40 min were performed with the same load. The plastic (irreversible) part $d_p(t)$ of the total indentation $d(t)$ produced during these shorter creeps was read off after a recovery period 10 times longer than the creep period. The acquired experimental data were fitted to $d_p(t) = yt^z$ (where $y$ and $z$ are constants) in order to calculate $d_p(t)$ for any time of the creep scale in the interval 0.1–100 min. If the final value of indentation depth $d(t)$ in an auxiliary creep was different from the value $d(t)$ obtained in the 100 min experiment, then the irreversible component $d_p(t)$ of the indentation depth from the auxiliary creep was multiplied by the $d(t)/d_p(t)$ ratio.

4. Results and discussion

The creep behaviour of impact modifiers was not studied because test specimens could not be prepared. In general, we can presume that ordinary impact modifiers are rubber-like or leather-like materials having a much higher compliance than thermoplastics. Thus, the contribution of a dispersed IM to the creep resistance of a thermoplastic blend will be very small or virtually negligible. It is obvious that the viscoelastic properties of PET/IM blends are mainly determined by the properties and the fraction of PET. In a simplified manner, such an IM can be viewed as a “diluent” in the PET matrix.

The application of the theory of linear viscoelasticity to the creep of polymeric materials requires experiments at very low stresses in order to produce dependencies of $\log D(t)$ vs. $\log t$ independent of the applied stress. However, such an approach is impractical, because (i) the linearity limit between stress and strain is usually very low (below 1%) and uncertain, (ii) the relative accuracy of creep measurements is poor and (iii) the effect of stress remains unspecified, which may be a serious shortcoming from the viewpoint of possible applications. This communication deals mainly with experiments performed at relatively high pressures, which produce reversible (elastic and viscoelastic) and irreversible (plastic) deformations. Analysing the obtained results we should take into account that (i) the stress decreases with time in all experiments and that (ii) stress levels differ in individual experiments owing to differing penetration
depths (although the load and the sphere radius are the same for all measurements). While the former factor causes the performed experiment not to be “true” creep, the latter factor makes a comparison of various materials more complicated.

Fig. 1 visualizes the decrease in the acting stress with log $t$ due to rising penetration depth and contact area in the course of individual measurements. Interestingly enough, these dependencies are approximately linear for all measured samples. At the same time, we can see that the pressure decreases (the penetration depth rises) for a selected creep time with the content of IM in the blends. It should be pointed out that applied pressures were almost one order of magnitude higher than the stresses in tensile creep measurements reported in our previous work [20]. In this way, we can explain the extensive plastic component of the indentation because the yield strength was obviously exceeded for all tested materials.

Fig. 2 shows the indentation depth $d(t)$ and its plastic (irreversible) component $d_p(t)$ as functions of time (as mentioned before, the points on the time dependence $d_p(t)$ were calculated on the basis of six independent measurements and do not correspond to experimental data points). As can be seen, an essential part of the indentation depth corresponds to the plastic component, which occurs within 10 s after the initiation of loading. Further rise in $d(t)$ and $d_p(t)$ with time is relatively small; the difference $d(t) - d_p(t)$ corresponding to the reversible penetration depth remains almost constant throughout the whole interval of measurements. On the other hand, both $d(t)$ and $d_p(t)$ rise with the fraction of impact modifier in the blends, which means that the creep resistance of the materials is perceptibly reduced. The penetration depths related to the sphere radius, $d(t)/R$ and $d_p(t)/R$, are approximately linear functions of the logarithm of time (Fig. 3). It is worth noting that the values of $d(t)/R$ were in all cases smaller than 0.10. Both quantities grow with the IM content in blends, while their time dependencies are very similar. Obviously, if the pressures were the same in all experiments (cf. Fig. 1), the differences in $d(t)/R$ or $d_p(t)/R$ (i.e. the effect of blend composition) would be more evident.

Also, the strain $e(t)$ and its plastic component $e_p(t)$ calculated from Eq. (6) are approximately linear functions of log $t$ (Fig. 4). In most cases, $e_p(t)$ grows faster with time than $e(t)$, which means that the reversible

![Fig. 1. Mean indentation pressure as a function of time. Weight fraction of impact modifier: (○) 0%; (□) 7%; (△) 10%; (△) 15%. (a) Lucalen; (b) Paraloid.](image)

![Fig. 2. Total indentation depth (empty points) and irreversible indentation depth (full points) as functions of time. Weight fraction of impact modifier: (○) 0%; (□) 7%; (△) 10%; (△) 15%. (a) Lucalen; (b) Paraloid.](image)
component slightly diminishes with the creep time. Besides, $e_p(t)$, which is small for neat PET, rises with the IM fraction more than $e(t)$. After 100 min creep, $e_p(100 \text{ min})$ corresponds to about one third of $e(100 \text{ min})$ for the PET/IM = 85/15 blends.

The log $D(t)$ vs. log $t$ plots are quite linear for all studied materials (Fig. 5). Although there is no a priori reason for such linearity, it seems to be a "standard" feature of the studied materials. As the decrease in pressure during a creep measurement does not account for a (more) complex log $D(t)$ vs. log $t$ dependency, it seems that the materials behave as if they were in the region of the linear pressure–strain relationship (despite produced plastic deformation). Thus, the values of $D(t)$ are not noticeably affected by the decreasing indentation pressure (in the interval indicated in Fig. 1), so that the indentation creep under a constant load can be viewed as an acceptable approximation of the indentation creep under a constant pressure. As can be seen, the compliance of blends is proportional to the IM fraction. Also, analogous plots of log $D_p(t)$ vs. log $t$ appear to be quite linear in the time interval of measurements. Irreversible component $D_p(t)$ makes 5–20% of the (total) compliance $D(t)$.

The rising IM fraction accounts for a higher $D_p(t)$, while the slopes of the log $D_p(t)$ vs. log $t$ dependencies decrease in a somewhat irregular way. It is important to note that all plotted dependencies (Figs. 2–5) are quite smooth, without any "break" between the loading and creeping intervals (cf. Ref. [36]).

The values of $n$ from Eq. (9b) read off as the slope of the log $D(t)$ vs. log $t$ dependencies rise slightly with the IM fraction in blends, though they remain very low (Table 1). On the other hand, log $C$ from Eq. (9b), which corresponds to the compliance at $t = 1$, displays a clear growth with the fraction of IM. Analogous values of $n_p$ and $C_p$ were read off from the log $D_p(t)$ vs. log $t$ dependencies. The effect of IM on log $C_p$ is similar to that observed for log $C$; in contrast, $n_p$ rather diminishes with the fraction of IM. Considering the scatter of data, we can only say that the effects of the two types of IM are practically identical from the viewpoint of the indentation creep of PET blends, though Paraloid seemingly accounts for somewhat higher values of $n$ than Lucalen.

It is worth noting that the high values of $R^2$ evidence a very good accuracy of the fitting of experimental data with Eq. (9)).

The resistance of the PET blends to plastic defor-
Fig. 5. Compliance (empty points) and irreversible component (full points) as functions of time. Weight fraction of impact modifier: \((/H17034)0\%; (/H17040)7\%; (/H17003)10\%; (/L50773)15\%. (a) Lucalen; (b) Paraloid.

Table 1
Parameters of Eq. (9b) characterizing the indentation creep compliance of poly(ethylene terephthalate) and its blends with impact modifiers

<table>
<thead>
<tr>
<th>PET/IM</th>
<th>Average stress (MPa)</th>
<th>log C</th>
<th>n</th>
<th>(R^2)</th>
<th>log (C_p)</th>
<th>(n_p)</th>
<th>(R^2_p)</th>
</tr>
</thead>
<tbody>
<tr>
<td>100/0</td>
<td>141</td>
<td>−0.0753</td>
<td>0.0317</td>
<td>0.9883</td>
<td>−1.2842</td>
<td>0.2985</td>
<td>0.9999</td>
</tr>
<tr>
<td>93/7 L*</td>
<td>134</td>
<td>−0.0384</td>
<td>0.0301</td>
<td>0.9865</td>
<td>−0.7565</td>
<td>0.1294</td>
<td>0.9998</td>
</tr>
<tr>
<td>90/10 L</td>
<td>126</td>
<td>−0.0012</td>
<td>0.0307</td>
<td>0.9833</td>
<td>−0.6591</td>
<td>0.1492</td>
<td>0.9999</td>
</tr>
<tr>
<td>85/15 L</td>
<td>107</td>
<td>0.1140</td>
<td>0.0256</td>
<td>0.9917</td>
<td>−0.4730</td>
<td>0.1278</td>
<td>0.9999</td>
</tr>
<tr>
<td>93/7 P†</td>
<td>132</td>
<td>−0.0314</td>
<td>0.0290</td>
<td>0.9919</td>
<td>−0.7231</td>
<td>0.1236</td>
<td>0.9998</td>
</tr>
<tr>
<td>90/10 P</td>
<td>127</td>
<td>−0.0026</td>
<td>0.0315</td>
<td>0.9920</td>
<td>−0.6683</td>
<td>0.1400</td>
<td>0.9998</td>
</tr>
<tr>
<td>85/15 P</td>
<td>115</td>
<td>0.0546</td>
<td>0.0383</td>
<td>0.9955</td>
<td>−0.3954</td>
<td>0.0762</td>
<td>0.9998</td>
</tr>
</tbody>
</table>

Subscript p denotes the dependencies for the plastic component of the indentation creep; \(C, n\): parameters of Eq. (9b); \(R^2\): reliability values.
* Lucalen A3110 MQ 244.
† Paraloid EXL.

5. Conclusions

The indentation of a sphere into a flat support under a constant load was found suitable for studying the resistance of PET/IM blends to pressure. The existing theoretical background for indentation tests in the elastic–plastic regime was adapted for creep measurements. The indentation creep measured over the interval 0.1–100 min was amended by five auxiliary measurements lasting 0.4, 1, 4, 10 and 40 min. After a recovery period 10 times longer than the creep time, an irreversible part of the indentation depth was read off; the acquired experimental data were fitted with a suitable function in order to calculate irreversible deformation for any time in the interval 0.1–100 min. The indentation proceeded mainly in a short period after the initiation of loading, while a further rise in the penetration depth was much slower; however, no “break” between loading and creep intervals was observed on the time dependencies of the measured properties. An essential part (up to 50%) of the indentation depth was found to be irreversible; it grew with creep time somewhat faster than the total indentation.
depth. The penetration depth/sphere radius ratio was in all cases smaller than 0.10; the acting pressure was found to decrease linearly with the logarithm of time due to rising penetration depth and contact area in the course of individual measurements. The logarithm of compliance was a linear function of the logarithm of creep time for all samples. Although the slope of these dependencies was rather small, it grew with modifier content. Thus, both the compliance and creep rate were proportional to the fraction of the impact modifier. In other words, the produced creep rose with the IM content and its time dependence became more visible. The effects of two selected impact modifiers on the indentation creep and/or dimensional stability of PET were virtually identical.

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