Phase structure and tensile creep of recycled poly(ethylene terephthalate)/short glass fibers/impact modifier ternary composites

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Abstract. Binary and ternary composites of recycled poly(ethylene terephtalate) (rPET), short glass fibres (SGF) and/or impact modifier (IM) were prepared by melt compounding and injection moulding. SEM images of rPET/IM fracture surfaces indicated that IM particles of about $1-2 \mu m$ in diameter were uniformly distributed in the rPET matrix, but with a poor adhesion level. Microphotographs of PET/SGF composites evidenced brittle fracture proceeding through the matrix and strong adhesion between components. In ternary composites SGF were evenly distributed, while IM particles were no longer detectable.

Tensile creep of rPET and prepared composites was investigated under short and long term testing conditions at various stress levels. Main part of the tensile creep corresponded to the elastic time-independent component, while the time-dependent component was rather limited even at relatively high stresses. While SGF accounted for a significant decrease in the overall creep compliance, the incorporation of IM induced a small decrease in the compliance and a non-linear vis-coelastic behavior. In ternary composites, the reinforcing effects of SGF was dominating. Under a constant stress, the log-arithm of compliance grew linearly with the logarithm of time. The creep rate, which resulted to be generally very small for all tested materials, was slightly reduced by SGF and increased by IM.

Keywords: polymer composites, poly(ethylene terephthalate), recycling, creep

1. Introduction

Due to the rapid growth in its use, poly(ethylene terephthalate) (PET) has become one of the most important engineering polymers in the past two decades [1, 2]. PET is used widely in several massive applications such as textile fibres for apparel, films for packaging, bottles and containers for beverage drinks [3]. While neat (non-reinforced) PET has a rather limited use as an engineering plastic, short fibre reinforced PET is increasingly adopted for high demanding applications, like automobile components, lighting products, power tools, mate-

rial handling equipment, sporting goods and housewares [2]. As a result of environmental pressures and for economical reasons, several strategies have been explored for physical or chemical recycling of post-consumer PET waste [1, 4–6]. Physical recycling of PET waste usually involves melting of solid flakes or granules in an extruder for pelletization into chips or for direct melt processing into value-added product. Through melt compounding in a twin screw extruder, recycled PET (rPET) chips can be used to obtain engineering plastics

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such as blends/alloys or fibre reinforced compounds [7–16].

Many applications of thermoplastics are limited by their poor dimensional stability under long-lasting dead loads (constant external forces). Thus, systematic studies of the creep stability over appropriate intervals of time, stress and temperature are of great practical importance [17, 18]. As generally known, the creep of thermoplastics can be reduced by various types of reinforcing agents. Short glass fibres rank among very efficient discontinuous reinforcements, but their incorporation is usually accompanied by a significant decrease in toughness (impact resistance). For this reason, ternary composites have been developed which combine the effects of reinforcing and toughening components [19–22]. In view of the wide structural variability of ternary composites, it is necessary to acquire detailed information on their mechanical (physical) properties in correlation to their composition. A deficiency of many thermoplastics, in particular crystalline ones, consists in non-linear viscoelastic behaviour, which means that the produced strain rises more than acting stress (the compliance rises with stress). In creep experiments it is manifested by creep rate increasing in the course of the exposition, particularly after long creep periods, say 1000-10 000 min.

The objective of this communication is to study (i) phase structure, (ii) short-term creep and (iii) long-term creep of recycled poly(ethylene terephthalate) (PET) and its binary and ternary composites containing short glass fibers (SGF) and/or impact modifier (IM).

2. Format for the creep compliance of thermoplastics

Creep strain $\varepsilon(t,\sigma,T)$ depending on time *t*, stress σ and temperature *T* is usually viewed as consisting of three components [17, 18], as shown by Equation (1):

$$\varepsilon(t,\sigma,T) = \varepsilon_e(\sigma,T) + \varepsilon_v(t,\sigma,T) + \varepsilon_p(t,\sigma,T)$$
(1)

where $\varepsilon_e(\sigma, T)$ is the elastic (instantaneous, timeindependent) component, $\varepsilon_v(t, \sigma, T)$ is the viscoelastic (reversible, time-dependent) component, and $\varepsilon_p(t, \sigma, T)$ is the plastic (irreversible, time-dependent) component. In practical applications of thermoplastics, the conditions should be avoided where $\varepsilon_p(t,\sigma,T) > 0$ because any plastic deformation usually accounts for irreversible damage of end-products. Therefore, the tensile compliance $D(t,\sigma,T) = \varepsilon(t,\sigma,T)/\sigma$ corresponding to reversible deformation reads as shown by Equation (2):

$$D(t,\sigma,T) = D_e(\sigma,T) + D_v(t,\sigma,T)$$
(2)

Storage of experimental creep data in a graphical form is impractical, though it is widely used [23]. If the creep curves can be fitted by an equation, then evaluation of the creep rate, interpolation or extrapolation of the creep deformation, and quantitative description of the effects of external variables are facilitated. The creep compliance is generally viewed as a product of independent functions of time or stress or temperature, i.e., $D(t,\sigma,T) = C_p g_1(t) g_2(\sigma) g_3(T)$, determined *a posteriori* by fitting experimental data. Of numerous empirical functions proposed for $g_1(t)$ and $g_2(\sigma)$, the Equation (3) was found suitable for both short- and long-term tensile creeps of polypropylene and its blends [24–26]:

$$D(t,\sigma) = W(\sigma) \left(\frac{t}{\tau_{rm}}\right)^n$$
(3)

where $W(\sigma)$ is a function of the stress, τ_{rm} is the mean retardation time, and $0 \le n \le 1$ is the creep curve shape parameter reflecting the distribution of retardation times. We assume that the retardation time τ_{rm} is controlled by the available fractional free volume whose changes obey the Equation (4):

$$\log a_{\varepsilon}(t) = \log \left[\frac{\tau_{rm}(f_2)}{\tau_{rm}(f_1)} \right]$$
(4)

where $\log a_{\varepsilon}(t)$ is a shift factor and $f_2 > f_1$ are the fractional free volumes related to deformations $\varepsilon_2 > \varepsilon_1$. Obviously, if the free volume changes are small during creep measurements, the value of $\log a_{\varepsilon}(t)$ is negligible.

Combining Equations (3) and (4) we obtain the Equation (5) [24]:

$$\log D(t,\sigma) = [\log W(\sigma) - n \log \tau_{rm} - n \log a_{\varepsilon}(t)] + n \log(t) = \log C(t,\sigma) + n \log(t)$$
(5)

3. Experimental

3.1. Materials

Recycled poly(ethylene terephthalate) (rPET) was produced by Eco Selekta Italia Srl (Salorno, Italy) 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).

A commercial nucleating agent (Procaster by Polichem) was used (3.5 wt% related to rPET) in order to obtain fully crystallized rPET matrix under the selected moulding conditions.

Short (chopped strand) E-glass fibres (SGF) type 952 produced by Saint Gobain – Vetrotex were used as a reinforcing agent in percentage of 15 and 30 wt%. Filament diameter was 10 μ m, fibre length 4.5 mm. The fibres are surface treated with a proprietary sizing agent specifically designed for thermoplastic polyester and polycarbonate resin systems. According to previous investigations [10], the fibre, added in the feeding zone of the extruder, degraded from their initial length to average fibre lengths of 0.71±0.14 mm and 0.55±0.08 mm for rPET-15GF and rPET-30GF, respectively.

A commercial core/shell acrylic impact modifier (IM) (ParaloidTM EXL by Rohm and Haas) was added (15 wt%) in some formulations to improve the impact properties of rPET and its composites.

All components, i.e., rPET, nucleating agent, short glass fibres and/or impact modifier, were mixed in a single screw extruder (Eco Extruder, model EEGT/35/L-D36/ESI) working at 160 rpm and at temperature in the range 280-310°C. Produced pellets were used for feeding a Sandretto injection moulding machine, model 310/95, (average barrel temperature range: 270–300°C; injection pressure: 20 MPa; mould temperature: 130°C) to produce ASTM D638 dumb-bell specimens (length: 210 mm; thickness: 3.3 mm; gauge length: 80 mm; gauge width: 12.8 mm) for the measurements of mechanical properties. Specimens used for creep studies were stored for more than six months at room temperature to avoid any interfering effect of the physical aging during measurements.

3.2. Electron microscopy

Injection moulded dumb-bell specimens were fractured in the middle of their length in liquid nitrogen. Fracture surfaces, which were perpendicular to the injection direction, were sputtered with a 4 nm layer of Pt in a vacuum sputter coater (SCD 050, Balzers) and observed in a scanning electron microscope (SEM; Vega TS 5130, Tescan). All SEM micrographs are secondary electron images, taken at 30 kV.

3.3. Tensile creep measurements

Tensile measurements were performed by using a home-made experimental apparatus equipped with a mechanical stress amplifier (lever) 11:1. The initial length of the test specimens was about 80 mm; their cross-section was 12.8 mm×3.3 mm. The displacement was measured with a mechanical gauge with an accuracy of about 2 μ m, i.e., of about 0.0025%. Mechanical conditioning before each creep measurement consisted in applying a stress (for 1 min), which produced a strain larger than the expected final strain attained in the following measurement; the recovery period after the mechanical conditioning was more than 1 h. All creep tests were implemented at room temperature, i.e., 21–23°C.

Short-term tensile creep measurements in the interval 0.1–100 min were performed with one test specimen at four gradually increasing stress levels (between 5 and 30 MPa) in order to estimate the linearity limit between stress and strain. Each shortterm creep measurement was followed by a 22 h recovery before another creep test (at an increased stress) was initiated. Long-term tensile creep experiments under a selected stress extended from 0.1 to 10 000 min.

4. Results and discussion

4.1. Phase structure of PET/SGF/IM composites

Microphotographs of the rPET/IM fracture surfaces (Figure 1a) show that IM particles of about $1-2 \mu m$ in diameter are uniformly distributed in the rPET matrix. The adhesion between rPET and IM is weak because imprints (holes) of many torn-off particles can be observed. Fracture surfaces of PET/SGF composites evidence (Figures 1b and 1c) that the brittle fracture mainly proceeds through the matrix. As no fractured (protruding) fibres or circular holes as imprints of pulled out fibres can be seen



Figure 1. SEM microphotographs of fracture surfaces of binary systems: a) rPET/IM15; b) rPET/SGF15; c) and d) rPET/SGF30

on fracture surfaces, it is evident that the adhesion between rPET and SGF is strong. SEM images of fractured surfaces of ternary composites (Figure 2) clearly visualize SGF, while the identification of the IM particles is no longer possible, probably because the fracture path is controlled by SGF rather than IM particles. Other possible reasons could be related to the fact that the presence of glass fibers may induce higher shear stresses in the melt during compounding thus enabling a substantial reduction of the dimensions of IM particles below the resolution of the SEM observations. Moreover a localization of the IM at the fibermatrix interface could also be supposed. As commonly encountered in injection moulded SGF reinforced thermoplastics [27], a skin-core microstructure was observed, with a prevalent orientation of SGF in the direction of injection moulding in the outer (skin) layers (see Figures 1d and 2c).

4.2. Short-term tensile creep of PET/SGF/IM composites

It can be expected that incorporation of a reinforcing component will increase the elastic modulus of the material and suppress viscoelastic character of the creep. With regard to expected lower compliance of the prepared composites, it was inevitable to perform the creep experiments at relatively high stresses in order to produce reasonable strains. Otherwise, the relative accuracy of creep measurements would be low and the effect of stress would remain unspecified, which would be a serious shortcoming from the viewpoint of possible applications. Therefore, this communication deals mainly with experiments performed at relatively high stresses, which may be of practical importance.

Tensile compliance D(t) of rPET (Figure 3) is virtually independent of applied stress (up to 30 MPa), which indicates linear viscoelastic behavior. Main



Figure 2. SEM microphotographs of fracture surfaces of ternary composites: a) rPET/SGF15/IM15; b) and c) rPET/SGF30/IM15

part of the compliance corresponds to the elastic time-independent component, which occurs immediately after loading. Viscoelastic component $D_v(t)$ is low, representing only a few percent of D(t); for this reason, the $D_v(t)$ vs. log t plots are somewhat irregular.



Figure 3. Short-term creep of rPET: effect of stress on compliance D(t) (upper part) and its viscoelastic component $D_v(t)$ (lower part). Lines are polynomial regressions. Applied tensile stress [MPa]: (0) 7.40; (\Box) 14.81; (∇) 22.21; (\diamondsuit) 29.62.

The effect of four selected stress levels on the time dependencies of D(t) of binary and ternary systems is summarized in Figures 4–8 and in Table 1. As expected, the binary composites with 15 and 30 wt% of SGF (samples rPET/SGF15 and rPET/SGF30) have a much lower compliance than rPET, but they display stress-strain non-linearity because their compliance noticeably rises with applied stress. Also the creep rate characterized by the parameter *n* (Table 1) is higher than that found for the rPET matrix. On the other hand, the effect of applied stress on $D_v(t)$ is somewhat irregular, because the values of $D_v(t)$ are very low. In contrast, the incorporation of 15 wt% of IM into PET (sample rPET/IM15) brings about an increase in



Figure 4. Short-term creep of rPET/SGF15: effect of stress on compliance D(t) (upper part) and its viscoelastic component $D_v(t)$ (lower part). Lines are polynomial regressions. Applied tensile stress [MPa]: (O) 6.73; (\Box) 13.46; (∇) 20.19; (\diamond) 26.91.



Figure 5. Short-term creep of rPET/SGF30: effect of stress on compliance D(t) (upper part) and its viscoelastic component $D_v(t)$ (lower part). Lines are polynomial regressions. Applied tensile stress [MPa]: (O) 6.73; (\Box) 13.46; (∇) 20.19; (\diamond) 26.91.

the compliance, but the latter is insensitive to the applied stress as it is observed for the neat rPET. However, the parameter n (Table 1) is much higher than that of rPET (although the absolute values of n still remain low), thus indicating an augmented creep rate.



Figure 6. Short-term creep of rPET/IM15: effect of stress on compliance D(t) (upper part) and its viscoelastic component $D_{\nu}(t)$ (lower part). Lines are polynomial regressions. Applied tensile stress [MPa]: (O) 7.47; (\Box) 14.94; (∇) 22.42; (\diamond) 29.89.

Comparing the creep stability of the prepared composites we can say that the compliance of the ternary composites rPET/SGF15/IM15 and rPET/ SGF30/IM15 is noticeably lower than that of rPET. Creep patterns of ternary composites are different from the previous ones in that they show decrease in D(t) and $D_v(t)$ with rising stress (samples

Sample	SGF content [wt%]	IM content [wt%]	Applied stress [MPa]	log C	n	R ²
rPET	0	0	7.40	-0.4618	0.0057	0.9328
			14.81	-0.4733	0.0041	0.9371
			22.21	-0.4987	0.0045	0.9013
			29.62	-0.4972	0.0054	0.9732
rPET/SGF15	15	0	6.73	-1.1406	0.0164	0.8979
			13.46	-1.0369	0.0112	0.8781
			20.19	-0.9653	0.0100	0.9659
			26.91	-0.8217	0.0055	0.9457
rPET/SGF30	30	0	6.73	-1.3812	0.0226	0.9109
			13.46	-1.1039	0.0087	0.8906
			20.19	-1.0431	0.0071	0.9698
			26.91	-0.9824	0.0059	0.8327
rPET/IM15	0	15	7.47	-0.3437	0.0124	0.9588
			14.94	-0.3180	0.0098	0.9612
			22.42	-0.3425	0.0138	0.9613
			29.89	-0.3204	0.0228	0.9445
rPET/SGF15/IM15	15	15	7.47	-0.6046	0.0084	0.9289
			14.95	-0.6597	0.0065	0.9590
			22.42	-0.6812	0.0059	0.9578
			29.90	-0.6979	0.0073	0.9703
rPET/SGF30/IM15	30	15	7.40	-0.7426	0.0056	0.9487
			14.80	-0.8053	0.0055	0.8965
			22.20	-0.8580	0.0049	0.8892
			29.61	-0.8847	0.0080	0.9763

Table 1. Effect of tensile stress on the parameters characterizing the short-term compliance of composites rPET/SGF/IM

C, n: parameters of Equation (5); R: correlation coefficient



Figure 7. Short-term creep of rPET/SGF15/IM15: effect of stress on compliance D(t) (upper part) and its viscoelastic component $D_v(t)$ (lower part). Lines are polynomial regressions. Applied tensile stress [MPa]: (O) 7.47; (\Box) 14.94; (∇) 22.42; (\diamond) 29.89.





rPET/SGF15/IM15 and rPET/SGF30/IM15). This effect is more pronounced in the composite with 30 wt% of SGF than in the composite with 15 wt% of SGF. Thus we can presume that the unexpected result could be related to a 'strain hardening' of the composite structure or to a strain-induced fibre orientation effect. In other words, the presence of IM seems to enhance strain hardening of single rPET macromolecules (orientation of single macromolecules of the polymer matrix) and/or strain-induced orientation of SGF (orientation of whole fibres in the matrix); these processes probably lead to energy dissipation and decrease in tensile compliance. The creep rate of ternary composites is decreasing as the SGF content increases, while the creep rate of rPET/SGF30/IM15 composites is virtually the same as rPET.

4.3. Long-term tensile creep of PET/SGF/IM composites

The results obtained for the long-term creep tests (Figures 9 and 10, Table 2) are in a good agreement with those obtained for the short-term creep. The compliance is substantially reduced by SGF and



Figure 9. Long-term creep of rPET composites: effect of SGF and IM on compliance D(t) (upper part) and its viscoelastic component $D_v(t)$ (lower part). Lines are polynomial regressions. (O) rPET, applied tensile stress 22.21 MPa; (\Box) rPET/IM15, applied tensile stress 20.82 MPa; (∇) rPET/SGF15, applied tensile stress 20.19 MPa; (\Diamond) rPET/SGF30, applied tensile stress 20.19 MPa.



Figure 10. Long-term creep of rPET composites: effect of SGF and IM on compliance D(t) (upper part) and its viscoelastic component $D_v(t)$ (lower part). Lines are polynomial regressions. (O) rPET, applied tensile stress 22.21 MPa; (\Box) rPET/IM15, applied tensile stress 20.82 MPa; (∇) rPET/SGF15/IM15, applied tensile stress 20.93 MPa; (\diamond) rPET/SGF30/IM15, applied tensile stress 20.72 MPa.

Sample	SGF content [wt%]	IM content [wt%]	Applied stress [MPa]	log C	n	R ²
rPET	0	0	22.21	-0.5008	0.0108	0.8384
rPET/SGF15	15	0	20.19	-0.8450	0.0081	0.8814
rPET/SGF30	30	0	20.19	-1.0219	0.0068	0.9600
rPET/IM15	0	15	20.92	-0.3326	0.0297	0.8581
rPET/SGF15/IM15	15	15	20.93	-0.6678	0.0128	0.8368
rPET/SGF30/IM15	30	15	20.72	-0.8442	0.0075	0.9640

Table 2. Effect of the composition on the parameters characterizing the long-term compliance of composites rPET/SGF/IM

noticeably enhanced by IM. Thus the composite with 30 wt% of SGF and 15 wt% of IM (rPET/ SGF30/IM15) has approximately the same compliance as the composite with 15% of SGF (rPET/ SGF15). The creep rate is also reduced by SGF, but markedly enhanced by IM. The creep rate of ternary composites decreases with the content of SGF, but its values are rather close the value found for rPET.

A noticeable upswing of the $\log D(t)$ with $\log(t)$ plot for longer creep periods (t > 1000 min), which was typical of the rPET/IM blends described in a previous communication [8], can be seen also for the sample rPET/IM15. On the other hand, all composites (binary and ternary) containing SGF show a linear $\log D(t)$ vs. $\log(t)$ dependence in almost all the measured time interval. Thus a positive effect of SGF can be seen in that the creep rate does not rise at long creep periods.

The recovery following the long-term creep would require extremely long periods of experimental time (about 70 days). As possible plastic deformation (flow) is proportional to the creep time, it should be 100 times larger in the long-term creeps than in the corresponding short-term creeps. While no permanent deformation was detected after the recovery of short-term creeps, a very small residual deformation (of selected specimens) was observed after 20 days of the recovery in the creep apparatus. Thus, we believe that no irreversible deformation was produced in the executed long-term creep experiments.

5. Conclusions

Microphotographs of the rPET/IM fracture surfaces showed that the IM particles of about $1-2 \mu m$ in diameter were uniformly distributed in the PET matrix, but their adhesion to PET was weak because imprints (holes) of many torn-off particles

can be seen. Fracture surfaces of rPET/SGF composites evidenced brittle fracture mainly proceeding through the matrix. Absence of pulled out fibres confirmed strong adhesion between rPET and SGF. SEM images of fractured surfaces of ternary composites clearly visualized SGF evenly distributed, while the IM particles were no longer visible, probably because the paths of fracture surfaces was controlled by SGF rather than IM particles.

Main part of the tensile creep of rPET and prepared composites corresponded to the elastic time-independent component, while the time-dependent component was rather limited even at relatively high stresses. SGF accounted for a significant decrease in the compliance, non-linear stress-strain behaviour and a small increase in the creep rate. In contrast, the incorporation of IM provoked an increase in the compliance and creep rate, even if the linear viscoelastic behaviour of the rPET matrix was preserved.

In prepared ternary composites, the reinforcing effect of SGF was dominating. All composites containing SGF showed a linear $\log D(t)$ vs. $\log t$ dependence throughout the whole experimental time interval. Thus a positive effect of SGF could be seen in that the creep rate did not rise at long creep periods. The creep rate, which was generally very small for all tested materials, was slightly reduced by SGF and increased by IM. Creep patterns of the ternary composites rPET/SGF/IM showed a decrease in D(t) and $D_v(t)$ with rising stress, which could be tentatively related to a 'strain hardening' of the composite structure.

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