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Effects of compatibilization on rheological properties of PS/PB blends and investigation of Doi–Ohta scaling relationship in double start-up of shear experiments

Received: 6 September 2005
Accepted: 15 September 2005
Published online: 6 October 2005
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Abstract The rheological properties for the blends of polystyrene and polybutadiene were investigated and the effect of compatibilizer styrene butadiene rubber (SBR), on the blends were studied and the results compared with the non-compatibilized blends. The frequency sweep, step shear strain and shear stress growth experiments were carried out for the blends. The results showed that with addition of compatibilizer the changes in behavior of the rheological properties of blends are observed. These rheological variations could be related to the

reduction of interfacial tension and size of dispersed phase. Furthermore, the validity of Doi–Ohta scaling relationship in double start-up experiments was studied. It is shown that this scaling relationship becomes more reliable with increasing the amount of PB and compatibilizer.

Keywords Rheology · Immiscible polymer blend · Polystyrene · Polybutadiene · Doi–Ohta theory

Introduction

The melt blending of polymers is a suitable and cost-effective technique to produce materials with specific applications. Thermoplastic elastomers prepared from rubber–plastic blends are materials, which combine the excellent processability characteristics of plastics at high temperatures and the wide range of physical properties of elastomers at service temperatures. These materials have gained great importance in recent years, because of their many end-use applications (Joseph et al. 2003). The blend of PS and PB is one of the well-known thermoplastic elastomers that many studies focused on its morphological and rheological properties (Joseph et al. 2002, 2003; Joseph and Thomas 2003; Barzegari et al. 2004).

In immiscible polymer blends, morphology plays a major role and affects the properties of blend. With the use of premade block copolymers as compatibilizer, it is

possible to control final morphology of immiscible blends which the details could be found in the extensive studies of Macosko and his co-workers (Sundararaj and Macosko 1995; Macosko et al. 1996; Maric and Macosko 2002; Van Hemelrijck et al. 2004; Jeon et al. 2004; Galloway et al. 2005; Moldenaers et al. 2005). Compatibilization also could affect the rheological properties by reduction of interfacial tension and change in size and interfacial area of dispersed phase (Jafari et al. 2005). Hence, because of these major effects, it is important to study the effect of addition of compatibilizer on rheological properties of immiscible blends. The various studies have been done so far and the results are such that one may not draw a specific conclusion. Iza et al. (2001) reviewed effects of the compatibilizer so the authors indicated that the results showed no increase or decrease of melt viscosity, and elastic modulus of the blend. They concluded some important factors about the effect of presence of

compatibilizer. Such previous complex results motivated us to study and to check effect of the compatibilizer on the blends, which are elaborated in a detail manner.

In this work, the effect of compatibilizer on the rheological properties of commercial PS/PB blends was studied at three shear flow experiments as: (1) frequency sweep in linear viscoelastic regime; (2) stress relaxation after a sudden finite strain and (3) transient experiments. The commercial SBR was used as compatibilizer due to its molecular structure, which consists of both styrene and butadiene in a block form (Joseph et al. 2002). Furthermore, the experiments of double start-up stress growth were carried out to check the validity of Doi–Ohta scaling relationship in transient shear flows (Doi and Ohta 1991). To the best of our knowledge, it is the first time that a blend consists of a thermoplastic and a rubber used for such a study. In previous similar studies either a couple of thermoplastics or nearly Newtonian rubbers were used and it was concluded that Doi–Ohta scaling predictions are at least approximately valid (Takahashi et al. 1994a, b; Vinckier et al. 1996; Guenther and Baird 1996; Vinckier and Laun 2001; Iza et al. 2001). On the other hand, the original Doi–Ohta theory has not been considered any surfactant; but Iza et al. (2001) showed that for the compatibilized blends, these scaling relationships, particularly for transient flows, work nicely. Considering their results, it encourages us to check the scaling relationship in terms of the effect of compatibilization on the blends.

Experiment

The blends of commercial polystyrene and polybutadiene compatibilized with commercial SBR were used in this study. All polymers were delivered from Iranian Petrochemical Industries; polystyrene (GPPS grade 1160) from Tabriz petrochemical, polybutadiene (1441 grade) from Arak petrochemical and SBR (1502 grade) (block copolymer with 23.5% (mole/mole) styrene as supplier contended) from Bandar Imam petrochemical complexes. The main characteristics of these materials are given in Table 1.

Table 1 The characteristics of the polymers

	PS	PB	SBR
Supplier	Tabriz petrochemical	Arak petrochemical	Bandar Imam petrochemical
M_w (g/mol)	248,000	460,000	–
M_n (g/mol)	110,000	110,000	–
Mooney	–	–	46–58 (ASTM D-1646)

Blend preparation

All blends were prepared by melt blending in a Brabender internal mixer at 180°C and a rotor speed of 60 rpm for approximately 10 min (time to reach a constant torque). Eight types of the blends were selected: 95/5/0, 95/5/1, 90/10/0, 90/10/1, 90/10/2.5, 80/20/0, 80/20/2.5 and 80/20/5. The first and second terms stand for PS and PB weight fractions, respectively, on free compatibilizer basis. The last term denotes weight fraction of SBR calculated with respect to total weight of blend. For example, the 90/10/1 blend, consists of 35.64 g PS, 3.96 g PB and 0.4 g SBR (total weight is 40 g). The PS granules were conducted into the mixer at first in order to turning into a melt, and then PB (and SBR) (cut into small bits) was (were) introduced.

Morphological analysis

Blend morphologies were examined by scanning electron microscopy using a Philips model XL300 microscope. The samples were cryo-fractured in liquid nitrogen in order to have a brittle fracture, thus avoiding large deformations in the surface to be examined by the SEM. The resulting fractured surfaces were coated with gold prior to observations. In Figs. 1, 2, 3, the SEM micrographs of all blends used in this study are showed. The dark areas related to PS-rich phase. As evident in these micrographs, the minor phase (PB) distributed with complex patterns. For a rough quantitative comparison of morphologies, we assumed that the distributed domains as spherical droplets, so the number average (\bar{R}_n) and volume average (\bar{R}_v) radiuses of these hypothetical droplets were calculated by the following equations:

$$\bar{R}_n = \frac{\sum n_i R_i}{\sum n_i} \quad (1a)$$

$$\bar{R}_v = \frac{\sum n_i R_i^2}{\sum n_i R_i} \quad (1b)$$

The results of such calculations are shown in Table 2.

Rheological measurements

The rheological measurements were performed with a stress/strain controlled rheometer, UDS 200 made by Paar Physica. The experiments for blends were carried out using disk type parallel plates with 25-mm diameter and 1-mm gap at 165°C. During the tests, the temperature was chosen in which to hold thermal stability of the samples under the air atmosphere. Three types of the experiments were performed as frequency sweep, step shear strain and shear stress growth in both single and

double start-up modes. In each test we used a fresh sample that was preheated at least 10 min to insure morphological similarity.

Results and discussion

Frequency sweep

The frequency sweep tests were performed in linear viscoelastic region, i.e., at strain rate 5%. The tests were carried out at 100, 120, 140 and 160°C for PB and 140, 160, 180 and 200°C for PS. So the master curves of PS and PB at reference temperature 140°C are constructed by horizontal shifting and shown in Fig. 4a and b, respectively. As Fig. 4a shows that $G' \propto \omega^2$ and $G'' \propto \omega$ at low frequencies, so PS reaches its terminal region but PB does not. Figure 5a, b and c show complex moduli of 95/5, 90/10 and 80/20 blend series, respectively. As the

figures showed the addition of compatibilizer has a minor effect on frequency sweep experiments (Iza et al. 2001), however the tests were reproduced and the

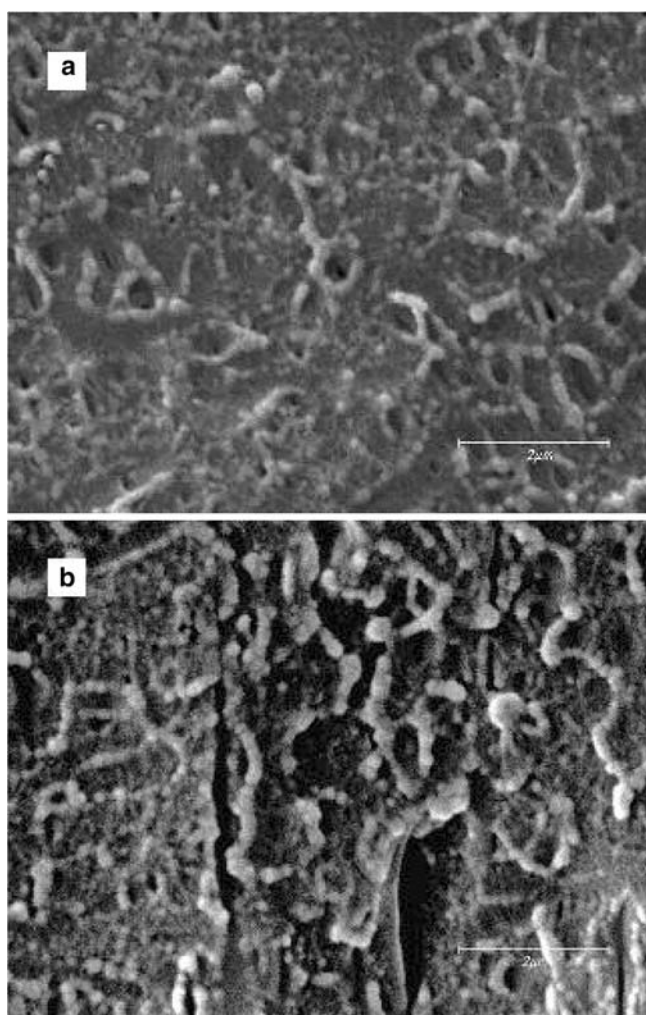


Fig. 1 The SEM micrograph of: a 95/5/0; b 95/5/1 blends

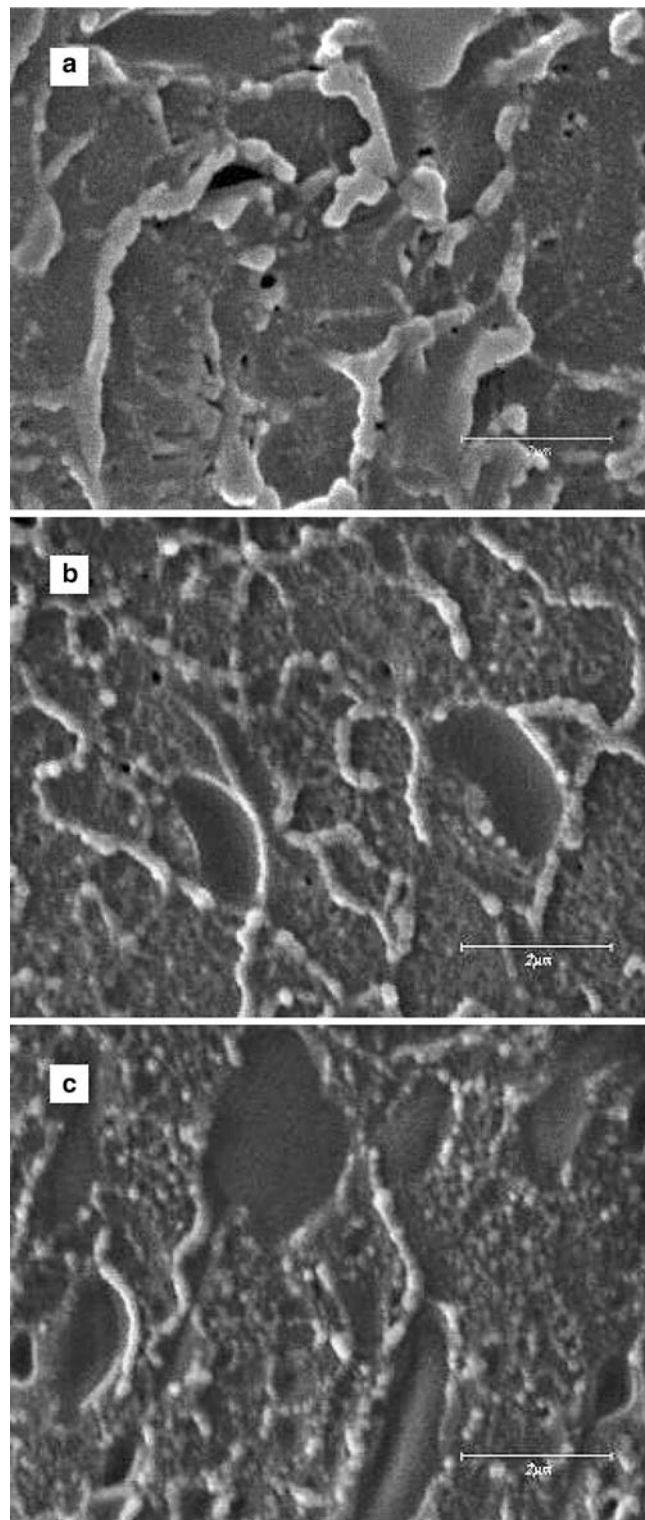


Fig. 2 The SEM micrographs of: a 90/10/0; b 90/10/1; c 90/10/2.5 blends

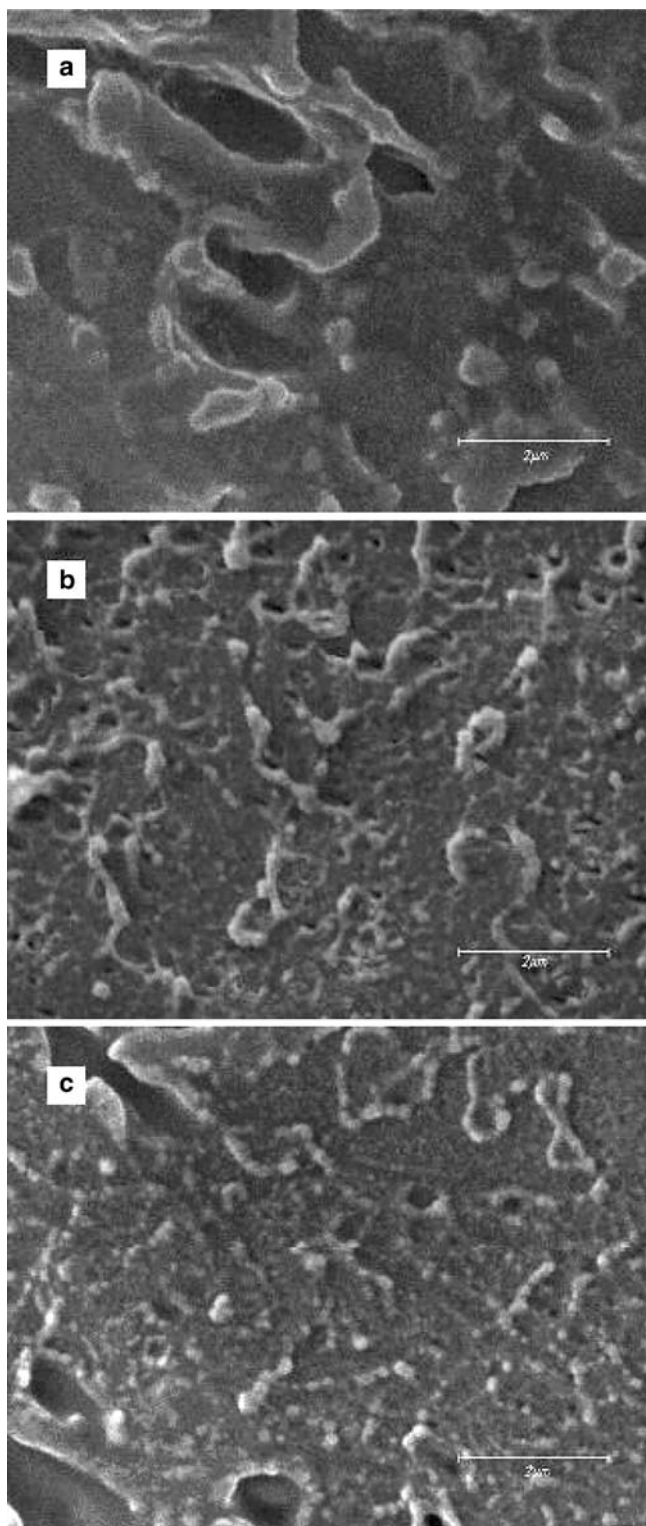


Fig. 3 The SEM micrographs of: **a** 80/20/0; **b** 80/20/2.5; **c** 80/20/5 blends

differences were observed. It is seen that by increasing compatibilizer, the complex modulus behaves differently in the three blends, i.e., it increases, not change and decreases in 95/5, 90/10 and 80/20 series, respectively. This complex behavior could be justified as below.

As explained in introduction, the interface alters rheological properties through interfacial tension and interfacial area. The main feature of interfacial area is size of disperse domains which determined with coalescence and break-up processes. So the parameter α/R , ratio of these two variables, plays a major role in the models, which were proposed for immiscible blends, particularly in emulsion models (Taylor 1934; Choi and Schowalter 1975; Paliarne 1990; Bousmina 1999a). Where α and R are interfacial tension and radius of dispersed phase spherical droplets, respectively. Bousmina (1999b) has shown that at first stages with increasing α/R , the complex modulus increases in a plateau region and then decreases toward the terminal zone. The radius of droplet, R , may be assumed as the size of dispersed domains, characteristic length scale, the term which has ignored in phenomenological models (Okamoto et al. 1999). It should be noted that in the present frequency sweep results, the changes in moduli have occurred in terminal zone and thus it is expected for our systems, stress increases with decreasing in α/R . In fact, the present results did not show any plateau. As Table 2 shows that for 95/5 blend series, the size of domains has not changed mainly by addition of compatibilizer. On the other hand, interfacial tension has been reduced, so α/R should be decreased. Hence, as experimental results show, modulus increases. Conversely, for 80/20 blends, a large reduction in dispersed domain sizes is clear. However due to presence of interfacial agent, it prevents further reduction of interfacial tension so in result the α/R increases. This means that modulus decreases for these types of blends. The same results could be found in Iza et al. (2001), which they studied the 50/50 blend of PS/HDPE. Addition of

Table 2 The number and volume averages of radius of dispersed domains for all blends

Blend	$\bar{R}_n (\mu\text{m})$	$\bar{R}_v (\mu\text{m})$
95/5/0	0.1352	0.1420
95/5/1	0.1268	0.1306
90/10/0	0.2590	0.2818
90/10/1	0.1557	0.1607
90/10/2.5	0.1284	0.1295
80/20/0	0.2536	0.2789
80/20/2.5	0.1674	0.1755
80/20/5	0.1189	0.1231

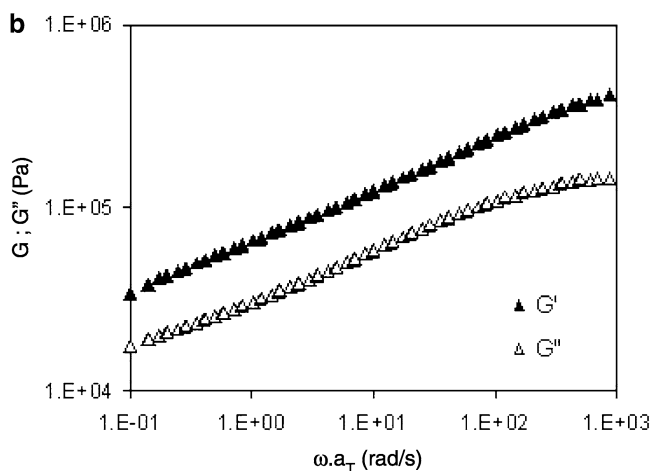
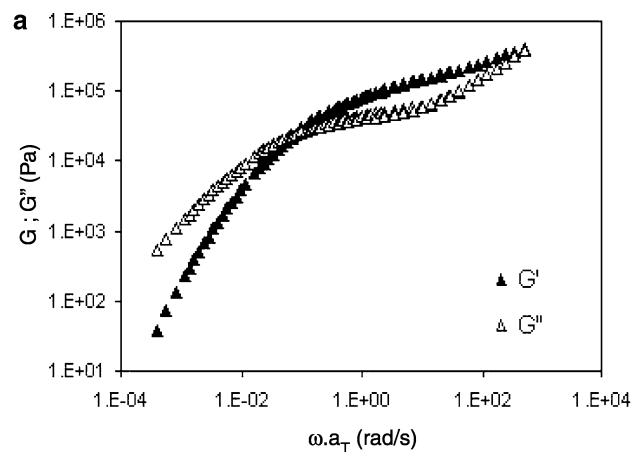


Fig. 4 The master curves of: **a** PS; **b** PB; at $T_{ref}=140^{\circ}\text{C}$

two types of the compatibilizer to the blend made the morphology too finer that caused the modulus to decrease. In addition, for 90/10 blends, reduction of α and R may be occurred at the same scales and not to demonstrate any essential change in α/R . Thus, there are no clear differences between the experimental curves.

Step strain

The step strain experiments were carried out for five strains: 0.05, 0.5, 1, 2 and 5. Due to using the fixture, i.e., parallel disks, at high strain, the deformations were not homogenous through the samples and a correction should be done. Soskey and Winter (1984) proposed such a correction for this type of test as:

$$G(t, \gamma_R) = \chi(\gamma_R) G_a(t, \gamma_R) \quad (2a)$$

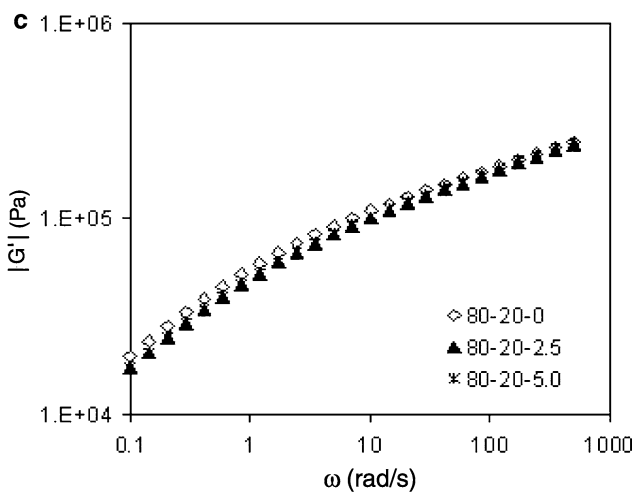
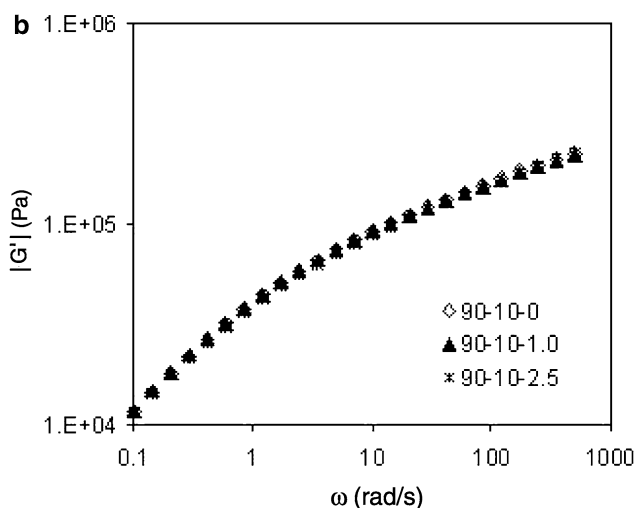
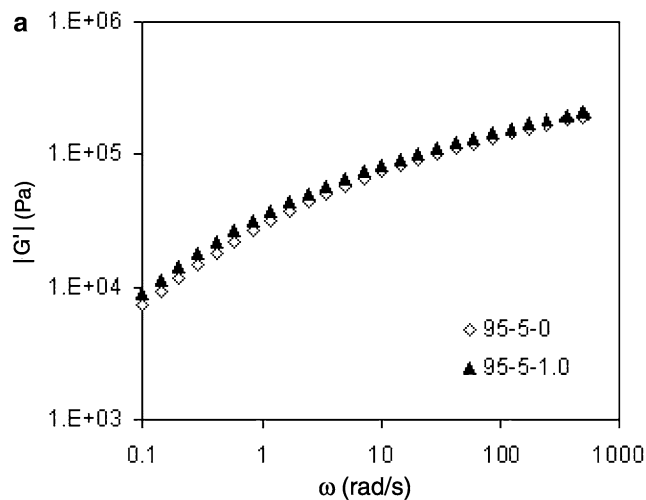


Fig. 5 The complex moduli of PS/PB: **a** 95/5; **b** 90/10; **c** 80/20 blends series

$$\chi(\gamma_R) = \frac{h(\gamma_R)\gamma_R^4}{4\int_0^{\gamma_R} h\gamma^3 d\gamma} \quad (2b)$$

where G_a , γ_R and h are apparent relaxation modulus, maximum strain imposed on disks circumference and damping function, respectively. So all of the data were refined with this procedure.

In Fig. 6, the relaxation modulus of all blends for $\gamma = 0.05$ is shown. By adding the compatibilizer, almost the same behavior as oscillatory test is seen for this type of experiment, i.e., increasing and decreasing in relaxation modulus for 95/5 and 80/20 blends, respectively. The only difference is for 90/10 blends, which there is a little increase in moduli due to interfacial agent. However, it was noted that the moduli are the same below a certain time around 10 s and following it begins to diverge. This value may be equivalent with the lowest frequency, which is used in dynamic measurements (0.1 s^{-1}) that indicates the similarity of data in both experiments.

Iza and Bousmina (2000) have performed the step strain experiments for PB/PDMS blend and their results showed that a fast relaxation process pertaining to relaxation of components has been occurred, followed by a slow one. For the present systems, the relaxations of components are comparable with relaxation of interface and so there are no two distinct processes as for PB/PDMS system.

Figure 7 shows the relaxation modulus of 95/5/0 blend at various strains. The linear data obtained at strains below 0.5. Haghtalab and Sodeifian (2002), Sodeifian and Haghtalab (2004) used a nonlinear regression algorithm to correlate relaxation modulus of the different blends PS/PB and PVC/NBR. In this work, we used the same method to correlate the relaxation

modulus of the blends. Therefore, a generalized Maxwell model is applied as (Dealy and Wissbrun 1990):

$$G(t) = \sum_{i=1}^n G_i e^{-t/\lambda_i} \quad (3)$$

where G_i and λ_i are the modulus and relaxation time of i th mode, respectively. By assuming five Maxwell elements ($n=5$) the best-fit values of G_i and λ_i for linear data of all blends were obtained and summarized in Table 3. The corresponding fit for blend 95/5/0 is also shown in Fig. 7.

It is clear from the figure that the curves are nearly parallel at long times. The same results were obtained for the other systems. The damping functions are calculated by vertical shifting of relaxation moduli curves onto linear data for 95/5, 90/10 and 80/20 blends and shown in Fig. 8a–c, respectively. The two-parameters damping function (Soskey and Winter 1984) is used to correlate the damping function as:

$$h(\gamma) = \frac{1}{1 + a\gamma^b} \quad (4)$$

This equation fits the present data approximately. Okamoto et al. (1999) and Iza and Bousmina (2000) correlated their results with a special form of this equation, i.e., $b=2$ which attributed to Zapas equation (Dealy and Wissbrun 1990), and obtained parameters “a” equal to 0.2 and 0.085, respectively. For comparison with experiment for the 95/5/0 blend, fitting of the data with this form of equation was also performed. Figure 9 shows the comparison of these two equations and Table 4 presents the parameters for the both cases. Again, the same effects of compatibilizer in damping functions are observed, but as shown in Fig. 8b it is clearly a transient behavior is seen for 90/10 series. The occurring of this phenomenon may be attributed to

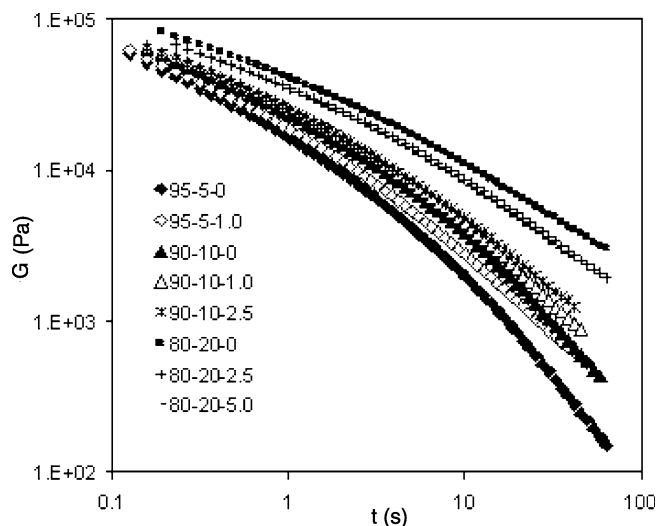


Fig. 6 The relaxation moduli for the blends of PS/PB at $\gamma = 0.05$

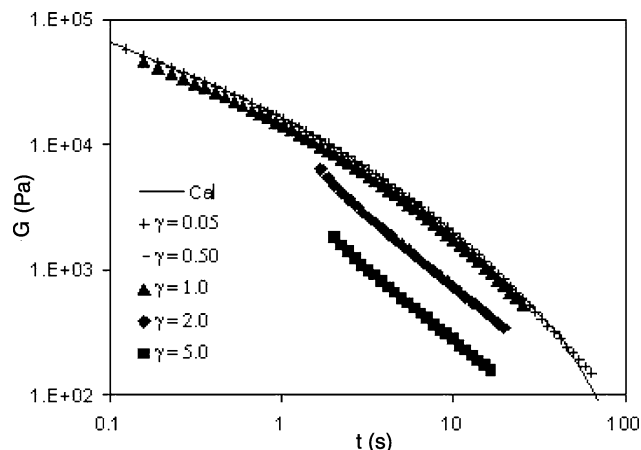


Fig. 7 The relaxation moduli for the blend PS/PB: 95/5/0 at different strain rates

Table 3 The parameters of generalized Maxwell model

i	λ_i (s)	G_i (Pa)
Blend 95/5/0		
1	0.071	69665.52
2	0.294	32402.46
3	1.135	18324.88
4	4.634	7506.71
5	23.325	1711.24
Blend 95/5/1		
1	0.051	72385.40
2	0.181	39503.18
3	0.786	25937.62
4	3.762	11589.55
5	26.626	2795.31
Blend 90/10/0		
1	0.092	62794.33
2	0.375	34265.45
3	1.532	20474.12
4	6.981	9754.61
5	38.603	1947.19
Blend 90/10/1		
1	0.086	57471.91
2	0.348	34781.91
3	1.389	22144.97
4	5.848	11472.79
5	34.708	3171.50
Blend 90/10/2.5		
1	0.095	57166.48
2	0.375	32268.10
3	1.470	20536.65
4	6.270	10650.55
5	41.726	3332.18
Blend 80/20/0^a		
1	0.160	54521.84
2	0.694	35085.30
3	2.897	23851.73
4	14.122	12941.81
5	204.817	4047.49
Blend 80/20/2.5		
1	0.139	49384.12
2	0.543	31677.37
3	2.114	21854.33
4	8.942	12909.36
5	65.396	5014.85
Blend 80/20/5		
1	0.144	49882.85
2	0.558	33316.26
3	2.398	22186.91
4	12.879	11788.29
5	1168.639	2097.00

changing in the morphology of the blend through increasing the dispersed domain sizes due to coalescence under high strains, which it causes to increase the stresses.

The shear stress growth experiment

The stress growth experiments were carried out at shear rate 0.2 s^{-1} . As the results are shown in Fig. 10a–c, the

Table 4 Zapas and Soskey-Winter parameters used to fitted damping functions for all blends

	Zapas	Soskey-Winter	
	a	a	b
95/5/0	0.4838	0.2353	2.5822
95/5/1	0.1394	0.1149	2.2907
90/10/0	0.5049	0.2220	2.7677
90/10/1	0.1638	0.3429	1.6250
90/10/2.5	0.1102	0.3926	1.1774
80/20/0	0.0364	0.1571	1.1534
80/20/2.5	0.0899	0.4409	1.0786
80/20/5	0.0904	0.3483	1.2347

same trends are observed as the previous tests, but effect of the compatibilizer is pronounced so our previous explanations are valid for interpretations of these results. In comparison with other experiments results, these significant effects of compatibilizer in transient region of the shear stress growth experiment confirms the conclusion of Iza et al. (2001), which they noted that the main effects can be seen in transient regions. Moreover, similar to these authors' results, the positions of occurring overshoots are independent of compatibilizer amount. It is worth noting to emphasis, the value of overshoots depend significantly on weight fraction of the compatibilizer.

Double start-up and Doi–Ohta scaling relationship

Using the double start-up experiment allows one to validate the Doi–Ohta scaling relationship. Doi and Ohta (1991) proposed a phenomenological model for describing the effect of interfacial area on rheological properties of a blend consists of two Newtonian fluids with equal viscosities, densities and volume fractions. In this model, the interfacial region, without considering its complexities, is assumed as the third component with its own properties. The interface is modeled with two mathematical parameters: a scalar “ Q ” which denotes the total interfacial area per unit volume and tensor “ q ” that it indicates the degree of anisotropy of interfacial area per unit volume. The mathematical descriptions of these parameters are as below:

$$Q = \frac{1}{V} \int_S dS \quad (5a)$$

$$q_{ij} = \frac{1}{V} \int_S \left(n_i n_j - \frac{1}{3} \delta_{ij} \right) dS \quad (5b)$$

Where V , n_α , δ_{ij} and S are total volume of system, α component of unit vector normal to the surface, Kronecker delta and total interfacial area, respectively.

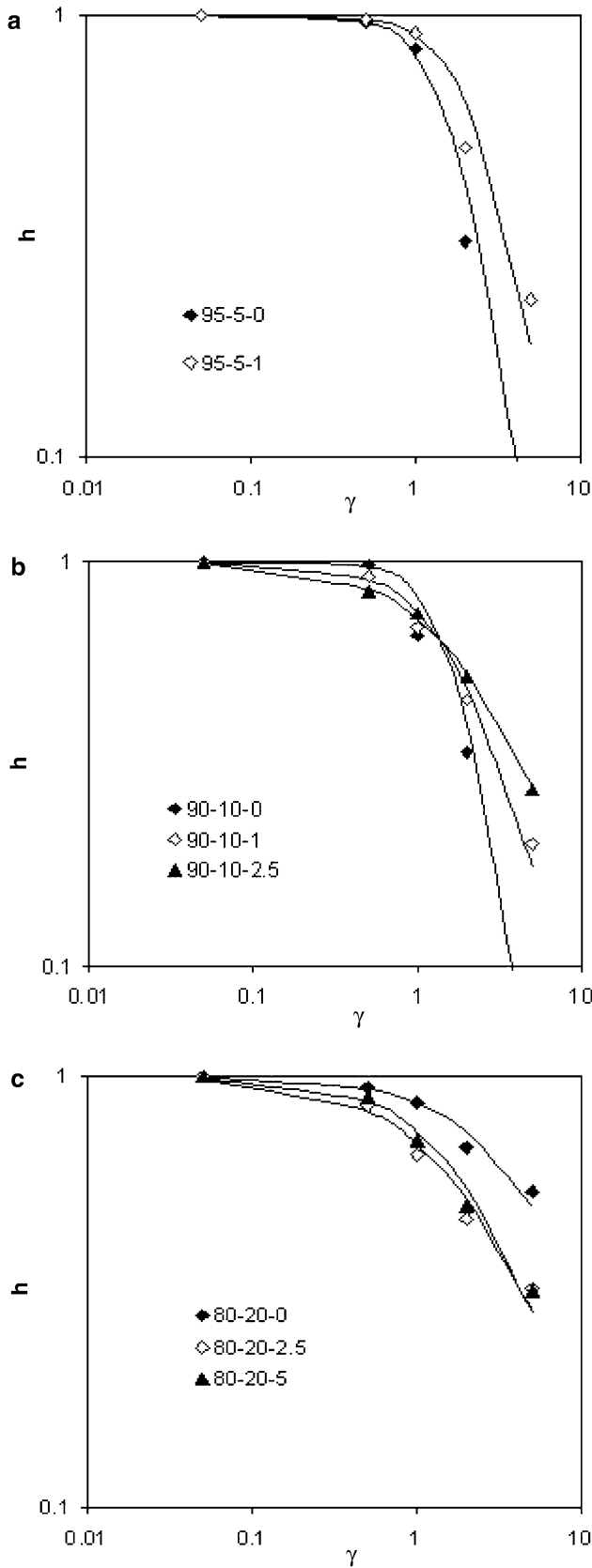


Fig. 8 The log-log plot of damping function vs. strain: **a** 95/5; **b** 90/10; **c** 80/20 blends series. The curves are the best fitting with Soskey-Winter equation for each data series with parameters shown in Table 4

These authors indicated that the two quantities affect the interface region in opposite directions, i.e., flow, which increases anisotropy and magnitude of interfacial area, and interfacial tension that tends to decrease area of interface and makes it isotropic. By assuming independency of these phenomena with each other and using a mathematical route with an ad-hoc manner for determination the effects of flow and interfacial tension, respectively, Doi and Ohta (1991) proposed the following equations for time developments of Q and q :

$$\begin{cases} \frac{\partial}{\partial t} q_{\alpha\beta} = -q_{\alpha k} \kappa_{k\beta} - q_{\beta k} \kappa_{k\alpha} + \frac{2}{3} q_{ij} \kappa_{ij} \delta_{\alpha\beta} - \frac{Q}{3} (\kappa_{\alpha\beta} + \kappa_{\beta\alpha}) \\ \quad + \frac{q_{ij} \kappa_{ij}}{Q} q_{\alpha\beta} - \lambda Q q_{\alpha\beta} \\ \frac{\partial}{\partial t} Q = -q_{\alpha\beta} \kappa_{\alpha\beta} - \lambda \mu Q^2, \end{cases} \quad (6a, b)$$

where λ and μ are two phenomenological parameters which related to relaxation of size and shape, respectively. By solving these nonlinear differential equations with proper initial conditions, and substituting the values of q into the following equation (Batchelor 1970; Onuki 1987), one can calculate the stress as:

$$\sigma_{ij} = \eta_0 (\kappa_{ij} + \kappa_{ji}) - \alpha q_{ij} + p \delta_{ij}, \quad (7)$$

where η_0 , κ_{ij} , α and p are Newtonian viscosity, macroscopic velocity gradient tensor, interfacial tension and hydrostatic pressure, respectively. The assumption of 50/50 mixture consequences to a co-continuous morphology allows one to use Doi-Ohta theory to obtain better results for blends, which have this type of morphology.

It is worth mentioning that Doi-Ohta theory is applied for some interesting scaling relationships such as: (1) It predicts constant viscosity despite of presence of interface in the system. (2) For a steady shear flow, both shear stress and first normal stress difference are proportional to the magnitude of shear rate. (3) In transient shear flow, when the shear rate is changed from an initial value $\dot{\gamma}_i$ to a final one $\dot{\gamma}_f$ at time $t=0$, the resulting transient stress $\sigma^+(t, \dot{\gamma}_i, \dot{\gamma}_f)$ divided by the initial steady stress $\sigma(\dot{\gamma}_i)$ superimposed onto a single master curve in terms of strain γ_f , regardless of shear rate, providing that the shear rate ratio ($\dot{\gamma}_f/\dot{\gamma}_i$) is kept constant:

$$\frac{\sigma^+(t, \dot{\gamma}_i, \dot{\gamma}_f)}{\sigma(\dot{\gamma}_i)} = f\left(\gamma_f, \frac{\dot{\gamma}_f}{\dot{\gamma}_i}\right) \quad (8)$$

Many studies on immiscible polymer blends have shown that these scaling relationships are in good agreement with the experimental data as cited in the introduction. Although the scaling law originally was

developed for 50–50 mixtures of Newtonian fluids, however, as indicated by Doi and Ohta (1991), the validity of this law is independent of the initial assumptions and should hold quiet generally for systems that include no intrinsic length scale. So the extensive works have been carried out for viscoelastic and non 50–50 blends as indicated in the introduction. In addition, Iza et al. (2001) presented the results for compatibilized PS/HDPE blends and validated the data with Doi–Ohta scaling relationship; however, the Doi–Ohta theory worked out for non-compatibilized blends. With careful insight, one may notice that the validity of these relationships hold better with introducing compatibilizer so this motivated us, to investigate this model more extensively. The major question was whether Doi–Ohta scaling relationship in transient flow holds better with increasing compatibilizer and weight fraction of dispersed phase.

In this study, all double start-up experiments were carried out with constant shear rates ratio set on 2: 0.05–0.1 s⁻¹, 0.1–0.2 s⁻¹ and 0.2–0.4 s⁻¹. Figures 11 and 12 show the results of these experiments on 90/10 and 80/20 blend series, respectively. With comparison of Figs. 11a and 12a, as one can observe, Doi–Ohta relationship validity hold better with increasing weight fraction of dispersed phase. Justification of this result may be referred to increasing of continuity index of PB (Joseph et al. 2003) that satisfy more the assumptions of the theory. On the other hand, by using compatibilizer the validity of the scaling law was improved as shown by Figs. 11 and 12. Based on the theory assumptions, validity of this scaling law normally holds for non-compatibilized blend. However, our results show the reverse behavior, i.e., the law is

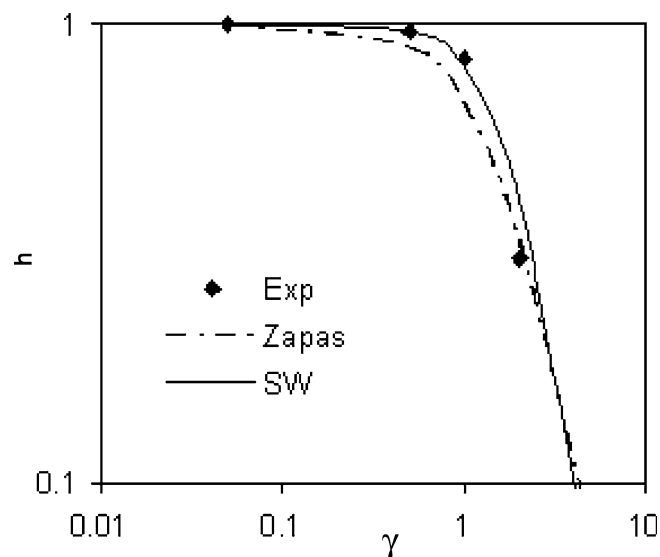


Fig. 9 The log–log plot of damping function versus strain: comparison between SW and Zapas equations and experimental results for blend 95/5/0

validated for compatibilized blends. It should be noted that the same conclusion were reported for scaling exponent of the shear stress and the first normal stress difference versus shear rate (Iza et al. 2001).

Conclusions

In this study, he has investigated the effect of compatibilizer on rheological properties of PS/PB blends. Using SBR as compatibilizer, it was observed that composition

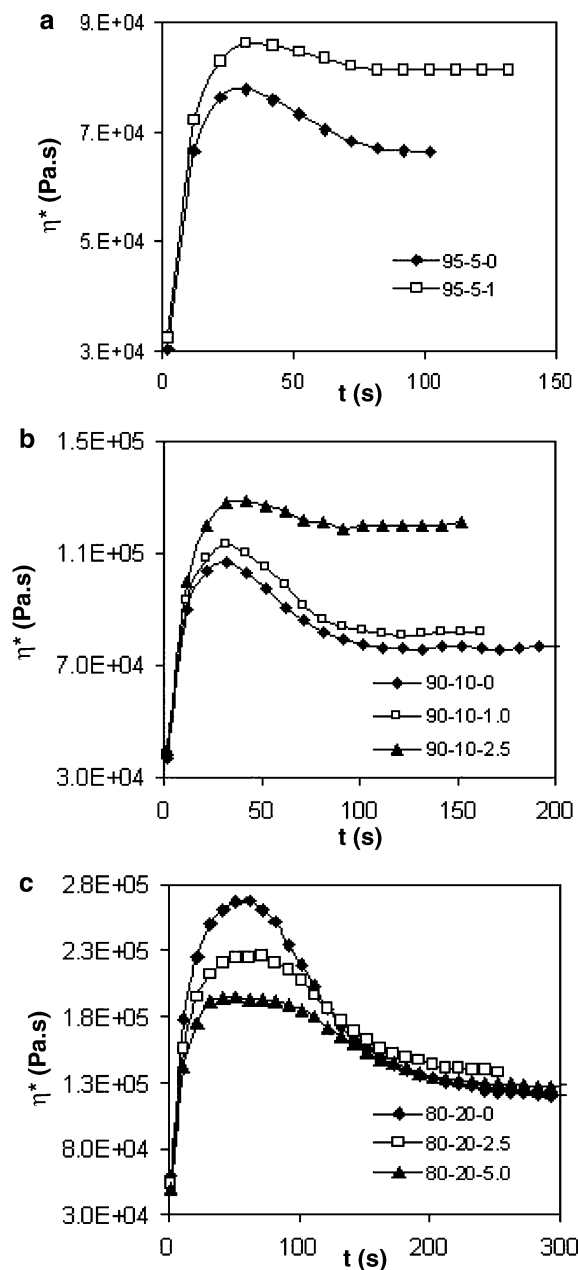


Fig. 10 The stress growth coefficients of: a 95/5; b 90/10; c 80/20 blends series

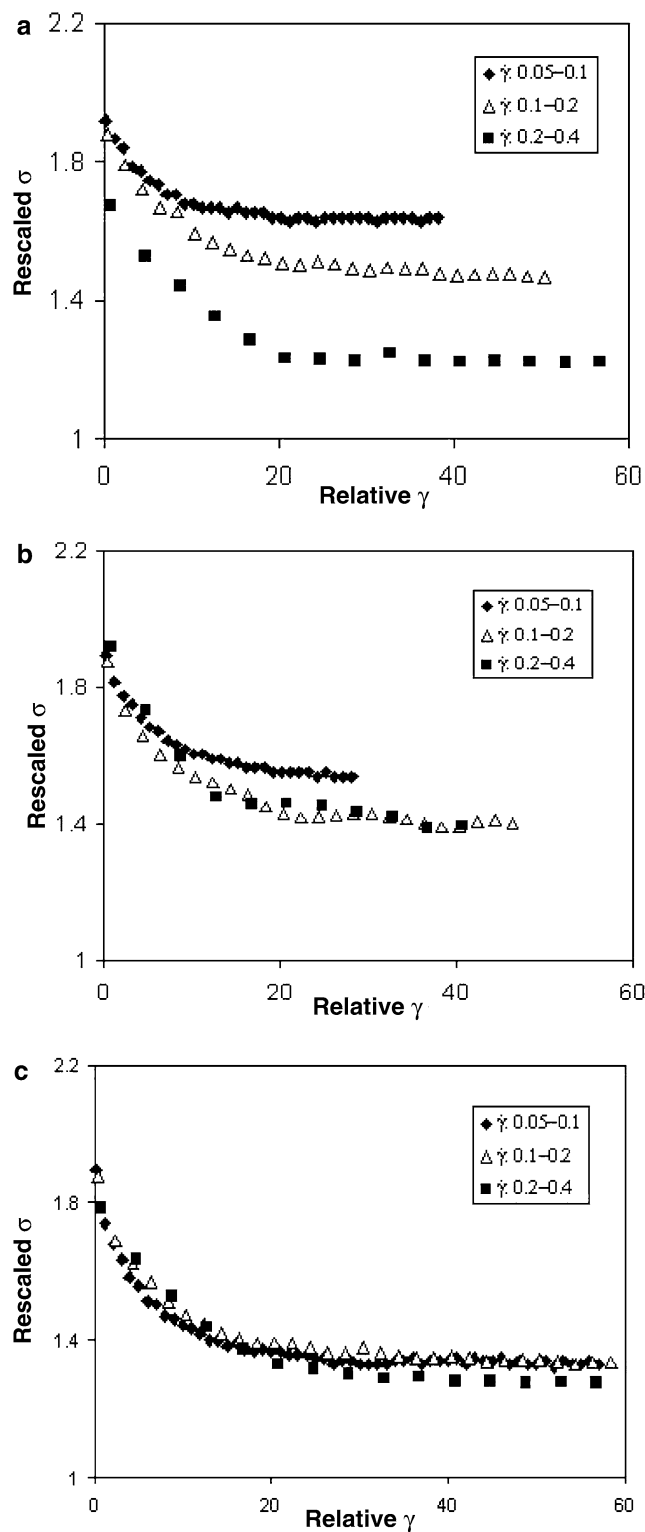


Fig. 11 The double start-up transient experiments for: a 90/10/0; b 90/10/1; c 90/10/2.5 blends

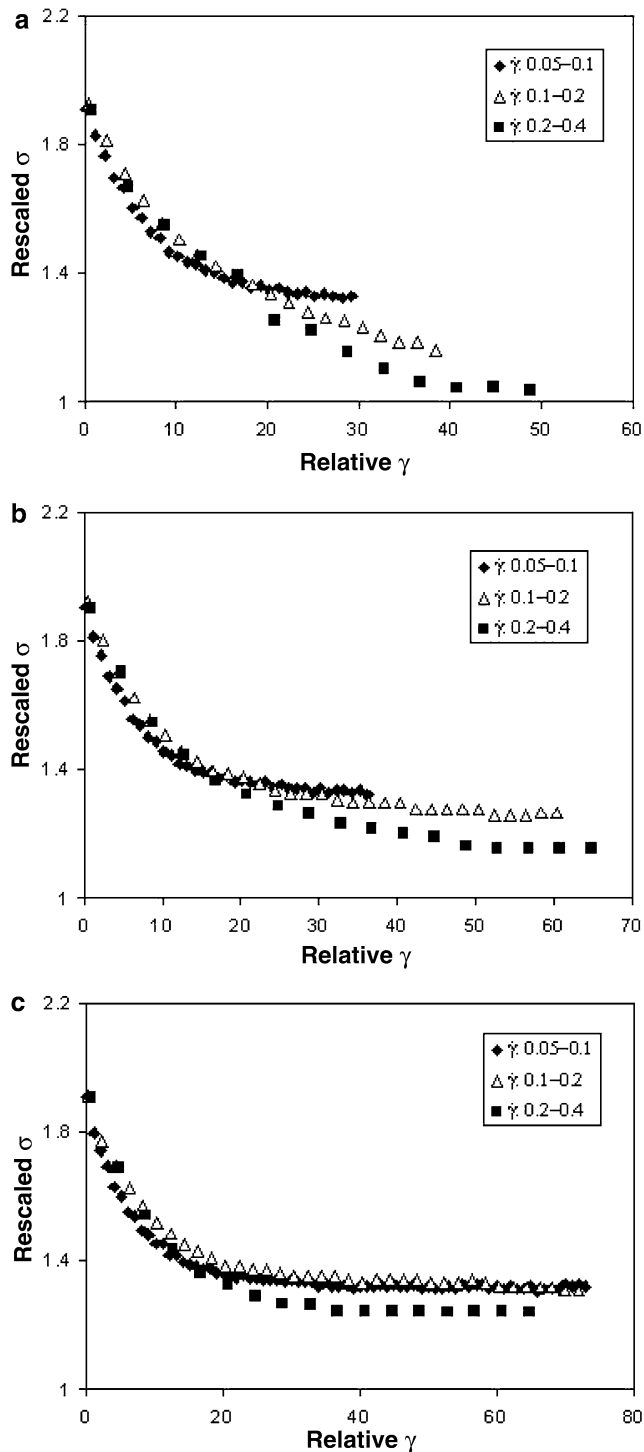


Fig. 12 The double start-up transient experiments for: a 80/20/0; b 80/20/2.5; c 80/20/5 blends

of compatibilizer influences on the modulus of the blends so that for the 95/5, 90/10 and 80/20 blends it increases, low rise or no change and decreases, respectively. These observations demonstrated themselves with

various level of intensity with the other experiments as low in frequency sweep and high for transient tests as reported in previous studies. The origin of these phenomena comes back to the morphology of the blends, so two factors, dispersed phase domain size and interfacial tension, have major influence on the modulus of the blends. If reduction of the interfacial tension is much more than reduction in the dispersed domains size, where their ratio becomes greater than original blend ratio, then resulting stress will be greater than non-compatibilized original blend; and vice versa. It is concluded that it is possible to have a qualitative sense

of effect of compatibilizer on morphology of the blend, with a rheological test.

In addition, in the double start-up experiments, validity of Doi–Ohta scaling relationship has investigated. As expected, with increasing the amount of compatibilizer and weight fraction of minor phase, this correlation holds better and the curves superimpose onto each other. This is due to enhancement of continuity index of minor phase for non-compatibilized blends.

Acknowledgements Authors acknowledge Dr. G.H. Sodeifian for his kindly helps and to M. Yavarizadeh for GPC data.

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