Investigation and Prediction on the Nonlinear Viscoelastic Behaviors of Nylon1212 Toughened with Elastomer

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ABSTRACT: Studies on the nonlinear viscoelastic behaviors of nylon1212 toughened with styrene-[ethylene-(ethylene-propylene)]-styrene block copolymer (SEEPS) were carried out. The linear relaxation curves at relatively low shear strains show good overlap, the relaxation time and modulus corresponding to the characteristic relaxation modes were also acquired through simulating the linear relaxation modulus curves using Maxwell model. The nonlinear relaxation curves of nylon1212 blends at different shear strains have been obtained and their damping functions were evaluated. Meanwhile, it is found that most blends in the experimental windows follow the strain-time separation principle and Laun double exponential model can predict damping curves well. The successive start-up of shear behavior was investigated. The results showed that Wagner model, derived from the K-BKZ (Kearsley-Bernstein, Kearsley, Zapas) constitutive equation, could simulate the experiment data of nylon 1212 blend with 10 wt % SEEPS well, but there exists some deviation for experiment data of nylon1212 blends with high SEEPS concentrations. © 2011 Wiley Periodicals, Inc. J Appl Polym Sci 123: 1283–1292, 2012

Key words: nonlinear viscoelastic behavior; damping function; Wagner model; nylon1212; blends

INTRODUCTION

Multicomponent polymer blending has attracted everlastingly increasing attention because it is an efficient way to improve the properties and enlarge the applicative range of origin materials. A lot of complex polymeric materials have been developed to satisfied different requirements. Polymer blends can be divided into homogeneous (miscible) and heterogeneous (immiscible) blends. Homogeneous blends are relatively rare because the change in the combinatorial entropy of mixing two high molecular weight polymers is negligible. Heterogeneous blends are two phase that exhibit usually advantages over the single phase polymers.[1] High toughness polymeric materials are always acquired through blending engineering plastics with suitable elastomeric materials, which has become one of the most effective methods to improve the toughness of a brittle polymer. The degree of toughness can be determined by the blending ratio, the size of phase domain, the interaction between matrix and elastomer, the blending approach, processing parameters etc.[2–9]

A lot of researches concerning nylon/elastomer blends have mostly focused on the aspects of mechanical and thermal properties.[10–15] To our knowledge, a few reports have been dealt with rheological behaviors of their melts, especially for their nonlinear rheology.[16–20] Due to its highly polar amide end-groups, nylon is uncompatible with SEEPS elastomer, resulting in heterogeneous blends. The rheological measurements not only give much useful information for material molding and processing but also is an important way to characterize structure and properties of polymer materials.[21,22] Processability of polymers is directly related to the rheological behaviors. For homopolymers, the rheological behaviors depend mainly on chain and agglomeration structure. In the case of polymer blends, the rheological behaviors are related with not only the viscoelastic behaviors of blending compositions but also phase morphologies, interactions in the interface, so it is complex to probe the essence of rheological behaviors of the polymeric blends. The linear viscoelastic behavior of polymers and their blends have attracted ever-increasing interests of researches, and the correlations of their microstructure and linear viscoelastic behavior have been reported.[23–27] However, up to now, the thorough understanding for nonlinear viscoelastic behavior has not been achieved, especially for the polymeric blending systems.

It is well-known that most polymers exhibit the nonlinear viscoelastic behavior under relatively large strains, whereas the nonlinear viscoelastic behavior can also be observed under relatively small strains for suspension, gel, and polymer composite systems. Recently, to probe the essence of nonlinear viscoelastic behavior, many works had been done.[28–34] Among them, one way is that people employed some constitutive equations to simulate the nonlinear viscoelastic behavior of polymer, in which Kearsley-Bernstein Kearsley Zapas (K-BKZ) equation is the integral one that has been most widely used. Based on K-BKZ constitutive equation, Wagner et al. investigated the variation of stress with time under various flow conditions, and proposed a new model, i.e., the so-called "Wagner model," for simulating the nonlinear viscoelastic behavior of polymer.[35,36] We have discussed the linear viscoelastic behavior in the previous article,[37] the aim of this article is to investigate the nonlinear viscoelastic behaviors of the nylon1212/elastomers blends, obtain the damping function according to time-strain separability principle, and predict the nonlinear viscoelastic behaviors based on Wagner model.

Theoretical background

According to rubber-liquid theory,[38] Bernstein, Kearsley, and Zapas proposed a K-BKZ equation to predict the nonlinear viscoelastic behavior as given as[39]

$$\sigma'_{ij} = \int\limits_0^\infty [m_1(s, I_1, I_2)C_{t\,ij}^{-1} + m_2(s, I_1, I_2)C_{t\,ij}]ds, \quad (1)$$

in which C_t^{-1} and C_t are the Finger and Cauchy tensor respectively; m_1 and m_2 are assumed to be memory functions of the time difference s; s is time from present to past defined as $s = t - t'$. As the secondary normal stress difference is not considered here, m_2 is set equal zero. m_1 can be separated into strain-dependent and time-dependent functions in the nonlinear viscoelastic region as follows,

$$m_1(s, I_1\,I_2) = \mu(s)h(I_1, I_2), \quad (2)$$

in which $\mu(s)$ is a linear memory function having time-dependence which can be obtained through simulating the linear relaxation modulus with the generalized Maxwell model, and $h(I_1, I_2)$ is called a damping function.

Based on K-BKZ equation, Wagner introduced a new constitutive equation for describing the strain history applied to the samples.

$$\sigma(t - t') = \int\limits_{-\infty}^t m(t - t')h(I_1, I_2)C_t^{-1}(t, t')dt'. \quad (3)$$

The memory function in the eq. (3) can be represented as

$$m(t - t') = \frac{\partial G(t - t')}{\partial t} = \sum_{i=1}^n a_i e^{-(t-t')/\lambda_i}$$
$$= \sum_{i=1}^n \frac{G_i}{\lambda_i} e^{-(t-t')/\lambda_i}, \quad (4)$$

in which $G(t - t')$ is stress relaxation modulus, λ_i and G_i are characteristic values determining stress relaxation behavior. If the volume of sample is constant, a simple shear flow at constant shear rate $\dot{\gamma}$ can be described in terms of the shear deformation, $\gamma(t) = \dot{\gamma} \cdot t$, and the Finger tensor is given by

$$C_t^{-1}(t') = \begin{pmatrix} 1 + \dot{\gamma}^2(t - t')^2 & \dot{\gamma}(t - t') & 0 \\ \dot{\gamma}(t - t') & 1 & 0 \\ 0 & 0 & 1 \end{pmatrix}. \quad (5)$$

Hence, the invariants of $C_t^{-1}(t')$ are

$$I_1 = I_2 = \dot{\gamma}^2(t - t')^2 + 3 = \dot{\gamma}^2 s^2 + 3$$
$$I_3 = 1. \quad (6)$$

From eq. (3), the shear viscosity $\eta(t, \dot{\gamma}_0)$ are calculated as

$$\eta(t, \dot{\gamma}_0) = \frac{\sigma_{12}(t, \gamma_0)}{\dot{\gamma}_0} = \int\limits_0^t G(s)h(\gamma(s)^2)s\frac{\partial}{\partial s}\ln(h(\gamma(s)s)ds. \quad (7)$$

If the relaxation modulus curves of samples under different large strains are parallel each other, the viscoelastic behaviors of samples obey the time-strain separation principle.[40] The nonlinear viscoelastic modulus $G(\gamma, t - t')$ can be divided into a linear viscoelatic modulus $G(t - t')$ and a strain-dependent function as given as,

$$G(\gamma, t - t') = G(t - t') \times h(\gamma), \quad (8)$$

in which $h(\gamma)$ is referred to as "damping function," which can be used for describing the nonlinear rheological response. In fact, eq. (8) is only a factorization method allowing us to separate the time and the strain effects and to extract the contribution of the strain to the total relaxation modulus. As for simple shear, Wagner proposed the following expression for the damping function[35]

$$h(\gamma) = e^{-k\gamma}. \quad (9)$$

For complex fluid system, Soskey, Laun, and Papanastasiou proposed three damping functions as follows[41–43]

$$h(\gamma) = \frac{1}{1 + \alpha\gamma^{\beta}}, \qquad (10)$$

$$h(\gamma) = Ke^{-a_1\gamma} + (1-K)e^{-a_2\gamma}, \qquad (11)$$

$$h(\gamma) = \frac{b}{b + \gamma^2}, \qquad (12)$$

in which γ is shear strain, K, α, β, a_1, a_2 and b are fitting parameters, which are determined by the chemical structure, the molecular weight, the molecular weight distribution and flow types. If the linear viscoelastic region and the damping function of sample are determined, eq. (3) can be used to predict the nonlinear viscoelastic behavior. When the damping function follows Wagner equation, $\eta(t, \dot{\gamma}_0)$ can be calculated as

$$\eta(t, \dot{\gamma}_0) = \frac{\sigma_{12}(t, \dot{\gamma}_0)}{\dot{\gamma}_0}$$
$$= \sum_{i=1}^{n} \frac{a_i\lambda_i^2}{(1 + n\dot{\gamma}_0\lambda_i)^2}[1 - e^{-t_{r,i}}(1 - n\dot{\gamma}_0\lambda_i t_{r,i})],$$
$$(13)$$

in which

$$t_{r,i} = \frac{t}{\lambda_i} + n\dot{\gamma}_0 t, \qquad (14)$$

It is noted that other three types of damping functions mentioned above can also be used to calculate the corresponding nonlinear viscoelastic functions.

EXPERIMENTAL

Materials

Nylon1212 (melt-flow index = 22.43 g/min) used here is a product from Shandong Dongchen Engineering Plastic Co., China. Styrene-[ethylene-(ethylene-propylene)]-styrene block copolymer (SEEPS 4033, polystyrene end-blocks = 30 wt %, the density = 0.91 g/cm^3. The melting index (MI) $\leq$ 0.1 under 2.16 kg pressure at 230°C, the viscosity = 50 mPa s in the 10 wt % toluene solution, $\overline{M}_n = 67,240$, $\overline{M}_w = 91,365$, $\overline{M}_z = 122,487$, $\overline{M}_w/\overline{M}_n = 1.3588$) is a product of Kuraray Co., Japan. The antioxidant (B215, the relative molecular weight = 647, $T_m = 453$–458 K) is a product of Ciba-Geigy Co., Switzerland.

Preparation of samples

Nylon1212 and SEEPS and 1 wt % antioxidant were blended in a Haake torque rheometer (Rheoflixer Polylab) at 190°C for 10 min. The samples were compressedly molded at 190°C into disks of 25 mm in diameter and 1.2 mm in thickness.

Rheological measurements

Melt rheological tests were conducted on an ARES rheometer (Rheometrics, Piscataway, NJ) in parallel plates mode. To record the relaxation modulus varying with time, the stress relaxation tests were performed under condition of the amplitude being 0.1–100% and the test time being 500 s. The successive start-up of steady shear tests have been carried out in the shear rate 0.01 s^{-1}, 0.05 s^{-1}, 0.1 s^{-1}, 0.5 s^{-1}, the test time for each shear rate is 100 s. At the end of that time, the shear rate increases to the next level without allowing the samples to relax.

RESULTS AND DISCUSSIONS

Evaluation of the linear relaxation behavior

Figure 1 presents the linear relaxation modulus curves of nylon1212 blends at various shear strains. Linearity can be obtained for stain values up to 5%, namely the linear relaxation modulus $G(t)$ is independent of the applied strains. For the blends with low concentration of elastomer, the relaxation behaviors are controlled by matrix of virgin nylon1212, exhibiting a fast relaxation process, while the relaxation behaviors of the blends with high concentration of elastomer firstly show fast and then slow relaxation processes. The former is corresponding to the relaxation behavior of hard block (PS), while the latter can be attributed to the relaxation behavior of random copolymer block.[33] In addition, it can be observed that the overlapping curves after about 10 s for nylon1212/SEEPS (70/30 and 60/40) blends are not better than those of other blends, which should be related with the inhomogeneous structures. The morphologies of blends change from droplet-matrix structures to double phase cocontinuity structures.[37]

In the linear viscoelastic range, $G(t)$ can be simulated by Maxwell model as given as,

$$G(t) = \sum_{i=1}^{n} G_i e^{-(t-t')/\lambda_i} = \sum_{i=1}^{n} a_i\lambda_i e^{-(t-t')/\lambda_i}, \qquad (15)$$

in which, G_i and λ_i are the modulus and the relaxation time of the ith relaxation mode, respectively. The knowledge of the set (G_i, λ_i) is very useful because it allows one to predict the behavior of the material in any standard experiment, the transient viscosity, plateau modulus and other viscoelastic parameters can be determine by the set (G_i, λ_i). In the section of theoretical background, the memory function in the Wagner function can be obtain from simulation on the linear stress relaxation modulus curves using Maxwell model. Here, to evaluate easily the transient viscosity, we make the relaxation times equal to 0.01, 0.1, 1, 10, 100 and obtain the five
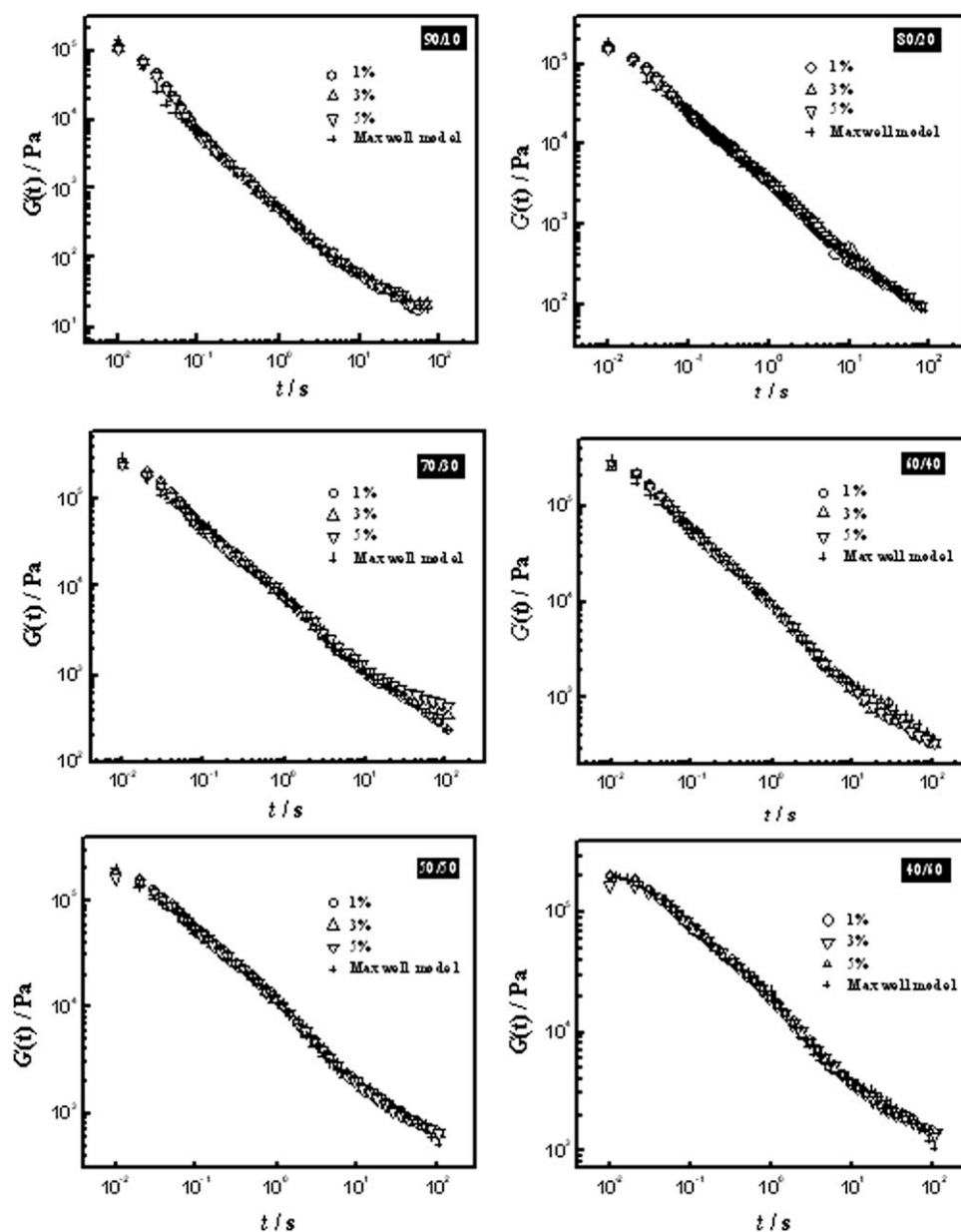
Figure 1 The linear relaxation modulus curves of nylon1212/SEEPS blends and their predictions by Maxwell model.

sets (G_i, λ_i). Of course, the relaxation behaviors of blends are complex, the characteristic relaxation times can be attributed to polymers of two phases and the interface region between two phases. The best fitting values of G_i and λ_i are summarized in Table I and corresponding results are shown in Figure 1. It can be seen from Figure 1 that the simulated curve can describe the relaxation process of nylon1212 blends well in the range of whole time scales.

Evaluation of the damping function

Usually, for homopolymers, the damping function can be determined by shifting $G(t)$ vertically at the longer relaxation time region.[25] However, for multi-

phase or multicomponent polymeric materials, the superposition becomes to be breakdown. The reason for this is that $G(\gamma, t)$ data in the examined range of t are resulted from different relaxation processes. For nylon1212 blends, the relaxation behavior can be separated into two relaxation processes. The former can be attributed to the relaxation of nylon 1212 matrix and polystyrene block in SEEPS; while the latter is the relaxation behavior of random copolymer block in SEEPS. On the other hand, in our experiment window, SEEPS elastomer does not relax fully, and as a result, the longer relaxation behaviors cannot be accurately examined. So, in this article, we evaluate the damping function at the reference time, $t = 10$ s, at which the nylon1212 matrix and PS block

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TABLE I
The Results of Prediction Using Maxwell Model for nylon1212/SEEPS Blends

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λ_i (s)	G_i (Pa)					
	90/10	80/20	70/30	60/40	50/50	40/60
10^{-2}	319500	351950	533490	495920	260460	203850
10^{-1}	16247	49967	95684	109720	89670	118930
10^{0}	1213	8455	16254	20703	23858	39000
10^{1}	74	700	1506	1307	2254	3413
10^{2}	38	215	638	1024	1384	3253

relaxations almost complete, and the relaxation of random block hardly begins.

Figure 2 gives the nonlinear relaxation modulus curves of nylon1212 blends at various shear strains. It can be seen that $G(\gamma, t)$ for all the blends decrease with increase of the relaxation time, and the relaxation curves can shift vertically along the direction of modulus reduction with γ increasing. With the SEEPS concentration increasing, nylon 1212 blends show strong sensitivity to shear strains. When the relaxation time is over 10 s, all the relaxation processes of blends become slow, which is related to the multiphase structures of blends. The whole relaxation processes of blends include the rapid relaxation of nylon 1212 matrix, the slow relaxation of SEEPS elastomer and the interface relaxation.

Figure 3 presents the relationship between $G(\gamma, t)/h(\gamma)$ and time for nylon1212 blends. It can be seen that the curves of nonlinear relaxation modulus $G(\gamma, t)$ for most nylon1212 blends show relatively good superposition in the whole examined range of time, indicating the validity of the time-strain separation principle. However, it also can be observed that some curves of $G(\gamma, t)$ at high shear strains have some deviation from master curves in the longer time region because the interfacial interactions between two phases have been destroyed by the applied large strains, the time-strain separation principle is valid when the time is shorter than 10 s. In addition, it is noted that the shear rate in the transient test is low and the test time is short. Hence, we believe that the damping function evaluated by this method can be used to predict the nonlinear viscoelastic behavior of SEEPS.

It is noted that determination of the damping function is the key to develop the constitutive equation of the nonlinear viscoelastic behavior. Figure 4 presents the curve of the damping function $h(\gamma)$ with γ. It is known that many different forms of the damping function have been used to fit the experiment data. Among them, Wagner equation predicts a much quicker decrease in the damping function with increasing strain in the nonlinear viscoelastic region than that observed in the experimental data for most blends. Moreover, Laun equation can predict the experiment data of all the bends well, Papa-

nastasiou equation and Soskey and Winter equation only can simulate the curves of some nylon1212 blends. The parameters in the four models were determined through the *Origin7.0* software using the nonlinear least squares fitter analysis mode. Table II gives the fitting parameter values of four damping functions for nylon 1212 blends. It can be seen that every parameter doesn't exhibit rules with the increase of SEEPS concentration, because the fitting parameters are related with chemical structure, the molecular weight, the molecular weight distribution and flow types. Here, we choose Laun damping equation to describe the effects on nonlinearity of viscoelastic functions, the other three models also can be introduced into the Wagner model to acquire the expressions of transient viscoelastic behavior. Here, those results are not presented in this article.

Evaluation of successive start-up of steady shear behavior

Figure 5 illustrates the results of start-up steady shear experiments for nylon1212 blends. It is noted that the applied shear rates varies from 0.01 to 0.5 s^{-1} and the time at each of shear rates is 100 s. At the end of that time, the shear rate increases to the next level without allowing the sample to relax. It can be seen that the nonlinear transient shear viscosity $\eta^+(t, \dot{\gamma})$ decreases with increase of the shear rates and increases rapidly to a stable plateau, the decreasing degree of $\eta^+(t, \dot{\gamma})$ increasing with SEEPS concentration increasing, which is related with relative weak interfacial interaction and mutiphases structure. In addition, introduce of SEEPS elastomer improve $\eta^+(t, \dot{\gamma})$ of blends and the sensitivity of $\eta^+(t, \dot{\gamma})$ to shear rates. In general, stress overshoot is a relatively common phenomenon, especially for the nonlinear viscoelastic behavior of polymers appearing at relatively high shear rates. Here, obvious stress overshoot behavior is observed at 0.5 s^{-1}, while this phenomenon can't be observed at other applied shear rates for all the blends.

From the experimental data, the changes of structure can be shown. when the shear rate is low, the transient viscosity increases rapidly to a stable plateau, which exhibits that the strain history make the
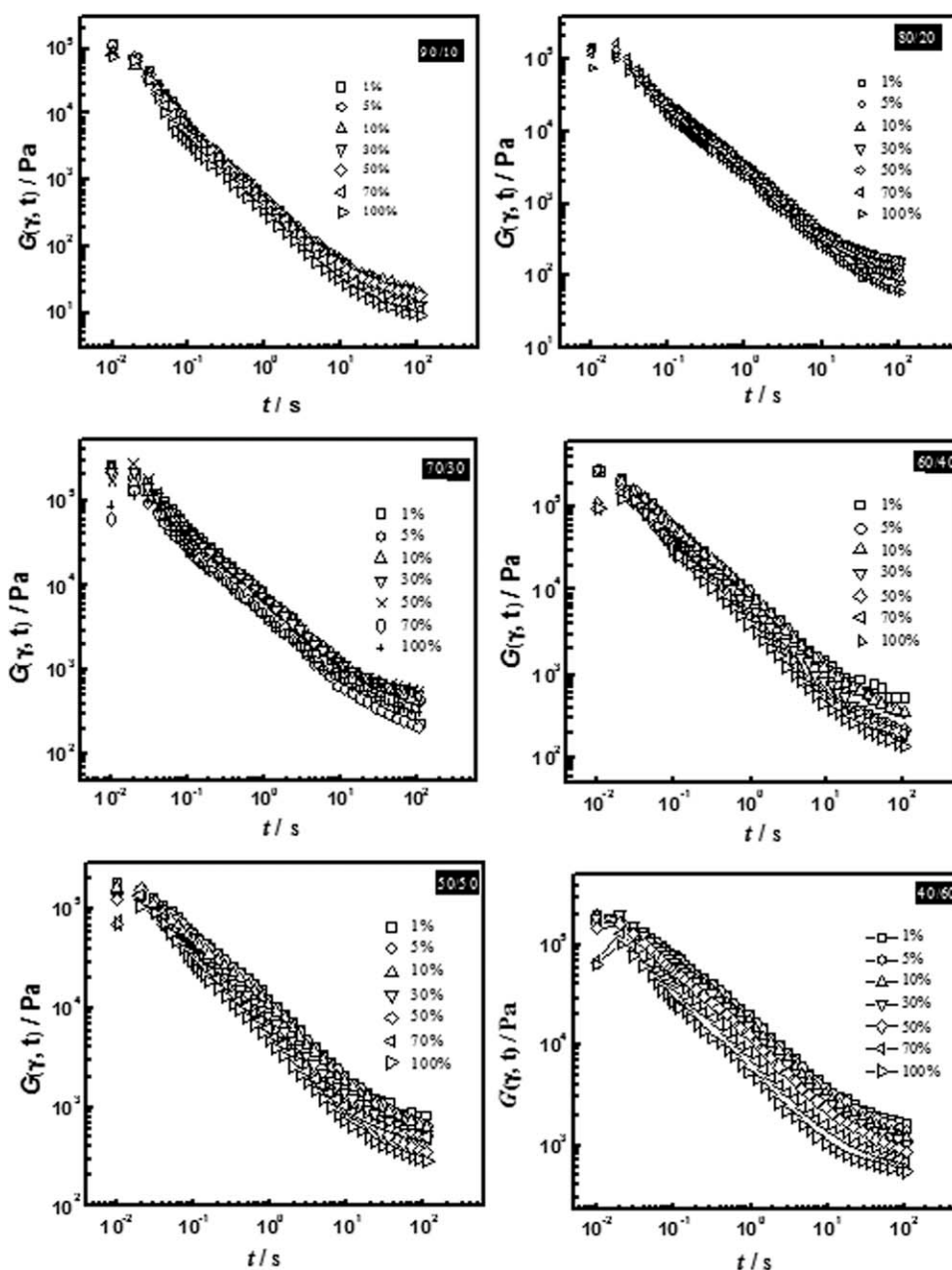
Figure 2 The nonlinear relaxation modulus curves of nylon1212/SEEPS blends at different shear strains.

structure of this sample derivate from the stable state while the sample tries its best to revert to original state. If the applied time is enough long, the structure of this sample will approach a new dynamic equilibrium state and form a viscosity plateau. When the shear rate is high, the transient viscosity decreases gradually after a plateau, indicating that the accumulative strain history further to destroy the structure of this system. If the shear rate increase further, the "stress overshoot" can be considered as a label of father destruction of structure.

For successive start-up steady shear behavior, $\eta^+(t, \dot{\gamma})$ can be expressed using Wagner model as

$$\eta^+(\dot{\gamma}, t) = \int\limits_0^t G(s) \left[s \frac{\partial h(s)}{\partial(s)} + h(s) \right] ds. \qquad (16)$$

Taking the relaxation modulus $G(s)$ defined by eq. (15) and the damping function defined by eq. (11) into eq. (7), $\eta^+(t, \dot{\gamma})$ can be calculated as

$$\eta^+(t, \dot{\gamma}_0) = \sum_{i=1}^n \frac{G_i \lambda_i K}{(1 - a_1 \dot{\gamma}_0 \lambda_i)^2} [1 - e^{-t_{r,i,1}}(1 - a_1 \dot{\gamma}_0 \lambda_i t_{r,i,1})]$$
$$+ \sum_{i=1}^n \frac{G_i \lambda_i (1 - K)}{(1 - a_2 \dot{\gamma}_0 \lambda_i)^2} [1 - e^{-t_{r,i,2}}(1 - a_2 \dot{\gamma}_0 \lambda_i t_{r,i,2})],$$
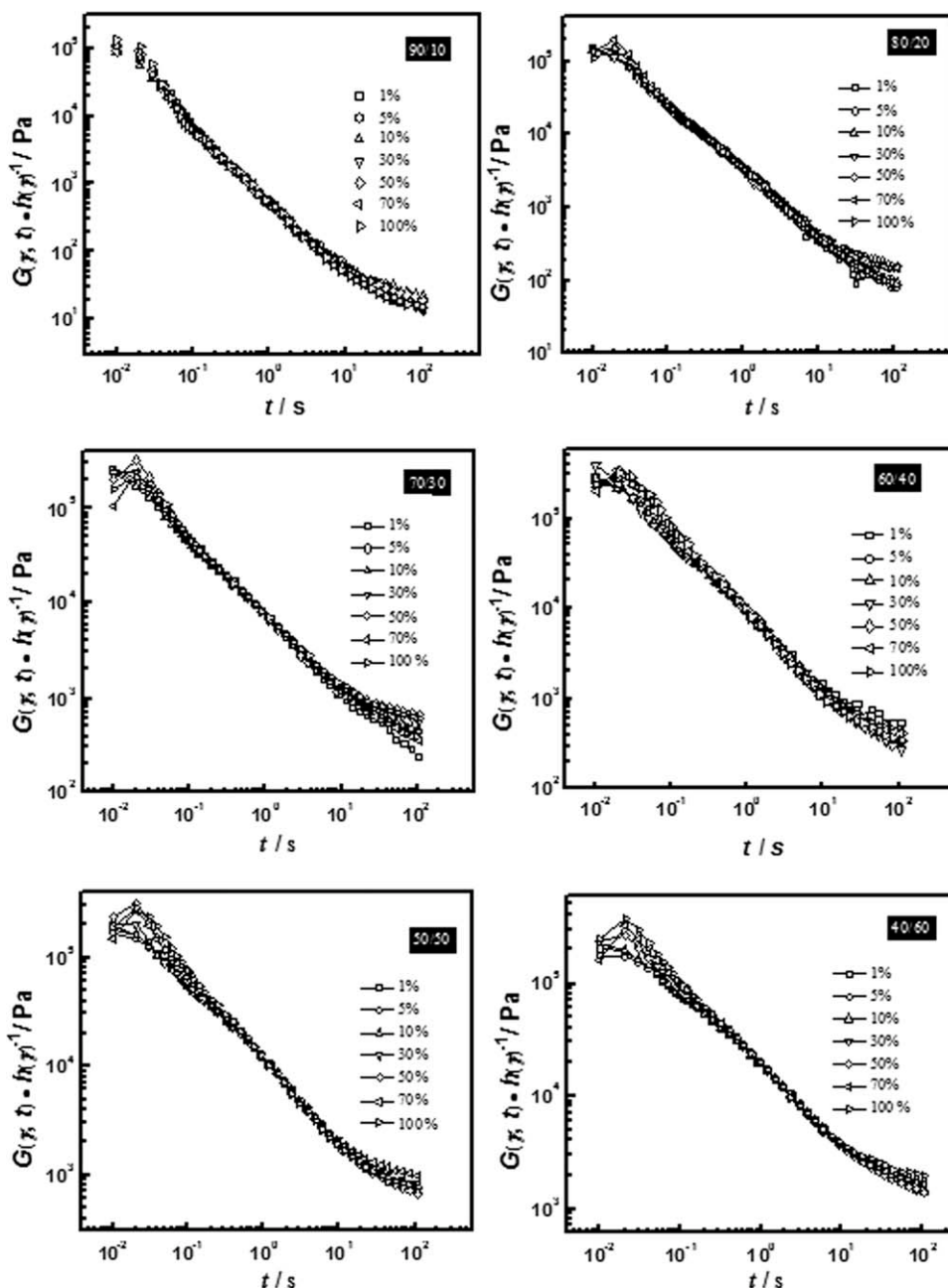
$$(17)$$

Figure 3 The relationships between $G(\gamma, t)/h(\gamma)$ and time for nylon1212 blends.

in which

$$t_{r,i,1} = \frac{t}{\lambda_i} + a_1\dot{\gamma}_0 t, \quad t_{r,i,2} = \frac{t}{\lambda_i} + a_2\dot{\gamma}_0 t. \quad (18)$$

Figure 5 illustrates the effect of shear rate and time on the transient viscosity for nylon1212 blends through fitting nonlinear viscoelastic behavior based on Wagner model. It can be seen that nonlinear Wagner model can predict not only experimental data of nylon 1212/SEEPS (90/10) blend at 0.05 s^{-1} well, but also the plateau value at 0.05 s^{-1} and 0.1 s^{-1} and the stress overshoot behavior at 0.5 s^{-1}.

As for blends (80/20), although Wagner equation can describe the changing tendency of $\eta^+(t, \dot{\gamma})$, the simulating curves are higher than experiment data, which suggests that Wagner model cannot predict the nonlinear viscoelastic behavior of this blends. Based on the simulating results of other nylon1212 blends, it can be concluded that Wagner model cannot simulate the nonlinear nylon1212 blends with high SEEPS concentration well, which is similar to the simulating results of linear viscoelastic behavior using Palierne model.[37]

It has been discussed in our previous articles that when the concentration of SEEPS is relatively low,
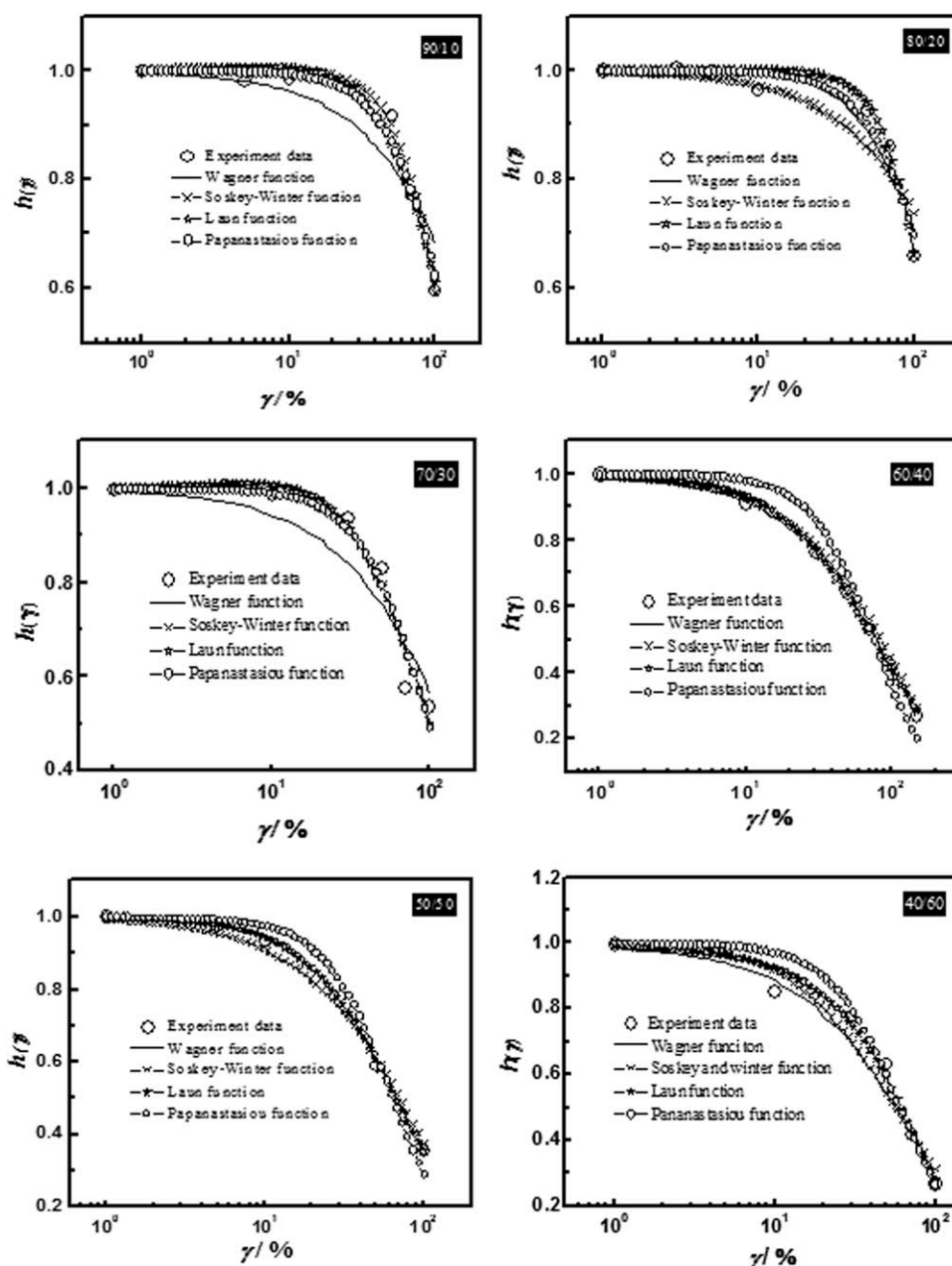
Figure 4 The damping functions of nylon1212/SEEPS blends and predictions with four mathematic functions.

the morphologies of blends show droplet-matrix structure and interactions between the disperse phases and matrix is weak.[37] Therefore, the linear

and nonlinear rheological behaviors of blends with low concentration of SEEPS are controlled by the nylon1212 matrix, the relaxation curves and

TABLE II
The Fitting Parameters of Four Damping Functions for Nylon1212/SEEPS Blends

	$\alpha \times 10^{-4}$	β	$a_1 \times 10^{-3}$	$a_2 \times 10^{-2}$	K	$b \times 10^3$	$k \times 10^{-3}$
90/10	12	1.5	7.3	18	1.1	9.21	6.5
80/20	1.0	2.3	7.3	2.6	1.5	22.9	2.9
70/30	1.1	3.6	9.5	5.4	1.3	18.1	5.7
60/40	34	1.3	8.5	1.7	1.0	5.71	8.2
50/50	19	1.5	11	12	1.1	4.07	4.8
40/60	6.3	1.3	26	4.4	2.0	1.80	1.2
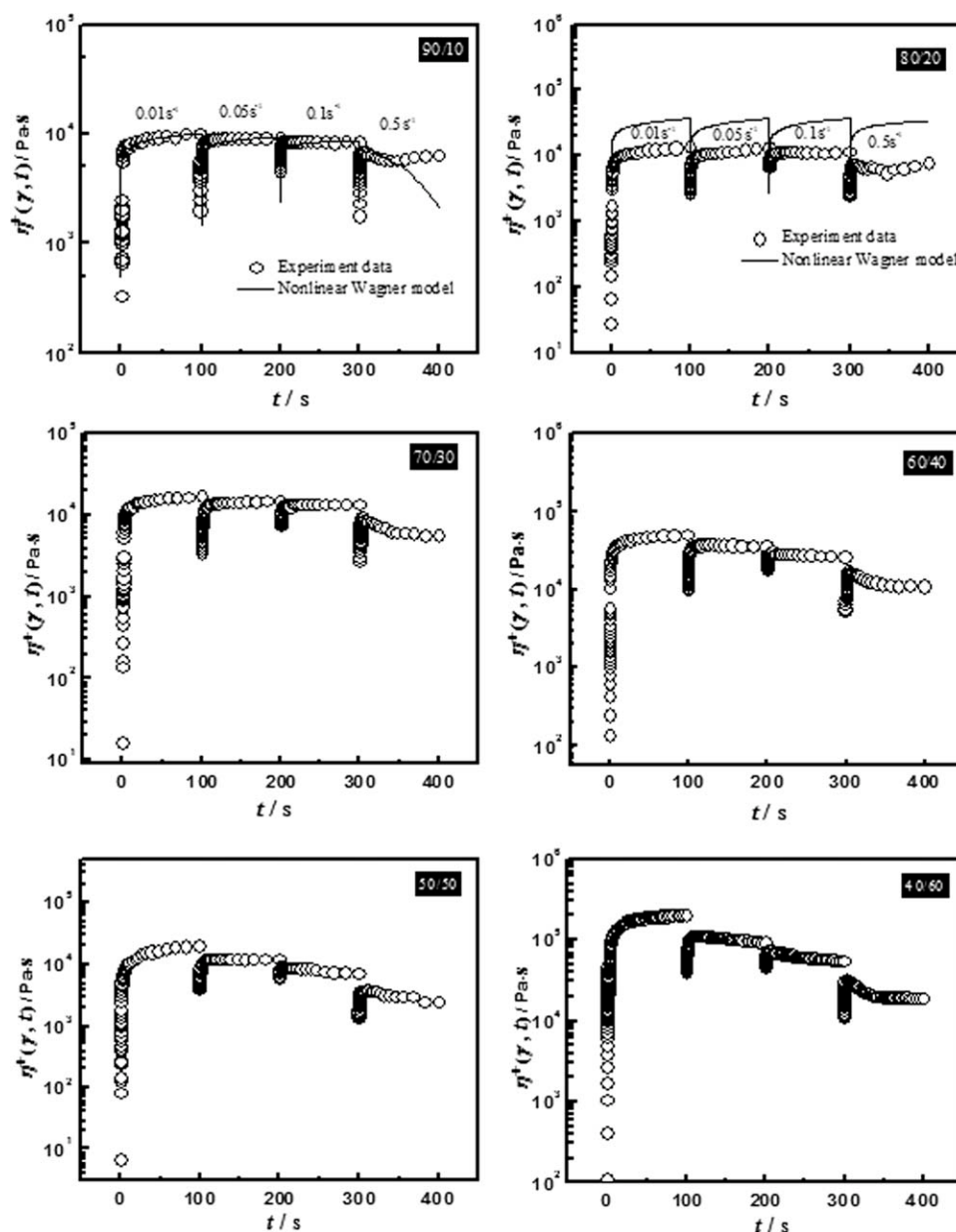
Figure 5 The effects of shear rate and time on the transient viscosity of nylon1212/SEEPS blends, the fit of nonlinear Wagner model is illustrated in the figure with a solid line.

damping functions of nylon1212 blends with low SEEPS concentration are similar to those of matrix, we can use Wagner model acquired from homopolymer to predict their nonlinear viscoelastic behavior. When the concentrations of SEEPS increase, the blends enter gradually into phase transition region and the morphologies of blends change from droplet-matrix structure to double phase cocontinuity structures and the interactions between two phases strengthen obviously. The nonlinear viscoelastic behavior can not only be controlled by the matrix and disperse phases, but the interaction between matrix and disperse phases can affect the nonlinear viscoelastic behavior. Therefore, it is difficult to

obtain an appropriate damping function to describe the viscoelastic behaviors of blends.

CONCLUSIONS

The nonlinear viscoelastic behaviors of nylon1212/SEEPS blends were carried out. In the linear region, Maxwell model was used to evaluate the relaxation behavior and the time and modulus corresponding to the characteristic relaxation modes was achieved. The damping function can be obtained from the nonlinear and linear relaxation curves and Laun double exponential function could be used for describing experimental data well. Moreover,

Wagner model was applied to predict the nonlinear rheological behavior under the successive start-up of shear, the results show that Wanger model is appropriate to predict not only the homogeneous polymer, but also that of nylon1212 blends with low SEEPS concentration. The reasons are that the relaxation curves and damping functions of nylon1212 blends with low SEEPS concentration are controlled mainly by the matrix. As for the blends with high SEEPS concentration, complex morphologies and interaction in the interface can affect obviously the nonlinear viscoelastic behaviors, so it is difficult to obtain an appropriate damping function to describe the viscoelastic behaviors of blends and fail to simulate the nonlinear viscoelastic behaviors with Wagner model.

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