<연구논문>

비상용성 고분자 블렌드의 전단응력과 법선응력

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(1997년 4월 14일 접수)

Steady Shear and Normal Stresses of Immiscible Polymer Blends

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(Receibed April 14, 1997)

요 약

비상용성 고분자는 그 계면의 특성에 의해서 복잡한 유변학적 거동을 갖는데, 그러한 거동을 해석하기 위해서, 최근에 제안된 구성방정식을 토대로 소폭 진동 전단 흐름장에서의 유변학적 물성과 몰폴로지 전개를 예측한 바 있다. Takahashi[2] 등은 이런 흐름하에서의 계면은 거의 변하지 않음을 보여 주었고, 따라서 이 경우는 상대적으로 쉬운 편이었다. 본 연구에서는 정상 전단흐름장하에서의 몰폴로지 전개에 대한 구성방정식을 간단한 형태로 표현함으로써, 실제 산업계에서 이용될 수 있도록 하였다. 그러한 해석을 통하여, 원래의 모델에서 제시되었던 3개의 실험 변수를 2개로 줄일 수 있었으며, 계면의 특성을 잘 나타내 주는 새로운 변수 (κ)를 도입하였다. 계면의 특성을 잘 나타내 주는 이 새로운 변수를 통하여, 그 계면의 영향을 예측할 수 있었다. 한편, 분산상의 파괴,변형,합체 메카니즘을 모델에 제시되었던 변수값들을 통하여 해석하였고, 이를 실험적인 데이타와 비교해 보았다.

Abstract — Recently we proposed a new set of constitutive equation that can describe the interfacial contributions on the rheological properties of immiscible polymer blends with complex interfaces and successfully compared with dynamic experiments[1]. As Takahashi et. al. [2] have noticed, the domain structures remain unchanged under oscillatory flows with appropriate frequency and strain. So, it is relatively easy to consider such cases. Here we would like to explore it further to obtain the compact expressions for the steady shear properties. Furthermore we would like to compare them with experimental observations for the understanding of the morphological differences in the interfaces. In case of the shear flow, the domains can be deformed, ruptured, and aggregated due to the competition of flow and interfacial tension. That is why we proposed a phenomenological scheme to take into account such effects previously[1]. Three different characteristics of the interfaces have been successfully demonstrated as a conjunction with theoretical parameters and excess properties due to the interfaces.
Keywords: Immiscible polymer blends, Drop morphology, Complex Interface, Steady shear viscosity, Normal stress difference

1. Theory

First of all, let us briefly explain the constitutive equation proposed previously. If two-phase system with complex interfaces is shown in Fig. 1, then the total stress tensor can be expressed as a sum of three terms:

$$\frac{\eta_b}{\eta_m} = 1 + \frac{6}{10}\frac{(\eta_i - \eta_m)}{(\eta_i + \eta_m)}\phi + \frac{\sigma_{ex}}{\sigma_m} \qquad (1)$$

$$\frac{N_b}{N_m} = 1 + \frac{6}{10}\frac{(N_i - N_m)}{(N_i + N_m)}\phi + \frac{N_{ex}}{N_m} \qquad (2)$$

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where η, σ, N and ϕ is the viscosity, shear stress, normal stress difference and volume fraction of inclusion phase, respectively. The subscript b,m,i and ex indicate the blend,matrix,inclusion phase and excess property due to the interfaces, respectively. First term is the matrix contribution and second is due to the difference of properties between matrix and inclusion. Therefore, if we deal with polymer blends with the same rheological properties, then it will vanish. However, when we deal with polymer blends with different properties, it is very important to choose a proper mixing rule in the first place. The linear mixing rule has been adopted by several authors[2,3], but there is no rigorous logical background on that. Instead, we proposed a different mixing rule in the previous communication[1] which

can provide us a proper limit in case of dilute concentration of inclusions. The details can be found in the previous paper. However, in the previous communication, only equation (1) was proposed, but here we assume the similar mixing rule for the first normal stress difference as equation (2) as well without any further investigations in order to consider the materials with normal stress difference. However, the interfacial contribution on the normal stress difference is so dominated in the range of experiment, first two terms in equation (2) can be neglected in most cases. The third term is the excess shear stress and normal stress difference due to the interfaces that can be separately obtained from the theoretical consideration[1]. In order to do that, it is necessary to solve the time evolution equations for Q and q_{ij}. These are defined in Fig. 1 as the interfacial area per unit volume and the anisotropy of such quantity. They are solely defined as mathematical quantities of the interface. The first question is how to obtain them and whether it is possible to have a steady state solution of such quantities.

If so, they must be results of balance between shear flow deformations and interfacial relaxations. Once we have such quantities, it is necessary to have a relationship between these and excess stress tensors. Here we are going to use the linear theory proposed by Doi and Ohta [4].

$$\sigma_{ex,ij} = -\alpha q_{ij} \qquad (3)$$

with σ_{ex}, the excess shear stress tensor and α, the interfacial tension.

If we apply a 3-dimensional simple shear flow given by the velocity gradient tensor
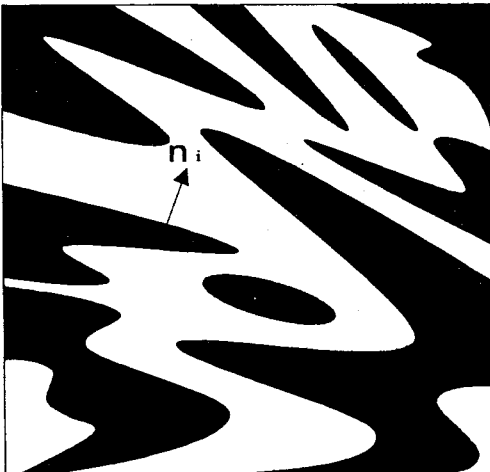
$$q_{ij} = (1/V)\int dS \{n_i n_j - (1/3)\delta_{ij}\} : \text{Anisotropy}$$
$$Q = (1/V)\int dS : \text{Interfacial area}$$

Fig. 1. A concentrated mixture of two immiscible fluids with the complex interfaces.

$$d_{ij} = \dot{\gamma}\delta_{i1}\delta_{j2} \qquad (4)$$

to the previously proposed theory[1] and define

$$\tau = \dot{\gamma}t \qquad (5)$$

$$A = \frac{\alpha\lambda}{\eta_m \dot{\gamma}} \qquad (6)$$

$$P_1 = \frac{q_{11}}{Q}, P_2 = \frac{q_{22}}{Q}, P_3 = \frac{q_{12}}{Q} \qquad (7)$$

$$P^2 = 2(P_1^2 + P_2^2 + P_3^2 + P_1 P_2) \times Q^2 \qquad (8)$$

Here, τ represents the strain and λ is corresponding to the degree of total relaxation as defined by the proposed theory[1].

Then, the time evolution equations for 3-D simple shear flow on 1-2 plane will be:

$$\frac{dQ}{d\tau} = -P_3 Q - A\mu Q^2 - A\nu P^2 \qquad (9)$$

$$\frac{dP_1}{d\tau} = 2P_1 P_3 + \frac{2}{3}P_3 + A(\mu-1)P_1 Q \qquad (10)$$

$$\frac{dP_2}{d\tau} = 2P_2 P_3 - \frac{4}{3}P_3 + A(\mu-1)P_2 Q \qquad (11)$$

$$\frac{dP_3}{d\tau} = 2P_3^2 - P_1 - \frac{1}{3} + A(\mu-1)P_3 Q \qquad (12)$$

Here, μ and ν are model parameters which are related to the coalesence and break-up relaxation in the proposed theory[1]. To combine eq (10) and (11), it is easily seen that P_2 always equal to - 2 times of P_1 at any time. Therefore we do not need to solve for P_2, and just drop the eq (11). At steady state, equations for Q, P_1, and P_3 will be:

$$2P_3 P_1 + \frac{2}{3}P_3 = A(1-\mu)P_1 Q \qquad (13)$$

$$2P_3^2 - P_1 - \frac{1}{3} = A(1-\mu)P_3 Q \qquad (14)$$

$$-P_3 = A\mu Q + 2A\nu(3P_1^2 + P_3^2)Q \qquad (15)$$

By combining (13) and (14), we come up with a relationship between P_1 and P_3.

$$3P_1^2 + P_1 + 2P_3^2 = 0 \qquad (16)$$

To eliminate Q and P_3, we end up with a P_1 equation that looks like:

$$18\nu P_1^3 + (3+3\mu-2\nu)P_1 + 2\mu = 0 \qquad (17)$$

Once we have P_1, it is easy to calculate P_3 and Q and therefore we can evaluate excess stress tensor contribution by equation (3). For simple shear flow, it looks like:

$$\sigma_{ex} = -\alpha q_{12} \tag{18}$$

$$N_{ex} = -3\alpha q_{11} \tag{19}$$

It is possible to obtain an explicit expression for P_1 from equation (17) in principle, but it is rather complicated. Instead, it is rather convenient to introduce the ratio of excess normal stress difference to the excess shear stress such as

$$k = \frac{N_{ex}}{\sigma_{ex}} = \frac{(q_{11} - q_{22})}{q_{12}} = 3\frac{P_1}{P_3} \tag{20}$$

With the aid of this quantity, we can obtain the following expressions:

$$P_1 = \frac{-k^2}{3(k^2+6)} \tag{21}$$

$$\sigma_{ex} = 12\frac{\eta_m \dot{\gamma}}{\lambda(1-\mu)(6+k^2)^2} \tag{22}$$

$$N_{ex} = 12\frac{k\eta_m \dot{\gamma}}{\lambda(1-\mu)(6+k^2)^2} \tag{23}$$

v can be is related to k and μ as followings:

$$v = \frac{(k^2+6)^2[k^2-(k^2+12)\mu]}{8k^2(k^2+3)} \tag{24}$$

If we put these two quantities into eq (1) and (2), then the shear properties of blends are established like (25) and (26).

$$\frac{\eta_b}{\eta_m} = 1 + \frac{6}{10}\frac{(\eta_i - \eta_m)}{(\eta_i + \eta_m)}\phi + \frac{12}{\lambda(1-\mu)(6+k^2)^2} \tag{25}$$

$$\frac{N_b}{N_m} = 1 + \frac{6}{10}\frac{(N_i - N_m)}{(N_i + N_m)}\phi + \frac{12\sigma_m k}{N_m\lambda(1-\mu)(6+k^2)^2} \tag{26}$$

It is nothing but two parameters fitting with κ, $\lambda(1-\mu)$. The original theory is dealing with three different kinds of phenomenological interfacial relaxations that can be related with λ, μ, and v. Here we only have two parameters and these two are enough to describe the interfacial relaxations. In fact, these two quantities will be used to qualify the characteristics of the interfaces later. Mathematically, we can put $\mu=0$ and it does not make any qualitative difference in the excess rheological properties. If we recall the physical meaning of three dimensionless parameters concerning the interfacial relaxation from the previous paper [1], λ comes from the total intensity of such interfacial relaxation and μ is from the contribution of size relaxation and v is from the contribution of break-up and shape relaxation. Therefore near steady state, the average size of the domains is almost fixed so that the size relaxation term μ does not make any influence any more. Therefore, in order to explain the steady properties, μ and λ are not enough but κ and $\lambda(1-\mu)$ should be included in the first place. It is a fun-

damental shortage of the original Doi and Ohta model[4] to neglect v term(or κ term) in the first place. Average domain size so-called 'surface to volume equivalent diameter', d can be predicted if we simply assume the quasi-spherical domains, but in the range of high shear rates, it does not have a significant meaning since they are usually deformed a lot from the spherical shape. The average domain size, d is represented by the following expression.

$$d = \frac{\phi k(k^2+6)}{2}\frac{\lambda(1-\mu)\alpha}{\eta_m \dot{\gamma}} \tag{27}$$

One more thing which is worthy to be mentioned is that in order to have a proper steady state solution, v should be larger than 1.5 or κ should be positive, and we can put $\mu=0$. It means that even we neglect coalescence for some cases, it is impossible to ignore the break-up of inclusions in the first place.

We have also tried the other type of closure approximation and corresponding nonlinear stress with q_{ij} as suggested by M. Grmela and A. Ait-Kadi[5] in order to satisfy the requirement of the compatibility of dynamics with thermodynamics. No qualitative difference, however, was found as seen in the Appendix A.

2. Comparison with experiments

Experimental data was obtained for Polyacrylic acid solution (hereafter called as PAAS) and Polydimethylsiloxane (hereafter PDMS) blend with 20/80, 50/50 and 80/20 compositions. The experimental procedure and the detailed data will be reported in the separate communication[6] and here, we would like to compare them with the theoretical expressions obtained above. The rheological datas of pure component are shown in the Fig. 2. The parameters that have been used for the fitting are tabulated in Table 1. For simplicity we put $\mu=0$ then v are 1.502,
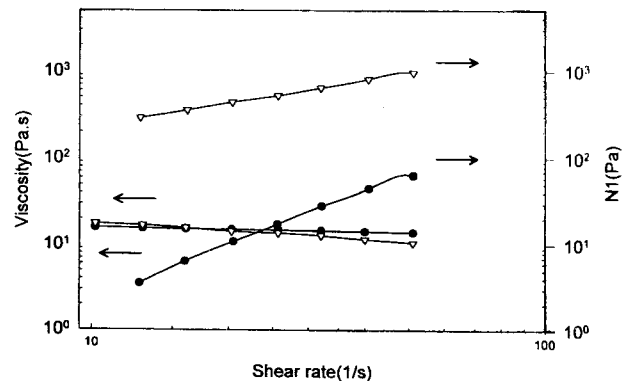

Fig. 2. Rheological data of pure component($\bullet$; PDMS, ∇; PAAS).

Table 1. Model parameters of PAAS/PDMS blend for data fitting

Blend	20PAAS/80PDMS	50PAAS/50PDMS	80PAAS/20PDMS
σ_{ex}(Pa)	77.1	393.2	109
N_{ex}(Pa)	34.7	377.5	453.3
κ	0.45	0.96	4.16
$\dot{\gamma}$(1/sec)	12.6	12.6	12.6
η_m(Pa.s)	15.7	15.7	16.8
N_m(Pa)	3.5	3.5	293
matrix	PDMS	PDMS	PAAS
λ(1-μ)	0.801	0.126	0.0433
α(mN/m)	55.2	55.2	55.2
ν	7.51(0.2-12.2μ)	1.64(0.93-12.9μ)	0.193(17.3-29.3μ)

1.525, and 3.34 respectively.

2.1 20PAAS/80PDMS-Simple Interface

We compared the experimental data with model prediction of 20PAAS/80PDMS in Fig. 3. This blend exhibits a kind of weak contribution of interfaces not only to shear stress but also to the normal stress difference. It may be classified as a simple interface case. The reason why we have very weak contribution on shear stress is that we started from the very similar viscosity materials with a relatively high relaxation of the interface. It is confirmed by O(1) value of λ shown in the Table 1. The normal stress differences are not too high compared with the con- tribution of pure components but a little underestimated. One of the possible reason for the discrepancy is that the original difference in normal stress differences of pure components is too high.

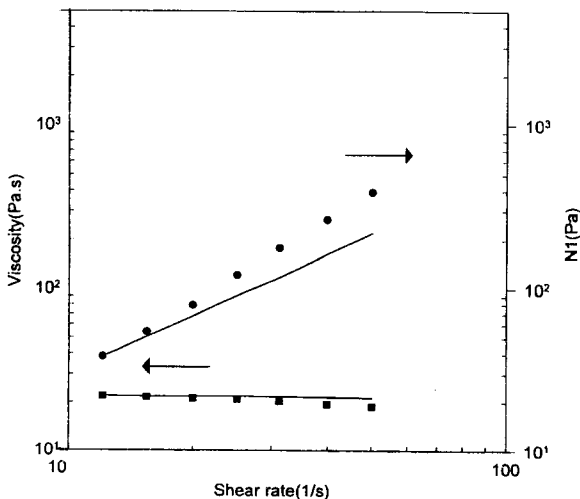### 2.2 50PAAS/50PDMS-Viscous and Elastic Interface

Fig. 3. Comparison experimental data with model prediction of 20PAAS/80PDMS(■; experimental viscosity, ●;ex- perimenta first normal stress difference ─────; theoret- ical prediction).
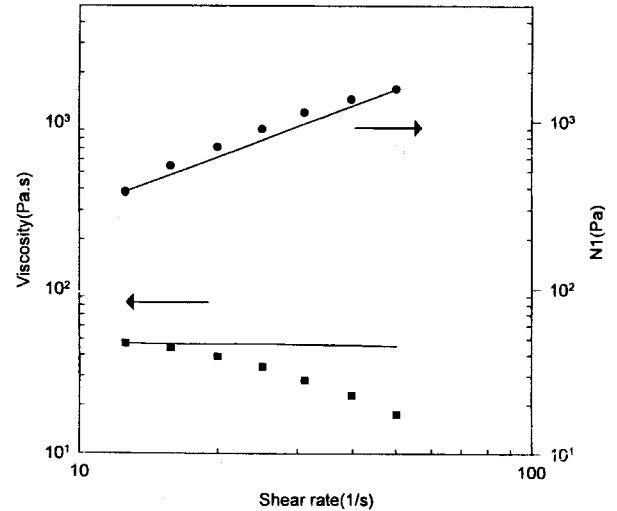

Fig. 4. Comparison experimental data with model prediction of 50PAAS/50PDMS(■; experimental viscosity, ●;ex- perimenta first normal stress difference ────; theoret- ical prediction).

We compared the experimental data with model prediction of 50PAAS/50PDMS in Fig. 4. This blend shows a kind of strong contribution of interface not only to shear stress but also to the normal stress difference. The reason why we have very strong contribution on shear stress is that even though we start from very similar viscosity materials, a relatively weak relaxation of the interfaces play a role. It is confirmed by lower vale of λ shown in the table. Thus it gives us very complex interfaces that contributes not only to the shear stress but also to the nor- mal stress difference. The normal stress differences are very high compared with the contribution of pure components even though it is a little underestimated. One of the possible reason is the original difference in normal stress differences of pure components is too high. Therefore we can call it a viscoelastic interface.

2.3 80PAAS/20PDMS --Purely Elastic Interface

We compared the experimental data with model prediction of 80PAAS/20PDMS in Fig. 5. This blend has a kind of weak contribution of interface to shear stress but very strong to the normal stress difference. The reason why we have very weak contribution on shear stress is that we start from very similar viscosity materials. The normal stress differences of blends are too high compared with those of pure components and a little overestimated. The possible reason is that a relatively low relaxation of the interfaces contributes a lot that is con- firmed by λ value shown in the table. The original difference in normal stress differences of pure components has some con- tribution, too. So it can be classified as a purely elastic in-
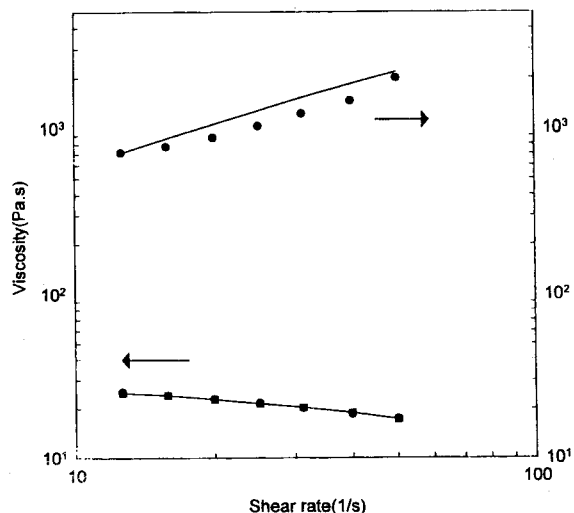
Fig. 5. Comparison experimental data with model prediction of 80PAAS/20PDMS(■ ; experimental viscosity, ● ;experimenta first normal stress difference ——————; theoretical prediction).

terface case.

2.4 Other Experimental Data

PDMS/PB blends studied by Takahashi et. al. [2,7] can be interpreted via same parameters and the results are summarized in Table 2. We can clearly see the differences in the properties of interface contribution on the rheological properties in blends. As seen in the PDMS90/PB10 blends, O(1) value of $\lambda(1-\mu)$ means a simple interface. Other four cases can be classified as viscous and elastic interfaces with relatively weak relaxations of interfaces. Same calculation for Vinckier data for PDMS/PIB blends can be considered along the same line and the results are in Table 3.[3]. Here we can notice that 9:1 and 1:9 blends exhibit the simple interface characteristics but 7:3 and 3:7 blends are the typical cases for the purely elastic and viscous-elastic interfaces, respectively. All 13 blends can be shown in Fig. 6 as k vs $\lambda(1-\mu)$. This plot can be used for the determination of interfacial charac-

Table 2. Model parameters for other system; blend1(PDMS : PB), blend2(PDMS : PI)   (Evaluated at $\dot\gamma$=1/sec)

Blend	blend1(9:1)	blend1(7:3)	blend1(5:5)	blend1(7:3)	blend1(5:5)
σ_{ex}(Pa)	21.5	43.5	102	34.7	32
σ_m(Pa)	107	107	97.8	97.8	60
matrix	PDMS	PDMS	PB	PB	PI
N_{ex}(Pa)	24.9	104	198	72.6	61.5
N_m(Pa)	2.24	2.24	0.35	0.35	0.08
κ	1.16	2.39	1.94	2.09	1.92
$\lambda(1-\mu)$	1.06	0.207	0.109	0.283	0.24
Interface Properties	simple	elastic viscous	elastic viscous	elastic viscous	elastic viscous

Table 3. Model parameters for PDMS:PIB system   (Evaluated at $\dot\gamma$=1/sec)

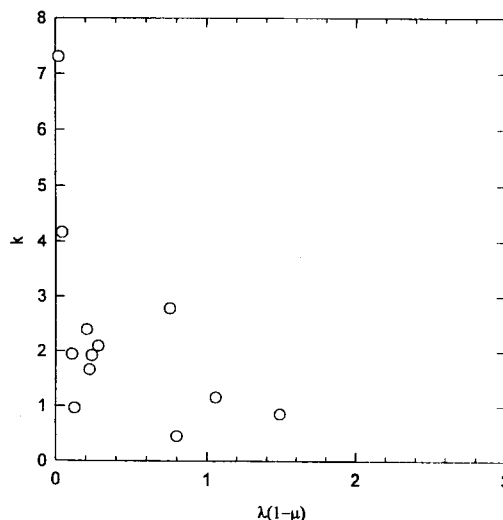| Blend	90/10	70/30	30/70	10/90
σ_{ex}(Pa)	16.8	31.3	60.3	15.4
σ_m(Pa)	200	200	86.5	86.5
matrix	PDMS	PDMS	PIB	PIB
N_{ex}(Pa)	46.8	229	99.8	13.2
N_m(Pa)	10	10	0.133	0.133
κ	2.78	7.31	1.66	0.854
$\lambda(1-\mu)$	0.758	0.022	0.225	1.49
Interface Properties	simple	elastic	elastic viscous	elastic

Fig. 6. Map of model parameters between κ and $\lambda(1-\mu)$.

teristics.

3. Conclusions

Three different kinds of interfacial contributions on the shear stress and normal stress difference of polymer blend are well explained along the line of the previously proposed theory[1]. First of all, very simple analytic expressions and the method of obtaining the relevant parameters are demonstrated to be easily compared with experiments. Three different characteristics can be revealed by the estimation of the phenomenological values from experimental data. Specifically, 20PAAS/80PDMS blend has a weak viscous and elastic interface classified as a simple interface, but 50PAAS/50PDMS has a viscous and elastic interface. 20PDMS/80PAAS shows a purely elastic interface. Nonlinear theory for the interfacial contribution was tried but no qualitative difference was observed. It can be expanded to the case of extensional flow and for the reference we just put them in Appendix B. for further comparison with experiments since we do not have any experimental data right now.

Acknowledgments

Symbols

η_i : Viscosity of inclusion phase

η_m : Viscosity of matrix phase

η_b : Viscosity of blend

N_i : First normal stress difference of inclusion phase

N_m : First normal stress difference of matrix phase

N_b : First normal stress difference of blend

ϕ : Volume fraction

σ : Shear stress

σ_{ex} : Excess shear stress due to interface

N_{ex} : Excess first normal stress difference due to interface

$\dot{\gamma}$: Shear rate

τ : Shear strain

α : Interfacial tension

κ : N_{ex}/σ_{ex}

λ : Degree of total relaxation

μ : Coalescence relaxation

ν : Break-up relaxation

d : Drop size

$\dot{\varepsilon}$: Extensional rate

η_E : Extensional viscosity

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Appendix A

In order to accommodate the suggestion of M. Grmela, the extra stress tensor due to the interface will be calculated by

$$\sigma_{ij} = -\alpha q_{ij} - \frac{6\alpha}{Q} q_{jk}\, q_{ki} + \frac{6\alpha}{Q^2} q_{lm}\, q_{lm}\, q_{ji} \tag{A1}$$

In this case, we should use a different closure for 4th order tensor so that the governing equations for q_{ij} are slightly different from equations (10), (11) and (12)

$$\frac{dP_1}{d\tau} = 3P_1 P_3 + \frac{2}{3}P_3 + A(\mu - 1)P_1 Q \tag{A2}$$

$$\frac{dP_2}{d\tau} = 3P_2 P_3 - \frac{4}{3}P_3 + A(\mu - 1)P_2 Q \tag{A3}$$

$$\frac{dP_3}{d\tau} = 3P_3^2 - P_1 - \frac{1}{3} + A(\mu - 1)P_3 Q \tag{A4}$$

It is possible to define the ratio of excess normal stress to the excess shear stress as before and final results will be given by

$$\frac{\eta_b}{\eta_m} = 1 + \frac{6}{10}\frac{(\eta_i - \eta_m)}{(\eta_i + \eta_m)}\phi + \frac{12}{\lambda(1-\mu)}\frac{(36 + 12\kappa^2 - \kappa^4)}{(6 + \kappa^2)^4} \tag{A5}$$

$$\frac{N_b}{N_m} = 1 + \frac{6}{10}\frac{(N_i - N_m)}{(N_i + N_m)}\phi + \frac{12\sigma_m}{N_m\lambda(1-\mu)}\frac{\kappa(36 + 12\kappa^2 - \kappa^4)}{(6 + \kappa^2)^4} \tag{A6}$$

The surface to volume equivalent diameter can be expressed as

$$d = \frac{6\phi\kappa(\kappa^2 + 6)}{(12 - \kappa^2)}\frac{\lambda(1 - \mu)\alpha}{\eta_m\dot{\gamma}} \tag{A7}$$

As you see, there is no qualitative difference noticed except some quantitative change depending on the ratio κ.

Appendix B

Three-dimensional extensional flow given by $d_{ij} = \varepsilon(\delta_{i1}\delta_{j1} - 1/2\,\delta_{i2}\delta_{j2} - 1/2\delta_{i3}\delta_{j3})$ is applied to the immiscible polymer blend, then the following equations can be easily obtained after defining a few parameters and variables as before.

$$A = \frac{\alpha\lambda}{\eta_m\dot{\varepsilon}} \tag{B1}$$

$$\tau = \dot{\varepsilon}t \tag{B2}$$

$$q = q_{11}, \quad p = \frac{q}{Q} \tag{B3}$$

$$\frac{dQ}{d\tau} = -\frac{3}{2}q - A\mu Q^2 - \frac{3}{2}A\nu q^2 \tag{B4}$$

$$\frac{dq}{d\tau} = -q - \frac{2}{3}Q + \frac{3}{2}\frac{q^2}{Q} - Aq^2 - \frac{3}{2}Av\frac{q^3}{Q} \tag{B5}$$

Here, $\dot{\varepsilon}$ represents the extensional rate

It is pretty straightforward to see that $q_{22} = q_{33} = -\frac{1}{2}q_{11}$. By combining (B4) and (B5) into p - equation will give us

$$27vp^4 - 9vp^3 + (9 + 9\mu - 6v)p^2 - 6\mu p - 4\mu = 0 \tag{B6}$$

Once p is obtained, Q and q can be given as

$$Q = \frac{-3p(2\mu + 3vp^2)}{A}, \quad q = pQ = \frac{3p^2(2\mu + 3vp^2)}{A} \tag{B7}$$

Therefore,

$$\frac{\eta_E}{3\eta_m} = 1 + \frac{6}{10}\frac{(\eta_i - \eta_m)}{(\eta_i + \eta_m)}\phi + \frac{1}{2v\lambda(1 + 2\frac{\mu}{3vp^2})} \tag{B8}$$

Here, $\mu = 0$ will give us a simple excess extensional viscosity with one combined parameter $v\lambda$. In that case, the domain size of inclusion may be

$$d = \frac{4v(2v - 3)}{[v + (9v^2 - 12v)^{\frac{1}{2}}]}\frac{\alpha\lambda}{\eta_m\dot{\varepsilon}} \tag{B9}$$

It will be very nice to be compared with experimental data if available.