

Effect of morphology on the brittle ductile transition of polymer blends: 1. A new equation for correlating morphological parameters

Z. H. Liu*, X. D. Zhang†, X. G. Zhu, Z. N. Qi and F. S. Wang

State Key Laboratory of Engineering Plastics, Institute of Chemistry, Academia Sinica, Beijing 100080, P.R. China

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The relationships among morphological parameters of binary polymer blends have been studied. A new equation for correlating morphological parameters: particle size (d), particle size distribution (σ), particle volume fraction (ϕ), and matrix ligament thickness ($T(d, \sigma, \phi)$) is derived. The equation is generally applicable to the binary polymer blends with the log-normal distribution of particle size and the configuration of well-dispersed particles in matrix. The effects of σ and particle configuration on $T(d, \sigma, \phi)$ are discussed. The new equation predicts that $T(d, \sigma, \phi)$ increases with increasing d and σ , and with reducing ϕ . The effect of σ is found to be influenced by ϕ . The higher the ϕ is, the more significant the effect of σ on $T(d, \sigma, \phi)$ is. The new equation is applied to the poly(vinyl chloride)/nitrile rubber, polypropylene (PP)/EPDM, and PP/EVA blends. The theoretical predictions agree well with the experimental results. The equations neglecting or underestimating the effect of σ on $T(d, \sigma, \phi)$, however, cause great errors. Compared with the effect of σ , the effect of particle configuration of cubic lattice assumed in deriving the new equation is much less important in the above blends. Therefore, σ is also an important morphological parameter. © 1997 Elsevier Science Ltd.

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INTRODUCTION

Toughness is one of the most important properties of polymeric materials. The impact toughness of polymer blends has been found to be influenced by many factors¹, e.g. morphology, interfacial adhesion, the physical and mechanical properties of each component, etc., even for a given mode, frequency and temperature of deformation. These factors are, however, usually interrelated. It is necessary to establish a quantitative method to correlate these factors so that the effect of each factor on the impact toughness can be understood. The effect of morphology should be first recognized since it is more basic than others. So far, there have been few papers concerning the relationships among morphological parameters, and there has been no reliable equation for correlating morphological parameters.

The particles of dispersed phase are usually distributed randomly in a polymer matrix. This morphology is very typical in the binary polymer blends, and thus is particularly interesting here. In this case, the morphological parameters include particle size (d), particle size distribution (σ), particle volume fraction (ϕ), configuration of particles in matrix and matrix ligament thickness (surface to surface interparticle distance) (T). The influences of these parameters on the impact toughness

of polymer blends also coexist, and thus their effects should be separated.

It has been well established that the particles of dispersed phase act as the stress concentrators to initiate shear bands or crazes². T has been shown to be an important factor dominating the interacting stress fields between neighbouring particles³. Wu^{4,5} proposed the criterion of critical matrix ligament thickness (T_c) for rubber toughening in which the effects of d and ϕ have been formulated as the effect of T using an idealized equation to relate T to d and ϕ . According to the criterion, the effects of d and ϕ have been suggested to be separated. For a given d , the impact toughness of polymer blends increases with ϕ . But it decreases with increasing d for a given ϕ . So far, the effect of σ on the impact toughness has not been well understood though an unfavourable effect has been expected⁵.

T is also an important parameter relating the effects of other factors, e.g. the chain structure of polymers^{6,7}, interfacial adhesion⁸, the configuration of particles in the matrix⁹ and the modulus of matrix⁷, on the impact toughness of polymer blends. To better understand the effects of these factors, a more approximate equation for calculating T is also needed.

This paper is the first of our serial ones. In this work, a new equation for relating T to d , σ and ϕ is derived. The T values for poly(vinyl chloride) (PVC)/nitrile rubber (NBR), polypropylene (PP)/ethylene-propylene diene monomer rubber (EPDM) and PP/ethylene-vinylacetate

* To whom correspondence should be addressed

† Present address: PO Box 13, Taiyuan 030051, Shanxi, P.R. China

copolymer (EVA) blends are measured. A comparison between the experimental results and the theoretical predictions is made to find out the relationships among morphological parameters. The effect of σ on T is discussed.

EVALUATION OF MATRIX LIGAMENT THICKNESS

The definition of particle size distribution

For the majority of binary polymer blends, the particle size distribution of dispersed phase has been found to fit log-normal distribution^{5,8-10}. So, the frequency $f(d_i)$ of a particle size d_i is defined as¹¹

$$f(d_i) = \frac{1}{\sqrt{2\pi} \ln \sigma} \exp \left[\frac{-(\ln d_i - \ln d)^2}{2 \ln^2 \sigma} \right] \quad (1)$$

where d and σ are given by¹¹

$$\ln d = \frac{\sum_{i=1}^N n_i \ln d_i}{\sum_{i=1}^N n_i} \quad (2)$$

and

$$\ln \sigma = \sqrt{\frac{\sum_{i=1}^N n_i (\ln d_i - \ln d)^2}{\sum_{i=1}^N n_i}} \quad (3)$$

In the case of monodispersity, $\sigma = 1$; and $\sigma > 1$ for polydispersity.

The monodisperse system

In order to relate T to d and ϕ , the configuration of dispersed particles in matrix must be considered. As a first approximation, assuming particles occupying cubic lattices, Wu⁴ obtained

$$T(d, \phi) = d [(\pi/6\phi)^{1/3} - 1] \quad (4)$$

The polydisperse system

The effect of σ on the number of particles per unit volume ($n_V(\sigma)$). The relationship between the number of particles per unit volume in the polydisperse sample ($n_V(\sigma)$) and the number of particles per unit volume in the monodisperse sample ($n_V(1)$) is key in relating σ with T , and should be found. In a well-dispersed blend, we divide the total sample into a number of unit volumes. Each of them has the same morphological parameters as the total sample. In other words, they have the same d , σ , ϕ , T and configuration of particles in the matrix. We consider two different unit volumes which have the same d , ϕ , and particle configuration, but different σ and T . Moreover, one unit volume has the uniform sized particles while another has the heterogeneous sized particles. The ratio $n_V(\sigma)/n_V(1)$ is derived below.

From the limit of the same particle volume fraction,

we have

$$\sum_{i=1}^N n_{V,i} \left(\frac{\pi}{6} d_i^3 \right) = \frac{\pi}{6} d^3 n_V(1) \quad (5)$$

where $n_{V,i}$ is the number of particles with diameter d_i . Equation (5) can be rewritten as

$$\sum_{i=1}^N n_{V,i} d_i^3 = d^3 n_V(1) \quad (6)$$

Since the particle size obeys log-normal distribution, the left-hand side of equation (6) gives¹¹

$$\sum_{i=1}^N n_{V,i} d_i^3 = n_V(\sigma) \exp(3 \ln d + 4.5 \ln^2 \sigma) \quad (7)$$

Combining equations (6) and (7), we have

$$\frac{n_V(\sigma)}{n_V(1)} = \exp(-4.5 \ln^2 \sigma) \quad (8)$$

The ratio $n_V(\sigma)/n_V(1) \leq 1$ because of $\sigma \geq 1$. It should be pointed out that there is no limit of particle configuration to equation (8).

The evaluation of matrix ligament thickness. In order to obtain a simple equation for calculating the average matrix ligament thickness, we assume that the particles in matrix occupy the cubic lattices. The number ($n_L(\sigma)$) of particles within the unit length is given by

$$n_L(\sigma) = \sqrt[3]{n_V(\sigma)} \quad (9)$$

In the case of $\sigma = 1$, equation (9) becomes

$$n_L(1) = \sqrt[3]{n_V(1)} \quad (10)$$

The $T(d, \sigma, \phi)$ can be expressed as

$$T(d, \sigma, \phi) = \frac{l - \sum_{i=1}^{N_L} n_{L,i} d_i}{n_L(\sigma)} \quad (11)$$

where $n_{L,i}$ is the number of particles with size d_i within the unit length. Because the particle size distribution fits log-normal distribution, we have¹¹

$$\sum_{i=1}^{N_L} n_{L,i} d_i = n_L(\sigma) \exp(\ln d + 0.5 \ln^2 \sigma) \quad (12)$$

Insertion of equations (9) and (12) into equation (11) yields

$$T(d, \sigma, \phi) = \frac{l - \sqrt[3]{n_V(\sigma)} \exp(\ln d + 0.5 \ln^2 \sigma)}{\sqrt[3]{n_V(\sigma)}} \quad (13)$$

When $\sigma = 1$, equation (13) becomes

$$T(d, \phi) = \frac{l - \sqrt[3]{n_V(1)} d}{\sqrt[3]{n_V(1)}} \quad (14)$$

Combination of equations (8), (9), (10), (13) and (14) gives

$$T(d, \sigma, \phi) = [T(d, \phi) + d] \exp(1.5 \ln^2 \sigma) - \exp(\ln d + 0.5 \ln^2 \sigma) \quad (15)$$

Inserting equation (4) in equation (15), we have the final result

$$T(d, \sigma, \phi) = d[(\pi/6\phi)^{1/3} \exp(1.5 \ln^2 \sigma) - \exp(0.5 \ln^2 \sigma)] \quad (16)$$

where d , σ , and ϕ can be measured experimentally. So, $T(d, \sigma, \phi)$ can be calculated from the experimental results.

EXPERIMENTAL

Materials

PVC (S-1000 from Qilu Petrochem Company, P.R. China) was a commercial product with the number-average molecular weight $M_n = 62\,500$. NBR was a commercial rubber from Lanzhou Chemical Company, P.R. China. The acrylonitrile level was reported by the manufacturer to be 18% by weight. PP was a commercial polymer (2401 from Yanshan Petrochem Company, P.R. China). EPDM (4045) was a commercial elastomer (from Mitsui Petrochemical Industries Ltd., Japan). EVA (H2020) was also a commercial elastomer (from Polyolefin Company Pte. Ltd., Singapore).

Blend preparation

PVC was first mixed in a mixer with the processing additives, 0.4 phr lubricator, 3 phr stabilizer and 5 phr plasticizer, and was then mixed with NBR on a laboratory two-roll mill for 6 min at 160°C. PP was blended respectively with EPDM and EVA on the mill for 8 min at 155°C. These milled sheets were stacked and compression-moulded for 10 min at 160°C, then cooled slowly down to the room temperature to give 4 mm thick plates. The bars for the morphology observations were cut from these plates.

Morphological observations

The samples were cryofractured. For the PVC/NBR blends, the fracture surfaces were etched in the oxidizer composed of 100 ml H_2SO_4 , 30 ml H_3PO_4 , 30 ml H_2O and 3 g $K_2Cr_2O_7$ for 5 min at 30°C to remove the NBR phase. For the PP/EVA blends, the fracture surfaces were etched with hot toluene of 50°C to remove the EVA phase. For the PP/EPDM blends, the fracture surfaces were etched with *n*-heptane at room temperature to remove the EPDM phase. Then, the etched surfaces were coated with Au. The morphologies were observed in a Hitach S-530 SEM scanning electron microscope.

Analysis of morphological parameters

The SEM photographs were used to analyse the particle size, particle size distribution and matrix ligament thickness using a computer image analyser. The numbers of particles and of matrix ligament thickness for each sample were between 300–400.

RESULTS AND DISCUSSION

Comparison between the experimental results and the theoretical predictions

The d and T values for PVC/NBR, PP/EPDM and PP/EVA blends were measured using SEM photographs and computer image analyser. The accumulative number densities of particles or ligaments are plotted against d or T in the probability-log coordinate. The average values

of d and T are those at the probability of 50%. The σ is the ratio of the particle size at the probability of 84% to that at 50%.

Figures 1a–c show that the typical particle sizes for the PVC/NBR, PP/EPDM and PP/EVA blends fit log-normal distribution. For the PVC/NBR blend with $\phi = 0.136$ (see Figure 1a), $d = 0.073 \mu\text{m}$ and $\sigma = 1.58$. For the PP/EPDM blend with $\phi = 0.243$ (see Figure 1b), $d = 0.51 \mu\text{m}$ and $\sigma = 2.31$. For the PP/EVA blend with $\phi = 0.243$ (see Figure 1c), $d = 0.32 \mu\text{m}$ and $\sigma = 1.59$. The typical σ values for PVC/NBR blends are between 1.4 and 1.6. But the σ values for PP/EPDM blends range from 1.6 to 2.4, and are much larger than those for PVC/NBR blends. It is clear that the σ values vary dramatically from one blend system to another.

Figures 2a–c display that the matrix ligament thicknesses of above three samples also obey log-normal distribution. The T values for them are 0.08, 1.22 and $0.25 \mu\text{m}$, respectively. Above experimental results are listed in Table 1.

In order to estimate the effect of σ on $T(d, \sigma, \phi)$, Wu⁵ proposed

$$T(d, \sigma, \phi) = d[(\pi/6\phi)^{1/3} - 1] \exp(\ln^2 \sigma) \quad (17)$$

According to the d , σ and ϕ values listed in Table 1, the appropriate T values for the above samples are also calculated from equations (4), (16) and (17). The corresponding T s are T^* , T_1 and T_2 . The calculated and experimental results are listed in Table 1 too. For the PVC/NBR blend, they are 0.041, 0.076 and $0.051 \mu\text{m}$, respectively. Compared with the experimental result of $0.080 \mu\text{m}$, the errors of equations (4), (16) and (17) are 49, 5 and 36%, respectively. For the PP/EPDM blend, they are 0.15, 1.16 and $0.30 \mu\text{m}$ predicted from the corresponding equations, and the experimental result is $1.22 \mu\text{m}$. So, the errors of these equations are found to be 88, 5 and 75%. For the PP/EVA blend, the T values calculated from equations (4), (16) and (17) are 0.093, 0.21 and $0.12 \mu\text{m}$. The experimental T is $0.25 \mu\text{m}$. Thus, the corresponding errors are 63, 16 and 52%. Equation (16) derived in this work has the smallest errors (5, 5 and 16%, respectively) for the above blends. Equations (4) and (17), however, have great errors. Equation (4) has the greatest errors (49, 88 and 63%, respectively) for the three samples. A little improvement is made by equation (17) since the corresponding errors are reduced only by 13, 13 and 11% compared with equation (4).

The effect of σ on $T(d, \sigma, \phi)$

Equation (8) derived in this work gives the exact relationship between σ and $n_V(\sigma)$. The effect of σ on $n_V(\sigma)$ can be seen more clearly by plotting $n_V(\sigma)/n_V(1)$ vs. σ at the same d and ϕ , as shown in Figure 3. The ratio $n_V(\sigma)/n_V(1)$ decreases with increasing σ . This trend becomes more evident especially when σ is between 1.2 and 2. It can be expected that at the same d and ϕ , $T(d, \sigma, \phi)$ increases with increasing σ because of the reduction of $n_V(\sigma)$.

From equations (4) and (16), and at the same d and ϕ , the normalized $T(d, \sigma, \phi)$ is

$$\frac{T_1(d, \sigma, \phi)}{T(d, \phi)} = \frac{(\pi/6\phi)^{1/3} \exp(1.5 \ln^2 \sigma) - \exp(0.5 \ln^2 \sigma)}{(\pi/6\phi)^{1/3} - 1} \quad (18)$$

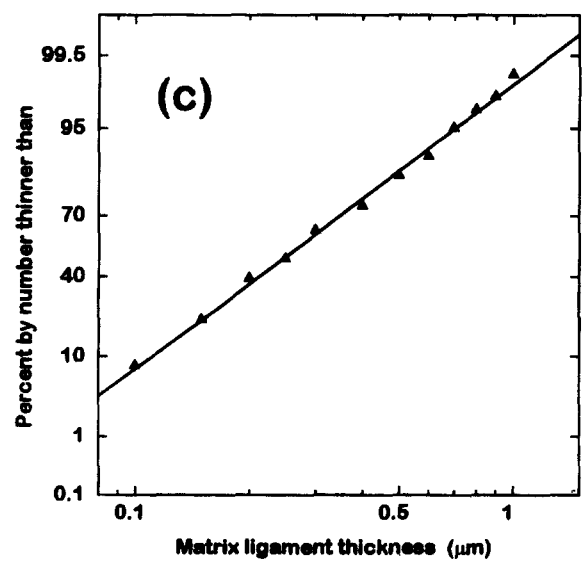
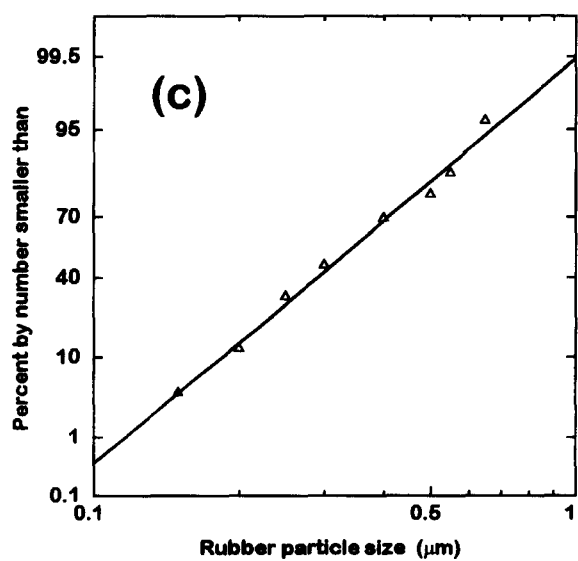
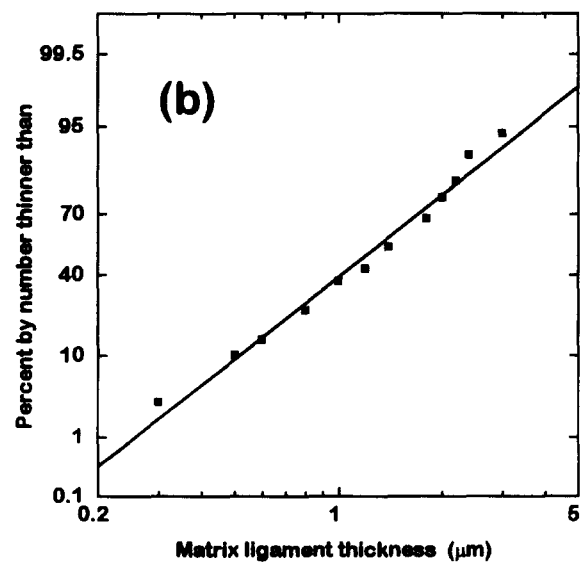
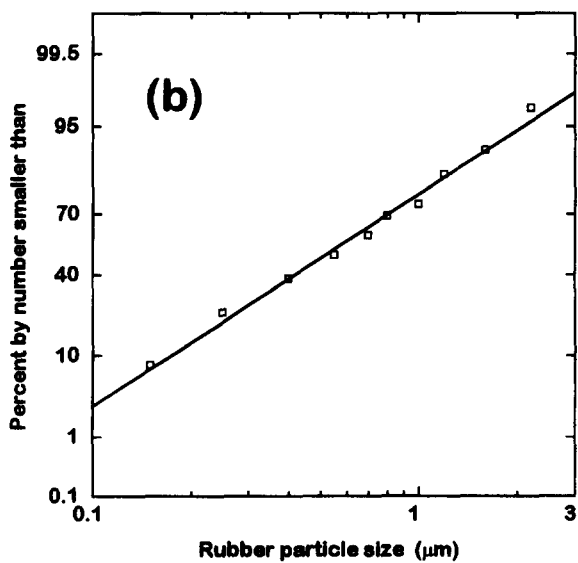
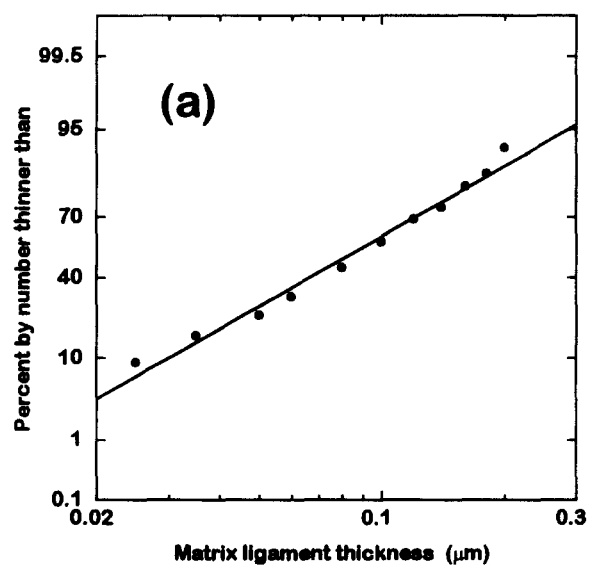
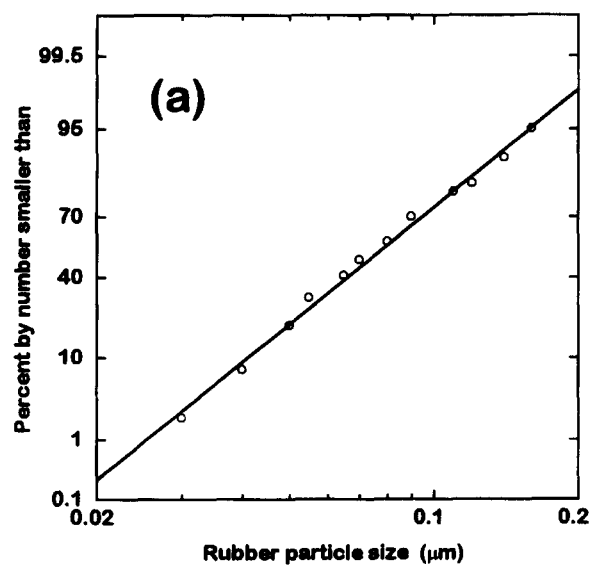


Figure 1 Log-normal distributions of rubber particles in (a) a PVC/NBR blend with $\phi = 0.136$, (b) a PP/EPDM blend with $\phi = 0.243$ and (c) a PP/EVA blend with $\phi = 0.243$

Figure 2 Log-normal distributions of matrix ligaments in (a) the PVC/NBR blend, (b) the PP/EPDM blend and (c) the PP/EVA blend corresponding to the blends as shown in Figure 1

The effect of σ on $T(d, \sigma, \phi)$ can be predicted using equation (18). For the purpose of comparison, the equation for evaluating the effect of σ on $T(d, \sigma, \phi)/T(d, \phi)$ reported in the literature is cited here. Under the same conditions as assumed in deriving equation (18), Wu⁶ suggested

$$\frac{T_2(d, \sigma, \phi)}{T(d, \phi)} = \exp(\ln^2 \sigma) \quad (19)$$

Figure 4 shows the effect of σ on $T(d, \sigma, \phi)/T(d, \phi)$. Equation (18) predicts that the effect of σ on $T(d, \sigma, \phi)/T(d, \phi)$ is controlled by ϕ . $T(d, \sigma, \phi)/T(d, \phi)$ increases with increasing σ at the same ϕ . Moreover, it increases more quickly in the case of a higher ϕ . The predictions by equation (18) are consistent with the experimental results. Comparing the PP/EPDM blend with the PP/EVA blend, one may find that both have the same $\phi (= 0.243)$, but the effects of σ on $T(d, \sigma, \phi)$ for them are quite different. The ratio ($T/T(d, \phi)$) of the experimental T to $T(d, \phi)$ for the PP/EVA blend is 2.7. It is, however, 8 for the PP/EPDM blend due to a larger σ . Thus, at the same ϕ , the larger the σ is, the more significant the effect of σ on $T(d, \sigma, \phi)$ is. The effects of σ on $T(d, \sigma, \phi)$ for the PVC/NBR blend and the PP/EVA

blend are also different though both have the same σ ($\cong 1.58$). The $T/T(d, \phi)$ value for the former is 2. It is 2.7 for the latter, which is slightly larger because of a higher ϕ . So, at the same σ , the higher the ϕ is, the more significant the effect of σ on $T(d, \sigma, \phi)$ is. But equation (19) predicts that the effect of σ on $T(d, \sigma, \phi)/T(d, \phi)$ is independent of ϕ . That is why equations (4) and (17) cause a great error in the case of polydisperse polymer blends.

At least, two experimental error sources can be identified. The resolution of the real average matrix ligament thickness of polymer blends is a problem of three-dimensions. The two-dimensional SEM or TEM photographs, however, are used to measure the average matrix ligament thickness since so far we have had no better method. It is no doubt that this simplification will introduce an error. The error from the two-dimensional measurement is inevitable. There are two types of theoretical errors from the assumptions made in deriving equations. If one of equations (4), (16) and (17) is applied, the configurational error arises from the cubic lattice. Moreover, if equation (4) is used, the error due to neglecting the effect of σ on $T(d, \sigma, \phi)$ exists. Therefore, the error ($e_{eq.(4)}$) of equation (4) between the calculated $T(d, \phi)$ and the experimental T cannot be purely attributed to the neglect of the effect of σ on $T(d, \sigma, \phi)$. The error (e_σ) arising from the neglect of the effect of σ on $T(d, \sigma, \phi)$ may be given by

$$e_\sigma = e_{eq.(4)} - e_{eq.(16)} \quad (20)$$

where $e_{eq.(16)}$ is the error from equation (16).

For the PVC/NBR, PP/EPDM and PP/EVA blends, the corresponding e_σ values are 44, 83 and 47%.

The effect of particle configuration on $T(d, \sigma, \phi)$

The discussion of the effect of particle configuration on $T(d, \sigma, \phi)$ should be based on the roles of rubber particles. In the brittle polymers, such as polystyrene and poly(styrene-co-acrylonitrile), the rubber particles promote crazing in the matrix, whereas in the pseudoductile

Table 1 Comparison of the matrix ligament thickness between the experimental results and the theoretical predictions

Morphological parameters	Blends		
	PVC/NBR	PP/EPDM	PP/EVA
$d, \mu\text{m}$	0.073	0.51	0.32
σ	1.58	2.31	1.59
ϕ	0.136	0.243	0.243
T (experimental), μm	0.080	1.22	0.25
T_1 [by equation (16)], μm	0.076	1.16	0.21
Error ^a , %	5	5	16
T_2 [by equation (17)], μm	0.051	0.30	0.12
Error ^a , %	36	75	52
T^* [by equation (4)], μm	0.041	0.15	0.093
Error ^a , %	49	88	63

^a Note: the error is defined as $|T^C - T| \times 100/T$, where T^C is the calculated value

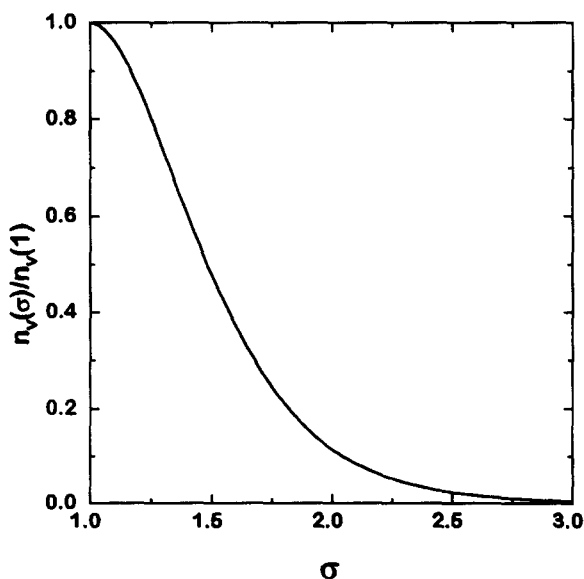


Figure 3 Plot of $n_v(\sigma)/n_v(1)$ vs. σ for rubber particles conforming to log-normal distribution

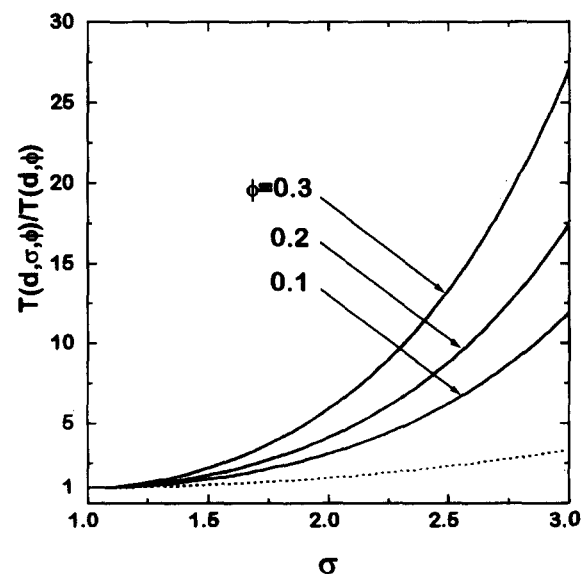


Figure 4 Plot of $T(d, \sigma, \phi)/T(d, \phi)$ vs. σ for rubber particles conforming to log-normal distribution. The solid and dotted lines are calculated from equation (16) derived by this work and (17) reported in the literature, respectively

polymers, such as polycarbonate, polyamide, PP and PVC, shear yielding is usually the major energy absorbing mechanism. The rubber particles act as the stress concentrators to initiate crazes or shear bands. The

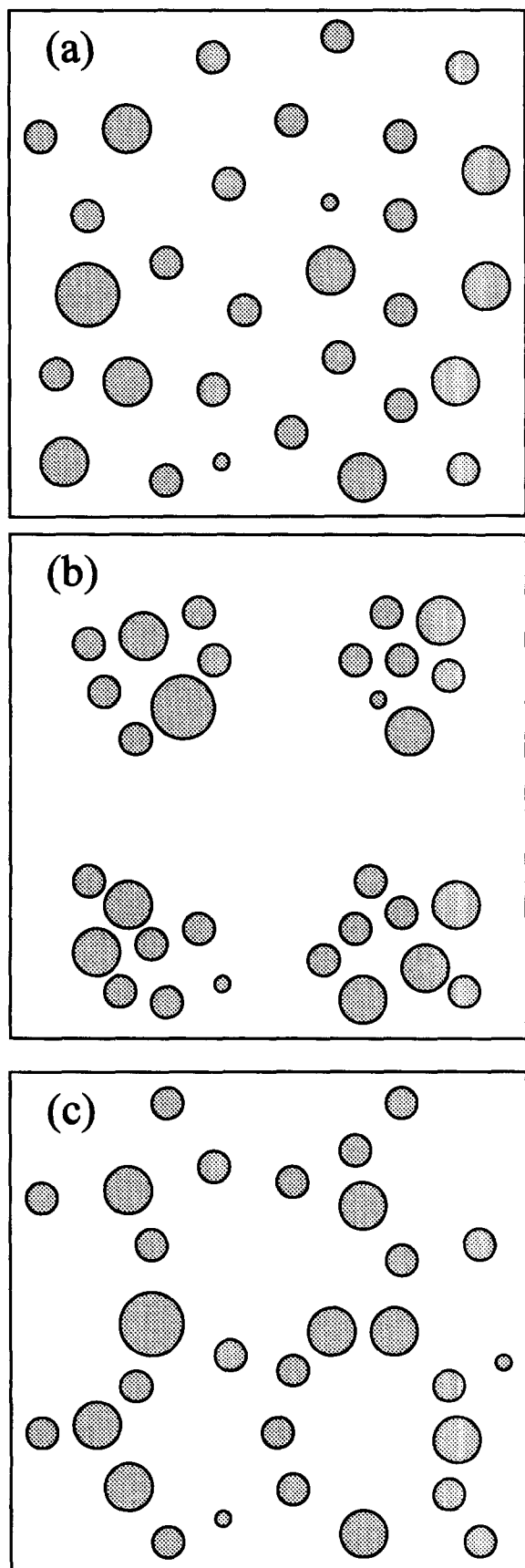


Figure 5 Schematics of particle configuration: (a) well dispersed, (b) flocculated and (c) pseudonetwork

brittle ductile transition of polymer blends is a critical phenomenon which is associated with the connectivity of spherical stress volumes. $T(d, \sigma, \phi)$ is an important factor determining the connectivity. It has been well established by the percolation theory that the connectivity depends on the particle configuration. So, the toughening efficiency is influenced by the particle configuration and $T(d, \sigma, \phi)$.

Figures 5a–c show three types of morphologies. The most widely observed morphology is the well-dispersed particles, as shown in Figure 5a. The ligaments are uniformly distributed, and ligament yielding can propagate. Numerous studies have shown that this configuration is effective in polymer toughening. The new equation (16) well predicts the experimental results though the particle configuration of cubic lattice has been assumed in obtaining the equation. Since equation (16) has included the effect of σ on $T(d, \sigma, \phi)$, the configurational error $e_{\text{configuration}}$ arising from the assumption of cubic lattice can be calculated by

$$e_{\text{configuration}} = e_{\text{eq.(16)}} \quad (21)$$

For the PVC/NBR, PP/EPDM and PP/EVA blends, the appropriate $e_{\text{configuration}}$ values are 5, 5 and 16%. The particles of NBR, EPDM and EVA rubbers are well dispersed in the matrices, but the $e_{\text{configuration}}$ values are different. The change in the $e_{\text{configuration}}$ values may be attributable to the different dispersions. Compared with the e_{σ} values, the $e_{\text{configuration}}$ values are much smaller. In other words, the effect of σ on $T(d, \sigma, \phi)$ is much more significant than that of particle configuration in a well-dispersed blend. Therefore, the random configuration of well-dispersed particles can be simulated with the cubic lattice.

Figure 5b displays the flocculated particles. Flocculation forms clusters of particles. Within a cluster, the ligaments are thin, but the clusters are surrounded by very thick ligaments. Therefore, ligament yielding cannot propagate, the blend is brittle. This particle configuration must be avoided in polymer toughening. In this case, it is expected that equation (16) if applied will result in a great error. PVC/rubber blends can exhibit a much more effective morphology (pseudonetwork structure) in the toughening of PVC than the well-dispersed particles, as shown in Figure 5c. The PVC primary particles force the rubber particles to be scattered in between the PVC primary particles. The fourth paper of this series is focused on the effect of particle configuration on the brittle ductile transition of PVC/NBR blends. We will show that equation (16) cannot be directly used to calculate the average matrix ligament thickness of polymer blends with the pseudonetwork configuration.

CONCLUSIONS

Based on the assumptions of the log-normal distribution of particle size and the particle configuration of cubic lattice, a new equation (16) for correlating morphological parameters, particle size, particle size distribution, volume fraction of particles and matrix ligament thickness, has been derived.

The new equation predicts that the average matrix ligament thickness increases with increasing particle size and particle size distribution, and with decreasing particle volume fraction. It also predicts that there is a great influence of particle size distribution on the average

matrix ligament thickness. This effect is connected with the particle volume fraction. The higher the particle volume fraction is, the more significant the effect of particle size distribution on the average matrix ligament thickness is. Equations (4) and (17) reported in the literature, however, cannot predict this effect.

The experimental results show that the particle sizes of dispersed phases of PVC/NBR, PP/EPDM and PP/EVA blends fit log-normal distribution, and that the matrix ligament thicknesses also obey log-normal distribution in these blends. The values of the average matrix ligament thickness calculated from the new equation are quite near the experimental values. But the realized equation (4) without including the effect of particle size distribution on the average matrix ligament thickness causes the greatest errors. There remain big errors from equation (17) due to underestimation of the effect of particle size distribution.

The assumption of particle configuration of cubic lattice has been made in obtaining the new equation though the rubber particles of the above blends are irregularly dispersed in the matrices. On the other hand, the new equation well predicts the experimental results. The error from the particle size distribution is much larger than that from the configurational assumption of the cubic lattice. Therefore, the particle configuration of random dispersion can be simulated with the cubic lattice in polymer blends with well-dispersed particles. It should be pointed out that the new equation is invalid when the particles of dispersed phase flocculate. The configuration of flocculated particles will greatly reduce the toughening efficiency, and thus is not interesting in polymer toughening.

The experimental results also show that the particle size distribution varies from one blend system to another. The effect of particle size distribution on the average matrix ligament thickness should be considered since the average matrix ligament thickness is an important parameter determining the toughness and toughening behaviour of polymer blends.

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