

Polymer blends with a liquid crystalline polymer dispersed phase

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Abstract

Immiscible polymer blends containing a liquid crystalline polymer dispersed phase can be described by existing blend theories when the dispersed-phase droplets are large relative to the orientation correlation length ("domain size") of the LCP. There does not appear to be an interfacial contribution to the linear viscoelastic properties of the blend from droplets smaller than the correlation length. Polyester blends, where interfacial interactions occur between the LCP and the matrix, exhibit a reduction in viscosity to below the viscosity of either component at low shear rates, where the droplet morphology is spherical. These anomalies cannot be explained in the context of existing theory.

1. Introduction

Liquid crystalline polymers (LCPs) contain rigid backbone units that lead to molecular order in concentrated solutions (*lyotropic LCPs*) and melts (*thermotropic LCPs*), typically of a nematic type. The correlation length for nematic order in polymeric liquid crystals is rarely greater than five microns; when viewed with polarized light, a quiescent nematic melt appears to be made up of "domains," with a high degree of order within each domain but little or no order on a macroscopic scale. The morphology during flow is usually characterized by Onogi and Asada's (1980) "three region flow curve:" At low deformation rates, where the flow is dominated by deformation of the domain structure, the viscosity typically decreases with a power-law dependence of approximately 1/2. At intermediate rates, the domains appear to decrease in size with increasing shear rate, and the viscosity is relatively insensitive to shear rate. At high deformation rates, the domain structure is not evident and the viscosity is again shear thinning; LCPs typically have much lower viscosities at high shear rates than flexible polymers of similar chemical structure and comparable molecular weight. The domain structure reappears upon cessation of shear; time constants for morphological change are much longer than time constants for stress relaxation.

Blends containing small amounts of a thermotropic LCP in a matrix of a flexible engineering thermoplastic are of

technological interest for two reasons: Firstly, by processing in a way that causes the LCP dispersed phase to develop a fibrillar morphology, it may be possible to develop "self-reinforced" composites that exploit the outstanding tensile properties of fibers made from LCPs (see, for example, Weiss *et al.* (1987), Handlos and Baird (1995)). Secondly, it is known that LCPs can act as "flow modifiers" for conventional thermoplastics, effecting substantial reduction in extrusion pressure (Cogswell, 1983); the usual explanation for the effectiveness of LCPs as flow modifiers is that elongated low-viscosity inclusions act as internal "slip surfaces," but there is no theory to support this concept.

Viscosity data for LCP blends at low shear rates are limited, since this is not a regime of technological significance. It is, however, the regime in which comparisons of experiments with theories of blend rheology are most likely to be fruitful. Conventional theories of blends, which have not been formulated to include possible liquid crystallinity in either phase, predict an increase in the zero-shear viscosity of a blend relative to the matrix following the addition of any dispersed phase. For some blends with LCP dispersed phases, however, there is a decrease in the magnitude of the complex viscosity relative to that of the matrix in small-strain oscillatory measurements and in the magnitude of the shear viscosity relative to the matrix at low shear rates; the morphology is expected to be nearly spherical in these experiments, and the concept of an internal slip plane is unlikely to be meaningful (Kim and Denn, 1992; Van Eijndhoven-Rivera *et al.*, 1998). In other LCP blends, the effect of the dispersed phase is considerably reduced relative to that predicted by existing blend theories; in partic-

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ular, the influence of droplets with a size scale of the order of a liquid crystalline domain on the blend rheological properties seems to be negligible (Riise *et al.*, 1999; Lee and Denn, 1999). We review these observations here in the context of existing theories, and identify the major outstanding problems in understanding the behavior of blends with a LCP dispersed phase.

2. Rheological Models

The introduction of a droplet with radius R into a continuous liquid results in a new term with dimensions of stress, the Laplace pressure α/R , where α is the interfacial tension. If the droplet and matrix fluids are both newtonian and inertial effects can be neglected, it follows from dimensional analysis that the time constant τ_D associated with the response of the droplet when subjected to a small deformation is

$$\tau_D = \frac{R\eta_M f(k)}{\alpha} \quad (1)$$

where k is the viscosity ratio, η_D/η_M , and M and D refer to the matrix and dispersed phase, respectively. In the limiting case $\eta_D \gg \eta_M$, $f(k) \rightarrow 1$ (i.e., the droplet response time is independent of the dispersed phase viscosity), while if $\eta_M \ll \eta_D$, $f(k) \rightarrow k$ (the response time is independent of the matrix viscosity). (These limits omit scale factors, of course.) Similarly, droplets will break up in a shear field of intensity $\dot{\gamma}$ and dimensional analysis requires that the steady-state droplet size be

$$R = \frac{\alpha}{\eta_M \dot{\gamma}} g(k) \quad (2)$$

The same limits should apply to $g(k)$ as to $f(k)$, except that there are bounds on the regime over which shear can effect droplet breakup. [The classic work is by Grace (1982). For a review, see Rallison (1984), and for recent work and references, see Grizzuti and Bifulco (1997) and Tsakalos *et al.* (1998)].

The dynamics of a blend with viscoelastic continuous and dispersed phases have been analyzed by Paliarne (1990; 1991), building on earlier analyses for newtonian fluids by Taylor (1932), Oldroyd (1953) and Choi and Schowalter (1975); Paliarne's general result for the characteristic response time of a monodisperse blend is

$$\tau_D = \frac{R\eta_M(19k+16)[2k+3-2\phi(k-1)]}{4\alpha[10(k+1)-2\phi(5k+2)]} \quad (3)$$

where ϕ is the volume fraction of the dispersed phase. It is readily shown that for $\phi \ll 1$ the Paliarne equation interpolates almost linearly between the two limits $R\eta_M/\alpha$ and $R\eta_D/\alpha$ of Eq. 1, where the coefficients in both limits are close to unity. Paliarne further showed that the linear viscoelastic response of the blend is given in terms of the complex modulus $G^* = G' + iG''$ as

$$G^* = G_M^* \frac{1+3\sum\phi_i H_i^*}{1-2\sum\phi_i H_i^*} \quad (4)$$

where ϕ_i is the volume fraction of dispersed phase with radius R_i and

$$H_i^* = \frac{4(\alpha/R_i)[2G_M^*+5G_D^*]+[G_D^*-G_M^*][16G_M^*+19G_D^*]}{40(\alpha/R_i)[G_M^*+G_D^*]+[2G_D^*+3G_M^*][16G_M^*+19G_D^*]} \quad (5)$$

When the droplet polydispersity is less than 2.3, the volume-average radius can be used in Eq. 4 in place of the distribution (Graebling *et al.*, 1993). The Paliarne theory does a good job of describing the linear viscoelastic response of immiscible blends of flexible polymers (for example, Graebling *et al.*, (1993); Vinckier *et al.*, (1996); Kitake *et al.*, (1997); Lacroix *et al.*, (1998); Yamane *et al.*, (1998); Lee and Denn, (1999)). The general characteristic of these blends is that G'' is relatively insensitive to the presence of the dispersed phase for small ϕ , as is G' at high frequency. G' increases to a value significantly above the matrix value, however, at a frequency near the reciprocal of the time constant given by Eq. 3.

The zero-shear viscosity η_0 of the blend can be obtained from Eq. 4 by allowing the frequency to go to zero. The result is

$$\eta_0 = \eta_M \left[\frac{10(1+k)+3\phi(2+5k)}{10(1+k)-2\phi(2+5k)} \right] \cong \eta_M \left[1 + \phi \frac{2+5k}{2(1+k)} + O(\phi^2) \right] \quad (6)$$

This result, first obtained by Taylor (1932), is independent of the droplet size and the interfacial tension. In the linear limit, the zero-shear viscosity increases by a factor between $1 + \phi$ for $k \ll 1$ and $1 + 2.5\phi$ for $k \gg 1$; the latter is the Einstein equation for the viscosity of a dispersion of rigid spheres.

Blend theories for finite deformations have been developed only for newtonian fluids. The theory of Choi and Schowalter (1975) predicts the following for the shear viscosity and first normal stress difference:

$$\eta = \eta_M \left[1 + \phi \times \frac{2(5k+2)-10(k-1)\phi^{7/3}}{4(k+1)-5(5k+2)\phi+42k\phi^{5/3}-5(5k-2)\phi^{7/3}+4(k-1)\phi^{10/3}} \right] \cong \eta_M \left[1 + \phi \frac{2+5k}{2(1+k)} + O(\phi^2) \right] \quad (7a)$$

$$N_1 = N_{11} - N_{22} = \eta_M \phi \dot{\gamma}^2 \left(\frac{\eta_M R}{\alpha} \right) \left(\frac{m}{1+(h\dot{\gamma})^2} \right) \quad (7b)$$

m and h are rational functions of k and ϕ . This result is exact to first order in the deviation from a spherical droplet; a shear-rate dependence of the viscosity occurs at higher order.

The Paliarne and Choi-Schowalter zero-shear viscosities agree through the linear term, but the quadratic term is considerably larger in the latter theory. Equation 7b requires a value for the droplet size, and it is clear that the appropriate radius is the one corresponding to the given shear rate. The scaling from Eq. 2 then suggests that the first normal stress difference should be linear at low rates. At high rates one would expect the droplet size to approach a limiting minimum value, so the normal stress should approach a constant. This qualitative reasoning permits a possible maximum in the normal stress, but it does not account for possible large deviations from sphericity.

The Doi-Ohta (1991) theory and an extension by Lee and Park (1994) describe the evolution of the interface as well as the rheology in a blend of newtonian fluids; the theory predicts linear shear and normal stresses that are proportional to the interfacial tension and the interfacial area per unit volume, and the steady-state droplet size is given by Eq. 2 with $g = \text{constant}$. Takahashi and coworkers (1994) proposed that the theory could be applied to the extra stresses arising from the interface for blends of non-Newtonian fluids, as follows:

$$\Delta\tau(\dot{\gamma}) = \tau(\dot{\gamma}) - \phi_D \tau_D - (1 - \phi_D) \tau_M \sim \dot{\gamma} \quad (8a)$$

$$\Delta N_1(\dot{\gamma}) = N_1(\dot{\gamma}) - \phi_D N_{1D} - (1 - \phi_D) N_{1M} \sim |\dot{\gamma}| \quad (8b)$$

This scaling has been shown to hold for immiscible flexible polymer blends (for example, Takahashi *et al.* (1994a; 1994b); Vinckier *et al.* (1996)); the size scaling has been observed for isotropic viscoelastic blends with different viscosity and volume ratios by Vinckier and coworkers (1996), but not by Lacroix and coworkers (1998).

3. Polyester Blends

Kim and Denn (1992) studied a blend of Vectra-A, a liquid crystalline copolyester of 73 mole% p-hydroxynaphthoic acid and 27% p-hydroxybenzoic acid and polyethylene terephthalate (PET). Thermal analysis showed a large depression of the PET melting temperature in LCP-rich compositions, indicating interactions between the two blend components, and nuclear magnetic resonance spectroscopy showed evidence of interfacial mixing (Tang *et al.*, 1993). In addition, the presence of the LCP affected the ratio of trans to gauche conformations in the PET (Tang *et al.*, 1993) and the rate and extent of crystallization (Kim and Denn, 1992; Chang-Chien and Denn, 1996). According to Flory's (1978) thermodynamic analysis, blends of rodlike molecules with flexible coils are immiscible, but of course the LCP is not perfectly rodlike and PET is stiffer than a random coil. Ester exchange reactions at the interface are a possible explanation of the interfacial mixing, but no evidence of interfacial reactions has been found for this system

(Chang-Chien and Denn, 1996; Magagnini *et al.*, 1998).

At Vectra-A fractions of 20% and below, the shear viscosity at low shear rates was less than that of either PET (the lower-viscosity component) or Vectra A; the dispersed phase droplets appeared to remain spherical in these experiments, although the system is opaque and some retraction might have occurred during the cooling step required for sample observation. A similar reduction in the magnitude of the complex viscosity was seen in linear viscoelastic measurements, where the droplet morphology is certainly spherical. These results are qualitatively different from the predictions of all blend theories. It is clear that the standard processing aid explanation of an internal slip surface created by elongated low-viscosity inclusions cannot apply. Similar results were obtained on another polyester LCP/polyester flexible matrix by Van Eijnhoven-Rivera and coworkers (1998), as well as by Weiss and coworkers (1987) on a LCP/polystyrene blend.

The results for the polyester system are suggestive of a strong effect of the LCP dispersed phase, but they are ambiguous because of the possibility that undetected interfacial chemistry is playing a role in the rheological anomaly. In addition, the polyester systems require high temperatures, and transient effects resulting from chemical change are always a concern. In particular, the possibility that volatiles from the LCP might have plasticized the PET, though unlikely, cannot be completely ruled out (compare Giles and Denn (1994)).

4. HPC/PDMS Blend

Riise and coworkers (1999) studied a blend in which the dispersed phase was an aqueous solution of hydroxypropylcellulose (HPC) and the continuous phase was polydimethylsiloxane (PDMS). Aqueous HPC is lyotropic; at low HPC concentrations it is isotropic at rest, while above a critical concentration it is liquid crystalline, with an intermediate biphasic region. The liquid crystalline state is cholesteric at rest, but HPC is generally assumed to behave as a nematic in flow. This system was chosen because it was expected to be nonreactive and could be studied at and below room temperature. (A reduced temperature was required to ensure that the LCP was beyond the biphasic region of the phase diagram.)

A blend containing 10% of an isotropic HPC dispersed phase behaved as predicted by the Paliarne and Doi-Ohta theories. A blend containing 20% of a liquid crystalline dispersed phase was also described by the Paliarne theory, but there were deviations from the Doi-Ohta scaling at the highest shear rates studied. A blend containing 10% of a liquid crystalline dispersed phase, however, behaved in a surprising manner: The Paliarne theory substantially over-predicted G' , and in fact the data were fit best by a curve calculated assuming a zero interfacial tension. The 10%

LCP blend did follow the Doi-Ohta linear scaling for steady shear. The only obvious difference between the two LCP blends was the droplet size; the average radius for the 10% blend was 7 μm , while in the 20% blend the average droplet radius was 15 μm . The characteristic length of a typical liquid crystalline domain for the bulk aqueous HPC liquid crystalline phase is roughly 5 μm .

The average droplets in the 20% LCP blend contained many domains, while the average droplets in the 10% blend were comparable in size to one or two domains. It is tempting to conclude that the dynamics of the LCP droplets were very different on the two length scales; the larger droplets would have been macroscopically isotropic, while the smaller droplets were likely highly oriented. Whether this size effect, if indeed a consequence of orientation, is a manifestation of a bulk or surface phenomenon cannot be determined from the data at hand. There is in fact one confounding experimental detail. Water is slightly soluble in PDMS. The small amount of possible water absorption into the PDMS did not appear to have been a problem, but the extraction of any water from the concentrated HPC droplets could have had a significant effect on their bulk rheological and interfacial properties.

5. Vectra-B/FEP Blends

Lee and Denn (1999) studied the rheology of a blend of Vectra-B, a liquid crystalline copoly(ester amide) consisting of 60 mole% hydroxynaphthoic acid, 20% hydroxybenzoic acid, and 20% aminophenol, with a continuous phase of Teflon FEP 100, a copolymer consisting of 87 mole % tetrafluoroethylene and 13% hexafluoropropylene. This system is completely immiscible and without any possibility of interfacial reactions, although the LCP is likely to be slightly reactive at experimental conditions. They analyzed data in the context of the Paliarne theory by plotting the ratio of G' of the blend to G' of the matrix; according to the Paliarne theory there will be a maximum in this curve at a frequency in the neighborhood of the inverse of τ_D (Eq. 3). The theory predicted the location of the maximum quite well, but it substantially underpredicted the magnitude for LCP concentrations of 6.5 and 12.5 vol. %; the prediction of the magnitude was accurate only for a 24.4% blend. The volume-average droplet radii for these concentrations were 4.4, 5.2 and 14.3 μm , respectively. The characteristic length of a liquid crystalline domain in quiescent bulk Vectra-B is less than 5 μm .

The Paliarne theory does fit the experimental data for all three compositions if it is assumed that the interfacial tension between the LCP and the flexible polymer is zero for droplets with radii less than 1.5 μm , but equal to the bulk value for larger droplets. This is consistent with the treatment of the HPC/PDMS data, where the data were fit by assuming that the interfacial tension was zero for drop-

lets of the order of a domain, and confounding experimental issues are absent. There is, however, an alternative treatment of the data. The ratio of the blend storage modulus to that of the matrix has a maximum with respect to interfacial tension for a fixed droplet size, and the difference between taking the interfacial tension to be zero or infinity is small. The Vectra-B/FEP data can also be fit by assuming that the interfacial tension is infinite for droplets with radii less than 1.5 μm , although the fit is not quite as good as that obtained by setting the interfacial tension to zero.

The Choi-Schowalter theory, Eq. 7a, is a better fit to the zero-shear viscosity than the Paliarne model, Eq. 6, but the theory underpredicts the viscosities of the two lower concentrations and overpredicts the viscosity of the 24.4% blend. There is a large negative deviation from the Doi-Ohta viscosity scaling for the lowest concentration (corresponding to the smallest mean droplet size) in a frequency range that is equivalent to a longer time scale than that given by Eq. 3. The excess normal stresses appear to be initially linear, with a decreasing slope with increasing shear rate.

6. Perspective

The limited data available on immiscible blends with an LCP dispersed phase show an unambiguous droplet-size effect that is absent in flexible polymers. When the droplets are large relative to the orientation correlation length (i.e., contain many "domains"), the approaches developed for blends of flexible polymers appear to be adequate; these approaches fail when droplet length scales are comparable to the orientation correlation length. The underlying physical phenomena are not transparent, however, since the data can be interpreted in diametrically-opposed ways: the effective interfacial tension is either very large or very small relative to the bulk value. Indeed, considering the limit of very large apparent interfacial tension, the phenomenon might reflect the bulk properties of the highly oriented "domain." At present, an explanation of this interesting and possibly important behavior is unavailable.

The polyester blends present an additional complication because of the interfacial interactions, as well as the possibility that the observed behavior may be a consequence of volatiles from the LCP plasticizing the continuous phase. This is the only system of the three discussed here for which a reduction in the viscosity below that of either phase is observed at low shear rates, and this behavior does not appear to be a general property of LCP/flexible polymer blends. The mechanism does need to be understood, however, because thermotropic LCPs are potentially reactive with many candidate matrix polymers.

Finally, the theory of blends of flexible polymers is itself well-developed only for linear viscoelasticity, where all deformations are infinitesimal. A theoretical foundation is

lacking for liquid crystalline dispersed phases even in this limit, and without such a theory the unusual behavior observed for these blends cannot be exploited with confidence.

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