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The effect of miscibility on the morphology and properties of blends of poly(ether imide) with liquid crystalline polymers

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A poly(ether imide) (Ultem), which is an amorphous engineering thermoplastic, was melt blended with two liquid crystalline polymers: one based on *p*-hydroxybenzoic acid and 2-hydroxy-6-naphthoic acid (Vectra A900) and the other based on phenylhydroquinone, ethylphenylhydroquinone and terephthalic acid (Granlar). The miscibility of these two blend systems was studied by means of differential scanning calorimetry (d.s.c.), dynamic mechanical thermal analysis (d.m.t.a.), scanning electron microscopy (s.e.m.) and wide-angle X-ray scattering (WAXS). It was found that the Ultem/Vectra system is immiscible, while the Ultem/Granlar system is partially miscible. As revealed by d.m.t.a., the partial miscibility of the latter can be improved by increasing the degree of mixing in a single-screw extruder. The flexural and tensile moduli of the *in situ* reinforced composites based on these two blend systems were determined as a function of blend composition. For similar mixing conditions it was found that the flexural moduli of the Ultem/Granlar system showed a positive deviation from the simple rule of mixtures while those of the Ultem/Vectra system showed a negative deviation. The tensile moduli of the Ultem/Vectra system also showed a pronounced positive deviation only for blends with high Vectra compositions. These results are interpreted in terms of the differences in miscibility.

(Keywords: miscibility; poly(ether imide); liquid crystalline polymers)

INTRODUCTION

In situ composites based on blending of liquid crystalline polymers (LCP) with engineering thermoplastics have recently received considerable attention[1–9]. As pointed out in a recent review article[10], morphology and properties of *in situ* composites would be affected by many factors, one of which would be the miscibility between the liquid crystalline polymer and the thermoplastic matrix. Studies dealing with the miscibility of the blend of a thermotropic liquid crystalline copolymer based on *p*-hydroxybenzoic acid and ethylene terephthalate (PHB/PET) with various thermoplastics can be found in the literature. Nakai *et al.*[11] reported that PHB/PET 60/40 (where 60 denotes the mole percentage of PHB in the copolyester) formed a miscible blend with polyethylene terephthalate (PET) for samples that were solvent cast. However, a phase separation of this blend occurs upon thermal treatment. As reported by Paci *et al.*[12], when PHB/PET 60/40 was blended with polyethylene terephthalate (PET), a transesterification reaction was evident. Zhuang *et al.*[13] studied blends of PHB/PET with polycarbonate (PC), PET and polystyrene (PS). On the basis of differential scanning calorimetry and dielectric and dynamic mechanical analysis, a claim was made that PS was totally immiscible, PC was partially miscible and PET was completely miscible with the PET component of the LCP. The presence of fibrils of the liquid crystal phase was observed. Improvements were also found in the tensile properties of these blends. No attempt was made to explain the reason for the observed morphology or correlate it with the miscibility. Recently, it has been found that no single phase can be prepared with PHB/PET and PC through solvent casting[14]. The blends of PHB/PET with PC were originally reported to be partially miscible[13].

In this paper poly(ether imide) (PEI), commercially known as Ultem, has been used as an amorphous matrix polymer in the preparation of blends with LCPs to prepare high-modulus *in situ* composites. While it has been reported that the Ultem is miscible with several thermoplastics[17–19], there has been no work reported in the literature dealing with its miscibility with liquid crystalline polymers. The results reported in this paper, for Ultem which was melt blended with two thermotropic liquid crystalline systems, are concerned with the blend miscibility and its effect on the morphology and mechanical properties of the corresponding *in situ* composites.

EXPERIMENTAL

Materials and sample preparation

The amorphous matrix polymer used was a PEI (Ultem 1000) sold by the General Electric Company. The

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*To whom correspondence should be addressed reinforcing phase was one of two LCPs: a liquid crystalline aromatic copolyester consisting of 73 mol% hydroxybenzoic acid and 27 mol% 2-hydroxy-6-naphthoic acid moieties (Vectra A-900) sold by Hoechst-Celanese Corporation and another liquid crystalline aromatic copolyester consisting of terephthalic acid, phenyl hydroquinone and phenyl ethyl hydroquinone (Granlar) provided by Istituto Guido Donegani (Montedison).

Samples were prepared for analysis by either extruding films or injection moulding plaques. First, pellets of the Ultem were dried at 70°C while the liquid crystalline polymers, Vectra and Granlar, were dried at 150°C. All drying processes were carried out in a vacuum oven for at least 24 h. The dried pellets were then tumbled together in predetermined weight ratios in a steel container before being blended in a single-screw extruder or the screw of an injection moulder. A laboratory single-screw extruder (Killion KL100) connected to a 102 mm wide film die (gap = 0.5 mm) was used to generate the films. The temperature of the melting section of the extruder was set at 370°C. The temperature of the connector between the extruder and die was set at 360°C while the die temperature was set at 345°C and 360°C for Ultem/Vectra and Ultem/Granlar, respectively. To obtain thicker specimens, rectangular plaques measuring approximately 75 mm × 85 mm × 1.75 mm were also moulded using an Arburg (221-55-250 Allrounder) injecting moulding unit. Temperatures were set at 300, 360, 370°C in zones 1, 2 and 3 of the barrel, respectively, and at 370°C at the nozzle.

To study the effect of mixing history, the mixture of Ultem and Granlar pellets was tumbled and extruded in a single-screw extruder (Killion KL-100) connected to a pelletizing die. The extrudates were quenched in ice-water and continuously pelletized. In order to impart additional mixing history, the pellets were re-extruded in the single-screw extruder and then pelletized again. The blended pellets were then used to make injection-moulded plaques, and hence the samples were subjected to two additional mixing histories.

Thermal analysis

The dynamic mechanical response of the thin film was obtained by means of a Rheovibron (model DDV-2C) which was automated by a system developed by the Imass Company. The heating rate was 2°C min^{-1} and the frequency was 11 Hz. For the thicker injection-moulded plaques dynamic mechanical analyses were carried out in the torsional mode in a Rheometrics Mechanical Spectrometer (RMS 800). For the torsional modulus measurements, rectangular samples with dimensions of 45 × 12.5 mm were cut from the injection-moulded plaques. Tan δ of the samples was monitored and recorded as a function of temperature. The frequency of oscillation was 10 rad s^{-1} and the strain was 0.05%. The temperature was raised at a rate of 5°C min^{-1} until the modulus of a given sample dropped appreciably.

A Sieko DSC (model 210) was utilized for the thermal analysis of these blends. The heating rate was fixed at 10°C min^{-1} for all the samples and prepurified nitrogen was used as the purge gas.

Structure and properties

Wide-angle X-ray diffraction (WAXD) was carried out by using a Phillips 1720 table top X-ray generator equipped with a fixed copper target X-ray tube and Warhus camera. The diameter of the pin-hole collimator was 0.5 mm and the sample-to-film distance was 76 mm. The beam conditions were 40 kV and 20 mA and the patterns were recorded under vacuum.

The morphology of the blends was determined by scanning electron microscopy (s.e.m.), using a Cambridge Stereoscan S200 with an accelerating voltage of 25 kV. All samples were fractured perpendicular or parallel to the draw direction after immersing them in liquid nitrogen. The fractured surfaces were sputter coated with gold to provide enhanced conductivity.

The flexural modulus of the injection-moulded plaques was measured according to ASTM test D 790-86 using an Instron mechanical tester (model 4204) at room temperature. The test samples were strips approximately 80 mm long, 12.5 mm wide and 1.75 mm thick. The crosshead speed was 2 mm min^{-1}. The tensile tests were done using an Instron mechanical tester (model 4204). The crosshead speed for tensile tests was 1.27 mm min^{-1}. The samples for tensile tests were injection moulded as per specifications given in ASTM Standard D 638-87b (Type V). The arithmetic average and the standard deviation of the tensile properties were calculated using a minimum of five samples. The toughness reported is the area under the stress–strain curve.

RESULTS AND DISCUSSION

Dynamic mechanical and thermal analysis

To study the miscibility of the Ultem/Vectra and Ultem/Granlar blends, their glass transition temperatures were measured as a function of the blend composition. The dynamic torsional test results for the Ultem/Vectra blends of various compositions show a glass transition temperature (T_g) at 228°C for the pure Ultem and 105°C for the pure Vectra. These results reveal that Vectra is immiscible with Ultem (*Figure 1*). As seen from the dynamic torsional analysis of the Ultem/Granlar blends shown in *Figure 2*, for the Ultem/Granlar blend at a ratio of 90 wt% Ultem and 10 wt% Granlar (90/10) only one T_g can be detected and its value is lower than that of the neat Ultem. As the Granlar content in the blend increases,

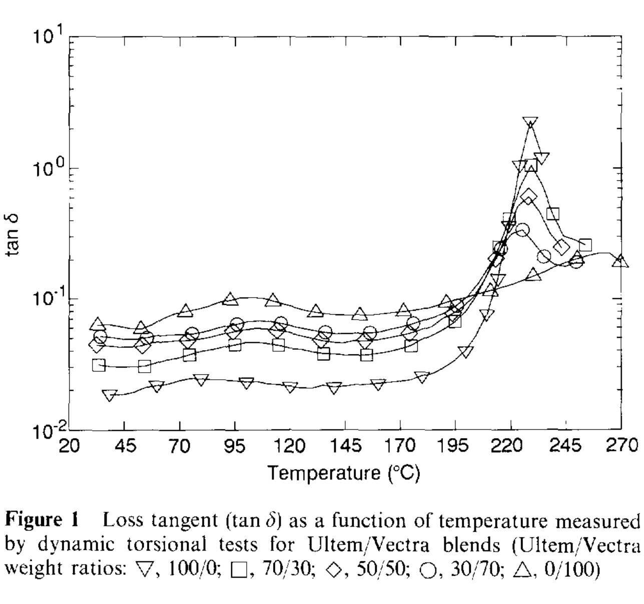

Figure 1 Loss tangent (tan δ) as a function of temperature measured by dynamic torsional tests for Ultem/Vectra blends (Ultem/Vectra weight ratios: ▽, 100/0; □, 70/30; ◇, 50/50; ○, 30/70; △, 0/100)

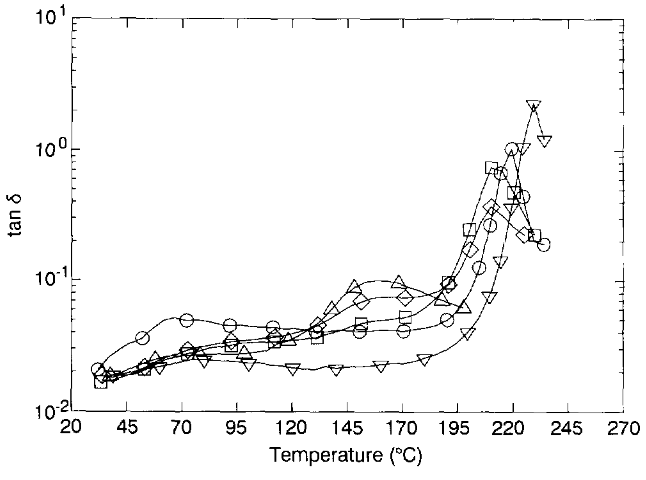

Figure 2 Loss tangent (tan δ) as a function of temperature measured by dynamic torsional tests for Ultem/Granlar blends (Ultem/Granlar weight ratios; ▽, 100/0; ○, 90/10; □, 70/30; ◇, 50/50; △, 0/100)

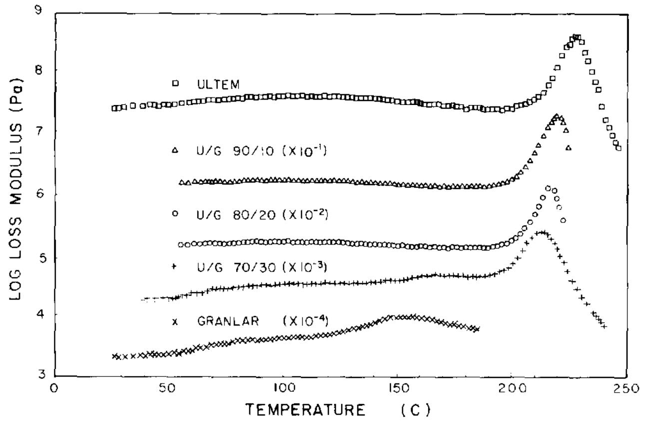

Figure 3 Loss modulus *versus* temperature measured by dynamic stretching tests for Ultem/Granlar blends of various compositions

two separate T_gs can be observed and the higher one (corresponding to the T_g of the Ultem phase) remains lower than that of the neat Ultem and becomes relatively insensitive to the Granlar content.

To verify this interesting phenomenon, the extruding films were investigated by utilizing the Rheovibron in a stretching mode (*Figure 3*). For the pure Granlar, a broad maximum in the tan δ curve is observed at 155°C. This transition temperature agrees well with that obtained from the torsional results. For the blends that contain 10 and 20 wt% Granlar, there is no evidence of a glass transition at 155°C. In fact, the spectra are very similar to that of the pure Ultem except that the onset of the glass transition decreases as the Granlar content increases. As the Granlar content increases to 30 wt%, the T_g at 155°C can be observed again which indicates the existence of a Granlar phase in the blend. The lack of a Granlar T_g at concentrations less than 20 wt% Granlar cannot be attributed to the lack of sensitivity of the dynamic mechanical analysis as two T_gs are readily observable for the Vectra/Ultem systems for similar levels of Vectra.

For further confirmation of the shift of the glass transition temperature of the Ultem phase, differential scanning calorimetry (d.s.c.) thermograms of this system were also obtained (see *Figure 4*). Two points are obvious from this figure. First, there is a shift in the Ultem T_g up to about 20 wt% Granlar with no further shift as the percentage of Granlar is increased. Second, d.s.c. is not sensitive enough to pick up the T_g of the Granlar phase for the blend compositions shown.

The T_g of the Ultem-rich phase of these blends obtained from the d.s.c. is plotted *versus* the Granlar concentration and compared with those determined from the RMS and the Rheovibron (*Figure 5*). The trend of the T_g dependence on the Granlar concentration from these three measuring techniques is in good agreement. However, the T_g values obtained from dynamic mechanical analysis are higher than those obtained from thermal analysis. Such a difference is reasonable when one takes the features of these measuring techniques into consideration. In *Figure 5*, curves based on the Fox equation, which is often used to estimate the T_g of a perfectly miscible polymer blend, are also represented. One can note that for the Ultem/Granlar 90/10 blend, the experimental

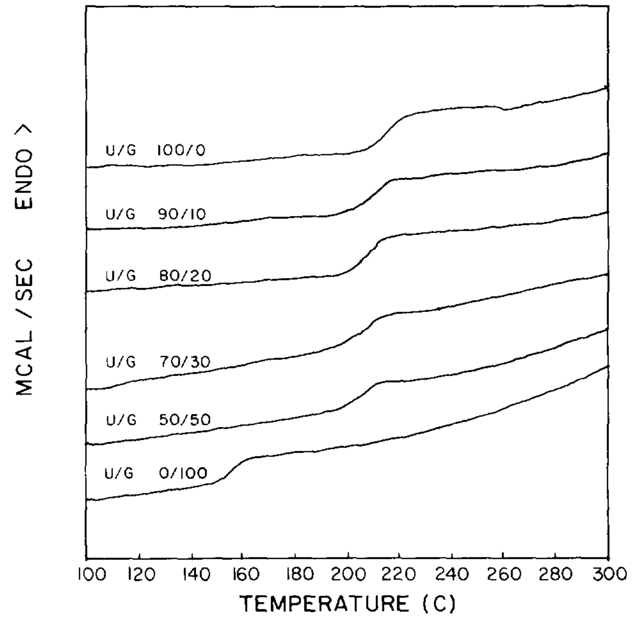

Figure 4 D.s.c. thermograms of Ultem/Granlar blends of various compositions

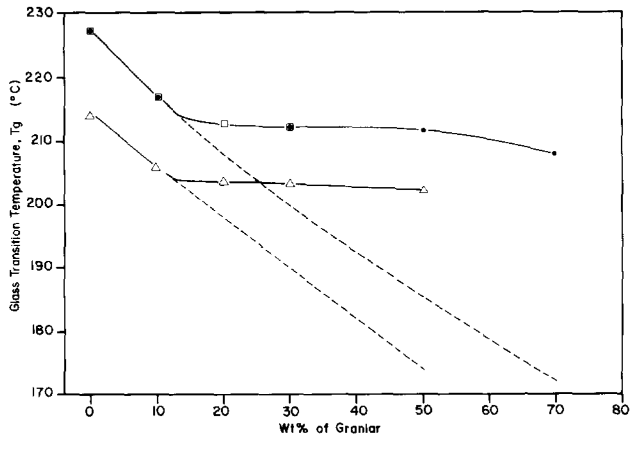

Figure 5 Glass transition temperature *versus* weight percentage of Granlar in Ultem/Granlar blends (△, values obtained from d.s.c.; □, values obtained from Rheovibron; ●, values obtained from dynamic torsional tests; ---, values calculated from the Fox equation)

values of T_g obtained from the three methods agree well with those predicted by the Fox equation. This suggests that at 10% Granlar loading, the Ultem is miscible with the Granlar. As the Granlar content increases to 20 wt%, the T_g of the Ultem-rich phase becomes relatively insensitive to the Granlar content as it exceeds 20 wt%. These results indicate that the two components are partially miscible. Furthermore, all three methods suggest that the extent of miscibility is somewhere between 10 and 20 wt% of Granlar content.

It is also interesting to know if the extent of miscibility of the Ultem/Granlar system is affected by the melt blending history. As blending on a molecular level is not possible in the melt state, mixing history could play a significant role. For this purpose the Ultem/Granlar 80/20 blend was chosen, and the glass transition behaviour of an injection-moulded sample prepared by mixing in the screw of the injection-moulding unit (single pass material) was compared with that of a sample prepared by first mixing in a single-screw extruder and then injection moulding the pellets (double pass) (*Figure 6*). For the plaques prepared only by mixing in the injection-moulding unit (single-pass blend plaques), two T_gs can be detected. The lower one characterizes the segmental motion of the Granlar, while the higher one characterizes the glass transition of the Ultem-rich phase. These results imply that a free Granlar phase and a miscible Ultem–Granlar phase coexist in the given blend. After mixing and then injection moulding (double-pass blend), the T_g of the free Granlar phase vanished from the dynamic mechanical spectrum, and the T_g of the Ultem-rich phase shifted to a lower temperature (the shifted T_g matched the Fox equation well at this blend composition). These facts not only confirm the partial miscibility of this blend system, but also show that the extent of miscibility can be improved with mixing. Although the data are not shown, it was found that additional mixing time did not lead to any further shifting of the T_g.

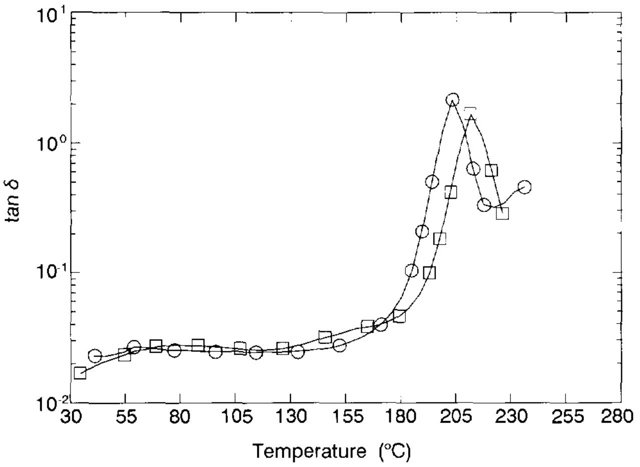

Figure 6 Loss tangent (tan δ) *versus* temperature for Ultem/Granlar 80/20 blend as a function of mixing history (□, one pass; ○, two passes)

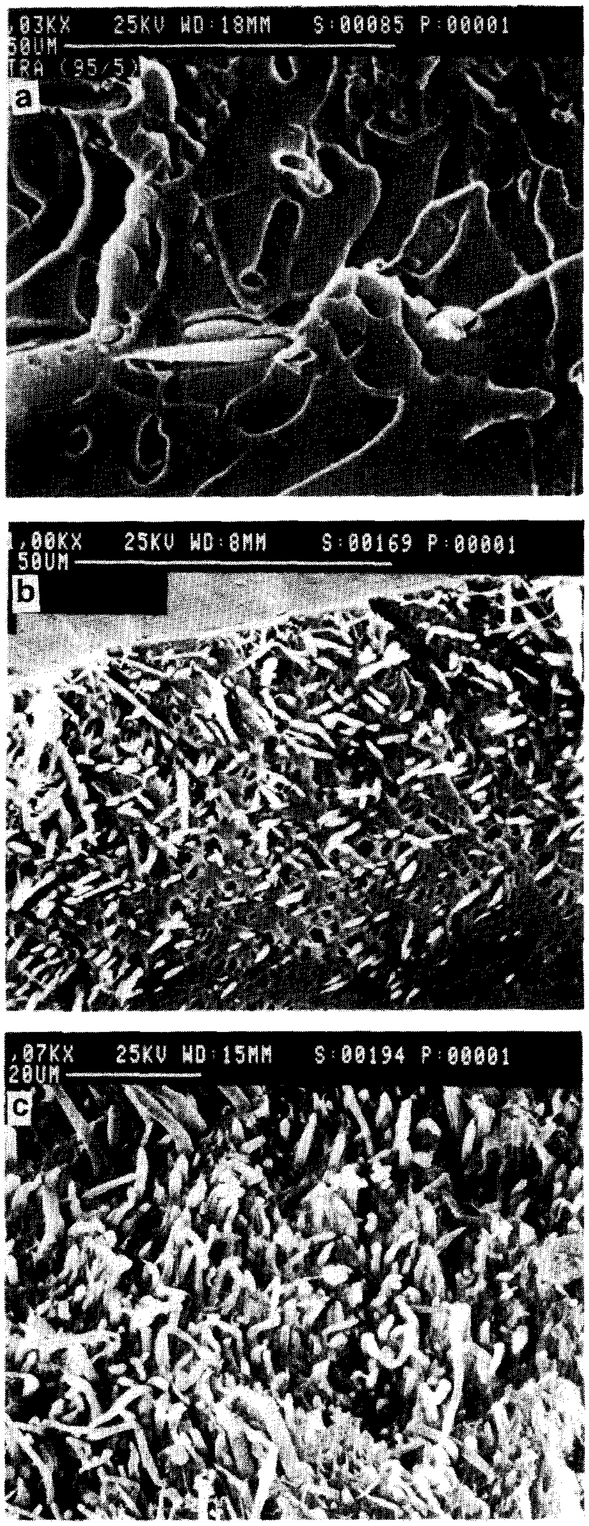

Figure 7 S.e.m. micrographs of the fractured surfaces of extruded Ultem/Vectra films of various blend compositions: (a) Ultem/Vectra 95/5; (b) 90/10; (c) 70/30

Morphology and structure

The difference between the Ultem/Vectra and Ultem/Granlar blends in terms of miscibility can also be observed clearly from the s.e.m. and WAXD studies. The phase separation of extruded Ultem/Vectra blend films is distinct, even when the content of Vectra in the blend is as low as 5% (*Figure 7*), but no phase separation can be detected in the Ultem/Granlar system, at least at the level of magnification used here, even when the Granlar content in the blend reaches 10% (*Figure 8*). The WAXD patterns of the Ultem/Vectra and Ultem/Granlar blend sheets for various compositions are shown in *Figures 9* and *10*. For the samples of the Ultem/Vectra for two composition ratios of 90/10 and 70/30, a sharp

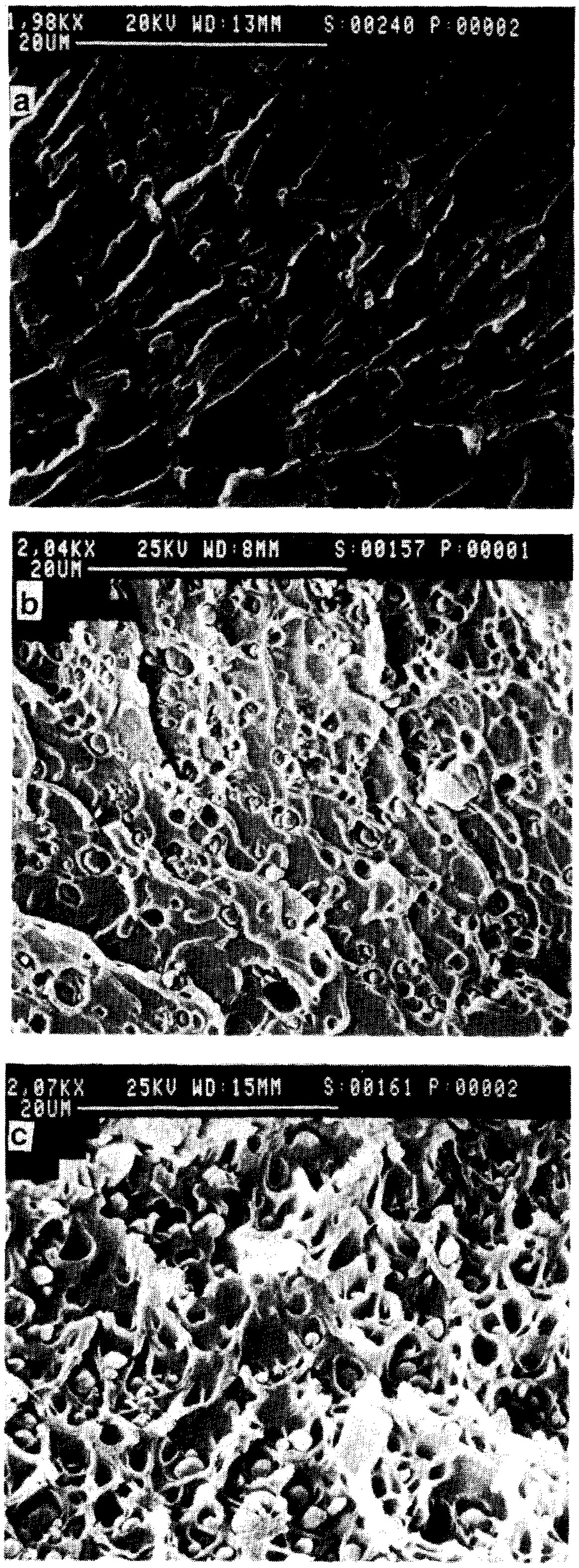

Figure 8 S.e.m. micrographs of the fractured surfaces of extruded Ultem/Granlar films of various blend compositions: (a) Ultem/Granlar 90/10; (b) 80/20; (c) 70/30

Debye ring corresponding to a *d*-spacing of 0.46 nm is observed (*Figure 9*). This is believed to be caused by the phase-separated Vectra domains in the blends. Debye arcs are not observed for the case of the drawn Ultem/Granlar 90/10 blend sheet (see *Figure 10a*) but are evident for the case of the drawn sheets of higher Granlar concentrations (see *Figure 10b* and *c*). This again suggests that Ultem and Granlar are miscible with each other and no phase separation occurs at the 10% Granlar concentration level. Both the s.e.m. and WAXD results are in good agreement with those obtained from dynamic mechanical and thermal analysis.

Mechanical properties

It is apparent that the Ultem/Granlar system is at least partially miscible. The question that then arises is whether the difference in miscibility would affect the morphology and mechanical properties of the composites based on these Ultem/LCP blends. To understand the effect on mechanical properties, the flexural moduli of both the Ultem/Vectra and the Ultem/Granlar injection-moulded plaques were measured along the machine direction as a function of the LCP fibril volume fraction, V_f. The experimental results are also compared with the modulus values predicted by the rule of mixtures (parallel model) which is given below:

$$E_{1b} = E_m V_m + E_f V_f \qquad (1)$$

where E_{1b}, E_m and E_f represent the flexural moduli of the *in situ* composite, the matrix polymer and a pure injection-moulded LCP plaque, respectively, and V_m and V_f represent the volume fraction of the matrix polymer and the LCP phase in the composites, respectively. In the case of Ultem/Vectra (*Figure 11*) the moduli show a negative deviation, while in the case of Ultem/Granlar (*Figure 12*) the moduli show a positive deviation from equation (1).

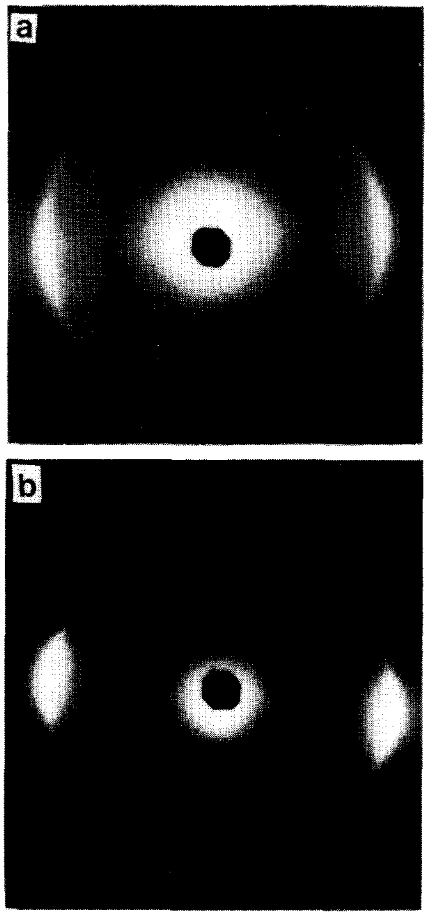

Figure 9 WAXD patterns of Ultem/Vectra blend sheets. (a) Ultem/Vectra 90/10; (b) 70/30

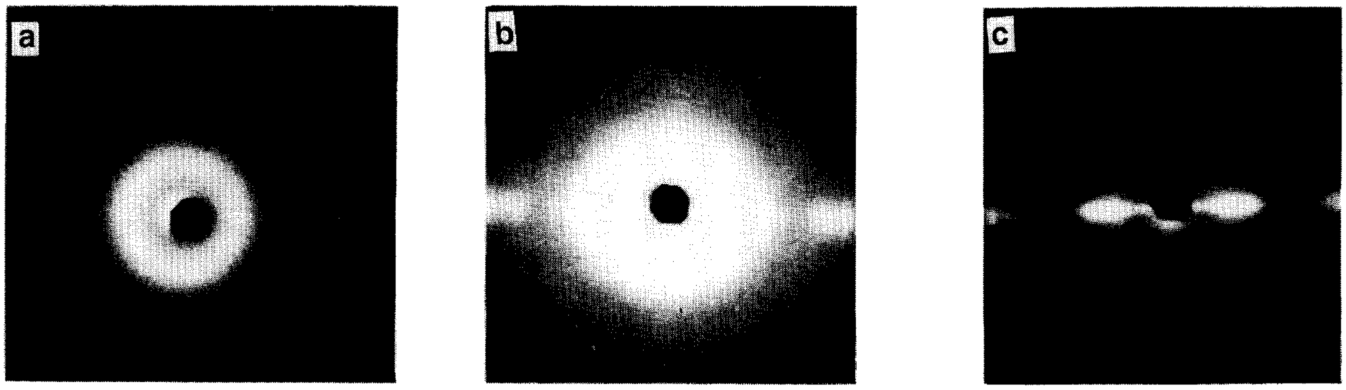

Figure 10 WAXD patterns of Ultem/Granlar blend sheets. (a) Ultem/Granlar 90/10; (b) 80/20; (c) 70/30

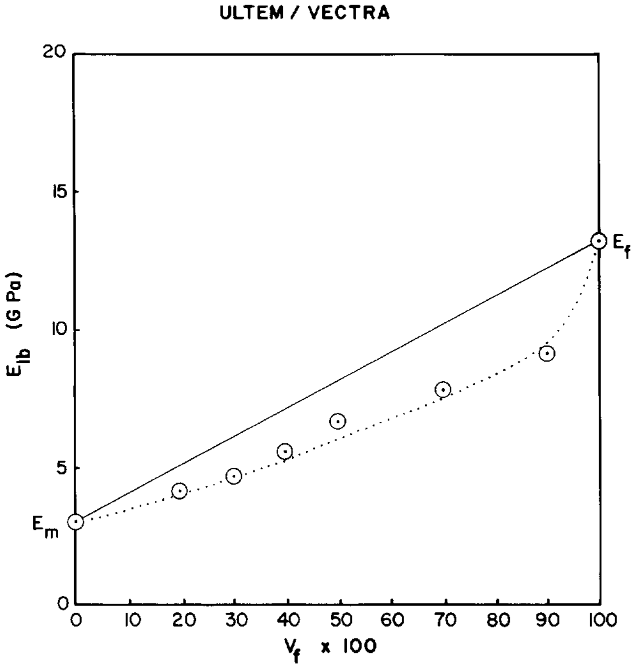

Figure 11 Machine direction flexural modulus *versus* volume fraction of Vectra phase for injection-moulded Ultem/Vectra plaques. ○, Experimental data; ——, law of mixtures

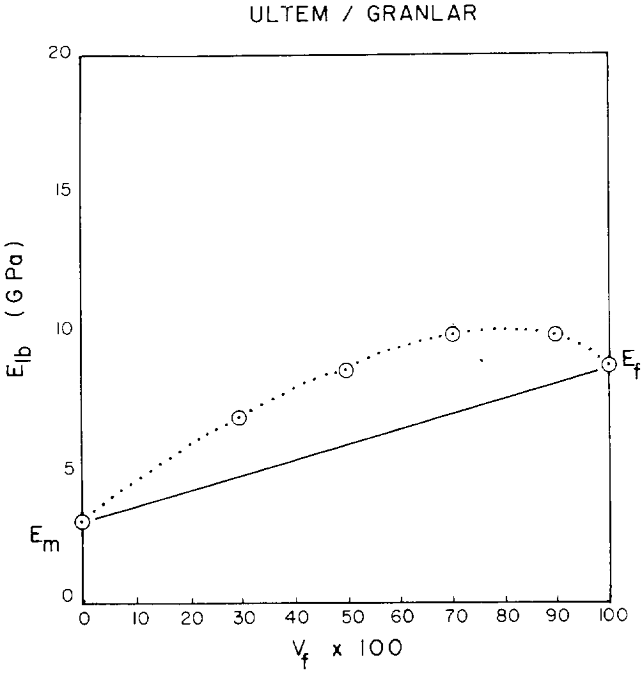

Figure 12 Machine direction flexural modulus *versus* volume fraction of Granlar phase for injection-moulded Ultem/Granlar plaques. ○, Experimental data; ——, law of mixtures

The tensile properties are listed in *Table 1*. In *Figure 13* it is shown that for the Ultem/Granlar blend, the tensile modulus shows a positive deviation from the rule of mixtures, just as the flexural modulus did. However, the behaviour of the tensile properties of the Ultem/Vectra blend is considerably more complex. In *Figure 14* it is shown that the tensile modulus of the Ultem/Vectra blend shows large deviations from the rule of mixtures when the Vectra content in the blend is high (greater than 70%). The tensile modulus for the Ultem/Vectra A 1/9 blend (13.5 GPa) is even higher than that of pure Vectra A itself (11.0 GPa)! The reason for this is not clearly understood. The tensile strength for either blend does not show any simple trend. The tensile strength of blends with the higher Vectra content (greater than 70%) is more than that of either pure Ultem or pure Vectra. The tensile strength for the Ultem/Vectra A system shows a pronounced maximum when the Vectra A weight fraction is 0.9. The elongation at break for the

Table 1 Tensile properties of injection-moulded Ultem/Vectra and Ultem/Granlar blends

Composition	Modulus (GPa)	Strength (MPa)	Elongation (%)	Toughness (kJ m^{-3})
Ultem/Vectra 10/0	3.0 (0.2)	100 (1)	60[a]	7752 (198)[b]
Ultem/Vectra 7/3	5.8 (0.6)	135 (6)	4.42 (0.86)	3826 (661)
Ultem/Vectra 5/5	7.0 (0.5)	129 (10)	3.15 (0.22)	2447 (416)
Ultem/Vectra 3/7	10.0 (0.5)	187 (11)	4.48 (0.48)	5550 (851)
Ultem/Vectra 2/8	10.8 (0.3)	194 (14)	4.42 (0.36)	5896 (703)
Ultem/Vectra 1/9	13.5 (0.4)	240 (6)	4.64 (0.23)	7577 (314)
Ultem/Vectra 0/10	11.0 (1.0)	234 (17)	4.41 (0.12)	5942 (131)
Ultem/Granlar 10/0	3.0 (0.2)	100 (1)	60[a]	7752 (198)[b]
Ultem/Granlar 7/3	9.0 (0.5)	129 (6)	2.05 (0.08)	1574 (205)
Ultem/Granlar 5/5	10.2 (0.5)	103 (10)	1.21 (0.21)	726 (128)
Ultem/Granlar 3/7	11.4 (0.4)	74 (12)	0.66 (0.13)	397 (137)
Ultem/Granlar 0/10	12.5 (1.2)	85 (18)	0.75 (0.21)	534 (199)

Standard deviations are given in parentheses
[a] Data from GE Ultem Properties Guide
[b] Calculated at 10% elongation

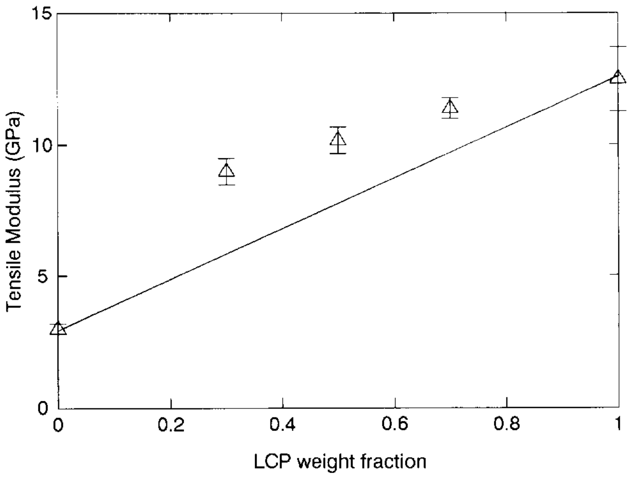

Figure 13 Tensile modulus *versus* Granlar weight fraction of injection-moulded Ultem/Granlar tensile bars

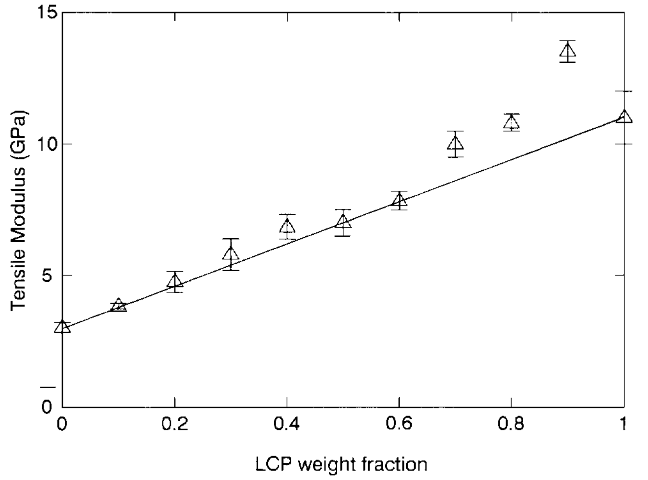

Figure 14 Tensile modulus *versus* Vectra weight fraction of injection-moulded Ultem/Vectra tensile bars

blends is more than an order of magnitude less than that of pure Ultem (the elongation at break for Ultem is 60%). The toughness (defined as the area under the stress–strain curve) for the Ultem/Granlar blends is much less than that for pure Ultem (which is amorphous). The toughness for the Ultem/Granlar blends seems to show a minimum at about 70 wt% Granlar. The toughness for the Ultem/Vectra blends is also less than that of pure Ultem, but the toughness of the Ultem/Vectra A blend at a ratio of 1/9 shows a maximum toughness value. Hence, Ultem/Vectra A 1/9 composition seems to show exceptional properties because it shows a maximum in the tensile modulus, strength and toughness.

At present the reason for these results is not quite clear. It seems that miscibility may be at least one of the factors affecting the modulus. As reported by Paul[20] and Kleiner *et al.*[21] a synergism has been observed for the modulus in some miscible blends. In the immiscible Ultem/Vectra blend two separate phases, Ultem and Vectra, exist, while in the partially miscible Ultem/Granlar system, the Ultem–Granlar solid solution phase coexists with the free Granlar phase. Therefore, one can expect that in the latter case the matrix itself is reinforced to some extent by the LCP on a molecular level. Also due to the partial miscibility, the interfacial adhesion between the Ultem–Granlar solid solution phase and the free Granlar phase (existing as reinforcing fibrils in the composite) can be higher. It may result in a higher reinforcing effect and, consequently, higher moduli as compared with those predicted from the law of mixtures. The assumption of higher matrix/fibril interaction in the partially miscible *in situ* composite is supported by s.e.m. micrographs of the fracture surfaces of injection-moulded plaques (*Figure 15*). For the immiscible Ultem/Vectra composites numerous microfibrils are pulled out from the matrix due to the poor interfacial adhesion, but this is not observed for the partially miscible Ultem/Granlar composite. Another possibility is that because of partial miscibility there is lower interfacial surface tension, which leads to finer fibrils with high aspect ratios which would provide more surface area.

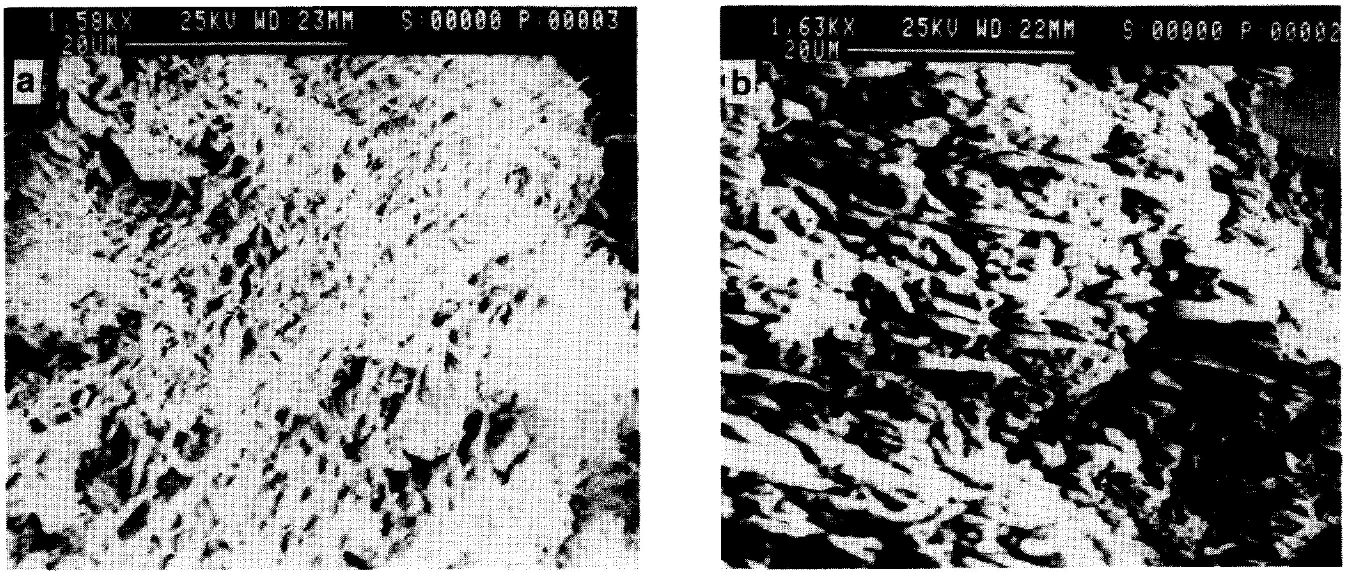

Figure 15 S.e.m. micrographs of the fractured surfaces of injection-moulded samples. (a) Ultem/Vectra 70/30; (b) Ultem/Granlar 70/30

CONCLUSIONS

By using d.m.t.a. and d.s.c. it has been found that the Ultem/Vectra blend is immiscible, but the Ultem/Granlar blend is partially miscible. These results were further confirmed by s.e.m. and WAXD studies. For Ultem/Granlar blends prepared by one pass through a single-screw extruder, the extent of miscibility was found to be around 13 wt% of Granlar. The extent of miscibility could be improved by intensifying the mixing process. The flexural moduli of the *in situ* reinforced composites prepared from the immiscible Ultem/Vectra blend showed a negative deviation from those predicted by the rule of mixtures, while in the case of the partially miscible Ultem/Granlar system, a positive deviation was observed. The tensile moduli of the Ultem/Granlar system also showed a positive deviation from the rule of mixtures but those of the Ultem/Vectra system showed a pronounced positive deviation only for blends with high Vectra compositions (greater than 70%). This difference in behaviour may be due to the partial miscibility, and hence the higher interfacial adhesion between Granlar and Ultem. It may also be due, at least indirectly, to the lower interfacial tension between Granlar and Ultem, which leads to the possibility of forming finer fibrils with higher aspect ratio and more surface area. The origin of the maximum in properties of the Ultem/Vectra system is not known at this time. However, it is interesting to note that miscibility does not necessarily guarantee enhancement of all properties. The properties of the LCP such as strength and toughness still play a significant role in affecting the properties of the blend.

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