



# Rheology–morphology relationships in nylon–LCP hybrid composites

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Received 20 July 2005; received in revised form 8 February 2006; accepted 8 February 2006

## Abstract

Rheological behaviors of hybrid composite systems consisting of liquid crystalline polymer (LCP), short glass fibers and toughened nylon 66 were characterized with capillary rheometry. The results were compared with the rheological behavior of untoughened nylon/glass fiber/LCP hybrid composites to establish the role of elastomers and glass fibers on LCP fibrillation. Results showed that, compatibilization of nylon and LCP in the presence of MA-grafted elastomer and glass fibers is the primary factor promoting LCP fibrillation in toughened glass fiber filled hybrid composites. Glass fibers serve as inert fillers modifying the interfacial interaction between the toughened nylon 66 and LCP phases. Morphology of the rheometer extrudates observed with scanning electron microscopy was consistent with the predictions from rheological results. The influence of rheology and morphology on the mechanical properties was also examined. It was found that tensile strength of toughened hybrid composites improved with the addition of glass fibers and on the other hand the strength of untoughened hybrid composites deteriorated with the glass fiber addition.

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**Keywords:** Hybrid composites; Liquid crystalline polymer; Reactive compatibilization; Rheology

## 1. Introduction

Concept of in situ hybrid composites was developed in which inexpensive inorganic fillers were used in conjunction with liquid crystalline polymer (LCP) as reinforcements for polymer matrices. Distinctive advantages were reported in processability and mechanical properties for in situ hybrid composites [1]. Nevertheless, it was found that filler loadings severely curtailed the ductility of thermoplastics. It will be interesting to invoke the concept of in situ hybrid composites for rubber toughened polymers. The distinctiveness of such materials is that, it will assimilate the processability and reinforcing advantages of liquid crystalline polymers [2], the cost effectiveness of glass fibers, and the utility of elastomeric phase to promote crack propagation toughness.

In this paper, we investigate the potential of a complex hybrid system consisting of rubber toughened nylon 66, short glass fibers, and a thermotropic LCP (Vectra A950).

One important parameter critical to the performance of in situ hybrid composite is the fibrillation of LCP phase during processing and its interfacial compatibility with the polymer matrix. In our previous studies, we observed enhanced fibrillation of LCP phase in the system with glass fibers in comparison to the one without glass fibers [3]. At 20-wt% LCP loading, synergistic improvements in tensile strength and modulus were observed.

In condensation polymers like polyamides with relatively low matrix viscosities, the viscosity ratio of LCP to that of matrix is unfavorable for LCP deformation [4]. Hence research efforts were directed at inducing LCP fibrillation in such thermoplastics by enhancing the capillary number. Critical capillary number defined by Taylor [5] determines droplet deformation of a minor LCP phase in a biphasic polymer blend. Capillary number (Ca) is the ratio of the viscous stress in the fluid to the interfacial stress intending to prevent its deformation. Specifically,

$$Ca = \frac{\eta_m \dot{\gamma} d}{\sigma} \quad (1)$$

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where  $\eta_m$  is the matrix viscosity,  $\dot{\gamma}$  is the shear rate,  $d$  the initial droplet radius, and  $\sigma$  the interfacial tension. A critical capillary number ( $Ca_{crit}$ ) exists, below which no droplet deformation takes place. Capillary number could be enhanced either by addition of suitable compatibilizers [6] which reduces the interfacial surface tension ( $\sigma$ ) or through blending extra fillers with the matrix to increase the matrix viscosity ( $\eta_m$ ) [7,8].

In our early work [9], we assessed the role of maleated rubber in compatibilizing LCP with nylon matrix. The maleic anhydride group was found to act as an efficient reactive compatibilizer for toughened nylon and LCP phase. Maleic anhydride groups are able to react with the amino groups of nylon 66 (Fig. 1(b)) as well as with the hydroxyl groups of Vectra A950 copolyester (Fig. 1(a)) [10]. The product of such a reaction will be a graft block copolymer, which is able to

interact with nylon 66 and LCP at the interface, facilitating interfacial interaction between nylon 66 and LCP. Hence, it could be anticipated that, LCP fibrillation in toughened nylon/glass fiber/LCP hybrid composites could either be due to the compatibilization effect or because of the matrix viscosity enhancement of glass fibers.

In this paper, we will report a systematic rheological study to identify the roles of glass fibers and elastomers on LCP fibrillation in glass fiber filled-rubber toughened nylon hybrid composites. Rheological measurements were also carried out on untoughened hybrid composites to ascertain the role of elastomer on LCP fibrillation.

Broadly, three classes of blends were prepared each from toughened nylon and untoughened nylon. Denotations of blends are explained in Fig. 2 and their compositions are presented in Tables 1 and 2. LCP loading was fixed at 20 wt% in

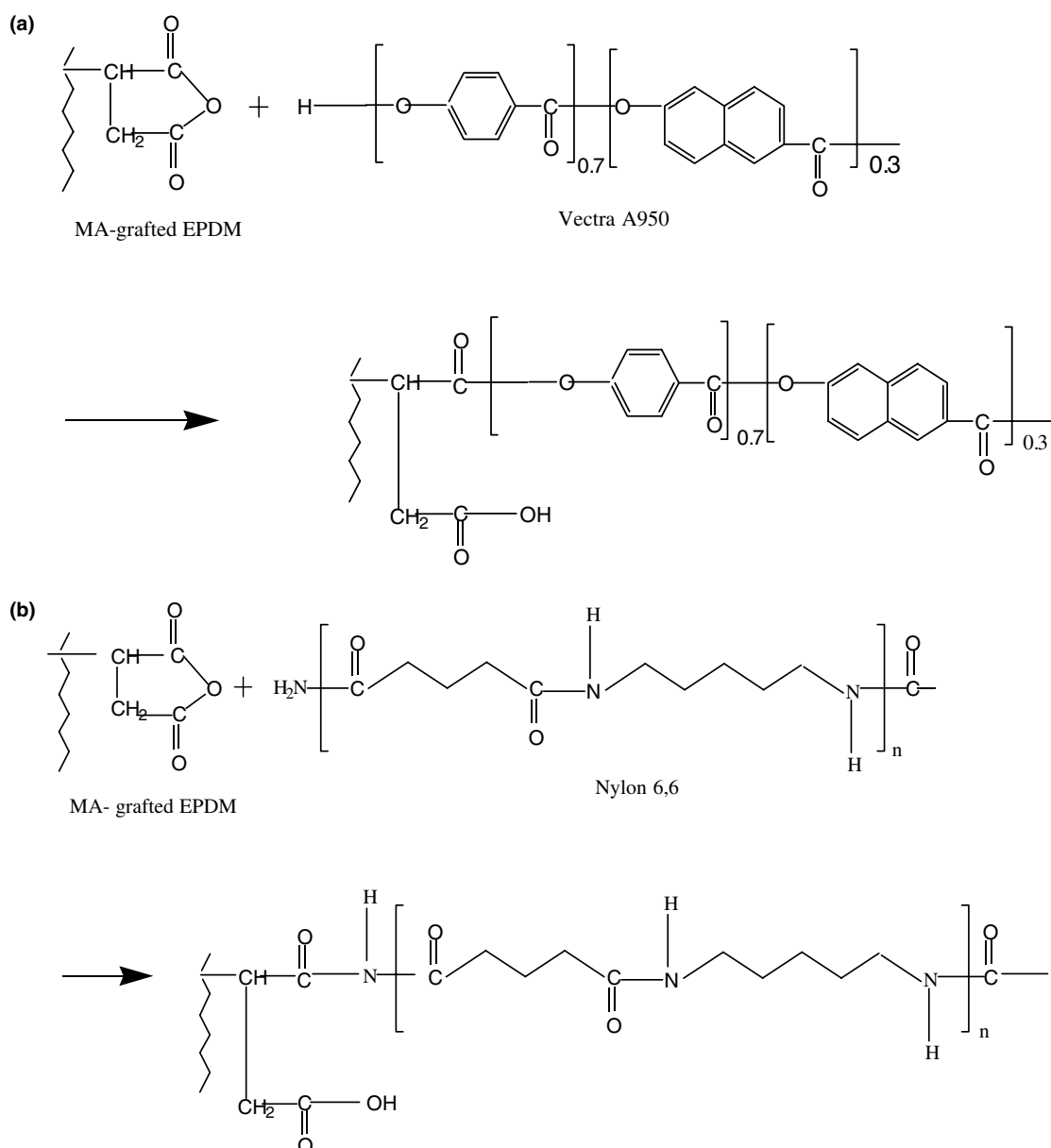


Fig. 1. Possible interfacial reaction between MA-grafted EPDM with (a) Vectra A(950); (b) nylon 66.

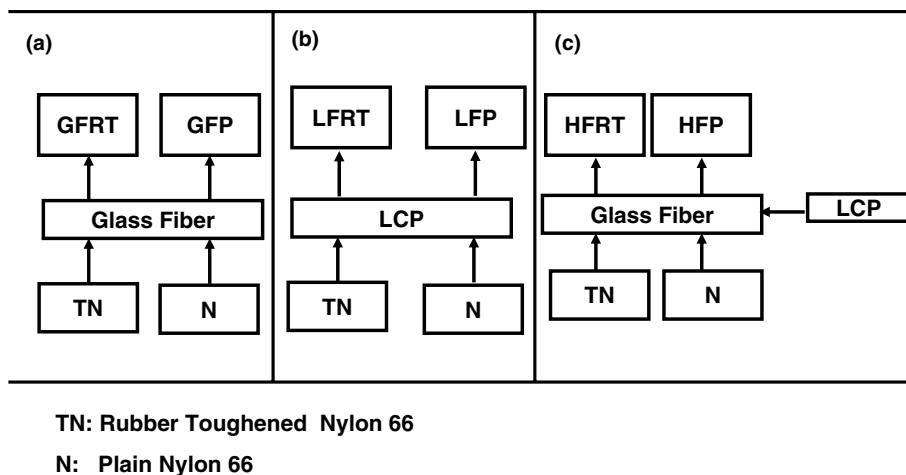


Fig. 2. Flow charts explaining the denotations of blend compositions used in this study: (a) blends containing only glass fiber; (b) blends containing only LCP; (c) blends containing both glass fiber and LCP.

Table 1  
Compositions of toughened hybrid materials

Sample name	Toughened nylon 66 (wt%)	LCP (wt%)	Glass fiber (wt%)
LFRT 20	80	20	0
GFRT 10	90	0	10
GFRT 20	80	0	20
HFRT 10	70	20	10
HFRT 20	60	20	20

Table 2  
Compositions of untoughened hybrid materials

Sample name	Nylon 66 (wt%)	LCP (wt%)	Glass fiber (wt%)
LFP 20	80	20	0
GFP 20	80	0	20
HFP 20	60	20	20

the blends. All the rheological results were correlated with the morphology of rheometer extrudates. Tensile properties were also evaluated to support the rheological results.

## 2. Experimental

### 2.1. Materials

The polymers used in this research were rubber toughened nylon 66 (Zytel ST801 from DuPont), plain nylon 66 (Zytel 101L from Dupont), and liquid crystalline polymer (LCP), (Vectra A950 from Hoechst Celanese). Zytel ST801 comprises nylon 66 with 20 vol% MA grafted EPDM rubber particles. Vectra A950 is made up of 27-mol% 2-hydroxy-6-naphthoic acid (HNA) and 73-mol% *p*-hydroxybenzoic acid (HBA). The short glass fibers used were E-glass fibers with a length of 12 mm and diameter of 17  $\mu\text{m}$ . However, the number average length is estimated as 300  $\mu\text{m}$  after processing. Detailed study on the fiber length distribution in the composites has been reported [3].

### 2.2. Specimen preparation

Blend compositions by weight are shown in Tables 1 and 2. Materials were pre-compounded using a high shear rate, inter-meshing, co-rotating twin-screw extruder (Leistritz Micro 18; with a screw diameter of 18 mm and  $L/D$  ratio = 30). In the extruder, barrel temperatures were set at 260/280/285/285/292  $^{\circ}\text{C}$  and a screw speed of 200 rpm was used. The dried extruded pellets were injection molded into 3.5 mm thick dog bone specimens using a Battenfeld BA 300 CD<sup>Plus</sup> injection-molding machine for tensile tests.

### 2.3. Rheological characterization

Rheological properties were measured using a Gottfert Rheograph 6000 capillary rheometer. The length and diameter of the capillary die were 30 and 1 mm ( $L/D$  ratio = 30), respectively. End corrections were not applied; hence the viscosity values obtained are apparent viscosities. Measurements were carried out at 290  $^{\circ}\text{C}$  over a range of shear rates of 50, 200, 500, 1000, and 3000  $\text{s}^{-1}$ .

### 2.4. Microscopy

The morphology of lateral cross sections of capillary rheometer extrudates were studied using a JEOL 5410LV scanning electron microscope (SEM). Extrudates collected at specified shear rates were cryogenically fractured after immersing them in liquid nitrogen for 20 min and the surfaces were coated with a thin layer of gold using an SPI sputter coater.

### 2.5. Tensile tests

Tensile tests were conducted according to ASTM D638 on 3.5 mm-thick dumbbell specimens with an Instron 5565 machine using a 30 kN load cell. Test speed was kept at 5 mm/min. An extensometer was used to precisely monitor

the elastic modulus. Values reported were averages of a minimum of five measurements.

### 3. Results

#### 3.1. Melt rheology

The flow curves of toughened hybrid composites containing 10 and 20 wt% glass fibers are shown in Fig. 3a and b. Viscosity of pure LCP is the highest of all the material compositions. In both the curves the in situ hybrid composites (HFRT 10 and HFRT 20) exhibit intermediate viscosities between that of glass fiber reinforced composites (GFRT 10 and GFRT 20) and rubber toughened nylon 66. Among the in situ hybrid composites, HFRT 20 is having a higher viscosity than that of HFRT 10. The composition consisting of only 20 wt% LCP, LFRT 20, exhibits the lowest viscosity of all the hybrid compositions. It is even lower than that of the plain rubber toughened nylon 66. The flow curves of untoughened hybrid composites are presented in Fig. 4. In contrast with the toughened systems, viscosity of untoughened in situ hybrid composite (HFP 20) containing both glass fibers and LCP is the lowest of all the studied compositions, even lower than that of the plain nylon 66/LCP binary blend.

#### 3.2. Morphology

Fig. 5a and b presents the SEM micrographs of lateral cross sections of rheometer extrudates of HFRT 20 and LFRT 20 collected at a shear rate of  $500 \text{ s}^{-1}$ . In HFRT 20 (Fig. 5a), the LCP phase has deformed into fine fibrous structures. In LFRT 20 (Fig. 5b), the LCP phase exists as spherical droplets. A cross sectional SEM micrograph of the rheometer extrudate of HFP 20 shows predominantly spherical droplets of LCP with a weak interface (Fig. 6).

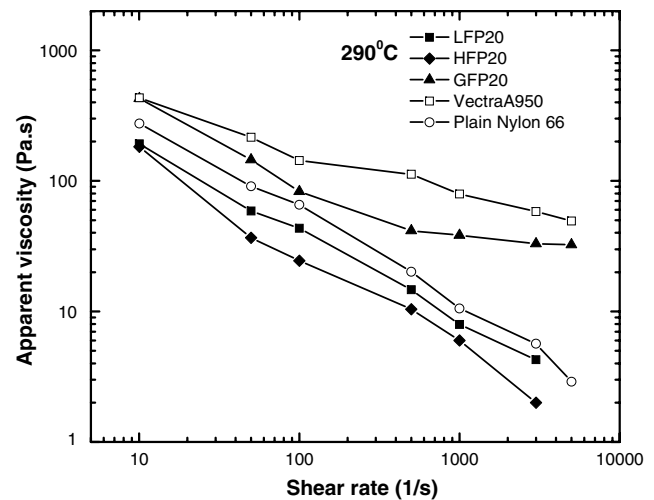


Fig. 4. Rheological curves for untoughened hybrid composites. The characterizations were carried out at 290 °C. HFP 20 shows the least viscosity.

#### 3.3. Tensile properties

The stress–strain behavior of toughened and untoughened hybrid composites is presented in Fig. 7a and b. Toughened hybrid composites gain in both strength and stiffness with the addition of glass fibers. LFRT 20 shows considerable elongation of 32% after achieving the peak stress. Peak stress was referred to the maximum stress attained by the specimen after whom the yielding process takes place. Young's modulus increases from 2.6 GPa in LFRT 20 to 5.2 GPa in HFRT 20. HFRT 20 shows a tensile strength of 70 MPa, which is 1.75 times that of LFRT 20 (40 MPa).

The overall strength of untoughened hybrid composites is higher than that of the toughened ones. Unlike the situation with toughened systems, HFP20, the composite containing both LCP and glass fibers, shows a lower

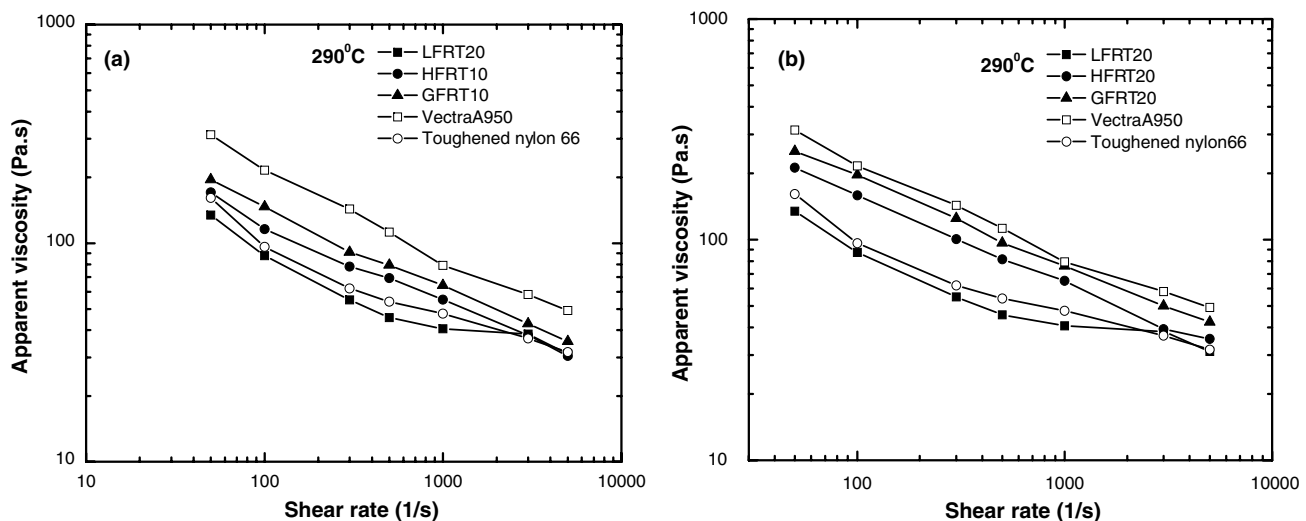


Fig. 3. Rheological curves for (a) toughened hybrid composites containing 10 wt% glass fiber (b) toughened hybrid composites containing 20 wt% glass fibers. The characterizations were carried out at 290 °C. HFRT 10 and HFRT 20 show intermediate viscosities.



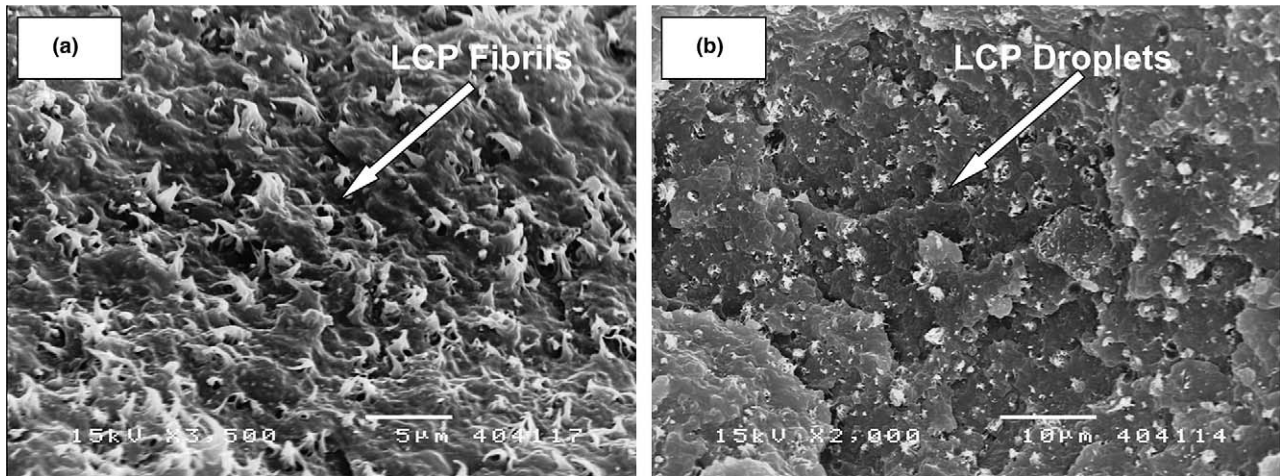


Fig. 5. SEM micrographs of rheometer extrudates of (a) HFRT 20 (b) LFRT 20. Extrudates were collected at shear rate of  $500 \text{ s}^{-1}$ . LCP phase exhibits fibrillar morphology in HFRT 20.

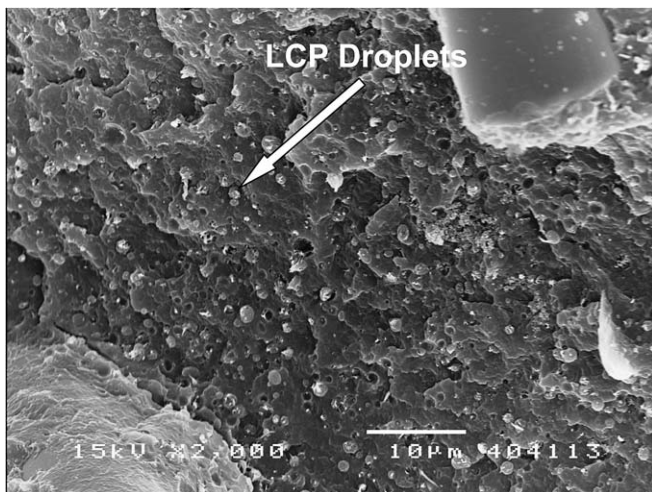


Fig. 6. SEM micrograph of the rheometer extrudate of HFP 20 collected at a shear rate of  $500 \text{ s}^{-1}$ .

strength of 60 MPa when compared to that of LFP 20 (65 MPa).

#### 4. Discussion

Capillary rheometry is a powerful tool to establish the flow characteristics of polymer blends, which is influenced by the deformation and interfacial compatibility of the constituent minor phase. Viscosity reduction phenomenon in thermoplastic/LCP blends could be ascribed to:

- Deformation of LCP phase into fibrils and orientation of deformed fibrils in the direction of flow.
- Interfacial slip due to incompatibility between the blend components.

Comparing the rheological results presented in Figs. 3 and 4, glass fiber addition has enhanced the matrix

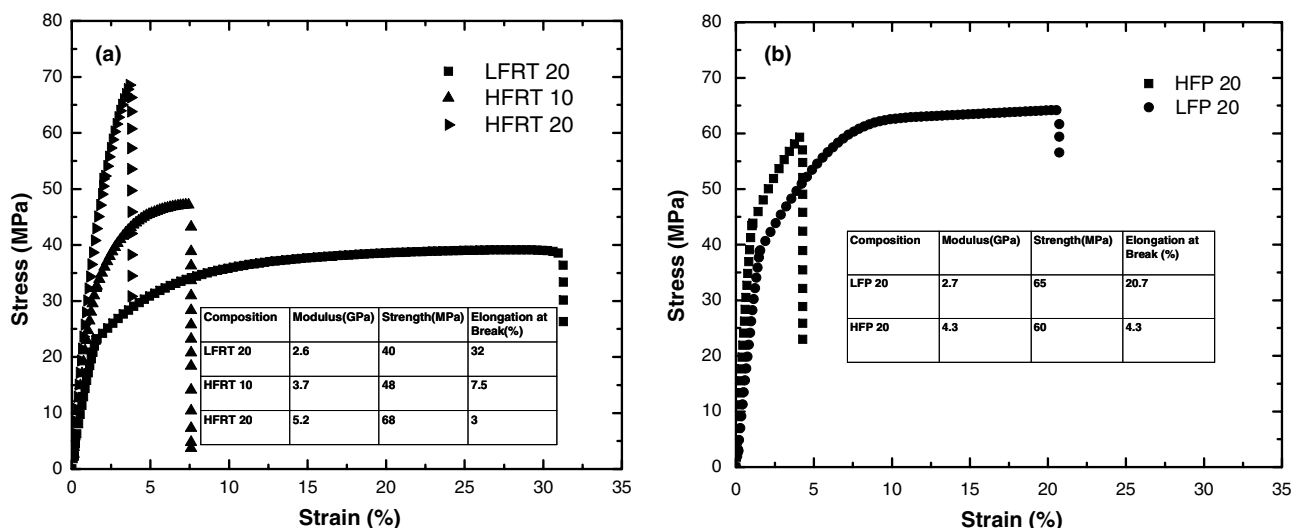


Fig. 7. Stress–strain curves of (a) toughened hybrid composites (b) untoughened hybrid composites.

viscosity of both toughened nylon and untoughened nylon. Hence, if the determining factor for the rheological behavior of HFRT 20 is the LCP fibrillation promoted by the viscosity enhancement effect of the matrix, HFP 20 should also exhibit a similar flow behavior. However, interestingly, HFRT 20 and HFP 20 exhibit different rheological behaviors. The only difference in the composition of HFRT 20 and HFP 20 is the absence of elastomer in the latter. This result implies that, rheology of toughened nylon hybrid composites is controlled by the LCP phase deformation induced by compatibilization effect of the elastomers present in toughened nylon.

Reactive compatibilization is a widely accepted strategy to improve the compatibility and thus the mechanical performance of immiscible polymer blends can be improved [11]. The extent of reactive compatibilization varies with the relative amount of reactive functional groups. In our situation, relative amount of toughened nylon 66 to that of LCP varies with the addition of glass fibers to LFRT 20. Hence, as we move from LFRT 20 composition to HFRT 20 composition, variations in degree of reactive compatibilization is expected. To assess this, we had performed a dynamic mechanical analysis on LFRT 20 and its glass fiber containing hybrid composites [3]. Results indicated that in LFRT 20, the elastomer phase is more miscible with the LCP phase. This leaves behind nylon 66

with little amount of compatibilizer to undergo reaction with LCP, resulting in poor compatibility between them. On the other hand, in HFRT 20, the elastomer content gets diluted with the addition of glass fibers, reducing its miscibility with LCP and providing better compatibilization action. Therefore, it could be concluded that, MA-grafted EPDM rubber in toughened nylon 66 promoted a better degree of compatibilization between nylon 66 and LCP in a hybrid system containing short glass fibers in comparison with the one without glass fibers. Fig. 8 presents the effect of different extent of compatibilization on the morphology of LCP phase of HFRT 20 and LFRT 20.

Phenomenon of interfacial slip [12] is a direct consequence of the interfacial incompatibility between the blend components. In incompatible polymers, the polymer chains are less entangled at the interface than that in the bulk, forming a lower viscosity region near the interface. When shear stresses are imposed parallel to the interface the resulting shear rate at the interface will be higher than that in the bulk phases. This difference is referred to as interfacial slip and the lower viscosity of the interfacial region reduces the apparent blend viscosity [13]. Consequently, among the toughened hybrid composites, the lowest viscosity of LFRT 20 could be attributed to the interfacial slip among the blend components due to weak interfacial compatibility. Addition of glass fibers to LFRT 20 improves

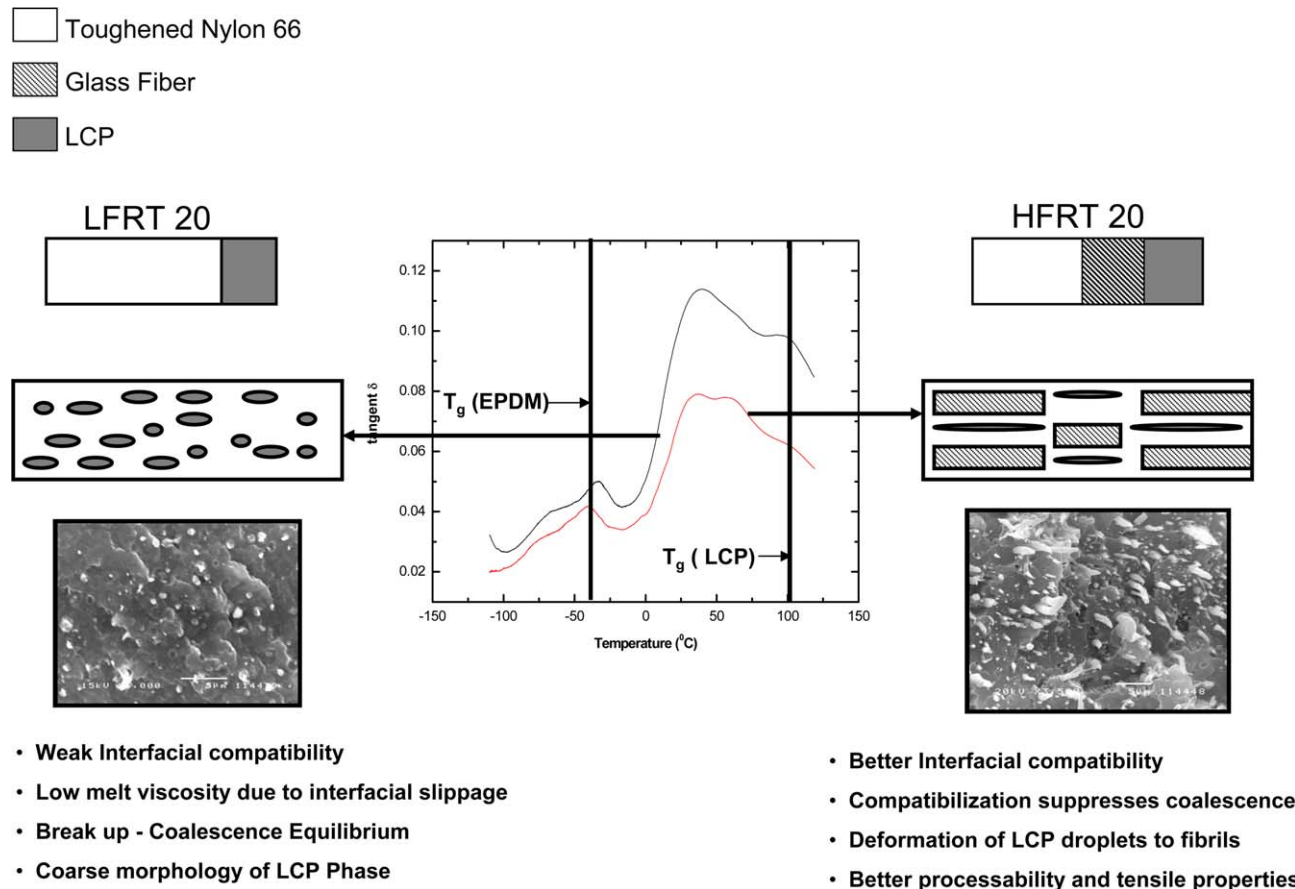


Fig. 8. Schematic explaining the effect of compatibilization on the morphology of LFRT 20 and HFRT 20.

the compatibilization action, thus increasing the interfacial width among the blend components. A wider interface promotes interpenetration of polymer chains across the interface [14]. This process suppresses the interfacial slip and increases the apparent blend viscosities of HFRT 10 and HFRT 20.

On the other hand, for the untoughened hybrid composites, in the absence of any compatibilization action, the addition of glass fibers to LFP 20 further deteriorates the interface leading to very low viscosity for HFP 20. It is interesting to note that, the magnitude of interfacial slip in HFP 20 dominates over the viscosity enhancement effect of glass fibers.

The difference in the interfacial compatibility is also reflected in the cross sectional SEM micrographs of rheometer extrudates of HFRT 20 and LFRT 20 presented in Fig. 5a and b. It is well known that, in melt blending operations, the dispersed phase morphology depends on the establishment of equilibrium between droplet break up and coalescence [15,16]. For incompatible polymer blends, the final particle size of the dispersed phase is coarse due to the prevalence of coalescence. In compatible systems, compatibilizer molecules act as springs at the interface, thus suppressing the coalescence phenomenon, resulting in a finer morphology of the dispersed phase.

In LFRT 20, where the interfacial compatibility between nylon 66 and LCP is weak, a weak interface leads to coalescence of LCP droplets resulting in larger domain size of LCP phase. Improved interfacial compatibility, between the blend components in HFRT 20, suppresses the coalescence resulting in a finer LCP phase. A stronger interface also provides an efficient stress transfer from the matrix to deform the LCP phase into thinner fibrils. The thin fibrils could orient in the direction of flow, which tends to lubricate the melt. When the LCP domains are oriented, interlayer slip takes place within the LCP phase leading to reduction in melt viscosity [17]. This is the possible reason for the lower melt viscosity of HFRT 20 compared to that of GFRT 20. Thus the morphological results are consistent with the conclusions on interfacial compatibility from rheological measurements.

The interfacial issues will be apparently reflected in the tensile properties. In a rigorous fracture mechanics characterization of hybrid composites [3], we observed that, in LFRT 20, weak interfacial compatibility between toughened nylon 66 and LCP promoted interfacial debonding. Function of interfacial debonding between the rigid phases [18] is analogous to rubber particle cavitation in rubber toughened polymers leading to ductile yielding. The cavitation process serves to relieve the hydrostatic tension around the flaws and promotes matrix yielding. This is the reason for LFRT's higher strain at break. For HFRT 20, the interfacial debonding mechanism is inhibited due to the stronger interface between toughened nylon 66 and LCP. This results in constrained plastic deformation and lower strain at break.

Modulus is directly proportional to the volume fraction of the fillers in the composite is independent of interfacial adhesion [19]. Hence, stiffness of both toughened and untoughened hybrid composites increases with the addition of high modulus glass fibers and LCP to the nylon matrix.

It is well known that tensile strength of toughened and untoughened nylon 66 could be improved by the addition of short glass fibers [20]. On the other hand, to obtain a synergistic performance in tensile strength from a LCP in situ hybrid composite, interfacial compatibility between the thermoplastic and LCP is also a critical parameter. In HFRT 20, both LCP phase and glass fibers could positively contribute to the overall tensile strength of the hybrid composite supported by better interfacial compatibility between toughened nylon 66 and LCP. However, in HFP 20, LCP phase negatively contribute to the overall tensile strength due to poor interfacial compatibility between nylon 66 and LCP. Poor interfacial compatibility leads to massive interfacial debonding leading to premature failure.

Table 3 compares and contrasts toughened and untoughened hybrid systems in terms of their differences in rheology, morphology and mechanical properties. Results indicate that, in toughened hybrid composites, glass fibers not only serve as inert fillers, but also help to establish an engineered interface between toughened nylon 66 and LCP, thereby providing desirable properties.

Table 3  
Comparison of toughened and untoughened hybrid systems

Hybrid system	Results			Conclusions
	Rheology	Microstructure	Mechanical	
Toughened	HFRT shows intermediate melt viscosity between that of GFRT and rubber toughened nylon 66	Fibrillar morphology of LCP phase due to effective interfacial stress transfer	High tensile strength and stiffness	Improved interfacial compatibility with the addition of glass fibers to LFRT
	LFRT shows the least melt viscosity among toughened hybrid composites	Droplet size morphology of LCP due to the predominance of coalescence	Low strength stiffness and higher strain at break	Weak interfacial compatibility between toughened nylon 66 and LCP
Untoughened	HFP shows the least melt viscosity among untoughened hybrid composites	LCP droplets with a weak interface	Low tensile strength and low strain at break due to massive interfacial debonding	Enhanced interfacial slippage with the addition of glass fibers to LFP



However, hybridization of untoughened nylon 66 and LCP with glass fibers leads to hybrid composites with poor tensile strength.

It is instructive to compare the hybridization method of addition of fillers to LCP systems based on high and low matrix viscosity polymers. For polymers with higher melt viscosities than LCP, addition of fillers is an effective strategy to induce LCP phase fibrillation and improving mechanical properties [7,21]. Our research indicates that, for polymers with low matrix viscosity, filler addition is not a successful hybridization strategy if not in the presence of an efficient reactive compatibilizer. Therefore, to effectively implement the concept of in situ hybrid composites onto polymers with low matrix viscosities, hybridization methods should be directed at improving the interfacial compatibility between the blend components.

## 5. Conclusions

A systematic rheological study was carried out to establish the roles of elastomer and glass fiber on the deformation of LCP domains in toughened nylon 66/glass fiber/LCP hybrid composites, the compositions of which are shown in Tables 1 and 2. Based on the presented results in this paper the following conclusions can be drawn:

1. In the toughened hybrid materials, hybrid compositions containing both LCP and glass fibers (HFRT 10 and HFRT 20) exhibited intermediate viscosities between that of glass fiber reinforced composites (GFRT 10 and GFRT 20) and pure rubber toughened nylon 66. A binary blend of toughened nylon 66 and LCP (LFRT 20) showed the lowest viscosity. The higher viscosities of HFRT 10 and HFRT 20 compared to LFRT 20 were ascribed to the improvement in interfacial compatibilities between toughened nylon 66 with the addition glass fibers as reported earlier [3]. On the other hand, for the untoughened system, composition containing both LCP and glass fiber (HFP 20) showed the least viscosity, which is even lower than that of the less viscous nylon 6,6/LCP binary blend (LFP 20). This observation was related to the interfacial slip phenomenon observed in uncompatibilized polymer blends. Rheological results clearly established that, compatibilization effect from MA-grafted elastomer phase in toughened nylon 66 is the primary factor for LCP deformation in toughened hybrid composites and the possibility of LCP deformation by the matrix viscosity enhancement effect of glass fibers was minimal.
2. Morphological results were consistent with the rheological predictions on interfacial compatibility of blend components. As a result of the improved compatibilization effect in HFRT 20, LCP domains were observed to deform to fibrillar structures with good interfacial adhesion between the component phases. However, in HFP 20, the LCP phase exhibited spherical domains with a weak interface resulting in interfacial slippage.

3. Toughened hybrid composites could gain from the reinforcement effect of both glass fibers and LCP supported by better compatibility between nylon and LCP in the presence of maleated elastomer and glass fibers. Nevertheless, for untoughened hybrid composites, the overall tensile strength deteriorates with the addition of glass fibers.

## Acknowledgement

The authors acknowledge the funding from the Nanyang Technological University through a Ph.D. scholarship for Sreekumar Pisharath.

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