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Frictional wear in high temperature thermotropic liquid crystalline polymers: correlation with glass transitions

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Abstract

Wholly aromatic thermotropic main chain liquid crystalline copolymers (LCPs) with varying glass transitions (T_g) were tested for wear resistance, particularly under high friction conditions, where surface temperatures can rise. Dynamic mechanical spectroscopy and DSC were used to characterize molecular relaxations. Three copolyester LCPs which all contain a substantial fraction of main chain 1,4-phenyl groups were chosen for this study. These included semi-crystalline Vectra® A900, a semi-crystalline LCP containing phenyl hydroquinone (phHQ–LCP), and a low crystallinity LCP containing *t*-butyl substituted hydroquinone (*t*-butylLCP). These have glass transitions of 100, 160 and 175 °C, respectively, and heat deflection temperatures (HDTs) of 170, 260 and 174 °C, respectively. HDT is dependent in part on crystallinity. The wear performance was found to depend mainly on T_g and not HDT, suggesting a microscopic failure mechanism related to the amorphous phase. This is supported by the relatively poor elevated temperature wear performance of Vectra® compared to the higher T_g LCPs. Shear strength measurements on the neat LCP resins did not correlate with wear properties of the blends, most likely because the measurements were made at room temperature and not elevated temperatures.

Keywords: Dynamic mechanical spectroscopy; Heat deflection temperature; Glass transition

1. Introduction

Our main impetus for characterizing the glass transitions and other properties of semi-crystalline Vectra® A900 (Hoechst-Celanese). phHQ–LCP, and 'amorphous' liquid crystalline polymers (LCPs) such as *t*-butylLCP, is an interest in understanding and defining subtle differences in the relaxation properties as these are related to elevated temperature wear properties. The systematic study of mechanical relaxation spectra also allows us to define important chain structural attributes in determining the structure property correlations of high temperature properties of these LCPs.

Under high pressure–velocity (*PV*) wear conditions, the surface temperature is raised to ca. 130 °C or more (Table 1). As the wear surface temperature is elevated, LCPs [1] and isotropic thermoplastics [2–4] show large differences in wear performance depending on their thermal properties such as glass transition temperature (T_g), crystallinity, and heat deflection temperature (HDT). The latter is affected by both T_g and crystallinity, in addition to reinforcing fillers. Isotropic thermoplastics such as high T_g semi-crystalline polymers like PEEK ($T_g = 150$ °C) show exceptional high *PV* wear performance [3]. High temperature wear performance is generally improved by fiber reinforcement due to improved 'thermal' resistance [3–5], although in LCPs wear performance is decreased by fiber fillers [4]. High T_g isotropic amorphous polymers such as bisphenol-A polycarbonate and Ultem® 1000 poly(ether imide) also show good wear performance as do low T_g-high crystallinity polymers such as poly(oxymethylene) [2]. While glass fiber, carbon fiber reinforcement, and other fillers or reinforcing agents significantly improve wear in isotropic polymers, only 'lubricants' such as Teflon® improve wear in thermoplastic LCPs [4,6]. Apparently adhesion is not sufficient with fibers and other fillers in LCPs [4], leading to accelerated damage rates due to debris in the wear zone. Thus, raising the HDT in LCPs by fiber reinforcement, does not necessarily improve high *PV* wear performance because

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Table 1
Wear and friction data for blends

LCP in blend	P (MPa)	V (m/min)	PV	$K \times 10^{-8}$ (cm^2/kg)	Co. F (d/s)	Surface temperature (°C)	HDT (°C)	T_m (°C)	T_g (°C)
Vectra® A900	2.76	15.2	42	68	0.17/0.07	81	170	280	100
	2.76	23.4	65	33,380	0.20/0.12	132			
t-butylLCP	2.76	15.2	42	31	0.17/0.12	85	174	none	175
	2.76	23.4	65	11	0.13/0.07	99			
	2.76	30.4	84	23	0.13/0.07	136			
	2.76	38.4	105	9578	0.17/0.10	153			
phHQ-LCP	2.76	23.4	65	12	0.12/0.09	99	260	305	160
	2.76	30.4	84	39	0.11/0.06	136			
	2.76	38.1	105	12,017	0.17/0.10	152			

P is the pressure and V is the velocity; K is the volumetric wear rate coefficient with the smaller values indicating lower wear rates (see text); Co. F: coefficients of friction in arbitrary units.

of competing mechanisms. For related reasons discussed below, crystallinity in LCPs can raise HDT, but crystallinity is only of secondary importance in improving wear performance under high PV wear conditions. Thus, LCPs are quite different in these important aspects as compared to isotropic polymers. The main advantages of LCPs are that they are available with high T_gs [1], yet still have relatively low melt viscosities and are easy to process.

Thermoplastic LCPs such as Vectra® are known to have good wear performance because of their low friction, high modulus, and high thermal properties [4,6]. Early studies have shown some of the novel wear properties of PTFE/LCP blends [7]. The non-crystalline or 'amorphous' LCP (*t*-butylLCP, Fig. 1) has unusually good wear properties under high PV wear conditions when blended with 30 wt% Teflon® micro-particles [1]. The wear properties of the two semi-crystalline LCPs, phHQ–LCP and Vectra® (Fig. 1), when blended with Teflon®, will also be discussed here. Teflon® is known to be a 'permanent' self-lubricant in these systems [1,4]. During the wear test it readily coats the contacting surfaces and reduces friction and wear.

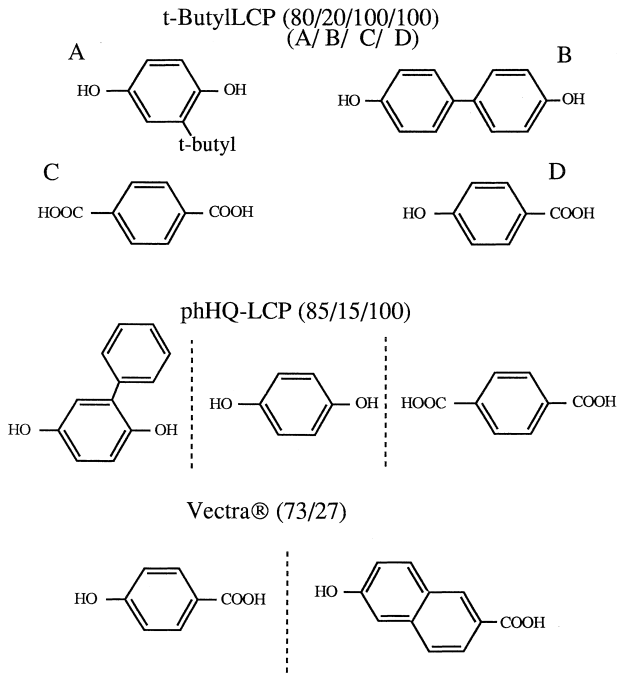

Fig. 1. Structures of the thermotropic LCPs.

2. Materials

The structures of *t*-butylLCP, phHQ–LCP, and Vectra® A900 are given in Fig. 1. Vectra® contains 1,4-hydroxybenzoic acid (HBA) and 6-hydoxy-2-naphthoic acid (HNA) in a 73/27 ratio. In addition to phenyl hydroquinone, phHQ–LCP also contains 1,4-dihydoxy benzene and terephthalic acid. In addition to *t*-butyl substituted hydroquinone, *t*-butylLCP contains biphenyl, terephthalic acid, and HBA (Fig. 1). For the wear data in Table 1, LCP blends with 30 wt% PTFE micro-particles (DuPont's Teflon® MP 1500 with average particle size of about 20 μm) were used. Because of the high density of PTFE, this corresponds to ca. 15 vol% fraction. Blends were prepared by compounding in a twin-screw extruder [1]. Injection molded 3.2 mm thick test bars were used in this study. The MP 1500 is expected to be an inert 'filler' in the LCP and has a melting point equal to that of PTFE (= 340 °C) which is above the LCP processing temperatures used. Thermal analysis and shear strength data were generally obtained for the neat LCP resins. In one case, representative DMA data are presented below for *t*-butylLCP/MP1500 blend.

3. Methods

Wear testing was carried out using a thrust bearing wear test: 6.35 mm square 'pins' were cut from the center edge of a 3.2 mm thick flex bar which had been injection molded. Three pins were mounted on a 3.18 cm circular holder spaced 120° apart, with the original longitudinal axis of the flex bar oriented tangentially to the circumference, as described previously [1]. The pins were loaded axially at

pressure P against a steel washer of the same outer diameter, and the washer rotated at a velocity V, measured at the mean diameter of the washer. The washer temperature was measured as the surface temperature of the LCP–PTFE blend. Also measured were the wear factor, K, defined as the volumetric wear rate divided by PV, and the stationary (s) and moving (d) coefficients of friction (Table 1). The lower the value of K, the better the wear resistance of the blend.

A TA Instruments 9900 tation with a DMA-983 module was used for the dynamical mechanical analysis (DMA) of the glass transition. Molded test bars (1/8″) were studied in the with the TA instrument. A polymer labs dynamical mechanical thermal analyzer (DMTA) was also used. DMTA measurements were obtained on 0.1–0.2 mm films pressed at 280–330 °C. Modulus (E') and dissipation factor ($\tan\delta = E''/E'$) were measured, where E'' is the mechanical loss. HDT measurements were obtained on flex bars of blends at 1.8 MPa load by a standard ASTM D648 deflection test.

The samples for the interface shear strengths using the micro-debond method [8] were prepared by dipping large diameter glass fibers into a very small disk of molten LCP at about 350 °C, and then cooling to allow the LCP to solidify. PTFE blends could not be used for this test, because the PTFE interfered with the adhesion of the LCP. The sample preparation method is a modified form of that used for the micro-bead-debond technique [9] and is designed for high temperature materials which do not flow well because of high viscosity [8]. A standard 'micro-vise' [9] and an Instron® were used to debond the samples at room temperature. The debond force (F) is converted to an apparent interface shear strength (τ) using; $\tau = F/A$, where A is equal to the imbedded length of fiber times the fiber circumference [8,9].

4. Results

4.1. Friction and wear results

The data in Table 1 indicate that t-butylLCP and phHQ–LCP, both with 30% MP 1500 Teflon® micro-particles, have the lowest wear factors, (K, in units of cm2/kg × 10^8), at high PV (in units of MPa m min^{-1}) values. Low values of K indicate good wear performance and the results in Table 1 show that the PV wear limits at $PV = 84$ have not been reached for t-butylLCP or phHQ–LCP, while by $PV = 65$ the wear limit of Vectra® has been reached as is indicated by the high values of K. The wear mechanism of these blends consists of failure of the LCP by surface macro-fibrillation [4] because of the well-known hierarchical structure of LCP organized in domains [10]. The shear forces in injection molding are known to induce macro-orientation of LCP fibrils. The surface temperature during the wear measurement was measured and is shown in Table 1. It is about 130 °C for the higher PV conditions (i.e. $PV = 65$), which is still appreciably below T_g of ca. 170 °C for t-butylLCP and phHQ–LCP (Table 1), while well above the T_g of Vectra® as will be discussed below. The difference is still surprising since the glass transition in Vectra® is weak and its HDT is substantially above 130 °C because of crystallinity, while the HDT is about equal to the glass transition for t-butylLCP (Table 1). phHQ–LCP has both high T_g and high HDT, the latter because of its crystallinity. The T_gs of both these are elevated over that of Vectra® because of side group substitution of the main-chain aromatic groups as will be discussed below. The high T_gs seem to be the unique feature which lead to the many-fold improved wear resistance under high PV conditions as compared to Vectra® blends with Teflon®.

4.2. Thermal analysis

4.2.1. DSC

The glass transition is detected by DSC for the non-crystalline t-butylLCP sample at 173 °C (Fig. 2). Hints of a small crystallization exotherm were seen at ca. 240 °C in this sample followed by a melting endotherm. Melting endotherms become more substantial upon annealing around 230 °C for tens of minutes (data not shown). Thus, a small amount of crystallization can be induced in t-butylLCP and this has consequences for the thermal and mechanical relaxation properties above T_g as compared to truly amorphous materials. The glass transitions in phHQ–LCP and Vectra® are broad and are detected by DSC at ca. 170 and 100 °C, respectively (Fig. 2). Weak and broad endotherms are also seen for phHQ–LCP and Vectra® with some indication of multiple melting. The percent crystallinity in unannealed Vectra® is believed to be of the order of 20–30% [11,12]. The value for the heat of fusion in LCPs is

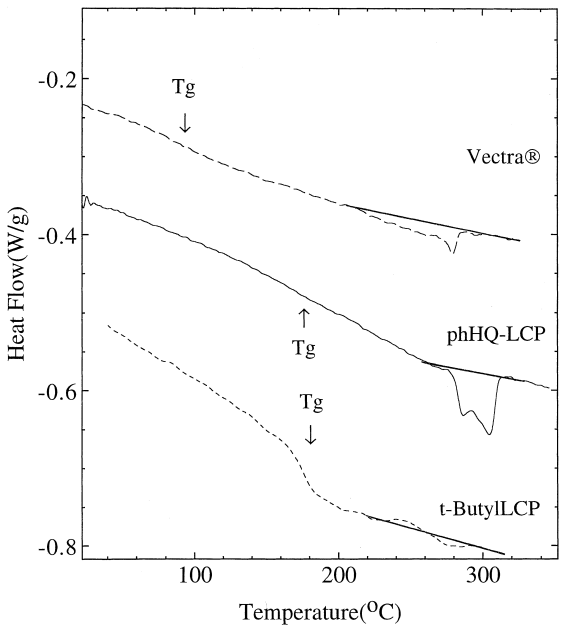

Fig. 2. DSC heating data at 10 °C/min for the indicated polymers.

typically low because of the small entropic change upon melting [11], i.e. the crystal to anisotropic melt transition involves only a small change in chain order or conformation as compared to melting of normal isotropic polymers. The crystals are also small and high in surface area [11] which leads to a highly constrained non-crystalline phase in these 'rigid' aromatic copolyesters. This is the cause of the broad and somewhat weak glass transitions even though the crystallinity is moderate.

4.2.2. DMA

Typical DMA results obtained for 1/8″ test bars for neat resins at 1 Hz are given in Fig. 3. Glass transitions for *t*-butylLCP and phHQ–LCP are seen at 175 and 163 °C, respectively. A smaller lower temperature peak is seen at ca. 75 °C for *t*-butylLCP in Fig. 3 which has been shown recently to be a very weak second glass transition [13]. This was discussed recently in a study of a series of related wholly aromatic copolyesters including *t*-butylLCP [13]. The multi-frequency DMTA data (Fig. 4) for thinner films of *t*-butylLCP, originally fast quenched from the melt at 310 °C, show a similar trend. The modulus drop above T_g is not as large as for typical amorphous polymers. The DSC results discussed above indicate that some crystallization occurs in the temperature region of 220 °C. Thus, a small degree of crystallization during cooling of the injection molded flex bar could explain the relatively high modulus

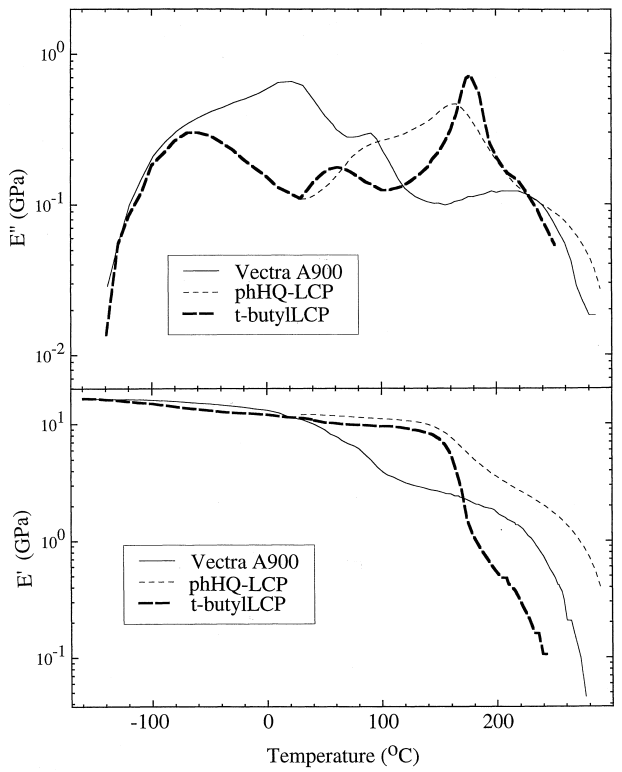

Fig. 3. DMA modulus (E'') and loss factor (E') taken at 1 Hz on 1/8″ test bars.

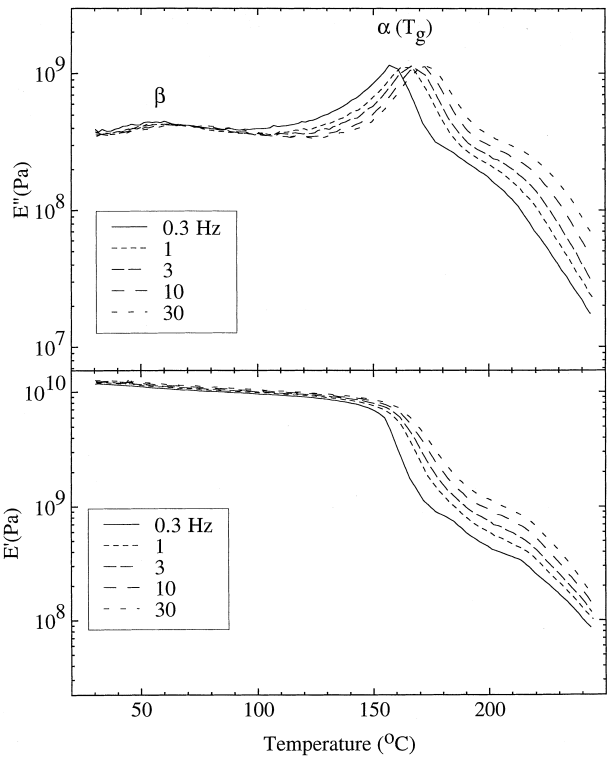

Fig. 4. Multi-frequency DMTA data for *t*-butylLCP on 0.2 mm thick films.

above T_g and the small shoulder on the E' data around this temperature (Fig. 3).

The Arrhenius relaxation map of the DMTA frequency vs $1/T_{max}$ data for *t*-butylLCP is given in Fig. 5, where the slopes are proportional to the apparent activation energies (E_a). The glass transition (α) is characterized by a high value of E_a of ca. 170 kcal/mol. The β relaxation in Fig. 5 is also characterized by a relatively high value of E_a of 56 kcal/mol. It has been proposed that this β transition is a weak second glass transition due to main chain motions of hydroxy benzoic acid (HBA) and/or biphenol rich segments in these chemically heterogeneous copolyesters [13]. The low T_g of these unsubstituted species is due to their low

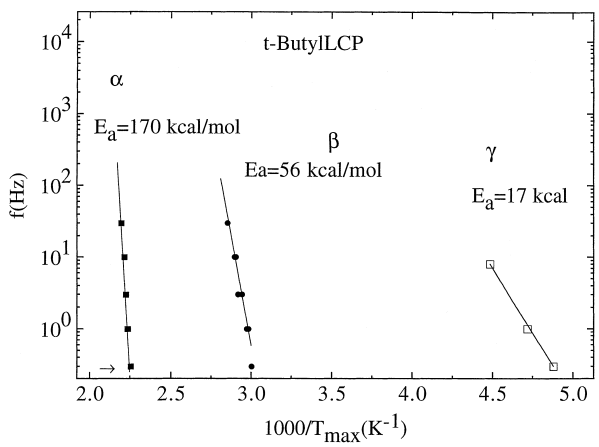

Fig. 5. Arrhenius relaxation map of frequency vs reciprocal peak transition temperature ($1/T_{max}$) for *t*-butylLCP.

rotational barriers, i.e. for the same reason Vectra® has a lower 'main' T_g because of its lack of vinyl substitution relative to *t*-butylLCP with its higher T_g.

The γ transition in *t*-butylLCP is a typical low activation energy local mode motion of the HBA related units. A comparable low activation energy γ transition is seen in Vectra® [14,15] although the γ transition is stronger dielectrically than mechanically [15]. An Arrhenius relaxation map has been given for Vectra® A900 previously [15].

Returning to the high temperature or premelting region, DSC shows that the melting endotherm extends down to temperatures of ca. 265 and 205 °C for phHQ–LCP and Vectra®, respectively. Corresponding sharp drops in modulus can be seen in the E' data (Fig. 3, bottom) for these two LCPs at about the same temperature, respectively. The tan δ plot (not shown) exhibits broad peaks in phHQ–LCP and Vectra® at ca. 260 and 210 °C, respectively. We have found that this DMA 'transition' (at low frequencies) roughly correlates with the HDTs which are 260 and 170 °C, respectively (Table 1). Thus, early melting of defective crystals has the important consequence of contributing to significant softening well before the main melting region.

The magnitudes of the modulus drops across the glass transition are compared in Table 2. The modulus drop can be represented as E'_0/E'_∞, where E'_0 is the low frequency (high temperature) modulus and E'_∞ is the high frequency (low temperature) modulus. The glass transition is mechanically the broadest in Vectra® and phHQ–LCP due to crystallinity. The data indicate that *t*-butylLCP shows more than an order of magnitude drop in modulus across its glass transition, as would be expected for this mostly amorphous material. This will be discussed further below, where it is found that the elevated temperature wear properties are more dependent on T_g than the strength of the glass transition.

The DMA data for 1/8″ test bars of neat *t*-butylLCP are compared in Fig. 6 with those for a blend of *t*-butylLCP/Teflon® (30 wt% or ca. 15 vol% MP1500 PTFE particles). The glass transitions are identical, indicating that there is no mixing of PTFE with LCP on a molecular scale. Within experimental error, both E' and E'' are quite similar in magnitude. To explain why the modulus is not decreased compared to the pure LCP at any temperature in Fig. 6, it is possible that the PTFE particles are relatively high modulus due to their high crystallinity. DSC measurements of the PTFE heat of fusion in the LCP/PTFE blend indicate that the crystallinity in the PTFE particles is about 60%.

Table 2
Strengths of the glass transitions determined by DMA

	E'_0 (GPa)	E'_∞ (GPa)	E'_0/E'_∞
t-butylLCP	4.45	0.33	13.6
phHQ–LCP	7.5	2.4	3.1
Vectra® A900	2.0	1.0	2.0

Estimations of the strength ($E'_0-E'_\infty$) of the glass transitions. The limiting low frequency (E'_0) and high frequency (E'_∞) values of the modulus, E' are given for the glass transition regions. Glass transitions are evaluated in the vicinity of ca. 175 °C for *t*-butylLCP, 160 °C for phHQ–LCP, and ca. 100 °C for Vectra®. There is also a weaker second glass transition at ca 70 °C in *t*-butylLCP which is not considered in this table.

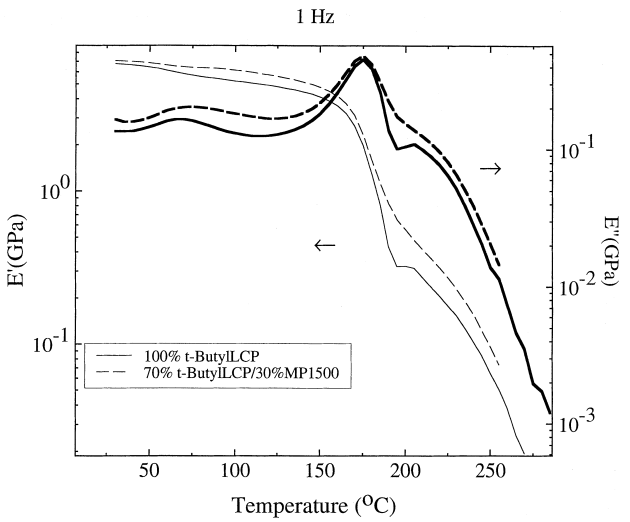

Fig. 6. DMA data for neat *t*-butylLCP and *t*-butylLCP with 30% Teflon® micro-particles (1 Hz).

4.3. Shear strength measurements

Because the wear mechanism consists of macro-fibrillation at high enough surface temperatures for injection molded LCPs including PTFE filled LCPs [4], it was suspected that the 'shear strength' of the LCP would follow the same trend as the wear data. 'Interface shear strengths' on the pure LCPs were measured at room temperature using the modified micro-debond method described previously [8]. It was shown that these interface shear strengths correlate with independently measured bulk transverse (transverse to flow, i.e. weak direction) shear strengths [8]. The measurement was not possible with the blends because the PTFE interfered with adhesion which was shown to be 'good' for the pure LCP resin [8]. Shear strength data taken at room temperature (Table 3) do not correlate with the wear tests. At high *PV* values, Vectra® is the poorest performer (highest *K* in Table 1), yet it has the highest interface shear strength amongst the LCPs. As was discussed above, Vectra® has a significantly lower T_g than the other two LCPs and this is likely the important factor in governing the wear performance. Shear strength measurements made at elevated temperatures should be more

Table 3
Interface shear strengths determined using micro-debond methods

	Interface shear strength, τ (MPa)
Vectra® A950	23 ± 2
Vectra® A900	24 ± 2
t-butylLCP	19 ± 3
phHQ–LCP	13 ± 2

relevant for the high *PV* wear tests at elevated temperatures but these data are not available.

5. Discussion

In the semi-crystalline LCPs such as Vectra® and phHQ–LCP, DMA [11,16] seems to be the most sensitive technique because of the low frequencies, lack of conductivity effects, and lack of overlapping relaxations, which tend complicate dielectrical spectra [13]. DSC can be used to study the glass transitions in some related semi-crystalline LCPs [17–22]. The origin of the weak and broad glass transitions in Vectra® and phHQ–LCP is the unique crystal morphology which consists of very fine, high surface area crystallites which restrict amorphous motions [11,19–22].

The rather low T_gs in LCPs such as Vectra® A900 containing a large fraction of unsubstituted main chain 1,4-phenyl groups (e.g. HBA), can be explained in terms of the low rotational barriers associated with the non-substituted aromatic ester species. The T_g is about 100 °C (Fig. 3) [11,15] for Vectra® and for other series of related LCPs. The 100 °C glass transition in Vectra® is characterized by a high apparent activation energy as was shown previously by low frequency dielectric [15] and DMA [13] studies consistent with a cooperative glass transition like motion. On the other hand, *t*-butylLCP has a high T_g because of the high rotational barriers associated with the *t*-butyl substitution of hydroquinone even though it is also rich in unsubstituted main chain phenyl groups (Fig. 1) [13].

The 100 °C glass transition for Vectra® is broad and relatively weak. One could expect that the crystalline network would improve thermal resistance in the context of elevated temperature wear properties. Crystallinity seems to have little effect. It is the low value of T_g which correlates with the decrease in high *PV* wear performance for the Teflon®/Vectra® blend at elevated surface temperatures. It is not unreasonable to think that softening and the corresponding weakening of the amorphous phase would promote macro-fibrillation at the oriented surface of these molded bars due to the decrease in shear strength of LCPs with temperature [4]. Other examples of high HDT, related semi-crystalline polymers with rather low T_gs of around 120 °C have been given and the wear results are consistent with those for Vectra® [1]. For Vectra®, the surface temperature is measured to be ca. 130 °C at $PV = 65$. Under these conditions, the *PV* limit of the Vectra® blend has been exceeded (Table 1) leading to large values of *K* which represent poor wear behavior. The amorphous *t*-butylLCP and the semi-crystalline phHQ–LCP when blended with Teflon® show exceptional performance at these elevated *PV* wear conditions. They have DMA glass transitions of 175 and 160 °C, respectively (Table 1). Although the T_g of Vectra® is low, the HDT of 170 °C is relatively high due to reinforcement by the crystal network. One may have expected that crystallinity combined with the weak glass transition would make the HDT more important than T_g. The HDT of 173 °C for *t*-butylLCP is about the same, yet the elevated temperature high *PV* wear limit is much higher because of its higher T_g. phHQ–LCP has both a high T_g and a high HDT explaining its excellent high temperature wear properties (Table 1). The HDT is high because of its crystallinity but its wear limit at surface temperatures of 153 °C ($PV = 105$, Table 1) is closer to its T_g.

We have shown by DMA (e.g. Fig. 6) that there is no molecular mixing of the inert-highly crystalline Teflon® particles with the LCPs. Therefore, this does not contribute to variations in wear properties between the different LCPs. The stainless steel wear surfaces and the polymer surface both are found to have a thin lubrication layer of PFTE as the wear process progresses. This would obviously lower friction and also significantly reduces the tendency for surface fibrillation [4]. Another contribution is the change in the macrostructure of the LCP domains in these thermotropic LCPs [10]. Fillers are known to reduce the anisotropy of the liquid crystalline domains [4,6] and the Teflon® micro-particles should disrupt the anisotropy and the level of macro-fibril LCP orientation at the surface. This could reduce the tendency for the LCP surface to fibrillate during the wear test. The modification in LCP domain structure by Teflon® particles could be different for the various LCPs, but we have no evidence of such variations in wear performance from material to material due to domain disruption.

6. Conclusions

Dynamic mechanical spectroscopy (DMA) and DSC were used to characterize molecular relaxations and the effects of the level of crystallinity and the breadth of the transitions. Main chain LCPs studied here included semi-crystalline Vectra® A900, another semi-crystalline LCP containing phenyl hydroquinone (phHQ–LCP), and a very low crystallinity LCP containing *t*-butyl substituted hydroquinone (*t*-butylLCP) with glass transitions of 100, 160, and 175 °C, respectively, and HDTs of 170, 260, and 174 °C, respectively. The wear performance of injection molded test bars was significantly better for *t*-butylLCP compared to Vectra® even though the former is non-crystalline. The results suggest that even though the semi-crystalline network in Vectra® contributes to a HDT significantly higher than its T_g, under elevated temperature, wear conditions the crystal network cannot prevent surface defibrillation. Thus, the wear performance was found to be dependent mainly on T_g and not HDT. The results suggest a microscopic failure mechanism related to softening of the amorphous phase leading to reduced shear strength and macro-defibrillation at elevated temperatures.

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