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# High-performance Composites Based on All-aromatic Liquid Crystal Thermosets

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#### **Abstract**

In this study, a new high-performance liquid crystal ester-based thermoset for composite applications was investigated. All-aromatic liquid crystalline thermosets (LCTs) are a promising class of polymers that offer a unique combination of properties such as solvent resistivity, high modulus, high strength, low coefficient of thermal expansion and high after-cure glass-transition temperatures ( $T_g \ge 150$  °C). Fully cured LCTs offer superior thermo-mechanical properties over high-performance thermoplastic polymers such as PPS, PEEK and PEI. For this study we used a 9000 g.mol<sup>-1</sup> ester-based LCT based on cheap and readily available monomers, i.e. 4-hydroxybenzoic acid (H), isophthalic acid (I) and hydroquinone (Q), abbreviated by us as HIQ-9. Composite panels prepared from T300 carbon fiber (5-harness satin weave) showed in-plane shear strength of 154 MPa and an in-plane shear modulus of 3.7 GPa. The tensile strength and modulus were measured to be 696 MPa and 57 GPa, respectively. A post-mortem inspection showed that the interfacial strength was excellent and no delamination was observed in the test specimen. Preliminary results show that LCT based composites exhibit a better combination of (thermo)mechanical properties over PPS and PEI based composites.

#### 1. Introduction

The demand for light-weight structures in the aerospace and automobile industry has increased considerably over the years. Due to their high specific strength and stiffness, fiber-reinforced composites have become an important class of structural materials. Fiber-reinforced composites based on thermoplastic polymer matrixes are now in use for more than twenty years. A special class of fiber-reinforced thermoplastic composites is based on all-aromatic semi-crystalline polymers such as PPS, PEEK and PEI. They offer several desirable properties such as unlimited shelf life, low manufacturing costs and can be used in all but aggressive chemical environments.[1-4] Despite these properties they also have several short comings. Due to the semi-crystalline nature of these polymers the thermal history has a pronounced effect on the degree of crystallinity, which consequently has an effect on the overall mechanical properties of the final composite.[5] The microstructure, or the morphology of semi-crystalline thermoplastic composites, is highly dependent on the applied processing parameters.[6] In thick laminates, for example, the outer layers will cool at a much faster rate while the layers at the center of the composite will cool at a lower rate. This results in thermal stresses at the fiber resin interface, which again weakens the final composite structure.[7] Another point of concern is the low glass-transition temperature (Tg) of most all-aromatic commercial thermoplastics. Carbon-fiber based composites based on PPS (T<sub>g</sub> = 85 °C) loose 60% of their flexural strength at 140 °C while the in-plane shear strength decreases already at 45 °C, which is far below T<sub>g</sub>. A similar effect was also observed for PEEK-based composites.[8] Amorphous high T<sub>g</sub> poly(ether imide)s (PEIs) and thermosetting PEIs, on the other hand, offer advantages for high temperature applications but their use is limited due to high cost and processability issues.[9]

In order to provide a useful alternative we are currently exploring all-aromatic liquid crystal thermosets, which can be processed as thermoplastic polymers and cured into so-called liquid crystal networks or liquid crystal thermosets (LCTs).[10-12] An important advantage is that our LCT concept could be useful for the production of thick composites structures because in fully cured LCTs the molecules are closely packed, which is a consequence of liquid crystallinity, but lack crystallinity, which we anticipate, will result in lower thermal stresses in the final composite structure. LCTs combine both the advantages of thermoplastic liquid crystalline polymers as well as that of thermosetting polymers. The advantages include improved processability, high glass transition temperatures, excellent thermal stability, high strength and stiffness, outstanding interfacial properties and low shrinkage after curing. Herein we will present the properties of composites based on T300 Carbon fibers (5HS) and a 9000 g.mol<sup>-1</sup> reactive random co-polymer based on 4-hydroxybenzoic acid, isophthalic acid and hydroquinone, labeled by us as HIQ-9. Thermal, mechanical and interfacial properties were investigated and will be discussed.

## 2. Experimental Methods

## 2.1 Materials

Carbon fabric T300 (5 HS) with areal density of 280 g/m $^2$  and thickness of 0.35 mm was provided by Ten Cate Composites and the LCT precursor HIQ-9 was synthesized in our laboratory. The synthetic details can be found elsewhere.[13] The reactive liquid crystal oligomer used for this study has a maximum after-cure  $T_g$  of 190

°C, which depends on the applied temperature-time (T-t) cure profile. The structure of the reactive HIQ-9 oligomer is shown below.

## 2.2 Composite Manufacturing

Laminates (6-plies, 40 x 40 cm) were prepared using a simple resin film infusion process. A 5-harness satin weave T300 Carbon fabric was selected and the reactive oligomer was distributed evenly between the individual layers. The resin-fiber content at this point was 40/60 (40 wt% resin and 60 wt% fiber). The whole assembly was placed in a Joos press and heated to 350 °C at a heating rate of 9 °C.min<sup>-1</sup> and at a pressure of 2 bar. At 350 °C, the pressure was increased to 10 bar and the composite was kept at this temperature and pressure for 15 min, followed by increasing the temperature to 370 °C and a pressure of 10 bar for 30 min. The complete curing cycle will be discussed in more detail in section 3. To study the effect of curing on thermal and mechanical properties neat resin thin films were also cured at different temperatures *i.e* 340, 370 and 400 °C.

## 2.3 Analysis

A Leica DMLM optical microscope equipped with a hot stage was used to investigate the oligomer melt behavior. A powder sample was investigated between two glass slides and heated to 450 °C. Optical microscopy was also used to investigate the

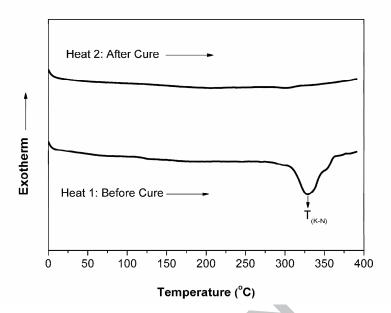
void content in the fully cured T-300/HIQ-9 composite specimen. A Perkin-Elmer Sapphire differential scanning calorimeter (DSC) and thermogravimetric analyzer (TGA) were used to investigate the thermal properties of the reactive oligomers and their fully cured thermosets. The heating and cooling rates used were 20 °C.min<sup>-1</sup> for DSC and 10 °C.min<sup>-1</sup> for TGA experiments. All DSC experiments were performed under a nitrogen atmosphere whereas all TGA experiments were conducted in the presence of nitrogen or air. LCT samples were cured at 370 °C for 45 min. in a nitrogen atmosphere and cooled to room temperature before measurement. The dynamic mechanical analysis (DMTA) was performed using a PerkinElmer Diamond dynamic mechanical thermal analyzer (DMTA) under a nitrogen atmosphere and at a heating rate of 2 °C.min<sup>-1</sup>. The tensile and in-plane shear tests were performed on a Zwick 250 kN machine according to ASTM standards (Tensile D 3039, In-plane shear D3518 and Flexural D7264). The flexural tests were performed on a Zwick 20 kN machine. Five specimens were used for each test. A Jeol JSM-7500F, high resolution scanning electron microscopy (HRSEM) was used to study the composite fracture surfaces after failure. A Midas 3 x 1.5m single throughtransmission ultrasonic transmission scanner with a probe diameter of 10 mm and a natural focusing transducer was used to inspect the defects in the laminates and density variations at a frequency of 10 MHz.

## 3. Results and Discussion

## 3.1 Thermal and thermo-mechanical properties of HIQ-9

In order to investigate the thermal behavior of the reactive HIQ-9 oligomer we used a differential scanning calorimeter (DSC). The samples were heated to 370 °C at a

heating rate of 20 °C.min<sup>-1</sup> and isothermally cured at this temperature to allow chain extension/crosslinking to take place via the reactive end-groups. After a 45 min. hold the sample was quenched and cooled to room temperature, followed by reheating from room. temperature to 400 °C. In the first heating cycle a melt transition (T<sub>K-N</sub>) was observed. In the second heating cycle no melt transitions were detected, which is a strong indication that the resulting polymer was fully crosslinked. The DSC heating traces of the HIQ-9 oligomer, before and after cure, are shown in Figure 1. Thermo gravimetric analysis (TGA) was used to investigate the thermal stability and decomposition temperatures. A heating rate of 10 °C.min<sup>-1</sup> was used for these measurements. The thermal stability was evaluated in terms of 5% weight loss (T<sub>d</sub><sup>5%</sup>) both in air and nitrogen. HIQ-9 showed excellent thermal stabilities (> 400 °C) both in air and nitrogen. The storage modulus (E') and glass transition temperatures were measured using dynamic mechanical thermal analysis (DMTA) in the temperature range of -100 to 500 °C at 1Hz. The fully cured thermosets exhibit high glass-transition temperatures and show excellent moduli at elevated temperatures. With the reactive oligomer approach, we achieved a significant improvement in the glass transition temperature as compared to commercially available LCPs such as Vectra,  $T_g = 110$  °C.[11] and other well-known high-performance polymers such as PPS. The thermal and thermo-mechanical properties of HIQ-9 are summarized in Table 1.



**Figure 1.** DSC thermogram of the HIQ-9 oligomer measured at a heating rate of 20  $^{\circ}$ C.min<sup>-1</sup> under a nitrogen atmosphere. The lower curve represent the first heating cycle, showing a melting endotherm ( $T_{K-N}$ ). The upper curve represent the second heat after curing and is consistent with a nematic thermoset.

**Table 1.** Thermal properties of a fully cured HIQ-9 liquid crystal thermoset film.

Sample	*T <sub>m</sub> (°C)	**T <sub>d</sub> (°C) N <sub>2</sub> /Air		E' (GPa) at 100 °C		***T <sub>g</sub> (°C)
HIQ-9	330	466/427	3.3	2.4	0.07	189

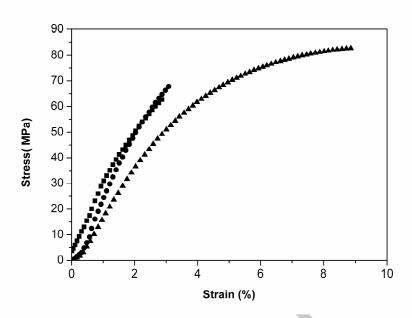
<sup>\*</sup> Tm as measured before cure (DSC, first heat at 20 °C.min<sup>-1</sup>)

<sup>\*\* 5%</sup> weight loss (TGA, first heat at 10 °C.min<sup>-1</sup>)

<sup>\*\*\*</sup> $T_g$  taken at maximum of Tan  $\delta$  (DMTA, 2  $^o\!C.min^{\text{--}1}\!)$ 

## 3.2 Mechanical Properties of Neat Resins.

The effect of curing temperature on the thermal and mechanical properties of neat HIQ-9 films was investigated by preparing three films, each cured at a different temperature, i.e. 340, 370 and 400 °C. Room temperature tensile tests were performed to determine the stress-strain behavior of these LCT films. It was shown by Roberts et al. that phenylethynyl end-capped oligomers chain-extend below 350 °C while above 350 °C crosslinking becomes the dominating reaction.[14] A tensile strength of 83 MPa and elongation at break of ~10% were achieved by curing the LCT at 370 °C for 45 minutes. PPS and PEI have similar tensile properties but their elongation at break is significantly less, i.e. 3% and 7% respectively.[15] When we cured the reactive oligomer at a temperature above or below 370 °C, a decrease in the tensile properties was observed, i.e. a tensile strength of 69 and 64 MPa was obtained for the curing temperature of 340 and 400 °C, respectively. The elongation at break also decreased to ~2% when the polymer was cured at 400 °C and is due to a high crosslink density. Based on these results the curing temperature for this HIQ-9 oligomer was set at 370 °C for our initial composite fabrication trials. The stress-strain behavior of the thin films cured at the different temperatures is shown in Figure 2.

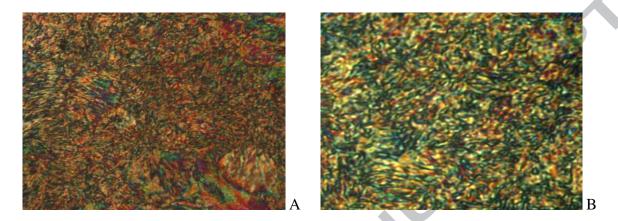


**Figure 2.** Tensile properties of HIQ-9 -based thin films cured at different temperatures for 45 min., *i.e.* at 340 °C ( $\bullet$ ), 370 °C ( $\blacktriangle$ ) and 400 °C ( $\blacksquare$ ).

## 3.3 Melt Behavior by Polarized Optical Microscopy.

Polarizing optical microscopy was used to study the phase behavior of this reactive oligomer and its cured polymer. Since HIQ-9 is a liquid crystal oligomer it will display a birefringent melt when investigated under crossed polarizers whereas the melt of a classic thermoplastic polymer, such as PEEK or PPS, will show no birefringence. The HIQ-9 oligomer displays a classic nematic texture over a broad temperature range. The nematic phase stability of this oligomer was excellent and no nematic-to-isotropic transition (N-I) could be observed. When this oligomer was heated and cured above 370 °C, the viscosity of the melt started to increase and solidified after a 45 min. hold at this temperature exhibiting a fixed nematic texture. At this point it was no longer possible to

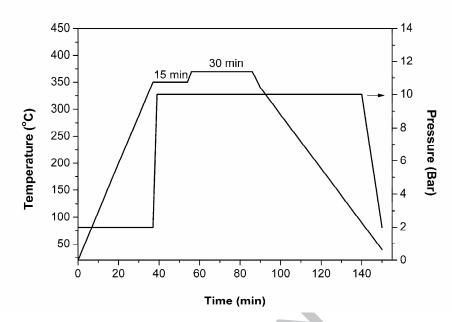
shear the sample and the texture observed remained stable till decomposition (> 400 °C). The nematic texture of HIQ-9 before and after cure is shown in Figure 3.



**Figure 3.** Microphotographs of HIQ-9. *A*- low viscous nematic melt of the oligomer at 370 °C, *B*- Solidified nematic thermoset after a 45 min. cure at 370 °C.

## 3.4 Curing of Composites.

A typical cure cycle for a 6 ply T-300/HIQ-9 composite is shown in Figure 4. The first ramp and isothermal hold at 350 °C and a pressure of 10 bar is used for resin infiltration and allows trapped gasses to escape. Polymerization will take place, albeit at a low rate. After 15 min the cure temperature is increased to 370 °C and the composite is kept at this temperature for 30 min to allow full polymerization to take place. Applied heating and cooling rates were 9 and 5 °C.min<sup>-1</sup>, respectively. There are no volatiles or out gassing associated with the curing of the reactive HIQ-9 oligomer. The curing cycle used for HIQ-9 is practically the same as applied for consolidating composites based on high-performance thermoplastics such as PPS and PEEK.

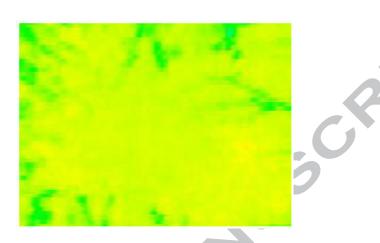


**Figure 4.** Typical curing cycle for a 6-ply T-300/HIQ-9 composite. Resin infiltration takes place at 350 °C and 10 bar followed by a 30 min consolidation step at 370 °C to complete the polymerization

## 3.5 Ultrasonic C-Scan Analysis.

The laminates consolidated according to the curing cycle discussed above were inspected using a C-scan apparatus. In order to achieve the best possible composite properties the composite needs to be well consolidated and void free. In thermosetting polymers, pressure, temperature and time are important parameters. Voids are usually formed during processing and can be reduced by using appropriate consolidation conditions.[16] Initial results showed that the laminates prepared according to the processing cycle shown in Figure 4 had some variation in density. Figure 5 shows a C-scan of one of our best composite panels after cure and the scan clearly shows the presence of voids, green areas in Figure 5, and this void content was calculated to be ~4

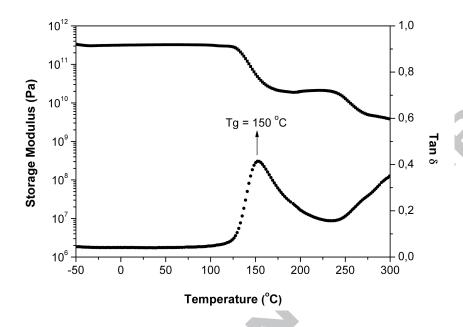
% by the threshold method where attenuation of the reference plus 6 dB was considered as a defect or indicating the presence of micro-voids.[17, 18]



**Figure 5.** C-Scan of a T-300/HIQ-9, 6-ply laminate, processed according to the curing cycle as shown in Figure 4. The yellow area's are well consolidated whereas the green area's are indicative of voids present in the composite panel.

## 3.6 Dynamic Mechanical and Thermal Analysis (DMTA).

The storage modulus (E') of the T-300/HIQ-9 composites was measured as a function of temperature using DMTA at a heating rate of 2 °C.min<sup>-1</sup>. The fully cured composite showed a high initial storage modulus, which is typical for an all-aromatic resin/carbon fiber composite. The storage modulus remains fairly constant up to 150 °C, the apparent  $T_g$  of HIQ-9, after which the rubber plateau is reached with a modulus of 21 GPa. The storage modulus and Tan  $\delta$  as a function of temperature are shown in Figure 6.



**Figure 6.** ( $\blacksquare$ )The storage modulus (E') and ( $\bullet$ ) Tan  $\delta$  of a fully cured T-300/HIQ-9 composite as measured by DMTA using a heating rate of 2 °C.min<sup>-1</sup> and measured at 1 Hz.

## 3.7 Mechanical Properties of T-300/HIQ-9 based Composites.

In order to investigate whether LCTs are usefulness composite resins we prepared several 6-ply, 40 x 40 cm panels, using a standard T300 carbon fiber fabric (5HS) and our HIQ-9 resin. Composite panels prepared from this LCT showed a tensile strength of 696 MPa and a tensile modulus of 57 GPa. When the tensile properties of LCT based composites were compared with that of high-performance composites based on PPS and PEI, we found that the tensile strength and tensile modulus are better than PPS or PEI composites based on the same fiber, fabric and resin content.[15] An in-plane shear strength and modulus were measured to be 154 MPa and 3.7 GPa respectively. The in-

plane shear strength of LCT based composites is significantly higher, *i.e.* 154 MPa as compared to 119 MPa for PPS and 118 MPa for PEI based composites.[15] The in-plane shear strength depends mainly on the fiber matrix adhesion and the properties of the neat resin. During these tests no delamination was observed, which suggests a high interfacial LCT/C-fiber strength. The flexural strength and modulus of T-300/HIQ-9 composites was found to be 817 MPa and 58 GPa, respectively. When these values were compared with PPS and PEI based composites, it was observed that the flexural modulus of the LCT-based composites is similar or better than that of PPS and PEI-based composites. The flexural strength of LCT-based composites is relatively low, which can be attributed to the presence of voids in the laminates.[16] Based on the encouraging room temperature properties, we performed tensile tests at 150 °C, which is close to the polymer glass transition temperature. The results showed that the composite can retain more than 75% of its room temperature strength at this temperature. The composite properties are summarized in Table 2.

**Table 2.** Mechanical properties of a 6-ply T-300/HIQ-9 composite. PPS and PEI-based composites are shown for reference purposes.

	T-300/HIQ-9	Carbon/ PPS*	Carbon/ PEI*
Tensile Strength (MPa)	696	592#	656
Tensile Strength (MPa) at 150 °C	527	-	
Tensile Modulus (MPa)	57	54	56
In-Plane shear Strength (MPa)	154	119	118
In-Plane shear modulus (GPa)	3.7	4.04	3.3
Flexural Strength (MPa)	817	1027	870
Flexural Modulus (GPa))	58	60	50

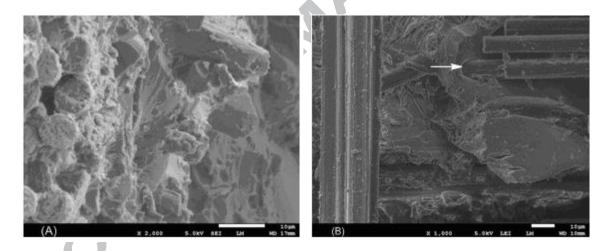
data provided by TenCate[15]

## 3.8 Fracture behavior of the composites

The mechanical performance of fiber-reinforced composites is directly related to the quality of the fiber-resin interface.[20] High-resolution scanning electron microscopy (HRSEM) analysis was used to inspect the composite fracture surfaces after tensile and compression failure. Franco *et. al.* reported that the presence of clean, resin free, fibers at the fracture area is an indication that composite failure has occurred at the fiber/matrix interface.[21] No clean fibers were found in the T-300/HIQ-9 test specimen after tensile experiments. From Figure 7 it is clear that failure occurred mainly in the fibers and that

<sup>\*</sup> Pantelakis et.al. [19]

the fibers remain present in the form of bundles. At the fracture interface the carbon fibers are still surrounded by the polymer matrix. The same was observed when we inspected fractured tensile specimen tested at 150 °C. During both tensile tests, i.e. at room temperature and at 150 °C, no delamination was observed. These results are consistent with strong resin-fiber interfacial bonding, which is supported by the high inplane shear strength found for these composites. The HRSEM micrographs of the fracture surfaces are shown in Figure 7A and 7B. Kline *et.al.* showed that during compression tests, graphite epoxy specimens failed due to delamination.[22] Similar compression tests performed by us showed that there were only very few cracks in the specimen but no clear signs of delamination.



**Figure 7**. Scanning electron micrograph (HRSEM) of a T-300/HIQ-9 composite after tensile failure. Both figures show the fracture interface. **A**- After tensile testing at room temperature, **B**- After tensile testing at 150 °C.

## 4. Conclusions

Although far from being optimized, we have successfully demonstrated the fabrication of an all-aromatic liquid crystal thermoset/carbon-fiber (T300 5HS) composite using a simple resin powder infusion process. The composite shows excellent room temperature tensile properties, i.e. a tensile strength of 696 MPa and tensile modulus of 57 MPa. The high in-plane shear modulus of 154 MPa seems to be indirect evidence of good interfacial bond strength between the carbon fibers and the polymer matrix. The composites were also tested close to their T<sub>2</sub> of 150 °C and we found a tensile strength of 527 MPa. The flexural strength of 817 MPa is low and can be explained by the presence of voids. A *Post-mortem* inspection of the fractured tensile and compression samples with high-resolution SEM showed that failure during tensile and compression testing occurred within the fiber and not at the fiber-matrix interface. The initial thermo-mechanical and mechanical properties of this T-300/HIQ-9 composite are promising but additional composite manufacturing and composite testing is needed. After this proof-of-concept phase we will continue this work using new high Tg all aromatic LCTs (Tg> 350 °C), which have been developed by us recently.

## 5. Acknowledgements

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