HIGH MODULUS, RECYCABLE, FULLY THERMOPLASTIC COMPOSITES FOR AERONAUTICAL APPLICATIONS

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Abstract

High-performance engineering thermoplastics are essential in modern aerospace applications. Polyetherimide (PEI) is widely used in aircraft due to its excellent chemical resistance, electrical insulation, and heat resistance. However, its mechanical properties hinder its more widespread use in other aeronautical components. Thermotropic liquid crystalline polymers (TLCPs) are a promising reinforcement for engineering thermoplastics like PEI, as they possess high tensile strength, dimensional stability, and exceptional processability due to their rod-like molecular structure. Under high shear and extensional stresses, TLCPs elongate into fibrils aligned with the stress direction, significantly reinforcing the matrix. Furthermore, TLCPs can regenerate their fibrillar morphology upon reprocessing, enabling mechanical recycling with minimal loss of mechanical properties. This study develops a fully thermoplastic, recyclable, high-strength composite using PEI as the matrix and HX3000 TLCP as the reinforcement. The thermal and rheological properties of both materials were characterized to establish processing windows for injection molding. The highest tensile modulus, tensile strength, and creep resistance were observed in the 50 wt% HX3000-PEI composite after the initial processing step. However, tensile properties across recycling steps were most consistent in the 25 wt% HX3000-PEI composites, as recycling progressively reduced the tensile performance of the 50 wt% composite at each step.

1 - Introduction

Plastics and aeronautics have been closely intertwined since the early days of modern aviation. Today, engineering thermoplastics such as polyether ether ketone (PEEK), polyphenylene sulfide (PPS), and polyetherimide (PEI) are widely used in aerospace applications due to their chemical resistance, electrical insulation, and heat resistance[1]. However, their broader adoption is hindered by their relatively poor mechanical properties compared to metals such as aluminum, titanium, and steel. Given the critical role of high strength-to-weight ratio materials in aviation, improving the mechanical performance of these thermoplastics could significantly expand their usability in aerospace applications. Furthermore, as sustainability becomes a growing priority, ensuring these plastics can be reprocessed and recycled without significant degradation in performance is essential.

One notable high-performance thermoplastic used in aeronautics is polyetherimide (PEI). PEI is known for its chemical resistance, electrical insulation, and flame resistance properties[1]. Many PEI grades comply with Federal Aviation Administration (FAA) regulation 25.853, which dictates material standards for aircraft, making it a common choice for aerospace applications. A notable example of its use was by LSG Sky Chefs, the world's largest provider of in-flight catering, which developed PEI-based thermoplastic trolleys that were 40% lighter than their metal counterparts, leading to estimated yearly fuel savings of $65,000 per commercial aircraft[2]. Despite such successes, PEI's applications remain largely limited to interior components due to its relatively low strength and susceptibility to creep over long time scales when compared to metals. Enhancing the mechanical properties of PEI could expand its use beyond interior applications, making it a more viable alternative to traditional aerospace materials.

A promising approach to improving the mechanical performance of PEI is through the incorporation of reinforcement. Prior studies have demonstrated enhanced mechanical properties in PEI composites reinforced with carbon fibers [3] and glass fibers [4]. However, a key limitation of these reinforcements is fiber shortening during recycling as repeated reprocessing can shorten the fibers, ultimately reducing composite performance. An alternative reinforcement strategy that offers both mechanical property enhancement and improved recyclability is the incorporation of thermotropic liquid crystalline polymers (TLCPs), which have shown the ability to strengthen thermoplastic polymers without the same compromise to recyclability. TLCPs are a class of thermoplastic polymers that exhibit oriented mesophase upon entering the melt. This molecular orientation results in high tensile properties, coupled with low viscosity and ease of processing[5]. Under shear or extensional stresses—such as during extrusion or injection molding—TLCPs can elongate and form highly oriented fibrils when mixed with a separate thermoplastic polymer matrix, serving as in-situ fibril formation. Most importantly, because TLCPs are thermoplastic, the fibril forming process reoccurs upon subsequent processing steps with minimal decrease in mechanical performance; opening opportunities in recycling[6–8]. By creating a TLCP fibril reinforced composite with an already FAA approved material like PEI, the mechanical properties can be increased without compromising recyclability.

One critical factor in evaluating aerospace-grade plastics is their creep resistance, or their ability to resist time-dependent deformation under constant stress. Plastics—especially amorphous polymers like PEI—are particularly susceptible to creep, which can compromise the materials' long-term reliability. Previous studies have demonstrated that incorporating short carbon fibers into PEI can significantly improve its creep resistance[9,10]. Panin *et al*[9]. found that when incorporating the longer the length of the short carbon fibers added, the better improvement of creep properties was attained. PEI loaded with 10 wt% 2000 µm short carbon fibers showed the most improvement, with the rate of change of cyclic creep decreasing by a factor of 4.5 and the cycles before failure increased by a factor of 5.1.

In this work, we aim to improve the mechanical performance of PEI by incorporating TLCPs to create the next generation of recyclable aerospace composites. TLCPs are known to form fibrils when processed within a polymer matrix, offering a potential advantage over traditional short fiber reinforcements. Unlike short fibers, TLCP fibrils are continuous and can be aligned in the direction of applied load, making them particularly effective at enhancing creep resistance. By leveraging TLCP reinforcement, our goal is to develop a high-modulus, creep-resistant composite with a PEI matrix—an FAA-approved thermoplastic commonly used in aerospace. This material system has the potential to expand the application space of PEI while contributing to weight reduction and improved fuel efficiency in modern aircraft. Composite samples containing 0 wt%, 25 wt%, and 50 wt% TLCP reinforcement were produced and subjected to five recycling steps. The evolution of tensile properties across multiple reprocessing cycles, as well as the composites' creep performance, were evaluated to assess the recyclability and long-term performance of these materials for aerospace applications.

2 - Experimental Section

2.1 Materials

Poly(etherimide) (PEI), Ultem 1010 grade, was supplied by Sabic (Houston, Texas, USA). Its glass transition temperature was determined to be 220°C using differential scanning calorimetry (DSC) at a 10°C/min ramp rate. HX3000, supplied by DuPont, is a proprietary TLCP believed to be composed of hydroxybenzoic acid, terephthalic acid, hydroquinone, and hydroquinone derivatives in unspecified ratios. Its melting temperature was determined to be 330°C using DSC at a 10°C/min ramp rate.

2.2 Material Characterization

Torsional Rheology

Measurements were performed using an HR-30 rheometer equipped with 25 mm parallel plates or cone and plate geometries depending on the specific test. All tests were conducted under nitrogen. Strain sweeps were ran from 0.1% to 100% at 360°C and 10 rads/s. The temperature ramp on PEI was run from 400°C to 270°C at 5°C/min ramp rate at 3% strain and 10 rads/s for PEI. The temperature ramp on HX3000 was run from 360°C to 300°C at 5°C/min ramp rate at 0.1% strain and 10 rads/s. Time sweeps were ran on PEI at 360°C, 370°C, and 380°C at 10 rads/s and 10% strain for 30 minutes. Shear step strain tests were ran on HX3000 using a 25 mm cone and plate geometry at 0.1% strain at 350°C and 360°C. Frequency sweeps on recycled PEI samples were ran at 1% strain and 360°C

2.3 Composite Generation

TLCP and PEI pellets were dried separately in a vacuum oven at 130°C overnight before processing. The dried pellets were then weighed to prepare 25 wt% and 50 wt% TLCP compositions. To enhance mixing before injection molding, these compositions were compounded using a Killion KL-100 single-screw extruder equipped with a 24 L/D screw and a 6 mm die. The thermal profile in the extruder was set to 315°C, 330°C, and 360°C, corresponding to the feed zone, compression zone and metering zone respectively. The residence time of the extruder was roughly 8 minutes. The extruded filament was then pelletized and dried again before injection molding. Samples were injection molded with a BOY 35E into Type V dogbone specimens following ASTM D638 standards. The thermal profile for injection molding was adapted from Sabic's processing guide for Ultem 1010. After producing enough dogbones for characterization, the remaining material was pelletized and reprocessed utilizing the same injection molding procedure. This recycling process was repeated five times to simulate multiple recycling steps for each sample: neat PEI, 25 wt% HX3000-PEI, and 50 wt% HX3000-PEI.

2.4 Composite Material Characterization

Composite Composition Verification

To verify that the TLCP loading composition matched the intended values of 25 wt% and 50 wt%, a selective dissolution method was employed on the pellets following mixing in the single screw extruder. Since TLCPs are insoluble in most solvents, a solvent that selectively dissolves the PEI matrix while leaving the TLCP fibrils intact was used. Three glass vials, each containing one gram of composite pellets, were filled with approximately 25 mL of dimethylformamide (DMF) and stirred continuously for 24 hours. Once the PEI matrix was fully dissolved, the remaining TLCP was collected via vacuum filtration using pre-weighed filter paper. The filter paper was then dried overnight in a fume hood before measuring the mass of the residual TLCP and calculating the weight percentage of TLCP in the composite is calculated in equation (1).

$$TLCP\ \% = \frac{(m_{measured} - m_{filter\ paper})}{1\ g} \tag{1}$$

Scanning Electron Microscopy

Samples were cryo-fractured in liquid nitrogen, perpendicular to the extrusion or injection direction. They were mounted on aluminum stubs using carbon tape and sputter-coated with 10 nm of platinum-palladium to increase conductivity for high-quality imaging. Cross-sectional morphology was analyzed using a JEOL IT-500HR scanning electron microscope at an accelerating voltage of 5 kV.

Tensile Testing

Tensile properties were evaluated following ASTM D638 using a 5969 Instron universal testing machine. Type V dogbone specimens were tested at a grip separation rate of 1 mm/min. The strain on the samples was measured with an Instron 2630-100 Series clip-on extensometer.

Creep Characterization (TTS)

Creep tests were conducted using a TA Instruments DMA 850 to evaluate the long-term deformation behavior of the composites. Samples were subjected to tensile creep under a constant stress of 2 MPa at 30°C. The stress level was chosen because it was the highest achievable without approaching the machine's force limit.

3 – Results

Torsional rheometry is a powerful characterization method that provides molecular-level insights into polymer behavior under flow conditions. This technique helps us understand how the material responds during processing methods such as extrusion and injection molding, enabling process optimization.

Strain sweeps were performed on both PEI and HX3000, as shown in Figure 1 andFigure 2, respectively. These tests were essential for identifying the linear viscoelastic region (LVR) and determining the appropriate strain level for subsequent oscillatory tests. Both materials were tested at a frequency of 10 rad/s and temperature of 360°C, as this was the anticipated upper processing temperature. From Figure 1, the critical strain for PEI was determined to be approximately 30% as determined from a 5% decline in storage modulus. Figure 2 shows a critical strain of roughly 1% for HX3000.

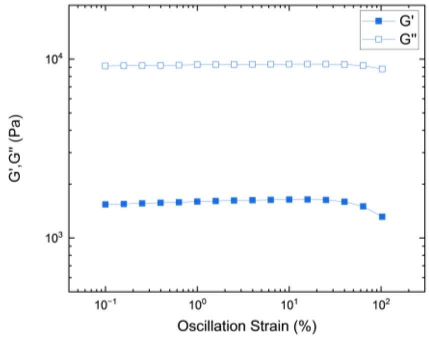

Figure 1: Strain sweep from 0.1% to 100% strain on PEI matrix material to determine the LVR.

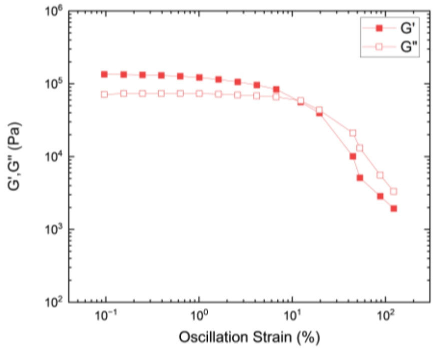

Figure 2: Strain sweep from 0.1% to 100% strain on HX3000 TLCP to determine the LVR.

Once the LVR was established, further rheological characterization of PEI and HX3000 was conducted. For PEI, a temperature ramp test was performed to evaluate its temperature-dependent behavior. Figure 3 displays the storage and loss moduli as a function of temperature.

At 275°C, the storage modulus is higher than the loss modulus, indicating that PEI behaves primarily as a viscoelastic solid. As the temperature increases beyond 275°C, the moduli intersect, with the loss modulus surpassing the storage modulus—signifying a transition to the material behaving as a viscoelastic fluid. Both moduli continue to decrease with increasing temperature until approximately 370°C, where the storage modulus begins to rise, suggesting the possibility of the onset of degradation. PEI is known to undergo crosslinking at temperatures above 360°C, which increases solid-like behavior and leads to a rise in storage modulus as observed. This observation aligns with the expected thermal degradation characteristics of PEI.

Figure 4 shows the magnitude of the complex viscosity of PEI as a function of temperature from the same temperature ramp as Figure 3. As expected, the magnitude of the complex viscosity decreases with increasing temperature, consistent with typical thermoplastic behavior.

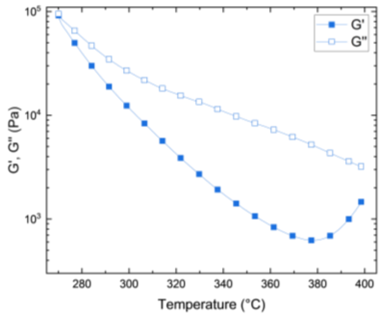

Figure 3: Temperature ramp on PEI at 10 rads/s, 3% strain, and 5°C/min ramp rate. Storage and loss modulus are plotted with respect to temperature from 270°C to 400°C

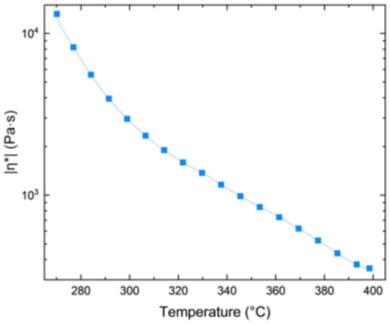

Figure 4: Temperature ramp on PEI at 10 rads/s, 3% strain, and 5°C/min ramp rate. The magnitude of the complex viscosity ($|\eta^*|$) is plotted with respect to temperature from 270°C to 400°C

After characterizing the temperature-dependent behavior of PEI, its thermal stability was evaluated. TLCPs are generally more thermally stable than thermoplastics, making it essential to confirm that PEI does not undergo significant degradation at higher processing temperatures. Based on prior DSC measurements, the anticipated upper processing limit for HX3000 was 360°C–380°C. To assess whether PEI remains stable at these temperatures, oscillatory time sweeps were performed at 360°C, 370°C, and 380°C for 30 minutes. Although the residence time in the extruder used in this study is approximately 8 minutes, the tests were extended to 30 minutes since shear rates in an extruder are significantly higher than those achievable in a torsional rheometer. Higher shear rates accelerate polymer degradation[11], so demonstrating stability over an extended duration at lower shear rates suggests that the material can withstand the processing conditions. The tests were conducted under a nitrogen atmosphere at 10 rad/s and 10% strain, with the results shown in Figure 5.

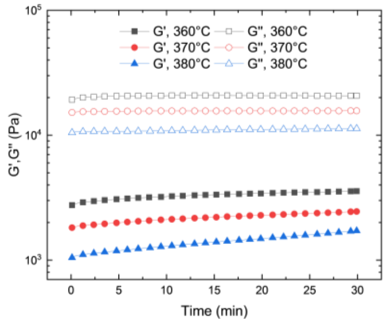

Figure 5: Time sweeps at 360°C, 370°C, and 380°C on PEI under nitrogen, at 10 rads/s, and 10% strain to see the thermal stability of the material.

From the data presented in Figure 5, the loss modulus and storage modulus do not intersect for any of the tests, suggesting that PEI remains thermally stable for short time scales at the tested temperatures. However, it is important to note that PEI begins to crosslink at 360°C, meaning that exposure to or above this temperature should be minimized to prevent unwanted degradation.

With the processing range for PEI established, similar rheological tests were performed on HX3000 TLCP to characterize its thermal stability and processing window. A temperature sweep was conducted on HX3000 from 360°C to 300°C at 0.1% strain, 10 rad/s, and a temperature ramp rate of 5°C/min.

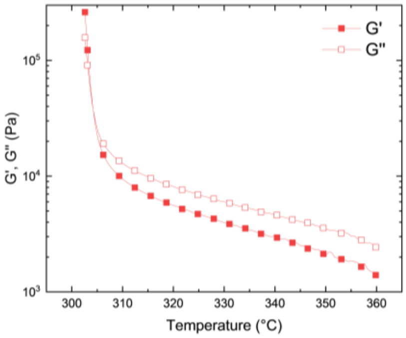

Figure 6: Temperature ramp on HX3000 TLCP at 10 rads/s, 0.1% strain, and 5°C/min ramp rate. Storage and loss modulus are plotted with respect to temperature from 300°C to 360°C

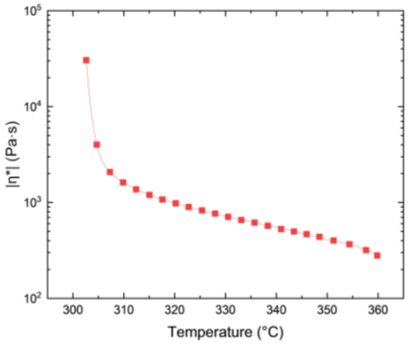

Figure 7: Temperature ramp on HX3000 TLCP at 10 rads/s, 0.1% strain, and 5°C/min ramp rate. The magnitude of complex viscosity $(|\eta^*|)$ *is plotted with respect to temperature from 300°C to 360°C*

Figure 6 presents the temperature-dependent behavior of HX3000 TLCP during a temperature ramp from 360°C to 300°C. The storage and loss modulus crossover occurs around 305°C, suggesting this as the minimum allowable processing temperature. Similarly, Figure 7 illustrates the complex viscosity as a function of temperature, showing the expected decrease in viscosity as temperature increases.

While Figure 6 and **Error! Reference source not found.** provide insight into the thermal behavior of HX3000, they do not fully determine the optimal processing temperature for the TLCP. Relaxation curves for TLCPs typically show an initial rapid decay followed by a long relaxation tail which is attributed to an unmelted solid phase still present within the melt[12]. When this relaxation tail is not present within the relaxation curve, all solid phases are eliminated.

To assess the presence of solid phases, a shear step strain test was conducted at 350°C and 360°C as shown in Figure 8. This test applies a sudden strain at $t = 0$ after which the material's stress response is measured. TLCP pellets were loaded onto the bottom plate and allowed to melt for roughly 3 minutes, before trimming the sample and beginning the test.

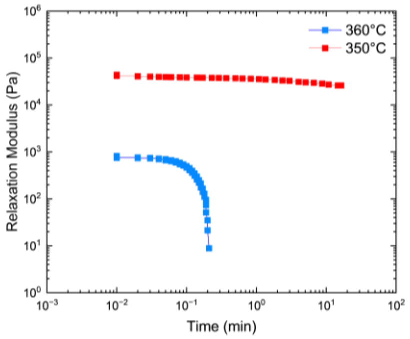

Figure 8: Shear step strain test at 0.1% strain and under nitrogen with a cone and plate geometry on HX3000 TLCP to determine the minimum processing temperature where all TLCP crystallites are melted.

At 350°C, the relaxation modulus shows the relaxation tail indicating the presence of solid phases within the melt. However, at 360°C, the relaxation modulus steadily decreases, confirming that all solid phases have been eliminated. Based on this result, the TLCP must be processed at a minimum of 360°C at some point in the extruder or injection molder to melt all solid phases, as processing at a temperature below this value can lead to worse mechanical properties[13]. Furthermore, by leveraging the sub cooling behavior of TLCPs, once heated to 360°C, the material can be cooled down to a temperature below this value, and still flow, without the presence of crystallites[14]. As a result, when developing a thermal profile for both the extruder and injection molder, the material must be heated to 360°C at some point during processing; however, it can subsequently be cooled to a lower temperature to help minimize degradation of the polymer matrix

Following detailed characterization of the selected materials and processing of TLCP composites via extrusion and injection molding, scanning electron microscopy (SEM) was employed to examine the cross-sections of both the extruded filament and injection molded samples in order to verify TLCP fibrillation. Recall more fibrillation indicates more continuous fiber reinforcement, thus enhancing the mechanical properties Figure 9 displays SEM images of the TLCP composites.

Before pelletizing and injection molding, the cross-section of the 50 wt% TLCP-PEI composite filament, extruded once, is shown in Figure 9a. This image reveals extensive fibrillation, with fibril diameters ranging from 1 to 5 µm. After one injection molding cycle, cross-sectional images of both the 25 wt% HX3000-PEI and 50 wt% HX3000-PEI composites are shown in Figure 9b and Figure 9d, respectively. After five recycling steps, the 25 wt% TLCP and 50 wt% HX3000-PEI composites are shown in Figure 9c and Figure 9e, respectively

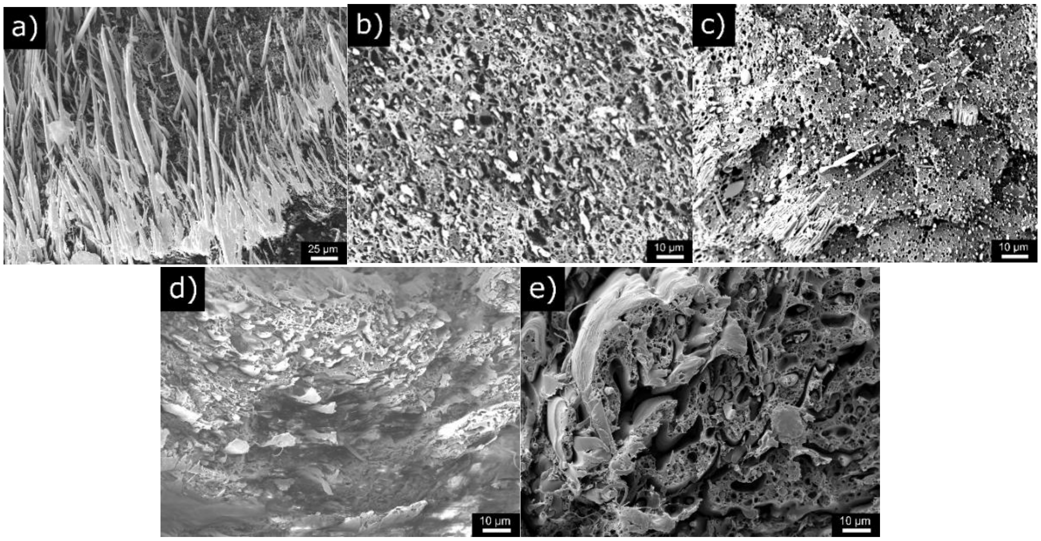

Figure 9: a) cross-section of 50 wt% HX3000-PEI composite filament after extrusion b) cross section of 25 wt% HX3000-PEI injection molded composite dogbone before any recycling steps c) cross section of 25 wt% HX3000-PEI injection molded composite dogbone after 5 recycling steps d) cross section of 50 wt% HX3000-PEI injection molded composite dogbone before any recycling steps e) cross section of 50 wt% HX3000-PEI injection molded composite dogbone after 5 recycling steps

To verify the TLCP loading percentage in each extruded composite, a selective dissolution method was employed. This technique involved dissolving the PEI matrix in DMF while leaving the TLCP fibrils intact. The results of this analysis, shown in Table 1, demonstrate that the measured TLCP content was close to the expected values, confirming that the composites were successfully created with the targeted loading percentages.

Table 1: Measured loading percentage of TLCP composites from the selective dissolution procedure

	25 wt%	50 wt%
Measured Loading	25.08% ± 0.61	51.53% ± 0.25

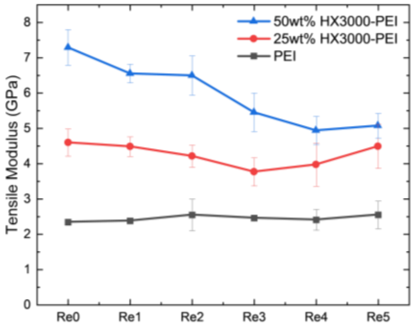

Figure 10: Tensile modulus of neat injection molded PEI, 25 wt% HX3000-PEI, and 50 wt% HX3000-PEI over 5 recycling steps.

In addition to characterizing the morphological features of the composites using SEM, the mechanical properties of the composites across recycling steps were evaluated, as shown in Figure 10 and Figure 11. Both the tensile modulus and the ultimate tensile strength increased with the incorporation of TLCP into PEI. The mechanical properties of both the PEI and the 25 wt% HX3000-PEI composite remained relatively constant over the recycling steps. However, a decrease in mechanical properties was observed over the recycling steps for the 50 wt% HX3000-PEI composites. This phenomenon suggests that interfacial adhesion between the matrix and TLCP becomes increasingly important at higher TLCP loadings. At lower loadings, the greater availability of matrix material facilitates encapsulation and promotes fibrillation of the TLCP phase during processing. In contrast, higher TLCP loadings limit the amount of matrix available to promote this behavior, resulting in less distinct phase separation and reduced fibrillation, as observed in Figure 9d and 9e.

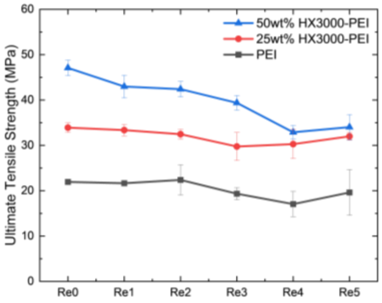

Figure 11: Ultimate tensile strength of neat injection molded PEI, 25 wt% HX3000-PEI, and 50 wt% HX3000-PEI over 5 recycling steps.

A dynamic mechanical analyzer (DMA) was employed to characterize the creep properties of the composites. During the test, samples were equilibrated at the testing temperature for one hour before a stress of 2 MPa was applied and held for 10 hours at 30°C. The resulting strain was calculated and plotted as shown in Figure 12. A second axis is also plotted to show the instantaneous applied load put on the samples by the DMA. Incorporation of TLCP into the matrix significantly lowers the amount that the material creeps during the extent of the test.

Frequency sweeps were also performed over the recycling steps on neat PEI, with the data shown in Figure 13. As expected, the zero-shear viscosity decreases with each recycling step, which is most likely attributed to a reduction in average molecular weight due to chain scission of the matrix during processing.

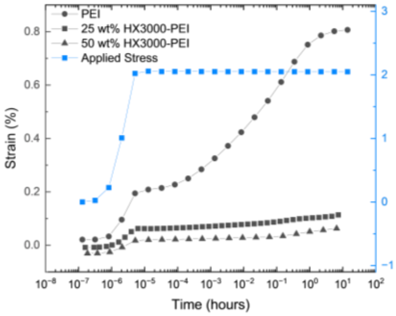

Figure 12: Stress and strain as a function of time at 30°C for injection molded PEI, 25 wt% HX3000-PEI, and 50 wt% HX3000-PEI

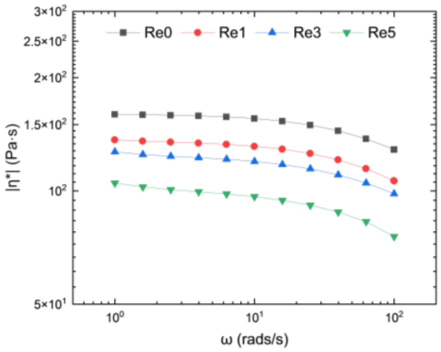

Figure 13: Frequency sweeps on recycled, injection molded PEI at 1% strain and 360°C

As the average molecular weight of the matrix material decreases, the degree of chain entanglements of the matrix decreases; thus, the viscosity of the matrix would go down.

4 – Discussion

The thermal properties of both the TLCP and matrix were extensively characterized using rheometry to evaluate compatibility and processing parameters. The processing temperature range for PEI was determined to be between 280°C and 380°C. Additionally, the complete melting of crystallites in the HX3000 TLCP was observed at 360°C, indicating that the composite must reach this temperature during processing to achieve optimal mechanical properties. This thermal characterization informed the development of a precise temperature profile for both the extruder and injection molder to enhance fibrillation and mechanical performance.

Scanning electron microscopy (SEM) of the composite cross-sections revealed notable differences in fibril formation between processing methods. The most well-developed fibrillation was observed in the extruded filament samples. Over multiple recycling steps, fibril formation remained apparent in the 25 wt% injection-molded samples, whereas the 50 wt% composite exhibited less distinct phase separation. The higher TLCP content in the 50 wt% composite likely obscured phase boundaries between PEI and HX3000, making fibril identification more challenging. Because of this, no well-defined fibrils were observed in SEM images of the 50 wt% composite.

Mechanical testing demonstrated significant improvements in tensile modulus, ultimate tensile strength, and creep resistance with TLCP incorporation. The 25 wt% HX3000-PEI composite retained its mechanical properties across multiple recycling steps, whereas the 50 wt% composite experienced a decline. This decrease is likely due to changes in viscosity in the matrix material. As shown in Figure 13, PEI's average molecular weight decreased with each reprocessing step, leading to a decrease in the degree of chain entanglements within the matrix. This is most likely only observed in the 50 wt% composite because of the importance of interfacial heating between the matrix and TLCP at higher loading. At lower loadings, the greater availability of matrix material facilitates encapsulation and promotes fibrillation of the TLCP phase during processing. In contrast, higher TLCP loadings limit the amount of matrix available to promote this behavior, resulting in less distinct phase separation and reduced fibrillation. Furthermore, with decreasing average molecular weight of the matrix at each recycling step, the adhesion between the phases, is likely to decrease, which could explain why decreasing mechanical properties at higher TLCP loading is observed.

5 – Conclusion

The incorporation of TLCPs into PEI significantly enhanced the tensile and creep properties of the composite. With 25 wt% TLCP addition, the tensile modulus approximately doubled, and the ultimate tensile strength nearly doubled as well. While 50 wt% TLCP further improved mechanical properties initially, these benefits diminished with reprocessing due to decreasing extensional viscous forces on the TLCP as the PEI matrix degraded.

Creep resistance also improved with TLCP addition, as evidenced by significantly lower strain under load compared to neat PEI. Because PEI is amorphous, continuous loading led to chain disentanglement and material creep. The stiff TLCP reinforcement limited strain development, reducing creep. Future studies should explore long-term creep behavior using time-temperature superposition or extended-duration tests to better understand the material's performance over extended use.

Overall, this study demonstrates that TLCP incorporation into FAA-approved PEI enhances mechanical properties, broadening its potential applications in aviation. Increased adoption of TLCP-reinforced PEI in aircraft could contribute to weight reduction, improved fuel efficiency, and lower emissions, aligning with industry goals for sustainability and performance optimization.

6 - Acknowledgements

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