Development and Characterization of Supercooled Polyethylene Naphthalate

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Abstract: The utilization of undercooled or supercooled polymers presents a promising approach for the creation of single-polymer composites (SPCs), applicable not only to compaction processing but also to extrusion, injection molding, and 3D printing techniques. This study focuses on the development and characterization of supercooled polyethylene naphthalate (PEN) through differential scanning calorimetry (DSC) and rheological measurements. By employing predetermined conditions, a supercooling degree of 50 ˚C for PEN was achieved. The impact of maximum heating temperature, cooling rate, and shear rate on the supercooling degree was examined, revealing that higher supercooling degrees of PEN can be attained by increasing these factors. Additionally, the flow behavior of supercooled polymer melts at various temperatures was analyzed. The supercooling state of PEN exhibited remarkable stability for a minimum duration of half an hour at temperatures exceeding 250 ˚C.

Keywords: Undercool, supercooling, single-polymer composites, polyethylene naphthalate.

1. INTRODUCTION

The utilization of reinforcement fibers such as glass and carbon in polymer matrices has garnered significant academic and commercial interest in recent decades. While these composites have achieved excellent mechanical properties, their limited recyclability poses challenges in terms of life cycle assessment. Additionally, traditional composites face interface issues due to the use of different materials for reinforcement and matrix components. To address the need for environmentally friendly composites with strong adhesion properties, the concept of single-polymer composites (SPCs) has emerged. SPCs are characterized by having both the reinforcement and matrix derived from the same polymer material. The key advantage of SPCs lies in their ease of recyclability, as the reinforcement and matrix can be reprocessed together. Furthermore, SPCs ensure excellent fiber/matrix adhesion without the need for coupling agents, as identical materials exhibit the best adhesion and highest interfacial shear strength. The future of SPCs appears promising due to ongoing advancements in their preparation, properties, market growth, and recyclability [1].

However, in the case of true SPCs, the matrix and fiber must originate from the same polymer and have similar melting temperatures. This requirement presents difficulties in combining the fiber with the matrix using conventional methods, as melting the oriented fiber can lead to a loss of stiffness and strength acquired during processing. The difference in melting temperatures determines the processing window, and various techniques have been developed to expand this temperature range for SPC production. These methods include hot compaction of fibers or tapes [2-7], film stacking [8-11], a combination of hot compaction and film stacking [12], co-extrusion [13-16], extrusion-calendering [17,18], insert injection molding [19-23], and 3D printing [24,25].

To further widen the processing temperature window, our previous work [26,27] explored the feasibility of utilizing undercooled polymer melts in the manufacturing of SPCs. Polypropylene (PP) was used as a model system to demonstrate the method. By introducing the fiber into a liquid matrix at a temperature well below the matrix's melting temperature, a processing temperature window of at least 25 ˚C was established. Undercooling or supercooling refers to the process of cooling a substance below its phase-transition temperature without transitioning to the next phase. Polymers, with their high molecular weight and long molecular chains,

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can be easily supercooled [28]. It is known that semi-crystalline polymers can be supercooled to temperatures well below their melting point without significant crystallization [28]. The so-called mesophase of PP can be formed at high supercooling (below 60 ˚C) [29]. While existing methods for preparing SPCs are primarily limited to compaction processes, the use of supercooled polymer melts opens the door to industry-standard production methods such as extrusion [17,18], injection molding [19-23], and 3D printing [24,25], which are more widely employed.

Although the supercooled temperature is below the polymer's melting temperature (T_m), directly heating the polymer to the supercooled temperature may not result in a molten state. Therefore, two main processes determine the formation of supercooled polymer melts: the heating process and the cooling process. The polymer must first be heated into a molten state, after which it can be rapidly cooled to achieve a supercooled state. The heating temperature is typically above the T_m, while the desired supercooled temperature lies between the T_m and the crystallization onset temperature (T_x), defining the supercooling degree. The larger the supercooling degree, the smaller the potential for heat damage to the strength of the polymer fiber [26]. Moreover, even with a significant supercooling degree, the supercooled polymer melt must remain stable without undergoing crystallization to allow for the addition or mixing of reinforcements and the formation of SPCs. Hence, it is crucial to focus on the development and characterization of supercooled polymers.

Previous investigations on supercooled polymers primarily centered around PP. However, different polymers exhibit distinct supercooling behaviors. Polyethylene naphthalate (PEN), also known as poly(ethylene 2,6-naphthalate), is a polyester that is synthesized from ethylene glycol and naphthalene-2,6-dicarboxylic acid. While it shares similarities with poly(ethylene terephthalate), PEN possesses two condensed aromatic rings, which provide it with enhanced strength, modulus, chemical and hydrolytic resistance, gaseous barrier properties, thermal and thermo-oxidative resistance, as well as resistance to ultraviolet (UV) light barriers, surpassing those of polyethylene terephthalate (PET). PEN is specifically designed to serve as a substitute for PET, particularly when utilized as a substrate for flexible integrated circuits [30,31]. Its application in textile and industrial fibers, films, foamed articles, containers for carbonated beverages, water, and other liquids, as well as thermoformed applications, has led to significant commercial markets. High-performance fibers, boasting exceptional modulus and superior dimensional stability compared to PET or Nylon fibers, are manufactured using PEN. In addition, we developed the supercooling/undercooling melt film stacking method to prepare PEN SPCs and realized the production of easily recyclable PEN composites with enhanced mechanical properties [32]. However, the exact supercooling degree of the PEN was not investigated in detail.

In order to understand the formation process of supercooled PEN and determine their supercooling behavior, in this work, differential scanning calorimetry (DSC) and rheological measurements were employed. The effects of various process variables, including maximum heating temperature, cooling rate, and shear rate, on the supercooling degree were investigated. Additionally, the flow behavior of supercooled PEN at different temperatures was characterized. Comparison with PP revealed different properties, allowing for detailed experimental results and key theoretical principles regarding supercooled polymers to be applied to the processing of PEN SPCs.

2. EXPERIMENTAL

2.1. Materials

Polyethylene naphthalate (PEN) used in this study was sourced from Goodfellow Corporation and exhibited a density of 1.36 g/cm^3 at room temperature. As a comparative material [26], polypropylene (PP) was provided by Phillips Sumika Polypropylene Company and had a density of 0.905 g/cm^3 at room temperature. The melt flow rate of the PP was 3.8 g/10 min at 230 ˚C. Figure **1** shows the chemical structures of PP and PEN.

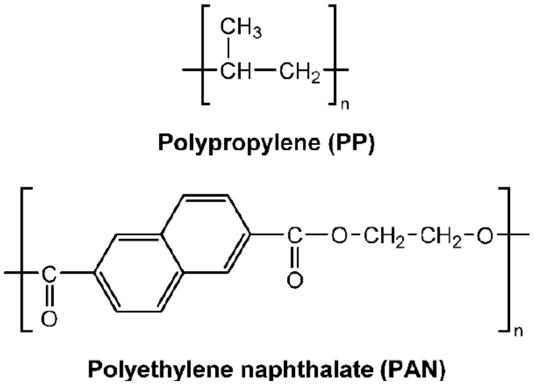

Figure 1: Chemical structures of PP and PEN.

2.2. Thermogravimetric Analysis (TGA)

Prior to the calorimetric analysis, the thermal stability of PEN was evaluated through thermogravimetric analysis (TGA) using a TA Instruments Q5000 thermogravimetric analyzer. The samples were heated at a rate of 10 ˚C/min from 50 to 600 ˚C under a nitrogen atmosphere with a flow rate of 60 mL/min. The TGA curve of PEN is illustrated in Figure **2**. The decomposition temperature of PEN was determined to be 368 ˚C at a heating rate of 10 ˚C/min. Consequently, in the calorimetric analysis, it is imperative to set the maximum temperatures below these decomposition temperatures to ensure the integrity of the samples.

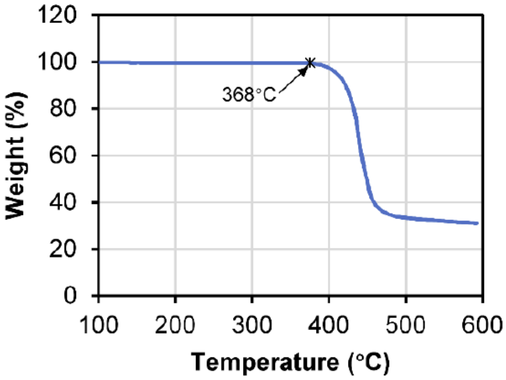

Figure 2: TGA curves of PEN.

2.3. Differential Scanning Calorimetry (DSC)

The thermal characteristics of PEN were analyzed using a TA Instruments Q200 differential scanning calorimeter (DSC). The T_m and T_x were determined to calculate the supercooling degree. To examine the impact of cooling rate on the supercooling degree, various cooling rates were employed. The PEN samples were initially heated from 40 to 350 ˚C at a rate of 10 ˚C/min and held for 2 min. Subsequently, they were cooled back to 40 ˚C using different cooling rates of 5, 10, 15, and 20 ˚C/min. Following this, the PEN samples were immediately reheated to 350 ˚C at a rate of 10 ˚C/min and cooled to 40 ˚C at a cooling rate of 10 ˚C/min.

The isothermal crystallization behavior of supercooled PEN at different temperatures was also investigated. The PEN samples were heated to 350 ˚C at a rate of 10 ˚C/min and maintained at that temperature for 2 min. They were then rapidly cooled to various temperatures of 220, 230, 240, 250, and 260 ˚C for isothermal crystallization. After a 30-min duration, the samples were cooled to 40 ˚C at a rate of 10 ˚C/min.

2.4. Rheological Measurement

The dynamic rheological properties of the samples were assessed using a parallel plate rotational rheometer (AR2000ex, TA Instruments). The diameter of the plates was 25 mm, and a strain level of 1% was applied. The initial gap for PP was set at 1 mm plus 50 μm, while for PEN it was 0.5 mm plus 50 μm. Initially, the samples were melted at the maximum heating temperature. Excessive squeezed-out material was trimmed, and then the upper plate was adjusted to achieve a final gap of 1 mm for PP and 0.5 mm for PEN. To eliminate existing crystallization and residual stress, the specimens were allowed to relax for approximately 10 min for PP and 3 min for PEN. Subsequently, the samples were cooled at a rate of 10 ˚C/min for temperature ramp rheological measurements.

To investigate the influence of the maximum heating temperature on the supercooling degree, different temperatures were utilized. For PP, the chips were melted at 200, 220, and 240 ˚C, while for PEN, the chips were melted at 280, 300, and 320 ˚C, respectively. The applied frequency during these measurements was 10 Hz. The selection of these three temperatures was based on that the material was melted but not degraded under these three temperatures.

To examine the effect of shear rate on the supercooling degree, three different frequencies (1, 10, and 100 Hz) were employed for PP and PEN at maximum heating temperatures of 200 ˚C and 300 ˚C, respectively.

Furthermore, the dynamic rheological properties of supercooled PEN at various temperatures were investigated. Initially, a temperature sweep was conducted to obtain the supercooled melt, and then the PEN chip was cooled to predetermined temperatures at a rate of 10 ˚C/min for subsequent time sweep rheological measurements. The predetermined temperatures for this analysis were 230, 240, 250, and 260 ˚C, while the maximum heating temperature remained at 300 ˚C. The frequency applied during these measurements was 10 Hz.

3. RESULTS AND DISCUSSION

3.1. Formation of Supercooled PEN

Figure **3** illustrates the DSC thermograms of PEN obtained using heating and cooling rates of 10 ˚C/min and a maximum heating temperature of 350 ˚C. In the heating process curve, a distinct step at 123 ˚C indicates the glass transition temperature (T_g), followed by a peak at 210 ˚C resulting from cold crystallization of the amorphous phase. The endothermal peak at 264 ˚C corresponds to T_m, marking the completion of melting. In the cooling process curve, a small peak at 198 ˚C (T_c) is attributed to crystallization, with PEN initiating crystallization at 213 ˚C (T_x). These observations reveal that PEN exhibits incomplete crystallization, with certain portions remaining in an amorphous state within the semicrystalline structure [33]. The supercooling degree can reach up to 51 ˚C, which was calculated as the difference between T_m and T_x (264 ˚C - 213 ˚C). In comparison to the findings for PP [26], PEN exhibited a slower crystallization rate and a larger supercooling degree.

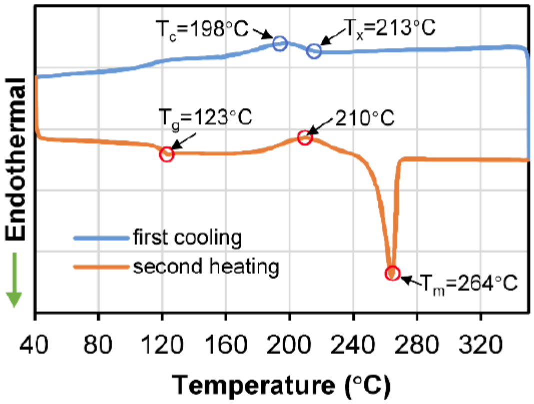

Figure 3: DSC thermograms of PEN determined at heating and cooling rates of 10 ˚C/min.

Crystallization of polymers involves the exothermic transition from a molten state to a solid state. The supercooling phenomenon can be determined through dynamic rheological measurements during the cooling process. The complex viscosities of PEN during cooling were assessed using a parallel plate rotational rheometer. Figure **4** displays the cooling behavior of PEN as a plot of complex viscosity against temperature. The PEN sample was cooled from 300 ˚C at a cooling rate of 10 ˚C/min and a frequency of 10 Hz. The complex viscosity of PEN increased by 15.2 kPa·s (from 1.5 to 16.7 kPa·s) within the temperature range of 80 ˚C (from 300 to 220 ˚C). Further cooling resulted in a viscosity increase of 67.3 kPa·s (from 16.7 to 84 kPa·s) over a temperature range of 60 ˚C (from 220 to 160 ˚C). The latter stage of viscosity increase can be attributed to crystallization.

Figure **4** does not exhibit a distinct turning point, as the viscosity increase of PEN is slower compared to that of PP [26]. The crossover point between the loss (G”) and storage (G’) modulus, which occurs around the transition from liquid to solid, was used to determine T_x. Figure **5** presents the storage and loss modulus of PEN as a function of temperature during cooling from 300 ˚C. Both G’’ and G’ increased as the temperature decreased. At the initial stages of cooling, G” was significantly larger than G’, but at the crossover point, this relationship reversed. The crossover point occured at 213 ˚C, which is considered to be T_x. This result is consistent with the DSC findings in Figure **3**.

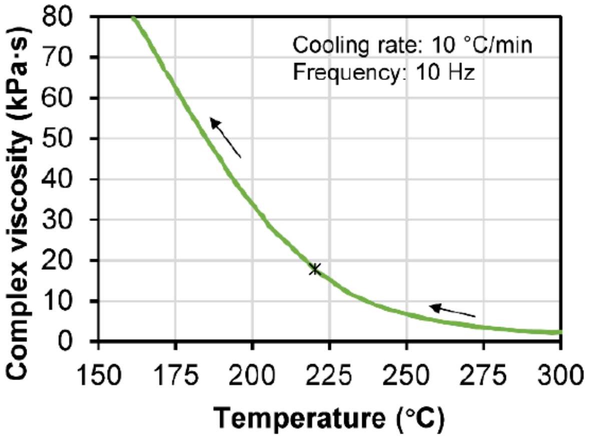

Figure 4: Complex viscosity of PEN as a function of temperature during cooling from 300 ˚C.

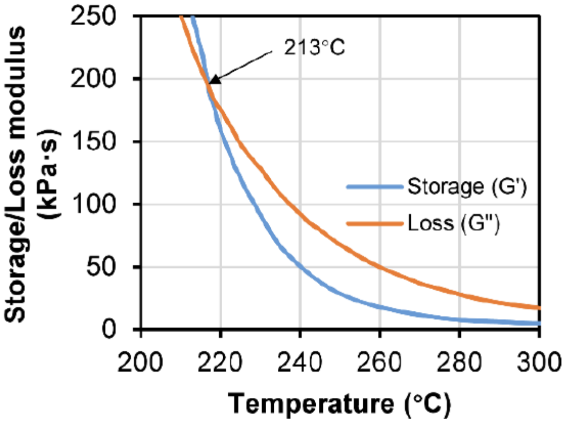

Figure 5: Storage and loss modulus of PEN as a function of temperature during cooling from 300 ˚C.

In summary, the supercooling degree of PEN, determined through DSC or rheological measurements, was found to be 50 ˚C under the specified conditions. However, it is important to note that several key factors, such as the maximum heating temperature, cooling rate, and shear rate, can influence the formation of the supercooled melt. Further investigations were conducted to explore these aspects.

3.2. Effect of Maximum Heating Temperature on Supercooling Degree

To create a supercooled polymer melt, the heating temperature must exceed the T_m of the polymer during the heating process. To understand the impact of the maximum heating temperature on the supercooling degree, rheological measurements were conducted using different maximum heating temperatures. The cooling rate remained at 10 ˚C/min, and the frequency was set at 10 Hz. Figure **6** depicts the complex viscosities of PP and PEN as a function of temperature during cooling from various maximum heating temperatures. The viscosity-temperature relationship varied with different maximum heating temperatures. Both supercooled PP and PEN exhibited a significant decrease in viscosity as the frequency or shear rate increases. Furthermore, the complex viscosities of supercooled PP and PEN decreased as the maximum heating temperature increased.

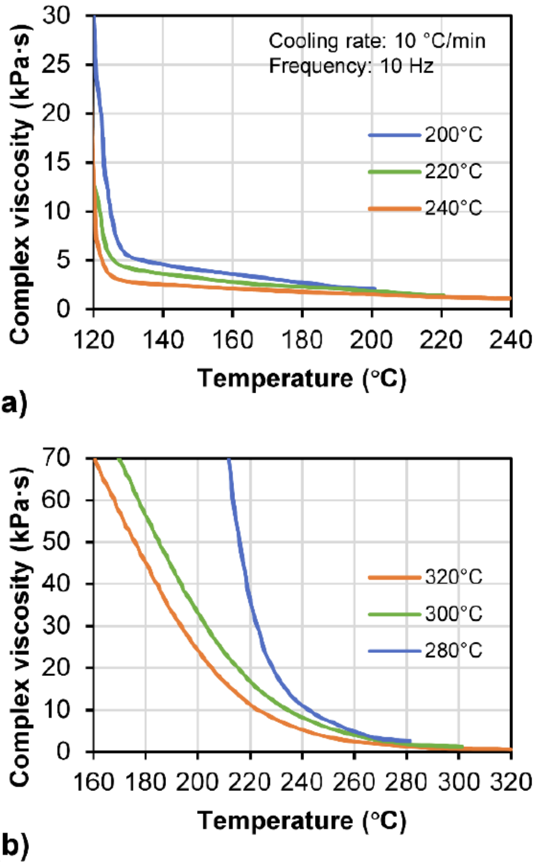

Figure 6: Complex viscosities of PP (**a**) and PEN (**b**) as a function of temperature during cooling from different maximum heating temperatures: 200, 220 and 240 ˚C for PP; 280, 300 and 320 ˚C for PEN.

Figure **6a** reveals that the turning points of all three curves were around 125 ˚C. When the temperature droped below 125 ˚C, there was a sharp increase in the complex viscosity for all three curves. This indicates that the maximum heating temperature had little influence on the supercooling degree of PP. In contrast, as shown in Figure **6b**, during the cooling process within the temperature range of 20 ˚C (from 240 to 220 ˚C), the complex viscosity of PEN increased by 169.4 kPa·s (from 96.6 to 266), 8.7 kPa·s (from 8 to 16.7), and 5.9 kPa·s (from 5.1 to 11) when the maximum heating temperatures were 280, 300, and 320 ˚C, respectively. This behavior differed from PP. The viscosity increase during the cooling process was much more rapid when PEN was cooled from 280 ˚C compared to 300 and 320 ˚C. This demonstrates that increasing the maximum heating temperature can slow down the crystallization of PEN.

To determine the value of T_x, Figure **7** displays the storage and loss modulus of PEN as a function of temperature during cooling from different maximum heating temperatures. T_x values of 217, 213, and 206 ˚C were observed for various maximum heating temperatures of 280, 300, and 320 ˚C, respectively. The T_x of PEN decreased with an increase in the maximum heating temperature, resulting in an increase in the supercooling degree. This occurred because at higher maximum heating temperatures, the crystals fully melt, making it challenging for them to re-crystallize. Similar findings were reported by Buchner *et al*. [34]. According to their study, the half-times of crystallization measured after directly transferring samples from the melt at 320 ˚C to T_c were much longer compared to those after melting at 280 or 300 ˚C. This indicates that the maximum heating temperature enhanced the increase in the supercooling degree of PEN.

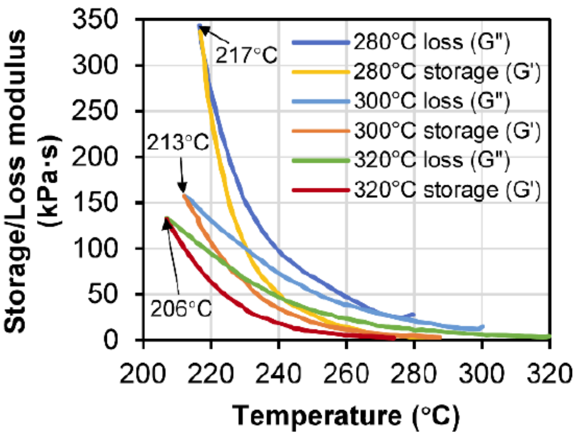

Figure 7: Storage and loss modulus of PEN as a function of temperature during cooling from different maximum heating temperatures: 280, 300 and 320 ˚C.

3.3. Effect of Cooling Rate on Supercooling Degree

The supercooling degree of PP increases with higher cooling rates. A cooling rate of 10 ˚C/min is sufficient for processing SPCs with supercooled PP [26]. To investigate the influence of cooling rate on the supercooling degree of PEN, DSC analysis was conducted. Figure **8** illustrates the DSC thermograms of PEN obtained at different cooling rates: 5, 10, 15, and 20 ˚C/min. In the heating process, all melting peaks occurred at 264 ˚C. However, the second heating process, which followed the cooling process, was also affected by the cooling rate. Comparing to the curve at 5 ˚C/min with the curves at higher cooling rates, no glass transition step or cold crystallization peak was observed in Figure **8**, indicating that PEN can fully crystallize at a cooling rate of 5 ˚C/min. Exothermal peaks can be observed during the cooling process at cooling rates of 5 and 10 ˚C/min. The PEN started to crystallize at 230 and 213 ˚C, respectively, for these cooling rates, indicating an increase in the supercooling degree with higher cooling rates. However, the curves at the other two cooling rates of 15 and 20 ˚C/min show no significant exothermal peaks but an indistinct step during the cooling process. This is because the cooling rates of 15 and 20 ˚C/min were too high, making it difficult for PEN to crystallize and resulting in an amorphous state. At these cooling rates, the supercooling degree can reach more than 100 ˚C. Table **1** provides the values of T_m, T_x, and the supercooling degree of PEN as a function of cooling rate.

Table 1: Supercooling Degree of PEN as a Function of Cooling Rate

Cooing rate (˚C/min)	T_m (˚C)	T_x (˚C)	Supercooling degree (˚C)
5	264	230	34
10	264	213	51
15	264	-	>100
20	264	-	>100

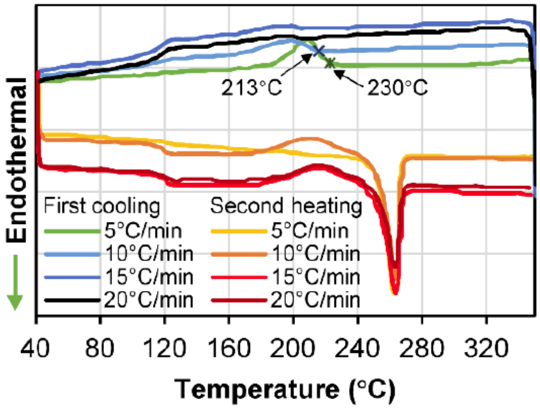

Figure 8: DSC thermograms of PEN determined at different cooling rates of 5, 10, 15 and 20 ˚C/min.

3.4. Effect of Shear Rate on Supercooling Degree

In addition to temperature, the flow behavior of supercooled polymer melt is also influenced by shear rate, which is particularly relevant in real industrial production where different device geometries generate varying shear rates. In the rheological measurements using a parallel plate rheometer, different frequencies of 1, 10, and 100 Hz were employed to investigate the impact of shear rate on the supercooling degree. Both supercooled PP and PEN exhibited a significant decrease in viscosity as the frequency/shear rate increased. Figure **9a** illustrates the complex viscosities of PP as a function of temperature during cooling from 200 ˚C at different frequencies, with a cooling rate of 10 ˚C/min. As the shear rate increased, the turning point shifted to higher temperatures, indicating a slight reduction in the supercooling degree of PP. This can be attributed to the increased ease of polymer chain orientation with higher shear rates, facilitating the solidification or crystallization of supercooled PP.

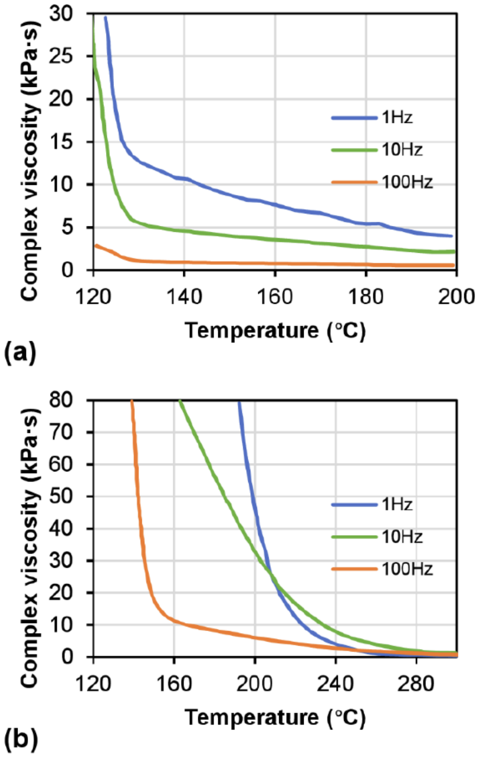

Figure 9: Complex viscosities of PP (**a**) and PEN (**b**) as a function of temperature during cooling with different scanning frequencies: 1, 10 and 100 Hz.

Figure **9b** demonstrates the complex viscosities of PEN as a function of temperature during cooling from 300 ˚C at different frequencies, with a cooling rate of 10 ˚C/min. In contrast to PP, the turning point shifts to lower temperatures as the shear rate increases for PEN. This suggests that the T_x of PEN decreased and, consequently, the supercooling degree increased. At a shear rate of 100 s^{-1}, a significant turning point occured at 150 ˚C, indicating rapid crystallization of PEN at higher shear rates. This behavior of PEN is opposite to that of PP. Due to the faster crystallization rate of PP, increasing the shear rate enhances the orderly arrangement of PP molecular chains, promoting PP crystallization. However, PEN crystallizes at a slower rate, making it susceptible to disruption of the internal structure with increasing shear rate, resulting in difficulties in crystallization. As a result, the supercooling degree of PEN increased at higher shear rates.

Therefore, different supercooled polymer melts exhibit distinct shear rate-dependent behaviors. In the processing of SPCs with supercooled PP, it is preferable to minimize the shear rate. Conversely, shear rate facilitates the formation of supercooled PEN, and device geometry can be designed accordingly based on this principle.

3.5. Flow behavior of Supercooled PEN at Different Temperature

During the processing of SPCs, it is crucial to develop a supercooled polymer melt as the matrix to penetrate the fabric or wet the fibers. Since the supercooled polymer melt is processed below its T_m, the key focus is to maintain a stable supercooling/undercooled state without crystallization. Ensuring the stability of the supercooled polymer melt is of utmost importance.

The isothermal crystallization behavior of supercooled PEN was investigated at various temperatures within the temperature range of the supercooled melt. The PEN samples were heated to 350 ˚C and held for 2 min, then rapidly cooled to the desired temperatures and held for 30 min before being further cooled to 40 ˚C at a rate of 10 ˚C/min. Figure **10** displays the heat flow curves of PEN during isothermal crystallization at different temperatures: 220, 230, 240, 250, and 260 ˚C. It can be observed that supercooled PEN rapidly crystallized at 220 ˚C, completing the crystallization process in just 5 min. At 230 ˚C, crystallization began after 4 min and was completed after 18 min. However, no crystallization peak was observed between 30 to 40 min during the cooling process following isothermal crystallization at 220 and 230 ˚C. This indicates that supercooled PEN completed its crystallization entirely during the isothermal crystallization stage before the cooling process, and it was unable to maintain its supercooling state at 220 and 230 ˚C. On the other hand, no crystallization peak was observed during the 30-min isothermal crystallization at 240, 250, and 260 ˚C, indicating that supercooled PEN remained stable and maintained its supercooling state.

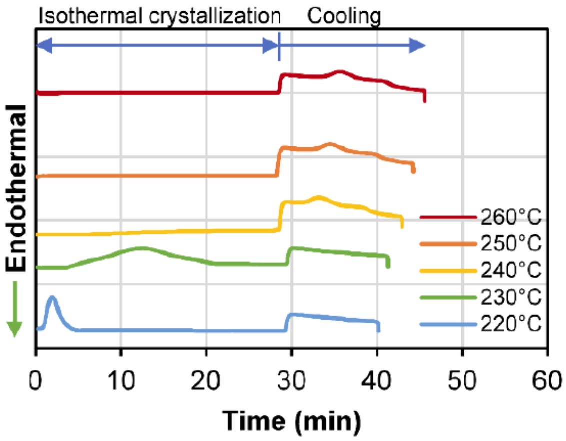

Figure 10: The heat flow curves of PEN during isothermal crystallization at different temperatures: 220, 230, 240, 250 and 260 ˚C.

In the processing of SPCs, a high fluidity of the matrix is desirable for effective fiber wetting. Temperature is the primary parameter influencing the fluidity of the supercooled melt. The dynamic rheological properties of supercooled PEN at different temperatures were examined. Figure **11** illustrates the complex viscosities of PEN as a function of time during cooling from 300 ˚C to different predetermined temperatures at a rate of 10 ˚C/min. Since PEN solidifies at 220 ˚C, the predetermined temperatures include 230, 240, 250, and 260 ˚C. Similar to PP [26], it can be observed that the complex viscosity of PEN increased as the temperature decreased. The fluidity of PEN at 230 and 240 ˚C was unstable, with only 4 and 7.5 min of stable time, respectively. However, both 250 and 260 ˚C were suitable temperatures for supercooled PEN to maintain a fluid state.

Based on the obtained experimental results from Figure **10**, it was found that the supercooled melt did not crystallize within 30 min at 240 ˚C. However, the complex viscosity at 240 ˚C, as shown in Figure **11**, exhibited significant variation. Rheological measurements have demonstrated an advantage in detecting viscosity variations even in the absence of crystallization. Mastering the fluidity of the supercooled melt is necessary in the actual production of SPCs. For polymer processors, it assists in optimizing processing conditions such as mold temperature and holding time. It is advisable to choose a higher temperature slightly below the melting point to maintain good fluidity of the supercooled melt. However, if a lower temperature is used to expand the processing temperature window, faster fiber wetting time should be employed accordingly.

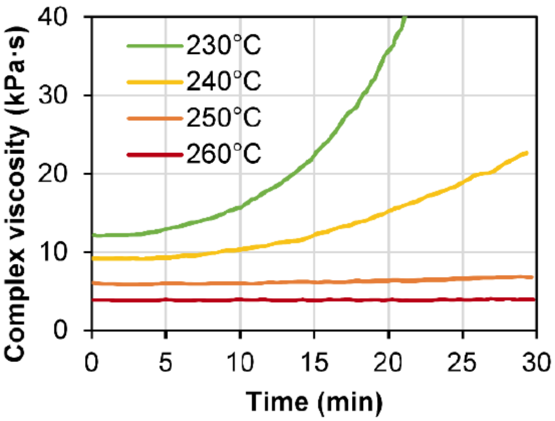

Figure 11: Complex viscosities of PEN as a function of time during cooling to different predetermined temperatures: 230, 240, 250 and 260 ˚C.

4. CONCLUSIONS

The potential of the undercooled or supercooling concept for manufacturing SPCs has been identified. The development and characterization of supercooled PEN were thoroughly investigated. The suitability of supercooled melt in SPCs processing largely depends on the supercooling degree, which can be determined using DSC or rheological measurements. PEN exhibited a supercooling degree of 50 ˚C under predetermined conditions. However, various factors including maximum heating temperature, cooling rate, and shear rate significantly influence the supercooling degree, and a comparison with PP revealed distinct properties. Understanding the flow behavior of supercooled PEN as a function of temperature is also crucial. The following conclusions can be drawn:

The maximum heating temperature has little impact on the supercooling degree of PP, whereas increasing the maximum heating temperature promotes a higher supercooling degree of PEN.

The supercooling degree of both PP and PEN increases with higher cooling rates. Supercooling of over 100 ˚C was achieved for PEN at cooling rates of 15 and 20 ˚C/min.

Different supercooled polymer melts exhibit varying shear rate-dependent behaviors. The supercooling degree of PP slightly decreases with increasing shear rate, whereas increasing shear rate enhances the supercooling degree of PEN.

Rheological measurement proves to be a superior method for assessing the stability of the supercooling state compared to DSC. DSC is unable to detect viscosity variations when no crystallization occurs. The supercooling state of PEN can be maintained for at least 30 min at temperatures above 250 ˚C, which is desirable for the preparation of PEN-based SPCs.

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