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Thermal Analysis on Carbon Fiber Polyphenylene Sulfide Composite Under Thermoforming Process

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ABSTRACT

The aerospace sector is transitioning from thermoset to thermoplastic composites to reduce weight, improve fuel efficiency, and accelerate manufacturing output. Carbon Fiber Reinforced Polyphenylene Sulfide (CF/PPS) composites offer advantageous properties, including improved weldability, enhanced storage stability, and compliance with fire-smoke toxicity (FST) standards. This study systematically evaluates the thermal behavior of 6-ply and 8-ply CF/PPS laminates thermoformed at 150, 160, 170, 180, and 195°C. Differential Scanning Calorimetry (DSC) analyses indicated a positive correlation between tooling temperature and both the crystallization temperature (T_{cc}) and degree of crystallinity, reflecting enhanced polymer chain mobility and molecular orientation during thermoforming. The 8-ply laminates exhibited earlier crystallization onset and higher crystallinity (~21.18%) due to reduced cooling rates and improved thermal retention, compared to the 6-ply laminates, which showed delayed crystallization kinetics. Thermogravimetric Analysis (TGA) results demonstrated significant improvement in thermal stability post-thermoforming, with char residue increasing from approximately 44-47% in unpressed states to 70-75% in pressed specimens, accompanied by elevated degradation onset and offset temperatures. These enhancements underscore the critical role of optimized tooling temperatures and ply thickness in modulating crystallization dynamics and thermal degradation resistance. The findings substantiate the suitability of CF/PPS composites for aerospace applications requiring stringent thermal durability and structural integrity, including interior panels and load-bearing components subjected to elevated in-service temperatures.

Keywords: Carbon Fiber Reinforced Polyphenylene Sulfide (CF/PPS); thermoforming; crystallization behavior; thermal stability; aerospace composite; DSC; TGA

INTRODUCTION

In the past four decades, thermoset polymers have been extensively utilized in the aerospace sector due to their well-established processing routes, high stiffness, and good dimensional stability (Hamdan et al. 2022; Sharif et al. 2024). Their widespread adoption in primary and secondary aircraft structures is largely attributed to their ability to form strong covalent crosslinked networks, which provide excellent mechanical integrity under static loading conditions. However, despite these favorable mechanical characteristics, thermosets present several inherent limitations that restrict their broader application in next generation aerospace structures. These include the requirement for low-temperature storage conditions to prevent premature curing, limited shelf life particularly when manufacturing durations exceed approximately 12 hours for large panel fabrication and their inherently irreversible curing nature, which prevents reshaping,

recycling, or reprocessing once polymerization has occurred (Zuhaira, 2020). In addition, thermoset materials such as epoxy systems often exhibit relatively high stiffness but low toughness, which may lead to brittle failure modes under dynamic or impact loading conditions. This behavior becomes especially critical in high-vibration aerospace environments, where cyclic stresses can initiate microcracking and progressive delamination, ultimately leading to structural degradation (Vieille et al. 2017). Moreover, thermosets typically possess inherently low thermal conductivity, which limits their ability to dissipate heat efficiently. This characteristic can constrain their performance in high-temperature or thermally fluctuating environments, where thermal management is a key design requirement (Sharif et al. 2024).

In contrast, thermoplastic polymers have emerged as promising alternatives to thermosets in advanced aerospace applications due to their superior processability, recyclability, and damage tolerance (Kiss et al. 2021; Kang et al. 2022; Sharif et al. 2024). Thermoplastics, which are commonly used in everyday applications such as containers, films, and bottles, offer a significant environmental advantage because they can be reprocessed and recycled multiple times without undergoing permanent chemical degradation (Saccani et al. 2019). Unlike thermoset resins, thermoplastics can be reheated after solidification, allowing reshaping and remolding, which not only provides recycling opportunities but also contributes to reducing material waste sent to landfills (Zuhaira, 2020). This re-meltable characteristic also enables advanced joining techniques such as fusion bonding, in which the material is heated to a temperature near or above its melting point and consolidated under controlled pressure to form a welded interface (Velmurugan et al. 2020). Furthermore, thermoplastics generally exhibit a combination of advantageous properties, including good chemical resistance, wide service temperature capability, and superior impact and damage resistance compared to conventional thermosets, making them highly attractive for structural applications in demanding aerospace environments (Sharif et al. 2024).

Thermoplastic materials are available in a wide range of chemistries, among which high-performance thermoplastics represent a particularly important subgroup for structural engineering applications. Within this category, polyether ether ketone (PEEK), low-melt polyaryletherketone (LMPAEK), and polyphenylene sulfide (PPS) are among the most widely utilized due to their balance of thermal stability, mechanical performance, and processability (Wilkins and Strauss 2023; Pohl et al. 2024; Sharif et al. 2024). In particular, PPS is a semi-crystalline aromatic polymer composed of benzene rings interconnected through sulfide linkages, which contributes to its excellent mechanical strength, chemical resistance, and thermal stability (Hassan et al. 2022).

In aerospace and various industrial sectors, PPS is frequently reinforced with carbon or glass fibres to further enhance its mechanical performance, stiffness, and fatigue resistance (Quan et al. 2020). PPS is especially favored for aircraft structural components due to its unique combination of toughness, low density, abrasion resistance, and high resistance to chemicals and elevated temperatures. As a semicrystalline thermoplastic, PPS exhibits a melting temperature in the range of 280–290°C and maintains stable mechanical performance under harsh operational conditions. In addition, it demonstrates inherent flame-retardant properties, high hardness, and sustained structural integrity under long-term thermal exposure, making it highly suitable for aerospace environments where safety and reliability are critical design requirements (Hamdan et al. 2022). The chemical structure of PPS is illustrated in Figure 1.

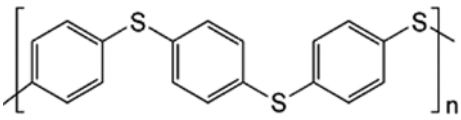


FIGURE 1. Chemical structure of PPS (aromatic ring and sulfur) (Yeole et al. 2020).

Crystallinity and structural integrity, which govern the mechanical performance of thermoplastic composites such as CF/PPS, are strongly influenced by processing conditions, particularly during thermoforming. During the forming stage, the tooling temperature plays a critical role in defining the thermal history of the laminate, thereby controlling heat transfer efficiency, cooling rate, and polymer crystallization kinetics (Hassan, Elagib and Ahmed 2022). A higher tooling temperature generally slows down the cooling process, allowing polymer chains more time to rearrange into ordered crystalline regions, while lower temperatures promote faster solidification and reduced molecular mobility, leading to lower crystallinity. These thermal variations directly influence the internal microstructure of the composite, which ultimately governs its stiffness, toughness, and overall mechanical integrity.

Such microstructural evolution is effectively characterized using thermal analysis techniques, particularly differential scanning calorimetry (DSC) and thermogravimetric analysis (TGA) (Carpier et al. 2020). DSC provides detailed insight into thermal transitions, including melting temperature, crystallization temperature, degree of crystallinity, and heat flow behavior, all of which are sensitive indicators of changes in molecular ordering. In parallel, TGA evaluates the material's thermal stability by measuring weight loss as a function of temperature,

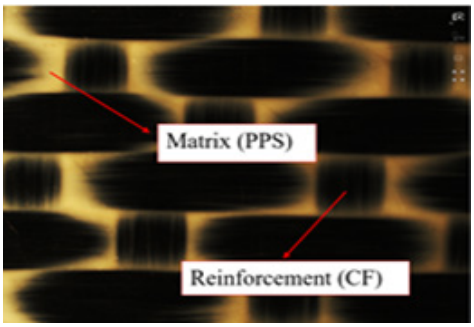
thereby identifying degradation onset and thermal resistance. When tooling temperature is varied during stamping or thermoforming, both DSC and TGA responses exhibit corresponding shifts, reflecting changes in crystallite formation, thermal stability, and overall structural arrangement within the polymer matrix.

These thermally induced structural changes are directly correlated with the final mechanical performance and quality of thermoformed CF/PPS composites. Therefore, precise optimization of tooling temperature, guided by DSC and TGA analyses, is essential to ensure consistent crystallization behavior, improved interfacial bonding, and reliable mechanical properties for high-performance aerospace components (Khan et al. 2016).

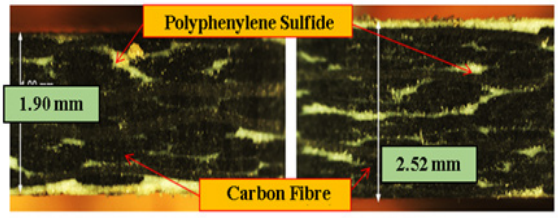
METHODOLOGY

MATERIAL PREPARATION

To characterize the behavior of Carbon Fiber Reinforced Polyphenylene Sulfide composites, this research focused on laminates with nominal thicknesses of 1.90 mm for 6 ply [(0°,90°)/(±45°)/(0°,90°)]s and 2.52 mm for 8 ply [[(0°,90°)/(±45°)]$_2$]s configurations. The composites were subjected to five distinct tooling temperatures of 150 °C, 160 °C, 170 °C, 180 °C, and 195 °C. The thickness of CF/PPS, characterized by a 5-harness satin weave, as illustrated in Figure 2.



Front View CF/PPS



Lateral View CF/PPS

6 ply (b) 8 ply

FIGURE 2. CF/PPS Composite (a) 6 ply and (b) 8 ply

The thermoforming procedure was carried out on the hot-pressed integrated automation machine. The detailed thermoforming process is illustrated in Figures 3 and 4. At first, CF/PPS in the form of Reinforced Thermoplastic Laminate (RTL) is attached to the spring. Next, the integrated rail inside the hot press machine transports the RTL into the Infrared (IR) heater to ensure that the RTL reaches the melting temperature (320°C under Infrared (IR) heater ≤ 3 minutes). Next, the RTL is transported to the pressing area for stamping (135 seconds) at tool temperatures of 150, 160, 170, 180, and 195°C. Lastly, the stamped RTL of CF/PPS undergoes demoulding and cut to specific dimensions for thermal testing by TGA and DSC, following their ASTM standards.

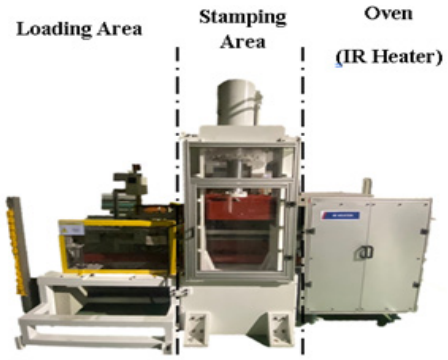


FIGURE 3. Hot Press Machine.

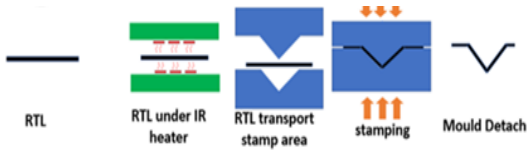


FIGURE 4. Thermoforming Process (Hamdan, Razali, Hamid, et al. 2022).

MEASUREMENT METHOD

The stamped CF/PPS samples were characterised using Thermogravimetric Analysis (TGA) and Differential Scanning Calorimetry (DSC) in accordance with ASTM E1131 and ASTM E3418 to evaluate their thermal stability and crystallisation behaviour. TGA was conducted using a PerkinElmer STA 6000 instrument (USA) with approximately 10 mg of each specimen to ensure consistent thermal response. The samples were heated from 30°C to 992°C at a controlled heating rate of 10°C/min under a nitrogen atmosphere with a flow rate of 100 mL/min. The inert nitrogen environment was used to prevent oxidative degradation and to ensure that the observed mass loss accurately reflected the thermal decomposition behaviour

of the CF/PPS composite. This analysis provides key information such as degradation onset temperature, thermal stability range, and residual char formation.

For DSC analysis, a TA Instruments Q2000 (USA) was employed to investigate the melting and crystallisation behaviour of the stamped products. Samples weighing between 10 and 15 mg were carefully sealed in aluminium pans using a crimping process to prevent material loss during heating. The tests were performed over a temperature range of 30°C to 330°C at a scanning rate of 10°C/min under a nitrogen atmosphere. The degree of crystallinity (DOC) was determined using Equation (1), based on the measured melting enthalpy from the DSC thermogram. This method enables quantitative evaluation of the crystalline fraction within the PPS matrix, which is critical for understanding the thermal history and processing structure relationship of the stamped CF/PPS laminates.

$$x = \frac{\Delta H}{\Delta H_{100\%}(1-W_f)}\, x\ 100\% \quad (1)$$

Where x is the DOC, ΔH is the enthalpy from the melt peak from the DSC curve, $\Delta H_{100\%}$ is the enthalpy of melting of a theoretical 100% crystalline polymer (150.4 J/g), and W_f is fiber content as a percentage of an initial mass from the acid digestion test (0.58). For 6-ply, the W_f is 58.4%, while 8-ply recorded a W_f of 58.2%.

RESULTS AND DISCUSSION

For TGA and DSC, the initial results of the control sample (before stamping) were recorded for comparison and observation before and after the thermoforming process.

TGA TESTING

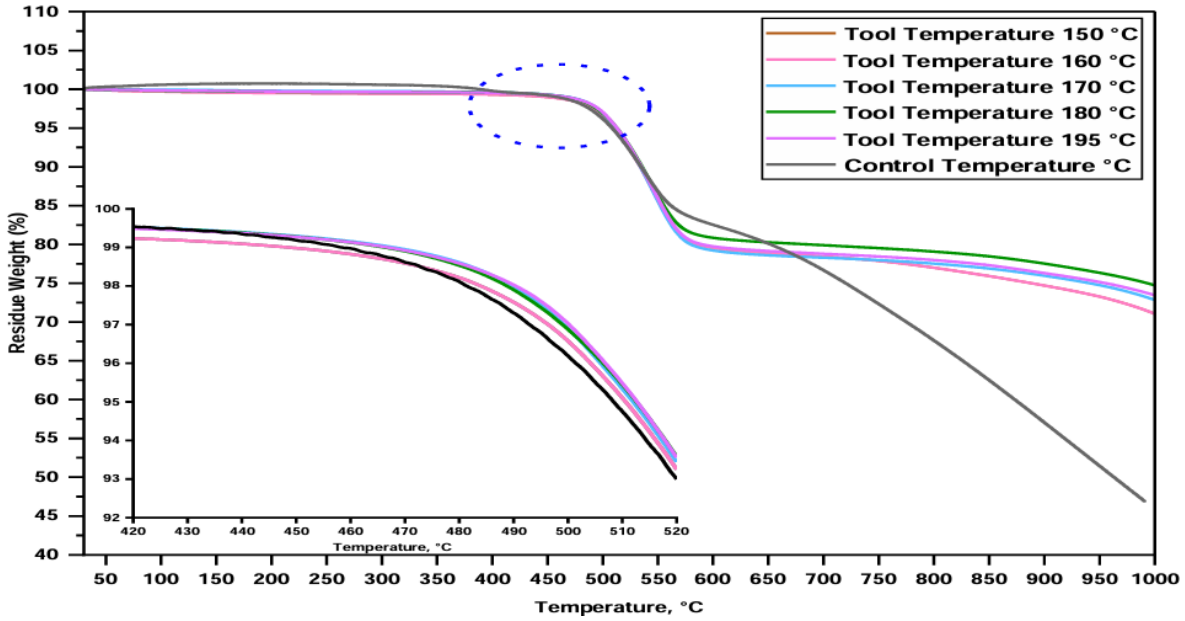


FIGURE 5. TGA curve for 6-ply.

Based on Figure 5, the onset temperature of the pressed CF/PPS 6-ply samples remained consistent at 448.06°C for tool temperatures of 150 and 160°C. As the tool temperature increased, the onset temperature also rose slightly to 450.15°C at 170°C, 455.73°C at 180°C, and 456.30°C at 195°C. In terms of endset temperature, the pressed samples showed a value of 656.74°C at both 150 and 160°C. At 170°C, the endset dropped to 628.62°C, followed by a further decrease to 600.8°C at 180°C, before increasing again to 661.17°C at 195°C. The residue weight also increased with temperature, starting at 71.07% at 150 and 160°C, rising to 72.85% at 170°C, 74.74% at 180°C, and then slightly decreasing to 73.46% at 195°C. When compared to the unpressed sample reported by Hamdan et al. (2022), which recorded an onset of 453.20°C, an endset of 591.09°C, and a much lower residue weight of 46.92%, it is proven that pressing at various tool temperatures enhances the thermal stability and char retention of the CF/PPS composite.

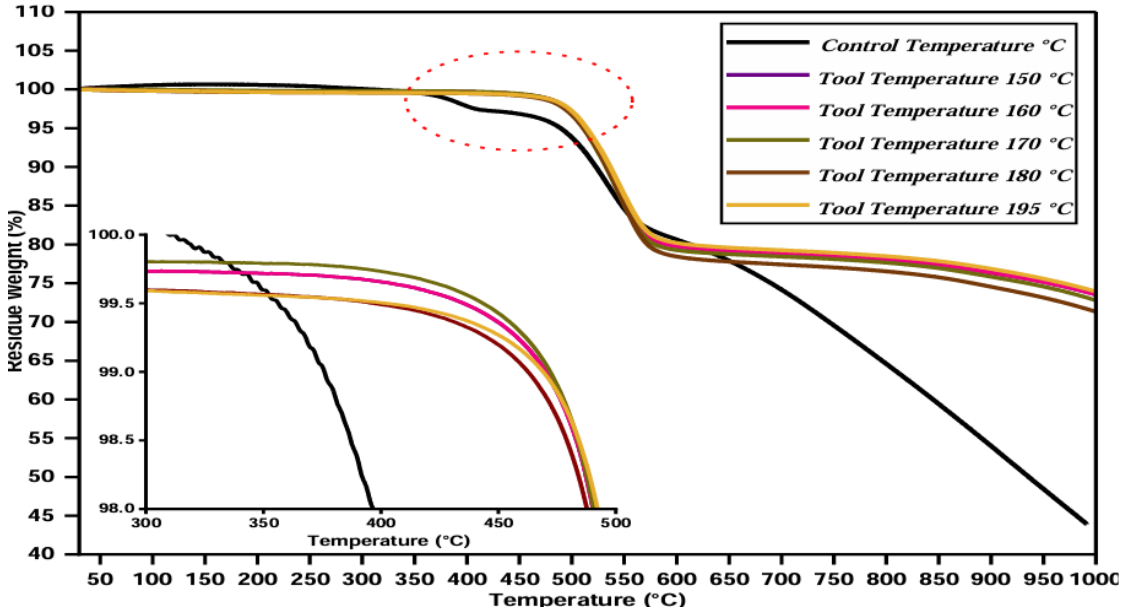


FIGURE 6. TGA curve for 8-ply.

As shown in Figure 6, the thermal behavior of the 8-ply CF/PPS composite also varied with the tooling temperature during thermoforming. At 150 and 160°C, the onset temperature of the pressed samples remained constant at 427.33°C. As the tool temperature increased to 170°C, the onset temperature rose to 438.47°C, followed by 447.14°C at 180°C and 462.16°C at 195°C, reflecting improved thermal resistance with higher pressing temperatures. The endset temperature was highest at 150°C (722.08°C), followed by 667.51°C at 180°C, 655.70°C at 170°C, 624.42°C at 160°C, and the lowest at 195°C (623.10°C). Residue weight remained relatively stable, ranging from 71.32 to 73.93%, with the highest char yield recorded at 195°C. Compared with the unpressed sample reported by Hamdan et al. (2022), which recorded an onset temperature of 412.63°C, an endset of 492.98°C, and a residue weight of 43.94%, the pressed 8-ply samples showed significantly better thermal performance and char retention.

In summary, thermogravimetric analysis (TGA) was employed to evaluate the thermal degradation behavior of CF/PPS composites as a function of temperature for both 6-ply (1.90 mm) and 8-ply (2.52 mm) laminates. Tooling temperature during thermoforming had a significant effect on onset temperature, endset temperature, and final residue weight. For the 6-ply samples, the onset temperature increased from 448.06 to 456.30°C, and the residue weight reached 74.74% at 180°C. Similarly, for 8-ply samples, onset temperatures rose from 427.33°C at 150°C to 462.16°C at 195°C, and char residue reached a maximum of 73.93%. Compared to unpressed controls by Hamdan, Razali, Kamarolzaman et al. (2022), the thermal stability of both pressed configurations was notably improved. The 6-ply control had only 46.92% residue, while the 8-ply control showed 43.94%, confirming that pressing improves char retention. Although both plies demonstrated similar degradation trends, the 6-ply configuration showed a more thermally stable molecular structure. This is evident from the Initial Decomposition Temperature (IDT), which was 453.20°C for the 6-ply and only 347.95°C for the 8-ply. These findings align with those of Rahate et al. (2013), who reported that degradation of pure PPS and PPS composites begins at around 450°C and 465°C, respectively, reinforcing the conclusion that the 6-ply laminate has superior thermal stability.

According to Khan et al. (2016), the thermal degradation of PPS/CF composites involves chain scission, followed by depolymerization and oxidation of the char. Carbon fiber enhances thermal resistance by delaying matrix degradation, as evidenced by higher residue values after pressing. While unpressed samples retained less than 50% of their mass after degradation, pressed samples retained between 70 and 75% of their original weight. The lowest residues were found at 150°C for both 6-ply (70.72%) and 8-ply (69.95%), confirming that higher tool temperatures promote better char formation and improved thermal stability. These observations are consistent with literature reports. Schuhler et al. (2018) and Carpier et al. (2020) stated that PPS-based composite degradation typically begins between 480 and 500°C. The high thermal and dimensional stability of PPS is attributed to its structured backbone of alternating phenylene and sulfide units (Liu and Wang, 2019; Zuo et al. 2019). Most of the thermal degradation in PPS occurs between 350 and 600°C,

primarily through random chain scission and subsequent oxidation of the carbonaceous residue (Vieille et al. 2014). Moreover, Ucpinar and Aytac (2019) reported residue values as high as 76% for PPS/CF composites, closely matching the values recorded in this study. In comparing pressed versus control samples, the increase in final residue was substantial: +59.29% for 6-ply at 180°C and +68.31% for 8-ply at 195°C.

DSC TESTING

TABLE 1. Result of DSC Curve for both 6 and 8 plies CF/PPS.

Thickness	Tool Temperature (°C)	T_g, °C	T_{cc}, °C	T_m, °C	ΔH_{cc}, J/g	ΔH_m, J/g
6-ply (1.90 mm)	150	96.75	227.99	279.59	16.57	13.53
	160	96.15	229.72	279.54	15.99	13.14
	170	97.03	229.89	279.47	16.94	15.05
	180	95.75	232.92	279.92	17.15	12.73
	195	95.24	228.24	279.15	16.57	13.36
8-ply (2.52 mm)	150	97.03	228.24	279.13	16.01	12.72
	160	94.61	232.41	279.96	17.95	14.47
	170	98.39	231.03	279.20	16.89	14.03
	180	95.25	232.21	279.35	18.52	14.67
	195	96.72	231.19	279.91	17.00	13.45

As shown in Table 1, the cold crystallization (T_{cc}) behavior of CF/PPS composites is strongly influenced by the tool temperature. In the 6-ply samples, cold crystallization temperatures increased from 227.99°C at 150°C to 229.72°C at 160°C and peaked at 229.89°C at 170°C, indicating enhanced nucleation and crystalline structure formation. At higher temperatures of 180 and 195°C, the crystallization temperatures increased to 232.92°C and then decreased slightly to 228.24°C, respectively. These shifts suggest that beyond a certain point, competing thermal effects such as partial melting and polymer relaxation begin to influence crystallization kinetics.

The crystallization enthalpy in the 6-ply composite peaked at 16.939 J/g at 170°C, indicating maximum crystal growth at this temperature. The corresponding crystallinity index also increased with tool temperature, reaching its highest at 180°C before slightly declining at 195°C. These patterns suggest that while increasing tool temperature initially improved crystallinity, excessive heat might lead to thermal disruption of ordered molecular alignment, limiting further crystallization. These trends are consistent with the observations of Frolov et al. (2018), who reported incomplete crystallization during rapid cooling at high processing temperatures.

Compared to the 6-ply laminate, the 8-ply samples exhibited consistently higher crystallization temperatures at each corresponding tool temperature. For example, cold crystallization began at 228.85°C at 150°C and peaked at 232.41°C at 160 °C, indicating earlier and more active crystallization in thicker composites. The enthalpy also peaked here, at 17.946 J/g, suggesting optimal nucleation and growth conditions under moderate processing temperatures. As the tool temperature increased to 170, 180, and 195°C, crystallization temperatures remained relatively high at 231.03, 231.21, and 231.19°C, respectively. This stability in thermal response implies that thicker laminates benefit from prolonged heat exposure, facilitating molecular rearrangement and enhancing crystallization.

TABLE 2. Comparison of pressed DOC from DSC curves for both 6 and 8 plies CF/PPS in this study with unpressed samples from literature (Hamdan et al. 2022).

Thickness	Tool Temperature (°C)	Pressed Sample DOC	Unpressed Sample DOC (Hamdan et al. 2022)
6-ply (1.90 mm)	150	20.52	19.10
	160	19.68	
	170	22.84	
	180	20.43	
	195	20.28	

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8-ply (2.52 mm)	150	20.70	18.20
	160	22.82	
	170	21.67	
	180	21.18	
	195	20.90	

Table 2 presents the DSC results for both 6 and 8 plies of CF/PPS composites, illustrating how composite thickness influences the thermal behavior. The crystallinity index remained consistently above that of the 6-ply samples, with values of 20.70, 21.18, and 20.90% at 150, 180, and 195°C, respectively.

These findings align with the work of Seki et al. (2022), who observed that the high thermal conductivity of carbon fibers enables more uniform, albeit slower, heat distribution in thicker laminates. Consequently, heat is retained longer, allowing more time for molecular chains to crystallize. Glass transition temperatures also reflected this behavior, with the 8-ply laminate showing a slightly higher T_g of 98.39°C compared to 97.03°C in the 6-ply sample. Tsukada et al. (2017) similarly reported that increased composite thickness lowers the cooling rate and enhances crystal formation.

In this study, the crystallization process in thermoplastics like CF/PPS occurred when amorphous regions began to form ordered molecular structures, releasing heat. At 150°C, both 6-ply and 8-ply CF/PPS composites exhibit the onset of crystallization, with nucleation initiating small crystalline clusters. However, due to limited molecular mobility, the degree of crystallinity remains relatively low at this stage. Batista et al. (2016) emphasized that carbon fiber surfaces facilitate crystallite nucleation under slow cooling, which supports these observations.

As the tool temperature increases to 160 and 170°C, nuclei expand in both size and quantity, transforming more of the amorphous phase into crystalline regions. This is reflected in the rising crystallization temperatures observed in both ply configurations. Wang et al. (2018) found that slower heating leads to more pronounced cold crystallization, which explains the elevated crystallization peaks at these temperatures.

At elevated tool temperatures of 180 and 195°C, both plies demonstrate continued increments in crystallinity index. However, thermal events such as partial melting and relaxation began to interfere with crystallization, as indicated by a slight drop in crystallization temperatures, particularly in the 6-ply configuration. Theoretical support from Chen, Fu, and Li (2021) confirms that such reductions may result from incomplete crystallization under rapid or excessive heating.

Furthermore, the thermal conductivity differences between carbon fiber and PPS significantly influenced heat flow during DSC testing. As the number of plies increased, the laminate's ability to absorb and redistribute heat increased. This prolonged thermal exposure, particularly in the 8-ply composite, facilitated greater molecular alignment and crystallinity. The observed crystallinity differences of 0.18, 0.75, and 0.62% higher in 8-ply at 150, 180, and 195°C, respectively, reinforce the conclusion that thickness plays a crucial role in thermal behavior and structure development.

CONCLUSION

The tooling temperature and composite thickness play a significant role in governing the thermal behaviour of CF/PPS composites, which is essential for their suitability in aerospace applications. The DSC results indicated that both crystallisation temperature T_{cc} and degree of crystallinity increased progressively with higher tooling temperatures. This trend suggests enhanced molecular mobility and improved chain alignment within the PPS matrix, leading to a more ordered crystalline structure. In addition, thicker laminates (8-ply) generally exhibited earlier onset of thermal transitions and higher crystallinity compared to thinner laminates (6-ply). This behaviour is attributed to slower cooling rates and greater heat retention in thicker samples, which provide sufficient time for crystal growth. In contrast, thinner samples cooled more rapidly, resulting in slightly delayed crystallisation and reduced crystalline development. TGA results further confirmed the beneficial effect of thermoforming on thermal stability. The residual char yield increased significantly from approximately 44–47% in unpressed samples to about 70–75% in pressed samples, accompanied by higher onset and endset degradation temperatures. These improvements indicate enhanced thermal resistance and structural integrity following optimized processing conditions. Overall, the combined DSC and TGA findings demonstrate that appropriate control of tooling temperature and laminate thickness effectively improves crystallisation behaviour and thermal stability. These characteristics are critical for aerospace components such as interior panels, structural brackets, and other load-bearing thermoplastic parts, ensuring dimensional stability, durability, and safe performance under elevated service temperatures.

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DECLARATION OF COMPETING INTEREST

None.

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