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Process–Structure–Property Relationships of CF/PPS Composites Formed by Hot Pressing: Influence of Tool Temperature and Cooling Rate

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ABSTRACT

Carbon fibre-reinforced polyphenylene sulfide (CF/PPS) thermoplastic composites are increasingly adopted in aerospace applications owing to their high specific strength, chemical resistance, and reprocessability. However, the coupled influence of hot-press tool temperature (Tt) on microstructural evolution, interfacial properties, and mechanical performance remains poorly characterised, limiting the ability to define predictive process–structure–property relationships for this material system. This study systematically investigates the effect of tool temperature (150–195 °C) on the cooling rate, degree of crystallinity (DoC), void content (Vc), surface energy and tensile strength of CF/PPS 5-harness satin laminates formed by hot pressing. Increasing Tt from 150 °C to 195 °C reduced the laminate cooling rate from 21.09 °C/min to 14.64 °C/min, representing a 31% reduction. Composite performance responded non-monotonically, with an optimum condition identified at 180 °C. At this temperature, the lowest cooling rate that preceded the performance peak (15.93 °C/min) yielded the highest DoC (22.82%), lowest Vc (0.21%), highest surface energy (35.13 mJ/m²), and maximum tensile strength (794 ± 19.7 MPa). Exceeding this threshold at 195 °C degraded all four properties simultaneously, attributed to excessive molten dwell time promoting void reformation and reducing fibre-matrix interfacial continuity. The results demonstrate that Vc influences fibre–matrix surface energy through contact continuity, which in turn determines tensile performance through load transfer efficiency. This cascade relationship establishes a process–structure–property relationship that identifies 180 °C as the optimal Tt and provides a quantitative basis for process optimisation of hot-pressed CF/PPS composites.

Keywords: Thermoplastic composite; carbon fibre/polyphenylene sulfide (CF/PPS); tool temperature; cooling rate; degree of crystallinity

INTRODUCTION

Carbon fibre-reinforced polyphenylene sulfide (CF/PPS) thermoplastic composites have attracted considerable attention in aerospace and other high-performance structural applications due to their excellent specific strength, chemical resistance, weldability and recyclability. These characteristics also make them attractive for sustainable aerospace manufacturing (Koerdt et al. 2022; Zhao et al. 2022). The thermoplastic nature of polyphenylene sulfide (PPS) permits reversible melting, enabling fast

processing cycles and flexible manufacturing strategies that are advantageous in industrial production (Higuchi et al. 2021; Nakagawa et al. 2019). As the aerospace sector pursues more sustainable manufacturing pathways, the reprocessability of thermoplastic composite systems further strengthens their appeal relative to thermoset alternatives (Ruiz et al. 2023). In addition to their excellent chemical resistance and mechanical performance, CF/PPS composites contribute to aircraft weight reduction and component integration, addressing the growing demand among original equipment manufacturers for improved fuel efficiency and lower lifecycle carbon emissions.

The mechanical performance of CF/PPS laminates is governed not only by fibre architecture and fibre volume fraction but also by several microstructural factors, including matrix crystallinity, fibre–matrix interfacial adhesion and the presence of manufacturing defects such as voids. These parameters collectively influence stress transfer efficiency, damage initiation, and overall structural integrity (Gao et al. 2019; Osti de Moraes et al. 2024). The degree of crystallinity *(DoC)* exerts a particularly strong influence on the stiffness, strength, and dimensional stability of the semi-crystalline PPS matrix: increased crystallinity promotes improved molecular packing and more effective intra-matrix stress transfer, while excessive crystallinity may reduce fracture toughness by increasing chain rigidity (Osti de Moraes et al. 2024; Takayama et al. 2024). Conversely, even modest void contents (*Vc*) act as stress concentrators that accelerate crack initiation and cause disproportionate reductions in tensile strength (de Melo Morgado et al. 2024; Harris et al. 2020). In addition to crystallinity and *Vc*, surface energy governs fibre–matrix wetting and interfacial adhesion, directly influencing consolidation quality and load transfer efficiency. Despite its importance, the effect of processing conditions on surface energy in CF/PPS systems and its relationship to mechanical performance remain poorly characterised (Alipour & Jayaraman 2024; Banerjee et al. 2022).

In hot-press forming, the tool temperature (*Tt*) governs the thermal boundary condition imposed on the laminate during consolidation, thereby determining the cooling rate, crystallisation kinetics, resin flow behaviour and void evolution. Processing above the PPS melting temperature (*Tm*) ensures complete matrix melting and adequate resin flow, after which the cooling rate — controlled primarily by *Tt* — dictates the final DoC and *Vc* of the consolidated laminate (Hamdan et al. 2023; Koerdt et al. 2022; Nakagawa et al. 2019). Slower cooling, achieved through higher *Tt*, extends the crystallisation window and provides greater molecular mobility for crystalline ordering and void consolidation; however, excessively slow cooling may promote detrimental microstructural changes such as gas entrapment or heterogeneous void reformation (Hong & Cho 2023; Yi et al. 2024). Consequently, *Tt* must be carefully optimised to balance crystallisation behaviour, consolidation quality and demoulding efficiency, as inadequate thermal control leads to variability in *DoC*, heterogeneous void distributions, and inconsistent mechanical performance (Hamdan et al. 2023; Koerdt et al. 2022).

Although prior studies have examined crystallinity, void formation and mechanical performance in thermoplastic composites individually, few investigations have systematically characterised the coupled influence of *Tt* on both microstructural evolution and mechanical behaviour, creating a gap in process–structure–property understanding for CF/PPS laminates (Ruiz et al. 2023; Zhao et al. 2021). In particular, the role of surface energy as an intermediate variable linking consolidation quality to mechanical performance has not been quantitatively established for hot-pressed CF/PPS systems. This gap limits the ability to predict and optimise composite performance based on processing parameters alone. Filling this knowledge gap is particularly important as hot-press consolidation is increasingly adopted as a rapid and tooling-efficient alternative to autoclave processing, where robust process control is essential to ensure consistent part quality.

To address this gap, the present study pursues three specific objectives: (i) to quantify the effect of hot-press *Tt* (150–195 °C) on the cooling rate and *DoC*; (ii) to assess the influence of cooling rate and *Tt* on *Vc* and surface energy; and (iii) to establish quantitative correlations between these microstructural and interfacial parameters and the resulting tensile strength, with the aim of identifying the optimal processing condition and establishing a quantitative process–structure–property relationship for hot-pressed CF/PPS laminates.

METHODOLOGY

MATERIAL

The material used in this study was a CF/PPS laminate comprising six plies of 5-harness satin (5HS) woven carbon fibre fabric impregnated with a PPS matrix and arranged in a symmetric stacking sequence of [(0,90)/(±45)] s, with a nominal thickness of 1.90 mm. The fibre volume fraction was 50%. The densities of the carbon fibre and PPS matrix were 1.55 g/cm³ and 1.35 g/cm³, respectively. The PPS matrix exhibited a glass transition temperature (*Tg*) of 90 °C and a *Tm* of 280 °C (Toray Advanced Composites 2024).

HOT PRESS FORMING PROCESS

The CF/PPS composite laminates were fabricated using the hot-press forming method as shown in Figure 1. Prior to forming, laminates were heated to 320 °C using an infrared oven to ensure complete melting of the PPS matrix. Consolidation was performed using a hydraulic hot press under constant pressure, pressing time (135 s) and closing speed (24 mm/s). Tool temperatures of 150 °C, 170 °C, 180 °C, and 195 °C were investigated to isolate the influence of thermal boundary conditions. Temperature evolution during cooling was monitored using a surface-mounted thermocouple. The cooling time interval was measured from the laminate temperature during hot pressing (considered as Tt) until the laminate reached ambient temperature (34 °C).

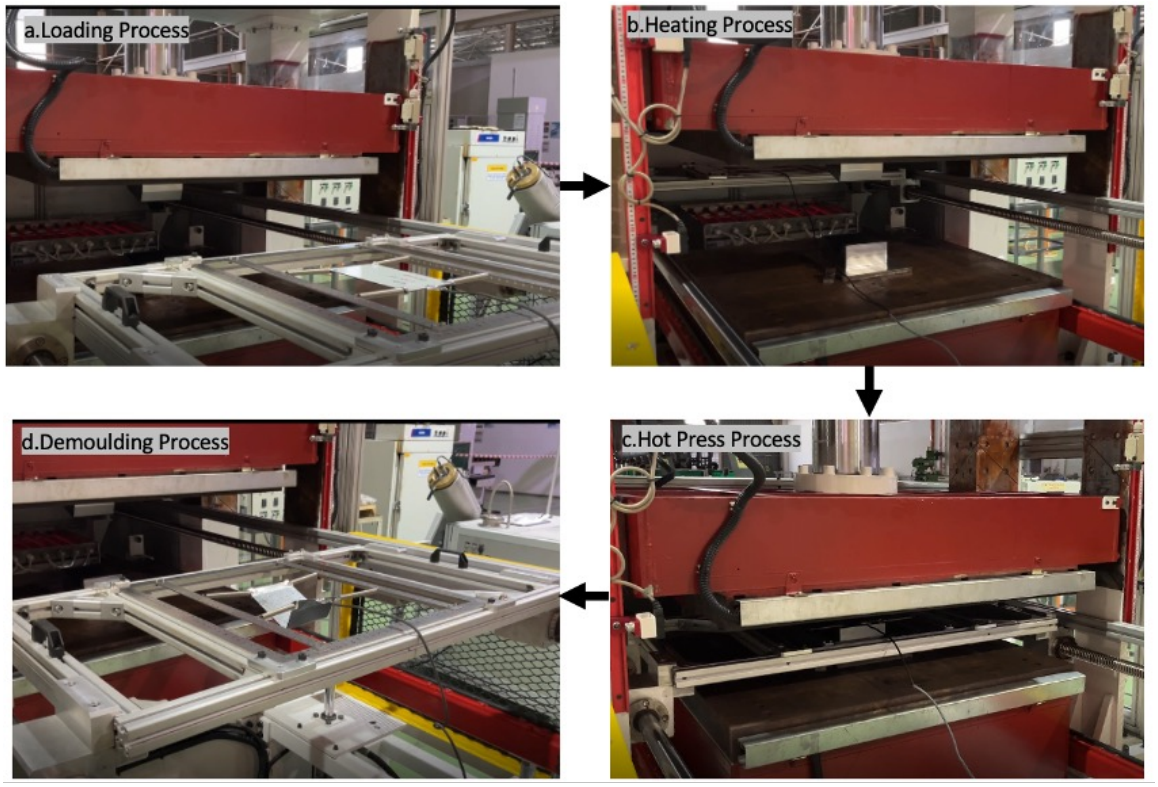


FIGURE 1. Schematic illustration of the hot-press forming process used for CF/PPS laminate fabrication

CHARACTERISATION TECHNIQUE

All specimens were extracted from the consolidated CF/PPS panels manufactured at each *Tt* to ensure material consistency. Waterjet cutting was employed to produce smooth specimen edges without inducing thermal damage to the matrix.

The *DoC* was determined using Differential Scanning Calorimetry (DSC) based on the melting enthalpy derived from the DSC curve, with a mass of 7–10 mg. The samples were sealed in aluminium pans and tested under a nitrogen atmosphere. Then, the samples were heated from room temperature to 320 °C at a rate of 20 °C/min, followed by a 5 min isothermal hold and subsequent cooling at a rate of 20 °C/min. The *DoC* was calculated using the reference enthalpy of fully crystalline PPS of 150.4 J/g (Chadwick et al. 2021) and the fibre mass fraction of 0.58 obtained from the acid digestion test (*Vc* test). The *DoC* was calculated using the following equation:

$$DoC, \% = \frac{\Delta H_m}{\Delta H100\% \ (1 - W_f)} \ X \ 100\%$$

where:
ΔH_m = melting enthalpy in J/g obtained from the DSC curve
W_f = fibre content as a percentage of initial mass obtained from acid digestion test
$\Delta H100\%$ = melting enthalpy of 100% crystalline PPS (150.4 J/g)

Vc was determined in accordance with EN 2564 using the acid digestion method, with a minimum sample mass of 1 g. Surface energy was evaluated by the sessile drop method using distilled water and acetone as probe liquids, with values calculated using the Owens–Wendt approach.

Tensile properties were characterised in accordance with EN 2561 using a universal testing machine, with specimen dimensions of 250 mm × 15 mm. For each characterisation technique, two replicate specimens were tested per condition for *Vc* and DSC measurements and five specimens per condition for tensile testing, with the mean value reported in all cases.

RESULTS AND DISCUSSION

The results presented in Table 1 demonstrate the coupled influence of *Tt* on the cooling rate, *DoC*, *Vc*, surface energy and tensile strength of hot-pressed CF/PPS laminates. As the *Tt* increased from 150 °C to 195 °C, the cooling rate progressively decreased from 21.09 °C/min to 14.64 °C/min, representing a reduction of approximately 31%. This slower cooling promoted changes in crystallinity, consolidation quality, fibre–matrix interfacial properties and ultimately tensile performance. Importantly, the results reveal an optimum processing condition at 180 °C rather than a continuously improving trend, indicating that a balance between crystallisation kinetics and consolidation behaviour governs composite performance.

TABLE 1. Summary of experimental results for CF/PPS laminates fabricated at different tool temperatures

Tool Temperature, ºC	Cooling Rate, ºC/min	Degree of Crystallinity, %	Void Content, %	Surface Energy, mJ/m²	Tensile Strength, MPa
150	21.09	20.70	0.65	24.88	746 ± 21.9
170	16.16	20.90	0.52	29.11	780 ± 31.7
180	15.93	22.82	0.21	35.13	794 ± 19.7
195	14.64	21.18	0.41	29.28	789 ± 34.7

The cooling rate exhibited an approximately linear decrease with increasing *Tt*, indicating that *Tt* effectively governs the thermal history during hot-press consolidation (Hamdan et al. 2023; Yi et al. 2024). The slower cooling conditions extend the crystallisation window and provide more favourable conditions for molecular rearrangement and resin flow. However, as discussed in subsequent sections, further reductions in cooling rate beyond a critical threshold do not yield additional structural or mechanical benefits, and may in fact compromise consolidation quality.

As shown in Figure 2, increasing the *Tt* progressively reduced the laminate cooling rate from 21.09 °C/min at 150 °C to 14.64 °C/min at 195 °C. This decrease is attributed to the reduction in the thermal gradient between the molten CF/PPS laminate (approximately 320 °C) and the forming tool. A smaller temperature difference lowers the rate of heat extraction, extending the crystallisation window and allowing the polymer chains additional time for molecular rearrangement before solidification (Hong & Cho 2023; Nakagawa et al. 2019). The observed relationship between *Tt* and cooling rate appears linear across the evaluated range, indicating that the *Tt* effectively controls the thermal history during hot-press consolidation (Hossain et al. 2025; Ren et al. 2024).

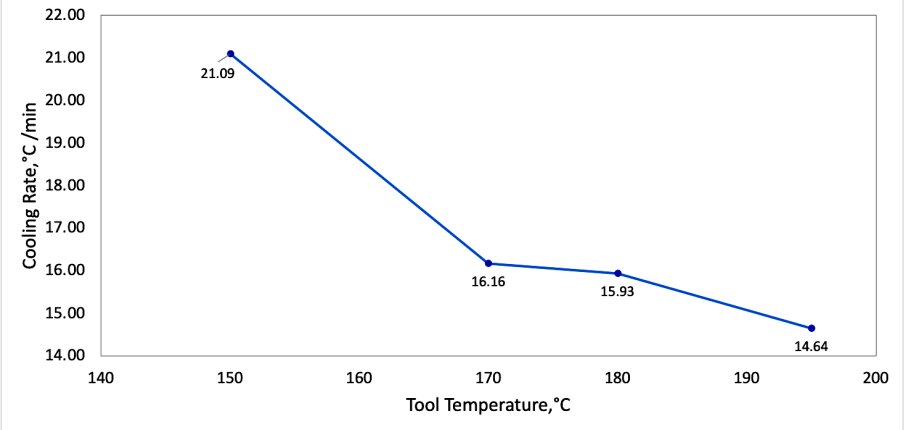


FIGURE 2. Effect of *Tt* on CF/PPS laminate cooling rate during hot-press consolidation

DEGREE OF CRYSTALLINITY

The crystallisation behaviour of the PPS matrix is strongly "dependent on cooling rate which is governed by the *Tt"*, as shown in Figure 3. At the highest cooling rate of 21.09 °C/min (150 °C), the *DoC* was 20.70%, indicating that the relatively rapid cooling restricted sufficient molecular diffusion for crystallisation (Shi et al. 2025). As the cooling rate decreased to 16.16 °C/min at 170 °C, the *DoC* increased marginally to 20.90%, suggesting that this cooling rate remained above the threshold required to substantially extend the crystallisation window. A pronounced increase in *DoC* to 22.82% was recorded at a cooling rate of 15.93 °C/min (180 °C), representing the peak crystallinity across all conditions and an increase of approximately 10% relative to the 150 °C condition. The extended time allowed PPS chains greater mobility to rearrange into ordered crystalline regions, thereby improving the structural organisation of the matrix (Yu et al. 2023). However, as the *Tt* increased to 195 °C and the cooling rate decreased further to 14.64 °C/min, the *DoC* fell to 21.18% with a reduction of 1.64% from the 180 °C optimum despite the slowest cooling conditions in the study. This decline suggests that the relationship between cooling rate and crystallinity in PPS is not monotonic, showing that an optimum cooling window exists for PPS crystallisation, beyond which further reductions in cooling rate provide limited structural benefit (Ren et al. 2024; Zhai et al. 2023). This trend is consistent with the crystallisation behaviour of semi-crystalline polymers, whereby an optimal cooling rate is required to balance nucleation and spherulitic growth, while cooling rates that are too rapid or too slow reduce the final degree of crystallinity.

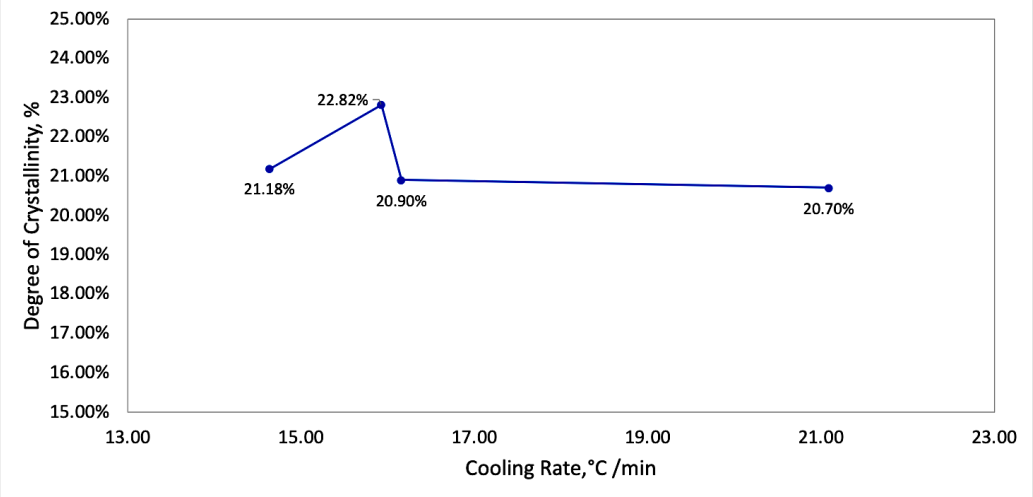


FIGURE 3. Effect of cooling rate on *DoC* of CF/PPS laminates

VOID CONTENT AND SURFACE ENERGY

The influence of *Tt* on laminate consolidation is illustrated in Figure 4 through the measured *Vc* and surface energy. *Vc* decreased substantially from 0.65% at 150 °C to 0.21% at 180 °C, representing a reduction of approximately 68%. This improvement is attributed to enhanced resin flow, improved fibre wetting and more effective consolidation resulting from the slower cooling conditions (Hong & Cho 2023; Shi et al. 2025). Correspondingly, the surface energy increased from 24.88 mJ/m² to 35.13 mJ/m², an increase of approximately 41%, indicating enhanced interfacial adhesion and stronger fibre–matrix adhesion (Yu et al. 2023). Higher surface energy is consistent with improved molecular interaction at the interface, facilitating more efficient stress transfer during mechanical loading.

At 195 °C, however, a reversal was observed: *Vc* increased to 0.41% and surface energy decreased to 29.28 mJ/m², indicating a deterioration of consolidation quality despite the further reduction in cooling. This behaviour is likely associated with the competing effects of extended molten dwell time at very low cooling rates. While moderate reductions in cooling rate favour void consolidation through improved resin flow, excessively prolonged time in the fully molten state reduces the matrix viscosity to a level at which the applied consolidation pressure becomes less effective at collapsing residual voids. Furthermore, the extended thermal exposure may increase the probability of thermally dissolved gases or residual moisture nucleating as stable bubble populations before the matrix viscosity rises sufficiently to suppress their growth during solidification (Backmann et al. 2025;

Nakagawa et al. 2019). The resulting increase in *Vc* at 195 °C reduces fibre–matrix contact area and therefore lowers the measured surface energy, consistent with the view that surface energy in fibre-reinforced laminates is sensitive to interfacial continuity at the microscale. This finding emphasises that void content should be considered not merely as a measure of manufacturing defects but as a governing parameter that regulates interfacial wetting, thereby providing important guidance for balancing dwell time and consolidation pressure to achieve optimal laminate consolidation.

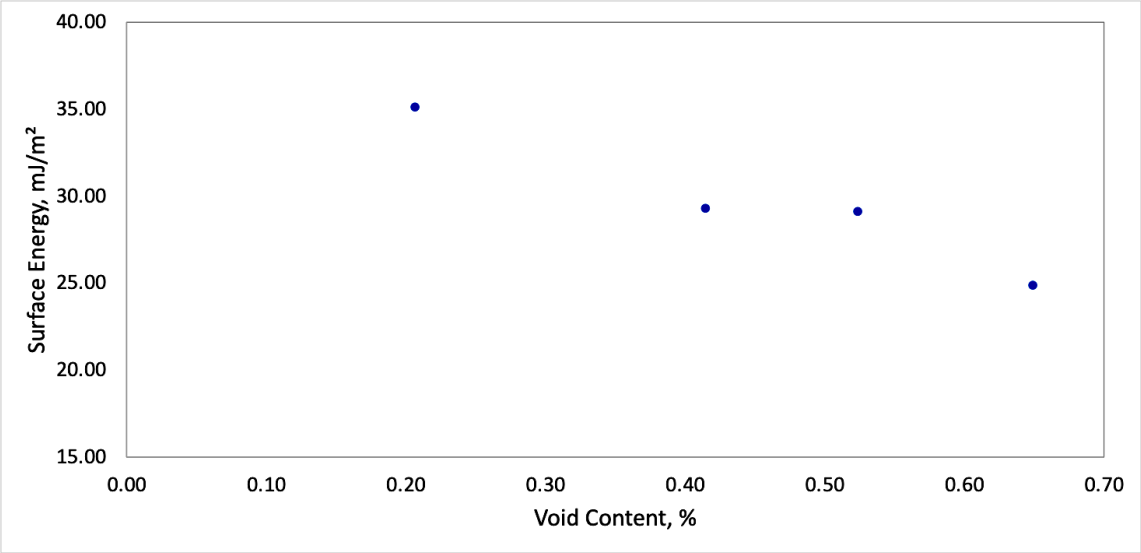


FIGURE 4. Relationship between *Vc* and surface energy of CF/PPS laminates

VOID CONTENT AND TENSILE STRENGTH

The tensile strength results presented in Figure 5 demonstrate a consistent increase from 746 ± 21.9 MPa at 150 °C to a maximum of 794 ± 19.7 MPa at 180 °C, representing an improvement of approximately 6.4%. The enhanced tensile performance is aligned with lower *Vc* and improved surface energy across the same temperature range. This increase suggests that *Vc* reduces the density of stress concentrators within the laminate, while higher surface energy reflects improved fibre–matrix interfacial adhesion, both of which contribute to more efficient load transfer across the fibre–matrix interface during tensile loading (Shi et al. 2025).

When the *Tt* was increased to 195 °C, tensile strength decreased slightly to 789 ± 34.7 MPa. Although the absolute reduction of 5 MPa is modest, it corresponds directly with the increased *Vc* (0.41%) and reduced surface energy (29.28 mJ/m²) observed at this condition, suggesting that the optimum processing window had been exceeded. These results demonstrate that maximum tensile performance in hot-pressed CF/PPS laminates is governed not by the lowest achievable cooling rate, but by the optimal balance between crystallisation extent, void consolidation and interfacial adhesion (Osti de Moraes et al. 2024; Zhao et al. 2021).

These results reflect the combined influence of *DoC*, *Vc*, and fibre–matrix interfacial bonding on the tensile properties of CF/PPS laminates. *Vc* is closely associated with fibre–matrix contact continuity, which in turn determines surface energy and, ultimately, the efficiency of load transfer and tensile strength. This cascade relationship is consistent with previously reported observations of microstructure–mechanical property linkages in thermoplastic composite systems (Zhai et al. 2023).

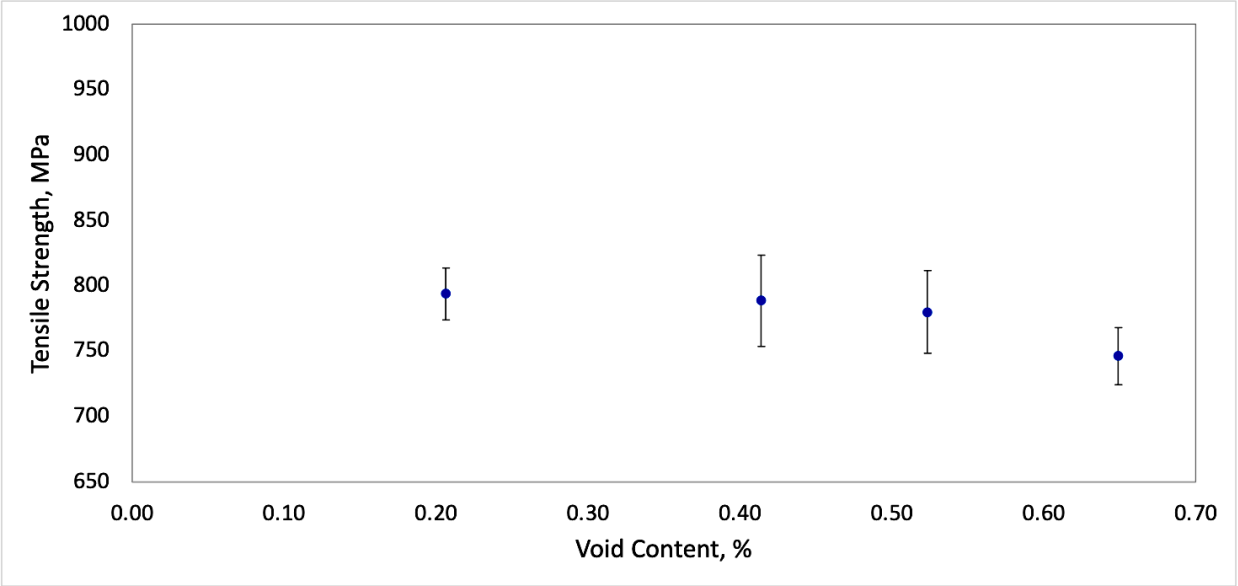


FIGURE 5. Relationship between *Vc* and tensile strength of CF/PPS laminates

PROCESS–STRUCTURE–PROPERTY TREND

To provide an integrated comparison of the relative influence of *Tt* on thermal, structural, interfacial and mechanical properties, the experimental results were normalised where 150 °C as the baseline condition, as shown in Figure 6. The integrated analysis illustrates a typical process–structure–property relationship. Normalising the experimental data reveals that while cooling rates monotonically decrease with increased *Tt*, structural and mechanical properties exhibit non-linear trends. At the identified optimal processing condition of 180 °C, the peak in normalised *DoC*, surface energy and mechanical integrity aligns with minimal *Vc*.

Increasing the *Tt* first modifies the processing condition by reducing the cooling rate. The slower cooling subsequently influences the microstructure by promoting crystallisation and reducing void formation through improved resin flow and fibre wetting. These structural improvements enhance the interfacial properties, as reflected by increased surface energy, which facilitates more efficient stress transfer across the fibre–matrix interface (Bodaghi et al. 2023; Ma et al. 2025). However, further increasing the *Tt* to 195 °C provides only limited additional crystallisation while promoting a slight increase in *Vc* and a reduction in surface energy. These competing effects ultimately reduce the mechanical benefits, suggesting that composite performance is regulated by process–structure–property relationships rather than by *Tt* alone (Shi et al. 2025; Yu et al. 2023). Overall, the results demonstrate that cooling rate acts as the critical link between processing conditions and composite performance. Careful control of *Tt* enables optimisation of crystallisation behaviour, consolidation quality, interfacial bonding and mechanical properties, thereby establishing a clear process–structure–property relationship for hot-pressed CF/PPS composites (Parodo et al. 2024; Zhai et al. 2023). From an industrial perspective, these findings demonstrate the potential of cooling-rate monitoring as a real-time, non-destructive tool for assessing laminate quality during hot-press consolidation. This approach may minimise the need for extensive destructive post-fabrication characterisation, such as acid digestion and DSC analysis, by providing an efficient means of monitoring process-induced material changes.

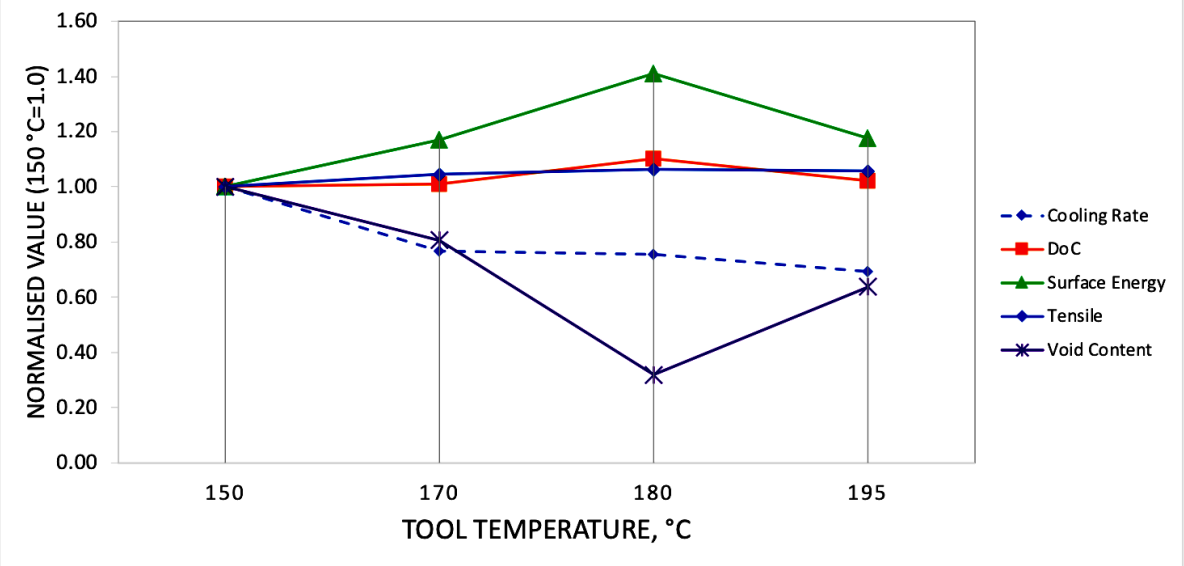


FIGURE 6. Normalised process–structure–property trends showing the influence of *Tt* on cooling rate, *DoC*, *Vc*, surface energy and tensile strength, normalised to the 150 °C condition

CONCLUSION

This study established quantitative process–structure–property relationships for hot-pressed CF/PPS laminates across *Tt* of 150–195 °C. Increasing *Tt* reduced the cooling rate from 21.09 °C/min to 14.64 °C/min, but CF/PPS performance responded non-monotonically. At 180 °C, the optimal balance between crystallisation kinetics and consolidation quality was achieved, yielding the highest *DoC* (22.82%), lowest *Vc* (0.21%), highest surface energy (35.13 mJ/m²) and maximum tensile strength (794 ± 19.7 MPa). Exceeding this threshold at 195 °C degraded all four properties simultaneously, suggesting that excessively slow cooling may compromise, rather than improve laminate quality.

The results demonstrate that CF/PPS performance is governed by the coupled interaction of cooling rate, matrix crystallinity, void consolidation, and fibre–matrix interfacial bonding. Within the investigated range, 180 °C was identified as the optimum *Tt*, establishing a clear process–structure–property relationship for hot-pressed CF/PPS laminates. The outcomes of this study offer practical insights for aerospace and industrial manufacturers seeking to optimise hot-press cycle conditions. The findings provide an evidence-based approach for selecting suitable tool temperatures to enhance laminate performance while reducing processing time and energy requirements.

Notwithstanding these findings, several limitations should be acknowledged. The study examined only four discrete *Tt values*, limiting the resolution of the optimum identification, and mechanical characterisation was restricted to uniaxial tensile properties. In addition, the absence of SEM analysis limited the direct correlation between the observed mechanical behaviour and microstructural features, such as fibre–matrix interfacial bonding, fracture mechanisms, and void morphology. Finally, a single laminate architecture was investigated, and the established process–structure–property relationship may not transfer directly to other lay-up configurations or loading conditions. Future work should expand the temperature range with finer increments, increase replication across all characterisation techniques, and validate the process–structure–property relationship across additional CF/PPS architectures.

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DECLARATION OF COMPETING INTEREST

None.

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