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Physical, Thermal and Mechanical Properties of Carbon Fibre/Polyphenylene Sulfide (CF/PPS) Composite at Different Tool Temperatures Fabricated by Hot Press

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ABSTRACT

Carbon fibre/polyphenylene sulfide (CF/PPS) composites have gained the attention in the aerospace industry due to its excellent impact strength, fire/smoke/toxicity (FST) performance, and chemical resistance. This study investigated the effect of hot press tool temperatures (T_t) on the physical, thermal, and mechanical on CF/PPS composites. The experimental procedure involved using PCL of CF/PPS composite and subjecting it to hot press forming at different tool temperatures ranging from 150°C to 195°C. The physical properties was characterized by surface energy measurement using the sessile drop method. Thermal properties were evaluated through the degree of crystallinity (*DoC*) determination using differential scanning calorimetry (DSC) and thermogravimetric analysis (TGA) where the percentage of the residual weight is calculated. The mechanical properties was assessed using tensile test. From the results, increasing the T_t will enhances the surface energy, improving the adhesion and bonding between the matrix and CF, and the thermal stability of the composite materials. At Tt 180°C, the physical, thermal, and mechanical properties of CF/PPS composite component is at their best with the value of surface energy is 35.13%, the DoC is 25.00%, TGA is 74.74% and tensile strength is 793.80 MPa. This study provides valuable insights into the relationship between tool temperature and the physical, thermal, and mechanical properties of CF/PPS composites, offering guidance for manufacturing processes and composite material design in aerospace and other industries.

1. Introduction

In recent years, carbon fibre reinforced thermoplastic resin (CFRTP) with aerospace grades such as carbon fibre/polyphenylene sulfide (CF/PPS), carbon fibre/polyetheretherketone (CF/PEEK),

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carbon fibre/polyetherketoneketone (CF/PEKK), and the newest carbon fibre/low melt polyaryletherketone (CF/LMPAEK) had gained the attention in aerospace industries for manufacturing of advanced composite components. These materials are semi-crystalline engineering thermoplastics. It exhibits higher impact strength, fire/smoke/toxicity (FST) performance, and chemical resistance. The chain structure within the polymer enables them to retain a significant portion of their strength and stiffness above their glass transition temperature (T_g). On the other hand, thermoset such as epoxy decomposes when exposed to temperatures above its T_g [1–3]. A comparison between the thermoplastic composite and thermoset composite manufactured via compression moulding has been investigated. Based on the results, thermoplastic composite is more recommended for aerospace applications due to it having better mechanical and thermal properties compared to thermoset composite [4].

The main driver for using pre-consolidated laminate (PCL) and thermoforming technologies is the potential for faster cycle time, low recurring cost of manufacturing, and high level of process control and automation. The process parameters are crucial to producing a good quality of final product. It is not only limited to the state of the art on the physical properties of the final product such as visual appearance, thickness, and dimensionality [5–7] but of utmost importance is the mechanical properties. Studies showed that tool temperature affects the mechanical properties of laminates. This is because it influences the cooling rate of the formed components, resulting in different crystallization levels [8–10]. The investigation on CF/PPS composites manufactured using automated fibre placement by Zhao *et al.,* [11] revealed that the tool temperature is the main factor influencing the crystallinity, and the properties of the CF/PPS composite are significantly affected by the crystallinity. Lona Batista *et al.,* [8] also found that lower cooling rates during hot compression moulding processing resulted in higher *DoC* in the CF/PPS composites with the mechanical properties improved for the lower cooling rates. It showed that by changing the cooling rate during processing, it is possible to induce a significant improvement in the mechanical resistance of the CF/PPS composite. As reported by Saenz-Castillo *et al.,* [12], the CF/PEEK composites fabricated by different manufacturing technologies including vacuum bag only, hot press, and automatic lay-up with in-situ consolidation showed different behaviors in terms of mechanical properties and void content. It is crucial to understand the effect of tool temperature on the physical, mechanical, and thermal properties of CF/PPS composites manufactured by the hot press forming process. Additionally, the components made of CF/PPS composites uphold a significant cost reduction for manufacturing and operational as well as environmental sustainability. However, limited studies have been conducted locally in Malaysia on aerospace-grade thermoplastic composite materials.

Therefore, this study aims to investigate the effect of hot press tool temperature on the physical, mechanical, and thermal properties of CF/PPS composite. CF/PPS composite in the form of PCL was used to manufacture the components by the hot press forming process at different tool temperature ranges from 150°C to 195°C. The characterization of physical properties was evaluated in terms of surface energy (SE) while the degree of crystallinity (DoC) and thermogravimetric analysis (TGA) were conducted for thermal characterization. Tensile test was performed using a universal testing machine (UTM) to determine the mechanical properties of CF/PPS composites. Finally, the correlation between SE, DoC, TGA and tensile strength was discussed.

2. Methodology

2.1 Material

CF/PPS composites in the form of PCL with size 300 X 300 mm were used for this study. The resin content by volume (RC), fibre content as a percentage of an initial mass (W_f), and thermal properties

such as T_g and melting temperature (T_m) were evaluated, and the data obtained is summarized in Table 1.

Table 1
Detail of the Incoming PCL

Material	Thickness	RC	W_f	T_g	T_m
5 harness (5H) CF/PPS	1.90 mm	43%	58 %	93°C	282°C

2.2 Manufacturing Process

The test panels were manufactured using a hot press machine with an infrared oven for heating the material. The machine had two hot platens (upper and lower) with aluminium tools installed on them. The upper and lower tools were aligned using an aligning block. The machine was programmed with a specific process cycle tabulated in Table 2.

Table 2
Processing Parameters

Sub-Process	Stages	Parameter
Oven	Heating Temperature	320°C
Hot Press	Tool Temperature	150°C, 170°C, 180°C and 195°C
	Tool Closing Speed	24mm/s
	Pressing Time	135s
Cooling	Cooling Temperature	Natural Cooling at Ambient

Before the pressing started, the tools were heated to desired temperatures, between 150°C and 195°C. The material was held in frames by springs. The material was then heated in an oven and transferred to the press area. In the press area, the heated material was pressed by the upper tool for 135 seconds. After that, the test panel was allowed to cool down at room temperature before the demoulding process. Figure 1 gives an overview of the entire process.

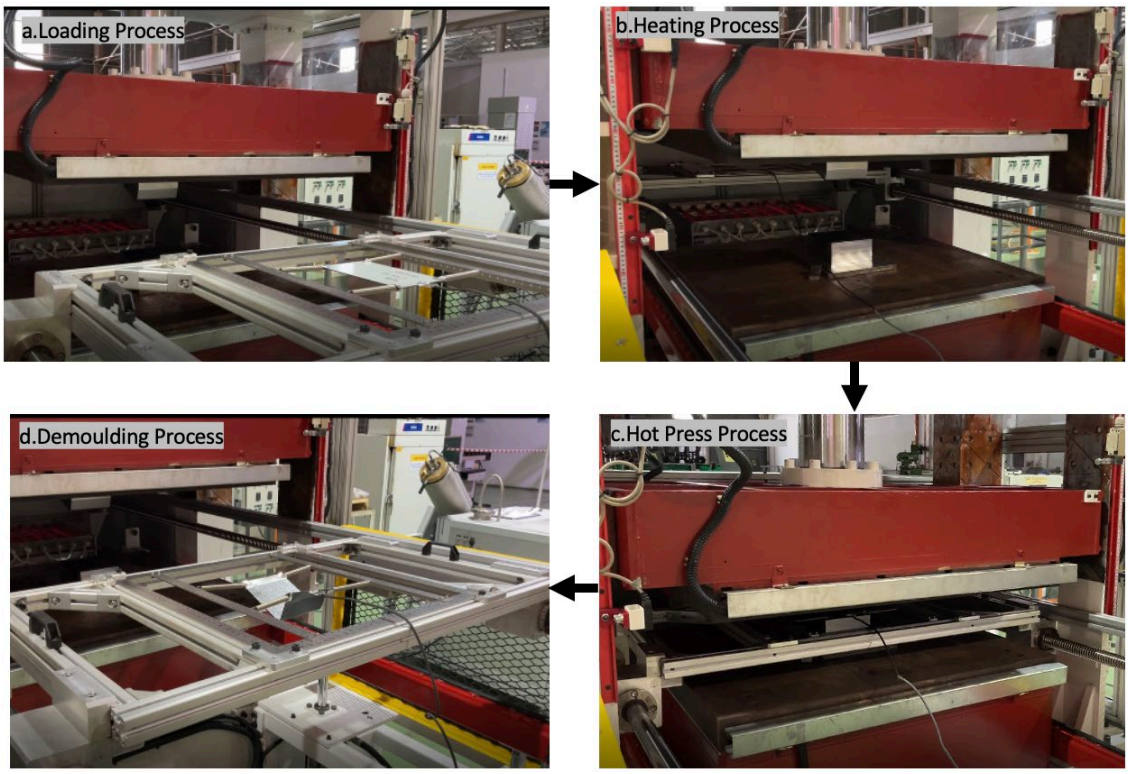

Fig. 1. Overview of Hot Press Forming Process

2.3 Evaluation and Characterization

The press-formed CF/PPS composite components were identified as in Table 3. Based on the data, components Tt150, Tt170, Tt180, and Tt195 all exhibited relatively similar pressure measurements ranging from 4.10 bar to 4.60 bar.

Table 3
Identification of Press-Formed CF/PPS Composite Components

Tool Temperature, °C	Component Identification (ID)	Pressure Measured, bar
150	Tt150	4.1
170	Tt170	4.1
180	Tt180	4.6
195	Tt195	4.5

2.4 Physical Properties Characterization

The sessile drop method was used to determine the surface energy (SE). Contact angle measurements were obtained using water and acetone as the test liquids. The droplet volumes used for the experiment were 5 microliters (µL) for water and 10 µL for acetone. A self-fabricated contact angle measurement tool was utilized to capture the droplet image, and the contact angle was determined using ImageJ software, as depicted in Figure 2. The SE was calculated following Owens – Wendt calculation. The dispersion and polar components for water and acetone from Gurau *et al.,* [13] were used for the calculation as shown in Table 4.

Table 4
Dispersive and Polar of the Test Liquids

Liquid	Liquid Dispersion, γ^d_L	Liquid Polar, γ^p_L	Surface Energy, γ_L
Water	19.9 mJ/m^2	52.2 mJ/m^2	72.1 mJ/m^2
Acetone	17.3 mJ/m^2	6.4 mJ/m^2	23.7mJ/m^2

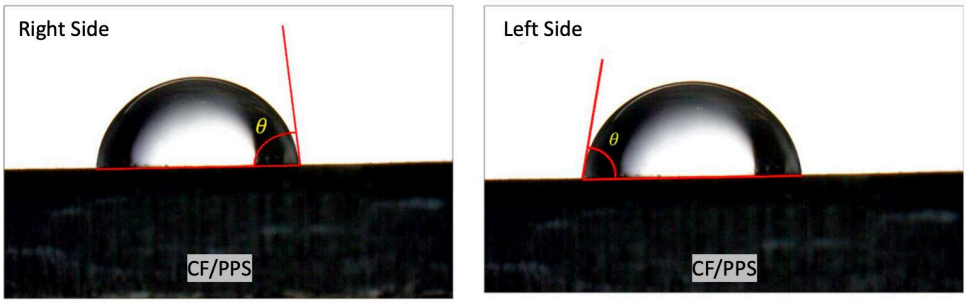

Fig. 2. Contact Angle of Water droplet at Right Side and Left Side

2.5 Thermal Properties Characterization

The degree of crystallinity (DoC) was determined using DSC equipment from TA Instruments. Each sample with a mass of 7-10 mg was placed in a sample pan with good thermal contact with the pan. The sample was tested under a dry inert gas with a heating rate of 20°C/min starting at room temperature (RT) up to 320°C followed by isothermal for 5 minutes and a cooling process. The data obtained from the thermal curved was used for the calculation of DoC using Eq. (1)

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

where ΔH_m is the melting enthalpy in J/g obtained from the DSC curve, W_f is the fibre content as a percentage of an initial mass attained from the acid digestion test (0.58) and $\Delta H100\%$ is the 100% crystalline PPS in J/g based on the recent data of 150.4 J/g [12]

Thermogravimetric Analysis (TGA) test was conducted by using TGA Perkin Elmer STA 6000 machine in accordance with ASTM E1131. The sample was tested under a dry inert gas with a heating rate of 10 C/min between temperatures 30°C to 990°C until a mass loss plateau is reached and then the environment was switched from inert to reactive (oxygen or air) environment. The ash content was calculated using Eq. (2)

$$Ash\ Content\ (A), \% = \frac{W_r}{W_i}\ X\ 100\% \quad (2)$$

where W_r is the mass measured at plateau temperature and W_i is the original mass of samples.

2.6 Mechanical Properties Characterization

The tensile test was conducted in accordance with ASTM D3039 with a test speed of 2mm/min using a Universal Test Machine (UTM) Instron® 5585 INSTRON. Untabbed specimen with size 150 (*l*) X 22 (*w*) mm as shown in Figure 3 was assembled into the test jig and aligned with the loading axis of the machine. The length was parallel to the fibre direction.

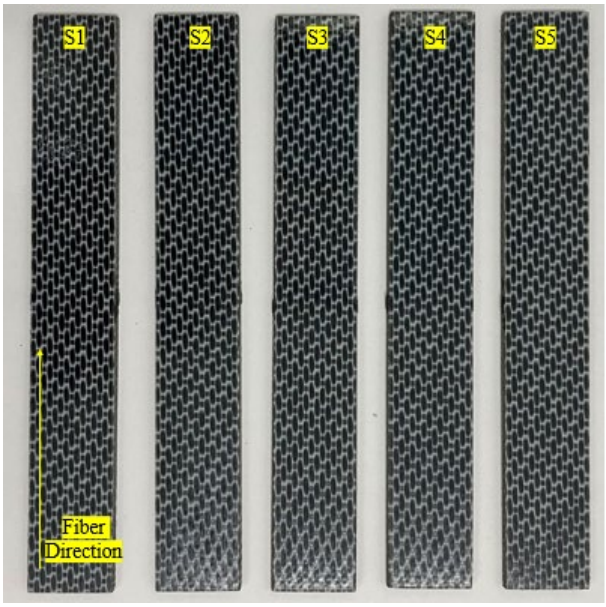

Fig. 3. Set of Tensile Test Specimens

3. Results and Discussion

3.1 Surface Energy (SE)

Surface energy (SE) refers to the amount of energy required to increase the surface area of a material. In the context of thermoplastic composites (TPC), surface energy plays a crucial role in determining their adhesion properties, bonding characteristics, and interactions with other materials. The SE of CF/PPS composites affects their adhesion to other materials, such as adhesives or coatings. A higher surface energy promotes better wetting and adhesion, resulting in stronger bonds between the composite and other materials. This is particularly important in applications where CF/PPS composites are used as structural components or in joining technologies [14–17]. Besides, CF/PPS composites rely on the interfacial bonding between the CF and the polymer matrix (PPS) for effective load transfer and mechanical performance. The surface energy of the PPS and the CF influences the interfacial bonding strength. A well-bonded interface, achieved through appropriate surface energy matching or surface treatments, enhances the composite's mechanical properties and resistance to delamination or fibre-matrix debonding [18]. This is because raising the temperature during forming facilitates the thermal movement of polymer molecules, thereby aiding interlaminar diffusion and penetration of the polymer. Consequently, this improves the strength of bonding between layers of the material.

In this study, the contact angles of water and acetone tabulated in Table 5 were used to derive the SE using Owens – Wendt calculation. Figure 4 shows the image of the water and acetone droplets on the CF/PPS composite component.

Table 5
Measured Contact Angle of the Test Liquids

Component ID	Contact angle (°)		Surface Energy (mJ/m^2)
	Water	Acetone	
Tt150	80.50	21.43	24.88
Tt170	74.29	14.63	29.11
Tt180	67.85	16.60	35.13
Tt195	74.07	15.28	29.28

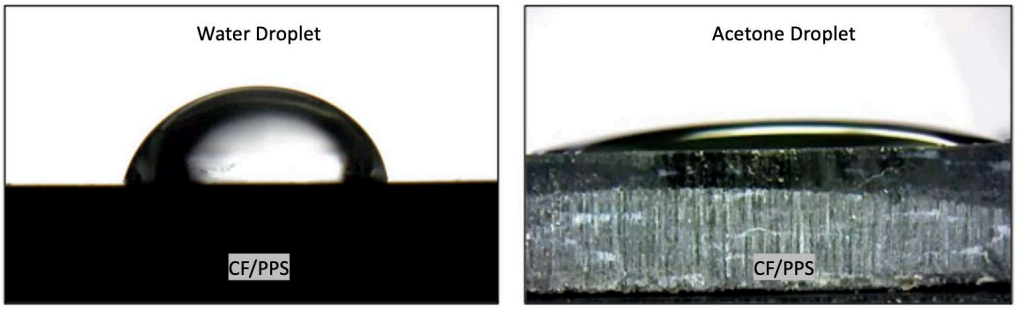

Fig. 4. Water and Acetone Droplets on the CF/PPS Composite Component

From the results in Figure 5, the values of SE increase as the tool temperature increases. Tt150 showed the lowest SE of 24.88 mJ/m², while Tt180 showed the highest SE of 35.13 mJ/m². Tt170 showed almost similar results to Tt195 with the value of 29.11 mJ/m² and 29.28 mJ/m², respectively. Even though the SE decreases at Tt195, the value is still higher than Tt150. These results suggest that as the temperature increases, the surface energy of the material tends to increase as well. According to a study by Liu *et al.,* [19], it was observed that increasing the surface roughness and surface energy of CF could lead to an improvement in the mechanical properties at the interface between the reinforcement and the matrix material. This is due to contact surface between the CF and matrix increased and good wetting can be achieved. This can be observed in similar trends of SE and tensile strength in this study. Enhanced wetting and adhesion occur due to greater surface energy, facilitating improved mechanical properties and effective load transfer in the composite. In contrast, insufficient surface energy can result in inadequate adhesion, weakened interfacial strength, and limited capability for load transfer. The findings by Mahat *et al.,* [20] provided evidence of a significant correlation between the processing parameters and the physical characteristics of CF/PPS composites.

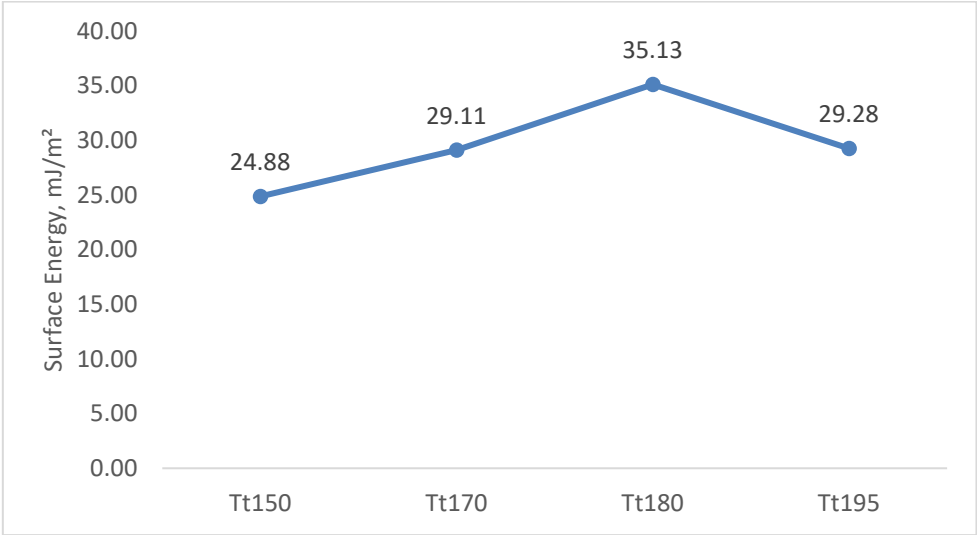

Fig. 5. SE of CF/PPS Composite subjected to Different Tool Temperature

3.2 Degree of Crystallinity (DoC)

The degree of crystallinity (DoC) refers to the proportion of crystalline regions within a polymer composite. It is a measure of the order and arrangement of polymer chains, affecting various

properties such as mechanical strength, stiffness, thermal stability, and chemical resistance. The DoC is influenced by factors such as processing conditions, cooling rates, and temperature [21,22].

Temperature plays a crucial role in the melting and crystallization behaviour of CF/PPS composites. As the temperature increases, the polymer chains in the PPS matrix experience increased molecular mobility, allowing for enhanced chain rearrangement and crystallization. Higher temperature promotes the melting of crystalline regions, leading to a decrease in the DoC . The temperature during the processing of CF/PPS composites, such as moulding or curing, can significantly influence the DoC. Different temperature profiles can result in variations in cooling rates, which affect the rate of crystallization and subsequent DoC [23,24] .

From the obtained results, the DoC represents the extent to which a material's structure consists of ordered, crystalline regions. The results of DoC in Figure 6 exhibited minimal variations among the different components. Tt180 had the highest value of 25.00%, while Tt150 had the lowest value of 24.30% with a range value of 0.70%. Overall, the DoC remains relatively consistent across the different tool temperatures in this study. The CF/PPS composite component likely had reached a saturation point in terms of crystalline regions, and further temperature increase has minimal impact on the crystalline structure. A group of researchers conducted an experiment, and the results showed that higher processing temperatures enhanced the crystallinity of the polypropylene (PP) matrix and strengthened the interfacial adhesion between the fibre and resin, which in turn increased the tensile modulus [25]. This seems to correlate with the tensile strength and TGA test results in this study. The DoC in almost every component was directly proportional to the tensile strength proving that strong correlation between DoC and mechanical properties as well as physical properties of the CF/PPS composite component. The crystallisation window of CF/PPS composites have a crystallisation window of 87-270°C. When the tool temperature in the range of ithe crystallization window, the CF/PPS composite undergo the isothermal crystallization, which could increase crystallinity even further. The mechanical characteristics improved as the tool temperature increased due to improved matrix self-adhesion, whereas the fracture toughness declined due to lower matrix ductility [11,26].

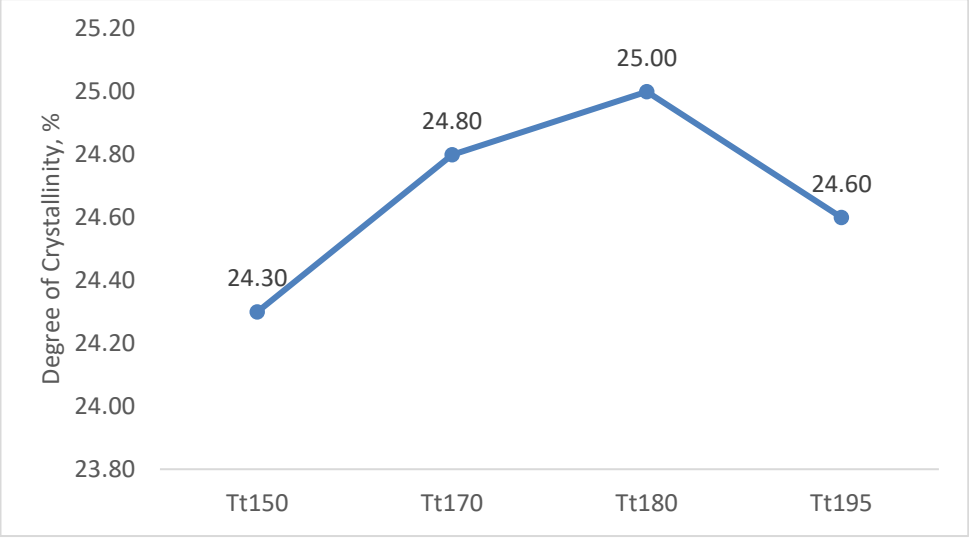

Fig. 6. DoC of CF/PPS Composites Subjected to Different Tool Temperature

3.3 Thermogravimetric Analysis (TGA)

Thermogravimetric analysis (TGA) is a technique that measures the weight change of a material as a function of temperature. It provides information about the thermal stability, decomposition

kinetics, and weight loss of materials under controlled heating conditions. TGA is commonly used to characterize the thermal behaviour of CF/PPS composites and assess their suitability for high-temperature applications. TGA provides insight into the degradation behaviour of CF/PPS composites. The weight loss profile obtained during TGA can reveal multiple decomposition steps, each associated with different components within the composite system [27,28]. Temperature variations during TGA can affect the rate and extent of degradation, providing information on the composite's overall thermal behaviour.

In TGA analysis, the weight percentage of the residue remaining after thermal degradation is typically measured. The residue represents the non-volatile or more stable components of the CF/PPS material. According to Figure 7, an increasing trend in the TGA values as the temperature increases. Tt150 exhibited the lowest TGA value of 71.07%, while Tt180 showed the highest value of 74.74%. Tt170 and Tt195 had slightly below Tt180 with TGA values of 72.85% and 73.46%, respectively. These results indicate that increasing the tool temperature generally leads to more stable components of the CF/PPS material. The results are aligned with the simulation generated by Miao *et al.,* [29] where the chemical reaction occurring at the co-cured interface is the key factor contributing to the enhancement of composite material. Furthermore, the strength of the interface increases proportionally with the number of chemical processes taking place upon contact. Thermal stability such as temperature transmission from end to end, load capacity at certain temperatures, material behaviour, and dimensional stability at high temperatures all play an equal role in structural applications [30]. Based on a study performed by Sathees Kumar [31], good adhesion between the fibres and matrix is responsible for the effective resistance capability. Further, thermal properties such as thermal stability were correlated to the tensile properties.

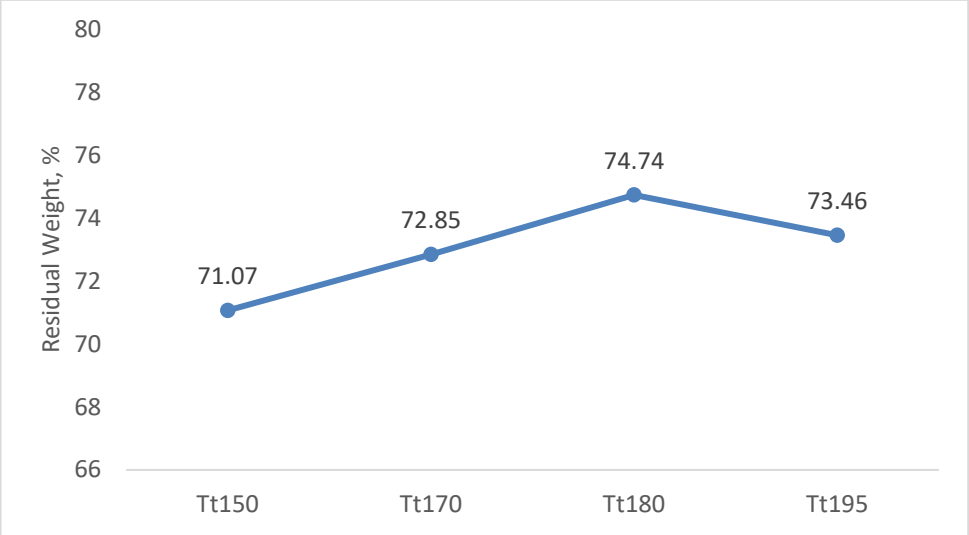

Fig. 7. TGA of CF/PPS Composites Subjected to Different Tool Temperature

3.4 Tensile Strength

Tensile strength refers to the maximum stress a material can withstand under tension before it fractures or breaks. It is a crucial mechanical property that determines the structural integrity and load-bearing capacity of CF/PPS composites. Understanding the effect of temperature on tensile strength is essential for assessing the material's performance in different temperature environments and optimizing its applications.

Tensile strength is a measure of a material's ability to resist deformation under tension. The tensile strength values increase as the temperature increases as presented in Figure 8. The lowest value was obtained from Tt150 (746.40 MPa) and the highest value was obtained from Tt180 (793.80 MPa). Tt170 and Tt195 showed almost identical values of 779.80 MPa and 788.60 MPa, respectively. These results indicated that increasing the temperature can enhance the tensile strength of the material. Tt180 exhibits the highest tensile strength where it shows good agreement with the results of SE, DoC and TGA. High SE promotes better wettability, and consequently improved load transfer. The fracture morphology of tensile specimens under scanning electron microscopy (SEM) is presented in Figure 9. It can be observed that good adhesion of matrix (PPS) on the fibre interfacial for Tt180 as compared to Tt150. The PPS was firmly contacted and glued the CF on Tt180 while apparently the CF was debonded from the PPS on Tt150. Zhang *et al.,* [32] proved that high surface energy shows good agreement for the high interfacial strength in a composite. Gao *et al.,* [33] also reported on the influence of cooling rate on final CF/PEEK crystallinity and its relation to mechanical properties is directly related. In the experiment performed by Tatsuno *et al.,* [34], they found that the part's ultimate strength increased with a slower cooling rate. The degree of resin and fibre bonding is influenced by the cooling rate, which in turn impacts the final composite part's mechanical properties. [35–37].

Temperature variations can affect the interfacial bonding between CF and the PPS matrix. If interfacial debonding occurs, the interfacial strength between the CF and the PPS matrix increases with crystallinity [38]. At elevated temperatures, the thermal expansion mismatch between the fibres and the matrix may induce stress concentrations at the interface, potentially weakening the interfacial adhesion and leading to a decrease in tensile strength. CF in CF/PPS composites may also exhibit temperature-dependent behavior. At higher temperatures, the mechanical properties of the CF can be affected, including potential reductions in strength and stiffness [26]. In addition, Chadwick *et al.,* [9] found that with increasing temperature, CF/PPS composite laminate produced using heated tool exhibited a considerable rise in both interlaminar shear and perpendicular tensile characteristics.

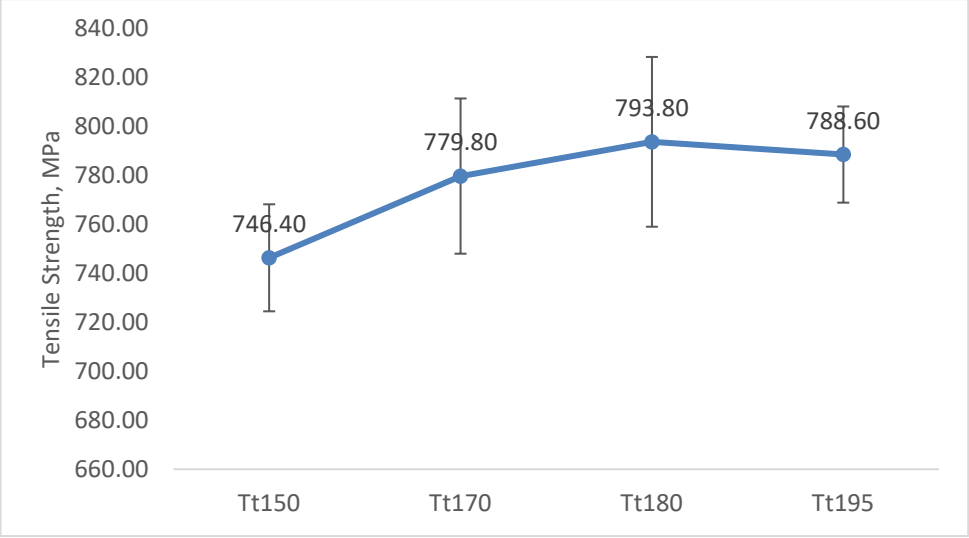

Fig. 8. Tensile Strength of CF/PPS Composites Subjected to Different Tool Temperature

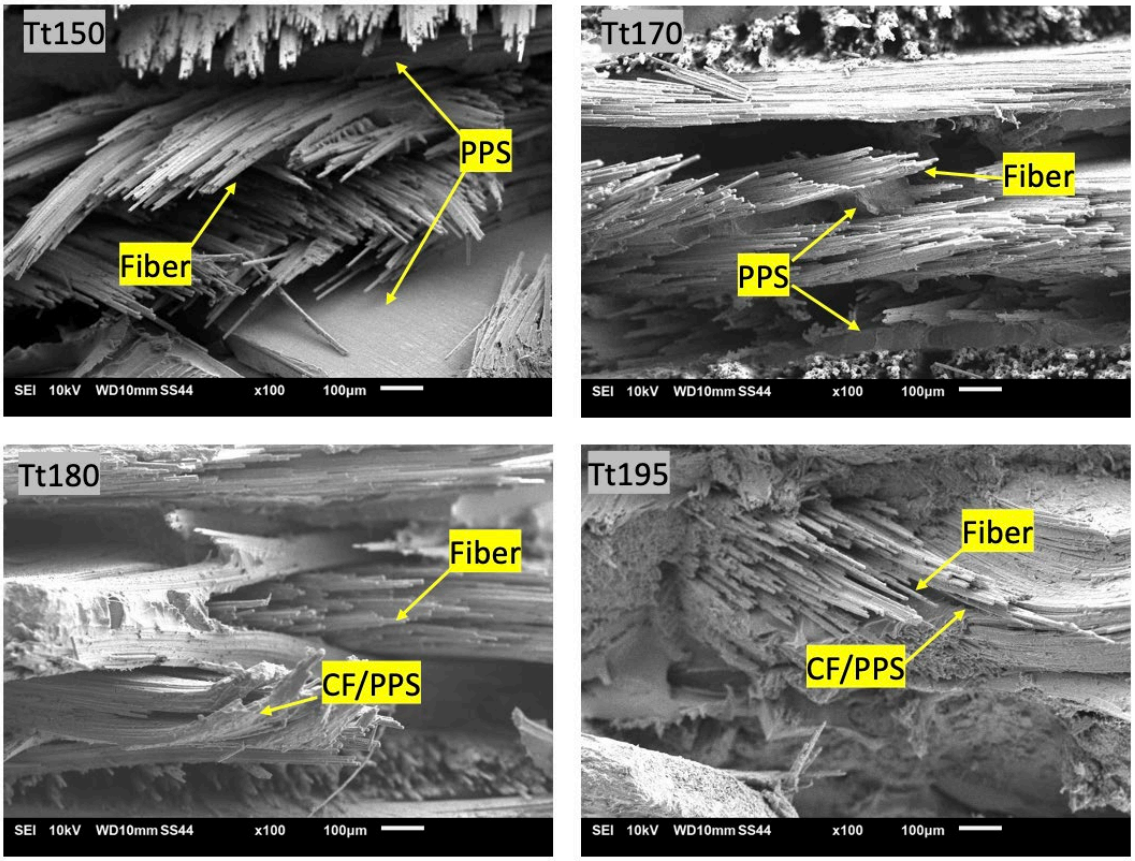

Fig. 9. Failure Mode of Tensile Specimens under SEM

4. Conclusion

In this study, we examined the physical, thermal, and mechanical properties of CF/PPS composites thermoformed at different tool temperatures by the hot press forming process. Based on the results, it can be concluded that:

i. As the tool temperature increases, the physical, thermal, and mechanical properties of the thermoformed CF/PPS composite component increase. Increasing the hot press tool temperature enhances the surface energy, improving adhesion and bonding between the matrix and CF and developing the composite materials' thermal stability.
ii. The recommended hot press tool temperature for optimal composite performance is around 180°C. At this temperature, the physical, thermal, and mechanical properties of the CF/PPS composite component are the highest with the value of surface energy is 35.13%, the DoC is 25.00%, TGA is 74.74% and tensile strength is 793.80 MPa.
iii. In conclusion, selecting the right processing temperature is important for achieving strong adhesion, enhanced mechanical properties, and overall superior performance in composite materials.

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