UNIVERSITI TEKNIKAL MALAYSIA MELAKA

EFFECT OF HOTPRESS TOOL TEMPERATURE ON PHYSICAL, THERMAL, AND MECHANICAL PROPERTIES OF POLYPHENYLENE SULFIDE COMPOSITE

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A thesis submitted in fulfillment of the requirements for the degree of Master of Science in Mechanical Engineering

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2025

DECLARATION

I declare that this thesis entitled "Effect of Hotpress Tool Temperature on Physical, Thermal, and Mechanical Properties of Polyphenylene Sulfide Composite" is the result of my own research except as cited in the references. The thesis has not been accepted for any degree and is not concurrently submitted in candidature of any other degree.

Signature : ....

Name : Emy Aqillah Sharif

Date : 17 Julai 2025

APPROVAL

I hereby declare that I have read this thesis and in my opinion this thesis is sufficient in terms of scope and quality for the award of Master of Science in Mechanical Engineering.

Signature	:	....
Supervisor Name	:	PM Dr. Nadlene Binti Razali
Date	:	18-7-2025

DEDICATION

This research is dedicated to my family and friends who have never failed to give attention and continuous support throughout my research journey.

This research is also dedicated to my supervisor, Associate Professor. Dr. Nadlene Binti Razali who has guided me with great attention and motivated me to set a higher goal to produce outstanding research.

Finally, I dedicate this research to all UTeM lecturers, staff, and my research teammates who were involved in giving their cooperation for this study.

ABSTRACT

This study investigates the influence of hot press tooling temperature on cooling rates and examines how these rates impact the physical, thermal and mechanical properties of carbon fiber-reinforced polyphenylene sulfide (CF/PPS) composites. CF/PPS composites are increasingly used in aerospace applications due to their high-to-weight ratio, chemical resistance and recyclability. The research focuses on understanding how different tool temperatures of 150°C, 170°C, 180°C, and 195°C during the hot press forming process affect key properties of the composite material. Test panels were fabricated using six plies of 5-harness CF/PPS pre-consolidated laminates with a [(0,90)/(±45)/(0,90)]s stacking sequence. Physical characterization involved determining the surface energy through the sessile drop method and void content (Vc) via acid digestion by following the EN2564 standard. Mechanical properties including flexural and tensile strengths were evaluated using universal testing machine (UTM) by following the EN2562 and EN2561 standards respectively. The degree of crystallinity (DoC) was analysed through differential scanning calorimetry (DSC) to assess thermal properties. The results indicate that increasing tool temperature improves the DoC and enhances mechanical properties up to an optimal temperature of 180°C. At this tool temperature, the composite exhibited the highest flexural and tensile strengths as well as minimal void content. However, further increasing the tool temperature to 195°C led to slight degradation in material properties, likely due to excessive resin flow and potential thermal degradation. To strenghen the findings, morphological analysis was conducted by using Scanning Electron Microscopy (SEM). Fractured tensile samples of CF/PPS after hot press forming process were investigated to observe the interfacial bonding between carbon fibres and polyphenylene sulfide. Lastly, a strong correlation was determined in order to analyse the relationship between cooling rate, crystallinity and mechanical performance by using SPSS software. The correlation revealed strong relationships, notably a significant negative correlation between cooling rate and flexural strength, indicating that faster cooling tends to reduce the material's flexural performance. Furthermore, a very strong inverse relationship was observed between void content and degree of crystallinity, alongside strong positive intercorrelations among surface energy, degree of crystallinity, tensile strength, and flexural strength. These findings provide beneficial input for optimizing the hot press forming process to achieve superior CF/PPS composites for aerospace applications.

KESAN SUHU ACUAN PENEKAN PANAS TERHADAP SIFAT FIZIKAL, TERMA, DAN MEKANIKAL KOMPOSIT POLIFENILENA SULFIDA

ABSTRAK

Kajian ini meneliti pengaruh suhu acuan mesin penekan panas terhadap kadar penyejukan serta kesannya terhadap sifat fizikal, terma dan mekanikal komposit polifenilena sulfida yang diperkukuh dengan gentian karbon (CF/PPS). Komposit CF/PPS kian mendapat perhatian dalam aplikasi aeroangkasa berikutan nisbah kekuatan terhadap berat yang tinggi, ketahanan kimia serta kebolehannya untuk dikitar semula. Dalam kajian ini, suhu acuan sebanyak 150°C, 170°C, 180°C dan 195°C digunakan semasa proses pembentukan tekan panas untuk memahami pengaruhnya terhadap ciri-ciri utama panel CF/PPS selepas ditekan. Panel ujian dihasilkan menggunakan enam lapisan lamina CF/PPS jenis 5-harness yang telah dikonsolidasikan dengan jujukan lapisan [(0,90)/(±45)/(0,90)]s. Ciri-ciri fizikal dikaji melalui penentuan tenaga permukaan menggunakan kaedah titisan sessile dan kandungan rongga (Vc) menggunakan penghadaman asid mengikut piawaian EN2564. Ujian mekanikal dijalankan bagi menilai kekuatan lentur dan tegangan menggunakan mesin ujian universal (UTM) mengikut piawaian EN2562 dan EN2561. Ciri terma pula dianalisis menggunakan kalorimetri imbasan pembezaan (DSC) bagi menentukan tahap penghabluran (DoC). Hasil kajian menunjukkan bahawa peningkatan suhu acuan meningkatkan DoC dan prestasi mekanikal sehingga suhu optimum 180°C, di mana bahan menunjukkan kekuatan lentur dan tegangan tertinggi serta kandungan rongga yang minimum. Namun, peningkatan lanjut sehingga 195°C menyebabkan sifat bahan panel menjadi merosot akibat aliran resin berlebihan dan kemungkinan disebabkan oleh degradasi terma. Bagi mengukuhkan penemuan kajian, analisis morfologi turut dijalankan menggunakan Mikroskopi Elektron Imbasan (SEM). Sampel patah (sampel tegangan) CF/PPS setelah proses pembentukan tekan panas telah dikaji bagi memerhati ikatan antara muka antara gentian karbon dan polifenilena sulfida. Akhir sekali, hubungan statistik yang kukuh dianalisis menggunakan perisian SPSS bagi menilai hubungan antara kadar penyejukan, tahap penghabluran dan prestasi mekanikal. Analisis korelasi menunjukkan hubungan yang kuat, khususnya korelasi negatif yang signifikan antara kadar penyejukan dan kekuatan lentur, menunjukkan bahawa kadar penyejukan yang lebih pantas cenderung mengurangkan prestasi kelenturan bahan CF/PPS. Tambahan pula, hubungan songsang yang sangat kuat turut diperhatikan antara kandungan rongga dan tahap penghabluran, disamping hubungan positif yang ketara antara tenaga permukaan, tahap penghabluran, kekuatan tegangan dan kekuatan lentur. Dapatan ini memberikan input yang bernilai bagi pengoptimuman proses pembentukan mesin penekan panas dalam penghasilan komposit CF/PPS yang berkualiti tinggi untuk aplikasi dalam industri aeroangkasa.

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LIST OF ABBREVIATIONS

AFP		Automated Fiber Placement
CF/LMPAEK	-	Carbon Fiber/ Polyaryletherketone
CF/PEEK	-	Carbon fiber/Polyetheretherketone
CF/PEKK	-	Carbon Fiber/Polyetherketoneketone
CF/PPS	-	Carbon Fiber/Polyphenylene Sulfide
CFRTP	-	Continuous Fibre-Reinforced Thermoplastic Composites
DoC		Degree of Crystallinity
DSC		Differential Scanning Calorimetry
FST	-	Fire/Smoke/Toxicity
PCL		Pre-consolidated Laminate
PPS		Polyphenylene Sulfide
RT		Room Temperature
Tg	-	Glass Transition Temperature
Tm		Melting Temperature
Tp		Processing Temperature
TPC	-	Thermoplastic Composite
UTM		Universal Testing Machine
UTeM	-	Universiti Teknikal Malaysia Melaka
V_c		Void Content Percentage

LIST OF SYMBOLS

γsg	-	Surface energy
ρ_c		Specimen density
ρ_f		Fiber density
ρ_r		Resin density
$\gamma^d{}_L$		Liquid dispersion
$\gamma^p{}_L$		Liquid polar
γ_L		Surface energy of test liquid
P		Load at failure
S		Distance between supports
w		Specimen width
t		Specimen thickness
$\boldsymbol{F_f}$		Flexural strength
$\boldsymbol{T_f}$		Tensile strength
ΔH_m		Melting enthalpy
$\Delta H100\%$		100% crystalline PPS enthalpy

LIST OF APPENDICES

LIST OF PUBLICATIONS

The followings are the list of publications related to the work on this thesis:

1. Emy Aqillah Sharif, Nadlene Razali, Hamdan , H. ., Ismail, N. ., Shamsullizam , N. H. ., Esa , M. D. ., Abd Aziz, N. Z., Mohamad , M. K. A. ., & Syazwan Ahmad Rashidi. (2024). Physical, Thermal and Mechanical Properties of Carbon Fibre/Polyphenylene Sulfide (CF/PPS) Composite at Different Tool Temperatures Fabricated by Hot Press. *Journal of Advanced Research in Applied Mechanics, 117(1)*, 190–203. https://doi.org/10.37934/aram.117.1.190203

2. Rashid, B., Razali, N., Zakaria, M.S., Ramlan, M.Z.H., Hamdan, H., Sharif, E.A., Muhammad, N. and Rashidi, S.A., A Study on the Thermal Distribution of the Thermoforming Process for Polyphenylene Sulfite (Polyphenylene Sulfide) PPS Composites Towards Out of Autoclave Activity. *Pertanika Journal of Science & Technology, Volume 32, Issue S2, December 2024*. https://doi.org/10.47836/pjst.32.S2.04

3. Hamdan, Hasanudin, Nadlene Razali, Anita Akmar Kamarolzaman, Nurfaizey Abdul Hamid, Emy Aqillah Sharif, Nur Farhana Mohd Yusoff, Nur Umairah Noriman, Syazwan Ahmad Rashidi, and Sarah Othman. "Layering Effect on Mechanical, Thermal & Physical Properties Carbon Fibre Reinforced Polyphenylene Sulfide." *Journal of Advanced Research in Fluid Mechanics and Thermal Sciences 98, no. 2 (2022)*: 128-145.

4. Hamdan, Hasanudin, Nadlene Razali, Nurfaizey Abdul Hamid, Anita Akmar Kamarolzaman, Emy Aqillah Sharif, Syazwan Ahmad Rashidi, and Sarah Othman. "Validation of thermoforming process for pressed formed two folded clip panel simulated by aniform software." *In Proceedings of SAKURA Symposium on Surface*

Technology and Engineering Materials 2022, vol. 2022, pp. 37-39. Malaysian Tribology Society, 2022.

5. Hamdan, Hasanudin, Nadlene Razali, Anita Akmar Kamarolzaman, Nurfaizey Abdul Hamid, Siti Hajar Sheikh Md Fadzullah, Emy Aqillah Sharif, Syazwan Ahmad Rashidi, and Sarah Othman. "Characterization of Carbon Fibre Reinforced Polyphenylene Sulfide Composite Under Interlaminar Shear Strength." *Journal of Advanced Research in Applied Mechanics 102, no. 1 (2023)*: 1-9.

CHAPTER 1

INTRODUCTION

1.1 Background

Over the previous few decades, most fiber-reinforced polymers utilized in automotive, aerospace, and renewable energy constructions have been based on thermoset matrix fiber-reinforced composites. Because of their superior strength and stiffness compared to their metallic equivalents, thermoset matrices are widely used, allowing structural engineers to produce lighter components that are more economical in terms of operational expenses influenced by weight reduction (Quan *et al.,* 2023).

Aircraft designers employ materials with increased specific strength, specific modulus, fatigue strength, and other qualities to create lighter aircraft structures. Innovative design employs advances in engineered materials as well as manufacturing methods to reduce the number of subassemblies and fasteners, resulting in weight reduction. Aircraft weight reduction is critical for lowering fuel consumption and thereby lowering pollution. It also enhances fuel economy and performance (Yıllıkçı and Findik, 2013; Krishnadas Nair, 2019).

As environmental and energy concerns increase, a variety of industrial sectors are increasingly adopting lightweight materials. In an era when new materials are developed and manufacturing processes are optimised to satisfy industrial demands, thermoplastic composite (TPC) plays a significant role in the industry. In addition to being able to be thermoformed and heat-welded, TPC offers extended storage life, durable construction, and simple maintenance and recycling. A wide range of industries, including aerospace, automotive, construction, and energy, utilise them to achieve automated, high-speed mass production with minimal reliance on auxiliary operations (Krawczak and Maffezzoli, 2020). In comparison to thermoset and metallic alternatives, TPC has the ability to lower the weight of aeroplane structures by 20 % to 50%. Parts can be created in minutes rather than hours, and customers can benefit from up to an 80% decrease in production cycle time for the same lighter part. Due to their unique welding capabilities, integrated TPCs such as access doors, engine nacelles, flight control surfaces, and interiors are lighter in weight and less expensive due to a combination of materials, manufacturing processes, and unrivalled expertise (Collins Aerospace, 2023).

TPC provide numerous benefits, including enhanced bonding, lightweight construction, durability, design flexibility, and accelerated production. The high strength-to-weight ratio of these composites qualifies them for use in aerospace applications. Their exceptional resistance to chemicals, impact, and fatigue ensures their durability in harsh environments. The capacity to manipulate TPC into intricate configurations enables increased autonomy in design and tailoring. The ability of TPC to be rapidly heated and cooled reduces processing periods, thereby enhancing production efficiency. In contrast to thermoset composites, TPC is more cost-effective and environmentally sustainable due to their ability to be reprocessed and dissolved. Additionally, TPC demonstrates enhanced adhesion to other materials, which simplifies the process of integrating various components during aircraft manufacturing.

There are a few manufacturing technologies for aircraft components using TPC materials such as pultrusion, continuous compression moulding, automated fibre placement

(AFP), automated tape laying (ATL), and thermoforming. Three basic stages are needed to fabricate TPC: heating to melt the polymer matrix, pressurisation to solidify and shape, and cooling to solidify the polymer. A quick manufacturing method called thermoforming is utilised to transform a two-dimensional (2D) panel into a three-dimensional (3D) object. Consolidation can be done using an autoclave or a press. The technique is appealing because of its very short cycle time. Usually, a semi-manufactured item needs to be used. A pre-consolidated laminate (PCL) is fed into the thermoforming phase to maximise production efficiency (Marco *et al.*, 2020).

The TPC press-forming process is similar to that of metals; the difference in TPC processing is that the material is heated to a high temperature and processed in a very short period of time. The process consists of a few sub-processes starting from the heating process to the demoulding of the component. The heating process in an oven above its melting temperature (T_m) where the temperature is called processing temperature (T_p) to make it pliable for forming. The melted material is transferred into the hot press area for the forming process using a set of preheated tools under constant pressure with a specific press time until it is solidified followed by the de-moulding process (Schug, 2019).

Several literature reviews about TPC studies that are available are mainly conducted overseas. Limited literature with background on the manufacturing of TPC material for aircraft components is performed in Malaysia. Therefore, considering the growing visibility of TPC nowadays and to stay updated with global development, an investigation in this field is beneficial to the local aircraft industries.

Thus, this research is carried out to evaluate the physical properties and to characterize the thermal and mechanical properties of CF/PPS composite fabricated by the hot press forming process at different tool temperatures (T_t) ranging from 150°C to 195°C. CF/PPS composite made of six plies of 5-harness (5H) carbon layers in the form of PCL with stacking sequence [(0,90)/(±45)/(0,90)]s is used for the test panels manufacturing. Four test panels are manufactured and each test panel represents each specified T_t. The physical characterization of press-formed test panels is determined by means of surface energy and void content percentage (V_c). Flexural and tensile tests are performed using the universal testing machine (UTM) for the characterization of mechanical properties while differential scanning calorimetry (DSC) equipment was used to evaluate the degree of crystallinity (*DoC*) for the characterization of thermal properties.

1.2 Problem Statement

Although TPCs are introduced to the market many years ago, they have started to find use in recent years with the further development of materials and the development of production technologies, such as thermoforming, AFP, press forming, and welding. The hot press forming technology is one of the methods commonly used in the production of high volume of TPCs such as ribs, brackets, clips, stringers and stiffeners used in aircraft fuselage and wings (Slange, 2019).

Hot press forming allows relatively complex shapes to be formed comparatively quickly from pre-consolidated composite laminate. It involves heating a material (usually to above the matrix melting temperature, T_m) before transferring the material to a matched metal die or mould set. The two halves of the mould are brought together, and the heated material is deformed into the shape of the mould. After a given holding time, the mould sections move apart and the formed part is removed. Nevertheless, the hot press forming process involved a series of temperature-dependent sub-processes. This parameter is crucial in order to produce good quality of the final product (Brooks *et al.*, 2022).

High-performance TPC such as CF/PEEK and CF/PPS carbon have been successfully applied in the aircraft industries in recent years due to their numerous advantages. These materials are semi-crystalline polymers. Semi-crystalline resin crystallization is primarily influenced by solidification circumstances during the production process, such as temperature and pressure histories, as well as resin flow. Furthermore, inhomogeneous crystallization in carbon fiber reinforced thermoplastic (CFRTP) is much more complex than homogeneous crystallization in neat resins because the presence of CF influences the morphological structures of resin within the CFRTPs. Among the solidification circumstances, various experimental research on the cooling rate has been reported to date. According to the kinetics theory of polymer crystallization, the crystal growth rate of semi-crystalline resins is mostly determined by temperature Oshima *et al.,* (2023).

However, due to the long molecular chain structure of thermoplastic resins, the high viscosity of the molten resin made thorough infiltration of the fibers impossible. Furthermore, thermoplastic resin comprises amorphous domains and crystallites that can convert to each other when temperature or shear flow is changed. As a result, the temperatures have a major impact on the performance of TPCs. Furthermore, thermoplastic resins have a high T_m and a limited processing window. These extreme temperatures have posed significant problems to the forming process of high-performance TPC. Several studies have investigated the effect of the processing parameter on the physical, mechanical, and thermal characteristics of TPC (Zuo *et al.*, 2019).

Based on the literatures, research on the TPC are mainly conducted abroad. Therefore, a study shall be conducted locally (in Malaysia) on the manufacturing of CFRTP components to align with the recent global development. Nevertheless, the hot press forming process involved a series of temperature-dependent sub-processes. This parameter is crucial in order to produce good quality of the final product. Understanding the effect of T_t on the physical, mechanical, and thermal properties of CF/PPS composites formed by the hot press forming process is crucial due to different T_t result in different cooling rates which will influence the *DoC* that will impact the physical, thermal and mechanical properties of the press-formed components.

1.3 Research Question

This research is conducted to answer the following questions:

i) How are the physical properties of CF/PPS composite produced by the hot press forming process at varying tool temperatures?

ii) What are the mechanical and thermal properties of CF/PPS composite fabricated by the hot press forming process at varying tool temperatures?

iii) What is the correlation between the physical, thermal and mechanical properties of CF/PPS composite produced by the hot press forming process at varying tool temperatures?