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**A STUDY OF THE FEASIBILITY OF ADVANCED
HYBRID THERMOPLASTIC COMPOSITES FOR
AEROSPACE AND AUTOMOTIVE APPLICATIONS**

BY

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**A thesis submitted to the Faculty of Engineering and the
Built Environment, University of Cape Town in fulfilment of
the requirement for the degree of Master of Science in
Applied Science in Materials Engineering.**



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June 2008**



Declaration:

I know the meaning of plagiarism and declare that all the work in the document, save for that which is properly acknowledged, is my own.

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“What we know today will be obsolete tomorrow. If we stop learning we stagnate.”
Dorothy Billington



ABSTRACT

The development of new technologies and the demand for improved performance has resulted in the need for materials with high specific strength and stiffness. Fibre reinforced polymer materials have found increased application in the automotive and aerospace industries by virtue of their excellent specific properties, good corrosion resistance, toughness and fatigue properties. This study aimed at the feasibility of developing an advanced hybrid thermoplastic composite for aerospace and automotive applications. This was done by first researching the types and functions of current thermoplastic composites used in the aerospace and the automotive industries. Thereafter, an advanced thermoplastic resin was selected as matrix to develop the hybrid composites which were then characterised by mechanical and physical testing.

Two advanced hybrid thermoplastic composites based on polyphenylene sulphide (PPS) as matrix with glass fibre and talc filler have been developed. Tensile, flexural and impact tests were performed on them and the results compared with those of unfilled PPS and 40% glass fibre filled PPS. The tensile tests were performed at different temperatures and test speeds. The test temperatures varied from room temperature to 150°C. At each temperature, test speeds of between 2mm/min and 500mm/min were employed to investigate the temperature and strain rate effect on the composites. Scanning electron microscope (SEM) was used to study the morphology of the fractured surfaces in order to understand the failure modes and failure mechanisms of PPS and its composites.

The literature survey shows that PEEK, PPS, PAI, PES, PEI and PEKK dominate the advanced thermoplastic composite industry currently. PEEK, PPS and PEI reinforced with glass or carbon fibres are the most widely known and used materials because their mechanical properties have been well characterised. The addition of glass fibres and talc enhanced the mechanical properties, dimensional stability and the failure strain of the PPS hybrid composites as well as the 40% glass fibre filled PPS. The PPS composites appear to be more sensitive to test speed changes than the unfilled polymer. Increasing temperature resulted in a decrease in strength of all the PPS materials. The flexural and impact strength of the 30% glass fibres and 10% talc filled



PPS hybrid composite (631 PPS hybrid) are comparable to that of the 40% glass fibres filled PPS composite. The PPS materials were found to be generally creep resistant, with the 40% glass fibre filled PPS being more creep resistant than the hybrid composites. The SEM studies revealed that the PPS composites exhibited a brittle failure mode with fibre pullout, fibre fracture and in most cases matrix shear being the dominant mode of failure.

The development of hybrid composites has demonstrated that optimum tailoring of composite structures can result in mechanical property enhancement and reduced cost as a result of appropriate fibre selection and combination.

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GLOSSARY / LIST OF ABBREVIATIONS

PMC	Polymer matrix composite
OEM	Original equipment manufacturer
AMTS	Advanced manufacturing technology strategy
g/cm ³	grams per cubic centimetre
TMC	Thermoplastic matrix composite
FRP	Fibre reinforced polymer/plastic
PEEK	Polyether ether ketone
GMT	Glass mat thermoplastic
ATC	Advanced thermoplastic composite
Pa.s	pascal second
PAN	Polyacrylonitrile
HDPE	High density polyethylene
PP	Polypropylene
PPS	Polyphenylene sulphide
PAI	Polyamide imide
HDT	Heat distortion temperature
PEI	Polyether imide
PES	Polyether sulphone/sulfone
FST	Fire, smoke and toxicity
ASTM	American Standard for Testing Materials
CO	Carbon monoxide
CO ₂	Carbon dioxide
HCN	Hydrogen cyanide
NO ₂	Nitrogen dioxide
SO ₂	Sulphur dioxide
NH ₃	Ammonia
Cl ₂	Chlorine
HCl	Hydrogen chloride
FAA	Federal aviation administration
OSU	Ohio State University



EHC	Effective heat of combustion
HRR	Heat release rate
TGA	Thermo gravimetric analysis
DCS	Differential scanning calorimetry
T _g	Glass transition temperature
23°C	Room Temperature
DTA	Differential thermal analysis
CSIR	Council for Scientific and Industrial Research
ISO	International Standards Organisation
LVDT	Linear variable differential transducer
SEM	Scanning electron microscopy

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CHAPTER ONE

GENERAL INTRODUCTION

1.1 BACKGROUND TO THE RESEARCH

High performance polymer matrix composites (PMCs) based on thermoplastic matrices reinforced with fibres are finding increasing use in high technological applications [1]. Structural fibres that may be used as reinforcement include glass, carbon, aramid, and boron. PMCs are attractive because they are lighter, stronger and stiffer than conventional materials, with the additional advantage that their properties and form can be tailored to meet the needs of a specific application.

High performance PMCs were initially developed in the 1960s for use in military applications such as aerospace and missile systems. As the price of high performance fibres declined in the 1980s due to process improvements, PMCs began to find applications in commercial aircrafts, industrial applications, sports and leisure equipment and in automobile applications [2]. The figure 1.1 below illustrates the breakdown of Western European market according to application sector for PMC finished products in 2001 [3].

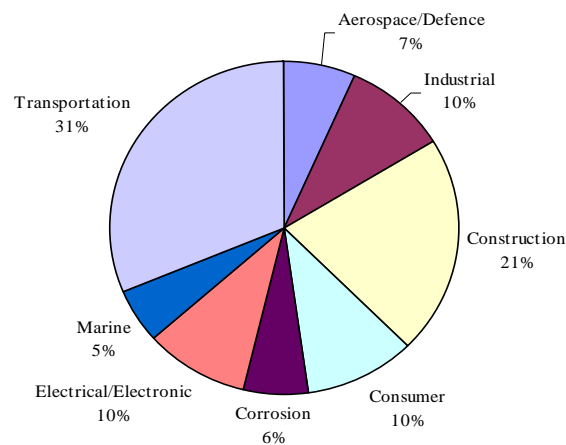


Figure 1.1 Western European PMC finished product by sector [3].

It is therefore not surprising that the transportation, construction, industrial application, electrical/electronic, consumer goods and aerospace/defence sectors take up a sizeable proportion of the Western European PMC market.

In a market survey of more than 50 professionals working with original equipment manufacturers (OEMs) in the aircraft industry, the consensus was that the demand for composites in the aerospace market alone is expected to grow to more than 10% in the next five years from now, due to several new aircraft models with high percentages of composite usage [4, 5]. This opens up opportunities for the development of new composites with properties and characteristics that build on the underlying advantages of composites such as high specific strength and stiffness.

Extensive studies have been carried out on PMCs based on thermoset and thermoplastic matrices. Different researchers have reported on different aspects of thermosetting and thermoplastic matrix composites; however, not much has been reported on hybrid composites based on advanced thermoplastic matrix which this study intends to research.

1.2 RESEARCH MOTIVATION

In 2004 the South African government accepted an invitation from Airbus Military to participate in the design and manufacture of the A400M multi-role mission transport aircraft programme. In return the government committed to procure and take delivery of eight of the aircraft between 2010 and 2014 [6]. This means that South African aerospace companies will design and produce components and sub-components for every Airbus A400M military transport aircraft that is built [7, 8, 9]. In view of this, the State's Integrated Manufacturing Strategy and the Advanced Manufacturing Technology Strategy (AMTS) emphasised the importance of building globally competitive capabilities in knowledge-intensive industries, of which automotive and aerospace are a prime example [6].

It is important to note that many countries consider aerospace as a strategic industry for reasons of national security, economic strength and technological advancement. Therefore, it is understandable for a country like South Africa to seek a competitive position in aerospace. Composites, considered high tech materials, will play an important role in achieving a competitive edge in aerospace.

As part of the AMTS composites projects, a proposal to perform a detailed investigation into the development of a polymer based hybrid composite material based on advanced thermoplastic resin as matrix for aerospace and automotive applications was made. This will provide a better understanding of the mechanical integrity and the behaviour of these hybrid composites.

1.3 RESEARCH OBJECTIVES

The objectives of this study are to:

- Research the types and function of current thermoplastic composites used in the aerospace and automobile industries.
- Develop a hybrid composite material using high performance thermoplastic as a matrix.
- Perform a feasibility study through experimentations to characterise the new hybrid thermoplastic composite that has been developed and correlate the results.

1.4 OUTLINE OF THE THESIS

The work conducted during this research is presented in various chapters. Chapter one has been used to introduce the research. The second chapter is a review of valuable research papers and books in the literature which relates to this study. The experimental techniques used to conduct this research are outlined in chapter three. This is followed by the results obtained from the experiments in chapter four. A detailed interpretation and discussion of the experimental results are stated in chapter



five. Conclusions drawn from the study are presented in chapter six and recommendations for future work are stated in chapter seven. All the references that were used in this study are listed in chapter eight and the appendix section contains some of the data that were not included in the results chapter.

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CHAPTER TWO

LITERATURE REVIEW

2.1 INTRODUCTION

Fibre reinforced polymer (FRP) composite materials are finding increasing use in a wide range of high technology applications. The main reasons for the usage of fibre reinforced polymer composite in these areas is the need to improve structural efficiency and reduce structural mass without compromising on structural strength and stiffness.

Polymer matrix composite (PMC) materials offer a number of distinct advantages over conventional engineering materials. The most significant advantage of polymer matrix composite derives from the fact that they are low density materials with high specific strength and high specific stiffness. The lightweight of PMCs is due to the low densities of their constituents. Polymers used in polymer matrix composites have densities between 0.9 and 1.5g/cm³ and their reinforcing fibres have densities between 1.4 and 2.6g/cm³ [10].

The use of these composite materials gives the designer the possibilities to tailor the structural advantages in a specific application. The possibility of using different fibre orientation to control the behaviour of the component under a specific loading condition can enhance the overall performance of the component. Fibres in a PMC can be selectively placed to resist load in any direction, thus producing directional strength and modulus instead of equal strength and modulus in all directions as in isotropic materials such as metals and unreinforced polymers [10].

Another advantage is that PMCs can be combined with aluminium honeycomb, structural plastic foam, or balsa wood to produce sandwich structures that are stiff and at the same time lightweight. Two or more different types of fibres and fillers can be used to produce a hybrid composite with high flexural stiffness and impact resistance.



There are several other advantages of PMCs that make them desirable in many high tech applications. They have damping factors that are higher than those of metals, which mean that noise and vibrations are damped in PMC structures more effectively than in metal structures. They also do not corrode. However, depending on the nature of the matrix and fibres, their properties may be affected by environmental factors such as elevated temperatures, moisture, chemicals and ultraviolet light [10].

Polymer matrix composites (PMCs) are fibre reinforced polymers in which either a thermoset or a thermoplastic polymer is used as the matrix. The development of PMCs for structural applications started in the 1950s, and they are by far the most common fibre reinforced composite materials in use today [10]. One reason for their growing use is that their processing is relatively simple and does not require very high temperatures and pressures. The equipment required for processing PMCs is also relatively simple and less expensive than that required for other types of composites and conventional materials. There are also a variety of processing techniques available for producing parts made up of PMCs. Depending on the part being produced; some of these techniques can be highly automated [10].

2.2 COMPOSITE MATERIALS

A composite material is a heterogeneous mixture of two or more homogeneous materials that have been bonded together. The mixing of the separate materials is done in a controlled way such that one material is dispersed in the other to achieve optimum properties. The properties of the composite are superior to the properties of the individual components [11, 12]. Composite materials are not new, they were being employed centuries ago when man was making dwellings from mud and straw but the types of composite being considered here are relatively new [12, 13].

The development of PMCs has been such that there has been extensive research into improving the matrix materials. The original glass fibre reinforced components were manufactured using polyester resin as matrix but they had restricted properties and polymer composites were improved by the introduction of epoxy resins. A further

progression was the development of preimpregnated materials using resins which cured at higher temperatures with consequent improvement of properties, particularly regarding higher working temperatures and ductility. The preimpregnates also allowed more control over processing, particularly in producing a more consistent end product [13].

The introduction of newer matrices has extended the range of composite materials even more and this has considerably extended the range of its applications. Resins such as polyimide allow composite components to be subjected to temperatures in the range of 250°C or more and polyetheretherketone is a thermoplastic matrix with excellent chemical resistance and greater damaged tolerance than epoxy based composites [13].

Composites are used in a wide variety of applications since there is a considerable scope of tailoring their structure to suit the service conditions. This is because the fibre reinforcement would allow designers to tailor the strength of a component in the direction most needed by strategic orientation of the fibres. It was realised that this technology had the potential of reducing the weight of components [12, 14].

In addition to the benefits associated with composite materials, it also pose challenges for the vast majority of engineers whose education and work experience have focused on metals. Beyond the challenges posed by the development and application of advanced composite materials, continual advances in structural analysis and design, fatigue and fracture analysis and computational methods, require that engineers refresh their knowledge of the fundamentals and keep up with new developments [15].

Composites make up a very broad and important class of engineering materials. The world annual production is over 10million tonnes and the market has in recent years been growing at 5 – 10% per annum [12, 14]. Figure 2.1 shows the market size of PMC production for each of the main global regions as at 2001 [3]. Asia-Pacific, North America and Western Europe are the leading producers of PMCs with Africa contributing only 2% of the world's PMC production. However, South Africa produces 87% of Africa's PMC production with the rest of Africa producing 13% [3].



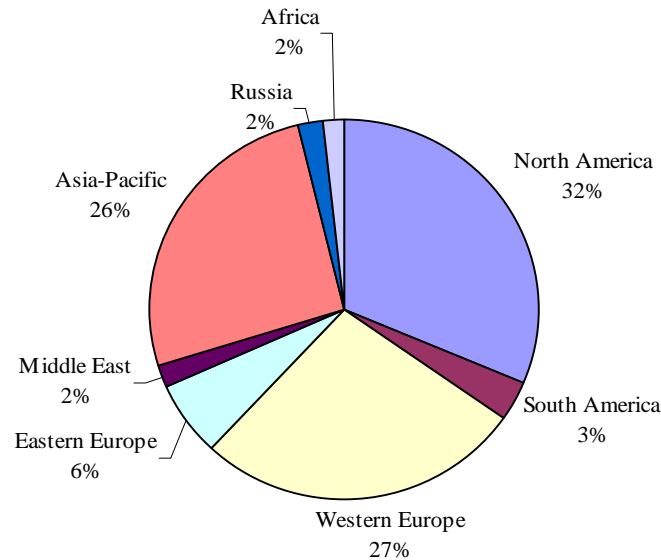


Figure 2.1: World composites finished product by region in tonnes [3].

2.3 COMPOSITE APPLICATIONS IN AIRCRAFT

Composite materials offer tremendous advantages to aircraft and automobile designers. Compared to aluminium, these lightweight materials use less fuel, resist fatigue and prevent corrosion. They have been in use for aircraft applications over the past 35 years, but there has been a rapid increase in their use over the last 20 years [12, 13]. Over the past 20 years, military aircraft have driven the development of advanced composite materials, but today an increasing share of new commercial airplane development is devoted to composites [15].

The amount of polymer matrix composite materials used in aircraft and helicopters has risen dramatically since the 1970s. The figure 2.2 below shows the rapid growth in the use of these composite materials in large civil aircrafts, military aircrafts and rotorcrafts. In these three classes of aircrafts, the use of PMCs has increased many folds over the past thirty years, and the trend is set to continue as these materials continue to replace aluminium and other aerospace alloys in some primary structures, secondary structures and control surfaces [16, 17].

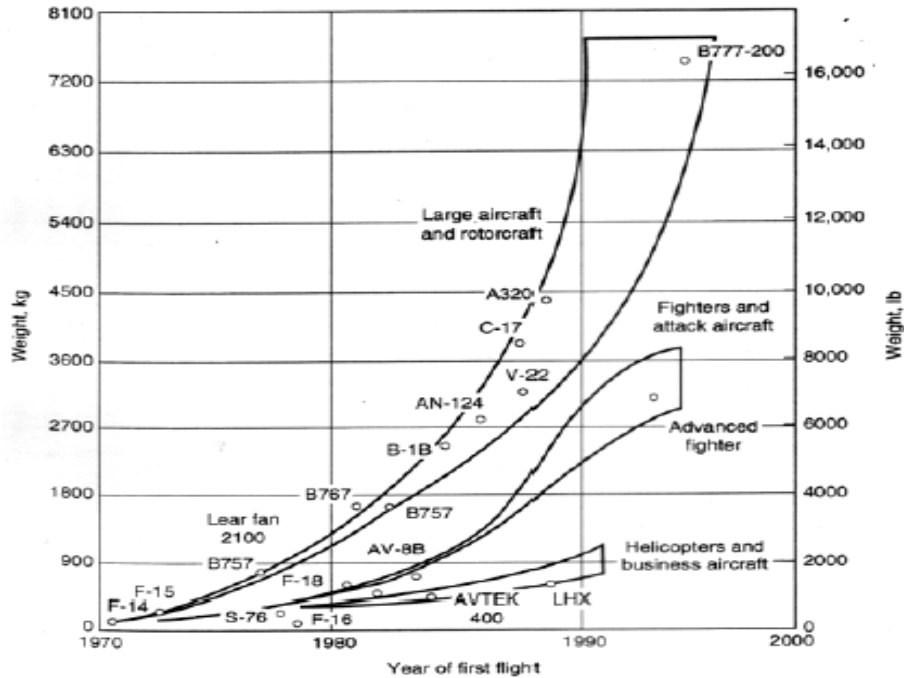


Figure 2.2: Composite aircraft structure by weight [16, 17].

Composite materials are now used extensively in Class 1 structures. A modern aircraft has a high proportion of its structure manufactured from composite materials [13]. The figure 2.3 below illustrates the parts of modern aircraft manufactured from PMCs [18].

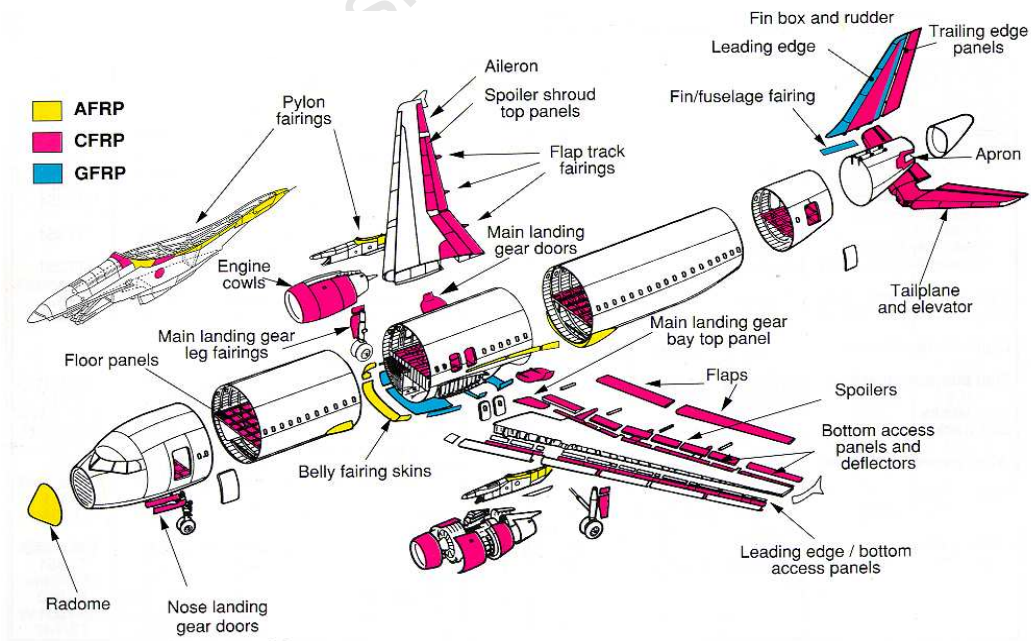


Figure 2.3: Proportion of composite structures in modern aircraft [18].

The value of aircraft components made from composite materials in 2004 was estimated at over \$130 billion. The market value is expected to grow in the coming years provided, among other things, composites do not pose an increased fire safety hazard to aircraft. Boeing and Airbus expect the amount of composites used in their aircraft to increase in the next 10 to 15 years. The percentage of the structure of large passenger aircraft made using composite is currently 10 to 15%, although this is expected to increase to over 25% and possibly higher by 2010 as shown in figure 2.4 below [19]. Both the new A380 and B787 aircraft will make extensive use of composite materials [17].

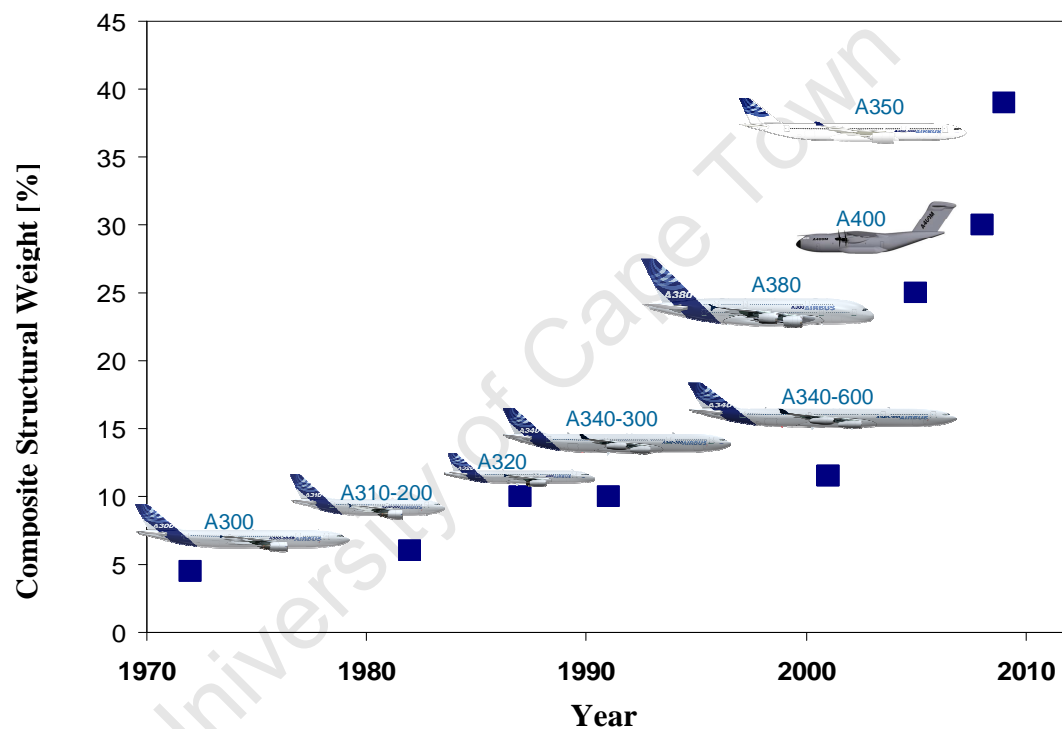


Figure 2.4: Projected Airbus composite structural weight in percentages [18].

The use of composites in cabins is expected to increase with the growing use in passenger electronics and telecommunication equipment. The growth in the usage of composites is due to several factors such as light weight, high specific strength and stiffness, fatigue endurance, design flexibility and corrosion resistance [17]. The two major types of polymer matrix composites used in aircraft are described below.

2.4 THERMOSET MATRIX COMPOSITES

The early thermosetting systems for composites were polyester and epoxy. Of these, epoxy has always been the preferred choice for use with carbon or glass fibres due to its better mechanical properties and it has been the standard material for aerospace applications. In simple terms it is possible to identify three generations of epoxy resins for composite materials. The first generation epoxies were essentially single phase untoughened systems which gives the predominant mechanical properties as being good strength but relatively low toughness. These had low fracture toughness with the result that when loaded in tension, they fail at strengths well below the matrix yield strength due to the brittleness of the resin [20, 21].

Building on these early systems, tougher epoxy resins were developed with reduced strength, but enhanced tensile strain to failure and toughness. In some cases these property changes were associated with the presence of a thermoplastic modifier. In making these improvements the systems tended to compromise, to varying extents, some of the other properties such as hot/wet performance and compression strength in particular. The current systems offer yet a further improvement in toughness through the incorporation of a thin “interlayer” between plies, introduced by the addition of fine tough resin particles on the surfaces of the fibre-epoxy base prepreg. Tougher systems may show yielding and subsequent plastic deformation behaviour when bulk samples of resins are loaded in tension [21].

With regard to temperature capability, the routine application of most current high temperature cure thermoset epoxy composites with glass transition temperature typically between 175°C and 200°C, is limited to less than 120°C. Some blended systems such as epoxy cyanates, have glass transition temperatures approaching 250°C. Other resins have been developed such as bismaleimide and a range of other systems based on polyimides. Some of these later systems which have glass transition temperatures in excess of 300°C can operate in service environments in which there are short term exposures to temperatures of around 250°C. An important consideration for some of these materials is the level of residual thermal stress introduced as a result of processing. Due to the higher processing temperatures, these stresses are generally



higher than in epoxy systems and unless the composite has adequate toughness and an appropriate fibre-matrix interface, problems such as matrix micro-cracking may result [21]. This led to the development of thermoplastic composites.

2.5 THERMOPLASTIC MATRIX COMPOSITES

Thermoplastic matrix composites (TMCs) evolved from structural polymer composites. Structural polymer composites such as epoxy and polyester thermosetting resins reinforced with continuous fibres had many beneficial properties such as low density, good mechanical properties combined with good insulation and environmental resistance. However, they suffer from chemical instability and their impregnated intermediates (prepregs) have limited shelf life.

TMCs on the other hand do not suffer from these problems as they use thermoplastic material as matrix. When heated they soften and can be remoulded without degradation. When they cool they harden into the finished shape. This cycle of heating and cooling can be repeated several times, giving the product an almost indefinite shelf life. They can be shaped using techniques derived from wood and metalworking industries and they have increased damage tolerance due to the tough nature of the matrix material. They also lend themselves to recycling, unlike thermoset composites [22, 23].

The composite industry has seen a continuous growth and will continue to extend its market share. However, it is foreseen that a move towards thermoplastic composites will occur thanks to their benefits over thermosets. The control of environmental pollution, the high performance properties and the recycling opportunity make thermoplastic composite very attractive. Thermoplastic matrices offer high performance properties compared to thermosetting resins. Thermoplastics are attractive thanks to their superior toughness and impact resistance, their post-process formability and improved reparability [21].

The development of thermoplastic resins for fibre reinforced polymers (FRPs) originated from the early 1980s with the development of PEEK as a matrix material. The attraction of thermoplastic systems includes their inherent toughness and their flexibility in processing compare to thermosets. It is important to note that the success in developing thermoplastic systems to meet these objectives have in turn acted as a driver in the development of improved thermosetting systems. The various thermoplastic resin systems have a large range of melting temperatures with that of PEEK, about 400°C, being at the upper end of the range. The high processing temperature means that the glass transition temperature of fibre/PEEK systems is high, in excess of 200°C but that residual thermal stresses are significant [21, 24].

A further issue with thermoplastic composites is matrix crystallization. The cooling rate during manufacture is a critical parameter which influences the degree of crystallinity and this in turn affects the mechanical properties [21, 24, 25].

Thermoplastic composites are expected to replace metals and thermosets in numerous applications as they offer remarkable advantages over these traditional materials. They are generally recyclable, offer high impact strength and are environmentally acceptable as there are no solvent used or volatiles emitted during processing. These vary from low pressure processes such as vacuum moulding, filament winding and pultrusion to high pressure processes like co-moulding, extrusion, compression and injection moulding. Most thermoplastic composites cannot be disposed off; their post-industrial or even post consumer scrap can easily be recycled and converted into a valuable high performance moulding material [22, 23].

Thermoplastic composite components achieve a typical weight saving of 40% over steel and 20% over aluminium. Innovative polymers and composites are dramatically broadening the range of applications and commercial production of thermoplastics. Made from both well-established and novel polymers, materials such as long and short fibre reinforced thermoplastics; wood-plastic composites and nanocomposites are experiencing high growth. These durable, light, environmentally friendly and chemically stable composites also offer a myriad of possibilities in terms of optimising materials to suit applications. They have the ability to create more

complex shapes from different forming operations. Compared to thermosets, most thermoplastics are extremely ductile. This ductility gives the final composite greater impact resistance and damage tolerance [22, 23].

With the increasing interest in using fibre reinforced thermoplastic matrix composites, there is also an increasing demand for knowledge and basic understanding of the development and processing technology for these new materials. The biggest differences in the fabrication technology of fibre composites having thermoplastic matrices compared with composites having thermosetting matrices are the much higher viscosity and higher processing temperature of the thermoplastic composites. The high melt viscosity of the thermoplastic matrices makes it difficult for molten plastic to penetrate the fibre bundles and ensure complete wetting of all individual fibres [26].

Thermoplastic composites can be split into two broad categories viz Glass Mat Thermoplastics (GMT) and Advanced Thermoplastic Composites (ATC).

2.5.1 GLASS MAT THERMOPLASTICS (GMT)

GMTs uses nearly any thermoplastic as the matrix material, however, in practice choices have been limited to polyvinyl chloride, polypropylene, polyamide, polyesters, polycarbonate and polyphenylene sulphide, with polypropylene accounting for about 90% of commercial use. It is also a suitable material when service temperatures remain below 110°C. When higher temperatures are required, and service conditions are harsher, the more expensive polyphenylene sulphide can be used [22, 23].

E-glass fibre in the form of chopped fibres, random chopped fibres or continuous mats are the most common reinforcing phase. Due to the light weight and high toughness, GMTs have been adopted by the automotive industry. Their application include seat frames, battery trays, bumper beams, load floors, front ends, valve covers, rocker panels and under engine covers [22, 23, 27]. It also has potential applications in other fields such as shipping containers, blower housing and door skins [28].

2.5.2 ADVANCED THERMOPLASTIC COMPOSITES (ATCs)

Originally, advanced thermoplastic composites (ATCs) used amorphous resins such as polyethersulphone and polyetherimide as the matrix material. However, where increased solvent resistance is required, semi-crystalline polymers such as polyether ether ketone and polyphenylene sulphide could be employed. The reinforcement phase may be in strand, woven, knitted or braided fabric forms and can either be made from carbon, aramid and/or S-, R- and E-glass. Carbon is the most popular material for higher temperature applications, while E-glass dominates lower temperature applications.

ATCs have found limited use in the aerospace industry as tougher composite materials. They are analogous to thermoset composites with fibre contents above 50% (v/v) with a highly aligned continuous fibre structure. Actual applications include missile and aircraft stabiliser fins, wing ribs and panels, fuselage wall linings, overhead storage compartments, and helicopter fairings [22, 23].

ATCs offer the following significant advantages over conventional metal alloys. They have outstanding strength, low density, and high impact values, are thermally stable, exhibit low friction factors, are electrically and thermally insulating, and show outstanding abrasion resistance. ATC materials are used in numerous applications across a broad range of industries. Typically, ATCs are used to replace products made of metal alloys. In some applications ATCs can offer comparable or better strength requirements than metal parts, while providing a reduction in weight. This is particularly important in the aerospace and automotive industries, where the use of composite materials results in lighter, faster and more fuel-efficient aircraft and automobiles. Modern civilian jet aircraft comprises of about 25% of composite material, with military aircraft even higher [22].

2.6 HYBRID COMPOSITES

Composite designers tend to specify a single type of fibre for a given component, although the use of a combination of reinforcement forms to reduce manufacturing cost is becoming very common. Multi-component compounding such as combining different fibres in the same structure produces a hybrid composite. This is done to;

- reduce the amount of individual components in the composite
- develop a cost effective and a wide range application for the composite.

It is an effective approach to improve properties and reduce the overall cost. Hybrid composites offer a suitable alternative in the design of structures where a single fibre type composite does not satisfy all the requirements [29].

A novel use of hybrid composite is the development of the F-14 overwing fairing, which combined boron and glass fibres to produce a component that was 25% lighter and 40% cheaper than the original metal design [30, 31]. Another example is the B-1 horizontal stabilizer developed primarily from carbon fibres with boron added in the skins to provide stiffening and improved strength [31, 32].

Alternatively, some components such as helicopter rotor blades may require exact stiffness and strength and this can be achieved more readily with hybrid composites, usually with mixtures of glass and carbon fibres [33]. The concept of hybridization is an extension of the composite principle of combining two or more materials so as to optimise their properties. As a result any combination of dissimilar materials could in fact be thought of as a hybrid. In this study it will be restricted to the mixing of fibres and fillers in a single resin.

An important aspect of using hybrids is that they allow closer tailoring of the composite properties to suit specific requirements that cannot be achieved with single fibre type composites. This is possible if there is adequate understanding of the underlying mechanisms of stiffening, strengthening and toughening [34]. In addition to the choice of individual fibre species or fillers for hybridization, there is also the important question of arrangement or dispersion of the separate species within the

composite. A brief description of the different forms of hybridization in composites is presented below.

2.6.1 SKIN-CORE STRUCTURE

In this type of hybrid composite arrangement the outer plies must sustain tensile and compression loads in bending applications. They are also intended to protect the load bearing core against impact or abrasion damage [34].

2.6.2 PLY-BY-PLY LAMINATION

In many tensile or tensile-compression applications, the composite plies may be placed in some balanced arrangement throughout the sheet thickness so as to achieve a predictable elastic response. In such situations it may be advantageous to place the plies with higher strain to failure on the outside to improve resistance to external damage or crack initiation [34].

2.6.3 TOW-BY-TOW HYBRIDIZATION

This type of hybridization calls for a prefabricated hybrid reinforcement, either woven or nonwoven, of given composition. This provides a more homogeneous dispersion of reinforcement species in the composite [34].

2.6.4 INTIMATE MIXTURE OF FIBRE HYBRIDIZATION

This is the finest level of hybridization where two species of fibres/fillers are intimately mixed in random fashion. The degree of intimacy here is difficult to achieve with continuous fibres. However, the short fibre fabrication process readily permits the preparation of random, intimate mixtures of two or more species of fibres in prepreg sheets [34].

2.7 SHORT FIBRE REINFORCED COMPOSITES

Short fibres are discontinuous with an average length of less than 8mm for injection moulded thermoplastic polymer matrix composites and between 8 to 50mm for compression moulded polymer matrix composites agents [35]. Depending on the part thickness and the processing method used, the fibre orientation in the short fibre reinforced composite can either be two dimensionally random or three dimensional random. The fibre orientation is two dimensionally random if the fibre length is, in general, greater than the part thickness. These composites are usually compression moulded or transfer moulded. Three dimensional fibre orientations is likely to occur in short fibre reinforced injection moulded polymer matrix composites if the part thickness is greater than the fibre length. However, depending on the processing condition and the flow properties of the material, there may be a preferred orientation of fibres in these composites [33, 35].

Compared with continuous fibre reinforced polymers, short fibre reinforced polymers have much lower strength properties and modulus values; yet they are used in large quantities in a variety of applications such as consumer goods, electrical components, automotive structures, sports and leisure equipment as well as in aerospace applications. The reason for their widespread use is that:

- They can be mass produced relatively easily using processes such as injection moulding and compression moulding. These processes which involve short moulding times are much less labour intensive than those used for continuous fibre laminates [33, 35].
- The mechanical properties obtained from short fibre reinforced polymers are better than those of unreinforced polymers. For structural and semi-structural applications, their specific strength and stiffness are sufficiently high to compete with steel and aluminium alloys [33, 35].
- Unless there is a preferred orientation due to processing conditions, short fibre reinforced polymers can be considered isotropic if fibre orientation is truly three dimensionally random. Planer isotropy will be observed if the random fibre orientation is two dimensional [33, 35].

The properties of short fibre reinforced composites depend as much on the fibre length. In fact, the efficiency of strengthening by the fibres depends on the fibre length. For effective strengthening, the fibre length must be equal or greater than the critical fibre length (l_c). For a given loading on a composite the maximum stress occur away from the fibre ends as indicated in the diagram below.

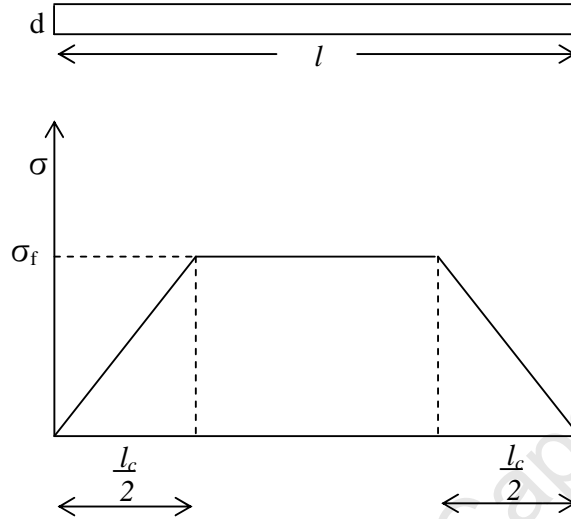


Figure 2.5: Variation of tensile stresses along a fibre length [33].

As the shear effect builds up from the fibre ends, the stress carried increases until it reaches a maximum (σ_f). To achieve this level of stress in the fibre the fibre length must be at least equal to l_c . The critical fibre length (l_c) is the minimum fibre length for a given diameter (d) which will allow tensile failure of fibre instead of shear failure of the interface. The critical fibre length may be determined by considering a force balance in the fibre when the fibre stress is σ_f [33]:

$$\text{Tensile force in fibre} = \frac{\sigma_f \pi d^2}{4} \quad 2.1$$

$$\text{Shear force at interface} = \frac{\tau \pi d l_c}{2} \quad 2.2$$

$$\text{By equating these two quantities: } l_c = \frac{\sigma_f d}{2\tau} \quad 2.3$$

where τ in the interfacial shear strength.

The stress transfer from the matrix to the fibre occurs by a shear transfer mechanism. The efficiency of the transfer depends on the shear strength and the orientation of the fibre relative to the stress direction [36, 37]. The stronger the interfacial bond strength the better the stress transfer from the matrix to the fibre. This is an indication that the l_c is dependant on the shear strength.

2.8 MODELS TO PREDICT ELASTIC PROPERTIES OF COMPOSITES

It is convenient to predict performance of different materials under various loading conditions by the use of simple performance indices [38]. The elastic properties of a composite can be predicted by the rule of mixtures. The rule of mixtures assumes that the matrix is intimately bonded to the fibres, and when a composite is loaded parallel to the reinforcing fibres, the rule of mixtures also assumes that the strain in the matrix is the same as that in the fibres. For such a system, the elastic modulus of the composite (E_c) can be predicted by;

$$E_c = E_f V_f + E_m V_m \quad 2.4$$

where E_f is the elastic modulus of the fibres and E_m is the elastic modulus of the matrix, while V_f , V_m are the fibre volume fraction and matrix volume fraction, respectively.

When a composite is loaded perpendicular to the reinforcing fibres, the rule of mixtures assumes that the stress in the composite is the same as the stress in the fibres and in the matrix. The elastic modulus of such a system is given by;

$$E_c = \frac{E_f E_m}{E_f V_m + E_m V_f} \quad 2.5$$

These equations are used to predict the modulus of continuous fibre reinforced composites. The effective modulus for short fibre reinforced composites is lower than that of long fibre materials since the reinforcing efficiency for short fibres is lower than that of long fibres. This is due to the role played by the fibre length and fibre



orientation in the short fibre reinforced composite [12, 33]. To predict the effective modulus of short fibre reinforced composite, the rule of mixtures needs to be modified to include fibre length and fibre orientation efficiency factors.

For unidirectional aligned materials with fibre length l , a modified rule of mixtures often used is;

$$\mathbf{E}_c = \eta \mathbf{E}_f \mathbf{V}_f + \mathbf{E}_m (1 - \mathbf{V}_f) \quad 2.6$$

where η is the length efficiency parameter and it is given by:

$$\eta = 1 - \left[\frac{\tanh\left(\frac{1}{2}\beta\ell\right)}{\left(\frac{1}{2}\beta\ell\right)} \right] \quad 2.7$$

$$\text{and } \beta = \left[\frac{8G_m}{\mathbf{E}_f \mathbf{D}^2 \log_e(2R/D)} \right]^{\frac{1}{2}} \quad 2.8$$

where $2R$ is the interfibre spacing, G_m is the shear modulus of the matrix and D is the fibre diameter.

For non-aligned systems there is a distribution of fibre orientation that reduces the reinforcing efficiency even further. An additional fibre orientation efficiency factor (η_o) is introduced. Therefore;

$$\mathbf{E}_c = \eta_o \eta \mathbf{E}_f \mathbf{V}_f + \mathbf{E}_m (1 - \mathbf{V}_f) \quad 2.9$$

Values for η_o have been calculated for simple fibre orientation distributions assuming elastic deformation of the matrix and fibres and equal strain are tabulated below.

Table 2.1: Orientation efficiency factor η_o for some systems [33].

Fibre orientation	Stress direction	η_o
Longitudinally aligned	Parallel to fibres	1
Two-dimensional random	Any direction in the plane of fibres	0.375
Three-dimensional random	Any direction	0.2

2.9 FIBRE REINFORCEMENTS

The most common types of fibre reinforcements used in advanced PMC for high tech applications are mostly glass, carbon, aramid and boron fibres. E-glass is selected in the majority of automotive, durable goods and consumer goods applications because of its low cost. For most aerospace applications glass and carbon fibres are selected for their low cost, high modulus and strength. Aramid fibres, such as Kevlar 49, are finding applications in both aerospace and marine composites because they have a higher strength to weight ratio than carbon fibres [10, 39].

In high performance composites, the mechanical performance is controlled by the choice of reinforcing fibre and the fibre architecture. The choice of matrix has comparatively little influence on basic stiffness or strength, although it might profoundly influence impact, fatigue and creep behaviour. The best performing systems utilise a high volume fraction (V_f) of a high performance fibre, with the fibre orientation aligned to the predicted service loading [40].

The efficiency of the reinforcement increases with its aspect ratio, reaching maximum level for continuous fibre. By increasing the reinforcement length the impact strength of the composite may also be improved, but the possibility to flow during processing to create complex shapes is generally reduced considerably [41, 42, 43, 44].

2.9.1 GLASS FIBRES

Continuous glass filaments are manufactured by allowing molten glass to pass through minute holes in platinum-rhodium alloy bushings under gravity and drawing the filaments so produced. Before the filaments are drawn and spun into fibres, they are coated with coupling agent to protect them from abrasive damage during handling and also to improve their compatibility with the matrix during composite forming [11, 45, 46]. A schematic diagram for the manufacturing process of glass fibre is shown in figure 2.6 below [47, 48].

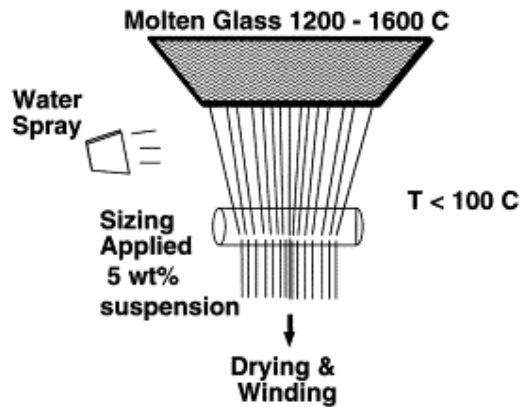


Figure 2.6: Schematic diagram for the manufacturing and sizing of glass fibres [47, 48].

There are different types of glass fibres available due to the wide variety of properties they offer. Some of the common ones are A-glass, C-glass, D-glass, E-glass, R-glass and S-glass. The designation letter is given to a particular glass because of its superior properties. E-glass for instance has a low electrical conductivity whereas C-glass has high chemical resistance and S-glass has a high strength. One type of glass differs from the other by the percentage composition. Glass fibres are isotropic; meaning for a particular glass fibre type, the elastic modulus and strength as well as thermal expansion coefficients is the same in all directions [11]. The diagram in figure 2.7 below shows the internal structure of glass [12, 46].

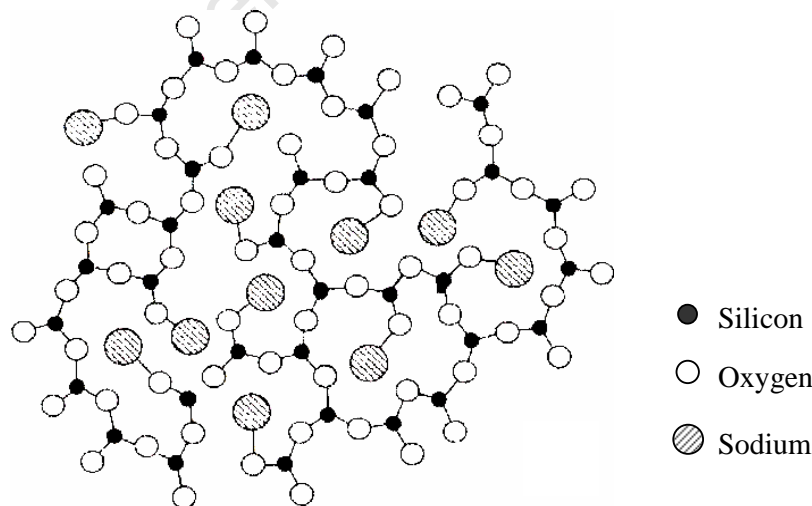


Figure 2.7: Schematic diagram showing the internal structure of glass fibre [12, 46].

The type of glass fibre mostly used for composite manufacturing is E-glass. This material has a calcium aluminium borosilicate base and is a good general purpose fibre. However, in the aircraft industry, S-glass fibre is the most extensively used for composite manufacturing. This material has a higher strength and modulus than E-glass (see table below) and its strength is retain at higher temperatures [13].

Table 2.2: Mechanical properties of E-glass and S-glass [49].

Typical Properties	E-Glass	S-Glass
Density (g/cm ³)	2.60	2.50
Young's Modulus (GPa)	72	87
Tensile Strength (GPa)	1.72	2.53
Tensile Elongation (%)	2.4	2.9

2.9.2 CARBON FIBRES

Carbon is the most widely used reinforcing fibres in composites for the aircraft industry. It has probably had a greater influence on the use of composites in the aircraft industry than any other fibre. Carbon fibres can be produced from a number of precursors to offer different properties but the two which are mainly used are made from polyacrylonitrile (PAN) and pitch [13].

The PAN based carbon fibres are produced through a multi-stage process. The initial stage converts the linearly oriented fibre to a ladder oriented fibre. The next stage is an oxidation process during which the fibres are heated to 350°C in an atmosphere of oxygen. This is followed by a condensation stage where the fibres are heated to about 1200°C in an inert atmosphere. This produces fibres which are strongly aligned but have poor crystal arrangement. The fibres are then subjected to a graphitization stage by heating in an argon atmosphere to temperatures above 1500°C. The temperature of treatment at this stage modifies the fibre properties; therefore it is necessary to tailor the heat treatment to obtain the optimum properties for the fibre [13].

The pitch-based carbon fibres are also produced by a multi-stage process. The first stage polymerises the pitch to produce a highly anisotropic product with a high degree of orientation along the fibre axis. The fibres are then oxidized and carbonized at 2000°C. The final stage involves heating the fibres to 2900°C to increase the orientation of the fibres even further. This process produces fibres with very high modulus without the necessity of high temperature stretching [13].

Carbon fibres also have good mechanical properties and can sustain temperatures as high as 400°C under oxidizing conditions and even higher temperatures if the supply of oxygen is limited. Carbon fibres are available in three moduli of elasticity as shown in Table 2.3 below. The modulus and strength of the fibre depend on the final heat treatment temperature. Depending on the modulus of elasticity the failure strain of the fibre also varies as shown in the table below [50].

Table 2.3: Mechanical properties of the different forms of carbon fibre [49].

Typical Properties	High Strength	High Modulus	Ultra-High Modulus
Density (g/cm ³)	1.8	1.9	2.0 – 2.1
Young's Modulus (GPa)	230	370	520 – 620
Tensile Strength (GPa)	2.48	1.79	1.03 – 1.31
Tensile Elongation (%)	1.1	0.5	0.2

2.9.3 ARAMID FIBRES

These were the first organic fibres to be produced which had the properties that made them suitable for reinforcing structural composites and were introduced in the early 1970s. The first fibres produced by Du Pont under the trade name Kevlar contain the chemical polyparaphenylene terephthalamide.

The aromatic ring structure contributes to its high thermal stability and the para configuration leads to stiff rigid molecules resulting in high strength and modulus. It is a liquid crystal polymer, therefore if it is taken into solution then extruded through a

spinneret it produces a fibre which has straight polymer chains and is extremely well aligned parallel to the fibre axis. Aramid fibres have high tensile properties and since they have a low density their specific strength and modulus are very high. They do, however, have rather low compression properties and are affected by moisture [13].

2.9.4 FIBRE VOLUME FRACTION

The fibre volume fraction (V_f) is the most important factor that controls the mechanical behaviour of composites. Higher fibre volume fractions lead to higher strengths and stiffness. In the case of polymer matrix composites, very high fibre volume fraction of the order of 60 to 65% may be achieved with uniaxial prepreg, while a volume fraction of typically 50 to 55% may be realised for the woven biaxial prepreg [40, 50]. In high performance aerospace PMCs, the fibre volume fraction is typically of the order of 55 – 60%. Lower volume fractions are used for random fibre PMCs [10]. When designing composite materials, it is important to ensure that the desired volume fraction of both matrix and fibres are achieved for optimal properties.

2.9.5 FIBRE – MATRIX INTERFACE

The fibres in a composite material provide the basic strength of the composite but, if full strength and stiffness is to be achieved, it is essential that they are well bonded to the matrix. The efficiency of the composite is therefore greatly affected by the interface between the matrix and the reinforcing fibres. If there is a weak interface then the laminate will have a low stiffness and strength but it will have a greater resistance to fracture. Conversely, a composite laminate with strong interface will have a high strength and stiffness but the composite will tend to be brittle. In order to enhance the bond between fibre and matrix, it is often necessary to apply a coupling agent to the fibres [12, 13]. The role of the coupling agent is to enhance the chemical and physical bonding between the matrix and the reinforcement materials. It also protects the fibre surface from environmental degradation due to moisture or exposure to reactive fluids [10]. Coupling agents are also believed to promote better wetting and matrix infiltration of the fibres resulting in lower void content, especially in composites with high reinforcement volume fractions [51].

The properties of the interface between the fibre reinforcement and the polymer matrix have a significant influence on the mechanical properties of the FRP. To produce a high modulus composite requires adequate adhesion between the fibre and the matrix. The fibre-matrix interface is also important in influencing load transfer in the vicinity of a broken fibre and therefore has a role in determining the tensile strength [21]. The interfacial properties also influence the resistance to crack propagation in a composite and therefore its fracture toughness.

The function of the interface is to transfer any stress acting on the matrix to the fibres. If the interfacial bond is not strong enough, any stress applied to the matrix may not be properly transferred to the fibres. This may result in premature failure of the matrix with the fibres not having been significantly stressed [52]. The methods of enhancing the interface for the different fibres are given below.

2.9.6 LOAD TRANSFER BETWEEN FIBRE AND MATRIX

The strain capacity of organic polymers is typically much larger than the strain capacity of high strength fibres. This larger strain capacity of the matrix plays an important role in load transfer mechanism between fibres. The matrix does not crack till the failure of the composite and therefore, there is an efficient transfer of forces between fibres through shearing of matrix. This is particularly true for samples that fail in tension because in both tension and flexure tests, there is no stress concentration on fibres due to cracking of the matrix [50].

2.9.6.1 Glass Fibres

Glass fibres tend not to bond well with polymers and because of this they are normally coated with a size which has a two-fold effect: it protects the fibres from damage and it provides a chemical link between the fibre and the matrix. Most of the sizes currently used in polymer-based composite are silanes. Hydrolysis of the silane coupling agent forms silanol solution. The silanol and water compete with one another to form hydrogen bonds with hydroxyl groups on the surface of the fibre. Once the fibres have dried a condensation reaction occur and polysiloxane layer is

bound to the fibre surface and the R groups are exposed to the polymer matrix as shown in figure 2.8 below [12, 33].

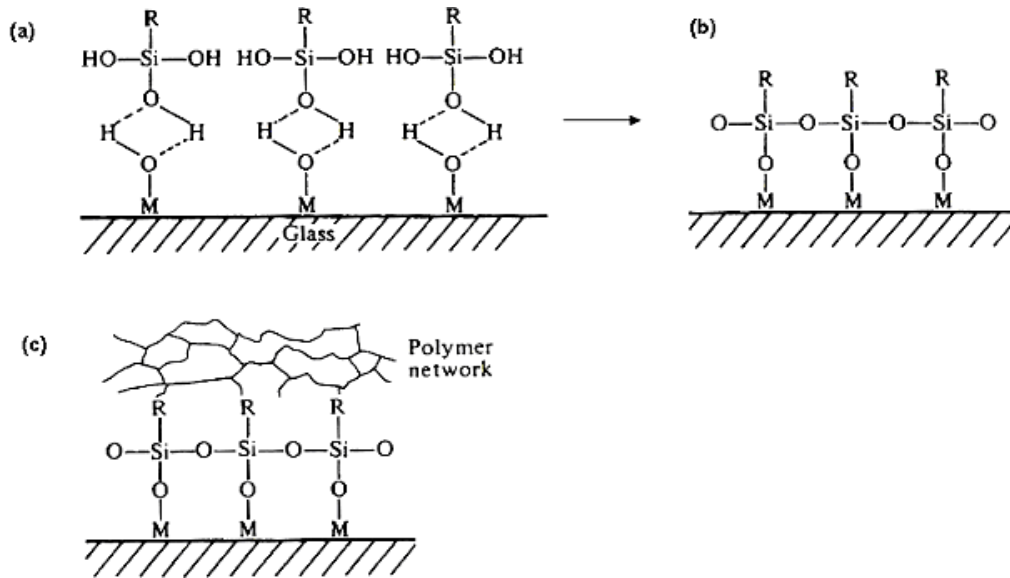


Figure 2.8: Illustration showing (a) hydrogen bonding between silanol and hydroxyl groups (b) polysiloxane bound to the surface of glass and (c) the bond between the polymer network and the R group of siloxane [12, 33, 45].

The silane group will give a chemical link to the glass fibre and the polymer group to the polymer matrix (epoxy). Although it is more difficult to bond in the same manner to a thermoplastic matrix, it is still normal to size the fibre with a silane [13].

2.9.6.2 Carbon Fibre

Carbon has a highly active surface and a range of functional groups can be produced at this surface by different oxidation treatment such as heating in oxygen or treatment in nitric acid or sodium hypochlorite. These functional groups can form chemical bonds directly with unsaturated resins and unsaturated groups in thermoplastic resins. Adhesion promotion has also been obtained by using polymer or silane coatings on the fibres [13].

2.9.6.3 Aramid Fibres

In order to avoid surface damage during processing, aramid fibres can be coated with a polyvinyl alcohol size. Conventional coupling agents have not proved to be very effective but fibres can be given a light coating of an epoxy resin to promote adhesion between the fibres and the matrix [13].

2.10 PARTICULATE FILLERS

Particulate reinforced polymers are commonly referred to as filled polymers in the polymer and composite industries. Particulates or fillers are low cost, micrometer sized solid particles that have traditionally been used to reduce the material cost. However, fillers also influenced the mechanical, thermal and flow properties of the polymers and in some cases, they even act as reinforcing agents [35, 53]. Fillers are often used to control mould shrinkage, improve dimensional stability and, increase part stiffness [35, 54]. Fillers are usually characterised by their shape, size and hardness. Based on their shapes they can be grouped as plate-like (e.g. talc and mica), fibrous (e.g. wollastonite), spherical (e.g. glass micro-spheres, calcium carbonate) and three dimensional irregular shaped (silica) [35, 55]. In general, fibrous and plate-like fillers tend to increase the strength of the filled polymer, provided there is a good wet-out and bonding with the matrix [35]. The filler of interest in this study is the plate-like talc.

2.10.1 TALC FILLER

Talc in its pure form is a hydrated magnesium silicate with a chemical formula of $3\text{MgO}\cdot 4\text{SiO}_2\cdot 3\text{H}_2\text{O}$ [12, 35]. Talc has a layered plate-like structure consisting of a magnesium hydroxide layer sandwiched between two silica layers. These three are held together by weak Van der Waals forces and therefore they can slide past each other relatively easily when subjected to shearing. This is responsible for the soft slippery feel of talc [35].

The incorporation of talc as a filler in thermoplastics is a common practice in the plastics industry. It is done with the purpose of reducing production cost and to improve the toughness, stiffness and strength of the product [56, 57, 58].

Talc is the softest mineral filler used in the plastic industry. It has high resistivity, low gas permeability, low abrasiveness and high lubricating property. All these qualities are attributed to the plate-like structure of talc. The aspect ratio of talc fillers can be as high as 20:1. Talc with a high aspect ratio has shown reinforcing ability in high-density polyethylene (HDPE), polypropylene (PP) and other thermoplastics, especially when they are treated with coupling agents to promote better bonding with the polymer [35, 56, 59]. The addition of talc provides the following properties to the thermoplastic composite:

- Increase stiffness and flexural modulus
- Increase surface hardness and improve scratch resistance
- Reduces shrinkage and warping
- Provides good surface aesthetics and appearance
- Lower cost and
- Improve processability [53, 60, 61].

2.11 MATRIX MATERIALS

The main function of the matrix is to transfer load to the fibres. The matrix is also responsible for providing a fairly rigid medium to hold the reinforcement in place. In the case of continuous fibre composites, the matrix keeps the fibres aligned. It also provides protection against friction between fibres or between fibres and an external medium. The matrix is usually made of tough materials and can act as a crack arrestor. It also provides chemical protection to the composite.

The properties of the matrix that affect the mechanical behaviour of composites are:

- The modulus of elasticity
- Stress-strain behaviour

- Strain capacity
- Bond strength between fibres and matrix as well as
- Strength in tension, compression and shear

2.11.1 THERMOSET RESINS

Thermosetting resins, such as epoxies, vinyl esters, and polyesters are the most commonly used resins in continuous fibre PMC. Phenolics, bismaleimides and polyimides are used for high temperature applications. The starting materials for these and many other thermoset resins are low viscosity liquid-like pre-polymers. Long continuous fibres can be relatively easily coated and wetted by these pre-polymers before curing reaction starts that changes them into solid thermoset polymers [10, 12]. During the curing process the liquid resin is converted into a hard rigid solid by chemical cross linking, which leads to the formation of a tightly bound three dimensional network. This is usually done while the composite is being formed. Mechanical properties depend on the molecular units making up the network and on the length and density of the cross links. Curing can be achieved at room temperature, but it is usual to use a cure schedule which involves heating at one or more temperatures for predetermine times to achieve optimum cross linking and hence optimal properties. A relatively high temperature final post cure treatment is often given to minimise any further cure and changes in properties during service. Shrinkage during curing and thermal contraction on cooling after cure can lead to residual stresses in the composite [12].

The most commonly used thermoset resins is epoxy. This is because epoxies in general are tougher than unsaturated polyesters and vinyl esters. They also shrink less during curing than polyesters. Epoxies can have good resistance to heat distortions. They can be partially cured, so that prepregs can be supplied. Epoxies are superior in most respects to the alternative thermosetting systems, which are sometimes preferred simply on the grounds of lower cost [12]. Although epoxies can be expensive, it may be worth the cost if high performance is required.

Thermoset are more thermally stable and chemically resistant. They have high glass transition temperatures [10]. They are however brittle and have much lower strains to failure than thermoplastics.

2.11.2 THERMOPLASTIC RESINS

Unlike thermosetting resins, thermoplastics are not cross-linked. They derived their strength and stiffness from the inherent properties of the monomer units. In amorphous thermoplastics there is a high concentration of molecular entanglements, which act like cross-links whereas in semi-crystalline thermoplastic there is a high degree of molecular order and alignment. Heating of amorphous material leads to disentanglement and a change from rigid solid to a viscous liquid. In crystalline materials heating results in melting of the crystalline phase to give an amorphous viscous liquid. Depending on the conditions of solidification, both amorphous and semi-crystalline polymers may have anisotropic properties [12].

Thermoplastic polymers have very high viscosity even at high processing temperatures. Wetting long continuous fibres with thermoplastic polymer has been a problem for a long time. Although several innovative processes such as melt impregnation and powder coating have been developed for coating long continuous fibres with thermoplastic resins, high processing and material costs have prevented a wider use of continuous fibre thermoplastic composites [10]. Short fibres are used more commonly with thermoplastic resins because they can be compounded relatively easily by extrusion with these polymers and then injection moulded into desired shapes. Thermoplastics have high tensile strain to failure, greater crack resistance, and higher impact strength. They have unlimited storage life and require less processing time although their processing temperatures are higher. Thermoplastics can be directly recycled, which is a distinct advantage for thermoplastic resins in this environmentally conscious age [10].

Since thermoplastics are fully polymerised before they combine with reinforcing fibres their glass transition and melting temperatures are fully developed. Consequently, the thermoplastic must be melted at high temperature in order to wet

the fibre. Furthermore, once the thermoplastic is melted its viscosity is orders of magnitude higher than the low molecular weight pre-polymers used in thermoset composite processing. Therefore combining thermoplastic matrices with reinforcements and ensuring good fibre wet out and bonding is a significant technical challenge [62].

Since thermoplastics are not cured during the fabrication of parts, rapid cycle times can be realized. The ultimate limitation on cycle times is the heat transfer required to solidify the thermoplastic. For thin cross sections, cycle times in the range of seconds are possible. However, many thermoplastic pre-forms consist of unconsolidated commingled fibres or powder coated fibres. In these cases the time required for complete fibre wet out would extend the cycle time and the cycle times may be in the range of minutes. In contrast, the cure times for some thermosets can be hours long [62].

In addition to their high failure strain, they tend to exhibit good resistance to attack by chemicals. The advanced thermoplastics tend to have generally good thermal stability. The stiffness and strength of PEEK for example are very little affected by temperatures up to 150°C. A composite containing 60% volume carbon fibre in a PEEK matrix has found extensive use in aerospace applications. Other high performance thermoplastics include polyether sulphone, polyphenylene sulphide and polyimide [12].

2.12 HIGH-TEMPERATURE THERMOPLASTICS

Synthetic polymers are remarkably resistant to tension, compression, abrasion, impact, repeated flexure, discolouration and effect of sunlight and corrosive chemicals. They were designed to withstand almost any destructive agent except intense heat. It was not anticipated that they would ever have to maintain their rigidity, shape and strength in aviation-fuel fires, curing ovens, industrial smokestacks and other high temperature environments [63]. Today, the demand for light, strong and formable materials that

can considerably withstand higher temperatures has led to the development of new families of polymers, some of which are discussed below.

2.12.1 POLYETHER ETHER KETONE (PEEK)

PEEK is a semi-crystalline high temperature thermoplastic polymer with a glass transition temperature of 140°C, a melting point of 342°C and recommended processing temperature range of 380 – 400°C [26]. PEEK contains oxygen bonded to carbon in their backbone. The ether group imparts flexibility which makes melt processing easier. It also has aromatic rings in its backbone to increase the transition temperature [62]. Figure 2.9 below shows the molecular structure of PEEK [64].

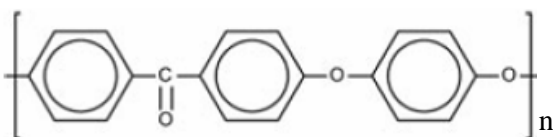


Figure 2.9: The molecular structure of PEEK [64]

PEEK has greater strength and rigidity than many of the other advanced thermoplastics, making it tough over a wide range of temperatures. It has good mechanical properties, including impact resistance, low wear rate and a low coefficient of friction. These properties are also retained over a wide temperature range. PEEK has excellent resistance to burning and very low flame spread rate, even when burning, the material has one of the lowest smoke generation characteristics of the engineering thermoplastics [64].

The use of PEEK in advance thermoplastic composite is well known. The appeal of PEEK is its high strength, high modulus, high impact strength, good fibre adhesion, excellent chemical resistance and non-flammability. Unfortunately it is quite expensive [62]. PEEK is mainly used in the electronic and electrical industry for printed circuit boards. In the aircrafts and aerospace industries and in military applications it is used as an alternative to metals [65].

2.12.2 POLYPHENYLENE SULPHIDE (PPS)

Polyphenylene sulphide (PPS) is a thermoplastic polymer made up of alternating sulphur atoms and phenylene rings in a para substitution pattern as shown in figure 2.10 below. The sulphur in the backbone provides flexibility. The highly stable chemical bonds of its molecular structure impart a remarkable degree of molecular stability toward both thermal degradation and chemical reactivity. The molecular structure is linear with little branching and readily packs into a thermally stable crystalline lattice, hence PPS is a semi-crystalline polymer with a high melting point of about 285°C. Due to its molecular structure, PPS also tends to char during combustion, making the material inherently flame retardant. PPS has not been found to dissolve in any solvent at temperatures below about 200°C [62, 66].

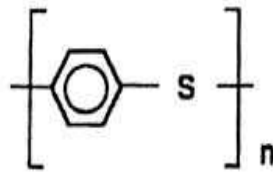


Figure 2.10: The molecular structure of PPS [65, 66].

The main properties of PPS are;

- High strength, hardness and stiffness
- High heat distortion stability
- Good electrical properties
- Low water absorption high chemical and oxidation resistance
- Non-flammable and self-extinguishing.

Processing is by means of injection and compression moulding. The processing temperature is between 290°C – 350°C. During processing good venting of the cavities must be ensured and the mould temperature should be about 130°C [65]. When blended with glass fibres and other fillers, PPS produces engineering plastics having a unique combination of properties including:

- A remarkable combination of both long-term and short-term thermal stability
- Exceptionally high modulus and creep resistance
- Outstanding resistance to a wide variety of aggressive chemical environments

- Precision moulding to tight tolerances with high reproducibility
- Inherent non-flammability without flame retardant additives
- Dielectric and insulating properties over a wide range of conditions [65, 66].

PPS is used frequently as a thermoplastic composite matrix because of its excellent chemical resistance, low moisture absorption and high strength and stiffness. Without modification its impact strength is low. PPS is used in applications such as automobile, aerospace and other technical applications such as pump and bearing housings. Due to its high melting point and good electrical properties it is used in a variety of electronic applications and electronic parts that have to withstand high temperatures [62].

2.12.3 POLYAMIDE IMIDE (PAI)

PAI is an amorphous polymer that performs well at high temperature and stress. It has a very high viscosity in its processing temperature range and can be processed by injection moulding, compression moulding and extrusion. PAI has an outstanding tensile strength and modulus combined with a good elongation to failure, leading to high impact strength. It also has good creep resistance and an inherent flame resistance with low smoke generation. Even without reinforcement its HDT exceeds 275°C. PAI contain an imide and an amide groups as shown in figure 2.11. The amide group included in PAI is to facilitate processing [62, 67].

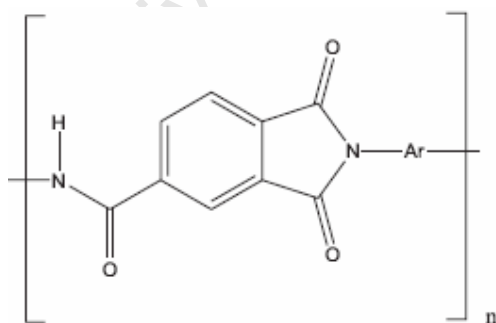


Figure 2.11: The molecular structure of PAI [67]

It is used in the aircraft and automobile industries, electrical and electronic applications and also in applications where high temperature is of great importance.

2.12.4 POLYETHER IMIDE (PEI)

Polyether imide is an amorphous polymer with high strength and rigidity at elevated temperatures. It has long term heat resistance, dimensional stability and good electrical properties. Creep resistance over the long term allows PEI to replace metals and other materials in many structural applications [62, 68]. The molecular structure of PEI is indicated in the figure below [68].

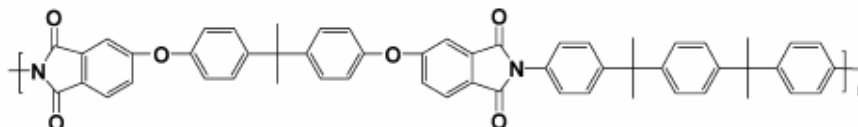


Figure 2.12: The molecular structure of PEI [68]

The presence of an imide group in polyimides makes it much more rigid, leading to high transition temperatures. In PEI an ether group is included in the backbone in order to have a polyimide that can be processed as melt. PEI is amorphous polymer due to its irregular structure. It has a relatively high glass transition temperature. PEI is often selected for composite applications because of its high service temperatures, toughness, good interfacial adhesion and non-flammable behaviour [62].

2.12.5 POLYETHER SULPHONE (PES)

PES is a heat resistant non-crystalline polymer with a high glass transition temperature. It has good strength and stiffness and has shown excellent creep resistance. It is non-flammable and self extinguishing. It also has high tensile and impact strength, high heat distortion stability, good dielectric properties up to 200°C and high chemical resistance [62, 65, 69]. The molecular structure of PES is shown in the figure 2.13 below [69, 70].

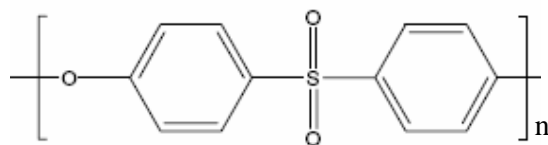


Figure 2.13: The molecular structure of PES [69, 70].

PES can be processed by means of injection moulding, extrusion, compression moulding, and solution casting and sintering. Pre-drying of PES before moulding is recommended [69]. Films and sheets can be thermoformed. It is used in applications where high temperature is of great importance, for example in components of hair dryers, press iron handles and cooking utensils, condenser foils, printed circuits boards and lamp sockets [65].

2.13 DESIGNING WITH COMPOSITE MATERIALS

The rate and progress of technological innovations is dependent on the development of new and better materials. Modern aircraft structures for example have only been made possible by the development of suitable aluminium alloys. The whole progress of the development of aero-engines has been dependent on new and better materials for its construction.

For a material to be used for load bearing applications, it must satisfy certain requirements. The three most important requirements for materials to be used for load bearing applications at normal temperatures are;

- a high resistance to plastic deformation
- a high resistance to fracture and
- a high elastic stiffness.

If it combines the first two properties it is said to be tough. The development of composite over the past fifty years has been an attempt to produce a material with all three properties [71].

2.14 DRIVING FORCE FOR COMPOSITES

Common driving forces for the use of composite materials include the ability to save weight, increase mechanical properties, reduce the number of elements in a component, increase shaping freedom and obtain a unique combination of properties.



Increasingly, composites are being used for all of the above while also achieving a reduction in part cost. Many of these driving forces, together with the manufacturing cycle, often offset the higher raw material costs of the composite constituents to produce a commercially viable end product [44].

The criteria on which composite materials are selected for a particular application are naturally dependent on the industrial sector for which they are intended. For example, aerospace has traditionally been driven by performance. Emphasis is therefore placed on strength, stiffness, weight and quality control when selecting a composite material for the aerospace industry. The automobile industry is typified by high volume applications which require rapid and highly automated techniques. The full potential of the composite in terms of mechanical properties is seldom reached [44].

2.14.1 THE AEROSPACE INDUSTRY

A leading role in the development of both composite materials and processing technology has been taken by the aerospace industry. The high specific strength and stiffness of the composite material offered the potential for reduced fuel consumption and increased range for passenger aircraft and increased performance for military aircraft. The main driving forces for the aerospace industry are therefore primary weight reduction by using a material with higher specific mechanical properties, facilitating secondary weight savings, leading to considerable additional weight reduction. The strong demand for weight savings in aerospace applications as well as the lower sensitivity of this industry to production rates and material costs has led to the development of finely-tuned high performance materials and processing techniques [44].

Reinforcement orientations are specified to suit the principal stresses in the component and prepregs are carefully placed to maintain the desired orientations. The stresses carried in the aerospace structures are often high, requiring continuous fibres for maximum strength and creep resistance. This limits processing techniques to those where fibre lengths are maintained in highly controlled directions and where stiffness is achieved through the intrinsic properties of the composite [44].

The first composite primary structure to enter production on a commercial aircraft was the all composite rudder introduced in 1985 for the A300/A310. This consisted of a single piece rudder assembled from a hollow triangle of three honey-comb sandwich panels with carbon fibre/epoxy skins. This replaced its metal counterpart without design changes to the aircraft, reducing 2000 parts including fasteners, for the metal system to less than 100 for the composite system with 20% weight saving and overall cost savings, despite the high raw material cost [44]. Combined with other design changes, the composite rudder and the vertical fin lead to reduced fuel consumption, illustrating the commercial reality of composites in the aerospace industry. Hence traditional aerospace driving forces for weight reduction, which yield improved range and reduced operational costs, are increasingly being extended to the additional requirement of total weight savings [44].

2.14.2 THE AUTOMOTIVE INDUSTRY

The transportation industry represents a potentially large application area for fibre reinforced composites and is driven by a complex set of interacting driving forces. The needs of the aerospace industry led to the development of advanced composites, and the needs of the automotive industry dominated the development of engineering composites, with increased shape complexity and a strong emphasis on decreasing cost.

In the automotive industry vehicle mass reductions necessary to improve fuel economy and reduce emissions interact with improved safety, functionality and refinement. Studies have shown that up to 40% of a vehicle's fuel consumption is related to factors attributed to vehicle inertia losses. Additionally, the location of the weight reduction will have an influence on the safety, comfort and manoeuvrability of the vehicle. By reducing the weight away from the centre of gravity, the handling of the vehicle can be significantly improved [44].

Composite materials have been used commercially in the automotive industry as a means of reducing fuel consumption and cost. The driving force in the automotive industry has been weight reduction, increased safety, and niche products.

Development in the automotive industry have not only been focused on material performance, as in the aerospace industry, but also on the constraints imposed by manufacturing techniques for large parts and the need to reduce production costs. For composite materials to compete with steel and alloys, higher value must be added and cost reductions made in manufacturing cycles to balance the higher raw material cost. The ability to form complex shapes with high design-based stiffness, together with the incorporation of many additional features in the same piece, offers maximum added value to the raw materials through a flexible manufacturing process and the increased design freedom that this offers. This is how composites have provided cost-effective solutions within the boundary of complex driving forces [44].

2.15 FIRE SAFETY OF POLYMER COMPOSITES

Polymers and polymer composites are widely used in many applications. However, most of these materials will burn readily in air or oxygen. The flammability of polymers is a serious issue and severely limits their applications [72, 73]. Recent fire safety concerns put even more stringent requirements for materials used in enclosed and inescapable areas, such as electronic enclosures, high-rise buildings, and submarines, ships, and aircraft cabins [74]. Light-weight, high performance polymeric materials offer many advantages in these applications over conventional metal and ceramic materials, but they greatly increase the fire risk because of their flammability and possible release of toxic fumes and by-products when they burn.

The growing use of polymer composite materials in the aerospace industry has the potential to increase the fire hazard due to the flammable nature of the organic matrix. Without careful management and strict safety regulations, the risk of aircraft fires could increase with the growing use of fibre reinforced polymer composite materials in aircraft. Many polymer composites rapidly ignite when exposed to fire and generate high amounts of heat, blinding smoke and choking fumes. The careful development of fire resistant composite materials is essential to aircraft safety [17, 75].

2.15.1 FIRE SMOKE AND TOXICITY (FST)

There is an increase interest in the use of composite materials in a host of applications or industries. Composite materials are lighter, more resistant to corrosion and may be less expensive than many metal alloys in some applications. In addition to a host of other criteria, any design strategy that replaces metals with composite must address combustion characteristics. Major concerns regarding the use of composites in aerospace applications are to characterise the smoke density as well as the emission of toxic products from composite materials during fire [76].

One of the main safety concerns with polymer composites is the generation of dense smoke caused by fire. Smoke is defined by the ASTM Fire Standards Committee as a visible airborne suspension of solid and liquid particles evolved when materials undergoes pyrolysis and combustion [17]. The particles suspended in smoke are ultra fine, typically between 0.3 to 3 microns in size. The quantity and size of smoke particles is dependent on the chemical composition of the material, char yield and the nature of the combustion process. The smoke produced by a burning composite is a mixture of small fragments of fibre and ultra fine carbon soot [17]. The table below consist of toxic gases that may be found in smoke and their minimum exposure limits.

Table 2.4: List of selected toxic gases that may be found in smoke [76].

Compound	10min emergency exposure limit (ppm)
Carbon monoxide CO	1500
Carbon dioxide CO ₂	50 000
Hydrogen cyanide HCN	50
Nitrogen dioxide NO ₂ (or NO _x)	30
Sulphur dioxide SO ₂	30
Ammonia NH ₃	400
Chlorine Cl ₂	30
Hydrogen chloride HCl	30

The smoke measurement procedure with the cone calorimeter has the ASTM approval as a proposed method [77]. The major immediate toxic threats in a fire situation are

carbon monoxide, a multitude of irritating organic chemicals in the smoke, oxygen depletion and heat. An analysis of toxicological findings in fire and non-fire deaths indicates that CO is still likely to be the major toxicant in modern fires [78].

2.15.2 HEAT RELEASE OF POLYMER COMPOSITES IN FIRE

One of the main safety concerns with using fibre reinforced polymer composites is their combustibility. Many polymer composites ignite when exposed to heat flux, releasing heat that can, in some circumstances contribute to the growth of the fire. Significant quantities of smoke and toxic fumes may also be released, limiting visibility and posing a health hazard. For this reason stringent fire regulations govern the use of composites in aircraft, automobiles, ships, buildings, oil and gas facilities and other applications. These regulations require that the fire reaction properties meet specified levels. The reaction properties that are often used to define the fire hazard include the heat release rate, time-to-ignition and flame spread rate. Other important properties include smoke density, yield of carbon monoxide and smoke toxicity because these determine survivability [79, 80].

Of the many fire reaction properties, it is generally recognised that heat release rate is the single most important in controlling fire hazard. As mentioned before, heat release rate is the critical fire reaction property because it is the driving force for fire spread. It also controls other reaction properties [79]. Heat release rate is defined as the mass loss rate of the material multiplied by its heat of combustion [79]. The amount of heat released from a polymer composite is controlled by the combustion of flammable gas products resulting from the decomposition of the organic components.

In most cases, high strength composites are made with carbon, glass or Kevlar fibres and organic matrices. In spite of the excellent mechanical properties, these composites cannot be used in high temperature applications. In certain cases such as aerospace and automobile structure applications, exposure to high temperature during accidents not only reduces the mechanical properties but also results in toxic fumes and smokes. In applications that require more than 200°C temperature exposure, most organic matrix composites cannot be used [50].

2.15.3 FST TESTING USING THE CONE CALORIMETER

The federal aviation administration (FAA) mandates that the FST properties of non-metallic materials must be measured using the Ohio State University (OSU) cone calorimeter test operated at a heat flux of 35kWm^{-2} , as described in ASTM E906 [17]. As part of the safety regulations, the test material is required to have a total heat release of less than or equal to 65kWm^{-2} over two minutes and a peak heat release rate of less than or equal to 65kWm^{-2} over five minutes duration of test [17].

The FST testing is done to determine the fire worthiness of a material. It is achieved by observing the changes in the fire parameters such as;

- The ratio of CO to CO₂ base on the mass of materials burnt. The CO/CO₂ ratio indicates the efficiency of the combustion process. High CO/CO₂ ratio implies an incomplete combustion.
- Effective heat of combustion (EHC) in MJ/kg. It is the efficiency of emitted gaseous products to combustion. A low EHC means less efficient combustion, therefore better flame retardancy.
- Heat release rate (HRR) in kW/m². It is the rate of fire growth and intensity of the fire. A low HRR implies better fire retardancy.
- Mass loss rate in %·s⁻¹

The rate of smoke formation/released in cm²·s⁻¹ measures incomplete combustion of volatile product [79, 80, 81, 82].

2.16 THERMAL PROPERTIES OF POLYMER COMPOSITES

Thermal properties, such as expansivity and conductivity must be taken into account. This is because composite materials are subject to temperature changes during manufacturing and/or in service. The mismatch between thermal expansivities of constituents may leads to internal residual stresses. These can have a strong effect on the mechanical behaviour [12]. Some of the methods to determine thermal properties and if a polymeric material can survive a particular temperature are discussed below.

2.16.1 THERMO-GRAVIMETRIC ANALYSIS (TGA)

TGA is one of the thermal analysis techniques used to characterise a wide variety of materials. TGA measures the amount and rate of change in mass of a sample as a function of temperature or time in a controlled atmosphere. In this method the specimen is weighed as the temperature is increased. The amount of weight lost is assumed to reveal how much the material has decomposed [63, 83].

TGA measurements provide valuable information that can be used to select materials for certain end-use applications predict product performance and improve product quality. The technique is used to determine thermal stability, oxidative stability, moisture and volatile content, effect of reactive atmosphere and decomposition kinetics of a material [63, 83].

2.16.2 DIFFERENTIAL SCANNING CALORIMETRY (DSC)

DSC is widely used to characterise the thermo-physical properties of polymers. DSC can be used to measure important thermoplastic properties such as melting or softening temperature, percentage crystallinity, glass transition temperature (T_g) and the presence, composition and compatibility of recyclates, regrinds and polymer blends.

DSC is a thermo-analytical technique in which the difference in the amount of heat required to increase the temperature of a sample and a reference are measured as a function of temperature. Both sample and reference are maintained at the same temperature throughout the experiment. The basic principle underlying this technique is that when a sample undergoes a physical transformation more or less heat will need to flow to it than the reference to maintain both at the same temperature. By observing the difference in heat flow between the sample and the reference, differential scanning calorimeters are able to measure the amount of heat absorbed or released during such transition [63, 83].

2.16.3 DIFFERENTIAL THERMAL ANALYSIS (DTA)

DTA is a thermo-analytical method in which a specimen and an inert reference material are heated concurrently at a linear rate, each having its own temperature sensing and recording apparatus and noting the temperature at which it absorbs or gives off heat, indicating some thermal reaction. The thermal energy changes which occur in the course of heating are plotted. The thermogram provides data on the chemical and physical transformation that have occurred, such as melting, sublimation, glass transitions, crystal transitions and crystallization [63].

2.17 MECHANICAL PROPERTIES

Every material requires some degree of standard testing to determine its mechanical properties. The three most common purposes for mechanical property determination are research and development, quality control and design data generation. In the research and development, testing is done to support the study of material performance in comparison to the program objectives, competitive materials, or other developmental materials. Once a material is developed, the tests can also be used to produce the first generation of property data used to market the new material [84].

Quality control characterization is defined primarily by the customer's acceptance test method and product specifications. The specimen sampling, preparation and test methods must rigorously follow documented specifications. Quality control tests usually characterize a couple of the most critical properties that define a product and strive for comparison against historical values rather than absolute properties [84].

Design data generation strives to produce absolute property data. The data must represent the actual mechanical performance of the material under loading conditions like those that will be encountered in service. The test used must be capable of measuring the material property desired, and specimen preparation must rigorously conform to the standards specified in the test methods [84].

Different testing methods are appropriate to meet the range of objectives that one may encounter in material characterization. The mechanical properties of advanced composite materials are determined by specifically designed test methods. These methods are mechanically simple in concept but are extremely sensitive to the composite material and the test execution procedures [84]. The mechanical properties of polymer composites depend mainly on three major factors, namely those of the matrix, reinforcement and the interfacial region between the matrix and the reinforcement. Reinforcements, depending on their shape, size, and distribution in the matrix affect the composite properties. In order to strike a proper balance in addressing testing needs, the engineer must fully understand the cause and effect relationship between all aspects of the specimen preparation and testing procedures and the quality of the results. This section attempts to elaborate on some of the key concepts of mechanical property determination for composite materials.

2.17.1 TENSILE TESTING

The most common mechanical test is the tensile test; used to determine the strength of materials. It is a measure of the resistance of the material to pulling stresses. For most structural materials, the tensile properties are essential elements of materials design requirements. The tension test is used to measure the tensile strength, tensile modulus and the ultimate strain to failure. During testing, adequate gripping of the test specimen is a major issue. The test specimens are dumbbell shaped which consist of gripping regions where loads are introduced to the specimen, a gauge section of reduced cross-sectional area to promote failure away from the grips. The specimen is tested to failure while recording load, crosshead displacement, and strain. The test data is used to generate a stress-strain curve as shown in figure 2.14 below [85]. From the stress-strain curve, the tensile properties such as the yield strength (σ_y), the ultimate tensile strength (σ_t), the tensile modulus (E) and the ultimate strain to failure (ϵ_f) can be obtained [84].

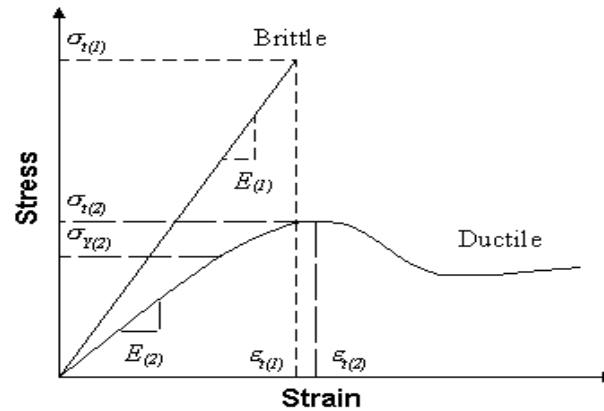


Figure 2.14: Stress-strain curves illustrating a brittle and a ductile behaviour [85].

2.17.2 FLEXURAL TESTING

Flexural or bending test is usually performed to determine the flexural strength and modulus of a material. During testing, the top part of the specimen is under compression while the bottom surface is under tension. Flexural testing can be done in either the three-point or four-point bending method. The four-point bend test is commonly performed with two load noses located at a quarter span or a third span points. The three-point test is performed with the load nose at the mid-span location as illustrated in the diagram below [85].

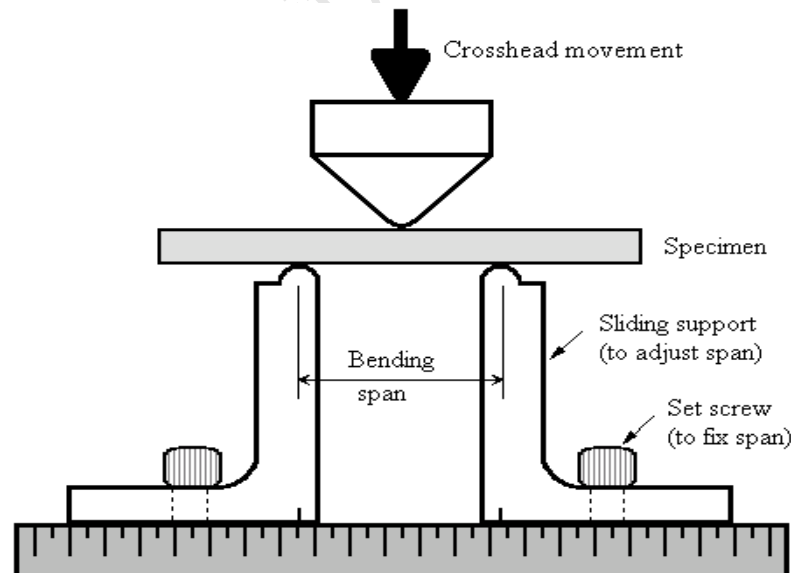


Figure 2.15: A schematic diagram for three points bend test set up [85].

For the three-point loading the flexural stress and flexural modulus of the material can be calculated from the following equations.

$$\text{Flexural stress, } \sigma = \frac{3PL}{2bh^2} \quad 2.10$$

$$\text{Flexural modulus, } E = \frac{PL^3}{4bh^3\delta} \quad 2.11$$

where, P , L , b , h , and δ are the load, support span, specimen width, specimen thickness and deflection at the centre of the specimen, respectively [84, 85].

2.17.3 IMPACT TESTING

This form of testing is used to determine the ability of a material to withstand a sudden impact. The impact damage depends on the velocity, mass, shape, hardness and initial height of the impacting object as well as the properties of the target material [86]. The impact behaviour of a material is usually determined using several test methods. The test methods of interest in this study are the Izod and Charpy, impact test methods.

The Izod and Charpy impact tests are performed on the same instrument with different clamps and pendulums. The two tests differ in the manner in which the test specimen is placed during testing. The Izod test specimen is clamped vertically with the notch facing the pendulum while the Charpy test specimen is placed horizontally with the notch facing away from the pendulum [87].

2.17.4 CREEP

Polymer composites can be considered as both elastic solids and viscous fluids, therefore they are classified as viscoelastic materials [88, 89]. Their ambient operating temperatures coincide with or are close to their viscoelastic phase; therefore creep becomes a significant factor in assessing their long term load carrying capacity [90]. Creep is the time dependent deformation of a material subjected to a constant load and

at constant temperature. The stress that causes creep in a material is usually less than its yield stress and for the effects of creep to be observed, time must be allowed [88, 91]. The effect of creep is a permanent, irreversible deformation. All materials do creep under constant load, but some creep so small that it is impossible to see with the naked eye. During material creep the strain does not only depends on stress but it also depend on time and temperature the material is exposed to. This can be represented mathematically as [88, 89];

$$\varepsilon = f(\sigma, t, T) \quad 2.12$$

where ε is creep strain, σ is applied stress, t is time allowed for creep and T is the temperature at which the materials has been exposed to during its operation. Elevated temperatures and high stresses increases the creep rate while longer time allows the material to run to rupture.

Most polymers do creep at room temperature and at stresses far below their ultimate strengths [91, 93]. Therefore in order for the creep resistance and mechanical stiffness of the polymeric material to be satisfactory, it is necessary to ensure that the service temperature of the materials is below its glass transition temperature (T_g) [88]. The creep behaviour in randomly oriented fibre reinforced polymer composites is largely controlled by the matrix behaviour as opposed to unidirectional oriented composites where fibres control the viscoelastic behaviour in the fibre direction [91, 92]. Creep of polymer composites also depends on the type, orientation and volume fraction of the reinforcing fibre. The creep performance of short fibre reinforced thermoplastic composites is affected by the nature of stress transfer, which in turn is influenced by the fibre-matrix adhesion [91, 92].

Creep testing is done by subjecting a specimen to a load at constant temperature. The deformation or strain is measured and plotted as function of time elapsed. There are two common creep phenomena as shown below; tensile creep and flexural creep. Tensile creep is uniaxial while flexural creep is multi-axial. Creep testing is done to generate engineering information that provides a better understanding of the creep behaviour of the material.

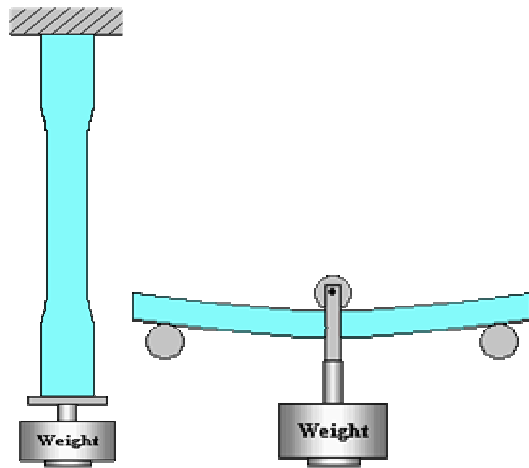


Figure 2.16: The tensile creep test and flexural creep test [94].

Data from the creep test and creep rupture test are necessary to predict the creep modulus and strength of the materials under long term loads and to predict dimensional changes that may occur as a result of such loads [90 94].

2.18 FAILURE MECHANISMS

The rapid progress in composite material technology continues to raise their performance potential with the development of stronger fibres and tougher matrix systems. An understanding of the failure mechanisms in composites is developing, which is providing guidance to material developers as well as structural designers. The failure mechanisms in composite materials depend on the characteristics of the fibre and matrix materials as well as the interfacial bond strength [51, 95]. The most common mechanisms of failure in short fibre reinforced composites are; matrix cracking, fibre-matrix debonding, fibre bridging, fibre pullout, fibre fracture and matrix deformation. These processes do not occur in sequence but interact with each other as summarised in figure 2.17 below [96].

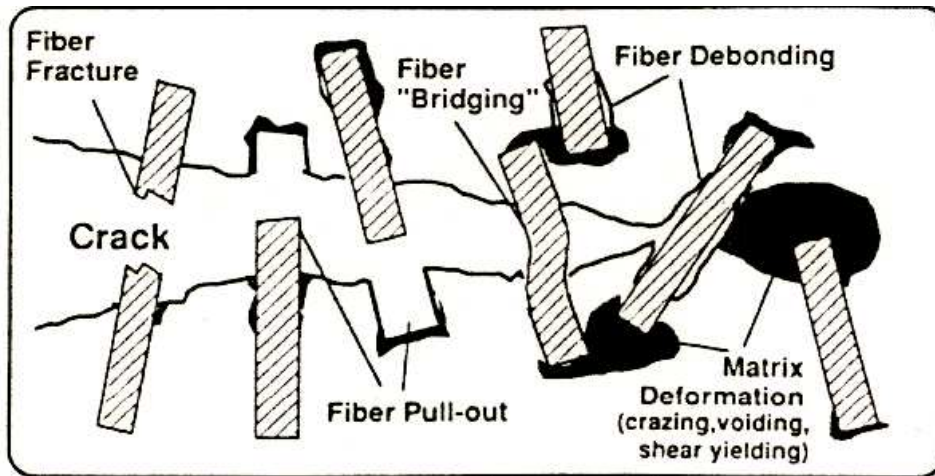


Figure 2.17: Failure mechanisms in short fibre reinforced composites [96].

Matrix cracking is one of the common forms of failure that occur in fibre reinforced composites. The formation of matrix cracks is influenced by the internal and external geometries of the laminate. This includes fibre orientation and composite thickness. Due to the presence of fibres in a composite, matrix cracking is likely to be arrested and blunted at some reinforced boundary. This crack arresting mechanism may result in the initiation of another matrix crack elsewhere in the composite [95].

When an advancing matrix crack approaches a neighbouring fibre, the stress field ahead of the crack tip induces a shear stress at the fibre-matrix interface. If the interfacial shear strength τ is too high, matrix and fibre fracture without significant interfacial debonding occur, resulting in low toughness and catastrophic failure. If τ is not too high, interfacial debonding will occur [95]. Debonding is one of the potential toughening mechanisms for the composite. The two most important energy absorbing failure mechanisms in fibre reinforced composite are debonding at the fibre-matrix interface and fibre pullout. Conversely, if τ is too low, extensive interfacial debonding occurs with decoupling of fibres and matrix. This result in a poor stress transfer between the fibre and the matrix; which lead to a failure of the basic concept of fibre reinforcement. [95, 97].

Fibre bridging between two crack surfaces often occurs in composites that have a matrix of low strength. Fibre bridging can contribute to toughening of the composite

as it allows further debonding at the fibre-matrix interface, further elongation of the fibre and reduction of the crack tip stress [95].

As a matrix crack advances, the fibres bridging the crack are progressively elongated until fracture. Relaxation of the strain energy in the fibre following the fracture causes contraction in the axial direction and expansion in the radial direction leading to the fibre snapping back into its matrix socket. Further crack opening leads to fibre pullout. This is usually resisted by frictional forces at the fibre-matrix interface [95]. If the interfacial bond is relatively weak, the crack propagation is interrupted by the debonding process and instead of moving through the fibre, the crack moves along the fibre surface, allowing the fibre to carry higher loads. Fibre pullout occurs because fibres do not all break at the crack plane. Since they break at random locations away from the crack plane, one of the broken fibres ends pulls out from the matrix as the two crack faces open up with increasing load. The process of debonding instead of fibre failure, and subsequently fibre pullout, are essential in achieving high fracture toughness [97].

Fibre fracture generally occurs at the final stages of composite failure. Debonding between fibre and matrix usually occur at the site of the fibre breakage. During impact loading for instance, fibre fracture may occur as a result of a heavier impactor. The fibre failure may be caused by either high local contact stress or micro-buckling [95].

2.18.1 EFFECT OF TEMPERATURE ON FAILURE

The fracture behaviour of polymers and polymer composites has been found to be temperature dependent. The failure mode and performance can be very different depending on the temperature. A given polymer or polymer composite can break in a brittle, semi-brittle or ductile manner depending on the temperature and the glass transition of the material. Brittle fracture usually results in the shattering of the component. In this case, the elastic energy stored in the part is much higher than the energy dissipated in the fracture process. In large structures, this excess energy can assist a crack to propagate without external loading [98].

Ductile fracture usually occurs under stable crack propagation, with more plastic deformation. In such a situation, after crack initiation, the crack can only propagate with additional supply of energy by external loading [98, 99]. This type of failure is less catastrophic. Raising the temperature increases the internal energy of the material, resulting in easier molecular movement which lead to ductile fracture.

University of Cape Town



CHAPTER THREE

EXPERIMENTAL TECHNIQUES

3.1 INTRODUCTION

This research project is looking at the feasibility of developing an advanced hybrid thermoplastic composite for aerospace and automobile applications. In order to achieve the objectives of this research, several experiments and mechanical tests were conducted. The procedure followed in preparing the advanced hybrid thermoplastic composites and in conducting the experiments and tests are presented in this chapter. The advanced hybrid thermoplastic polymer composites specimens were subjected to tensile tests at different temperatures and different test speeds (strain rates). These were done to study the effect of temperature and test speed on the thermoplastic polymer and its composites. Three-point bend tests, Izod and Charpy impact tests were also performed to determine the flexural properties and impact properties of the polymer and its composites. Creep testing was also performed to determine the short term creep resistance of the materials. Scanning electron microscope (SEM) was used to study the morphology of the fractured surfaces of the test specimens to understand the failure mode and mechanisms.

3.2 MATERIALS USED

Two types of polyphenylene sulphide (PPS) compounds were used in this study. The PPS compounds were purchased from Chevron Phillips Chemicals (CPChem) in Belgium. The materials were PPS neat resin and 40% glass fibre filled PPS. They were designated as;

Ryton PR28	PPS neat resin.
Ryton R4200NA	PPS 40% glass fibre filled

Two types of hybrid composites were prepared from the PPS neat resin (Ryton PR28) with glass fibres and talc as reinforcements. The reinforcing materials suitable for



reinforcing PPS that were used to prepare the hybrid composites are short glass fibres of fibre length 3.5 – 4 mm and talc. They were supplied by Plastamid (Pty) Ltd and they were designated as;

PFG3540	Chopped strand glass fibre
Talc	Particle size 5 – 12 μm

Among the high temperature polymers, commonly used as matrix in the advanced thermoplastic composites industry, PPS has a moderate processing temperature profile and also with a relatively lower cost as indicated in the table below. It is based on this fact that PPS was selected as the matrix in this study.

Table 3.1: Morphology, processing temperature and relative cost of common thermoplastic matrices for advanced thermoplastic composites.

Matrix	Morphology	T _g (°C)	Processing Temperature (°C)	Relative Cost (\$) [100]
PEEK	SC*	143	360 – 400	\$97.00
PEI	A**	217	330 – 390	\$14.24
PPS	SC	90	293 – 343	\$14.09
PEKK	SC	156	340 – 382	\$94.72
PAI	A	293	304 – 370	\$60.63
PES	A	225	343 – 380	\$8.85

* Semi-crystalline and ** Amorphous.

3.3 PREPARATION OF THE HYBRID COMPOSITE

Two hybrid composites based on PPS neat resin, talc and short glass fibres were prepared for this study. Talc is mostly incorporated into thermoplastics with the purpose of reducing production cost. However, talc with a high aspect ratio has been found to show some reinforcing ability when added to thermoplastics. It also improves the toughness, stiffness and strength balance of the composite. Glass fibres strengthen the matrix when it is incorporated into it.

Prior to compounding and moulding of the composites, a thermogravimetric analysis was performed on the unfilled PPS powder to determine its thermal stability and the temperature at which it begins to degrade. The compounding and processing of the PPS composites into test specimens went through a couple of thermal cycles. Therefore knowledge of the thermal stability of the PPS matrix was very critical.

3.3.1 THERMO-GRAVIMETRIC ANALYSIS (TGA)

The thermal decomposition of the PPS resin was investigated by TGA in an inert atmosphere. The TGA testing was conducted at CSIR using a Metlar Toledo, SDTA851 thermo-gravimetric analyser shown in figure 3.1. Approximately 4.5mg of the neat PPS resin was placed in the sample holder of the instrument. It was then heated at 10°C/min to a maximum temperature of 600°C in a nitrogen environment. Three separate TGA runs were performed on the PPS powder. The data obtained was plotted as mass loss versus temperature from which the thermal stability and the temperature at which the PPS resin begins to degrade were determined.



Figure 3.1: The Metlar Toledo SDTA 851 thermogravimetric analyser used for the TGA tests of the PPS resin.

3.3.2 COMPOUNDING THE COMPOSITES

The processing and moulding of the hybrid composites, the 40% glass fibre filled PPS composite and the virgin PPS were done at the Polymers, Ceramics and Composites Unit at CSIR in Pretoria and at Plastamid (Pty) Ltd in Cape Town.

The PPS neat resin (Ryton PR28) was compounded with short glass fibres and talc into two different hybrid composites. Hybrid 1 consisted of 60% w/w PPS, 20% w/w glass fibres and 20% w/w talc and Hybrid 2 consisted of 60% w/w PPS, 30% w/w glass fibres and 10% w/w talc. The constituent materials were weighed in the above ratios and mixed thoroughly using a high speed Papenmeier mixer. The composite mixture was then compression moulded into sheets using Pasadena hydraulic compression moulders. The composite sheets obtained were granulated into granules and then injection moulded into test specimens using the Engel 80t and Demag D60 injection moulders. The Ryton PR28 powder was also compression moulded and granulated and then processed as above to obtain the unfilled PPS test specimens.

3.3.3 MOULDING THE TEST SPECIMENS

Prior to injection moulding, each of the compounds was dried at 135°C for 4 hours to eliminate any moisture that may be present. The moulding of the unfilled PPS and its composites into test specimens were done at the CSIR and Plastamid using the Engel 80t and Demag D60 injection moulders. A schematic of the recommended moulding parameters provided by the supplier of the PPS compounds is shown below [101].

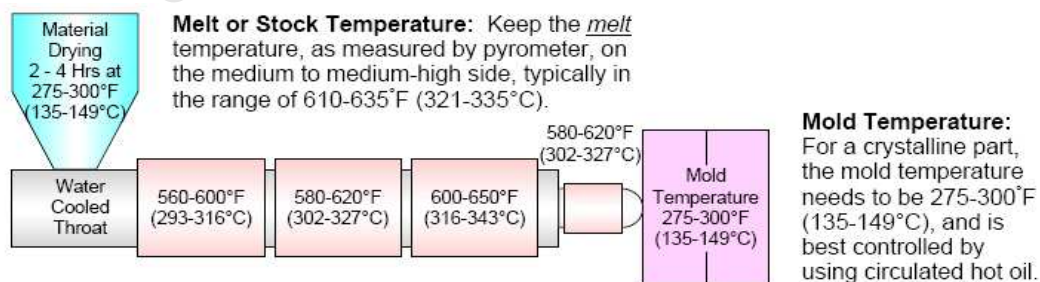


Figure 3.2: Temperature profile for injection moulding of the PPS compounds for maximum crystallinity [101].

The actual moulding parameters for the processing of the PPS and its composites are listed in the table below.

Table 3.2: Processing parameters for moulding the PPS and its composites.

Processing Parameter	Set Value
Rear Temperature	305°C
Middle Temperature	315°C
Front Temperature	325°C
Nozzle Temperature	320°C
Mould Temperature	120°C
Injection Fill Pressure	28.0 bar
Injection Holding Pressure	Max
Back Pressure	7.0 bar
Injection Fill Time	5 s
Injection Holding Time	15 s
Screw Speed	100 rpm
Injection Rate	Medium

To achieve ultimate crystallinity, the moulded specimens were annealed at 210°C for 4 hours since the recommended mould temperature could not be attained during the moulding process. The specimens were then subjected to mechanical testing to determine the tensile, flexural, impact and creep properties of the composite materials.

3.4 MECHANICAL TESTING

Several mechanical tests were conducted on the PPS composites to determine their mechanical properties. The polymer composites were subjected to tensile testing at room and high temperatures, flexural testing, Izod and Charpy impact tests and creep testing. The room temperature (23°C) tensile tests, the three-point bend tests and the creep tests were conducted at the Centre for Materials Engineering, University of Cape Town. The high temperature tensile tests were done at the SA Naval Base Materials Laboratory in Simonstown. The Izod and Charpy impact tests were

conducted at (Plastamid) Pty Ltd in Elsies River, Cape Town. The ASTM standards were followed in conducting all the mechanical tests except for the impact tests where ISO standards were followed. The fractured surfaces of the specimens tested were examined under the Scanning Electron Microscope to study the failure mode and failure mechanisms of the advance thermoplastic composites.

3.4.1 TENSILE TESTING

Tensile testing of the materials was done by pulling the tensile specimen in tension to failure at room temperature, 50°C, 100°C and 150°C. At each temperature the specimen were tested at 2mm/min, 50mm/min, 100mm/min, 200mm/min, 250mm/min and 500mm/min. These test speeds corresponds to strain rates of 4.2×10^{-4} ; 10^{-2} ; 2×10^{-2} ; 4.2×10^{-2} ; 5×10^{-2} and 10^{-1} s^{-1} , respectively. This was done to study the effect of temperature and strain rate on the PPS polymer and its composites. All the tensile tests were performed according to ASTM D638 and D3039. The data obtained from all the tests were presented in stress-strain curves and bar charts form which the tensile properties were obtained.

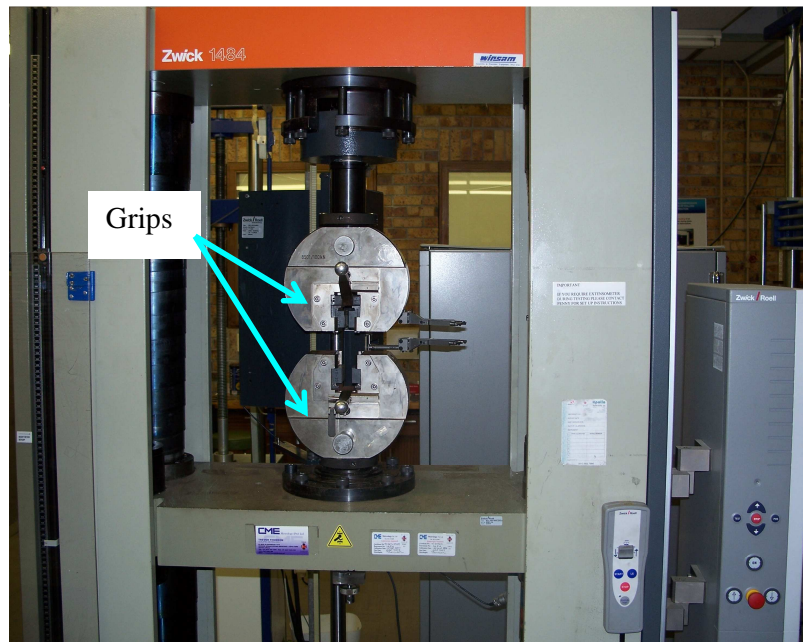


Figure 3.3: A photograph of the Zwick 1484 universal tensile tester used for both tensile and three-point bend tests.

The room temperature tensile tests were performed on the Zwick 1484 universal tensile tester. Prior to testing, the specimen dimensions were entered into the computer that is attached to the Zwick universal tensile tester. The dimensions were; 80mm × 10mm × 4mm which correspond to gauge length × width × thickness. At least five specimens were tested for each test speed to ensure repeatability of the testing. The figure 3.3 above show the photograph of the Zwick 1484 universal tensile tester used.

The high temperature tensile tests were performed on the Hounsfield tensile tester at the SA Naval Materials Laboratory in Simonstown. At least five specimens were tested for each of the test speeds at a particular temperature. The figure 3.4 below shows the Hounsfield tensile tester and the furnace for the high temperature tensile testing.

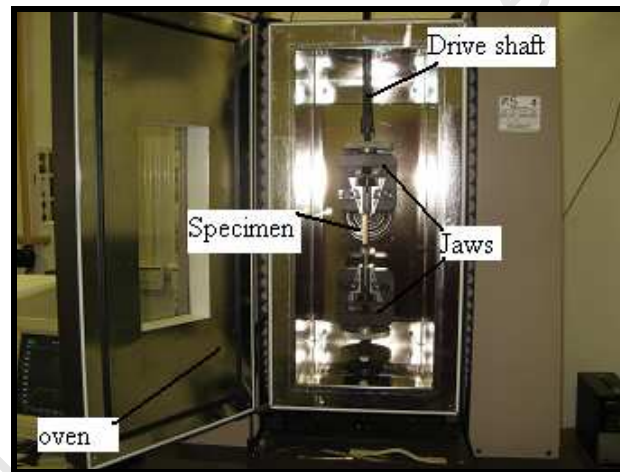


Figure 3.4: The Hounsfield tensile tester showing the main components.

The temperature inside the furnace was monitored by a temperature controller shown in the figure 3.5 below.



Figure 3.5: The temperature controller connected to the Hounsfield tensile tester.

3.4.2 THREE-POINT BEND TESTING

Three-point bend testing was conducted on some specimens of the PPS and its composites. The three-point bend test was conducted on the Zwick 1484 universal tensile tester by replacing the tensile grips with appropriate three-point bend test supports shown below.

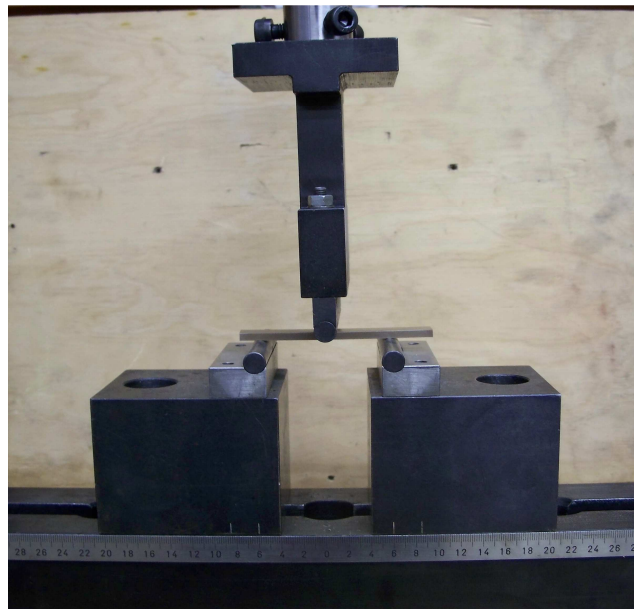


Figure 3.6: Three-point bend test set up on the Zwick 1484 Universal tensile tester.

This test was done to determine the flexural properties of the materials under investigation. The test specimens were machined from the gauge length section of the tensile specimen. The dimensions of the specimen were 80mm x 10mm x 40mm, corresponding to the length, width and thickness respectively. The three-point bend test was performed according to the ASTM standard D790 which is the standard test method for three-point bend test of unreinforced and reinforced plastics. The test was done by placing the specimen on a support of span 60mm. It was then subjected to flexural loading at the central point of the specimen between the two lower stationary supports. At least ten specimens were tested for each material type at a test speed of 5mm/min. The load-deflection data that was obtained were analysed and presented in stress-deflection curves from which the flexural properties were obtained

3.4.3 IMPACT TESTING

Impact tests were performed on the PPS and its composites specimens to determine their resistance to sudden impact. Izod and Charpy impact tests were performed on the Zwick/Roell pendulum impact tester shown in the figure 3.7 below.



Figure 3.7: The Zwick/Roell impact tester for Izod and Charpy impact testing.

The Izod impact tests were performed according to ISO standard 180. The Izod impact test was used to determine the impact energy required to cause the specimen to fail upon impact at the free end while the other end is clamped as shown below. A 2.75 J pendulum was released from an angle of 124° to strike the free end of the specimen. The impact energy in J and impact resistance in kJ/m^2 was recorded.

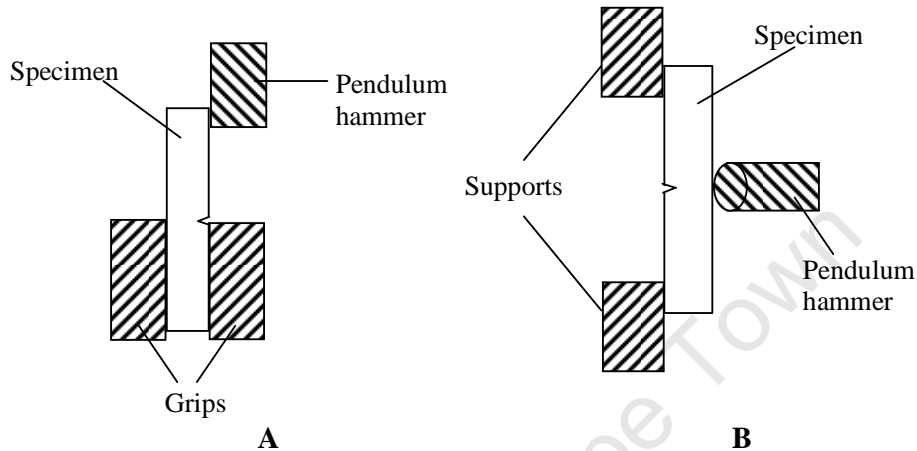


Figure 3.8: A schematic representation of Izod impact (A) and Charpy impact (B) test configuration

The Charpy impact test was also performed according to ISO standard 179. It was also used to determine the amount of energy needed to cause the specimen to fracture upon sudden impact. The Charpy specimen was placed horizontally as shown above. A 4.0 J pendulum was released from an angle of 160° to strike the specimen. The dimensions of the specimens used for both Izod and Charpy impact tests are shown in the table below.

Table 3.3: Specimen dimensions for the Izod and Charpy impact tests

Dimensions	Izod and Charpy Test
Length (mm)	80
Width (mm)	10
Thickness (mm)	4
Notch length (mm)	2
Notch radius (mm)	0.25
Notch angle ($^\circ$)	30

The Izod and Charpy impact tests were performed on both notched and un-notched specimens to study the effect of notching on the impact strength of the PPS and its composites.

3.4.4 CREEP TESTING

It is a well known fact that high performance polymer matrix composites exhibit a time-dependent degradation in modulus and strength as a result of their viscoelastic behaviour. Creep test is aimed at measuring dimensional changes at constant temperature and constant load. It provides useful information for the long term applications which are strain limited. One of the factors that must be considered when selecting a material for long term load bearing application is the materials ability to resist distortion during service. It is therefore important to determine the creep resistance of a material to be used for such applications.

A short term (five weeks) creep testing was performed on each of the material type using a custom built in-house creep rig, engineered for testing polymers and polymer composites (see figure 3.9). The purpose of this part of the study is not to undertake an in-depth study of the creep properties for PPS and its composites under various conditions, but rather to rank the materials according to their creep resistance. If the data is to be used for design purposes, multiple tests will have to be done and statistically analysed. Two tensile specimens were mounted inside the furnace of the creep rig under a constant load of 0.592 MPa each and at a temperature of 100°C. A linear variable differential transducer (LVDT) was used to measure the change in length which was recorded as change in voltage from the voltmeter connected to the LVDT. The voltage readings were converted into extension (in mm) using the LVDT calibration equation;

$$\Delta l = \frac{\Delta V \times 10^3}{97.32} \quad 3.1$$

From this the creep strain was calculated as;

$$\epsilon = \frac{\Delta l}{l} \quad 3.2$$

The results obtain was plotted as a strain-time curve.

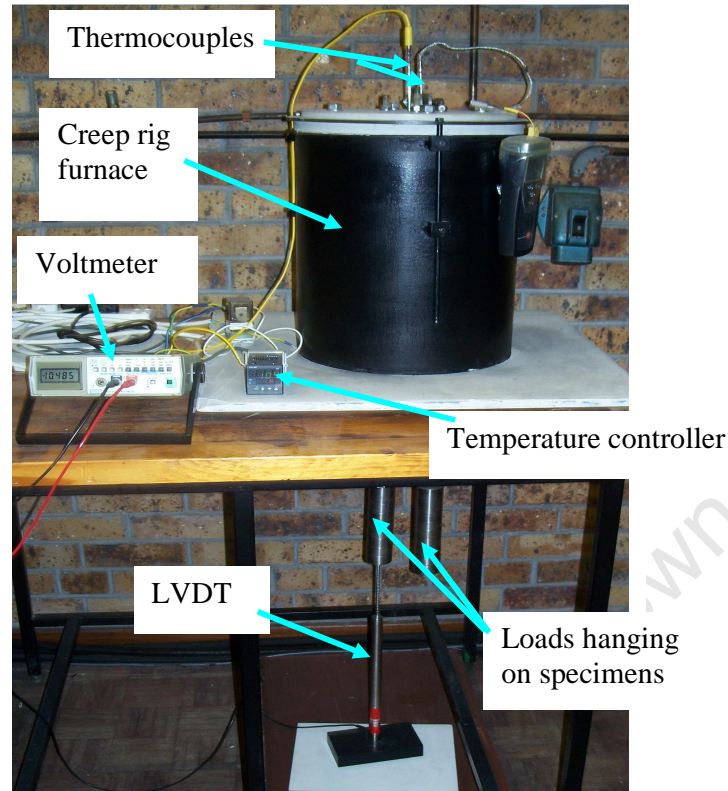


Figure 3.9: A set up of the in-house creep rig built for polymer and polymer composites.

3.5 SCANNING ELECTRON MICROSCOPY (SEM)

Examination of the fractured surfaces of the tensile, impact and flexural test specimens was carried out using the Leo Stereoscan 440 scanning electron microscope. It was operating at an acceleration voltage of 10 kV. This was done to study the morphology of the fractured surfaces in order to understand the failure mode and failure mechanisms of the PPS and its composite materials. In preparing the samples for the analysis, the specimens were mounted on aluminium stubs. They were then sputter coated with gold and palladium mixture to ensure electrical conductivity before being examined in the SEM and to avoid electrical charge accumulation during the examination. Micrographs of the fractured surfaces were used to study the failure mode and failure mechanisms and the nature of the fibre-matrix interfacial bond in the PPS composites.

CHAPTER FOUR

EXPERIMENTAL RESULTS

4.1 INTRODUCTION

From the various techniques employed in developing the hybrid thermoplastic composites based on PPS resin as matrix and the experimentations to determine their mechanical properties, the following results have been obtained. The results obtained for all the mechanical tests are presented along with the fractographic analysis of the fractured surfaces of the tensile, impact and flexural test specimens. The results obtained for the hybrid composites are presented along with that of the unfilled PPS and the 40% glass fibre reinforced PPS composite.

4.2 THERMO-GRAVIMETRIC ANALYSIS (TGA)

TGA is a thermal analysis technique used to measure changes in the mass (weight) of a sample as a function of temperature or time. It is commonly used to determine among others, polymer degradation temperature, absorbed moisture content and the amount of non-combustible fillers in a polymer composite. TGA tests were performed on the neat PPS resin to determine its degradation temperature before compounding the hybrid composites and processing them into the various test specimens. The results obtained are presented in curves of mass loss versus temperature in figure 4.1 below. It can be observed from the graphs in figure 4.1 that in all three TGA test runs for the PPS resin, the material remained thermally stable until about 470°C when it started to degrade. This is evident from the typical plateau of the TGA profile at a fairly constant weight level until 470°C when the polymer starts to degrade.

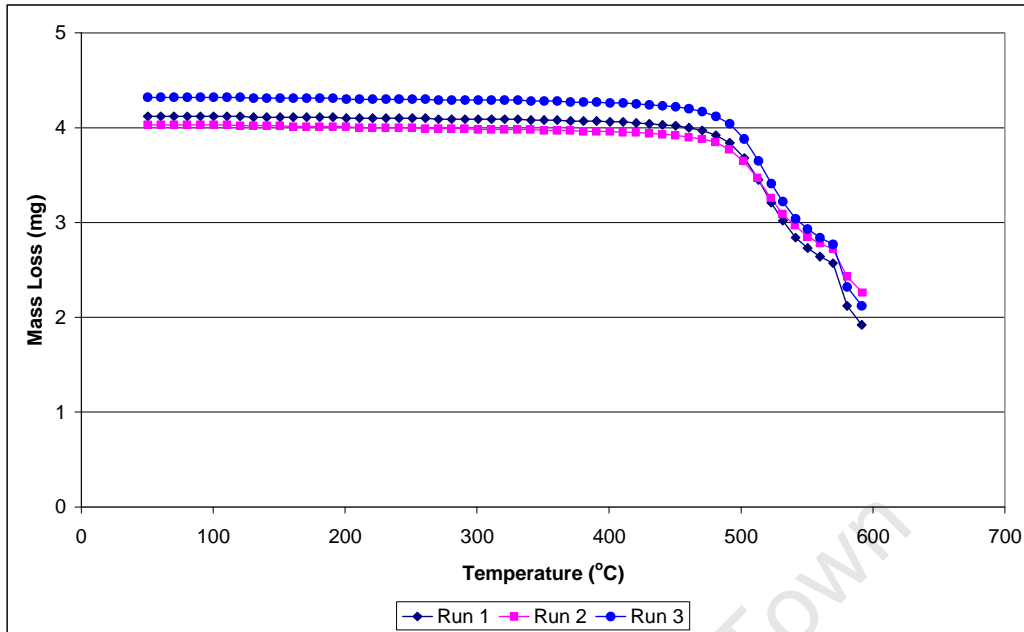


Figure 4.1: TGA results showing the thermal degradation of unfilled PPS resin.

Although TGA is an analytical technique that can be used to determine a host of other properties of a material, in this particular instance, the emphasis was to determine the polymer degradation temperature of the PPS resin which was used as a matrix in developing the hybrid composites. The information on the thermal stability of the PPS matrix was a guiding principle during the processing and moulding of the PPS and the hybrid composites as it went through a couple of thermal cycles.

4.3 TENSILE TESTING

Tensile tests were performed at different temperatures and test speeds (strain rates) on the unfilled PPS, Hybrid 1 (622 PPS composite), Hybrid 2 (631 PPS composite) and the 40% glass fibre reinforced PPS (40% GF PPS) as mentioned in section 3.4.1. The test temperature varied from room temperature to 150°C. Corresponding to each temperature, the test speeds of between 2mm/min and 500mm/min were employed to investigate the strain rate effects. In order to ensure repeatability at each test speed, five specimens were tested for each test condition. The results obtained are presented in stress-strain relationships and bar graphs.

4.3.1 EFFECT OF TEST SPEED (STRAIN RATE) ON TENSILE TESTING

The behaviour of each of the PPS materials under tension was studied by performing tensile testing at six different test speeds (strain rates). The results obtained for each of the materials are presented in this section.

4.3.1.1 Effect of Test Speed on the Tensile Properties of Unfilled PPS

The effect of test speed (strain rate) on the tensile properties of the unfilled PPS at room temperature is given in figures 4.2 – 4.3. The effect of temperature and test speed on the UTS of the unfilled PPS is also presented in figure 4.5. The averages of the tensile properties with their standard deviations are also presented in table 4.1. It can be observed from figure 4.2 that the polymer exhibits a brittle failure because there was no necking and plastic deformation during tensile testing. The percentage strain to failure of below 3% (see table 4.1) indicate the brittleness of the material. The brittleness of the material was observed at all the test temperatures except for 150°C where it exhibited necking and plastic deformation during the tensile testing. From figure 4.3 and table 4.1 it can be seen that there is a marginal decrease in tensile strength along with percentage strain to failure of the unfilled PPS as the test speed (strain rate) increases. This behaviour was observed for tensile tests at room temperature while at 50°C the polymer is not that sensitive to test speed. However, at 100°C the tensile strength appeared to increase with test speed (see figure 4.5) although at 150°C the polymer become insensitive to test speed changes. Figure 4.5 summarises how test speed and temperature affect the tensile properties of the unfilled PPS. Figure 4.4 indicates the effect of test speed on the elastic modulus of the unfilled PPS. It can be seen from the graph that test speed has little influence on the elastic properties of the unfilled PPS, although the elastic modulus tend to increase with test speed from 2mm/min to 100mm/min after which it becomes somewhat constant with increasing test speed.

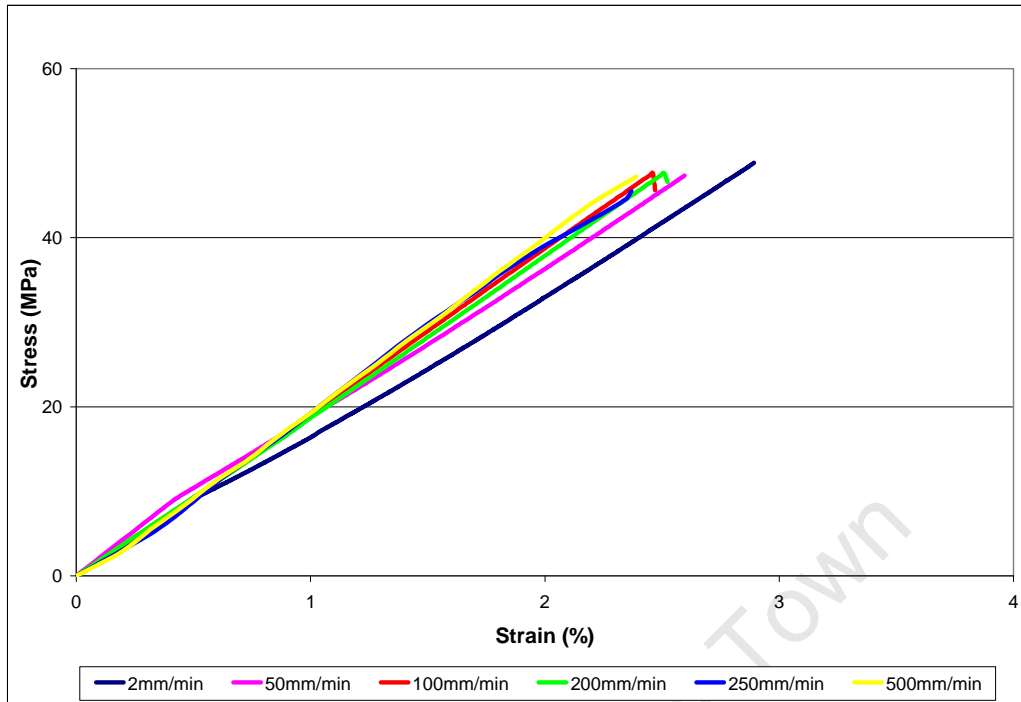


Figure 4.2: Stress-strain curves for unfilled PPS at room temperature and different test speeds.

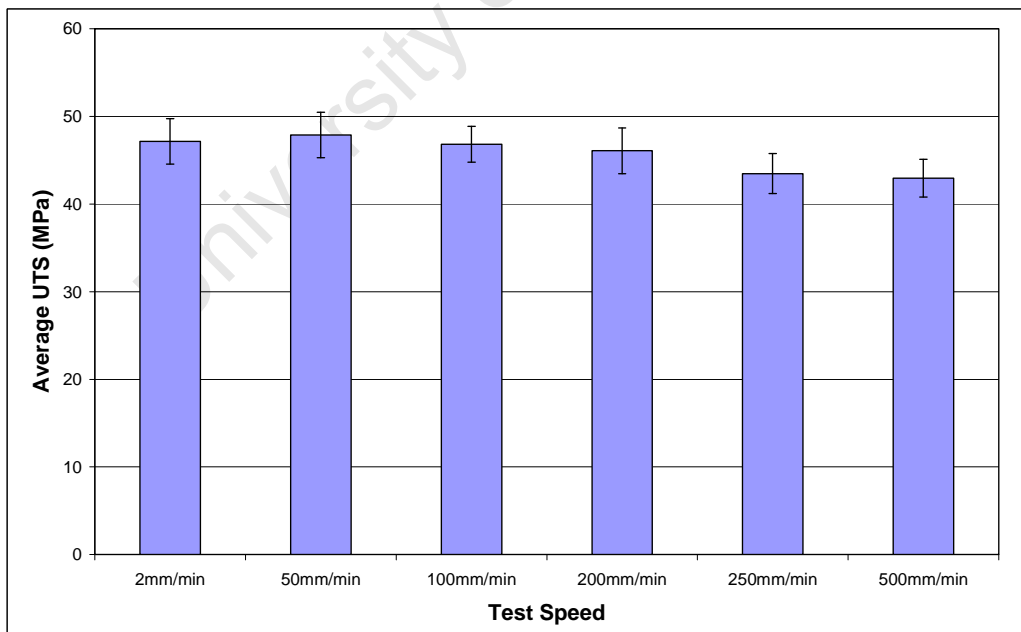


Figure 4.3: Average UTS against test speed for unfilled PPS at room temperature.

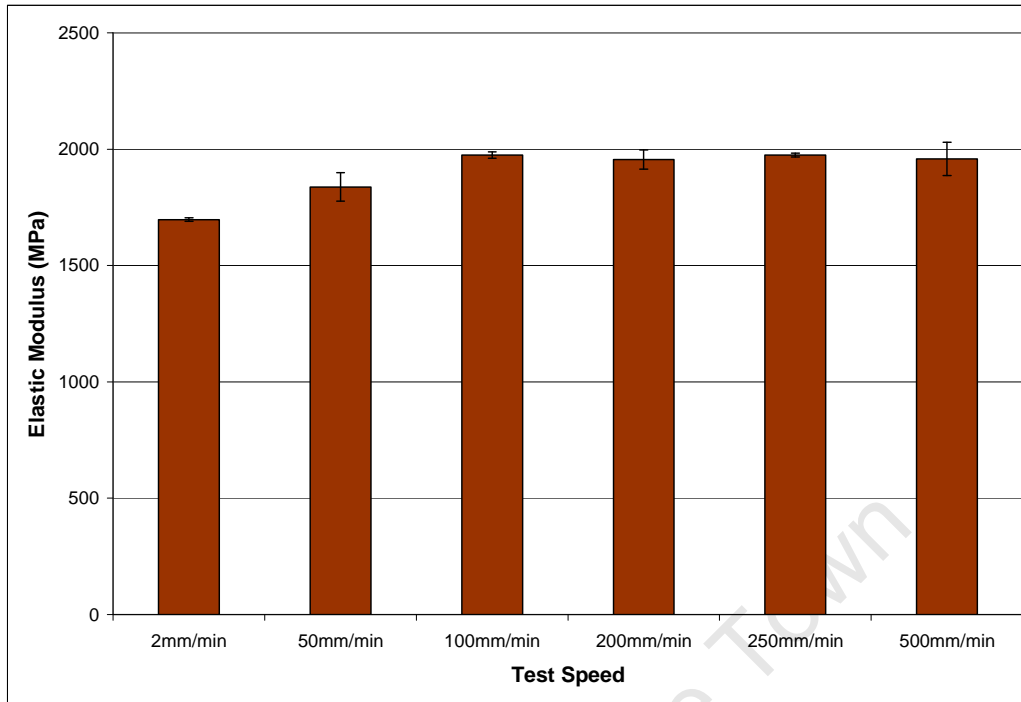


Figure 4.4: Elastic modulus against test speed for unfilled PPS at room temperature.

Table 4.1: Average tensile properties of unfilled PPS at room temperature and different test speed.

Test Speed	UTS (MPa)	Modulus (MPa)	Strain (%)
2mm/min	47.2 ± 2.6	1697 ± 7	2.8 ± 0.2
50mm/min	47.9 ± 2.6	1837 ± 61	2.7 ± 0.2
100mm/min	46.8 ± 2.0	1975 ± 13	2.4 ± 0.3
200mm/min	46.1 ± 3.0	1955 ± 41	2.4 ± 0.2
250mm/min	43.5 ± 2.3	1974 ± 8	2.3 ± 0.2
500mm/min	42.9 ± 2.2	1958 ± 72	2.2 ± 0.3

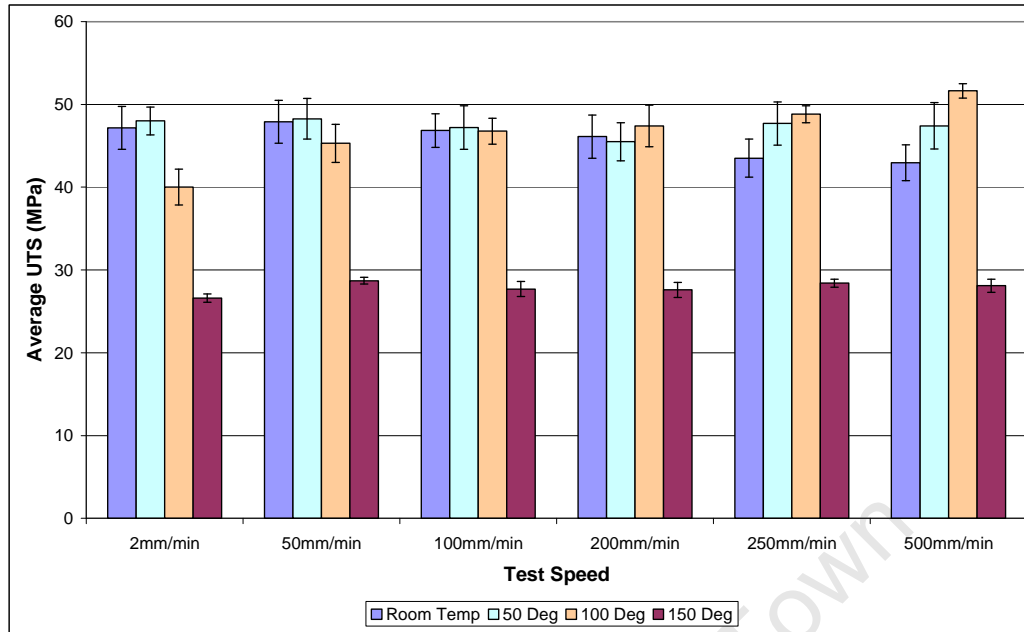


Figure 4.5: Average UTS versus test speed at different temperatures for unfilled PPS.

4.3.1.2 Effect of Test Speed on the Tensile Properties of the 622 PPS Hybrids

The room temperature tensile properties of the 622 PPS hybrid composite at different test speeds are presented in figures 4.6 – 4.8. The effect of temperature and test speed changes on the UTS of the 622 PPS hybrid is also presented in figure 4.9. Table 4.2 contains the averages of the tensile properties at the different test speeds. From the stress-strain relationship of the 622 PPS hybrid composite (figure 4.6), this hybrid composite exhibited a brittle fracture during tensile testing just like the unfilled PPS. However, unlike the unfilled PPS, the percentage strain to failure of the 622 PPS hybrid composite has increased by about 2% from the addition of glass fibres and talc. It is apparent from figures 4.6 and 4.7 that the UTS of this PPS hybrid composite appears to be insensitive to test speed changes, even at different temperature as in figure 4.9. The modulus of elasticity of this hybrid composite appears to be unaffected by test speed changes (see figure 4.9 and table 4.2). With the addition of 20% w/w glass fibre and 20%w/w talc to the PPS matrix, the strength of the composite increased to about three fold that of the unfilled polymer, while contrary to what was expected the ductility also increased.

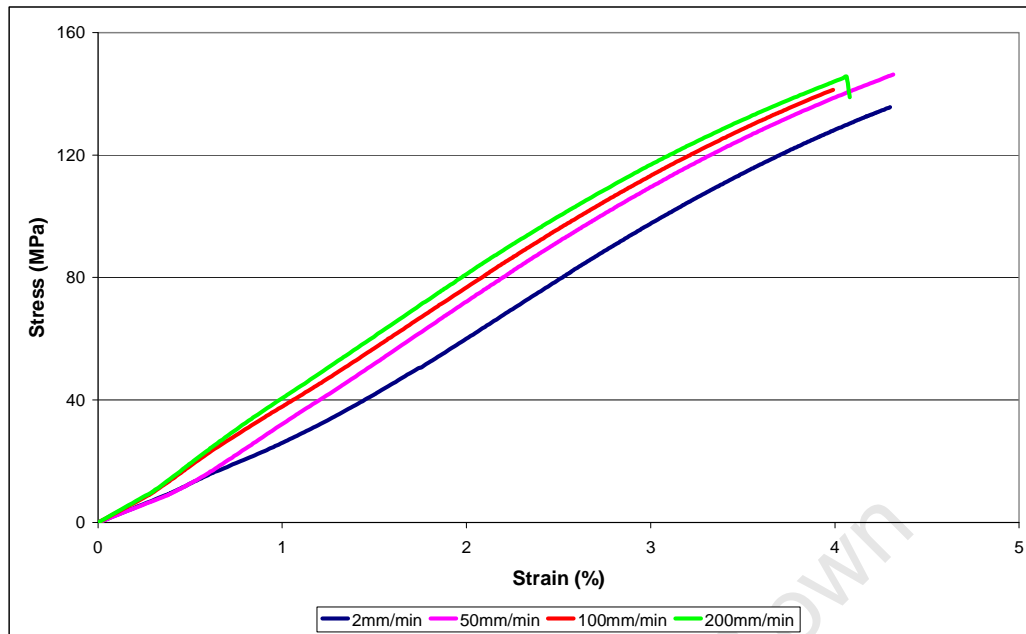


Figure 4.6: Stress-strain curves for 622 PPS hybrid composite at room temperature and different test speeds.

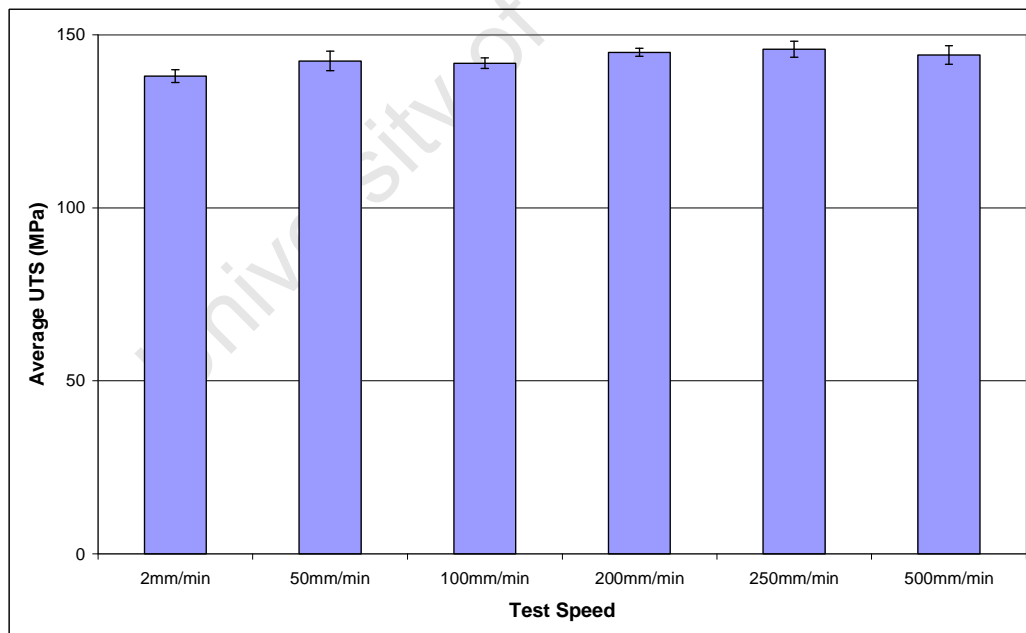


Figure 4.7: Average UTS versus test speed for the 622 PPS hybrid composite at room temperature.

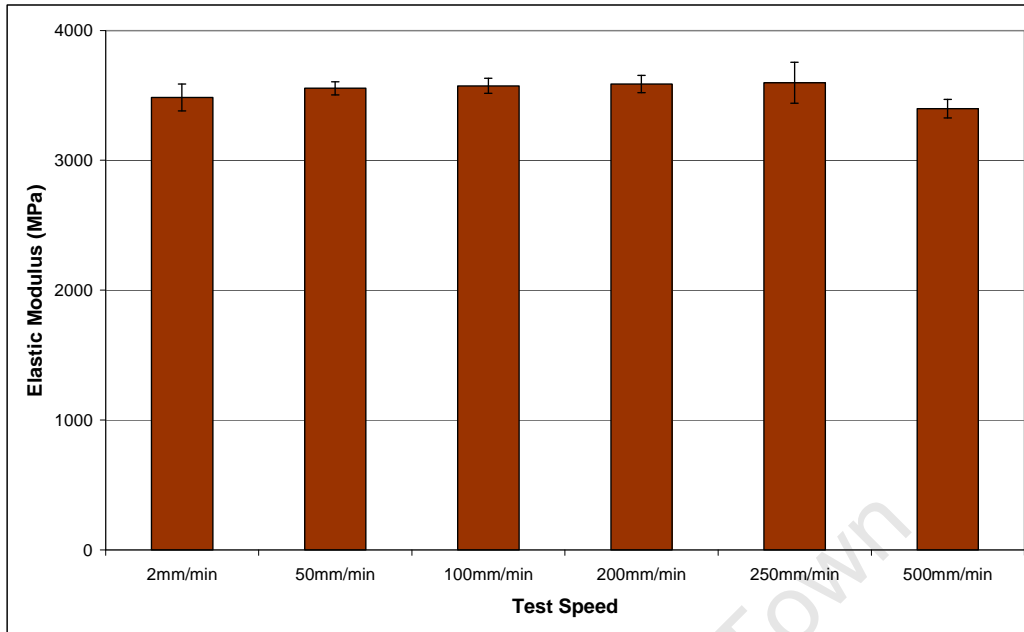


Figure 4.8: Elastic modulus of 622 PPS hybrid versus test speed at room temperature.

Table 4.2: The average tensile properties of the 622 PPS hybrid composite at room temperature and different test speed.

Test Speed	UTS (MPa)	Modulus (MPa)	Strain (%)
2mm/min	138.0 ± 1.9	3484 ± 105	4.3 ± 0.3
50mm/min	142.4 ± 2.8	3555 ± 50	4.4 ± 0.1
100mm/min	141.8 ± 1.5	3573 ± 58	4.4 ± 0.3
200mm/min	144.9 ± 1.2	3587 ± 67	4.4 ± 0.3
250mm/min	145.8 ± 2.3	3597 ± 158	5.0 ± 0.3
500mm/min	144.2 ± 2.7	3398 ± 72	4.9 ± 0.2

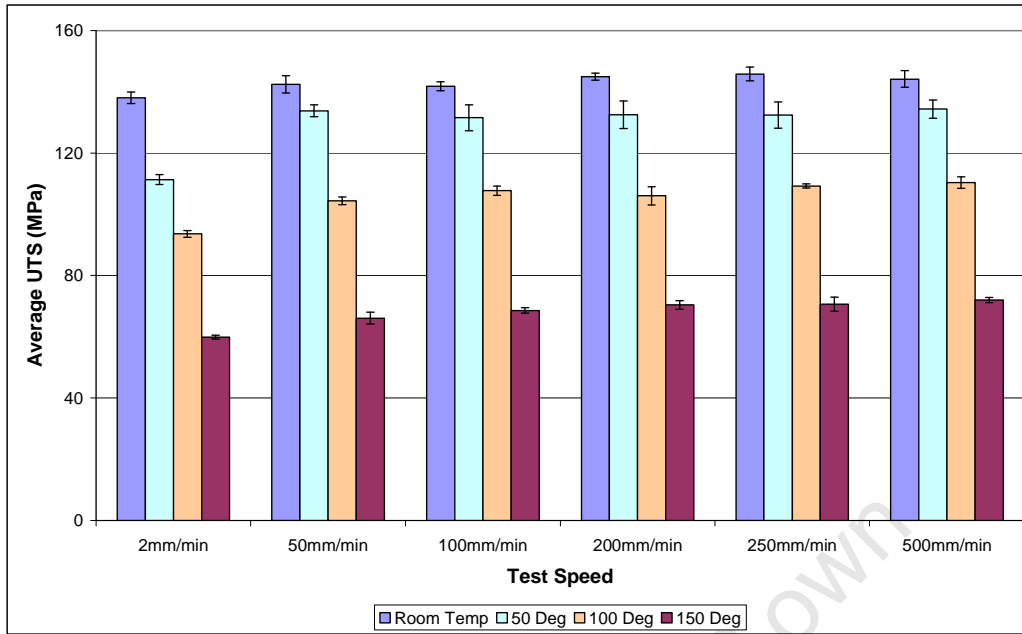


Figure 4.9: Average UTS versus test speed at different temperatures for 622 PPS hybrid composite.

4.3.1.3 Effect of Test Speed on the Tensile Properties of the 631 PPS Hybrids

Upon performing tensile tests at different test speeds for the 631 PPS hybrid composite, the tensile stress-strain curves are presented in figure 4.10 along with a bar graph of the average UTS at different test speeds in figure 4.11. The elastic moduli at different test speeds are also presented in a bar graph in figure 4.12 and the averages of the tensile properties tabulated in table 4.3. The effect of temperature and test speed changes on the UTS of the 631 PPS hybrid is also presented in figure 4.13. Inspection of figures 4.10, 4.11 and 4.13 shows that there is a small increase in strength as test speed increases across the temperatures tested. Like the unfilled PPS and the 622 PPS hybrid composite, this hybrid composite also exhibited brittle failure during tensile testing. It can be seen from figure 4.12 and table 4.3 that the modulus of elasticity seems to be increasing with test speed up until 200mm/min after which there appears to be a drop in the modulus. The percentage strain to failure for this particular hybrid composite is very similar to that of the 622 PPS hybrid composite. It is important to note that the addition of 30% w/w glass fibre and 10% w/w talc resulted in an increase in strength of the composite to over three times that of the unfilled PPS polymer.

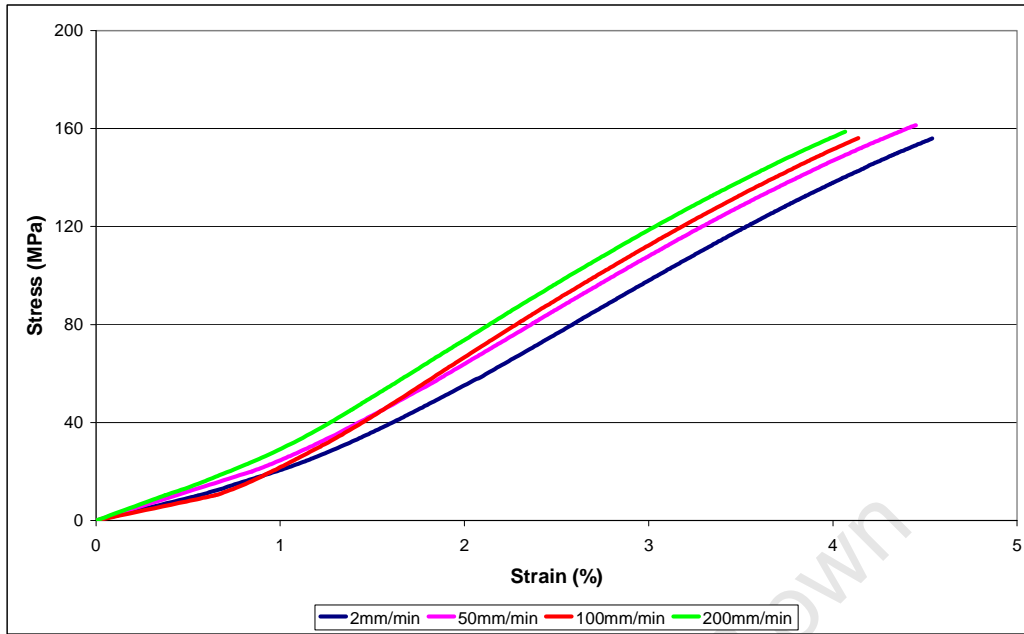


Figure 4.10: The stress-strain curves for 631 PPS hybrid composite at room temperature and different test speeds.

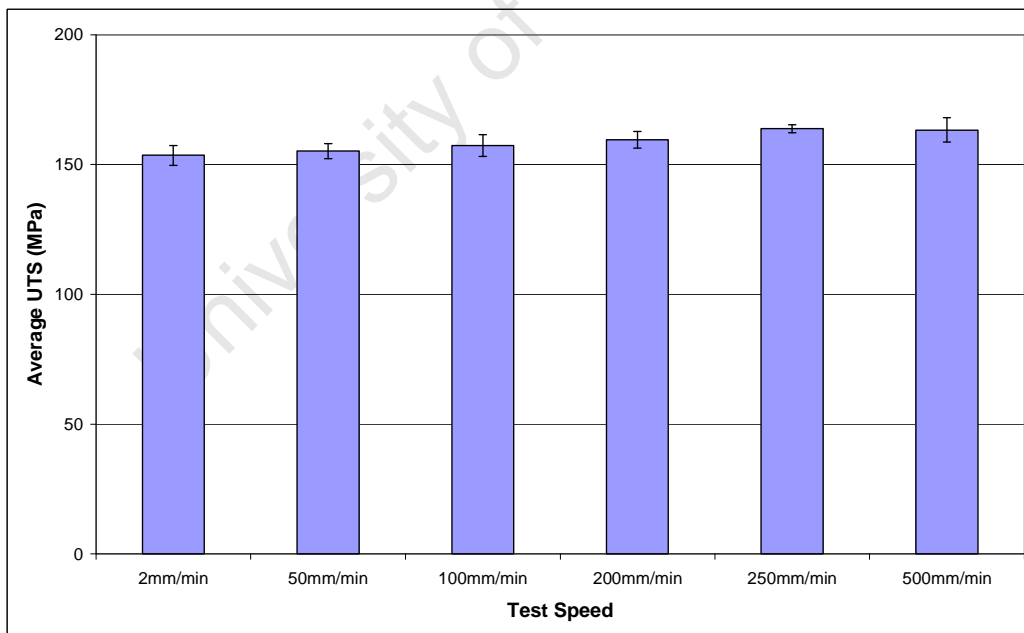


Figure 4.11: The average UTS versus test speed for 631 PPS hybrid composite at room temperature.

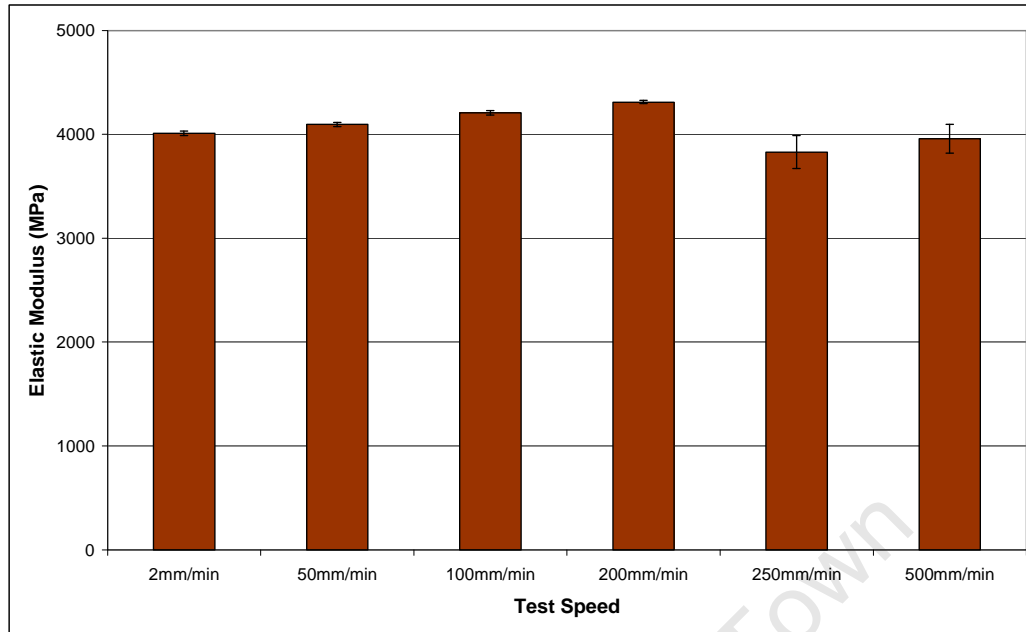


Figure 4.12: Elastic modulus versus test speed for 631 PPS hybrid at room temperature.

Table 4.3: Average tensile properties of 631 PPS hybrid at room temperature and different test speed.

Test Speed	UTS (MPa)	Modulus (MPa)	Strain (%)
2mm/min	153.5 ± 3.8	4011 ± 22	4.3 ± 0.4
50mm/min	155.2 ± 2.9	4094 ± 21	4.3 ± 0.2
100mm/min	157.3 ± 4.2	4207 ± 23	4.2 ± 0.2
200mm/min	159.5 ± 3.1	4315 ± 15	4.4 ± 0.2
250mm/min	163.8 ± 1.5	3829 ± 158	4.8 ± 0.3
500mm/min	163.3 ± 4.7	3958 ± 138	4.8 ± 0.3

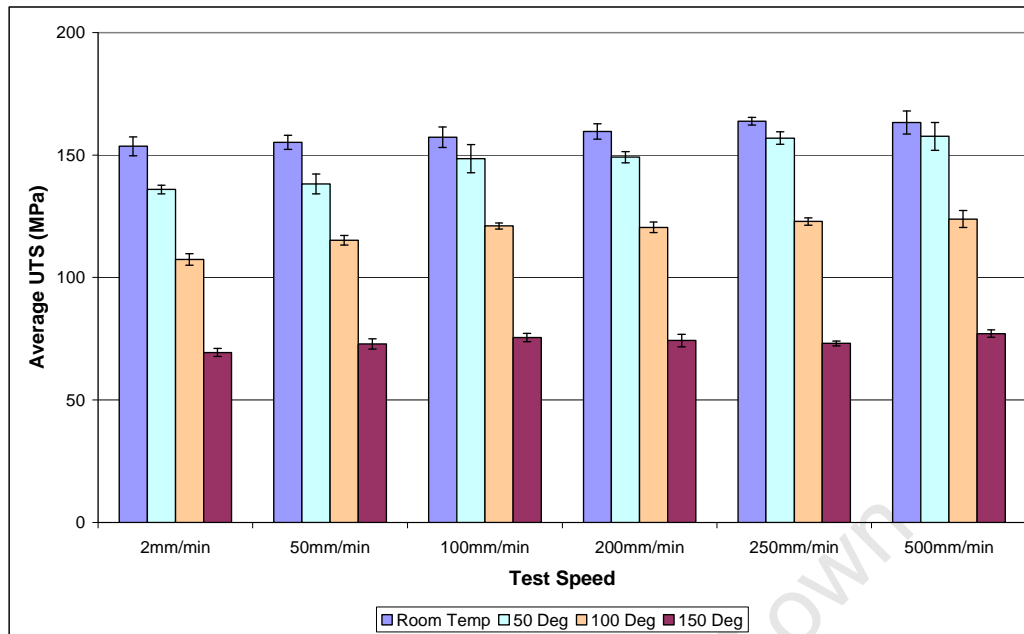


Figure 4.13: Average UTS versus test speed at different temperatures for 631 PPS hybrid composite.

4.3.1.4 Effect of Test Speed on the Tensile Properties of 40% GF PPS

The effect of test speed on the tensile properties at room temperature for 40% GF PPS composite are presented in figures 4.14 to 4.16 and table 4.4 below. The effect of temperature and test speed on the UTS of the 40% GF PPS composite is also presented in figure 4.17. Just as the unfilled PPS and the PPS hybrid composites, the 40% GF PPS also experienced brittle fracture during tensile testing. This is depicted in the stress-strain relationship in figures 4.14. It is apparent from figure 4.15 and table 4.4 that for the 40% GF PPS, the tensile strength tend to increase with increasing test speed. This is an indication that this composite is sensitive to test speed. The ductility of the 40% GF PPS is comparable with those of the PPS hybrid composites mentioned in sections 4.3.1.2 and 4.3.1.3 above. The elastic modulus of this composite tend to increase marginally with test speed up to 200mm/min and then drops a little at the higher test speeds as seen in figure 4.16 and table 4.4. The effect of test speed on the tensile properties of the 40% GF PPS at different temperatures (see figure 4.17) appears to give the same effect as those at room temperature. From figure 4.17 tensile strength tend to increase with increasing test speed at all the test

temperatures.



Figure 4.14: Stress-strain curves for 40% GF PPS composite at room temperature and different test speeds.

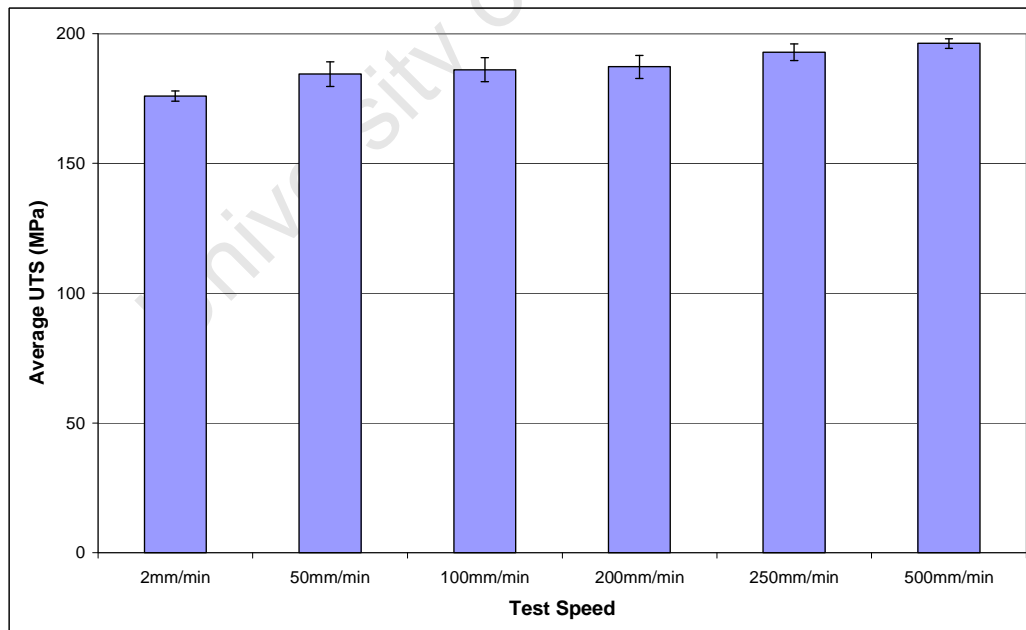


Figure 4.15: Average UTS versus test speed for 40% GF PPS composite at room temperature.

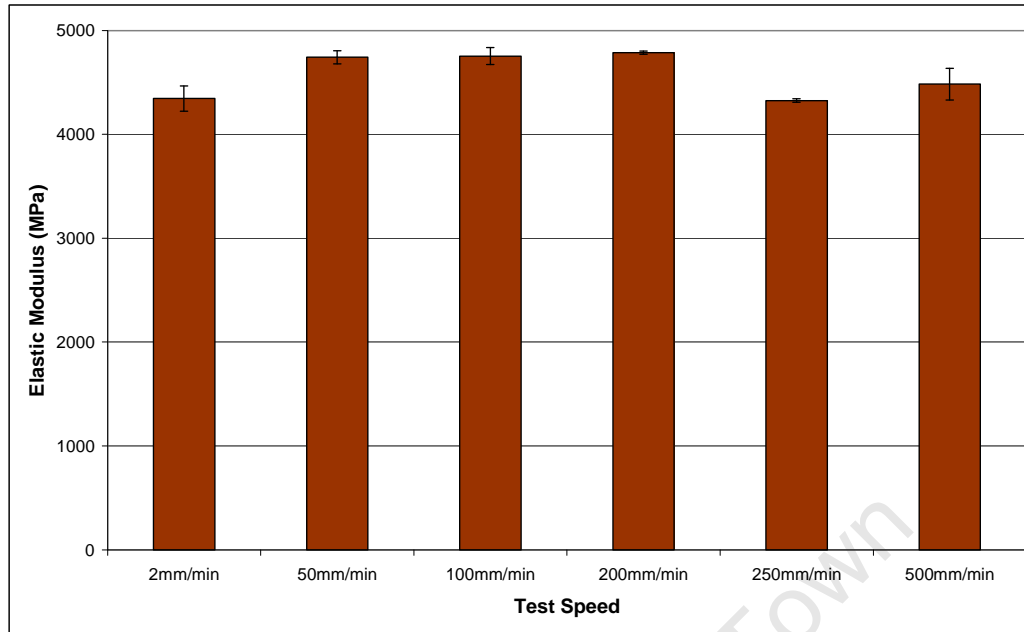


Figure 4.16: Elastic modulus at different test speed for the 40% GF PPS at room temperature.

Table 4.4: Average tensile properties of 40% GF PPS at room temperature and different test speed.

Test Speed	UTS (MPa)	Modulus (MPa)	Strain (%)
2mm/min	176.0 ± 1.2	4344 ± 122	4.2 ± 0.3
50mm/min	184.4 ± 4.7	4742 ± 64	4.2 ± 0.1
100mm/min	186.0 ± 4.6	4754 ± 82	4.3 ± 0.3
200mm/min	187.2 ± 4.4	4786 ± 15	4.1 ± 0.1
250mm/min	192.8 ± 3.2	4325 ± 16	4.4 ± 0.6
500mm/min	196.2 ± 1.9	4484 ± 152	4.4 ± 0.3

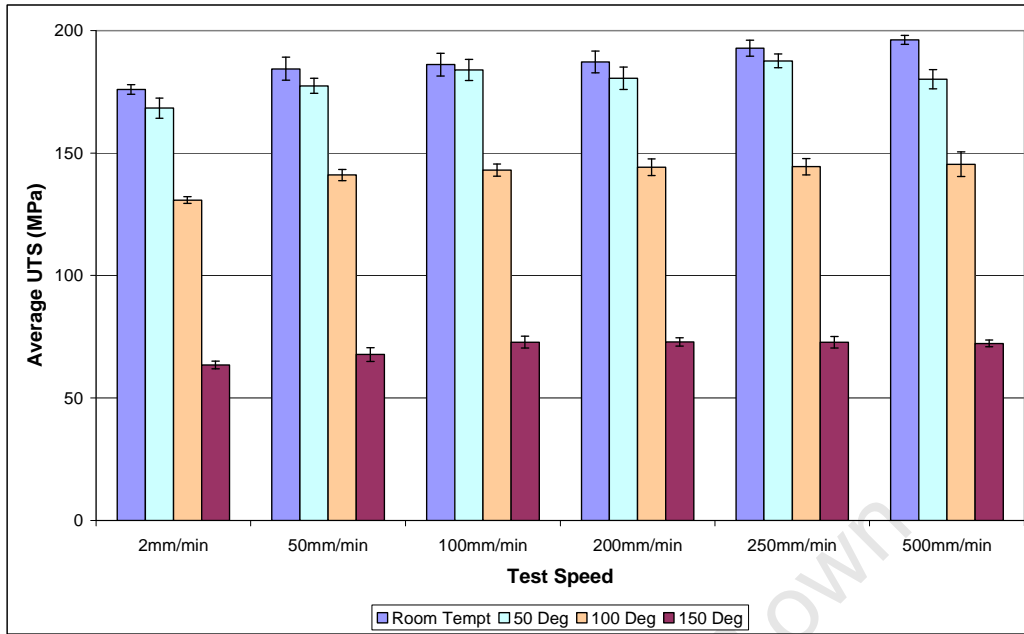


Figure 4.17: Average UTS versus test speed at different temperatures for 40% GF PPS composite.

4.3.2 EFFECT OF TEMPERATURE ON TENSILE TESTING

The effect of temperature on the tensile properties of the PPS polymer and its composites has been studied and the results obtained are presented in this section. As mentioned earlier the tensile tests were conducted at different test speeds for temperatures ranging from room temperature to 150°C for each of the materials. The results are presented in the form of stress-strain curves and bar graphs of the UTS versus temperature at the different test speeds. This gives a clear representation of how temperature and test speed affect the UTS of the PPS materials.

4.3.2.1 Temperature Effect on the Tensile Properties of Unfilled PPS

The stress-strain relationship of the unfilled PPS at 2mm/m and different temperatures are shown in figure 4.18 below. Figure 4.19 is a representation of the average UTS against temperature at different test speeds. Inspection of figure 4.18 shows that the tensile strength decreases with increasing temperature; however, the

decrease in strength of the material from room temperature to 50°C is negligible and this may be because 50°C is still below the T_g (~90°C) of the polymer. The strain to failure increased with temperature. At 100°C, the material experienced a brittle failure with some plastic yielding leading to a considerable loss in strength. This was observed at all the test speeds. But at 150°C the unfilled PPS material went through plastic yielding and necking with a considerable amount of plastic deformation until 62.4% strain when the test was terminated before failure.

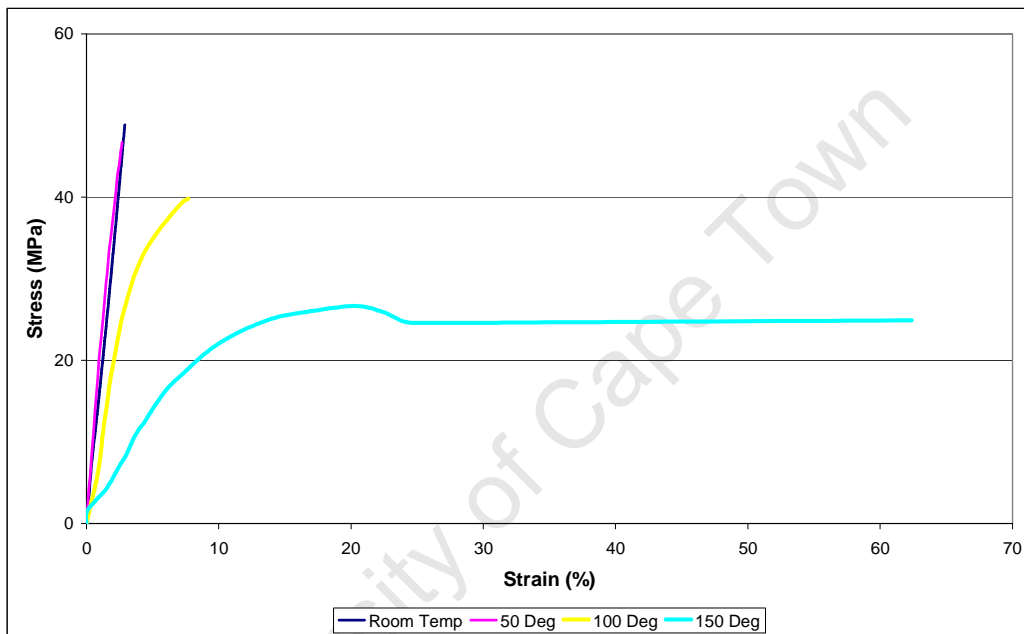


Figure 4.18: The stress-strain curves for unfilled PPS at 2mm/min and different test temperatures.

The results in figure 4.19 indicate the general response of the unfilled PPS to test speed. It can be seen that at room temperature, the average UTS of the unfilled PPS tend to decrease marginally with increasing test speed as mentioned in section 4.3.1.1 above. However, at 100°C the average UTS tend to increase with increasing test speed while at 50°C and 150°C it is not sensitive to test speed.

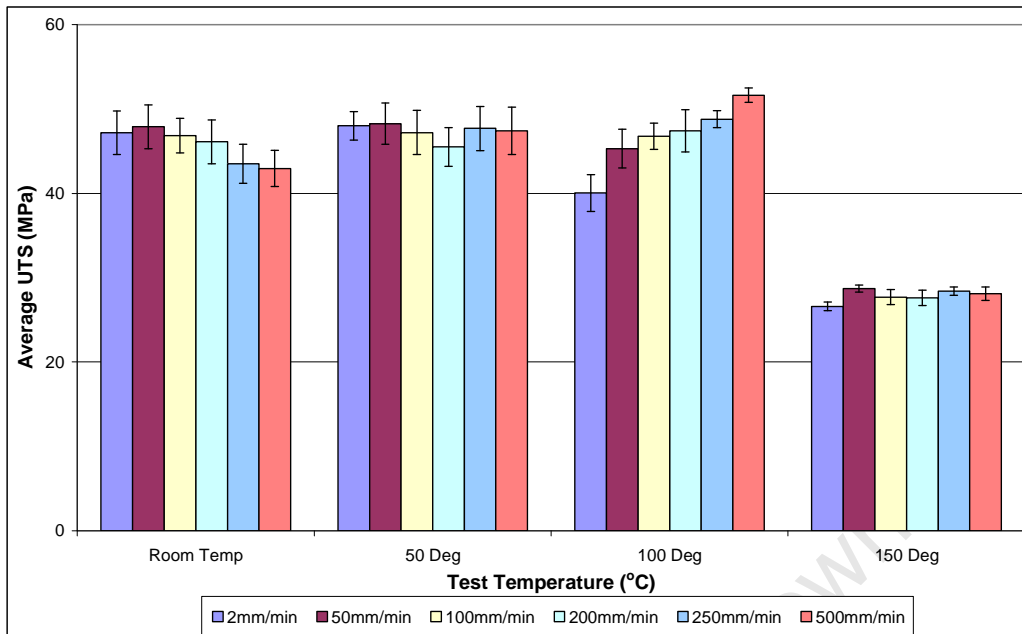


Figure 4.19: Average UTS versus temperature of unfilled PPS at different test speeds.

4.3.2.2 Temperature Effect on the Tensile Properties of 622 PPS Hybrid

A representative of the effect of temperature on the tensile properties of the 622 PPS hybrid composite is given in the stress–strain curves of figure 4.20. The effect of temperature and test speed on the UTS of this hybrid composite is also presented in the bar graphs of figure 4.21. In figure 4.20 the test speed was 2mm/m while in figure 4.21 the tests were conducted at all the test speeds and test temperatures. As expected, it is apparent from figure 4.20 that the tensile strength of the 622 PPS hybrid composite decreased with increasing temperature with a slight increase in strain to failure. During testing the material failed in a brittle mode at all the test temperature and test speeds. It can be seen from figure 4.21 that the material seems to be generally slightly sensitive to increasing test speed at room temperature and the other test temperatures.

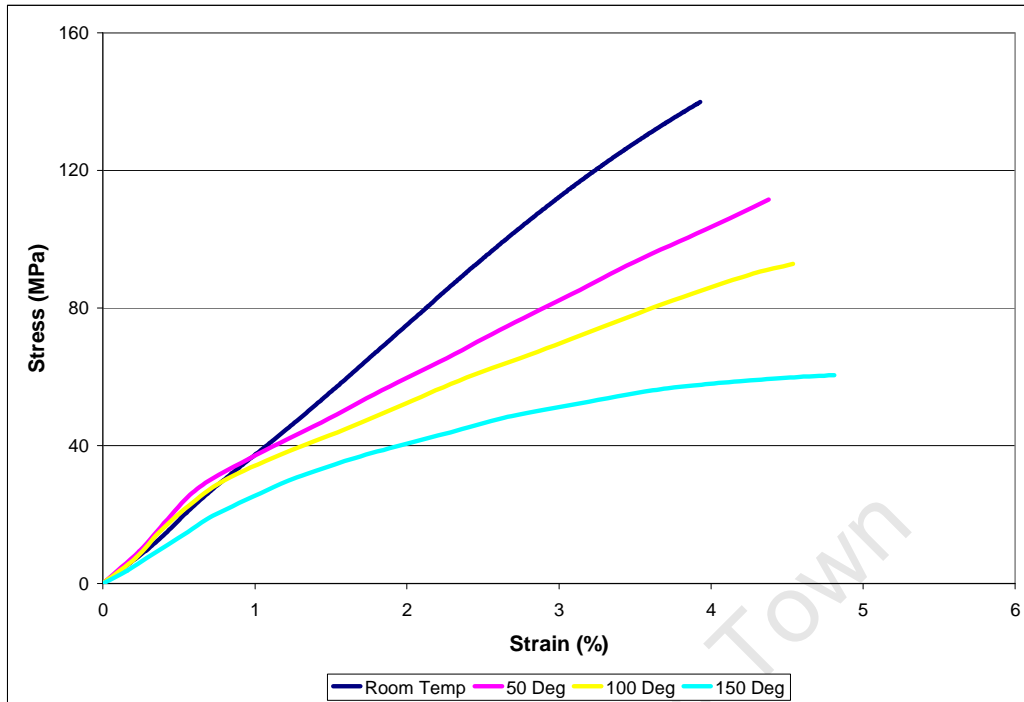


Figure 4.20: The stress-strain curves for 622 PPS hybrid at 2mm/min and different temperatures.

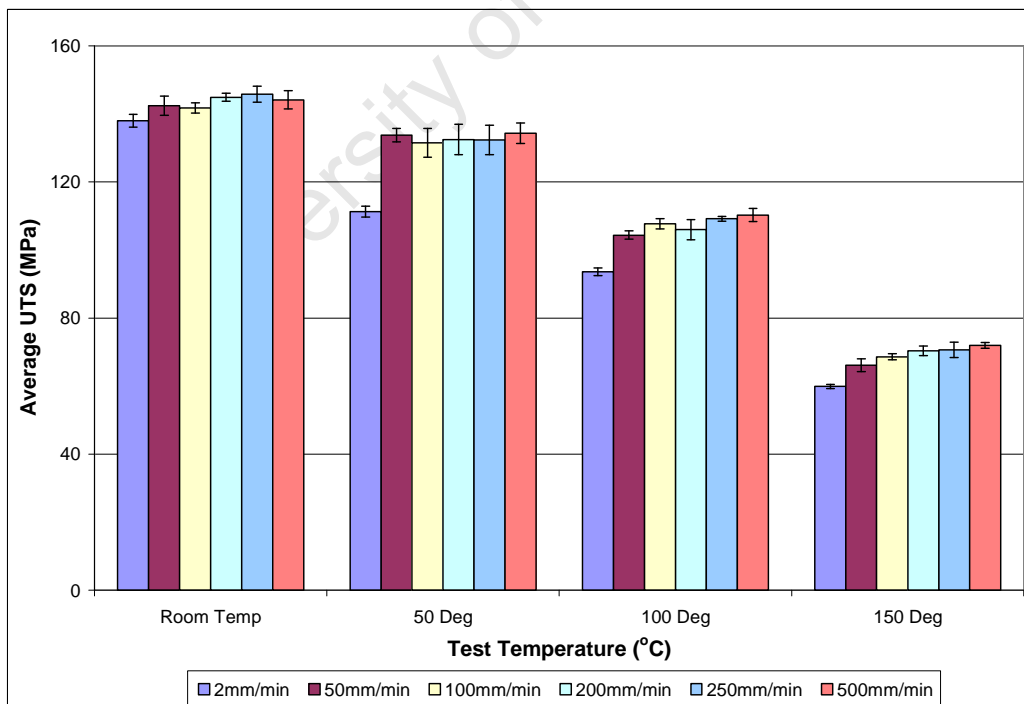


Figure 4.21: Average UTS versus temperature for 622 PPS hybrid at different test speeds.

4.3.2.3 Temperature Effect on the Tensile Properties of 631 PPS Hybrid

The results below indicate a representative stress-strain relationship and the average UTS versus temperature of the 631 PPS hybrid composite in figures 4.22 and 4.23, respectively. Inspection of the results in figure 4.22 shows a decrease in tensile strength of the 631 PPS hybrid composite as temperature increases with a little change in ductility. The 631 PPS hybrid composite seems to be sensitive to test speed changes as can be seen from figure 4.23 below. The average UTS increases with increasing test speed at all the test temperatures except for 150°C where it appears to become insensitive to test speed changes. The sensitivity of the UTS of the PPS materials to test speed seems to be affected by temperature changes.

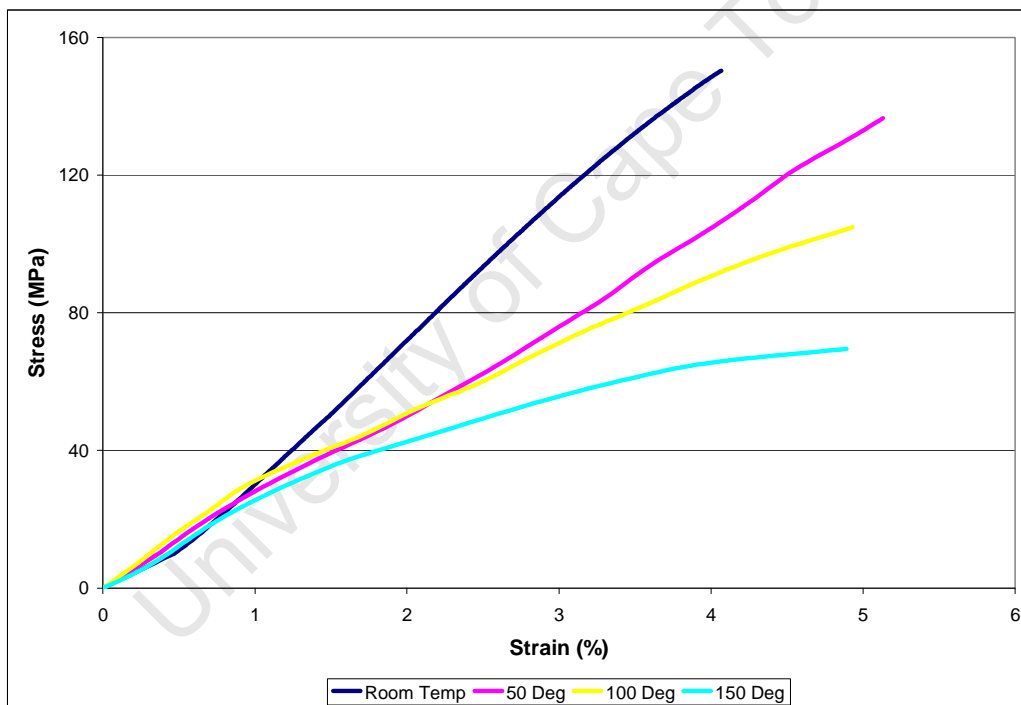


Figure 4.22: The stress-strain curves for 631 PPS hybrid at 2mm/min and different temperatures.

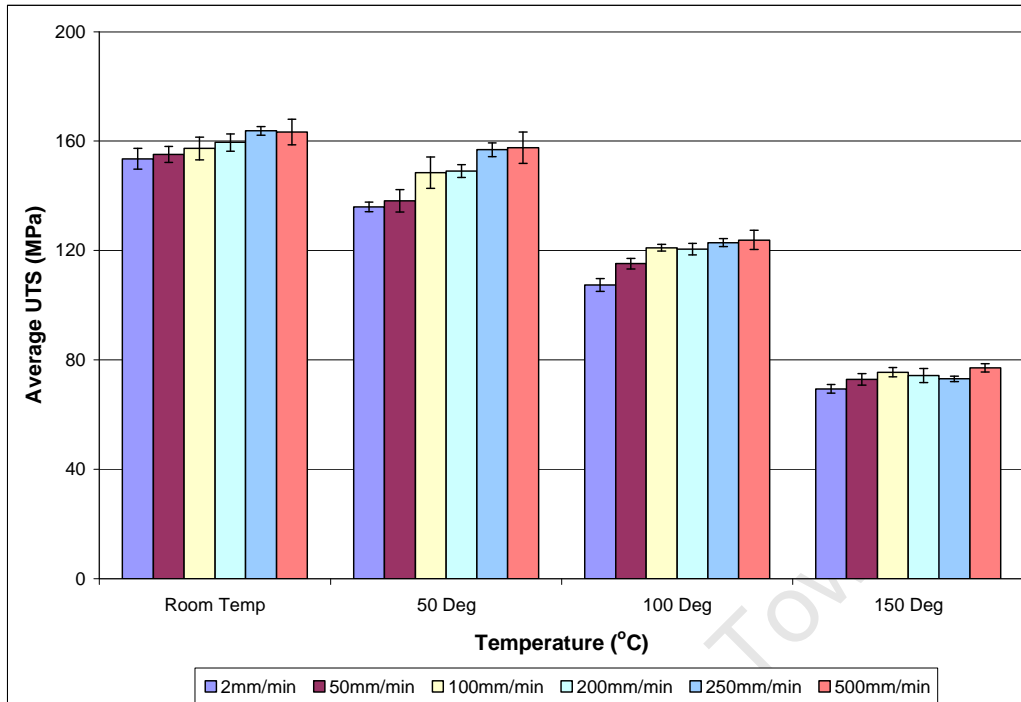


Figure 4.23: Average UTS versus temperature for 631 PPS hybrid at different test speeds.

4.3.2.4 Temperature Effect on the Tensile Properties of 40% GF PPS

The effect of temperature on the tensile properties of the 40% GF PPS composite is given in figure 4.24, while figure 4.25 gives the effect of temperature and test speed on the 40% GF PPS composite. It can be seen from figure 4.24 that the tensile strength of the 40% GF PPS composite decreases as temperature increases with no significant increase in strain to failure. It can be seen from figure 4.25 that the sensitivity of this composite to test speed is very much affected by temperature. At room temperature, the strength tend to increase with increasing test speed but this seems not to be the case at 50°C and 100°C although there is a general increase in strength as test speed increases at these temperatures.

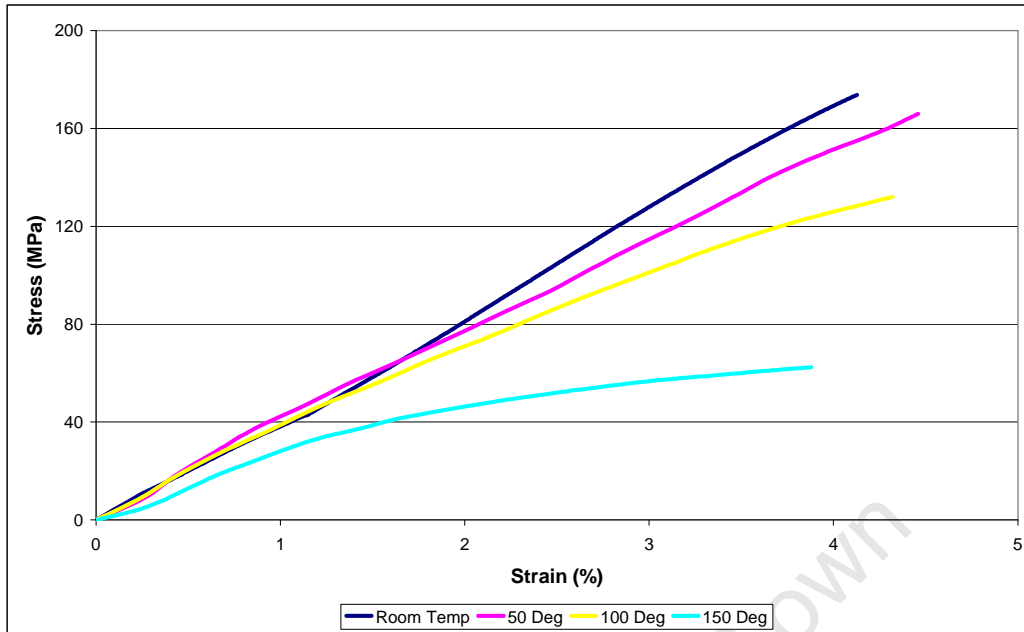


Figure 4.24: The stress-strain curves for 40% GF PPS composite at 2mm/min and different temperatures.

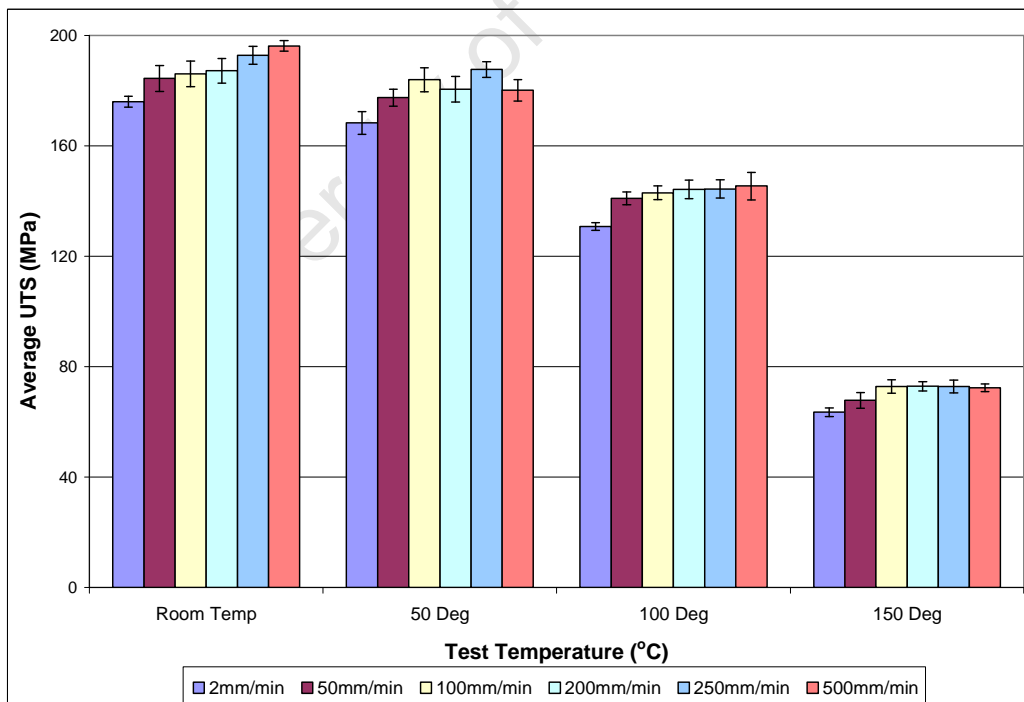


Figure 4.25: Average UTS versus temperature for 40% GF PPS at different test speeds.

4.3.2.5 Tensile Properties of the PPS Materials at Different Temperatures

The results in figure 4.26 below compare the average UTS of the PPS materials with each other at the different test temperatures. Each of the tests was conducted at 2mm/min. Inspection of figure 4.26 shows that the 40% GF PPS composite exhibit a higher strength at all the test temperatures except at 150°C where 631 PPS hybrid composite showed higher strength than the 40% GF PPS composite. It can also be seen that at each of the test temperatures, the unfilled PPS showed the least strength followed by the 622 PPS hybrid composite, then the 631 PPS hybrid composite. This is an indication that the amount of glass fibre in the composite contributed greatly to the strength of the composite. It is also apparent that the strength of each of the PPS material decreased with increasing temperature as expected. A similar result was obtained at all the test speeds for the PPS materials at the different test temperatures.

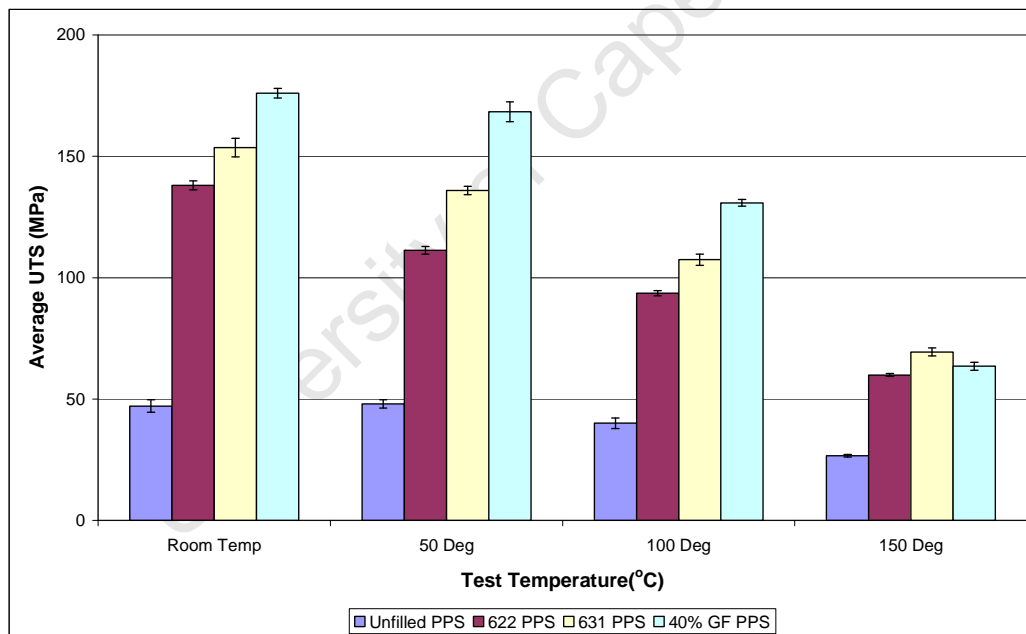


Figure 4.26: Average UTS versus temperature at 2mm/min of the different PPS materials.

4.4 THREE-POINT BEND TESTING

Three-point bend tests were performed on the PPS and its composites. The materials tested were unfilled PPS, the 622 PPS hybrid composite, the 631 PPS hybrid composite and the 40% glass fibre reinforced PPS. Each of the specimens was tested to failure and a representative stress versus deflection curves are shown in figure 4.27. For each of the materials, ten bend tests were carried out to ensure repeatability. The averages of the bend strength and bending modulus with their standard deviations are tabulated in table 4.5 and also presented in bar graphs in figures 4.28 and 4.29. The error bars indicate the standard deviations. Inspection of figures 4.27 to 4.29 shows that the 40% GF PPS has higher flexural strength and modulus than the 631 PPS and the 622 PPS hybrid composites. As expected the unfilled PPS displayed the least flexural strength and modulus. This observation is in agreement with the tensile properties of the PPS materials.

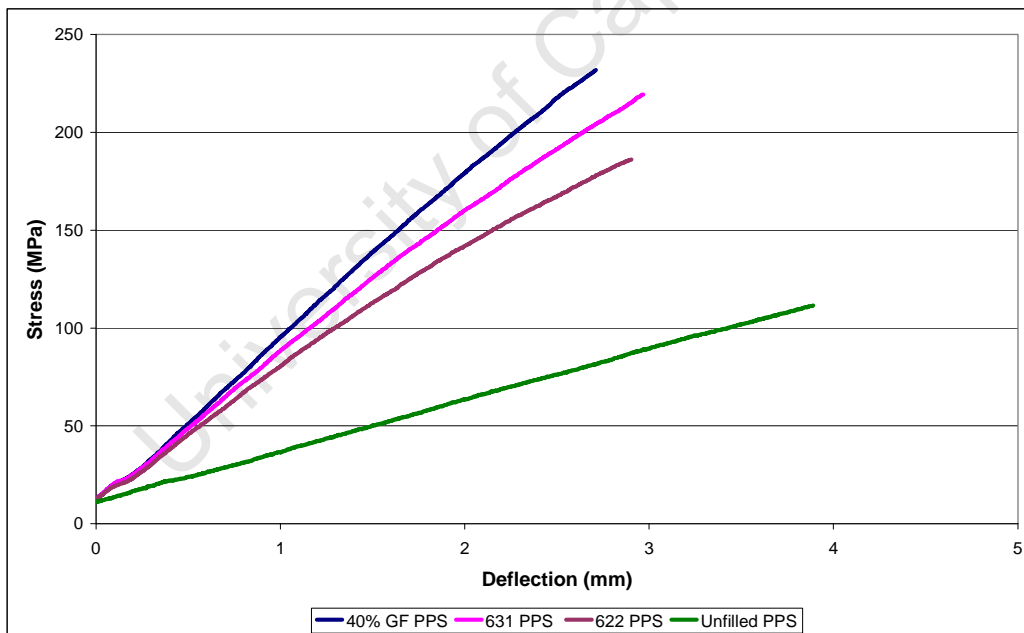


Figure 4.27: Flexural stress-deflection curves for PPS and its composites tested at 5mm/min and room temperature.

Table 4.5: The room temperature flexural properties of PPS and filled PPS materials.

Material Type	Flexural Strength (MPa)	Flexural Modulus (GPa)
40% GF PPS Composite	241.2 ± 3.2	13.6 ± 0.1
631 PPS Hybrid Composite	218.4 ± 5.4	11.2 ± 0.1
622 PPS Hybrid Composite	184.3 ± 3.5	9.4 ± 0.2
Unfilled PPS Resin	111.5 ± 6.6	4.3 ± 0.3

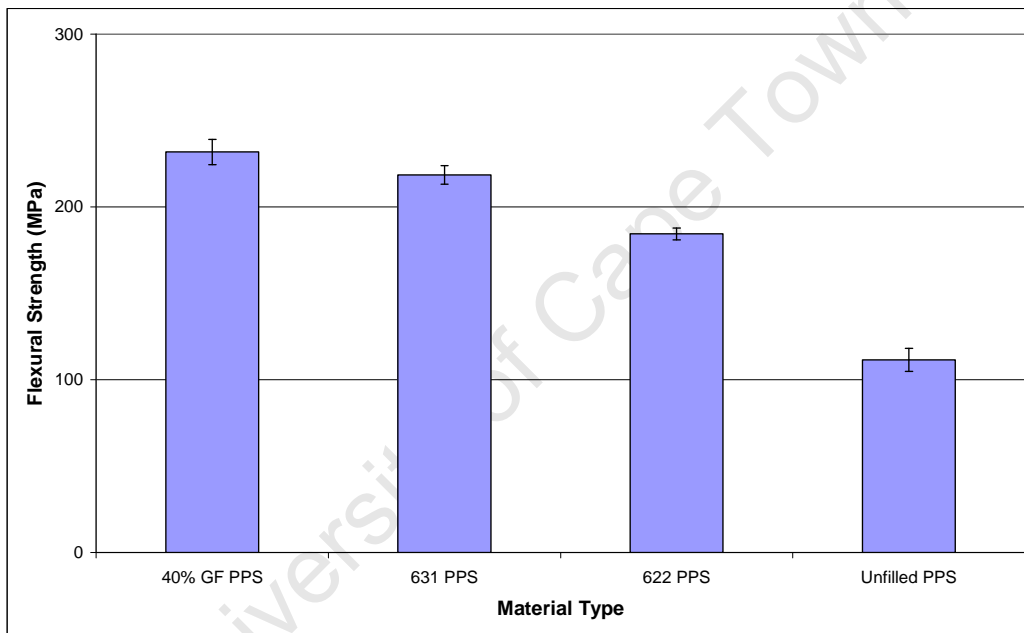


Figure 4.28: Average flexural strength of PPS and its composites tested at 5mm/min and room temperature.

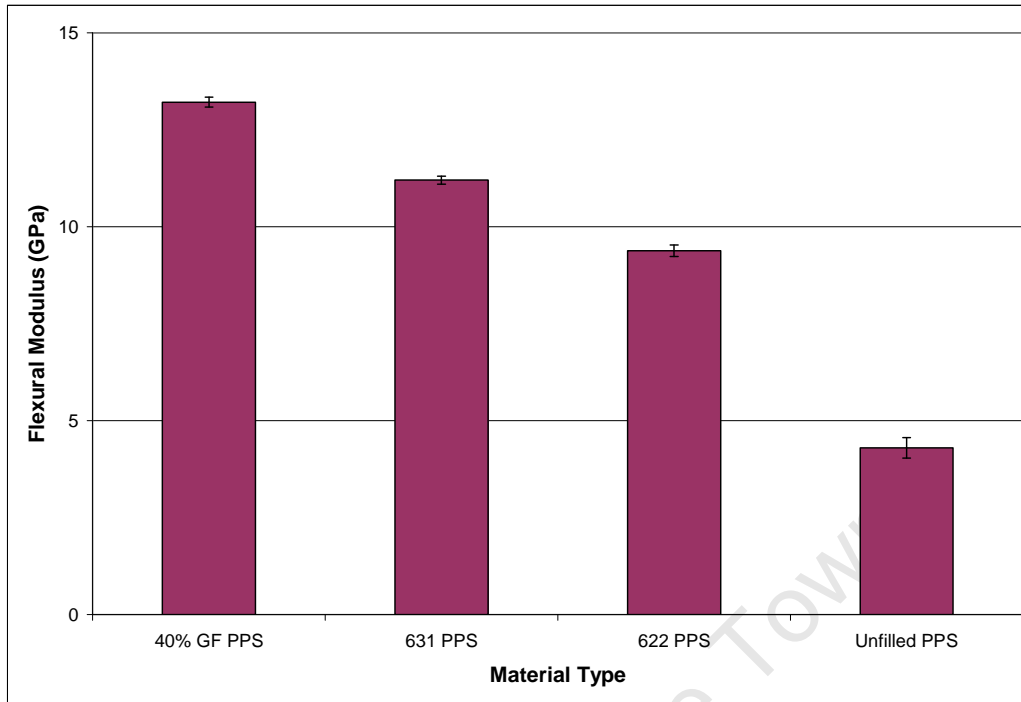


Figure 4.29: Flexural modulus of PPS and its composites test at 5mm/min and room temperature.

4.5 IMPACT TEST RESULTS

An investigation of the impact behaviour of the high performance PPS and its composites has been conducted. The approach used to assess the toughness of the materials was by Izod pendulum impact testing using a 2.75J hammer and Charpy pendulum impact testing using a 4J hammer. The impact behaviour in terms of impact resistance and the sensitivity of the materials to notching as well as their fracture behaviour were established. The results of the Izod and Charpy impact tests performed on the PPS and its composites are presented in figures 4.30 to 4.32 and in table 4.6. Figures 4.30 and 4.31 indicate the Izod and Charpy impact resistance of the notched and un-notched specimens of PPS and its composites. It can be observed from figures 4.30 and 4.31 that the un-notched impact resistance is three times greater than the impact resistance of the notched specimens. This is an indication of how detrimental the presence of a crack (notch) can be to the impact resistance of a material. This indicates how sensitive the PPS materials under consideration here are to notching.

In comparing the Izod and Charpy impact resistances, it is clear from figure 4.32 and table 4.6 that both the notched and un-notched Izod impact resistances are marginally higher than the Charpy impact resistance of all the material types except for the 622 PPS hybrid composite where the notched Izod and Charpy impact resistances are similar. The un-notched Izod impact resistance of the 631 PPS hybrid composite is also similar to that of the 40% GF PPS composite.

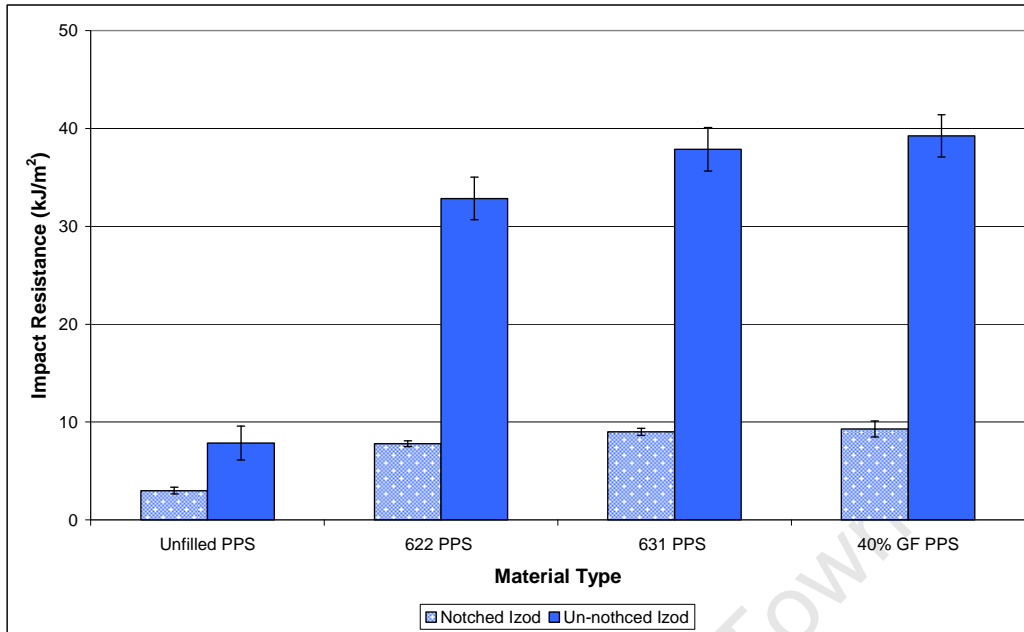


Figure 4.30: The Izod impact resistance of PPS and its composites.

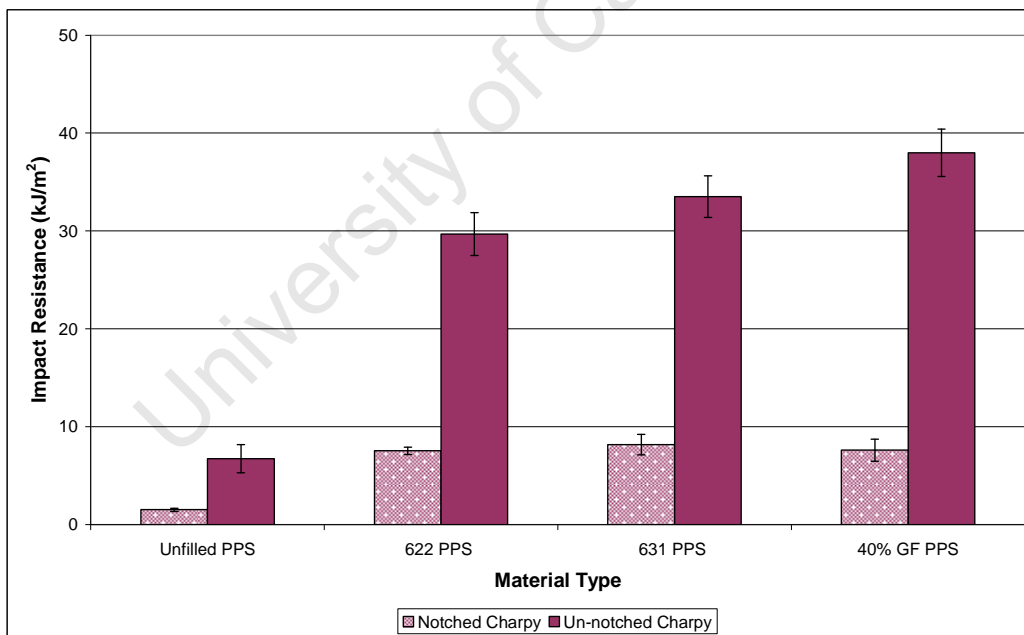


Figure 4.31: The Charpy impact resistance of PPS and its composites.

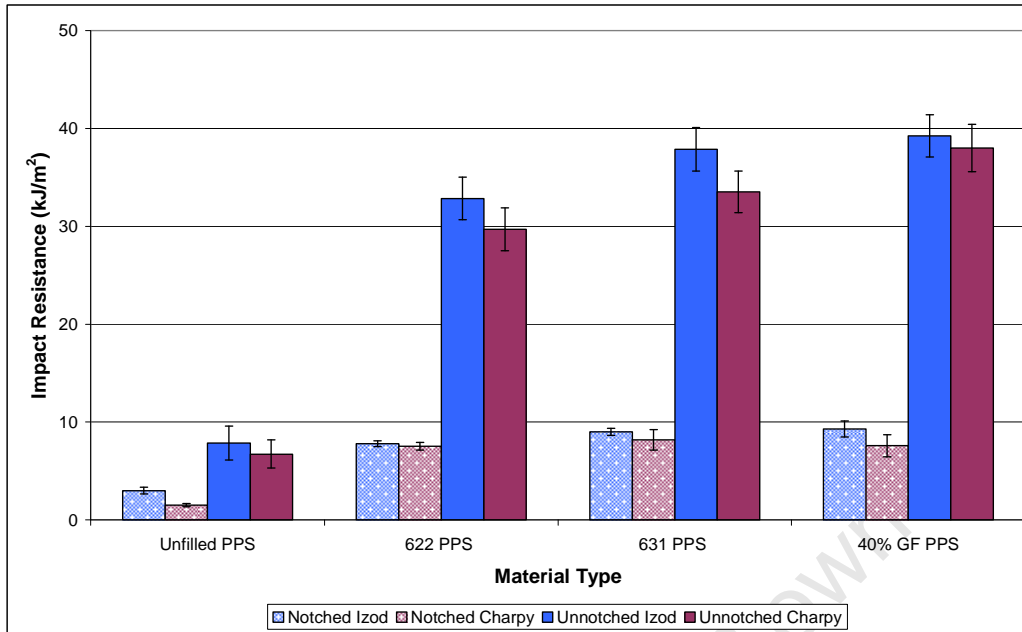


Figure 4.32: Izod and Charpy impact resistance of PPS and its composites.

Table 4.6: Izod and Charpy impact resistance of the PPS materials.

Materials Tested	Izod Impact Resistance (kJ/m ²)		Charpy Impact Resistance (kJ/m ²)	
	Notched	Un-notched	Notched	Un-notched
40% GF PPS	9.3 ± 0.8	39.2 ± 1.2	7.6 ± 1.1	38.0 ± 2.3
631 PPS	9.1 ± 0.4	38.9 ± 3.5	8.2 ± 1.1	33.5 ± 2.1
622 PPS	7.8 ± 0.3	32.8 ± 3.2	7.5 ± 0.4	30.0 ± 2.2
Unfilled PPS	3.0 ± 0.4	7.9 ± 1.7	1.5 ± 0.2	6.7 ± 1.4

4.6 CREEP TESTING

Creep tests were conducted on all the PPS material types using the custom built in-house creep rig for polymers and polymer composites. Experimental data obtained from the creep testing were analysed and represented in a plot of creep strain as a function of time. In this representation each curve represents the creep strain response at constant values of temperature and stress as mentioned in section 3.4.4 of chapter 3.

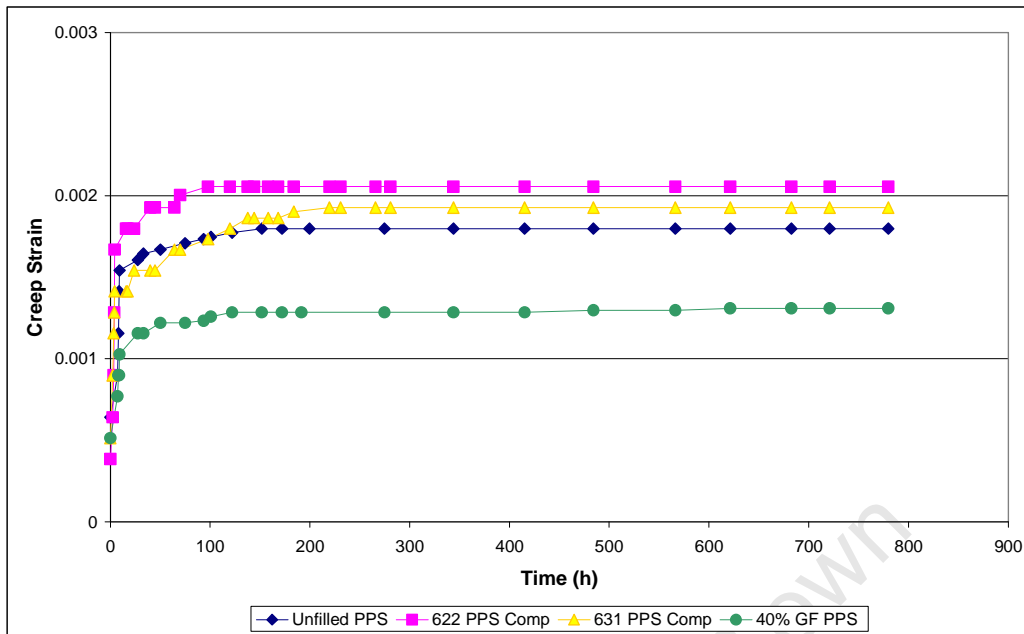


Figure 4.33: Creep strain-time curves for the different PPS materials conducted at 100°C at a load of 2.414kg.

The results in figure 4.33 indicate that the PPS materials are creep resistant in general. The maximum creep strain for the duration of the testing is just above 0.002. The 40% GF PPS is more creep resistant than the unfilled PPS which in turn is more creep resistant than the hybrid composites. It can be observed from the graph that the 40% GF PPS and the unfilled PPS reached the steady state quicker than the 631 PPS and 622 PPS hybrid composites. This may be partly due the presence of talc in the hybrid composites.

It is important to mention that the purpose of this part of the research is not to undertake an in-depth study of the creep properties for the PPS materials, but rather to rank the materials according to their creep response/resistance. If creep data of the PPS materials are needed for engineering design purpose, then a multiple tests at each temperature and creep stress are to be conducted. The data obtained are then analysed statistically and mean strain versus time curves developed for use in design.

4.7 SCANNING ELECTRON MICROSCOPY ANALYSIS

The rapid progress in composite materials technology and its continuous use in high technological applications demand an understanding of the failure mechanisms in composite materials. This will provide guidance to composite material developers as well as structural designers. The deformation behaviour in polymer composite materials occur as matrix cracking, fibre–matrix debonding, fibre pullout, fibre fracture, matrix shear yielding and matrix plastic deformation. These failure mechanisms do not occur in isolation or in a sequence, they interact with each other.

Characterization of the morphology of the fracture surfaces of short fibre reinforced composites is often conducted with scanning electron microscopy (SEM). The morphology of the fractured surfaces of the tensile, flexural and impact tests specimens were examined using the scanning electron microscope (SEM) to understand the failure mechanisms of the PPS and its composites materials. These morphologies provide evidence to study the strengthening and toughening mechanisms of the composites.

The fracture surface morphology of short fibre reinforce thermoplastics has often been used to assess the degree of fibre-matrix interfacial bond/adhesion. The SEM micrographs of the fractured surfaces of the failed specimen were captured to study the mode of failure and the failure mechanisms. Mechanical properties such as tensile strength, fracture toughness and failure strain are then correlated with the morphology.

4.7.1 TENSILE FRACTURE SURFACES AT ROOM TEMPERATURE

The micrographs in figure 4.34 below illustrate the tensile fracture surfaces of the PPS materials tested at room temperature and 2mm/min. Micrograph (A) shows a brittle fracture of the unfilled PPS. There is no sign of plastic deformation on the fracture surface of the unfilled PPS material. The fracture surface shows a fast fracture mode. The micrographs (B), (C) and (D) illustrate the tensile fracture behaviour of PPS composites. The fracture surfaces show the different adhesion of the polymer matrix material to the short glass fibres. A relatively weak interface between PPS matrix and glass fibres can be seen in micrograph (D). This results in failure mechanisms such as fibre-matrix debonding and fibre pullout. Although these failure mechanisms can be observed in micrographs (B) and (C), the fibres in (B) and (C) appear to be surrounded with some matrix material, indicating some interfacial bond between fibres and matrix. This may be due to the presence of talc in these composites.

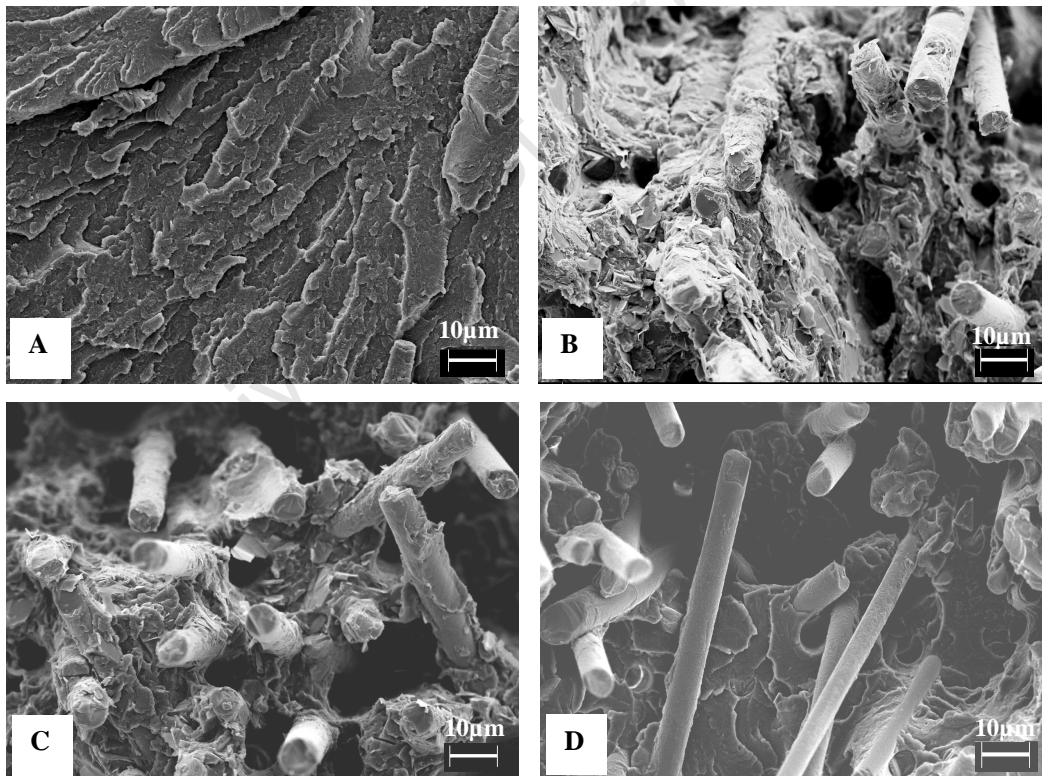


Figure 4.34: SEM micrographs of the tensile fractured surfaces of: unfilled PPS (A); 622 PPS hybrid composite (B); 631 PPS hybrid composite (C) and 40% GF PPS composite (D) at room temperature and 2mm/min.

4.7.2 TENSILE FRACTURE SURFACES AT 50°C

The tensile fracture behaviour of the PPS materials at 50°C and 100mm/m are given in figure 4.35. It can be seen from the fracture surface in micrograph (A) that the unfilled PPS failed in a brittle fast fracture mode. Fibre-matrix debonding and fibre pullout can be observed in micrographs (B) and (C) of figure 4.35. There is a less amount of the matrix material surrounding these fibres than those surrounding the fibres in figure 3.34 (B) and (C). This is due to the difference in test temperatures and test speeds. In addition to fibre-matrix debonding and fibre pullout, there is also fibre breakage in micrograph (D).

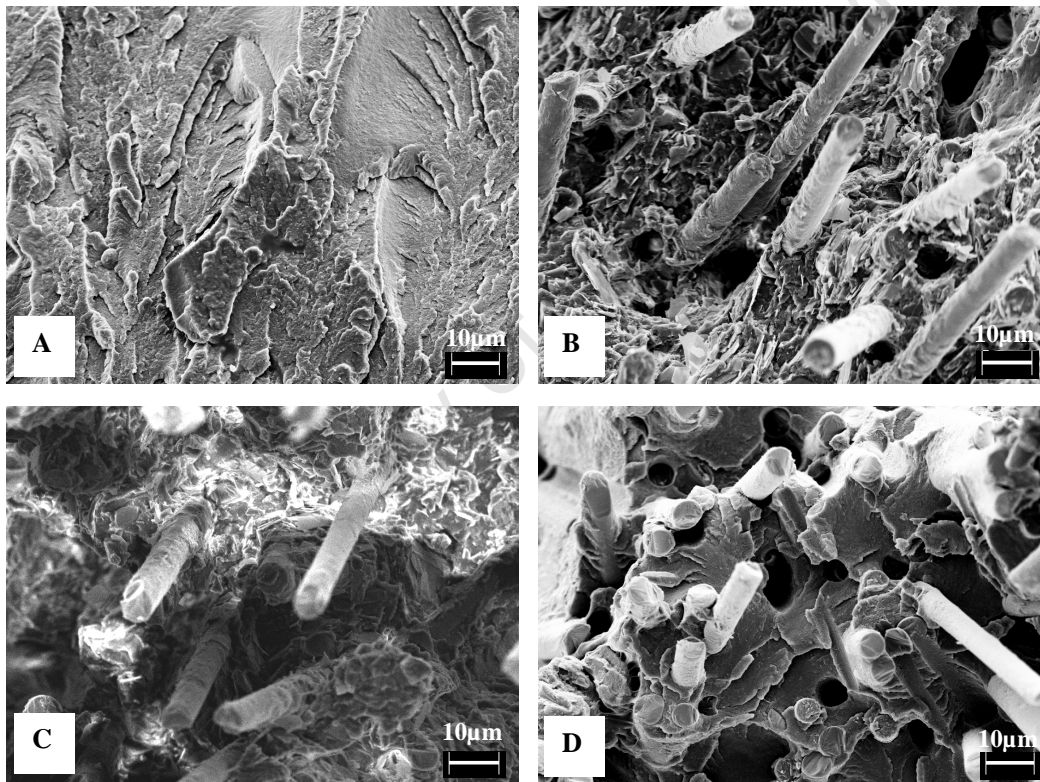


Figure 4.35: SEM micrographs of the tensile fractured surfaces of: unfilled PPS (A); 622 PPS hybrid composite (B); 631 PPS hybrid composite (C) and 40% GF PPS composite (D) at 50°C and 100mm/min.

4.7.3 TENSILE FRACTURE SURFACES AT 100°C

The micrographs in figure 4.36 are the fracture surfaces of the PPS materials tested at 100°C and 2mm/m. The fracture surface of the unfilled PPS in micrograph (A) shows some plastic deformation which resulted in some form of shear yielding of the material. This results in a ductile fracture which leads to a higher failure strain. The fracture surfaces of micrographs (B), (C) and (D) show some fibre pullout and fibre breakage. However, the predominant mode of failure in (B) and (C) is a combination of fibre pullout and breakage while in micrograph (D) the predominant mode of failure is fibre breakage. This is an indication of a strong fibre-matrix interfacial bond.

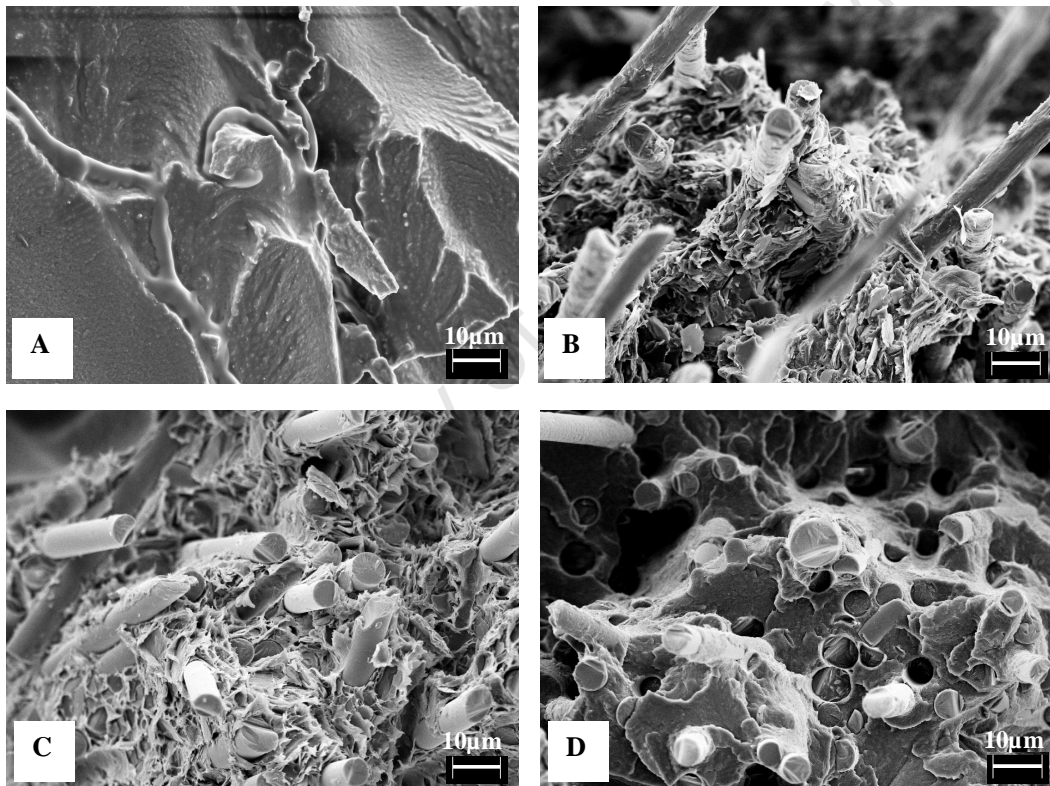


Figure 4.36: SEM micrographs of the tensile fractured surfaces of: unfilled PPS (A); 622 PPS hybrid composite (B); 631 PPS hybrid composite (C) and 40% GF PPS composite (D) at 100°C and 2mm/min.

4.7.4 TENSILE FRACTURE SURFACES AT 150°C

The micrographs of the fracture surfaces of the PPS composites tested at 150°C and a test speed of 2mm/m are shown in figure 4.37. Micrographs (A), (B) and (C) illustrate the tensile fracture behaviour of the PPS composites since the unfilled PPS testing at this temperature was terminated before failure. The fracture surfaces show the different adhesion of the polymer matrix material to the short glass fibres at 150°C. A relatively weak interface between PPS matrix and glass fibres can be seen in micrograph (A) and (B). This leads to fibre-matrix debonding and fibre pullout. The presence of fibres with little or no matrix material adhered to it indicate a weak fibre-matrix interfacial bond. In micrograph (C) the fibres appear to be surrounded with some matrix material, indicating some interfacial bonding between the fibres and matrix. This is evident from the fracture surface of 40% GF PPS which shows a cluster of fibres covered with the polymer matrix.

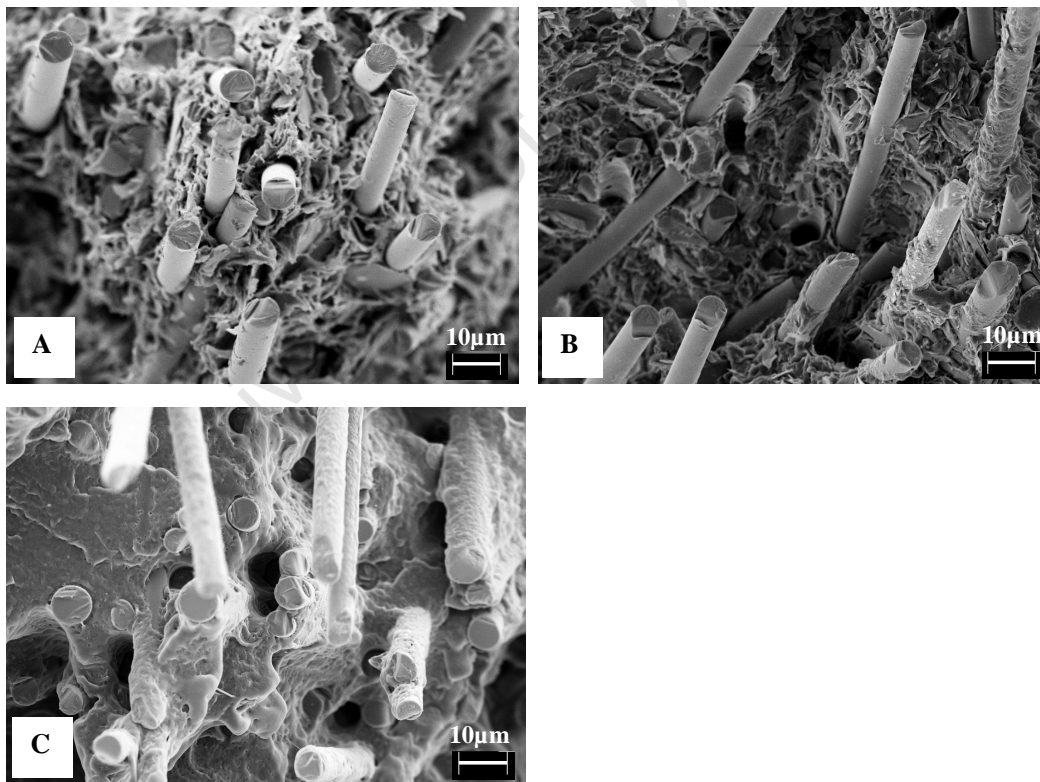


Figure 4.37: SEM micrographs of the tensile fractured surfaces of: 622 PPS hybrid composite (A); 631 PPS hybrid composite (B) and 40% GF PPS composite (C) at 150°C and 2mm/min.

4.7.5 FRACTURED SURFACES OF THE FLEXURAL TEST SPECIMENS

The three-point bend fracture surfaces of PPS and its composites are shown in the micrographs of figure 4.38 below. The fracture surface of the unfilled PPS shows a brittle fast fracture mode. The micrographs of the fracture surfaces of the PPS composites shows different adhesion of the polymer matrix to the short glass fibres as illustrated in figure 4.38 (B), (C) and (D). Fibre pullout and bulk fibre pullout as well as fibre breakage can be observed in the fracture surfaces of the composites. The fracture surface of the 622 PPS hybrid composite displayed a strong fibre-matrix interfacial bond. This is evident from the amount of matrix material adhering to the fibres. Compared to micrograph (B), there is some matrix material surrounding the fibres in micrographs (C) and (D), indicating some level fibre matrix interface adhesion.

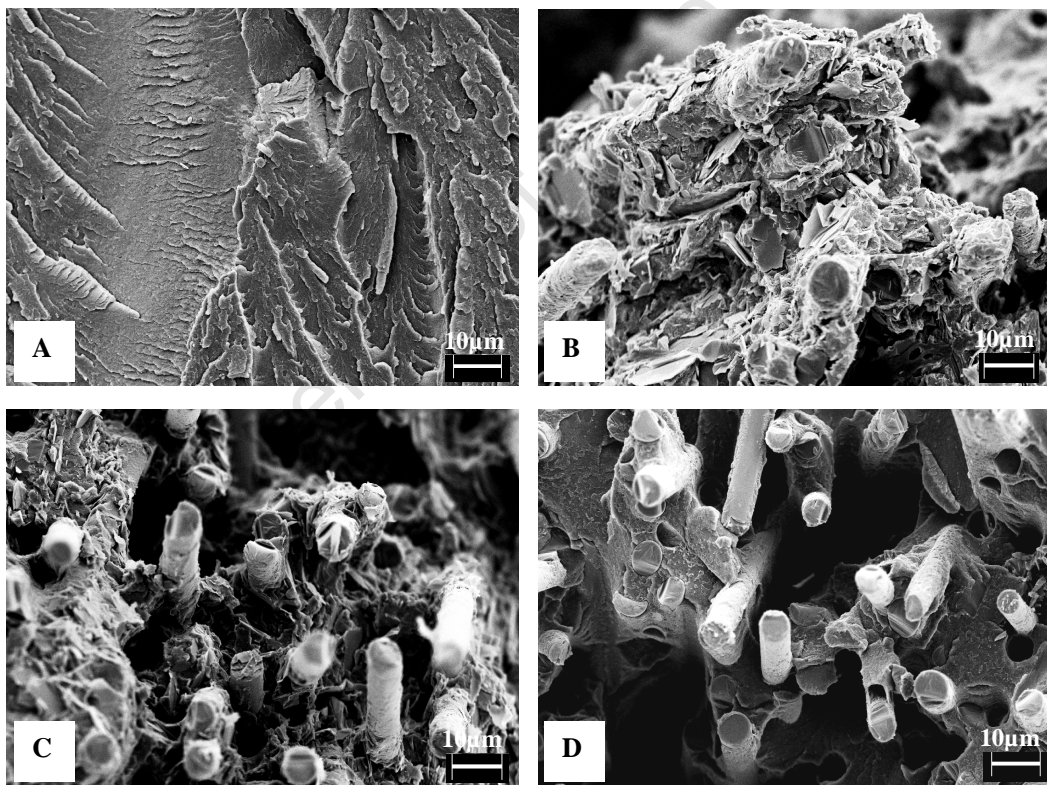


Figure 4.38: SEM micrographs of the room temperature flexural fractured surfaces of: unfilled PPS (A); 622 PPS hybrid composite (B); 631 PPS hybrid composite (C) and 40% GF PPS composite (D).

4.7.6 FRACTURED SURFACES OF THE IMPACT TEST SPECIMENS

The micrographs of the fracture surfaces of the PPS materials subjected to Izod and Charpy impact tests are shown in figures 4.39 and 4.40, respectively. The micrographs of the unfilled PPS in both figures reveal a brittle fracture. Similarly, the fracture surfaces of the PPS composite materials in figures 4.39 and 4.40 show fibre breakage as the predominant mode of failure. This indicates a brittle mode of failure of the composites. The impact fracture surfaces of PPS and its composites show a fast fracture mode of failure.

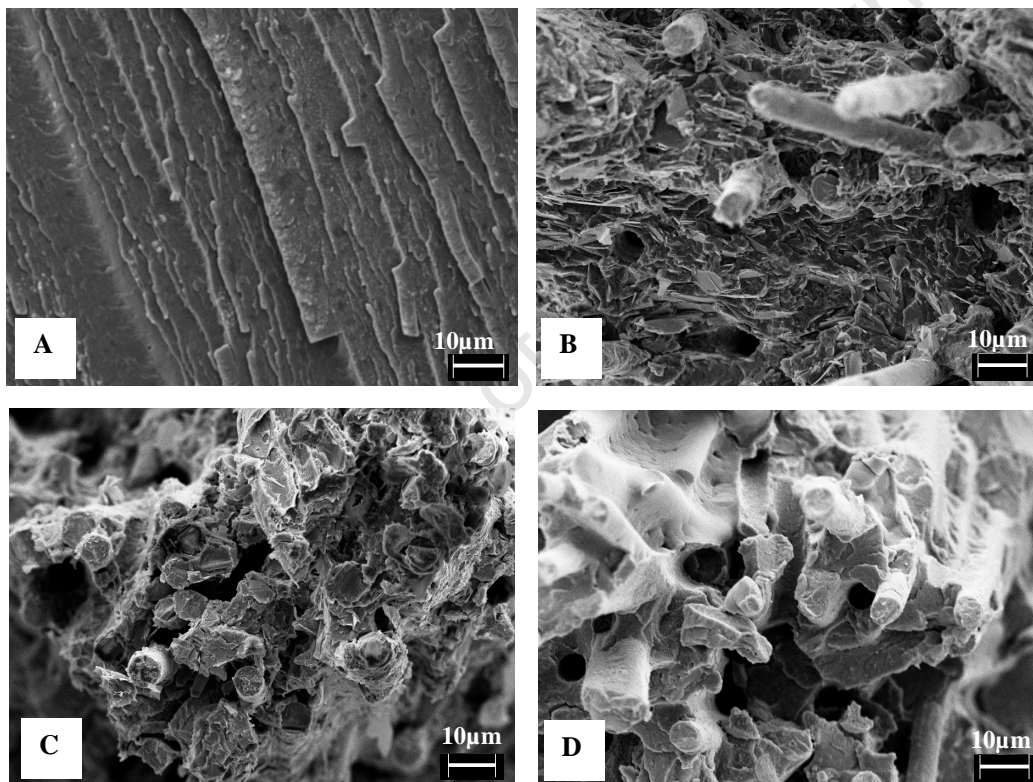


Figure 4.39: SEM micrographs of the Izod Impact fractured surfaces of: unfilled PPS (A); 622 PPS hybrid composite (B); 631 PPS hybrid composite (C) and 40% GF PPS composite (D).

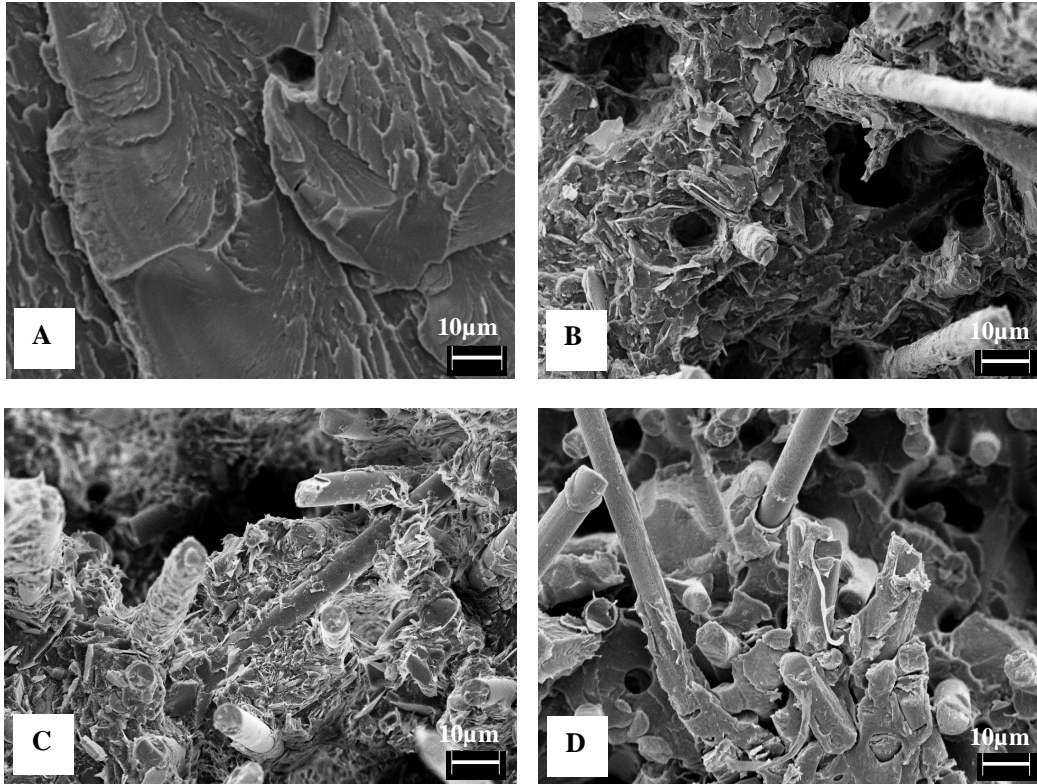


Figure 4.40: SEM micrographs of the Charpy Impact fractured surfaces of: unfilled PPS (A); 622 PPS hybrid composite (B); 631 PPS hybrid composite (C) and 40% GF PPS composite (D).

CHAPTER FIVE

DISCUSSION

5.1 INTRODUCTION

The development of new technologies and the need for improved performance has resulted in the necessity for materials with high specific strength and stiffness. By virtue of their excellent specific properties, good corrosion resistance, toughness and fatigue properties, fibre reinforced polymers have found increased application in the automotive as well as aerospace industries.

Advanced hybrid thermoplastic composites based on PPS matrix were developed and mechanically tested to provide some characterisation for them. This chapter discusses and analyse all the findings and results obtained in this research study.

5.2 CURRENT ADVANCED THERMOPLASTICS AS MATRICES

The evolution of thermoplastics as matrices for polymer matrix composites resulted from the fact that the thermosetting matrices such as epoxy and polyester resins reinforced with fibres suffered from chemical instability with their prepregs having limited shelf life. Thermoplastics prepregs on the other hand do not suffer from these problems. In addition to the many inherent beneficial properties of polymer matrix composites, thermoplastic matrix composites also lend themselves to recycling which makes them very attractive. According to Smith [21], thermoplastic matrices offer attractive properties such as superior toughness and impact resistance, post-process formability and improved repairability over their thermoset counterparts.

Currently only a few advanced thermoplastic polymers have emerged as matrix

material for composites that dominate the aerospace field. These high temperature polymers are PEEK, PPS, PAI, PES, PEI and PEKK. Of these polymers PEEK has become one of the most widely known and used material. This is because its mechanical properties have been well characterised and effects of various environmental agents including solvents, hydraulic fluids and fuels have been assessed with favourable results. Other materials that have been well characterised are PPS and PEI, reinforced with either glass or carbon fibres. Although PEKK exhibits excellent properties, its applications have progressed at a slower rate [102].

Work to apply thermoplastic composites to commercial aircraft has been underway since the early 1990s. An early application was the main landing gear door of the Fokker 50 aircraft [103, 104]. Thermoplastic composites are gaining recognition for their toughness and their attributes that can enable recurring cost savings. A wide variety of applications have been made that ranges from tertiary and secondary to primary structures that has received Federal Aviation Administration (FAA) certification. To date, applications of thermoplastic composites have ranged from small, simple, structural details such as ribs or spars up to relatively large structures [103, 104].

5.3 MATRIX FOR THE HYBRID COMPOSITES

To develop an advanced hybrid thermoplastic composite, there is the need to select a matrix material. From the literature survey, there are a couple of high temperature polymers that are used as matrix in the advanced thermoplastic composite industry. The choice of a matrix material for this research was based on many factors such those as mentioned in section 3.2. In section 2.12, several candidate thermoplastics such as PEEK, PPS, PAI, PES, PEI and PEKK were compared as far as their characteristics are concerned. Processability, mechanical properties and availability were used as the selection criteria. The price and glass transition temperatures of the thermoplastics were considered as the most critical attributes. Based on the above, PPS was selected as the thermoplastic resin to be used as a matrix material to develop the advanced hybrid thermoplastic composite.



PPS is an organic polymer consisting of aromatic rings linked with sulphides. It offers an excellent balance of properties including high temperature resistance, chemical resistance, flowability, dimensional stability and electrical characteristics. Muzzy [62] stated that PPS is frequently used as a matrix material in the thermoplastic composite industry because of its ease of processing, excellent chemical resistance, low moisture absorption and high strength and stiffness. According to Yilmaz et al [105] PPS must be filled with fibres and fillers to overcome its inherent brittleness. Due to its low viscosity, PPS can be moulded with high loading of fillers and reinforcements. These fillers and reinforcements will make a difference in the strength, surface properties, dimensional stability and overall cost.

PPS is a semi-crystalline polymer with highly stable chemical bonds and a remarkable degree of thermal stability. However, knowledge of the thermal stability and the temperature at which it degrades was essential for the development of the hybrid composites. This is because the compounding and processing of the PPS hybrid composites was taken through a couple of thermal cycles.

5.4 THERMAL STABILITY OF THE PPS RESIN

A thermogravimetric analysis technique was used to determine the thermal stability of the PPS resin. The results obtained are presented in figure 4.1 of chapter 4. It can be seen from the graphs of the three TGA test runs in figure 4.1 that the material remained thermally stable until 470°C before it started to degrade. This is evident from the typical plateau of the TGA profile at a fairly constant weight level until 470°C. Figure 4.1 shows that decomposition of the PPS commences at about 470°C. Above this temperature the resins are rapidly decomposed in an endothermic process that is dominated by random chain scission of the main polymer chain [80].

The high thermal stability of the PPS resin observed in the TGA testing is due to the highly stable chemical bonds of its molecular structure which impart a remarkable degree of molecular stability toward thermal degradation and chemical reactivity [62, 66]. The molecular structure of PPS is linear with little branching which packs into a



thermally stable semi-crystalline polymer. Although PPS melt at about 285°C, due to the stable chemical bonds within its molecular structure, it remained thermally stable until about 470°C before starting to degrade as observed during the TGA testing.

5.5 THE HYBRID PPS THERMOPLASTIC COMPOSITES

Advanced hybrid composites based on PPS thermoplastic resin as matrix were developed during the research project. As mentioned in section 2.6, the main reason for developing a hybrid composite is to reduce the amount of individual components in the composite with the aim of reducing the overall cost. By mixing two or more different types of fillers and reinforcements, it is possible to combine their advantages while at the same time mitigating their less desirable qualities [106]. Normally, one of the fibres/fillers in the hybrid composite provides the stiffness and load bearing qualities, while the other makes the composite more damage tolerant and keeps the material cost low, thereby tailoring the composite properties to requirements.

In this study, short glass fibres and talc were used as the reinforcements and fillers for the hybrid composites. Glass fibres were used to provide strength and stiffness while talc was used among other things to improve dimensional stability, control mould shrinkage and lower cost. The PPS resin was intimately mixed in a random fashion with the glass fibres and talc fillers to develop the hybrid composite as discussed in section 3.3. The developed hybrid composites were mechanically tested to characterise them alongside unfilled PPS and 40% glass fibre filled PPS composite.

5.6 MECHANICAL PROPERTIES

The mechanical properties of polymer composites depend mainly on three major factors, namely those of the matrix, reinforcement, and interfacial region between the matrix and the reinforcement. The reinforcements, depending on their shape, size, quantity, distribution and orientation in the matrix affect the properties of the composites. When a composite material is put in service, it experiences some kind of



mechanical loading. Tensile, flexural, impact and creep are some of the common mechanical loading a composite material in service may experience. Therefore once a material is developed, testing need to be done to determine the mechanical integrity of the material. This section gives detail analysis and interpretations of the results and observations from the mechanical tests reported in chapter four.

5.6.1 EFFECT OF REINFORCEMENTS AND FILLERS ON THE MECHANICAL PROPERTIES OF THE PPS COMPOSITES

The addition of short glass fibres and talc to the PPS matrix caused an increased in the tensile and flexural strength as well as the elastic and flexural modulus of the composites. There was a dramatic increase in the Izod and Charpy impact resistance of the composite as a result of addition of the glass fibres and talc. This is because when the composite is loaded, the low strength matrix transfers the applied load to the glass fibres. The glass fibres in this case carry most of the stress that the composite is subjected to. The quantity of glass fibres present in the composite also contributed to the improvement of the strength properties.

The addition of talc filler into thermoplastics is a common practice and it is done with the aim of reducing cost and improves the toughness, stiffness and strength balance [56, 57, 58]. Talc fillers do not always enhance the strength and impact properties of thermoplastics. This is due to the geometry of talc particles. However, according to Mallick [35] and Maiti et al [59] talc with a high aspect ratio has shown reinforcing ability in HDPE, PP and other thermoplastics. Unal [61] found out in his studies that the strength of PA 6 linearly increased with talc until 10wt % after which the strength decreased with an increase in talc. The modulus of elasticity also increased linearly with the addition of talc and glass beads, with talc having the higher influence than glass beads. He explained it with the percolation theory which states that a matrix zone around each particle is affected by stress concentration. Therefore, if the distance between particles is small enough, these zones join together and form a percolation network, which increases the modulus. According to Unal [61], in semi-crystalline thermoplastics, the addition of talc can initiate a heterogeneous crystallisation, thus changing the morphology of the matrix and conferring significant property changes.

The introduction of fine particles of talc provides a strong nucleating effect by multiplying the number of crystalline units in the material.

5.6.2 EFFECT OF FIBRE-MATRIX INTERFACIAL BOND

The fibre-matrix interface has a crucial role in the performance of a composite material. The reinforcement is effective in strengthening the matrix only if a strong interfacial bond exists between the reinforcement and the matrix. The interfacial bond between fibres and matrix depends also on the coupling agents used. The role of the coupling agents is to enhance the chemical and physical bonding between the matrix and the reinforcement materials. It also protects the fibre surface from environmental degradation due to moisture or exposure to reactive fluids [10]. Coupling agents are also believed to promote better wetting and matrix infiltration of the fibres resulting in lower void content, especially in composites with high reinforcement volume fractions [51].

It has been reported that fracture surfaces of short fibre reinforced composites containing fibres surrounded with a substantial amount of matrix material imply strong interfacial bond between fibres and matrix, which in turn increase the impact strength. Conversely, smooth fibre surfaces signify weak interfacial adhesion between fibres and matrix [97, 107]. The morphology of the fracture surfaces has often been used to correlate microstructure with the mechanical properties of the composite.

The SEM investigations revealed fibres pullout for the room temperature fracture surfaces of the 40% GF PPS composite whereas the high temperature fracture surfaces contain fibres surrounded with substantial amount of matrix material. On the other hand the room temperature fracture surfaces of the hybrid composites revealed profound matrix-adhered fibres while the high temperature fracture surfaces revealed clean fibres. The morphology of the fracture surfaces of the PPS composites indicates that the fibre-matrix interfacial bond is temperature dependant. This may be due the difference in the heat capacities of the fillers (glass fibres and talc) and the PPS matrix material.

High interfacial bond strength results in matrix shear yielding leading to matrix material around fibres. Low interfacial bond strength results in interfacial debonding taking place before matrix shear yielding, little or no matrix material is adhered to the fibre surfaces and thus smooth fibre surfaces are observed [97].

5.6.3 EFFECT OF TEST SPEED (STRAIN RATE) ON THE TENSILE PROPERTIES

The sensitivity of the PPS materials to test speed (strain rate) changes tends to be dictated by temperature changes. It was observed from this study that the room temperature tensile strength of the unfilled PPS appears to decrease marginally as the test speed increases. At 50°C, the polymer seem not be sensitive to test speed changes. However, at 100°C the tensile strength of the unfilled PPS increased with test speed. The PPS composites displayed strain rate sensitivity, with the rate of sensitivity increasing with glass fibre content. It has been shown that increasing strain rate leads to an increase in failure strength of the composites. It follows that the ability to absorb energy by sustaining damage increases as the loading goes from quasi-static to the impact domains [51, 108, 109].

Glass fibres exhibits rate dependence behaviour, the failure strength increase with increasing strain rate and tensile modulus of elasticity of glass fibres increases with strain rate [51] [108, 109]. It then follows that the observed rate dependence of the failure strength of the composites follows from the increased strength of the glass fibres. Agbossou et al [51] found out that for polymeric matrices, increasing strain rate leads to an increase in failure strength; however, in this study the failure strength of the PPS matrix tend to decrease with increasing test speed at room temperature.

In a tension type failure, propagation direction of a crack is perpendicular to the direction of the applied load. It was observed that for the unfilled PPS and its composites, load values drop rapidly to zero in a short time after reaching its maximum. It is clear that sudden drop in load after small displacement means unexpected failure indicating a brittle type failure.

5.6.4 EFFECT OF TEMPERATURE ON THE TENSILE PROPERTIES

From the tensile stress–strain curves at room temperature and 50°C, it can be seen that once the applied stress reaches the yield stress of the material, it breaks immediately, with no visible plastic deformation. When the temperature is raised to 100°C the stress-strain relationship remains basically linear without any great influence of temperature and the load bearing capacity of the composite reduces. The value of maximum stress in this case is smaller than that at room temperature and at 50°C. The magnitude of the elastic modulus also decreased with increasing temperature. At 150°C, however, the unfilled PPS softens greatly and displays non-linear deformation. The whole stress-strain curve of the unfilled PPS looks smooth and a large amount of plastic deformation is observed. The stress-strain curves of the composites displays non-linear relationship with no visible plastic deformation and a very much reduced loading bearing capacity at 150°C.

The load bearing capacity of PPS and its composite gradually decreased as temperature increases. Raising the temperature increases the internal energy of the material, resulting in easier molecular movement. When the temperature has risen up to 150°C, which is higher than the glass transition temperature of the pure resin, the reduction in strength of the composite is large, irrespective of the content of reinforcing fibres and fillers.

From the tensile curves of the composites it can be seen that the tensile stress–strain curves show linear deformation up to 100°C and non-linear deformation at 150°C. The deviation of the curves from linear to non-linear deformation corresponds to either interfacial debonding or matrix shear yielding deformation [97, 110]. Inspection of the SEM micrographs shows that for the hybrid composites, the deformation moved from matrix shear yielding to interfacial debonding as temperature increases. This is evident from the matrix material adhering to fibres on the room temperature fracture surface and clean fibres on those at 150°C. The 40% GF PPS composite on the other hand displayed interfacial debonding to matrix shear yielding as temperature increases. This is indicative of the clean fibres on the fracture surface at room temperature and matrix material adhering to fibres on the fracture surface at 150°C.

5.6.5 FLEXURAL PROPERTIES

Flexural loading introduces tensile and compressive stresses and strains across the thickness of the sample. As seen in equation 2.10, the flexural stress is directly proportional to the load on the composite. The maximum load of the composite depends on the elastic modulus and the ultimate failure strain of the composite. The bending test results show that the flexural strength and modulus of the PPS and its composites are higher than their tensile strength and modulus. It can also be observed that the flexural strength of the 40% GF PPS composite is only marginally higher than that of the hybrid composite with 30% glass and 10% talc (631 PPS).

The flexural failures in composites require attention because of the various possible failure modes. The flexural of thermoplastics has been investigated in recent years. In thermoplastic composites the failure normally propagates in a compressive mode, owing to the low fibre/matrix interfacial strength [111, 112]. A study of flexural failure of carbon/thermoplastic composites showed that failure invariably occurred in the compressive mode [112]. The failure was catastrophic in most cases and revealed no damage before the drop in load. In all of the tests conducted, the failure initiated in the compressive mode and was catastrophic.

5.6.6 IMPACT PROPERTIES

The impact performance of polymer matrix composites is one of the critical properties that need emphasising because low velocity impacts from tools, hail, runway debris and ground equipment can damage the polymer matrix composite. With sufficient kinetic energy, these impacts can damage the composite without readily visible evidence and can significantly reduce the strength. It is important for composite structures to carry ultimate load with no visible impact damage. The very low impact resistance obtained from the impact test results for the notched specimen of the PPS and its composites is an indication of the brittle nature of the materials and as such very sensitive to notching or the presence of a crack. The impact resistance of the unfilled PPS is very low; this indicates the brittle nature of the material. The maximum impact resistance of a specimen with a defect is much smaller than that of a

specimen without a defect [99]. The impact resistance of the un-notched specimen of the composites were approximately four times higher than that of the notched specimen. The study also revealed that the 40% GF PPS and hybrid 2 (631 PPS) composites showed comparable impact properties. They have a higher capacity for energy absorption during impact loading.

The higher impact resistance of the filled polymers can be explained by the observation of Sinmazcelik [113] in his study when he incorporated calcium carbonate particles in addition to short glass fibres into a PPS matrix. He found that the crack length increased considerably during the process of fracture due to the presence of calcium carbonate and the short glass fibres. The growing crack front, during the process of fracture, had to change its path numerous times after interaction with particles leading to higher absorption of energy in the process of fracture. The smaller the size of filler particles, the larger their surface area, hence the crack length increase will be greater leading to an increase in energy absorption before fracture.

A weak fibre-matrix interfacial interaction results in high impact energy absorption, while a strong fibre-matrix interfacial interaction leads to low damage tolerance. If glass fibres adhere poorly to the polymer matrix, extensive fibre-matrix debonding and pullout is observed. These micro-mechanical processes result in high energy absorption during impact. As the fibre-matrix bond strength is increased, energy absorption during impact decreases. Significant fibre breakage during impact dominates the failure and less fibre-matrix debonding and pullout is observed [114]. The SEM study of the impact fractured surfaces of the composites showed both fibre pullout and fibre breakage.

5.6.7 CREEP TESTING

The creep results obtained in figure 4.33 indicate that in general, the PPS materials are creep resistant. This is because for the duration of the testing, the maximum creep strain obtained was just over 0.002. The 40% GF PPS is more creep resistant than the unfilled PPS which in turn is more creep resistant than the hybrid composites. The creep resistance of the 40% GF PPS is due to the presence of the glass fibres which is

an elastic material and has virtually no creep response compared with the thermoplastic matrix. The creep resistance of the unfilled PPS may be due to the strong interaction between the rather bulky lethargic PPS molecules which inhibit chain slippage. It can be observed from the graph that the 40% GF PPS and the unfilled PPS attained steady state creep much quicker than the 30% glass and 10% talc (631 PPS) and the 20% glass and 20% talc (622 PPS) hybrid composites. This may be partly due the presence of talc in the hybrid composites. The results indicates that the presence of talc fillers in the hybrid composites considerably affect the creep behaviour of the PPS hybrid composite. The presence of the talc may have decreased the interactions between the bulky PPS molecules thereby promoting chain slippage making them less creep resistance than the unfilled PPS. Talc has a plate-like structure which can slide past each other easily when subjected to loading. This soft slippery property of talc may have contributed to the relatively poor creep performance of the hybrid composites as compared to the unfilled PPS and the 40% GF PPS.

In this research the creep behaviour of the PPS materials was found to be relatively similar. This is in agreement with Boey et al [115] in their study of non-linear bending creep rate of PPS composites, where they found that the creep deformation of both 20 and 40% glass fibre reinforced PPS samples were relatively similar despite the difference in the amount of fibre reinforcement.

The mechanism of creep in fibre reinforced thermoplastics need to be researched further. This is because thermoplastic matrices can contain both amorphous and crystalline regions. The amorphous regions could re-align themselves by molecular sliding easily. The crystalline regions on the contrary provide stiff resistance to creep, particularly in the longitudinal direction. In the transverse direction, the crystalline phase is more likely to creep. This is due to the creep of the amorphous interlayer that exists between the crystalline lamellar layers [115]. The introduction of fibres into a creeping semi-crystalline thermoplastic matrix complicates the understanding of the behaviour. Most fibres such as glass fibres have virtually no creep response at all compared to the matrix material. When glass fibre reinforced composite is loaded, the stress is transferred to the fibres in the composite. This reduces the stress level in the

matrix considerably, thereby reducing the matrix creep.

5.7 FRACTOGRAPHIC ANALYSIS

The tensile, flexural and impact fracture surfaces of the PPS and its composite materials were studied using the SEM to understand the failure mechanism of the materials. The fracture behaviour of the specimens was used to explain the mechanical behaviour of the materials.

Figures 4.34 – 4.37 are the SEM micrographs of the tensile testing at different test temperatures showing fibre pullout. The extensive fibre pullout seen on the fractured surfaces indicates a weak fibre-matrix interface. The fracture surfaces are rough with protruding broken fibres. The fibre ends indicate a brittle failure mode. However, there are signs of matrix material adhering to some of the fibres which indicate a good bond in the fibre-matrix interface. This brings about brittle failure in the fibres. The observed pullout of fibres is dependent on the bond strength and the load transfer mechanism from the matrix to the fibre.

The hybridization of the composites resulted in changes in the fracture modes from clean fibre pullout as in the 40% GF PPS composite, to glass fibres with matrix material attached in the hybrid composites at room temperature. The opposite of this observation was seen on the fracture surface of the materials tested at higher temperatures. With regard to the fibre-matrix interface stresses, they become severe as temperature rises because of the difference in their thermal expansion coefficients.

Addition of glass fibre increased the ductility of the composites slightly. This is evident from the extensive fibre pullout on the fractured surfaces of the composites. It can be said that the introduction of glass fibres to the PPS matrix to form a composite changes the fracture mode from brittle to relatively ductile. This is in agreement with Khatri et al [106] in their studies of thick-section AS4-graphite/E-glass/PPS hybrid composites.

From room temperature to 100°C, the matrix morphology of the fracture surfaces of the unfilled PPS is basically flat with a cusp, which exists in a shear band with features of brittle behaviour. River marks are also visible, showing the direction of crack propagation in the matrix. The crack has propagated through the thickness of the specimen without significant branching. This is indicative of brittle failure.

The fractography of flexural failures in composites requires attention because of the various possible failure modes. Flexure results in tensile and compressive stresses that are perpendicular to the load. The stresses are greatest at the outside surfaces. In thermoplastic composites the failure normally initiates from the compressive mode, due to the low fibre-matrix interfacial strength [111]. The deep pockets by the pullout of fibre bundles can be clearly seen on the fracture surfaces of the micrographs in figure 4.38. This confirms the debonding of the fibre-matrix interface. The damage in the composite originates from a combination of test speed and interfacial bond strength. The pullout of fibre bundles shows that at this loading rate, the fibre-matrix interfacial bond strength was exceeded before the failure strength of the composite was attained [116]. The failure was catastrophic in most cases and revealed no damage before the drop in load. The smooth surface of the unfilled PPS polymer matrix shows no signs of plastic flow, which indicate a brittle failure.

The fracture surface of the impact specimens of the composites are rough with protruding broken fibres as shown in figures 4.39 – 4.40. The fibre ends indicate a brittle failure mode. Increased damage in the composites brought about by a combination of increased test speed and the interfacial bond strength can be observed on the impact fracture surfaces. The result of this is an extensive fibre pullout and fibre breakage. However, there are signs of matrix material adhering to some of the fibres which indicate a good bond in the fibre-matrix interface. This brings about brittle failure in the composites. The observed fibre pullout is dependent on the bond strength and the load transfer mechanism from the matrix to the fibre. As fibres pullout, matrix debonding occurs, resulting in cracking and disintegration of the matrix. It was pointed out earlier that the two most important energy absorbing failure mechanisms in fibre reinforced composite are fibre-matrix debonding and fibre pullout. It is therefore not surprising that the PPS composites show substantial impact

resistance than the unfilled PPS.

5.8 FIRE SMOKE AND TOXICITY (FST)

A wide variety of composite materials are being developed and evaluated for use in aircraft and automotive components. These include composites with high temperature thermoplastic polymers, thermosets and nanocomposites. Because of stringent legislation requirements, each of these advanced composites has to be evaluated for its fire worthiness.

The FST properties of the PPS hybrid composites that were developed could not be determined due to the unavailability of appropriate equipment such as the cone calorimeter within the country. However, PPS is known to be a semi-crystalline thermoplastic polymer, when reinforced with inorganic fibres and fillers, the resulting composite have been found to possess an inherent flame resistance with low smoke emission [17, 66].

The fire safety of enclosed and inescapable areas such as submarines, ships, high-rise buildings, and aircraft is of high priority. A major hazard with the use of many types of polymer composite materials in enclosed areas like aircraft cabins and structures is their flammability. When these composites are exposed to high temperatures above 300 – 500°C, the organic matrix decomposed with the release of heat, smoke, soot and toxic volatiles. Composites also soften, creep and distort when heated to moderate temperatures. The damage caused by the heat and flame can cause distortion, buckling and failure of load bearing structures. The heat, smoke and gases released by burning composites and the degradation in their structural integrity can jeopardise the safety of the aircraft or automotive or ship or high-rise building in which it is used. The susceptibility of many conventional composites to fire has been a key issue in many applications.

5.9 ACCEPTABILITY OF THE EXPERIMENTAL RESULTS

The reliability of the experimental results obtained during this research is a function of two aspects. The first aspect is a material property which depends on the material homogeneity and the fibre-matrix interfacial bonding. The second aspect depends on the experimentation process and the equipment used during the experimentation. If we assume that the homogeneity of the material is consistent and there is negligible variation in the properties of the material, then the reliability of the experimental results will depend on the process and equipment used during experimentation. Therefore, to analyse the reliability of the experimental results, a process of worst case scenario will be followed in each of the experimentation processes.

5.9.1 ANALYSIS OF THE TENSILE TEST RESULTS

During the tensile testing, five identical tests (X_i) were performed at each test speed and temperature. The average of the UTS's of the five identical tensile tests is represented by \bar{X} . The standard deviation (σ_x) for the five UTS values was also calculated. The test speed and test temperature were varied to include the different testing conditions into the statistical analysis. The test condition with the largest error band in the tensile test results is used in the analysis to determine the confidence level of the tensile test results.

The figure 5.1 below is the average UTS as a function of test speed at different test temperatures for the unfilled PPS. The bars with the largest error band are indicated with a ring. Table 5.1 is a summary of the individual tensile results including the average and standard deviations obtained for the three ringed bars on the graph.

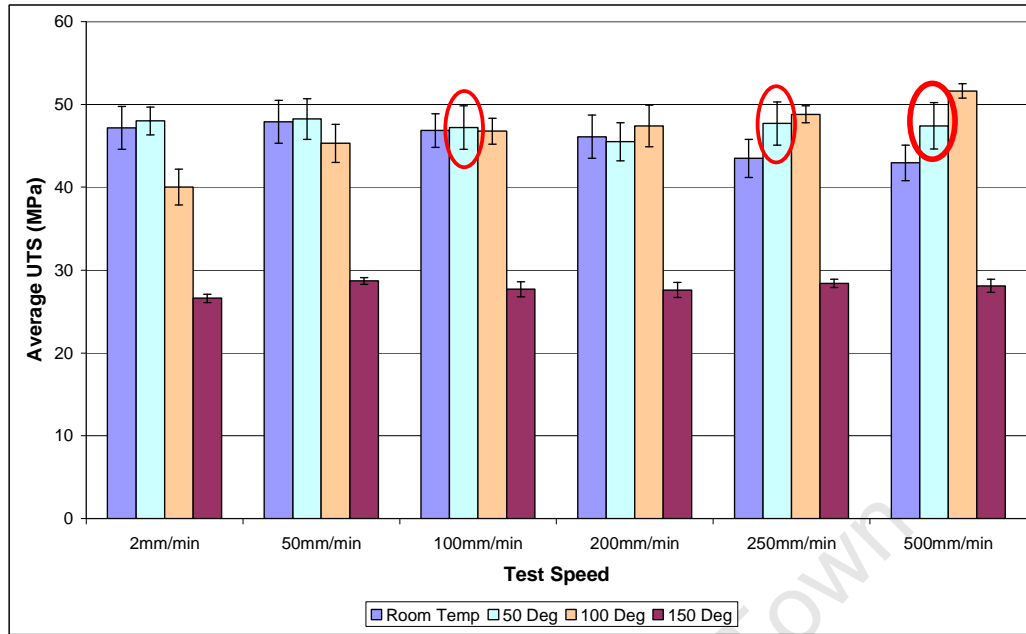


Figure 5.1 Average UTS versus test speed at different temperatures for unfilled PPS.

Table 5.1: Selected individual tensile test results at 50°C and different test speeds.

Tensile Test Number	UTS (MPa) at Different Test Speeds		
	100mm/min	250mm/min	500mm/min
X_1	44.6	48	46.7
X_2	50.6	44.3	52.3
X_3	44.7	51.6	47.1
X_4	48.8	47.5	45.3
X_5	47.4	47	45.5
\bar{X}	47.2	47.7	47.4
σ_x	2.61	2.62	2.85

To determine the acceptability of the experimental results, the average UTS is used as a measure of the expected result (\mathbf{X}_{exp}). The discrepancy between the expected value and the worst UTS value ($\mathbf{X}_{\text{worst}}$) is calculated from equation 5.1 below.

$$\text{Discrepancy} = \left| \mathbf{X}_{\text{worst}} - \mathbf{X}_{\text{exp}} \right| \quad 5.1$$

Since the data with the largest error band/standard deviation is used to determine the confidence level of the tensile tests, the 500mm/m test results are analysed since they show the largest standard deviation. The discrepancy value for the selected data would therefore be 4.92 MPa.

$$\mathbf{t} = \left\{ \frac{\left| \mathbf{X}_{\text{worst}} - \mathbf{X}_{\text{exp}} \right|}{\sigma_x} \right\} \quad 5.2$$

Equation 5.2 above is used to determine the number of standard deviations (\mathbf{t}) by which $\mathbf{X}_{\text{worst}}$ deviate from \mathbf{X}_{exp} . In the case of the data for the 500mm/m the calculated value for \mathbf{t} would be 1.73. Therefore using the table of normal error integral, the probability of obtaining a UTS value that differs from \mathbf{X}_{exp} by \mathbf{t} or more standard deviations can be found. This probability is given by equation 5.3 below;

$$\begin{aligned} \text{Probability (outside } \mathbf{t}\sigma_x) &= 1 - \text{Probability (within } \mathbf{t}\sigma_x) & 5.3 \\ 8.36\% &= 100 - 91.64\% \end{aligned}$$

It can therefore be stated that under the assumptions of complete homogeneity, excellent fibre-matrix interfacial bond of the materials and the Gaussian distribution of the test data, there exists a 91.64% probability that the next UTS value obtained would lie within the distribution of the average. Considering that this is the worst case scenario, the tensile data of the other test conditions would even show a higher probability of falling within the upper and lower limits of the data. Hence the equipments used and the experimentation process can confidently be used to determine the tensile properties of the different PPS materials.

5.9.2 ANALYSIS OF THE FLEXURAL TEST RESULTS

During the flexural testing, ten identical tests (X_i) were performed for each of the PPS materials. The average of the flexural strength of the ten identical tests was calculated and represented as \bar{X} . The standard deviation (σ_x) for the ten flexural strength values was also determined. The individual flexural results including the mean and standard deviations obtained for the different PPS materials are recorded in table 5.2 below.

Table 5.2: Flexural text results of the different PPS materials

Flexural Test Number	Flexural Strength (MPa) of PPS and its Composites			
	Unfilled PPS	622 PPS	631 PPS	40% GF PPS
X_1	123	182	223	238
X_2	113	184	221	240
X_3	112	178	213	242
X_4	105	186	223	239
X_5	114	187	219	241
X_6	106	183	229	240
X_7	122	188	212	244
X_8	110	190	215	247
X_9	107	181	216	244
X_{10}	105	185	213	237
\bar{X}	112	184	218	241
σ_x	6.7	3.5	5.4	3.2

The test result with the largest standard deviation is used to determine the confidence level of the flexural test results. The data for the unfilled PPS is analysed since it shows the largest standard deviation relative to the others. To determine the confidence level of the flexural test results, the mean of the flexural strengths is used as a measure of the expected result (X_{exp}). Equation 5.2 is used to calculate the number of standard deviations (t) by which X_{worst} deviate from X_{exp} . From the selected data, t was found to be 1.69.

From the table of normal error integral and using equation 5.3 the probability of obtaining a flexural strength value that differ from X_{exp} by t or more standard deviations is 9.1%

Based on the above assumption and making use of the Gaussian distribution of the test data, the probability that the next flexural strength would lie within the distribution of the average is 90.9%. Considering that this is the worst case scenario, the flexural data of the other PPS composites would even show a higher probability of falling within the upper and lower limits of the data. Hence the equipment and the process of experimentation can confidently be used to determine the flexural properties of the different PPS materials.

5.9.3 ANALYSIS OF THE IMPACT TEST RESULTS

The Izod and Charpy impact tests were conducted on each of the PPS materials. In each case, five notched and five un-notched samples were tested. The impact resistance of the five identical tests (X_i) were recorded along with their average (\bar{X}) and standard deviation (σ_x). Figure 5.2 is the average impact resistance of the different PPS materials. The test condition with the largest error band is used to determine the confidence level of the impact test results. The individual impact test results including the mean and standard deviations for the ringed bars on the graph are tabulated in table 5.3.

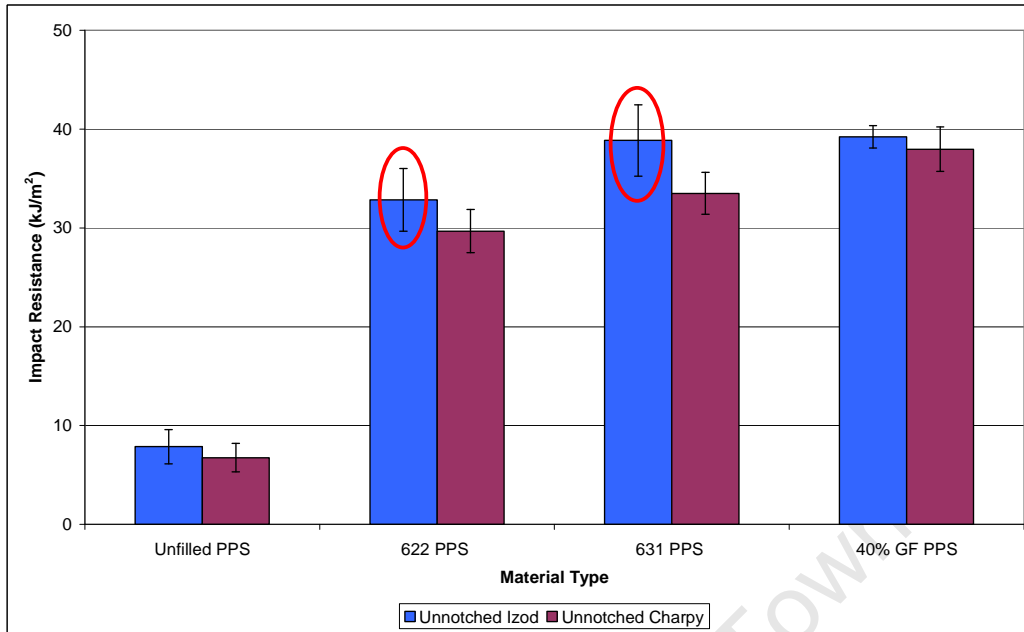


Figure 5.2: Izod and Charpy impact resistance on un-notched specimen

Table 5.3: The Izod impact resistance of un-notched PPS hybrid composites

Impact Test Number	Izod Impact Resistance (kJ/m ²)	
	Un-notched 622 PPS	Un-notched 631 PPS
X_1	36.5	40.2
X_2	27.9	36.2
X_3	32.1	35.1
X_4	33.6	44.2
X_5	34.1	38.6
\bar{X}	32.8	38.9
σ_x	3.18	3.51

To determine the confidence level of the impact test results the average of the impact results is used as a measure of the expected value (X_{exp}). Equation 5.2 is used to determine the number of standard deviations (t) by which the worst impact value (X_{worst}) deviate from X_{exp} . From the selected data, the value of t was calculated to be 1.53. From the table of normal error integral and using equation 5.3, the probability of

obtaining an impact resistance that differ from X_{exp} by t or more standard deviations is 12.6%.

Similarly, based on the assumptions and making use of the Gaussian distribution of the test data, the probability that the next impact resistance would lie within the distribution of the average is 87.4%. Since this is the worst case scenario, the impact resistance data of the other test conditions would show an even higher probability of falling within the upper and lower limits of the experimental data. Therefore, the equipment and the experimentation process can be confidently be used to determine the impact properties of the PPS materials.

5.10 MECHANICAL PROPERTY ANALYSIS

In this study two thermoplastic hybrid composites based on PPS matrix has been developed. It is important to note that once a material is developed, mechanical testing is done to obtain the first generation of mechanical property data for the new material. The mechanical characterisation through experimentation has been done. The mechanical properties and cost of these PPS hybrid composites are being compared with 30% glass fibre PEI, 40% glass fibre/25% $CaCO_3$ hybrid PPS and 30% glass fibre PAI composites that are on the market at the moment. Given the importance of mechanical properties in the characterisation of a material, a remark is made that a conclusion can only be drawn from the results obtained for the PPS hybrid composites developed in this research. The mechanical properties of these composites are summarised in tables 5.4 and 5.5 below.

It can be seen that mechanical properties of the PPS hybrid composites developed in this research compare favourably well with the 30% glass fibre PES and the 40% glass fibre/25% $CaCO_3$ hybrid PPS composites that are on the market at the moment. In the case of tensile and flexural strength, the new PPS hybrid composites performed better than the 30% glass fibre PES and the 40% glass fibre/25% $CaCO_3$. The 30% glass fibre PAI has superior properties than all the composites under discussion here.

Table 5.4: Mechanical properties and cost of the PPS hybrid composites along with other composites on the market [66, 67, 69, 100, 117].

Composite Materials	Tensile Properties (MPa)		Flexural Props. (MPa)		Cost \$/kg[100]
	Strength	Modulus	Strength	Modulus	
622 PPS Hybrid	138 – 144	3398 – 3597	184	9380	9.10
631 PPS Hybrid	153 – 163	3829 – 4315	218	11200	9.18
40% GF PPS	175 – 196	4325 – 4786	241	13610	11.46
40% GF PPS*	195	–	280	14000	11.46
[66]	137	9800	190	8800	11.57
30% GF PES [69]					
40% GF/25%	130	19000	210	18800	–
CaCO ₃ PPS [117]	205	10800	338	11700	55.12
30% GF PAI [67]					

Table 5.5: Impact properties of the PPS hybrid composites along with other composites on the market [66, 67, 69, 117].

Composite Materials	Izod Impact Strength (kJ/m ²)		Charpy Impact Strength (kJ/m ²)	
	Notched	Un-notched	Notched	Un-notched
622 PPS Hybrid	7.8	32.8	7.5	29.7
631 PPS Hybrid	9.1	38.9	8.2	33.5
40% GF PPS	9.3	39.2	7.6	38
40% GF PPS* [66]	9	35	–	–
30% GF PES [69]	–	90 J/m	–	–
40% GF/25%				
CaCO ₃ PPS [117]	6	20	7	20
30% GF PAI [67]	–	79 J/m	–	–

* Mechanical property data given by the supplier of the PPS compounds.

The anisotropy and inhomogeneity of composite materials make the characterisation of their engineering properties a complex issue. According to Wilson and Carlsson [84], the mechanical properties of advanced composite material systems are determined by specially designed test methods. These test methods are mechanically simple in concept but are extremely sensitive to specimen preparation and test execution procedures. The mechanical properties of the 40% glass fibre PPS that was processed and determined in this research is in agreement with that of the same composite provided by the supplier. This also gives an indication that the mechanical properties of the hybrid composites developed in this study can be confidently depended on.

5.11 COST ANALYSIS

The cost of a composite component is influenced by both material cost and fabrication method. The cost comparison of the hybrid composite materials and the other composites are listed in table 5.4. At first glance, the hybrid composites look much cheaper than the other composites. However, the cost quoted here is purely that of materials used to develop the hybrid composites. The cost of compounding and processing is not included in the above cost of the hybrid. In spite of that the hybrid composites developed in this research would be cost effective compared to the 30% glass fibre PEI and the 40% glass fibre/25% CaCO₃ PPS for the same application because the mechanical properties of the hybrid composites compare favourably well with those of 30% glass fibre PEI and the 40% glass fibre/25% CaCO₃ PPS.

According to Mallick [10], one major obstacle to the widespread use of advanced composites is their cost. Although composite applications in the aerospace and automobile industries have increased significantly in recent years, the high cost is still a major concern. The high cost of fibres is not the only reason for the high cost of advanced composites. The cost of matrix can also be very high, particularly for high temperature resins. For the total material cost, one must also include the cost of incorporating the fibres into the matrix as well as processing and fabrication cost.

The automotive and aerospace industries placed more emphasis on endurance, reliability, ease of manufacture and lower cost, in addition to weight savings. Therefore, the development of polymer based hybrid composites to reduce cost and weight through rational design is the way to go. Many countries consider aerospace a strategic industry for reasons of national security, economic strength and technological advancement. It is therefore proper for South Africa to seek a competitive position in aerospace. Composites in general and hybrid composites in particular considered high-tech materials will play an important role in achieving this competitive edge in aerospace. The aerospace industry can be compared to the automotive industry in the race to achieve greater affordability. These industries as well as composite manufacturers are focusing on cost. Initial cost is only a part of the equation as the emphasis is shifting to life cycle costs. The superiority of composite materials is seen when their inherent advantages are used, such as their very low assembly cost due to the high potential for parts consolidation and their significantly lower life-cycle costs. According to Parker [5], OEMs require composite component to be manufactured with cost reduction of up to 30% over the production lifecycle.

CHAPTER SIX

CONCLUSIONS

The principal objective of this study has been to investigate the feasibility of developing an advanced hybrid composite based on a thermoplastic matrix. From the procedure that was followed in this study through to the experimentation and analyses of the results obtained, the following conclusions can be drawn:

- The current thermoplastic materials that dominate the advanced thermoplastic matrix composite industry are PEEK, PPS, PAI, PES, PEI, and PEKK. Out of these polymers PEEK, PPS and PEI reinforced with either glass or carbon fibres are the most widely known and used materials. This is because their mechanical properties have been well characterised and they have favourable resistance to environmental agents such as solvents, hydraulic fluids and fuel. PEKK on the other hand exhibits excellent properties but its application has progressed at a slower rate.
- The thermal stability of the PPS resin was found to be as high as 470°C. The PPS resin remained stable during thermal analysis until above 470°C before it starts to degrade.
- As a result of fibre/filler reinforcement, the mechanical properties of the PPS composites remarkably changed. The load bearing capability of the PPS composites were observed to be higher than that of the unfilled polymer. Addition of short glass fibres enhanced the tensile strength and modulus of the PPS composites. The presence of talc in the PPS hybrid composites improved the dimensional stability, controlled the mould shrinkage and reduced the cost and moulding cycles of the materials. The introduction of glass fibres and talc filler resulted in an enhancement of the failure strain of the composite.

- At the temperature and test speeds used, the PPS composites appear to be more sensitive to test speed changes than the unfilled polymer. This can be attributed to the sluggishness of the molecules to uncoil from the bulk material. Test speed changes seem not to have any significant effect on the elastic modulus of the PPS and its composites.
- The effect of temperature on the strength performance of the composites is very obvious. Increasing the test temperature resulted in a decrease in strength of all the PPS materials that were investigated. With an increase in temperature, the strength reduction of the composites is large and the strength at 150°C is less than half of the value at room temperature.
- The flexural strength and modulus of the PPS and its composites are higher than the tensile strength and modulus. The flexural strength of the composite with 30% glass fibres and 10% talc (631 PPS hybrid) is comparable to that of the 40% glass fibre filled.
- The incorporation of rigid fillers into brittle matrices can also produce a significant toughening effect. The impact resistance of these composites increased with increasing glass content. However, the impact resistances of the composite with 30% glass fibres and 10% talc (631 PPS hybrid) and the 40% GF PPS composites are comparable in both the notched and the un-notched conditions.
- The failure mode in the composites evaluated changes from brittle failure with fibre pullout at quasi-static cross-head speeds, to brittle failure with a considerable matrix damage preceding final fracture as the cross-head rates increases from intermediate to high. The composites exhibited a brittle failure mode with fibre pullout, fibre fracture and in most cases matrix shear failure.

- The short term creep testing revealed that the PPS materials are generally creep resistant. The tensile creep testing of the composite specimens showed that the 40% GF PPS is more creep resistant than the hybrid composites under the testing conditions, although their creep behaviour is similar.
- Polymer matrix composites reinforced with glass, carbon, or aramid fibres are established aerospace and automobile materials. The technology has advanced to the point where it is possible to design and fabricate efficient and reliable high performance structures. The durability of production components has been at least as good as their metal counterparts. The development of hybrid composites has demonstrated that optimum tailoring of composite structures can result in the mechanical property enhancement as a result of appropriate fibre selection and combination.



CHAPTER SEVEN

RECOMMENDATIONS FOR FUTURE WORK

The following recommendations are made with the anticipation that a further investigation into the hybrid composites developed in this study would extend its potential.

- Although PPS is known to have excellent fire resistance, it will be appropriate if FST testing could be done on the materials to determine its fire hazard reaction properties such as heat release rate, time to ignition, flame spread rate as well as smoke density and yield of carbon monoxide. This is because these properties influence the temperature and spread of fire as well as determining the survivability. This in turn determines the fire worthiness of the PPS and its composites.
- An in-depth creep testing under various conditions of temperature and stresses be undertaken to study the creep properties of the PPS and its composites materials. The data obtained from such tests could be use to develop a mathematical model to predict the creep behaviour of the PPS and its hybrid composites as well as the 40% PPS composite.
- Additional mechanical and physical tests other than those performed in this research could be carried out in order to investigate the composites response to properties such as fatigue and moisture absorption.
- A further development of a hybrid composite with high loading of different fibres/fillers combinations could be investigated to study how it would influenced the strength, surface properties, dimensional stability and overall cost of the composite.

CHAPTER EIGHT

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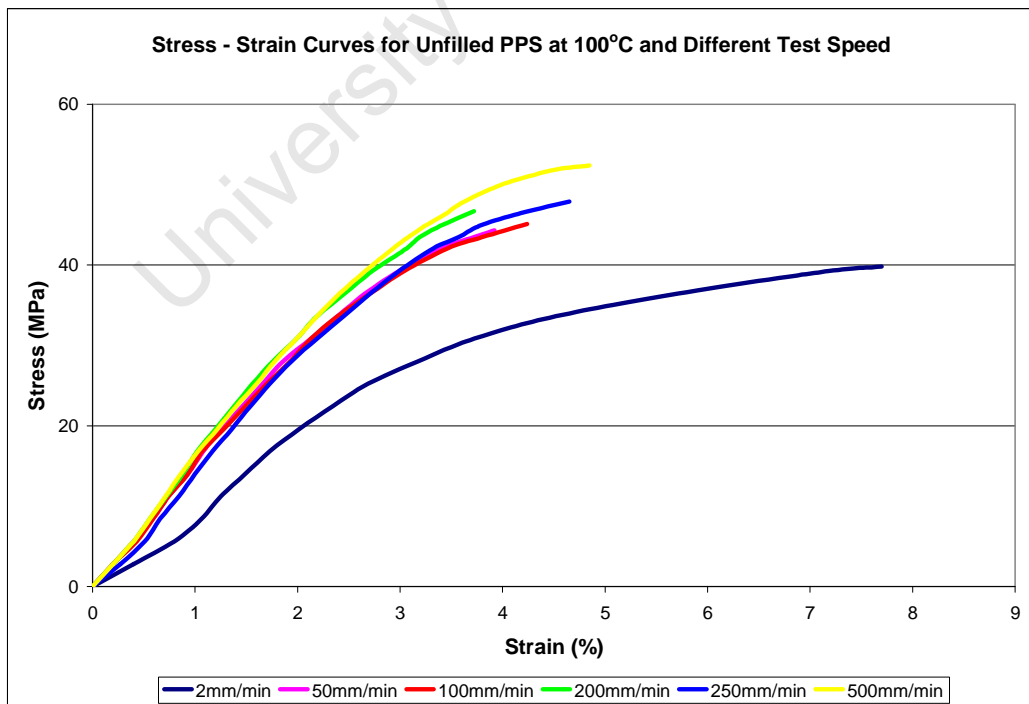
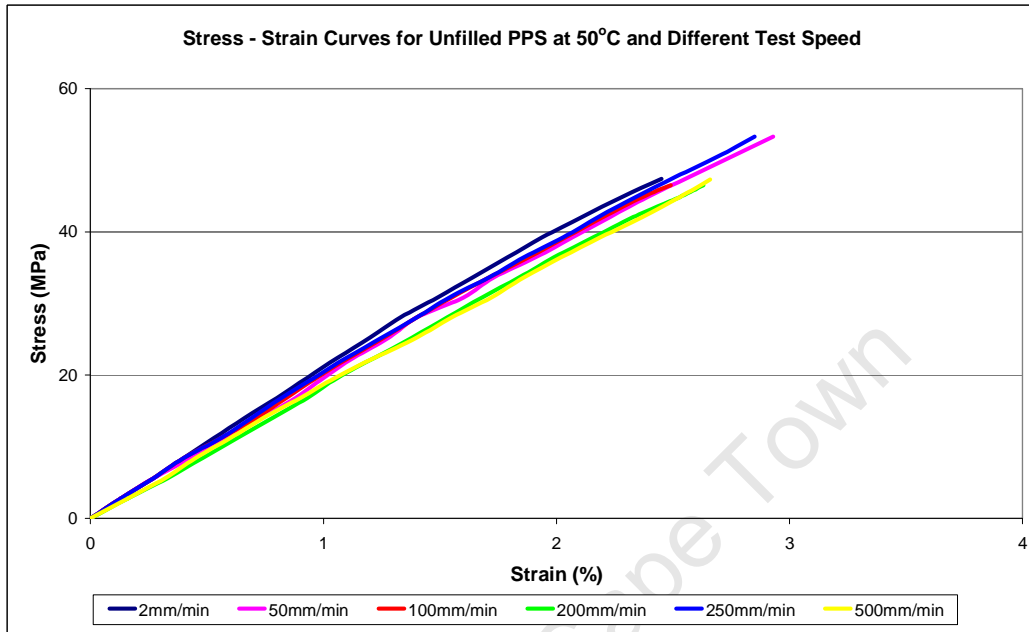


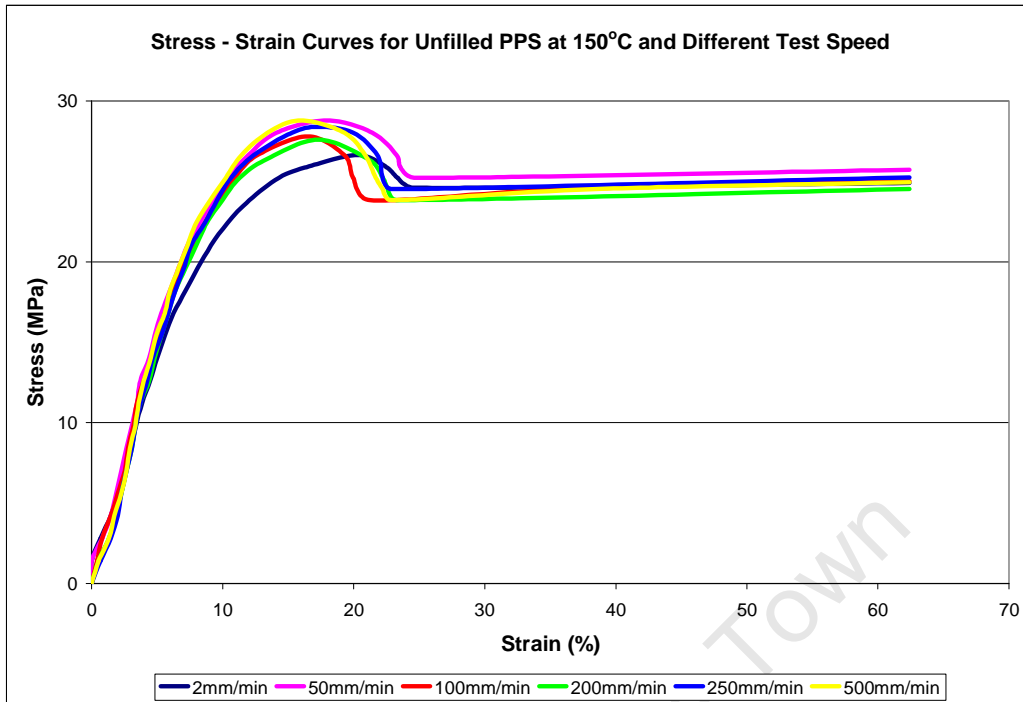
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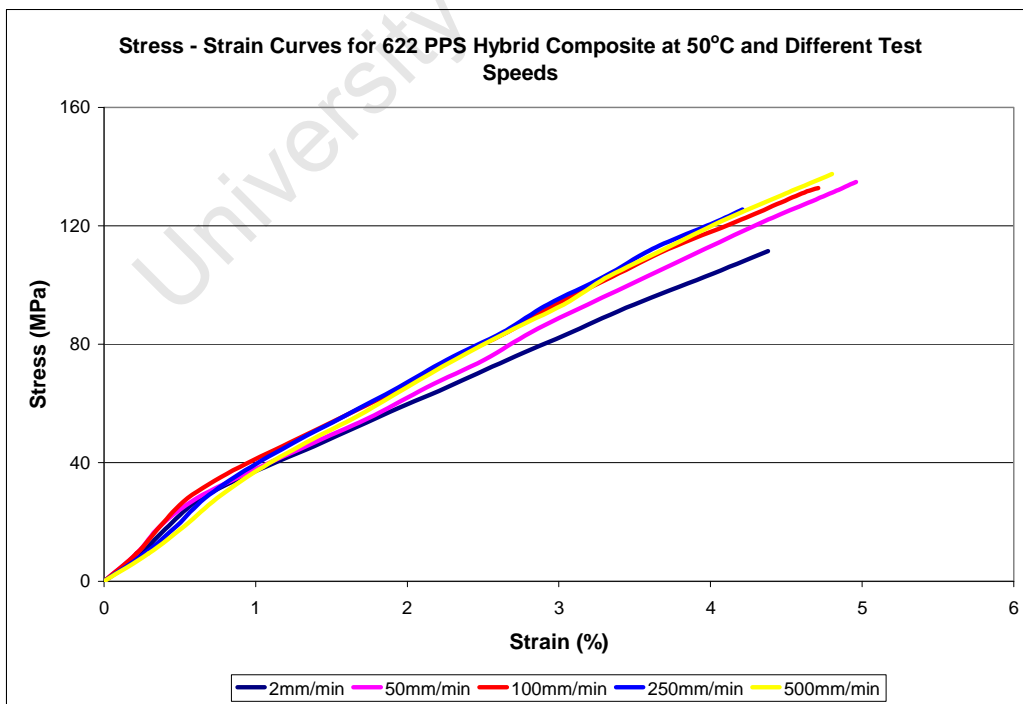
TENSILE STRESS-STRAIN CURVES FOR THE PPS COMPOUNDS

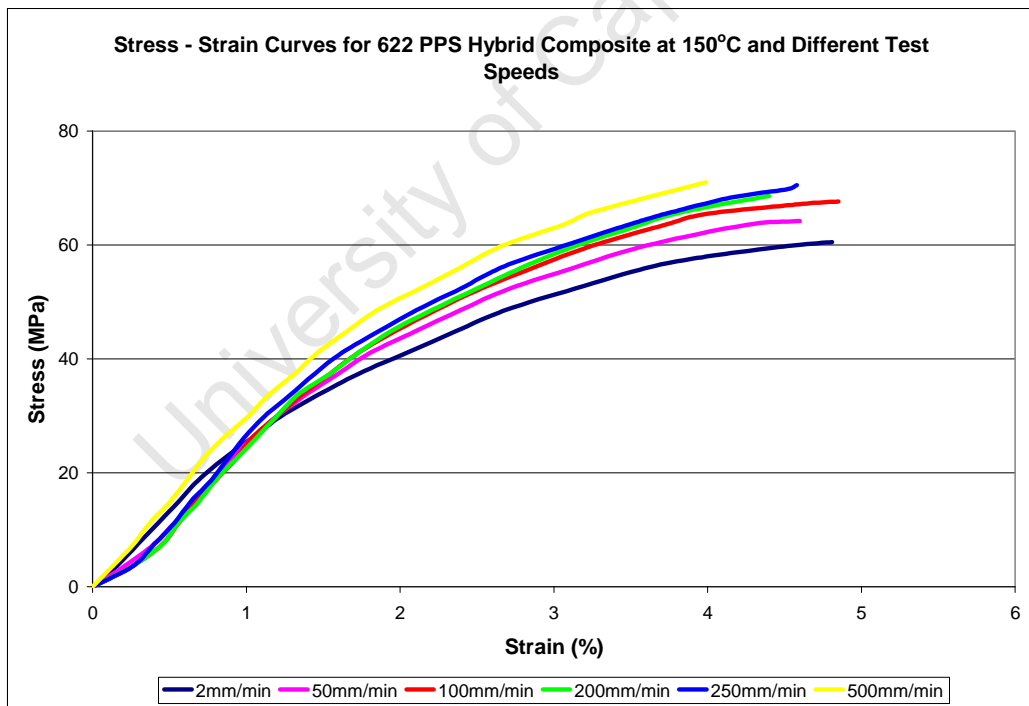
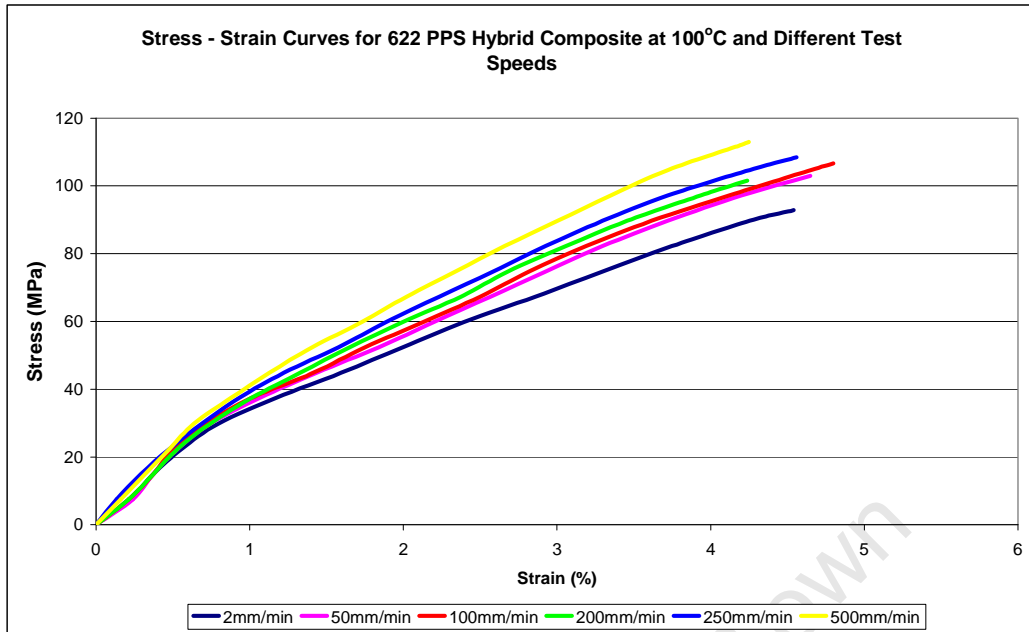
Unfilled PPS at a Particular Temperature and Different Test Speed



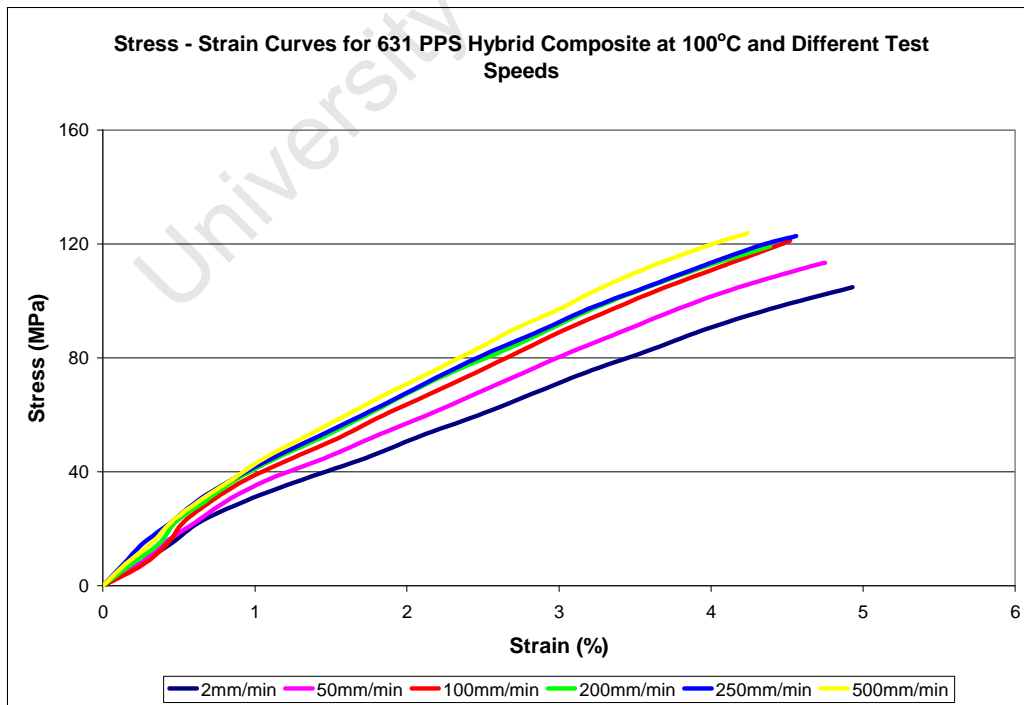
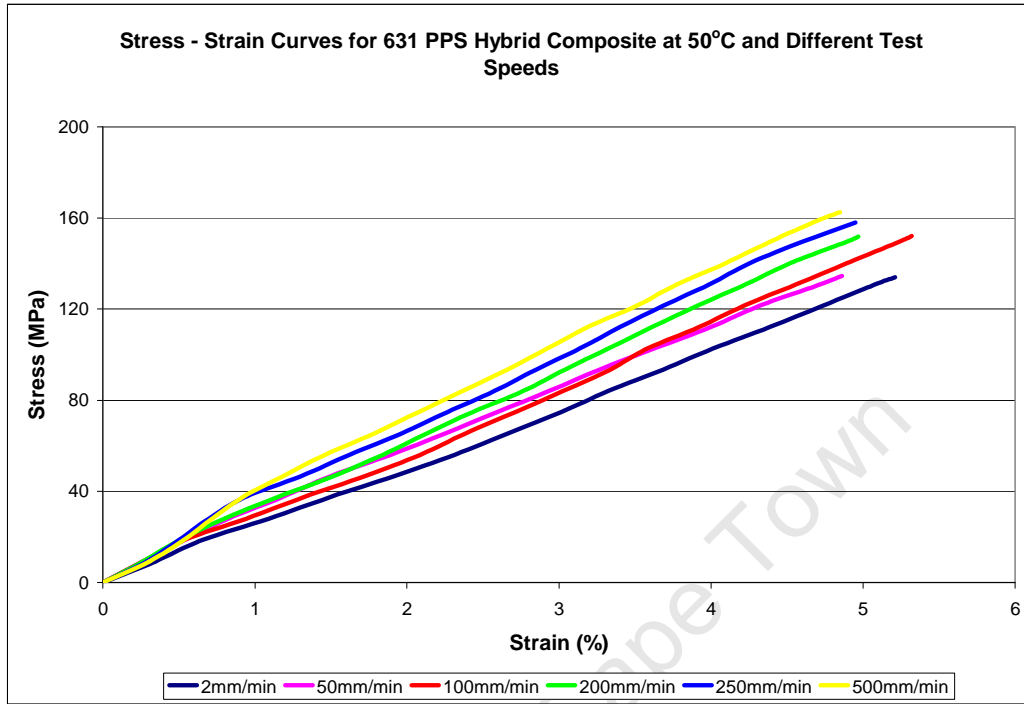


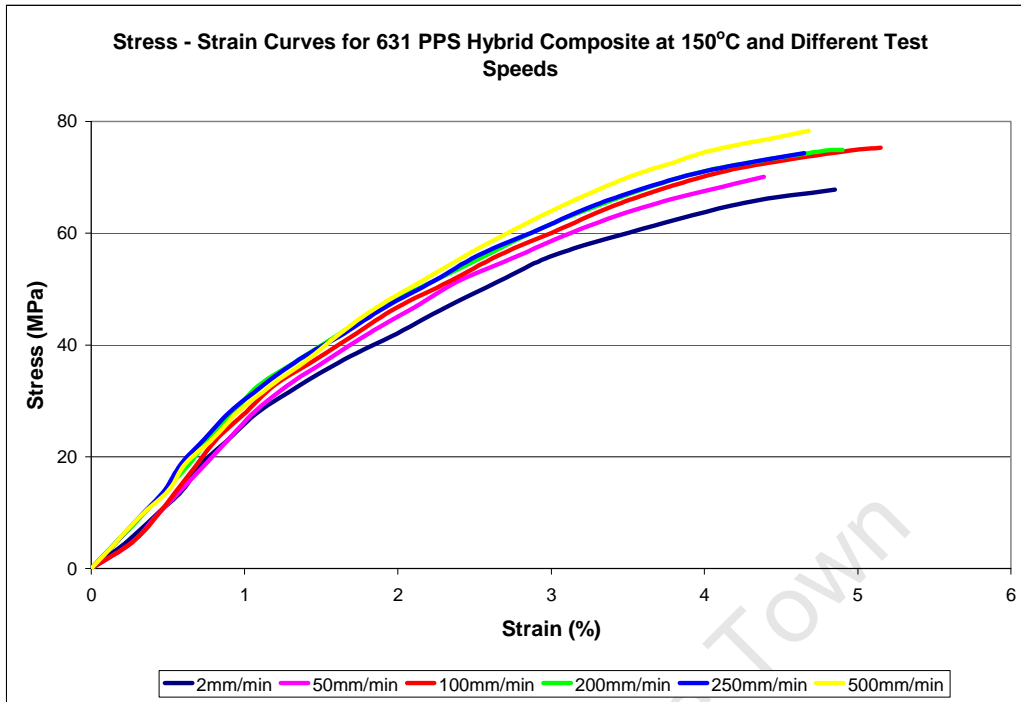
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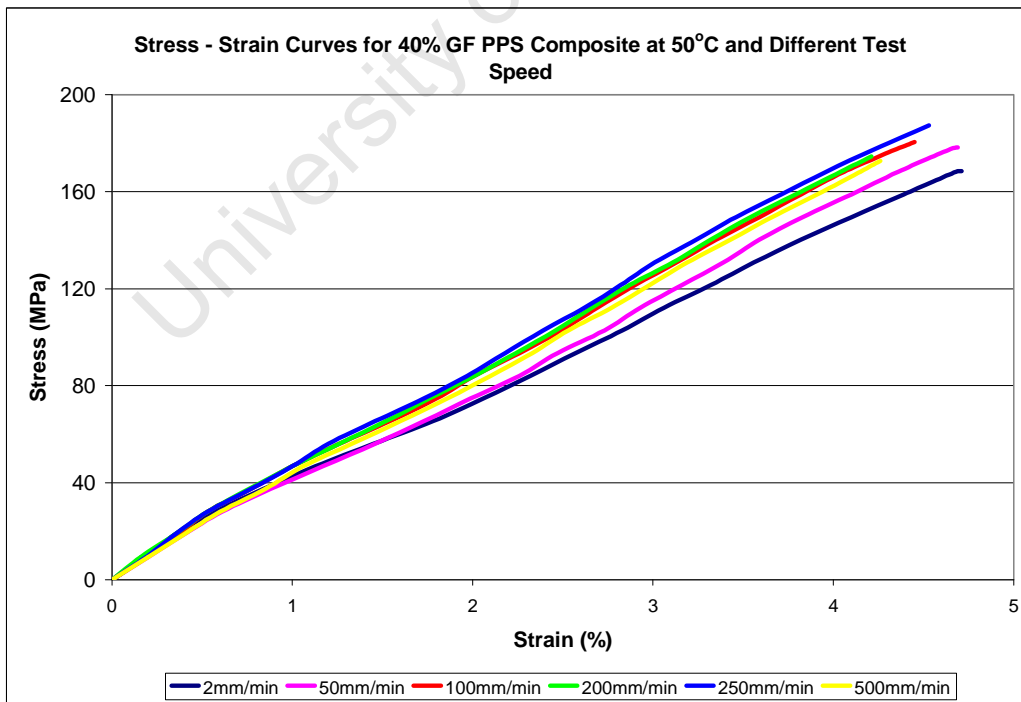


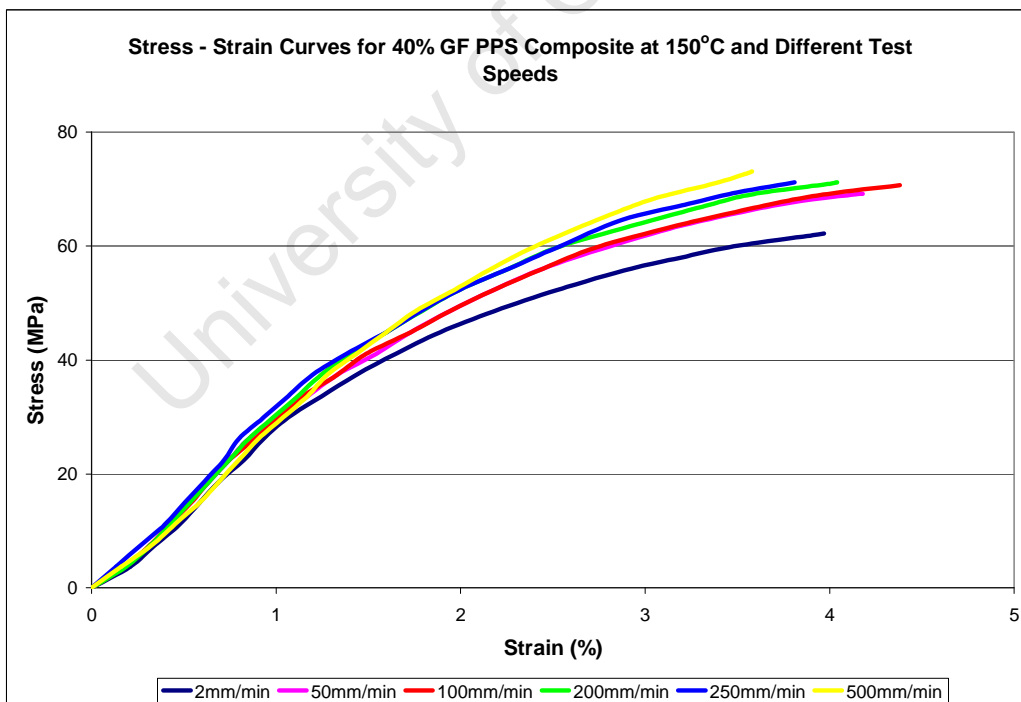
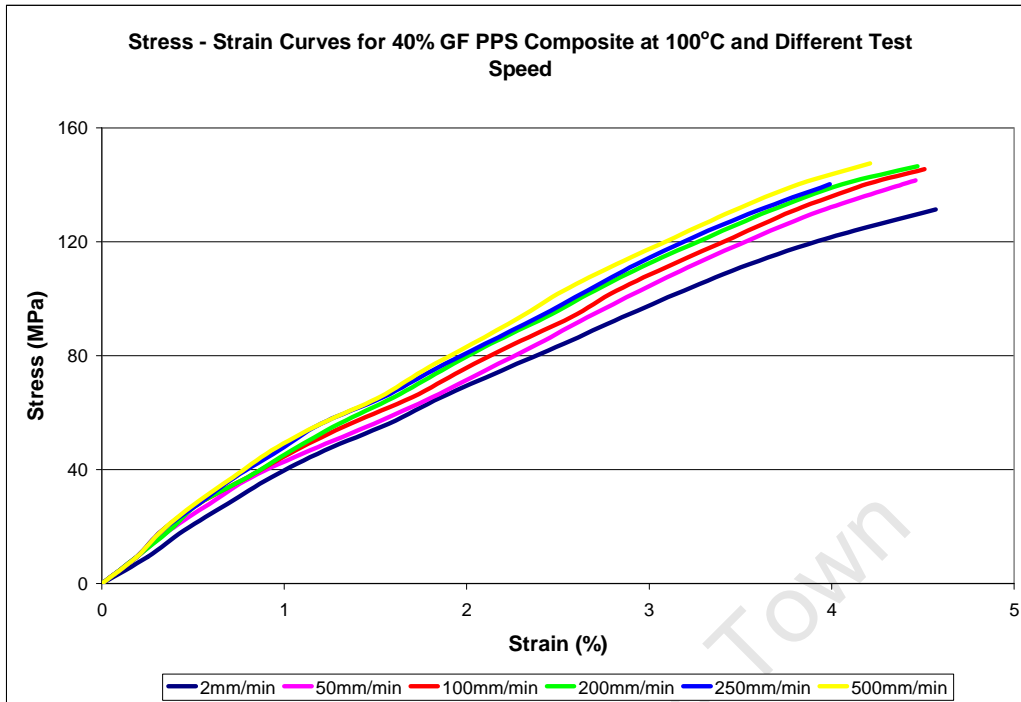
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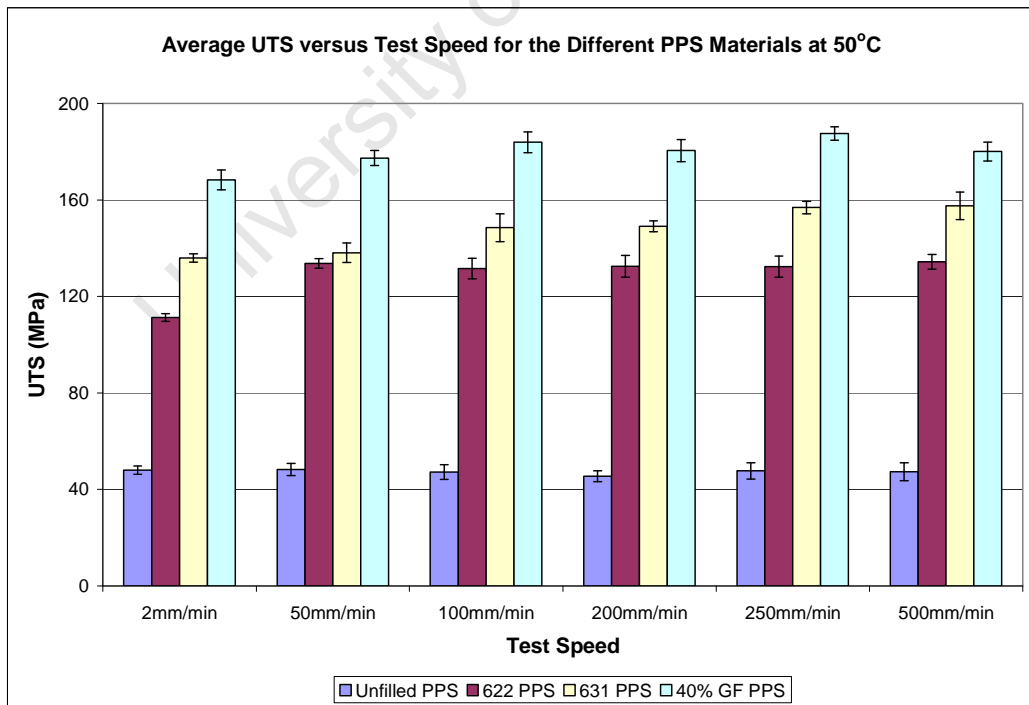
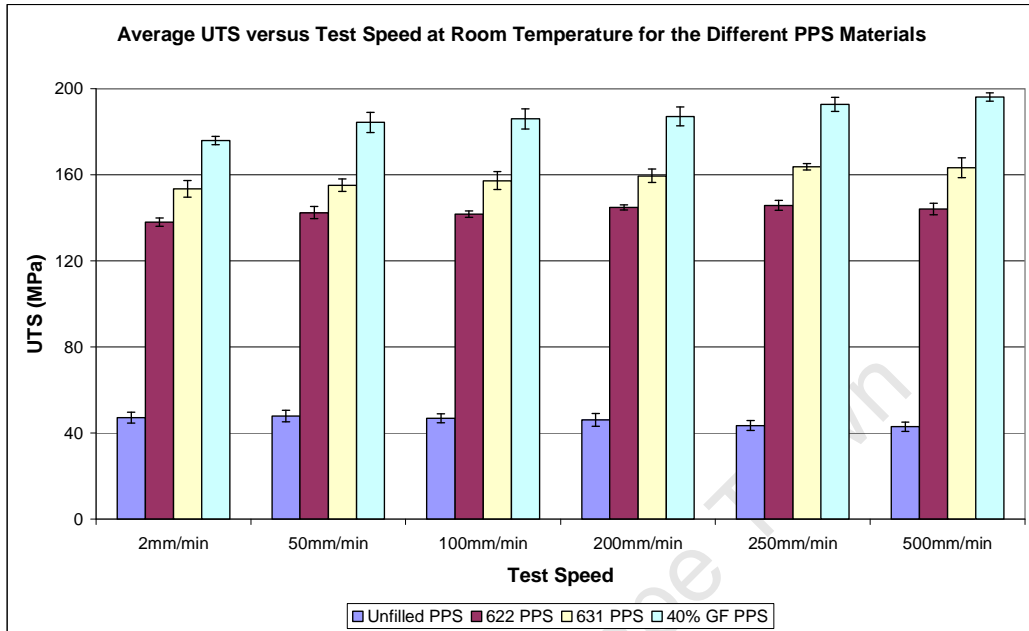


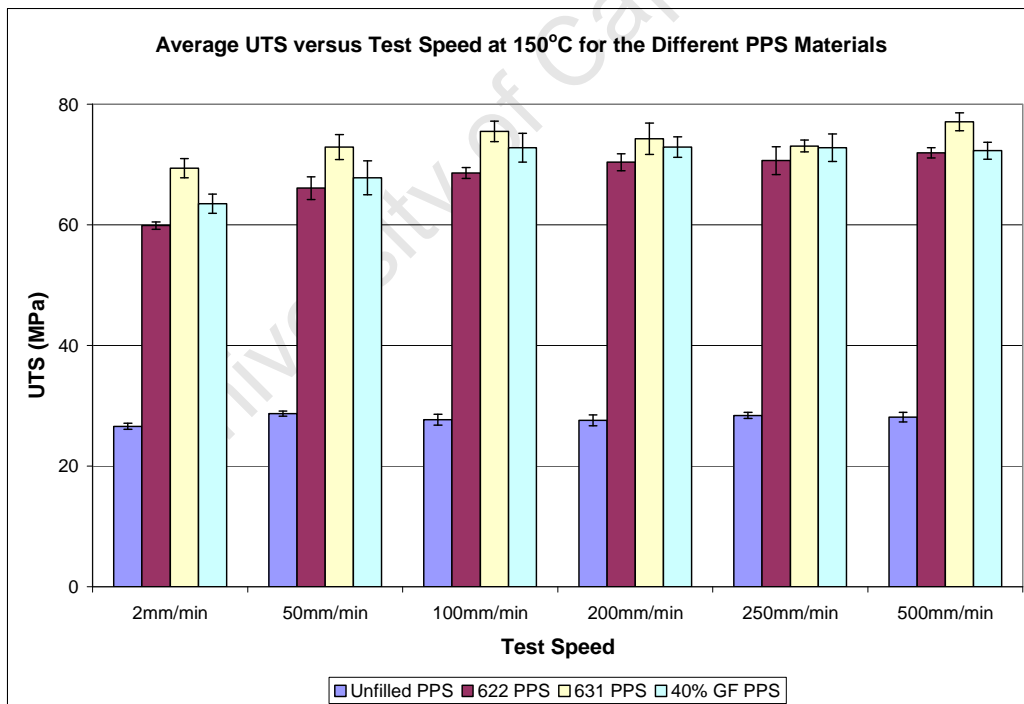
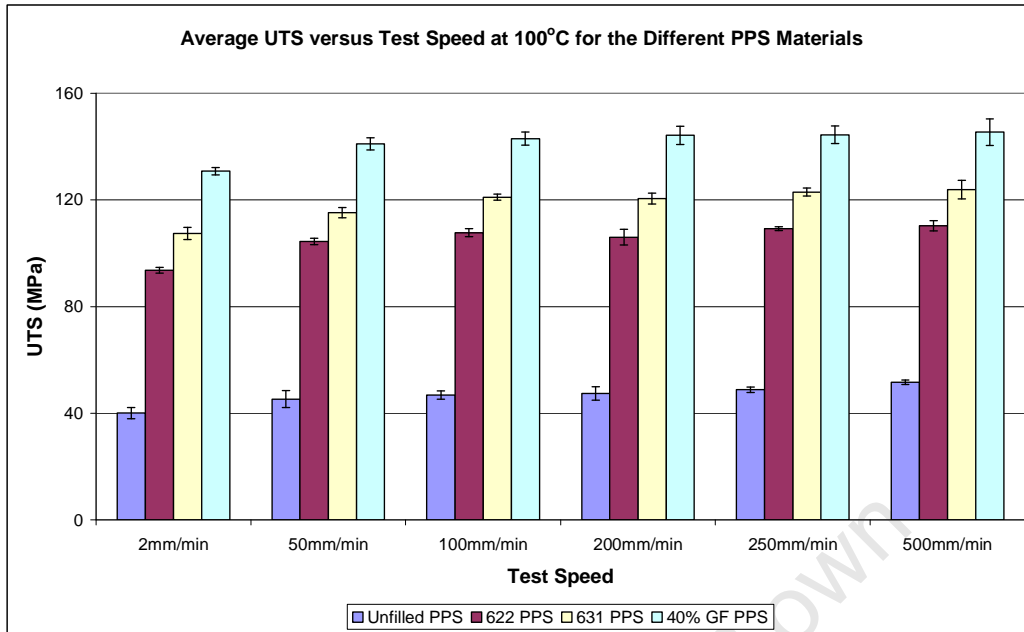
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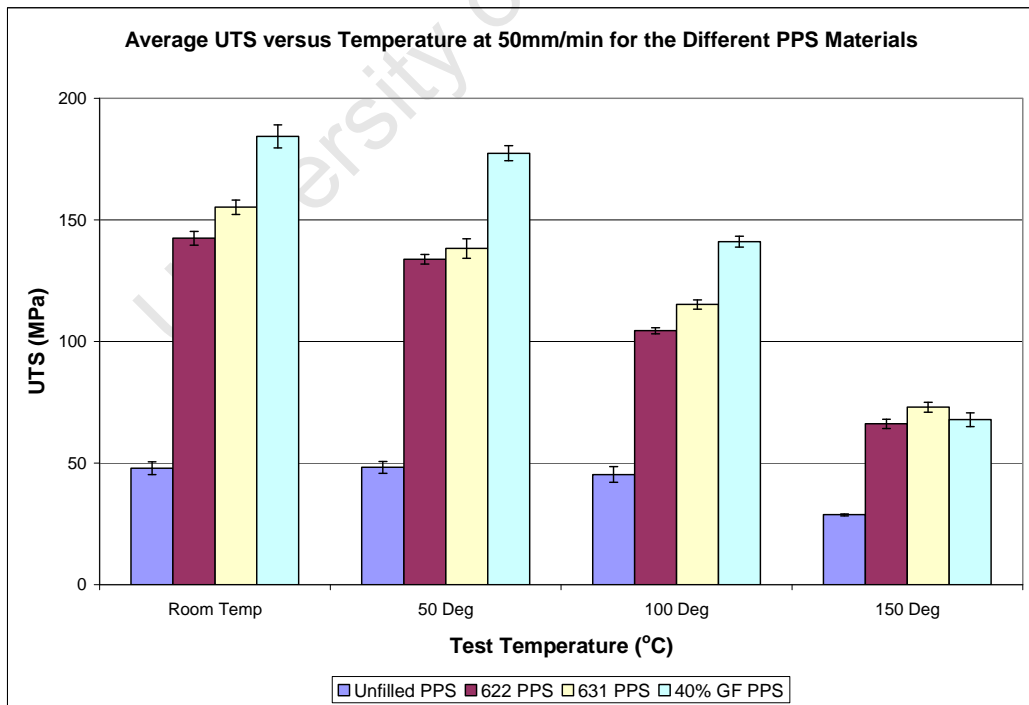
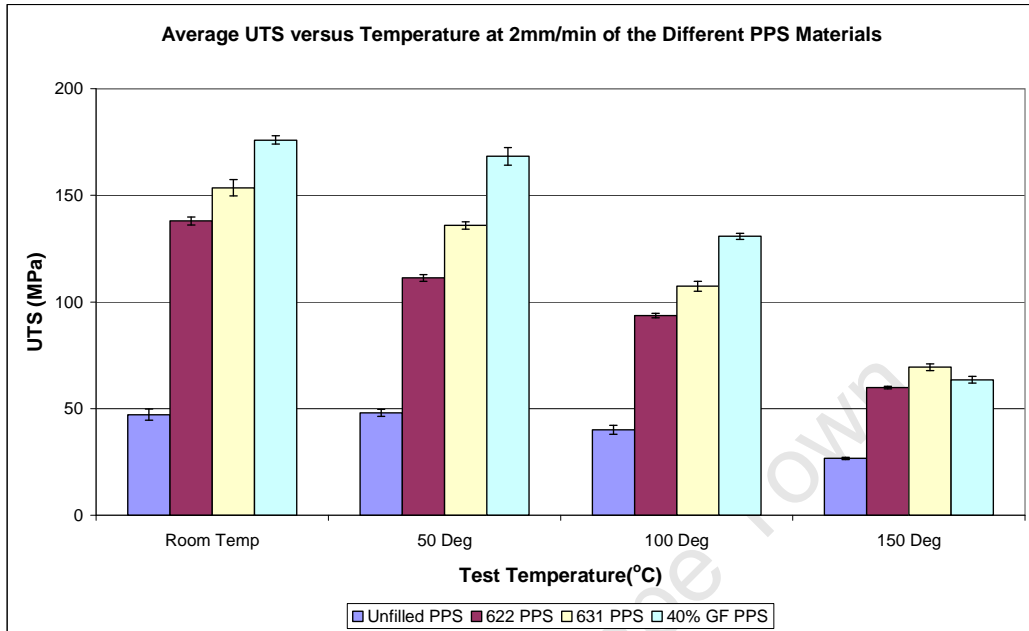


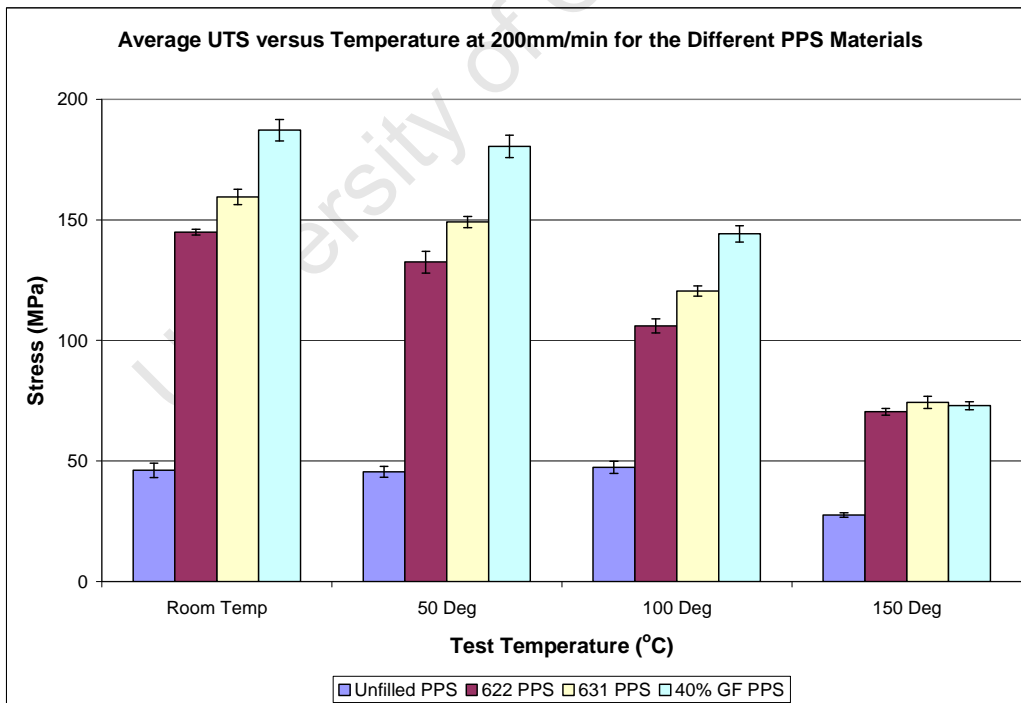
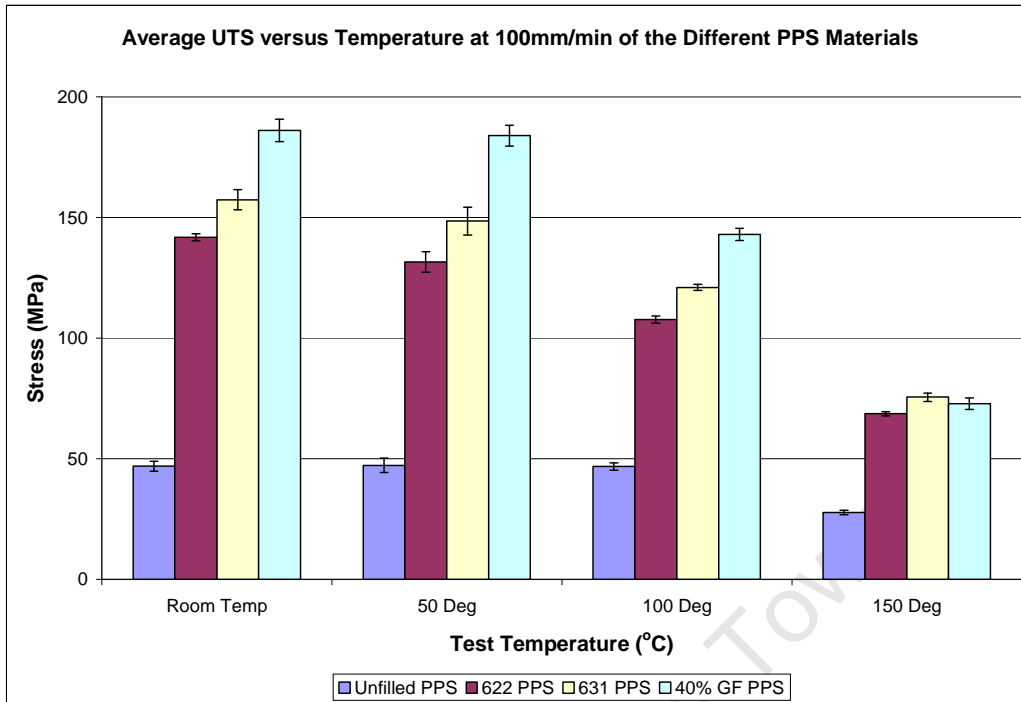
AVERAGE UTS AGAINST TEST SPEED AT DIFFERENT TEMPERATURES FOR THE PPS COMPOUNDS

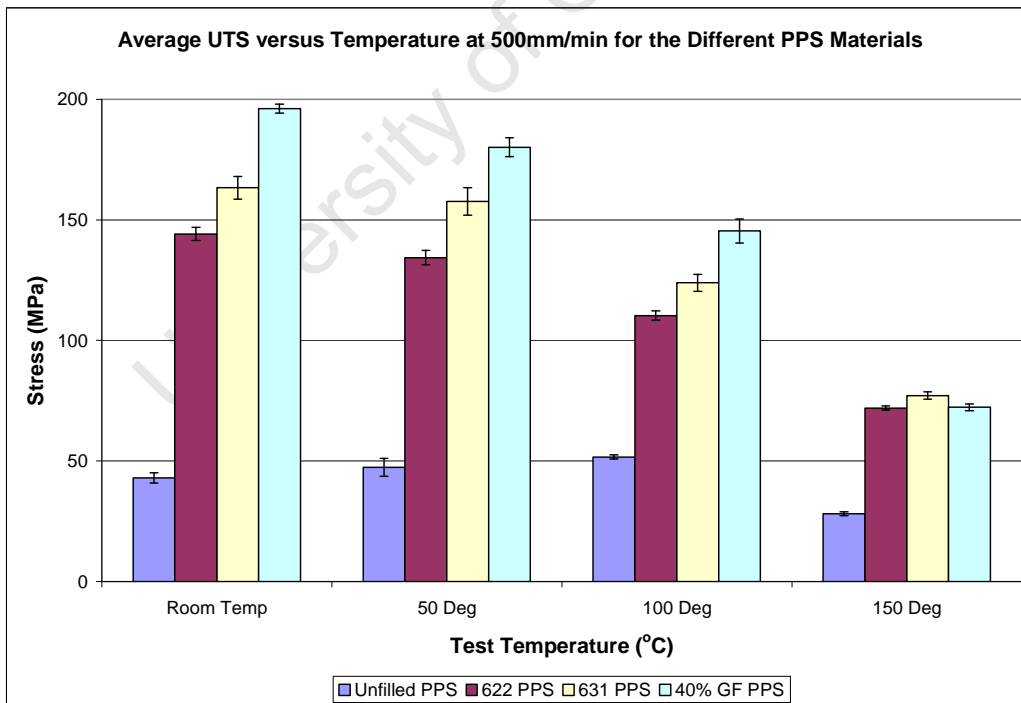
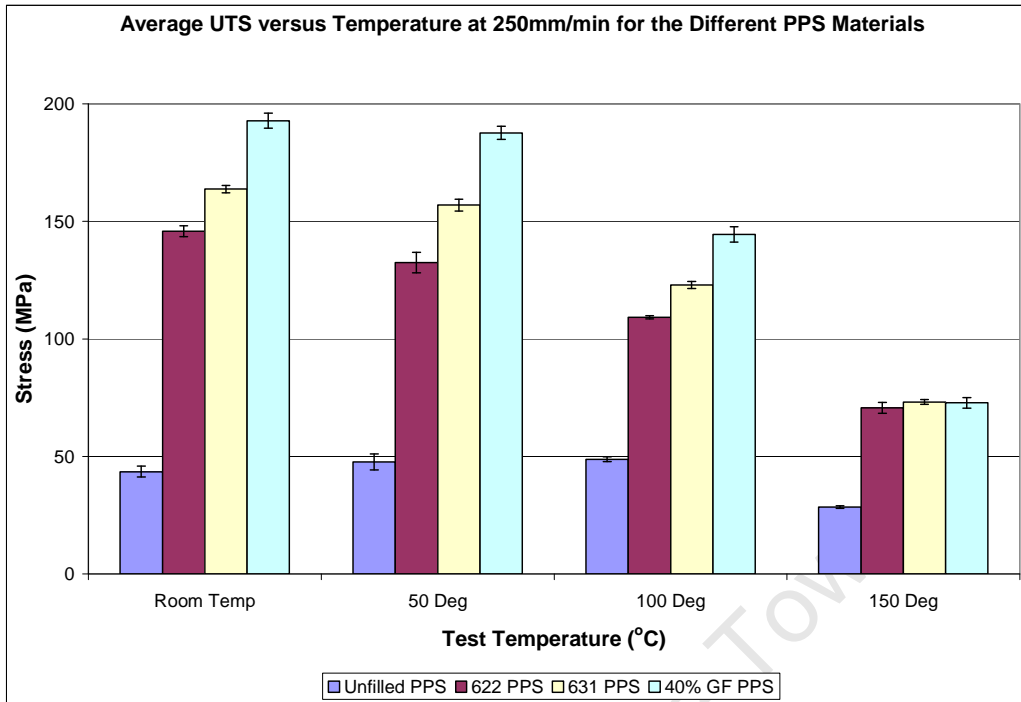




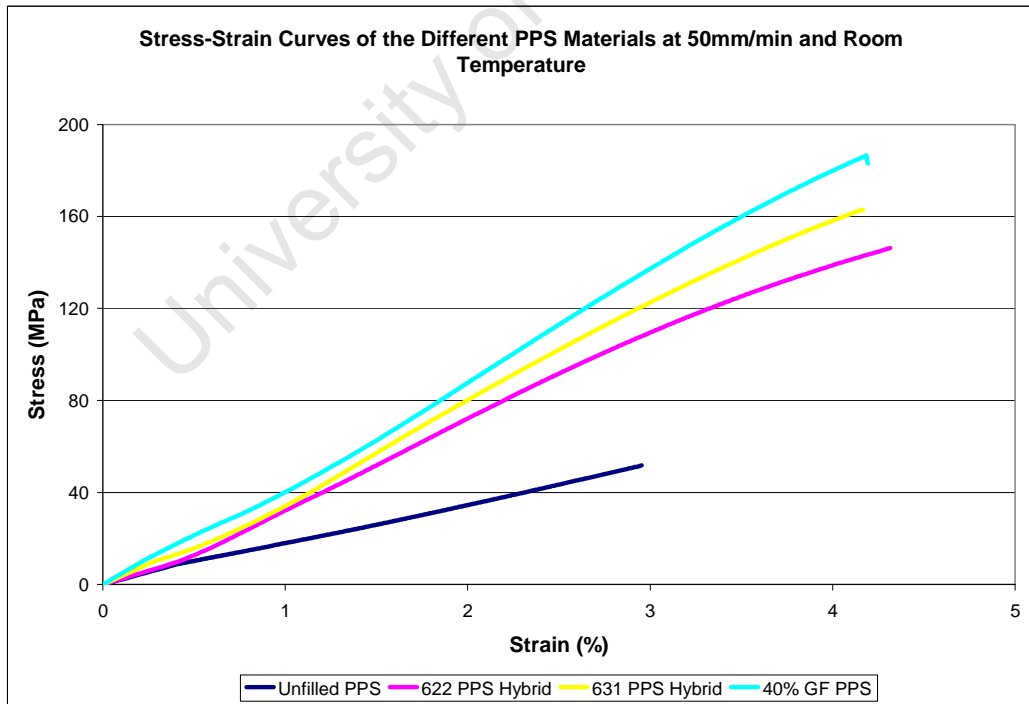
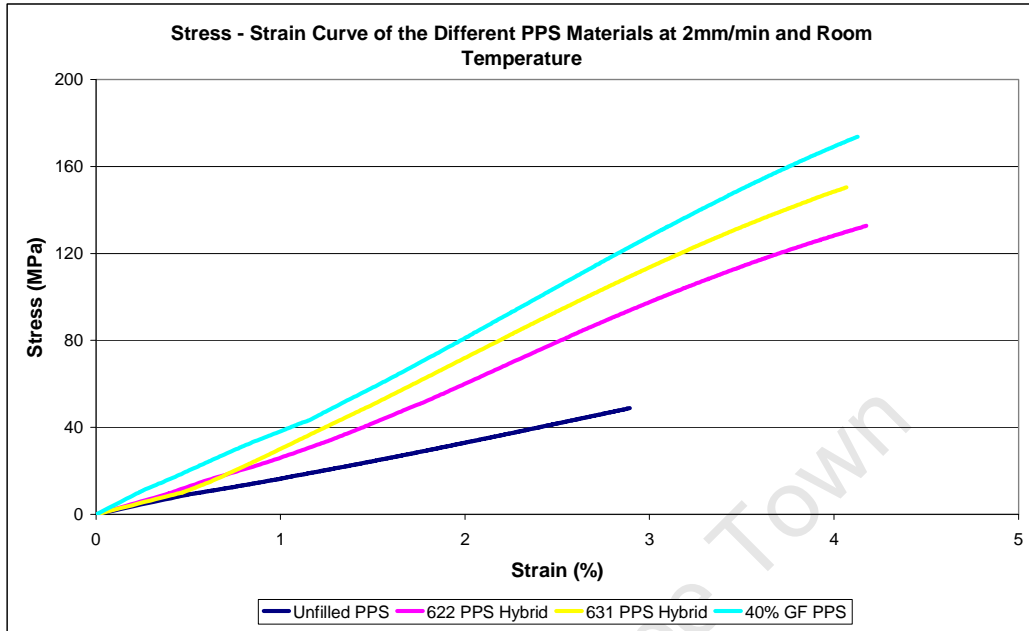
AVERAGE UTS AGAINST TEMPERATURE AT DIFFERENT TEST SPEEDS FOR THE PPS COMPOUNDS

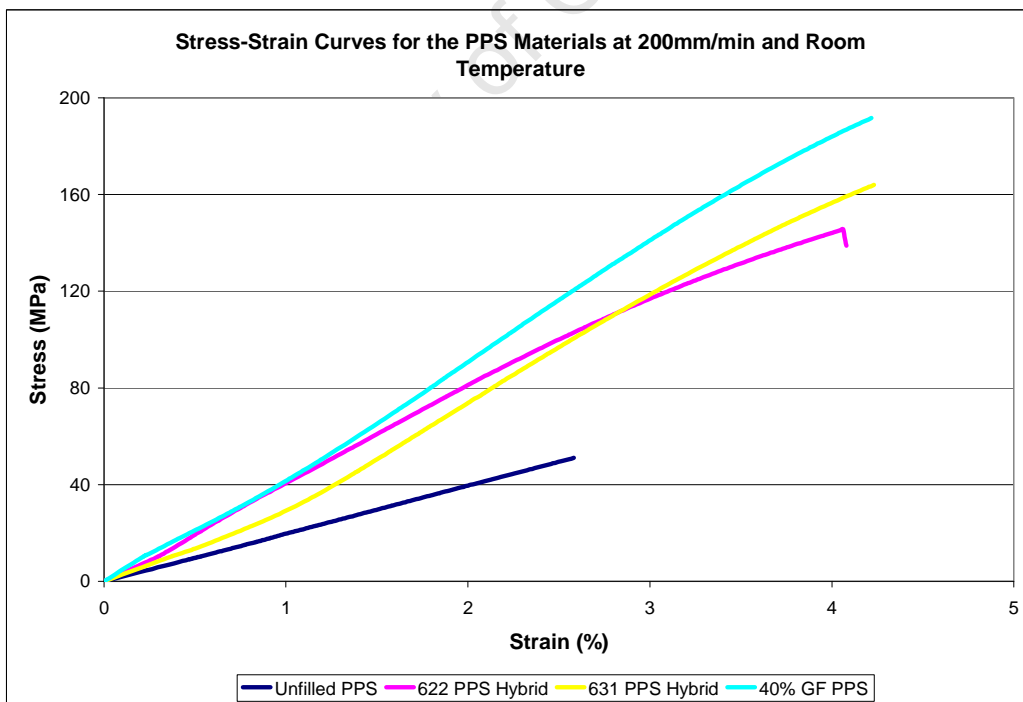
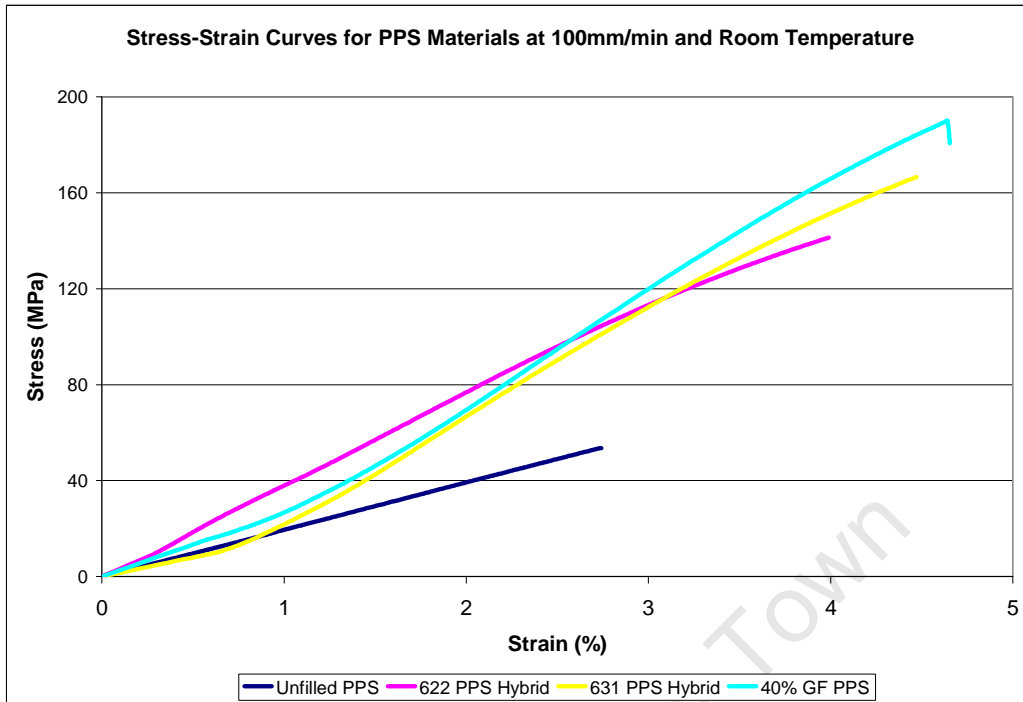


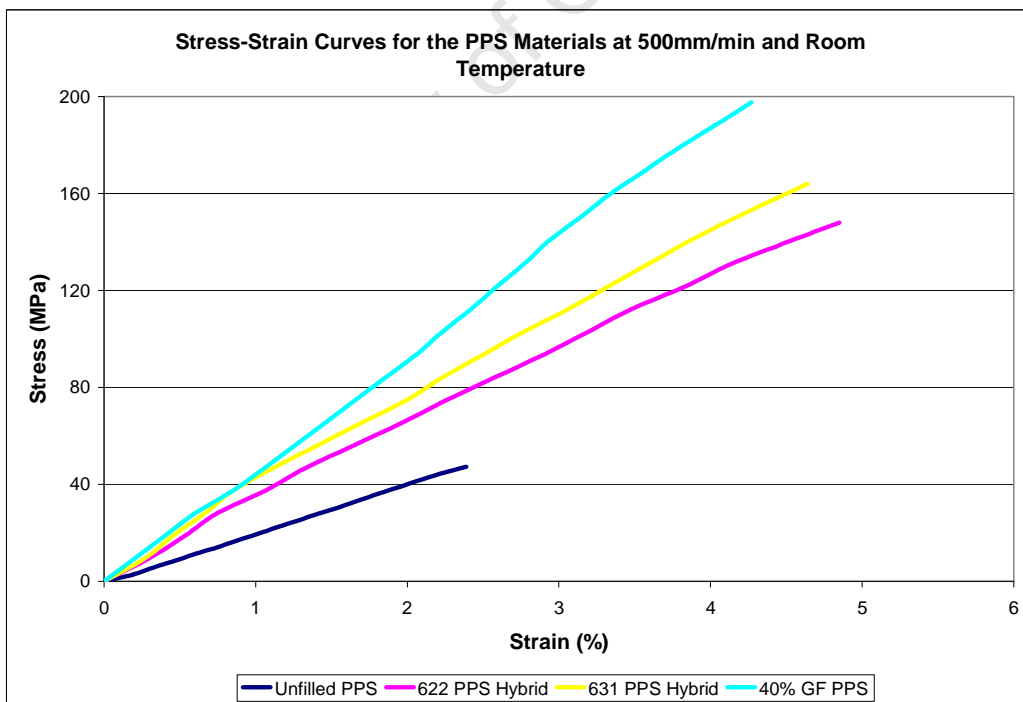
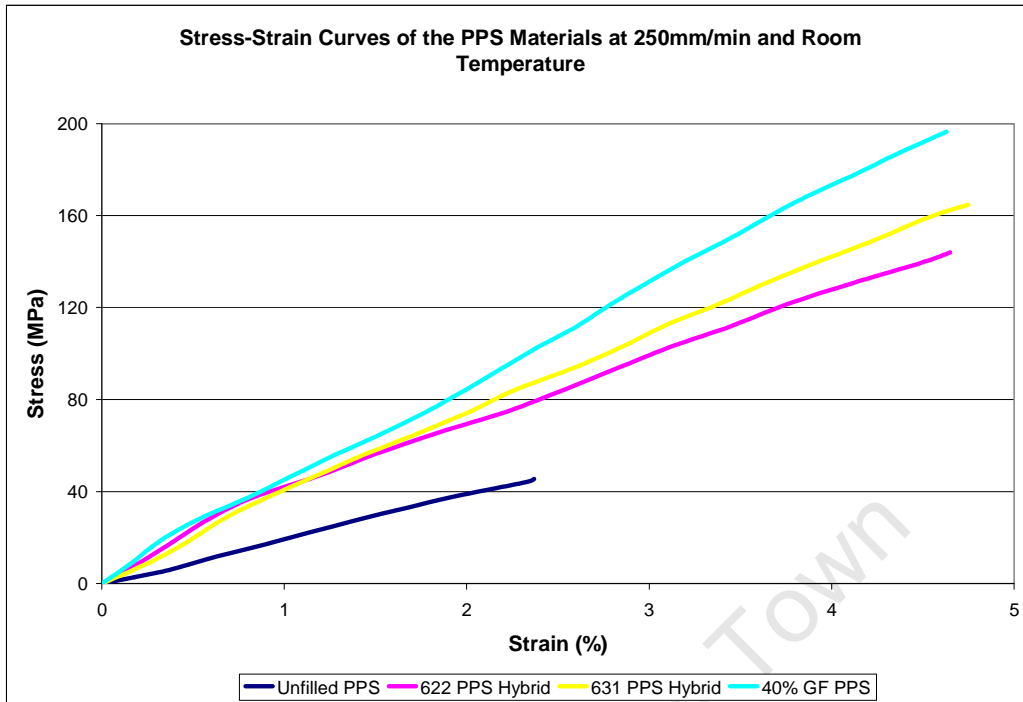


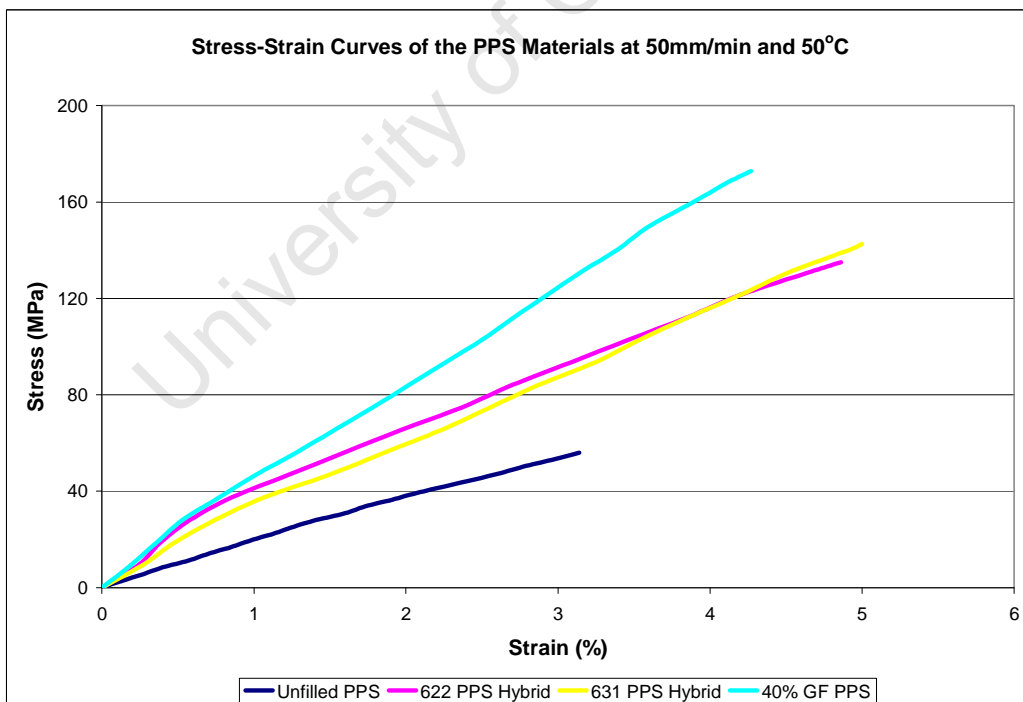
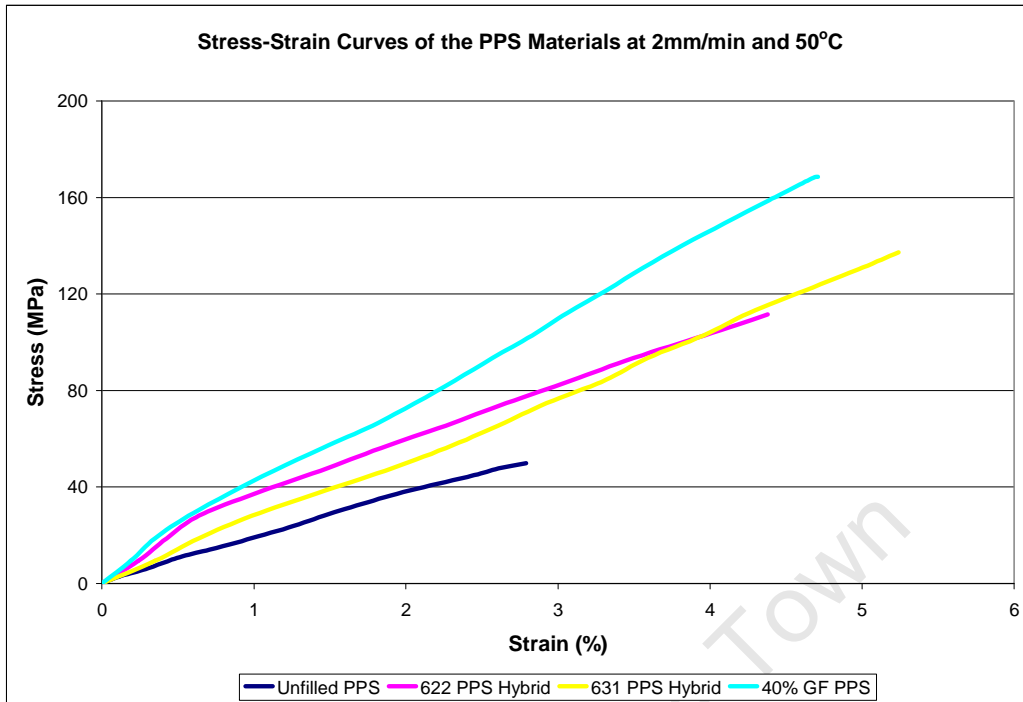


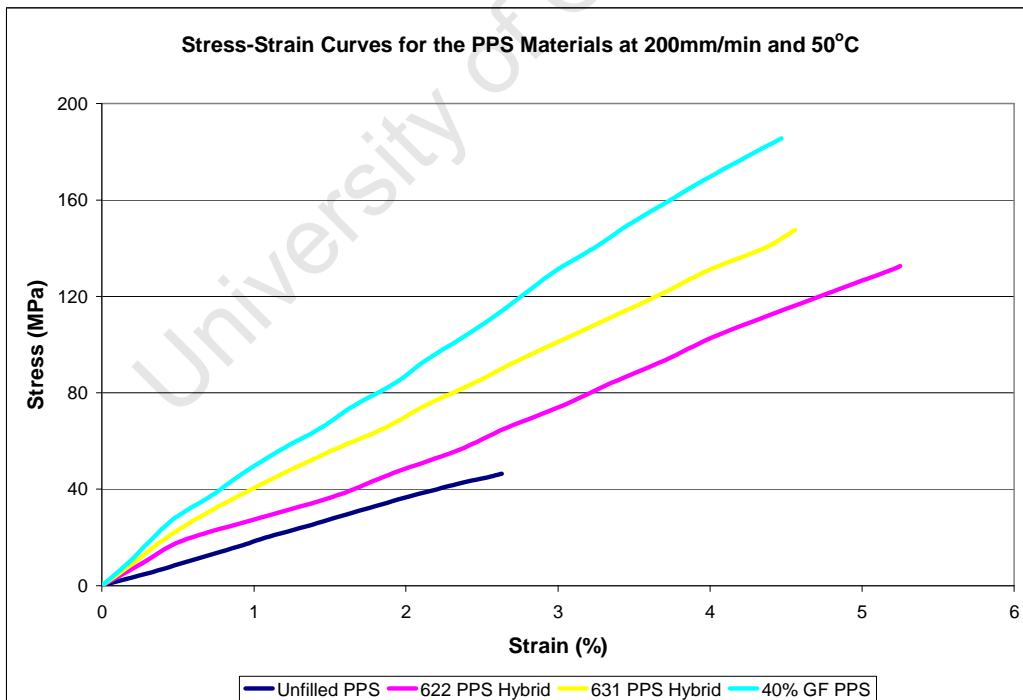
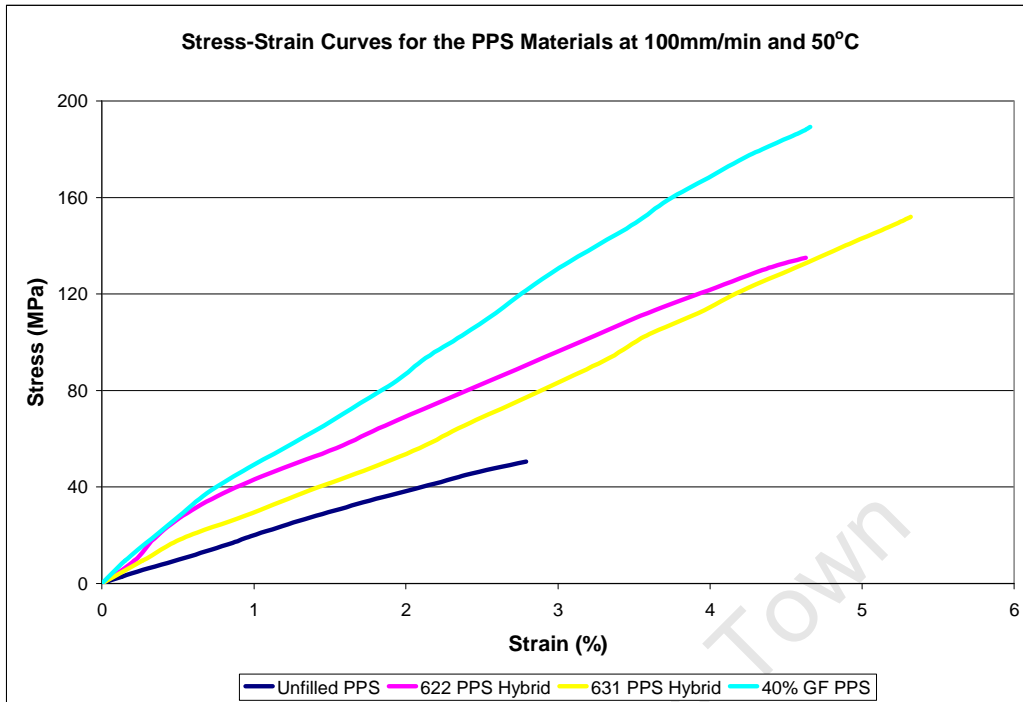
TENSILE STRESS-STRAIN CURVES FOR THE PPS COMPOUNDS

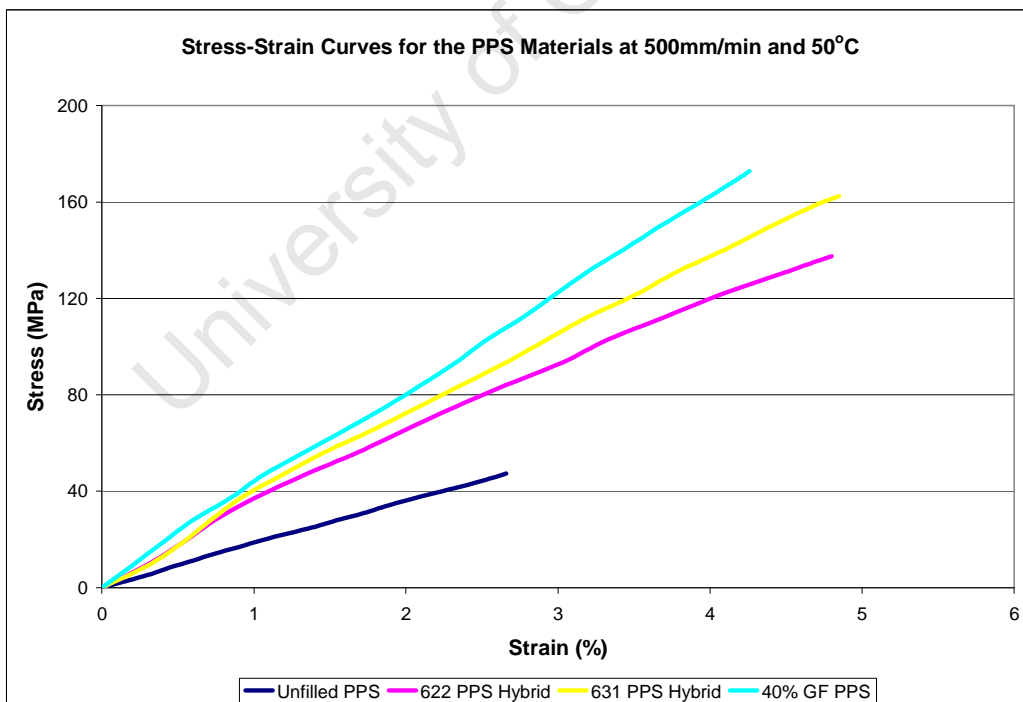
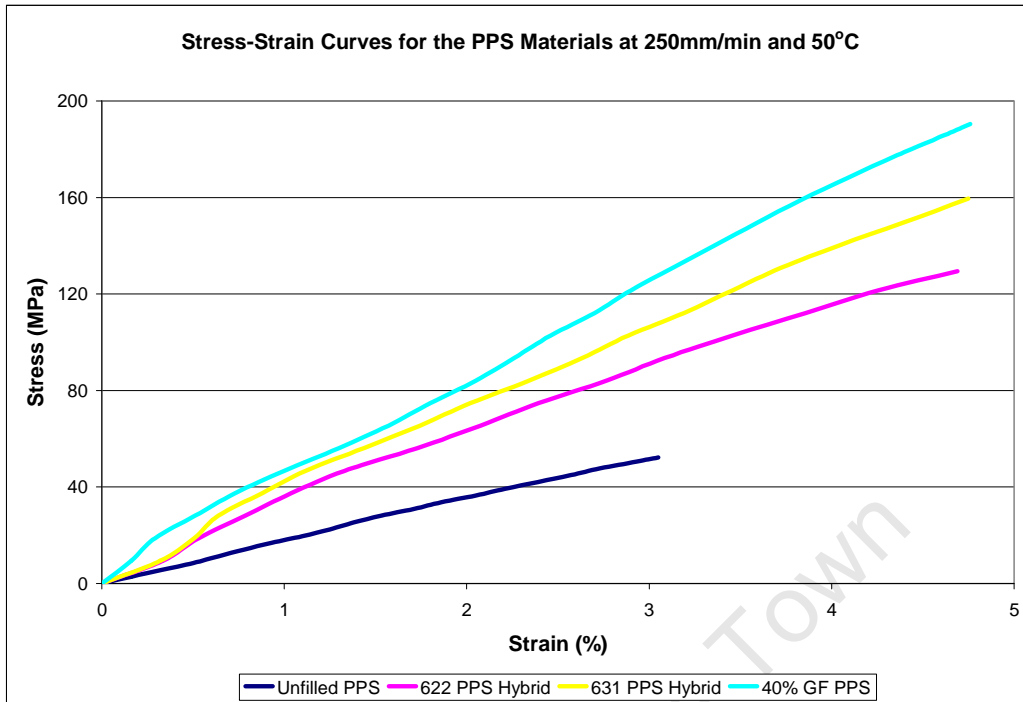


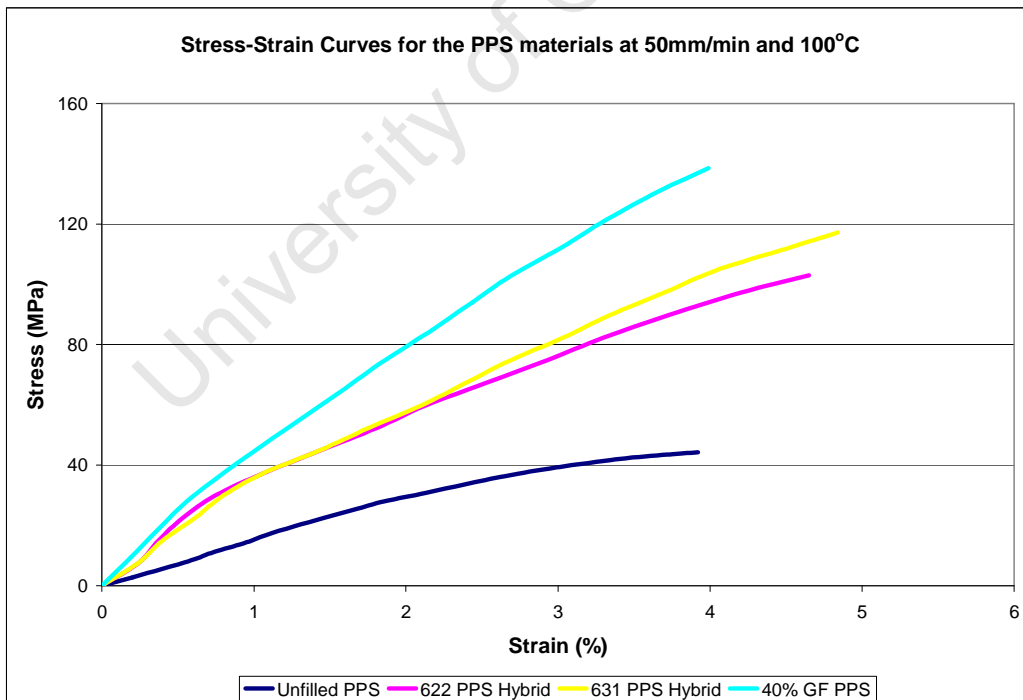
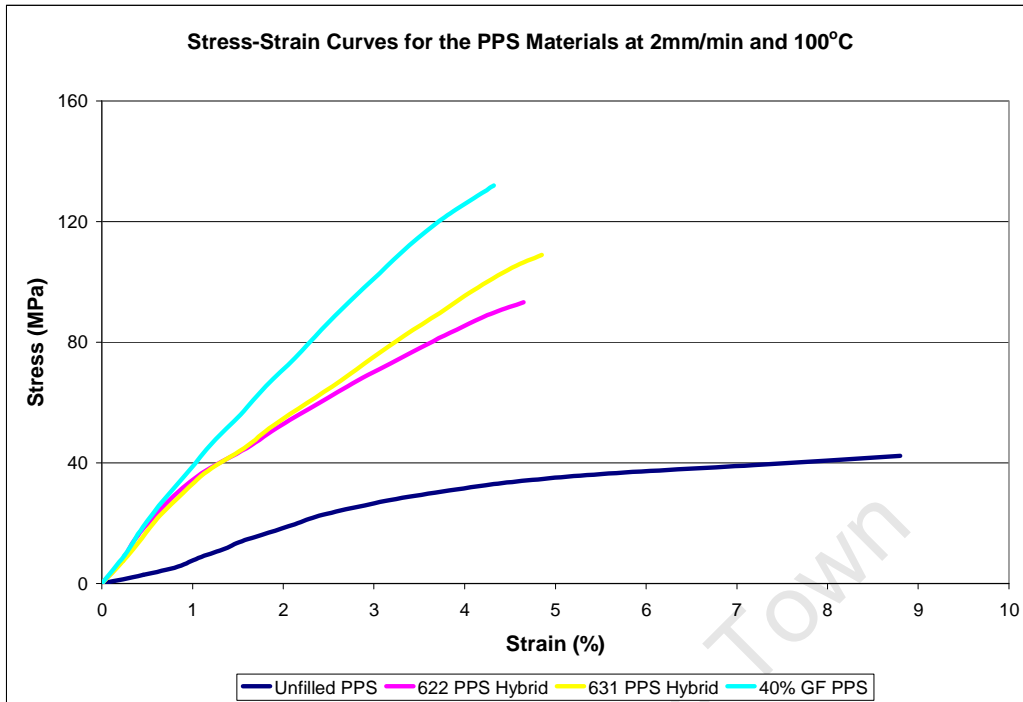


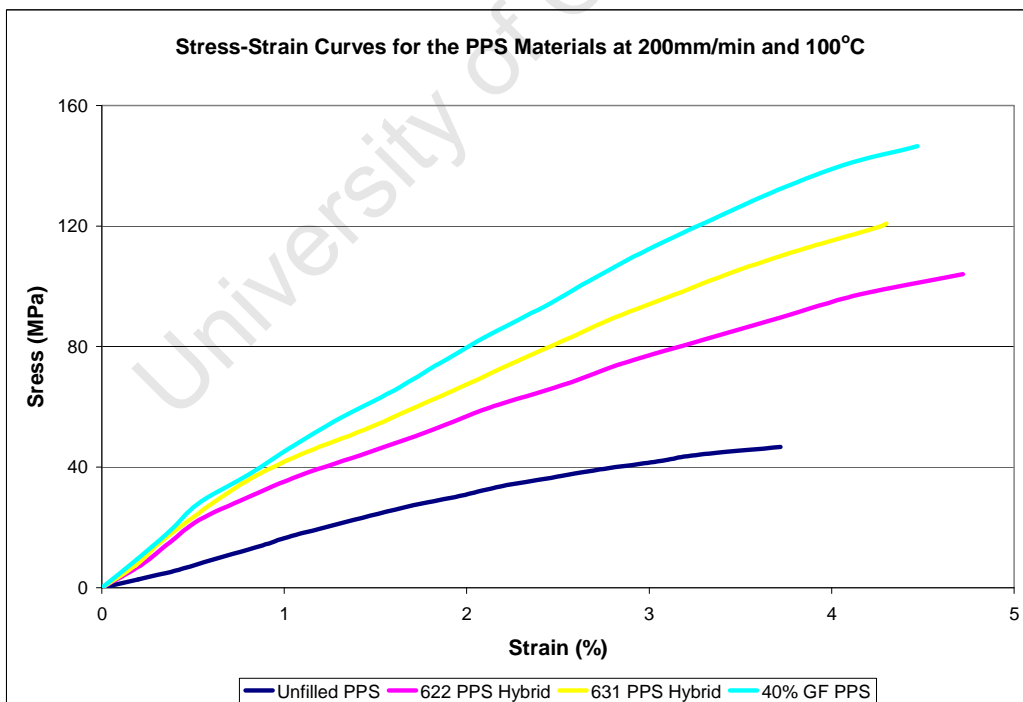
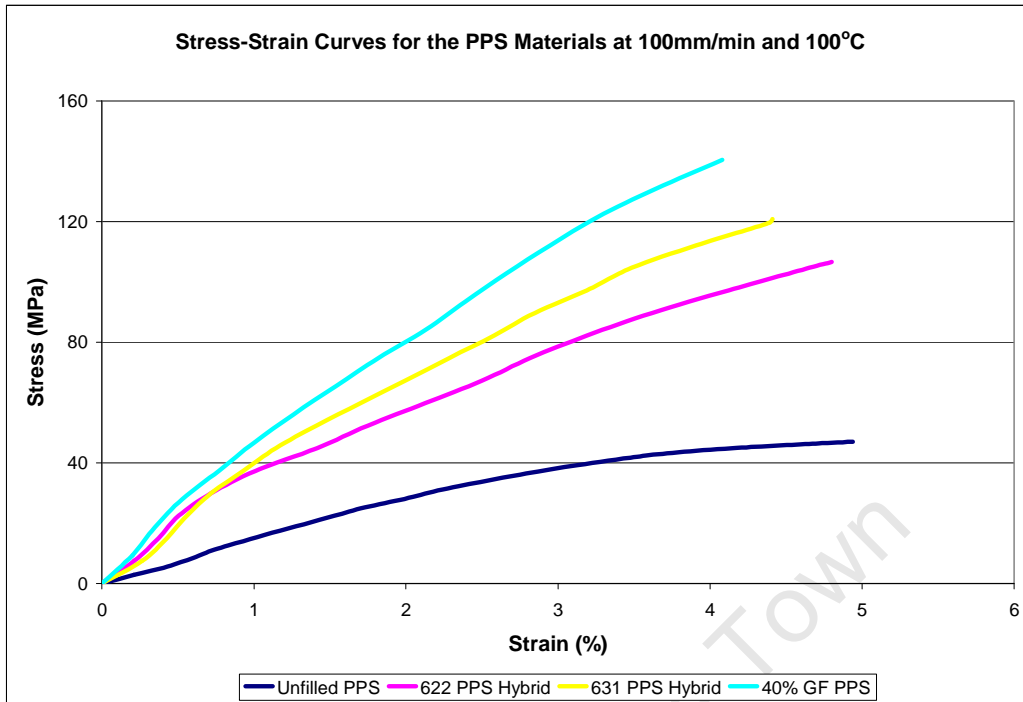


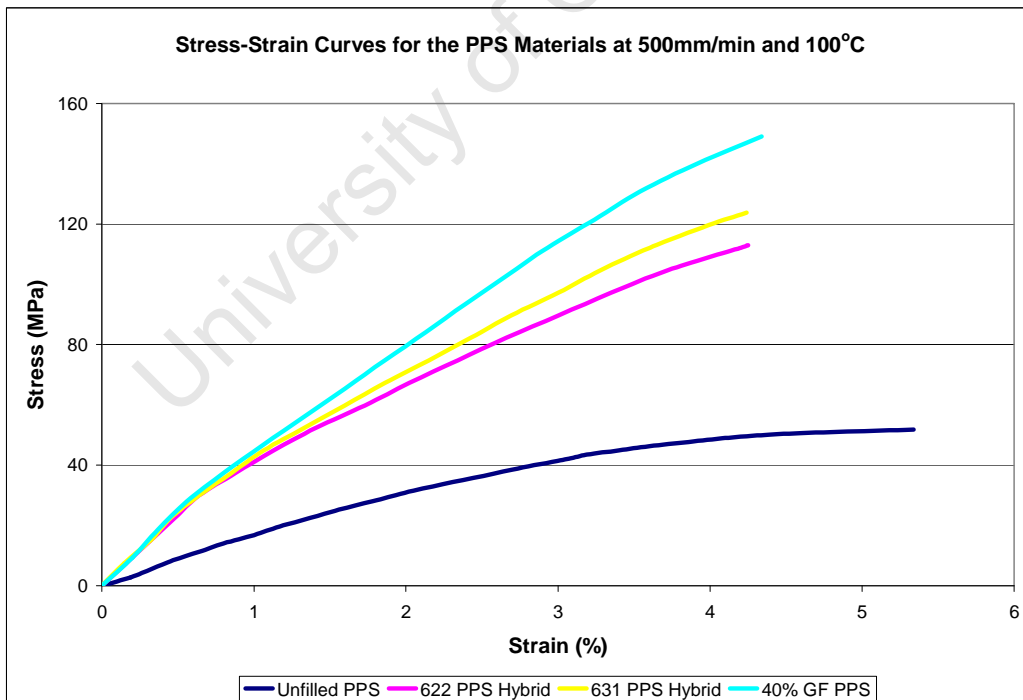
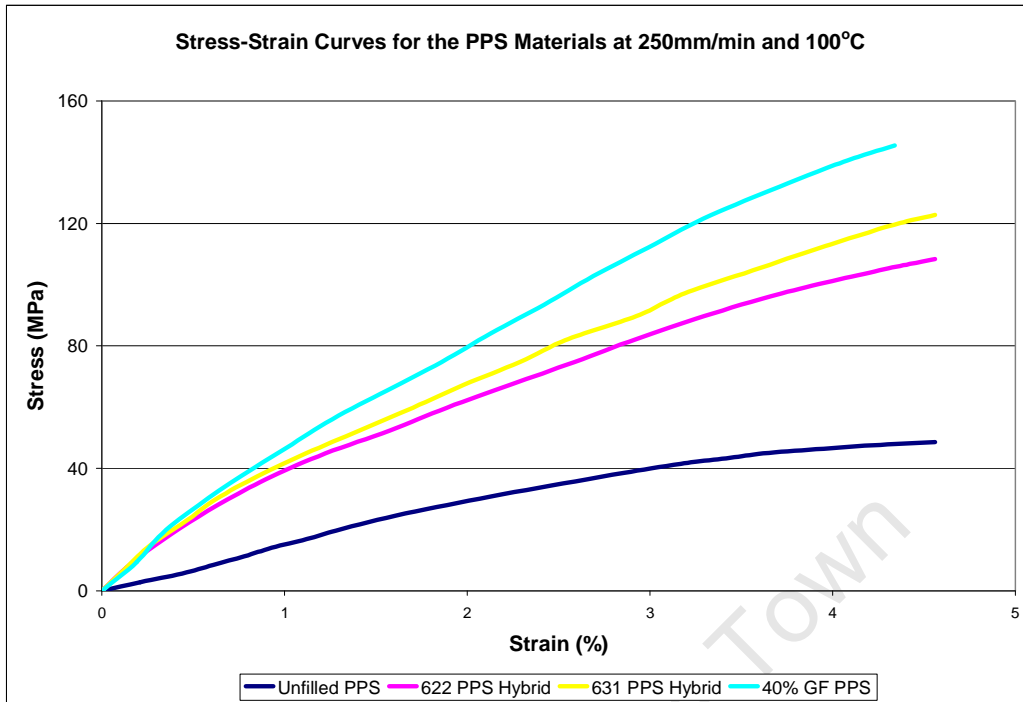


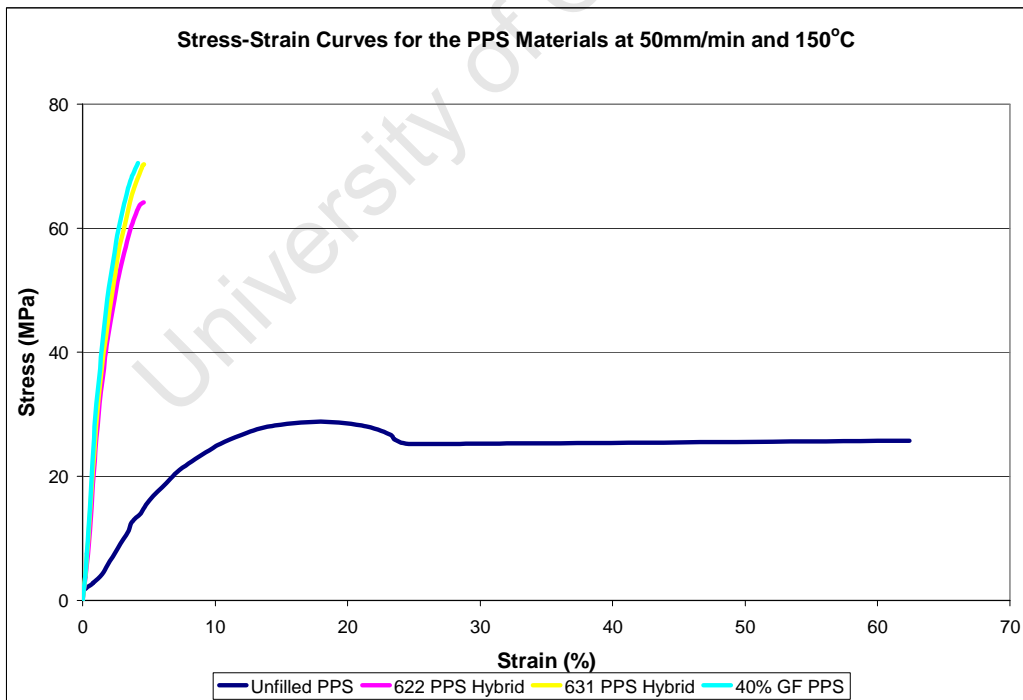
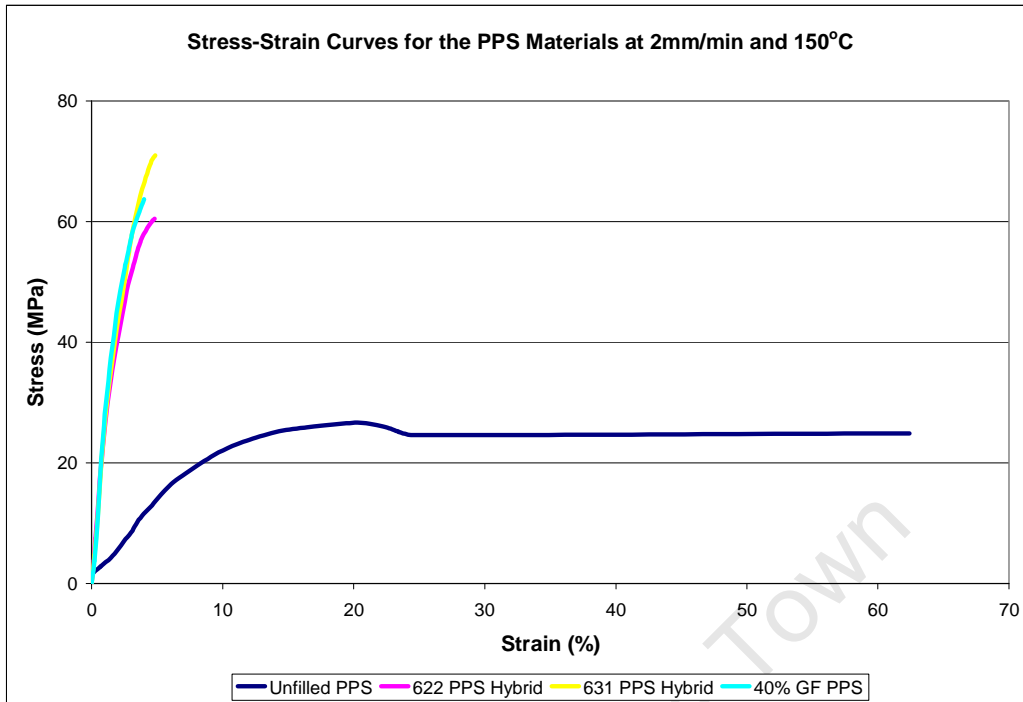


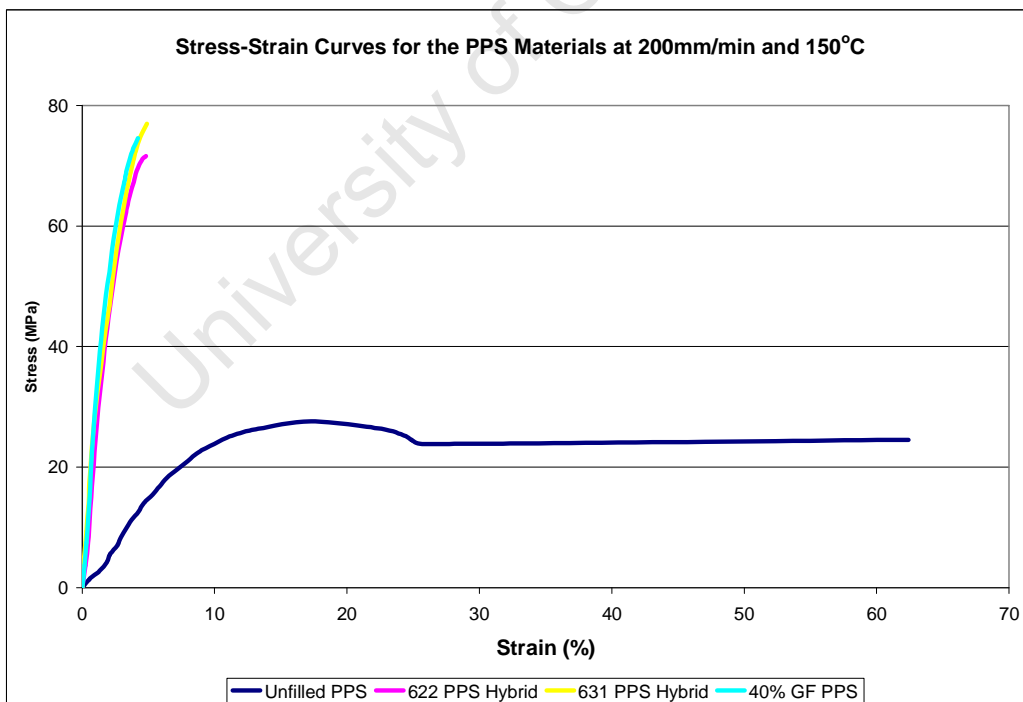
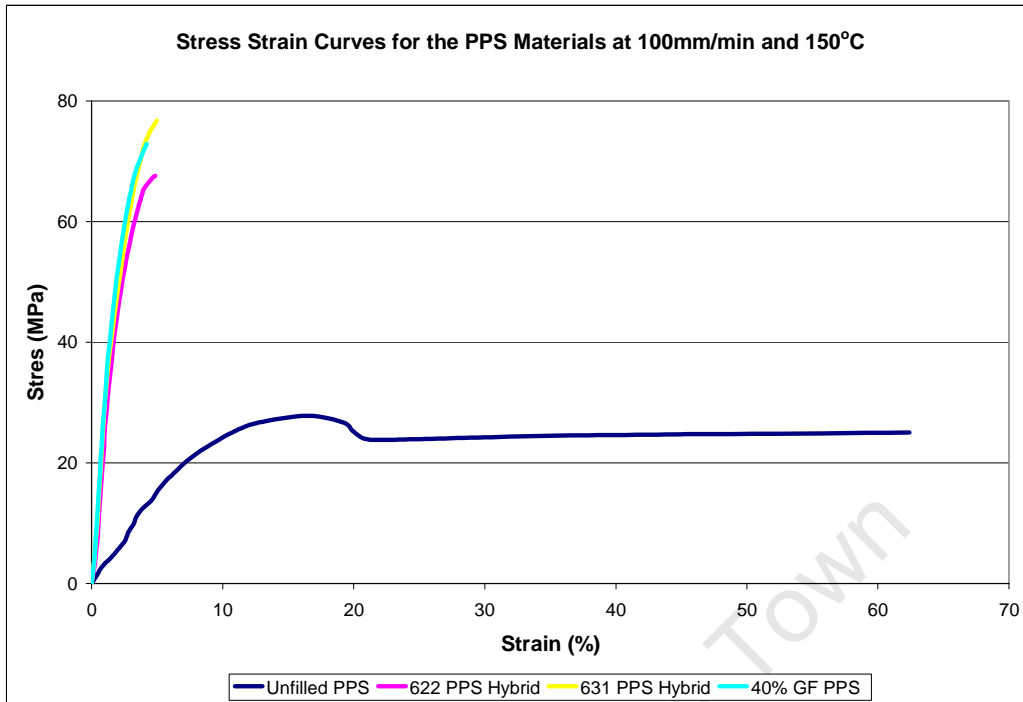


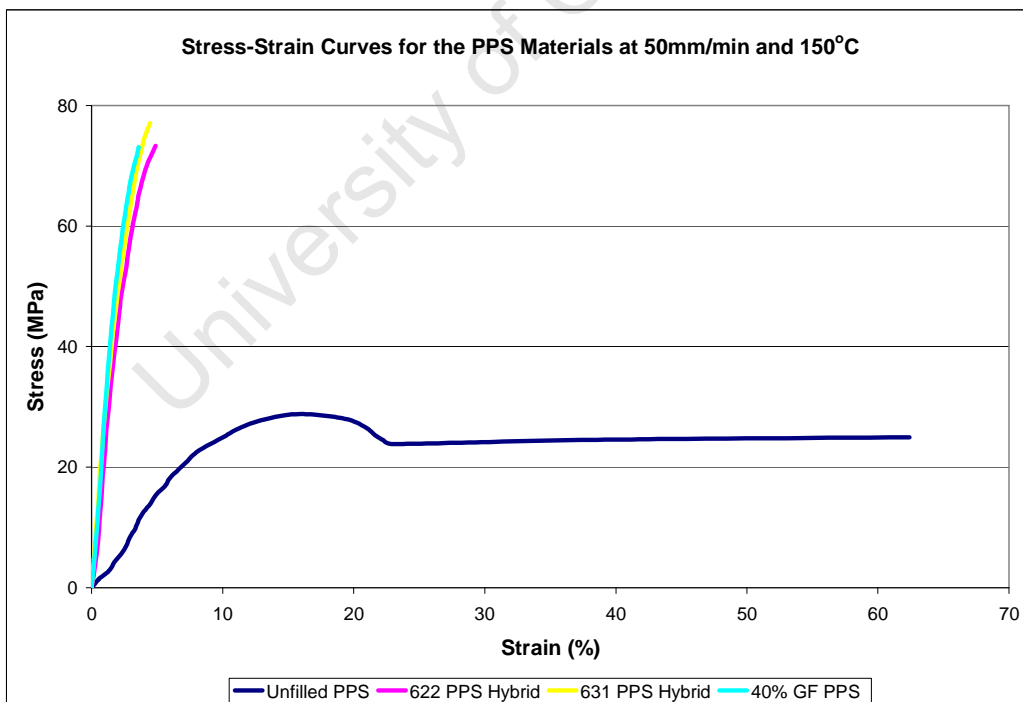
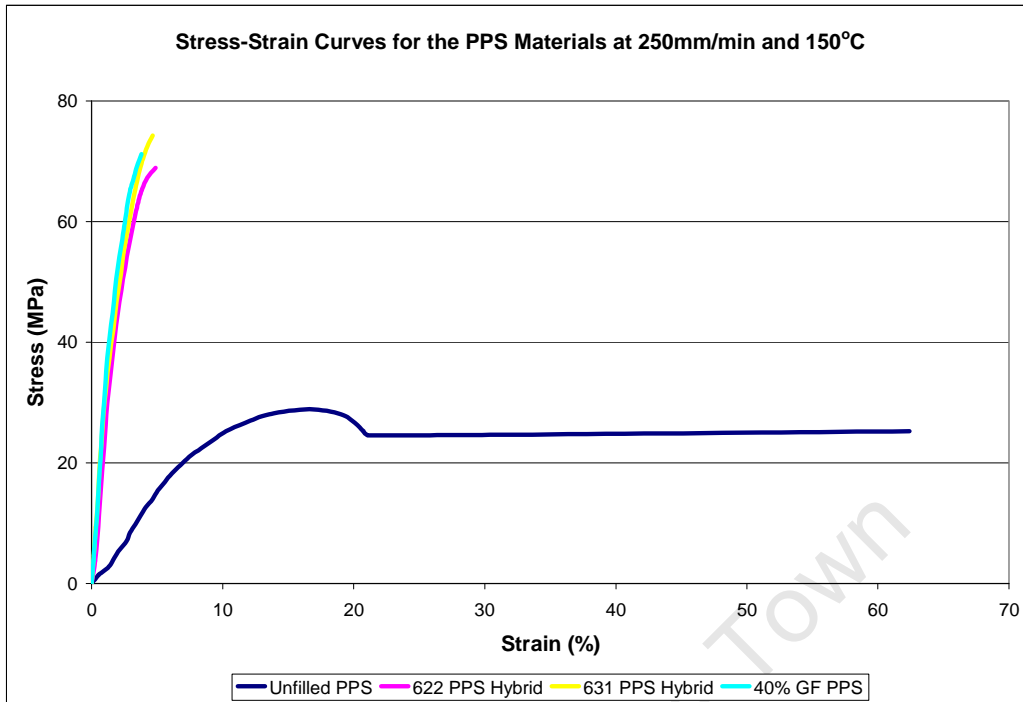






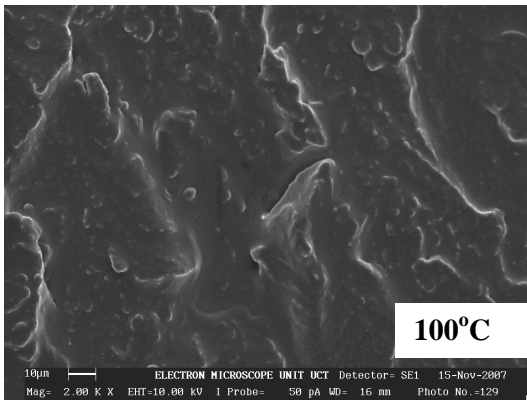
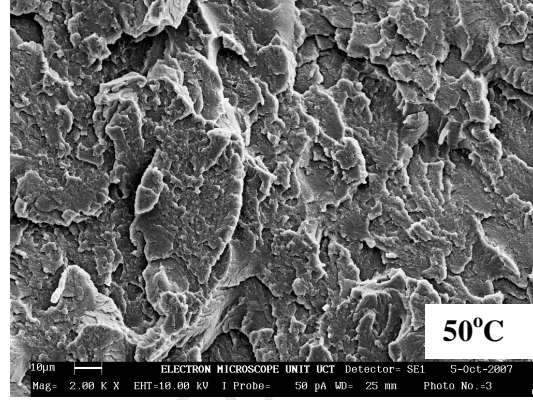
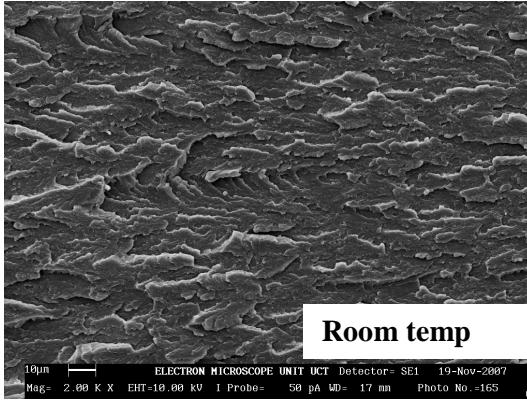




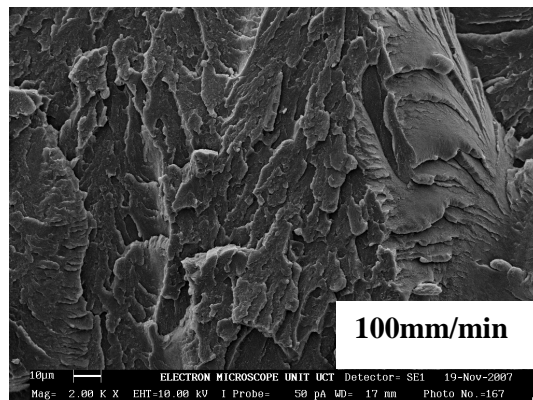
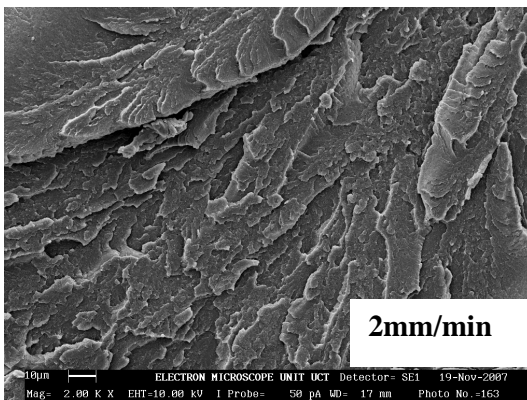


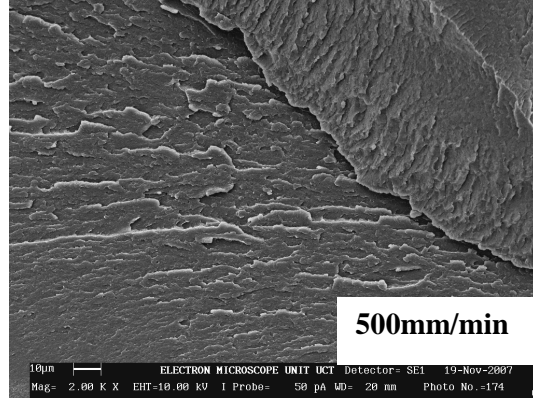
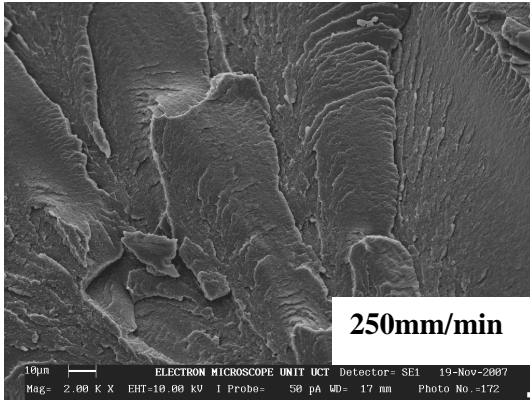
SEM MICROGRAPHS FOR THE TENSILE FRACTURED SURFACES OF THE DIFFERENT PPS COMPOUNDS

Unfilled PPS Tested at 50mm/min and Different Temperatures

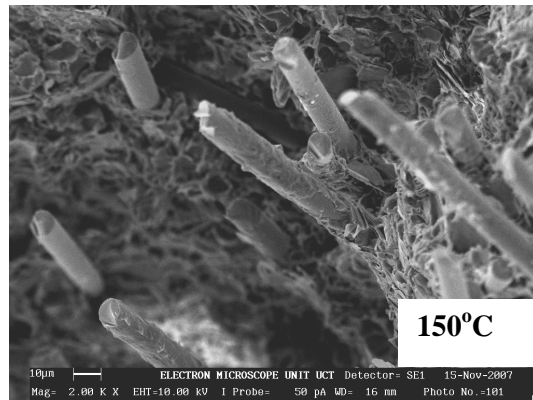
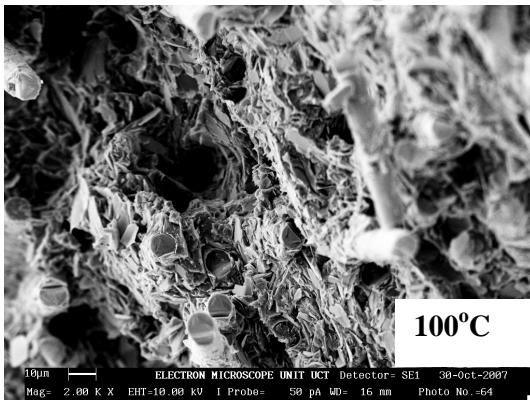
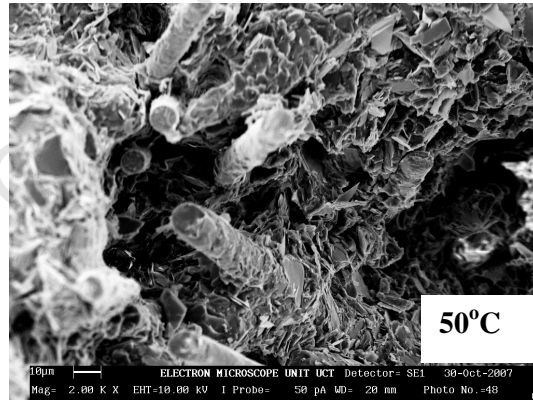
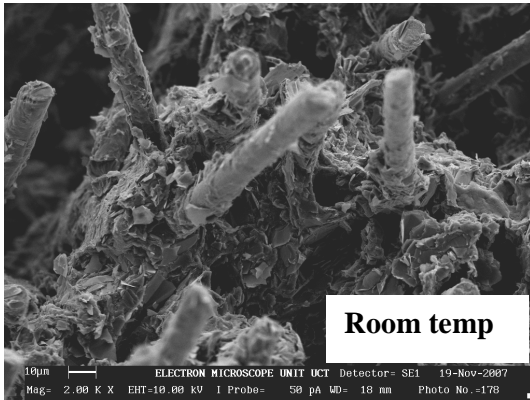


Unfilled PPS Tested at Room Temperature and Different Test Speeds

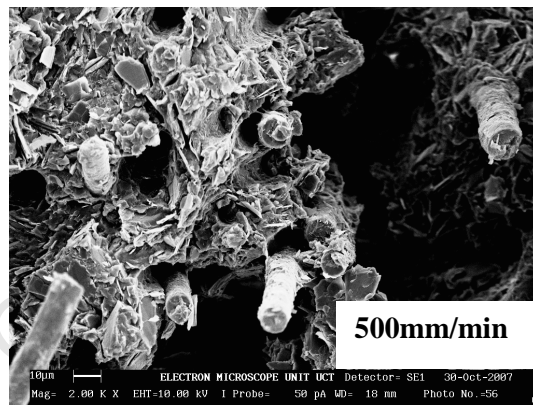
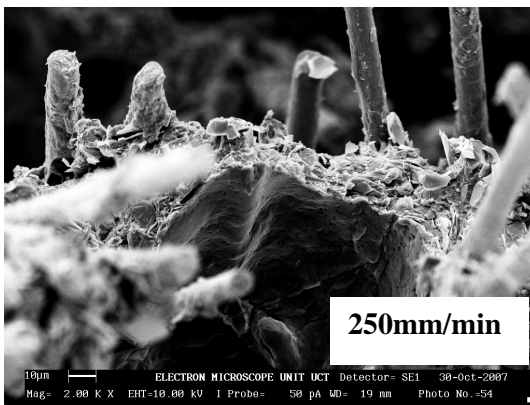
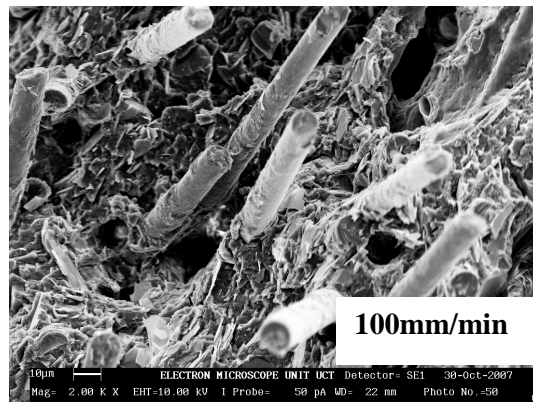
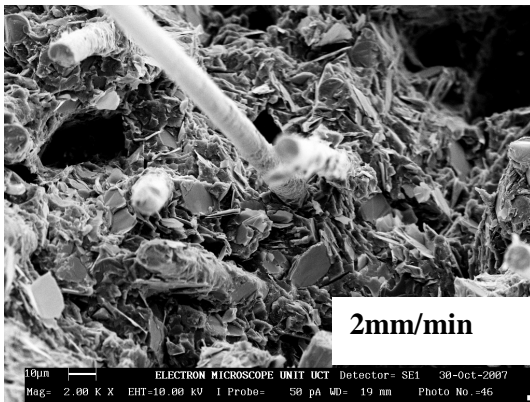




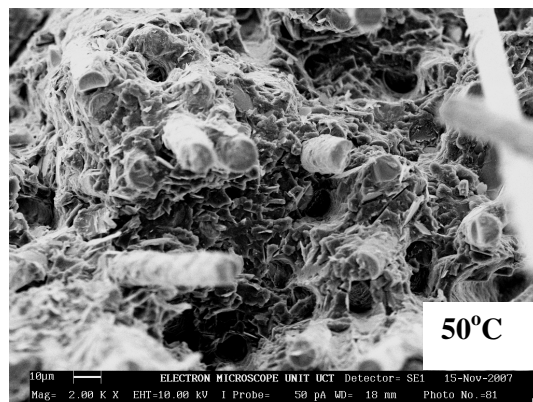
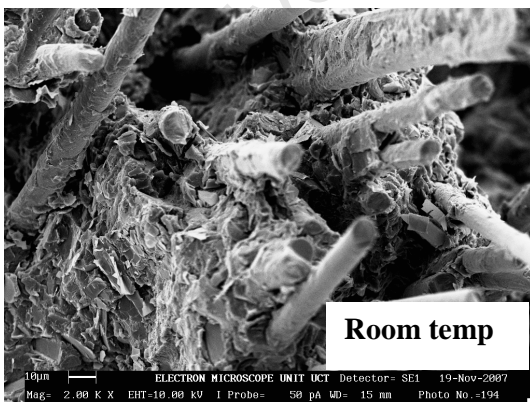
622 PPS Hybrid Composite Tested at 50mm/min and Different Temperatures

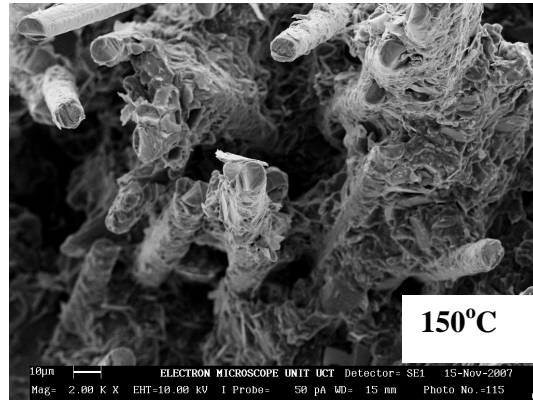
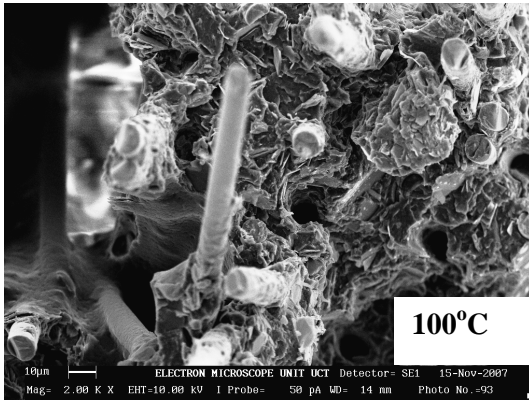


622 PPS Hybrid Composite Tested at 50°C and Different Test Speeds

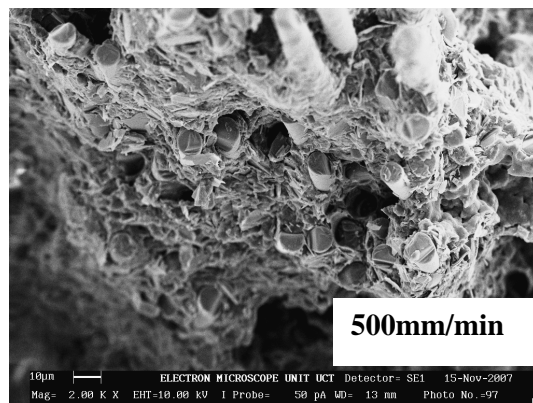
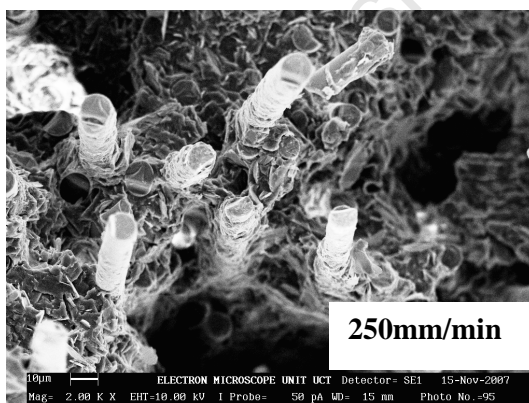
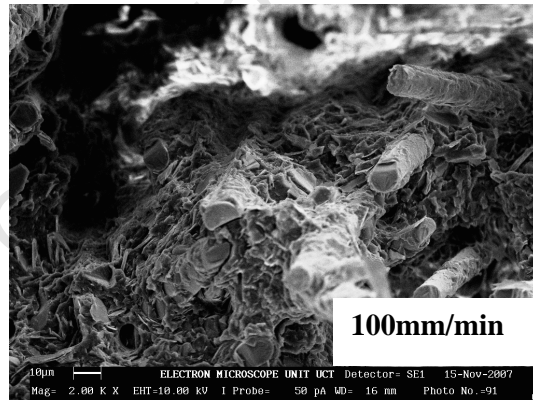
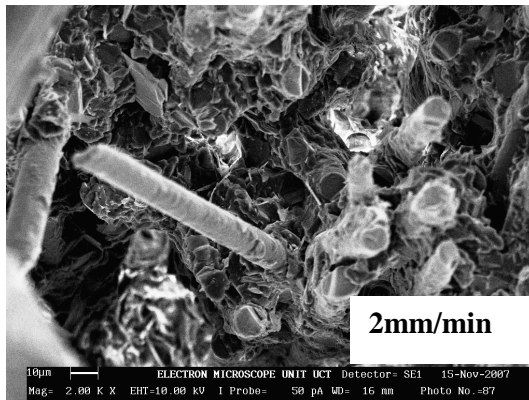


631 PPS Hybrid Composite Tested at 200mm/min and Different Temperatures

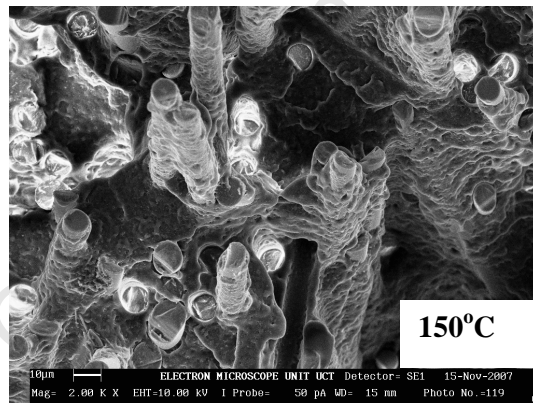
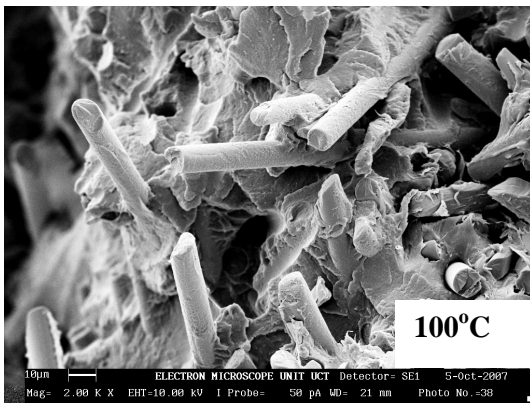
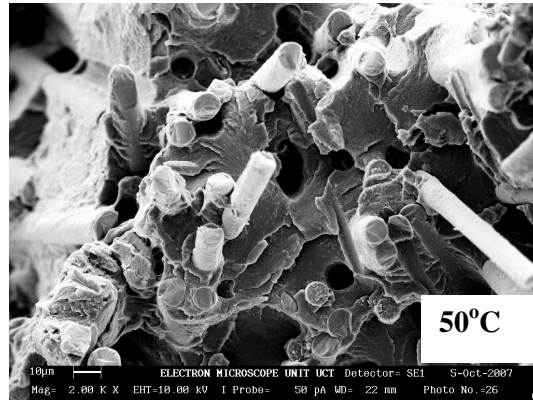
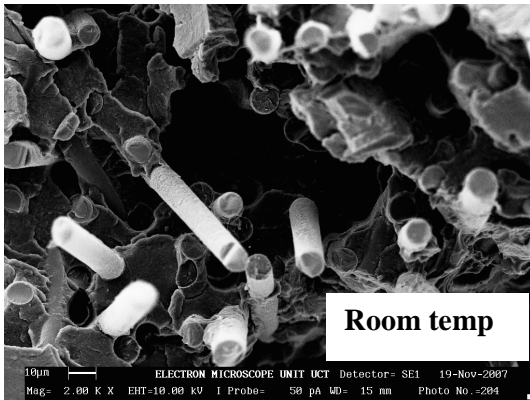




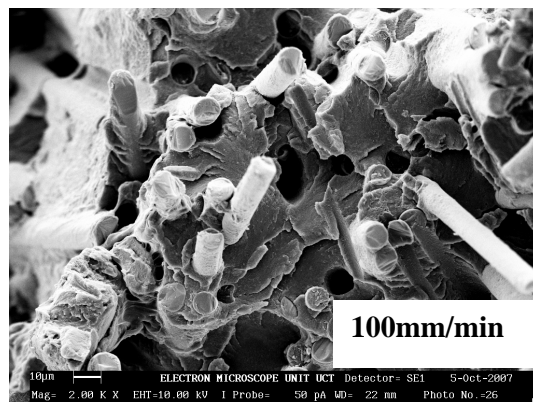
631 PPS Hybrid Composite Tested at 100°C and Different Test Speeds

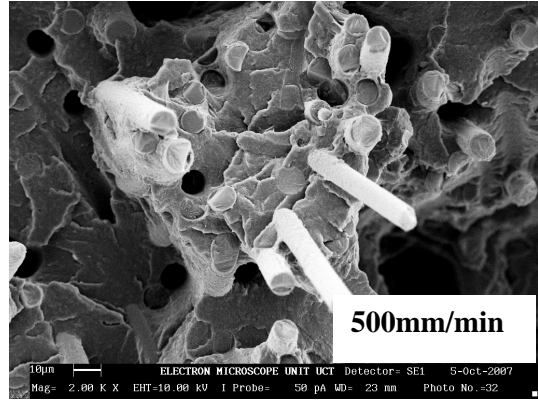
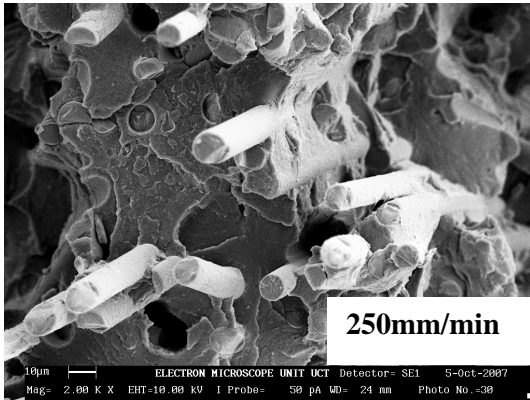


40% GF PPS Composite Tested at 100mm/min and Different Temperatures



40% GF PPS Composite Tested at 50°C and Different Test Speeds





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MARKET PRICE OF SOME THERMOPLASTIC RESINS AND COMPOUNDS AS AT MID APRIL 2008 [100]

Resin Material	Price (\$/kg)
PEEK	97.00
30% GF PEEK	72.75
PEI	14.24
30% GF PEI	11.71
PPS	14.09
40% GF PPS	11.46
PAI	60.63
30% GF PAI	55.12
PEKK	94.72
Chopped Strand Glass Fibre (PFG3540)	1.55 – 2.00
Talc (5 - 12µm Particle Size)	1.25



APPENDICES

APPENDIX C

(THE MARKET PRICE OF SOME ADVANCED THERMOPLASTIC RESINS AND COMPOUNDS)

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