# EFFECTS OF PROCESSING PARAMETERS ON THE MECHANICAL BEHAVIOR OF CONTINUOUS GLASS FIBER/POLYPROPYLENE COMPOSITES

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# ABSTRACT


## EFFECTS OF PROCESSING PARAMETERS ON THE MECHANICAL BEHAVIOR OF CONTINUOUS GLASS FIBER/POLYPROPYLENE COMPOSITES

Fiber reinforced polymeric composite materials have an increasing demand in industrial applications. Easy and rapid processing capability, high impact and delamination resistance, low moisture absorption and infinite shelf life of the raw materials are the attractive properties of continuous fiber reinforced thermoplastic composite materials. Therefore, thermoplastic based composites find in many application areas in automobile, aerospace, construction, defense, transportation and marine industries.

In recent years, hybrid fabrics; composed of continuous glass fibers and polymer fibers such as polypropylene (PP), have been used to fabricate thermoplastic composite with higher fiber volume fraction and improved performance.

In this study, hybrid fabrics were developed by commingling the continuous PP and glass fibers using air jet and direct twist hybrid yarn preparation techniques. The hybrid commingled fabrics obtained with $\pm 45^0$ fiber orientation and non-crimp fabric pattern. Non-crimp fabrics were obtained various fiber sizing that are compatible and in-compatible with PP matrix to investigate the effect of interfacial adhesion on the properties of the thermoplastic composites. Composite panels were produced from these fabrics via hot press compression method. Microstructural properties of the composites were investigated by matrix burn-out test and optical and scanning electron microscopy (SEM) analyzes. Tensile, compression, flexural and interlaminar peel tests were used to investigate the mechanical properties of the composites. Impact properties of the composites were examined by charpy impact test.

Results showed that laminates of the fabrics fabricated by air jet hybrid yarn preparation technique exhibit superior properties to those fabricated by direct twist covering hybrid yarn preparation technique. The results also showed that the fabrics with polypropylene compatible sizing results with enhanced composite properties.

# ÖZET

## SÜREKLİ CAM ELYAF/POLİPROPİLEN KOMPOZİTLERİN MEKANİK DAVRANIŞLARINA ÜRETİM PARAMETRELERİNİN ETKİLERİ


Elyaf destekli polimerik kompozit malzemeler giderek artan oranda endüstriyel uygulamalara girmektedir. Sürekli elyaf destekli termoplastik malzemelere olan ilginin artmasını bu malzemelerin; hızlı ve kolay işlenebilmesi, yüksek darbe dayanımı ve delaminasyon direnci, düşük nem emme özelliği, hammaddelerinin sınırsız raf ömrünün olması ve düşük maliyet gibi özelliklerinin olmasına bağlanabilir. Bu yüzden bu malzemeler otomotiv, havacılık ve uzay, inşaat, savunma, taşıma ve yat sektöründe giderek artan oranda kullanılmaktadırlar.

Son zamanlarda yüksek fiber hacim oranı ve yüksek performanslı kompozit malzeme üretimi amacı ile sürekli formda cam fiber ve polimer fiberlerinden (örnek olarak polipropilen (PP)) oluşan hibrit kumaşlar kullanılmaya başlanmıştır.

Bu çalışmada hava jeti tekniği ve direkt büküm tekniği adı verilen hibrit iplik geliştirme teknikleri kullanılarak PP ve cam elyaf liflerinin sürekli formda bir araya getirilmesiyle üretilen hibrit, örgüsüz kumaşlar geliştirilmiştir. Bu kumaşlar ± $45^0$ fiber yönlenmesinde ve örgüsüz formdadır. Fiber matriks ara yüzündeki bağlanmanın kompozit mekanik özelliklerine etkisini incelemek amacı ile PP uyumlu ve uyumsuz olmak üzere çeşitli cam fiber kaplamalı örgüsüz cam elyaf/PP kumaşlar geliştirilmiştir. Geliştirilen bu kumaşlardan sıcak presleme yöntemi ile cam elyaf/PP kompozit paneller üretilmiştir. Kompozit malzemelerin mikroyapısal özellikleri fiber yakma testi, optik mikroskop ve taramalı elektron mikroskobu (SEM) analizleriyle incelenmiştir. Çekme, basma eğme ve lamineler arası ayrılma testleri malzemelerin mekanik özelliklerinin belirlenmesi için uygulanmıştır. Malzemelerin darbe dayanımlarının incelenmesinde charpy darbe testi kullanılmıştır.

Sonuçlara göre hava jeti tekniği ile geliştirilen kumaşlardan üretilen kompozit malzemenin mekanik özelliklerinin diğer teknikle üretilenlere göre daha yüksek olduğu gözlenmiştir. Ayrıca kullanılan cam elyaf kaplamasının ara yüzdeki yapışmaya etkisi incelenmiş ve polipropilen reçine uyumlu kaplama uygulanmış cam fiber kullanılan kompozit malzemelerin daha üstün mekanik özeliklere sahip olduğu gözlenmiştir.

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# CHAPTER 1

# INTRODUCTION

High performance polymeric composite materials have many advantages including lighter weight, superior mechanical properties, corrosion resistance, ability to tailor lay-ups for optimum strength and stiffness, and flexibility in design capabilities. Disadvantages of composites include high raw materials, fabrication and assembly costs. Polymeric composites are adversely affected by both temperature and moisture and they are weak in the out-of-plane direction.

Polymeric composites consist of two parts; matrix and reinforcement. The matrix holds the fibers in their proper position; protects the fibers from abrasion; transfers loads between fibers; and provides interlaminar shear strength. Reinforcements provide strength and stiffness, and significantly improve the structural characteristics of thermoplastics and thermosets.

Polymer composites typically fall in to two categories with respect to the nature of the matrix material: thermoplastic and thermoset matrix composites. Thermoset resins such as epoxy and polyester, crosslink during cure stage to form rigid intractable solids. Due to the immobile chemical bonds and high crosslink densities of the thermoset systems, they are inherently brittle. On the other hand, thermoplastics such as polypropylene (PP) and polyamide (PIA) are saturated polymers which are fully reacted prior to processing and do not form crosslink during processing (Campbell 2006). There are a number of advantages of usage of thermoplastic composites as compared with thermoset based. One of the important features of the thermoplastic composites is their re-processability and recyclability (Ferreira, et al. 1997, Greco, et al. 2007, Ishak, et.al 2007). They are inherently much tougher than thermosets, therefore thermoplastics are damage tolerant and resistant to low velocity impact (Trudel-Boucher, et al. 2006). Processing of thermoplastics is relatively simpler, faster, healthier and environmentally friendly (Brueu and Denault 2004, Ferreira, et al. 1997). The common reinforced thermoplastics are nylons and polypropylenes. Other thermoplastics used to prepare composites are particularly PET, PBT, PC and PPS.

Reinforcements are available in continuous forms and chopped forms having different lengths, or discontinuous in form (whiskers, flakes, spheres, etc.) to meet different properties and processing methods (Rosato and Rosato 2004). A wide variety of fiber materials are available for the polymer composites. The most commonly used fibers in polymer matrices are carbon, glass and aramid type fibers (Hull 1995). Glass fiber reinforced thermoplastic materials fall into mainly two categories; aligned thermoplastic composites (ATC) and glass mat thermoplastic composites (GMT). GMTs are non-woven textile technology that are being used as a typical chopped strand mat or a continuous swirl mat form, impregnated typically with polypropylene. ATCs are prepregs fabricated from aligned glass fibers which are suitable for weaving process and impregnation during final forming. Aligned thermoplastic composites have better mechanical properties than glass mat thermoplastics.

Most current applications utilize glass mat thermoplastic GMT, based on usually on random fibers within a thermoplastic matrix. These materials are used for semi-structural components since fiber content is limited to up to 40% by mass. To achieve higher mechanical properties, materials based on aligned fibers, usually in the form of textile preforms, have been developed. High viscosity of the thermoplastic resin causes to some problems during impregnation in to the glass fibers. To overcome this problem, hybrid yarns have been recently developed. Hybrid yarns also offer an ideal opportunity to achieve short cycle times (Mader, et al. 2008). Furthermore, textile preforms manufactured from hybrid yarns, are being used in thermoplastic composite manufacturing. These preforms may be in the form of woven fabrics, knitted fabrics, braided fabrics and non-woven fabrics (Alagirusamy, et al. 2006). Current techniques to manufacture thermoplastic composites from these textile preforms and hybrid yarns include; compression molding, filament winding, pultrusion, autoclave molding, inflation molding and injection molding. However, there is very limited work reported in the literature about the development of hybrid continuous fabrics, fabrication technologies to prepare composite from these fabrics. The information on the properties of these composites is also relatively limited. Zhao, et al. (2009) have been reported tensile and impact properties of glass/PP woven fabrics, Perrin, et al. (2003) have been investigated the mode I interlaminar fracture toughness of unidirectional continuous glass fiber/PP composites and there are some other similar studies. In our study, we investigated the effect of processing parameters on the mechanical behavior of continuous glass fiber/PP composites manufactured from hybrid fabrics of glass fiber and PP.

Objectives of this study are; 1) development of commingled, non-crimp glass fiber/polypropylene hybrid fabrics, 2) manufacturing thermoplastic based composite material from these developed fabrics, 3) characterization of the mechanical and microstructural properties of the manufactured thermoplastic composite materials, 4) relating process parameters to composite properties.

# CHAPTER 2

# THERMOPLASTIC MATRIX COMPOSITES

An interest in thermoplastic composites dates from the late 1960s with the appearance of 'Azdel'™ a random reinforced GMT, in the USA. Thermoplastic composites caught the attention of researchers and become a hot spot of study since the 1980s (Ford 2004). Over the last years European industry has generally been far more receptive to thermoplastic composites, primarily due to environmental concerns and legislation in areas such as processing emissions and end-of-life recycling. Partly because of these factors, the growth rate of thermoplastic composites is widely reported as being twice that of thermoset composites, and therefore seen significantly increased interest and activities in thermoplastic composites in all over the world.

Thermoplastics have the simplest molecular structure with chemically independent macromolecules. By heating, they are softened or melted, and solidified when cooled. Multiple cycles of heating and cooling can be repeated without severe damage, allowing reprocessing and recycling (Biron 2007).

During the heating cycle, care must be taken to avoid degrading or decomposition (Rosato and Rosato 2004). Thermoplastics offer a wide range of matrix materials for reinforcement by fibers, flakes, beads, or particulate materials such as talc and mica. They bring the great advantage that they are more easily molded in mass production quantities (such as injection molding) than are reinforced thermosets. Most types of thermoplastics can readily be compounded with reinforcing materials. Among the fibers, glass is the main reinforcement (Rosato and Rosato 2004).

Before considering the potential advantages of thermoplastic composite materials, it is necessary to understand the difference between a thermoset and thermoplastics. As shown in Figure 2.1, a thermoset crosslinks during cure to form a rigid intractable solid. Prior to cure, the resin is relatively low molecular weight semi-solid that melts and flows during the initial part of the cure process.

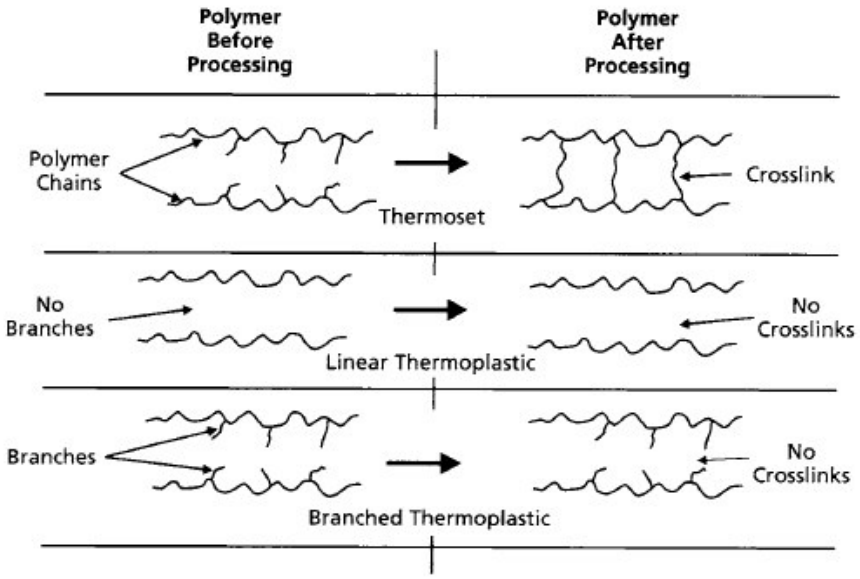



Figure 2.1. Comparison of Thermoset and Thermoplastic Polymer Structures (Source: Campbell 2006)

As the molecular weight builds during cure, the viscosity increases until the resin gels, and then strong covalent bond crosslinks form during cure. Due to the high crosslink densities obtained for high performance thermoset systems, they are inherently brittle unless steps are taken to enhance toughness (Campbell 2006). On the other hand, thermoplastics are high molecular weight resins that are fully reacted prior to processing. They melt and flow during processing but do not form crosslinking reactions. Their main chains are held together by relatively weak secondary bonds. However, being high molecular weight resins, the viscosities of thermoplastics during processing are orders of magnitude higher than thermosets (e.g. $10^4$-$10^7$ P for thermoplastics vs. 10 P for thermosets) (Campbell 2006, Ishak, et al. 2007). This high viscosity requires high process temperature and pressure to prevent non-wetting problems. Since thermoplastics do not crosslink during processing, they can be reprocessed, for example they can be thermoformed in to structural shapes by simply reheating to the processing temperature (Ferreira, et al. 1997, Greco, et al. 2007, Ishak, et al. 2007). On the other hand, thermosets, due to their highly crosslinked structures, cannot be reprocessed and will thermally degrade, and eventually char, if heated to high enough temperatures. However, there is a limit to the number of times a thermoplastic can be reprocessed.

The structural difference between thermosets and thermoplastics yields some insight into the potential advantages of thermoplastics. Since thermoplastics are not crosslinked, they are inherently much tougher than thermosets (Ferreira, et al. 1997, Ishak, et al. 2007, Trudel-Boucher, et al. 2006). Therefore, they are much more damage tolerant and resistant to low velocity impact damage than the untoughened thermoset composites (Greco, et al. 2007, Trudel-Boucher, et al. 2006).

Since thermoplastics are fully reacted high molecular resins that do not undergo chemical reactions during cure, the processing for these materials is theoretically simpler and faster (Ferreira, et al. 1997, Greco, et al. 2007, Ishak, et al. 2007, Trudel-Boucher, et al. 2006, Ye, et al. 1995). For example, the cycle time for heating, forming and cooling a PEEK/AS4 (APC2) composite is approximately 120 min for maximum process conditions of 420 $^{0}$C and 0.69 MPa in an autoclave or press. When volume production is sought, the composite can be preheated separately and then formed and shaped in less than 10 mins. To process a typical 177 $^{0}$C cure epoxy and carbon fiber composite would take approximately 3-4 h to heat, cure and cool in autoclave (Gutowski 1997). Another advantage of thermoplastic composites involves health and safety issues. Since these materials are fully reacted, there is no danger to the worker from low molecular weight unreacted resin components. During processing of thermoplastic composites there are no VOC emissions (Bureau and Denault 2004, Ferreira, et al. 1997). In addition, thermoplastic composite prepregs do not require refrigeration. They have essentially an infinite shelf life (Greco, et al. 2007, Ishak, et al. 2007), but may require drying to remove surface moisture prior to processing.

Another potential advantage of thermoplastics is low moisture absorption (Greco, et al. 2007). Cured thermoset composite parts absorb moisture from the atmosphere that lowers their elevated temperature (hot-wet) performance. Since many thermoplastics absorb only very little moisture, the design does not have to take as severe a structural "knock down" for lower hot-wet properties (Campbell 2006). However, since thermosets are highly crosslinked, they are resistant to most fluids and solvents encountered in service.

## 2.1. Types of Thermoplastic Composites

Thermoplastic composites have the potential to replace metals in structural applications. The materials are therefore characterized by a relatively high fiber content, which ideally be well aligned. Typical examples, at the top end of the performance spectrum, are the highly aligned thermoplastic composites, (ATC), such as unidirectional prepregs and laminates derived from them. The materials at the bottom end are conventionally called short and long fiber composites. Glass mat thermoplastics (GMT) are between these two composites as their performance. Performance spectrum of thermoplastic composites is shown in Figure 2.2 (Ford 2004).

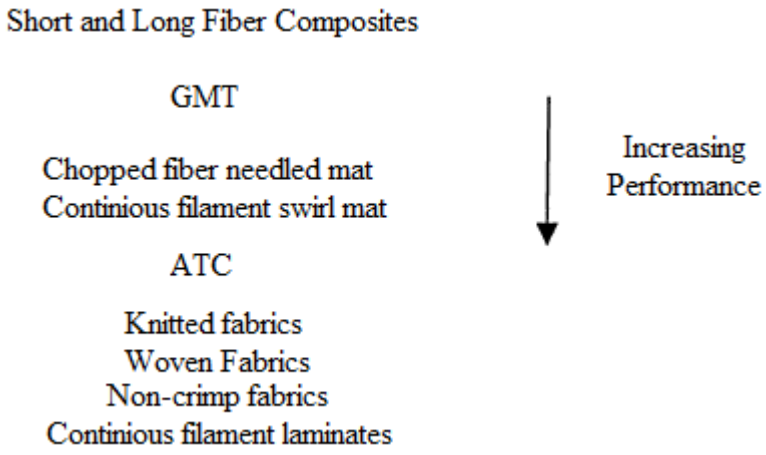
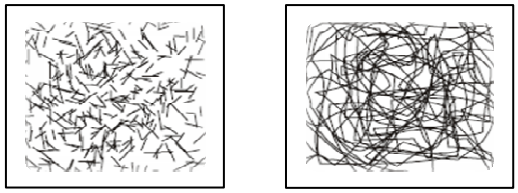



Figure 2.2. Performance Comparison of the Thermoplastic Composites According to reinforcement type (Source: Ford 2004)

### 2.1.1. Glass Mat Thermoplastic Composites (GMTs)

The compression moulding of glass-mat thermoplastics is used widely for the production of complex semi-structural components, notably for the automotive industry. The technology to manufacture in volume to necessary glass fiber mat from continuous filament material was available in the form of well established non-woven textile technology. Two types of mat are being used; a typical chopped strand mat or a continuous

swirl mat form, impregnated typically with polypropylene. Types of glass mats used in glass mat thermoplastics are shown in Figure 2.3.



(a) (b)

Figure 2.3.Schematic representation of GMTs a) chopped strand mat b)continuous swirl mat (Source: Wakeman et. al. 1999)

After heating to higher than the polymer melting point, the GMT sheets can be moulded relatively easily, providing great freedom of design. Also the process can be totally automated (Ford 2004).

GMT composites have optimum impact resistance and highly rigid structures. Another advantage of the technology is the multiple possibilities for fiberglass distribution and configuration adaptable to the specific characteristics of each part. GMTs are 100% recyclable and have very low densities. The example of parts have good mechanical properties but creep behavior is not as good as thermosets (Biron 2007). Parts made using this technology are; cockpit carrier, spare wheel cavity, pedestrian protection, under-engine shields, deflectors, structural door panels and seat structures. Rear structural support produced from GMT for housing different components such as: spare wheel, jack, tool box, batteries, etc. As an example spare wheel housing produced from glass mat thermoplastic is shown in Figure 2.4.

Figure 2.4.Example of GMT: Spare wheel housing
(Source: Fpksa 2009)

### 2.1.2. Aligned Thermoplastic Composites (ATCs)

Early developments in continuous-aligned fiber thermoplastic composites were driven by the aerospace industry where melt impregnated carbon and PEEK improved toughness compared with conventional carbon and epoxy prepregs. Attempts were made to improve the formability of materials and the commingling of carbon and PEEK filaments led to a prepregs suitable for weaving processes and impregnation during final forming (Wakeman, et al. 1998). Glass mat reinforced thermoplastics were produced to fulfill the need for commodity materials utilizing cheaper raw materials. Production and processing techniques limit the glass fiber content of standard GMTs to 40% by mass, restricting usage to semi-structural applications. More recently, aligned-fiber glass polypropylene composites have been developed to increase fiber content over GMTs for increased structural loading. The important manufacturing techniques for aligned fiber composites include powder impregnation and the commingling of glass and polypropylene. There are several commercial fabrics used in ATC manufacturing and they are given below.

- Twintex R PP (from Saint-Gobain Inc.) is a roving composed of commingled E-glass and polypropylene filaments. It is suitable for filament winding and pultrusion. Consolidation is achieved by heating (180–230°C) under pressure.
- Twintex G PP is based on commingled E-glass and polypropylene rovings. Delivered in pellet form, it is suitable for injection or extrusion-compression. Consolidation is again achieved by heating (180–230°C) under pressure.
- Twintex T PP is a fabric woven with commingled E-glass and polypropylene rovings. It is suitable for pressure moulding. Consolidation is accomplished by heating (180 230°C) under pressure.
- SUPreM, Plytron, Quadrax (Gurit Suprem Inc.): consolidated tapes or fabrics are made from continuous fibres impregnated with thermoplastic powder. The fibres can be glass, aramid, carbon, steel and the matrices are polyethylene, polypropylene, polyamide, PPS, polyetherimide, PEEK, thermoplastic polyimide, or fluorothermoplastic. High levels of fibers can be obtained (Up to 65% in volume) (Biron 2007).

There are many studies performed about aligned glass fiber reinforced thermoplastics in the literature.

Bureau et al. (Bureau, et al. 2002) have been studied mod II interlaminar fatigue crack propagation behavior of unidirectional continuous glass fiber composites with a

polypropylene matrix obtained under three different molding conditions with the use of end-notch flexure geometry. They have confirmed that microstructure and mechanical performance, especially the interlaminar fatigue crack propagation, are strongly affected by the molding conditions.

Hangstrand, et al. (2005) have been studied influence of the void content on the flexural properties of beams manufactured by compression molding multiple unidirectional commingled glass/polypropylene fiber tows. By varying the time under moulding pressure, beams with void contents between 1 and 14% could have been manufactured by them. They have reported that voids had negative effect on flexural modulus and strength. However, voids actually had a clear positive effect on the beam stiffness which increased by about 2% for each 1% of voids.

Zhao, et al. (2009) have been investigated tensile and impact behaviors of stitched glass/polypropylene woven composites. Their data indicated that the stitching in through-the-thickness direction considerably increases the impact damage tolerance especially at low temperature. In addition, it has reported that glass sewing threads does not deteriorate the tensile performance of the stitched composite.

In the study of Perrin, et al. (2003), the mode I interlaminar fracture toughness of unidirectional continuous glass fiber/polypropylene composites above 23$^{o}$C and below (-40$^{o}$C) the glass transition temperature of the PP matrix was investigated. Three molding conditions, leading to different levels of fiber dispersion and matrix microstructure, were studied. They have performed fracture toughness testing employing double-cantilever beam (DCB) specimens. They have reported that molding conditions strongly influenced the fracture toughness of the composites. Similar values of fracture toughness were obtained at the two test temperatures investigated. Also they have reported that, crack propagation occurred either at the fiber-matrix interface or in the matrix interspherulitic regions.

Bigg and Bradbury (1992) have been produced sheet composites of polyethylene and poly(ethylene terephthalate) by melt consolidation of alternating layers of polymer films and glass fiber mats. The composites had a nominal glass content of 50 wt%. they have reported that flexural strength as high as 159 MPa for polypropylene composites and 313 MPa for poly(ethylene terephthalate) composites. The flexural modulus of the polypropylene composites reached 9.1 MPa, whereas the modulus of the stiffest poly(ethylene terephthalate) composite was 15 GPa. Also they have reported impact properties wherein polypropylene composites absorbed up to 257 J/cm during an

instrumented falling dart impact test, on the other hand poly(ethylene terephthalate) composites absorbed as much as 116 J/cm in the same test.

## 2.2. Raw Materials for Thermoplastic Composites

### 2.2.1. Matrix Materials

The common reinforced thermoplastic matrixes used to prepare composites are nylons and polypropylenes. Other reinforced thermoplastics are particularly PET, PBT,PC and PPS. The matrix holds the fibers in their proper position; protects the fibers from abrasion; transfers loads between fibers; and provides interlaminar shear strength. A properly chosen matrix is expected to provide resistance to heat, chemicals and moisture; have a high strain to failure and not be toxic (Campbell 2006, Ishak, et al. 2007).

#### 2.2.1.1. Polypropylene

Polypropylene (PP) has an increasing value as an injection moldable material with capability of offering usable relatively low cost engineering properties, is now the second most important reinforced thermoplastic, in volume terms, after reinforced nylon, and it could possibly overtake this group (Rosato and Rosato 2004).

Until 1954, most attempts to produce plastics from polyolefin's had little commercial success, and only the polyethylene (PE) family was commercially important. It was in 1955 that Italian scientist F.J.Natta announced the discovery of PP. It is not surprising that PP and PE have many of the same properties. Although they are similar in origin and manufacture, PP has become a strong competitor of PE (Richardson and Lokensgard 1997).

Particularly for the automotive and appliance industries (and in reinforced structural foam compounds) PP compounds reinforced with glass fiber, talc or mica are

widely used. To make it possible to bond glass fiber to a PP matrix, special chemical coupling materials and technologies have been developed. Long-fiber and continuous fiber reinforcement technology with PP produce molding materials with higher tensile strength and semi-finished materials such as sheet and tape which are beginning to find applications, mainly in structural parts. Both glass- and mineral-reinforced PPs appear to have greatest potential in the automotive industry, the former for lightweight structural parts such as bumper supports, where the mass-production advantage of injection molding can be utilized, and the latter for many general applications such as interior components, where acrylonitrile-butadienestyrene (ABS) is being partly replaced (Rosato and Rosato 2004). A growing advantage of reinforced PP is its facility for recycling, and many producers now have programs to take back used or scrapped parts for recovery and reprocessing.

Because of its characteristic low density, good process ability by all thermoplastic methods and excellent electrical insulation, PP is considered one of the most promising thermoplastic matrices for many industrial applications (Greco, et al. 2007, Richardson and Lokensgard 1997). Other advantages of the polypropylene are its low coefficient of friction, good fatigue resistance and good grade availability. Also polypropylene has excellent moisture resistance, very good chemical resistance, excellent flexural strength and good impact strength (Richardson and Lokensgard 1997). Fiber reinforcement enhances polypropylene mechanical properties, such as stiffness and fracture resistance, and limits the material deformation under creep loads (Greco, et al. 2007). Some typical properties of common thermoplastics are given in Table 2.1.

Table 2.1. Comparison of typical properties of three common thermoplastics used in composite materials at 20 $^{o}$C (Source: Hull 1995)

| Property | Units | Poly-propylene | Nylon 6.6 | Poly-carbonate |
|---|---|---|---|---|
| **Density** | Mg $m^{-3}$ | 0.90 | 1.14 | 1.06-1.2 |
| **Youngs Modulus** | GN $m^{-2}$ | 1.0-1.4 | 1.4-2.8 | 2.2-2.4 |
| **Poisson's ratio** | | 0.3 | 0.3 | 0.3 |
| **Tensile-Yield strength** | MN $m^{-2}$ | 25-38 | 60-75 | 45-70 |
| **Elongation to ubreak** | % | >300 | 40-80 | 50-100 |
| **Thermal Conductivity** | W $m^{-1}$ $^{o}$C | 0.2 | 0.2 | 0.2 |
| **Coefficient of thermal expansion** | $10^{-6}$ $^{o}$C | 110 | 90 | 70 |
| **Melting point** | $^{o}$C | 175 | 264 | - |
| **Water absorption 24h to 20 $^{o}$C** | | 0.03 | 1.3 | 0.1 |

The low glass transition temperature of PP severely limits its applicability when a long service life at moderate to high temperatures is required (Bureau and Denault 2004, Greco, et al. 2007). Other drawbacks of polypropylene are its low rigidity, risk of sensitivity to UV, flammability and difficult gluing (Biron 2007, Richardson Lokensgard 1997) .

### 2.2.2. Fiber Materials

The second important component of the composite is the fibers. The primary role of the fibers is to provide strength and stiffness (Campbell 2006). Reinforcement fibers can also significantly improve the structural characteristics of thermoplastics and thermosets. They are available in continuous forms and chopped forms having different lengths, or discontinuous in form (whiskers, flakes, spheres, etc.) to meet different properties and processing methods (Rosato and Rosato 2004). A wide variety of fiber materials are available for the polymer composites. The most commonly used fibers in polymer matrices are carbon, glass and aramid type fibers (Hull 1995). Although they are relatively more expensive, boron fibers also find some applications. Alumina, silicon carbide, silicon nitride, and other ceramic fibers and metal wires have still limited use as well. Typical properties for some fibers and some bulk materials are given in Table 2.2.

Table 2.2. Properties of some fibers and some bulk materials (Source: Gutowski 1997)

| Materials | $E_{11}$ (GPa) | $\sigma_{11}$ or $\sigma$ (MPa) | Maximum Strain (%) | Density ($g/cm^3$) | $T_{max}$ ($^oC$) |
|---|---|---|---|---|---|
| **Boron** | 400 | 3600 | 1 | 2.53 | 500 |
| **Graphite: stiff** | 300 | 5313 | 1.8 | 1.78 | 500 |
| **Graphite: strong** | 248 | 4071 | 1.65 | 1.8 | 500 |
| **Kevlar 49** | 138 | 3034 | 2.3 | 1.44 | 160 |
| **Kevlar 29** | 97 | 3275 | 3.9 | 1.44 | 160 |
| **S-glass** | 85 | 4585 | 5.7 | 2.48 | 650 |
| **E-glass** | 72 | 3448 | 4.8 | 2.54 | 550 |
| **Steel-SS410** | 200 | 1000 | 20 | 7.8 | 780 |
| **Aluminum (2024)** | 73 | 469 | 20 | 2.8 | 330 |
| **Wood (hickory)** | 15 | 76 | 0.5 | 0.7 | 100 |
| **Plate glass** | 70 | 70 | 0.1 | 2.5 | 500 |

### 2.2.2.1. Aramid Fibers

Aramid fiber (e.g., Kevlar) is an organic fiber that has a low density and is extremely tough, exhibiting excellent damage tolerance. Although it has a high tensile strength, it performs poorly in compression. It is also sensitive to ultraviolet light and should be limited to long-term service at temperatures less than 350F. Kevlar, developed by Du Pont Corp., is composed of poly(1,4-phenyleneterepthhalamide). Two forms, Kevlar 29 and Kevlar 49, are available. Another organic fiber is made from Ultra-High Molecular Weight Polyethylene (UHMWPE). That has a low density with excellent radar transparency and a low dielectric constant. Due to its low density, it exhibits a very high specific strength and modulus at room temperature. However, being UHMWPE, it is limited to temperature of about 290 $^{0}$C or lower. Similar to aramid, UHMWPE has excellent impact resistance; however, poor adhesion to the matrix is a problem. However, plasma treatments have been developed to improve the adhesion at the fiber/matrix interface (Campbell 2006).

### 2.2.2.2. Carbon Fibers

Carbon fiber contains the best combination of properties but is also more expensive than either glass or aramid. Carbon fiber has low density and coefficient of thermal expansion (CTE), and it is electrically and thermally conductive. It is structurally very efficient and exhibits excellent fatigue resistance. It is brittle (strain-to failure less than 2%) and exhibits low impact resistance. Being conductive, it may cause galvanic corrosion if placed in direct contact with aluminum. Carbon fiber is available in a wide range of strength and stiffness, with strengths ranging from 300 to 1000 ksi and moduli ranging from 30 to 145 msi. With this wide range of properties, carbon fiber is frequently classified either as: (1) high strength, (2) intermediate modulus, or (3) high modulus (Campbell 2006).

### 2.2.2.3. Glass Fibers

Glass fibers, the most widely used at over 90% of all reinforcements with thermoplastic or thermoset matrices, are available in many forms for producing different commercial and industrial products. They also include parts in aircraft to space vehicles and surface water to under water vehicles (Rosato and Rosato 2004). Many different compositions of mineral glasses have been used to produce fibers. The most common are based on silica ($SiO_2$) with additions of oxides of calcium, boron, sodium, iron and aluminum. These glasses are usually amorphous although some crystallization may occur after prolonged heating at high temperatures. This usually leads to a reduction in strength properties. Typical compositions of the three well known glasses used for glass fiber composite materials are given in Table 2.3.

Table 2.3. Composition of glass used for fiber manufacture (Source: Hull 1995)

| **wt%** | **E glass** | **C glass** | **S glass** |
|---|---|---|---|
| **$SiO_2$** | 52.4 | 64.4 | 64.4 |
| **$Al_2O_3$, $Fe_2O_3$** | 14.4 | 4.1 | 25.0 |
| **CaO** | 17.2 | 13.4 | -- |
| **MgO** | 4.6 | 3.3 | 10.3 |
| **$Na_2O$, $K_2O$** | 0.8 | 9.6 | 0.3 |
| **$Ba_2O_3$** | 10.6 | 4.7 | -- |
| **BaO** | -- | 0.9 | -- |

E glass (E for electrical) is the most commonly used glass because it draws well and has good strength, stiffness, electrical and weathering properties. C glass (C for corrosion) has a higher resistance to chemical corrosion than E glass but it is more expensive and has lower strength properties. S glass is more expensive than E glass but it has higher Young's modulus and temperature resistance. It is used in special applications such as the aircraft industry where the higher modulus may justify the extra cost (Hull 1995).

Glass fibers have elastic modulus in the range of 50-90 GPa, much higher than the polymer but lower than carbon fibers. All types of glass fibers have high thermal resistance, low coefficient of thermal expansion, high density and insulating properties. On

the other hand, glass fibers are brittle under high stresses during processing and their abrasive properties harmful for tools (Biron 2007).

## 2.3. Processing of Textile Preforms

Compared with traditional metals and laminated composites, textile composites have many advantages due to their high specific stiffness, high strength, low weight, nice integral performance, low thermal expansion and good corrosion resistance. Most important is that, textile composites are more flexible than metals and possess a high capacity to conform to complicated contours; therefore, they are particularly suitable for manufacturing components with complex shape (Zhu, et al. 2007). In textile process, there is direct control over fiber placements and ease of handling of fibers. Textile technologies also provide homogenous distribution of matrix and reinforcing fiber. Thus textile preforms are considered to be the structural backbone of composite structures. Textile industry has the necessary technology to weave high performance multifilament fibers such as glass, aramid and carbon, which have high tensile strength, modulus and resistance to chemicals and heat in to various types of preforms (Alagirusamy, et al. 2006, Pandiata, et al. 2002).

### 2.3.1. Hybrid Yarn Manufacturing Techniques

Hybrid yarn manufacturing has been developed recently for rapid and cost-effective processing of continuous fiber reinforced thermoplastic composites, aimed at light weight components for passenger and commercial vehicles, rail vehicles, agricultural machineries, as well as for aerospace vehicles. Because of the very short flow paths of the viscous thermoplastic melt, hybrid yarns offer an ideal opportunity to achieve short cycle times (Mader, et al. 2008). Hybrid yarn manufacturing techniques are given below.

### 2.3.1.1. "Direct Twist Covering" Technique

Fiber twisting is carried out by the new technique, which is called "Direct Twist Covering". With DirectTwist covering technique, two types of hybrid yarns are being produced. One of them is single (S) twist and the other is double (SZ) twist. In fiber twist technique, it is possible to adjust thermoplastic fiber (i.e.,PP) and glass fiber composition by controlling fiber and twist number (Agteks 2009). Schematic representation of direct twist covering technique and types of hybrid yarns are given in Figures 2.5 and 2.6, respectively.

Figure 2.5. Direct Twist, Covering technique
(Source: Agteks 2009)

**(a)** **(b)**

Figure 2.6. (a) Single twist (b) double twist
(Source: Agteks 2009)

In single twist method; hybrid yarn is produced by twisting thermoplastic fiber around the reinforcement fiber by making “S” shape. On the other hand, in double twist method, hybrid yarn is produced by twisting thermoplastic fiber around the reinforcement fiber by making both “S” and “Z” shapes (Agteks 2009).

#### 2.3.1.2. Air Jet Texturing Technique

Air-jet texturing is a purely mechanical process and can be used to combine reinforcing and matrix forming filaments. Figure 2.7 shows the schematic of the air-jet texturing process and the structure of the air-textured yarn is demonstrated in Figure 2.8. In this process, supply yarn is overfed in the turbulent zone where compressed air is directed mainly parallel to the yarn path, resulting in shifting of the filaments longitudinally together with the formation of filament loops. This action opens up filament bundles, and then builds mingling sections. The heart of the air-jet texturing process is the air nozzle. The purpose of the nozzle is to create a supersonic, turbulent and non uniform flow to entangle or blend the filaments forming them into loops to produce stable textured yarns. Some texturing nozzles have impact elements of different sizes and shapes at the exit of the nozzle, aiming to improve process stability and quality of the textured yarns (Alagirusamy, et al. 2006). Air jet texturing is an inexpensive and fully mechanical process, with great potential to lead the development of fiber reinforced composites with good adhesion properties (Koc, et al. 2008).

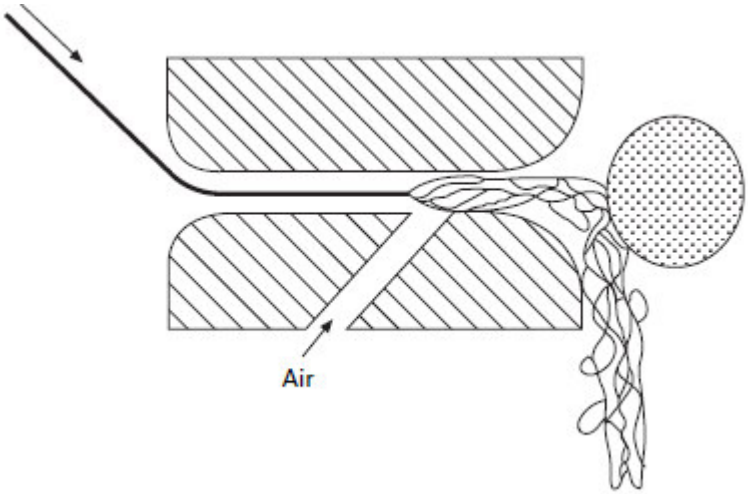



Figure 2.7. Air-jet yarn texturing
(Source: Alagirusamy, et al. 2006)

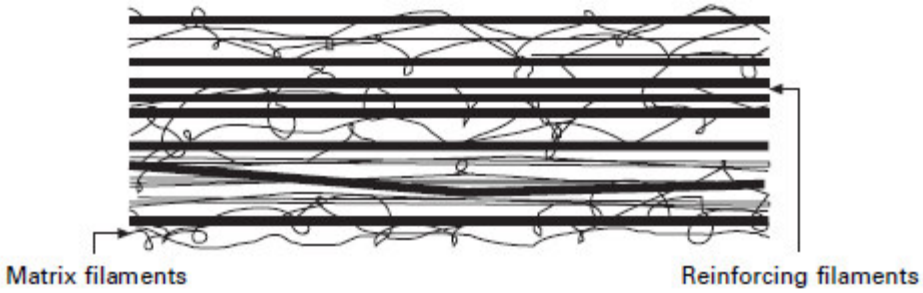



Figure 2.8. Structure of air-jet textured hybrid yarn
(Source: Alagirusamy, et al. 2006)

### 2.3.1.3. Commingling Technique

Commingling is one of the more promising routes for pre-impregnation, with relatively high and uniform pre-impregnation quality and low cost (Wysocky, et al. 2005). In the mingling process, rapidly moving air in an air jet is used to generate entanglements in and among filaments. Figure 2.9 shows a schematic of the intermingling process and the structure of the intermingled yarn.

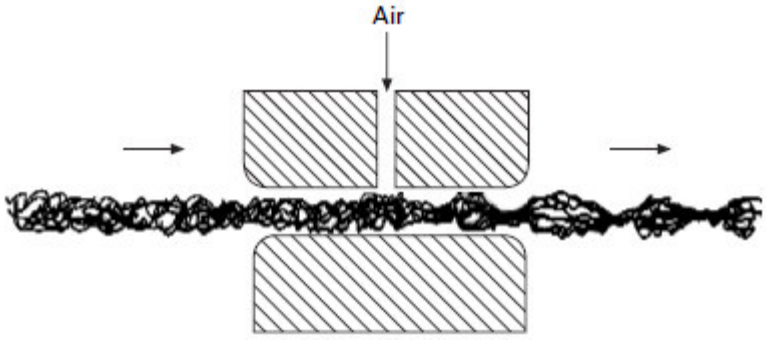



Figure 2.9. Hybrid yarn by commingling process
(Source: Alagirusamy, et al. 2006)

The mingling process of two or more yarns to form a single strand of yarn can be defined as commingling. Commingled yarn consists of a blended combination of reinforcing filament yarn and filament yarn spun from thermoplastic polymers, as represented in the Figure 2.10. The multifilament yarns are scattered amongst one another at filament level (Alagirusamy, et al. 2006, Ye, et al. 1995).

Long et al. (2001) have been performed series of experiments to determine the effects of rate, temperature and holding pressure on the consolidation of glass/polypropylene commingled fabric. They have reported that increasing rate resulted in increased consolidation pressure, although significant shear thinning occurred even at modest closure speeds. Also they have reported that increased rate led to an increase in void content at the end of the consolidation phase and application of pressure during cooling resulted in a dramatic decrease in void content. It was observed that at the end of consolidation the remaining voids were predominantly in the matrix rich regions between tows.

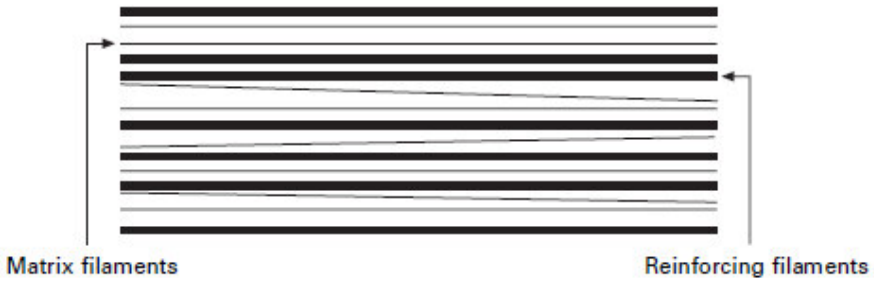



Figure 2.10. Structure of commingled hybrid yarn
(Source: Alagirusamy, et al. 2006)

### 2.3.1.4. Parallel Winding Technique

In this much simpler process, two components of hybrid yarns are led side-by-side to each other, as shown in Figure 2.11.

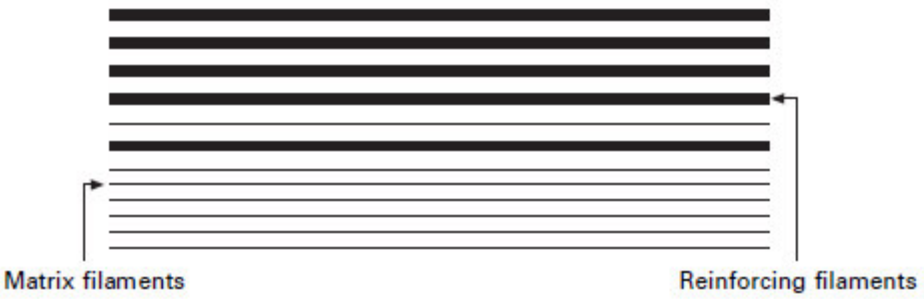



Figure 2.11. Structure of side-by-side hybrid yarn
(Source: Alagirusamy, et al. 2006)

### 2.3.1.5. KEMAFIL Technology

This technology has been developed by Saxon Textile Research Institute, Chemnitz, Germany. It is a turning thread technique. By means of mechanical interlacing of yarns into a knitted structure, linear textiles are produced. KEMAFIL machines are circular knitting machines operating with loopers that are arranged around a guide bar and give a tubular knitted structure which can cover any type of core yarn. In this type of hybrid yarn, a parallel arrangement of matrix fibers is surrounded by parallel reinforcing filaments. The entire structure of matrix and reinforcing filaments is placed in the core in a sheath of matrix fibers as the skin. The yarn structure is as shown in Figure 2.12 (Alagirusamy, et al. 2006, Lauke, et al. 1998).

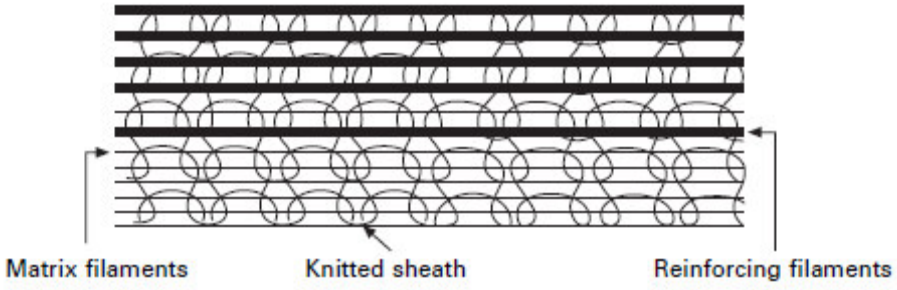



Figure 2.12. Structure of KEMAFIL hybrid yarn
(Source: Alagirusamy, et al. 2006)

## 2.3.2. Textile Preforming

### 2.3.2.1. Woven Fabrics

Woven fabrics are fabricated by the interlacing of yarns. There are hundreds of possible woven fabric combinations, which can be divided in to biaxial and triaxial woven structures according to in-plane fiber orientation (Alagirusamy, et al. 2006, Gutowski 1997). Woven-fabric reinforced composites have attracted a significant amount of attention from both industry and academia due to their high specific strength and stiffness, as well as their supreme formability characteristics (Cao, et al. 2008, Zhu, et al. 2007). In addition woven, textile composites are damage tolerant due to resistance offered by interlacing tows to crack propagation (Launay 2008). Also, woven fabrics have high fracture toughness and ease of handling. Woven fabric composites balanced in-plane properties and transverse tensile strength are much higher than that of unidirectional composites (Asi 2009). In Figure 2.13 basic 2D woven structures are shown.

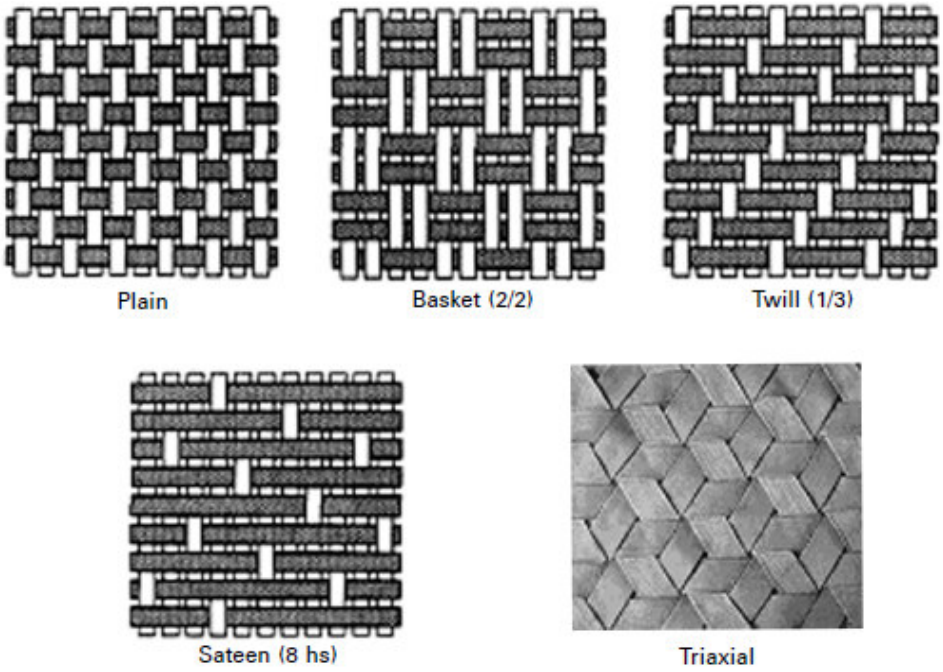



Figure 2.13. Basic 2D weave structures
(Source: Alagirusamy, et al. 2006)

The plain woven fabric is symmetrical, with good stability and reasonable porosity. Also this weave has the highest frequency of yarn interlacing. However, it is the most difficult of the weaves to drape and the high level of fiber crimp imparts relatively poor mechanical properties compared with the other weave styles (Cavallaro, et al. 2003). In twill fabrics, warp yarns alternately weave over and under two or more weft yarns in regular repeated manner. Superior drape is seen in the twill weave and also slightly better mechanical properties over the plain weave. Satin weaves are fundamentally twill weaves modified to produce fewer intersections of warp and weft. Therefore, the low crimps give good mechanical properties. Basket weave is fundamentally the same as plain weave except two or more warps alternately interlace with two or more wefts. Basket weave is stronger than plain weave but it shows poor stability. Triaxial weave has 90+/-60 yarns oriented in one plane, resulting in a high level of in-plane shear resistance. High levels of isotropy and dimensional stability can be achieved with triaxial weave at low fiber volume fractions (Alagirusamy, et al. 2006, Gutowski 1997). There have been several weaving techniques to produce multiaxial, multilayer 3D preforms that include lappet weaving, tri-axial weaving and pile weaving.

### 2.3.2.2. Knitted Fabrics

Knitted fabrics are interloped structures wherein the knitting loops are either produced by the introduction of the knitting yarn in the cross-machine direction (weft knit) or along-the-machine direction (warp knit). Large number of stitch geometries can be produced by knitting. In Figure 2.14 basic knitted structures are shown.

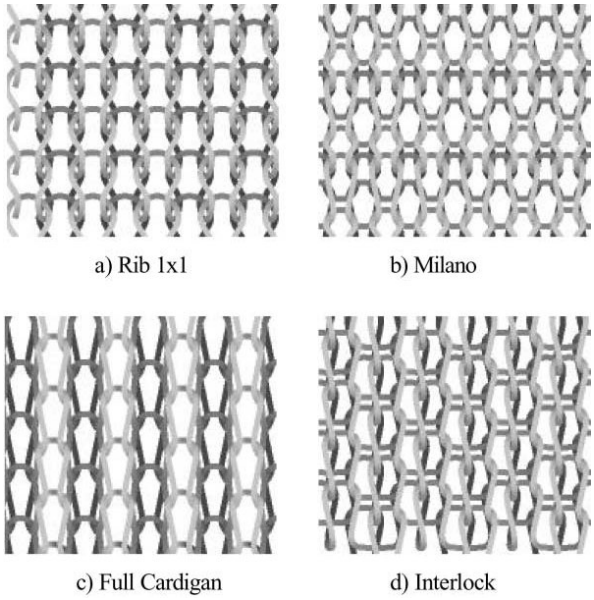



Figure 2.14. Examples for knitted structures
(Source: Pandiata, et. al. 2002)

Because of the interloped nature, the maximum fiber packing density of knitted structures is lower than that of woven fabrics. Knitted fabric composites are one of the textile fabric composites that are gaining interest, because of the excellent drapability of knitted fabric pre-pregs and hence the possibilities for near-net-shape manufacture of fiber preforms (Pandiata, et al. 2002). As fibers in knitted structures are oriented not only in the in-plane directions, but also in the thickness direction, the through the- thickness properties of knitted fabric composites are highest among unidirectional fiber reinforced composites and other textile fabric composites such as woven and braided fabric composites. However, the on-axis mechanical properties of knitted fabric composites are lower than the on-axis (weft or warp) mechanical properties of woven fabric composites. This is due to knitted fabric composites having a lower fiber volume fraction and fibers being less oriented to the wale or course directions. The on-axis mechanical properties of knitted fabric composites

are comparable to the off-axis mechanical properties of woven fabric composites like in the bias ($45^o$) directions (Khondker, et al. 2005, Pandiata, et al. 2002). Examples of 3D knitted preforms are shown in Figure 2.15.

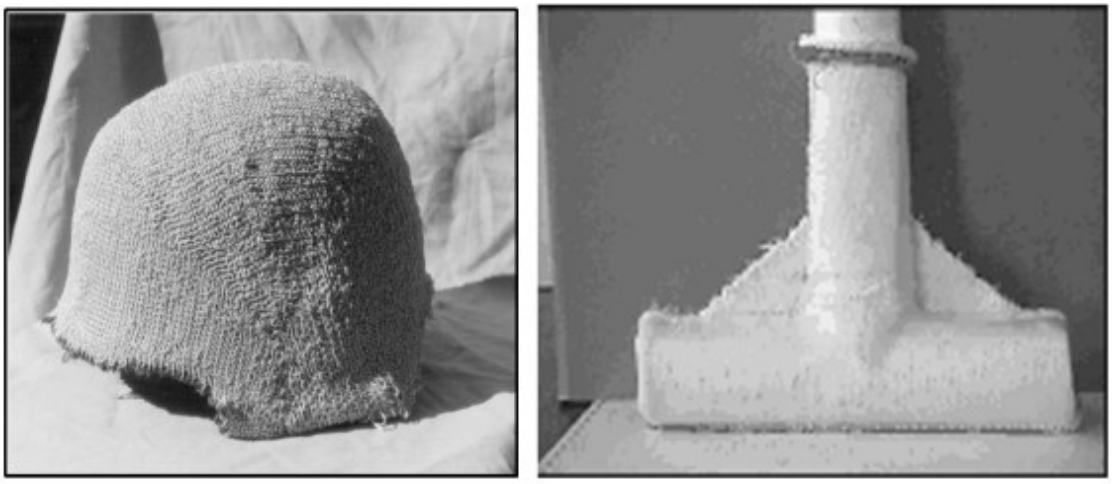


Figure 2.15. Examples of 3D flat knitted preforms
(Source: Alagirusamy, et al. 2006)

### 2.3.2.3. Braided Fabrics

Braiding is a composite material preform manufacturing technique where a braiding machine deposits continuous, intertwined, fiber tows to create desired reinforcing braid architecture before or during the impregnation of the fibers Ayrancı an Carey 2008). Braided fabrics can be produced in flat or tubular (Gutowski 1997). There are three commonly used braid architectures: Hercules braid, regular braid, diamond braid. Hercules braid is a braid where each yarn passes over and then above three other yarns, where in regular braid each yarn crosses over and below two yarns, and finally if each yarn crosses over and below one other yarn in a repeating manner, it is called a diamond braid (Ayrancı and Carey 2008). Adding axial fibers along the mandrel axis is called a triaxial braid, and it increases bending and tension strength and also stiffness of braided composite materials.

The feature of braided laminated composite is that reinforcing fiber yarns are continuously braided with a braiding angle. This continuous yarn alignment is capable to achieve fracture tolerance under multiple loading conditions as compared to conventional unidirectional fiber laminated composites. Hence, braided laminated composites have been

applied to severe stress applications such as I-beam and energy absorption rod (Fujihara, et al. 2007). Example of an Braid architecture is shown in Figure 2.16.

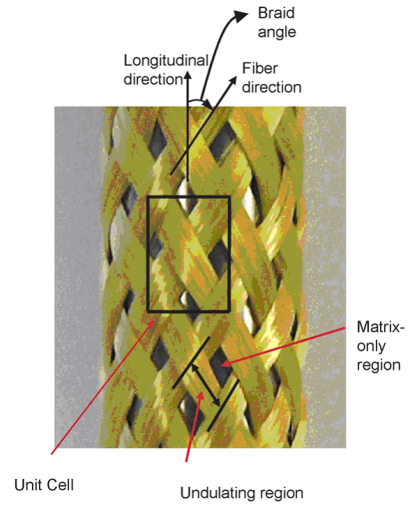


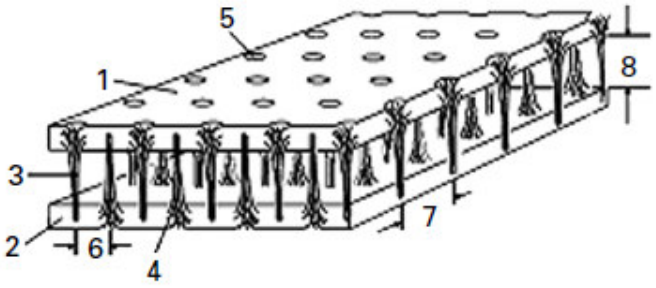

Figure 2.16. Braid architecture
(Source: Ayrancı and Carey 2008)

### 2.3.2.4. Non-Woven Fabrics

Nonwoven structures are fiber-to-fabric assemblies produced by chemical, thermal or mechanical means, or a combination of these (Figure 2.17). The thickness of sheets may vary from 25 μm to several centimeters, in weights from 10 g/cm$^2$ to 100 g/cm$^2$. Nonwovens have densities less than those usually demanded in structural composites. As nonwovens become more readily available with greater ranges of properties, the market for composites is also expected to increase (Alagirusamy, et al. 2006). They are flat, porous sheets that are made directly from separate fibers or from molten plastic or plastic film. They are not made by weaving or knitting and do not require converting the fibers to yarn.

Nonwoven fabrics are engineered fabrics that may be a limited life, single-use fabric or a very durable fabric. Nonwoven fabrics provide specific functions such as absorbency, liquid repellency, resilience, stretch, softness, strength, flame retardancy, washability, cushioning, filtering, bacterial barrier and sterility. These properties are often combined to create fabrics suited for specific jobs, while achieving a good balance between product use-life and cost. They can mimic the appearance, texture and strength of a woven fabric and can be as bulky as the thickest paddings. In combination with other materials

they provide a spectrum of products with diverse properties, and are used alone or as components of apparel, home furnishings, health care, engineering, industrial and consumer goods.




| | | | |
|---|---|---|---|
| 1, 2 | Nonwovens | 6 | feed space |
| 3 | bundle of fibre | 7 | space between rows of needles |
| 4, 5 | formation of funnels | 8 | space between nonwoven |

Figure 2.17. 3D needle punched nonwoven fabric
(Source: Alagirusamy, et al. 2006)

## 2.4. Manufacture of Thermoplastic Composites

Applications and the processing techniques are important for the structure of thermoplastics. Pellet and granule forms of short fiber reinforced and elastomer toughened thermoplastics are available for processing and injection or extrusion. Long or continuous fiber reinforcement is used in order to obtain the necessary mechanical properties. Pre-impregnated continuous fiber reinforced thermoplastics generally are in the form of a fabric. The fabric can then be molded using various stamping and thermoforming methods. Complex shapes are harder to process because of the oriented nature of the fibers. The major manufacturing techniques of thermoplastic composites are; compression molding, filament winding, pultrusion, injection molding, autoclave technique and diaphragm forming processes.

### 2.4.1. Compression molding

This process is a flow-forming process in which the heated composite sheet is squeezed between the mold halves to force resin and reinforcement fibers to fill the cavity. This is the only thermoplastic manufacturing process used in industry for making structural thermoplastic parts.

Another type of compression molding is the hot press technique in which intermediate thermoplastic materials such as prepregs and hybrid yarns are used. The fiber volume fraction achieved by this process is greater than 60% (Alagirusamy, et al. 2006). Schematic representation of compression molding is shown in Figure 2.18.

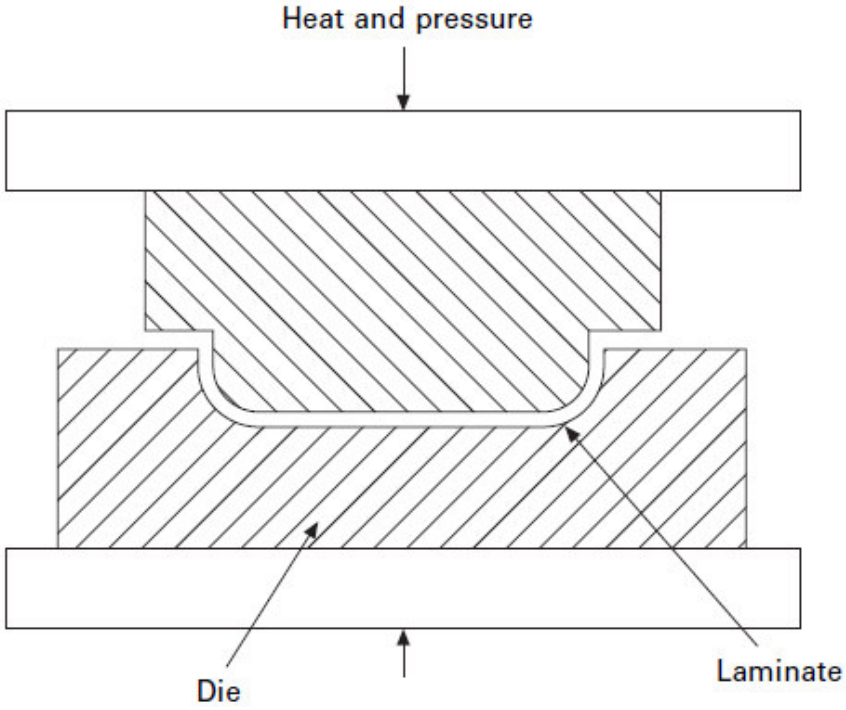



Figure 2.18. Hot press compression molding process
(Source: Alagirusamy, et al. 2006)

Trudel-Boucher (2006) have been investigated the stamp forming process of two unconsolidated PP/GF fabric for a simple mold geometry. The influence of stamping pressure, mold temperature, loading rate and holding time, have been determined on the void content and void distribution. They have shown that void distribution was very similar for most stamping pressure. They have found good correspond between variation of the flexural properties and variation of the void content

### 2.4.2. Filament winding

Thermoplastic filament winding, also called tape winding, is a process in which a thermoplastic prepreg tape or hybrid yarn is wound over a mandrel, and heat and pressure are applied at the contact point of the roller and the mandrel for melting of the thermoplastic and consolidation. A schematic representation of the process is shown in Figure 2.19. In this process, lay down, melting, and consolidation are obtained in one single step.

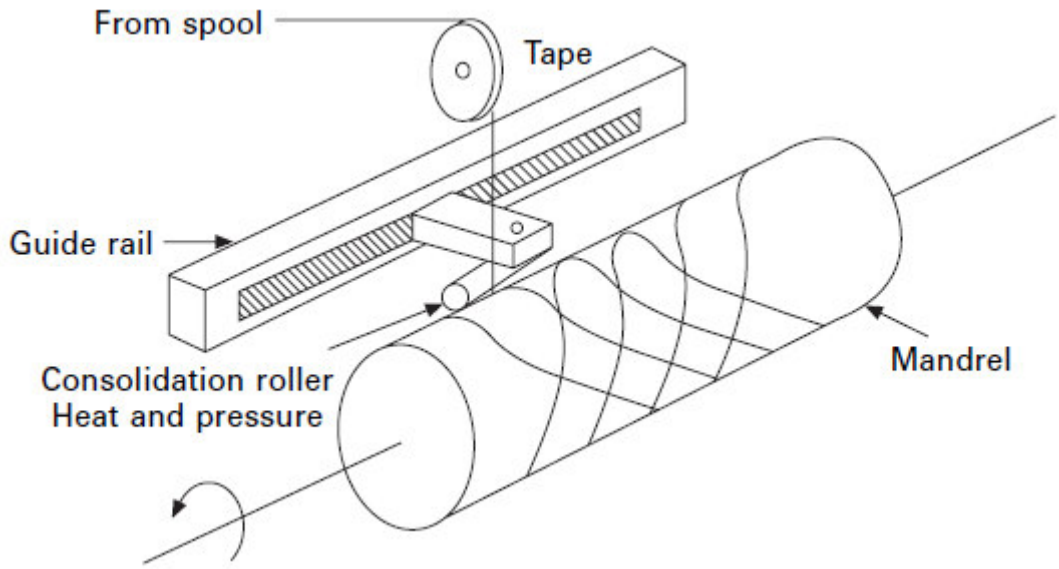



Figure 2.19. Filament winding Process
(Source: Alagirusamy, et al. 2006)

### 2.4.3. Pultrusion

Among continuous methods of composite manufacture, pultrusion is an important process in which composites with precision cross-sections are prepared. Although thermosetting resins were favored for this method, recent trends towards utilization of thermoplastic matrices are being adopted with intermediate commingled yarns to facilitate impregnation. A key requirement for the pultrusion of thermoplastic composites is a means of applying sufficient heat and pressure to consolidate the impregnated rovings as they are

formed by the die. The intermediate hybrid yarn is fed from the spools through a preheating chamber into a forming die. The forming die consists of two, heating and cooling; the thermoplastic matrix is melted in the heating section to achieve the impregnation and subsequently, the resin impregnated fibers are solidified by and formed into the desired cross sectional shapes by the cooling die. A schematic of such a thermoplastic pultrusion process is shown in Figure 2.20.

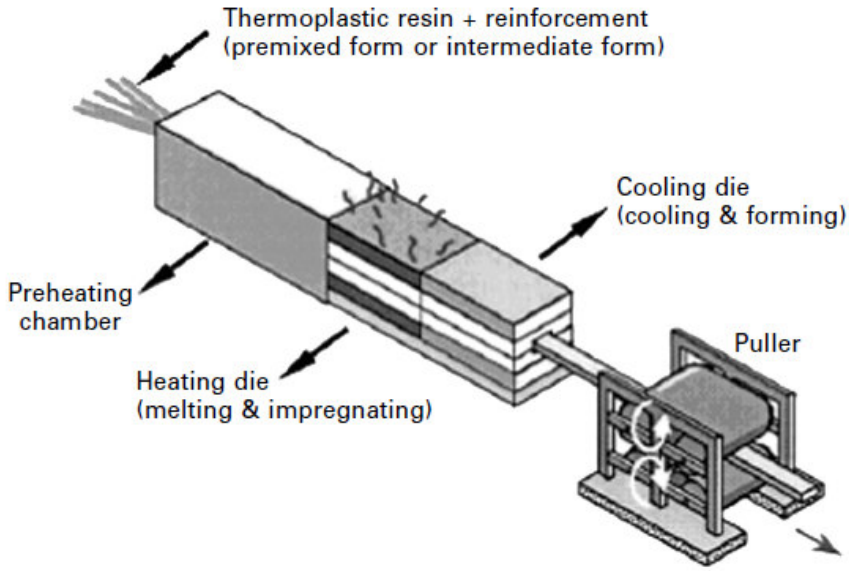



Figure 2.20. Pultrusion Process
(Source: Alagirusamy, et al. 2006)

### 2.4.4. Autoclave Molding

Autoclave molding is a process of thermoplastic composite manufacture in which the fibrous reinforcement and thermoplastic matrix are laid down on a tool in the desired sequence and is spot welded to make sure that the stacked plies do not move relative to each other. The entire assembly is then vacuum bagged and placed inside an autoclave. Following the process cycle, the part is removed from the tool. Intermediate forms of thermoplastic composites such as those from hybrid yarns and prepregs offer better process ability in this technique. The process is similar to the hot pres technique, the only difference being the method of applying pressure and heat. Composites for aerospace applications are the major manufactured products using this technique due to versatile fiber

orientation, and higher fiber volume fraction and quality of the material produced (Alagirusamy, et al. 2006).

### 2.4.5. Inflation Molding

The bladder inflation molding technique, also known as diaphragm forming, is an economically competitive process for the production of thermoplastic composites with complex hollow parts, which overcomes some of the limitations of filament winding, rotor molding and pultrusion. Inflation molding technique, which is shown in Figure 2.21, involves the placement of a composite preform around an expandable polymer mandrel, also known as a bladder. The composite/bladder assembly is then positioned in a mould and placed in a hot press. While the composite material is being heated, the bladder is inflated so as conform the preform to the shape of the mould cavity. Once the thermal cycle is completed, the part is removed from the mould and the bladder is extracted, leaving a thin-walled hollow composite structure.

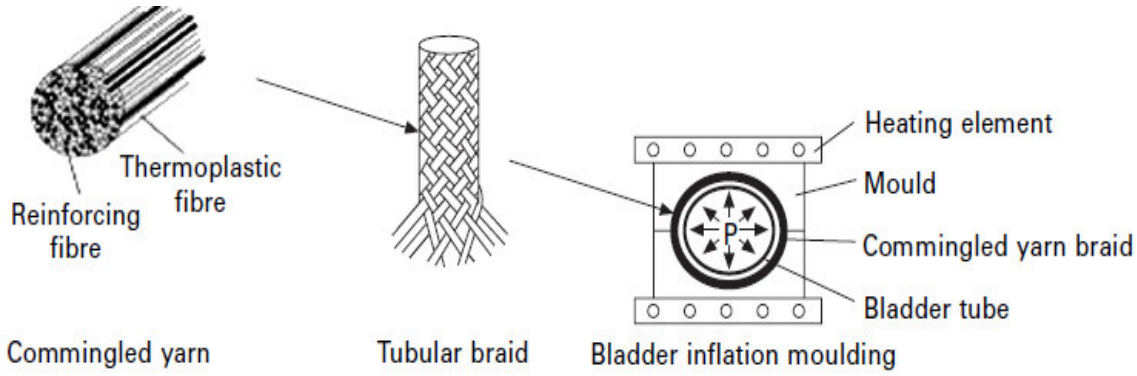



Figure 2.21. Inflation Process
(Source: Alagirusamy, et al. 2006)

### 2.4.6. Injection Molding

Injection molding is the predominant process for the production of thermoplastics into finished products. Applications are increasing with fiber-filled thermoplastics but the technique can only accommodate short fiber reinforcement, hence it has limitations for the adoption of hybrid yarn raw materials. Injection molding of thermoplastics is the process of choice for a tremendous variety of parts, and it is estimated that approximately 25% of all thermoplastic resins are used for injection molding (Alagirusamy, et al. 2006).

## 2.5. Applications of Thermoplastic Composites

Due to the light weight and toughness thermoplastic composites have been adopted by the automotive industry (Bureau and Denault 2004, Varatharajan, et al. 2006, Wakemen, et al. 1998). Thermoplastic composites are considered candidate structural materials for light weight and fuel efficient automobiles of the future. Potential applications of these materials include floor pans, body side interior panels, foot support demonstrator and prototype door cassette (Greco, et al. 2007, Trudel-Baucher, et al. 2006). Also seat frames, battery trays, bumper beams, load floors, front ends, valve covers, and under engine covers are made up of thermoplastic composites.

Thermoplastic composites have found limited use in the aerospace industry and came about due to the need for tougher composites. They are analogous to the first thermoset composites with fiber contents above 50 vol% and utilize a highly aligned continuous fiber structure. Actual applications include missile and aircraft stabilizer fins, wing ribs and panels, fuselage wall linings and overhead storage compartments, ducting, fasteners, engine housings and helicopter fairings (Ishak, et al. 2007, Varatharajan, et al. 2006, Wakeman, et al. 1998).

Thermoplastic composites are used in the construction industry for structural profiles, pipes, concrete rebars and lightweight structural and insulating panels (Wakeman, et al. 1998).

The materials handling industry benefit from these materials in the form of pallets and cargo containers. Thermoplastic composites are also being used in defense,

transportation and marine industries (Bureau and Denault 2004, Ishak, et al. 2007, Wakeman, et al. 1998).

## 2.6. Unique Contributions to Literature

In this study, polypropylene and glass fibers were commingled by direct twist and air-jet hybrid yarn preparation techniques and non-crimp fabrics were prepared from these yarns. Also thermoplastic composites were fabricated from the prepared fabrics. Mechanical and thermal properties of the laminates produced from these fabrics were investigated. The properties of the composites were compared based on the hybrid preparation technique, fiber types and composite processing parameters such as temperature and pressure.

Adhesion at the fiber matrix interface is very important on the mechanical properties of the composite materials. In this study effect of the type of the sizing applied on glass fiber on the mechanical and thermal properties were evaluated.

To our knowledge, there is no similar study on the investigation of effects of commingled fabric preparation technique, fiber type, sizing and processing parameters on the composite preparation.

# CHAPTER 3

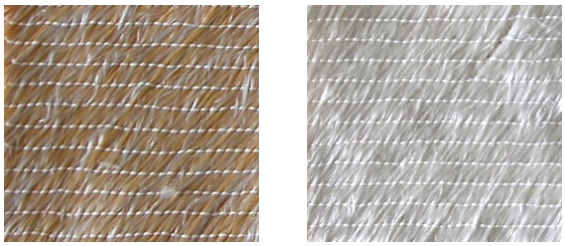
# EXPERIMENTAL

## 3.1. Materials

In this study, non-crimp glass fiber/PP fabrics (± $45^0$ biaxial glass) were used to manufacture thermoplastic based composite material. PP and glass fibers were used as a matrix and reinforcement constituent respectively. Used non-crimp fabrics were developed in collaboration with TELATEKS A.Ş.

At the first stage of the study, non-crimp fabrics were obtained by feeding glass and PP fibers from separate bobbins to the multiaxial machine in order to obtain textile fabrics. These fabrics were used to prepare composite material. Based on these first trials, it was decided to use textile fabrics which are produced from hybrid commingled yarns of polypropylene and glass fiber.

At the second stage, two different types of hybrid non-crimp glass fiber/PP fabrics, which differ from each other by the hybrid yarn manufacturing techniques, were prepared. The techniques used were direct twist covering technique (Agteks 2005) and air jet texturing technique (Alagirusamy, et al. 2006). Two types of hybrid yarn were produced with direct twist covering technique. One of them was single (S) twist and the other was double (SZ) twist. In single twist method; hybrid yarn was produced by twisting polypropylene fiber around the glass fiber by making "S" shape. On the other hand, double twist hybrid yarn was produced by twisting polypropylene fiber around the glass fiber by making both "S" and "Z" shapes. Produced hybrid fabrics are shown in Figure 3.1.



(a) (b)

Figure 3.1. Glass fiber/PP hybrid ± $45^0$ non-crimp fabrics a) air-jet technique b)direct twist covering technique

Properties of produced glass fiber polypropylene non-crimp hybrid fabrics are given in Table 3.1. In this table PES(E5) indicates that the type of rope used for sewing plies of non-crimp fabrics.

For fabric sample ID D2, D3, D4 and D5, glass fibers with polyester resin compatible sizing were used. On the other hand, for fabric D8, polypropylene resin compatible sizing applied glass fiber was used. Table 3.2.shows the differences of the fabrics according to hybrid yarn preparation technique and glass fiber sizing. Glass fiber weight percentages were calculated during fabric production.

Table 3.1 Properties of Glass fiber/PP non-crimp hybrid fabrics

| **Fabric Sample ID: D2** | | | | |
|---|---|---|---|---|
| **Fibers** | **Tex (g/10000m)** | **Composition by Weight (%)** | **Nominal Weight ($g/m^2$)** | **Weaving Angle** |
| Glass | 300 | 56.0 | 755 | +45°/-45° |
| PP | 200 | 43.2 | | |
| PES (E5) | | 0.8 | | |
| **Fabric Sample ID: D3** | | | | |
| **Fibers** | **Tex (g/10000m)** | **Composition by Weight (%)** | **Nominal Weight ($g/m^2$)** | **Weaving Angle** |
| Glass | 300 | 58.8 | 834 | +45°/-45° |
| PP | 200 | 40.4 | | |
| PES (E5) | | 0.7 | | |
| **Fabric Sample ID: D4** | | | | |
| **Fibers** | **Tex (g/10000m)** | **Composition by Weight (%)** | **Nominal Weight ($g/m^2$)** | **Weaving Angle** |
| Glass | 300 | 73.6 | 667 | +45°/-45° |
| PP | 200 | 25.8 | | |
| PES (E5) | | 0.9 | | |
| **Fabric Sample ID: D5** | | | | |
| **Fibers** | **Tex (g/10000m)** | **Composition by Weight (%)** | **Nominal Weight ($g/m^2$)** | **Weaving Angle** |
| Glass | 300 | 56.0 | 755 | +45°/-45° |
| PP | 200 | 43.2 | | |
| PES (E5) | | 0.8 | | |
| **Fabric Sample ID: D8** | | | | |
| **Fibers** | **Tex (g/10000m)** | **Composition by Weight (%)** | **Nominal Weight ($g/m^2$)** | **Weaving Angle** |
| Glass | 300 | 59.2 | 767 | +45°/-45° |
| PP | 200 | 40.0 | | |
| PES (E5) | | 0.8 | | |

Table 3.2. Differences of the hybrid non-crimp fabrics

| Fabric Code | Glass tex/PP tex | Hybrid Yarn Preparation Technique | Glass Fiber Sizing | Glass Fiber Weight Percent |
|---|---|---|---|---|
| D2 | 300/200 | Air Jet | PES resin compatible | 56 |
| D3 | 300/200 | Single Twist | PES resin compatible | 59 |
| D4 | 300/200 | Single Twist | PES resin compatible | 74 |
| D5 | 300/200 | Double Twist | PES resin compatible | 58 |
| D8 | 300/200 | Air Jet | PP resin compatible | 60 |

## 3.2. Thermoplastic Based Composite Manufacturing

Continuous glass fiber reinforced thermoplastic based composite materials were fabricated by hot press compression moulding of hybrid glass fiber/PP non-crimp fabrics. Schematic of the thermoplastic composite manufacturing is shown in Figure 3.2 and used hot press and finished composite material is shown in Figure 3.3.

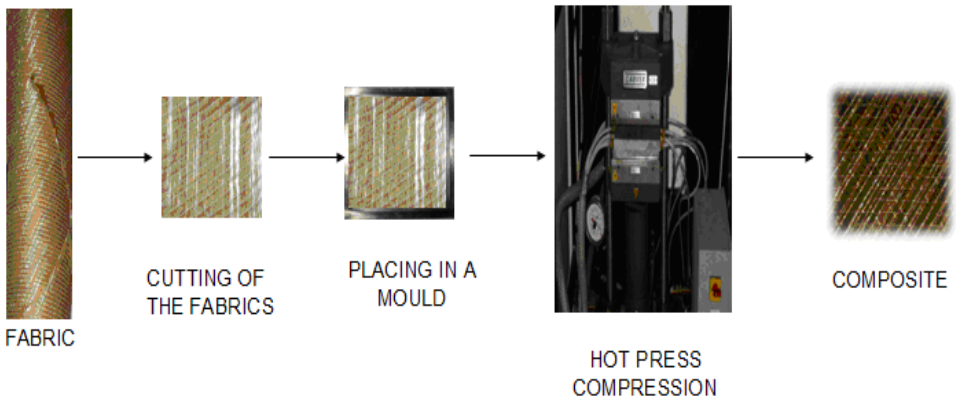



Figure 3.2. Composite manufacturing from Glass fiber/PP non-crimp hybrid fabrics

Figure 3.3. Hydraulic hot press, mould and finished composite part

The first step for composite fabrication was cutting fabrics in to the dimensions of the mould. In the second step, mould was transferred to hot press. Pressure was applied to fabrics while press was heating up to lamination temperature. In case of heating without pressure of the fabrics, it was observed that glass fiber orientations were effected negatively, because of the melting and shrinkage of the polypropylene matrix. Based on the melting temperature of PP fibers, process temperature was varied between 180 and 220 $^{0}$C. The lamination pressure was varied between 0.5 and 3MPa. Heating system was turned off after 30 min and system was cooled to room temperature under pressure. Finally glass fiber/PP composites were obtained after removal from the mold.

## 3.3. Characterization of Thermoplastic Composites

### 3.3.1. Microstructural Property Characterization

#### 3.3.1.1. Matrix Burn-Out Test

The burn-out test method was used to determine the fiber volume fraction of the glass fiber / PP composite panels. In this method, a small sample of composite is burned

off in a high temperature oven at 700 $^0$C. The remaining fiber is weighed. The volume of the fiber is calculated by dividing the mass of the fiber by the density of the fiber material. Calculation procedure is shown below.

$$V_f = \frac{v_f}{v_f + v_m} \times 100 = \frac{\frac{m_f}{\rho_f}}{\left(\frac{m_f}{\rho_f} + \frac{m_m}{\rho_m}\right)} \times 100 \qquad (3.1)$$

In this equation, $v_f$ and $v_m$ indicate the volumes of the fiber and matrix material, $m_f$ and $m_m$ indicate weights of fiber and matrix material and finally, $\rho_f$ and $\rho_m$ indicate densities of fiber and matrix material.

### 3.3.1.2. Optical and Electron Microscopy

Scanning electron and optical microscopy on the peel and fracture surfaces of tested specimens were performed in order to investigate the effect of sizing on the interface bonding. Also effect of process temperature on the consolidation quality and void content were investigated. For this purpose Philips™ XL 30SFEG SEM and Nikon™ optical microscopes were used.

## 3.3.2. Thermal Property Characterization

### 3.3.2.1. Differential Scanning Calorimetry (DSC)

Differential scanning calorimetry (DSC) is a thermal technique in which differences in heat flow into a substance and a reference are measured as a function of sample temperature while the two are subjected to a controlled temperature program (Skoog, et al. 1998). DSC (TA Instrument Q10 model) was used in order to investigate the

melting temperature of polypropylene. For this test, the samples of 8-10 mg of polypropylene samples were placed into the aluminum crucible, respectively. Indium was used to calibrate the thermal response due to heat flow as well as the temperature prior to analysis. The dynamic measurements were made at a constant heat rate of 2°C/minute from 25 to 200°C to determine the melting point of polypropylene matrix.

### 3.3.3. Mechanical Property Characterization

#### 3.3.3.1. Tensile Test

Tensile test technique, ASTM D 3039M-93 were used to determine tensile strength and modulus of the PP/Glass fiber composites. Test specimens were prepared using a diamond saw according to ASTM standard. As the composite exhibits similar behavior for $0^0$ and $90^0$ directions only one direction was tested. At least 5 samples were tested for each panel. The specimens were tested at room temperature using mechanical test machine (Shimadzu$^{TM}$ universal) at a cross head speed of 2 mm/min (Figure 3.4)

Tensile strength and strain values were calculated using following equations;

$$\sigma = \frac{F}{A} \tag{3.2}$$

$$\varepsilon = \frac{(L - L_0)}{L_0} \tag{3.3}$$

where F is the ultimate load, and A is the cross sectional area of the specimen. $L_0$ is the original distance between gage marks, and $L$ is the distance between gage marks at any time. Elastic modulus was obtained from the initial slope of stress-strain curves based on the equation below;

$$E = \frac{\sigma}{\varepsilon} \tag{3.4}$$

Figure 3.4. Tensile test specimen during test.

### 3.3.3.2. Flexural Test

The flexural test technique was used to determine the flexural strength and modulus of the composites. The flexural test technique and sample preparation was in accordance with ASTM D 790M-86. Specimens were tested in 3-point bending configuration with a span to thickness ratio of 16. Specimen length and width, span distance and test speed were adjusted according to specimen thickness. Figure 3.5 is the photo showing the flexural test specimen under load. At least five specimens from composites were tested using the universal test machine. Force vs. deflection at the center of the beam was recorded. Test specimen under load is given in Figure 3.5.

Figure 3.5. Flexural test specimen during test

The flexural strength *(S)* in the units of MPa was calculated using the following equation;

$$S = \frac{3PL}{2bd^2} \tag{3.5}$$

where *P* is the applied load at the deflection point, *L* is the span length, *d* and *b* are the thickness and the width of the specimen, respectively. The flexural modulus values $(E_b)$ were calculated using the following equation;

$$E_b = \frac{L^3 m}{4bd^3} \tag{3.6}$$

where *m* is the slope of the tangent to the initial straight line portion of the load-deflection curve.

### 3.3.3.3. Interlaminar Peel Test

The interlaminar peel test was used to determine the peel strength of the laminas. With the aid of this test method, effect of glass fiber sizing on the adhesion of fiber matrix interface was observed. The test technique and sample preparation was in accordance with ASTM D 5528-01. This standard is actually describes the determination of the opening Mode I interlaminar fracture toughness $G_{IC}$. However, adhesion problems of polypropylene with metal surfaces obliged us to screw metal blocks in to thermoplastic composites. Additionally difficulties in following the crack propagation in tough matrix composites did not let us to investigate the Mode I interlaminar fracture toughness $G_{IC}$. Therefore, only the peel strength of the laminas was determined.

Test laminates were fabricated from even plies and non-adhesive insert was inserted at the mid plane of the laminate during lay-up. Length of the insert was 63 mm and thickness of the insert was less than 13 μm. Test specimens were at least 125 mm long and nominally 20 to 25 mm wide. And the thickness of the specimens was between 3-5 mm. Also aluminum loading blocks were jointed to composite materials. The photo of test specimen is shown in Figure 3.6.

Figure 3.6. Interlaminar peel test specimen under load

### 3.3.3.4. Compression Test

Compression test method according to ASTM D 695-M was used to measure the ply-lay up and in-plane compressive strength, modulus and strain to failure values of the composite panels fabricated from glass fiber/PP non-crimp fabrics. For this purpose, compression test specimens were sectioned from larger panels and tests along these directions were performed using mechanical test machine (Shimadzu$^{TM}$ universal) at crosshead speed of 1.3 mm/min (Figure 3.7). At least 5 specimens for each set were tested and force versus stroke values was recorded. The compressive stress values were obtained by dividing load values with cross-sectional area of the specimens. The strain was estimated by dividing the adjusted stroke values with initial specimen thickness. The modulus values were estimated from the slope of stress-strain graph.

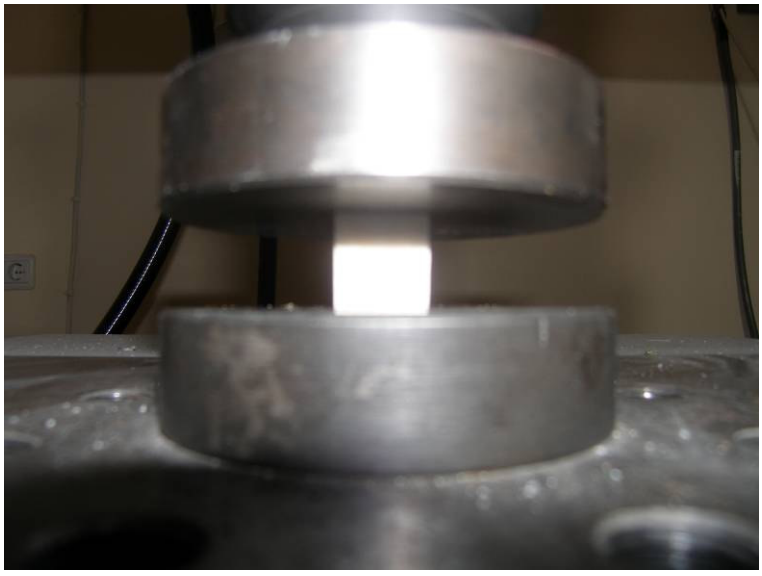


Figure 3.7. Compression test specimen during test

### 3.3.3.5. In-plane Shear Test

In-plane shear test method according to ASTM D 3518 M was used to measure the maximum shear and offset shear strength of the glass fiber/PP composite materials. Sectioned test specimens from larger panels were loaded in tension mode using mechanical

test machine (Shimadzu$^{TM}$) along the ± $45^0$ direction at a crosshead speed of 5 mm/min. At least 5 specimens for each set were tested and force versus stroke values was recorded. Shear stress values were estimated by dividing the estimates force values with twice of the specimen's cross-sectional area.

Figure 3.8. In-plane test specimen during test

### 3.3.4. Charpy Impact Test

The Charpy impact test, also known as the Charpy v-notch test, is a standardized high strain-rate test which determines the amount of energy absorbed by a material during fracture. This absorbed energy is a measure of a given material's toughness. It is widely applied in industry, since it is easy to prepare and conduct and results can be obtained quickly and cheaply. But a major disadvantage is that all results are only comparative.

Charpy impact test machine (Ceast Resil Impactor) was used to determine toughness of glass fiber/PP composite materials. The apparatus consists of a pendulum axe swinging at a notched sample of material.

The notch in the sample affects the results of the impact test, thus it is necessary for the notch to be of a regular dimensions and geometry. The size of the sample can also affect results, since the dimensions determine whether or not the material is in plane strain.

This difference can greatly affect conclusions made. Test machine and notch opening apparatus are shown in Figure 3.9.

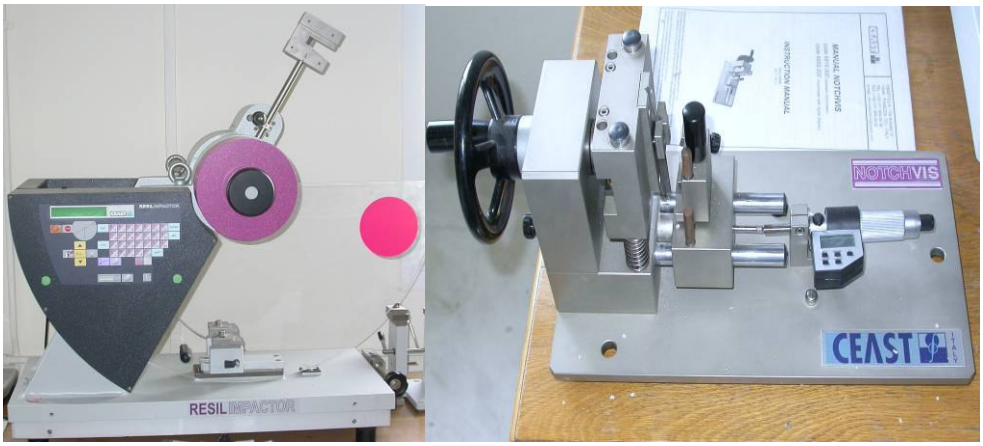



Figure 3.9. Charpy impact machine and notch opening apparatus

This test was performed in accordance with ISO 179 standard. 80 mm long, approximately 4 mm thick and 10 mm wide specimens were tested. Notch depth was 2 mm.

# CHAPTER 4

# RESULTS AND DISCUSSIONS

## 4.1. Microstructural Characterization

Non-crimp ± $45^0$ glass fiber reinforced polypropylene matrix composites were prepared by hot pressing. The fiber orientations, matrix rich regions and void fractions were characterized by optical microscopy. Fiber orientation after compression moulding is an important parameter in thermoplastic composite manufacturing from hybrid fabrics. Fiber orientation directly affects mechanical properties of the composite material. Optical microscopy lightened above and below was used before and after lamination in order to investigate the distortions in the fiber orientation during lamination.

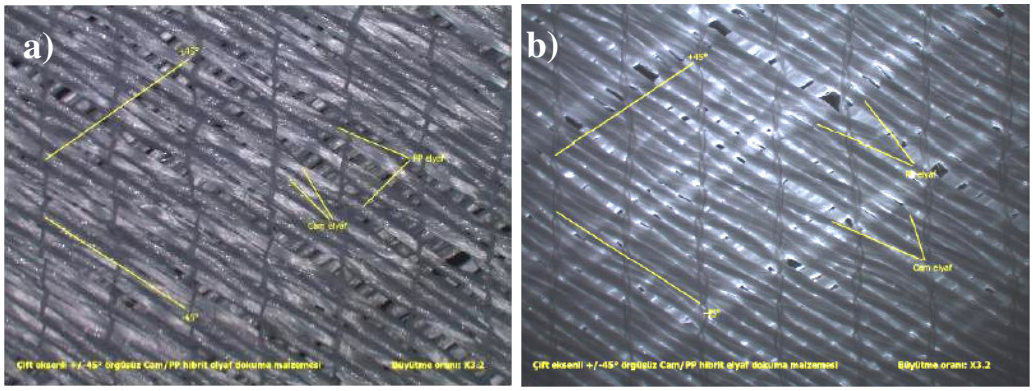



Figure 4.1. Optical microscope images of fabrics produced by single twist method (a) lightening from above (b) lightening from below. Magnification 3.2X

Based on these images, glass fiber bundles appear to be in order before lamination process. In Figure 4.2, optical microscope images of the composite produced from these fabrics by compression moulding are shown. As seen from these images, the orientation of the fiber bundles is distributed during the composite manufacturing by hot pressing. So, composites have less orientation as compared to those hybrid fabrics before consolidation. Thermal stresses during lamination cause this disordering.

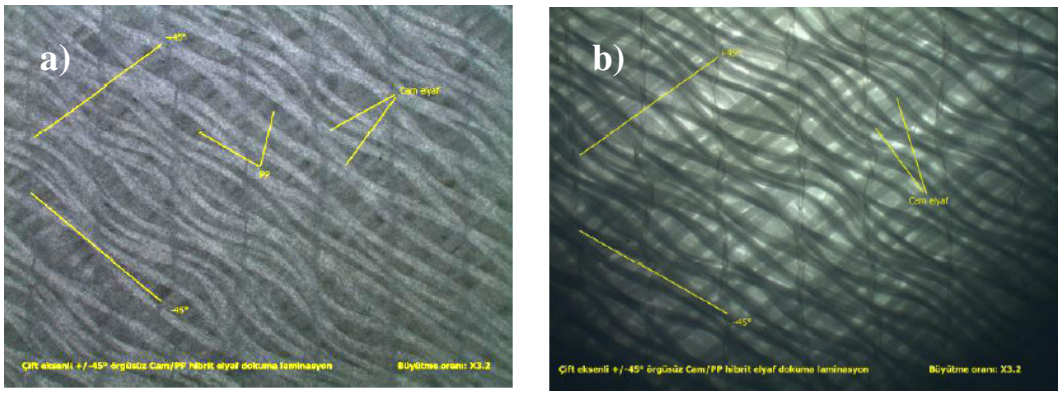



Figure 4.2. Optical microscope images of composites produced from single twisted fabrics (a) lightening from above (b) lightening from below. Magnification 3.2X

Laminates of three different fabrics were examined under microscope in order to compare the glass fiber orientations after laminations. Optical microscope images of the laminates of the fabrics prepared with single twist, double twist and air jet hybrid yarn preparation techniques are given in Figure 4.3.

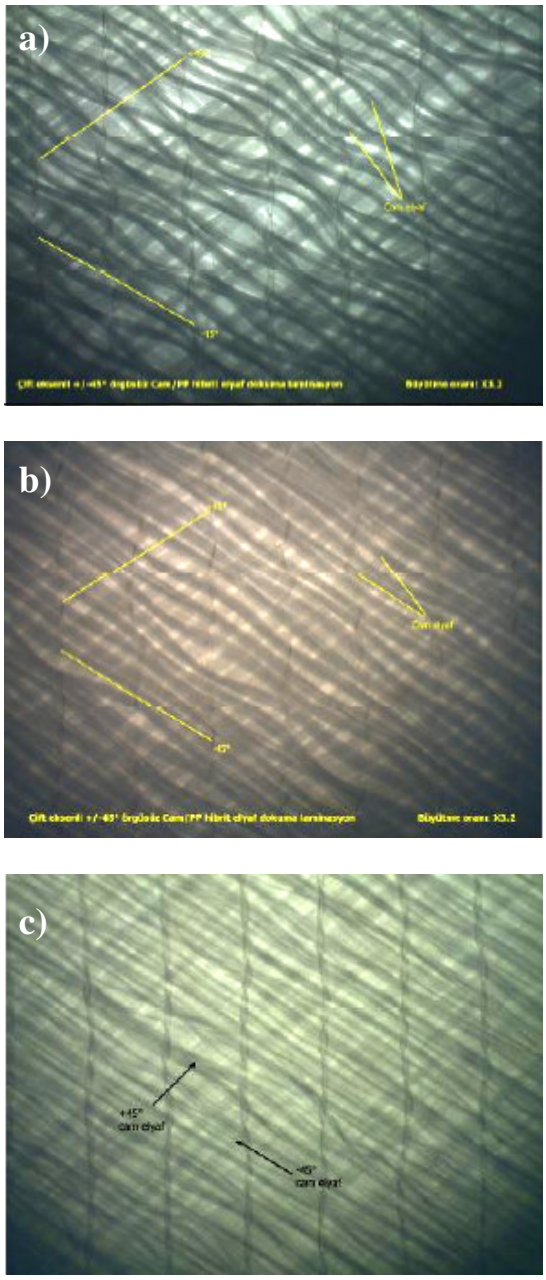



Figure 4.3. Optical microscope images of composites produced from different fabrics (a) Single twist (b) Double twist (c) Air-Jet Magnification 3.2X

According to Figure 4.3, the best glass fiber orientation was observed from the composite laminates that are produced with the fabrics of air-jet hybrid yarn preparation technique.

To examine the effect of process (hot-pressing) temperature on the void formation of the composites, SEM images were taken from cross-sections of three composite materials produced from fabric D2 at temperatures of 195, 200 and 205 $^{o}$C. For this purpose, composite samples polished using metallographic techniques.

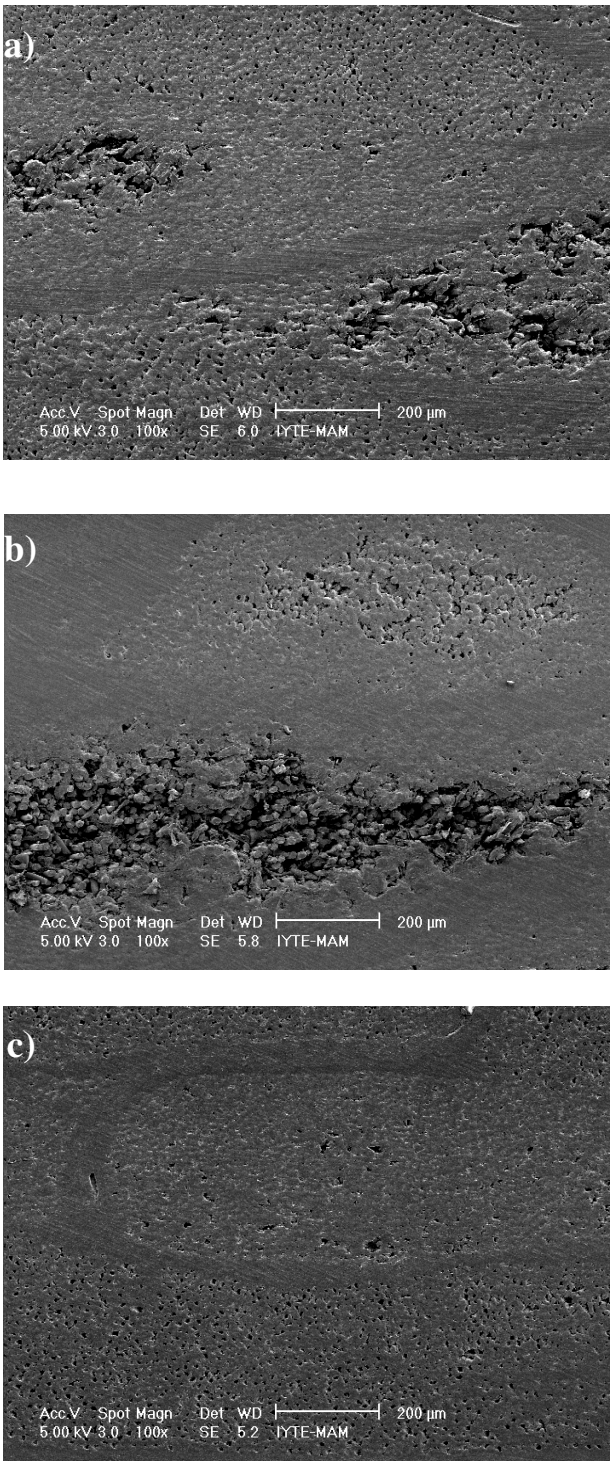



Figure 4.4. Effect of process (hot-pressing) temperature on the void formation of the Glass fiber/PP composites manufactured from fabric D2 at (a)195$^{o}$C (b)200 $^{o}$C (c) 205$^{o}$C

As seen in Figure 4.4, it was observed that presence of voids is less and penetration of polypropylene matrix in to the glass fiber bundles is better in the microstructure of the composites prepared at 205 $^{o}$C, as compared those prepared at lower temperatures.

## 4.2. Thermal Property Characterization

### 4.2.1. Differential Scanning Calorimetry (DSC) Analysis

DSC analyzes were performed to investigate the melting point of PP matrix used in glass fiber/PP hybrid non-crimp fabrics. Process (hot pressing) temperature was adjusted according to the result of this analysis. Temperature versus heat flow plot of PP matrix is shown in Figure 4.5.

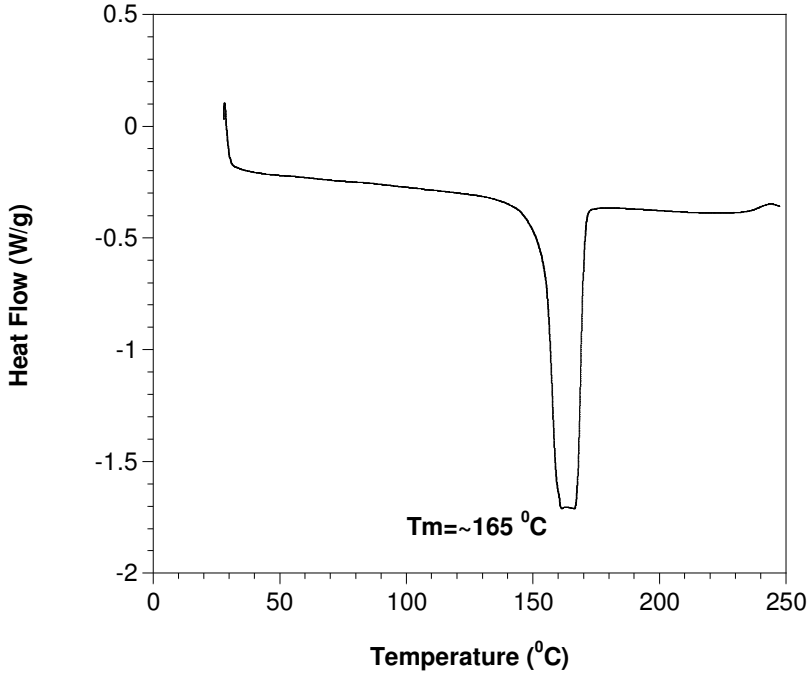



Figure 4.5. Heat flow vs. temperature plot of PP matrix used in this study

As seen in Figure 4.5, melting point of PP fiber used in glass fiber/PP hybrid fabrics is 165 $^0$C.

## 4.3. Mechanical Property Characterization

### 4.3.1. Tensile Properties

Tensile tests were performed in order to investigate the tensile properties of non-crimp PP/Glass thermoplastic composites. Figure 4.6 shows the plot of tensile stress-strain behavior of composites manufactured from different hybrid fabrics at 200 $^{o}$C temperature and 1.5 MPa compression pressure.

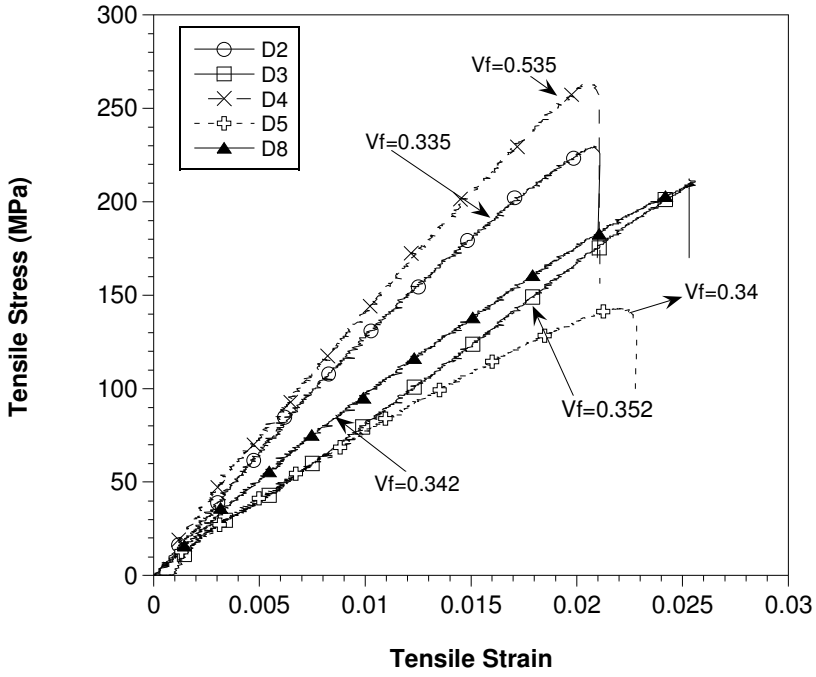



Figure 4.6. Tensile behavior of composites manufactured from different types of hybrid fabrics.

Tensile strength, tensile strain and tensile modulus values were calculated from force-stroke data and cross-sectional area of the test specimen and these values are given in Table 4.1. The effects of hybrid yarn preparation technique and used glass fiber sizing on the composite properties are revealed in Table 4.1. Additionally, fiber volume fractions of the composites obtained by matrix burn-out test are given on the same table.

Table 4.1. Tensile Properties of glass fiber/PP thermoplastic composites

| Fabric Code | Tensile Strength (MPa) | Tensile Modulus (Gpa) | Tensile Strain | Hybrid Yarn Preparation Technique | Fiber Volume Fraction | Glass Fiber Sizing |
|---|---|---|---|---|---|---|
| **D2** | 227.75 ±8.1 | 11.2 ± 0.41 | 0.022 ±0.001 | Air jet | 0.335 | PES resin compatible |
| **D3** | 211.9 ±16.1 | 10.7 ± 0.6 | 0.02 ±0.001 | Single Twist | 0.352 | PES resin compatible |
| **D4** | 262.7 ±15.3 | 13.83 ± 0.94 | 0.019 ±0.0018 | Single Twist | 0.535 | PES resin compatible |
| **D5** | 143.75 ±8.46 | 8.17 ± 0.59 | 0.0215 ±0.0026 | Double Twist | 0.34 | PES resin compatible |
| **D8** | 208.6 ±1.98 | 9.97 ± 0.53 | 0.023 ±0.0035 | Air Jet | 0.342 | PP resin compatible |

As seen in Table 4.1, maximum tensile properties were observed from the composite, prepared with fabric D4, as expected. This was due to the highest fiber volume fraction of the D4 coded composite. This composite have average tensile strength of 262.7 MPa (±15.3 MPa) and elastic modulus of 13.83 GPa (±0.94 GPa). When we compare composite panels with the similar fiber volume fractions, the highest tensile properties (with 227.75 MPa (±8.1 MPa) tensile strength and 11.2 GPa (± 0.41 GPa) elastic modulus) were obtained from D2 coded composite which was produced from fabrics prepared by air jet hybrid yarn preparation technique. It was observed that the effect of glass fiber sizing on the tensile properties of the glass fiber/PP composite materials was insignificant. Tensile test results showed that hybrid yarn preparation technique plays a dominant role on the tensile properties of the composite material. Composites made of fabrics produced by air jet hybrid yarn preparation technique give better results than those produced by direct twist covering hybrid yarn preparation technique. The lowest results (143.75 MPa (±8.46) tensile strength and 8.17 GPa (± 0.59 GPa) elastic modulus) were obtained from the composites made of fabrics produced by double twist covering hybrid yarn preparation technique. Santulli (2002) studied tensile properties of commingled E-glass/PP laminates with 60% weight glass fiber content and reported tensile strength and modulus 225 MPa and 13 GPa, respectively. Zhao, et al. (2009) studied tensile properties of commingled E-glass/PP laminates with 82.5% volume glass fiber content and reported tensile strength and modulus 220 MPa and 8.5 GPa, respectively.

SEM images of glass fiber/PP composites fabricated from fabrics D2 and D8 were taken from fracture surfaces and shown in Figures 4.7 and 4.8.

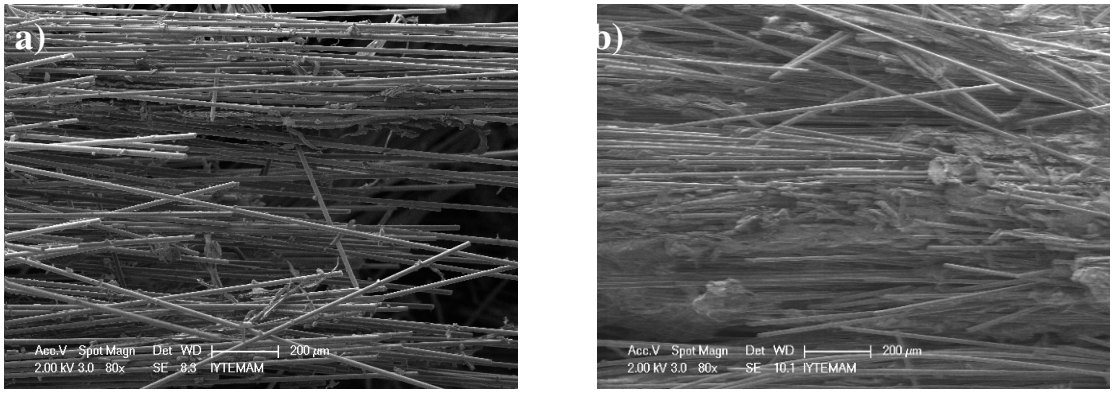



Figure 4.7. SEM images of the fracture surfaces of the composites coded with (a)D2 (b)D8 Magnification 80x

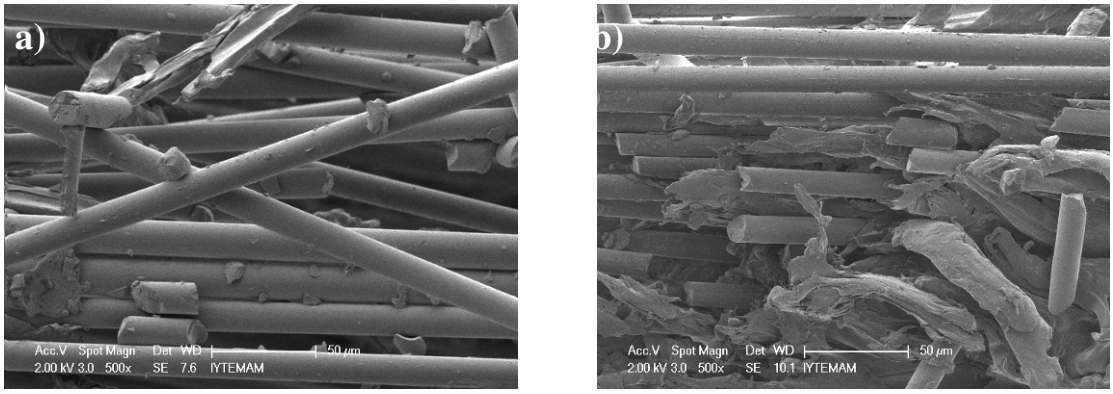



Figure 4.8. SEM images of the fracture surfaces of the composites coded with (a)D2 (b)D8 Magnification 500x

As seen in SEM images, more complex failure modes and deformation observed for composite with fabric D8. Due to better adhesion at the fiber/PP interface plastic deformation of the PP matrix around fibers was observed. On the other hand, smoother fracture surfaces were observed for composite with fabric D2. It is due to weak adhesion at the fiber/PP interface.

In order to investigate the effect of process (hot pressing) temperature on the tensile properties, composites produced from D2 coded fabric under 1.5 MPa pressure and process temperatures between 180-240 $^{o}$C were tested. Effect of process temperature on the elastic modulus values is shown in Figure 4.9.

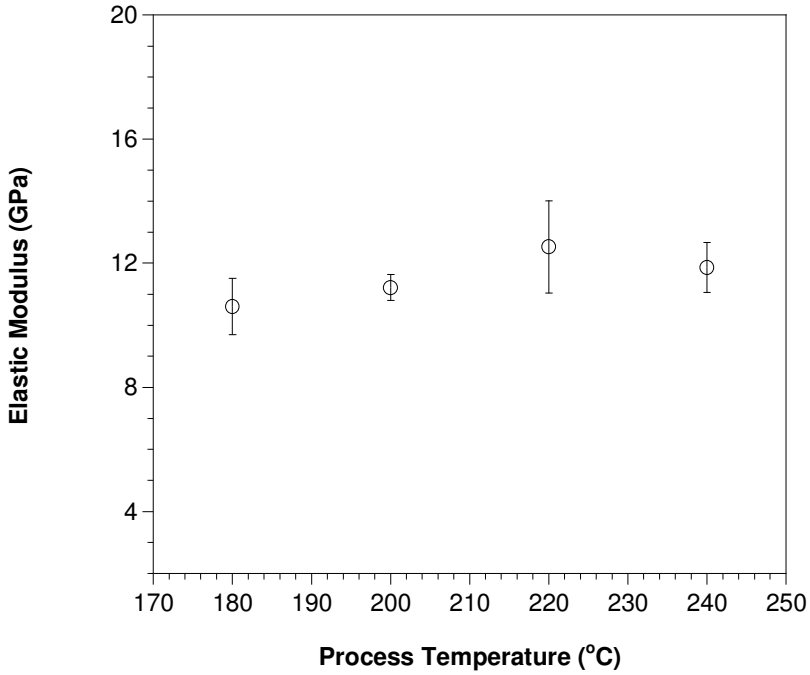


Figure 4.9. Effect of process (hot-pressing) temperature on the elastic modulus

Elastic modulus values differ in the range of 10.6-12.5 GPa. Elastic modulus values of glass fiber/PP composites slightly increases and reaches the maximum at 220 $^{o}$C. It was observed that process temperature does not have significant effect on the elastic modulus of the composite material. In Figure 4.10, effect of process pressure on the tensile strength of glass fiber/PP composite material is shown.

Tensile strength values changes in the range of 208-231 MPa.Tensile strength slightly increases with increasing process temperature up to 220 $^{o}$C. The tensile strength values drop slightly at 240 $^{o}$C. Both elastic modulus and tensile strength values slightly decreases at 240 $^{0}$C, it may be due to the increasing viscosity of the thermoplastic matrix and this increased viscosity may cause extensive flashing of the thermoplastic resin from composite parts with pressure.

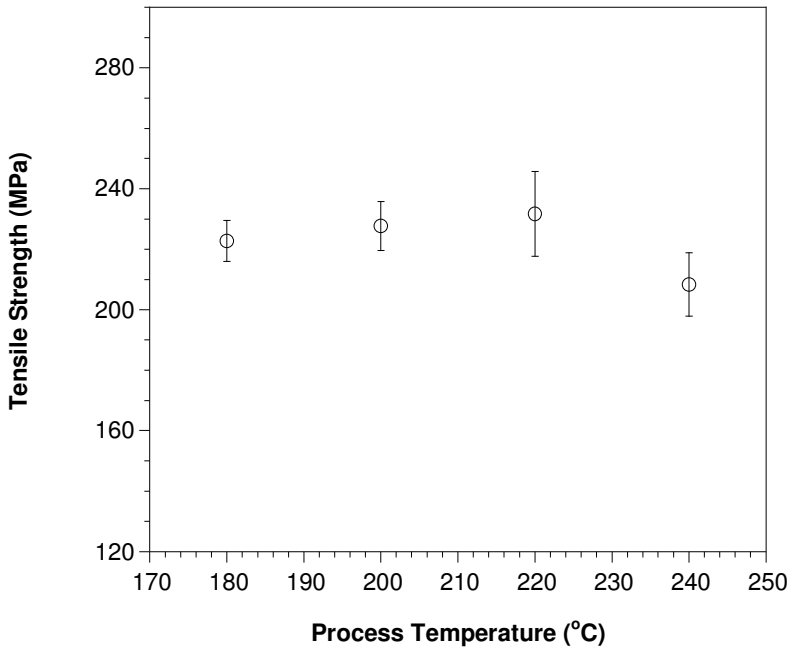



Figure 4.10. Effect of process (hot-pressing) temperature on the tensile strength

In Figure 4.11, strain at failure vs. temperature is given. It is shown that the strain at fracture slightly decreases with increasing temperature however there is no significant change observed. Strain values at failure changes in the range of 0.02-0.0242.

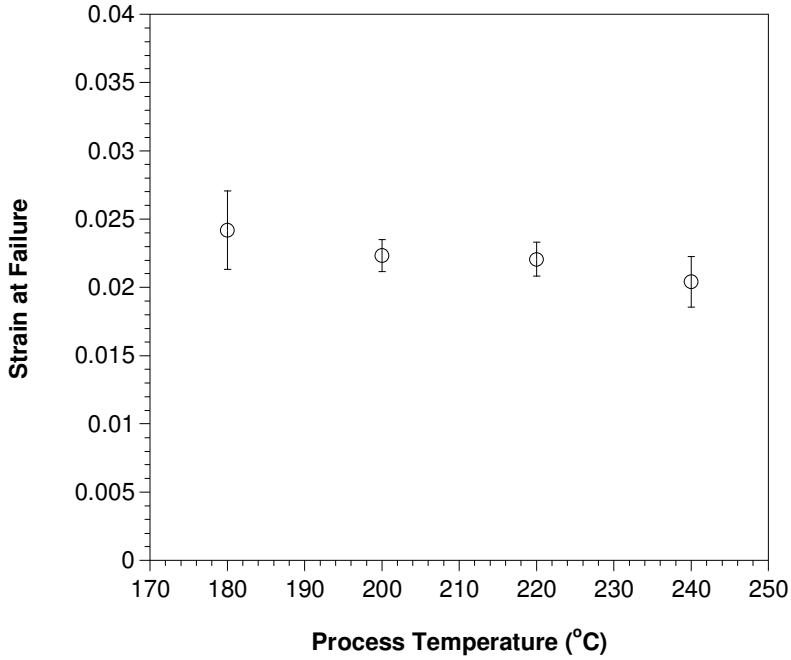



Figure 4.11. Effect of process (hot-pressing) temperature on the strain at failure

## 4.3.2. Flexural Properties

Figure 4.12 shows the plot of flexural stress-strain behavior of composites manufactured from various type of fabrics at 200 $^{o}$C temperature and 1.5 MPa compression pressure.

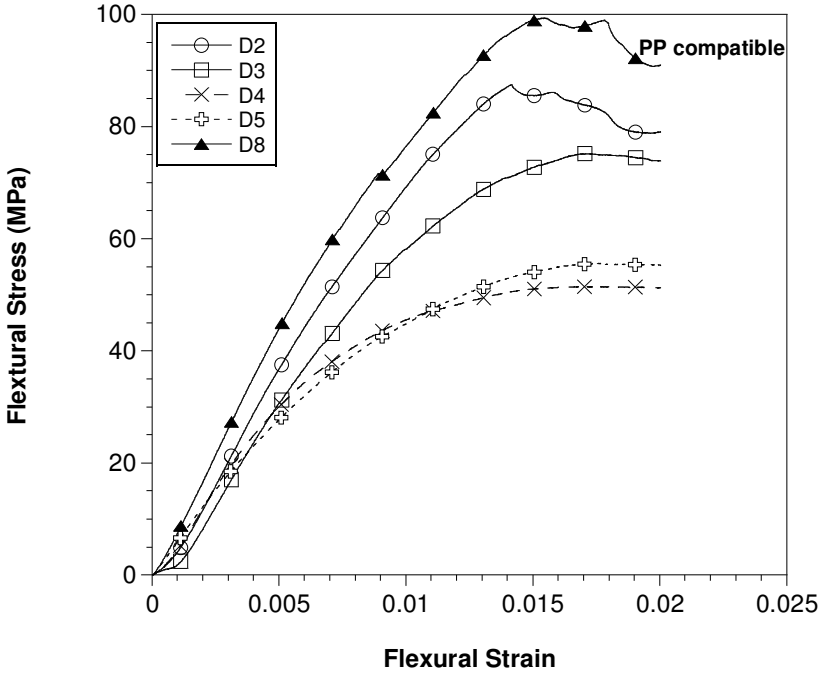



Figure 4.12. Flexural stress-strain behavior of composites manufactured from different types of fabrics.

Flexural strength, flexural strain and flexural modulus values were calculated from force-stroke data and these values are given in Table 4.2. In the same table, fiber volume fractions obtained by matrix burn-out test, type of the fabric obtained with various hybrid yarn preparation technique and the type of the sizing applied on the glass fibers are given.

Table 4.2. Flexural Properties of glass fiber/PP composites

| Fabric Code | Flexural Strength (MPa) | Flexural Modulus (Gpa) | Flexural Strain | Hybrid Yarn Preparation Technique | Fiber Volume Fraction | Glass Fiber Sizing |
|---|---|---|---|---|---|---|
| **D2** | 85.2 ±2.4 | 7.8 ± 0.4 | 0.016 ±0.0028 | Air jet | 0.335 | PES resin compatible |
| **D3** | 74.4 ±5.2 | 6.6 ± 0.4 | 0.019 ±0.0017 | Single Twist | 0.352 | PES resin compatible |
| **D4** | 48.4 ±6.5 | 5.4 ± 0.9 | 0.023 ±0.0026 | Single Twist | 0.535 | PES resin compatible |
| **D5** | 54.8 ±2.9 | 5.6 ± 0.4 | 0.017 ±0.0034 | Double Twist | 0.340 | PES resin compatible |
| **D8** | 99.1 ±4.2 | 9.6 ± 0.3 | 0.015 ±0.0013 | Air Jet | 0.342 | PP resin compatible |

Based on the results shown in Figure 4.12 and Table 4.2, it was observed that type of the hybrid yarn preparation technique and glass fiber sizing applied on the glass fibers has some important role on the flexural properties of glass fiber/PP non-crimp composites. The highest flexural properties (99.1 Mpa (± 4.2 MPa) flexural strength and 9.55 GPa (± 0.293 GPa) flexural modulus) was obtained from the composites manufactured from fabric D8. In these fabrics, PP resin compatible glass fiber sizing was used. It was observed that PP resin compatible sizing have positive effects on flexural properties of the composite as compared to those with PES resin compatible sizing. This is due to better adhesion at the interface of glass fibers and PP matrix. The results revealed that interfacial strength has more critical effect on the flexural properties as compared to tensile properties of the composites. In addition, for fabric D8, hybrid yarns were manufactured by air jet hybrid yarn preparation technique. The second highest results were (85.2 MPa (±2.4 MPa) flexural strength and 7.83 GPa (± 0.41 GPa)) flexural modulus) obtained from the composite which was manufactured from fabric D2. Common trait of the fabrics D2 and D8 is the hybrid yarn manufacturing technique. Fabric D2 was also produced with air jet hybrid yarn preparation technique. Therefore it can be concluded that, air jet hybrid yarn preparation technique contribute to the better mechanical properties due to relatively better commingling of the glass and PP fibers in the yarn.

When we compare composites which were produced from fabrics via direct twist covering hybrid yarn preparation technique, it was observed that (with similar fiber volume fractions) composite manufactured from single twisted hybrid yarns (D3) have better flexural properties as compared to those manufactured from double twisted hybrid yarns (D5), as seen in Table 4.2. Santulli (2002) studied flexural properties of commingled E-glass/PP laminates with 60% weight glass fiber content and reported flexural strength and modulus 230 MPa and 11.5 GPa, respectively. When we compared our flexural strength values with these values, it may be caused from different fabric properties.

In order to investigate the effect of process (hot-pressing) temperature on the flexural properties of glass fiber/PP composite materials, composite panels were produced from D2 coded fabric under 1.5 MPa process pressure and temperatures of 180, 190, 200, 210 and 220 $^{o}$C. Effect of process temperature on the flexural modulus of the composite material is shown in Figure 4.13.

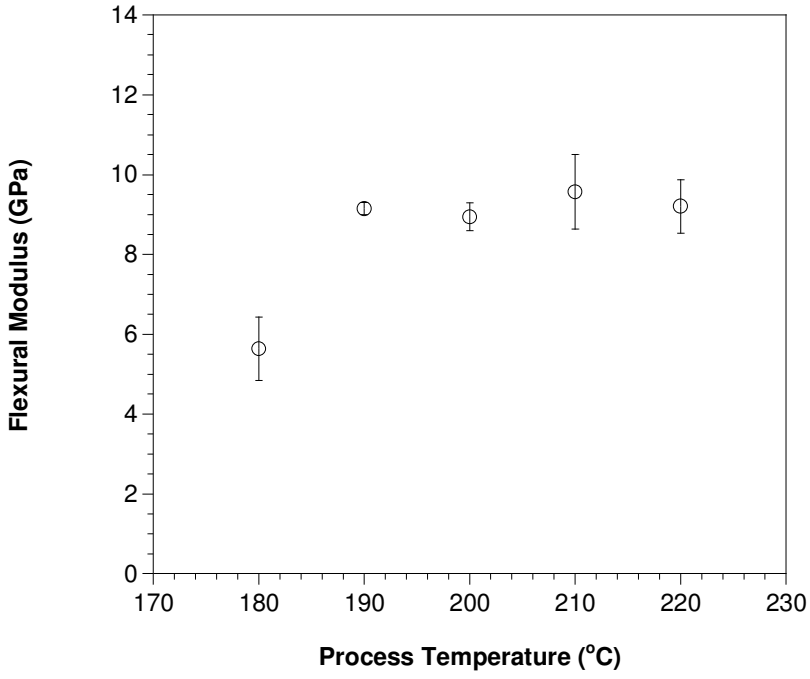



Figure 4.13. Effect of process (hot-pressing) temperature on the flexural modulus

It was observed that composites produced at 180 $^{o}$C exhibited the lowest flexural modulus value. It was concluded that the inefficient impregnation of the polypropylene matrix into the glass fibers at low temperatures results with lower mechanical properties. There is no significant change on the flexural modulus at process temperatures above 180

$^{o}$C. Flexural modulus values reach to approximately 9 GPa above 180 $^{o}$C processing temperatures.

In Figure 4.14, effect of process temperature on the flexural strength of glass fiber/PP composite materials is shown. Effect of process temperature on the flexural strength is similar to those with flexural modulus. Similar to modulus values, the lowest strength value (67 MPa) was obtained at 180$^{o}$C. Above 180$^{0}$C, the strength values increase up to 85 MPa and remain almost constant at their level at higher temperatures.

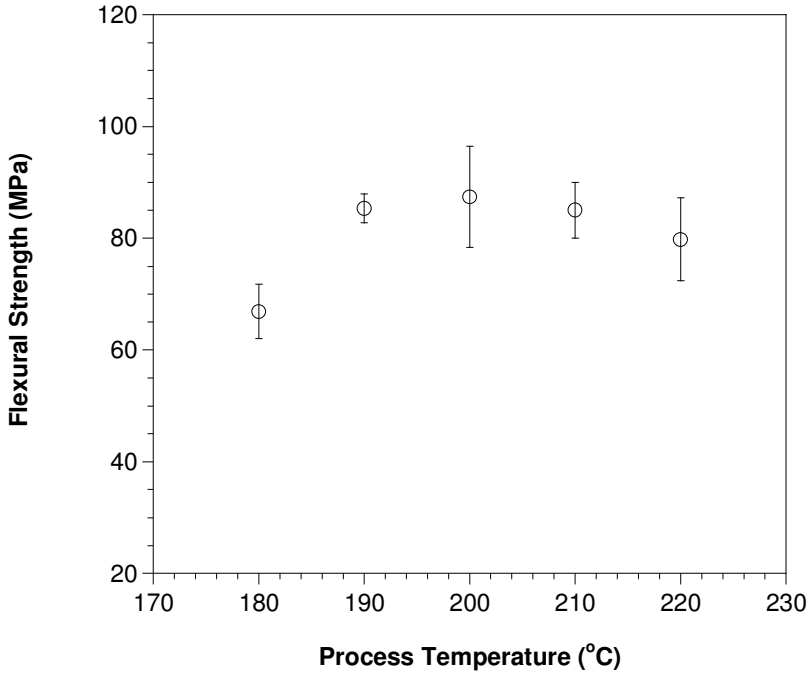



Figure 4.14. Effect of process (hot-pressing) temperature on the flexural strength

In order to investigate the effect of process pressure on the flexural properties of glass fiber/PP composite materials, panels were produced from fabric D2 at 200 $^{o}$C process temperature and under pressures of 0.5, 1, 1.5, 2, 2.5 and 3 MPa. The effect of pressure on the flexural modulus and strength of the composite material is shown in Figure 4.15 and 4.16, respectively.

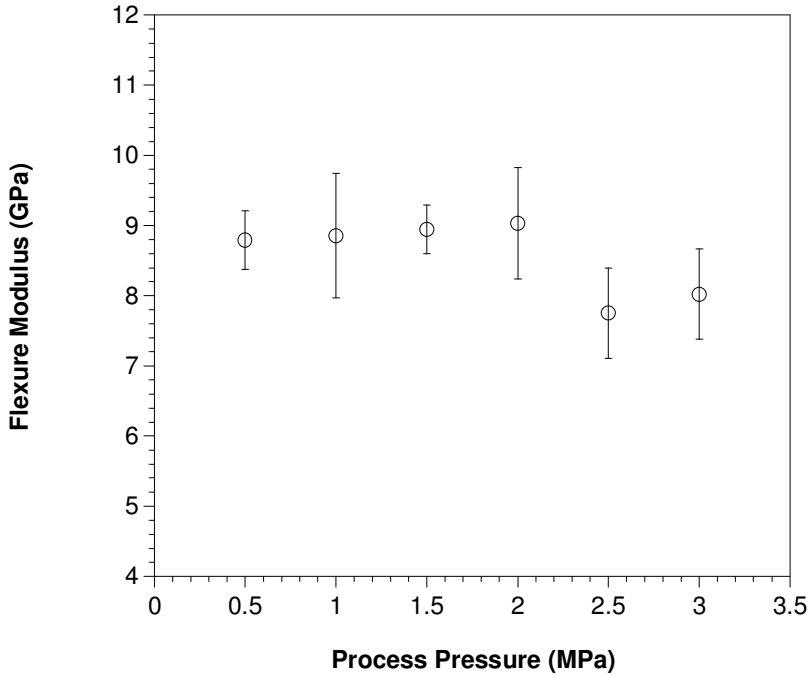



Figure 4.15. Effect of process (hot-pressing) pressure on the flexural modulus

It was observed that process pressure has no significant effect on the flexural strength and modulus of the glass fiber/PP composite material up to 2 MPa process pressure. On the other hand, flexural modulus and strength slightly decreases at pressures above 2 MPa. This was related with extensive flashing of the polypropylene matrix from the composite part as a result of high level of pressure.

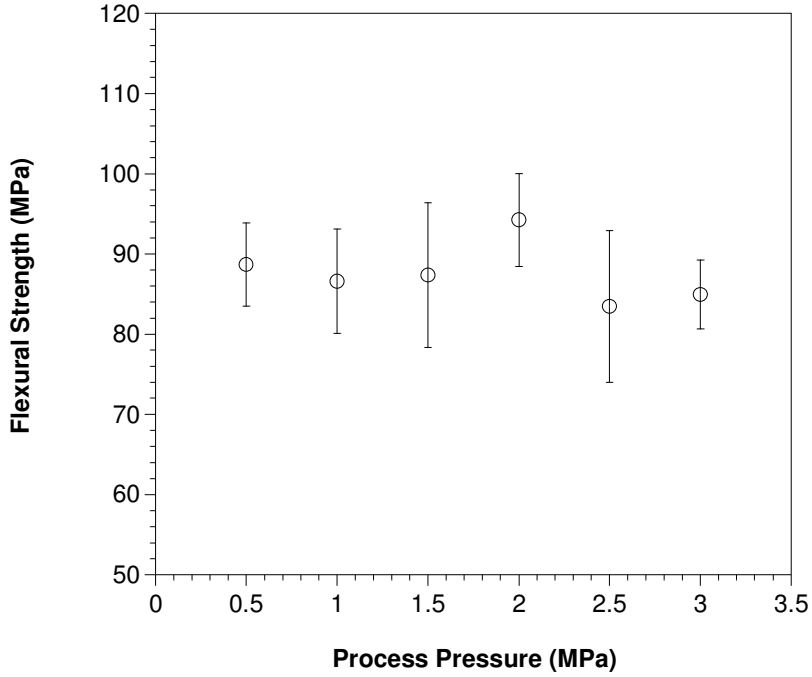



Figure 4.16. Effect of process (hot-pressing) pressure on the flexural strength

Fiber volume fractions of composite panels produced for investigating the effect of pressure and temperature on the flexural properties are given in Figure 4.17 and 4.18. In Figure 4.17 fiber volume fractions of composite panels produced under 1.5 MPa pressure and at various temperatures. Also, in Figure 4.18 fiber volume fractions of composite panels produced at 200 $^{o}$C under various pressures.

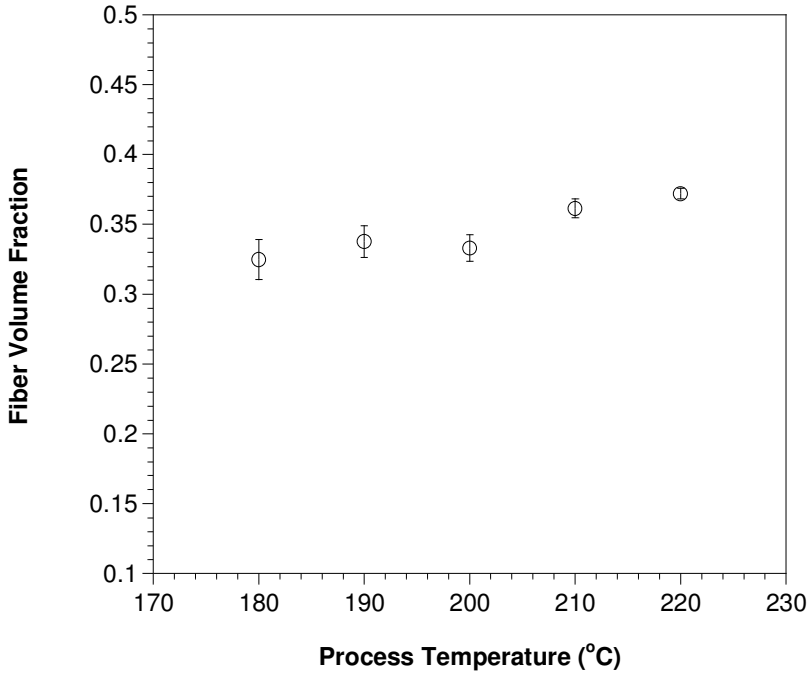



Figure 4.17. Fiber volume fractions of the composite panels fabricated at constant pressure of 1.5 MPa and various temperatures

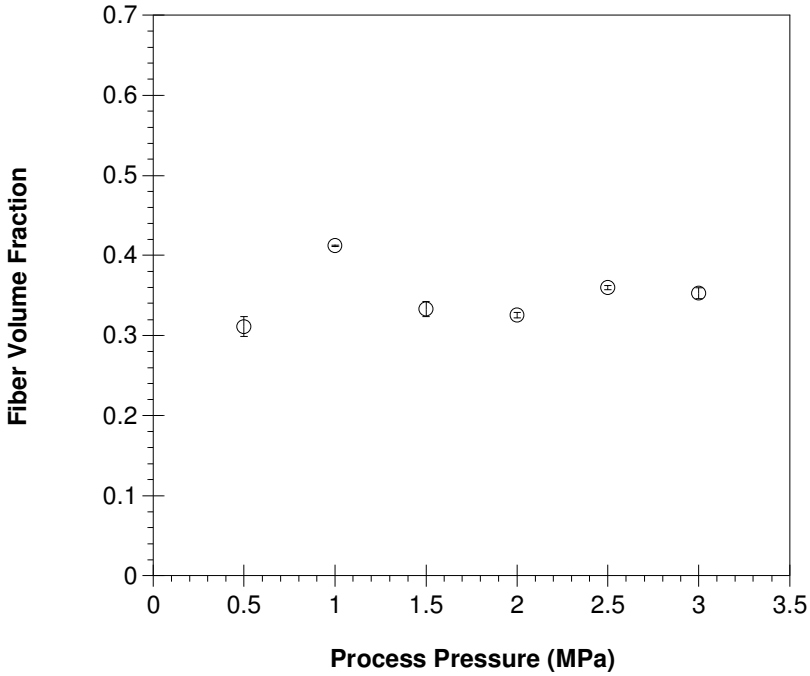



Figure 4.18. Fiber volume fractions of the composite panels fabricated at constant temperature of 200 $^{0}$C and various pressures

### 4.3.3. Interlaminar Peel Properties

Interlaminar peel test was performed in order to investigate effect of glass fiber sizing on the glass fiber-polypropylene matrix interface. All of the fabrics were fabricated under 1.5 MPa pressure and 200 $^{o}$C process temperature. Force-stroke values of the composites obtained during interlaminar peel test is shown in Figure 4.19. The strength values were also calculated based on dividing maximum force to specimen width. Fabric properties and interlaminar peel strength values are given in Table 4.3.

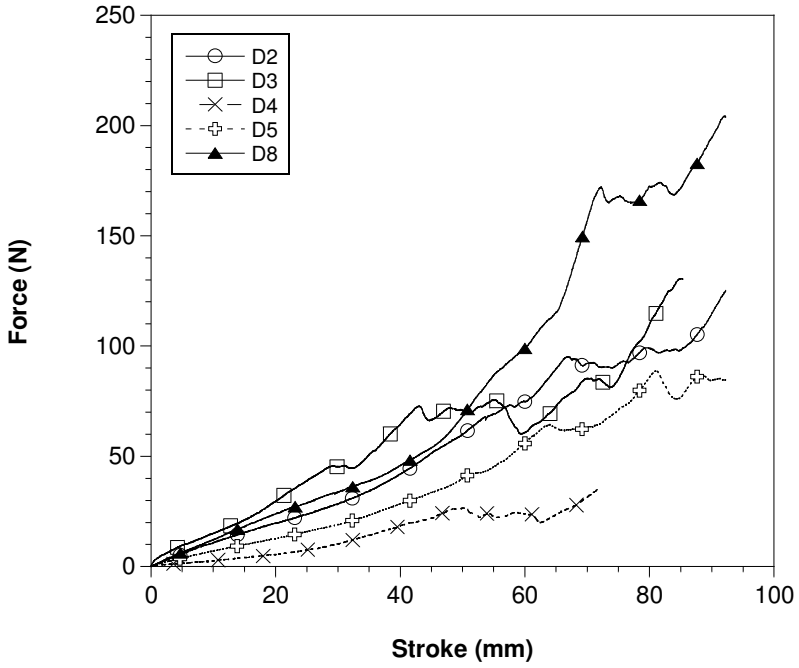



Figure 4.19. Force vs. stroke values of the composites obtained during the interlaminar peel test

It is clearly seen from Figure 4.19 that the composite fabricated from fabric with PP compatible sizing (sample named D8) exhibits the highest peel resistance to delamination than those of the composites made of other fabrics. Composites of fabric D8 have an interlaminar peel strength value of 5.87 N/mm. The nearest peel strength value 3.97 N/mm was observed with composites of fabric D2. Fabrics D2 and D8 were produced with the same hybrid yarn preparation technique, however, the only difference was the type of the glass fiber sizing. Based on these results it can be concluded that polypropylene resin compatible sizing improves adhesion at the interface of glass fiber and PP matrix.

Table 4.3. Properties of the fabrics used and interlaminar peel strength of the composites

| Fabric Code | Interlaminar Peel Strength (N/mm) | Hybrid Yarn Preparation Technique | Glass Fiber Sizing |
|---|---|---|---|
| **D2** | 3.97 | Air jet | PES resin compatible |
| **D3** | 2.58 | Single Twist | PES resin compatible |
| **D4** | 1.33 | Single Twist | PES resin compatible |
| **D5** | 2.45 | Double Twist | PES resin compatible |
| **D8** | 5.87 | Air Jet | PP resin compatible |

In order to support the results obtained by interlaminar peel test, peeled surfaces of the tested samples were examined with SEM. In Figures 4.20, 4.21 and 4.22 SEM images of the composites prepared with fabrics D2, D3 and D8 are given in different magnifications.

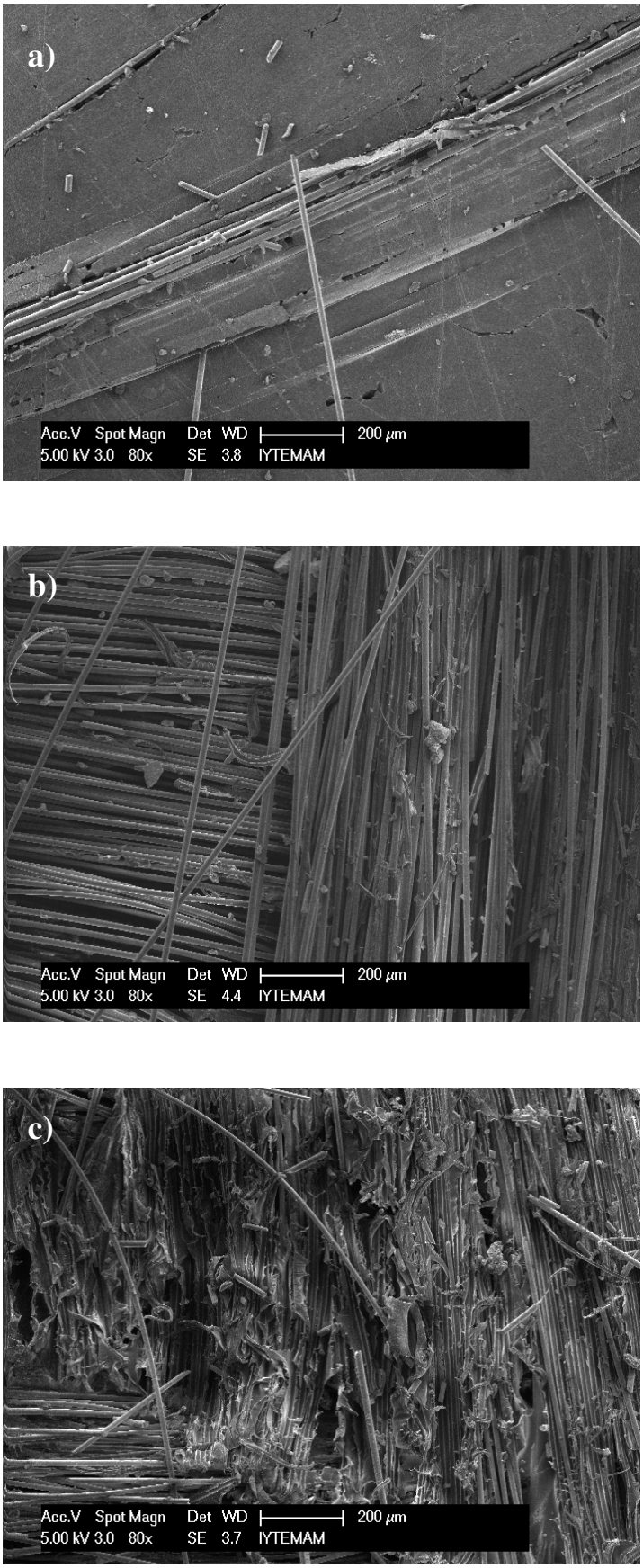



Figure 4.20. SEM images of the peeled surfaces of the composites coded with (a) D2 (b) D3 (c) D8 Magnification 80x

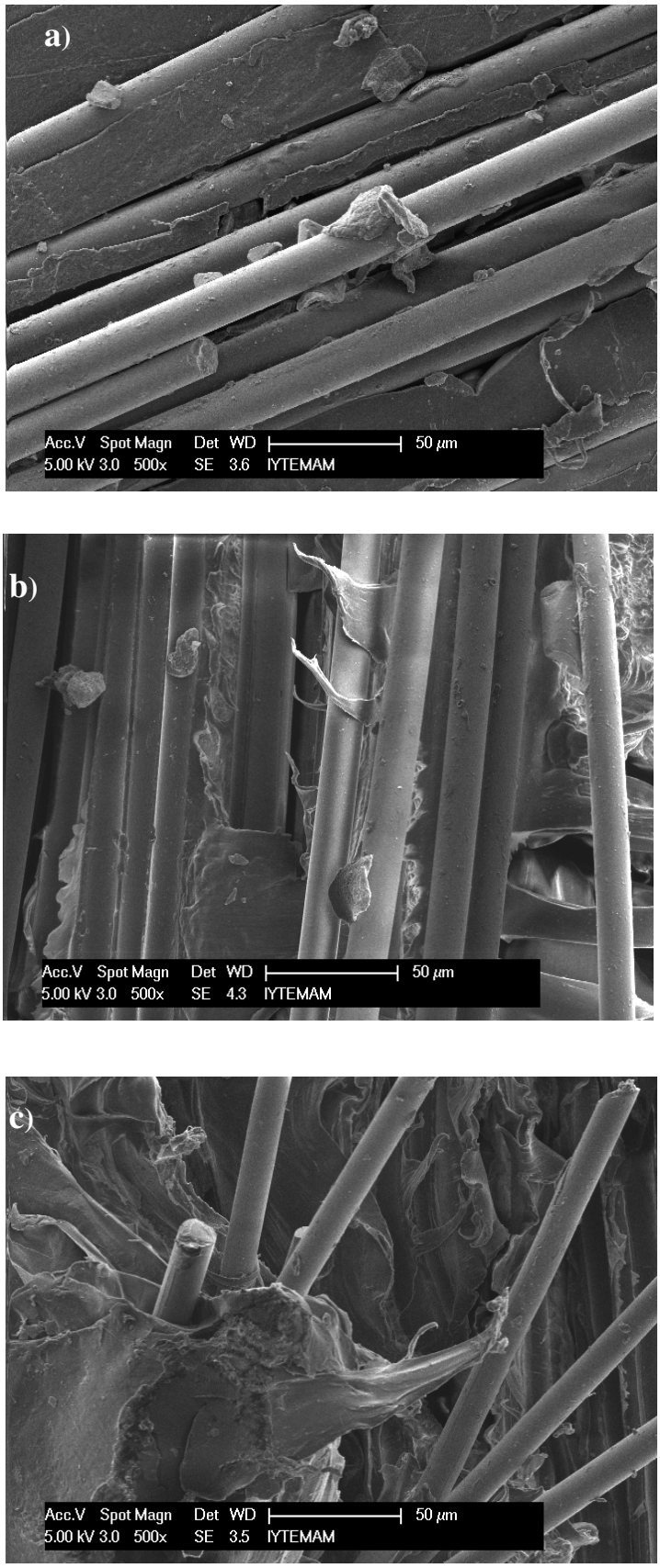



Figure 4.21. SEM images of the peeled surfaces of the composites coded with (a) D2 (b) D3 (c) D8 Magnification 500x

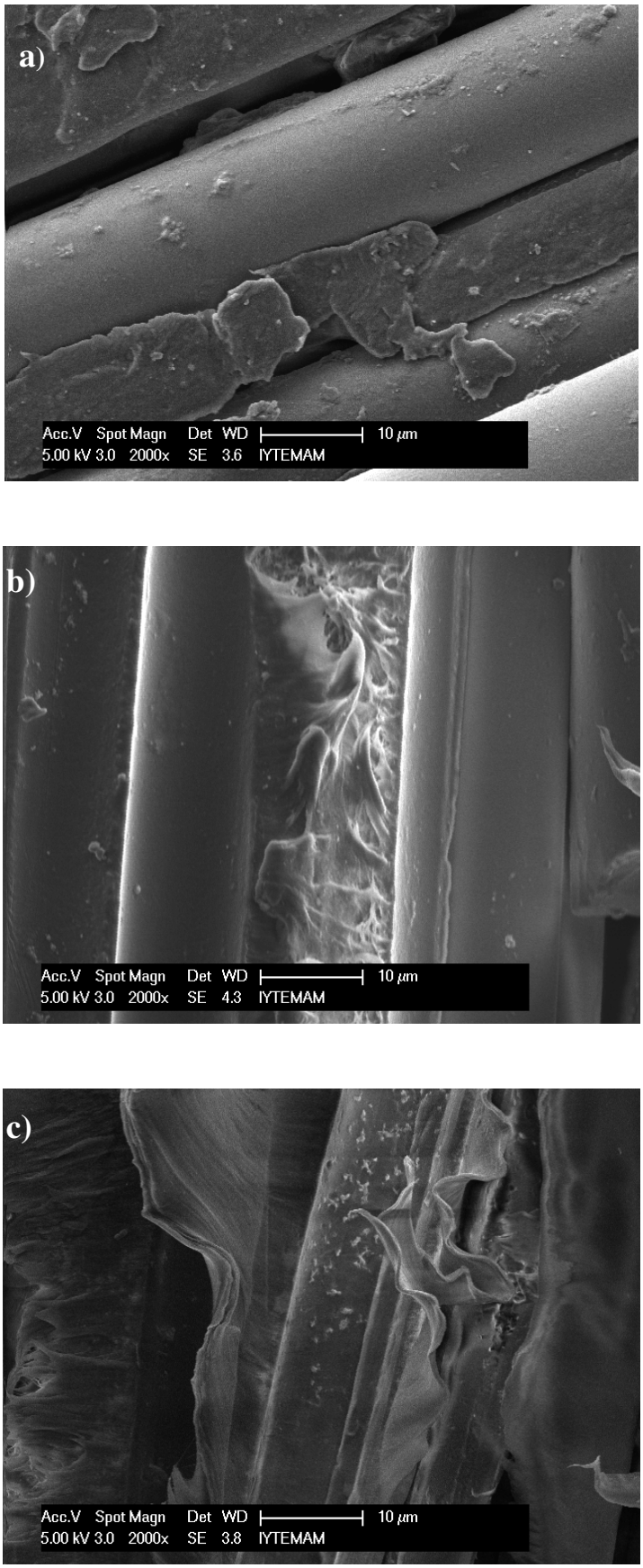



Figure 4.22. SEM images of the peeled surfaces of the composites coded with (a) D2 (b) D3 (c) D8 Magnification 2000x

SEM images taken from the peeled surfaces are in accordance with peel strength values obtained by interlaminar peel test. As seen in SEM images, a smoother fracture surface in which fracture occurred along the fiber surfaces or matrix material are observed for the composites prepared with fabrics D2 and D3 (in-compatible with PP). This indicates lower adhesion at the glass fiber-polypropylene interface. On the other hand, more complex failure modes and deformation of PP matrix are observed for composites with fabric D8. Due to better adhesion at fiber/PP interface, less amount of debonding of the interface and plastic deformation of the PP matrix around fibers was observed.

### 4.3.4. Compressive Properties

Compression test was performed to evaluate the compressive properties of the glass fiber/PP composites fabricated from fabric D8. Figure 4.23 shows typical compressive stress versus strain response of glass fiber/PP composite loaded along the ply-lay up direction.

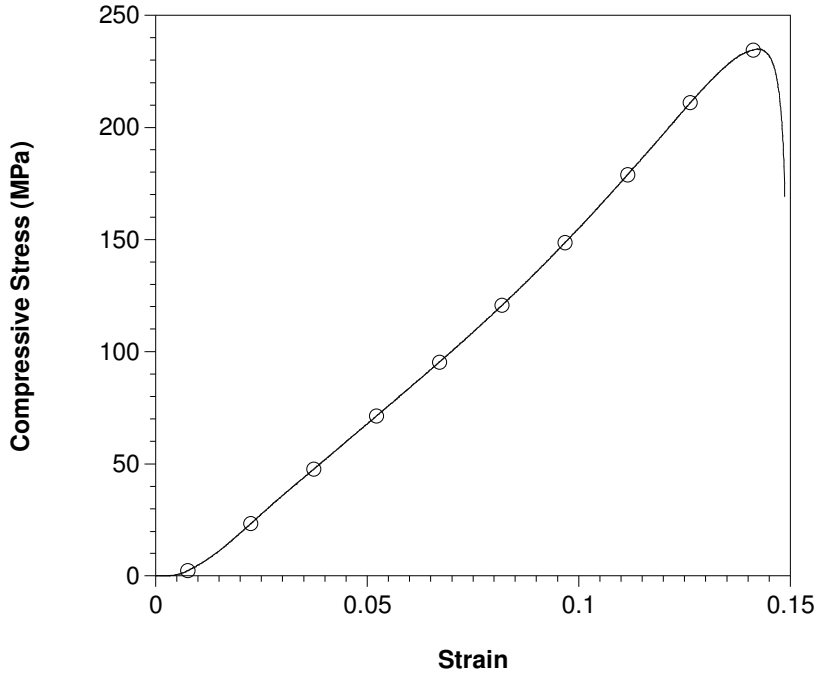



Figure 4.23. Typical compressive stress-strain curve of the glass fiber/PP composite loaded along ply-lay up direction

For loading in ply-lay up direction, stress-strain response of the composite is almost linear up to the maximum stress level at which failure initiates. There is a sudden drop of the stress after the maximum stress at which failure occurs and material loses its integrity. The average compressive strength and compressive modulus values of the glass fiber/PP composite loaded in ply-lay up direction were measured to be 240.18 (±3.6) MPa and 1.6 (±0.024) GPa respectively. It was found that the average strain values at the maximum stress for the specimens loaded along ply-lay up direction is about 0.171. Compression test specimen before and after along ply-lay up direction is shown in Figure 4.24.

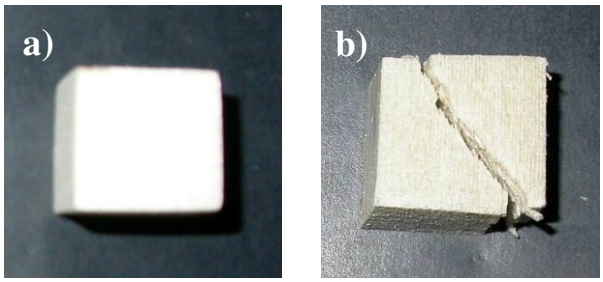



Figure 4.24.Compression test specimen before and after along ply-lay up direction

Compressive stress vs. strain curves of the glass fiber/PP composites loaded along in-plane direction is given in Figure 4.25. The stress-strain behavior of the composites loaded along the in-plane direction is less linear as compared to those for ply-lay up direction. The average maximum stress and compressive modulus values of the glass fiber/PP composite material loaded along the in-plane direction are 44.4 (±0.31) MPa and 0.98 (±0.089) GPa, respectively. It was found that the average strain value at the maximum stress for the specimens loaded along in-plane direction is about 0.074. The sudden decrease after maximum stress along ply-lay up direction may be due to the fact that the compressive strength is more matrix-property dominant in this direction. Along in-plane direction it is more related with interlaminar and interfacial bonding therefore sudden drop after max stress was not observed in this direction.

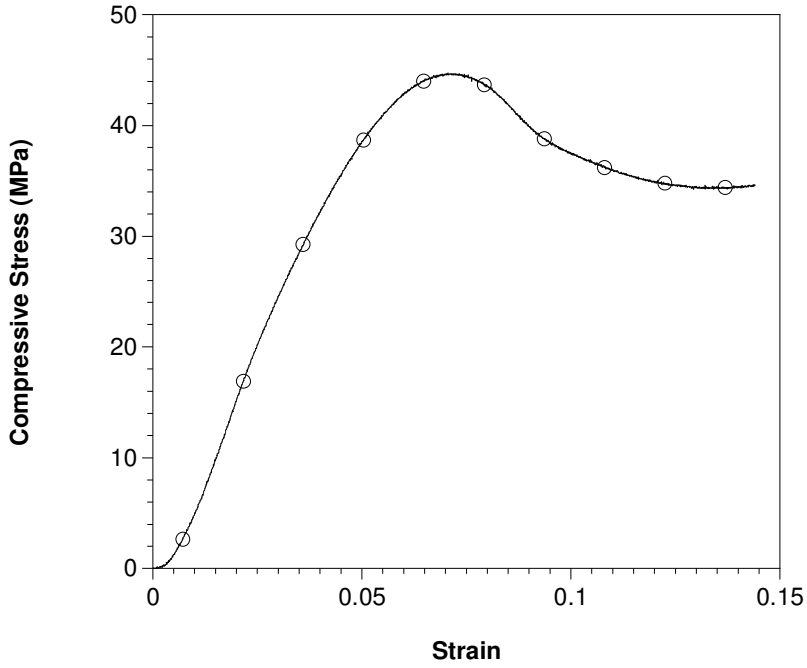



Figure 4.25. Typical compressive stress-strain curve of the glass fiber/PP composite loaded along in-plane direction

### 4.3.5. In-plane Shear Properties

In-plane shear tests were performed to evaluate the in-plane shear properties of the glass fiber/PP composites fabricated from non-crimp fabrics. Figure 4.26 shows the shear stress responses of the glass fiber/PP composites fabricated at 200 $^{0}$C process (hot pressing) and under 1.5MPa pressure. Maximum shear stress and offset shear strength of the glass fiber/PP composites are given in Table 4.4.

The highest offset shear strength was obtained from composite fabricated from fabric D8 (10.9 MPa). The second highest offset shear strength was obtained from the composite prepared with fabric D2. Offset shear strength values indicates that the composites made of fabrics fabricated by air-jet hybrid yarn preparation technique have higher in-plane shear properties than those fabricated by single twist hybrid yarn preparation technique. Maximum shear stress values of the composites also support this finding.

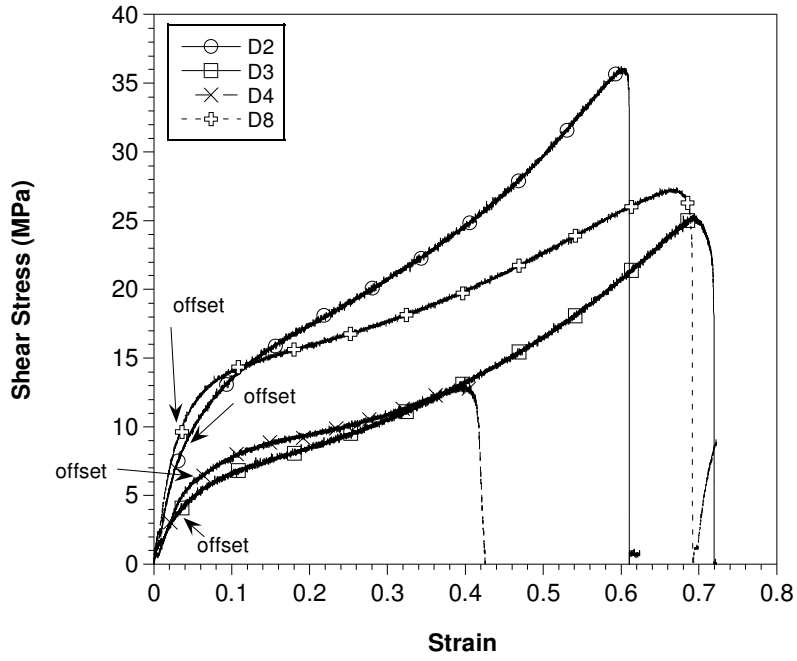



Figure 4.26. Shear stress vs. strain responses of the glas fiber/PP composites

Table 4.4. In-plane shear properties of glass fiber/PP composites

| Fabric ID | Max. Shear Stress (MPa) | Offset Shear Strength (MPa) | Hybrid Yarn Preparation Technique | Fiber Volume Fraction | Glass Fiber Sizing |
|---|---|---|---|---|---|
| **D2** | 36.1 | 7.8 | Air-Jet | 0.335 | PES resin compatible |
| **D3** | 25.1 | 5.4 | Single Twist | 0.352 | PES resin compatible |
| **D4** | 13.1 | 5.7 | Single Twist | 0.535 | PES resin compatible |
| **D8** | 27.5 | 10.9 | Air Jet | 0.342 | PP resin compatible |

Photo of in-plane test specimen before and after test is shown in Figure 4.27.

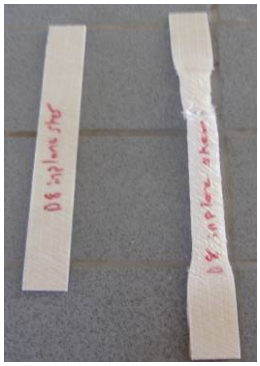


Figure 4.27. Photo of glass fiber/PP composite material before and after in-plane shear test

The thickness of individual plies is an important parameter that influences both the shear stress-strain response and ultimate failure load of the specimen. Fabric D4 has lower nominal weight and ply thickness as compared with other fabrics. Based on the results given in Figure 4.26 and Table 4.4 composites made of D4 have the lowest shear strength values due to the lowest thickness of the individual plies of the composite.

## 4.4. Impact Properties

Impact properties of the glass fiber/PP composite materials were evaluated with charpy impact tests. Absorbed energies of the composites are given in Table 4.5.

Table 4.5. Impact Properties of glass fiber/PP composites

| SAMPLE ID | Max.absorbed energy(kJ/m$^2$) | Min. absorbed energy (kJ/m$^2$) | Avg. absorbed energy (kJ/m$^2$) | Standard Deviation |
|---|---|---|---|---|
| **D2** | 224 | 146 | 197 | 32 |
| **D3** | 221 | 132 | 175 | 32 |
| **D4** | 133 | 104 | 115 | 11 |
| **D5** | 225 | 156 | 195 | 35 |
| **D8** | 225 | 153 | 198 | 26 |

Based on the results in Table 4.5, it can be concluded that hybrid yarn preparation technique and type of the glass fiber sizing have insignificant effect on the impact properties of the glass fiber/polypropylene composite. The average absorbed energy values of the composites D2, D5 and D8 are very close to each other and it is in the range of 195-198 KJ/m$^2$. These results are in agreement with the other studies in the literature. Composites made of fabric D4 exhibited lower impact properties than those of other composites. It may be due to poor in-plane properties and low thickness of individual plies. Santulli, et al. (2002) studied impact properties of commingled E-glass/PP composites fabricated by compression moulding. They have reported the charpy impact results of glass fiber/PP composites with glass fiber content similar to values used in this study. Similar to the present work, their composite's average absorbed energy value was found to be 196.9 KJ/m$^2$ (±24.9 KJ/m$^2$). In another study, Santulli (2002) have studied impact properties of glass fiber/PP composites at different tool temperatures and reported that absorbed energy value of the composites change between 180 and 197 KJ/m$^2$ depending on tooling temperature. Zhao, et al. (2009) studied impact properties of stitched glass fiber/PP composites with 82.5% volume fraction of glass fiber and reported absorbed energy of 250 KJ/m$^2$. Photo of test specimens after charpy impact test is given in Figure 4.28.

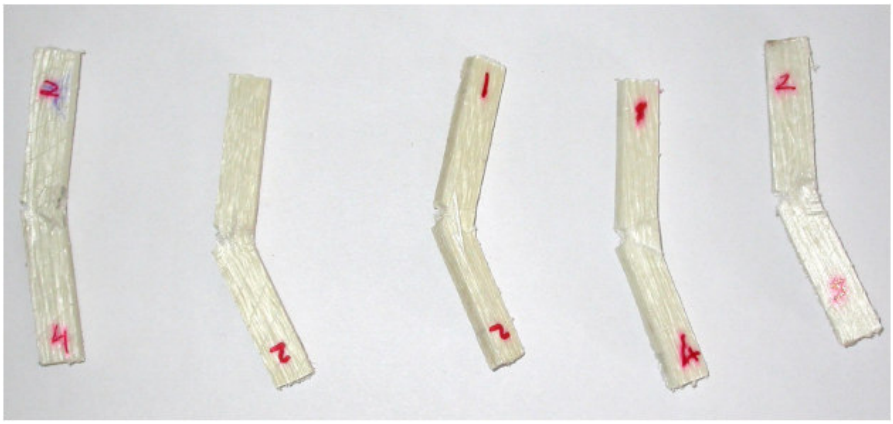


Figure 4.28. Photo of test specimens after charpy impact test

# CHAPTER 5

# CONCLUSIONS

In the present study, comingled glass fiber/ polypropylene (PP) hybrid non-crimp fabrics were developed. Glass fiber reinforced PP matrix composites were manufactured from these hybrid fabrics by compression moulding. Microstructural and mechanical properties of the manufactured thermoplastic composite materials were investigated. Also effects of fabric type, hybrid yarn preparation technique, glass fiber sizing and process conditions such as temperature and pressure, on the mechanical and micro structural properties of the composite materials were examined.

Glass fiber/PP composites were manufactured from three different types of fabrics. First type of fabric was produced by air jet hybrid yarn preparation technique. The other two fabrics were produced by direct twist covering hybrid yarn preparation technique. One of them was single (S) twist and the other was double (SZ) twist. In fabric D8, PP resin compatible sizing applied glass fiber used. In all the other fabrics polyester resin compatible sizing were used. Continuous fiber reinforced thermoplastic composite material was produced by hot press compression moulding of glass fiber/PP hybrid non-crimp fabrics.

Micro structural characterization was applied on fabrics before and after lamination. Based on optical microscope images, glass fiber bundles appear to be in order before lamination. However, after lamination of the fabrics by compression moulding, it was observed that fiber bundles was slightly distributed due to the thermal stresses occurred during melting process of the polypropylene matrix. As hybrid yarn preparation techniques are compared, the best glass fiber orientation was obtained with fabrics produced by air jet hybrid yarn preparation technique.

Tensile tests were performed in order to investigate tensile properties of the composites. The highest tensile strength and elastic modulus values (262.7 MPa tensile strength and 13.83 GPa elastic modulus) were obtained from composites panels produced from fabric coded as D4 which has the highest content of glass fiber. As composite panels with similar fiber volume fractions are compared, composite panels produced from the fabrics prepared by air jet hybrid yarn preparation technique (fabrics coded as D2 and D8) exhibited the highest mechanical properties than those of composites produced from the

fabrics fabricated by single twist hybrid yarn preparation technique (fabric coded as D3). The lowest values were obtained from the composite panels produced from the fabrics fabricated by double twist hybrid yarn preparation technique (Fabric coded as D5). It was observed that glass fiber sizing has insignificant effect on the tensile properties of the glass fiber/PP composites. Tensile test result showed that hybrid yarn preparation technique plays a dominant role on the tensile properties. Finally effect of process temperature on the tensile properties of the composites was investigated. Tensile properties slightly increase up to 220 $^{o}$C with increasing temperature and slightly decrease at 240 $^{0}$C.

Flexural test was performed in order to investigate flexural properties of the composites. Highest flexural properties were obtained with composite D8. It was observed that PP resin compatible sizing have positive effects on the flexural properties of the composites as compared to those with PES resin compatible sizing. Also composites of the fabrics produced by air jet hybrid yarn preparation technique exhibited higher flexural properties than those of composites of the fabrics produced by direct twist covering hybrid yarn preparation technique. Based on the flexural test results, it was observed that type of the hybrid yarn preparation technique and glass fiber sizing applied on the glass fibers have some important role on the flexural properties of glass fiber/PP composites. Effect of process (hot-pressing) temperature was also investigated. It was found that composites produced at 180 $^{o}$C exhibited the lowest flexural properties. It was concluded that the inefficient impregnation of the polypropylene matrix into the glass fibers at low temperatures results with lower mechanical properties. There is no significant change on the flexural properties at process temperatures above 180 $^{o}$C. Also, effect of process pressure was investigated and it was observed that process pressure has no significant effect on the flexural strength and modulus of the glass fiber/PP composite material up to 2 MPa process pressure. On the other hand, flexural modulus and strength slightly decreases at pressures above 2 MPa. This was related to extensive flashing of the polypropylene matrix from the composite part as a result of high level of pressure.

Interlaminar peel test was performed in order to investigate the effect of sizing applied on glass fibers. The best results were obtained from composite containing compatible sizing (coded as D8). Interlaminar peel strength of composite with D8 was found to be 1.47 times higher than those of composite with D2. Results were supported with SEM images. A relatively lower amount of debonding and higher amount of plastic deformation of the PP matrix around the fibers were observed for composites with D8

fabric. However, for composites with fabrics D2 and D3 (in-compatible sizing with PP) a smoother fracture surfaces with higher amount of debonding were observed.

Compression tests were applied to composite coded as D8 along ply-lay up and in-plane directions. The stress-strain behavior of the composites loaded along the in-plane direction is less linear as compared to those for ply-lay up direction sudden drop of stress was observed after the maximum stress at which failure occurs. The average compressive strength and compressive modulus values of the glass fiber/PP composite loaded in ply-lay up direction were measured to be 240.18 MPa and 1.6 GPa respectively. Strength and modulus values of the composite loaded along the in-plane direction were 44.4 MPa and 0.98 GPa respectively.

In-plane shear test was also performed to investigate the in-plane properties of the glass fiber/PP composites. The highest maximum and offset shear strength values were obtained from composites produced from fabrics fabricated with air-jet hybrid yarn preparation technique (composites D2 and D8). The lowest in-plane shear properties were obtained from composite coded as D4 due to lowest thickness of the individual plies of the composite, which is an important parameter that influences both the shear strain-stress response and ultimate failure load of the specimen.

Impact properties of the glass fiber/PP composites were evaluated with charpy impact tests and absorbed energy values were calculated. Based on absorbed energy values it is concluded that hybrid yarn preparation technique and type of glass fiber sizing have insignificant effect on impact properties of the glass fiber/PP composites. The average absorbed energy values of the composites for D2, D5 and D8 fabrics are very close to each other and it is in the range of 195-198 $KJ/m^2$. These results are in agreement with the other studies in literature. Composites made of fabric D4 exhibited lower impact properties due to lower in-plane and interlaminar properties.

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