STUDIES ON GLASS - POLYESTER COMPOSITES THROUGH FILM STACKING TECHNIQUE

A PROJECT REPORT

Submitted by

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BONAFIDE CERTIFICATE

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ABSTRACT

Glass fiber Polyester composites have been manufactured through the film stacking technique. Glass fabrics of different GSM were sandwiched between polyester films of varying thickness, in different stacking sequences, and hot pressed. The effect of material and process variables viz., time, temperature and pressure has been investigated on the mechanical properties of the composites. Effect of variation in the reinforcing and reinforced phase is studied and correlated with properties. Time, Temperature a,d pressure cycles has been optimised for the GF/PET composites.

High volume fraction of around 40% has been achieved with superior mechanical properties. The composites prepared had high load bearing capacity of ~55kg and high work of rupture of 13 kg-cm. The work of rupture increased by 550% for the lowest sample and elongation by 100% and the load bearing capacity by 225% when compared to the virgin glass fabric. The evidence of bridging fibers in the optical micrographs and composite failure in the ductile mode indicated good fiber matrix adhesion.

சாராம்சம்

கண்ணாடி இழை / பாலியெஸ்டர் குங்கிலிய தொகுப்பானது, மென் படல உத்தி மூலம் உற்பத்தி செய்யப்படுகிறது. அத்தொகுப்பானது நெசவு செய்த கண்ணாடித் துணி மற்றும் மெல்லிய பாலியெஸ்டர் குங்கிலியங்களின் வெவ்வேறு கொள்ளளவுகளில் அடுக்கி உயர் வெப்ப அழுத்தத்தின் மூலம் தயார் செய்யப்படுகிறது. இம்முயற்சியில் உற்பத்தி செய்த தொகுப்புகளின் பௌதிகத் தன்மைகளை கால நேரம், வெப்ப நிலை மற்றும் அழுத்தம் ஆகிய மூன்று செயல் வேறுபாடுகளைக் கொண்டு ஆராயப்படுகிறது மற்றும் ஒருங்கிணைப்பு மாற்ற நிலைகளும், ஒருங்கிணைப்பு படிகளும் இத்தொகுப்பின் தன்மைகளோடு ஆராயப்படுகிறது.

இதனால் இத்தொகுப்பின் பகுதி கொள்ளளவு 40% வரை உயர்ந்துள்ளது மற்றும் நாம் தயாரித்த தொகுப்பின் பாரம் தாங்கும் தன்மையை ஆராய்ந்த பொழுது 55 கிகி எடை வரை நீடித்து உழைக்கும் திறன் பெற்று 18 கிகி செ.மீ. தகர்வுத் திறனை கொண்டுள்ளது. கண்ணாடி இழை / பாலியெஸ்டர் குங்கிலய தொகுப்பின் விரிசல் ஆய்வுகள் இம்முயற்சியில் மேற்கொள்ளப்பட்டுள்ளது.

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S.No	Nomenclature	Symbol
1.	Microns-Thickness of the Polymer matrices	μ
2.	Volume Fraction of the Fibre	V_f
3.	Volume Fraction of the matrix	V_m

1. **Introduction**:

In the global market of industrial textiles, there is a great improvisation of textile product manufacturing strategies, absolute modern technology implementation procedures, which engraves the values of the product placing at higher standards in the market. Especially the role of composites plays an important part at this arena with its different shapes of applications in terms of raw material implied, technology followed during manufacturing, etc.

Mainly, the composites made out of Glass/Polyester as the basic raw materials play its part at various end applications such as Protective garments from impacts, chemicals, heat etc… and automobile interiors, shielding materials etc. The technique of manufacturing such composites depends upon its area of application.

In this project glass fabrics along with the polyester films as the polymer matrix which are stacked together in various sequence and the sandwich is subjected to high pressure and temperature resulting in the formation of Glass/Polyester composites. Various combinations of glass fabrics as well as polyester films are sandwiched by optimizing the parameters such as temperature, pressure and time.

The quality and characteristics of the manufactured composite materials are optimized in order to enhance the experimental procedures and the production techniques in a better way so as to obtain a quality product.

2. Fiber Composites

Fibrous composite materials typically have two or more distinct phases, which include high strength/stiffness reinforcing fibers and the encapsulating matrix material. Fibers can be either discontinuous (chopped) or continuous whereas the Polymer matrices typically fall into two categories: thermoplastic and thermosetting polymers. Thermoplastic polymers are distinguished by their ability to be reshaped upon the addition of heat (above the glass transition temperature of the amorphous phase or the melting temperature of the crystalline phase). This cycle can be carried out repeatedly. Thermosetting polymers, on the other hand, undergo chemical reactions during curing which crosslink the polymer molecules. Once crosslinked, thermosets become permanently hard and simply undergo chemical decomposition under the application of excessive heat. Thermosetting polymers typically have greater abrasion resistance and dimensional stability over that of thermoplastic polymers, which typically have better flexural and impact properties.

3. Thermoplastic Composite Materials

Throughout the prior two decades, fiber reinforced composite materials were principally fabricated using thermosetting matrices. Disadvantages stemming from the use of thermosets include brittleness, lengthy cure cycles and inability to repair and/or recycle damaged or scrapped parts. These disadvantages led to the development of the thermoplastic matrix composite system. Compared with thermosets, composites fabricated from thermoplastic materials typically have a longer shelf life, higher strain to failure, are faster to consolidate and retain the ability to be repaired, reshaped and reused as need arises.

However, as in many polymer composite systems, these materials frequently suffer from a lack of adequate fiber-matrix adhesion. In addition, the use of thermoplastics introduces the problem of adequate fiber tow penetration. Thermoplastic melts, as opposed to thermosetting resins, have a substantially higher viscosity. Thermoplastic matrices must be able to withstand high temperatures in order to effect a sufficient reduction in viscosity. Additional problems caused by high matrix viscosity during consolidation include de-alignment of reinforcing fibers during consolidation as well as the introduction of voids within the final composite product. All of these problems can be addressed by appropriate design regarding the fiber-matrix interface as well as optimization of composite fabrication procedures. Composites prepared with satisfactory matrix dispersion within the fiber tows as well as reasonable fiber-matrix adhesive interaction typically results in composites with good mechanical properties.

3.1 Fabrication techniques for Thermoplastic Composites

The first step in the design of a thermoplastic composite material is an appropriate choice of a fabrication method. There are several widely used methods to prepare thermoplastic composite materials, several of which are outlined below.

3.1.1 Solution Impregnation

The solubility of thermoplastics as opposed to thermosetting materials has led to the use of solution deposition and impregnation of matrix material onto fiber tows. 'Solution prepregging' is a common technique used to fabricate thermoplastic composite materials. This process entails solubilizing the matrix polymer at a suitable concentration and then immersing the fiber tow within the solution. In the ideal case, the polymer solution will individually wet each filament within the fiber tow. Once the fiber has been coated with solution, the fiber article is then removed from solution and the solvent must be evaporated before further fabrication steps are taken. Prepreg tows are then conventionally arranged in sheet form after which they are consolidated in a hot press to produce a composite article.

3.1.2 Powder Impregnation/Pultrusion

Another method of preparing composite preforms is powder-impregnation. In this case, the fabric or sheet reinforcement is impregnated with a suspension of fine (<5 micrometers) polymer particles. The prepregged sheet is then consolidated within a hot press. This method is restricted to polymer matrices that can be cryogenically ground to produce fine particles or precipitated from solution. Pultrusion operates in a similar manner. Fiber tow is pulled through powdered resin (30-250 micrometers) in a coating unit and then shaped and heated within a die. Pultruded tow is then arranged in sheet form and consolidated within a hot press.

3.1.3 Film Stacking

Film stacking is a third technique with which to prepare thermoplastic composites. In this case, fiber tows arranged in sheet form are sandwiched between matrix polymer films. This assembly is then placed within a press where temperature transforms the film into a polymer melt. Pressure is then applied and forces the melt to impregnate the fiber tow. Appropriate process conditions must be used in order to sufficiently reduce the matrix viscosity without thermally degrading the actual composite or de-align the fibrous reinforcement. Insufficient heat input and/or pressure will typically result in unwetted fiber and a high void content within the final material.

3.2 Consolidation of Thermoplastic Preforms:

After preparation, thermoplastic prepregs are then arranged in sheet form and stacked in either a unidirectional or cross-ply manner. This stack is then pressed within a mould under pressure and temperature. During pressing, a vacuum is frequently used in order to remove any air or solvents used to process the prepregs. The use of a vacuum during the final consolidation step can aid in the reduction of voids within the final composite article. Optimal process parameters, such as residence time, temperature and pressure must all be determined for the particular composite system and preform fabrication method.

4. Glass fibres:

Glass fibre is a generic name like carbon fibre or steel. Common glass fibres are silica based(~50-60% SiO_2) and contain a host of other oxides of calcium, boron, sodium, aluminium and iron. Table 1 gives the compositions of some commonly used glass fibres. The designation E stands for electrical because E glass is a good electrical insulator in addition to having good strength and a reasonable Young's modulus; C stands for corrosion because C glass has a better resistance to chemical corrosion; S stands for the high silica content that makes S glass withstand higher temperatures than other glasses. It should be pointed out that most of the continuous glass fibre produced is of the E glass type but, notwithstanding the designation E, electrical uses of E glass fibre only a small fraction of the market.

The following table illustrates about the composition of Glass fibres..

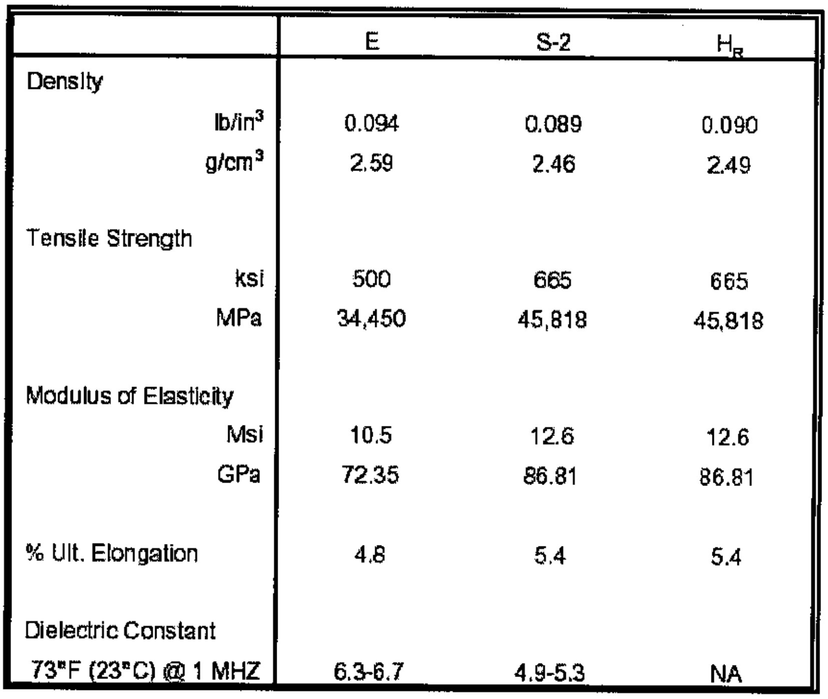
	(By Weight)	
	E-Glass	**S-Glass**
Silicone Dioxide	52 - 56%	64 - 66%
Calcium Oxide	16 - 25%	0 - .3%
Aluminum Oxide	12 - 16%	24- 26%
Boron Oxide	5 -10%	
Sodium Oxide & Potassium Oxide	0 - 2%	0 - .3%
Magnesium Oxide	0 - 5%	9 - 11%
Iron Oxide	.05 - .4%	0 - .3%
Titanium Oxide	0 - .8%	
Fluorides	0 -1.0%	

Table 4.1:Composition of commercial glass fabrics

4.1 Characteristics of Glass fibres:

A wide variety of properties can be achieved through proper selection of glass type, filament diameter, sizing chemistry and fiber forms (e.g., rovings, fabrics, etc.).

The reinforcing part in the study was glass plain woven fabrics. Some of the basic properties of Glass fibers are discussed as below:



	E	S-2	H_R
Density			
lb/in^3	0.094	0.089	0.090
g/cm^3	2.59	2.46	2.49
Tensile Strength			
ksi	500	665	665
MPa	34,450	45,818	45,818
Modulus of Elasticity			
Msi	10.5	12.6	12.6
GPa	72.35	86.81	86.81
% Ult. Elongation	4.8	5.4	5.4
Dielectric Constant 73°F (23°C) @ 1 MHZ	6.3-6.7	4.9-5.3	NA

Table 4.2: General characteristics of Glass fibres

Overall, glass fibers exhibit excellent thermal and impact resistance, high tensile strength, good chemical resistance and outstanding insulating properties. Fibers are silica-based glasses that contain several metal oxides, which can be tailored to create different types of glass fibers.

Because E-glass offers good strength properties at a low cost, it accounts for more than 90 percent of all glass fiber reinforcements. Named for its good electrical resistance, E-glass is particularly well-suited to applications where radio-signal transparency is desired, as in aircraft radomes and antennae. E-glass is also used extensively in computer circuit boards to provide stiffness and electrical resistance. Along with more than 50 percent silica oxide, this fiber also contains oxides of aluminum, boron and calcium, as well as other compounds.

4.1.1 Strength and corrosion characteristics:

When greater strength and lower weight are desired, high-strength glass or other advanced fibers, such as carbon, may be selected. High-strength glass is generally known as S-type glass in the United States; it is often called R-glass in Europe and T-glass in Japan. Originally developed for military applications in the 1960's. A lower cost version, S-2 glass, was later developed for commercial applications.

High-strength glass has appreciably higher silica oxide, aluminum oxide and magnesium oxide content than E-glass. Typically, S-2 glass is approximately 40 percent to 70 percent stronger than E-glass.

While glasses are generally considered to have relatively high chemical resistance, the fibers are eroded by leaching action when exposed to water. For instance, a 10-micron diameter E-glass filament typically loses .07 percent of its weight when left in hot water for 24 hours. This erosion rate slows significantly as leached glass forms a protective barrier on the outside of the filament; only 0.9 percent total weight loss occurs after seven days of exposure. Resin addition during composite formation further protects the fibers.

4.1.2 Thermal Characteristics:

Thermal properties are also an important consideration in selecting glass type. As the temperature increases, glass fibers lose tensile strength. C-glass has poor high-temperature performance and is not used in these applications. Both E-glass and S-type glass lose approximately 50 percent of their tensile strength as temperature increases from ambient temperature to 1000° F. Even so, these fibers exhibit good strength at high temperature.

Another temperature-related property is the coefficient of thermal expansion (CTE). A high CTE indicates that the fiber expands more as temperature increases. To prevent problems from occurring due to differential thermal expansion, the CTE's of the fiber and resin should be similar. S-type glass has a much lower CTE than either E-glass or C-glass.

4.1.3 Physical and Mechanical Characteristics

Table 3 elucidates the physical properties and the mechanical properties of glass fibres with respect to its type of application.

Fibre	**Softening Temp ^{0}C**	**Annealing Temp ^{0}C**	**Bulk Density annealed glass (g/cm^3)**	**Specific Heat cal/g/^{0} C**	**Tensile Strength at (23°C)**	**Young's Modulus (Gpa)**	**Filament Elongation at break (%)**
Boron containing glass	830-860	657	2.54-2.55	0.192	3100-3800	76-78	4.5-4.9
Boron-free glass	916	736	2.62	---	3100-3800	80-81	4.6
ECR glass	880	728	2.66-2.68	---	3100-3800	80-81	4.5-4.9
D-glass	770	---	2.16	0.175	2410	---	---
S-glass	1056	---	2.48-2.49	0.176	4380-4590	88-91	5.4-5.8
Silica/Quartz	---	---	2.15	---	3400	69	5

Table 4.3: Physical and Mechanical Properties of Glass Fibres

4.1.4 Style Alterations characteristics:

	What Happens To:				
If You	**Cost**	**Thickness**	**Weight**	**Strength**	**Porosity**
Increase the No. of Picks	+	+	+	+	-
Increase the No. of Ends	+	+	+	+	-
Increase Yarn Size (filament size or No.)	+	+	+	+	-
Change from Filament to Textured	+	+	+	-	+
Increase No. of Plies (e.g. 75 1/0 to 150 1/2)	+	0	0,+	0,+	+
Change from E to S glass	+	+	0	+	0

+ = increase, - = decrease, 0 = no change

Weave Rankings	**Cost**	**Thickness**	**Weight**	**Strength**	**Porosity**
Plain	1	3	1	3	1
Twill	1	2	1	4	2
4-Harness Satin (Crowfoot)	1	3	1	4	2
8-Harness Satin	1	1	1	7	4
Leno	7	7	7	1	7
Mock Leno	1	6	1	2	4

1 = lowest cost, 1 = least thick, 1 = lightest, 1 = weakest, 1 = closed

7 = highest cost, 7 = thickest, 7 = heaviest, 7 = strongest, 7 = open

Table 4.4: Style Alteration characteristics table of glass fibre/fabrics.

4.1.5 Characterisitcs of Woven Glass Fabrics:

Woven fabrics are fabricated on looms in a wide variety of weights, weaves and widths. Bi-directional woven fabrics provide good strength in the 0- and 90-degree directions. They also allow faster composite fabrication compared to using two laminates and applying one in each direction. However, woven fabrics provide lower tensile strength than separate laminates because fibers are crimped as they pass over and under one another. Under tensile loading, these fibers try to straighten out, causing stress within the resin matrix system.

Several different weaves are used for bi-directional fabrics. In plain weave, each fill yarn or roving alternately crosses over and under each warp fiber. Other weaves, such as harness satin and basket weave, allow the yarn or roving to cross over and under multiple warp fibers at a time. These weaves tend to be more pliable and conform more easily to curved surfaces. Woven roving is a thick fabric that is used for heavy reinforcement, especially in hand lay-up operations. Due to its relatively coarse weave, woven roving wets quickly and is relatively inexpensive. Exceptionally fine fiberglass fabrics can also be produced. One use for these fine fabrics is for reinforcing printed circuit boards.

4.2 Factors affecting end properties of the composite

4.2.1 Interphase

It has long been recognised that the mechanical properties of semicrystalline polymers and the composites made therefrom are dependent on the crystallinity and crystalline morphology, which are strongly affected by the processing conditions [i] [ii].

The interactions at the interphase region in semicrystalline thermoplastic composites depend on a number of factors, such as matrix morphology, fibre surface condition, presence of residual stresses, moduli of the fibre and matrix, as well as the presence of reactive functionalities. The majority of these characteristics are determined by processing conditions, including moulding temperature, cooling rate, holding time/temperature and annealing conditions.

4.2.1.1 Coupling agents for Glass/Polyester

In order for the composite to function properly there must be a chemical bond between the matrix and the re-inforcing fibres in order that the applied load (applied to the matrix) can be transferred to the fibres (which are expected to do all the work. However, the bond must not be too strong since the toughness of the composites comes from such sources as fibre pullout and fibre-matrix interfacial fracture. In 'fibre glass' the fibre is inorganic while the matrix is organic and the two do not bond readily unless the fibres are treated to modify their surface.

Silica (SiO_2) is hygroscopic ie. it absorbs water onto its surface where the water breaks down into hydoxyl (-OH) groups. It is impossible to avoid the water especially as the surface modifier or 'size' is applied in a water based solvent. It should also be stressed that water reduces the strength of SiO_2 by a stress-corrosion-cracking mechanism. The coupling agent takes the form of a silane ($R-SiX_3$) where R is an organic radical that is compatible with the polymer matrix (it may even react with the matrix polymer; for this reason styrene groups are favoured for polyesters while amine groups are preferred for epoxies) and X is a hydrolisable organic group such as an alcohol. The most common silane couplant is tri-ethoxy-silane.Heat will force the elimination of water between the -OH pairs at the hydrated silica surface and the silane as well as between the adjacent silane moecules.

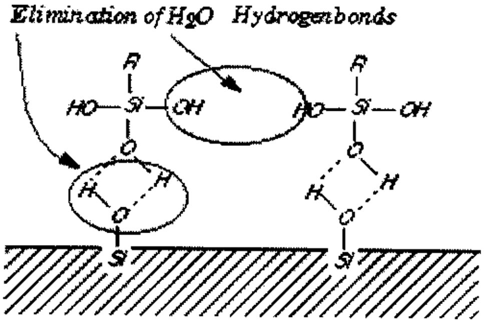


Fig 4.1 : Schematic Bonding of silica and polymer

4.2.2 Processing conditions

The interactions at the interphase region in semicrystalline thermoplastic composites depend on a number of factors, such as matrix morphology, fibre surface condition, presence of residual stresses, moduli of the fibre and matrix, as well as the presence of reactive functionalities.

The majority of these characteristics are determined by processing conditions, including moulding temperature, cooling rate, holding time/temperature and annealing conditions.

These are the three main parameters which affects the end properties of the composites made out of Glass/polyester combinations.

4.2.3 Presence of Moisture

Moisture penetration into composite materials is conducted by one major mechanism, namely diffusion. This mechanism involves direct diffusion of water molecules into the matrix and, in some cases, into the fibers. The other two common mechanisms of moisture penetration into composite materials are capillary flow along the fiber/matrix interface, followed by diffusion from the interface into the bulk resin, and transport by microcracks. Each of these mechanisms becomes active only after the occurrence of specific damage to the composite. Often, that damage, which enhances moisture penetration by activating those additional mechanisms, is in itself a direct consequence of the exposure of the composite to moisture.

5. Glass-Polyester composites:

Fiber preforms can be made either by introduction of the binder over the glass fabrics to bind them together or by weaving, braiding, knitting or stitching continous fibres[iii] [iv] [v]. In a work by Tanoglu et al[vi], the effects of the preforming binder on the mechanical and ballistic performance of E (electrical)-glass/polyester composite systems were investigated. It was found that the peel strength of the preforms increased with increasing binder content. The results also revealed that the flexural strength and modulus, Mode I interlaminar fracture toughness, ballistic performance and failure damage due to ballistic impact of the composite laminates are considerably affected by the presence of the binder.

Liquid molding (LM) processes such as resin transfer molding (RTM) and vacuum-assisted resin transfer molding (VARTM) have received considerable attention to manufacture high performance composite parts especially for automotive, civil engineering and military applications. RTM and VARTM processes have some unique advantages over all other composites manufacturing techniques[vii] [viii] [ix]. These techniques offer an opportunity to produce large and complex shapes with the desired fiber orientation into a single molding via fiber preforms that may have the shape and dimensions of the final parts. Fiber preforms can be made either by introduction of the binder over the glass fabrics to bind them together or by weaving, braiding, knitting or stitching continous fibres. The recent studies showed that net shape thermoformable preforms can be produced by introduction of plastic binders between reinforcement fabrics to supply brief compaction[x].

Compressive mechanical behaviour and failure modes of E (electrical)-glass/polyester composite laminates tailored with a thermoplastic preforming polyester binder were investigated by Metin Tano¢glu[xi] under ply-lay up and in-plane loading directions. Fiber preforms with various amount of the binder were consolidated under heat and pressure. The preform compaction experiments were performed by applying compressive pressure to the preforms, and the average thickness as a function of pressure was measured. It was found that the highest compaction of the preforms and therefore the highest fiber volume fraction can be obtained with 3 wt.% of the binder. Further increase of the amount of binder decreases the degree of compaction. Composite panels were fabricated by vacuum-assisted resin transfer molding using fabric preforms with various binder concentrations. The present investigation reveals that there are considerable effects of the binder on the compressive mechanical behaviour of the composites. Compression testing of the composites showed that the average strength values are in the range of 400–600 and 150–300 Mpa for ply-lay up and in-plane directions, respectively. Also, both the strength and modulus values increase up to 3 wt.% of the binder, and these values decrease with further addition of the binder. Scanning electron microscopy showed that failure modes of the composites are altered significantly by the presence of the binder.

Glass fabric/polyester composites of varying interfacial strengths have been subjected to water absorption cycles by S. Pavlidou,[xii]. Modification of the interfacial strength was achieved through different surface treatments of the fabrics. Composites containing clean glass fabrics, fabrics treated with a silane coupling agent and fabrics coated with a polydimethylsiloxane elastomer have been studied. Specimens were immersed in water for a short period of time, dried and subjected to three more reabsorption steps. Measurements of the interlaminar shear strength throughout the sorption–desorption cycles allowed estimation of the interfacial contribution to the absorption behavior. Results showed that a strong interface leads to 'matrix-dominated' absorption behavior, where in each successive sorption step the material absorbs higher amounts of water. This is attributed to matrix damage induced by moisture during previous absorption cycles. On the other hand, weak interfaces offer an easy path for water penetration in the composite. Thus, the absorption is an 'interface-dominated', more reversible phenomenon, since these cases, the matrix contributes less to the absorption process. The mechanical anchoring at the interface reforms on desorption and successive absorptions do not present pronounced differences.

Work by Dandekar et al[xiii] describes the compression and release response of a glass -reinforced polyester composite (GRP) under shock loading to 20 GPa. The diagnostic measurements fluctuate beyond the precision of the experimental measurements but they do permit determination of an average response of the material at the end state.

These experiments show that: (i) GRP deforms elastically in compression to at least 1.3 GPa; (ii) the deformation coordinates of shocked and re-shocked GRP lie on the deformation locus of initially shocked GRP to 4.3 GPa; (iii)

and the release path of GRP shocked to varying magnitudes of stresses indicate that the GRP expands such that its density when stresses are released in the range of 3–5 GPa from peak compressive stress of 9 GPa and above is lower than the initial density of GRP. Possible reasons for the observed lower density remain to be investigated.

Experimental Plan

Project methodology
Preliminary experiments
Characterisation

6. Project Methodology:

Materials (matrix and fabrics) used in this work are listed together along with their specifications in table 7. Glass fabrics as the core sheath and PET films as the matrices were selected for the production of Glass – Polymer composites by film stacking method. The glass fabrics and the PET films used were commercially procured under various specifications as per the experimental criteria.

Table 5.1: Raw material classification chart

Material	**PET film(PET-1)**	**PET film(PET-2)**	**PET film(PET-3)**	**Glass Fabric (GF-A)**	**Glass Fabric (GF-B)**	**Glass Fabric (GF-C)**
Thickness (mm)						
Nature	NA	NA	NA	Plain weave	Plain Weave	Plain Weave
Peak Melting point (^{0}C)	250	250	250	1600	1600	1600
Tenacity (MPa)	120.29	NA	NA	160	589	774
Elongation at break (%)	30.10	NA	NA	3.27	12.03	15.8

Composites were prepared in a compression-moulding machine. The mould was made of mild steel of dimensions 20x20x1 cm and also the samples as well. The glass fabric is sandwiched between two sheets of the polyester films and the entire sandwich is transferred to the die. Liquid silicon was sprayed all over the surface of the aluminium foil to enhance extraction of composites.

After the required temperature is reached, pressure is applied and the set up allowed to remain undisturbed for a particular time. The temperature, pressure and curing cycle was determined after the initial experiments. After the curing period the die is allowed to cool down in the atmospheric air until its temperature reaches 100 ^{0}C and the samples were taken out of the mould.

Process parameters of 1) Temperature 2) Pressure 3) Time, were varied during the preparation of the composites. The material parameters studied were gsm of matrices and fabric. Several combinations were tried and selected ones with good mechanical properties are reported.

6.1 Experimental Plan:

The experimental plan for the manufacturing of Glass/Polyester composite is shown in the Table 9. The various combinations of Glass Fabric/Polyester matrices were employed with respect to the variations in Temperature and Pressure as the main variables.

The entire work has been charted out with the help of a flow chart as follows.

6.2 Characterisation of composites

The composites fabricated were characterised in terms of the thermal, mechanical and morphological properties.

In order to undergo such processes the following test methods procures the resultant values with respect to the test principles.

6.2.1 Thermal Properties

6.2.1.1 Differential Scanning calorimetry studies:

The thermal behaviour of the samples was determined with a Perkin-Elmer Differential Scanning Calorimetry (DSC) in nitrogen atmosphere. The melting point (T_m) was determined from the peak of the endotherm resulting from scanning a sample (of around 5 mg weight) at 10^0 C per minute. The cooling cycle of the DSC scan was done to investigate the crystallization behaviour of the matrix.

6.2.1.2 Thermogravimetric Analysis:

Thermogravimetric Analysis (TGA) is a thermal analysis technique used to measure changes in the weight (mass) of a sample as a function of temperature and/or time. TGA is commonly used to determine polymer degradation temperatures, residual solvent levels, absorbed moisture content, and the amount of inorganic (noncombustible) filler in polymer or composite material compositions.

A simplified explanation of a TGA sample evaluation may be described as follows. A sample is placed into a tared TGA sample pan which is attached to a sensitive microbalance assembly. The sample holder portion of the TGA balance assembly is subsequently placed into a high temperature furnace. The balance assembly measures the initial sample weight at room temperature and then continuously monitors changes in sample weight (losses or gains) as heat is applied to the sample. TGA tests may be run in a heating mode at some controlled heating rate, or isothermally. Typical weight loss profiles are analyzed for the amount or percent of weight loss at any given temperature, the amount or percent of noncombusted residue at some final temperature, and the temperatures of various sample degradation processes.

The glass fabric and a composite sample were subjected to TGA under nitrogen atmosphere to investigate the thermal stability of the samples, till 1000^0C at a heating rate of $10^0C/min$.

6.2.2 Intrinsic property : volume fraction.

6.2.2.1 Polymer Digestion Test:

The composites made out of glass/polyester materials were subjected to the determination of the volume fraction of the material by the polymer digestion test. This test is carried out by dissolving the composite material with the help of the mixture of Phenol and Di-chloro methane in the 50:50 v:v ration at 100^0C. Before initializing the experiment the material taken should be weighed in a digital balance of weight X and immersed in the solution until the polymer gets dissolved from the composite sample. After dissolving, the final weight of the sample Y is measured . By evaluating the values of X and Y the Volume Fraction of the material is estimated by,

$$\text{Volume Fraction of the material } (V_f) = \left[\frac{X-Y}{X}\right] \times 100$$

Where,

X – Weight of the sample before dissolving the polymer in gms

Y - Weight of the sample after dissolving the polymer in gms

V_f - Volume fraction of the material in %

6.2.3 Mechanical Properties

6.2.3.1 Static Mechanical Properties:

Tensile properties were measured on a universal tensile tester according to ASTM D3039 at a speed of 30mm/min. The tensile specimen geometry recommendation as given for unidirectional composites were attained by proportionally reducing the test sample size to meet the ASTM geometry recommendations. Thus four rectangular specimens of 1cm by 14 cm were cut from the composites, the sample geometry detailed in the Fig 6.1.

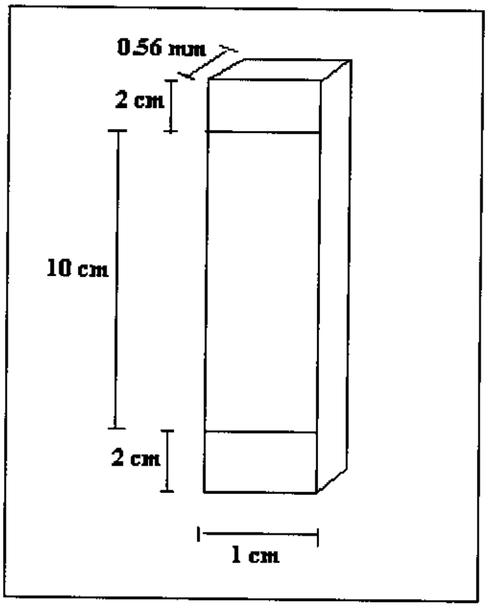


Fig:6.1 Sample dimensions

All the tensile properties reported represent the average value of four readings. The tenacity, elongation, peak load and breaking load of the sample were determined from the stress-strain curves. After the tests, fragments of the failed specimens were collected for fracture analysis.

6.2.4 Morphological Properties

6.2.4.1 Optical Microscopy:

The fracture samples were investigated using a ordinary microscope and the images were captured with the magnification of 50 X. the samples of fractured areas were placed under the lens and the image is caught in a film.

Results

7. Results:

7.1 Starting Process Flow chart:

Initial experiments were done to optimize temperature, time and pressure according to the following flow chart.

The cool mould is taken

⇩

Placing the Glass Fabrics + Polymer matrices placed in the mould

⇩

Mould is placed in the Compressing Machine

⇩

Application of heat without Pressure over the mould

⇩

Increase of Temperature

⇩

Application of pressure for desired time

⇩

Removal of mould from the heater and cooling in the open air

⇩

Extraction of the composite sample from the mould plate.

Table 7.1: Summary of GF-PET composite fabrication conditions(Initial)

Sample ID	Fabric	Matrix #	Temp ^{0}C	Pressure (Tonnes)	Comments
GF 2 PET 25	2 mil	(1+1)25 μ	280	30	D
GF 2 PET 25	2 mil	(1+1)25 μ	240	40	N
GF 2 PET 75	2 mil	(1+1)25 μ	240	40	N
GF 2 PET 75	2 mil	(1+1)25 μ	280	30	D
GF 2 PET 175	2 mil	(1+1)25 μ	260	30	T
GF 2 PET 175	2 mil	(1+1)25 μ	260	15	T
GF 7 PET 25	7 mil	(1+1)175 μ	260	30	T
GF 7 PET 175	7 mil	(1+1)175 μ	260	30	N
GF 7 PET 175	7 mil	(1+1)175 μ	260	30	N

Note: GF- Glass Fabric , PET- Polyester Resin, N- No adhesion, T- Torn samples, D- Degraded Samples.

- The composite stacking technique is given as (Film(s) on top+ Film(s) placed below) Matrix thickness

7.2 Observations:

Table 7.2 summarises the observations made during the experimentation of glass/polyester composites and their effects.The three main parameters affecting quality of the composites were 1. Temperature, 2.Pressure, 3. Curing Time.

7.2.1 Effect of Temperature:

Lower temperature results in poor adhesion due to incomplete melting of matrix. Higher temperature results in matrix degradation.

7.2.2 Effect of Pressure:

During the initial run of the experiment the load applied over the mould was 40 tonnes which results in the excess amount of polymer leakage from the mould during compression. Later the overflowing of the polymer continues even at the pressure level of 30 tonnes over the mould. Molten polymer matrices inside the mould, forces glass fibres to migrate away from their interlacements which results in a poor density sample. So the pressure level was reduced to the level of 20 tonnes to obtain a very clear moulded glass polyester composite without any polymer over flow.

7.2.3 Effect of curing time period:

The curing period of the matrix-fabric compression is also an important factor which affects the quality of the composite during compression moulding. As the curing period was maintained below 15 mins the polymer matrices doesn't have enough time to melt resulting in poor adhesion with the glass fabric,so that the polymer gets peeled out after the moulding process. Also as the time period exceeds 15-17 mins of curing, the polymer subjected to high temperature and pressure was observed to degrade which results in yellowing of the samples. As discussed earlier these three quality parameters have a major effect over the composites during compression moulding.

The effects and the results observed due to the changes made with the quality parameters is shown in the table 7.2:

Table 7.2 : Observation chart

Parameters	Effects	Result
Mould Temp below 250 ^{0} C	No melting of the Polymer Matrix	No sample adhesion
Mould Temp above 280 ^{0} C	Yellowing of the samples	Degraded Sample
Mould Dislocation	Matrix disturbed	Uneven Sample
Application of pressure above 20 tonnes	1.Leakage of molten matrices	Improper matrix protrusion
Curing time above 20 mins	Brittle samples	Sample Breakage
Extraction of samples without proper cooling	Brittle samples	Sample Breakage

In order to obtain a well-defined sample these parameters should be taken into keen monitoring process while experimentation.

Based on the above observations, the time-temperature-Pressure cycle has been optimised in the flow chart as follows:

7.2.4 Optimised Flow Chart:

The Mould is taken at room temperature

⇩

Placement of Glass Fabrics + Polymer matrices in required stacking sequence in the mould

⇩

Placement of Mould in the Hot press

⇩

Application of heat without Pressure over the mould

⇩

Temperature to reach 260 ^{0} C

⇩

Application of load of 15 tonnes for 10-15 mins

⇩

Cooling the mould in the open air until the temperature reaches the desired level

⇩

Extraction of composite sample from the mould plates.

The table below summarises the processing conditions for the optimized temperature, pressure, curing cycle for the composites.

Table 7.3: Sample Nomenclature and Temperature/Pressure conditions for optimised curing cycle.

Sample ID	**Fabric Thickness**	**Matrix # stacking sequence**	**Temp ^{0}C**	**Pressure (Tonnes)**
GF 2 PET 25	2 mil	(1+1)25 µ	260	15
GF 2 PET 75	2 mil	(1+1)25 µ	260	15
GF 2 PET 175	2 mil	(1+1)25 µ	260	15
GF 5 PET 25	5 mil	(1+1)75 µ	260	15
GF 5 PET 75	5 mil	(1+1)75 µ	260	15
GF 5 PET 175	5 mil	(1+1)75 µ	260	15
GF 7 PET 25	7 mil	(1+1)175 µ	260	15
GF 7 PET 75	7 mil	(1+1)175 µ	260	15
GF 7 PET 175	7 mil	(1+1)175 µ	260	15
GF 2 PET 25	2 mil	(3+3)25 µ	260	15
GF 2 PET 25	2 mil	(7+7)25 µ	260	15
GF 5 PET 75	5 mil(1+1)	(1+1+1)75µ	260	15

The composite stacking technique is given as (Film(s) on top+ Film(s) placed below) Matrix thickness in microns.

7.3 Volume Fraction of the Matrix:

Table 7.4: Volume fraction analysis chart.

S. No	**Sample ID**	**Volume fraction of Polyester(%)**
1.	GF 2 PET 25	49
2.	GF 2 PET 75	61
3.	GF 5 PET 25	40
4.	GF 5 PET 75	42
5.	GF 5 PET 175	44
6.	GF 7 PET 25	38
7.	GF 7 PET 75	45
8.	GF 7 PET 175	62
9.	GF 2 PET 25(3+3)	61
10.	GF 2 PET 25(7+7)	68
11.	GF 5(1+1) PET 75	48

The volume fraction of the matrix is a function of number of matrices and thickness of matrices used in the experiment. The following charts explains in detail about the Volume fraction to Matrix thickness ratio.

7.3.1 Analysis of Volume Fraction of the Polymer Matrices:

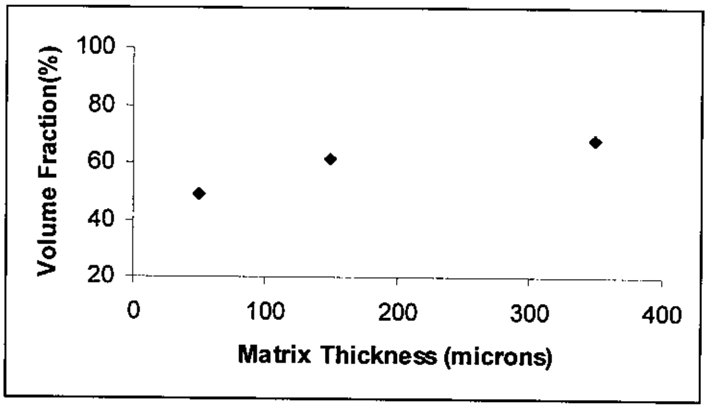


Fig. 7.1 **Volume Fraction vs Fabric Thickness(2 mil)**

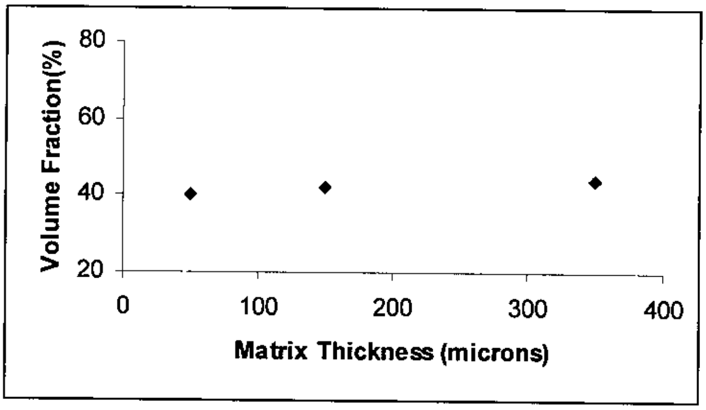


Fig. 7.2 **Volume Fraction vs Fabric Thickness(5 mil)**

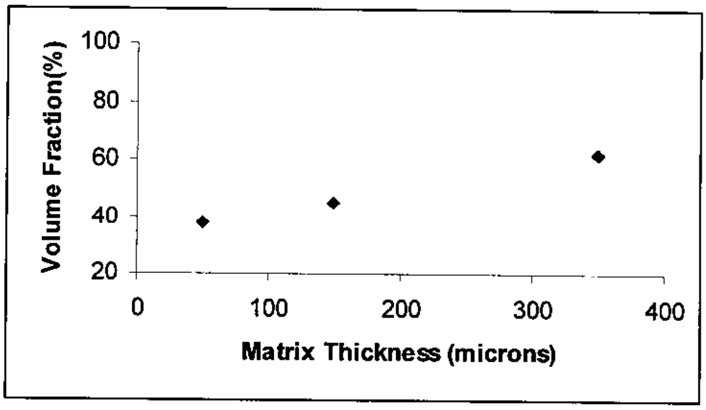


Fig. 7.3 **Volume Fraction vs Fabric Thickness(7 mil)**

It is clearly observed that the thicker matrix layers have resulted in higher V_m of the matrix, under identical curing cycle conditions

7.4 Mechanical Properties:

Table 7.5: Mechanical Properties of Glass/Polyester composites

Sample ID	Elongation (%)	Force (Kg)	Tenacity (cN/tex)	Work of Rupture(kg.cm)
2 mil	0.61	3.27	160.21	1.41
5 mil	1.49	12.03	589.78	2.01
7 mil	2.00	15.8	774.66	2.10
25 µ PET	30.10	2.9	142.29	9.27
GF 2 PET 25	1.50	6.17	302.40	0.70
GF 2 PET 75	1.86	12.36	606.19	1.51
GF 5 PET 25	2.56	27.33	1339.76	3.93
GF 5 PET 75	2.24	20.18	989.00	2.58
GF 5 PET 175	2.29	32.70	1602.75	4.28
GF 7 PET 25	4.08	57.09	2798.75	13.31
GF 7 PET 75	3.67	59.77	2930.07	11.78
GF 7 PET 175	3.47	50.96	2497.82	10.51
GF 2 PET 25(3+3)	2.15	17.95	880.04	2.34
GF 2 PET 25(7+7)	1.43	11.95	585.99	1.07
GF 5(1+1) PET 75	2.97	52.23	2560.21	8.74

All values are average of four tests, according to ASTM standards, With a C.V of (3-4)%.

Composite samples from Table 7.5 show excellent improvement in terms of tenacity & work of rupture. Highest work of rupture of 13.31 Kg.cm is obtained which is higher than the parent material.

7.5 Work of Rupture as a function of Matrix Thickness:

From Fig 7.4 it is observed that, as the matrix thickness increases the work of rupture of the composite also increases. As the thickness of the material increases due to higher volume fraction of the matrix, the work to rupture those materials have to be intensified due to the composite structure formed.

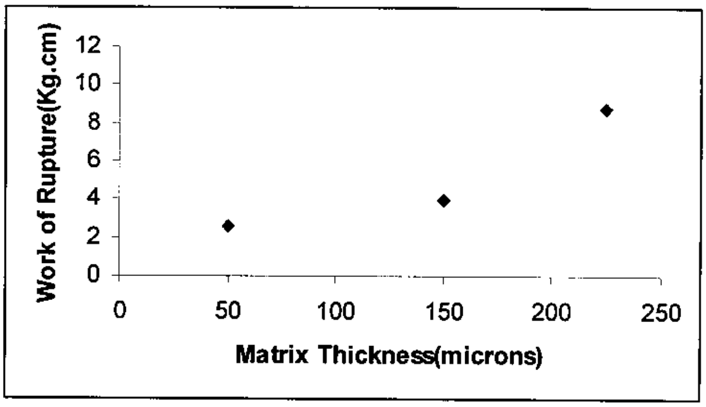


Fig: 7.4 Work of RuptureVs Matrix Thickness(2 mil)

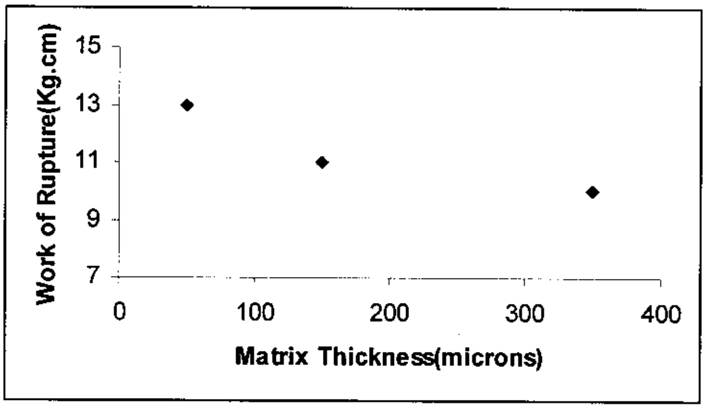


Fig:7.5 Work of RuptureVs Matrix Thickness(7 mil)

For 7 mil (Fig 7.5) the trend is just the opposite. Here increasing Volume Fraction of matrix results in lower elongation. The reason is more rigid samples. Higher matrix thickness results in more adhesion at yarn cross-over points resulting in a stiffer fabric with lower worrk of rupture.

7.6 Breaking load as a function of Matrix thickness:

As evident from Fig. 7.6 & 7.7, as the thickness of the matrices increases the breaking load obtained for the respective matrices was found to be higher.

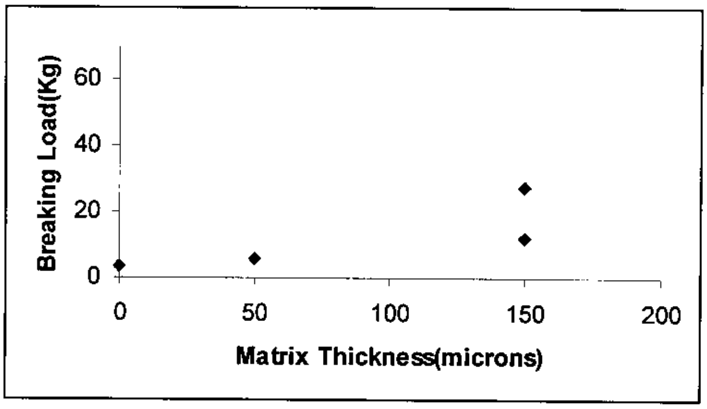


Fig:7.6 Breaking load Vs Matrix thickness(2mil)

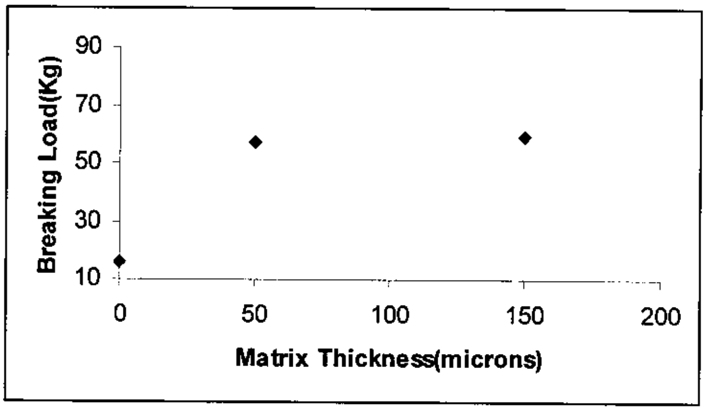


Fig:7.7 Breaking load Vs Matrix thickness(7mil)

7.7 Breaking Load as a function of No. of Matrices:

Fig: 7.8 clearly shows that the breaking load increases as the number of matrices increases.

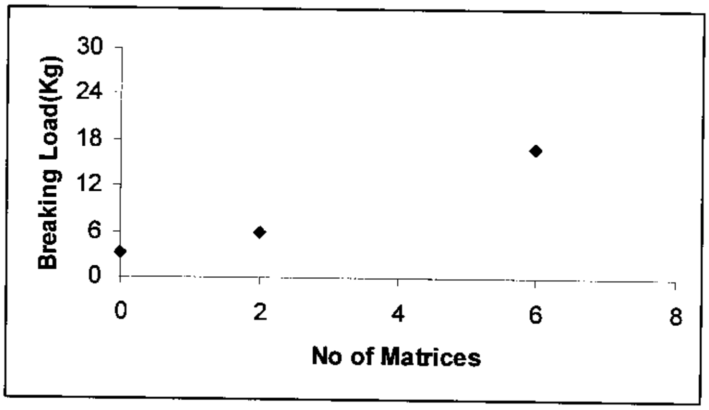


Fig: 7.8 Breaking Load Vs No. of Matrices(2 mil)

7.8 Breaking Load as a function of Fabric Thickness:

For the same matrix stacking sequence, increase in fabric thickness improves the breaking load shown in Fig 7.9-7.10.

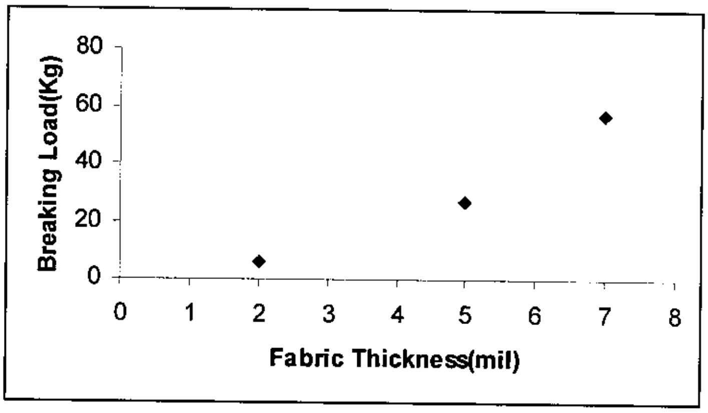


Fig:7.9 Breaking Load vs Fabric Thickness(25 microns)

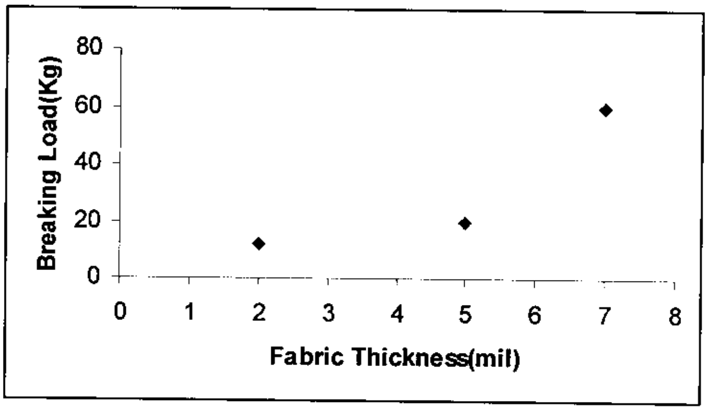


Fig:7.10 Breaking Load vs Fabric Thickness(75 microns)

7.9 Work of Rupture as a function of Fabric Thickness:

Similar trend is observed for the work of rupture shown in Fig 7.11-7.12.

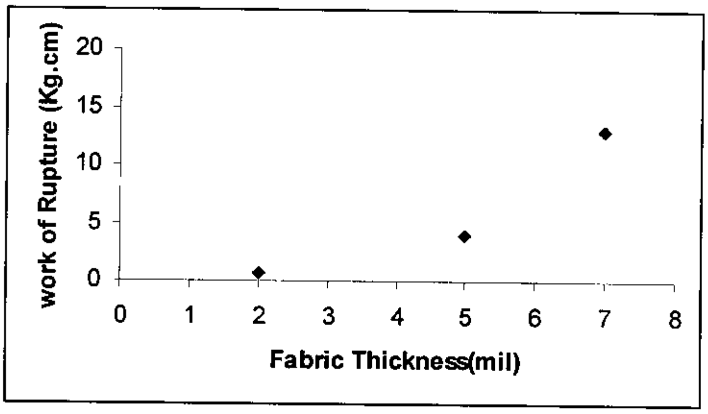


Fig:7.11 Work of Rupture vs Fabric Thickness (25 microns)

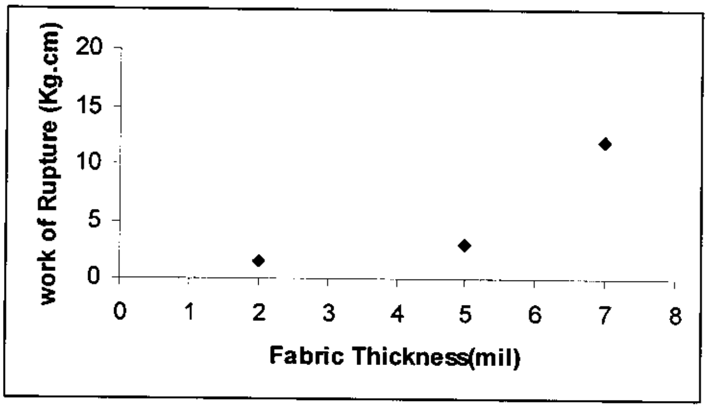


Fig:7.12 Work of Rupture vs Fabric Thickness (75 microns)

7.10 .Study of Fractured Surfaces:(Morphological Analysis):

The fractured samples obtained after the mechanical testing were subjected to morphological analysis with the help of an optical microscope. Images were observed at 50x magnification. Fractured samples indicated low fiber pull out and existence of bridging fibres across the fractured specimens. Overall they indicate fairly good amount of fiber- matrix adhesion.

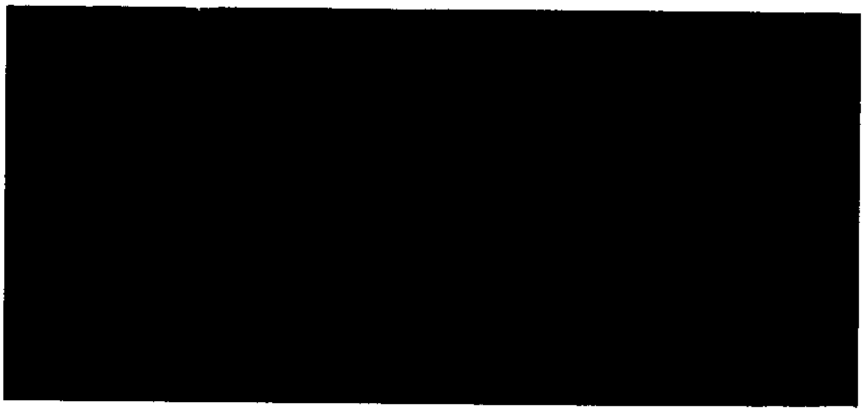


Fig: 7.13. Matrix binding the fibers well inside the layer, Fibers broken out but not pulled out indicating excellent adhesion.

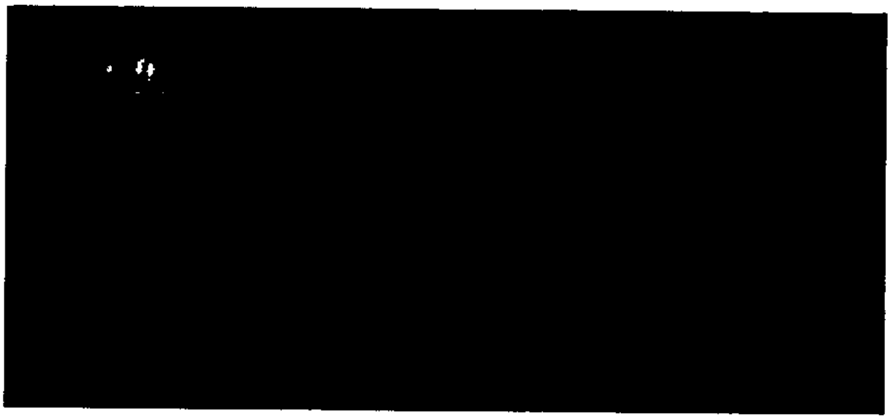


Fig 7.14: Ductile fiber break indicating good adhesion.

8. Conclusions:

Glass Polyester Composites have been successfully manufactured using the film stacking procedure and the curing cycle optimised for the GF/PET combination.

Significant improvement in tems of mechanical properties have been obtained. The composites prepared had high load bearing capacity of ~55kg and high work of rupture of 13 kg-cm. The work of rupture increased by 550% for the lowest sample and elongation by 100% and the load bearing capacity by 225% when compared to the virgin glass fabric. The work of rupture and breaking load were found to be a direct function of fabric thickness with the same stacking sequence. Reasonably high matrix Volume fraction were achieved as confirmed from the matrix digestion test. Optical Microscopy show minimum fibre pull-out, existence of bridging fibres indicating good adhesion between the fiber and the matrix.

Increased number of matrix layers improve the work of rupture when compared to equivalent single layer of matrix reinforcement. This is a very interesting finding and properties of the composites can be further investigated through subsequent study in terms of improvement of composite properties with multiple matrices and varying stacking sequence.

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