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Characterization of woven fabric composites

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Abstract

Composites have gained so much attention in most kinds of industries. They are greatly appreciated for their low weight and density compared to metals. These materials have not been fully adopted due to lack of knowledge of their capabilities. As a result interest in studying their properties is everywhere. The major problem with these materials is that as much as they have appreciable properties, these properties cannot be entirely predicted. Many conditions during their production affect the final properties. These production conditions may include choosing the appropriate matrix, curing temperature and ensuring even distribution of the matrix throughout the reinforcement.

Within the composites themselves, woven fabric composites have gained more interest due to their ease to handle during production as compared to their unidirectional composite counterparts. Woven fabrics have the advantage of having the yarns interlaced and tight, thus making them easy to handle. One most important thing though is to ensure that an appropriate resin is chosen for any particular reinforcement.

To enhance their properties, filler materials which contribute their effect to a micro and/or Nano scale are also being greatly investigated, so as to find out the properties they enhance and those they depreciate. Fillers enhance in increasing the volume of the matrix, since some of them like fly ash are far cheaper than polymers. Their addition is however crucial since their reaction with the matrix may also lead to undesired results.

In this study, interest has been given to effects caused by fly ash particles to some properties of woven glass/epoxy composites. The properties studied include; flexural strength, stab resistance, electrical resistance, dynamic mechanical analysis, EMI shielding efficiency and UV-transmission.

Results did show that whilst the addition of filler improves some properties, some properties are depreciated. As a result, the amount of filler and the type of filler to be added entirely depends on the final application of the composite. This means that before any composite material is produced, its intended application must be well understood so as to know which properties are of importance and which can be compromised.

Fly ash is one of the cheap filler materials because it is readily produced by burning coal in thermal industries. It has very low density with some elements that make them enhance performance of composites.

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1. Introduction

Composite materials provide a more interesting performance to weight ratio and are finding their way to more structural applications which were previously occupied by metal alloys. When compared to metals, composite materials can be made by relatively low tooling cost. Composites can be fiber reinforced or fabric reinforced. Fiberreinforced composites have good mechanical properties such as high specific stiffness and high specific strength. Specifically, laminated composite plates have been used extensively where the in plane properties are important. These laminated composites, however have poor mechanical properties in the thickness direction and have a tendency to show inter-laminar delamination. [1] In trying to overcome such difficulty in fiberreinforced composites, woven-fabric composites were developed to provide tridirectional reinforcement in a single layer.

Woven fabric composites represent a class of textile composites in which two or more yarn systems are interlaced in a regular pattern. These composites provide more balanced properties in the fabric plane and higher impact resistance than unidirectional composites. The interlacing yarn provides an out-of-plane strength which can take up the secondary loads due to load-path eccentricities, local bulking, etc. [2] The handling of woven fabrics is easier, so this makes the fabrication of woven fabric composites less laborious. Woven fabrics offer many advantages in terms of manipulative requirements including dimensional stability, good conformability and deep draw shape ability. Compared to unwoven unidirectional composites, the woven fabric composites provide more balanced properties, higher impact resistance, easier handling and lower fabrication cost, particularly for parts with complex shapes. [3]

Also, fabrics have the following advantages over other kinds of reinforcement; they offer uniformity of thickness and weight, consistency, improved tensile strength and modulus since more fibers can be contained within a given volume because of the precise arrangement of yarns, they are easy to lay-up and they have improved compatibility with resins because of a wide range of fabric finishes. This is because it is easier to apply finishes on fabrics since they are easy to handle. To overcome the problems associated with the production and mechanical behaviors of other kinds of composites, various methods have been used. These include the application of tough resin, interleafing, and chemical or plasma treatment of fibers in order to improve their adhesion strength with the resin. These methods, however, are superseded by the textile production of composites. This is probably due to the ability to produce large volumes of textile preforms in a short time thus reducing the manufacturing cost and the cycle times. There are currently a number of ways used to produce 3 dimensional textiles. They can be produced through stitching, weaving, knitting, braiding and non-woven structures. Woven structures are, however the mostly produced due to ease of production and diversity of different 3-dimensional structures to produce. A very novel and unconventional technique popularly known as 3-Dimentional (3D) weaving enabled researchers to create reinforcement material with industrial yarns like glass, aramids etc. in a 3 mutually perpendicular disposition, so as to develop a truly 3D fabric with fiber orientation along the thickness direction (Z direction) apart from the axial directions (X & Y directions) in a conventional 2D fabric. These are developed on specially designed prototype machines. Such woven structures completely eliminate the chances of delamination failure, which is commonly observed in conventional fiber/fabric reinforced composite materials.

With the advent of nano science and technology, researchers have found many applications where the advantages of very high surface area can be exploited to improve the functional performance properties substantially. Incorporation of Nano powder/nanoparticles in a composite material system is an added advantage to the enhancement of mechanical as well as functional characteristics. The characterization of Nano composites was done using Dynamic Mechanical Analysis (DMA), Thermal Mechanical Analysis (TMA), Impact testing , Tensile testing, Thermal conductivity and electrical conductivity measurements. The morphology of composites was characterized by Scanning Electron Microscopy (SEM). Experimental results show that some functional properties can be enhanced by the incorporation of nanoparticles.

The research work was carried out with samples developed on state of the art machines. The composites involving suitable resins and varieties of Nano particles have been evaluated for their mechanical and functional characteristics. The results ate encouraging and promising.

2. Literature Review

2.1 Composites

Technical textiles are currently mostly used in human life. This is due to their appreciated performance. Technical textiles are semi-finished or finished textiles and textile products manufactured mainly for their technical performance and functional properties rather than their decorative characteristics. Technical textile composites are generally growing in developing lightweight materials. Fabrics form the reinforcing component of a composite material. Fabrics used in the production of a composite material are called the preforms.

Textile structural composites have gained so much attention in various industries like civil and defense. This is due to their availability and cheaper production compared to classical materials like metals and ceramics. Textile preforms can be fabricated in different methods such as; weaving, knitting, braiding, stitching and nonwoven techniques. The fabrication used depends mostly on the end-use of the product.

Reinforcing fibers can be classified based on their structure. These classes are as follows; discrete fibers, continuous fibers, interlaced 2-dimensional structures, and finally the 3-dimensional structure.

Discrete fibers are short fibers which have no continuity. These fibers are randomly distributed. This is because these fibers are so short that it's hard to control their orientation. The integrity of the fiber preform comes from inter-fiber friction.

In the continuous fiber system, the fibers are laid in a uni-direction. Unlike in the discrete system where fiber's direction cannot be controlled, in this case fibers are lined facing the same direction. This structure has high level of continuity and linearity. This structure, however has the problem of inter-laminar and intra laminar weakness due to lack of in-plane and out of plane yarn interlacing.

The next category of fiber reinforcement is the interlaced 2-dimensional structure. Due to the interlacing, the intra-lamina failure problem experienced in the unidirectional fibers is solved.

The inter-lamina strength is, however, still limited by the matrix due to lack of out of plain reinforcement. 2D woven fabrics are the mostly used woven fabrics. These are formed by two sets of yarns, called the warp (which is at 0°) and fill yarns also called weft (at 90°). These two sets of yarns are interlaced to each other to form the surface. The production process of these fabrics can differ leading to different types of products namely, plain weave, satin weave and twill weave. These fabrics, however, has some short comings like; poor resistance to impact because of crimp, low in-plane shear properties because there is no off-axis fiber orientation and low delamination strength because of lack of binder fibers.[7]

The last classification, which is the 3 dimensional fabrics, has fibers oriented in in-plane and out of plane directions. The most outstanding feature for this structure is the through thickness reinforcement which eliminates delamination.

Three dimensional woven fabrics can be further divided into 3D orthogonal, 3D angle interlock and 3D multilayered fabrics. This difference is based on the positioning of warp yarns, weft yarns and the z-yarns. In the 3D orthogonal fabric, all the three yarns are interlaced to form the structure. The warp yarns are longitudinal whereas the others are orthogonal. The weft (filling) yarns are inserted between the warp layers leading to the formation of double picks. The z-yarns on the other hand are used to bind the other sets of yarns providing a structural integrity. [7] This structure provides better volume fraction, especially on the thickness direction.

The angle interlock fabrics are considered as layer-to-layer and through the thickness fabrics. According to Kadir, [7], the layer-to layer fabric has four sets of yarns as fillings, \pm bias and warp yarns. The \pm bias yarns are oriented at thickness direction and are interlaced with several filling yarns. The bias yarns make a zig-zag movement in the thickness direction of the structure. The through-the- thickness fabric also has four sets of fibers; \pm bias, warp and filling yarns. \pm Bias yarns are still oriented in the thickness direction and each bias yarn is oriented until it comes on top or bottom face of the structure. Each bias yarn is moved towards the top or bottom of the structure until it reaches the edge. These yarns are locked by several filling yarns depending on the number of layers. Compared to the other structures, this structure shows better pliability

and distortion capability, thus suitable for producing components with complex configuration.

The other type of 3D woven fabric is the multilayered woven fabric. In this type of weaving, the \pm bias yarns are inserted between the diagonal rows and columns of opening warp layers at a cross section of the woven structure. The process of producing such structure includes; \pm bias insertion needle assembly, warp layer assembly and hook holder assembly. The warp yarns are arranged in matrix array according to structural cross-section, a pair of multiple latch needle insertion system inserts \pm bias yarns at cross-section of the structure at angles about 60°. Loop holder fingers secure a bias loop for the next bias insertion and passes to the previous loop. [7]

2.2. 3-D weaving process

In order to ensure high quality products and faster production rates, producers of 3-D fabrics uses highly automated, computer controlled looms. The weaving process in the looms can be separated into the following production operations:

- Warp beam winding
- Warp let off
- Warp tensioning
- Shedding
- Weft insertion
- Beating and,
- Take up of finished fabric

The following schematic diagram shows how these operations are positioned in respect of one another.



Figure 1: schematic illustration of operation steps in Jacquard loom. [17]

a) Warp beam winding: This is a step before weaving. In this step, warp yarns are wind on the warp beams of the looms. The warp beams may vary depending on the looms being used. These beams hold the source of the warp yarns for weaving.

b) Warp let off: This is the process when weaving starts and warp yarns are pulled off the warp beams and moves towards the tensioning devices.

c) Warp tensioning: The purpose of this operation is to remove all slack from the warp yarns in preparation of the following step. This is achieved by letting the warp yarns pass through a series of tensioning devices which have guides that are loaded with weights hanging on to them.

d) Shedding: in this stage, the warp yarns pass through the eyes of heddles where the yarns are lifted and lowered in order to create a narrow gap through which the weft yarns will be inserted. Separate heddles are used to create similar gaps for the z-yarns.

e) Weft insertion: The weft yarns are pulled by a rapier through the gaps opened in between the warp yarns. Weft yarns are inserted at 90° to the warp yarns whilst the z-yarns are inserted in the trough the thickness direction, thus form a 3-D structure.

f) Beating: This is where weft yarns are packed tightly on the fabric by the reed(a combing device)

g) Fabric take up: When weft yarns are tightly packed to the fabric, a take up mechanism pulls out the fabric from the loom. At this stage, the 3-D fabric is complete and is ready for the production of composites. [17]

2-D fabrics used for composite laminates usually have 50% fiber in the warp direction and 50% in the weft direction. When this structure is combined with a suitable matrix, the result is a high strength composite structure. This, however, shows weakness under shear loads and has low through thickness strength. The 3-D fabrics on the other hand, have higher percentages of fiber in the warp and weft directions, and a lesser percentage in the thickness direction. The fibers in the thickness direction when combined with a matrix increase the inter-laminar shear strength of the composite lamina.

2.3 Reinforcement

The selection of the material to be used for reinforcement is based on four major principal criteria; weight, strength, stiffness and cost. For primary and secondary load bearing, high modulus fibers and yarns must be used. The mechanical properties of the woven fabric composites depend greatly on the yarn geometry. Also, the fiber chosen for the production of the fabric must meet the property requirements required for high performance composites. These properties include; high tensile strength, low density and good impact resistance. The most commonly used fibers for the production of woven fabric reinforcements are; fiber glass, carbon, basalt and aramid fibers. These are high modulus materials although they are usually stiffer and brittle than traditional materials. The latter can be corrected through modifications during textile processing.[8]

2.3.1 Carbon fibers

Carbon fibers are fibers containing at least 90% of carbon. These are produced by heat treatment of organic precursors such as nylon, polyacrylonitrile and pitch. The carbon atoms are bonded together in microscopic crystals which are aligned parallel to the long axis of the fiber. This crystal alignment makes this fiber strong for its size. Several thousand carbon fibers can be twisted together to form a yarn which can be used on its own or be woven into a fabric. These fibers have highest modulus and strength at both room and high temperatures. Carbon fibers have excellent tensile strength, heat resistance, electrical conductivity and chemical resistance. Due to its strength, it is put in the core of polyester resin mold as reinforcement. It is also used for heat insulation in high heated parts, filter material for high corrosive gases and liquids. The major problem to the usage of carbon is that it is still expensive thus only used for aviation machines, space rockets and atomic devises for golf, and fishing rods.

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According to Hegde. R et.al, [10] carbon fibers can be classified into groups based on the modulus, strength, precursor and final heat treatment.

Based on properties, they can be classified into;

- Ultra high modulus, type UHM (modulus>450Gpa)
- High modulus, type HM (modulus between 350-450Gpa)
- Intermediate modulus, type IM (modulus between 200-350Gpa)
- Low modulus and high-tensile strength, type HT

(modulus<100Gpa,tensile strength > 3.0Gpa)

• Super high tensile, type SHT (tensile strength>4.5Gpa)

Based on precursor fiber material, they can be classified into;

- PAN based carbon fibers
- Pitch based carbon fibers
- Isotropic pitch based carbon fibers
- Rayon based carbon fibers
- Gas-phase-grown carbon fibers
- Mesophase-pitch based carbon fibers

Based on final heat treatment temperature, they can be classified into;

• Type I, high heat treatment carbon fibers (HTT), final heat treatment should be above 2000°C and can be associated with high modulus type fibers

• Type II, Intermediate heat treatment carbon fibers (IHT), final heat treatment should be around or above 1500°C and can be associated with high strength type fibers.

• Type III, low heat treatment carbon fibers, where final heat treatment is not above 1000°C. These are low modulus and low strength fibers. [10]

2.3.2. Aramid fibers

These are organic aromatic fibers. The widely known of these is the Kevlar fiber produced by Dupont. There are various types of Kevlar fibers with slightly different properties. Kevlar 29 is used for high toughness, good damage tolerance and crash protection. Kevlar 149 has ultra-high modulus. There are other aramid fibers in the market like Twaron and Nomex. Aramid fibers in general are especially suitable for applications that require high tensile strength to weight such as missiles, pressure vessels and tension systems.

2.3.3. Basalt fibers

Basalt fiber is a material made of extremely fine fibers of basalt, which is composed of the following minerals; plagioclase, pyroxene, and olivine. This is an igneous rock which comes about as a result of volcanic eruption. Basalt fiber is produced in a continuous process similar to that used to make glass. The process is simpler than that of production of glass because basalt has a less complex composition. Basalt and glass are both silicates, but molten glass when cooled form a non-crystalline solid, whereas basalt, has a crystalline structure that varies based on the specific conditions during the lava flow at each geographical location. Ross, states that researchers say basalt fibers outperforms the E-glass fibers. Basalt fibers exhibit a higher breaking load and higher Young's modulus in chopped mat, roving and unidirectional fabric forms than E-glass.

Basalt fibers are naturally resistant to ultraviolet (UV) and high energy electromagnetic radiation, maintain their properties in cold temperatures and provide better acid resistance. Compared to glass fibers, basalt is also superior in terms of worker safety and air quality. Basalt fibers generally offers properties which are similar to S-glass fiber yet its cost is between that of E-glass and S-glass, and is also generally cheaper than carbon fibers. [11]

2.3.4. Glass fibers

Glass fibers are relatively cheaper yet they have good performance. Due to that, these fibers are mostly used in textile composites. Single filaments of glass fibers are produced by mechanically drawing molten glass streams. These filaments are then gathered into bundles called rovings. The strands maybe used in continuous form for filament winding; chopped into short lengths for incorporation into molding compounds; or woven into fabrics after which it can be coated with a coupling agent which enhances its adhesion to the matrix to be used. Glass reinforcing fibers are generally classified according to their unique properties. So far, they are five classes of the glass fibers, and each class differs from the rest by it specific property. These are; 1) A-glass, which is a high-alkali glass that contain 25% of soda and lime. This makes this glass to be highly resistant to chemicals but lower electrical properties; 2) C-glass, which is highly resistant to chemicals; 3) E-glass, which is a good electrical insulator and is highly resistant to water; S-glass, which a high strength glass. It has 33% higher tensile stress than E-glass; and lastly is the D-glass, which has lower dielectric constant with superior electric properties. Its mechanical properties are, however, not as good as the E- and S-glass.[9] Glass has good tensile strength, especially if it is made thinner. It does not absorb moisture thus its tenacity does not change due to moisture absorption. It is also resistant to acid and alkali and shows great electrical insulation properties.

Amongst the different kinds of glass, the E-glass and the S-glass are the mostly used in textile composites. E-glass is used in electronic boards because of its good electrical properties, dimensional stability, moisture resistant and lower cost.

The S-glass has higher tensile strength, higher elastic modulus and better thermal stability, but it is also more expensive. It is therefore used in advanced composites where the cost can be justified by the demand of the performance needed.[8]

2.4 The matrix

The matrix has various purposes in a composite. They bind fibrous materials together; hold them in particular positions and orientations giving the composite structural integrity. They also protect the fibers from environmental effects and handling. The matrix also strengthens the composite structure. The fibers and matrix must be compatible for all of these functions to be possible and for good bonding and penetration.

2.4.1. Properties of a desired matrix

The desired properties of a matrix are as follows:

- Low coefficient of thermal expansion
- Low shrinkage during curing
- Must be able to transfer load between fibers thus they must be elastic
- Must maintain shape
- Reduced moisture absorption from the environment
- Must possess good flow characteristics so that they can be applied evenly on to the matrix, penetrates it evenly and leave no voids during curing
- Depending on its application, it must be resistant to chemical damage
- Should be easily processable to required composite shape

Pandey, 2004, [12] classified matrices as follows;

- Polymer matrix, which can further be split into thermosets and thermoplastics.
- Metal matrix materials
- Ceramic matrix materials
- Carbon and graphic matrix material

2.4.2. Polymer matrices

Polymers are ideal materials for matrices and are widely used because the can be processed easily. They also have light weight which is highly appreciated in composites, and they possess good mechanical properties. They are two kinds of polymers used for reinforcements and these are; thermosets and thermoplastics.

Thermosets are materials that cannot be softened on heating. This polymer material cures irreversibly. The polymer chains in thermosets are joined by intermolecular bonding. They decompose instead of melting on hardening. Changing the basic composition of the resin alters the conditions suitable for curing and its other characteristics. Thermosets can be retained in a partially cured condition over a prolonged period of time. This makes them to be flexible.[12] Thermosets are the most popular of the fiber composite matrices. These include; epoxy, phenolic polyamide resin and polyester. Usually, these are produced by direct polymerization followed by rearrangement reactions to form heterocyclic entities. The voids which are a result of the production of water during the process have negative effects on the properties of the composites in terms of strength and dielectric properties.

The most important classes of thermosets are the polyesters and the epoxies. Epoxy resins are mostly used in filament wound composites and are suitable for molding prepress. They are stable to chemical attacks and are excellent adherents having slow shrinkage during curing.

They also have no emission of volatile gases during curing. They are cured by means of curing agents who may be called catalysts, hardener or an activator. Their use is, however, somehow expensive and they cannot be used beyond a temperature of 140°C.

Polyester resins, on the other hand are easily accessible and cheap thus they find their use in a variety of places. Liquid polyesters can be stored at room temperature for months and even years. The addition of a catalyst can cure the matrix material within a short time. The cured polyester can be rigid or flexible. They are stable against chemicals and can be used up to 70°C or higher. They are used in automobiles and structural applications.

A thermoplastic is a polymer material that turns to a liquid when heated and hardens when cooled. When sufficiently cooled, this material freezes to a glassy state subject to fracture. These characteristics of thermoplastics are reversible, meaning that it can be reheated, reshaped and frozen repeatedly. Examples of thermoplastics include; polyethylene, polystyrene, polyamides and polypropylene. The advantage of thermoplastics over thermosets is that there are no chemical reactions involved that may lead to the release of gases. All thermoplastic composites tend to lose their strength at high temperatures, but the addition of fillers may increase their heat resistance. Their outstanding qualities like, rigidity, toughness and ability to repudiate creep, places them in the important composite material's bracket. Thermoplastic resins are sold as molding compounds.

2.4.3. Metal matrix materials

These are not widely used like their plastic counterparts though they have gained much interest in research. They exhibit high strength, fracture toughness and stiffness. They can withstand elevated temperatures in corrosive environments compared to polymer composites.

Most metals and alloys can be used as matrices but they require reinforcement materials that are stable over a range of temperatures and which are also non-reactive.

2.4.4. Ceramic matrix materials

These are solid materials which exhibit ionic bonding in general but may have covalent bonding. They possess high melting point, good corrosion resistance, high compressive strength, and stability in elevated temperatures. These properties make ceramic matrices favorable for applications that require structural materials that withstand temperatures above 1500°C.

2.2.5. Carbon matrices

Carbon and graphite possess a special space in composites in that both are high temperature materials with strength and rigidity that are not affected by temperature up to 2300°C. Carbon-carbon composites have shown ability to retain their properties at room temperature as well as at temperatures in the range of 2400°C. This together with their dimensional stability makes them an oblivious choice in many applications related to military, industry and space.

2.4.6. Glass matrices

Glass matrices are in general reinforcement friendly. It is possible to obtain glass matrix composites with high strength and modulus and these can be maintained up to temperatures around 650°C.

These composites are considered to be superior in dimensions compared to polymer matrices and their low thermal expansion behavior. [12]

2.5 Filler material-Fly ash

Nano science and Nano technology has gained more recognition in recent years. This is because it impacts enhanced electronic, electrical, magnetic, and chemical properties to a level that cannot be achieved by conventional materials. A number of ways to synthesize Nano materials have been reported and some more methods are being studied by various companies. These methods may include plasma arcing, electro deposition, sol-gel synthesis, chemical vapor deposition and high intensity ball milling.[22] Amongst these methods, high energy milling is mostly used due to its simplicity, inexpensive to produce, applicable to most materials and easier to scale up to larger productions. In this type of production, the powder particles of the filler are exposed to plastic deformation due to repetitive compression loads arising from the balls and the powder.

Filler materials are generally inert materials which are used in composites to reduce material cost, improve mechanical properties and may improve process ability. Fly is one filler material that has low density and is mostly appreciated because it is inexpensive. Fly ash is one of the materials generated during combustion and it comprises of small particles that rises with the flue gases. In thermal power plants, fly ash is accumulated by the precipitation of the flue gases. During combustion in a coal thermal power plant, ash is carried forward in form of flue gases as fused particles. The components of fly ash vary depending on the source of the coal being burnt. But generally, all fly ash contains variable amounts of Silicon dioxide and calcium oxide.

2.6 Reinforcement-Matrix interface

The reinforcement-matrix interface plays a great role in the properties of the composite. A composite cannot be made from components that have divergent linear expansion characteristics. This means that it must be possible to combine the reinforcement and matrix for the composite to exist. The region where these two components meet is called an interface. This is a Nano-size region and its characteristics differ from those of both the matrix and the reinforcement. But its formation depends on the surface structure and properties of both phases.

2.7 Resin application

Various types of reinforcing fibers have been discussed above together with different matrices that can be used. Even with such a wide choice, it is still important to choose the right reinforcing fiber and correct matrix to suit a particular application. This means that before a particular composite is manufactured, its intended use must be well known, and expected performance requirements drawn. There is no easy rule for such selection but, apparently, the availability of materials and easy manufacturing procedures tends to be used. Reinforcement on its own only possesses high tensile strength with poor stiffness and compression performances. Due to that, matrices are needed to envelope the reinforcement thus protecting them from damage and overcoming some of their weaknesses.

The matrix applied binds the reinforcement and help transfer any stresses applied to the composite from one reinforcing fiber through the matrix to the next fiber. When polymeric matrices are used, the product is a composite structure with low density and good performance like high strength/weight and high stiffness/weight ratios. The distribution of the matrix on the fiber surface is of great importance. Also, variations of the fiber volume along the whole composite are important. These two factors may lead to variations in the final product properties.

To achieve uniform performance distribution and effective load transfer, the matrix must completely fill the interstices and all the spaces between the reinforcement and the reinforcing fibers must be completely wet. To make this possible, different ways are used to apply the matrix on the reinforcement. [13]

2.7.1. Hand impregnation

In this application, the resin is mixed by hand and applied by hand brush onto the reinforcement. To ensure even distribution, a hand driven roller is used. This is done in open space so all chemical reaction and crosslinking occurs in the presence of air. This process requires lot of labor and there are health risks involved due to any hazardous gases that may be released during this process. It is not possible to control the process since the atmosphere cannot be controlled so this leads to variations in the properties of the product. It is even harder to exclude all the air trapped in between the layers of reinforcing fibers since pressing is done by hand only. Due to lack of control of various parameters in this system, it is hard to achieve high volume ratios of the fibers thus it is difficult to obtain high performance composites. This method of application was mostly used in the early years of composite development when composites were mostly used for leisure goods.

2.7.2. Matched-die molding

As composites gained more commercial use, more automatic ways of application had to be developed in order to get a uniform matrix distribution throughout the reinforcement. The matched-die moulding is one of those developments. In this system, reinforcement is placed in closed matched cavity tools. Resin is first mixed with hardener and the mixture is then injected to the reinforcement under pressure from a pressure pot.

As the injection proceeds, the resin spreads out into the cavity, wetting the reinforcement inside until the whole cavity is filled with resin. It is however very important to understand the flowing patterns of the resin in the cavity to avoid having spaces which are left not wetted by the resin applied.

To avoid patches of dry reinforcement, high pressures are used. This high pressure, however leads to the compression of enclosed air thus leading to increase of temperature of the air. The increase in temperature can have positive and negative impacts in the process. Positively, it will accelerate the cross linking process but when these temperatures are too high, they may lead to thermal degradation of the matrix.

2.7.3. Pre-impregnation

In the process of trying to achieve uniform distribution of the resin and fiber, and less empty spaces, this system is used. This uses dip coating of the reinforcement in uncured resin and then using lick rolls to control and evenly distribute the resin on the fibers. The resin bath contains both the base resin and hardeners. The rolls of prepreg can be wrapped with film and be stored for quite some time before they can be cured to a complete product. These, however require being stored in low temperatures for instance -18°C for aerospace prepregs. This process ensures that the resin is uniformly distributed in the reinforcement. [13]

2.7.4. Autoclave

Another most important aspect of obtaining high performance composite is the achieving of high fiber volume ratio with even distribution of both fibers and matrix throughout the structure. To achieve this, an autoclave system is used. This system requires the use of a clean room and protective clothing so as to ensure that the composite is not contaminated as well as to ensure health and safety. The steps in the manufacturing process are as follows;

1. Prepreg is stored in refrigerator, preferably at -18°C. It is then taken out of the refrigerator and thawed. When it is at room temperature, the fabric is cut into required shape plies. These are labeled.

2. After cutting, the plies are hand laid in thoroughly degreased molding tools. When lay-up is done, a release film is placed on top of them and a breather cloth on top. The plies are then sealed.

3. After they are sealed and still under vacuum, they are placed under a computercontrolled autoclave, which is programmed to follow particular processing of both temperature and pressure. The system is designed in such a way that it ensures that all fibers are completely wetted and all interstices are filled with resin. Crosslinking of the resin then takes place slowly to ensure that the exothermic reactions are controlled.

4. When the autoclave process cycle has been finished, the component is cooled slowly and excess resin at the edges is trimmed off.

5. When all that is done, the components are assembled to form the final product.

Most kinds of fabric can be placed directly in the tool thus labor is reduced. As much as this processing does provide high performance composites, it is quite costly to run it.

2.7.5. Liquid molding with vacuum assistance

This process is used to produce high fiber volume composites with low voids. It uses either ply lay-up or more complicated 3-D structures. This process tries to overcome some shortcomings of the autoclave process. These autoclave short comings may include; high cost of operation and time consumption by the ply-cut outs, to name a few. The liquid molding process on its own can have some limitations but the presence of the vacuum assistance overcomes them. This enables the production of high performance composites by applying degassed resin onto reinforcement.

2.7.6. Resin film infusion

This method of composite manufacturing is rapid and can be used to produce more composite component in a short space of time. In this method, layers of resin are laid into the ply assembly, in film form. Heat, pressure or vacuum are applied to the resin film, causing it to be less viscous thus flowing into the reinforcement. This less viscous resin, as it flows into the reinforcement; it fills all the spaces between the filaments and all interstices within the reinforcement structure. Complicated 3-D reinforcement can also be used, and in such cases, the resin film is placed on both the top and bottom part of the complicated structure and the resin is drawn by pressure through the structure when heated.

2.7.7. Pultrusion

This method is amongst those that offer low-cost production of composites. A 3-D reinforcement structure is pulled through a heated die. As this reinforcement structure moves slowly along the die, heated polymer resin is forced into the structure. The impregnated structure is then passed through an oven or another second die for curing.

All these methods are ways of applying resin onto the reinforcement thus leading to the production of a composite material. A producer may choose any of these methods based on the availability of production tools and costs. Some methods are favoured over others

due to low operation costs or rapid production of required products with appreciable and expected functional properties. [13]

2.8 The forming process of composites

The forming process can also be an issue. According to Zhu et al, the fast and costeffective forming process is the stamping process. Compared to other methods of forming complex structures, this process can reduce the cycle time by several times. In this process, flat sheets are stamped into a particular shape with a pair of punch and die at elevated temperatures. This method, however, also has some failure mechanisms like wrinkling. In trying to find out the basis of wrinkling, experiments were done where the shear angle was measured. It was observed that the shear distribution in the preform was a combined result of both the mold shape and initial sample orientation. [5]

According to Boisse et al, forming of a composite material exploits the relative movement of fibers made possible by the absence of cohesion of the matrix. These deformations by textile reinforcements may lead to wrinkling, depending on their occurrence. Woven textile reinforcements are efficient in the case of double curve geometries because of the interlacing of weft and warp yarns. Such geometries are hard to obtain from unidirectional reinforcement. In the woven fabric reinforcement, there are two directions of yarns which are interlaced and this enables the fabric to reach very large in-plane shear strain and tensile strength. Also, the angles between the warp and weft yarns influence the permeability of the reinforcement and this in turn influence the filling of the resin in case of a liquid molding process.[6]

As far as fabric forming simulations are concerned, a number of codes have been developed based on geometrical approaches called fish-net algorithms. According to these methods, the knowledge of the shape on which the fabric is formed and sufficient initial kinematics conditions leads to the positions of the yarns in the final form. [6] These methods are fast and efficient, however they do not account for either the mechanical behavior of the fabric nor the static boundary conditions. These conditions which are not accounted for may be important in other forming processes, for instance, the static boundary conditions are important in the case of forming with punch and die.

2.9. Properties of composites

Composites in general have a problem when it comes to predicting their performance properties. This is due to the synergy effect. This effect explains that when a composite material is produced, at least one of its properties will be greater than pure summation of the individual component properties. This still poses as a problem to composite producers, as it has been stated earlier in this paper that before a composite is produced, the expected properties of the product must be known. A number of models have been developed in trying to overcome this. Some of the studied properties are the impact resistance, ballistic effect, stab resistance, electrical resistance and conductivity.

2.9.1. Impact resistance properties

It has been observed that composites structures in the form of laminates have a great problem of developing cracks and delamination. The initiation of cracks and the delamination has shown great effect on the strength and stiffness of the composites. These basically lead the reduction of in-plane strength and stiffness. They can be caused by the introduction of external loading in the form of bending, compression or impacts of low to medium energy. High energy impacts usually cause complete penetration with obvious damage that is greatly expressed on the surface. Low energy impacts; however tends to cause damage in the inside of the composite laminate. These damages may not be visible on the surface of the composite but causes internal damage which greatly reduces mechanical properties of both the reinforcement and the matrix, as well as the characteristics of the interface region.

According to Kim and Sham, woven fabrics in general shows lower maximum load, higher residual compression after impact strength, smaller damage area, and higher ductility index when compared to unidirectional laminates.

It was observed that these characteristics of woven fabrics were mostly due to, high inter-laminar fracture toughness, lower thermal and elastic mismatches as well as more ductile nature of these fabrics. They also observed that addition of silane agents as surface treatments on woven glass fabrics led to improvement of the impact performance of the composite. [1] Another observation was based on damage resistance of woven

carbon/epoxy composites. It was observed that the woven fabrics resisted damage due to low impact by suppressing the initiating of delamination at critical interfaces and by improving the residual compressive strength after sustaining the impact load.

In attempt of trying to improve the impact tolerance of composites, a number of techniques have been developed but all can be grouped into two groups. In one group, there are techniques in which this is achieved by modifying the fabric architecture; whilst on the second group are models which involve the improvement of the properties of the composite components. Improving fabric architecture involves the use of different structural preforms like braiding, knitting, weaving in producing 2or 3-dimentional fabrics. The latter approach involves improving the impact resistance of the matrix material and that of the fiber/matrix interface bond quality. [1]

2.9.2. Ballistic Effect

Composite materials undergo different loading conditions in their lifetime. Due to this, it is of crucial importance that their endurance under ballistic attack is studied and somehow understood. Some of these composites are produced for high-performance applications like; protective clothing for military, armor plating for military vehicles, transporting cylinders, aircrafts and military helicopters.

In all and more other fields, impact resistance is of crucial importance. Ballistic impact behavior of composites depends in more than one parameters, it depends on the shape of the target, in this case being a composite structure, its properties and the projectile parameters. This means that the ballistic impact resistance of one structure may differ if the projectiles differ. In order for a composite to be able to be resistant to ballistic impact, it must be able to absorb a significant amount of the kinetic energy of the projectile and must have specific strength and stiffness.

According to Naik, N.K et al, [14] ballistic impact is usually due to an object with a low mass travelling at a very high speed from the propelling projectile. When the slow mass objects hits or gets in contact with the target, energy transfer takes place. Energy is transferred from the moving projectile to the target and what happens after that depends on the properties of the target and the projectile parameters. Any of the following cases is possible;

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a) If the projectile's initial energy was more than the energy that can be absorbed by the target, the projectile will penetrate the target and exit with a particular velocity.

b) If the initial energy of the projectile is less than the energy that the target can absorb, the projectile will partially penetrate the target.

c) And if the energy of the projectile is equal to the amount of energy that the target can absorb, this projectile will fully perforates the target but will not exit it.[14]

During the occurrence of any of the above cases, there are different energy absorptions and damage mechanisms that take place within the target. There will be a cone formation on the back of the target due to the push exerted by the projectile as it penetrates.

The secondary yarns will be damaged whilst the primary yarns will experience tension. The matrix will crack and there will be delamination and friction between the projectile and the target. The extent of each mechanism will differ depending on the material properties of the target and the reinforcement architecture. This means that different materials like carbon, Kevlar, or glass will behave differently.

2.9.3. Thermal properties

The understanding of the behavior of materials when they are exposed to increased temperatures is of paramount importance. When materials are exposed to extreme temperatures, either due to design or by accidents, they have a tendency of changing their dimensions and sometimes their mechanical behavior. These parameters need to be greatly studied so as to be able to predict conditions under which a particular material will be suitable to use. Some of the thermal parameters which are important include thermo mechanical analysis (TMA) and dynamic mechanical analysis (DMA).

TMA can be defined as a measurement of a specimen's dimensions (length or volume) as a function of temperature whilst it is subjected to constant mechanical stress. From such studies, the thermal coefficients of materials can be deduced and their changes with temperature and/or time are monitored. Materials tend to deform under stress at a particular temperature as a result of melting or probably undergoing glass transition. [20]
DMA on the other hand deals with the measurement of mechanical properties (like mechanical modulus stiffness and damping) of materials or samples as a function of temperature.

This is a parameter that is sensitive and deals with the molecular mobility within the material. It is mostly used to study the glass transition temperatures and also changes of mechanical properties due to chemical reactions. To do such a measurement, the sample is exposed to an oscillating stress, usually following a sinusoidal wave form.

2.10. Uses of Composites

Composites have been part of human for a long time. Humans have been using composites, probably without even giving them the name composites. For instance, they used straw and mud for building walls. This formed a strong composite wall. Mud bricks are easy to break by bending. A piece of straw on the other hand has great strength when being stretched, but none when crumpled. When, however, the pieces of straw are embedded in a block of mud and this is allowed to dry hard, it gives a mud brick which resist both tearing and squeezing. With these properties, the mud brick becomes a very strong building material.

Nowadays, composites have gained attention from various industries and their use is spreading. In other industries, however, their use is still limited due to lack of trust and the fact that their capabilities are still under study. The following are some of the industries where composites are paving their way:

2.10.1. Construction

Reinforced composites have been used in construction for non-critical applications like baths, finishing and decoration. In the last decade, reinforced composites have been trying to find their way into the construction industry. Their advantages that are recognizable for their application in construction include; high specific strength, durability, high specific stiffness, good fatigue performance, lower maintenance costs and easy to form various structures. Due to these properties, the composites are finding way to construction applications like rehabilitation and retrofit, and as reinforcement for concrete. Homes can be framed using plastic laminated beams instead of traditional wood. This coating with plastics minimizes the risk of rot of the beams thus giving them a long life. On another application, fiber-reinforced shingles create strong maintenance-free roofing that could last for decades. Doors and flooring of composites are able to imitate the look of wood whilst offering an extended life expectancy at a lower cost sometimes. Composite laminates are also used in many bathrooms and kitchen walls to create a durable, waterproof and easy to clean surface.

2.10.2. Civil engineering

A small number of load bearing civil structures have been made of composites. These include compound curved roofs, pedestrian and vehicle bridges, bridge decks, energy absorbing roadside guard rails, building systems, modular roof top cooling towers. Their application is still not well accepted since it is still under experiment so as to see how possible it is to use them. They do show success in terms of structural performance but little success in terms of financial data. Some of the composite raw materials like carbon are still very expensive for companies even though they may show great structural performance.

2.10.3. Aircraft industry

For quite some time, aerospace engineers were coming with designs which seemed not possible to produce because the material s to construct them did not exist. An example could be the large spaceplanes like the space shuttle which some years back seemed impossible until there were the heat-resistant ceramic tiles which protect it during reentry.

Aerospace engineering now has changed. Traditionally, airplanes were made of metals, usually alloys of aluminium but that has changed now. Composite materials have gained recognition in this industry and have helped engineers to overcome obstacles that were met when individual materials were used. Composite materials can provide a much better strength to weight ratio than metals, which could be about 20% better. The lower weight has resulted in fewer fuel consumptions and emissions.

According to Dwayne, composites have not been a miracle for air craft structures. They are hard to inspect for flaws and some of them may absorb some amount of moisture. They are expensive mostly because they require complex and expensive fabrication methods. Another problem is that a metal, once dented can be hammered back to shape without replacing the whole structure yet a composite material once cracked needs to be fully replaced. Due to these reasons, composites are mostly adapted by military planes which are constantly being maintained than commercial air crafts which are not frequently maintained. In the production of composites for aviation, thermoplastic matrix seems to be better that thermosets, especially on light weight impacts.

For instance, if an object, say a spanner falls onto a wing accidentally, it could crack a thermoset composite because they a brittle but would bounce off on a thermoplastic material. [15]

2.10.4. Automobile

Composite properties such as their low density, durability, stiffness and strength have led to them gaining much recognition in various industries including the automotive industry. Like in the other industries, the obvious advantage of composites is the low weight due to their low densities. This comes with less fuel consumption thus saving both the economical aspect as well as the environmental aspect.

In manufacturing a composite component, the fibers can be oriented in the direction required so as to withstand the required stress thus less material could be used. Complicated shapes can be produced in a single step and some composites can be fabricated in lower temperatures thus saving on cost of production. Also, with their resistance to corrosion and rusting, composites are likely to give components with a prolonged lifetime.

Even in the automobile industry, composites still have limited usage. This is probably because most of their capabilities are still under study so no one wants to take the risk. Another thing could be that most existing production lines in industry are designed for the production of metals so putting up new composite production lines might take some time. There are still doubts on whether the use of the composites is really beneficial as far as cost is concerned since the low weight which comes with savings in fuel usage and

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better vehicle performance also comes with extra production cost of the composite materials as they need special equipment.

The use of composites in automobile is not a new phenomenon. Composites have been tried to be used as early as the 1930's when Henry Ford tried to use soya oil to produce a phenolic resin and hence produce a wood filled composite for car bodies. [16] The development however was slow since there was not enough knowledge of efficient producing technologies. As glass and polyester came commercially available, interest was taken into composites but till today composites are not yet fully explored. There seems to be so much to learn from them thus extended studies on their capabilities are taking place.

Engineers are trying to find ways of using composites in automobiles not just because composites are available but because they are trying to improve the performance of vehicles and reduce their maintenance. Some of the reasons why composites are of so much interest are as follows:

a) To improve safety and crashworthiness

Crash worthiness includes the following:

- Maintaining space that will improve survival of the occupants after crashing
- Be able to keep the occupants within that safe space after an accident
- Limit the deceleration of the people in the vehicle within acceptable levels
- Retain safety-cage integrity
- Minimize post-crash hazards

A material that has good crashworthiness must be able to absorb more energy resulting from the crash. In that case, less energy will reach the occupant.

b) Effect of reduction in weight when composites are used

They have observed that the reduction in weight of a vehicle leads to reduces fuel consumption and the performance of the vehicle will improve at a cheaper cost.

2.10.5. Sports

Most sport products now contain a composite component. This is done so as to improve performance in the sports and durability of the sport products. For instance, surfboards and skis contain composite materials that add flexibility and still maintain durability for good performance. Some baseball bats also may have a composite handle, so as to be strong and not to break easily.

2.10.6. Boating

Many recent boats are made from composites such as fiberglass or thermoplastics. These boats are a great achievement compared to traditional boats. Traditional wooden boats were likely to rot over time whilst metal boats were heavy and susceptible to rusting. Composites boats are light in weight thus stay afloat, yet are strong enough to resist punctures and cracking. Composite boats are also unlikely to corrode even after extensive exposure to salt water and sea air.

2.10.7. Ballistic

Composites have gained recognition in armor. They are mostly used for ballistic protection for light weight vehicles. To achieve this, composite materials of various hardness and elasticity are used for heat and shock absorption. Composites are used to provide protection against both kinetic and chemical weapons, for instance those that are hard and strong like Kevlar reinforces composites are used for kinetic protection like ballistic and those that are resistant to chemical like some type of glass fibers are used in protection against chemicals. Armor products can be passive or active and reactive. Passive armor materials are designed to absorb impact and stop penetration of ammunition whilst active and reactive materials are designed to deflect or destroy ammunition. These composites can also be used for protection against fire and some explosives since some of them are flame resistant. [20]

3. Materials and Methods

3.1 Objectives of study

1) To make composites using woven fabrics

2) To make composites containing fly-ash Nano-particles

3) To measure and analyze the mechanical properties of these composites

4) To compare measured properties of composites containing fly-ash and of those without.

3.2 Materials used

The following materials were used in the production of the composites

3.2.1. Reinforcement

The reinforcement used for the preparation of composites was based on glass fibers. Yarns of glass fiber were woven into four different structures. These are plain 2-D fabrics, 3-D multilayer fabrics; 3-D orthogonal fabrics and the 3-D angle interlock fabrics. All the glass woven material was prepared in India. The 2-D fabrics have a set of two yarns which are interlaced at 90°. One set of yarns is called the weft and the other called the warp yarns. All 3-D fabrics have three sets of yarns, the warp, weft and z-yarns. The third set of yarns (z-yarns) help to provide through the thickness dimension. The difference in the three kinds of 3-D fabrics is in the interlacing of the third set of yarns.

3.2.2. Epoxy matrix and hardener

The matrix used was an epoxy resin LH 288. This type of epoxy resin is characterized by low viscosity. It offers high quality properties such as high mechanical properties, good thermal properties and good chemical resistance. It is based on bisphenol A with viscosity 500-900 mPa.s.

There is no crystallization under normal storage temperature. The vapor pressure at 20°C is below 13mPa and density is from 1100 to 1200kg.m⁻³ at 25°C. It is insoluble in water, but soluble in most organic solvents. After being mixed with hardener, the resin has to be used within 15 minutes; otherwise it thickens, making application to any surface impossible.

3.2.3. Fly ash particles

Fly ash is a residue generated during combustion that contains fine particles that rises with flue gases. The fly ash used in these experiments was collected from a source in Plzeň, Czech republic. Before use, the fly-ash was mechanically activated by a high energy planetary ball mill.



Figure 2: SEM picture of fly ash

3.3 Methods

3.3.1 Weaving

To produce 2D fabrics, sets of two yarns were orthogonally interlaced. In the production of 3D fabrics, a third set of yarns had to be introduced in the system. The interlacing of yarns in this case occurs amongst the X, Y and Z set of yarns. In these weaving methods, the shedding of multi warp yarns is done in both the horizontal and through the thickness directions. This can be seen in the following pictures



a) 2D plain woven fabric



b) 3D multilayer woven fabric



c) 3D orthogonal woven fabriC



d) 3D Angle interlock

Figure 3: Weaving technologies: a) 2D-plain weaving, b) Multilayer, c) Orthogonal and d) Angle interlock

3.3.2. Preparation of fly-ash

As mentioned earlier, the fly ash used in the study was collected from a source in Plzeň, Czech republic. The fly ash was mechanically activated using a high-energy planetary ball mill of Fritsch Pulverisett 7 in a sinctered corundum container of 80ml capacity using zirconia balls of 3mm Ø under wet conditions in distilled water for 1, 2, 3, 4, and 5 hours. The ball mill was loaded with ball to powder weight ratio of 10:1. The rotation speed of the planet carrier was 850 rpm. In this mechanical treatment, powder particles are subjected to severe plastic deformation due to the repetitive compression loads arising from the impacts between the balls and the powder. The milled sample powder was taken out at a regular interval of every 1 hour of milling to test for particle size distribution on Malvern Zetasizer Nano based on dynamic light scattering principle. The dispersion medium was deionized water. The dispersion was ultrasonicated for 5minutes with bandeline ultrasonic before characterization. Refractive index of 1.55 used for fly ash to calculate particle size.

The quantitative analysis of the fly ash used is summarized in the table below:

Element	Atomic [%]	Standard deviation		
0	53.80	1.13		
Na	1.84	0.22		
Mg	1.06	0.26		
Al	14.27	0.65		
Si	20.25	1.87		
Р	0.09	0.02		
S	0.19	0.01		
К	0.25	0.04		
Ca	0.94	0.31		
Ti	1.95	0.22		
Fe	4.72	2.32		
Cu	0.16	0.02		
As	0.15	0.01		
Zr	0.34	0.04		

Table 1: Quantitativ	e elemental	analysis d	ata of fly ash
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The diameter distribution of the unmilled and milled fly ash particles is displayed in the figures below



(a) Unmilled and milled fly ash



(b) Unmilled fly ash



(c) Milled fly ash after 5 hr

Figure 4: Particle size distributions of unmilled and milled fly ash

3.3.3 Preparation of composites

The first set of composites prepared were composites with only the epoxy matrix. In preparing the matrix, 73% was the epoxy matrix whilst 27% was the activation hardener. These two were mixed in a beaker using a stirrer before the mixture could be applied on the fabric. To apply the matrix on the fabric, a hand brush was used to smear matrix on both sides of the fabric sample. Pressure was exerted, by hand during this application to ensure uniform penetration of the matrix through the fabric thickness. Epoxy matrix was applied to all the kinds of woven fabric produced. The ratio of fabric to matrix was 1:1.

Another set of composites prepared were containing the fly-ash described above. For this preparation, the required percentage of the fly ash was first mixed with the epoxy and hardener in a beaker using an ultrasonic stirrer. The mixture was then applied on the fabric by hand brush. The percentage of fly ash added ranged from 1 %, 3%, 5% and 10%.

In preparing all composites, the following steps were followed;

1) The weight of the fabric was measured.

2) 27% of hardener was added to epoxy matrix such that weight of matrix plus hardener were equal weight of the fabric, provided fly ash was not to be included.

3) In a case where fly ash was added, the required percentage of fly ash was added to the matrix and hardener such that the weight of fly ash plus hardener plus matrix was equal to the weight of the fabric.

4) Hardener and matrix were mixed by stirring in a beaker then applied on the fabric by brush.

5) After the thorough application of matrix on the fabric, it was placed in a mold, pressed and cured in the oven at 120°C for 20 minutes.

6) After curing, the composite was allowed to cool.

3.3.4 Testing and measurement

a) Mechanical properties

i) Stab-resistance test

To carry out this test, the testomeric tester M350-10CTwas used. This is a machine with an adjustable speed of the penetrating knife and a changeable type of stabbing knife. For the testing purposes of this experiment, a single sharp pointed knife, as shown in the figure below, was used. Each composite sample was placed on the plate of the machine. The machine head is dropped onto the sample thus holding the sample in place. The knife was then allowed to penetrate the fabric sample at a velocity of 1000mm/min. A recording monitor is connected to this machine and records the maximum force exerted on penetrating the sample.



a) Pointed knife blade before penetrating fabric



b) Testomeric stab resistance measuring instrument

Figure 5: *stab resistance measuring instrument: a) knife blade before penetration, b) blade after penetration and data analysing computer*

ii) Flexural strength

Flexural strength can be defined as the ability of material to resist deformation under load. One of the ways used to measure flexural strength is the three-point bending where force is applied onto the object hanging in between two end points. On the inside of the bend, the compressive stress will be at maximum (figure 6 at position B), whilst on the outer curve of the bend will be maximum tensile stress (see figure 6 position A).

The flexural stress, therefore represent the highest stress experienced within the object at rupture.



Figure 6: Three-point measuring of flexural strength

b) Thermal Properties

Dynamic Mechanical Analysis (DMA)

The dynamic mechanical analysis of the composites was performed on DMA DX04T RMI instrument. The test was performed in three point bending mode with gauge length and sample width of 30mm and 10mm respectively. The samples were subjected to an oscillating frequency of 1Hz and 100% oscillating amplitude in the temperature range 30°C to 300°C at the heating rate of 5°C per minute.



Figure 7: DMA measuring instrument

c) Electrical Resistance

Electrical resistivity is the measure of how much a material inhibits the flow of electric current. A low resistance is a characteristic of a material that allows the movement of electrons thus conducting electricity. An ohmmeter is used to measure electrical resistance of materials.

d) Electromagnetic interference (EMI) shielding

EMI shielding was measured by instrument Agilent E4991A, which has an electromagnetic wave creator and an electromagnetic wave receiver. The electromagnetic wave creator, created an electromagnetic wave at 2.4 GHz. The electromagnetic wave transferred was displayed in power flux mW/m2.

The electromagnetic wave transferred was recorded when there was no sample and when there was a sample placed in between the two. To calculate the shielding efficiency of the sample, the log equation is used as follows:

 $SE = 10log_{10} (P_R/P_L)$

Where P_R is power without the sample and P_L is power with the tested sample.

e) Scan Electron Microscope (SEM)

Scan electron microscope images of some cross sections of the composites were taken so as to view the distribution of both the matrix and fly ash particles within the composite structure. Below are the images of composite structure without fly ash and composite structure with infused fly ash particles.

The scanning electron microscopy uses a focused electron beam to scan small areas of solid samples. When the sample is placed in the machine, secondary electrons are emitted from the sample and are collected to create an area map of the secondary emissions. These secondary emissions are dependent on the local morphology, thus the area map is a magnified image of the sample.

The scan electron microscope used in this study was the Vega TS 5130 from Tescan. The magnification was 60x, 100x, 500x and 1000x.



SEM MAG: 100 × HV: 20.0 kV VAC: HIVac

DET: BE Det + SE Det L DATE: 03/30/12 50 Device: T85130

Vega ©Tescan TU Liberec



 SEM MAG: 65 x
 DET: BE Det + SE Det

 HV: 20.0 kV
 DATE: 03/30/12

 VAC: HiVac
 Device: T85130

Figure 8: SEM images

4. Results and discussion

After the preparation of composites, the above tests and measurements were performed, so as to obtain an idea of the performance of these composites. These tests were based on the comparison of the presence/absence of fly ash particles whereas others were based on the effect of increased fly-ash composition.

4.1 Stab-resistance test

The aim was to observe if the addition of filler does improve the knife penetration resistance of composites. The maximum force exerted by the knife in order to penetrate the composite was recorded and is shown in the tables below and graphs.

Table2: Maximum strength of 2D plain woven composite during knife penetration

Percentage of Nanoparticles of fly ash	Maximum strength for penetration (N)
0	530.533
10	647.267



Figure 9: Effect of nanoparticles on knife penetration strength of 2D woven composites

Table	3 :	Maximum	strength	of	3D	angle	interlock	woven	composite	during	knife
penetr	atio	on.									

Percentage of fly ash nanoparticles	Maximum strength for penetration (N)
0	551.90
10	581.7



Figure 10: Effect of nanoparticles on knife penetration strength of 3D angleinterlock woven composites

Table 4: Maximum strength of 3D multilayer woven composite during knife penetration

Percentage of fly ash nanoparticles	Maximum strength for penetration(N)
0	593.067
10	624.5



Figure 11: Effect of nanoparticles on knife penetration strength of 3D multilayer woven composites

Table 5: Maximum strength of 3D orthogonal woven composite with FA Nano particlesduring knife penetration

Percentage of fly ash nanoparticles	Maximum strength (N)
0	401.6
1	406.733
3	457.167
5	483.933
10	530.6



Figure 12: Effect of nanoparticles on knife penetration strength of 3D orthogonal woven composites

Table 6: Maximum strength of 3D orthogonal woven composite with micro particlesduring knife penetration

Percentage of fly ash micro-particle	Maximum strength (N)
0	401.6
1	452.825
	770.0
3	550.8
5	619.233
10	635.9



Figure 13: Stab resistance of woven fabric composites with micro particles of fly ash

Figures 9, 10 and 11, above all show that the force exerted during penetration increases with the addition of the filler material. Figures, 12 and 13 shows that force increases step by step. The resistance to penetration by sharp object is due to the spread of resin on the reinforcement thus holding the fibers together. The addition of filler to the system creates more particles on the surface and within the structure making it harder for the knife to pass through the fibers. Since the structure is not easy to penetrate, more force needs to be exerted to enhance the penetration. Both Nano and micro particles has the same effect.

4.2 Flexural strength

The flexural strength of each type of composite was measured, and for comparisons, the results were displayed in the following tables.

 Table 7: Flexural strength of 2D plain glass/epoxy composites

Percentage of nanoparticles	Flexural strength (MPa)
0	281.435
10	236.154



From table 7, the following graph was deduced.

Figure 14: Flexural strength of 2D plain woven composite

Table 8:	· Flexural	strength	of 3D	multilayer	glass/e	ероху сот	posite
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Percentage of nanoparticles of fly ash	Flexural strength (MPa)
0	251.685
10	218.777

From the above table, the following bar chart was drawn



Figure 15: Flexural strength of 3D woven multilayer composite

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Percentage of nanoparticles of fly ash	Flexural strength (MPa)
0	187.9041
10	178.9677

 Table 9: Flexural strength of 3D angle interlock glass/epoxy composite



Figure 16: Flexural strength of 3D woven angle interlock composite

For composites made from 3D orthogonal woven fabric, comparisons were made between the flexural strength of composites with increasing percentage of fly ash Nano particles.

 Table 10: Flexural strength of 3D orthogonal glass/epoxy composite with fly ash Nano

 particles

Percentage of nanoparticles of fly ash	Flexural strength (MPa)
0	171.4525
1	130.9
10	125.4879



Figure 17: Flexural strength of 3D orthogonal woven composite with fly ash Nano particles

A similar comparison was made on increasing percentage composition of Micro particles of fly ash in the composite.

 Table 11: Flexural strength of 3D orthogonal glass/epoxy composite with fly ash micro

 particles

Percentage of micro-particles of fly ash	Flexural strength (MPa)
referringe of mero particles of my ash	Tiokului suongui (ivii u)
0	171.4525
1	162.095
1	102.085
3	115.0831
5	106.096
	00.010
10	98.919



Figure 18: Flexural strength of 3D orthogonal woven composite with fly ash micro particles



Figure 19: Comparison of flexural strength of 3D orthogonal woven composites with Nano particles and micro particles of fly ash.

From the bar graphs shown above, it is clear that the increase in percentage content of the fly ash particles in the system leads to the decrease in flexural strength. This could be due to that as the content of fly ash is increased, their dispersion in the epoxy rich matrix increases thereby resulting in insufficient cross-linking of the epoxy and the hardener. When there is insufficient cross-linking between these two, the total strength of the whole structure is reduced since the resin-filler interaction is the major cause of stability and tightness of the composite.

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Another possibility of this decrease is due to non-wetting of the filler particles with matrix as their concentration increase in the system. This is due to insufficient resin to coat the filler thus reduction of filler – matrix interactions. These particles become randomly distributed in the cured epoxy system and thus there is poor stress transfer at interfacial region. The failure of transferring stress from one molecule to the other leads to structure failure. Also, due to this discontinuity in the system, the filler material fails to carry any load. Increasing filler content leads to increase in viscosity which in turn reduces ease of processing. This increase void content and reduces strength properties of the composite.

4.3. Dynamic Mechanical Analysis (DMA)

The dynamic mechanical analysis was done to compare the performance of the composites made from the four different kinds of woven fabrics. Below are the graphs which were obtained.



a) Storage modulus for woven composites with no fly ash particles



b) Damping factor to for woven composites with no particles of fly ash

Figure 20: a) Graph of storage modulus of neat composites, b) graph of damping factor(tan delta) of neat composites

The test was also performed on the composites produced from the different fabric structure, but having the same content of fly ash (10% fly ash).



a) Storage modulus of woven composites with 10% Nano particles of fly ash



b) Damping factor of woven composites with 10% fly ash Nano particles

Figure 21: *a*) storage modulus of woven composites with 10% fly ash Nano particles, b) damping factor of woven composites with 10% fly ash Nano particles

Another set of dynamic mechanical analysis was performed with respect to the change in Nano particle fly-ash content in composites produced from 3-D orthogonal fabric structure, and the following graphs were deduced from the results.



a) Storage modulus of woven orthogonal composite with increasing concentration of fly ash Nano particles



b) Damping factor of composites with increasing concentration of Nano particles

Figure 22: a) storage modulus of 3D orthogonal woven composites with increasing fly ash Nano particles, b) damping factor of 3D orthogonal woven composites with increasing fly ash Nano particles

Finally, the test was performed on composites produced from 3-D orthogonal fabrics with varying content of micro-particles of fly ash



a) Storage modulus of 3D woven composite with increasing concentration of micro particles of fly ash

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b) Damping factor of composites with increasing content of fly ash micro particles

Figure 23: a) storage modulus of 3D orthogonal woven composites with increasing fly ash Nano particles, b) damping factor of 3D orthogonal woven composites with increasing fly ash Nano particles

In the figures above, the storage modulus-temperature profiles and dissipation of energy (tan delta)-temperature profiles of composites with different concentration of Nano particles and micro particles of fly ash are shown. The aim of this measurement was to observe if there exist a relationship as far as storage modulus and transitions temperatures (T_{g}) and the increase of filler concentration in the composites.

Figure 21(a) and 22(a), shows that the storage modulus both in glassy and rubbery regions increases with increasing filler Nano particles. This makes the composite with 10% fly ash Nano particles has high storage modulus whereas the composite with no filler material has the lowest storage modulus. The increase in storage modulus is due to high aspect ratio of dispersed filler particles and their interaction with the polymer chains leading to high molar mass of the system. The addition of Micro particles of fly ash shows no particular trend, though it is clear that addition of these particles reduces the storage modulus of the composites, figure 23 (a). This reduction a result of reduced molar mass as the structure changes to being intercalated.

From this, it can be concluded that addition of fly ash Nano particles increases the storage modulus of the system whilst addition of micro particles of fly ash reduces it. In all the storage modulus graphs, there is a clear initial glassy region at lower temperatures. This could be an indication of stronger matrix/filler interaction. The steep drop of storage modulus at high temperature indicates that the increased thermal energy could easily exceed the matrix/filler interaction forces leading to a sharp decrease in storage modulus.

The glass transition temperature (T_g) in figures 22(b) and 23 (b)is clearly defined by maximum in the curves. In the addition of Nano particles, the transition temperature of the composite did not change, that is, the maximum of the curves is similar, though the relaxation (maximum tan delta) of 5% and 10% Nano fly ash is higher than that of the neat composite. The T_g of composites with filler Micro particles shows a little increase from that of neat composite. This increase is as a result of the restricted chain mobility due to the presence of filler particles

4.4. Electrical resistance

The electrical resistance of both volume and surface of the composites were measured and the following bar graphs were obtained;

Table 12: Electrical resistance of 2D plain woven glass/epoxy composite with Nanoparticles of fly ash

% of fly ash nanoparticles	Volume electrical resistance	Surface electrical resistance
	(x10 ¹⁰ Ω)	(x10 ¹⁰ Ω)
0	4.8335	7.19175
10	4.28525	7.0337



a) Volume electrical resistance of 2D woven composite



b) Surface electrical resistance of 2D woven composite

Figure 24: Electrical resistance of 2D woven composite; a) volume resistance, b) surface resistance

Table 13: Electrical resistance of 3D Multilayer woven glass/epoxy composite with Nanoparticles of fly ash

% of fly ash nanoparticles	Volume electrical resistance	Surface electrical resistance
	(x10 ¹⁰ Ω)	(x10 ¹⁰ Ω)
0	4.9885	6.849
10	4.1112	6.3051



a) Volume electrical resistance of 3D multilayer woven fabric



b) Surface electrical resistance of 3D multilayer woven fabric composite

Figure 25: *Electrical resistance of 3D woven multilayer composite; a) volume resistance, b) surface resistance*

Table 14: Electrical resistance of 3D angle interlock woven glass/epoxy composite withNano particles of fly ash

% of fly ash nanoparticles	Volume electrical resistance	Surface electrical resistance
	(x10 ¹⁰ Ω)	(x10 ¹⁰ Ω)
0	4.51895	6.90465
10	4.363175	6.5114



a) Volume electrical resistance of 3D angle interlock woven fabric composite



b) Volume electrical resistance of 3D angle interlock woven fabric composite

Figure 26: *Electrical resistance of 3D woven angle interlock composite; a) volume resistance, b) surface resistance*

The electrical resistivity of the composites showed to decrease with the increase in the filler content. This is because of interaction between the filler particles in the system. When these interact, they increase the flow of electricity in the system. This is as a result of the reduced barrier by the non-conductive epoxy matrix. When electrical conductivity is increased, the electrical resistance is directly reduced.

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This decrease in electric resistance is low at lower filler content because the conductive fillers tend to form isolated islands and do not contact well everywhere. This leads to narrow gaps separating them thus decreasing the continuous flow of current.[21] This could, however be expected to reach a certain thresh-hold where the increase of filler will no longer make any difference to resistivity due to channels of conduction being fully exploited.

4.5. Electromagnetic interference (EMI) shielding

The composite materials were tested for electromagnetic interference shielding. From the results obtained, the following tables and bar charts were deduced for the various composites.

Table 15: EMI shielding efficiency of woven composite with Nano particles of FA

Percentage of fly ash	EMI shielding efficiency		
0	0.8428215		
10	1.012748		



Figure 27: EMI shielding efficiency of 3D woven composite with Nano particles of FA

Table 16: EMI shielding efficiency of woven composite with micro particles	of FA
--	-------

% of particles	0	1	3	5
EMI shielding efficiency (dB)	1.031334	1.4752	1.518614	1.518614



Figure 28: EMI shielding efficiency of woven composite with Micro particles of FA

In the figures above, it is evident that fly ash addition to the composite increases EMI shielding efficiency of the composites. The increase of EMI shielding efficiency at lower concentrations of fly ash may be due to absorption and reflection of the electromagnetic waves as a result of the presence of Fe_2O_3 in the ash. Cao,J and Chung,DDL [23],confirmed the relation of EMI shielding efficiency to Fe_2O_3 by replacing the fly ash with Fe_2O_3 in their study. This addition led to increased shielding efficiency, thus confirming that the addition of fly ash which has Fe_2O_3 does increase efficiency.

4.6. Ultraviolet (UV) transmittance

The samples of composites produced by using different woven structures of glass fibers and epoxy resin with fly ash as filler were further studied for transmittance of UV-light. The UV-light transmittance of composites as function of wavelength is shown in figure 29, below:



a) UV-transmittance by 3D angle interlock woven composite with Nano particles of



.



b) UV-transmittance by 3D composite with micro particles of FA



c) UV-transmittance by woven composite with FA Nano particles

Figure29: UV-transmission of ; a) 3D woven angle interlock composite, b) 3D woven Orthogonal composite with fly ash Nano particles, c) 3D woven Orthogonal composite with fly ash micro particles

The graphs above shows that the addition of fly ash filler in the glass/epoxy composite increases the transmittance of UV light, though slightly. This could be due to that the presence of the filler particles in the system decreases the interaction of the resin molecules. With less interaction between these particles and the matrix creates voids and interstices in the structure thus allowing light (in this case UV-light) to be easily transmitted through. This basically means that fly ash cannot be used for improving UV protection of these composites. The transmittance of UV light through the structures is generally reduced as the wavelength increases. This is also enhanced by the fact that epoxy matrix on its own does not have good UV resistance.

5: Conclusion

The findings in this study indicate that addition of filler particles to the composite material does have effects. These effects can be either good or bad depending on the desired usage of the product. The addition should, however be monitored since too much concentrations may turn negative even if it had positive results at lower concentrations. This means that there is a maximum ratio between filler and matrix, so as to avoid too much interference between the resin, hardener and the filler particles.

The properties which were investigated in this study were mechanical, thermal and electrical properties. The stab resistance increased with addition of filler. This was due to the fact that the filler particles added obstruction on the surface and within the structure thus making penetration not easy. This is because stab or rather penetration of a sharp object like a knife is achieved by sliding through between the spaces in the fabric. When the resin coats the fabric, it closes the interstices in between the yarns. But when the filler particles are added, they do not only close the spaces, they also create obstruction due to their large surface area and enhanced interaction with the matrix.

The flexural strength on the other hand, decreased with the addition of filler materials. This was due to the particles weakening the resin/harder crosslinking. When the interaction between harden and resin is minimized, the whole structure is weakened leading to less stress transfer from one molecule to the other. At the same time, the filler particles fail to carry the stress leading to ease of failure of the structure.

The thermal property that was measured was the dynamic mechanical analysis (DMA). These results showed a distinction between the behavior on Nano particles of filler and micro particles of the same filler. Nanoparticles led to increase in storage modulus due to increase in molar mass. Addition of micro particles, however, reduces the storage modulus due to reduced molar mass. The transition temperature in micro particle composites is slightly shifted to higher temperatures due to restricted chain mobility in the resin structure.

Electrical resistance decreased as filler particles increased. This is because of the interaction between the filler particles. When the particles interact, they reduce the

barrier by the nonconductive epoxy resin thus leading to electric flow. The increase in electric flow reduces the electric resistance of the system.

The EMI shielding efficiency of the composites increased when filler was added. This is due to the presence of Si_2O_3 in fly ash. When shielding occurs, the electromagnetic waves are being absorbed and some reflected away.

The UV light transmission was also measured using the spectrophotometer. The UV transmission was increased. This was due to the filler disrupting resin molecule's interaction thus creating interstices which easily enable light rays to pass through.

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7: Appendix

SEM images



SEM MAG: 60 x HV: 20.0 kV VAC: HiVac

1 mm

SEM MAG: 100 x Vega ©Tescan HV: 20.0 kV TU Liberec VAC: HiVac

Vega ©Tescan TU Liberec





SEM MAG: 65 x HV: 20.0 KV VAC: HIVac

DET: BE Det + SE Det DATE: 03/30/12 Device: TS5130

1 mm Vega T

SEM MAG: 100 x Vega ©Tescan HV: 20.0 kV TU Liberec VAC: HiVac DET: BE Det + SE Det LLL DATE: 03/30/12 500 um Device: TS5130

Vega ©Tescan TU Liberec

