<u>Practical Workbook</u> (MY-308): Polymer & Composite Materials



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<u>Practical Workbook</u> (MY-308): Polymer & Composite Materials



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This is to certify that this practical book contains _____ pages.

APPROVED BY:

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Course Teacher

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1.		To study the safety procedures involved in polymer and composite processing.	
2.		Study the process cycle and main parts of injection molding machine.	
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PRACTICAL 1

OBJECTIVE: To study the safety involved in polymer & composite processing

THEORY

Composites are classified according to their matrix phase. There are polymer matrix composites (PMC's), ceramic matrix composites (CMC's), and metal matrix composites (MMC's). Materials within these categories are often called "advanced" if they combine the properties of high strength and high stiffness, low weight, corrosion resistance, and in some cases special electrical properties. This combination of properties makes advanced composites very attractive for aircraft and aerospace structural parts.

ELEMENTS

The feature common to all composite processes is the combining of a resin, a curing agent, some type of reinforcing fiber, and in some cases a solvent. Typically, heat and pressure are used to shape and "cure" the mixture into a finished part. In composites, the resin acts to hold the fibers together and protect them, and to transfer the load to the fibers in the fabricated composite part. The curing agent, also known as hardener, acts as a catalyst and helps in curing the resin to a hard plastic. The reinforcing fiber imparts strength and other required properties to the composite. Solvents may serve three purposes:

- as part of the resin mixture;
- as part of the process; and
- as a cleaning agent for removing residue from the process

MAJOR PROCESSES

The processes vary widely in type of equipment and potential worker exposure. Several of the processes are automated; however, some are manual and require worker contact with the part during manufacture. The basic process types are described below.

Formulation

Formulation is the process where the resin, curing agent, and any other component required are mixed together. This process may involve adding the components manually into a small mixing vessel or, in the case of larger processes, the components may be pumped into a mixing vessel. The potential hazards involve skin, eye, and respiratory contact with the ingredients or final formulation.

Prepregging

Prepregging is the process where the resin and curing agent mixture are impregnated into the reinforcing fiber. These impregnated reinforcements (also known as prepregs) take three main forms: woven fabrics, roving, and unidirectional tape. Fabrics and tapes are provided as continuous rolls in widths up to 72 inches and lengths up to several hundred feet. The fabric or tape thickness constitutes one ply in the construction of a multi-ply layup. Impregnated roving is wound onto cores or bobbins and is used for filament winding. Once the resin mixture has been impregnated onto the fibers, the prepreg must be stored in a refrigerator or freezer until ready for use in the manufacturing process. This cold storage prevents the chemical reaction from occurring prematurely. Prepreg materials are used widely in the advanced composite industry, particularly in aircraft and aerospace. Potential exposure is generally from handling of the fiber or resin.

Open Molding processes

Open Molding processes are those where the part being manufactured is exposed to the atmosphere. The worker typically handles the part manually, and there is a higher potential for exposure. The resin mixture may be a liquid being formed onto a reinforcing material or it may be in the form of a prepreg material being formed for final cure.

Closed Molding processes

Closed Molding processes are those in which all or part of the manufacture takes place in a closed vessel or chamber. The liquid resin mixture or prepreg material may be handled or formed manually into the container for the curing step. In the case of liquid resin mixtures, these may be pumped into the container, usually a mold of some type, for the curing step. These processes usually have less worker exposure potential, particularly if the entire process is closed.

POLYMER MATRIX COMPOSITE (PMC) RESIN SYSTEMS

In this workbook seven manufacturing processes are covered, along with two preliminary processes and two finishing processes. The number and variety of processes should give some indication of the wide spectrum of workplaces likely to be encountered by field personnel. Potential worker exposure obviously will also vary widely, depending on the size and type of process being used. Since the advanced composite industry is relatively new and still developing, other processes may be developing or changing to meet new performance requirements.

Resins

The resin systems used to manufacture advanced composites are of two basic types: thermosetting and thermoplastic. Thermosetting resins predominate today, while thermoplastics have only a minor role in advanced composites manufacture.

Thermoset

Thermoset resins require addition of a curing agent or hardener and impregnation onto a reinforcing material, followed by a curing step to produce a cured or finished part. Once cured, the part cannot be changed or reformed, except for finishing. Some of the more common thermosets include:

- epoxies
- polyurethanes
- phenolic and amino resins
- bismaleimides (BMI, polyimides)
- polyamides

Of these, epoxies are the most commonly used in today's PMC industry. The basic epoxy compounds most commonly used in industry are the reaction product of epichlorohydrin and bisphenol-A. Epoxy compounds are also referred to as glycidyl compounds. There are several types of epoxy compounds including glycidyl ethers (or diglycidyl ethers), glycidyl esters, and glycidyl amines. Several of these compounds are reactive diluents and are sometimes added to the basic resin to modify performance characteristics. The epoxy molecule can also be expanded or cross-linked with other molecules to form a wide variety of resin products, each with distinct performance characteristics. These resins range from low-viscosity liquids to high-molecular weight solids. Typically they are high-viscosity liquids.

Since epoxies are relatively high molecular-weight compounds, the potential for respiratory exposure is fairly low. The potential for respiratory exposure is increased when the resin

mixture is applied by spraying or when curing temperatures are high enough to volatilize the resin mixture. The potential for dermal exposure is typically much greater than respiratory exposure when working with epoxies. Several advanced composite processes involve some worker contact with the resin mixture.

The second of the essential ingredients of an advanced composite system is the curing agent or hardener. These compounds are very important because they control the reaction rate and determine the performance characteristics of the finished part. Since these compounds act as catalysts for the reaction, they must contain active sites on their molecules.

Some of the most commonly used curing agents in the advanced composite industry are the aromatic amines. Two of the most common are 4,4'-methylene-dianiline (MDA) and 4,4'-sulfonyldianiline (DDS). Like the epoxies, these compounds have a very low vapor pressure and usually do not present an airborne hazard unless in a mixture that is sprayed or cured at high temperatures. However, potential for dermal exposure is frequently high. The aromatic amines may permeate many of the commonly used protective gloves and thus may be particularly difficult to protect against.

Several other types of curing agents are also used in the advanced composite industry. These include aliphatic and cycloaliphatic amines, polyaminoamides, amides, and anhydrides. Again, the choice of curing agent depends on the cure and performance characteristics desired for the finished part.

Polyurethanes are another group of resins used in advanced composite processes. These compounds are formed by reacting the polyol component with an isocyanate compound, typically toluene diisocyanate (TDI); methylene diisocyanate (MDI) and hexamethylene diisocyanate (HDI) are also widely used. While the polyols are relatively innocuous, the isocyanates can represent a significant respiratory hazard as well as a dermal hazard.

Phenolic and amino resins are another group of PMC resins. With respect to the phenolformaldehyde resins, the well-known hazards of both phenol and formaldehyde must be protected against. In addition to traces of free formaldehyde, they may also contain free phenol, and contact with these resins in the uncured state is to be avoided. The urea- and melamine-formaldehyde resins present similar hazards. Free formaldehyde, which is present in trace amounts and may be liberated when their resins are processed, can irritate the mucous membranes.

The bismaleimides and polyamides are relative newcomers to the advanced composite industry and have not been studied to the extent of the other resins.

Thermoplastics

Thermoplastics currently represent a relatively small part of the PMC industry. They are typically supplied as nonreactive solids (no chemical reaction occurs during processing) and require only heat and pressure to form the finished part. Unlike the thermosets, the thermoplastics can usually be reheated and reformed into another shape, if desired.

Fiber Reinforcement

Fiber reinforcement materials are added to the resin system to provide strength to the finished part. The selection of reinforcement material is based on the properties desired in the finished product. These materials do not react with the resin but are an integral part of the advanced composite system.

Potential worker exposure is typically higher in facilities that manufacture the fibers or use them to produce prepreg material. Most of the fibers in use are considered to be in the nonrespirable range. However, they do have the potential to cause eye, skin, and upper respiratory tract irritation as a result of the mechanical properties of the fibers. The three basic types of fiber reinforcement materials in use in the advanced composite industry are carbon/graphite fibers, aramid fibers & glass fibers. Fibers used in advanced composite manufacture come in various forms, including yarns, rovings, ,chopped strands, woven fabric, mats, etc.

Each of these has its own special application. When prepreg materials are used in parts manufacture, woven fabric or mats are required. In processes such as filament wet winding or pultrusion, yarns and rovings are used.

The most commonly used reinforcement materials are carbon/graphite fibers. (The terms graphite and carbon are often used interchangeably.) This is due to the fact that many of the desired performance characteristics require the use of carbon/graphite fibers. Currently, these fibers are produced from three types of materials known as precursor fibers:

- polyacrylonitrile (PAN)
- rayon
- petroleum pitch

The carbon/graphite fibers are produced by the controlled burning off of the oxygen, nitrogen, and other noncarbon parts of the precursor fiber, leaving only carbon in the fiber. Following this burning off (or oxidizing) step, the fibers are run through a furnace to produce either carbon or graphite fibers. Carbon fibers are produced at furnace temperatures of 1,000-2,000° C, while graphite fibers require temperatures of 2,000-3,000° C. At these temperatures the carbon atoms in the fibers are rearranged to impart the required characteristics to the finished fiber. The PAN-based fiber is the more commonly used precursor in the advanced composite industry today.

Aramid fibers are another human-made product. These fibers are produced by manufacturing the basic polymer, then spinning it into either a paper-like configuration or into fiber. Aramid fibers have several useful characteristicshigh strength and modulus, temperature stability, flex performance, dimensional stability, chemical resistance, and textile processibility.

Textile (continuous filament) glass fibers are the type used in composite reinforcement. These fibers differ from the wool type in that they are die-drawn rather than spun. A number of solvents are used in the advanced composites industry. These may be introduced into the workplace in three basic ways as part of the resin or curing agent, during the manufacturing process, or as part of the cleanup process.

Solvents

Most of the solvents used may be introduced in any or all of the three ways above. For this reason it would be difficult, if not impossible, to separate the solvents into the categories of use. The solvents discussed in this section are grouped by chemical class ketones, alcohols, chlorinated hydrocarbons, others

Several solvents may be used in any one composite process. One or more may be introduced as part of the resin or curing agent, while another may be a part of the manufacturing process. Still another may be used for cleanup. Thus the hazard information for all products used in the process must be considered when evaluating potential exposures. The supplier's Material Safety Data Sheet (MSDS) should be consulted for more specific hazard information.

Composite residues are often difficult to clean from operation equipment and molds. Various solvents have been used for cleaning, with varying degrees of success. Solvents in the workplace may be found in several areas:

- in small containers near process equipment;
- in larger containers (drums or vats) for soaking and cleaning; and
- in process equipment containers (tanks, reactors, molds, etc.).

HEALTH HAZARDS

Potential health hazards associated with the use of advanced composites (Table _____.1) can be controlled through the implementation of an effective industrial hygiene program. Use of safe work practices, engineering controls, and proper personal protective equipment depends upon an appreciation of health hazard information for a safe work environment.

Resins

The resins used in advanced composite processes have high molecular weights (MW > 10,000) with low vapor pressures. High molecular weight is generally associated with decreased volatility. In an epoxy system, the resin components have very low vapor pressures and they are not present as a volatilized airborne hazard.

As discussed earlier, epoxy resins are currently the most commonly used resins in the advanced composite industry. The basic epoxy molecule is a reaction product of epichlorohydrin (ECH) and bisphenol-A (BPA). Some epoxies contain trace amounts of residual ECH typically in the range of <1 to 10 ppm (by weight). Industrial hygiene air monitoring for ECH has been done in a number of workplaces, involving a variety of epoxy resin end-uses. Most of the monitoring has shown no detectable levels of ECH in the air. Uncured epoxy resins can present a significant dermal exposure hazard. In many workplaces, manual processing results in potential skin exposure. This can result in skin irritation, rashes and, subsequently, dermatitis if contact is prolonged. Sensitization to the resins can also develop and may require a change of work assignment.

Polyurethane resins are reaction products of polyols and isocyanates. The significant hazard associated with these resin systems is the presence of isocyanates. Exposure to highly toxic isocyanates can have adverse health effects. Exposure to the vapor may cause irritation of the eyes, respiratory tract and skin. Irritation may be severe enough to produce bronchitis and pulmonary edema. Polyurethane resins contacting the eyes may cause severe irritation, and if polyurethane resins are allowed to remain in contact with the skin, they may produce redness, swelling, and blistering of the skin. Respiratory sensitization (an allergic, asthmatic-type reaction) may occur. Among the isocyanates, there is also evidence of cross-sensitization, in which a worker is sensitized to one isocyanate but reacts to others as well.

The phenol-formaldehyde resins must be handled with adequate ventilation. Traces of free formaldehyde and phenol may be present. Contact with these resins should be avoided because of the toxicity of these components and the skin-absorption potential of phenol. These components may also be given off during the curing process.

The acute toxicity of urea-formaldehyde resins is very similar to the phenol-formaldehyde resins. Free formaldehyde, which is present in trace amounts and may be liberated when the resins are processed, can have an irritating effect on mucous membranes. Skin sensitization to formaldehyde has been observed.

The health effects of bismaleimide resin systems have not been extensively studied. Manufacturers of these materials indicate that prolonged or repeated contact may cause skin irritation or sensitization. Dust or vapor from heated products may cause irritation of the eyes, nose, and throat.

Polyether and polyester polyols present no particular health hazard in industrial processing.

Thermoplastic resins in general are not considered harmful to workers' health. These resins appear harmless when ingested, and no skin irritation has been reported. No toxic effects are known to be associated with the inhalation of thermoplastic-resin dust. Treating it as nuisance or inert dust seems appropriate, although the presence of unreacted monomers may be of concern. These materials present a thermal hazard when handling. Molding operations may give off vapors which are irritating to the eyes and cause cold-like symptoms. Some thermoplastics are styrene-based, and presence of this monomer may be of concern.

Composite	Organ system target	Known (possible) health effect
component	(possible target)	
Resins		
Epoxy resins	Skin, lungs, eyes	Contact and allergic dermatitis, conjunctivitis
Polyurethane resins	Lungs, skin, eyes	Respiratory sensitization, contact dermatitis, conjunctivitis
Phenol formaldehyde	Skin, lungs, eyes	As above (potential carcinogen)
Bismaleimides (BMI)	Skin, lungs, eyes	As above (potential carcinogen)
Polyamides	Skin, lungs, eyes	As above (potential carcinogen)
Reinforcing materialss		
Aramid fibers	Skin (lungs)	Skin and respiratory irritation, contact dermatitis (chronic interstitial lung disease)
Carbon/graphite fibers	Skin (lungs)	As noted for aramid fibers
Glass fibers (continuous filament)	Skin (lungs)	As noted above
Hardeners and curing agent	S	
Diaminodiphenylsulfone		No known effects with workplace exposure
Methylenedianiline	Liver, skin	Hepatotoxicity, suspect human carcinogen
Other aromatic aminess		
Meta-phenylenediamine (MPDA)	Liver, skin (kidney, bladder)	Hepatitis, contact dermatitis (kidney and bladder cancer)
Aliphatic and cyclo- aliphatic amines	Eyes, skin	Severe irritation, contact dermatitis
Polyaminoamide	Eyes, skin	Irritation (sensitization)
Anhydride	Eyes, lungs, skin	Severe eye and skin irritation, respiratory sensitization, contact dermatitis

Curing agents

Curing agents, or hardeners, used with the epoxy resins are mostly amines, amides, or anhydrides. Two of the most widely used are the aromatic amines, MDA (4,4'-methylenedianiline) and DDS (4,4'-diaminodiphenyl-sulfone).

Analysis and review of epidemiologic data and human and animal toxicity data indicates that occupational exposure to MDA may result in reversible liver toxicity (hepatotoxicity). The retina of the eye might be damaged not only by direct contact but also from MDA absorbed through ingestion. MDA is an animal carcinogen and a suspect human carcinogen by any exposure route: ingestion, inhalation, or dermal.

Frequently, curing agents containing mixtures of these amines can cause skin staining in processes requiring dermal contact, even when protective gloves are used. Brown and orange stains on walls and ceilings have also been reported. The skin staining has been attributed to MDA; dermal absorption is approximately 2% per hour. Soap and water, rather than any organic solvent, should be used for skin clean-up to avoid any solvent increase of transdermal absorption.

The OSHA permissible exposure limits (PEL'S) for MDA are 10 ppb (parts per billion) expressed as an 8-hour time-weighted average, and a short-term exposure limit (STEL) of 100 ppb averaged over any 15-minute period for either general industry or construction uses of MDA.

Another of the amines, DDS, has a significant amount of toxicological data as its pharmaceutical grade, DapsoneTM, has been used for years to treat leprosy and certain types of chronic dermal inflammation. However, at low airborne concentrations, there are no known effects from workplace exposure.

Other aromatic amines used in the advanced composites industry include m-phenylene diamine and the various isomers of toluenediamine. These aromatic amines are considered to be only slightly irritating to the skin.

Aliphatic and cycloaliphatic amines are strong bases and are considered to be severe eye and skin irritants. Inhalation of these amines can cause irritation of the nose and throat, and lung irritation with respiratory distress. Some of these amines are also skin and respiratory-tract sensitizers. Vapors of the volatile amines may cause conjunctivitis and visual disturbances.

Polyaminoamide hardeners have a less irritating effect on the skin and mucous membranes than the aliphatic and cycloaliphatic amine hardeners, but may cause sensitization.

Amide hardeners generally have only a slight irritant effect. Should the handling of these hardeners generate dust, measures should be taken to prevent inhalation.

The most commonly used reinforcement materials are carbon/graphite fibers. (The terms graphite and carbon are often used interchangeably.) This is due to the fact that many of the desired performance characteristics require the use of carbon/graphite fibers. Currently, these fibers are produced from three types of materials known as precursor fibers:

- polyacrylonitrile (PAN)
- rayon
- petroleum pitch

REINFORCEMENT FIBERS

Most of the reinforcing materials used in the industry have the potential to cause eye, skin, and upper respiratory tract irritation as a result of the mechanical-irritant properties of the fibers. The potential synergism has not been clearly defined. The chemical irritation caused by resins can compound the mechanical irritation caused by the fibers.

Carbon/graphite fibers

Carbon/graphite fibers dominate the advanced composites industry and may be made from any of three precursors, as discussed in Section C. However, the PAN-based carbon fibers are the predominant form in use today. It is important to ascertain which type of carbon-fiber precursor is used in order to evaluate the hazards.

Pitch-based carbon fibers may be associated with an increased risk of skin cancer, although the evidence is weak. PAN-based carbon fibers did not cause tumors when the same test was conducted. Standard mutagenicity tests conducted on PAN-based carbon fibers were negative.

The principal hazards of carbon-fiber handling are mechanical irritation and abrasion similar to that of glass fibers. Skin rashes are common and reportedly more severe than from glass fibers. Carbon fibers commonly in use are also greater than six micrometers in diameter, making them unlikely to be respirable. An ongoing survey of workers in a carbon-fiber production plant shows no pulmonary function abnormalities and no evidence of dust-related disease.

Carbon fibers may be coated with a material to improve handling, known generically as sizing. The sizing materials are typically epoxy resins. They may be biologically active and cause irritation or sensitization.

Aramid fibers

Aramid fibers are made from a polymer, poly(p-phenylenediamine terephthalate). Animal and human skin tests of KevlarTM aramid fibers show no potential for skin sensitization and low potential for irritation. While KevlarTM fibers are too large to be inhaled (12-15 mm), they may be fractured into respirable fibrils in some composite manufacturing processes. Industrial process monitoring shows that airborne respirable fibril levels are low in typical operations. Measured exposure levels from composite machining are typically below 0.2 fibrils per milliliter of air (0.2 f/ml), as an 8-hour, time-weighted average (TWA), while continuous filament handling generates less than 0.1 f/ml. The physical structure of aramid fibers makes it extremely difficult to generate airborne concentrations.

Glass fibers

Glass fibers, used as reinforcement in PMC processes, are a continuous-filament form and not the glass-wool (random) type. Practically all glass fibers for composite reinforcement are greater than six microns in diameter. Airborne fiber of this diameter does not reach the alveoli and is nonrespirable. Glass fibers break only into shorter fragments of the same diameter. Their diameter cannot be reduced by machining, milling, or other mechanical processes.

Mechanical irritation of skin, eyes, nose, and throat are common hazards associated with glass-fiber exposure. Continuous-filament glass fiber is not considered fibrogenic. Lung clearance mechanisms are effective for glass fibers.

In June 1987, the International Agency for Research on Cancer (IARC) categorized continuous-filament glass fibers as not classifiable with respect to human carcinogenicity. The evidence from human and animal studies was evaluated by IARC as insufficient to classify continuous-filament glass fibers a possible, probable, or confirmed cancer-causing material.

Like carbon fibers, glass fibers may also be coated with a sizing material to improve handling. Sizing materials may be epoxy resins, polyvinyl acetate-chrome chloride, polyvinyl acetate-silane, polyester-silane, or epoxy-silane compounds. These materials may be biologically active and cause irritation or sensitization

Dusts

Dusts may be generated in several ways in advanced composite processes. The most common dust-generating processes are machining and finishing of cured parts and in repair of damaged parts. Much of the dust generated in these processes can be very fine and should be considered respirable. Studies of some graphite-epoxy finishing operations found respirable fractions ranging from 25% to 100%.

More dust is usually generated in finishing and repair processes since large surface areas are involved. Grinding, routing and sanding are frequently used methods in both processes. The repair process may require the use of abrasive blasting as well as sanding to remove existing paint or coatings. Typically, a synthetic blasting agent, e.g., plastic media blast, is used. Ingredients of the paint or coating being removed, such as lead or chromates, may also be of concern. The repair process may also require cutting or sawing to remove the damaged part area, and both may generate significant amounts of airborne dust.

In general, studies on composite dusts indicate that:

- The dusts are particulate in nature and usually contain few fibers;
- The dusts are thermally stable up to 250 °C and exhibit a high degree of cure; and
- Toxicology studies indicate the dusts should probably be controlled at levels below the PEL for inert dust, but not approaching the PEL for crystalline quartz.

Solvents

Many of the solvents used in advanced composite processes are volatile and flammable. Most are skin and eye irritants, and some may be readily absorbed through the skin. Precautions must be taken when using organic solvents because they can facilitate the entry of toxic materials into the skin and organ systems. They may also enhance skin sensitization caused by the resin systems. Some (such as methyl alcohol) are poisonous, and all are capable of extracting fat from skin. Harmful effects from industrial exposures come principally from skin contact and inhalation.

Selection of the proper glove for protection is important. Permeation data are available for many industrial chemicals, especially solvents. However, in the case of resins and curing agents, not much data are available. This also is true for mixtures of solvents, as little or no testing has been done. Often the glove selection process is one of trial and error. If a skin rash or dermatitis is observed there are several possible causes:

- the wrong gloves may have been selected;
- improper work practices are being followed;
- the employee is deficient in personal hygiene practices; or
- adequate washing facilities are absent.

Several of the solvent classes most commonly found in the PMC workplace are listed below, along with general hazard information.

Several ketones are frequently found in PMC manufacture. These include:

- acetone (DMK)
- methyl ethyl ketone (MEK)
- methyl isobutyl ketone (MIBK)

These solvents may cause eye, nose, and throat irritation, and prolonged contact with the liquid may result in defatting of the skin and resultant dermatitis. In high concentrations, narcosis is produced with symptoms of headache, nausea, light-headedness, vomiting, dizziness, incoordination, and unconsciousness. Ketones are volatile and flammable. Acetone is a popular solvent used for cleanup and may be found around the workplace in containers for this purpose.

Some of the lower-boiling alcohols are sometimes used in composites manufacture. These include:

- methanol (methyl alcohol)
- ethanol (ethyl alcohol)
- isopropanol (isopropyl alcohol)

These alcohols do not usually present serious hazards in the industrial setting. Toxicity is usually related to irritation of the conjunctivae and the mucous membranes of the upper airway. Contact with the liquid may cause defatting of the skin and dermatitis. These alcohols are volatile and flammable.

Three chlorinated hydrocarbon compounds in particular are found in the composites workplace:

• methylene chloride (dichloromethane)

- 1,1,1-trichloroethane (methyl chloroform)
- trichloroethylene

Health effects typical of the group include irritation of the eyes and upper respiratory tract, dizziness, confusion, drowsiness, nausea, vomiting, and occasionally abdominal pain. Visual disturbances may also occur. Due to the solvents' defatting properties, repeated or prolonged skin contact with these liquids may cause dermatitis. Ability to depress the central nervous system is a characteristic property of all members of this group.

These solvents are not particularly flammable. Many manufacturers have replaced the ketones with the above hydrocarbon solvents to reduce the risk of flammability.

Other solvents that may occasionally be used are:

- toluene
- xylene
- tetrahydrofuran (THF)
- dimethylsulfoxide (DMSO)
- dimethylformamide (DMF)
- gamma-butyrolactone (BLO)
- n-methyl pyrrolidone (NMP)
- n-butyl acetate
- glycol ethers

Technical literature including MSDS's from the solvent supplier should be consulted about these or any chemicals used with advanced composites.

WORKPLACE CONTROLS

Good workplace controls are essential in controlling exposure to process materials. Many of the materials, particularly the resins, curing agents, and fibers, present a potential dermalexposure hazard. Many of the solvents and some of the curing agents present a potential inhalation hazard. Some materials present both a dermal and inhalation hazard. Ingestion may be a potential exposure hazard, but usually involves poor personal hygiene or contamination of eating facilities. The various types of workplace controls described below may typically be found in the advanced composite workplace.

ENGINEERING CONTROLS

Isolation

Isolation (e.g., isolated storage, separate process areas, enclosures, closed systems) and local exhaust ventilation are the primary engineering controls found in advanced composites processes. These controls can be found in:

- Resin mixing areas;
- Heated curing areas including autoclaves;
- Finishing and repair areas; and
- Controlling off-gases from exotherms

WORK PRACTICE CONTROLS

Work practices

Work practices, as distinguished from engineering controls, involve the way a task is performed. Some fundamental and easily implemented work practices that can be used to minimize exposures when working with advanced composites are:

- good employee training and education;
- following the proper procedures for production, process and control equipment;
- proper use, maintenance, and cleaning of personal protective equipment;
- good personal hygiene program;
- housekeeping;
- periodic inspection and maintenance of production, process and control equipment; and
- good supervision.

PERSONAL PROTECTIVE EQUIPMENT

Gloves, protective clothing, and eye protection may frequently be required, especially when working with resins, curing agents, and solvents. Selection of the proper protective materials should be based on permeation data, if available. This type of data are often available for the solvents used, but very little data are available for the resins and curing agents.

In many advanced composites processes several chemicals or mixtures are involved. There are essentially no permeation data available for chemical mixtures. This means that, in many cases, glove and clothing selection must be a trial and error process.

Generally, the resins are of a larger molecular size and so are less likely to permeate protective materials than the curing agents and solvents. The aromatic amine curing agents are particularly difficult to protect against. In some advanced composites processes, close hand work and contact is required, and a glove must provide good tactility. Often this type of glove provides the least protection against the resin and curing agent.

Eye protection can be provided by standard safety glasses with side shields, goggles, or a face shield, as needed.

Respiratory protection is not required in many advanced composites processes, due to the low vapor pressure of the materials involved. However, respirators may be required where:

- Airborne solvent levels are high;
- Dust levels are high (resin mixing, finishing, repair);
- Large surface areas and significant hand work are involved; and
- Exotherms are experienced.

ADMINISTRATIVE CONTROLS

Employee exposures also can be controlled by scheduling operations with the highest exposures at a time when the fewest employees are present.

Reference

Occupational Health and Safety Administration, Directive Number: TED 01-00-015

EXERCISE

1. What are common elements used for composite processing?

2. What is a function of resins in composites?

3. What are hazards of solvents used in advanced composites processes?

4. Which reinforcement fiber is commonly used in composite processing?

5. How dust is generated in advanced composite processes? What hazards occurs from dust?

6. List the most dangerous solvents used in advanced composite processes?

7. List out the three basic reinforcement fibers used in composite processing?

8. What health effects are caused by Aliphatic and Cycloadiphatic Amines?

9. During Handling of solvents if a skin rashes or dermatitis, what are its possible causes?

10. Which personal protective equipments will you use while working in composite lab?

PRACTICAL 2

OBJECTIVE: Study the process cycle and main parts of injection molding machine.

THEORY

INJECTION MOLDING MACHINE

An Injection molding machine, also known as an injection press, is a machine for manufacturing plastic products by the <u>injection</u> molding process. It consists of two main parts, an injection unit and a clamping unit and within the clamping unit there is an important component is mold assembly.

INJECTION UNIT

The injection unit is responsible for both heating and injecting the material into the mold. The first part of this unit is the hopper, a large container into which the raw plastic is poured. The hopper has an open bottom, which allows the material to feed into the barrel. The barrel contains the mechanism for heating and injecting the material into the mold. This mechanism is usually a ram injector or a reciprocating screw. A ram injector forces the material forward through a heated section with a ram or plunger that is usually hydraulically powered. Today, the more common technique is the use of a reciprocating screw. A reciprocating screw moves the material forward by both rotating and sliding axially, being powered by either a hydraulic or electric motor. The material enters the grooves of the screw from the hopper and is advanced towards the mold as the screw rotates. While it is advanced, the material is melted by pressure, friction, and additional heaters that surround the reciprocating screw. The molten plastic is then injected very quickly into the mold through the nozzle at the end of the barrel by the buildup of pressure and the forward action of the screw. This increasing pressure allows the material to be packed and forcibly held in the mold. Once the material has solidified inside the mold, the screw can retract and fill with more material for the next shot.

CLAMPING UNIT

Prior to the injection of the molten plastic into the mold, the two halves of the mold must first be securely closed by the clamping unit. When the mold is attached to the injection molding machine, each half is fixed to a large plate, called a platen. The front half of the mold, called the mold cavity, is mounted to a stationary platen and aligns with the nozzle of the injection unit. The rear half of the mold, called the mold core, is mounted to a movable platen, which slides along the tie bars. The hydraulically powered clamping motor actuates clamping bars that push the moveable platen towards the stationary platen and exert sufficient force to keep the mold securely closed while the material is injected and subsequently cools. After the required cooling time, the mold is then opened by the clamping motor. An ejection system, which is attached to the rear half of the mold, is actuated by the ejector bar and pushes the solidified part out of the open cavity.



Cycle Time

The time that is required for one complete cycle of a manufacturing process. Typically, the cycle time is the time required to produce a single part. However, many processes allow for the production of multiple parts per cycle. For example, a mould may contain multiple cavities.

Clamp Force

The force that is applied to mold by the molding machine in order to keep it securely closed while the material is injected. The clamp force is typically some factor of greater than the separating force, which is the outward force, exerted on the mold halves by the injected material.

Separating Force

The outward force exerted on the mold halves caused by the injection of molten materil during the mold filling stage.

INJECTION MOLDING CYCLE

The injection molding process occurs cyclically. Typical cycle times range from 10 to 100 seconds and are controlled by the cooling time of the thermoplastic or the currying time of the thermosetting plastic. The plastic resin in the form of pellets or powder is fed from the hopper and melted. In a reciprocating screw type injection molding machine, the screw rotates forward and fills the mold with melt, holds the melt under high pressure, and adds more melt to compensate for the contraction due to cooling and solidification of the polymer. This is called the hold time. Eventually the gate freezes, isolating the mold from the injection unit, the melt cools and solidifies. Next the screw begins to rotate and more melt is generated for the next shot. In the soak time the screw is stationary and the polymer melts by heat conduction from the barrel to the polymer. The solidified part is then ejected and the mold closes for the next shot.



PROCESS PARAMETER

Melt temperature

Melt temperature control is critical to maintain melt viscosity. This ensures sufficient flow, surface wetting and void coverage in the mold cavities. Temperature must be sufficiently high enough to maintain melt fluidity without resulting in burn.

Injection metering & pressure

Injection metering and pressure control is critical as to ensure sufficient flow of melt into the mold cavities and to compensate for shrinkages and material creeping back during setting that occur during the cooling stages.

Cooling

Cooling time must be optimizing for cycle time without compromising quality of the finish product. Too rapid cooling may cause insufficient coverage of the mold cavities. Slow cooling rate however may increase cycle time.

Feature size & aspect ratio

Features shape, size and the aspect ratio of the required product play a significant role in the optimize selection of the above parameters.

PROCEDURE

1. Plasticizing describes the conversion of the polymer material from its normal hard granular form at room temperatures, to the liquid consistency necessary for injection at its correct melt temperature.

2. Clamping is where the moving and fixed platens of the injection molding machine hold the mold tools together under pressure.

3. Injection is the stage during which this melt is introduced into a mold to completely fill a cavity or cavities.

4. Compression Pressure is maintained for a short time (dwell time) to prevent the material creeping back during setting (hardening).

5. Chilling is the action of removing heat from the melt to convert it from a liquid consistency back to its original rigid state. As the material cools, it also shrinks.

6. Ejection is the removal of the cooled, molded part from the mold cavity.

EXERCISE

1. Differentiate between cooling time, curing time, holding time and cycle time?

2. Define thermoplastic and thermosetting plastic, and enlist few thermoplastic used in injections molding machine?

3. Can we use same grade thermoplastic in both injections molding machine and extrusion machine, If not why?

PRACTICAL 3

OBJECTIVE: Preparation of composite by Hand layup process.

APPARATUS

Glass fiber mat, Mould for desired shape, Epoxy Resin and Hardener

THEORY

In the early days, the wet lay-up process was the dominant fabrication method for the making of composite parts. It is still widely used in the marine industry as well as for making prototype parts. This process is labor intensive and has concerns for styrene emission because of its open mold nature. In this process, liquid resin is applied to the mold and then reinforcement is placed on top. A roller is used to impregnate the fiber with the resin. Another resin and reinforcement layer is applied until a suitable thickness builds up.

It is a very flexible process that allows the user to optimize the part by placing different types of fabric and mat materials. Because the reinforcement is placed manually, it is also called the hand lay-up process. This process requires little capital investment and expertise and is therefore easy to use.

On a commercial scale, this process is widely used for making boats, windmill blades, storage tanks, and swimming pools. Because of its process simplicity and little capital investment, this process is widely used for making prototype parts. Test coupons for performing various tests for the evaluation of reinforcements as well as resins are made using this process. Simple to complex shapes can be made using this process.

Woven fabrics of glass, Kevlar, and carbon fibers are used as reinforcing material, with Eglass predominating in the commercial sector. Epoxy, polyester, and vinyl-ester resins are used during the wet lay-up process, depending on the requirements of the part. Polyester resin is the most common resin in building boats and other commercial items.

The mold design for the wet lay-up process is very simple as compared to other manufacturing processes because the process requires mostly a room-temperature cure environment with low pressures. Steel, wood, GRP, and other materials are used as mold materials for prototyping purposes. The mold can be a male or female mold. To make shower bathtubs, a male mold is used. In the boating industry, a single-sided female mold made from FRP (fiber-reinforced plastic) is used to make yacht hulls. The outer shell of the mold is stiffened by a wood frame. The mold is made by taking the reversal of a male pattern. Several different hull sizes can be made using the same mold. The length of the mold is shortened or lengthened using inserts and mold secondaries such as windows, air vents, and propeller tunnels.

A schematic of the wet lay-up process is shown in Figure 1, where the thickness of the composite part is built up by applying a series of reinforcing layers and liquid resin layers. A roller is used to squeeze out excess resin and create uniform distribution of the resin throughout the surface. By the squeezing action of the roller, homogeneous fiber wetting is obtained. The part is then cured mostly at room temperature and, once solidified, it is removed from the mold.



Fig: 1 Schematic of the wet lay-up process

PROCEDURE

The major processing steps in the wet lay-up process include:

A release agent is applied to the mold.

- 1. The gel coat is applied to create a Class A surface finish on the outer surface. The gel coat is hardened before any reinforcing layer is placed.
- 2. The reinforcement layer is placed on the mold surface and then it is impregnated with resin. Sometimes, the wetted fabric is placed directly on the mold surface.
- 3. Using a roller, resin is uniformly distributed around the surface.
- 4. Subsequent reinforcing layers are placed until a suitable thickness is built up.
- 5. In the case of sandwich construction, a balsa, foam, or honeycomb core is placed on the laminated skin and then adhesively bonded. Rear-end laminated skin is built similar to how the first laminated skin was built up.
- 6. The part is allowed to cure at room temperature, or at elevated temperature.

OBSERVATION

Surface Finish

Time consumption

CALCULATION

Mass of Reinforcement	=
Mass of Composite Product	=
Mass of Matrix	=
Volume Fraction of Fiber	=
Volume Fraction of Matrix	=

RESULTS

EXERCISE

1. What are the application areas of Hand layup process on commercial scale?

2. What are the roles of Matrix and Reinforcement in the composite part fabrication?

3.List some Matrix and Reinforcement materials that are used in Hand Lay-up Process?

4. What are the advantages and limitations of Hand Lay-up Process?

5. What is a mould? Which materials can be used to make a mould for Hand Lay-up Process?



PRACTICAL 4

OBJECTIVE: To determine hardness of polymers using Shore Hardness Tester

APPARATUS

Shore Hardness Tester, Test Sample of rubber and plastics

THEORY

The hardness testing of plastics is most commonly measured by the Shore (Durometer) test or Rockwell hardness test. Both methods measure the resistance of the plastic toward indentation. Both scales provide an empirical hardness value that doesn't correlate to other properties or fundamental characteristics. Shore Hardness, using either the Shore A or Shore D scale, is the preferred method for rubbers/elastomers and is also commonly used for 'softer' plastics such as polyolefins, fluoropolymers, and vinyls. The hardness numbers are derived from a scale. Shore A and Shore D hardness scales are common; with the A scale being used for 'softer' rubbers while the Shore D scale is used for 'harder' ones. The Shore A Hardness is the relative hardness of elastic materials such as rubber or soft plastics. It can be determined with an instrument called a Shore A durometer. If the indenter completely penetrates the sample, a reading of 0 is obtained, and if no penetration occurs, a reading of 100 results. The reading is dimensionless. Each Durometer Type is made to a specific scale (i.e. A,B,C,D) and is capable of producing a value between 0 and 100.



Fig: 2 Digital and Analog Shore Hardness Testers

The Shore hardness is measured with an apparatus known as a Durometer and consequently is also known as 'Durometer hardness'. The hardness value is determined by the penetration of the Durometer indenter foot into the sample. Because of the resilience of rubbers and plastics, the hardness reading my change over time - so the indentation time is sometimes reported along with the hardness number. The ASTM test number is ASTM D2240 while the analogous ISO test method is ISO 868. The test specimens are generally 6.4mm (¼ in) thick. It is possible to pile several specimens to achieve the 6.4mm thickness, but one specimen is preferred.

The results obtained from this test are a useful measure of relative resistance to indentation of various grades of polymers. However, the Shore Durometer hardness test does not serve well as a predictor of other properties such as strength or resistance to scratches, abrasion, or wear, and should not be used alone for product design specifications.

PROCEDURE

- The specimen is first placed on a hard flat surface.
- The indenter for the instrument is then pressed into the specimen making sure that it is parallel to the surface.
- The hardness is read within one second of firm contact with the specimen.

OBSERVATION

MATERIAL	
SCALE	
HADDNESS	
nakdness	
MEAN HARDNESS	

RESULTS

EXERCISE

1._____ scale being used for softer and the _____ scale being used for harder materials

2. If the indenter completely penetrates the sample, a reading of ______ is obtained, and if no penetration occurs, a reading of _____ results.

3. The ASTM ______ is the Standard Test Method for Rubber Property which is analogous to ISO test method _____.

4. The test specimens are generally _____ mm thick.

5. Can we use Brinell and Rockwell Hardness Test for measuring hardness of polymers?

6. Name some polymers that can be tested using 'Scale A'.

7. Name some polymers that can be tested using 'Scale D'.

PRACTICAL 5

Objective

Joining of the fractured surface of fiber reinforced composites through Hand Layup process

Theory

Introduction

The bonding of FRP is a process requiring some degree of skill, which can readily be acquired by practicing the following recommended procedures. The methods described here can be applied to the bonding, welding, joining or repair of any fractured FRP'S

Recommended Tools

- 1. Sanding and Cutting Tools
- 2. Grit paper
- 3. Carbide grit cutting wheel

Bonding Tools

- 1. Vinyl ester or polyester promoted resin
- 2. MEKP Catalyst
- 3. Glass fiber reinforcing material; Chopped strand mat,
- 4. Acetone
- 5. Paint brushes from 4 to 6 inches in width
- 6. Cans for soaking tools in acetone
- 7. Utility knife or Scissors for cutting mat, and roving
- 8. Paper cups and half gallon plastic containers for mixing resin
- 9. Wooden or plastic paint sticks for mixing resin
- 10. Rubber gloves, safety glasses, respirators, and dust masks designed for FRP work.

Procedure

Surface Preparation

Thoroughly clean the area with a solvent agent of a brush to be joined to remove surface contamination. All of the surface area to be covered with fiberglass reinforcement and resin must be thoroughly sanded and roughened with a power sander. Make sure the glossy resin finish is removed. Thoroughly clean roughened areas with a brush. The surface must be dry. Apply resin and fiberglass as soon as possible after preparation to prevent possible recontamination of the prepared area.

Mixing Resin

Under no circumstance should the catalyst and promoter be mixed in the same container or poured into the resin at the same time. When mixed together, these two chemicals can react explosively. Resin used for repairing or joining should be the same type as the resin used in the original equipment. Weigh sufficient resin into a suitable container. (Avoid using more than one quart at a time.) Use this weight to determine the amount of promoter and/or catalyst. Measure the required amount of catalyst into a separate graduate and mix well into the promoted resin. See gel and cure data for vinyl ester resin included within this package. The process is affected dramatically by application temperature, and extra care is required at

temperatures below 40 degrees F to ensure adequate resin cure. High outdoor temperatures can also complicate joining and repair procedures by advancing the gelation and cure of the resins, leaving the operator with inadequate working time. We recommend that operators working under conditions of temperature extremes check the gelation and cure of a resin sample at the working temperature prior to starting any field joints.

Hardening or Cure

Resin must be allowed to harden or cure. The time to cure will vary according to weather conditions, temperatures and exact amount of promoter and/or catalyst used. An external heat source such as an infrared heat lamp will decrease hardening time and may be a necessity in cold weather. Generally, work should not be done at temperatures below 55 degrees Fahrenheit unless an outside source of heat is applied. Since most resins are FLAMMABLE liquids, external heat sources should be used with caution. Care must be exercised when using an external heat source to prevent overheating which can cause cracking and/or crazing or discoloration. Acetone lightly rubbed onto the exposed resin surface will determine if the system is adequately cured. If surface softens or becomes tacky, an external heat source can be applied to fully cure the resin. If surface does not cure, the entire bond process must be repeated.

EXERCISE

1. Why cleaning of the fractured surface is important before applying any reinforcement?

2. Which reinforcement fiber is commonly used in composite processing?

3. List out the three basic reinforcement fibers used in composite processing?

PRACTICAL 6

Objective

Preparation of composite part through VARTM process

Theory

Ever-increasing demand for faster production rates has pressed the industry to replace hand layup with alternative fabrication processes and has encouraged fabricators to automate those processes wherever possible. **Vacuum assisted resin transfer molding** (VARTM) is likely the most common acronym of all used in the discussion of low pressure closed mold reinforced composite molding. The often used VARTM acronym is most accurately applied to the process of vacuum infusion, that is where the composite is molded using a rigid mold to provide part geometry and a thin flexible membrane over the fiber, with outer atmospheric pressure compressing the fiber tight against the rigid mold surface. It should be noted however that VARTM simply means to add vacuum at the exit vent of the molding tool, thus any form of resin transfer molding in which vacuum is applied to the vent would qualify for the VARTM association.

How VARTM process is a variant of RTM process

Vacuum-assisted resin transfer molding (VARTM) refers to a variety of related processes, which represent the fastest growing new molding technology. The salient difference between VARTM type processes and standard RTM is that in VARTM, resin is drawn into a preform through use of a vacuum, rather than pumped in under pressure. VARTM does not require high heat or pressure. For that reason, VARTM operates with low-cost tooling, making it possible to inexpensively produce large, complex parts in one shot.

Operation

In the VARTM process, fiber reinforcements are placed in a one-sided mold, and a cover (rigid or flexible) is placed over the top to form a vacuum-tight seal. The resin typically enters the structure through strategically placed ports. It is drawn by vacuum through the reinforcements by means of a series of designed-in channels that facilitate wetout of the fibers. Fiber content in the finished part can run as high as 70 percent. Current applications include marine, ground transportation and infrastructure parts.

Procedure

- 1. Manufacture perform
- 2. Perform Lay Up and Compression via Vacuum
- 3. Checking for the Vacuum leaks
- 4. Resin injection
- 5. Resin cure
- 6. Demoulding

Observations

Applied Pressure (Bar)	
Infusion Time (sec)	
Curing Time (hour)	



EXERCISE

1. How VARTM process is a variant of RTM process

2. What is a function of distribution medium in VARTM process?

3. Which reinforcement fiber is commonly used in composite processing?

4. The properties results from the VARTM process are superior then Hand Layup discuss?

5. What is the function of Peel Ply in VARTM process?

PRACTICAL 7

OBJECTIVE: Determine the tensile properties of polymers & composite material.

STANDARD METHOD:

ASTM D412 (for elastomers) ASTM D638 (for plastics)

EQUIPMENT:

Tensile Testing Machine Instron 4301



Universal Testing Machine

SPECIMEN MATERIAL:



SPECIMEN GEOMETRY:

Specimens may be in any shape of dumbbells, rings or straight pieces of uniform crosssectional area. The test specimen is to be cut from a flat sheet not less than 1.3 mm (0.05 in) nor more than 3.3 mm (0.13 in) thick of any size. The specimen should be free of surface roughness, fabric layers, etc. Distance between gauge lengths should be 50 ± 0.5 mm, and the distance between grips should be 115 ± 1 mm.

THEORY:

Tensile Set: The extension remaining after a specimen has been stretched and allowed to retract in a specified manner, expressed as a percentage of the original length.

Tensile Strength: The maximum tensile stress applied in stretching a specimen to rupture. It is an intensive property therefore its value does not depend upon the size of the test specimen. **Ultimate Elongation:** The elongation at which rupture occurs in the application of continued tensile stress.

Yield Point: That point on the stress-strain curve, short of ultimate failure, where the rate of stress with respect to strain, goes through a zero value and become negative.

Yield Strain: The level of strain at the yield point.

Yield Stress: The level of stress at the yield point.

The tensile test is one of the most common procedures used to measure a material's behavior under pulling forces. The data from a tensile test can often be used directly in the design or to evaluate a design. The primary objective of the tensile test is to determine the maximum load a specimen can resist before taking a permanent set or before rupture occurs.

PROCEDURE:

- 1. Prepare specimens for testing.
- 2. Three measurements should be made for the thickness, one at the center and one at each end of the reduced section. The median of the three measurements should be used as the thickness in calculating the cross-sectional area.
- 3. Measure the width of the specimen.
- 4. Measure the length of the specimen.
- 5. Place the dumbbell in the grips of the testing machine, using care to adjust the specimen symmetrically to distribute tension uniformly over the cross-section.
- 6. Start the machine and note the distance between the bench marks; the two marks placed on the specimen and used to measure the elongation or strain are called the bench marks; taking care to avoid the parallax error.
- 7. Record the force at the elongation specified for the test and at the time of rupture.
- 8. At rupture measure and record the elongation to the nearest 10%.
- 9. Repeat the above procedure for different specimen materials & record the results.



FORMULA:

Cross-sectional Area:

 $A = T \times W$ Where; A = cross-sectional area, cm²T = Thickness, cmW = Width, cm

Tensile-strength:

$$TS = \frac{F}{A}$$

Where;

TS= tensile strength at rupture, kg_f/cm^2 F= the force magnitude at rupture, kg_f A= cross-sectional area of unstrained specimen, cm² Elongation:

$$E = \frac{100(L - Lo)}{Lo}$$

Where;

E= Elongation in %

L= Observed distance between bench marks on the extended specimen, mm $L_0=$ original distance between bench marks, mm

OBSERVATION:

S.No.	Specimen Material	Mean Thickness (mm)	Mean Width (mm)	Gauge Length (mm)	Load (kg _f)	Extension (mm)

CALCULATIONS:

S.No.	Specimen Material	Area (cm ²)	Tensile Strength (kg _f /cm ²)	Tensile Elongation (%)
1.				
2.				
3.				

RESULT:

The tensile strength of samples was found to be:



EXERCISE

1. What is the significance and use of the tensile strength and percentage elongation?

2.	What is the effect of the extension rate on the tensile properties of a material?
3.	What is the effect of the temperature of the test on the properties of a material?
4.	Distinguish between the proportional limit and the elastic limit of a material. Which is more important indicator of a material's mechanical behavior?
5. Sk	tetch the stress-strain curve and identify the following on the curve:
	a) Elastic limit
	b) Plastic region

- c) Yield point
- d) Fracture point
- e) Ultimate tensile stress
- f) Proportional limit

PRACTICAL 8

OBJECTIVE:

Determine the flexural strength of Plastic specimen by three-point loading system.

STANDARD METHOD:

ASTM D790

EQUIPMENT:

Universal Testing Machine, 3 point bending jig



ASTM D 790 SPECIMEN MATERIAL:

SPECIMEN GEOMETRY:

The specimens may be cut from sheets, plates, or molded shapes.

A variety of specimen shapes can be used for this test, but the most commonly used specimen is typically $1/8 \times 1/2 \times 4$ inches. For all tests the test span shall be 16 (tolerance +4 or -2) times the depth of the beam.

THEORY:

Flexure: Bending action in beam is defined as flexure. Bending may be accompanied by direct stress, transverse shear, or torsional shear. The term flexure applies to bending tests of beams subjected to transverse loading.

Flexural is the bending of a material specimen under load. The strength that the specimen exhibits is a function of the material of which the specimen is made and the cross sectional geometry of the specimen. Flexural properties may vary with specimen depth, temperature, atmospheric conditions and the rate of straining. Under loading conditions the material shows both tension and compressive deformations. The upper surface of the material is under compressive deformation and the lower surface of the material is under tension deformation.

The flexural test measures the force required to bend a beam under three point loading conditions. The data is often used to select materials for parts that will support loads without flexing. The test beam is subjected to compressive stress at the concave surface and tensile stress at the convex surface. The flexural strength is expressed in terms of modulus of rupture. Flexural modulus is used as an indication of a material's stiffness when flexed.

The parameters for this test are the support span, the speed of the loading, and the maximum deflection for the test. These parameters are based on the test specimen thickness and are defined differently by ASTM. For ASTM D790, the test is stopped when the specimen reaches 5% deflection or the specimen breaks before 5%, whichever occurs first.

PROCEDURE:

- 1. Use an untested specimen for each measurement.
- 2. Measure the width and depth of the specimen to the nearest 0.03 mm (0.001 in) at the center of the support span.
- 3. Determine the support span. Measure the span accurately to the nearest 0.1 mm (0.004 in.). Use the measured span for all calculations.
- 4. Align the loading nose and supports so that the axes of the cylindrical surfaces are parallel and the loading nose is midway between the supports.
- 5. Center the specimen on the supports, with the long axis of the specimen perpendicular to the loading nose and supports.
- 6. Apply the load to the specimen at the specified rate and take simultaneous load-deflection data.
- 7. Measure deflection by a gage under the specimen in contact with it at the center of the support span.
- 8. Terminate the test if the maximum strain in the material has reached 0.05 mm/ mm (in. /in.) or 5% deflection.
- 9. Repeat the above mentioned procedure for different specimens.
- 10. Record the results.

FORMULA:

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

Where;

S= stress in the outer fibers at mid span, MPa (psi)

P= load at a given point on the load deflection curve, kg_f

L= support span, mm

b= width of beam tested, mm

d= depth of beam tested, mm

OBSERVATION:

Span =	mm				
S. No.	Specimen Material	Mean Thickness (mm)	Mean Width (mm)	Speed (mm/min)	Load (kg _f)

CALCULATION:

S.No.	Specimen Material	Flexural Stress (kg _f /cm ²)

RESULT:

The Flexural Stress of specimen was found to be:

1	_ =	$kg_{\rm f}/cm^2 =$	MPa
2	_ =	$kg_f/cm^2 =$	MPa
3	_ =	$kg_{\rm f}/cm^2 =$	MPa

EXERCISE

- 1. What is the significance and use of flexural strength?
- 2. What is the effect of the following on the flexural properties of a material?
 - i. Depth of material.
 - ii. Temperature of material.
 - iii. Rate of straining.
 - iv. Span length.

- 3. Why "lengthwise" rather than "crosswise" position of specimen is used in the test?
- 4. What is the deformation of the material during loading conditions? Elastic or plastic?

PRACTICAL 9

OBJECTIVE:

Preparation of standard vulcanized molds of rubber.

EQUIPMENT:

Vulcanizer



THEORY:

Vulcanization is a chemical process for converting rubber into more durable materials via the addition of sulfur. These additives modify the polymer by forming cross links (bridges) between individual polymer chains. The vulcanized material is less sticky and has superior mechanical properties.

Apart from adding sulfur different compounds like plasticizers, antioxidants, activators, accelerators and fillers are also added. There function is defined below:

Plasticizers: Plasticizers or dispersants are additives that increase the plasticity or fluidity of the material to which they are added.

Antioxidants: An antioxidant is a molecule capable of inhibiting the oxidation of other molecules. Oxidation reactions can produce free radicals. In turn, these radicals can start chain reactions. Antioxidants terminate these chain reactions by removing free radical intermediates, and inhibit other oxidation reactions.

Activators: These are the compounds responsible for activating the process of curing.

Accelerators: The use of accelerators and activators lowers the activation energy of vulcanization reaction to 80-125kJ/mole from 210kJ/mole which is necessary if we use 'Sulfur' alone. Accelerators and activators break sulfur chains.

Fillers: These are the materials added in rubber to reduce cost and give strength. Common filler used in rubber industry is Carbon black.

PREPARATION OF STANDARD VULCANIZED MOLDS OF RUBBER

VULCANIZATION EQUIPMENTS

Press

The press should be capable of exerting a pressure of not less than 3.5 MP on the total cross sectional area of the cavities of the mold curing the entire period of vulcanization.

Mold

The size of the mold is 4 by 4 inches.

Rubber

Rubber is in the form of sheets. Rubber sheets are 30mm x 30 mm x approx. 6mm.

VULCANIZATION PROCEDURE:

- 1. Bring the mold to curing temperature within 0.5 °C in the closed press, and hold at this temperature for at least 20 min before the unvulcanized sheets are inserted. Verify the temperature of the mold by means of a thermocouple or other suitable temperature measuring device.
- 2. Open the press and insert the unvulcanized sheets into the mold and close the press in the minimum time possible. When the mold is removed from the press to inset the sheets, take precautions to prevent excessive cooling of the mold by avoiding contact with cool metal surfaces or by exposure to air drafts.
- 3. Consider the time of vulcanization to be the period between the instant the pressure is applied fully and the instant the pressure is released. Hold the mold under minimum pressure of 3.5 MPa on the cavity areas during vulcanization.
- 4. Open the press, immediately remove the vulcanized molds from the mold and cool in water (room temperature of lower) or on a metal surface for 10 to 15 min.

OBSERVATIONS:

Temperature = _____°C Pressure = _____ MPa Curing Time = _____ minutes

EXERCISE

1. Rubber cannot be used in raw or natural state. Why? What can be done to improve the properties of rubber?

- 2. Is there any cross linking agent other than sulfur?
- 3. Differentiate between curing and crosslinking?
- 4. Is it necessary to add anti-oxidant? Why?
- 5. A car is standing in sunlight for several days. The cracks appear on its tires. Explain reason?

PRACTICAL 10 Objective: Preparation of particulate composite

Theory

Particle-reinforced composites

This class of composites is most widely used composites mainly because they are widely available and cheap. They are again two kinds: dispersion-strengthened and particulate-reinforced composites. These two classes are distinguishable based upon strengthening mechanism – dispersion-strengthened composites and particulate composites.

In dispersion-strengthened composites, particles are comparatively smaller, and are of 0.01-0.1µm in size. Here the strengthening occurs at atomic/molecular level i.e. mechanism of strengthening is similar to that for precipitation hardening in metals where matrix bears the major portion of an applied load, while dispersoids hinder/impede the motion of dislocations. Examples: thoria (ThO₂) dispersed Ni-alloys (TD Ni-alloys) with high-temperature strength; SAP (sintered aluminium powder) – where aluminium matrix is dispersed with extremely small flakes of alumina (Al₂O₃)

Particulate composites are other class of particle-reinforced composites. These contain large amounts of comparatively coarse particles. These composites are designed to produce unusual combinations of properties rather than to improve the strength.

There are many different forms of particulate composites. The particulates can be very small particles (< 0.25 microns), chopped fibers (such as glass), platelets, hollow spheres, or new materials such as carbon nano-tubes. In each case, the particulates provide desirable material properties and the matrix acts as binding medium necessary for structural applications. The principal advantage of particle reinforced composites is their low cost and ease of production and forming.

Particulate composites are usually made of all three conventional engineering materials, namely-metals, polymers and ceramics.

E.g. Tungsten carbide(WC) or titanium carbide(TiC) embedded cobalt or nickel based cutting tools.

Aluminium alloy castings containing dispersed SiC particles are widely used for automotive applications including pistons and brake applications.

Equipment

- i. Spatula
- ii. Mold
- iii. Glass beaker
- iv. Ultrasonication process

Material used

1)

2)

Experimental Procedure

Calculation

Hardness

Result

EXERCISE

- 1. What are the advantages of particlualte composite?
- 2. In which material particulate composite can be used?
- 3. What are the main applications of particulate composite?



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NED University of Engineering & Technology Department of _____ Engineering Course Code and Title:

Psychomotor Domain Assessment Rubric-Level P3						
C1-:11 C at a	Extent of Achievement					
Skill Sets	0	1	2	3	4	
Equipment Identification Sensory skill to <i>identify</i> equipment and/or its component for a lab work.	Not able to identify the equipment.				Able to identify equipment as well as its components.	
Equipment Use Sensory skills to <i>demonstrate</i> the use of the equipment for the lab work.	Doesn't demonstrate the use of equipment.	Slightly demonstrates the use of equipment.	Somewhat demonstrates the use of equipment.	Moderately demon strates the use of equipment.	Fully demonstrates the use of equipment.	
Procedural Skills Displays skills to act upon sequence of steps in lab work.	Not able to either learn or perform lab work procedure.	Able to slightly understand lab work procedure and perform lab work.	Able to somewhat understand lab work procedure and perform lab work.	Able to moderately understand lab work procedure and perform lab work.	Able to fully understand lab work procedure and perform lab work.	
Response Ability to <i>imitate</i> the lab work on his/her own.	Not able to imitate the lab work.	Able to slightly imitate the lab work.	Able to somewhat imitate the lab work.	Able to moderately imitate the lab work.	Able to fully imitate the lab work.	
Observation's Use <i>Displays</i> skills to use the observations from lab work for experimental verifications and illustrations.	Not able to use the observations from lab work for experimental verifications and illustrations.	Slightly able to use the observations from lab work for experimental verifications and illustrations.	Somewhat able to use the observations from lab work for experimental verifications and illustrations.	Moderately able to use the observations from lab work for experimental verifications and illustrations.	Fully able to use the observations from lab work for experimental verifications and illustrations.	
Safety Adherence Adherence to <i>safety</i> procedures.	Doesn't adhere to safety procedures.	Slightly adheres to safety procedures.	Somewhat adheres to safety procedures.	Moderately adheres to safety procedures.	Fully adheres to safety procedures.	
Equipment Handling Equipment care during the use.	Doesn't handle equipment with required care.	Rarely handles equipment with required care.	Occasionally handles equipment with required care.	Often handles equipment with required care.	Handles equipment with required care.	
Group Work Contributes in a group based lab work.	Doesn't participate and contribute.	Slightly participates and contributes.	Somewhat participates and contributes.	Moderately participates and contributes.	Fully participates and contributes.	
Laboratory Session No Date:						

Weighted CLO (Psychomotor Score)	
Remarks	
Instructor's Signature with Date:	