A Technical Review on Deep Cryogenic Treatment on FRP Composite Materials & Experimental Analysis of its Effect

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Abstract— FRP composite materials offersvery good mechanical properties. The most valuable properties arecorrosion property, high toughness and strength,strength to density ratio, etc. During its service operation, it has to face many environmental conditions, different temperature etc. The present review based on the performance of FRP composites in different environment, different temperature and what will be its effect that is discussed here in this. Formation of irregularity due to separation or debonding and crack formation due to residual stresses, thermal shocks etc.
Key words: FRP composite, Mechanical behavior,Cryogenic exposure.

I. INTRODUCTION

Composites are the materials which consists of two or more physically and chemically separate phases parted by an interface. Composites have presently become one of the essential parts in today's materials. They provide promising mechanical properties like high strength to weight ratio, high corrosion resistance, low weight and many more. The major two phases include a) Matrix phase and b) Dispersed phase i.e. reinforcement. Matrix phase is the primary phase which is soft and ductile while the dispersed phase is the secondary phase (stronger than matrix) embedded in matrix. Composites can be classified on basis of matrix phase as Metal Matrix Composites (MMCs), Polymer Matrix Composites (PMCs) and Ceramic Matrix Composites (CMCs).

Composites are also classified on the basis of Reinforcement as a) Particulate composite, b) Fibrous composites and c) Laminated composites. Polymer Matrix Composites find application in Aerospace structures, Marine equipment, Parts of Automobile bodies, biomedical applications, Sports goods etc. The commonly used matrix and fibers for PMCs include Epoxy and Glass/Carbon/Kevlar respectively. The methods for fabricating the composites are Hand Lay-Up method, Bag Moulding Process, Pultrusion, Filament Winding, Preformed Moulding Compounds, Resin Transfer Moulding, Injection Moulding etc. Among these the most common method composites for fabricating the PMC is the Hand Lay-Up Process.

Another class of composite used presently is Hybrid composite. In hybrid there is either incorporation of two or more types of fibers or using nano fillers with matrix i.e. either fiber hybridisation or matrix hybridisation respectively. The behavior of a hybrid composite will be the weighted average of the individual components. Using hybrid composites with two or more types of fibers, the lack of properties in one fiber can be complemented with the other fibers. For example using Glass fiber reinforced Polymer (GFRP) composite can result in a soft laminate as compared to the Carbon fiber reinforced Polymer (CFRP) composite due to the high strength of carbon fibers. Hence in hybrid composite we use glass and carbon fibers both for appropriate properties. Likewise CFRP composite has a less impact resistance as compared to GFRP composite. Proper material design can lead to an improved composite. The types of hybrid composites include (1) Interply or tow by tow hybrid, (2) Sandwich hybrids, (3) Laminated hybrids, (4) Intimately mixed hybrids and (5) Intraply laminated. The judicious designing of hybrid composites result in a good balance of properties along with incurred cost. Environments like high temperature, low temperature, UV radiation exposure, humid environments etc. can cause degradation in mechanical properties of FRP composites and activation of micro failure mechanisms which ultimately results in composite failure [1].

The term composite stipulate a very broad class of materials, made of several (at least two) components. By opposition to alloys, the composite constituents are generally distinct at the macroscopic level. However, composite materials are often engineered to answer specific requirements. Commercial composites used in large markets such as, automotive components, boats, consumer goods and corrosion-resistance industrial parts. Advanced composites, initially developed for the military aerospace marked offer performance superior to that of conventional structural metals and now find applications in satellites, aircraft, sporting goods and in the energy sector in oil and gas exploration and wind turbine construction. Aircraft doors, fairings and interior panels are perfect for the FRP technology. Other potential applications could include the horizontal tail leading edge, pylon stub fairing and engine cowl panel troop door deflector. Largest aircraft the Airbus A380 have eleven times greater than the length and over 1000 times the weight of the Wright's 1903 Flyer, this super-jumbo will be the first airliner to have carbon fiber composite Centre-wing box. Besides that the rear pressure bulk-head, critical to passenger safety, will be of CFRP, as will the entire rear fuselage aft of the bulkhead. This is one of the largest carbon fiber parts yet produced by Airbus. The composite A500 carbon Aero from Adam Aircraft has now flown successfully in mid-2003. In helicopter, the use of composites material in several critical applications as Boeing-Sikorsky Comanche helicopter and Bell-Boeing V22 Osprey tiltrotor. The parts including exhaust doors, shrouds for transmission and tail rotor blades. That's why the composite deck is expected to last for more than 5o years with little to no maintenance. Currently employed to manufactures a family car where the metal bumpers, radiator end tanks, door handles and front and rear ends are substituted by engineering plastics [2].

World's oldest composite industry was found in Russia. Looking upon the future of this, Russians are fighting against few constrains of the gravity with the composite applications and there are some exploring opportunities are available. Russia has a very comprehensive large distance rail road network and major cities underground transport systems. As more and more cargo and people travel by rail, there is strong need for refurbishing the rolling stock. The coaches and wagons are very cold in temperature and face severe corrosion problems. It seems that composites material now the best choice for the refurbish old system.

A recent example is the Bridge-in-a-Backpack for 2014 Winter Olympics in Russia, an innovative inflatable composite-concrete arch bridge, which was developed to reduce construction time and costs, increase lifespan, reduce maintenance costs and reduce the carbon footprint of bridge construction. Polymer matrix key to an emerging breed of racing powerboats (V24) designed to make sport accessible. Here the hull bottom is stepped in two places to minimize wetted area as the boat rises in the water at speed and to help aerate the water's surface layer, so providing a more forgiving ride. Windmill blades made of 7 FRP are exposed to a large number of sustained and occasional environmental loads such as bending, vibrations, UV, cold and heat, moisture and also impact loading. Such loads combined with large dimensions perfectly illustrate the challenge of durability assessment of composite materials.

The fibrous polymeric composites at cryogenic temperature undergo inelastic deformation and damage when it subjected to mechanical loading. Despite such a restriction, the field of polymer composites still remains broad. Indeed, the nature and geometry of the materials constituents define to a large extent the response of the composite to external mechanical and environmental loads. At low temperature, the material might have some nucleating cracks and delaminated areas possibly by misfit strains at the interface region due to difference in coefficient of thermal expansion. In other aspect, matrix resin behaves brittle at low temperature unless it is modified by other modifier agent to increase its toughness value and retaining its strain capabilities. However, at low temperature the performance of fiber is not well-evaluated and critically characterized. Unless and until a complete and comprenshive understanding about the constituents (fiber, matrix resin and interphase) of FRP composites is being assessed and explored, the full potential of these material may not be fully utilized at cryogenic applications.

II. LITERATURE REVIEW

2.1 SanghamitraSethi*,Rathore D.K., Ray, B.C. (2014) [3] studied Effects of temperature and loading speed on interface-dominated strength in fibre/polymer composites: An evaluation for in-situ environment:

A. Glass fibre /epoxy composites:

1) Interlaminarbehavior

The variation of ILSS for glass/epoxy composite system in-situ conditioned at +50° C, +100° C, -50° C and -100° C temperatures, and tested at 1, 100, 200, 500, 700, and 1000 mm/min loading rates, is shown in Fig.

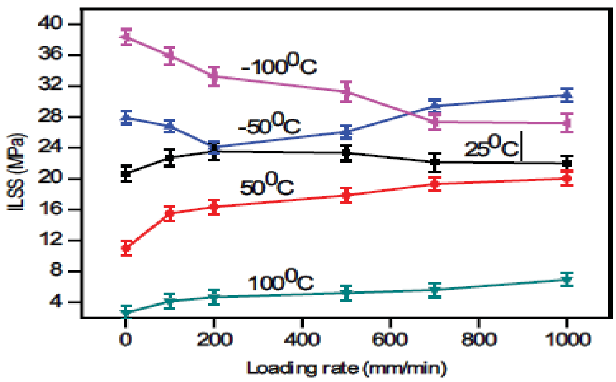

Fig. 2.1: Variation of ILSS with loading rate for glass/epoxy composite system at different temperatures.

The ILSS values at -100º C temperatures are better as compared to other conditioning temperatures but as the loading rate increased the ILSS decreased. The maximum ILSS for glass/epoxy composite is about 38.37 MPa, obtained at -100° C temperature and 1 mm/min loading speed, with an increase of 85.72% than the ILSS value (20.66 MPa) obtained at ambient temperature at 1 mm/min. Greater value of shear strength at low loading speed can be attributed to longer relaxation time resulting in improved interfacial integrity of the composite material. Higher crosshead speed during testing minimizes the relaxation process at the crack tip. This could be the reason for reduced ILSS values at higher crosshead speed. At -50° C temperature, initially the ILSS decreases from 1 mm/min to 200 mm/min and then increased with further increase in loading rate. The slight fall in the value at 200 mm/min conditioning could be related to the lower degree of cryogenic compressive stresses at fiber/matrix interface. At +50º C and +100º C temperatures the ILSS increased with increasing loading rate. The reason may be the induced thermal stresses in the matrix region. Thermal stress induced micro-cracks in the polymer matrix and/or, at the fiber/matrix interface may possibly grow without blunting at a steady state. Some micro cracks turn to potential cracks at low loading rates and cause significant reduction in inter laminar shear strength of the composite system while as the loading rate increases the time available to propagate the micro cracks is less. This can be attributed to higher ILSS at higher loading rates at these above-ambient temperatures. The effects of micro cracks and fiber breakage can nucleate the other form of damage such as delamination hence degradation in the thermos mechanical properties of the composite occurred. This interfacial separation caused by the delamination may lead to premature buckling of the laminates at high temperature. Further the effect of temperature on the ILSS is shown in Fig. 2.2 at 1 mm/min crosshead velocity.

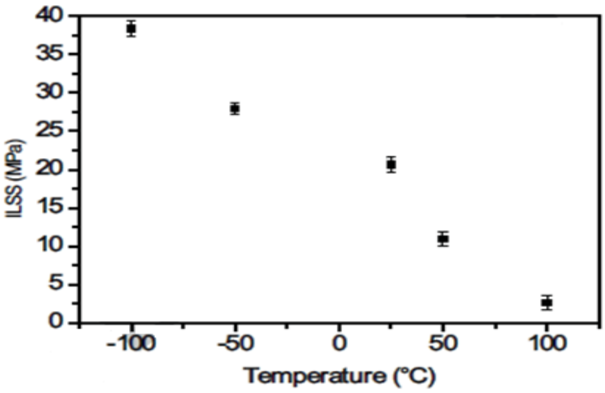

Fig. 2.2: Interlaminar shear strength of glass/epoxy composite at 1 mm/min for different temperature.

B. Carbon fibre /epoxy composites

1) Interlaminarbehavior

The interlaminar shear behavior of woven fabric carbon fibre/epoxy composite with loading rate at different temperature is shown in Fig. 2.3

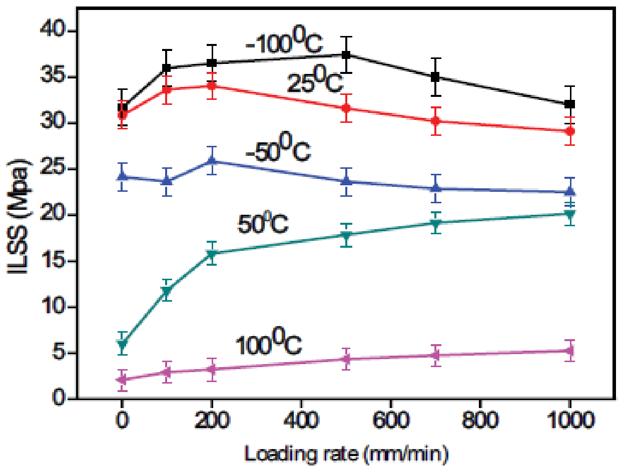

Fig. 2.3: variation of interlaminar shear strength with different loading rates at different temperatures for carbon fibre/epoxy composite system.

Interlaminar shear strength (ILSS) is one of the most important interfacial properties for composites. To better understand the interfacial strength between the carbon fiber/epoxy composites, three-point short beam shear test method was used to evaluate the interlaminar shear strength of the composites. It is readily observed that at -100° C temperature the carbon fiber/epoxy composites possess better ILSS compared to other testing temperatures. At -100° C temperature the variation of ILSS with loading rate is shawn in fig. 2.3. As the rate of loading increases the ILSS of the composite also increases up to 500 mm/min but, after 500 mm/min shear values decreases because micro crack density has exceeded the critical crack density for delamination. The energy release rate monotonically decreases as the delamination failure grows. Thus ILSS value decreases with increasing loading speed after 500mm/min. Here matrix behaves as brittle in nature which reduces the effective strain to failure and it is the source of matrix cracking. But at 200mm/min the shear value decreases with loading rate. From the relaxation behavior the thermal pre-stress would vanish after sufficient period of time. But with increasing loading speed the shear value shows no significant changes due to some microcracks behaves less dangerous since their stress concentrations are small. The laminates failed by very little delamination or no delamination because of low crack densities. However at high temperature +50° C and +100° C there is a significant change in ILSS values with loading speed. These results are probably due to the shear band propagation in matrix resin at high temperature. Here crack propagation in matrix resin prone to by crack jumping (unstable or stick-slip) mode at slow loading rate. Yield stresses decreases with decreasing strain rates and increasing temperature, stick-slip crack growth may be attributed to plastic deformation at the crack tip prior to crack initiation. Thus stress intensity factor is dependent on temperature and time. The size of the crack jump increases as the temperature approaches to glass transition temperature of the resin matrix. Thus shear value increases with increasing loading speed. But the ILSS value decreases with other testing conditions. At the vicinity of a glass transition temperature (Tg) viscoelastic processes decreases the modulus owing to unfreezing of molecular motion. At decreasing temperature due to thermal contraction a tighter packing and thus higher bond strength exist. As carbon fiber exhibit good interaction to matrix, they constitute good adhesion between fiber/matrix interface regions. When the force rises to a significant fraction of the force required to break a strong bond and threatens to break the backbone of the molecule, a domain unfolds. Thus, it could avoid the breaking of a strong bond in the backbone. Hence ILSS value increases with increasing loading speed. Matrix ductility increases the critical loads for delamination onset and delamination resistance in the composite laminates. At this temperature it is very difficult to find the delamination failure mode.

C. Kevlar fibre/epoxy composites

1) Interlaminarbehavior

The effects of temperature on ILSS of Kevlar fiber/epoxy composites are shown in Fig. 2.4. Here the specimens are subjected to in-situ testing at high and low temperatures. It is readily observed that at ambient temperature the specimens possess better ILSS compared with other conditioning temperature. The variation of ILSS here is the net result of good adhesion at interface by physical and mechanical bonding at the interface. The ILSS value increases with increasing loading rate but reduction of ILSS value occurred may be due to less post curing effect. The thermal conditioning is likely to change the chemistry at the fiber/matrix interface. The unique chemistry and morphology of Kevlar fiber is also manifested by the composite behavior. The bond between the fiber and the surrounding matrix can be weakened by exposure to active environments.

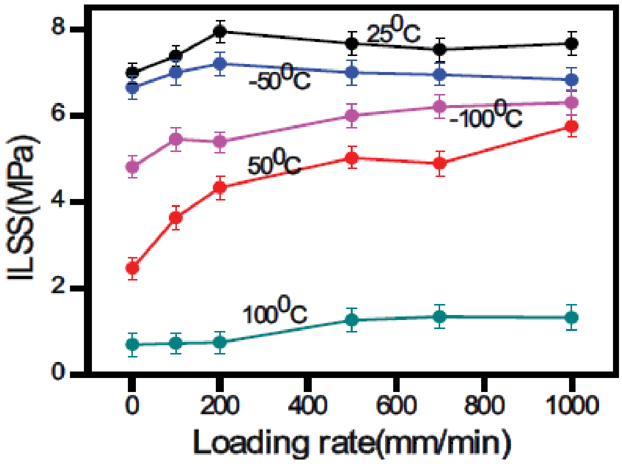

Fig. 2.4: Variation of ILSS with loading rate for Kevlar/epoxy composite system at various temperatures and loading rates.

At high temperature residual stress effects are negligible, mechanisms such as fiber/matrix debonding and matrix ductility become important. As the temperature increases the substantial segments of polymer chains have enough energy to surmount local barriers which hinders molecular motions and begins to move. Here deformation refers to change in shape without change in volume. A ductile tearing mode of failure may result when large-scale shear yielding occurs at a crack tip. Still at high temperature (close to glass transition temperature) molecular motion is so extreme that even chain entanglements are no longer effective in restricting molecular segmental flow. This change in flow behavior of polymer is due to decrease of the degree of chain interpenetration. In addition to the enhanced flow, confinement effects on the chain conformation can perturb the interfacial properties and ultimately the long term stability of the material. So there is presence of weak interface at high temperature. The weak interface readily allows crack deflection. Here the composites can sustain a large deflection by permitting the absorption of more energy. At low temperature a noticeable improvement of shear value was observed compared to high temperature. The result may be attributed to the development of greater amount of shrinkage compressive stress. The fiber/matrix debonding is dominant for the low temperature conditioned Kevlar/epoxy composites.

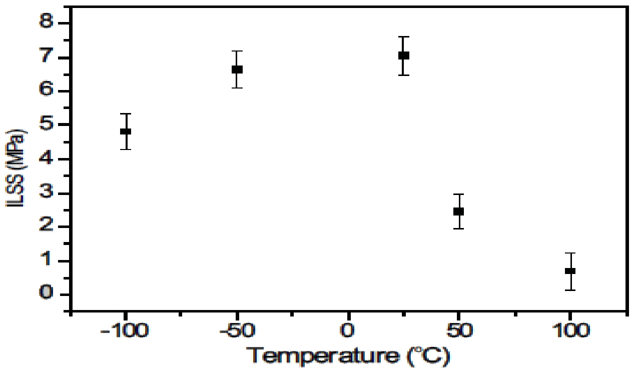

Fig. 2.5: Interlaminar shear strength of Carbon/epoxy composite at 1 mm/min for different temperature.

III. S. Disdier*, J.M. Rey*, P. Pailler* and A.R. Bunsell† (1998) [4] Studied Helium Permeation in Composite Materials for Cryogenic Application:

A disadvantage is the ease of permeability of He through the material at room temperature, and after damage accumulation at 4.2 K. For this study, a special experimental leak detector was built based on a controlled helium flow through a diaphragm and mass spectroscopic measurement. Temperature cycles between room temperature (RT) and 77 K have no effect on permeation rates at 300 K. Tensile tests producing damage at room temperature showed different effects on permeation flow of He, caused by cracking. At 4.2 K, the limit for using this material is given by matrix cracking. The glass fiber volume fraction is preponderant in controlling the coefficient of the permeation. It has been found that composite materials such as glass fiber reinforced epoxy can be used effectively for cryogenic applications using a vacuum as thermal insulation.

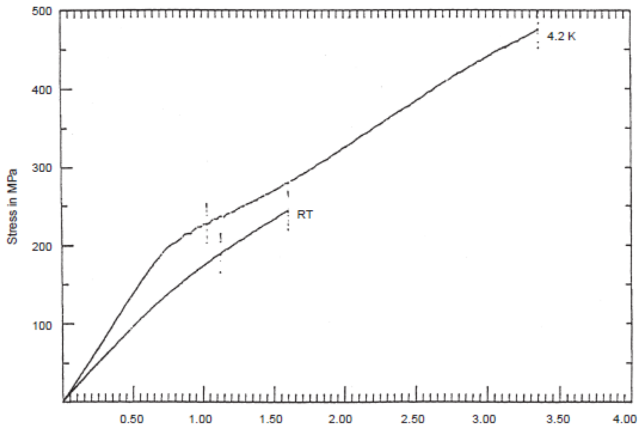

Fig. 2.6: Stress/strain curves for composite material (type U11) at RT and 4.2 K

The stress/strain curves of the tensile tests at RT and 4.2 K on the U11 specimens are shown in Figure 2.6. The curve obtained at 4.2 K reveals a change of slope at 200 MPa and an increase of tensile properties at cryogenic temperatures. The tests were realized in the warp direction of the weave.

IV. TakefumiHoriuchi and Tsutomu Ooi*(1995) Cryogenic Properties of Composite Materials [5]:

For the support materials of superconducting magnet systems, specifically for the transportable cryostat used for nuclear magnetic resonance spectroscopy, three kinds of organic composite materials were investigated: carbon-fibre-reinforced plastics (CFRP), aluminium-oxide-fibre-reinforced plastics, and silicon-carbide-fibre-reinforced plastics. Considering the temperature dependence of the thermal conductivities of glass-fibre-reinforced plastics and CFRP, the authors recommend the use of a combination of these two materials. The thermal and mechanical properties of these organic composite materials and support-system requirements for superconducting magnet systems are presented.

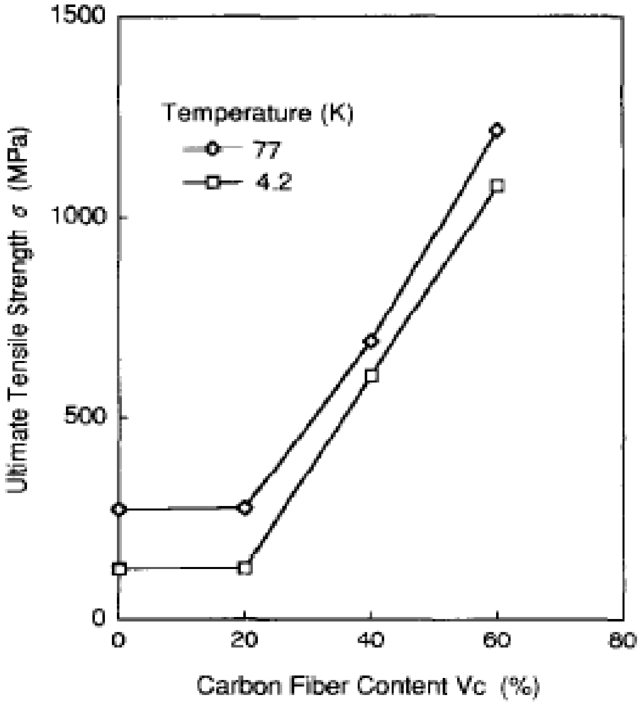

Fig. 2.7: Change in the ultimate tensile strength of CFRP with carbon-fiber content

The change in ultimate tensile strength of CFRP with carbon-fiber content is shown in Figure 2.7. At both 4.2 and 77 K, the ultimate strength is proportional to carbon-fiber content. The ultimate tensile strength at 77 K is higher than that at 4.2 K, by our measurement. The thermal stress caused by the difference in thermal contractions of the carbon fiber and the matrix influences the ultimate tensile strength. With carbon-fibre content less than 20%, the reinforcement by the carbon fibers is small.

V. Kamvouris JE, Roberts GD, Pereira JM (1997) studied Physical and Chemical Aging Effects in PMR-15 Neat Resin[6]:

found that both physical (reversible) aging effects and chemically induced (mass loss) effects occur during long term exposure at elevated temperatures and studied the feasibility of using shear stress relaxation for determining the extent of aging in PMR- 15. The stress relaxation curves measured after aging at 316oC for 800hrs could be shifted to form a master curve. The investigation revealed that the shift in stress relaxation curves and the extent of reversibility of aging effects can provide quantitative information about the physical and chemical aging which occurred in PMR-15 at temperatures up to 316°C.

VI. Hanson MP (1972), studied Effect of Temperature on Tensile and Creep Characteristics of PRD49 Fiber/Epoxy Composites [7]:

experimentally determined the strength and creep characteristics of PRD49 fiber in epoxy. In the range of temperature 20 to 477K, they found the mechanical properties like tensile strength and creep. Tensile strength properties were generally retained to 450 K (350 F); however, at 477 K (400° F), the tensile strength was about 73 percent of that at 297 K (75° F). The tensile modulus showed no significant change at elevated temperatures; however, at 20 K (-423° F), the modulus increased by about 40 percent as compared to the modulus at 297 K (75° F). In creep testing, the PRD49 fiber experienced an accelerated primary creep followed by a much lower secondary creep rate. At room temperature, the fiber exhibited a low secondary creep rate and sustained a tensile stress of about 80 percent of the ultimate tensile strength for 1000 hours without failure. Humidity caused a minor effect on creep behavior of the fiber. Birger et al. studied the effects of thermal and hygrothermal aging on the failure mechanism and concluded that aging of composites at high temperature is affecting both the mechanical as well as physical properties. FRPs are very sensitive to change in temperature due to the difference in the thermal expansion co-efficient of matrix and fiber. Hence this leads to development of thermal stresses in the composite. These thermal stresses may be released by formation of crack in the matrix and sometimes it may result in fiber failure. It was observed that there was reduction in shear strength and flexural strength due to thermal aging. Weakening of the interface takes place. On increasing the aging time, the failure changed from ductile appearance to a more brittle.

VII. Yentlswolfs, Larissa Gorbatikh, Ignaasverpoest (2014) studied Fiber hybridisation in polymer composites [8]:

Fiber-reinforced composites are rapidly gaining market share in structural applications, but further growth is limited by their lack of toughness. Fiber hybridisation is a promising strategy to toughen composite materials. By combining two or more fiber types, these hybrid composites offer a better balance in mechanical properties than non-hybrid composites. Predicting their mechanical properties is challenging due to the synergistic effects between both fibers.

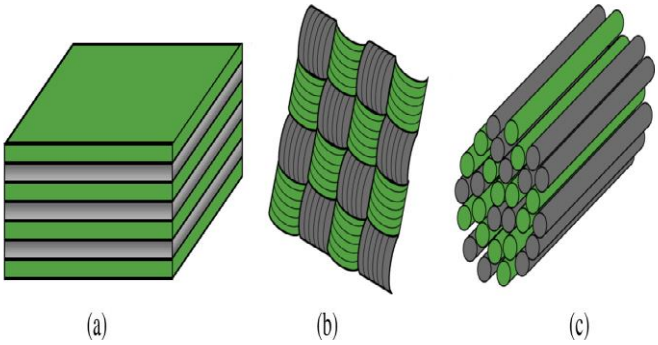

Fig. 2.8: The three main hybrid configurations: (a) interlayer or layer-by-layer, (b) intralayer or yarn-by-yarn, and (c) intrayarn or fibre-by-fibre. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

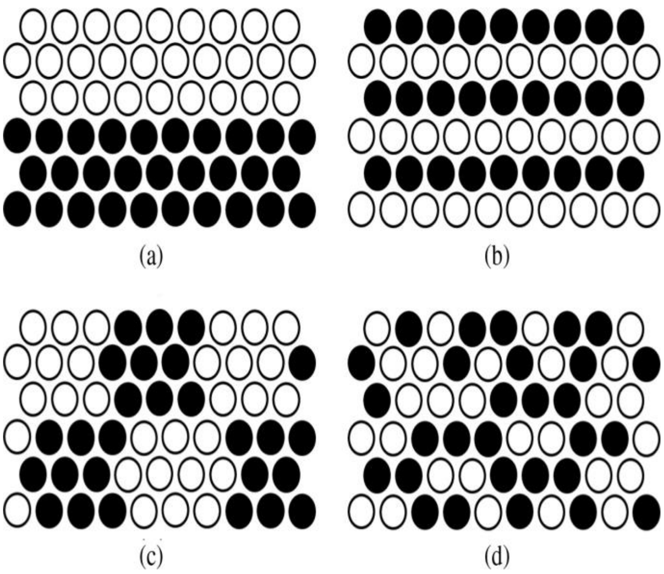

Fig. 2.9: Illustration of the various degrees of dispersion (a) two layers, (b) alternating layers, (c) bundle-by-bundle dispersion, and (d) completely random dispersion.

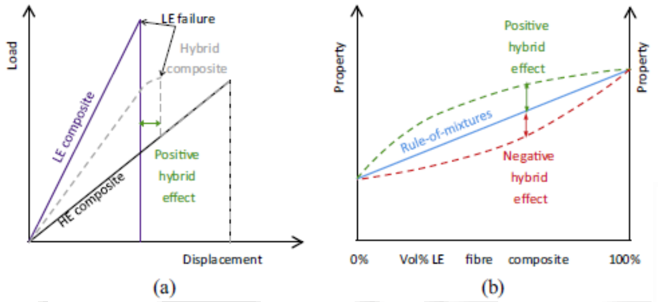

Fig. 2.10: Illustration of the definitions of the hybrid effect: (a) the apparent failure strain enhancement of the LE fibers, under the assumption that relative volume fraction is 50/50 and that the hybrid composite is twice as thick as the reference composites and (b) a deviation from the rule of mixtures. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

The two fibre types are typically referred to as low elongation (LE) and high elongation (HE) fibers. The first fibre to fail is normally the LE fibre. The HE fibre does not necessarily have a large failure strain, but it is always larger than the one of the LE fibre. The LE and HE fibers can be combined in many different configurations. The three most important configurations are visualised in Fig. 2.8. In the interlayer configuration, see Fig. 2.8a, the layers of two fibre types are stacked onto each other. This is the simplest and cheapest method for producing a hybrid composite. In the intralayer hybrid, the two fibre types are mixed within the layers. This is illustrated in Fig. 2.8b, where different yarns are co-woven into a fabric. Other intralayer configurations such as parallel bundles are also possible. The two fibre types can also be mixed or comingled on the fibre level, resulting in an intrayarn hybrid (see Fig. 2.8c). More complex configurations can be obtained by combining two of these three configurations. For example, an intrayarn hybrid can be woven together with a homogeneous yarn.

A crucial aspect in hybrid composites is the dispersion of the two fibre types. This is a measure for how well the two fibre types are mixed and is defined as the reciprocal of the smallest repeat length [10, 14]. Fig. 2.9 schematically illustrates the degree of dispersion. Fig. 2.9a shows a hybrid with a low degree of dispersion, as the two fibre types are in two distinct layers. This can be improved by increasing the number of layers or decreasing the layer thickness, as illustrated in Fig. 2.9b. Another way to increase the dispersion is by hybridising on the fibre bundle level, see Fig. 2.9c. The best dispersion is achieved if the two fibre types are completely randomly distributed, as in Fig. 2.9d.

This critical review of the mechanical properties of hybrid composites proved that in many cases positive hybrid effects can be achieved, although this is not always the case. For all the examined properties, the degree of dispersion was a crucial parameter. In general, increased dispersion leads to larger hybrid effects and better performance.

VIII. Kishore Kumar Mahato, Meet Jayesh Shukla, Devlingamsanthosh Kumar, Bankim Chandra Ray (2014) Studiedin- Service Performance of Fiber Reinforced Polymer Composite in Different Environmental Conditions [9]:

focuses on inservice performance of FRP composites in diversified environmental conditions. Subjecting the composite to high temperature may lead to significant mass loss and material shrinkage. Generation of residual stresses at low temperature accelerates delamination, debonding and matrix hardening.

A. Low temperature

Some researchers have been attracted by the application of glass fiber/ epoxy and carbon fiber/ epoxy composite materials in low temperature i.e. cryogenic environment experimentally studied the behavior of composite laminate at low temperature with the help of the following two types of specimens (1) E- glass fiber/ epoxy reinforced laminate with fiber volume fraction of 0.4. (2) Carbon fiber/ epoxy reinforced laminate with fiber volume fraction of 0.55. Then the observations were made. Stress vs. strain graph shows linearity when exposed to low and room temperature and this extends up to failure. Brittleness is the failure characteristic found. As compared to the strength of laminates at 296K, the strength is higher at 77K for each tested specimen. The value for carbon fiber/ epoxy is about 15- 20% and it is 30- 40% for glass fiber/ epoxy. Hence there is an improvement in the properties of laminates at low temperature. At 77K the laminates have more damaged area around the notch tips as compared to the damaged area obtained at 296K.The micro rupture events are responsible for the damage area, for example, fiber rupture around notch tip, matrix cracking, fiber- matrix interface split, delamination etc. Some energy is dissipated by each micro-rupture event. Hence more the micro- rupture events, larger are the damage areas and the large damage areas specify that during tension, the dissipation of energy is high at 77K. For all the test cases the density of energy dissipation is larger at 77K than at 296K. The Scanning electron micrograph of matrix cracking at cryogenic condition i.e. at 77K is shown in fig. 2.11.Differential thermal contraction between the fiber and the matrix during sudden cooling from room temperature to low temperature leads to the induction of residual stresses. There is an increment in the breaking load with increase in cross head speed up to 50 mm/min and above it the load reduces.

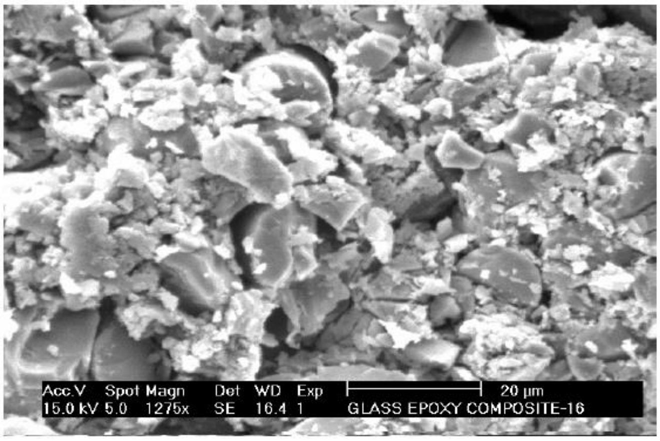

Fig.2.11: Schematic of Matrix cracking at cryogenic temperature i.e.77K.

IX. Conclusion and Remarks

As the temperature decreases, hardness is going to increase due to thermal stresses. Due to change in temperature condition, thermal stress form and due to this debonding presents between fiber and matrix. As the difference in coefficient of thermal expansion between different materials, there is presence of strain misfit.Cracks form due to stresses and debonding, impurities etc. Cracks are developed at fewirregularities in the interface region due tothermal stress.As the temperature cycle increasing, tensile strength is starts decreasing. Most critical part in performance of FRP is interfacing in structural applications. Hence key factor is interface to achieve long term reliability,durability and integrity for better performance, henceproper engineering and designing of the interface is the key factory.

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