MECHANICAL AND FATIGUE CHARACTERIZATION OF CARBON FIBER

REINFORCED COMPOSITE CONTANING RUBBER MICROPARTICLES

AND SILICA NANO-PARTICLES

by

Dinesh Kumar Kannabiran Vasudevan

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Committee Members:

Jintendra S.Tate, Chair

Vedaraman Sriraman

Kunal Kumar

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TABLE OF CONTENTS

Page

CHAPTER

LIST OF TABLES

LIST OF FIGURES

ABSTRACT

Composite materials play a vital role in a wide range of applications. Their adaptability to different situations and desirable properties attracted many industries. In the automotive industry the demand for lighter weight components is increasing day by day. Carbon-fiber reinforced epoxy composites are making inroads used in the automotive industry because of their superior properties such as high specific strength and stiffness, and chemical resistance. Since epoxy resins are brittle in nature, their toughness can be enhanced by reactive liquid rubbers and inorganic fillers. In this study carbon fiber reinforced composites were manufactured using epoxy resin modified by reactive liquid rubber particles carboxyl terminated butadiene acrylonitrile (CTBN) and nanosilica. Since fatigue failure of composite material is a complex phenomenon; the major aim of this study is to examine the effect of CTBN, nanosilica and hybrid (CTBN and nanosilica) under axial tension- tension fatigue performance. This research was first initiated by performing mechanical characterization (tensile and flexural) on neat, rubber and nanosilica modified resin specimens. Carbon fiber-reinforced nanomodified epoxy composite panels were manufactured by Vacuum Assisted Resin Transfer Molding (VARTM). To obtain a qualitative dispersion of rubber particles and nanoparticles in resin, centrifugal planetary mixer (THINKY™) was utilized in this research. Two different concentrations of rubber and nanoparticle particles were tested under tensile, flexural, and interlaminar shear loadings. Then the data was compared

against a control (0 wt% of rubber particles and nanoparticles) composite. Based on

mechanical characterization, one particular loading was selected for fatigue studies.

Extensive axial tension-tension fatigue testing was performed on control, rubber

modified, nano modified and hybrid composites.

1. INTRODUCTION

1.1 Motivation and Background

A combination of two or more materials at a macroscopic scale, which results in better properties, is known as composite materials [1]. Each composite material is isolated by their individual physical, chemical and mechanical properties. Major components of composite materials are reinforcement and matrix. The fiber, which is considered as reinforcement in the composite material, provides mechanical properties such as tensile, stiffness and impact strength. Moreover, composite materials are distinct from other materials, by allowing for a weight reduction in the finished part [1]

The application of composite material is expanding every day and the ability to predict the failure of structure is the major challenge in advance application like aerospace [2], automotive and other structural applications. Carbon fiber reinforced epoxy composite is a promising material in the automotive field. Presently, automotive field designers are developing components such as driver shafts, suspension systems and various kind of load-bearing for car bodies with carbon reinforced epoxy composite [3]. In lower weight application, carbon fiber reinforced epoxy composite system have superior or equal properties when compared to conventional materials like steel and aluminum, but for high load application it is inferior to other materials. Especially, the chassis of a vehicle will experience various loading which includes fatigue loading, torsion loading, lateral bending and longitudinal torsion [4]. A fundamental problem in the composite material is fatigue load, which is a major concern. Since composite materials are anisotropic and heterogeneous, the fatigue behavior is more complicated,

particularly in structural applications for high load. The composite materials exhibit a very complicated failure mechanism under fatigue loading. Fatigue failure in the composite materials occurs with the following main reasons: fiber breakage, matrix cracking, fiber bulking and their interaction [5]

The main motivation of this study is to understand the fatigue behavior of epoxy based composites with filler particles, which makes a vital contribution in the field of composite materials. Such composite materials could be used for engineering composite applications, those of which are subjected to cyclic loading. The mechanical properties of fiber-reinforced composites it is related to the fiber-domination. Properties related to matrix domination are fracture toughness and fracture energy [6]. Increasing the fracture toughness without any degradation of the other material properties will enhance the resistance towards the crack propagation and the lifetime of the composite material. (Zhang, 2004). Infusion of either rubber or nano particles will improve the fracture toughness of epoxy resin systems, which are naturally brittle in nature.

The lifetime of carbon fiber-reinforced composites are determined by the resistance towards the delamination growth and the interlaminar fracture toughness. The toughness of epoxy resin systems can be enhanced by mixing rubber fillers, such as liquid rubbers [7] [8], CTBN [9] and Core-shell [10]. Similarly, silica nanoparticles have some prosperity towards the enhancement of mechanical characteristics of epoxy resin. Even the fracture toughness of epoxy resin can be enhanced by the combination of both CTBN and nanosilica particles; this represents hybrid epoxy resin [11]. The following section will discuss the previous research studies based on the fracture toughness of epoxy resin.

1.2   Literature Review

1.2.1   Epoxy Resin

In the early 1950s, epoxy resins were commercially utilized in aerospace industries and rapidly penetrated into all industry sectors like automotive and microelectronics etc. Thermoset epoxy resins are defined by their excellent mechanical properties, electrical properties, chemical resistance, and low absorption of moisture. Due to their highly cross linked structure most of epoxy resins are naturally brittle, which dramatically limits the application of epoxy polymers [12] [13].

Since epoxy resins are brittle, they have poor fracture toughness, weak impact strength and low resistance towards   fatigue crack propagation [14]. As we discussed earlier, epoxy resins are toughened by the rubber particles. Kinloch et al. introduced CTBN as the tougheners for epoxy resin. Initially, a homogenous mixture was not attained by the CTBN particles, but later it was achieved by mixing an excess amount of epoxy resin [15]. Yet, the infusion of rubber particles decreases the strength, modulus and glass transition temperature of epoxy resin [16]. The toughness and the toughening mechanism of the epoxy resin are determined by the tougheners infused in epoxy resin [17].

Improvement in the strength and modulus of epoxy resin is observed by mixing nanoparticles such as nanosilica [18], carbon nanotubes [19] and graphene [20]. Zhang et al. observed some enhancement in the strength and modulus of the epoxy resin by silica nanoparticles. The impact strength was also improved by 5 wt% of the nanosilica content. Additionally, through SEM analysis it is observed that the major toughening mechanism is caused by the energy dissipation through the dimples caused by the nanoparticle [21].

For several years, improvement in mechanical properties of epoxy resin is achieved by the silica nanoparticle modification. Presently, the improvements in toughening of epoxy resin rely on the toughening mechanism of foreign particles incorporated in them. Certain toughening mechanisms have been suggested to explain the complex behavior of polymers with nanofillers, like crack deflection, crack branching, crack pinning, particle birding, particle deboning, micro cracking or crazing of the matrix and elastic deformation of the matrix [22].

Zappalorto et al. observed some improvement in the toughness of the epoxy matrix through nanoparticle debonding; when a crack occurs in the nanoparticle filled polymer and it's proved that nanoparticle debonding results in a limited toughness improvement [23]. This model accounts for the effects of the size and properties embedding the nanoparticles are created by their inter and supra-molecular interactions arising at the nanoscale [23]

In 2012, Zappalorto et al. observed that the improvement in toughness due to plastic yielding around nanovoids and proved that plastic yielding is a highly dissipative mechanism, which leads to high fracture toughness enhancement at low nanofillers content [24]. Even the fracture toughness can be enhanced by the combination of different mechanisms like particle debonding, plastic void growth of nanovoids and localized shear banding [25]

The major drawback with the rubber toughening is the relatively high viscosity of epoxy resin containing reactive liquid rubbers, which will be so critical for the infusion process during the composite manufacturing [22]. In contrast, nanosilica leads to a slight

increase in the viscosity of the resin mixture, but it can be utilized for injection or infusion process, which can easily saturate through the closed mesh fabrics.

Recently, the combination of CTBN rubber particles with nano-sized silica particles produced by the sol-gel process, observed a significant improvement in the toughness of epoxy resin and carbon fiber reinforced epoxy composites [26]. Through this hybrid epoxy resin system, a tough and stiff material can be formed. This formulation creates a well bonded soft layer around the particle that can transfer tensile load to the silica core, maintaining the modulus and strength of the composites [27]. Sprenger S et al, have also examined the combination of reactive liquid rubbers and nano silica particles in epoxy resin system, which resulted in enhancement of the properties [28].

Therefore, the amalgamation of nanoparticles and rubber particles into epoxy resin, which is considered to be a hybrid epoxy resin system, results in the improvement of fatigue crack growth resistance of reinforced epoxy composites. Yet, significant property improvement is only observed with hybrid neat resin, while considering the fiber/reinforced composite, only partial improvement is observed [29]

1.2.2 Rubber Modified Epoxy System

The modification epoxy resin system through rubber particles was identified in the late 1960's. The most successful method is the infusing of some reactive rubber, such as CTBN into a liquid epoxy resin system [30]. The aim of this initial experimentation is to enhance the fracture toughness of the material. [31].

The fracture toughness of composite materials is determined by the plain strain critical stress intensity factor (K1C). Different methods were approached to improve the

toughness of epoxy resin systems, but the most common method is the infusion of rubber particles into epoxy resin. The incorporation of CTBN liquid rubber which leads to the cross link of the molecular chain extension of epoxy resin has improved the ductility of the matrix [32] .Toughness in epoxy resins is enhanced by two phases; the first phase is the incorporation of dispersed toughners like butadiene-acrylonitrile of low molecular weight into the epoxy resin and then they are synthesized by carboxylic acid (CTBN) or amine groups (ATBN). When this formulation is cured, the rubber modifier urges out as a second phase. As a result, the fracture toughness of modified epoxy resin can significantly enhance with 10 phr of rubber modifier (second phase), but this toughening process will affect the glass transition temperature and modulus in a negative way.

Now, how is the toughening mechanism occurring in the system? Are the rubber particles absorbing the energy or is the matrix. The following section will provide some previous research studies regarding the toughening mechanism of epoxy resin systems.

1.2.3   Rubber Toughening Mechanism

According to Kinloch et al. toughening in epoxy resin is caused by the following mechanisms [33]

- Particle cavitation
- Void growth
- Shear banding
- Crack birding
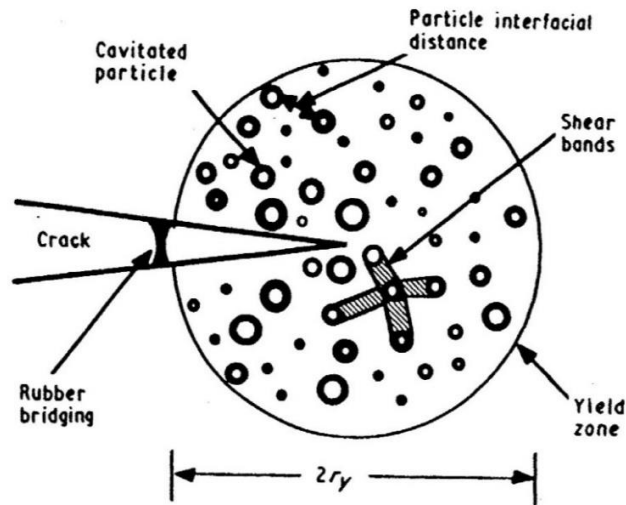
**Figure 1 : Schematic Representation of Toughening Mechanism
(http://www.vicbasechina.com/readnews.asp?id=29)**

In the past, each research group came out with different statements regarding toughening mechanism: Kunz et al. proposed that crack bridging of rubber particles makes a major contribution towards the toughening mechanisms [7]. Sultan et al. observed that the crazing of the epoxy matrix is the reason behind the toughening mechanism. The second phase of the toughening mechanism includes particle cavitation and matrix shear band growth. According to the previous studies the toughening mechanism is initiated by the cavitation of the rubber particles and pursued by the shear yielding of the epoxy matrix, and this process is observed by Rata et al. The particle cavitation leads to a reduction in hydrostatic tension and initiates the growth of shear bands [34].

Recently, Shamsiah et al. examined the fracture toughness with core-shell rubber particles. They observed voids of rubber particles and matrix deformation through SEM (Scanning Electron Microscopy) analysis. The voids in rubber particles will leads to the

cavity formation, which enhances their plastic deformation [35]. This action indicates improvement in the fracture toughness.

The toughening of rubber mechanisms is being attained through the stress field over the rubbery particles and matrix shield yielding. Bratczak et al. examined the external shear yielding in the matrix could be developed by the triaxial stress on the crack-tip and this mechanism is accessed by the rubber cavities [36]. Hence, the association of fracture toughness with the plastic deformation is advanced to the crack-tip.

In 2003, Kinloch et al. reported that the matrix materials have low deformability and shear yielding with high-cross linked epoxy [37]. Recently, Jianing Zhang et al. mentioned that the tough mechanism attained through pinning, cavitation and bridging of rubber particles will enhance toughness, but they are less efficient with low-cross linking density. In contrast, through their study they concluded, toughening of epoxy resin is not obtained by rubber separation, especially with low-cross linking density [38].

As we discussed earlier, the major objective of the rubber toughening system is to enhance the fracture toughness of the matrix material. Yet, the enhancement of fracture toughness is limited by a specific loading of the rubber particles and this phenomenon is observed by many research workers. In the past, Grag A.C et al. observed a drop in fracture energy with 20 phr rubbers [39] and this fact is applicable for the majority of the rubber particles. Below Figure 2 represents the effect of fracture toughness with the addition of CTBN rubber particles.
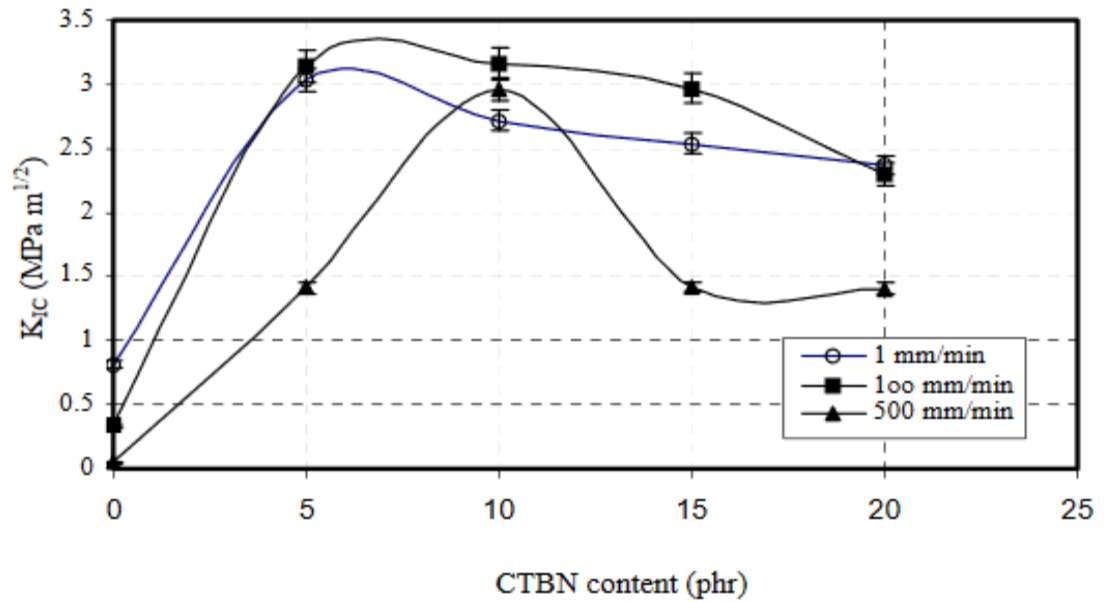
Figure 2: Effect of Fracture Toughness by CTBN rubber particles [40]

Even though the fracture toughness of the matrix is improved by the rubber particles, it has some disadvantages towards the mechanical characterization and glass transition temperature of the composite material and this phenomenon is observed by Dong et al and Ishak et al with core- shell and CTBN rubber particles respectively [40] [41].

The filler size plays a vital role in developing the toughness of the matrix system because smaller size fillers can play in the process zone; if the filler size is larger than the process zone, it could affect the toughening mechanism of the system. In the past, Kunz et al. compared the fracture toughness study between the 1 µm and 40 µm particle size and proved smaller size particles have an excellent effect on fracture toughness [42]. Presently, the rubber particle size varies from 0.1 – 5.0 µm, so filler size is not an issue towards the toughening mechanism. Yet, the mechanical properties of epoxy matrix are degraded by the rubber particles, but they can be sustained by the infusion of nanosilica

particles. Nanosilica will enhance the mechanical properties of the epoxy resin and also have influence towards toughness.

1.2.4 Silica-nanoparticles

In the past 13 years, many industrial composites applications are attracted towards silica nanoparticles due to their excellent mechanical characterization [6]. The infusion of silica nanoparticles in the epoxy matrix is the method to improve the mechanical characterization [43] and this scenario is proved by many researchers; we will discuss more about it further. On another track, CTE is decreased by addition of silica nanoparticles [44]

The effectiveness of silica particles is affected by following factors:

- Filler Size
- Filler Concentration

The above factors have major contributions towards the effectiveness of silica nanoparticles. In order to decide the optimum and loading content of silica nanoparticles for enhancing the mechanical properties, analysis has been performed with micron and nano-silica size particles. T.H. Hsieh et al. learned that micro-sized silica particles have significant growth towards toughness by plastic void growth and shear yielding [45]. This effect is observed due to local cluster and non-uniform distribution; the agglomeration of nanoparticles is more with smaller particles when comparing to larger particles. Certainly, Kinloch et al. observed such plastic voids will get closed when the epoxy polymer is heated above its glass transition temperature T_g [46]. During the debonding process of nanoparticles very low energy is observed when compared to the plastic

deformation of the polymer [47]. Yet, the debonding process is necessary, which reduces constrain at the crack tip and permits epoxy polymer to deform plastically though void-growth mechanism.

The property of epoxy composites can be enhanced with different filler concentration. It has been observed that an increase in the amount of silica particles will enhance the modulus, fracture toughness and flexural strength. The improvement in properties of the epoxy composite is observed with 10 wt% of the nanosilica concentration and it is a general trend. Jiaaning et al. concluded, that enhancement in toughening mechanism with 10 wt% of nanosilica carbon reinforced composite leads to an increase in fatigue delamination resistance [27]. Even Manjunatha et al. observed the similar trend with 10 wt% of nanosilica in glass fiber reinforced composite [18]. The main toughening mechanisms, shear banding and plastic void growth are observed stronger with 10 wt% of nanosilica [48]. In order to propagate, the cracks have to travel around the particles, so by increasing the concentration of nanosilica the crack would require more energy debond or move around the nanoparticles [49]. One more term which has a vital role towards the effect of filler concentration is "agglomeration". If particle agglomeration is observed it may lead to variation in interparticle distance, which is unfavorable to the toughening mechanisinsm. Sperenger S, et al. reported, due to the presence of smaller diameter nano-silica particles, the viscosity of resin is not significantly affected by the particles and lack of agglomeration [50]. This effect was observed by Kinloch et al. through atomic force microscopy (AFM) and transmission electron microscopy (TEM) analysis [51].

1.2.5 Silica Toughening Mechanism

Three general toughening mechanisms, which support silica toughening mechanism of epoxy-silica composites are debonding, cracking and matrix shear banding has been proved The toughening mechanism which acquires more contribution towards silica particle toughening is debonding. The concept of this mechanism is some amount of energy is dispersed in order to debond the particles from the matrix. Dittanet et al. observed debonding of particles with 10 wt% of silica nano particles by SEM analysis. Additionally, they predict that a decrease in particle size may reduce the number of debond particles [52]. In 2007, Johnsen et al. observed plastic void growth with 9.6 wt% of nanosilica, here the plastic void is initiated by the debonding of nanoparticles and this plastic void allows the matrix to deform plastically [53].
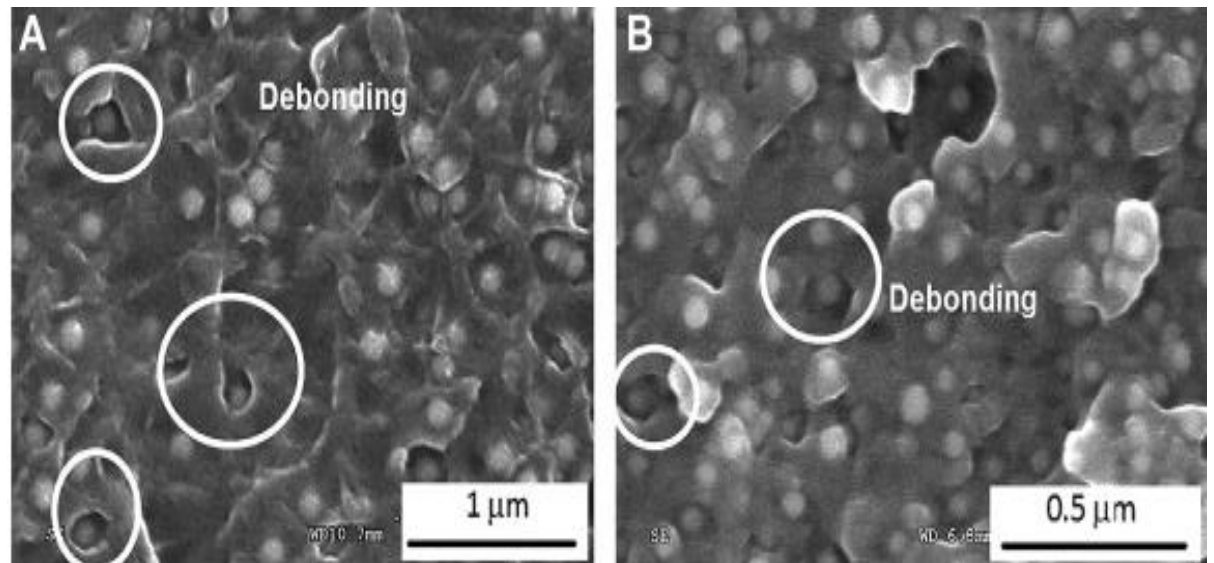
Figure 3 : SEM images of the fracture surface of DGEBA containing 10 vol% of different sized nanosilica particles: A) 170 nm, B) 74 nm [52]

The second toughening mechanism is crack pinning; the excursion of the crack is interfered by the presence of nanosilica particle in the matrix. Sometimes particles are

able to arrest the motion of the crack, but often the crack will deflect from the particular path and leads to the next weak interface. In order to be a valid crack mechanism, the particle size should be larger than the crack-opening displacement. [10].
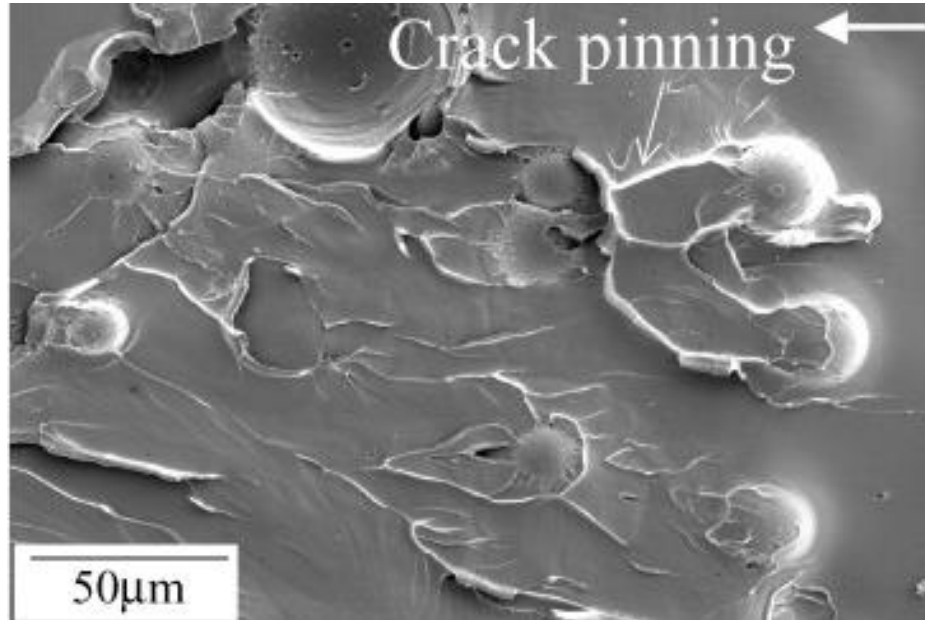
Figure 4: Crack Pinning observed by SEM analysis [54]

From the overview of the silicon toughening mechanism, improvement in fracture toughness, young modulus and yield strength were proved. As mentioned before, the specification of the particles involved in epoxy-silica composites plays vital role in enhancing the properties of the composite material.

Since, enhancement in fracture toughness and mechanical properties has been observed with both rubber and nano-silica particles; further research is involved with hybrid epoxy silica rubber composite. The valuable properties through this particular combination are very interesting. This particular hybridization will generate a composite material with excellent stiffness and toughness properties.

1.2.6   Hybrid Epoxy Composite

For the past few years, the combination of silica and rubber in the epoxy system in order to form tougher material has been investigated. In first industrial application, enhancement in lap shear strength is observed with the combination of reactive liquid rubbers and low nanosilica concentration 2wt%. The toughness seems to be not improved, when compared to the control formulation. This might be due to the formation of a very close meshed network by the curing agent, dicyandiamide [55]. The fracture toughness is improved to high level with this hybrid system and it has been observed in some cases

Both rubber and silica nanoparticles are involved in improving the toughening mechanisms. The cavitation of rubber particles results in reduction of hydrostatic stress in the matrix and this creates a lower driving force towards silica nanoparticles to debond which results with high efficiency in crack pinning by these particles [56]. In recent studies, Hesieh et al. observed similar toughening mechanism with cavitated rubber particles and with low debonding of the nanosilica particles [57]. The increase in matrix plasticity could blunt the crack tip with the small amount of silica nanoparticles. [58]. Overall, similar toughening mechanism is observed with hybrid system no new toughening mechanisms is evolved.

1.2.7   Static Mechanical Properties of Rubber and Silica Nanoparticle Modified

Composite

As mentioned earlier, epoxy resins are brittle, they have poor fracture toughness, weak impact strength and low resistance leads to fatigue crack propagation. The fracture toughness of epoxy systems can be enhanced by additives such as rubber, thermoplastic and inorganic particles [59]. The fracture toughness can be improved by adding rubber particles, but reduction in strength and modulus is observed. In the past, Bascom et al. observed fracture toughness improved by 30 times when compared to unmodified epoxy, yet the strength and modulus decreased drastically with an increase in the amount of rubber particles added to the epoxy system [60]. This indicates the plastisation effect of rubber particles, which exhibit low strength and modulus, but with the proper concentration amount of rubber particles, toughness can be enhanced without sacrificing the mechanical properties of epoxy resin.

The increase in fracture toughness of epoxy resins without sacrificing their basic mechanical properties can be achieved by adding inorganic additives such nanosilica, alumina and nanoclay particles. Epoxy resins modified by rigid nanoparticles have moderate property enhancement, particularly in fracture toughness, due to weak interfacial bonding and particle dispersion. Nanosilica particles are produced by the sol-gel process has an excellent dispersion with epoxy resin system which can improve fracture toughness and other mechanical properties [61]. In 2010, Manujnatha et.al with 10 wt% of nanosilica improved tensile strength of bulk epoxy from 73.3 to 86.9 MPa and modulus from 2.62 to 3.07 GPa; with laminates tensile strength improved from 365 to 382 MPa and modulus from 17.5 to 18.8 GPa [18]. Similarly, Tate et al. observed tensile

strength improved from 92.1 to 122.4 MPa at 6wt% of nanosilica, with 7 wt% 109.5 Mpa and 8 wt% 101.4 MPa and modulus improved from 10.3 GPa to 13.2 GPa with 6 wt% of nanosilica; 7 and 8 wt% achieved 12.8 GPa and 11.7 GPa, respectively [62]. In further investigation they observed improvement in interlaminar shear strength (ILSS), flexural strength and modulus.

1.2.8   *Fatigue of Carbon Fiber Reinforced Composites*

Carbon fiber reinforced composites (CFPRs) have very high strength and stiffness, dimensional and thermal stability. They are used as structural materials in load bearing applications. The primary reason for failure in composite structural materials is cyclic loading. Under cyclic loading, carbon reinforced composites exhibit gradual degradation of the mechanical and structural properties as a result of damage accumulation [63]. Even under relative low loading, debonding and matrix cracking will occur and it will grow as loading cycle increases. Fatigue failure occurs in composite materials with three stages, first stage is initial damage under low loading, which drastically decreases the stiffness and reduces strength in low level. In second stage, a gradual and slow material degradation will occur; the fatigue life of a material is relying on the second stage. At the final stage, the strength decreases rapidly and loses of stiffness in the damage zone leading to fatigue failure. Damage accumulation in composite material is occurred by collaboration of different damage modes like delamination, fiber fracture and matrix fracture. Fatigue failure in composite material is unpredictable, additionally other external factors which create more complexity in predicting fatigue failures are temperature [64], moisture and impact damages.

1.2.9   Fatigue of Rubber and Silica Nanoparticle Modified Composites

Generally, fatigue behavior of on-axis specimens was influenced by fiber breakage, where as in off-axis specimens it depended on interlaminar shear domination and crack propagation of the polymer matrix [65]. Since, epoxy resins are used in various structural applications, understanding the fatigue behavior of epoxy resin is essential. Curtis reported that toughened resin can enhance tensile fatigue behavior in low cycle fatigue system, while in high-cycle fatigue system, toughened epoxy is inferior when compared to standard epoxy resin [66]. In 1995 Masanobu et al. performed fatigue test on toughened epoxy and observed 10 times greater fatigue strength with 10 wt% CNBR particles. Manjunatha et al. observed three to four times improvement in fatigue properties with glass reinforced epoxy composite modified by 10 wt% of nanosilica; this enhancement is influenced by matrix cracking and plastic void growth mechanism [18]. Even, Tate et al. observed improvement into fatigue properties with 6 wt % of nanosilica particles in glass fiber reinforced epoxy composite for wind turbine blade application [67]

1.3   Objective of this Research

Since epoxy resins are naturally brittle toughening of epoxies has been studied extensively. Moreover, understanding the fatigue crack propagation behaviors of epoxy resin composite is more important for the composite materials involved in structural applications. The broad objective of this research is to determine the mechanical and fatigue performance of carbon fiber reinforced epoxy composite which is modified by 5 and 10 wt% of nanosilica and CTBN rubber particles, and their hybrid system. It is known that the infusion of micro-rubber and nanosilica particles will improve the fracture toughness of epoxy resin system. This scenario could improve the mechanical and fatigue

properties of laminate composite with epoxy resin system. So, this research is focused in understanding the mechanical and axial tension-tension fatigue behavior of CTBN rubber particles, nanosilica particles and their hybrid (nanosilica and CTBN rubber particles) in carbon reinforced epoxy composite. Additionally, this research could support the future work related to replacement of existing metallic materials with carbon fiber reinforced composite in the automotive field for high load application.

The flow charts in Figure 5 and 6 will represent the overall plan of this research.

1.3.1   Assumption, Limitation and Delimitation

From literature review, it is known that the fracture toughness of epoxy resin system could be improved by CTBN rubber particles and nanosilica particles with loading rate range between 2- 15 wt% and 2 – 20 wt% respectively. Considering the viscosity property of the resin and % elongation obtained during the benchmark study, the loading rates selected for carbon composite manufacturing. For this research 5 and 10 wt% of nanosilica and CTBN rubber particles were selected. This assumes that a loading rate beyond 10 wt% will increase the viscosity of the resin mixture which will affect the manufacturing process of carbon composite. And improvement in % elongation indicates good fracture toughness attained by CTBN and nanosilica particles.

The viscosity of epoxy resin system used in this study can be maintained at low level with temperature range between 40-48 °C. VARTM manufacturing technique at elevated temperature is utilized in this research to manufacture carbon composite panel. The temperature range is maintained between 40-48 °C throughout the manufacturing process. However, temperature- gradient within the mold will be an obstacle in this

manufacturing process, so there may be variations in thickness of the carbon composite

panel.
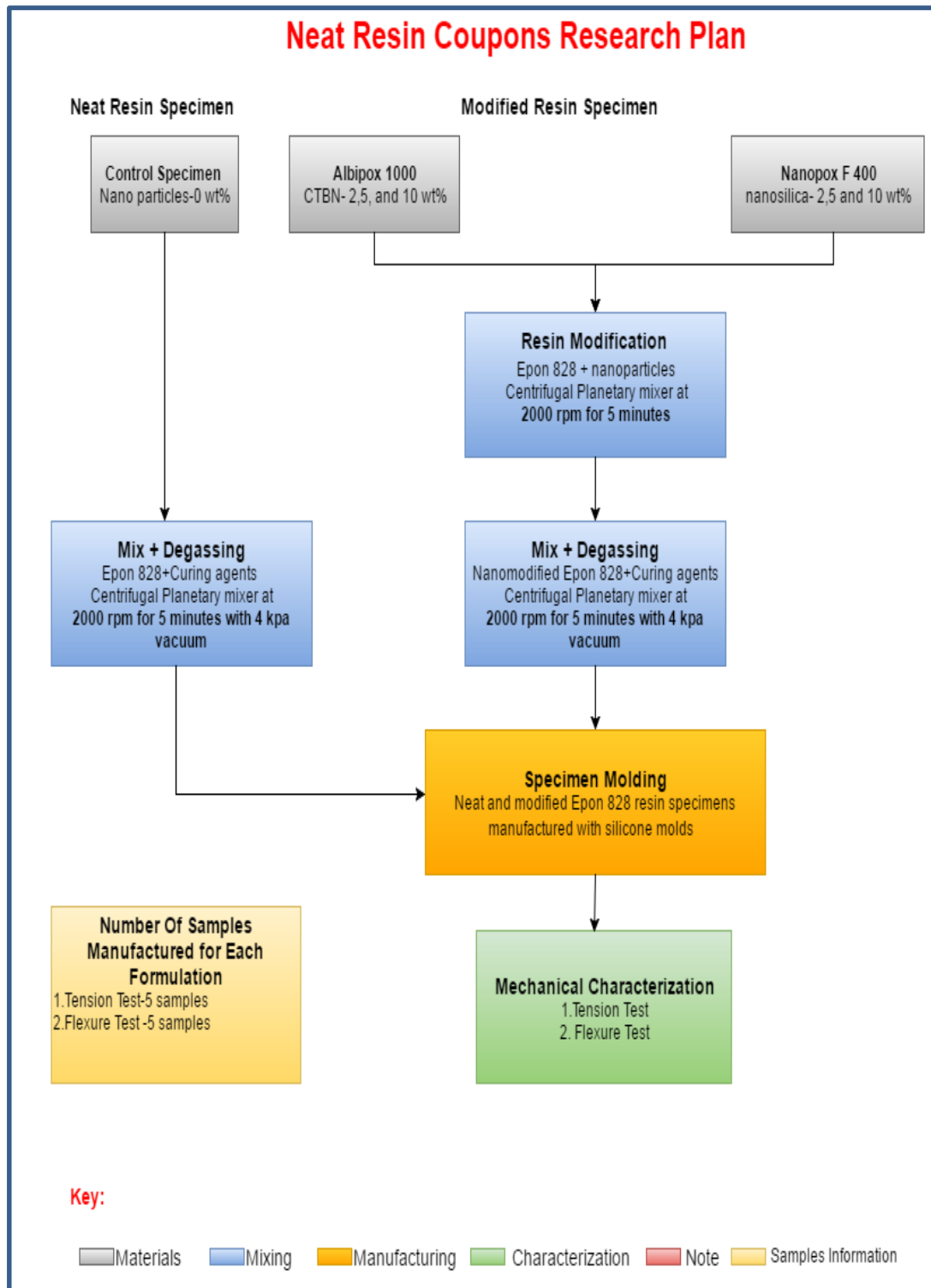
Figure 5 : Research plan of neat resin coupons
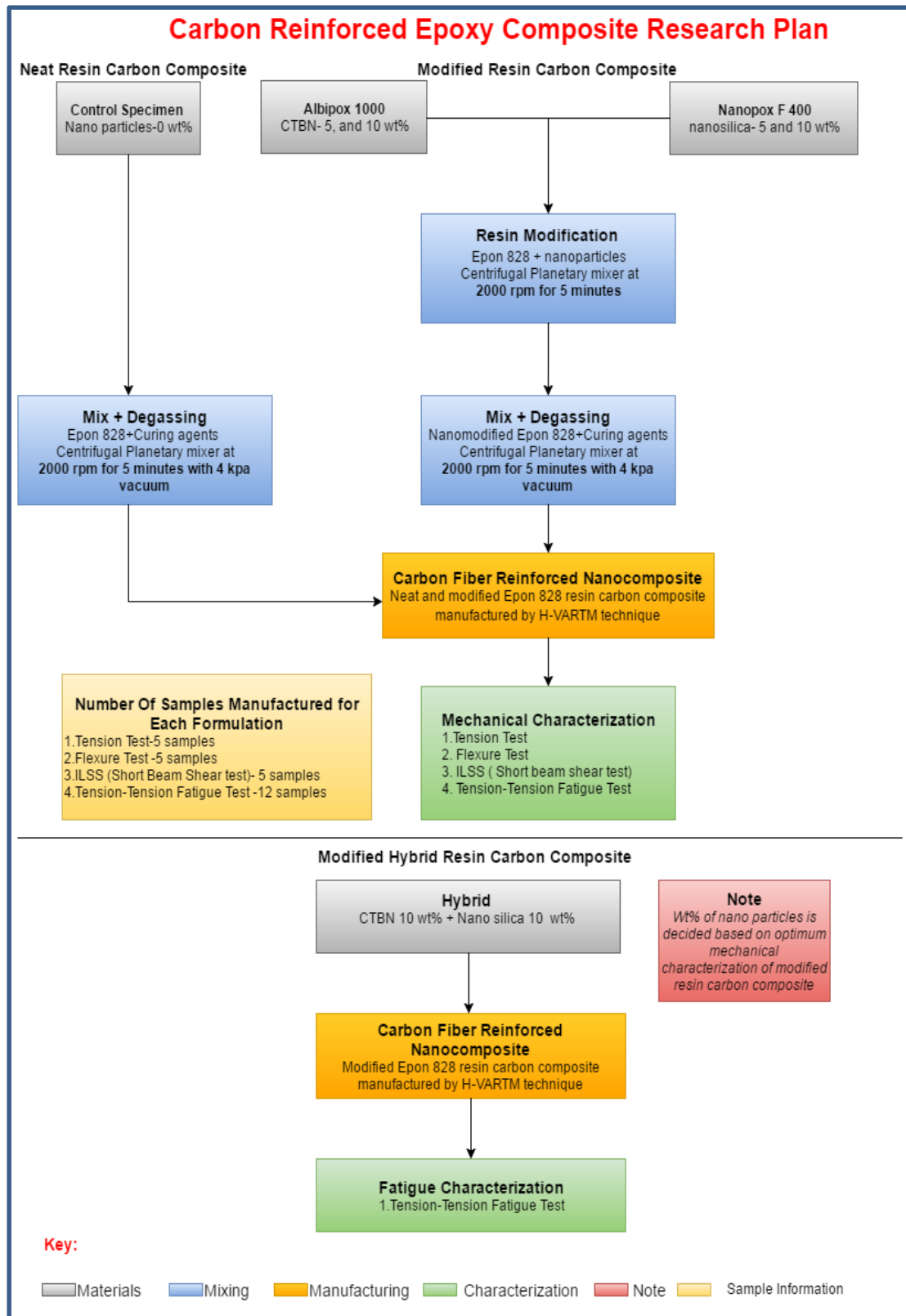
Figure 6 : Research plan of carbon reinforced epoxy composite

2. METHODOLOGY

2.1   Material System

2.1.1   Carbon Fabric

For this research, 3k- plain weave carbon fabric was purchased from Fiber Glast Development Corporation. The plain weave carbon fabric is selected for it fabric style, delivering uniform strength in both horizontal and vertical directions. Resin can soak through fabric quickly and can be handled efficiently. Plain weave carbon fabric acquires high specific strength and is suitable for aircraft, racing, marine and light industrial application. Moreover, plain weave is the tightest weave and is unlikely to fray at the ends.

Table 1: Properties of 3K-Plain Weave Carbon Fiber [68]

FIBER PROPERTIES	
Tensile Strength	4205.80- 4378.17 MPa
Tensile Modulus	227.5-240.6 GPa
Elongation	1.4-4.95 %
Carbon Content	95 %

2.1.2   Epoxy Resin (EPON™ 828)

Diglycidyl ether of bisphenol A (DGEBA) epoxy resin EPON™ 828 supplied by Hexion as free samples was selected in this research as it maintains excellent mechanical properties and is a standard resin used in the industry. By using appropriate curing agents, high strength and high performance can be obtained. Viscosity of EPON™ 828 resin at 25 °C is very high (110-150 P), but it has low viscosity at elevated temperatures, which is an advantage to achieve a quality dispersion of nanoparticles in the resin [69].

2.1.3 Curing Agents (EPIKURE™ 3300 and EPIKURE™ 3230)

For this research, EPIKURE™ 3300 and EPIKURE™ 3230 curing agents were provided by Hexion. The advantages of using EPIKURE agents are: convenient mix ratios and ease of handling; improved cure system strength, toughness and flexibility, improved impact and peel strength, improved cured system chemical, solvent and water resistance, ability to alter the reactivity of the system and finally blush and sweat-out free films. EPIKURE™ 3230 is compatible with modified epoxy resin blends and they enhance flexibility and good impact resistance [70]. EPIKURE™ 3300 is a low viscosity, amine curing agent used in formulating heat cured epoxies, and they have high distortion properties when they are cured properly.

2.1.4 Curing Agents (ANCAMINE® 2904 and ANCAMINE® 2678)

ANCAMINE® 2904 and ANCAMINE® 2678 were supplied by Air Products and Chemicals, which are designed to cure liquid epoxy resin at elevated temperature. ANCAMINE® 2904 is designed to heat-cure liquid epoxy resins at lower (~100°C) temperatures than typical cycloaliphatic amines with moderate pot life and rapid curing. Cured product exhibits excellent chemical resistance, high mechanical strength and high temperature tolerance [71] ANCAMINE® 2678 has excellent adhesive property with epoxy resin and ANCAMINE® 2904 have excellent mechanical and chemical resistance; moreover they have high heat distortion properties [72]

2.1.5 Rubber Micro Particles- CTBN (ALBIPOX® 1000)

ALBIPOX® 1000 is a high performance CTBN (butadiene-co-acrylonitrile) rubber modified (40 %) epoxy resin based on bisphenol A epoxy resin, and it was supplied by Evonik Corporation as a free sample. This resin system consists of a special nitrile rubber, which improves the toughness of brittle epoxy resin [73], at the expense of modulus and glass transition temperature (Tg) [74]

2.1.6 Silica Nanoparticles (NANOPOX® F 400)

NANOPOX® F 400 is supplied by Evonik Corporation as a free sample. This nanoresin system is a modified epoxy resin system consisting of surface-treated colloidal nanosilica particles, which is specially designed for fiber-reinforced applications. Because of its low viscosity it is suitable for infusion and the injection process. The weight percentage of nanosilica in this nanoresin system is 40% [75].

2.1.7 Planetary Centrifugal Mixer (THINKY^{TM} ARV-130)

A qualitative dispersion of rubber micro particles and nanosilica particles could be attained by Planetary Centrifugal Vacuum Mixer, "THINKY ^{TM}" ARV-130. The dispersion of rubber particles is achieved by the rotation and revolution motion, as shown in Figure 8. The maximum centrifugal force achieved by the mixer is 400 G, which is produced from 2,000 RPM and 9 cm diameter arc container rotation. The revolution speed is adjustable in a range of 200-2000 rpm and the ratio of revolution speed to the rotation speed of the cup holder is fixed at a 2:1 ratio.

Figure 7: THINKY ARV-310 Mixer

Figure 8: Planetary centrifugal mixing cycle

2.2   Manufacturing of Resin Coupons

2.2.1   Neat Resin Coupons

The neat resin coupons were manufactured according to the flow chart show in Figure 9. Neat resin coupons are control (0 wt%) specimens; they do not consist of rubber and nanosilica particles. The curing agents are mixed with Epon-828 epoxy resin at 2000 rpm for 5 minutes with 4 kPa vacuum to attain a degassing using centrifugal planetary mixer. After degassing, the resin mixture was poured into the silicone molds of tension and flexure specimens that are designed according to the ASTM standards. For curing, molds were placed in the oven for a curing cycle of 80°C for 2 hours and followed 150 °C for 3

hours. The cure temperatures and times were determined based on the recommended minimum curing temperatures and the times for each of the curing agents in the formulation.

Figure 9: Silicone Molds for Tension and Flexure Specimens
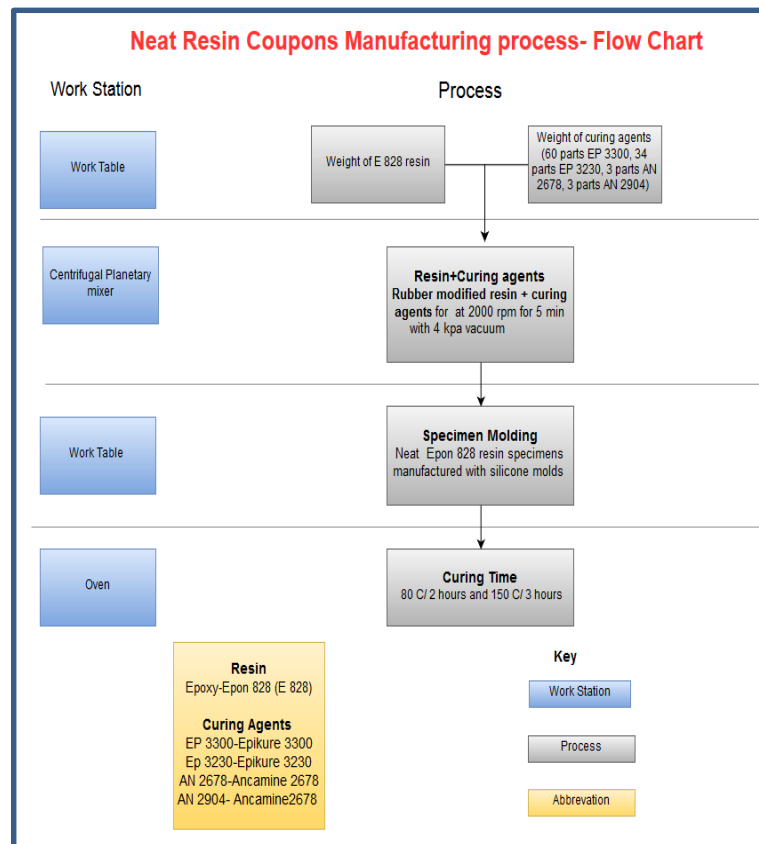
Figure 10: Manufacturing Flow Chart of Neat Resin Coupons

2.2.2 *Modified Resin Coupons*

The modified resin coupons were manufactured according to the flow chart shown in Figure 10**.** The same process was followed for both rubber and nanosilica modified resin coupons. The desired amount of ALBIPOX® 1000 / was mixed with Epon- 828 epoxy resin at 2000 rpm for 5 minutes in the centrifugal mixer. Same process was repeated with NANOPOX® F 400. Then, the curing agents were blended with the modified resin mixture at 2000 rpm for 5 minutes with 4 kPa vacuum. The modified resin mixture was then poured into the silicone molds, followed by the same curing process as that of neat resin coupons.
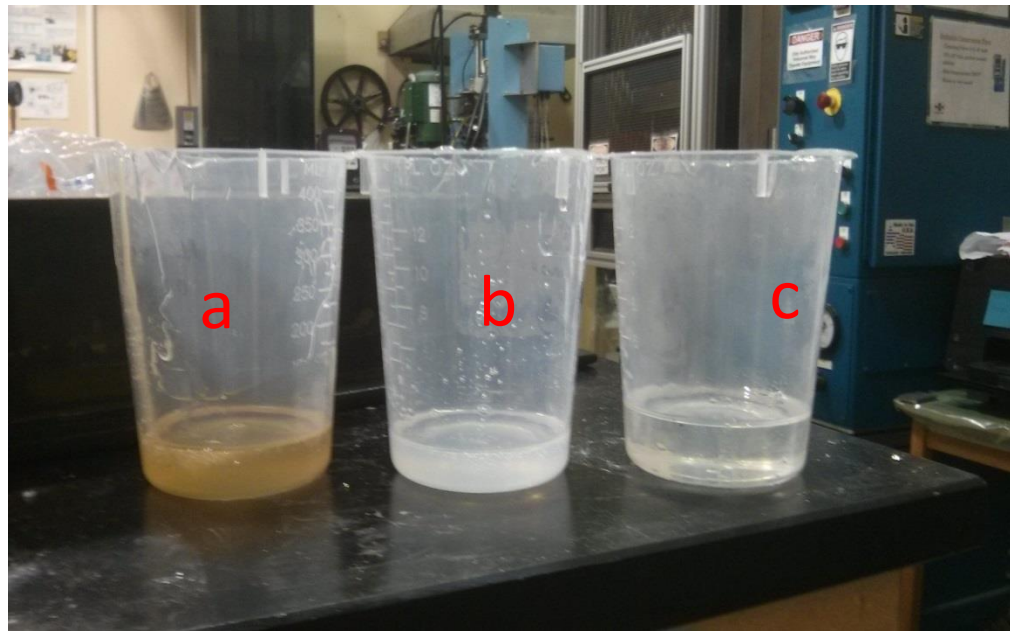
Figure 11: Modified Resin Coupons Materials: a) ALBIPOX® 1000 b) NANOPOX F 400 c) EPON™ 828- epoxy resin

Figure 12: Modified Resin Mixture in Silicone Molds
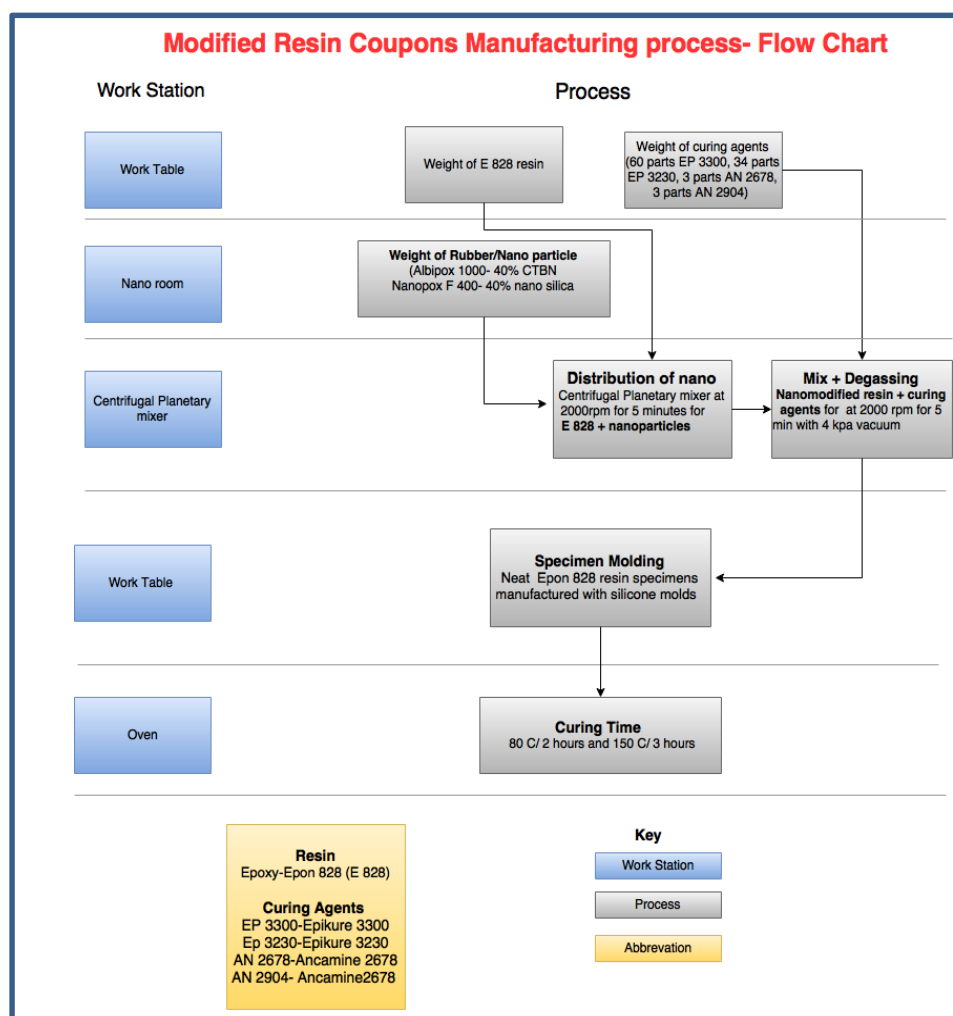
Figure 13: Manufacturing Flow Chart of Modified Resin Coupons

2.3   Manufacturing of Carbon Reinforced Epoxy Composite

2.3.1   Vacuum Assisted Resin Transfer Molding at Elevated Temperature

VARTM is a very cost-effective method in manufacturing composite materials. In this process, infusion of resin to carbon fibers is obtained through a vacuum. The low pressure attained by the VARTM lay-up, which creates a vacuum will support the resin to flow through the fabric. The viscosity of EPON™ 828- epoxy resin is very high (110-150 P); using high viscous in resin VARTM process may lead to uneven distribution of resin throughout the fabric.

Viscosity of epoxy resin can be reduced at elevated temperature. According to the technical data sheet, EPON™ 828 epoxy resin can attain low viscosity at an elevated temperature between 40 to 48 °C; with low viscosity, fiber volume fraction of composite material can be enhanced i.e. excellent adhesion of carbon fiber and resin is attained. To ensure low viscosity of resin throughout the process, the temperature is maintained between 40 to 48 °C. The molding materials used in the elevated temperature VARTM process can withstand a temperature level of 121 °C.

2.3.1.1   Mold Setup

Step 1: A Ceramic base considered to be an insulation material is placed on the table and the heating pad is placed above the ceramic base.

Step 2: The glass panel was placed on top of the heating pad.

Step 3: From this step, mold setup will follow a sequence of lay-up, starting with release film positioned on top of the glass panel followed by bottom peel ply, carbon fabric, top peel ply, resin distribution media and lines for resin and vacuum distribution.

Step 4: A yellow sealant tape is placed around the mold. Then, the vacuum bag is sealed on top of the mold setup using sealant tape.

Step 5: The vacuum lines are connected to the appropriate vacuum pump and the vacuum pressure by switching ON vacuum pump. Make sure there are no leaks

Step 6: To control the flow of heat, a thermocouple which is connected to a microcontroller, is placed above the vacuum bagging.
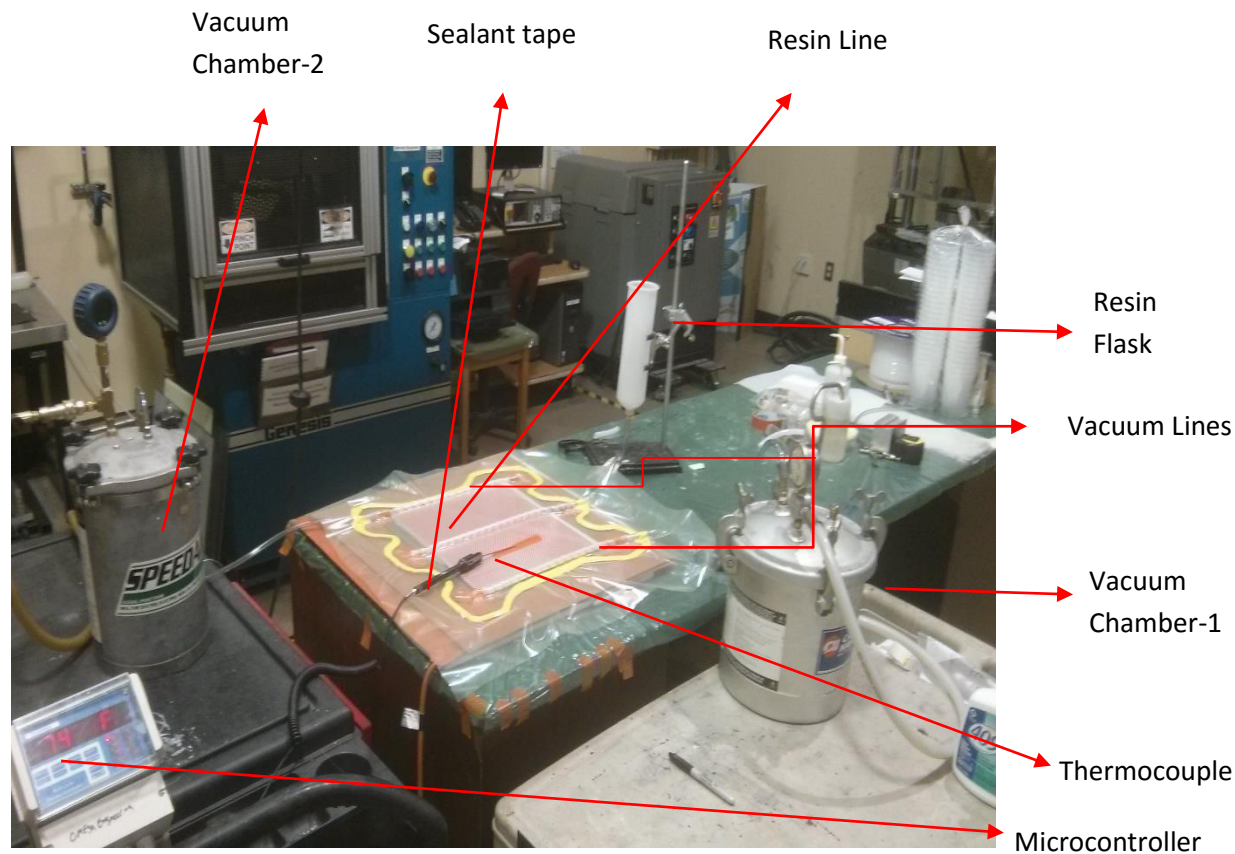
Figure 14: Mold Setup for VARTM at Elevated Temperature

Purpose of the Materials:

Ceramic Base: It is considered an insulating material, which is placed to avoid direct contact of the heating pad with the table.

Heating Pad: It is the heat resource for this VARTM process.

Glass Panel: A Pyrex glass plate is used as a mold. Since glass panels are bad conductor of heat, other bagging materials can be protected from overheating.

Release Film: In this process Dahlar Release Bag 125 is used as a release film. This release film provides easy removal of composite panel from the glass mold after curing.

Bottom Peel ply: It is a porous release material, which provides a surface suitable for secondary adhesive bonding (like tabbing) without further surface preparation.

Carbon Fabric: For this research, 12 layers of 3K plain weave carbon fabric were used.

Top Peel Ply: It is a porous material similar to bottom peel ply; it supports in resin flow and provide and provides surface which is suitable for secondary adhesive bonding (like tabbing) without further surface preparation.

Distribution Media: It is a red mesh made of LDPE/HDPE blend laid on top of the top release fabric. This helps maintain an even distribution of resin and facilitates the flow of resin through the thickness of the panel. The use of distribution media is a patented technology termed as SCRIMP (Seemann Composite Resin Infusion Manufacturing Process) that was invented by W. H. Seemann.

Resin and Vacuum Lines: Spirally cut HDPE (High Density Polyethylene) tubes are used in this research. Two vacuum lines are placed at the ends of the distribution media and one resin line is place at the center of the distribution media. Vacuum lines are connected to the appropriate vacuum chambers and resin line is connected to the resin supply (resin flask).

Microcontroller: A Multi-zone process controller from Omega Engineering is used in this research. A temperature set point has been programmed according to the requirement. The heating pad is connected to the microcontroller by universal relay module. If the thermocouple measures the set point temperature, the microcontroller will SHUT OFF the heating pad. If the temperature value reduces from the set point, the microcontroller will SWITCH ON the heating pad. Through this process, the temperature range will be maintained between 40 to 48 °C.
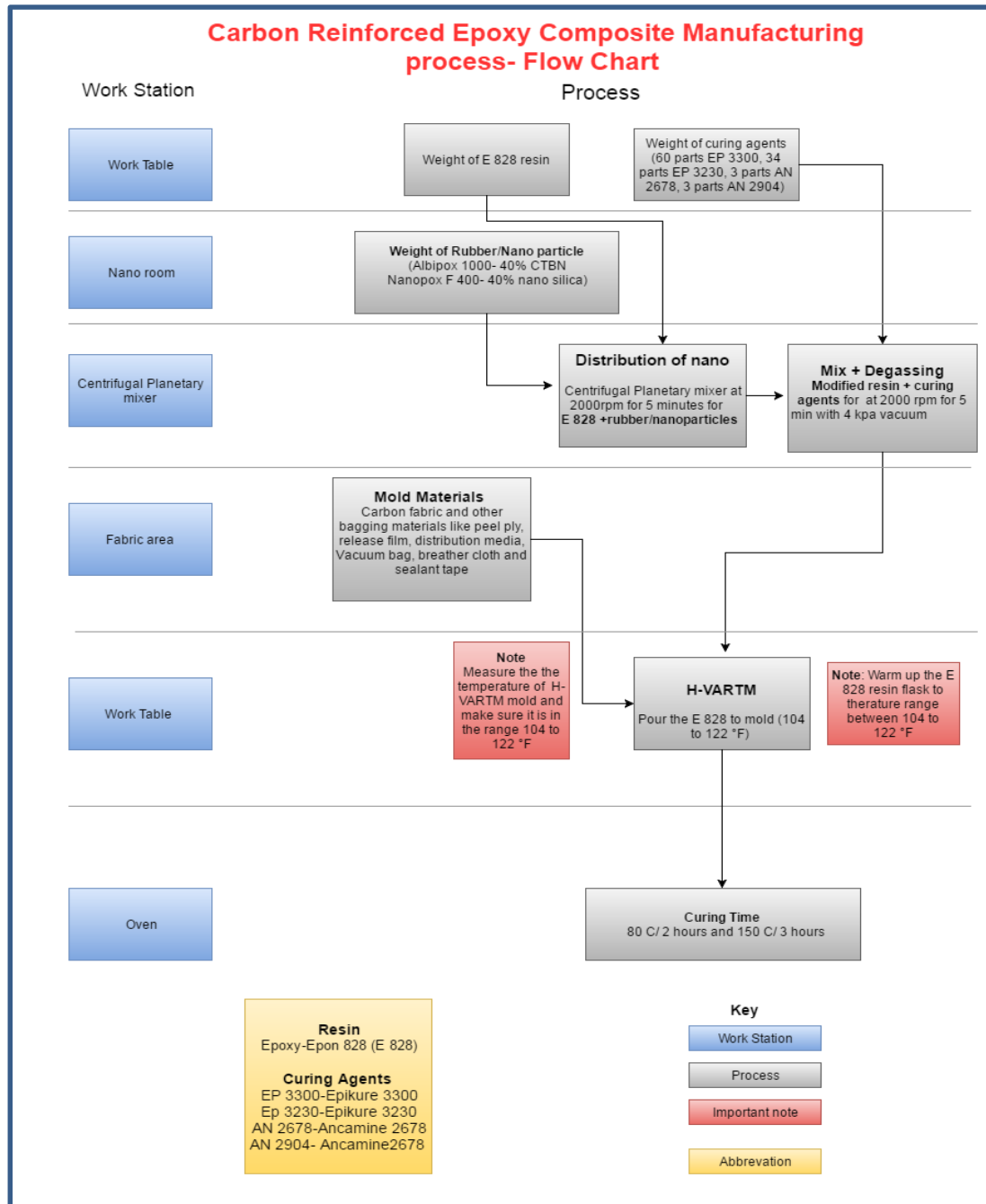
Figure 15: Manufacturing Flow Chart of Carbon Reinforced Epoxy Composite

2.3.1.2 Resin Formulation

Resin formulation for this research is calculated based on the equivalent weight of the resin and curing agents. By determining the equivalent weight of the blend (EPON™ 828 and ALBIPOX® 1000 / NANOPOX® F 400) and curing agents, the appropriate amount of curing agents and ALBIPOX® 1000 / NANOPOX® F 400 to be mixed with the resin is calculated.

Table 2 : Resin formulations for Composites modified by CTBN

CTBN (wt%)	EPON™ 828 (g)	ALBIPOX ® 1000 (g)	EPIKURE ™ 3300 (g)	EPIKURE ™ 3230 (g)	ANCAMINE® 2904 (g)	ANCAMINE® 2678 (g)
0	100	0	15.27	8.65	0.76	0.76
2	96.84	6.46	15.36	8.70	0.77	0.77
5	91.7	16.90	15.49	8.78	0.77	0.77
10	82.21	36.23	15.74	8.92	0.79	0.79

Table 3 : Resin formulations for Composites modified by Nanosilica

SiO_2 (wt%)	EPON ™ 828 (g)	NANOPOX ® F 400 (g)	EPIKURE ™ 3300 (g)	EPIKURE ™ 3230 (g)	ANCAMINE ® 2904 (g)	ANCAMINE ® 2678 (g)
0	100	0	15.27	8.65	0.76	0.76
2	96.8	6.51	15.42	8.74	0.77	0.77
5	91.74	16.82	15.67	8.88	0.78	0.78
10	82.1	36.46	16.12	9.14	0.81	0.81

After resin formulation, they are mixed by Planetary Centrifugal Vacuum Mixer, "THINKY ™" ARV-130, and even degassing of resin is performed with the same equipment. Degassing is the important step because the resin had to be free from entrapped air and/or gases that could cause voids in the composite panels. Functioning of THINKY™ centrifugal planetary mixer is explained in detailed in Figure 13.

2.4   Water Jet Cutting

After manufacturing composite panels, specimens were cut using 'Ingersoll Rand' water jet cutting system. According to ASTM standard, CAD diagrams were created with appropriate dimensions using 'AutoCAD 2013' in order to get cut. Then, the CAD file was transferred to the water jet cutting system.

Figure 16: Water-Jet System

Figure 17: Specimens Cut from Panel

2.5   Performance Evaluation

The mechanical and fatigue characterization of carbon fiber reinforced composites is determined were evaluated by '810- Material Testing System'. The load frame of the machine is 100 kN (22 Kips). This machine can conduct static tensile, compression, flexural or bending, and fatigue tests by adapting to different fixtures. It is controlled by 'Flex Test SE' controller with 'Multipurpose Testware' software developed by the MTS Corporation.

2.5.1   Neat and Modified Resin Coupons

2.5.1.1   Tensile Test of Resin Coupons

Tensile tests were performed according to ASTM D 638- "Standard Test Method for Tensile Properties of Plastics" [76] on neat and modified resin coupons. This test was conducted based on displacement control mode with a cross head rate of 1.5 mm/min. Since resin coupon specimens were brittle, pressure of the hydraulic MTS grips were maintained between 1 to 1.5 MPa. Axial strain was measured using Axial Extensometer-632.24 E. 50. According to the standard, a total five specimens were tested in each category. After the data collection, the axial stress vs axial strain graph was plotted to determine the tensile strength (UTS or S_u) and modulus for each specimen. The standard dumbbell-shaped specimen was used for this test.

Figure 18: Tension Test of Resin Coupon

Figure 19: Dumbbell-Shape specimen

2.5.1.2   Flexural Test of Resin Coupons

Flexural tests were performed according to ASTM D790 - Standard Test Methods for Flexural Properties of Unreinforced and Reinforced Plastics and Electrical Insulating Materials [77]. Four-point bend tests were conducted neat and modified resin coupons. The specimen used for this test is a rectangular flat specimen of 127 mm length, 12.7 mm width and 3 to 3.2 mm depth, with a span to depth ratio of 16:1. Loading span for the flexural test was 48 mm and rate of cross-head motion was 1.35 mm/min.

Figure 20: Four-Point bend test

Figure 21: Flat Flexural Specimens

2.5.1.3   Brief Conclusion

To have knowledge about toughening of epoxy resin system, static tests were performed on neat and modified resin coupons. Moreover, this study is considered as a benchmark for the composite research to evaluate the weight percentage of CTBN and nanosilica. Tension test was conducted to determine tensile strength and modulus; Flexural test was conducted to determine flexural strength and modulus. From the results, centrifugal planetary mixer "THINKY ᵀᴹ" ARV-130 considered as an excellent dispersion technique for CTBN rubber particles and silica nanoparticles.

2.5.2   Carbon Fiber Reinforced Epoxy Composite

2.5.2.1   Tension Test of Carbon Composite

According to ASTM D3039- 'Standard Test Method for Tensile Properties of Polymer Matrix Composite Materials' tensile tests were conducted on control and modified carbon fiber reinforced epoxy composite. Tensile property of reinforced composites is fiber dominant and this test determines in-plane tensile properties of reinforced composite materials. According to ASTM standard, the shape of the specimen is rectangular and 254 * 25.4 mm in dimension. Thickness of the specimen varied between 2.5 to 3.0 mm. To assure failure to occur in gauge area, while simultaneously t not to occur in grip area, tabs were attached to the tension specimens. By using 'Scotch-Weld Epoxy Adhesive DP 460 Off-White' tabs were attached to tension specimens. The pressure of the MTS hydraulic grips maintained at 5 MPa. Axial strain was measured using Axial Extensometer-632.24 E. 50. The tensile test was conducted based on displacement control mode with a cross head rate of 2 mm/min. According to the standard, a total of five specimens were tested in each category.

During the tension test, the parameters: Axial Force, Axial Displacement and Time are recorded. After data collection, the axial stress vs axial strain graph was plotted to determine the ultimate tensile strength (UTS or S_u) and modulus for each specimen. The maximum load endured by the specimen before breaking is considered as UTS, also known as tensile stress.

Tensile stress of the specimen can be calculated by the following equation:

$$\sigma_{max} = \frac{P_{max}}{A_o}$$

P_{max} = Maximum Load or Axial Force

A_0 = Area of the specimen before testing

According to standard, tensile modulus can be calculated from the slope of initial linear portion of the axial stress vs. axial strain graph called chord modulus (E^{chord}). The modulus of elasticity is calculated from axial strain values from 0.001 to 0.003 mm/mm.

$$E^{chord} = \frac{\Delta\sigma}{\Delta\varepsilon}$$

Figure 22: Tension test of Carbon Composite

Figure 23: Tension Specimen

2.5.2.2 *Flexural Test of Carbon Composite*

Flexural test on carbon reinforced epoxy composite is conducted according to ASTM D 790-92- 'Standard Test Methods for Flexural Properties of Unreinforced and Reinforced Plastics and Electrical Insulating Materials'. These test methods determine the flexural properties of unreinforced and reinforced plastics including high-modulus composites and electrical insulating materials in the form of rectangular bars molded directly or cut from sheets, plates, or molded shapes [77].

During the test, the top of the sample under the loading force is in compression, and the bottom opposite the loading force is in tension [78]. Since the specimen experiences compression loading on top and tension loading at bottom, it takes a long span to break; so this study was performed with span-to-thickness ratio 16:1. The specimen dimensions were manipulated based on the thickness. For this study, the dimension of the specimen was 76.2 * 25.4 mm; it is a flat rectangular specimen.

It is a three-point bend test; the specimen was placed on two supporting rollers and loaded by a single roller between the supports. The specimens were tested at a cross-head motion of 2 mm/min. During the test axial force, axial displacement and time were recorded. Flexural strength and modulus is determined by plotting a graph between axial force and axial displacement.

Flexural strength and modulus is calculated by the following equations:

Flexural Strength,

$$\sigma_{fs} = \frac{F_{max}.L}{b.d^2}$$

Where, F_{max} = Maximum axial force, L= Loading span of the specimen, b= width of the specimen, d= thickness of the specimen.

Flexural Modulus,

$$E_{fs} = \frac{m.L^2}{(4.b.d^3)}$$

Where, m= slope of initial linear portion on load-deflection curve, L= Length of the specimen, b= width of the specimen, d= thickness of the specimen.

Figure 24: Flexural Test of Carbon Composite

Figure 25: Flexure Specimen

2.5.2.3 Interlaminar Shear Strength (Short-Beam Test)

According to ASTM D2344 – 'Standard Test Method for Short-Beam Strength of Polymer Matrix Composite Materials and Their Laminates' short- beam tests were performed. The short-beam shear test is used to determine the fiber/matrix adhesion of composite material. This test is similar to the flexure test and differs only with the specimen dimension and span-to-thickness ratio is 4:1 to accuracy. The specimen span-to-thickness ratio which is constrained to 4:1, forcing the shear stress to attain the failure levels before tension and compression reach their ultimate values [79]. To observe the

43

shear strength of the material along the fiber/matrix adhesion plane, the short-beam test was performed. This test is considered as a quality check for the lamination process and matrix-dominated properties of the composite.

The dimension of the specimen is determined by the thickness of the specimen,

Specimen length (L) = thickness * 6

Specimen width (b) = thickness * 2.0

For this research, the dimension of the flat rectangular specimen was 16.8 * 5.6 mm. During the test axial force, axial displacement and time were recorded. A graph is plotted between axial force and displacement. Short-beam strength is calculated by using the following equation:

$$F_{sbs} = \frac{(0.75 * P_{max})}{b * h}$$

Where, P_{max} = Maximum axial force during the test, b= specimen width, h=specimen thickness

Figure 26: ILSS Test of Carbon Composite

Figure 27:ILSS Specimen

2.5.2.4 *Tension-Tension Fatigue Test*

Fatigue tests are used to determine durability of material under fluctuating stresses. When a material survives between 1 million and 10 million cycles it is considered to have a "infinite life" In this research, tension-tension fatigue tests were performed according to ASTM D3479/D3479M titled 'Standard Test Method for Tension-Tension Fatigue of Polymer Matrix Composite Materials'. According to standard, fatigue tests were performed under repetitive constant amplitude load cycles. The maximum and minimum stress was applied from 90% of the UTS and reduced until the specimens survived 1 million cycles. Since fatigue studies are time consuming, researchers often prefer 'Accelerated Testing'. Through this testing method material degradation mechanism can be studied. For this research, all tension-tension fatigue tests performed with 10 Hz frequency took 27 hours to reach 1 million cycle. Stress ratio (R) is 0.1

$$R = \frac{\sigma_{min}}{\sigma_{max}} = \frac{P_{min}}{P_{max}}$$

Where,

σ_{max} = Maximum stress and corresponding load is P_{max}

σ_{min} = Minimum stress and corresponding load is P_{min}

The geometry and dimension of the specimens were similar to ASTM- D 3039 standard 'Tension Test' i.e. a flat rectangular specimen with 254 * 25.4 mm in dimension. All the specimens were tabbed to avoid failure in the grip. Fatigue tests were performed on control (0 wt%), 10 wt% nansolica,10 wt% CTBN and Hybrid (10wt% of CTBN+10 wt% of nanosilica) specimens. Maximum stress applied to the specimens was percentage of ultimate strength of control composites. The percentage level of UTS applied in this study is 90%, 85%, 80% and 70%, and their respective stress values were 486.15, 459.14, 432.13 and 378.11MPa. In order to investigate the improvement in fatigue of the modified epoxy system, same stress levels were applied to other modified specimens. Under each category three specimens were tested.

Table 4: Number of Specimens Tested for Fatigue Study

Maximum Stress (MPa)	Control (0wt%) Composite	CTBN (10 wt%) Composite	Ns (10wt%) Composite	Hybrid (CTBN 10wt%+Ns 10wt%) Composite
486.15	3	3	3	3
459.14	3	3	3	3
432.13	3	3	3	3
378.11	3	3	3	3

S-N Curve:

S-N curve is used to determine the relationship between fatigue stress and fatigue cycles. This curve will describe the fatigue behavior of the material. Basically, S-N curves formed by a semi-logarithmic or logarithmic curve, and they do not consider stress ratio and other parameters; it is formed by maximum stress and the number of cycles withstood by the material. S-N curve does not have any rule, so the shape of a curve could vary for different materials. The results of the S-N curve could be accurate for one loading condition or system but poor for another [80].
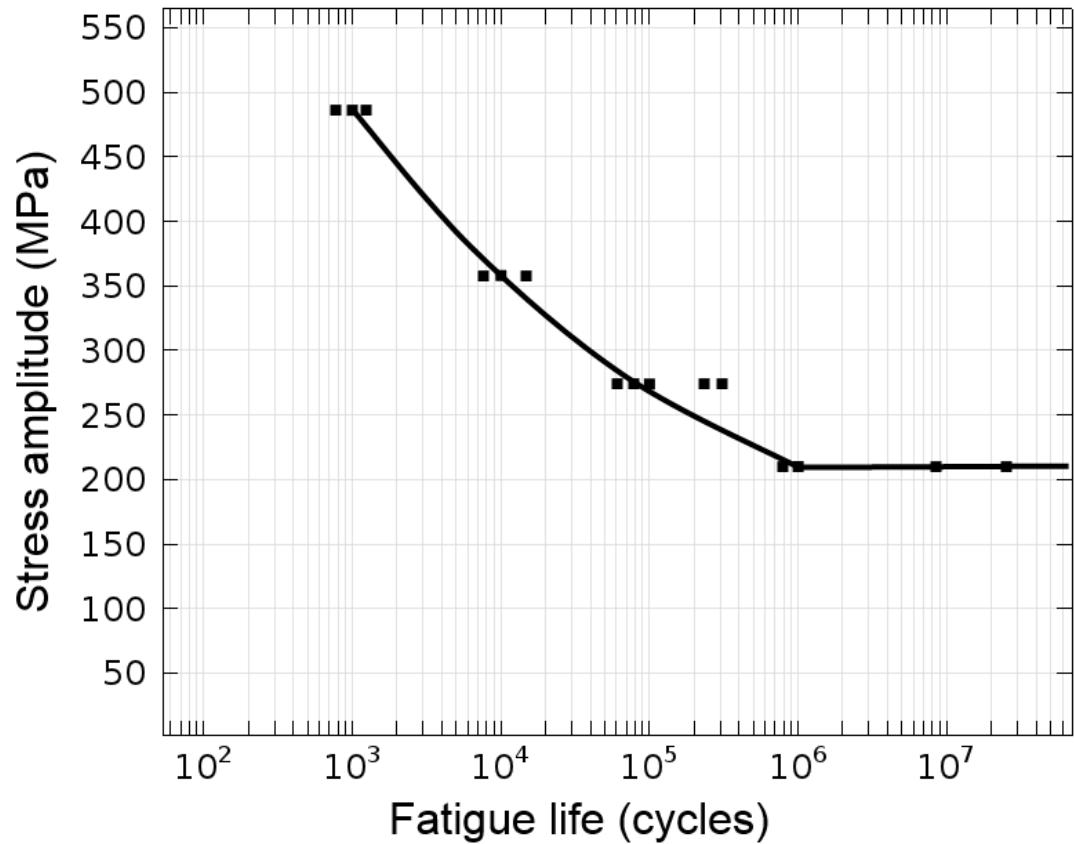
Figure 28: S-N Diagram [81]

Figure 27 represents a typical S-N curve of a material and dots in the diagram represent the number of samples tested at each load level. In composite materials, the plot above 10^6 fatigue cycles indicates the infinite life of the material with little or no crack propagation. The stress level at which materials show infinite fatigue life is called endurance limit.

Stiffness Degradation:

Since the damage mechanism of composite material is very complex, it can be studied through stiffness degradation method. There is a relationship between damage and stiffness degradation [82], because stiffness degradation is the only parameter used to evaluate the life of a component. Damage

accumulation and failure in composite materials occurs when the residual strength reduces to the maximum applied cycle stress. During fatigue test stiffness degradation will occur more when compared to reduction in residual strength. Damage accumulation in composite material occurs by collaboration of different damage modes like delamination, fiber fracture and matrix fracture. In Figure 28, P.K. Mallick explained the occurrence of different damage modes in composite against the fatigue life. Evaluation of fatigue life through residual strength cannot predict or track the fatigue damage. Stiffness degradation method is more applicable for designing of the composite structure.
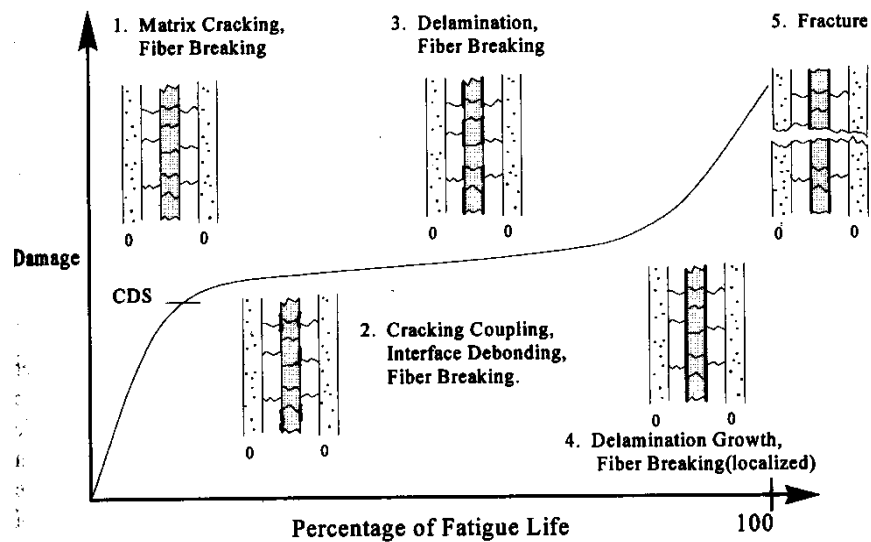
Figure 29: Damage Modes in Composite Material against Fatigue Life

Tate et al. represented stiffness degradation curve with 6 wt% of nanosilica in glass fiber reinforced composite.

49
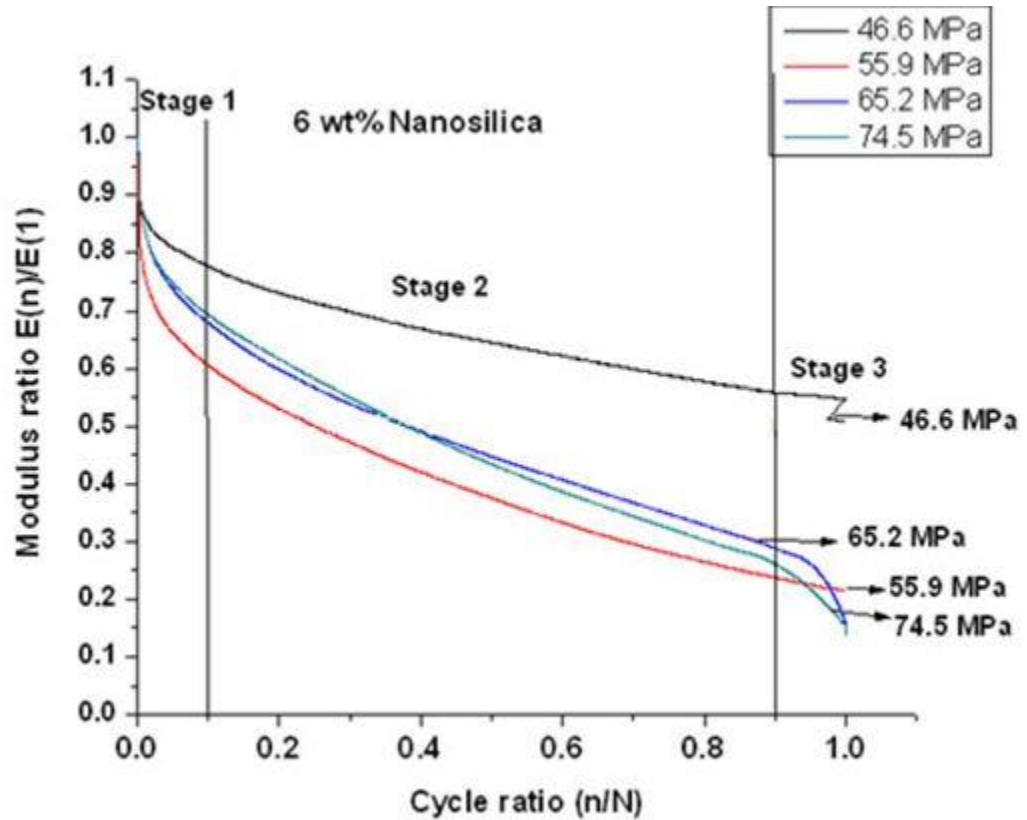
Figure 30: Stiffness Degradation Curve for 6 wt % Nanosilica Specimens [83]

Stage 1: In this stage, a number of matrix cracking increases rapidly along the fiber direction. This will continue until it reaches a saturation crack spacing called characteristic damage state (CDS). The modulus deceases rapidly in this stage.

Stage 2: In this stage, delamination and fiber breaking will occur. This causes a gradual modulus decrease in the specimen and 80% of the fatigue life constitutes in this stage

Stage 3: Fracture of the composite material occurs in this stage. The stiffness decreases rapidly in the last few cycles before the specimen fails.

Axial strain was measured using Axial Extensometer-632.24 E. 50. Increase in axial strain value during test indicates the loss of stiffness by the material.

2.5.2.5   *Brief Conclusion*

Static tests were performed on control and modified carbon composite to determine the behavior of epoxy resin with CTBN and nanosilica. Even though the tensile property of carbon composite is fiber dominant, matrix material does have some effect on tensile modulus and failure strain. Flexural properties are dependent on both fiber reinforcement and matrix. To determine the fiber/matrix adhesion of modified epoxy carbon composite material ILSS tests were performed.

Since, this research focused on improving the material standard related to the automotive field, the fatigue test is necessary. Prevention of fatigue failure is the most important parameter in designing the structural components. For example, a chassis of a truck supports all components of the vehicle which will experience fatigue and other kinds of load. Material which is used in manufacturing chassis frame should be light and rigid enough to withstand shock, twist vibration and other stresses. In this study, tension-tension fatigue test was conducted on carbon fiber reinforced epoxy composite, but based on the applications; tension-compression and compression-compression fatigue study could be conducted in future.

3. RESULTS AND DISCUSSION

3.1 Neat and Modified Resin Coupons

The following section will discuss about the static test results observed from neat and modified epoxy resin coupons. Tension and Flexural tests were performed on resin coupons. The toughening behavior of epoxy resin with CTBN and nanosilica is understood from the results.

	Tensile Strength (MPa)	Flexure Strength (MPa)
CTBN 2 wt%	44.74	55.09
CTBN 5wt%	36.88	55.78
CTBN 10 wt%	31.82	33.78
Control	59.84	51.48
Ns 2 wt%	62.132	51.79
Ns 5 wt%	65.3	53.86
Ns 10 wt%	67.78	54.94
Ns 20 wt%	74.014	67.28
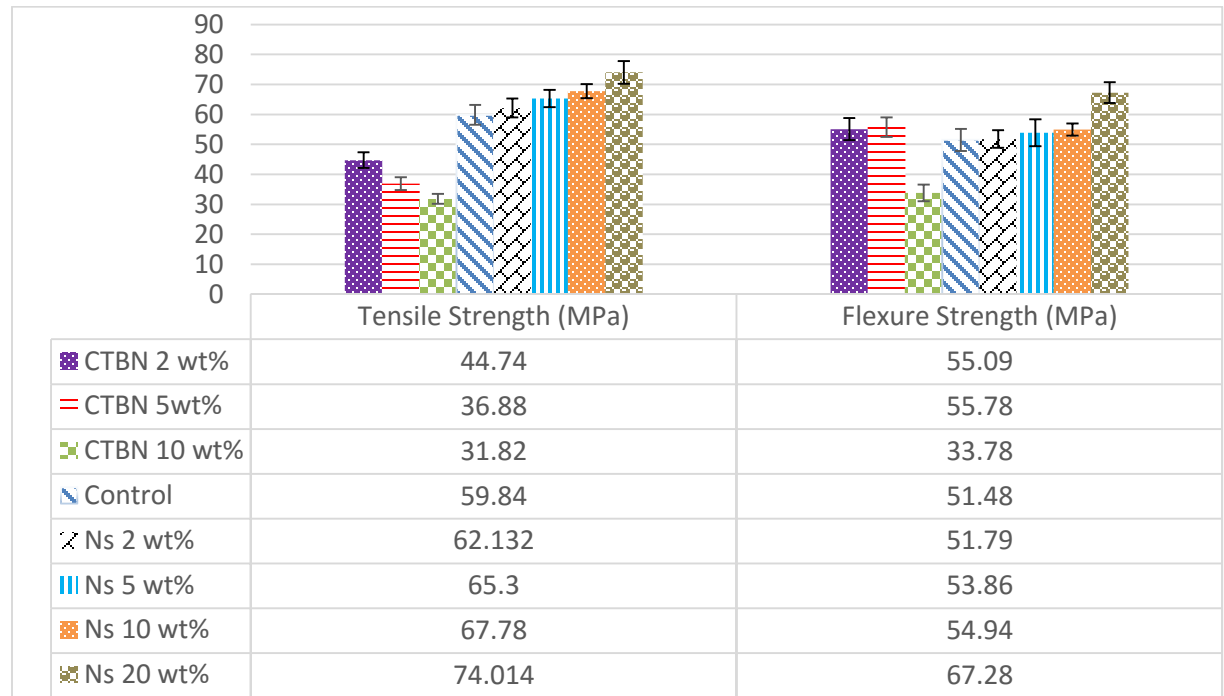
Figure 31: : Ultimate Tensile and Flexural Strength of Neat and Modified Epoxy Resin Coupons

	Tensile Modulus (GPa)	Flexural Modulus (GPa)
CTBN 2 wt%	2.45	2.55
CTBN 5wt%	1.978	2.01
CTBN 10wt%	1.578	1.24
Control	2.672	2.508
Ns 2 wt%	2.92	2.51
Ns 5 wt%	3	2.52
Ns 10 wt%	3.32	2.68
Ns 20 wt%	3.86	3.076
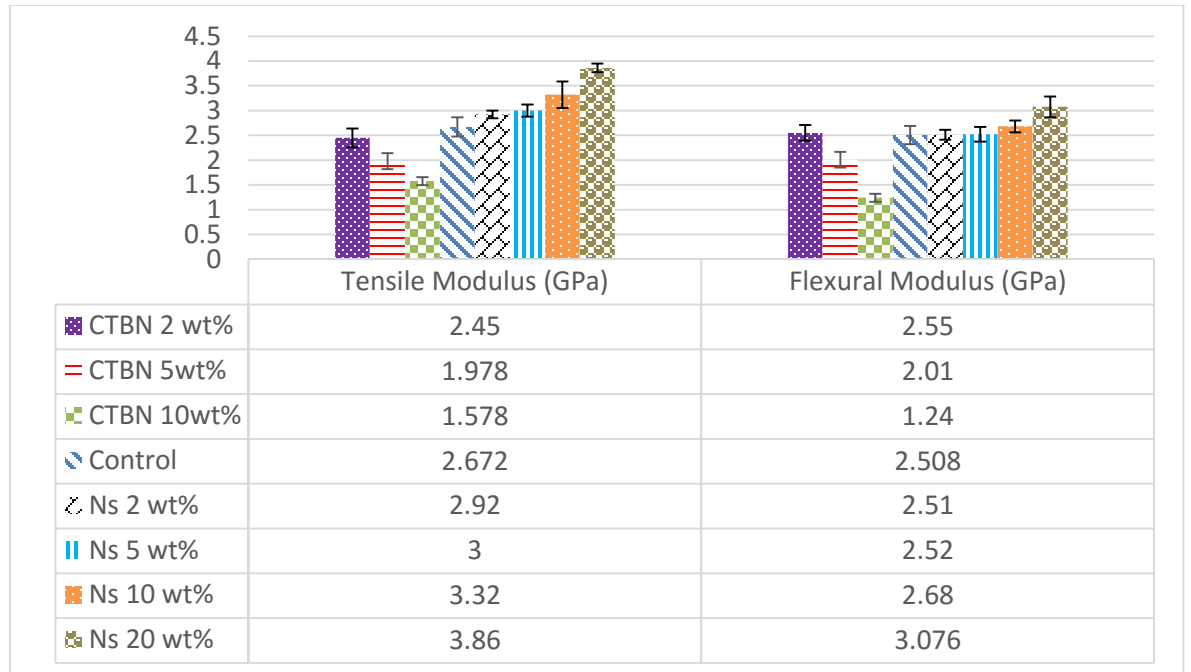
Figure 32: Tensile and Flexural Modulus of Neat and Modified Epoxy Resin Coupons

Table 5: Static Test Results of Neat and Modified Resin Coupons

Bulk Epoxy Resin Specimens	Control	CTBN 2 wt%	CTBN 5 wt%	CTBN 10 wt%	Ns 2 wt%	Ns 5 wt%	Ns 10 wt%	Ns 20 wt%
Tensile Strength MPa *(St.dev)*	59.84 *(3.33)*	44.74 *(2.65)*	36.88 *(2.11)*	31.82 *(1.66)*	62.132 *(3.14)*	65.3 *(2.89)*	67.78 *(2.38)*	74.014 *(3.77)*
Tensile Modulus GPa *(St.dev)*	2.672 *(0.195)*	2.45 *(0.19)*	1.978 *(0.161)*	1.578 *(0.081)*	2.92 *(0.078)*	3.0 *(0.122)*	3.32 *(0.27)*	3.86 *(0.086)*
Flexure Strength MPa *(St.dev)*	51.48 *(3.72)*	55.09 *(3.66)*	55.78 *(3.24)*	33.78 *(2.77)*	51.79 *(2.89)*	53.86 *(4.47)*	54.94 *(2.05)*	67.28 *(3.42)*
Flexural Modulus GPa *(St.dev)*	2.508 *(0.184)*	2.55 *(0.16)*	2.01 *(0.158)*	1.24 *(0.083)*	2.5 *(0.1)*	3.32 *(0.15)*	2.68 *(0.12)*	3.076 *(0.21)*

3.1.1   Tension Test on Epoxy Resin Coupons

Tension tests were conducted according to ASTM D 638 on epoxy resin coupons. Figure 30 and 31 show the ultimate tensile strength and tensile modulus of neat and modified resin coupons. As expected, the tensile strength and modulus of CTBN modified resin coupons was low when compared to neat resin coupons. Reduction in strength of the specimens indicates the presence of low modulus rubber particles and the plasticizing effect which is increased by rubber particles. Ratna D et al. observed the same results with different rubber particles [84].

The ultimate tensile strength and modulus of nanosilica modified specimens are improved when compared to control specimens. Increase in the percentage level of nanosilica improved the strength and modulus of epoxy resin coupons. Hong-Yuan et.al observed the same trend with nanosilica particles [43]; they also reported an increase in particle loading will improve modulus and toughness gradually.
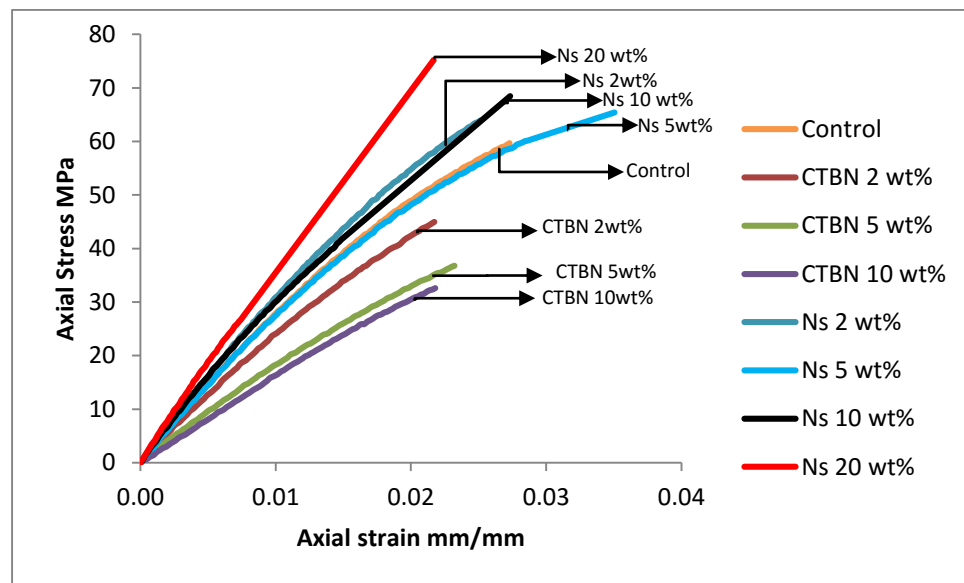
Figure 33: Stress vs. Strain Curve of Neat and Modified Resin Coupons

54

Figure 32, shows superimposed tensile stress-strain curves for neat and modified epoxy resin coupons. There was decrease in tensile strength and modulus of elasticity with 2, 5 and 10 wt% CTBN when compared to control composites. CTBN loading may undergo agglomeration of rubber particles, and it leads to the plastization effect which tends to show low strength and modulus [9]. Moreover, good dispersion of CTBN rubber particles in epoxy can be achieved with high amount of resin. The amount of resin used for casting of the samples was in the order of 141.46 ml. It can be concluded that there was not sufficient resin for uniform dispersion. With nanosilica modified resin coupons, tensile strength and modulus of elasticity are improved with all loading levels. This enhancement indicates excellent dispersion of silica nanoparticle in epoxy resin. Additionally, increase in % elongation with 5 wt% of nanosilica in epoxy resin determines the toughening mechanism evolved in the matrix system. Overall, nanosilica 20 wt% has achieved high tensile strength and modulus when compared to other specimens.

3.1.2   Flexural Test on Epoxy Resin Coupons

Flexural tests were conducted according to ASTM D 790 on epoxy resin coupons. Figure 30 and 31 show the Flexural strength and modulus of neat and modified resin coupons. A slight improvement in flexural strength was observed in epoxy resin coupons with CTBN 2 and 5 wt%. Nanosilica modification results were similar to the tensile test; flexural strength and modulus were improved with all loading levels when compared to control specimens.
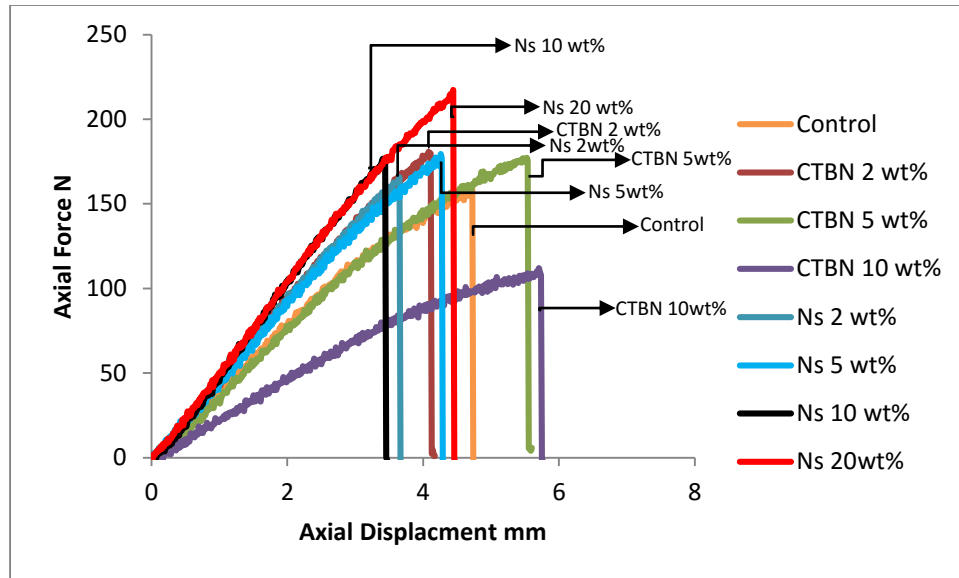
Figure 34: Load vs. Displacement Curve of Neat and Modified Resin Coupons

Figure 33, shows superimposed load vs displacement curves for neat and modified epoxy resin coupons. Even though the flexural strength and modulus of CTBN 2 and 5 wt% were enhanced, an excellent toughening mechanism towards bending moment was achieved by CTBN 10 wt%. From the above graph, we can observe that CTBN 10 wt% curve has travelled more towards x-axis (Axial displacement) when compared to other specimens, this trend indicates more bending moment has achieved by the particular CTBN loading level. This plastization effect is achieved by excess of CTBN loading in epoxy resin, which may undergo agglomeration of rubber particles. Through flexural test, nanosilica 20 wt% achieved high flexural and strength modulus when compared to other specimens.

3.2   Carbon Reinforced Epoxy Composite

The following section will discuss about the static and fatigue test results observed from control and modified carbon fiber reinforced epoxy composite. Static test on carbon composite were performed with control, CTBN 5 wt%, CTBN 10wt%, nanosilica 5 wt% and nanosilica 10 wt%. These loading levels were selected based on the static test results obtained from epoxy resin coupons. Analyzing the test results of the CTBN category, when compared to control specimen low modulus, was obtained by CTBN 5 and 10 wt% in both tension and flexural test; this scenario is an evident for toughening mechanisms occurred in epoxy resin system.

Nanosilica 20 wt% has attained high strength and modulus in overall static tests, but when considering the VARTM manufacturing process nanosilica 20 wt% would not suitable. Because the volume of resin mixture we use for carbon composite manufacturing is high when compared to resin coupons manufacturing. Since epoxy resin and other material systems used in this research possess very high viscosity, using 20 wt% nanosilica in carbon composite may affect the manufacturing process particularly with hybrid (CTBN + nanosilica) system. Moreover, nanosilica 5 and 10 wt% achieved high % elongation in tensile test when compared to other loading levels in nanosilica.

	Tensile Strength (MPa)	Flexure Strength (MPa)
☰ CTBN 5 wt%	535.81	390.57
▪ CTBN 10 wt%	610.7	398.85
◥ Control	540.17	564.65
‖‖ Ns 5 wt%	672.4	618.62
▦ Ns 10 wt%	681.16	687.97
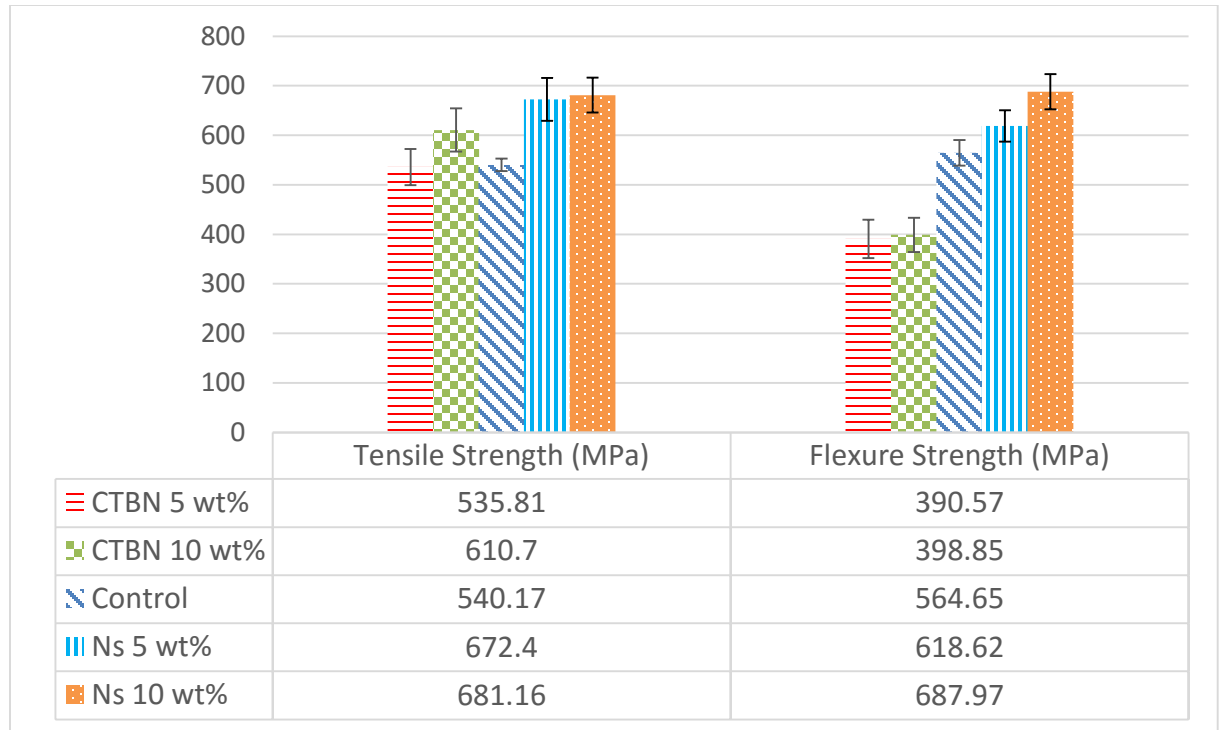
**Figure 35: Ultimate Tensile and Flexural Strength of Control and Modified Carbon
Reinforced Epoxy Composite**

	Tensile Modulus (GPa)	Flexural Modulus (GPa)
☰ CTBN 5 wt%	42.484	20.74
▪ CTBN 10 wt%	47.43	28.61
◥ Control	51.184	30.11
‖‖ Ns 5 wt%	52.28	34.046
▦ Ns 10 wt%	53.336	35.37
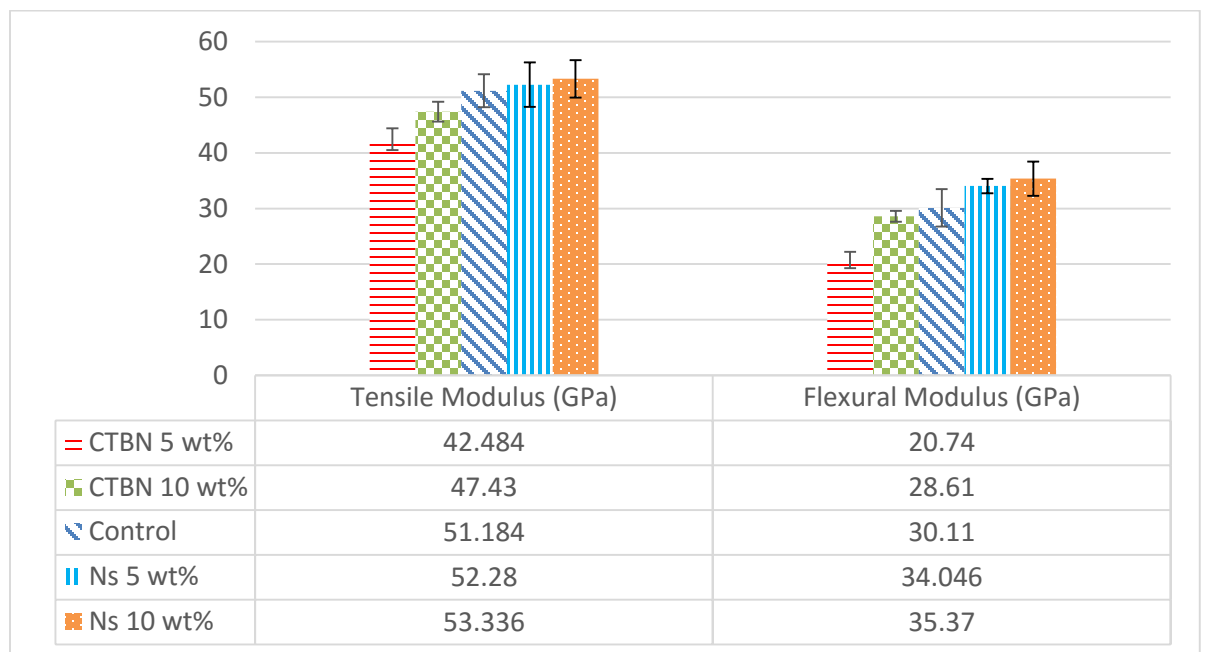
**Figure 36: Tensile and Flexural Modulus of Control and Modified Carbon
Reinforced Epoxy Composite**

Table 6: Static Test Results of Control and Carbon Reinforced Epoxy Composite

Epoxy Carbon Composite	Control	CTBN 5 wt%	CTBN 10 wt%	Ns 5 wt%	Ns 10 wt%
Tensile Strength (MPa) (St.dev)	540.17 *(12.71)*	535.81 *(36.47)*	610.7 *(43.55)*	672.4 *(43.44)*	681.16 *(35.4)*
Tensile Modulus (GPa) (St.dev)	51.184 *(2.95)*	42.484 *(1.95)*	47.43 *(1.78)*	52.28 *(4.01)*	53.336 *(3.37)*
Flexure Strength (MPa) (St.dev)	564.65 *(25.7)*	390.57 *(38.76)*	398.85 *(34.72)*	618.2 *(31.6)*	687.97 *(35.67)*
Flexural Modulus (GPa) (St.dev)	30.11 *(3.37)*	20.74 *(1.46)*	28.61 *(0.98)*	34.046 *(3.08)*	35.37 *(3.08)*

3.2.1   Tensile Test on Carbon Composite

Tension tests were conducted according to ASTM D 3039 on carbon composite. Figure 34 and 35 show the ultimate tensile strength and tensile modulus of control and modified carbon reinforced epoxy composite. From Table 6, we can observe the tensile strength of carbon composite with CTBN 10 wt% increased by 13 % when compared to control carbon composite specimen. Masanobu et al. observed similar results with CNBR (cross-linked acrylonitrile butadiene rubber) particles and they also mentioned the tensile strength will enhance with the loading rate range 5 -15% CNBR and it may decrease with increase in rubber content [85]. Moreover, the tensile strength in a composite material is fiber dominant property. With nanosilica 5 and 10 wt%, the tensile strength improved by 24.4 % and 26.1 % , the tensile modulus improved by 2.1 % and 2.15% respectively. In 2010, Manjunatha et al reported improvement in the tensile strength and modulus of epoxy polymer with 10 wt% nanosilica in glass fiber reinforced composite [86]
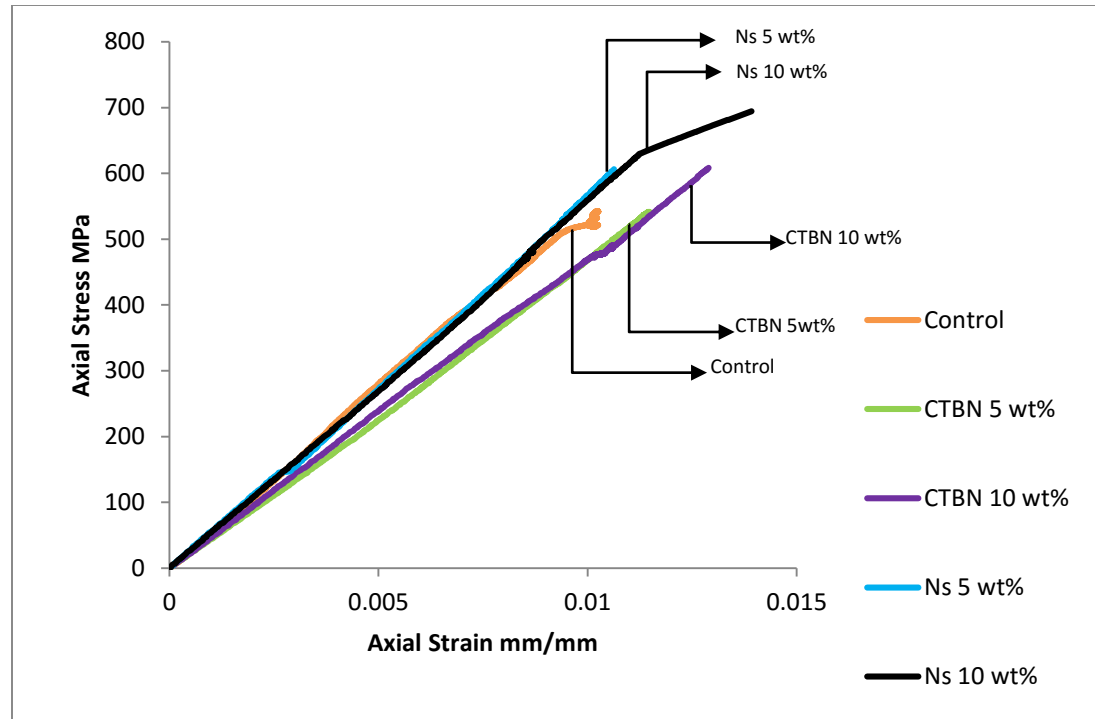
Figure 37: Stress vs Strain Curve of Control and Modified Carbon Reinforced Epoxy Composite

Figure 36, shows superimposed tensile stress-strain curves of control and modified carbon reinforced epoxy composite. When compare to control specimen, the tensile modulus of CTBN 5 and 10 wt% has dropped and % elongation has increased, but the tensile modulus of 10 wt% CTBN is greater than CTBN 5 wt%. Increase in % elongation indicates toughening of epoxy resin due to CTBN rubber particles. The volume of resin used for VARTM process was in order of 263.78 ml which is considerably excess for CTBN rubber particles to attain good dispersion. This scenario enhanced the tensile strength and % elongation of CTBN 10 wt% carbon composite. In nanosilica, the tensile modulus of 5 and 10 wt% carbon composite was high when compared to control and CTBNs carbon composite. Additionally, the % elongation of nanosilica 10 wt% was high when compared to other specimens. So, with this particular

60

loading level of nanosilica the tensile modulus is enhanced, simultaneously toughening of carbon composite is also achieved.

3.2.2   Flexural Test on Carbon Composite

Flexural tests were conducted according to ASTM D 790 on carbon composite. Figure 34 and 35 show the flexural strength and modulus of control and modified carbon reinforced epoxy composite. Results of flexural test are different from tension test because composite materials are anisotropic exhibit different strengths under tension, compression and flexural loadings. Moreover, flexural properties of composites depend on both fiber reinforcement and matrix. The flexural test applies tension load on bottom layer of the specimen and compression load on top layer of the specimen.
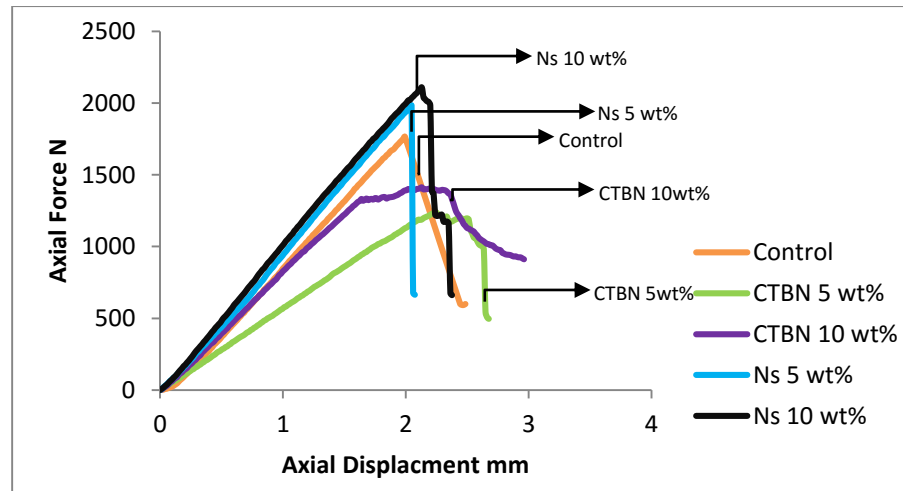
Figure 38: Load vs Displacement Curve of Control and Modified Carbon Reinforced Epoxy Composite

Figure 37, shows superimposed load vs displacement curves of control and modified carbon reinforced epoxy composite. Flexural strength and modulus of CTBN 5 and 10 wt% are decreased when compared to control specimens. With nanosilica 5 and

10 wt%, the flexural strength improved by 9.4% and 21.8%, the flexural modulus improved by 13% and 17.4% respectively.

3.2.3   Interlaminar Shear Strength

ILSS tests were conducted according to ASTM D 2344. This test is used to determine the fiber/matrix adhesion of composite material. As mentioned earlier, this test is similar to flexural test differs only with specimen dimension and span-to-thickness ratio i.e. 4:1. The specimen span-to-thickness ratio was constrained to 4:1, forced the shear stress to attain the failure levels before tension and compression reach their ultimate values [87]. ILSS plays a vital role in fatigue performance of the material.

	ILSS
CTBN 5 wt%	28.72
CTBN 10 wt%	12.21
Control	34.23
Ns 5 wt%	36.16
Ns 10 wt%	41.43
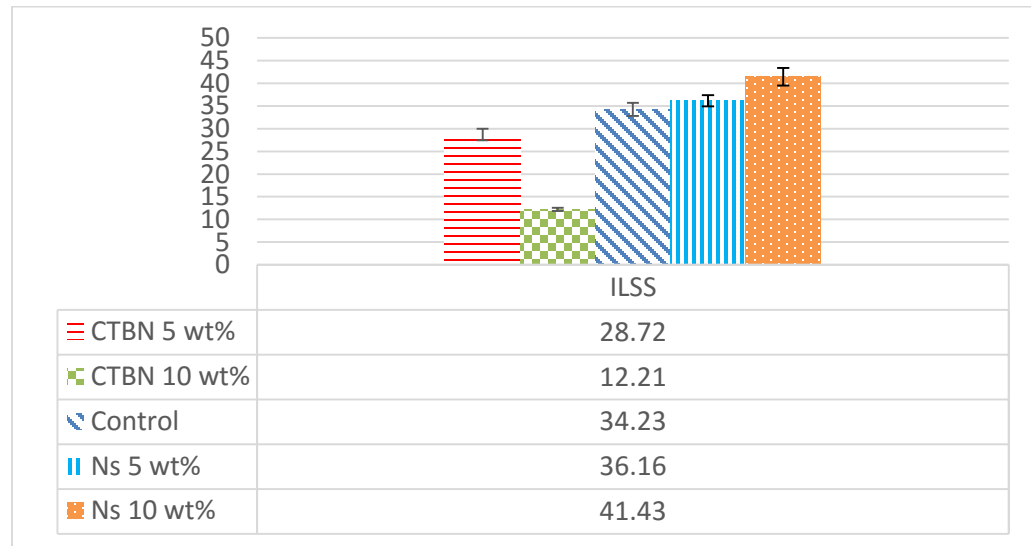
Figure 39: ILSS of Control and Modified Carbon Reinforced Epoxy Composite

Table 7: ILSS Results of Control and Carbon Reinforced Epoxy Composite

Epoxy Carbon Composite	Control	CTBN 5 wt%	CTBN 10 wt%	Ns 5 wt%	Ns 10 wt%
ILSS (Mpa) *(St.dev)*	34.23 *(1.46)*	28.72 *(1.29)*	12.21 *(0.37)*	36.16 *(1.21)*	41.43 *(1.93)*
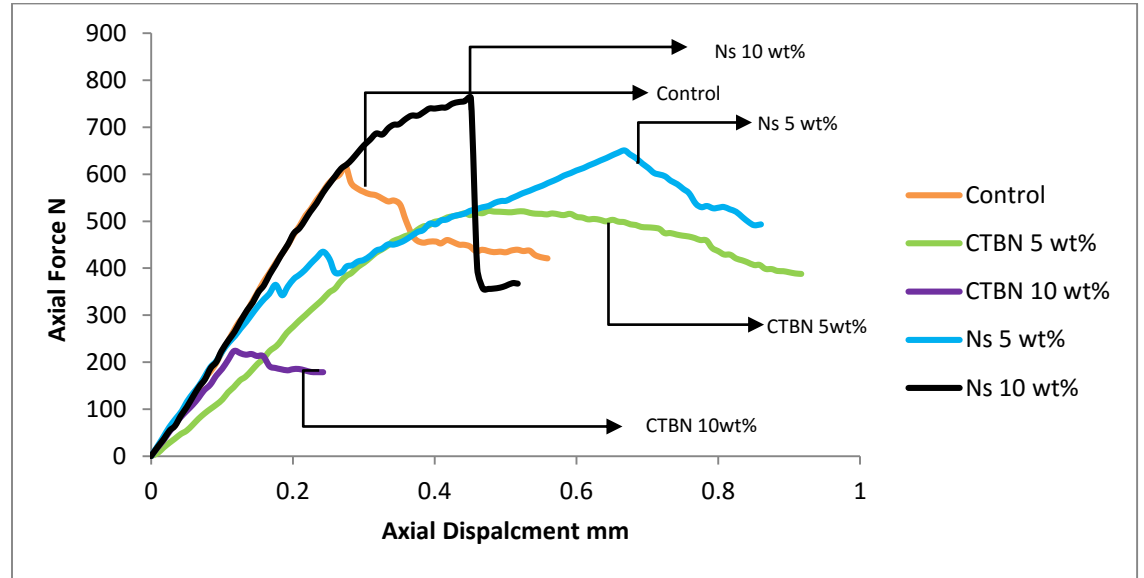
Figure 40: Load vs Displacement curve of Control and Modified Carbon Reinforced Epoxy Composite for ILSS

Interlaminar shear strength of CTBN 5 and 10 wt% specimens is significantly reduced when compared to control specimens. The agglomeration of CTBN rubber particles between carbon layers could reduce the adhesion between the matrix and reinforcement. At 10 wt% nanosilica, ILSS improved by 21 % when compared to control specimens.

3.2.4   Tension-Tension Fatigue on Carbon Composite

In this study, the tension-tension fatigue tests on carbon composites were performed according ASTM D 3479. Automotive components like vehicle frame and suspension systems are subjected to flexural fatigue. As mention earlier, the flexural load will apply both compression and tension loading on a component, so it will experience tension and compression loadings alternately. In comparing the load level stimulating is tension fatigue and flexural fatigue, tension fatigue will experience a high load level. If a material system withstands a high number of cycles in tension fatigue, certainly it has an excellent performance with flexural fatigue. From tension test results, the load levels selected for fatigue test are nanosilica 10 wt% and CTBN 10 wt%; even carbon composite with hybrid epoxy system is manufactured with the same load level.

Axial fatigue tests were performed under repetitive constant amplitude load cycles with stress ratio R= 0.1. In order to accelerate the fatigue study, all tension-tension fatigue tests were conducted with 10 Hz frequency, which took 27 hours to reach 1 million cycles. The percentage level of UTS applied in this study are 90%, 85 %, 80% and 70%; and their respective stress values are 486.15,459.14,432.13 and 378.11MPa. All tests were continued until the specimen break or it survives 1 million cycles. Number of cycles required for the failure determines the fatigue life of the material. Under each category three specimens were tested.

The modified carbon reinforced epoxy composites: nanosilica 10 wt%, CTBN 10 wt% and hybrid (CTBN 10wt% + nanosilica 10wt %) exhibited excellent fatigue life when compared to control composite. Similar results were observed by Masanobu et al and Manjunatha et.al with CNBR (cross-linked acrylonitrile butadiene rubber) rubber

64

particles and nanosilica respectively [85] [18]. All modified and control composites survived 1 million cycles at 378.11 MPa stress level. Table 8 shows, average fatigue life of control and modified carbon reinforced epoxy composites. Overall, nanosilica 10 wt% attained high fatigue life when compared to other categories.

Table 8: Fatigue Life of Carbon Reinforced Epoxy Composites at 10 Hz Frequency

Maximum Stress applied, MPa	Control (0 wt %)	CTBN 10 wt%	Ns 10 wt%	Hybrid (CTBN 10 wt% + Ns 10 wt%)
486.15	2013	240009	318813	2247
459.14	3391	301632	383557	257111
432.13	284616	1000000*	1000000*	542464
378.11	1000000*	1000000*	1000000*	1000000*

*Run out the specimen didn't break.

3.2.4.1 S-N Diagram

S-N diagram is used determine the relationship between fatigue stress and fatigue cycles (number of cycles). This diagram can predict the fatigue life of the material at a particular stress level. Figure 40 shows, the S-N diagram of control and modified specimens. All modified specimens showed enchantment in high and low cycle fatigue when compared to control specimens.
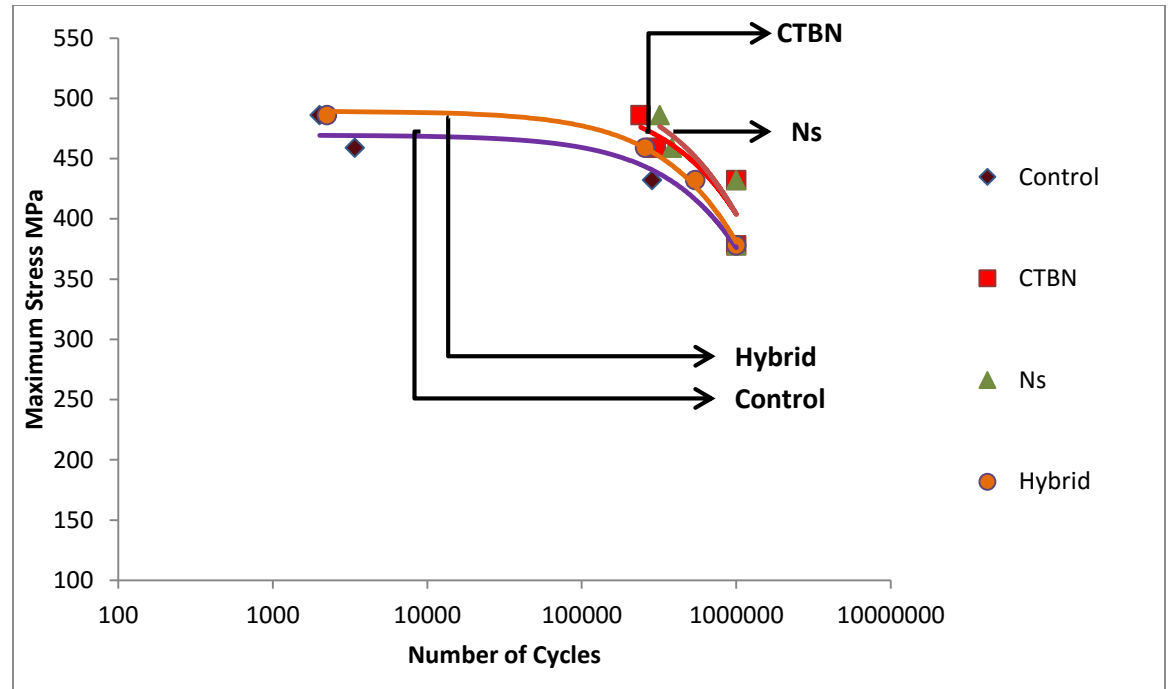
Figure 41: S-N Diagram of Control and Modified Carbon Reinforced Epoxy Composite

3.2.4.2 *Stiffness Degradation*

As mentioned earlier, the damage mechanism of a composite material is very complex. S-N curves are used determine fatigue life of the material, but it will not provide information about damage mechanism. Damage mechanism of a material can be studied through stiffness degradation method and it is the only parameter used to evaluate the life of a component. Stiffness of a material reduces when different damage mechanism like matrix cracking, fiber breaking and delamination occurs. Since, this study is focused on material improvement for high load application in the automotive field; stiffness degradation is focused only on high-cycle fatigue. Figure 41, represent the stiffness degradation curve of control and modified carbon reinforced epoxy composite at 378.11 MPa stress level; were all specimens reached 1 million cycles. If we observe S-N curve at this stress level all materials seem to have same fatigue life, but the stiffness

degradation curve will represent which material system has a superior fatigue life in three stages. In this curve X-axis represents cycle ratio n/N and Y-axis modulus ration E(n)/E(1).
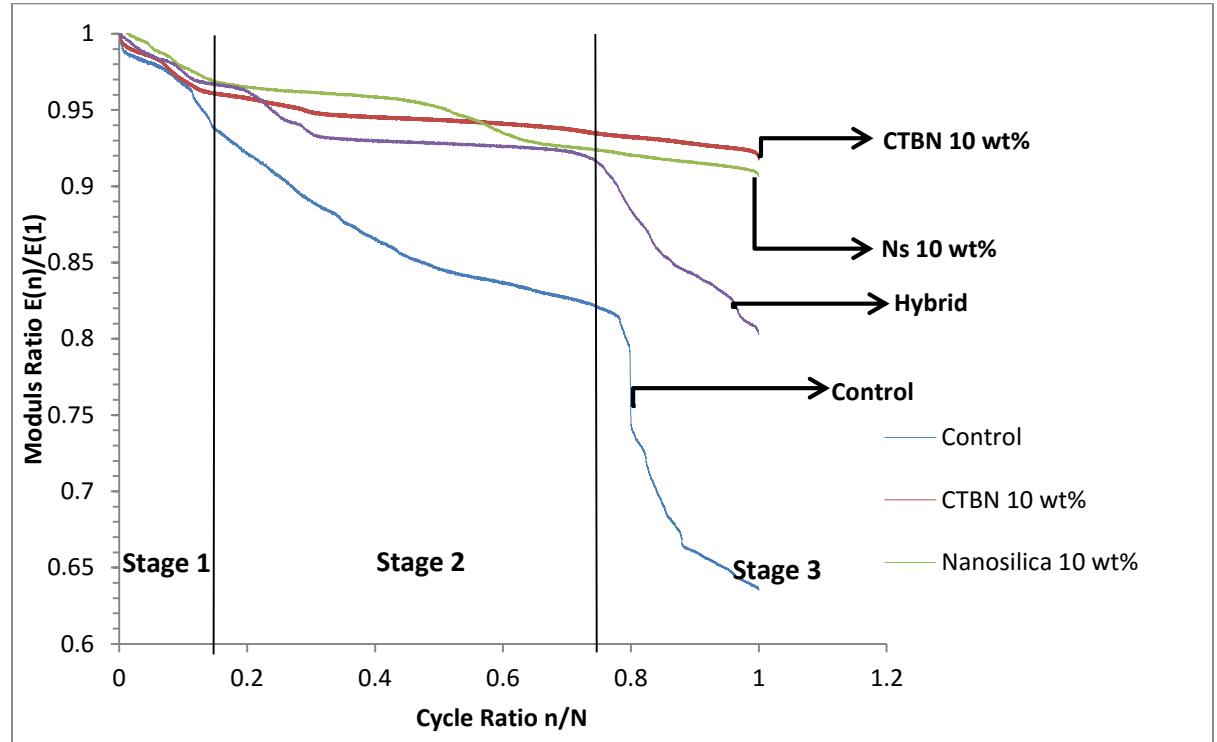
Figure 42: Stiffness Degradation Curve of Control and Modified Carbon Reinforced Composite at 378.11 MPa

Stage 1: In this stage, modulus was decreasing gradually for all material systems. This stage is about 15 % of the fatigue life. Control specimen lost 6 % modulus; CTBN specimen lost 4 % modulus; nanosilica and hybrid specimen lost 3% modulus.

Stage 2: Even in this stage, modulus was decreasing gradually, but the rate of the stiffness degradation is low when comparing to stage 1. The rate of stiffness degradation reduced for all composites except control composites, it maintained same rate in stiffness degradation as stage 1. Initially at this stage, hybrid and nanosilica marinated a rate in

stiffness degradation which is lower than CTBN. As cycles passed, the rate of stiffness degradation increased and become higher than CTBN. In this stage, control composites lost about 19 % modulus; CTBN composites lost 7% modulus; nanosilica and hybrid lost 8 % and 8.5% respectively. This stage covers 60 % of the fatigue life and there is an improvement in fatigue performance of modified carbon composites.

Stage 3: At this stage, the modulus of control and hybrid specimens suddenly dropped and it occurred because fiber delaminations. With nanosilica and CTBN the rate of stiffness degradation again increased gradually when compares to stage 2. This stage covers 25 % of the fatigue life. At the end of the stage, control composite lost 38 % of modulus; hybrid composite lost 20 % of modulus;
CTBN and nanosilica composite lost 8% and 9% of modulus respectively.

There is a significant enhancement of fatigue life with CTBN and nanosilica modified carbon reinforced epoxy composite when compared to control composite. With this same rate of stiffness degradation, nanosilica and CTBN carbon composites could even survive till 10 million cycles. Hybrid carbon composites might have high fracture toughness when compared to CTBN composites, but the fiber-matrix adhesion property could be lower than CTBN which affected its fatigue life. Stephan et al, observed reduction in ILSS of hybrid (CTBN + Nanosilica) laminates when compared to CTBN and control [22].It can be concluded that the agglomeration of rubber and nanosilica particles dropped the fiber-matrix adhesion of hybrid composite. Meanwhile, the agglomeration improved the fracture toughness of hybrid system which supported to have better fatigue life when compared to control specimens.

3.3   Analysis of Variance (ANOVA)

One-way analysis is performed to evaluate the variation observed in the results were statistically significant. Static test results of carbon composite showed superior enactment by adding CTBN and nanosilica 10 wt%. However, ANOVA shows that the variations observed with CTBN 10 wt% and nanosilica 10 wt% formulations are significant or not with alpha value 0.01.

Table 9: One -way analysis of variance with CTBN 10 wt% Formulation

Static Test	F	p-value	Recommendation
Ultimate Tensile Strength	12.08	0.00837	Reject
Tensile Modulus	5.985	0.0413	Fail to reject
Flexural Strength	73.64	2.63e-05	Reject
Flexural Modulus	0.906	0.369	Fail to Reject
Short-Beam Strength	1005	8.8e-10	Reject

Table 10: One-way analysis of variance with nanosilica 10 wt% formulation

Static Test	F	p-value	Recommendation
Ultimate Tensile Strength	70.24	3.12e-05	Reject
Tensile Modulus	1.15	0.315	Fail to Reject
Flexural Strength	39.33	0.00024	Reject
Flexural Modulus	0.873	0.238	Fail to Reject
Short-Beam Strength	43.88	0.000165	Reject

4. CONCLUSION

Carbon reinforced epoxy composites is a promising material in the present automotive field. Load bearing components like suspension, drive shaft and leaf springs are manufactured with carbon reinforced epoxy composite and they are superior to conventional materials like steel and aluminum in lower weight application. Since, epoxy resin systems are brittle in nature they are inferior to other conventional materials at high load applications. So, improving toughening property of epoxy resin system is considered to be an important study in present composite field. Infusion of either rubber or nano particles will improve the fracture toughness of epoxy resin systems. For this study, CTBN rubber particle and nanosilica particles were incorporated in epoxy resin. By performing static and fatigue test, the effects of CTBN and nanosilica particles in epoxy resin were evaluated.

Initially, static tests were performed on neat (0 wt%) and modified epoxy resin coupons. Three different loading levels 2, 5 and 10 wt% were used for CTBN and four different loading levels 2,5,10 and 20 wt% were used for nanosilica. Tensile strength, tensile modulus, flexural strength and flexural modulus were evaluated. A qualitative dispersion of rubber micro particles and nanosilica particles are attained by Planetary Centrifugal Vacuum Mixer, "THINKY ™" ARV-130. Nanosilica 20 wt% reported with 23.6% and 44.4 % improvement of tensile strength and tensile modulus respectively; 30.6 % and 48.8 % improvement of fatigue strength and modulus respectively when compared to control specimens. It is also observed that agglomeration of CTBN rubber particles in epoxy resin reported low strength and modulus, which indicates plastization effect of rubber particles.

Considering the viscosity of resin and the % elongation, the load levels selected for composite manufacturing are nanosilica 5 and 10 wt% , which are second and third highest in the strength and modulus of the static tests performed on epoxy resin coupons. From CTBN category the load levels selected for composite manufacturing process were 5 and10 wt%, which attained high % elongation. With VARTM process at elevated temperature high quality carbon composites were manufactured.

Both 5 and 10 wt% nanosilica with carbon reinforced epoxy composite improved in tensile strength, tensile modulus, flexural strength, flexural modulus and ILSS. Moreover, high % elongation of carbon composite is achieved by nanosilica 10wt%. The tensile strength of CTBN 10 wt% improved by 13 %, but the tensile modulus dropped with both CTBN 5 and 10 wt% and % elongation has increased

In flexural test, both nanosilica 5 and 10 wt% showed considerable improvement in strength and modulus. The flexural strength improved by 9.4% and 21.8%; flexural modulus improved by 13% and 17.4% respectively. With CTBN 5 and 10 wt% the flexural strength and modulus were decreased when compare to control specimens.

In ILSS, nanosilica 10 wt% showed 21 wt% when compared to control specimens. ILSS of CTBN 5 and 10 wt% has dropped; due to agglomeration of CTBN rubber particles between carbon layers reduced the adhesion between matrix and reinforcement.

Since the objective of this study is to discover a new material system for high load applications in the fields of automotive, fatigue study is necessary. Axial tension-tension fatigue tests (R=0.1) were conducted at 10 Hz frequency on control, CTBN 10 wt% , nanosilica 10 wt% and hybrid (CTBN 10 wt% and nanosilica 10 wt%). All material

systems reached 1 million cycle at 378.11 Mpa . From S-N curves, nanosilica 10 wt%

composite showed high fatigue life when compared to other materials systems. In order

to determine the damage mechanism of carbon composites, stiffness degradation curve

were formed on all specimens at 378.11 Mpa stress level. Overall, CTBN 10 wt% and

nanosilica 10 wt% showed significant improvement fatigue life when compared to

control specimens. Both CTBN and nanosilica carbon composite material systems could

even survive 10 million cycle at 378.11 MPa stress level.

The fatigue life of carbon reinforced epoxy composite with CTBN and nanosilica

particles is improved by the enchantment in fracture toughness of the epoxy resin system.

As mentioned in literature review section, different kinds of toughening mechanisms

would have been occurred to improve the toughening property of the epoxy resin system.

In this research, the enhancement in toughening mechanism is identified by the data

obtained from mechanical and fatigue characterizations. To identify the specific

toughening mechanism, SEM (Scanning Electron Microscopy) or TEM (Transmission

Electron Microscopy) analysis should performed in future

APPENDIX SECTION

HEXION

Technical Data Sheet

Re-issued September 2005

EPON™ Resin 828

Product Description

EPON™ Resin 828 is an undiluted clear difunctional bisphenol A/epichlorohydrin derived liquid epoxy resin. When cross-linked or hardened with appropriate curing agents, very good mechanical, adhesive, dielectric and chemical resistance properties are obtained. Because of this versatility, EPON Resin 828 has become a standard epoxy resin used in formulation, fabrication and fusion technology.

Benefits

- Fiber reinforced pipes, tanks and composites
- Tooling, casting and molding compounds
- Construction, electrical and aerospace adhesives
- High solids/low VOC maintenance and marine coatings
- Electrical encapsulations and laminates
- Chemical resistant tank linings, flooring and grouts
- Base resin for epoxy fusion technology

Sales Specification

Property	Units	Value	Test Method/Standard
Weight per Epoxide	g/eq	185 – 192	ASTM D1652
Viscosity at 25°C	P	110 – 150	ASTM D445
Color	Gardner	1 max.	ASTM D1544

Typical Properties

Property	Units	Value	Test Method/Standard
Density at 25°C	lb/gal	9.7	ASTM D1475
Denisty at 25°C	g/ml	1.16	
Vapor pressure @ 25°C (77° F)	mm Hg	0.03	
Refractive index @ 25°C (77° F)		1.573	
Specific heat	BTU/lb/°F	0.5	

Technical data sheet

Albipox 1000

Albipox 1000 is a high performance elastomer-modified epoxy resin based on Bisphenol A epoxy resin. It is used for the toughening of rigid and brittle epoxy resin systems. The elastomer used is a special nitrile rubber chemically linked to the epoxy resin. During the cure of the resin phase separation occurs, resulting in excellent properties of the end application.

A cured system compared with a similar unmodified epoxy resin shows improved properties:

- Improved toughness (fracture energy, fracture toughness, impact resistance)
- Much better mechanical properties at low temperatures
- Significantly improved adhesion to complicated substrates, e.g. oil-treated steel
- Nearly unchanged heat deflection temperature
- No or minimal reduction in hardness, thermal, chemical and ageing stabilities

Using Albipox 1000 toughened epoxy resin systems with excellent price/performance ratio can be formulated.

Albipox 1000 is silicone-free.

Albipox 1000 can be blended with all epoxy resins; no restrictions or incompatibilities exist. Any epoxy hardener desired can be used; the cure characteristics are not or nearly not affected by the toughening.

Fields of application

Albipox 1000 is used whenever a drastic improvement in toughness over the whole temperature range is required. This product is especially suitable for sophisticated structural adhesives. Some examples of applications are:

- Adhesives (very high toughness, significantly improved crash resistance, excellent adhesion, very good low temperature properties...)
- Reinforcement patches, e.g. for car construction
- Composites (improved interlaminar shear strength, increased punching resistance increased pressure resistance of pipes, improved processability...)
- Casts, structural foams, etc.

Application recommendations

Part of the epoxy resin used in the formulation to be improved is replaced by Albipox 1000. The amount of hardener is reduced corresponding to the new epoxy equivalent of the resin blend. For some non stochiometric hardeners like Dicyandiamide a change in hardener amount is unnecessary. Fillers and other ingredients of the formulation are used as usual. If the viscosity of Albipox 1000 should be

Technical data sheet- page 2

too high for the formulating procedure, we recommend to preheat the product to 60 – 80 °C. The viscosity will be decreased below 10.000 mPa·s.

Best improvements in properties are found usually in the range of 12 – 15 phr rubber (parts per hundred resin).

Formulation examples:

	Original formulation	10 phr NBR	12 phr NBR	15 phr NBR
Standard Bisphenol-A epoxy resin (EEW 185)	100	85	82	77,5
Albipox 1000 (EEW 330)	-	25	30	37,5
Total mass parts	100	110	112	115
EEW	185	206	210	216

Technical data Albipox 1000 (no specification)

Property	Units	Typical values
Appearance		yellowish resin
NBR rubber content	[%]	40
Base resin		Bisphenol A epoxy resin
Density @ 20 °C	[kg/m^3]	1080
Viscosity @ 25 °C	[mPa·s]	200.000
Epoxy equivalent weight		330
Shelf life	[months]	6 *
Packaging		180 kg steel drum, 25 kg can

*if stored in the original unopened container

75

Technical data NANOPOX® products (no specification)

Product	Base resin	SiO$_2$ content (wt%)	Viscosity (mPas)	Epoxy equivalent weight	Product specific
NANOPOX® F 400	DGEBA	40	60,000	295	standard grade
NANOPOX® F 440	DGEBA/DGEBF	40	45,000	290	crystallization-free
NANOPOX® F 520	DGEBF	40	20,000	275	standard grade
NANOPOX® F 631	cycloaliphatic epoxy	40	4,000	220	standard grade
NANOPOX® F 640	HDDGE	40	200	245	low viscosity
NANOPOX® FW 404	aqueous emulsion of NANOPOX® F 400	40 (on solid)	7,000	295 (solid)	56% solids content
NANOPOX® F 700	Epoxidized Novolac	40	20,000 (at 50°C)	310	high performance

Packaging

Steel drum, 25 kg
Steel drum, 250 kg
Other types of packaging available on request

Shelf-life

Six months if stored in the original unopened container.

Application Recommendations

Part of the epoxy resin used in the formulation to be improved is replaced with a NANOPOX® F product (up to 100%). The amount of hardener is reduced in proportion to the new epoxy equivalent of the resin blend. For some non-stochiometric hardeners like dicyandiamide a change in the hardener amount is unnecessary.

Fillers and other ingredients of the formulation are used as usual. If the viscosity of NANOPOX® products prove too high for the formulating procedure, we recommend preheating the product to 60–80 °C. The viscosity will be decreased below 10,000 mPas.

Best improvements in composites properties are found usually in the range of 8–10% silica nano-particles.

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