

**DYNAMIC MECHANICAL CHARACTERIZATIONS OF
CARBON FIBER - POLYURETHANE and EPOXY
COMPOSITES**

**M.Sc Thesis by
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Programme: Polymer Science and Technology

JANUARY 2008

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JANUARY 2008

**KARBON FİBER - POLİÜRETAN VE EPOKSİ
KOMPOZİTLERİNİN DİNAMİK MEKANİK
KARAKTERİZASYONU**

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LIST OF ABBREVIATIONS

CF	: Carbon Fiber
DMA	: Dynamic Mechanical Analysis
PU	: Polyurethane
SEM	: Scanning Electron Microscope

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DYNAMIC MECHANICAL CHARACTERIZATIONS OF CARBON FIBER - POLYURETHANE and EPOXY COMPOSITES

SUMMARY

The aim of this thesis is to investigate the dynamic mechanical properties of reinforced carbon fiber composites which contain polyurethane and epoxy. Carbon fiber was used reinforcement agent in chopped and bundle form. Curing method was used for epoxy composites and polymer systems; also casting method was used to prepare polyurethane composites. Two kinds of carbon fibers were used for epoxy composites. Mechanical properties of epoxy or polyurethane composites were studied by using dynamic mechanical analysis. Storage modulus, loss modulus, tan delta and complex modulus of composites were investigated at different temperatures. The stress-strain relationship was studied for five and fifth day left day samples for carbon fiber reinforced composites. There were significant developments obtained on modulus values after fifth days for epoxy composites. In the first part of study, epoxy has been reinforced with carbon fiber. Their mechanical properties were studied with dynamic mechanical analysis. From stress strain curves, values of young modulus were obtained. Scanning electron microscopy was used for morphologic study. Also, glass transition values of samples were obtained from tan delta curves with dynamic mechanical analysis. Glass transition values of samples were effected by carbon fiber content. In the second part, polyurethane and carbon fiber composite was presented. While increasing amount of carbon fiber in an epoxy composite, it affected the mechanical properties. In addition, young modulus, toughness, maximum stress and strain were developed with carbon fiber. There were results that using bundle of carbon fiber was resulted in significant increase for modulus of polyurethane composites. Carbon fiber improved the mechanical properties of polyurethane-carbon fiber composites. In the last part, both polyurethane and epoxy has been reinforced with chopped carbon fiber. Here, the amount of carbon fiber and hardener were changed. Glass transition of epoxy-polyurethane-carbon fiber composites were investigated, the values of glass transition were changed with the amount of hardener. Also, scanning electron microscopy was used for morphologic study. The differences on surface of composites were studied with SEM.

KARBON FİBER - POLİÜRETAN VE EPOKSİ KOMPOZİTLERİNİN DİNAMİK MEKANİK KARAKTERİZASYONU

ÖZET

Epoksi ve poliüretan içeren karbon fiberle güçlendirilmiş kompozitlerin dinamik mekanik özelliklerinin araştırılması amaçlanmıştır. Bundle şeklinde ve kısa kesilmiş karbon fiberler güçlendirme ajanı olarak kullanılmıştır. Epoksi kompozitleri için “curing” metot, poliüretan kompozitleri içinse “casting” metot kullanılmıştır. İki tür karbon fiber kullanılmıştır. Epoksi veya poliüretan kompozitlerin mekanik özellikleri dinamik mekanik analiz ile araştırılmıştır. Farklı sıcaklıklarda, kompozitlerin elastik modulus, loss modulus, kompleks viscosity and tan delta değerleri incelenmiştir. Ayrıca çekme uzama deneyleri de farklı sıcaklıklarda çalışılmıştır. Epoksi kompozitleri, elli gün sonunda yüksek bir modül değerine sahip olmaktadır. Çalışmanın ilk kısmı karbon fiber içeren epoksi kompozitlerini içermektedir. Dinamik mekanik analiz ile mekanik özellikleri araştırılmıştır. Çekme-gerilme eğrileri kullanılarak young modül değerleri elde edilmiştir. Taramalı elektron mikroskopu ile morfolojik araştırması gerçekleştirilmiştir. Dinamik mekanik analiz sonucu elde edilen tan delta eğrilerinden örneklerin camsı geçiş sıcaklıkları belirlenmiştir. Camsı geçiş sıcaklıkları karbon fiber içeriğiyle orantılı olarak değişmektedir. İkinci kısımda karbon fiberle güçlendirilmiş poliüretan kompozitleri üzerinde araştırma yapılmıştır. Karbon fiber miktarının artması mekanik özellikleri etkilemektedir. Buna ek olarak young modulus, dayanıklılık ve maksimum çekme ve uzama değerleri fiber miktarına bağlı olarak değişmektedir. Bundle halde kullanılan fiberle yapılan poliüretan deneylerinde, yüksek modül değerleri elde edilebilmiştir. Son bölümde, poliüretan ve epoxy birarada kullanılarak carbon fiber ile güçlendirilmiştir. Karbon fiber miktarı ve hardener miktarı değiştirilerek deneyler yapılmıştır. Numunelerin camsı geçiş sıcaklıkları tan delta eğrilerinden hesaplanarak hardener miktarının camsı geçiş sıcaklığı üzerine etkisi incelenmiştir. Hardener miktarı camsı geçiş sıcaklığını değiştirmektedir. Ayrıca, taramalı elektron mikroskopu kullanılarak morfolojik araştırması yapılmıştır, yüzeydeki değişimler araştırılmıştır.

1. INTRODUCTION

Composite materials are widely used for many areas for research and manufacturing due to their eligible, convertible and adjustable properties. Composites have replaced and changed traditional methods. Composite materials have been used widely for daily life applications for many years. Polymer composite systems have a large scale for industry or research area due to their light weight, design flexibility, and processability [1,2]. Using polymer composite is widespread to use for special engineering materials such as aerospace industry, automotive and civil engineering structures because of their outstanding mechanical properties. There is a great interesting to have knowledge of composite materials. Polymer composite theories were established on properties of composite constituents, volume fraction, shape, matrix-inclusion interface [3,4,1,2]. Mechanical properties of polymer composites are significant when they use for a building or an instrument which has critical points about temperature and strength properties [5-7]. Many types of composites in which have matrices and reinforcement materials are used in industry for instance, carbon and glass fiber composites [8]. Fiber and particulate reinforcement affect the mechanical properties of plastics positively, adding reinforcement material to composite reduces costs comparing similar material [9-21]. Composite materials manufactures for one or more industrial areas, and their manufacturing recipes must be prepared about their application area. The environmental conditions affect applications of composites and resistance to deformation in mechanical properties so it is important that applications area and methods for manufacturing a composite [22].

In composites, two materials are mixed to make improvement in the mechanical properties. Matrix material is dominant and including material. The second material is particle, filler or fiber. Composites are man made materials. Materials have properties such as strength (compressive, tensile, flexural, etc), toughness (impact resistance), stiffness (modulus of elasticity), wearing resistance, thermal insulation, fatigue life. These properties of materials can be improved by composites.

Composite materials properties have been developed such as chemical, physical and mechanical, so their application area comes much bigger.

Because of improvements on composites materials, their industry are growing on automotive, aerospace, electronics and biotechnology [23,24]. The important point of using nanocomposites is large scale, low cost, easy applicable [25].

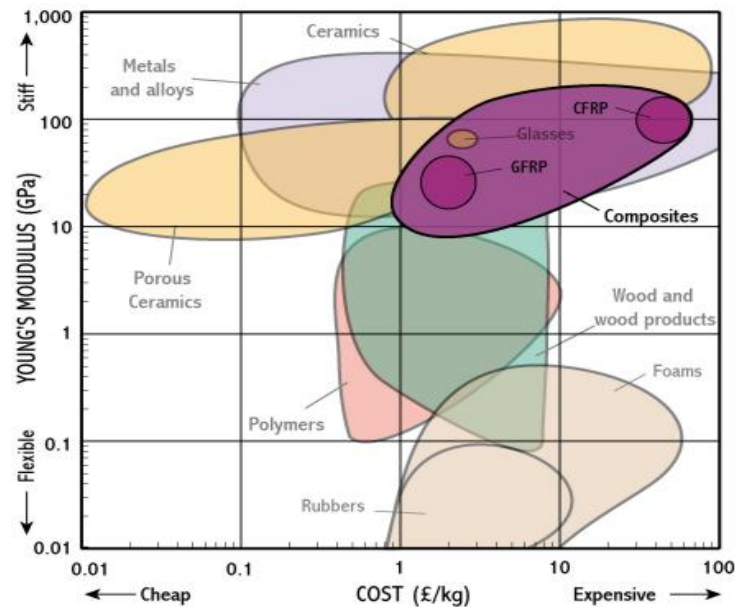


Figure 1.1: Classification of materials by young modulus

Carbon fiber (graphite fiber) is one of the popular materials that used preparing composites. The synthetic carbon industry starts with the foundation of National Carbon Company in Ohio, and continues Union Carbide Corp. High performance carbon fibers were developed at the Parma Technical Center by Dr. Roger Bacon in 1958. The atomic structure of carbon fiber is similar to graphite. Carbon fibers are classified by the tensile modulus of the fiber; low modulus, standard modulus, intermediate modulus, high modulus, ultrahigh modulus. Polyacrylonitrile is one of the raw materials that used to manufacture carbon fiber.

Fibers are used for plastic industry because of their considerable properties such as hardness, elastic modulus, mechanical strength, impact strength and dimensional control [26]. Fiber reinforced polymer matrix materials in which nano and micro-scale particles have been studied because of their potential and capacity of performance for twenty years [27]. Due to their high specific and stiffness properties, carbon fiber reinforced composites are widely used structural materials for aerospace, marine, armor, automobile, railways, civil engineering structures, sport goods industry [28]. Carbon fibers are used for many different industries also carbon fiber composites. For carbon fiber composites, matrix has an important role.

Carbon fiber is used for many polymer matrix composites. Carbon fiber and resin matrix interface is very important for determining properties of composites such as toughness or environmental endurance[29]. Nature of interface, bonding properties and surface morphology can change the composite fracture toughness[30]. Characterization of matrix resin is related with fiber, compression and thickness properties for composite polymer matrix [31]. Also characteristics, dimensions, shapes of the inorganic fillers and interfacial properties regulates attributes of polymer matrix composites [32]. The surface of fiber is about bonding force between plastic material and fiber [33]. Fiber and matrix interface is very important for the mechanical properties of polymer composites. Adjusting polymer composite properties is related with interphase characteristics and static and fatigue properties of composites [34]. An important point for an interaction is about transferring the load from matrix to fiber on composites, it effects mechanical properties of polymer matrix composites [35]. Akbar et al determined for all loading conditions, fiber/matrix behavior effects strength and toughness of composite materials [36].

Due to their low density, good processability and cost advantage, plastics are widely used for industry. Plastics can be separated by flexible and rigid plastics. Rigid plastics are called thermosetting polymers. Rigid plastics have high rigidity, high resistance to deformation, high modulus, high tensile strength and small elongation properties. Amorphous polymers have very rigid chains. Thermosetting polymers can not be reshaped by heating. They can be caused to undergo crosslinking to produce a network polymer. In these polymers, the resulting three dimensional networks lose its solubility and do not exhibit a melting point, since the individual chains may no longer flow past one another. Some of rigid plastics are epoxy, unsaturated polyester, phenol-formaldehyde, urea-formaldehyde, melamin-formaldehyde. Flexible plastics are also called thermoplastics. Flexible plastics have high degree of crystallinity wide range of T_g and T_m values. The elongation of a plastics results in permanent deformation. The plastic will retain its elongated shape when stress is removed. Thermoplastics can be softened and hardened reversibly by changing temperature. It can be heated until it becomes soft in order to process into a desired form. Also it can be recovered by application of heat and pressure(polystyrene, polyethylene, polypropylene, PVC). Fabrication processes like injection molding, extrusion molding and blowing are using to shape thermoplastic resins. The rigidity of thermoplastic resins changed at low temperature due to the existence of secondary forces between the polymer chains. Some of flexible plastics is polyethylene (HDPE, LDPE), poly(hexamethylenedipamide), polycarbonates, polymethylmethacrylate (PMMA) and polypropylene. But some composites have some negative attributes because of their characteristics [37].

Polypropylene (PP) is a semicrystalline polyolefin plastomer, and PP is known an outstanding polymer material. PP has good properties such as wide spectrum performance, easy processing, chemical resistance, heat resistance, and low cost in industry [38]. Polypropylene (PP) has a widely usage area used in packaging, textile, automobile due to its good processibility. But it has a limited usage because of poor impact resistance at room temperature [39]. PP has some disadvantages such as low toughness, service temperature, thermal stability, so researchers study to improve PP's mechanical properties by nanoscience [40].

PP is widely used for engineering functions by especially reinforced materials. It is used for containers, battery applications, and bumper applications except for automotive and textile industry [41,42]. It can be used fiber and fiber orientation (long or short fiber) on polymer composites for polymer mechanical properties. This can be balanced with every types of fibers [43]. Mechanical properties of PP can be improved using reinforcements such as carbon fiber and glass fiber by extrusion machine [44]. The mixture of carbon fiber and PP can be prepared in the required length by extrusion machine; they are called fiber reinforcement material [45]. It can be achieved composites which has different mechanical properties by using various fiber structure, size, distribution. The main parameter of composite materials is characteristics of polymers, contents especially fiber content and fiber length. The optimal choices about contents (fiber, PP etc.) provide appreciate products [11,12, 47,20]. Because of controlling the final properties of the composites, the ratio of reinforcement and its adhesion to matrix have significant [48]. When nanoparticle reinforcements use for polymer composites, reinforcements can demonstrate significant improvements compare to the others [49,50,51]. Due to sensor and actuator applications, smart materials and nanoparticles reply the requirements about environmental conditions [52,53].

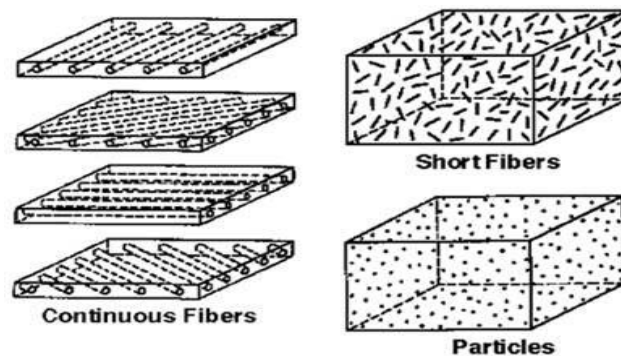


Figure 1.2: Different types of fiber composites

1.1. Epoxy Resins & Composite Applications

The first epoxy products were synthesized in 1891. In the 1930's, Pierre Castan (from Switzerland) and Sylvan Greenlee (in USA) patented their works at the same time, independently. The first commercial products marketed in the 1940's, and it was result of product was bisphenol A and epichlorohydrine reaction. One of the epoxy components is hardener, epoxy resins depend on the reactivity of hardeners [54]. Epoxy resins are cured with different curing agents like amines, amides, acids, acid anhydrides and amine adducts. Cycloaliphatic curing agents better properties for applications of epoxy area such as weather ability, low blush and water spotting, and chemical resistance.

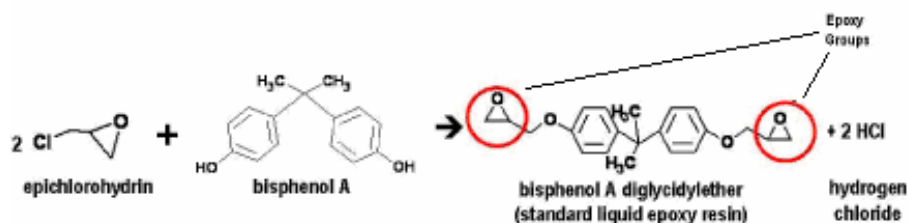


Figure 1.3: The reaction mechanism of standard epoxy resin

By using suitable epoxy resin and hardener we can obtain materials which have significant mechanical, physical and chemical properties in order to use in a widely usage area. Because of resin chemistry and available applications, epoxy resins have been available for many major applications such as surface coating, tooling, civil engineering, moulding compounds.

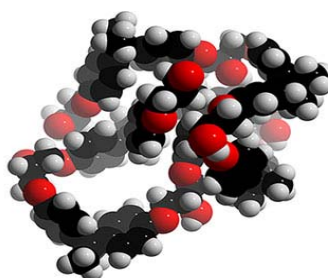


Figure 1.4: 3D View of Epoxy Resin

Epoxy resins can manufactured from epichlorohydrine, ,bisphenol A, bisphenol F, tetrakis phenylolethane, resorcinol, metyhlotated phenol, brominated and fluorinated phenols.

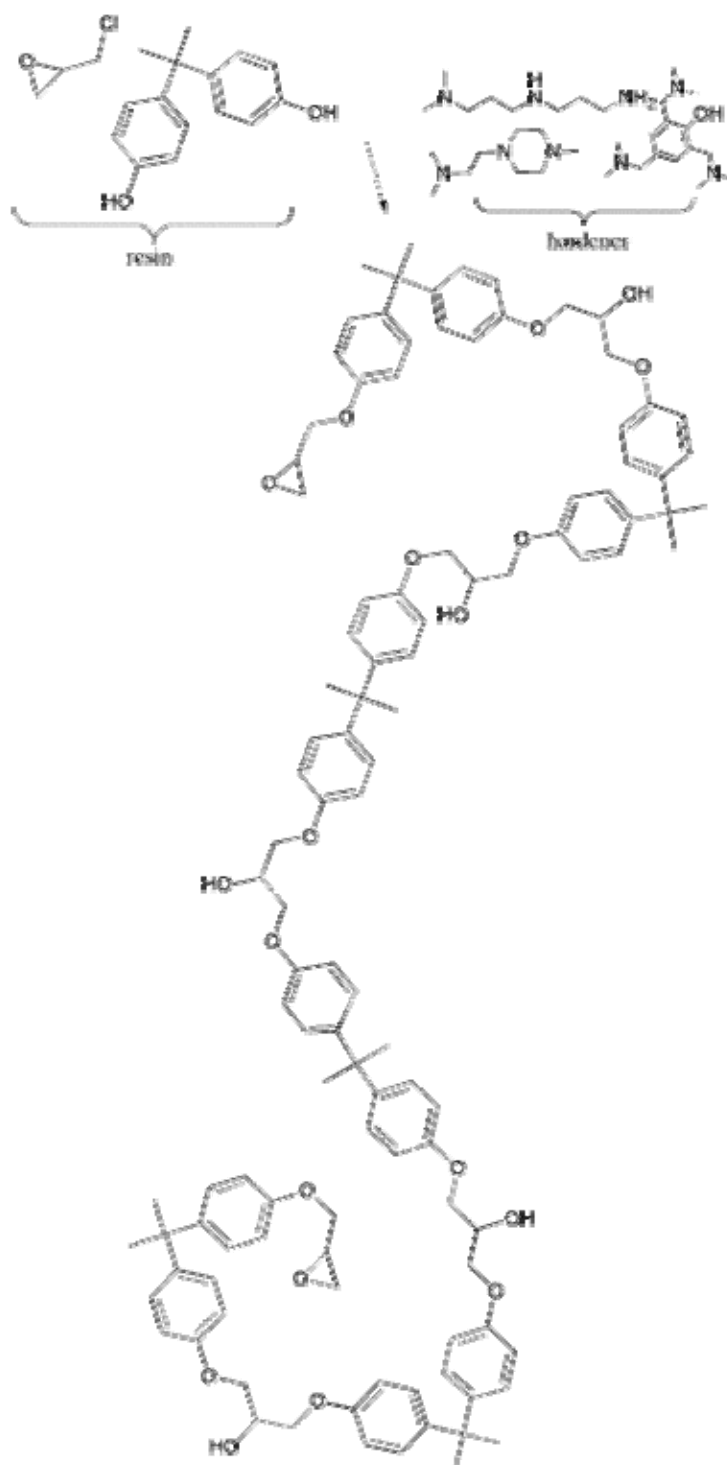


Figure 1.5: Schematic Diagram for Epoxy Synthesis From Resin and Hardener

Epoxy resins have widely been used in industry for sixty years such as aircraft applications, the car industry, reinforcement to various concrete structures also thin film coating, electronic circuits [55]. Epoxy resin is mostly used as a matrix in material engineering due to their high stiffness, resistance of creep and chemical, also good adhesion [56].

The mechanical properties of epoxy resins can be modified because of its structure. Mechanical properties can be improved by crosslink density increasing due to molecular architecture and structure to get high endurance [57]. It can be obtained resins which have properties: toughness, chemical resistance, high strength and hardness, high adhesive strength, good heat resistance and high electrical insulation connected with chemical structure of curing agent and conditions of curing [58]. Because of available variation of epoxy components and reactions, many different resins can be obtained [59,60]. Epoxy resin systems have wide usage industrial area in composite applications such as automotive and aerospace applications, for shipbuilding or electronic devices with surface coatings [61]. There are studies on polymer-based composites reinforced with filler to obtain high mechanical and high thermal polymer composites. There are some techniques such as effecting on processability, appearance, density, and ageing performance of the matrix to get these polymers [62]. Composite materials (graphite–epoxy etc) studied for industrial requirements whether the “neat” or pure resin because of their kinetics properties. In terms of ease of handling and sample, the composite is often easier to work with [63]. Epoxy resins are mostly used with fiber to obtain reinforced composites. An epoxy resin with fiber reinforced has advantages such as good stiffness, specific strength, stability, chemical resistance, and show good adhesion to the fiber [64]. Due to their strength, high modulus and light weight, epoxy-carbon fiber composites can be used for aircraft industries.

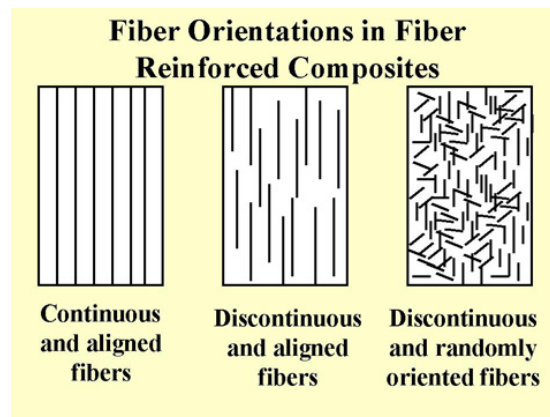


Figure 1.6: Types of using fiber for reinforced composites

1.2. Polyurethane and Carbon Fiber Composites

In the past twenty years, polyurethane industry has matured so much, reflecting the increasing and changing demands of a global market. Manufacturing of polyurethanes shows many variations and polyurethanes can be manufactured different variations; stiffness from flexible elastomers to rigid properties or density from 6 to 1,220 kg/m³. Polyurethane is made by mixing together the ingredient chemicals (isocyanate and polyol) in predetermined proportions. The simplest PU is linear in which the hydroxyl compound and the nitrogen compound each have a functionality of two. This can be represented by the following:

Isocyanate + Polyol = Polyurethane

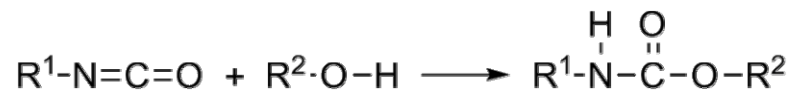


Figure 1.7: Synthesis of Polyurethane from isocyanate and polyol

A polyurethane formulation is significant to manufacture polyurethane composites. It can be achieved various composites content polyurethane with production methods such as the high speed, high density RRIM process. For polyurethane composites, there are two main steps in all processes, but all of processes depend on MDI(methylene diphenyl diisocyanate) and using high pressure machines. Many types of fibers are used polyurethane composites such as e-glass, a-glass, s-glass, c-glass, continuous fiber, woven glass cloth, woven roving (WR), chopped glass, chopped strand mat, natural fiber and carbon fiber. Polyurethanes are widely used for many applications such as automotive, furniture, construction, thermal insulation, footwear, and textile. The advantages of polyurethane composite are strength, stiffness and lightness properties; also it can be used for new applications for transportation, marine, electrical and construction products. Because of specific advantages in processability, material properties and condition of manufacture, polyurethane products increase. Polyurethanes are considerable materials due to their physical and chemical properties such as high tensile strength, weather resistance, low temperature resistance, wide range of rigidity after all, they are used for many applications for example biomedical, coatings, foams, adhesives, thermoplastic elastomers and composite [65].

Mechanical deformation is a significant problem for polyurethanes, and researchments on polyurethane can improve polyurethanes properties. Studies about PU with carbon fiber reinforcement have received attention because of elasticity of PU and rigidity of carbon fiber. PU-CF composites have a wide area for industry such as vehicle interiors, sporting goods, electronics, and constructing materials. Short carbon fibers can be put into composite matrix with molding etc [66-70].

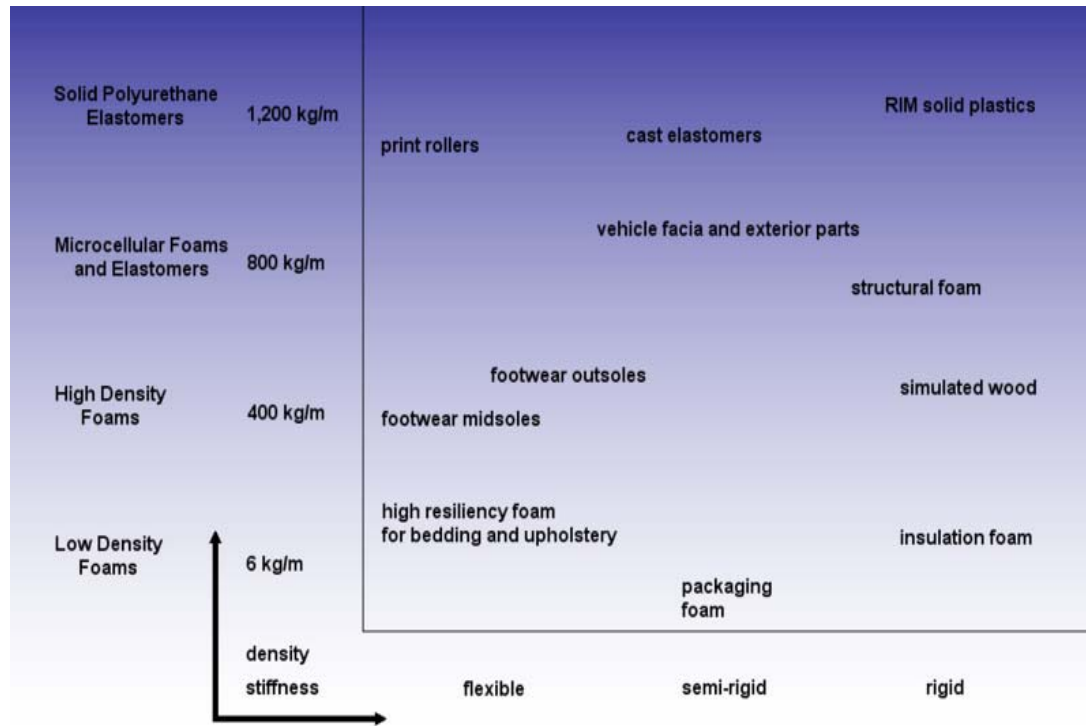


Figure 1.8: Application Areas of Polyurethane by Stiffness

1.3. Dynamic Mechanical Analysis

Dynamic Mechanical Analysis has become more popular because of their significant properties and to provide information about materials in particular polymers. The first experiments to measure elasticity of materials could be done by Poynting in 1909 [71]. In the 1950's, the Weissenberg Rheogoniometer and the Rheovibron instruments were invented for usage of commercially [72]. Viscoelastic properties of polymers published by Ferry in 1961 [73]. This book explains dynamic measurements and the best theory on viscoelastic measurements. "Torsional Braid Analyzer" was developed by J. Gilham and modern era of DMA began [74]. J. Starita, C. Macosko and Bohlin developed a commercial dynamic mechanical analyzer in the 1970's.

The first instruments have some disadvantages about using instrument and limited resulting properties. At the same time, characterization of material by dynamic mechanical analysis was reported by Murayama, Read and Brown [75,76]. After these developments on dynamic mechanical analysis, Polymer Labs, Du Pont and Perkin Elmer developed new instruments about dynamic mechanical analysis. With computer technology, dynamic mechanical analysis has become more effective and useful in the world of science.

There are several components which are critical to the design and resultant performance of a dynamic mechanical analyzer. These components are the drive motor (which supplies the sinusoidal deformation force to the sample material), the drive shaft support and guidance system (which transfers the force from the drive motor to the clamps which hold the sample), the displacement sensor (which measures the sample deformation [oscillation amplitude] that occurs under the applied force), the temperature control system (furnace), and the sample clamps.

DMA gives the information about rheological and thermal properties of polymers. Rheology is very sensitive to small changes of the material's polymer structure – thus ideal for characterization of polymers. The rheology structure relationship is the key to the development of new materials.

Dynamic Mechanical Analysis (DMA) measures the mechanical properties of materials as function of temperature, frequency and time and also it is a thermal analytical method by which the mechanical response of a sample subjected to a specific temperature program is investigated under periodic stress. Dynamic mechanical analyzer is a thermal analytical instrument used to test the mechanical properties of many different materials.

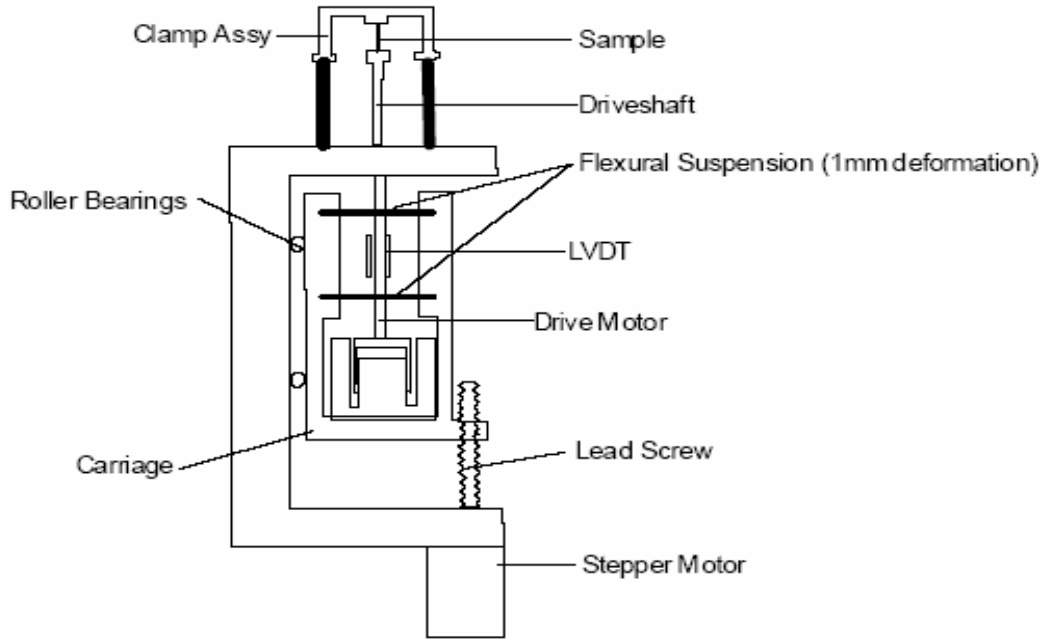


Figure 1.9: Traditional Dynamic Mechanical Analyzer Design

The Dynamic Mechanical Analysis is a high precision technique for measuring the viscoelastic properties of materials. Viscoelasticity is about elastic behaviors of material. Most real-world materials exhibit mechanical responses that are a mixture of viscous and elastic behavior.

1.4. Elasticity and Viscous Flow Behaviors

Materials are often referred to as solids or liquids, depending on whether or not they retain their shape under the force of gravity. An ideal solid is a material that is purely elastic. When adequate stress is applied to a purely elastic material, it undergoes a deformation, i.e., a change in shape instantaneously. When that stress is removed, the sample instantaneously regains its initial shape. The energy involved in the application of stress is stored within the material due to its elasticity, and in turn, it can use that energy to do work when the stress is released. It is important that there is no time dependence in the behavior of the material, in that the deformations occur the very instant the stress is changed (applied or removed in the example). This ideal mechanical behavior is described by Hooke's law in which stress and strain are related through a proportional constant called the modulus of rigidity or simply, modulus (E or G).

There is a constant ratio between stress and strain. This equation is also known as "Hooke's Law".

Hooke's Law:

$\sigma = E\varepsilon$ (Tension, Compression or Bending)

$\tau = G\gamma$ (Shear)

Where:

σ and τ are stress terms

ε and γ are strain terms

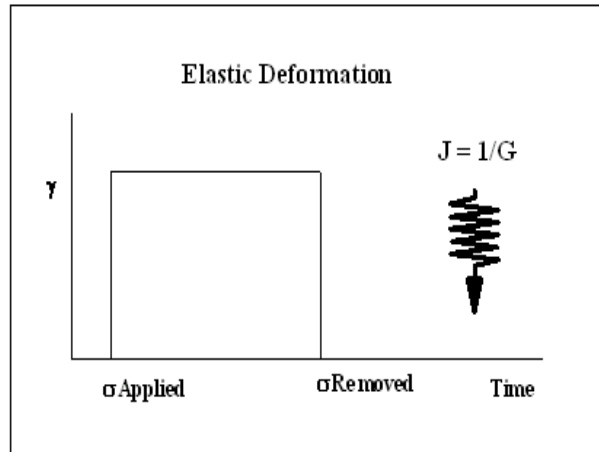


Figure 1.10: Hooke's Law and Elastic Deformation

An ideal liquid is a material that is absolutely devoid of any elasticity or rigidity. Thus, when placed in a container, an ideal liquid will conform to the shape of the container and find its own level under gravity. An ideal liquid is unable to store any energy imparted to it upon the application of a stress. Instead, the liquid undergoes continuous deformation until the stress is removed. Water is a good approximation to an ideal liquid. When placed in a beaker, it assumes the shape of the beaker and finds its own level in it. Next, consider an inclined plane, upon whose top end, one pours the water out of the beaker. Since gravity is acting as a stress on the water, there will be a continuous flow down the incline. If one could imagine that we are able to eliminate gravity, then the water would stop flowing farther down the incline and stay in that location. The absence of elasticity prevents it from flowing back up the incline to its initial location at the top. This kind of viscous behavior is addressed by Newton's Law of Viscosity.

Newton's Law:

$$\tau = \eta \frac{dy}{dt}$$

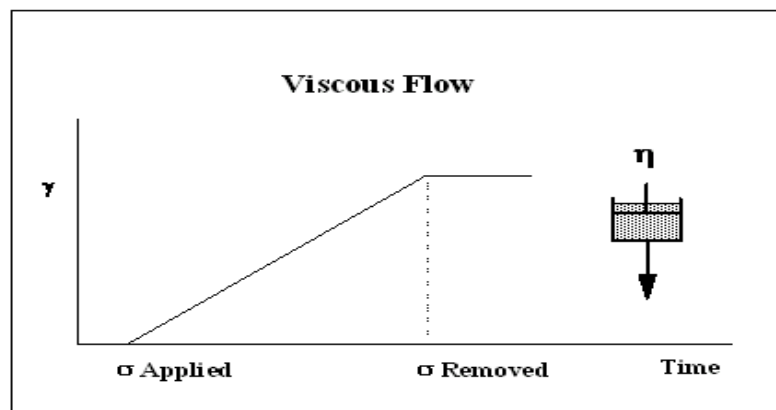


Figure 1.11: Newton's Law and graph of Viscous Flow Behaviour

A simple graphical representation of the behavior of an ideal fluid is shown in the Figure 1.11. An ideal fluid will deform continuously under the application of a stress but will not recover when the stress is removed. The strain developed under the application of the stress is a function of time until the stress is removed. The stress is independent of the strain but proportional to the rate of strain. The proportionality factor η is called the coefficient of viscosity. A Newtonian Fluid is one whose viscosity is independent of the applied shear rate.

Dynamic Mechanical Analyzer (DMA) deforms a sample mechanically and after that it measures the sample response. When a force is applied on a material it suffers a change in shape, that is, it deforms. The deformation can be applied sinusoidally, in a constant (or step) fashion, or under a fixed rate. The response to the deformation can be monitored as a function of temperature or time. A force to resist the deformation is also set up simultaneously within the material and it increases as the deformation continues. If the material is unable to put up full resistance to external action, the process of deformation continues until failure takes place. The deformation of a body under external action and resistance to deform are referred to by strain and stress respectively.

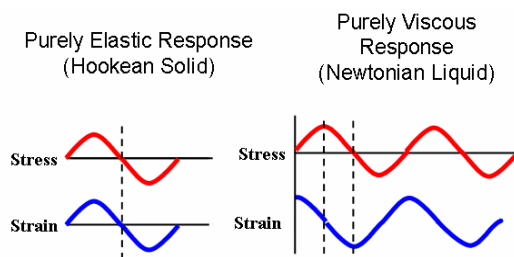


Figure 1.12: Purely elastic and viscous response functions

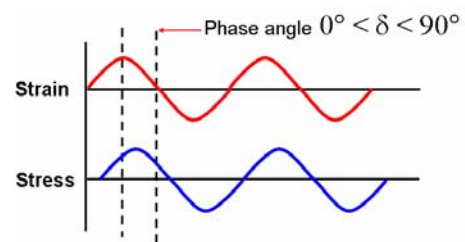


Figure 1.13: Function of viscoelastic response

DMA is a useful instrument to measure mechanical properties for materials. DMA results for solid polymers can be used to set the polymer morphology and structure for industrial end-use products. Polymers are viscoelastic fluids, which behave viscous or elastic, depending on how fast they flow or are deformed in the process. For instance, glass transition temperature and damping behavior can be used to determinate material's using conditions such as temperature, stiffness. In addition, DMA measurements explain how a material behaves at the moment and future. For some industrial products, it is more important to know how a material will behave weeks, months, and years. DMA is a non destructive technique. Small specimens can be used, so it is a decided advantage for evaluating experimental materials.

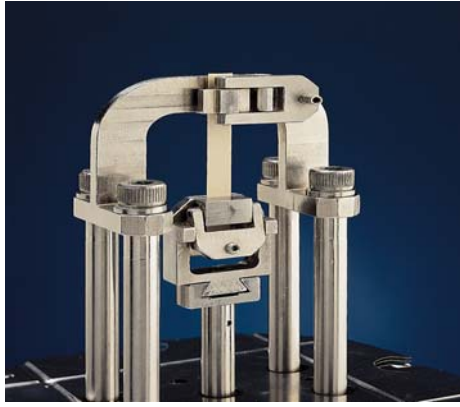


Figure 1.14: Tension Clamp of Dynamic Mechanical Analyzer

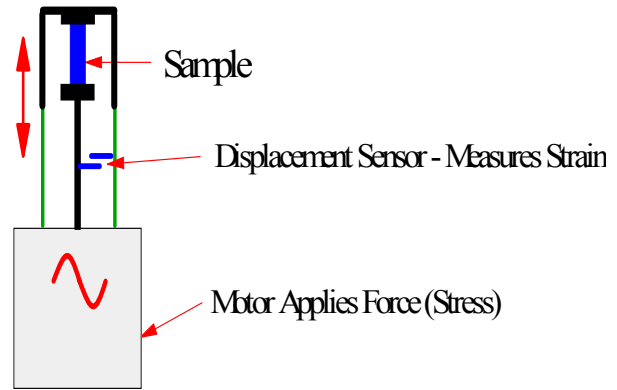


Figure 1.15: Schematic diagram for DMA measuring

Dynamic mechanical analysis can be applied to following materials: thermoplastic polymers, thermosetting polymers, elastomer, composites (including fillers, reinforcing fibers), wood, paper, glass ceramics, metals, alloys, food, cosmetics, etc.

Dynamic or oscillation experiments can be performed to characterize many properties of materials. The following is a partial list.

- Glass-transition temperature, T_g , and other secondary transitions - b, g, d
- Viscoelastic Response and Spectrum
- Linear Viscoelastic Region (Oscillatory Stress or Strain Sweep)
- Damping characteristics ($\tan \delta$)
- Stress fatigue
- Structure-related properties, especially with regard to:
 - ❑ Crosslinking, cure state
 - ❑ Crystallinity
 - ❑ Molecular Weight
 - ❑ Additives
 - ❑ Plasticizers
 - ❑ Blending
 - ❑ Aging
 - ❑ Orientation

Dynamic Mechanical Analyzer is useful for these tests: mechanical properties, morphology of polymers, loss factor (Tan delta), loss angle (delta), impact resistance, dynamic viscosity, curing kinetics, correlation with materials formulation, ageing, damping, glass transition temperature (T_g), industrial products stiffness, polymer compatibility, relationships mechanical properties/molecular structure, relaxation time, rheological properties, secondary transitions, specimen stiffness, stress relaxation test, thermal properties, viscoelastic properties, young modulus, thermal stability, prediction of long term mechanical behavior, optimization of curing process, dynamic viscosity, complex viscosity, modulus values, dynamic test, creep behavior, gel time, melting point, dimensional stability, impact resistance, secondary transitions, tension test, stress-strain.

1.5. Clamps of Dynamic Mechanical Analyzer

There are two classes of clamps for the DMA—tensioning and nontensioning. The 3-point bend, tension/film, tension/fiber, compression and penetration clamps are tensioning clamps, while the single/dual cantilever and shear sandwich clamps are nontensioning. There are seven types of clamps in DMA. Each of clamp is used with suitable materials. Single cantilever bending clamp, dual cantilever bending clamp, 3-point bending clamp, tension film clamp, tension fiber clamp, compression and shear clamp are used for DMA measurements. Single and dual cantilever clamps is useful for thermoplastics, elastomers, highly damped materials and evaluating the cure of supported materials, thermoplastics and elastomers above and below their glass transitions; uncured supported resins (supported with glass braid). It must be used for weak to moderately stiff samples. For example, thermosetting resins, elastomers below T_g , or lightly filled thermoplastic materials, polypropylene, polyvinylchloride, polycarbonate. 3-point bending is the best mode for measuring medium to high modulus materials and conforms with ASTM standard test method for bending also purest deformation mode since clamping effects are eliminated. Three-point bending is typically used with high-modulus materials such as thermoplastics, resins, epoxy and graphite laminates, metals, ceramics, prepreg laminates, PC boards, composites, and highly crystalline thermoplastics through T_g . Sample geometries include solid materials such as bar stock, test plaques, test coupons, sheet stock, wire, rods and tubes. Shear sandwich clamp is good for evaluating highly damped soft solids such as gels and adhesives & elastomers $> T_g$ and it can be used for high viscosity melts and resins. It is not suitable for viscous liquids to elastomers above glass transitions. It is available to use for soft solids and for materials above their glass transitions.

For example, cured and uncured rubber, polymer melts, gels, b-staged material. In addition, square sample configuration of shear sandwich clamp provides pure shear deformation. Compression clamp is a good mode for low to medium modulus materials (gels, weak elastomers). It can be used for soft solids, materials above the glass transition and highly viscous liquids can also be evaluated. For example, weak elastomers, foams, personal care products, toothpaste, hydrogels. Also it has some options for expansion and penetration measurements.

Tension film clamp is used for all kind of thin films. For example, thermoplastics, thermosets and elastomers in "sheet" form, fishing line. Tension Fiber can be used for Single and bundled fibers, thick fibers and tubular materials such as fishing line, sutures, monofilaments.

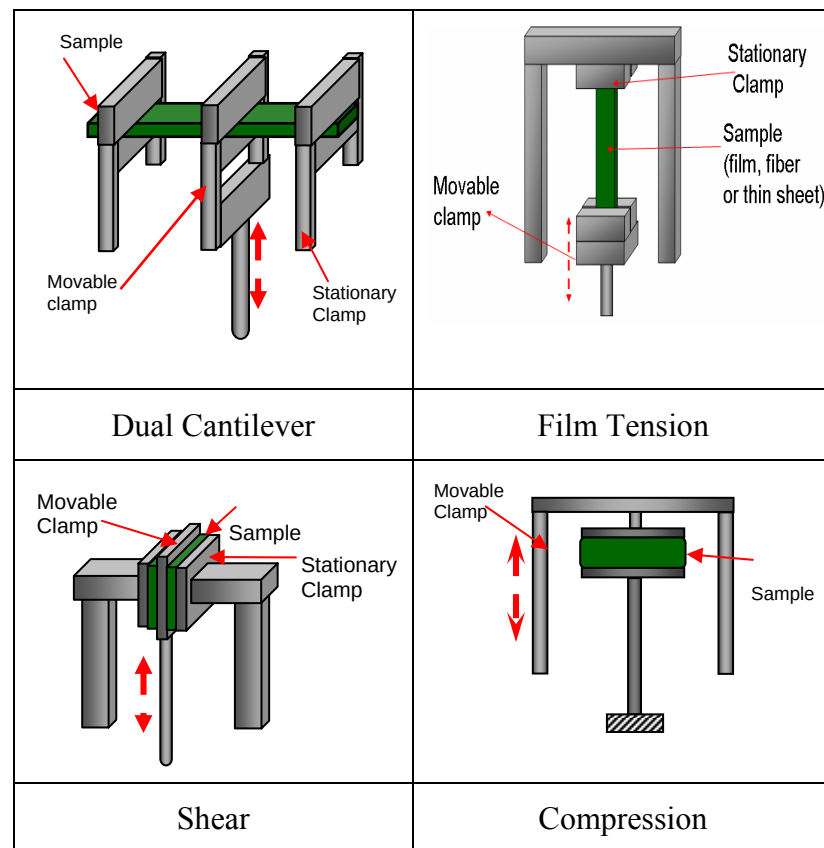


Figure 1.16: Clamps of Dynamic Mechanical Analyzer

Before all experiments, clamp calibration must be performed each time the clamp is changed. Clamp calibration has three steps (mass, zero, and compliance) depending on the clamp type installed. Firstly, clamp mass calibration is performed to allow the instrument to compensate for the mass of a specific clamp. Secondly, clamp zero calibration is needed to determine the point of zero sample length for automated sample length (tension) or thickness (compression and penetration) measurement.

Thirdly, compliance calibration is used to measure the flexibility of a clamp and calibrates the instrument to that flexibility.

Before clamp calibration, position calibration can perform. Position Calibration is used to calibrate the absolute position of the drive shaft by the optical encoder.

1.6. Film Tension Clamp Equations

Since a material's modulus is independent of its geometry, equations relating the sample stiffness to the modulus depend on the type of clamps used, the sample shape, and the mode of deformation. This part contains stiffness calculations for the different clamp types along with comments on appropriate correction factors. Also included are stress and strain equations, which can be used as a general guideline for calculations made from the force and amplitude of deformation. The equations for stress and strain assume linear viscoelastic behavior.

The fundamental measurement of the DMA is sample stiffness (K). Sample stiffness is defined as the force (in Newtons) applied to the sample divided by the deformation (in meters). For oscillation experiments, it is the force divided by the amplitude.

The stiffness of a material is dependent on its geometry (physical dimensions). The modulus of a material however is independent of its geometry. As an example, consider a piece of aluminum foil. It is very easy to bend the aluminum foil. If we take a bar of aluminum one-inch thick. This piece of aluminum is not easily bent.

Both the aluminum foil and bar are made of the same material but simply changing the physical dimensions of the material changes the amount of force required to deform the material. By measuring the modulus of both the foil and bar, we would get the same number. A good understanding of sample stiffness is important for understanding geometry selection when conducting DMA measurements.

The DMA determines the modulus (stiffness) of a material differently depending upon the clamp type installed on the instrument.

Modulus Equation

The stiffness model equation for a sample, analyzed on the film or fiber tension clamp, is as follows.

$$K = \frac{A \cdot E}{L} \quad (1.1)$$

K = Stiffness or spring constant
 E = Elastic modulus
 A = Sample cross-sectional area
 L = Sample length

Because the sample will have a very small area as compared to it's length, no end-effects correction is needed, the modulus equation is,

$$E = K_s \cdot \frac{L}{A} \quad (1.2)$$

E = Elastic modulus
 A = Sample cross-sectional area
 L = Sample length
 K_s = Measured stiffness

Stress and Strain Equation

$$\sigma_0 = \frac{P}{A_0}$$

$$\varepsilon_0 = \frac{\Delta L}{L_0} \quad (1.3)$$

σ₀ = Stress
 ε₀ = Strain
 P = Applied force
 ΔL = Cumulative change in sample length
 L₀ = Initial sample length
 A₀ = Initial sample cross-sectional area

1.7. DMA Modulus Parameters

The Modulus: Modulus is an intrinsic material property (does not change with material size or shape), defined as the ratio of stress/strain in a body under a particular mode of deformation (such as shear, bending, torsion, etc.). Thus modulus is a measure of materials overall resistance to deformation. The modulus is the ratio of a component of stress to a component of strain, in rheology.

$$G = \text{Stress/Strain} \quad (1.4)$$

The Elastic (Storage) Modulus: The storage modulus is measure of elasticity of material. It is also called “the ability of the material to store energy”. It is equivalent to the ability of a sample to store energy, i.e. its elasticity.

Energy storage occurs as molecules are distorted from their equilibrium position by application of a stress. Removal of the stress results in a return to equilibrium position of the molecular segments.

$$G' = (\text{stress/strain})\cos\delta \quad (1.5)$$

The Viscous (loss) Modulus: Loss modulus represents the capability of a material to dissipate energy (mechanical, acoustic) as heat, owing to viscous motions inside the material itself. It is limited to the molecular motion within the sample that dissipates energy as heat. In rheology, loss modulus is the imaginary part of the complex modulus.

$$G'' = (\text{stress/strain})\sin\delta \quad (1.6)$$

Tan Delta: It is measure of material damping - such as vibration or sound damping. Damping refers to damping the loss of mechanical energy as the amplitude of motion gradually decreases. It means also the ability of a material to dissipate mechanical energy by converting it into heat. Tan Delta is a useful index of material viscoelasticity since it is a ratio of viscous and elastic moduli. $\tan\delta$ is an important indication of viscoelasticity of materials, it is independent from the shape and dimension of samples and it is adimensional.

$$\tan \delta = \text{Loss Modulus/Storage Modulus} = G''/G' \quad (1.7)$$

Complex viscosity: Viscosity is the property of a material to resist deformation increasingly with increasing rate of deformation. Mathematically viscosity is defined as the shear stress divided by the rate of shear in simple shear flow. Complex viscosity is defined as mathematically the sum of a real part and an imaginary part. The real part is usually called the dynamic viscosity and the imaginary part is related to the real part of the complex shear modulus.

Complex viscosity has a dependence on the frequency in the denominator so that at 1 Hz, complex viscosity will overlap the complex modulus. Also note that converting E into G or the reverse requires the use of Poisson’s ratio, ν .

Out of these remaining properties, the most commonly used is the complex viscosity, η^* . The complex viscosity is given by:

$$\eta^* = G^* / \omega = E^* / 2(1 + \nu) \quad (1.8)$$

where G^* is the complex shear modulus, ω is the frequency. Like other complex properties, it can be divided into an in-phase and out-of-phase component:

$$\eta^* = \eta' - i\eta'' \quad (1.9)$$

where η' is a measure of energy loss and η'' is a measure of stored energy.

1.1 1.8. The Stress-Strain Curve

Stress expresses an applied force or system of forces that tends to strain or deform a body, strain means that a deformation produced by stress. Strain is defined the deformation from a specified reference state, measured as the ratio of the deformation to the total value of the dimension in which the change occurs. In another way, it defines the measurement of deformation, relative to a reference configuration of length, area, or volume. Strain is also called relative deformation.

The stress-strain curve characterizes the behavior of the material tested. It is most often plotted using engineering stress and strain measures, because the reference length and cross-sectional area are easily measured. Stress-strain curves generated from tensile test results help gain insight into the constitutive relationship between stress and strain for a particular material.

It can be obtained quantitative information that can be used for the constitutive relationship by stress-strain curves, in addition, the stress-strain curve helps to describe and classify the materials by qualitative. Typical regions that can be observed in a stress-strain curve are:

- a. Elastic region
- b. Yielding
- c. Strain Hardening
- d. Necking and Failure

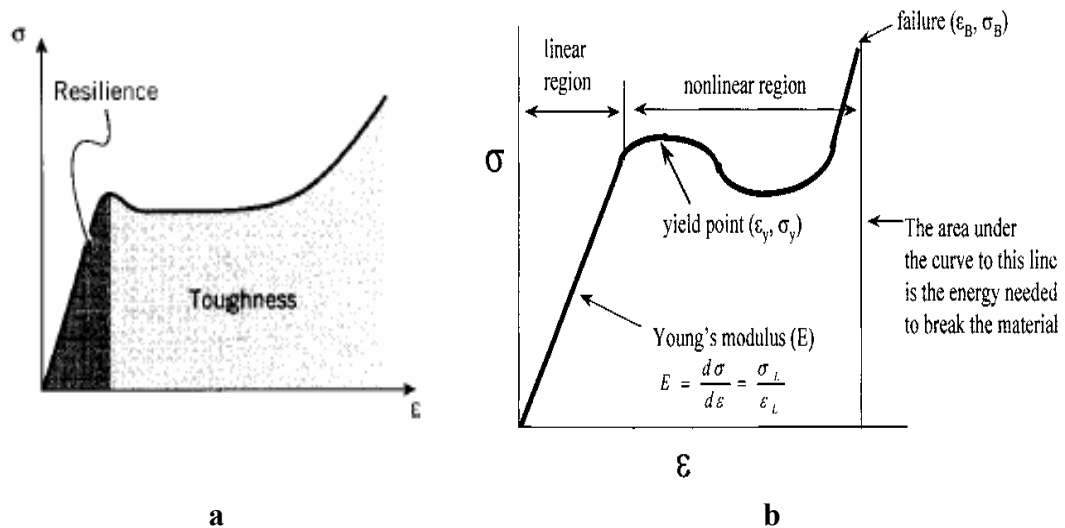


Figure 1.17: a) Resilience and toughness of modulus of stress strain curves
b) A typical stress strain curve

The modulus of resilience is then the quantity of energy the material can absorb without suffering damage. The integral of stress-strain curve gives us the energy spent to break the specimen (Figure 1.16). Toughness is really a measure of the energy a sample can absorb before it breaks. Similarly, the modulus of toughness is the energy needed to completely fracture the material. If a material higher the area of under the stress-strain diagram has a higher toughness (higher energy which is necessary to break specimen).

In Figure 1.17, up to yield point there is a homogenous deformation. After the yield point a necking can be seen. The ratio of necking increases and covers all part of specimen. In order to increase length of specimen we should increase stress to continue elongation.

Many materials show a linear relation for the initial part of the stress strain curves. The slope of this initial part's name is modulus of elasticity (Young modulus).

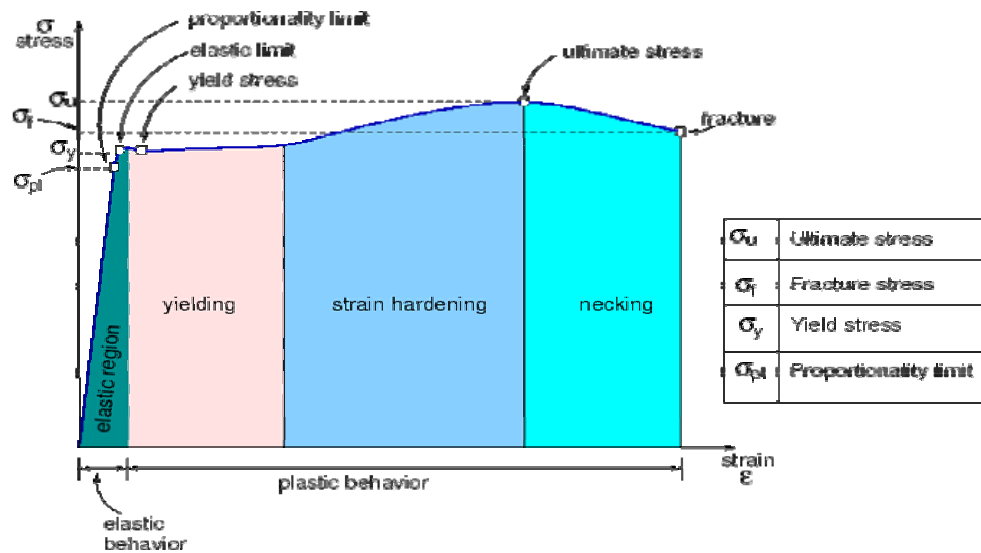


Figure 1.18: Various regions and points of stress strain curve.

The maximum stress level on the stress-strain curve corresponds to the strength of material while the maximum strain is defined as ultimate strain. If the slope is steep, the sample has a high tensile modulus and it resists deformation. If the slope is gentle, the sample has a low tensile modulus and it is easily deformed.

The behavior of materials can be broadly classified into two categories; brittle (steel, aluminum etc.) and ductile (glass, cast iron etc.). The two behaviors of materials can be distinguished by the stress-strain curves, shown in Figure 1.19. Ductile and brittle materials have qualitative and quantitative differences respect to their stress-strain response. For brittle materials, all deformation up to breaking is elastic character. They show no plastic deformation. Ductile materials show much higher toughness than brittle materials. Also they absorb energy much more than brittle. Ductile materials withstand bigger strains in contrast to brittle materials. Young's moduli and ultimate stress of brittle material has relatively higher than the ductile materials.

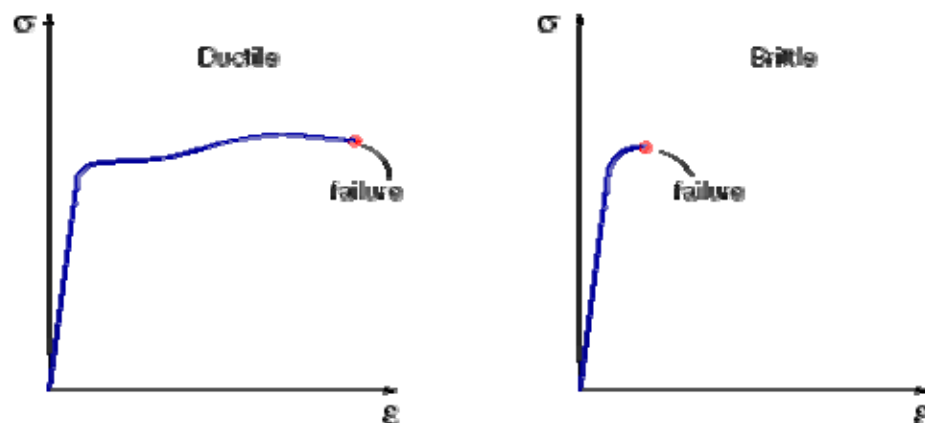


Figure 1.19: Behavior of ductile and brittle material

2. EXPERIMENTAL

2.1. Materials

There are mainly three different materials used in this research, epoxy, polyurethane and carbon fiber. The polymer matrix was epoxy and polyurethane. The reinforcing material was carbon fiber. Firstly, the polyurethane was taken from Flokser Co. (Istanbul, Turkey) with a weight average molecular weight of 30.000.

The solid content of PU was 35% wt in dimethyl formamide (DMF). It was the ester product of polyester polyol and diphenylmethane diisocyanate (MDI). The chain extender was 1,4 butanediol.

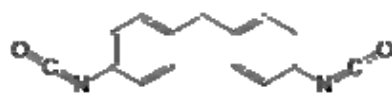


Figure 2.1: The Chemical Structure of MDI

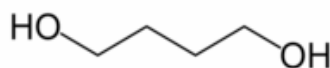


Figure 2.2: The Chemical Structure of 1,4-Butanediol

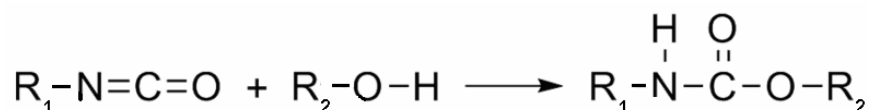


Figure 2.3: Synthesis Reaction of Polyurethane

Secondly, epoxy resin and hardener were used to prepare composites. Epoxy resin and hardener was taken from Tekno Construction Chemicals Co.(Istanbul, Turkey). Its commercial names are Teknobond 300 A and Teknobond 300 B. Epoxy resin is a product of bisphenol A-(epichlorhydrin)- Concentration %: 76.00 - 88.00 and 1,6-hexanedioldiglycidyl ether-Concentration %: 14.00 - 22.00.

It is obtained that epoxy resin's number average molecular weight has lower from 700. Hardener is a product of aliphatic and cycloaliphatic polyamines.

It is obtained from benzyl alcohol- Concentration %: 40.00 - 52.00, isophorone diamine-Concentration %: 30.00 - 42.00, trimethylhexamethylenediamine-Concentration %: 4.00 - 10.00 and phenol-Concentration %: 2.00-3.00.

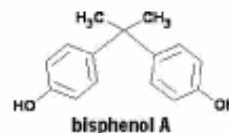
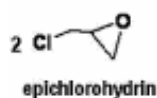


Figure 2.4: Structure of Epichlorohydrin **Figure 2.5:** Structure of Bisphenol A

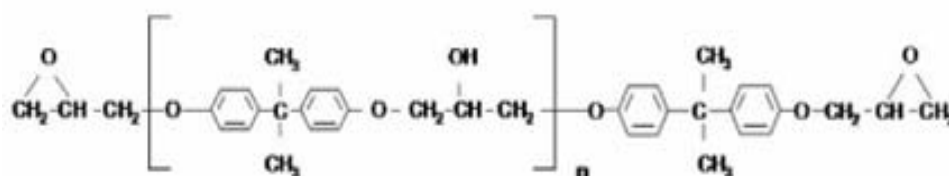


Figure 2.6: The Epoxy Resin from Bisphenol A and Epichlorohydrin

Carbon fibers were used to prepare composite materials from epoxy and polyurethane. Two types of carbon fibers (Torayca-T and Metyx-M) were used while preparing the composites. Carbon fibers were polyacrylonitrile based carbon fiber. The first carbon fiber (TORAYCA T700 12K, Warp = 12.000, Warp = 7 micron, tex (g/1000m) = 880) and the second one (Metyx Co., Tensile modulus=230 GPa, Elongation 2.1%, mass per unit 800 g/1000m) were used for mechanical analysis. The amount of Torayca CF used in these in one bundle is more than Metyx branded one and also investigated as shown in later sections.

2.2. Material Preparation

2.2.1. Preparation of carbon fiber reinforced epoxy composites by bundles

In a 50 ml flask, 10 g epoxy A and 3 g B components (hardener) were mixed for 20 minutes. After homogenous mixtures were obtained, the resins casted on the carbon fiber bundle(s) which were horizontally lay on the glass substrate as 1, 2, 3 bundles. After that, the prepared composite resins were cured in previously 100 °C heated oven for one hour.

2.2.2. Preparation of carbon fiber reinforced epoxy composites by using short fibers

Epoxy A and B components (hardener) were mixed for 10 minutes in a 50 ml flask. Torayca branded carbon fibers were cut into small species with a simple scissor and added to homogenous mixtures as 0.01 – 0.03 - 0.05 – 0.1 – 0.3 - 0.5% as weight. Then the continuous mixing was employed 20 minutes. After that, CF containing resins casted on the glass substrate and the prepared composite resins were cured in 100 °C heated oven for one hour.

Table 2.1: Carbon Fiber Reinforced Epoxy Composites with respect to Epoxy and Hardener Content

Sample Code	Epoxy A (w%)	Epoxy B (w%)	Carbon Fiber (w%)
Epoxy/Hard:(10/3) 1	76	23	0.01
Epoxy/Hard:(10/3) 2	76	23	0.03
Epoxy/Hard:(10/3) 3	76	23	0.05
Epoxy/Hard:(10/3) 4	76	23	0.10
Epoxy/Hard:(10/3) 5	76	23	0.30
Epoxy/Hard:(10/3) 6	76	23	0.50
Epoxy/Hard:(10/5) 1	66	33	0.01
Epoxy/Hard:(10/5) 2	66	33	0.03
Epoxy/Hard:(10/5) 3	66	33	0.05
Epoxy/Hard:(10/5) 4	66	33	0.10
Epoxy/Hard:(10/5) 5	66	33	0.30
Epoxy/Hard:(10/5) 6	66	33	0.50
Epoxy/Hard:(10/7) 1	58	41	0.01
Epoxy/Hard:(10/7) 2	58	41	0.03
Epoxy/Hard:(10/7) 3	58	41	0.05
Epoxy/Hard:(10/7) 4	58	41	0.10
Epoxy/Hard:(10/7) 5	58	41	0.30
Epoxy/Hard:(10/7) 6	58	41	0.50

2.2.3. Preparation of carbon fiber reinforced polyurethane composites

Polyurethane-DMF solution and bundle(s) of carbon fiber were used to prepare reinforced composites. The polyurethane casted on the carbon fiber bundle(s) which were horizontally lie on the glass substrate as 1, 2, 3 bundles respectively. After that, the prepared composites (polyurethane-carbon fiber) were left at room temperature over night to avoid bubbles.

Then the composites were taken and cut as compatible small specimens for DMA instrument. Two types of carbon fibers (Torayca and Metyx) were used while preparing the composites.

2.2.4. Preparation of Carbon Fiber Reinforced Epoxy-Polyurethane Polymer Systems

Polyurethane, epoxy A and epoxy B components (hardener) were taken in variants of amount and this mixture mixed for 30 minutes in a 50 ml flask. Then, Torayca branded carbon fibers were cut into small species and added to homogenous mixtures carefully, and the continuous mixing was employed half an hour. After that, the resins with carbon fibers casted on the glass substrate and the prepared composite resins were cured in 100 °C heated oven for half an hour.

Table 2.2: Different compositions of carbon fiber reinforced polyurethane-epoxy polymer system.

Sample Code	Polyurethane / g	Epoxy A / g	Epoxy B / g	Carbon Fiber / g
EPU_1	5	5	2	0.015
EPU_2	5	5	3	0.015
EPU_3	5	5	4	0.015
EPU_4	5	5	4	0.030
EPU_5	5	5	4	0.050
EPU_6	1	5	4	0.015
EPU_7	10	5	4	0.015

2.3. Characterization

2.3.1. Dynamic Mechanical Analyzer (DMA)

TA Q800 was used to measure modulus values of the epoxy-carbon fiber and polyurethane-carbon fiber composites and also polyurethane-epoxy-carbon fiber polymer system. The DMA T_g was found by examining the related modulus or tan delta curves. The material was heated from +30 °C to +100 °C with a heating rate 5 °C/min for polyurethane-carbon fiber and also +30 °C to +130 °C with a heating rate 5 °C/min for epoxy-carbon fiber composites. The applied frequency was 1 Hz. Materials were tested using tensile film clamp. The relationship with temperature and modulus also stress strain curves at different temperatures were also studied.

2.3.2. Scanning Electron Microscopy (SEM)

Morphology of carbon fiber reinforced of epoxy and polyurethane composites was investigated via a high resolution Supra Gemini 35VP Field Emission Scanning Electron Microscope from Leo. Before analysis, all samples were coated with carbon by Emitech, T950x Turbo Evaporate in order to have conductive samples to measure under SEM and avoid charging. Imaging was generally operated at 2 keV accelerating voltage, using the secondary electron imaging technique.

3. RESULTS AND DISCUSSION

3.1. Characterization of Bundle(s) Carbon Fiber Reinforced Epoxy Composites

Epoxy composites were prepared with bundle(s) of carbon fiber. Composites were investigated for dynamic mechanic analysis which values are storage modulus, loss modulus, tan delta and complex viscosity. Their modulus differences were studied and compared. The experiments were done for two types of carbon fibers, i.e., Toray and Metyx.

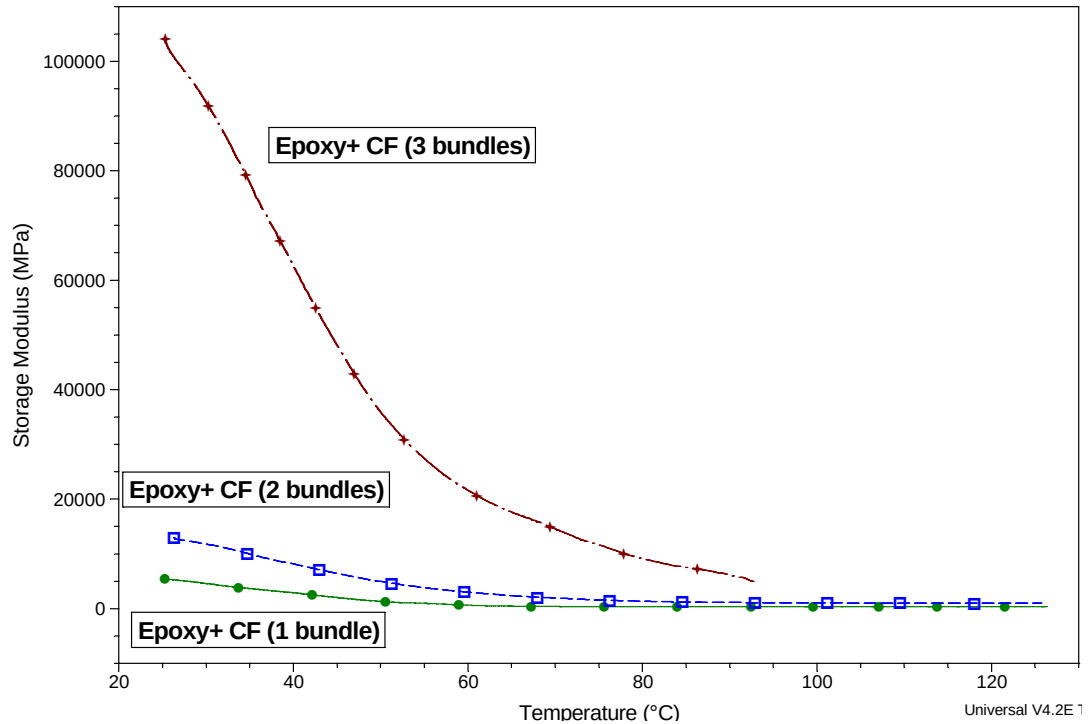


Figure 3.1: Variation of storage modulus of composites, [Epoxy/Hard:(10/3)] with amount of carbon fiber(T) (1 bundle, 2 bundles, 3 bundles)

Differences of storage and loss modulus between one bundle carbon fiber to three bundles carbon fiber can be seen in Figure 3.1 and 3.2. Composite with three carbon fiber bundles has the highest storage and loss modulus values.

The loss modulus peak of specimen with three bundles was 33.21 °C and the storage modulus value of sample with three bundles carbon fiber has the highest modulus.

The material was heated from +25 °C to +130 °C with a heating rate of 5 °C/min and the applied frequency was 1 Hz for storage, loss modulus and complex viscosity measurements.

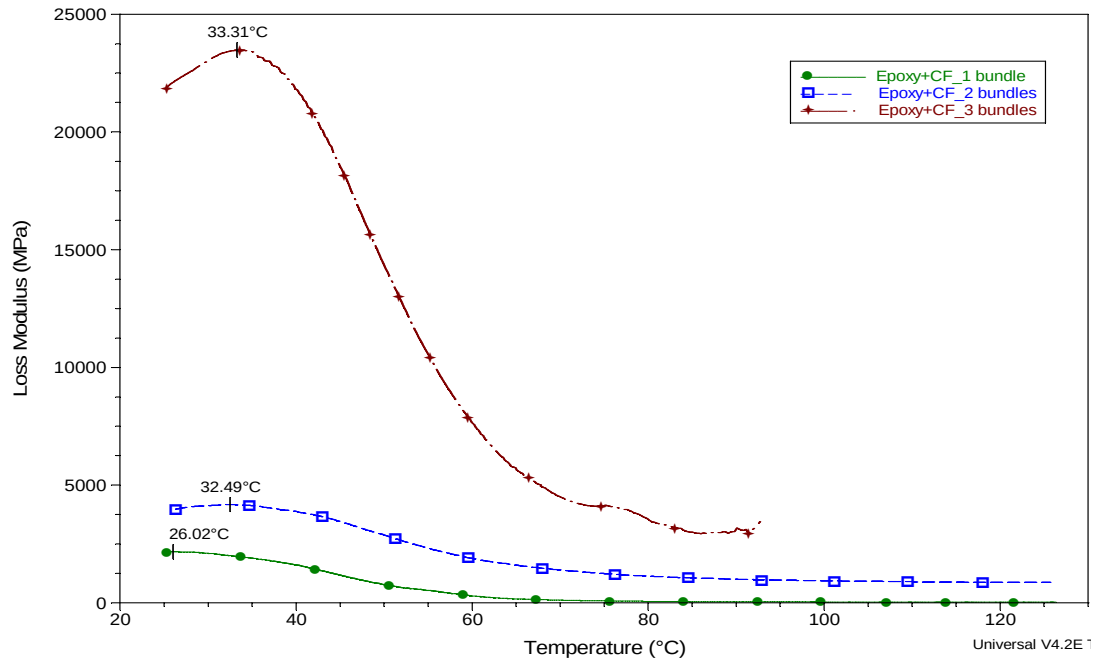


Figure 3.2: Variation of loss modulus of composites, [Epoxy/Hard:(10/3)] with amount of carbon fiber(T) (1 bundle, 2 bundles, 3 bundles)

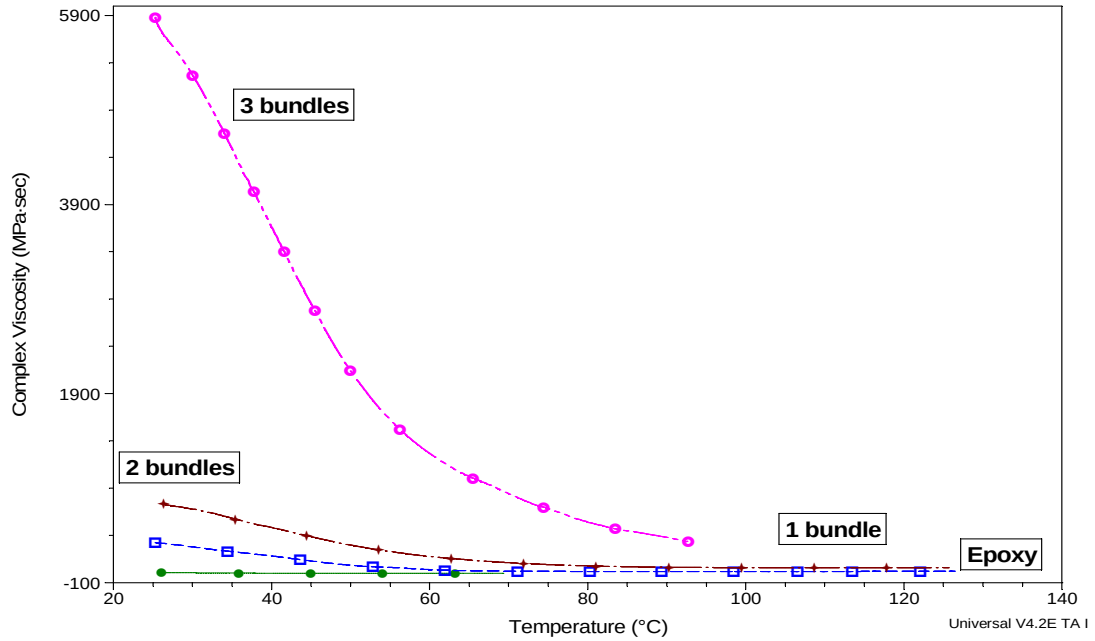


Figure 3.3: Plots of complex viscosity of composites, [Epoxy/Hard:(10/3)] with amount of carbon fiber(T) (1 bundle, 2 bundles, 3 bundles)

Complex viscosity values show similar properties with storage modulus values. Composite with three carbon fiber bundle has the highest complex viscosity value, 5882 Mpa.sec (Figure 3.3).

The data in Figure 3.3 demonstrate how the complex viscosity varies as a function of temperature. There were big shifts between samples. This large shift in complex viscosity, associated with change of carbon fiber content in the sample.

Table 3.1: Change of complex viscosity values for composites with different amounts of carbon fiber (T) at 25 °C [Reference: Pure Epoxy]

	Complex viscosity / MPa.sec
Epoxy	7.081
1 bundle of T	321.0
2 bundles of T	725.4
3 bundles of T	5882

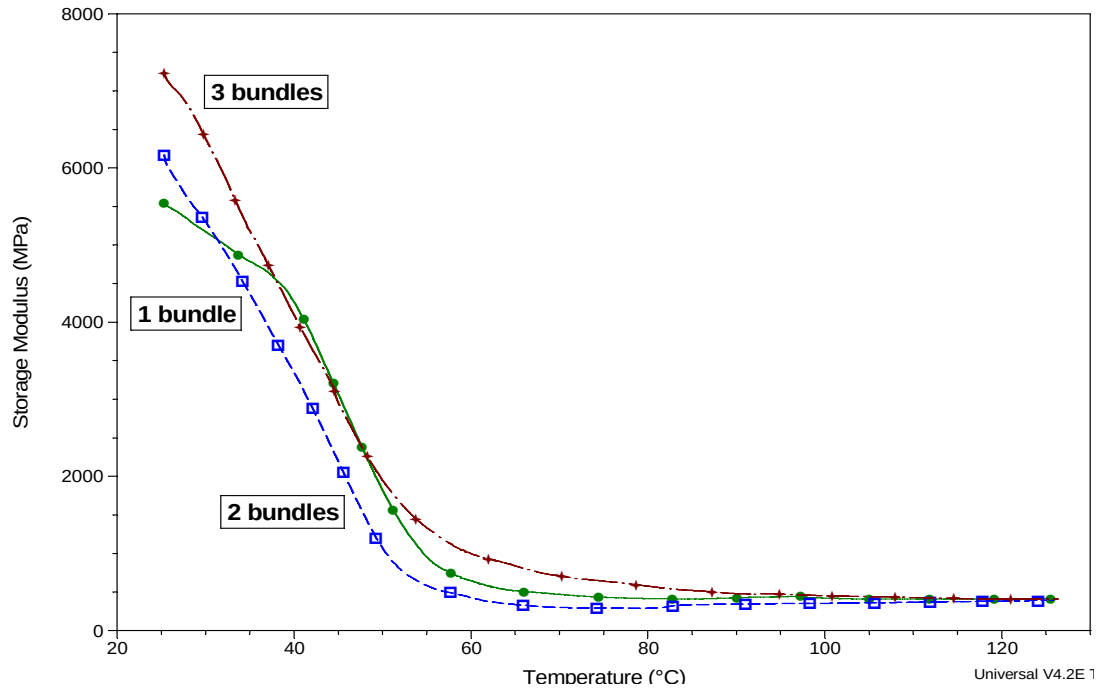


Figure 3.4: Temperature spectrum of storage modulus of composites, [Epoxy/Hard:(10/3)] with amount of carbon fiber(M) (1 bundle, 2 bundles, 3 bundles)

Figure 3.4 corresponds to loss modulus, storage modulus and complex viscosity values of the composites which are prepared by using Metyx branded carbon fibers instead of Toray carbon fiber.

Increase in the amount of carbon fibers as bundle for Metyx branded results an increase in storage modulus, comparison of these values with the corresponding Toray Carbon fibers results higher values (Table 3.1 and 3.2).

Table 3.2: Effects of carbon fiber contents on storage modulus of composites, [Epoxy/Hard:(10/3)] with amount of carbon fiber(M) (1 bundle, 2 bundles, 3 bundles) at 25 °C.

Storage Modulus / MPa	
Epoxy	101.4
1 bundle of M	5503
2 bundles of M	6118
3 bundles of M	7234

Table 3.2 shows the variation of modulus with carbon fiber content. It is observed that modulus of the composites increases continuously with increasing carbon fiber content.

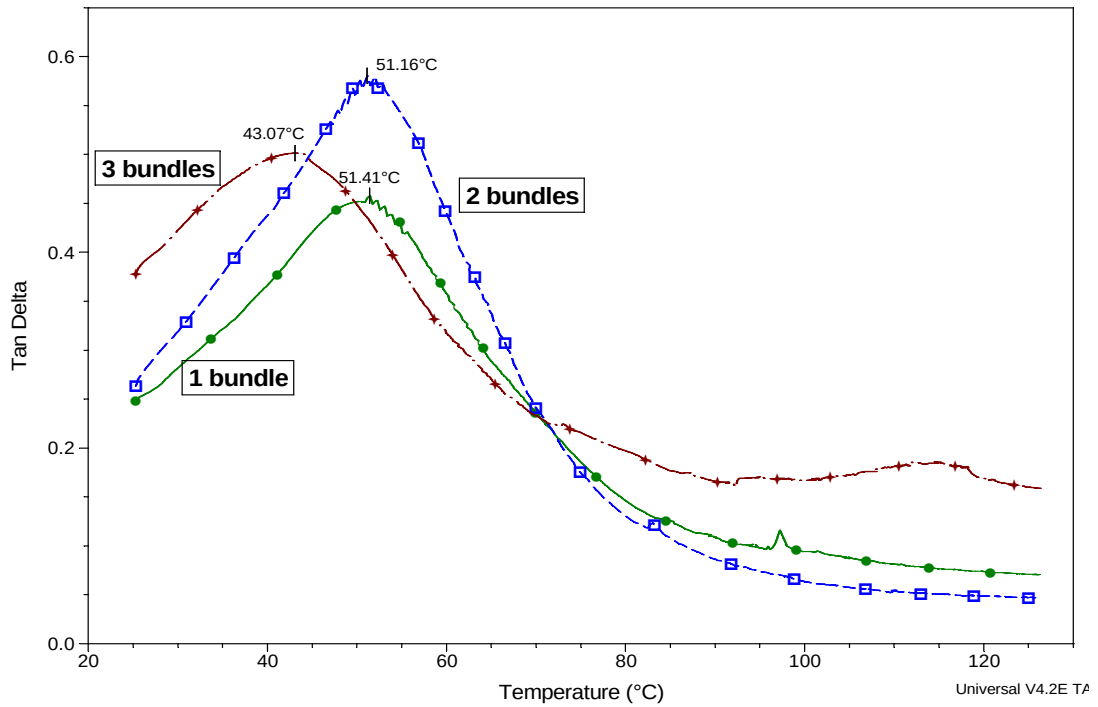


Figure 3.5: Plot of tan delta values with temperature at frequency 1 Hz for [Epoxy/Hard:(10/3)] with amount of carbon fiber(M) (1 bundle, 2 bundles, 3 bundles)

Carbon fiber reinforced epoxy composites were prepared to investigate if the bundle of carbon fiber (M) would improve the thermal properties and also to see the possible interaction between matrix and carbon fiber.

In Figure 3.5, the tan delta peaks were shown for all composites which have different carbon fiber content, have been shifted to higher temperatures compare to tan delta of pure epoxy peak, at 37 °C. Pure epoxy film was broken during the measurement at 70 °C.

Table 3.3: Complex viscosity and loss modulus obtained from DMA of composites, [(Epoxy/hard):(10/3))] with different amount of carbon fiber (Metyx) (1 bundle, 2 bundles, 3 bundles) at 25 °C.

	Complex Viscosity / MPa.sn	% Change	Loss Modulus / MPa	% Change
Epoxy	7.081	-	36	-
1 bundle	<u>315.3</u>	-	<u>1378</u>	-
2 bundles	352.5	12	1624	25
3 bundles	427.4	35	2734	100

Damping can be determined by the ratio between the loss energy and the storage energy (E''/E'), which is also named as tan delta. This ratio depends on the fiber and matrix adhesion. It can be seen that the peak of tan delta values are almost similar for composites that have one and two bundles of carbon fiber (M).

Also there was a similar increase on complex viscosity and loss modulus values. Pure epoxy has a 7.081 MPa.sec complex viscosity value at 25 °C, in addition composite which has three bundles of carbon fiber has 427.4 MPa.sec (Table 3.3). Similarly, loss modulus value of pure epoxy was 36 MPa, but three bundles of carbon fiber one was 2734 MPa.

Table 3.4: Comparison of storage modulus of epoxy composites prepared with Metyx and Toray at 25 °C.

Storage Modulus / MPa	1 bundle	2 bundles	3 bundles
Metyx CF	5503	6118	7234
Toray CF	5450	12700	104161

Storage modulus values are summarized in Table 3.4, in respect to increment of carbon fibers in each composite. However, the difference between the storage modulus values increases slightly for Metyx while Torayca increases extremely. In addition, there is a slight difference in one bundle CF added composites between Torayca and Metyx.

In contrast to storage modulus values, tan delta values arise with inverse proportionality.

Table 3.5: Comparison of tan delta values of epoxy composites prepared with Metyx and Toray at 25 °C

Tan Delta	1 bundle	2 bundles	3 bundles
Metyx CF	51.41	51.16	43.07
Torayca CF	26.02	32.49	33.31

3.2 Characterization of Chopped Carbon Fiber Reinforced Epoxy Composites

3.2.1. Dynamic Mechanical Analysis of Reinforced Epoxy/Hard:(10/3) Composites which include 0.01%, 0.03% and 0.05% Chopped Carbon Fiber by Weight

Three of samples which include chopped carbon fiber of 0.01%, 0.03% and 0.05% by weight, were prepared and the mix ratio of epoxy and hardener was 10:3.

In this study, DMA were performed on reinforced epoxy with carbon fiber to demonstrate the effect of carbon fiber on thermal and mechanical properties of the matrix.

The material was heated from +30 °C to +130 °C with a heating rate of 5 °C/min and the applied frequency was 1 Hz for dynamic mechanical analysis.

DMA results of composites such as storage modulus, loss modulus, and tan delta were taken after five days of preparation samples.

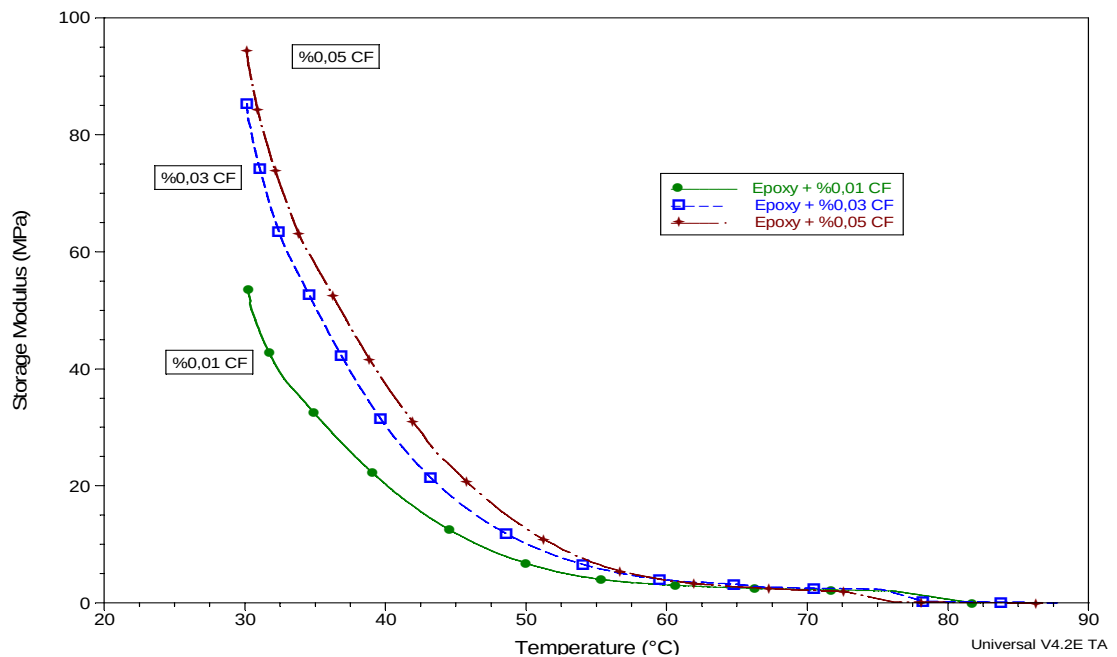


Figure 3.6: Storage modulus versus temperature of [Epoxy/Hard:(10/3)] with different amount of carbon fiber(T) (0.01% – 0.03% – 0.05%) after 5 days.

Figure 3.6 illustrates the DMA plots of storage modulus versus temperature as a function of carbon fiber loading. It can be seen that the storage modulus steadily increased with an increasing fiber weight percent.

The addition of 0.03% wt. of carbon fiber yielded a 75% increase of the storage modulus at 30 °C. The results of loss modulus and complex modulus were displayed similar values to storage modulus, after five days. The loss modulus of three samples were from 0.01 wt% to 0.05 wt.%; 45.9 MPa, 66.1 MPa, 68.9 Mpa at 30 °C.

Plot of complex viscosity and loss modulus has sharply decreasing from 30 °C to 60 °C, after this point plot has a slightly decreasing. These results indicate that loss modulus and complex viscosity were improved with addition of carbon fiber content up to 0.05% wt., after five days. Plot of tan delta 0.05% wt. has the highest peak value, 1.06 at 54.73 °C.

Table 3.6: Loss modulus, complex viscosity and tan delta values of carbon fiber Reinforced Epoxy Composites at 50 °C after five days.

	0.01%	0.03%	0.05%
	Carbon fiber	Carbon fiber	Carbon fiber
Loss modulus / MPa	12.05	8.371	6.000
Complex viscosity / MPa	6.376	5.897	3.901
Tan delta	0.901	0.880	0.951

3.2.2. Stress-Strain Curves of Reinforced Epoxy/Hard:(10/3) Composites which include 0.01%, 0.03% and 0.05% Chopped Carbon Fiber by Weight

Stress-strain curves were obtained for the composites which have 0.01%, 0.03% and 0.05% chopped carbon fiber by weight. The ambient temperature at 30 °C and 18 N maximum loads was applied with a ramp force rate of 1 N/min, after 5 days. Their stress-strain curves at different temperatures, 30 °C, 50 °C and 75 °C, and mechanical properties such as maximum stress, maximum strain, and young modulus at different temperatures were investigated.

Table 3.7: Young modulus values of carbon fiber reinforced epoxy composites at 30, 50 and 75 °C after five and fifth days.

Young Modulus / MPa	0.01%	0.03%	0.05%
After 5 days	Carbon Fiber	Carbon Fiber	Carbon Fiber
30 °C	3.357	4.272	4.551
50 °C	1.960	2.417	1.894
75 °C	2.387	2.812	1.742
Young Modulus / MPa			
After 50 days			
30 °C	-	-	-
50 °C	269.5	353.6	334.4
75 °C	9.950	9.276	9.444

After 50 days, there was a big increase on young modulus at 50 °C. For all the specimens, there was a significant improvement on young modulus values at 50 °C. Beside this, there was an important increase on young modulus of samples at 75 °C after fifth days, for instance; from 2.387 MPa at 75 °C to 9.950 MPa at 75 °C. This could be related to the formation of the crosslinking of epoxy which was formed by time.

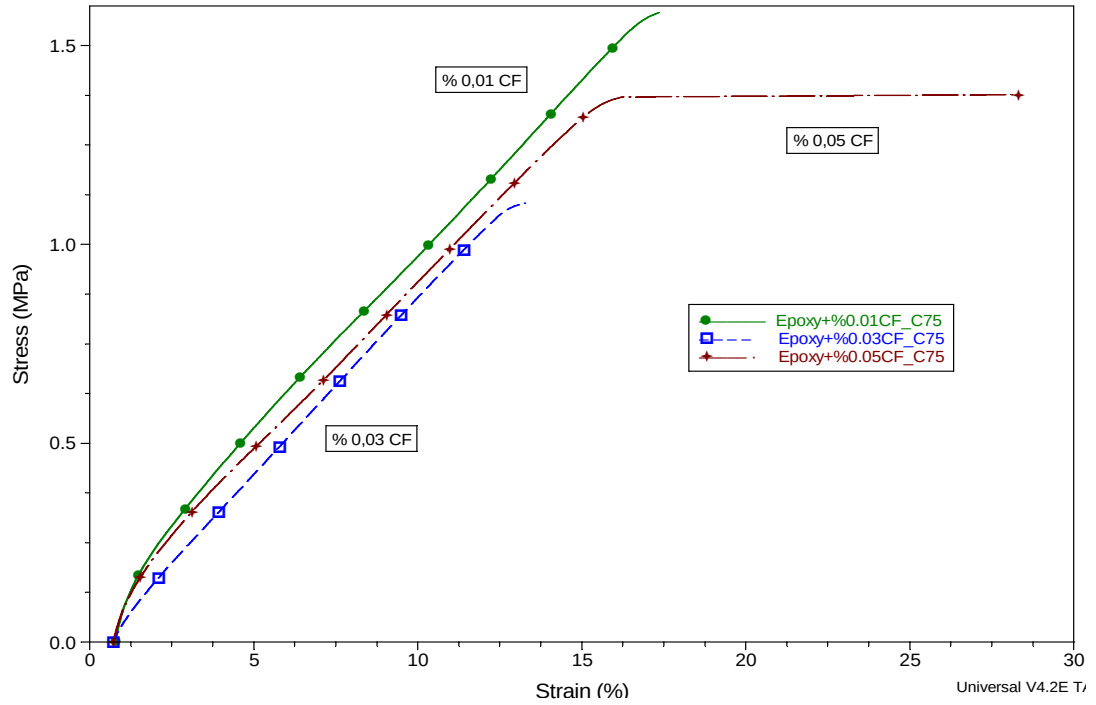


Figure 3.7. Stress–strain curves of composites: [Epoxy/Hard:(10/3)] with different amount of carbon fiber(T) (0.01 – 0.03 – 0.05%) at 75 °C after 50 days.

Table 3.8: Stress and strain values at break of Carbon fiber Reinforced Epoxy Composites at 30, 50 and 75 °C after five and fifth days.

	Stress at break / MPa	Strain at break / %
wt 0.01% Carbon fiber	1.583	17.33
wt 0.03% Carbon fiber	1.104	13.26
wt 0.05% Carbon fiber	1.377	28.30

From Table 3.8 and Figure 3.7, wt. 0.05% carbon fiber reinforced epoxy composite has maximum strain break at 75 °C, 28.30%. Maximum stress break value of composite was wt. 0.01% carbon fiber reinforced epoxy.

Composites were not displayed well proportional stress strain curves with their amount of carbon fiber content. But it was obvious that there were significant improvements on structure of composites after fifth days, and their mechanical properties were improved by time.

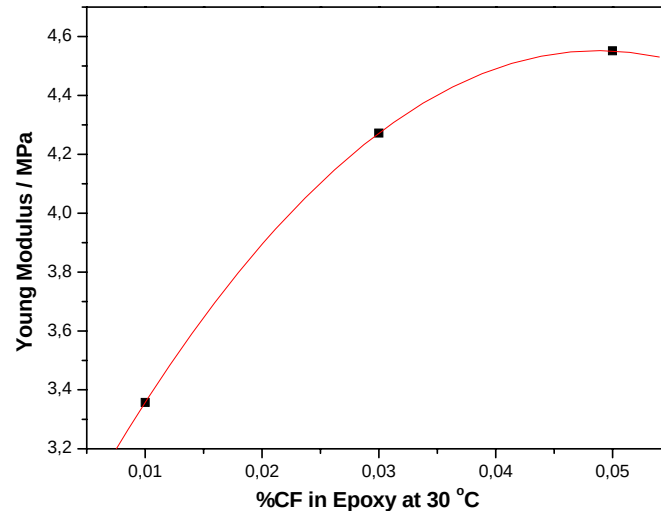


Figure 3.8: Increment of young modulus of epoxy with carbon fiber (T) composites (0.01-0.03-0.05% CF) at 30 °C after 5 days.

Stress strain curves of epoxy composites with variation of carbon fiber also indicate strengthen mechanical behavior as the amount of CF increased. The slope of stress strain curves are shown in Figure 3.8 as young modulus which tends to increase.

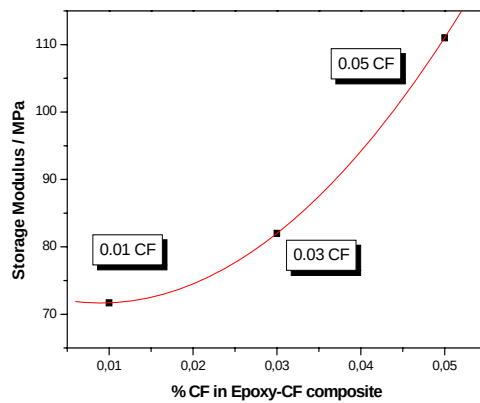


Figure 3.9.: Increment of young modulus of epoxy with carbon fiber (T) composites (0.01-0.03-0.05% CF) at 50 °C after 5 days.

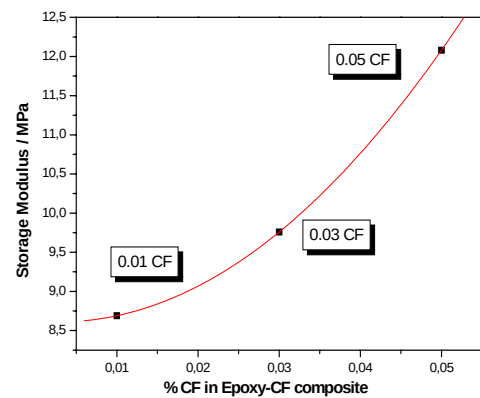


Figure 3.10: Increment of young modulus of epoxy with carbon fiber (T) composites (0.01-0.03-0.05% CF) at 75 °C after 5 days.

In Figure 3.9 and 3.10, while carbon fiber contents of composite increases the values of young modulus ascends.

Also if temperature of stress strain curve rises, young modulus values decreases because of temperature increasing. Carbon fiber effects composite structure and mechanical properties of material conformation.

3.2.3. Dynamic Mechanical Analysis of Reinforced Epoxy/Hard:(10/3) Composites which include 0.1%, 0.3% and 0.5% Chopped Carbon Fiber by Weight

In this part, amount of carbon fiber was increased in carbon fiber reinforced epoxy composite. Dynamic mechanical analysis of reinforced composites such as storage, loss modulus and complex viscosity, also stress-strain curves were studied. The results obtained after five and fifth days.

The comparison was made for the composites of wt. 0.01, 0.03, 0.05% and wt. 0.1, 0.3, 0.5% carbon fiber contents for complex viscosity, and the young modulus or maximum stress and strain was obtained from these curves.

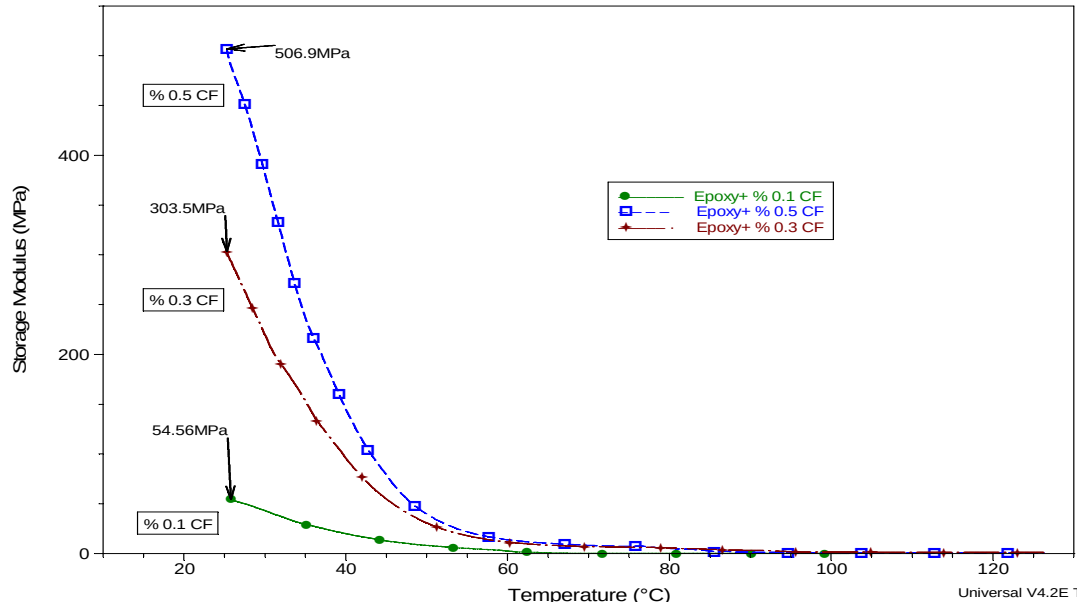


Figure 3.11: DMA spectra of storage modulus versus temperature for composites [Epoxy/Hard:(10/3)] with different amount of carbon fiber(T) (0.01 – 0.03 – 0.05%) after 5 days.

The storage modulus curve was obtained at 1 Hz for the composite samples in Figure 3.11, after five days. It is apparent that the composite samples have not similar modulus values and the reinforcement by amount of carbon fiber increases the modulus of the matrix polymer. The highest value belongs to the composite that has the biggest amount of carbon fiber. The storage modulus values were well-proportional to amount of carbon fiber.

The initial storage modulus (E' at 30 °C) is increased with 0.1%, 0.3% and 0.5% of fibers since the reinforcement of fibers allows transferring the strength at the interface level. Storage modulus decreases gradually at room temperature.

Table 3.9: Loss modulus, complex viscosity values of Carbon fiber Reinforced Epoxy Composites at 30 °C after five days.

	% 0.1 Carbon fiber	% 0.3 Carbon fiber	% 0.5 Carbon fiber
Loss modulus / MPa	37.73	142.5	188.5
Complex viscosity / MPa.sec	3.061	14.24	22.90

Loss modulus was defined as the calculated ability of a material to dissipate mechanical energy by converting it into heat, and it was limited to the molecular motion within the sample that dissipates energy as heat. So, amount of carbon fiber was effected the ability of dissipating mechanical energy. Also, complex viscosities were improved by carbon fiber in composites, positively. While increasing amount of carbon fiber, viscosity values become larger because of the final structure of composites.

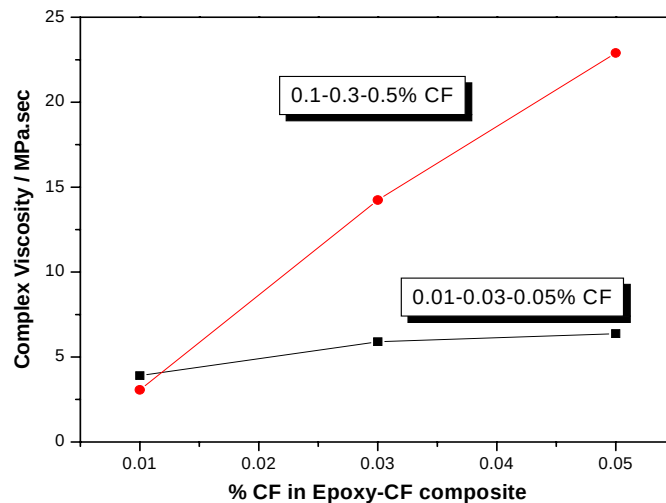


Figure 3.12: Increment of complex viscosity of epoxy with CF(T) composites (0.01 – 0.03 – 0.05% CF and 0.1 - 0.3 - 0.5% CF) after 5 days.

In Figure 3.12, it was seen that with increasing the amount of carbon fiber in composites ten times increases the complex viscosity. It was obvious that carbon fiber can result a change in the interaction of matrices which effects structure, so viscosity values was increased with the increase of amount of carbon fiber.

3.2.4. Stress-Strain Curves of Reinforced Epoxy/Hard:(10/3) Composites which include 0.1%, 0.3% and 0.5% Chopped Carbon Fiber by Weight

Maximum load of 18 N was applied with a ramp force rate of 1 N/min, after 5 days and 50 days. The stress-strain curves at different temperatures, 30 °C, 50 °C and 75 °C were obtained.

And a comparison was made for the composites include 0.01-0.03-0.05% and 0.1-0.3-0.5% carbon fiber by weight with dynamic analysis. A morphologic study can be seen at the end of this section, where compared with SEM figures for composites include wt. 0.05% and 0.5% carbon fiber.

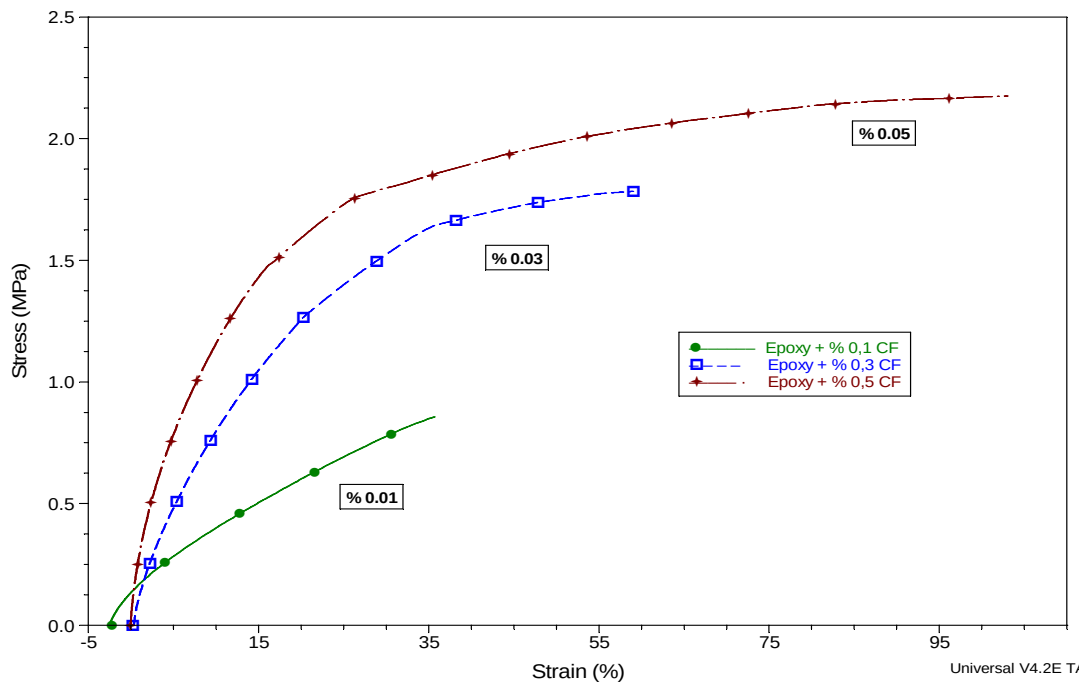


Figure 3.13: Stress–strain curves of composites: [Epoxy/Hard:(10/3)] with amount of carbon fiber(T) (0.1 – 0.3 – 0.5%) at 30 °C after 5 days.

Table 3.10: Young modulus of Epoxy composites with 0.1 - 0.3 – 0.5% carbon fiber at 30 °C, after 5 days.

	Young Modulus / MPa
[Epoxy/Hard:(10/3)] + 0.1% CF	3.652
[Epoxy/Hard:(10/3)] + 0.3% CF	8.005
[Epoxy/Hard:(10/3)] + 0.5% CF	21.690

Stress–strain curves shown in Figure 3.13 and from Table 3.14 revealed that by increasing amount of carbon fiber by weight, strength and failure strain were significantly improved. The composite with 0.1% carbon fiber shows the lowest young modulus values for mechanical characteristics.

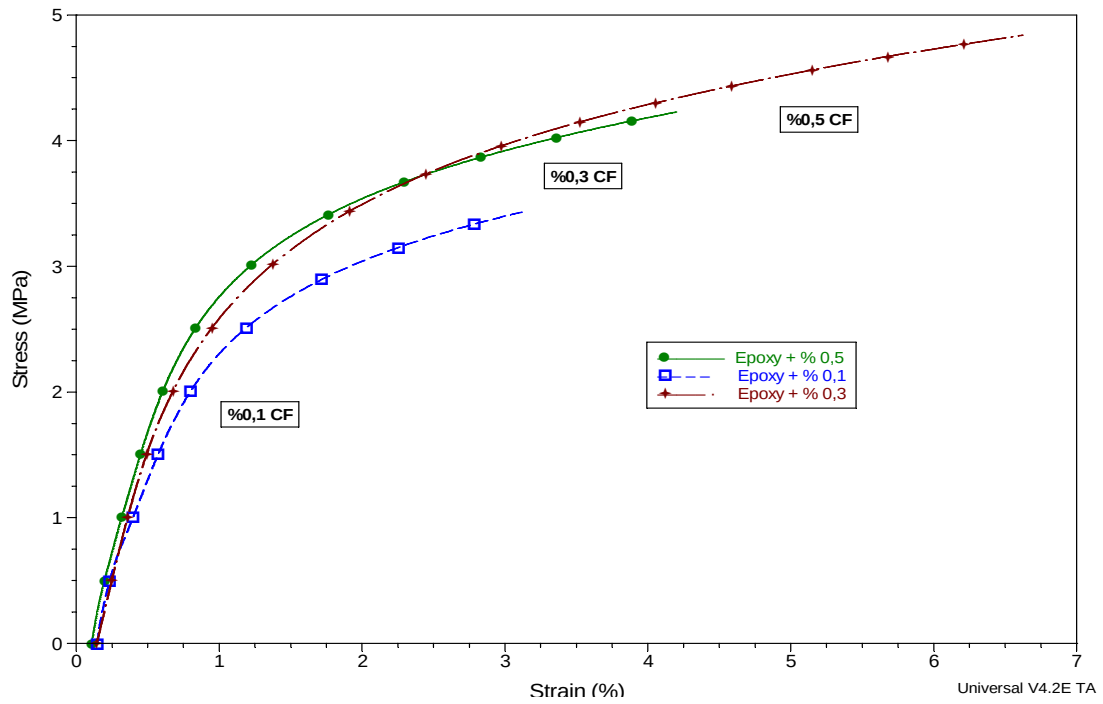


Figure 3.14: Stress–strain curves of composites: [Epoxy/Hard:(10/3)] with amount of carbon fiber(T) (0.1 – 0.3 – 0.5%) at 50 °C after 50 days.

As expected, stress strain curves of composites with carbon fiber were well proportional to amount of carbon fiber. Young modulus values of composites were slightly increased with carbon fiber content. It was a well known that the stress–strain response curves reveal the mechanical nature of the material. While composite with 0.5% carbon fiber's young modulus value was the biggest, composite with 0.5% carbon fiber's maximum strain was the highest. When compared to area under stress strain curve, composite with 0.3% has the highest value but composite with 0.1% has the lowest value. Area under the curve revealed the energy spent to break the specimens.

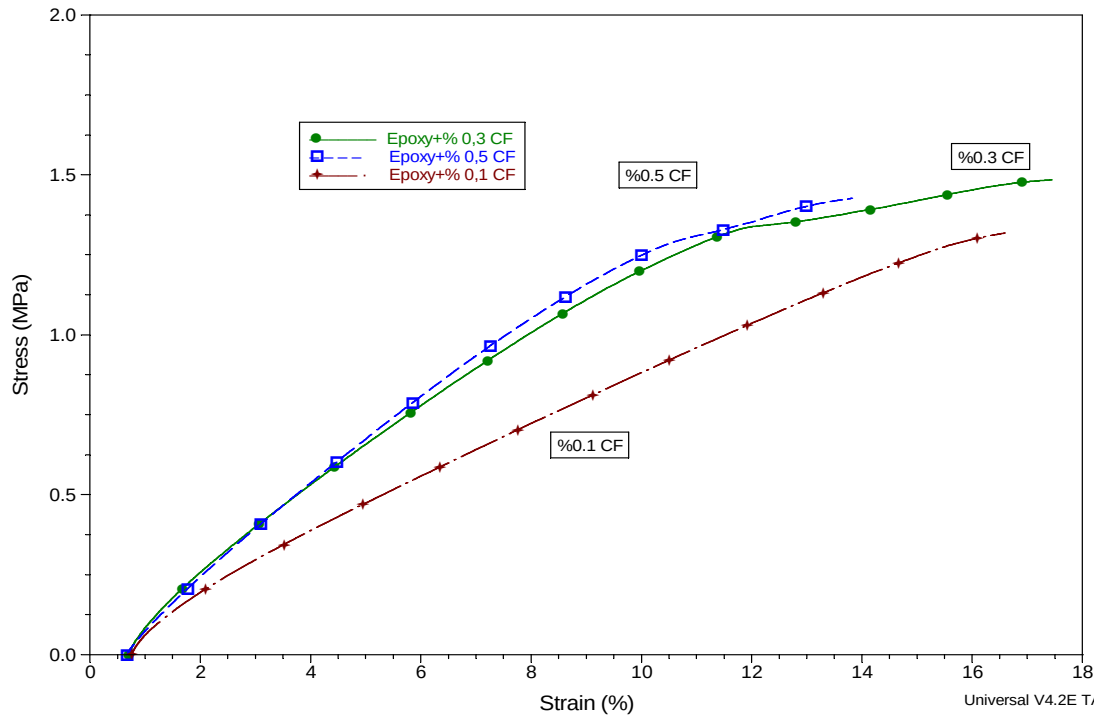


Figure 3.15: Stress–strain curves of composites: [Epoxy/Hard:(10/3)] with amount of carbon fiber(T) (0.1 – 0.3 – 0.5%) at 75 °C after 50 days.

Area under curve of composite with 0.3% carbon fiber has shown the largest area in Figure 3.15, although their young modulus results were nearly the same. In addition, composites with 0.5% and 0.3% carbon fiber has nearly maximum stress. Due to composite structure and matrix, sample with 0.1% carbon fiber has shown a lowest stress compared to others.

Table 3.11: Young modulus values of Carbon fiber Reinforced Epoxy Composites at 30, 50 and 75 °C after five and fifth days. (0.1% - 0.3% - 0.5%)

Young Modulus / MPa After 5 days	wt. 0. 1% Carbon Fiber	wt. 0.3% Carbon Fiber	wt. 0.5% Carbon Fiber
30 °C	3.652	8.005	21.69
50 °C	-	-	-
75 °C	-	-	-
Young Modulus / MPa After 50 days			
30 °C	-	-	-
50 °C	319.0	430.2	448.5
75 °C	11.69	16.47	16.73

Table 3.12: Young modulus of [Epoxy/Hard:(10/3)] + 0.01-0.03-0.05-0.1-0.3-0.5% carbon fiber at 50 °C and 75 °C, after 50 days.

Content of Composites	Young Modulus / MPa (After 50 days)	
	50 °C	75 °C
[Epoxy/Hard:(10/3)]+ 0.01% carbon fiber	269.5	9.950
[Epoxy/Hard:(10/3)]+ 0.03% carbon fiber	353.6	9.280
[Epoxy/Hard:(10/3)]+ 0.05% carbon fiber	334.4	9.440
[Epoxy/Hard:(10/3)]+ 0.1% carbon fiber	319.0	11.69
[Epoxy/Hard:(10/3)]+ 0.3% carbon fiber	430.2	16.47
[Epoxy/Hard:(10/3)]+ 0.5% carbon fiber	448.5	16.73

Variations of temperature in respect to the amount of carbon fiber in composites were compared after 50 days period. As shown in Table 3.12, the temperature dependency of the composites with various amounts of carbon fibers shows an increment of young modulus values. In contrast, it was found that, investigated young modulus results change inversely proportional with temperature.

The effect of the carbon fiber content on morphology was investigated.

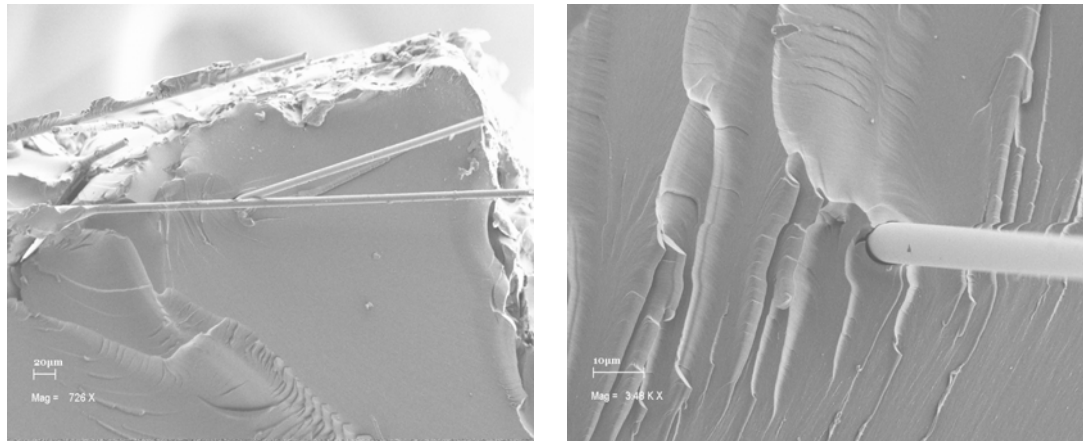


Figure 3.16: SEM micrographs of [Epoxy/Hard:(10/3)] with carbon fiber by weight 0.05 %.

In Figure 3.16 and 3.17, it was seen that ten times increase in the amount of carbon fiber in composites resulted fa not significant changes in the morphology except the numbers of fibers increased.

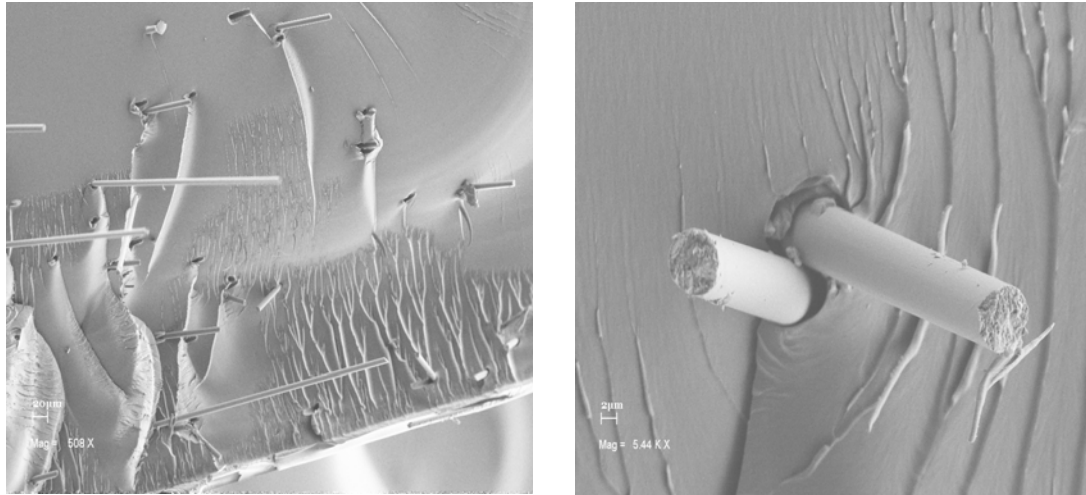


Figure 3.17: SEM micrographs of [Epoxy/Hard:(10/3)] with carbon fiber by weight 0.5% .

3.2.5. Dynamic Mechanical Analysis of Reinforced Epoxy/Hard:(10/5) Composites which include 0.01%, 0.03% and 0.05% Chopped Carbon Fiber by Weight

In this section, dynamic mechanical analysis of reinforced epoxy composites were investigated, in addition to these experiments T_g values were performed. The relationship between T_g (glass transition temperature) and dynamic mechanical results with in tan delta values were searched. In this research it was aimed to display relationships between carbon fiber content and mechanical properties using modulus results.

The storage modulus versus temperature curve, at 1 Hz, for the composites with different amounts of fiber was graphically represented in Figure 3.18. Among 1 the samples analyzed of all composites which have different carbon fiber by weight in this part, the storage modulus is higher for wt 0.05% carbon fiber one.

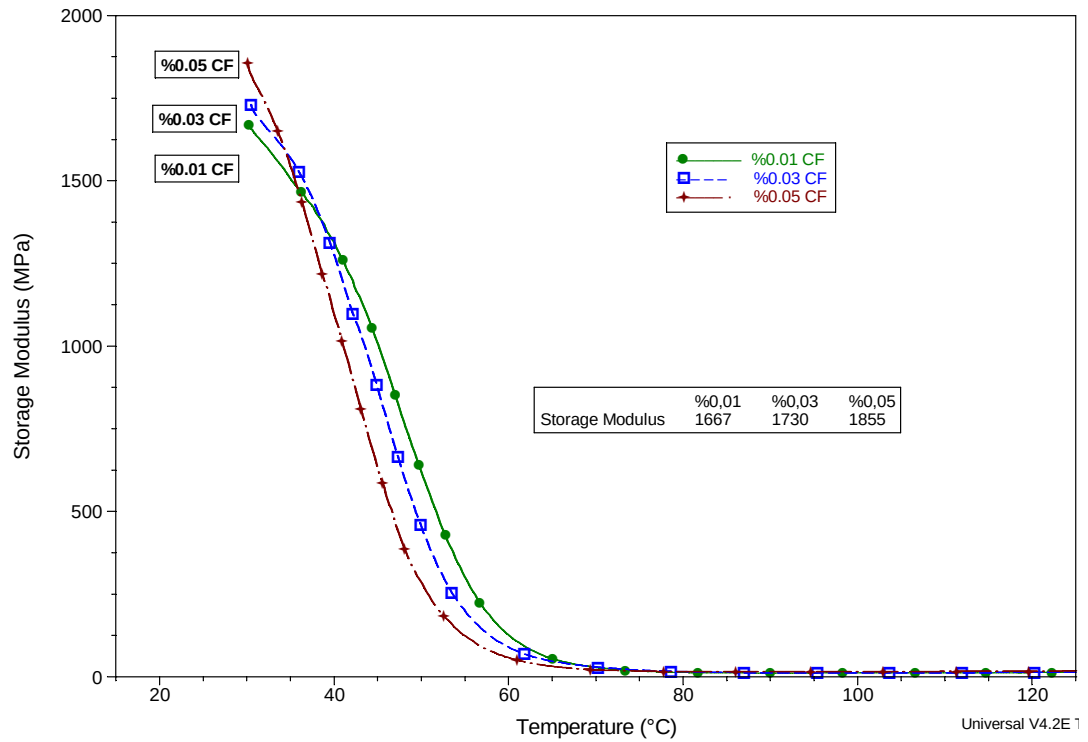


Figure 3.18: Storage modulus of composites [Epoxy/Hard:(10/5)] with amount of carbon fiber(T) (0.01 – 0.03 – 0.05% CF) as a function of temperature, after 5 days.

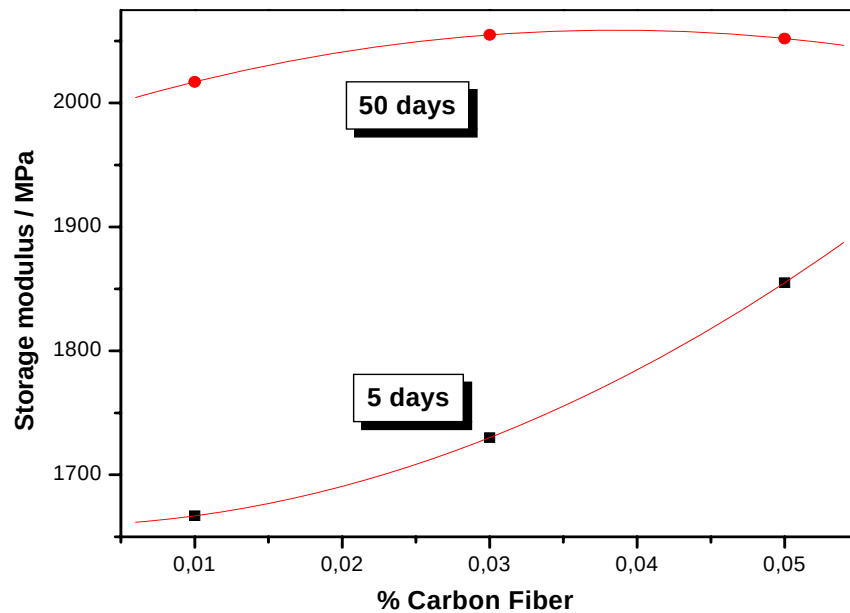


Figure 3.19: Storage modulus values of composites [Epoxy/Hard:(10/5)] with amount of carbon fiber(T) at 30 °C (for 5 days and 50 days)

According to Table 3.13, the storage modulus of epoxy with carbon fiber composites exhibited a linear increase with increasing amount of carbon fiber content up to 0.05% wt for five days sample. Fifty days specimen has an increment of storage modulus value about 25% according to five days sample. Storage moduli of samples for fifty days have similar values compared to each other. It was obvious that carbon fiber contents in composites improved mechanical properties for five days specimens.

Table 3.13: Storage modulus values of Carbon fiber Reinforced Epoxy Composites at 30, 50, 70 and 90 °C after five and fifth days. (0.01% - 0.03% - 0.05%)

Storage Modulus / MPa After 5 days	wt. 0.01% Carbon Fiber	wt. 0.03% Carbon Fiber	wt. 0.05% Carbon Fiber
30 °C	1670	1729	1856
50 °C	614.3	447.5	283.1
70 °C	28.08	29.70	20.83
90 °C	11.30	11.92	15.04
Storage Modulus / MPa After 50 days			
30 °C	2017	2055	2052
50 °C	1367	1570	1824
70 °C	45.48	159.1	283.4
90 °C	11.50	11.87	21.22

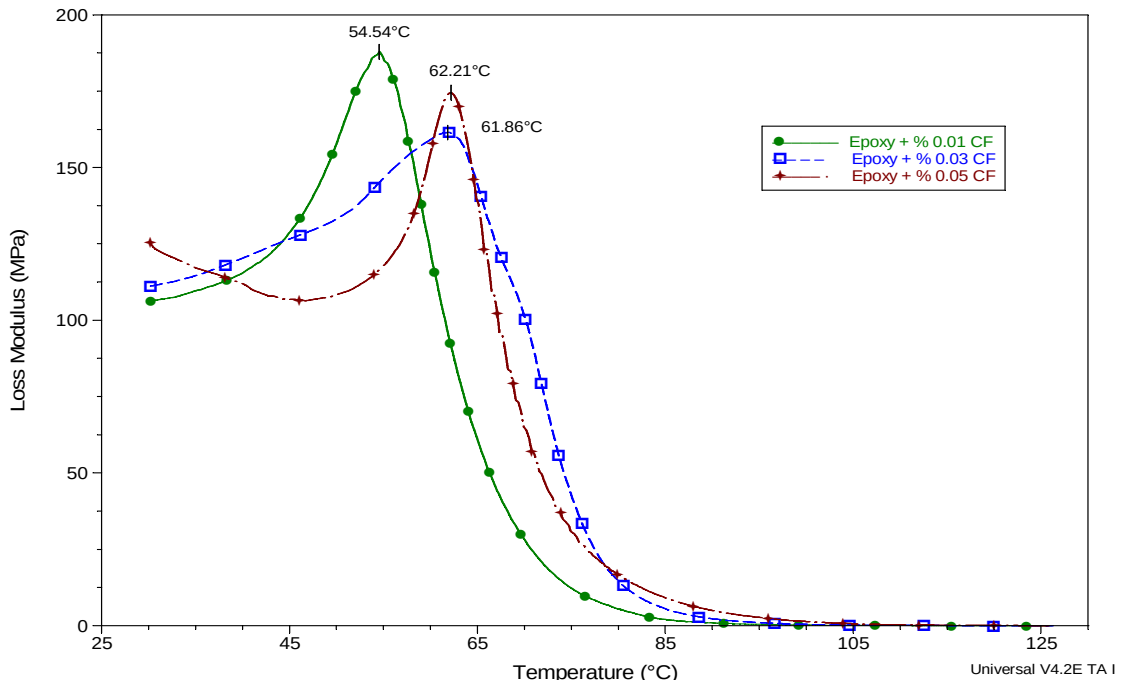


Figure 3.20: Loss modulus of composites [Epoxy/Hard:(10/5)] with different amount of carbon fiber(T) (0.01 – 0.03 – 0.05 % CF) as a function of temperature, after 50 days.

The glass transition temperature (T_g) denotes a change in the material physical property from a glassy state to a rubbery state. This property is important as it can indicate the maximum service temperature at which the material can be used and for thermoset systems, the degree of cure. Some authors suggest the use of the maximum value of E'' as the transition temperature (T_g) [77-79], whereas others assign this transition to the tan delta peak [80].

The peak of tan delta revealed T_g values of composites that [Epoxy/Hard:(10/5)] with amount of carbon fiber by weight 0.01 – 0.03 – 0.05%. The T_g values was obtained from peak of tan delta values. As seen in Figure 3.14, the peak values of composites 64.18, 58.72 and 58.09 °C, T_g values of composites obtained from DMA measurements.

Table 3.14: T_g values of carbon fiber reinforced epoxy composites from DMA

Carbon fiber by weight	T_g from DMA (from tan delta)
Epoxy - %0.01 CF	64.18
Epoxy - %0.03 CF	58.72
Epoxy - %0.05 CF	58.09

Table 3.15: The peaks of tan delta values of carbon fiber reinforced epoxy composites

Carbon fiber by weight	After 5 days	After 50 days
Epoxy - %0.01 CF	64.18	71.18
Epoxy - %0.03 CF	58.72	76.59
Epoxy - %0.05 CF	58.09	73.44

The peaks of tan delta values were increased after 50 days due to epoxy's crosslinking structure resulting a change in thermal properties of epoxy matrix. Dynamic mechanical modulus values that storage modulus, loss modulus, tan delta were increased after 50 days (Table 3.14, 3.15).

3.2.6. Stress-Strain Curves of Reinforced Epoxy/Hard:(10/5) Composites which include 0.01%, 0.03% and 0.05% Chopped Carbon Fiber by Weight

In this part, the amount of carbon fiber was increased ten times compared to the previous section. Dynamic mechanical properties of carbon fiber reinforced epoxy composites were studied. Curves were taken at different temperatures, 30 °C, 50 °C and 75 °C.

Five and fifth days specimens prepared for dynamic mechanical analysis for stress strain investigation.

In this section, a comparison was made for the composites which have a content of 0.01-0.03-0.05% and 0.1-0.3-0.5% carbon fiber by weight for dynamic analysis.

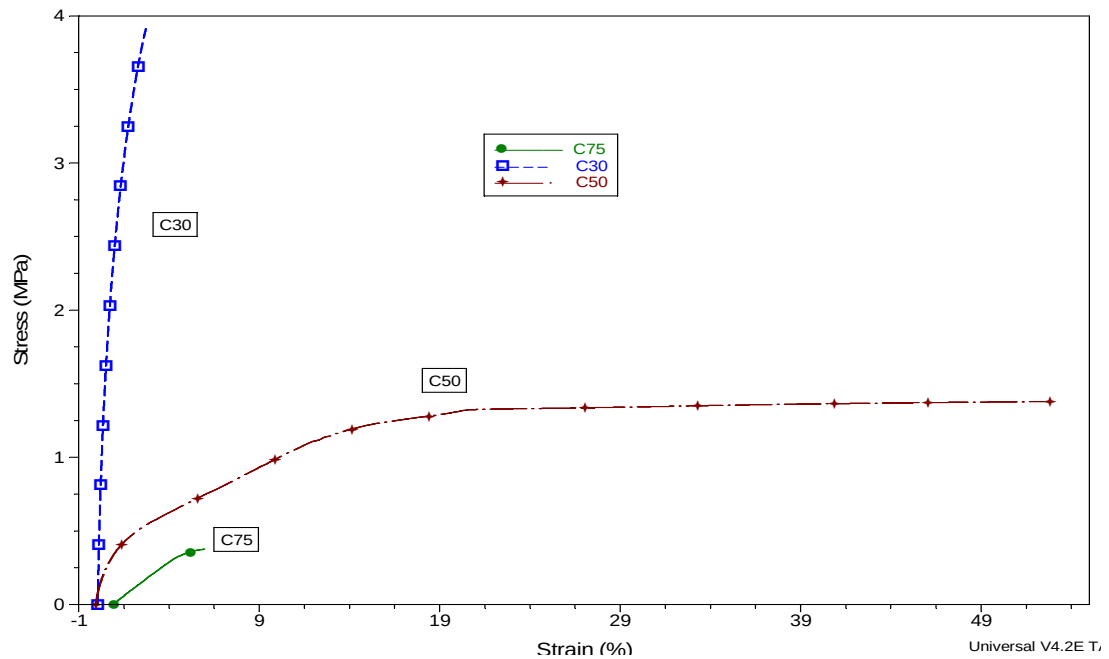


Figure 3.21: Stress strain curves of composites of [Epoxy/Hard:(10/5)] with amount of carbon fiber(T) 0.01% by weight at different temperatures, 30 °C – 50 °C – 75 °C, after 5 days.

While temperature increases, maximum stress of samples was decreased. With increasing temperature, motions related to mechanical properties of epoxy structure become important, and there were mechanical changes can be related to temperature increase

In Figure 3.21, It was seen that toughness (or called impact resistance) values of composite at 50 °C has the highest value and the other specimens have lower values at 30 °C and 75 °C, so the impact resistance of samples was lower for the samples prepared at 30 °C and 75 °C Impact resistance is a measure for the energy absorbed prior to fracture.

For the sample carbon fiber reinforced epoxy by wt 0.01%, the mechanical properties of composite was affected above T_g values and increasing temperature was resulted with decrease in strength, and also in toughness.

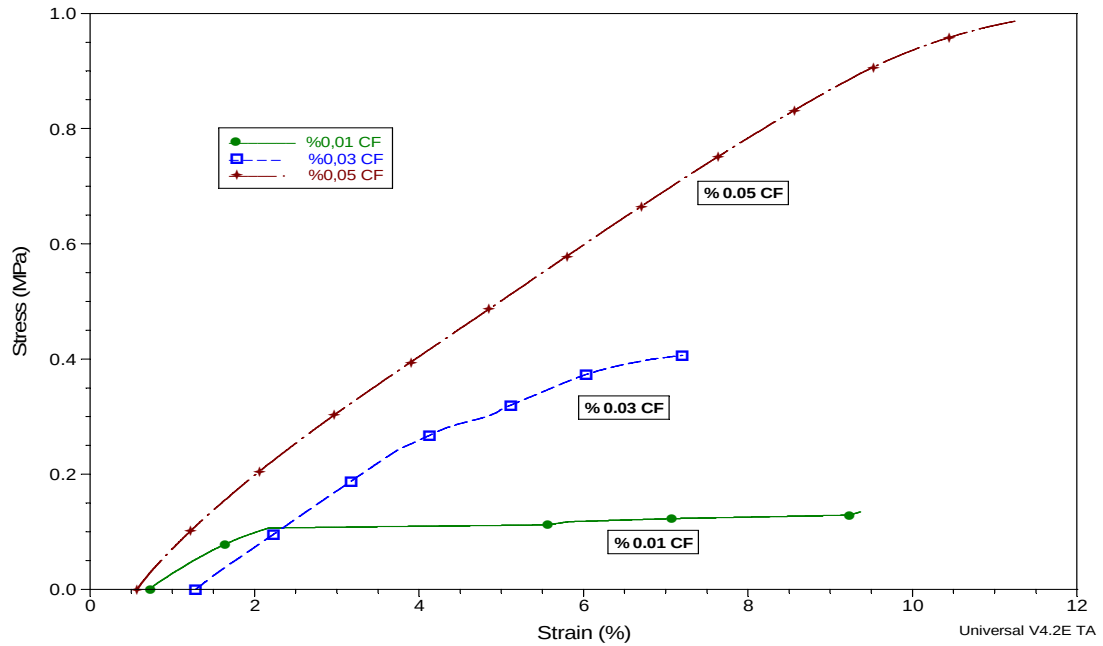


Figure 3.22: Stress strain curves of composite of [Epoxy/Hard:(10/5)] with different amount of carbon fiber(T) 0.01-0.03-0.05% (by weight at 75 °C), after 50 days.

Increase in the amount of carbon fiber resulted an increase in young modulus of composites (Figure 3.22). Carbon fiber content in composites was effected the stress-strain curves and mechanical properties of composites.

Figure 3.22 showed the toughness and strength values of carbon fiber reinforced epoxy composites. Reinforced composite (wt 0.05% carbon fiber content) has the highest toughness and strength values, besides Reinforced composite (wt 0.01% carbon fiber content) has the lowest value of impact resistance.

Table 3.16: Young modulus values of Carbon fiber Reinforced Epoxy(10:5) Composites at 30, 50 and 75 °C after five and fifth days. (0.01% - 0.03% - 0.05%)

Young Modulus / MPa After 5 days	wt. %0.1 Carbon Fiber	wt. %0.3 Carbon Fiber	wt. %0.5 Carbon Fiber
30 °C	324	295	235
50 °C	7.67	8.03	10.2
75 °C	8.69	8.36	0.01
Young Modulus / MPa After 50 days			
30 °C	778	791	890
50 °C	71.7	82.0	111
75 °C	8.68	9.75	12.0

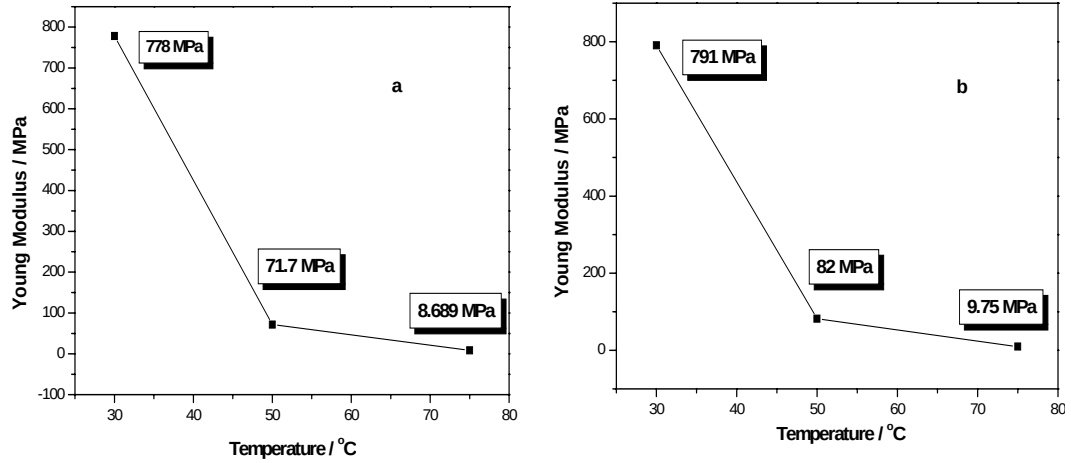


Figure 3.23: Young modulus values of composite of [Epoxy/Hard:(10/5)] with different amount of carbon fiber(T) a) 0.01% b) 0.03% by weight at 30-50-75 °C, after 50 days.

Variations of temperature in respect to the change of the amount of CF in composites were compared after 50 days period. As shown in Figure 3.23 and 3.24, the temperature dependency of the composites with various amounts of carbon fiber show an increment of young modulus values. In contrast, it was found that, investigated young modulus results change inversely proportional with temperature.

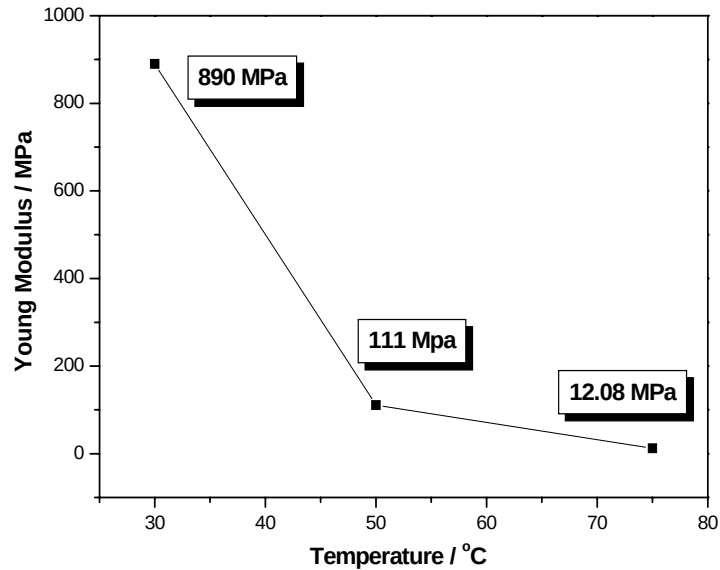


Figure 3.24: Young modulus values of composite that [Epoxy/Hard:(10/5)] with amount of carbon fiber(T) 0.05% by weight at 30-50-75 °C, after 50 days.

3.2.7. Dynamic Mechanical Analysis of Reinforced [Epoxy/Hard:(10/5)] Composites which include 0.1%, 0.3% and 0.5% Chopped Carbon Fiber by Weight

In his part, composites with different amounts of carbon fiber were investigated to explain the interaction between matrix and carbon fiber. Using the results dynamic mechanical analysis, mechanical properties of composites at different temperatures were studied. In this section, carbon fiber reinforced epoxy composites were used which include 0.1%, 0.3% and 0.5% chopped carbon fiber after five days.

The composite which was prepared with epoxy and wt 0.5% carbon fiber as weight, has the highest storage modulus value while the others are smaller. Carbon fiber content affected the results of storage modulus of composite (Figure 3.25).

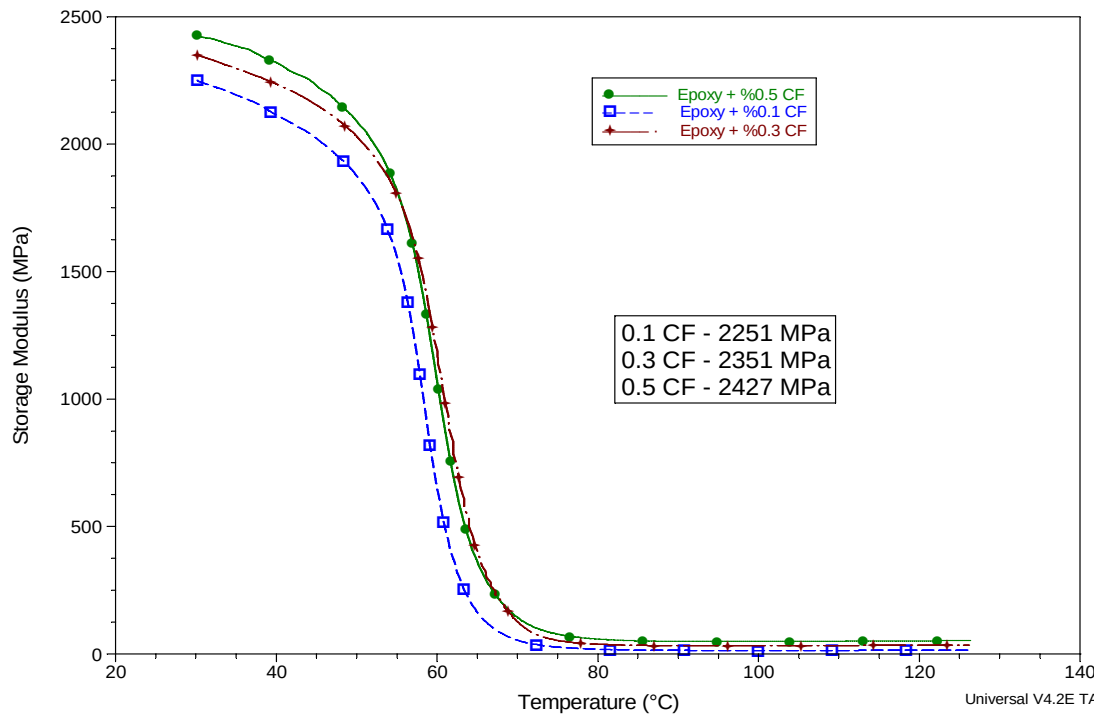


Figure 3.25: Storage modulus for [Epoxy/Hard:(10/5)] composites at various carbon fiber(T) contents (wt 0.1 – 0.3 – 0.5% CF), after 5 days.

Tan delta values of reinforced epoxy composites were important, T_g temperature. The results of T_g temperature were also obtained from DMA results. In Table 3.17, it can be clearly seen that the results of T_g values from tan delta values. T_g values were obtained from the peak of tan delta curves.(Table 3.17)

Table 3.17: T_g values of carbon fiber reinforced epoxy (10:5) composites from DMA.

Carbon fiber by weight	T_g from DMA / °C (from tan delta)
Epoxy - 0.1% CF	70.20
Epoxy - 0.3% CF	70.44
Epoxy - 0.5% CF	71.01

Figure 3.26 shows tan delta curves of three types of carbon fiber reinforced epoxy composites. From the dynamic mechanical analysis curves, peak of tan delta values were nearly same, the glass transition temperature (T_g) of the composite and reinforced epoxy composite with carbon fiber (0.1%, 0.3% and 0.5% wt).

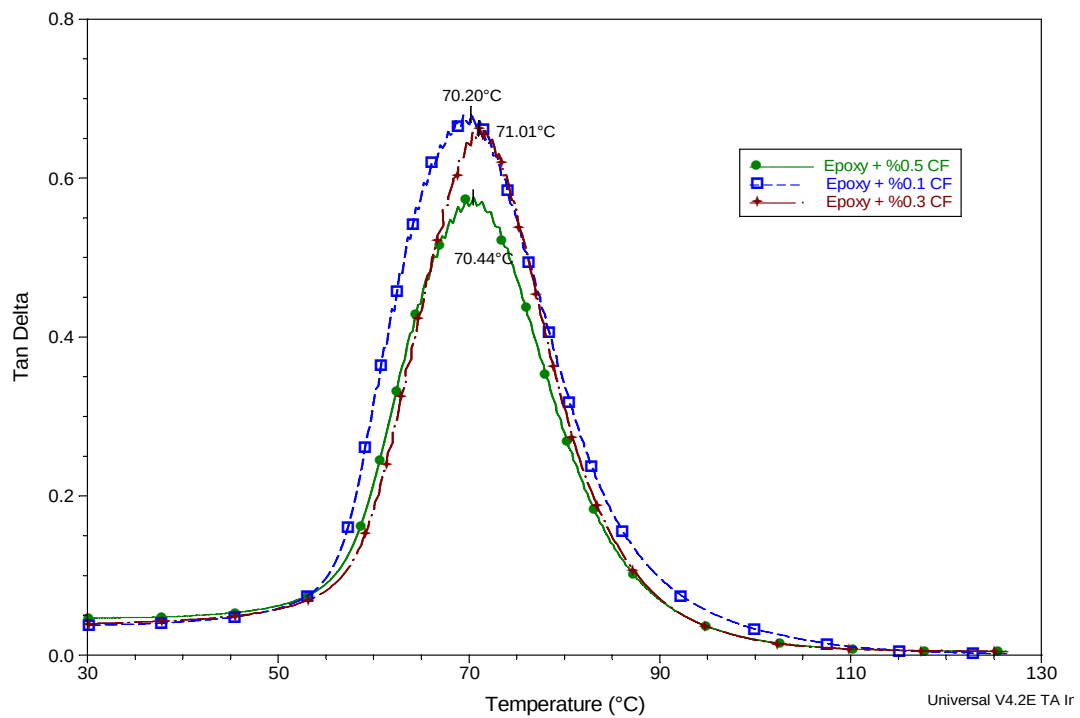


Figure 3.26: Tan delta for [Epoxy/Hard:(10/5)] composites at various carbon fiber(T) contents (wt % 0.1 – 0.3 – 0.5 CF) as a function of temperature, after 5 days.

From the plot of tan delta in Figure 3.26, it can be seen that polyester epoxy composite with carbon fiber wt 0.1% gives the highest tan delta peak followed by wt 0.3% and wt 0.5%. The peak maximum for epoxy composite with carbon fiber wt 0.1% was occurred at the lowest temperature and the peaks for wt 0.3% and wt 0.5% were occurred at around 70 °C confirming that T_g for epoxy composite with carbon fiber wt 0.1% was the lowest. At temperatures above the T_g of the composite, the molecular chains become highly mobile resulting in a damping (tan d) maximum at about 50 °C above T_g .

3.2.8. Stress Strain Curves of Reinforced [Epoxy/Hard:(10/5)] Composites which include 0.1%, 0.3% and 0.5% Chopped Carbon Fiber by Weight

Stress strain curves were taken at different temperatures, 30 °C, 50 °C and 75 °C for all reinforced epoxy composites with wt 0.1%, 0.3% and 0.5%, for five days left specimens.

A morphologic study can be seen at the end of this section. SEM figures for composites include wt 0.05% and 0.5% carbon fiber were compared with each other.

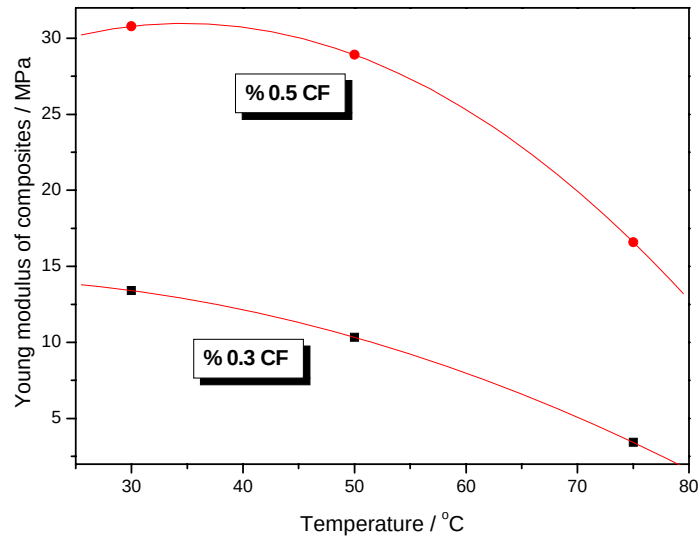


Figure 3.27: Young modulus of [Epoxy/Hard:(10/5)] composites at various carbon fiber(T) contents (wt 0.3% – 0.5% CF) at different temperatures, after 5 days.

By analyzing the young modulus values for different compositions with wt 0.3% and 0.5% carbon fiber, significant differences can be revealed due to composite's contents and experiment temperature (Figure 3.27). Also, values of young modulus were well proportional with the amount of carbon fiber contents.

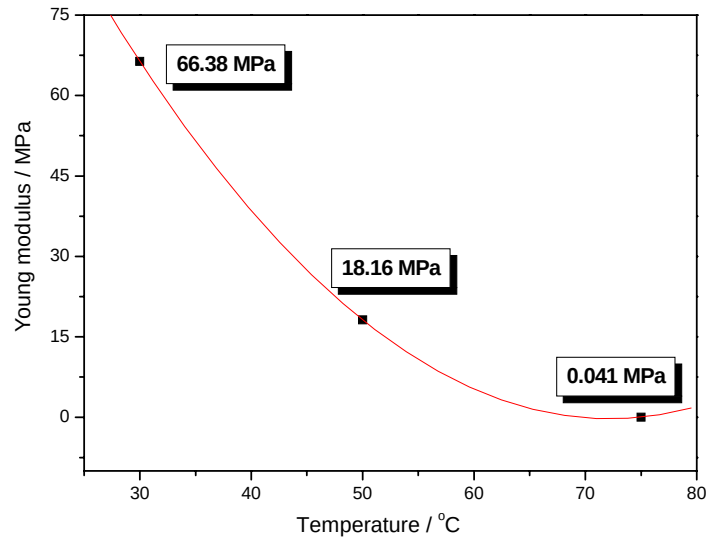


Figure 3.28: Young modulus of [Epoxy/Hard:(10/5)] composite carbon fiber(T) content (wt 0.1%) at different temperatures, after 5 days.

Additionally, the morphology of composites with different carbon fiber contents (wt 0.05% and 0.5%) was studied.

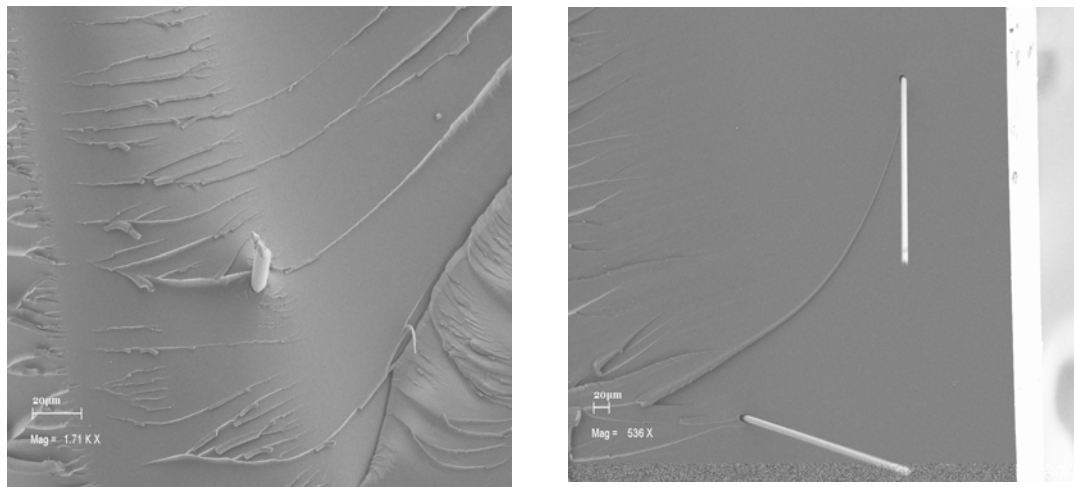


Figure 3.29: SEM micrographs of [Epoxy/Hard:(10/5)] with carbon fiber by weight 0.05 %.

It can be easily seen that epoxy surface morphology with carbon fiber in Figure 3.29, 3.30 that neat epoxy resin exhibits a relatively smooth surface.

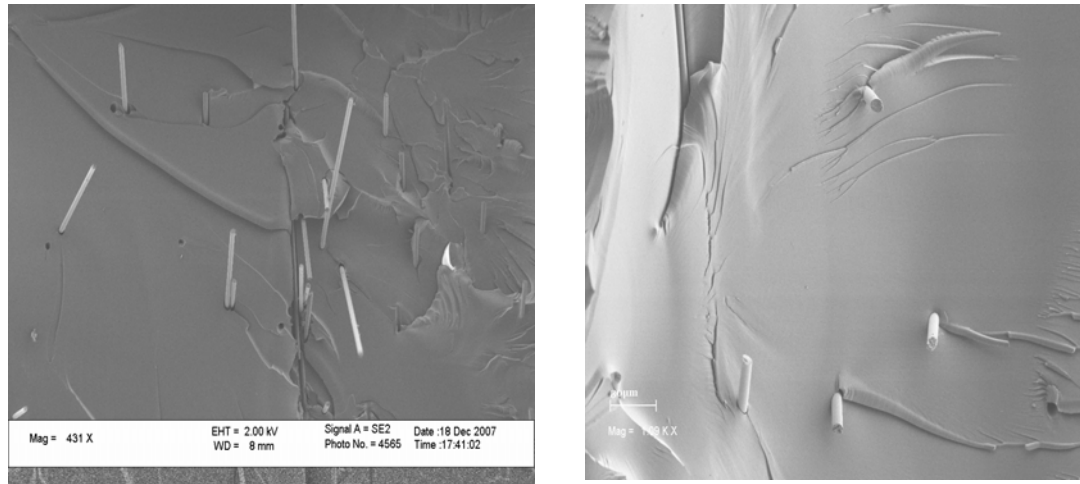


Figure 3.30: SEM micrographs of [Epoxy/Hard:(10/5)] with carbon fiber by weight 0.5 %.

3.2.9. Dynamic Mechanical Analysis of Reinforced [Epoxy/Hard:(10/7)] Composites which include 0.01%, 0.03% and 0.05% Chopped Carbon Fiber by Weight

Mechanical properties of the epoxy composites were investigated with dynamic mechanical analyzer (storage and loss modulus) as a function of temperature, at a constant frequency, 1 Hz.

By changing hardener content the rate of hardener for epoxy resin was changed, and epoxy/hardener:10/7 ratio was used in this part.

The measurements were performed after five days.

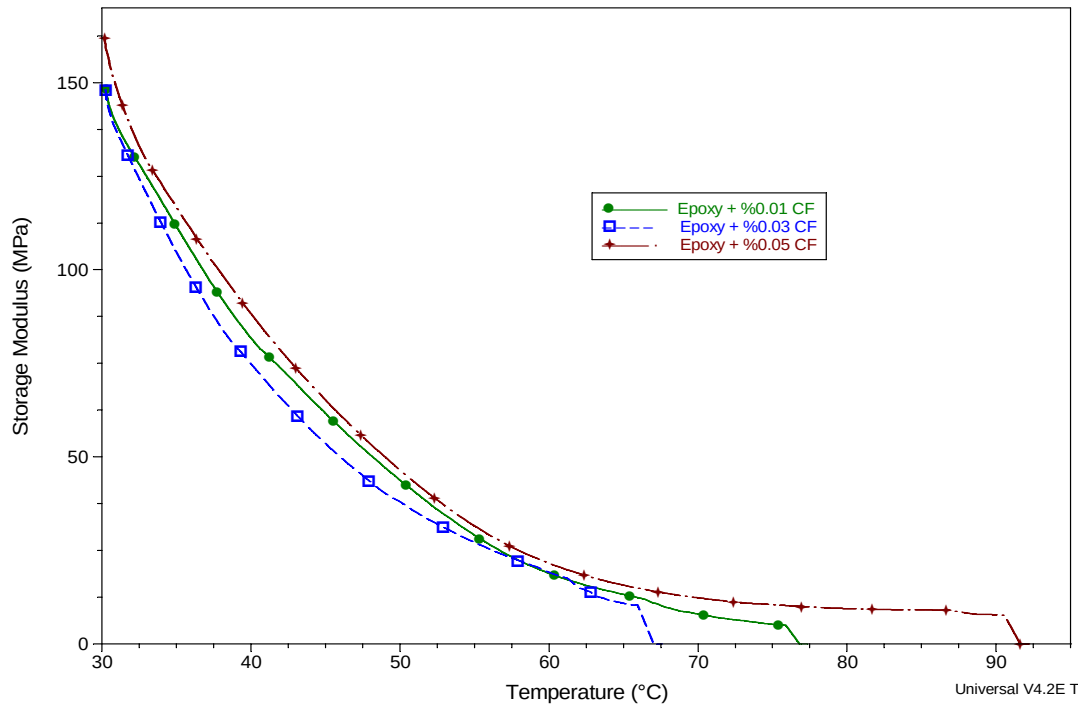


Figure 3.31: Storage modulus of [Epoxy/Hard:(10/7)] composites at various carbon fiber(T) contents (wt 0.01%–0.03%–0.05% CF) as a function of temperature, after 5 days.

All three composites with different amount of carbon fiber could not reach 100 °C and broke off starting from 68, 78 and 92 °C, wt 0.1% – 0.3% – 0.5% carbon fiber. Storage modulus values have not big differences for all the specimen at 30 °C, and nearly 150 MPa. However, the most resistive composite was found with the highest carbon fiber amount, 161 MPa. It was seen that there was a significant decrease on loss moduli values after point of 30 °C (Table 3.18).

Table 3.18: Loss moduli values of [Epoxy/Hard:(10/7)] composites which include 0.01%, 0.03% and 0.05% at different temperatures.

Amount of Carbon Fiber by weight	30 °C	50 °C	70 °C	90 °C
0.01 %	77.50	12.81	1.815	-
0.03 %	96.46	14.18	-	-
0.05 %	87.38	13.67	2.203	0.156

Table 3.19: Storage modulus values of composites which have different rate of epoxy and hardener with various carbon fiber amount, at 30 °C

<i>Amount of CF by weight</i>	Epoxy/Hard:10/3 (E')	Epoxy/Hard:10/5 (E')	Epoxy/Hard:10/7 (E')
% 0.01	53.5	1667	148
% 0.03	85	1730	146
% 0.05	94	1855	161

For all the samples analyzed from Table 3.19, the storage modulus is the highest for Epoxy/Hard:10/5 with 0.05% wt carbon fiber specimen. Minimum value of storage modulus was for Epoxy/Hard:10/3 with 0.01% wt carbon fiber specimen. It was evident that dynamic mechanical properties are influenced by rate of epoxy and hardener and with the amount of carbon fiber.

3.2.10. Stress Strain Curves of Reinforced [Epoxy/Hard:(10/7)] Composites which include 0.01%, 0.03% and 0.05% Chopped Carbon Fiber (by Weight)

The stress-strain curves at different temperatures, 30 °C, 50 °C were investigated. Maximum load of 18 N was applied with a ramp force rate of 1 N/min, after 5 days and 50 days.

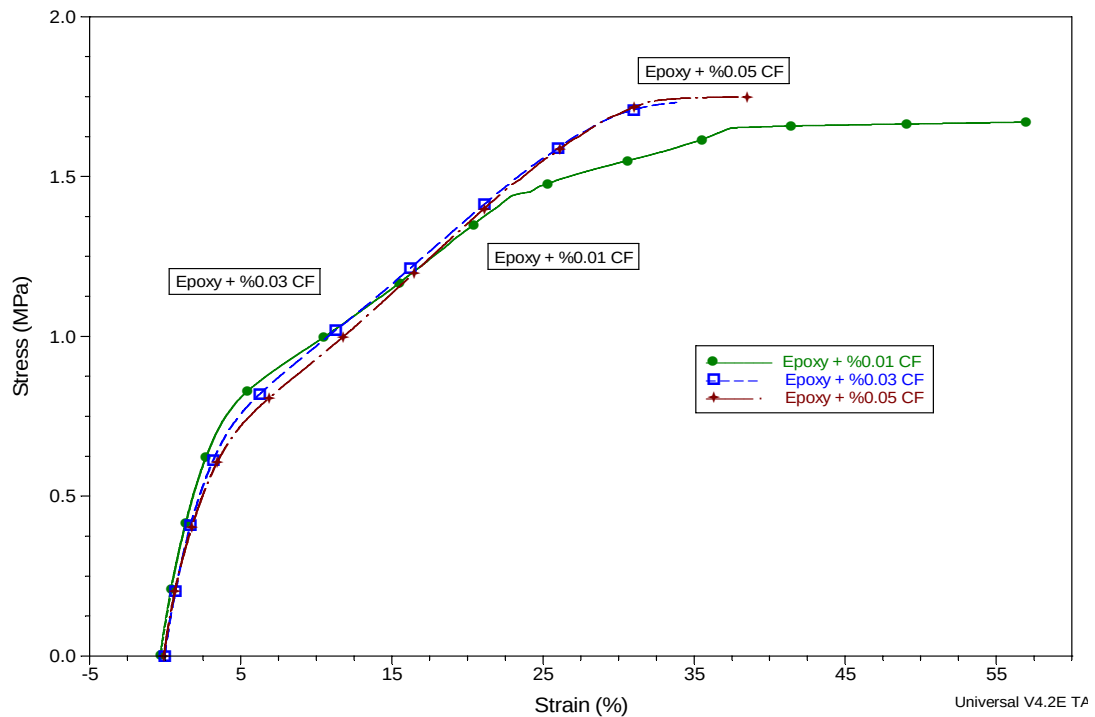


Figure 3.32: Stress strain curves of [Epoxy/Hard:(10/7)] composites at various carbon fiber(T) contents (wt 0.01 – 0.03 – 0.05% CF) at 30 °C, after 5 days.

The stress–strain curves of the composite containing 0.01, 0.03 and 0.05% wt. carbon fiber as epoxy composites at 30 °C were depicted in Figure 3.32, after 5 days. These results showed that the maximum strain to failure was about 57% for reinforced epoxy composite which has wt 0.01% carbon fiber content. Strength of composites was well proportional to composite carbon fiber contents. It was shown that the reinforcement effect reaches a maximum for strength and Young modulus at 0.05% wt. carbon fiber.

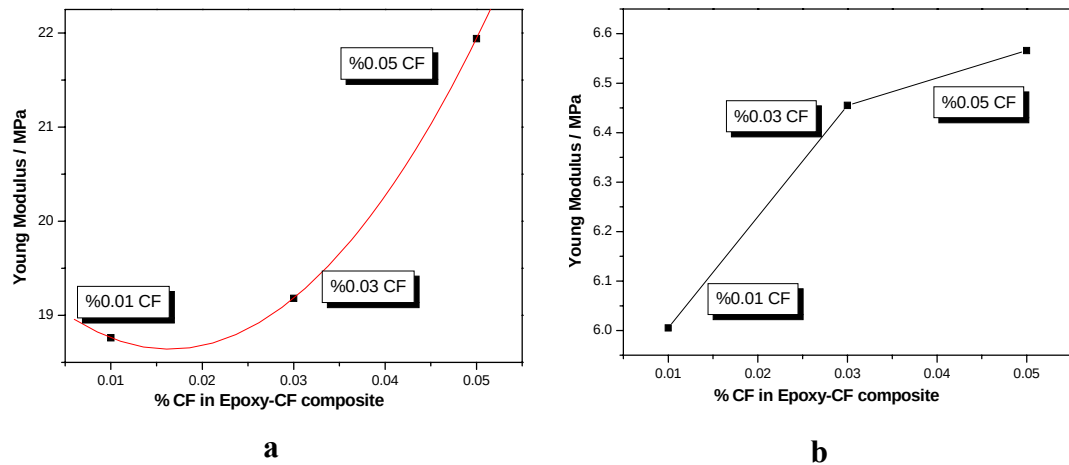


Figure 3.33: Young modulus of Epox/Hard:10/7 with wt 0.01-0.03% and 0.05% carbon fiber at a) 30 °C and b) 75 °C after 5 days.

The young modulus values of reinforced epoxy composites with %0.01, %0.03 and %0.05 carbon fiber by weight at 30 °C and 75 °C, after five days is shown in Figure 3.33. Increasing of young modulus values can be related with carbon fiber content at different temperatures. Increasing carbon fiber content for composites was resulted in developing of mechanical properties.

3.2.11. Dynamic Mechanical Analysis of Reinforced [Epoxy/Hard:(10/7)] Composites which include 0.1%, 0.3% and 0.5% Chopped Carbon Fiber by Weight

The carbon fiber reinforced epoxy composites were heated from +30 °C to +130 °C with a heating rate 5 °C/min. The applied frequency was 1 Hz. The samples were tested using tensile film clamp.

The experiments for epoxy composites were performed with dynamic mechanical analyzer, after five days.

T_g points of composites were determined with DMA.

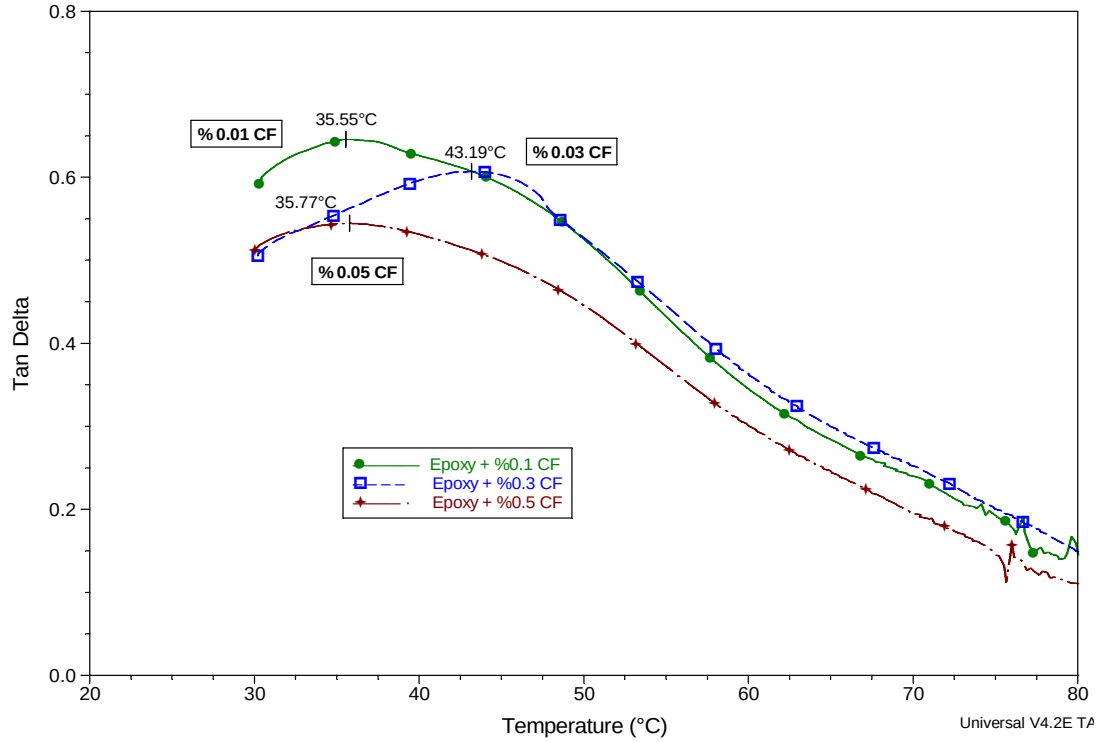


Figure 3.34: Dynamic mechanical thermal analysis of reinforced epoxy composites (0.1 - 0.3 – 0.5% carbon fiber) as a function of temperature of the tan delta, after five days.

Because of the restricted movement of molecules, the incorporation of carbon fibers in a composite structure reduces the tan delta peak height; 0.05% CF composite (Figure 3.34).

It was studied that tan delta values of composites with peak points of temperatures. Tan delta peak temperature of composites, T_g values were showed. There were differences on toughness or impact resistance of carbon fiber reinforced epoxy composites between 0.01%, 0.03% and 0.05% by weight.

T_g values from DMA showed differences for [Epoxy/Hard:(10/7)] composites which include 0.1%, 0.3% and 0.5% carbon fiber by weight. Composites with 0.1%, 0.3% carbon fiber have not similar T_g values obtained from dynamic mechanical analysis (Table 3.20).

Storage modulus of composites was not proportional to carbon fiber contents at 30 °C. The lowest value of storage modulus was 330 MPa at 30 °C for the wt. 0.03% carbon fiber content.

Table 3.20: T_g values of carbon fiber reinforced epoxy (10:7) composites from DMA.

Carbon fiber by weight	T_g from DMA (from tan delta)
Epoxy - 0.1% CF	35.5
Epoxy - 0.3% CF	43.1
Epoxy - 0.5% CF	35.7

Loss modulus of values for epoxy-CF composite increases with amount of carbon fiber but while temperature of experiment rise, storage modulus and tan delta values decreased. Carbon fiber influences mechanical properties of composite at different temperatures. There was a sharply decrease on values of loss modulus and complex viscosity. Values of loss modulus were proportional for all carbon fiber reinforced epoxy composites at 50 °C and 70 °C. Complex viscosity is proportional with storage modulus values (Table 3.21).

Table 3.21: Loss modulus and tan delta values of carbon fiber reinforced epoxy (10:7) composites.

<i>%0.1 Carbon Fiber</i>	30 °C	50 °C	70 °C	90 °C
Loss Modulus / MPa	184.1	26.24	5.035	0.433
Tan delta	0.477	0.440	0.210	0.037
Complex Viscosity / MPa.sec	23.78	4.780	0.997	0.582
<i>%0.3 Carbon Fiber</i>				
Loss Modulus / MPa	162.2	36.54	5.599	-
Tan delta	0.496	0.464	0.211	-
Complex Viscosity / MPa.sec	20,16	3.609	1.357	-
<i>%0.5 Carbon Fiber</i>				
Loss Modulus / MPa	184.4	36.70	6.045	1.066
Tan delta	0.432	0.400	0.126	0.028
Complex Viscosity / MPa.sec	25.58	5.936	2.630	2.125

3.2.12. Stress Strain Curves of Reinforced [Epoxy/Hard:(10/7)] Composites which include 0.1%, 0.3% and 0.5% Chopped Carbon Fiber by Weight

In this section stress strain curves at different temperatures, 30 °C, 50 °C and 75 °C for reinforced epoxy composites with wt 0.1%, 0.3% and 0.5% carbon fiber were presented(for five days specimens).

A morphologic study can be seen at the end of this section. SEM figures for composites include wt. 0.05% and 0.5% carbon fiber were compared.

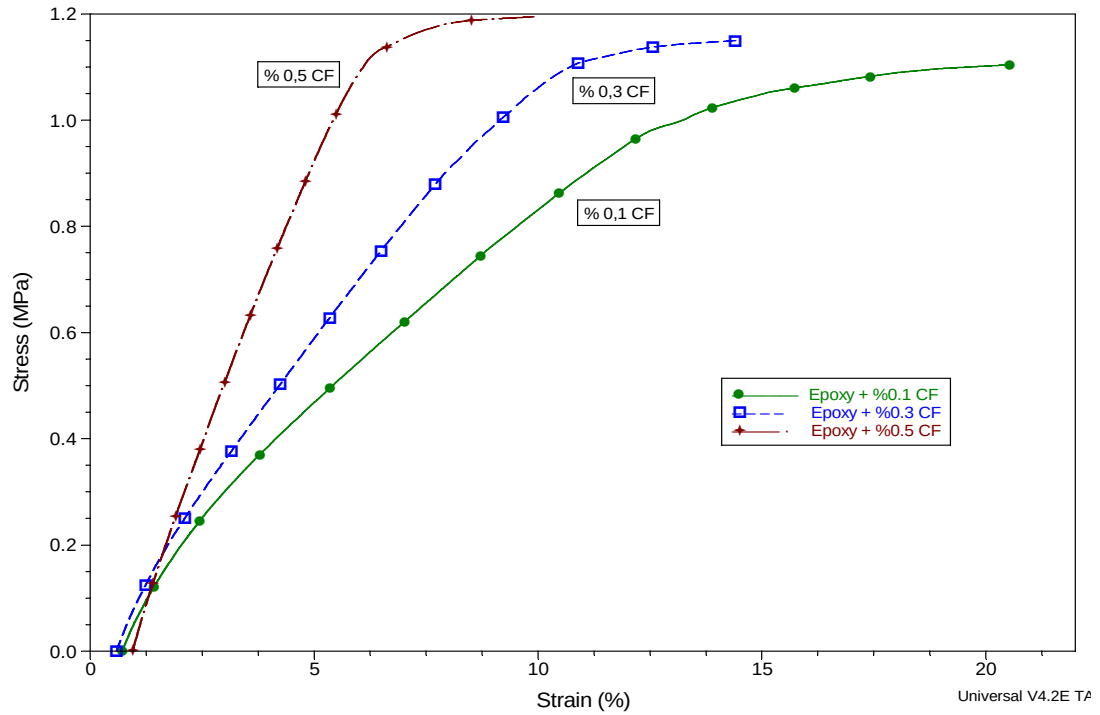


Figure 3.35: Stress strain curves of [Epoxy/Hard:(10/7)] composites at various carbon fiber(T) contents (wt 0.1% – 0.3% – 0.5% CF) at 50 °C, after 5 days.

Stress strain curves of reinforced epoxy composites were shown in Figure 3.35. Maximum stress and strain values can be seen easily and were proportional to carbon fiber contents. Epoxy composite with 0.5% carbon fiber has the highest stress value and young modulus. Also young modulus of composites was proportional to amount of carbon fiber in composites. On the other hand, epoxy composite with 0.1% carbon fiber has the highest maximum strain; it can be related to carbon fiber and their effect on composite matrix.

Mechanical properties of composites were developed with carbon fiber content. Carbon fiber generally increased the mechanical properties of samples.

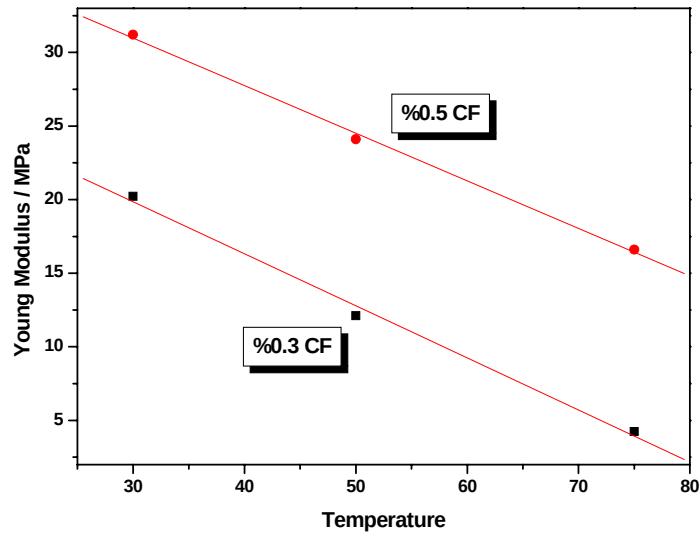


Figure 3.36. Young modulus of [Epoxy/Hard:(10/7)] composites at various carbon fiber(T) contents (wt 0.3 % – 0.5 % CF) at 30-50-75 °C, after 5 days.

In Figure 3.36, young modulus of composites increased with carbon fiber content. In contrast, young modulus values were decreased with increasing temperatures.

The morphology of epoxy composites was studied with scanning electron microscopy.

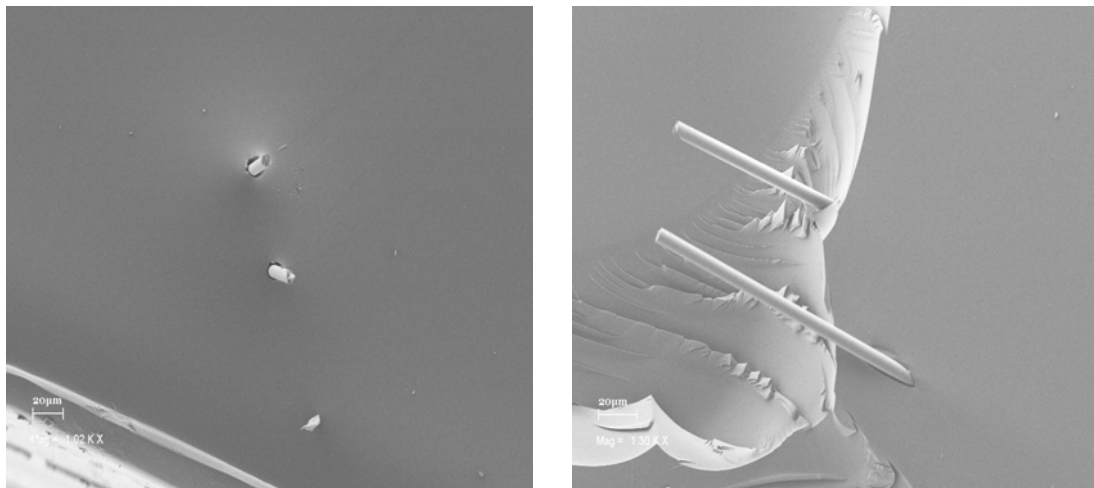


Figure 3.37: SEM micrographs of [Epoxy/Hard:(10/7)] with carbon fiber by weight % 0.05

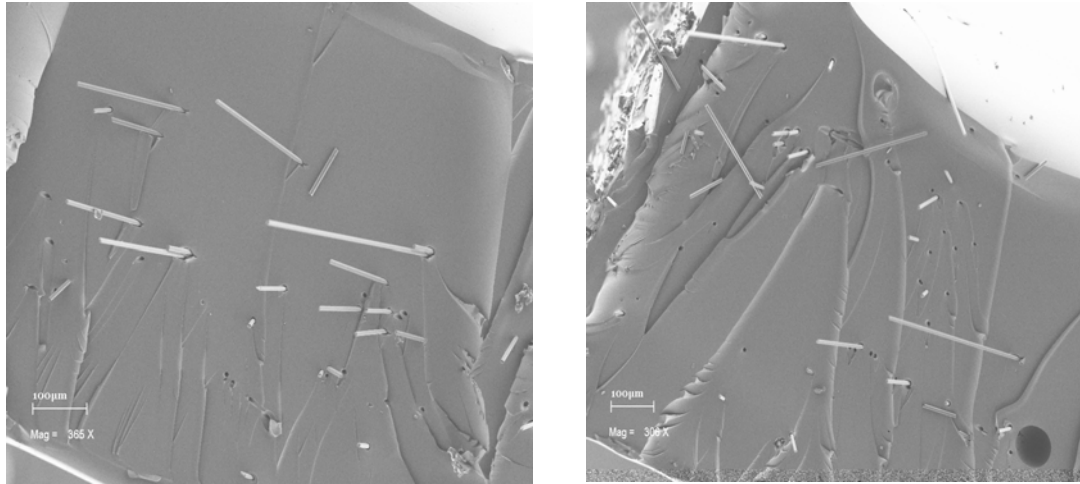


Figure 3.38: SEM micrographs of [Epoxy/Hard:(10/7)] with carbon fiber by weight % 0.5

3.3. Characterization of Bundle(s) Carbon Fiber Reinforced Polyurethane Composites

In this part , it was aimed to investigate the mechanical properties of carbon fiber reinforced polyurethane composites. The samples were prepared by bundle(s) of carbon fibers. Two types of carbon fibers (Torayca and Metyx) were used for the experiments.

Mechanical poperties of polyurethane composites were presented by storage modulus, loss modulus and tan delta values.

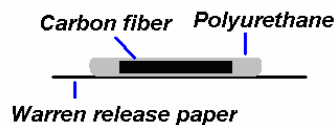


Figure 3.39: Crossectional view of one bundle of carbon fiber (horizontally) modified polyurethane composite.(1x1)

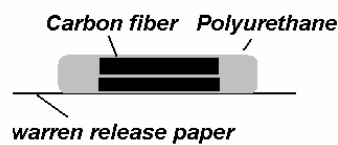


Figure 3.40: Crossectional view of two bundles of carbon fiber (horizontally) modified polyurethane composite.(1x2)

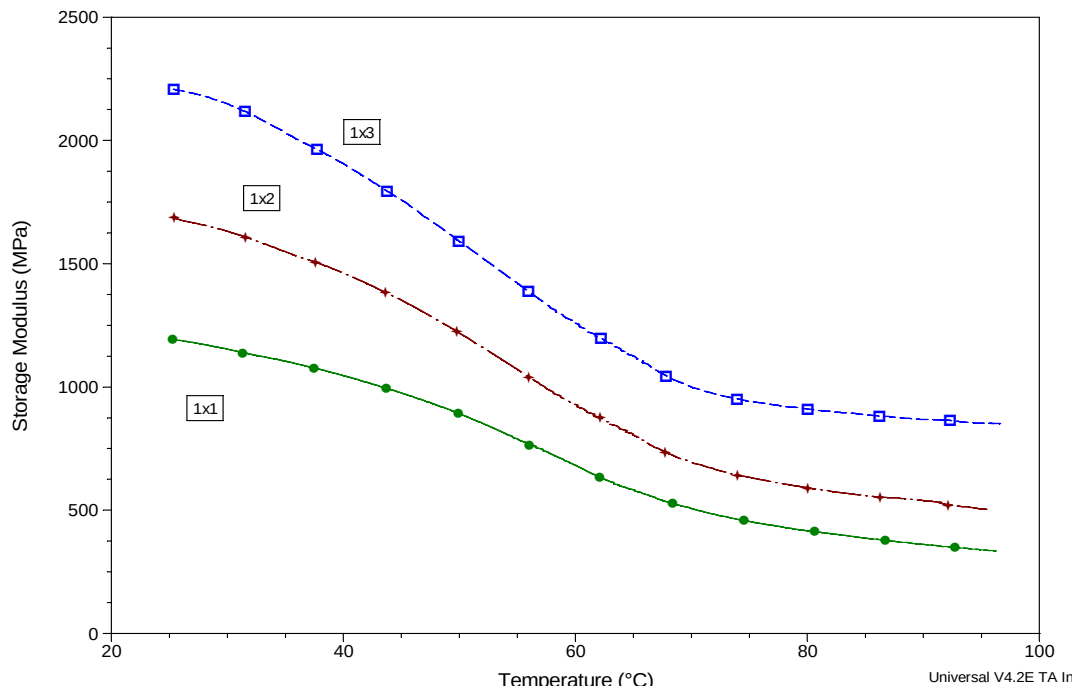


Figure 3.41: Storage modulus of PU – CF(T) composite versus temperature. (1x1, 1x2, 1x3)

It can be seen that the storage modulus of carbon fiber reinforced polyurethane composites steadily increased with an increasing bundle of fiber. The storage modulus (E') as a function of temperature is graphically represented in Fig. 3.41 for reinforced polyurethane composites (1x1, 1x2 and 1x3). As can be observed, the incorporation of both fibers were risen to a considerable increase of the polyurethane stiffness.

Table 3.22: Storage modulus, loss modulus and tan delta values of PU-CF(T) composites at different temperatures.

	Storage Modulus (E') / MPa			Loss Modulus (E'') / MPa			Tan Delta		
	30 °C	60 °C	90 °C	30 °C	60 °C	90 °C	30 °C	60 °C	90 °C
(1X1)	1153	680.5	361.2	219.5	151.8	77.14	0.190	0.222	0.213
(1X2)	1630	928.7	538.5	327.5	200.2	104.4	0.200	0.215	0.194
(1X3)	2149	1225	868.3	737	426.5	264.4	0.345	0.339	0.304

Variation of temperature in respect to amount of CF with different placement in composites were compared via 30 °C – 60 °C – 90 °C. As shown in Table 3.22, the temperature dependency of the composites with various amounts of carbon fiber does not show a significant change in storage, loss modulus and tan delta values. In contrast, it was found that temperature dependency of composites increases sharply after temperature increases.

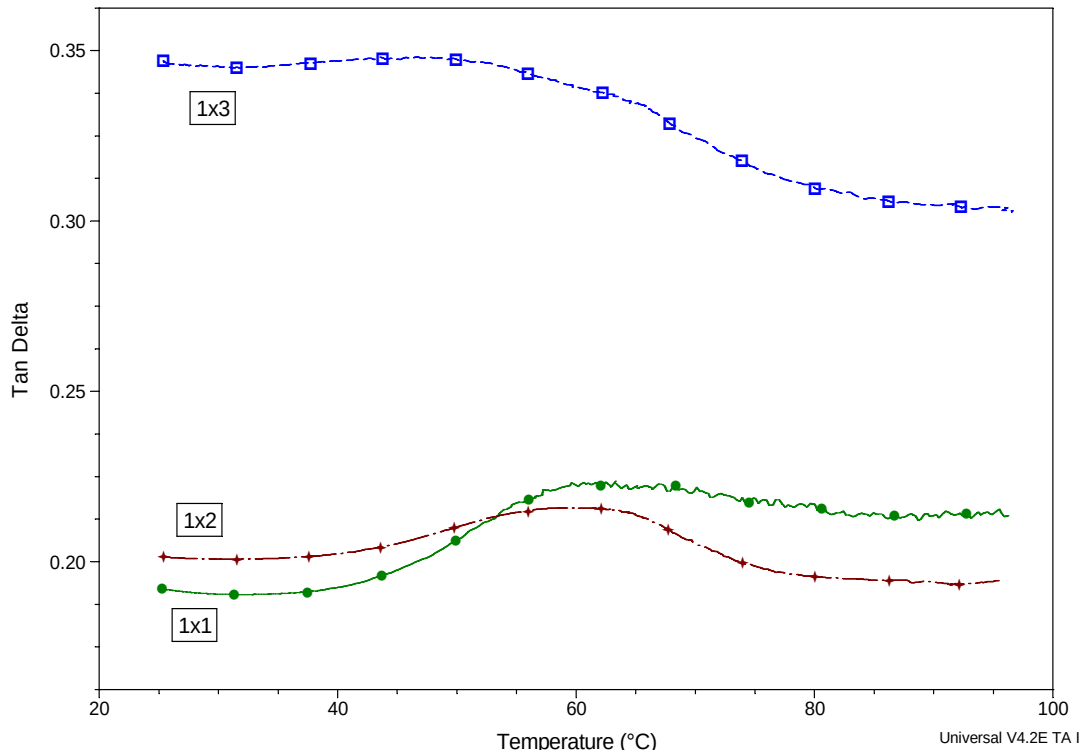


Figure 3.42: Tan delta values of PU – CF(T) composite. (1x1, 1x2, 1x3)

As shown in Figure 3.42, tan delta values of PU-CF composite shows an increment from 1x1 to 1x3 as the amount of CF increases and has the highest tan delta value in correlation with the storage modulus.

Table 3.23 : Complex viscosity values and increasing ratios of PU-CF(T) composites at 25 °C . (Reference: PU - (1x1) CF)

	Complex Viscosity / MPa.sn	% Change
PU	0.270	-
PU - (1X1) CF	<u>67.21</u>	-
PU - (1X2) CF	95.19	141
PU - (1X3) CF	129.1	192

Other samples were prepared with other carbon fiber, Metyx. All the same procedures were applied to specimens.

The material was heated from +25 °C to +130 °C with a heating rate 5 °C/min and the applied frequency was 1 Hz for storage, loss modulus and complex viscosity.

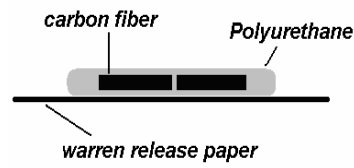


Figure 3.43 : Crossectional view of two bundles of CF(horizontally and vertically) modified polyurethane composite.

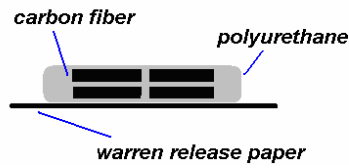


Figure 3.44 : Crossectional view of four bundles of CF(horizontally and vertically) modified polyurethane composite.

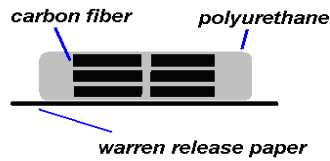


Figure 3.45: Crossectional view of six bundles of CF(horizontally and vertically) modified polyurethane composite

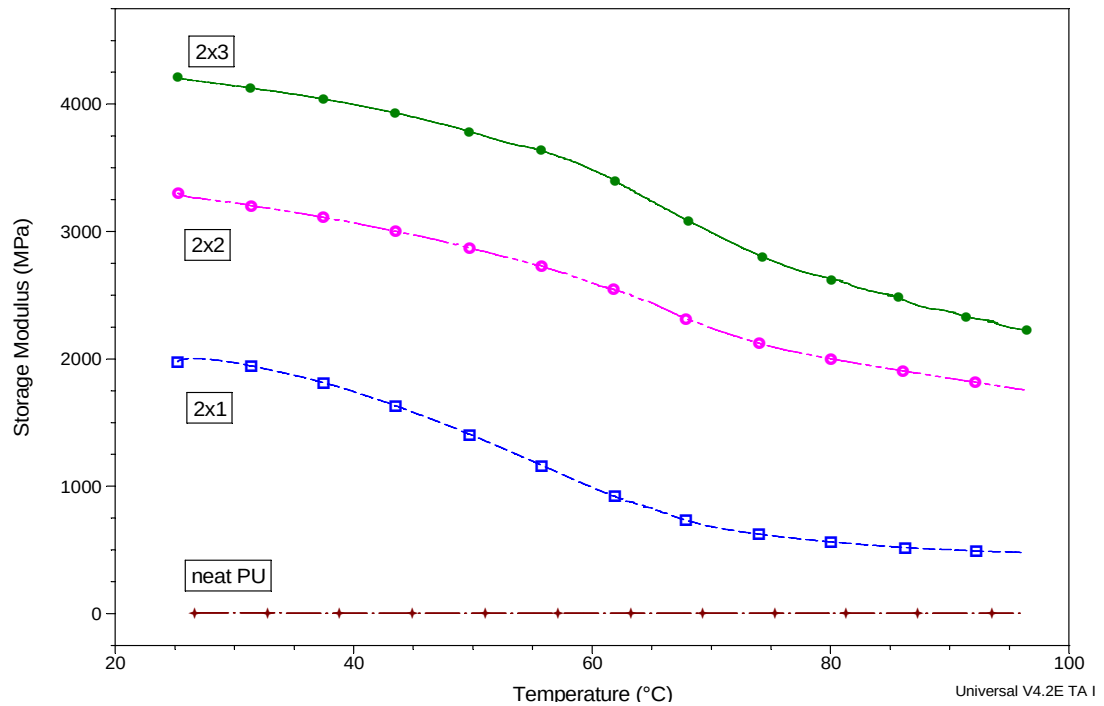


Figure 3.46: Storage modulus of PU – CF(M) composites. (PU, 2x1, 2x2, 2x3)

Figure 3.46 shows the storage modulus values of PU composites and compared with the neat polyurethane. (2x3) PU composite displays the most strength in comparison with the other investigated PU samples. However, (2x1) PU composite shows a storage modulus 500 times bigger than the neat PU composite as well.

Table 3.24 : Storage modulus and loss modulus of PU-CF(M) composites at 60 °C. (Reference: PU-(2x1)CF(M))

	Storage Modulus / MPa	% Change	Loss Modulus / MPa	% Change
PU	4.699	-	0.374	-
[PU-(2x1)CF]	1969	-	347.6	-
[PU-(2x2)CF]	3225	163	1933	556
[PU-(2x3)CF]	4144	210	3457	994

3.4. Characterization of Carbon Fiber Reinforced Polyurethane-Epoxy Polymer Systems

3.4.1. Dynamic Mechanical Analysis of Reinforced Polyurethane-Epoxy Polymer Systems which include 0.015 g, 0.030 g and 0.050 g Chopped Carbon Fiber

In previous studies, there were only carbon fiber reinforced epoxy or polyurethane composites. In this part epoxy, polyurethane and carbon fiber composites were presented. Mechanical properties of reinforced polyurethane-epoxy polymer systems were studied; storage modulus, loss modulus and tan delta values were investigated. Effect of carbon fiber content in reinforced polyurethane-epoxy was investigated on polymer systems.

Different amount of carbon fibers were used for reinforced polyurethane-epoxy polymer systems. Also, different amounts of polyurethane were used for reinforced polyurethane-epoxy polymer systems.

The samples of carbon fiber reinforced polyurethane-epoxy polymer systems were heated from +30 °C to +100 °C with a heating rate 5 °C/min. The applied frequency was 1 Hz. The samples were tested using tensile film clamp.

The experiments for polyurethane-epoxy polymer systems were performed with dynamic mechanical analyzer, after five days.

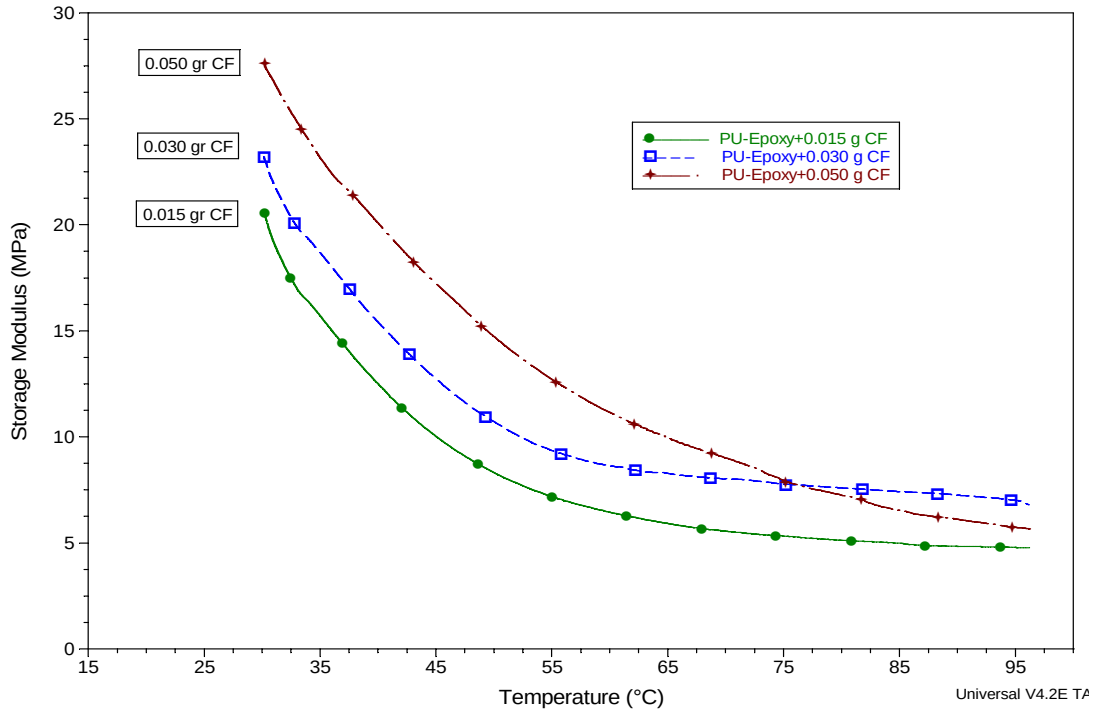


Figure 3.47: Storage modulus of Reinforced Polyurethane-Epoxy Polymer Systems which include 0.015 g, 0.030 g and 0.050 g Carbon Fiber

5 gram of polyurethane was used in these experiments (Figure 3.47). Differences of storage modulus values for carbon fiber reinforced polyurethane-epoxy polymer systems between 0.015 g carbon fiber to 0.050 g carbon fiber content can be seen in Figure 3.39. Carbon fiber content effected modulus of reinforced polyurethane-epoxy polymer systems. Epoxy composites show high storage modulus values. But addition of polyurethane decreases this storage modulus. However, the highest storage modulus value were obtained with the composites enrich in carbon fiber. From Table 3.25, polymer systems with 0.030 g CF and 0.050 g CF have increased their storage modulus values from 14% to 36%.

Table 3.25: Storage modulus of reinforced polyurethane-epoxy polymer systems with different carbon fiber contents at 30 °C.

<i>Contents of Samples</i>	Storage Modulus / MPa	% Change
PU-Epoxy + 0.015 g CF	20.32	-
PU-Epoxy + 0.030 g CF	23.16	14
PU-Epoxy + 0.050 g CF	27.64	36

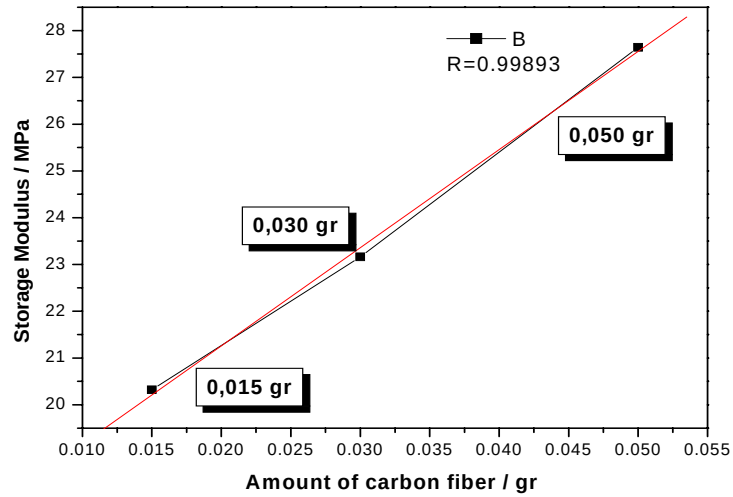


Figure 3.48: Storage modulus of samples with PU-Epoxy + 0.015 - 0.030 - 0.050 g carbon fiber (T) at 30 °C.

The increment of storage modulus in respect to amount of CF shows nearly perfect linear increments with a regression coefficient was very close to one (Figure 3.48).

Due to presence of polyurethane in polyurethane-epoxy polymer system, storage moduli of samples were decreased. In generally, epoxy composites have higher modulus values. It was a result of polyurethane particles inside epoxy crosslinking structure. These details can be followed with SEM micrographs (Figure 3.49, 3.50).

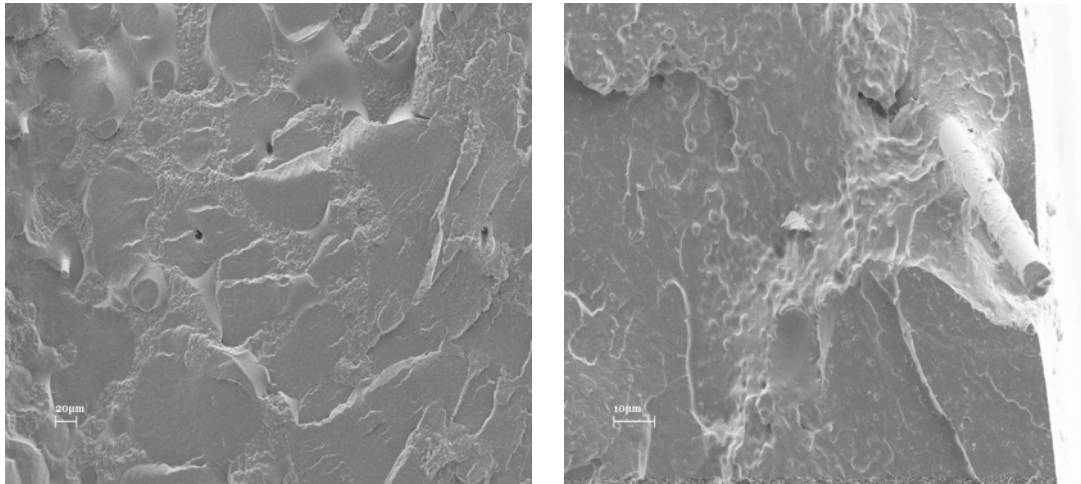


Figure 3.49: SEM micrographs of PU-[Epoxy/Hard:(5/4)] with 0.015 g carbon fiber

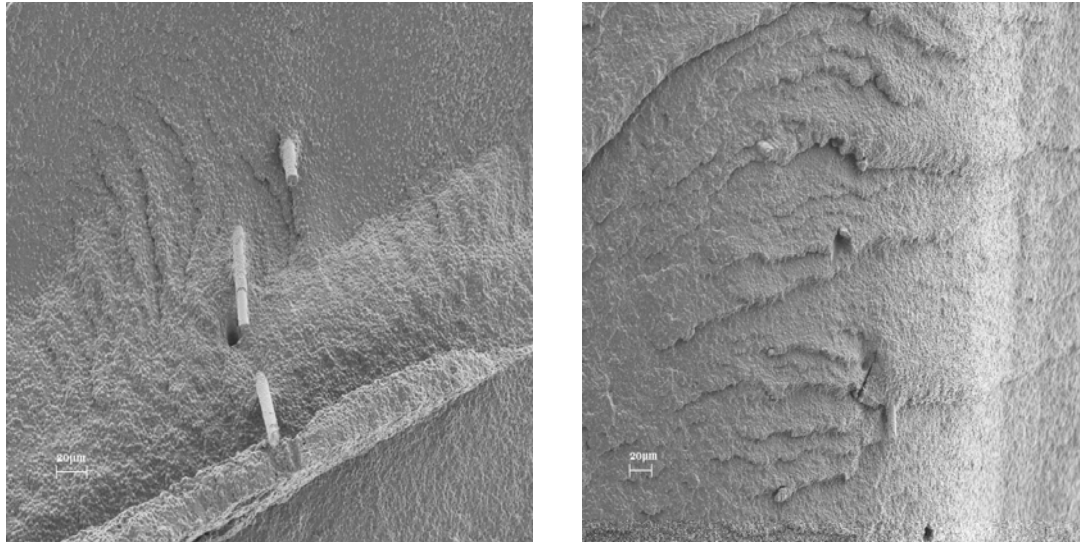


Figure 3.50: SEM micrographs of PU-[Epoxy/Hard:(5/4)] with 0.030 g carbon fiber

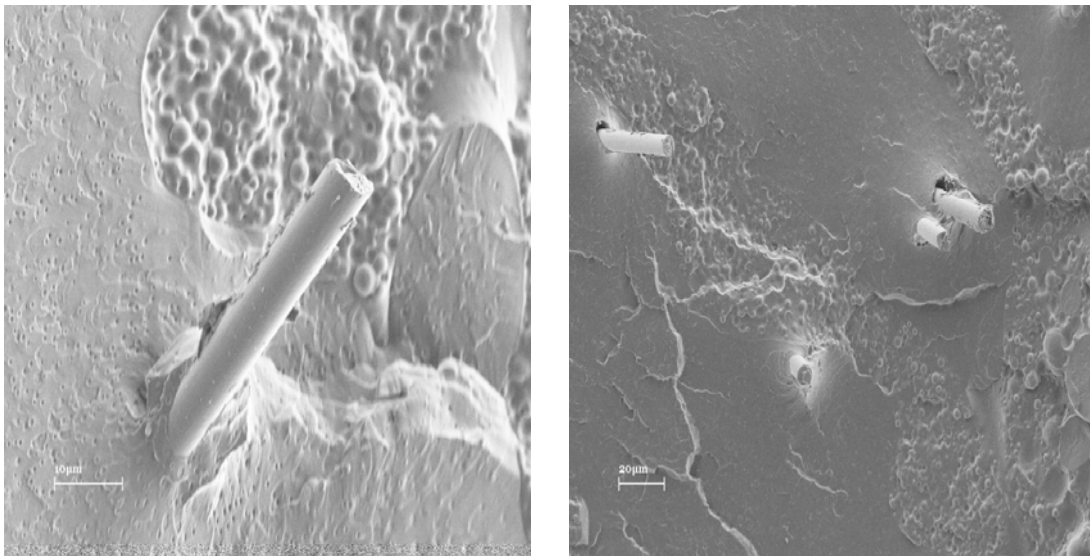


Figure 3.51: SEM micrographs of PU-[Epoxy/Hard:(5/4)] with 0.050 g carbon fiber

Because of increasing amount of polyurethane, there was some bubbles area on polymer system surface (Figure 3.51).

3.4.2. Stress Strain Curves of Reinforced Polyurethane-Epoxy Polymer Systems which include 0.015 g, 0.030 g and 0.050 g Chopped Carbon Fiber

In this section, the results of stress strain curves for reinforced PU-Epoxy polymer systems which has carbon fiber content of 0.015 g, 0.030 g and 0.050 g was investigated. The experiments were performed after five days.

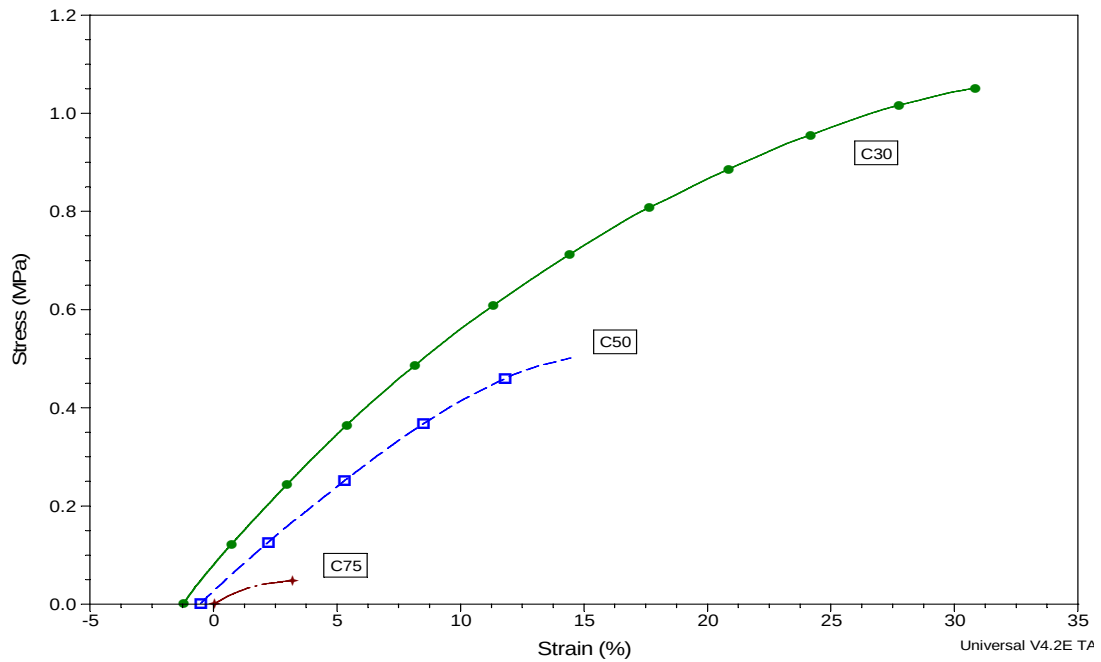


Figure 3.52: Stress strain curve of PU-Epoxy + 0.015 gr carbon fiber at 30 °C, 50 °C and 75 °C., after 5 days.

In Figure 3.52, it was shown that stress strain curve of PU-Epoxy + 0.015 gr carbon fiber at different temperatures, after 5 days.

The young modulus of samples affected with increasing temperature, so higher the temperature decreased the value of young modulus. In addition, maximum stress and strain values were changed in respect to temperature. The result of composite at 30 °C has the highest stress and strain value; in addition to this it has exhibited the biggest area under the stress strain curve. So, its toughness or impact resistance has the maximum.

For Figure 3.53, it can be seen that sample with in minimum temperature has the highest value of young modulus; similarly sample at higher temperature has lower value of young modulus and a relation between temperature and toughness was observed.

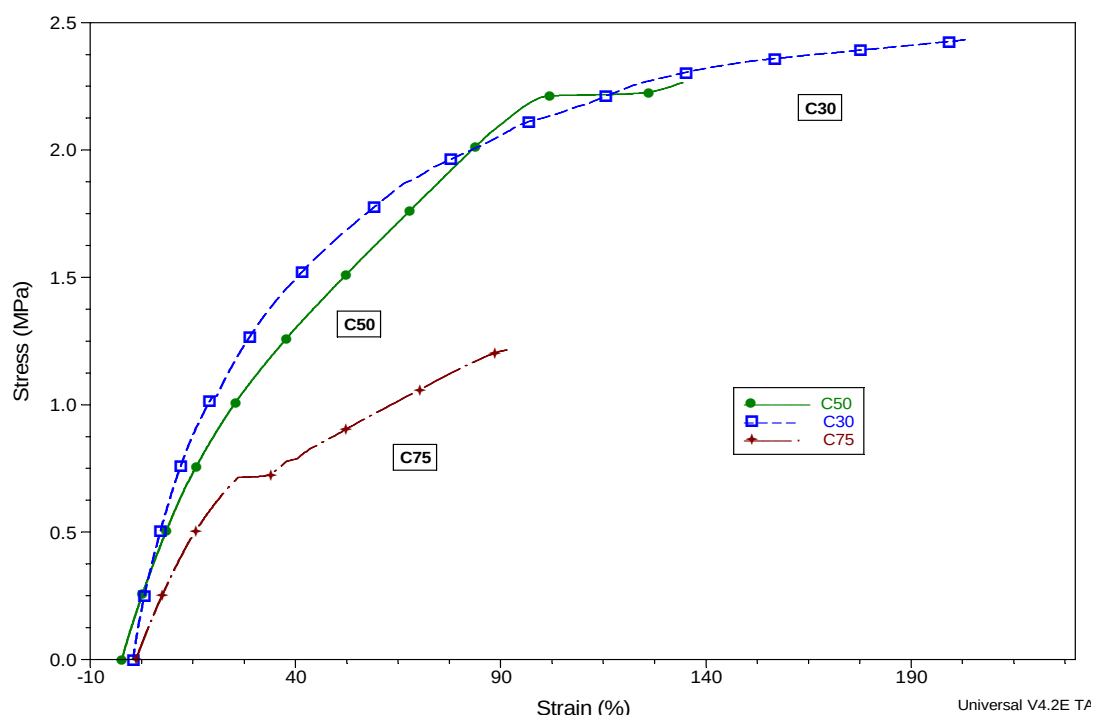


Figure 3.53: Stress strain curve of 10 g PU-Epoxy + 0.015 gr carbon fiber at 30 °C, 50 °C and 75 °C., after 5 days.

3.4.3. Hardener effect of Epoxy on Reinforced Polyurethane-Epoxy Polymer Systems which include Chopped Carbon Fibers

In this part, hardener effect on polymer systems which content polyurethane, epoxy and carbon fiber was investigated. Dynamic mechanical analysis of polymer systems was performed by DMA. Storage modulus, loss modulus and tan delta values were determined.

Stress strain curve of polymer systems was studied at different temperatures, and calculated young modulus of values. There were the results of toughness (impact resistance) for polymer systems.

T_g values of polymer systems which contain polyurethane, epoxy and carbon fiber were determined with the results of DMA.

Morphology of composites were studied and tried by SEM micrographs.

The experiments were performed after five days.

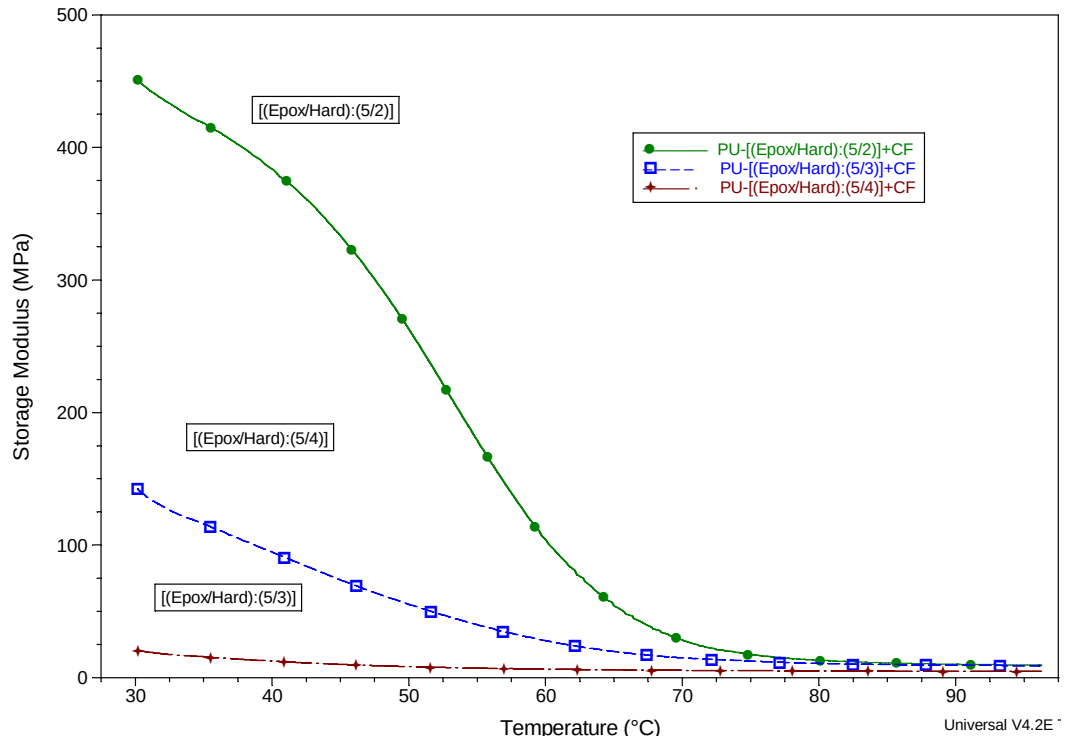


Figure 3.54: Effect of hardener on polymer systems which include polyurethane-epoxy with carbon fiber, after 5 days.

The amount of hardener effects storage modulus of polymer systems. While increasing amount of hardener resulted a decrease in values of storage modulus (Figure 3.54.) There was a big difference each of sample's storage modulus. Hardener of epoxy plays an important role in the polymeric composite system.

Table 3.26: Storage modulus of polymer systems which include different rate of hardener at 30°C.

Samples Content	Storage Modulus / MPa
5 g PU+ Epoxy(5 g A+ 2 g B) + 0.015 g CF	451.4
5 g PU+ Epoxy(5 g A+ 3 g B) + 0.015 g CF	142.4
5 g PU+ Epoxy(5 g A+ 4 g B) + 0.015 g CF	20.32

Table 3.27: T_g values of polymer systems which content different rate of hardener.

Contents of samples	T_g from DMA / °C (from tan delta)
PU-[(Epoxy/Hard):(5/2)] + Carbon Fiber	69.5
PU-[(Epoxy/Hard):(5/3)] + Carbon Fiber	64.0
PU-[(Epoxy/Hard):(5/4)] + Carbon Fiber	-

It is clearly observed that values of T_g was increased with decreasing hardener of epoxy in polymer systems (Table 3.27). It was also found that from dynamic mechanical analysis, tan delta curves of polymer systems. T_g values of polymer systems was obtained from Figure 3.55. Also, there was a relation with storage modulus and T_g values.

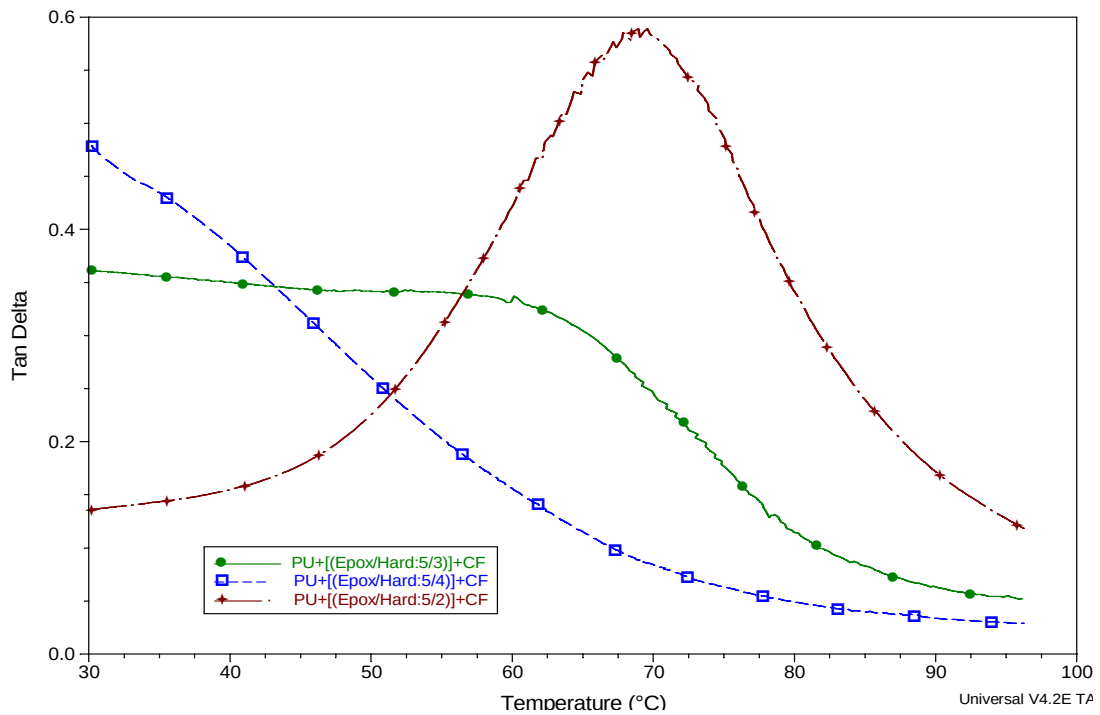


Figure 3.55: Effect of hardener on polymer systems which include polyurethane-epoxy with carbon fiber, after 5 days.

The stress strain curves of polymer systems which have amount of variety hardener were studied. The mechanical behaviors of polymer systems at different temperatures were investigated. Also, properties of toughness and young modulus were studied.

In Figure 3.56, while temperature increasing, young modulus of polymer systems decreased. It was observed that young modulus of PU-Epoxy polymer systems dramatically decreased at higher temperatures.

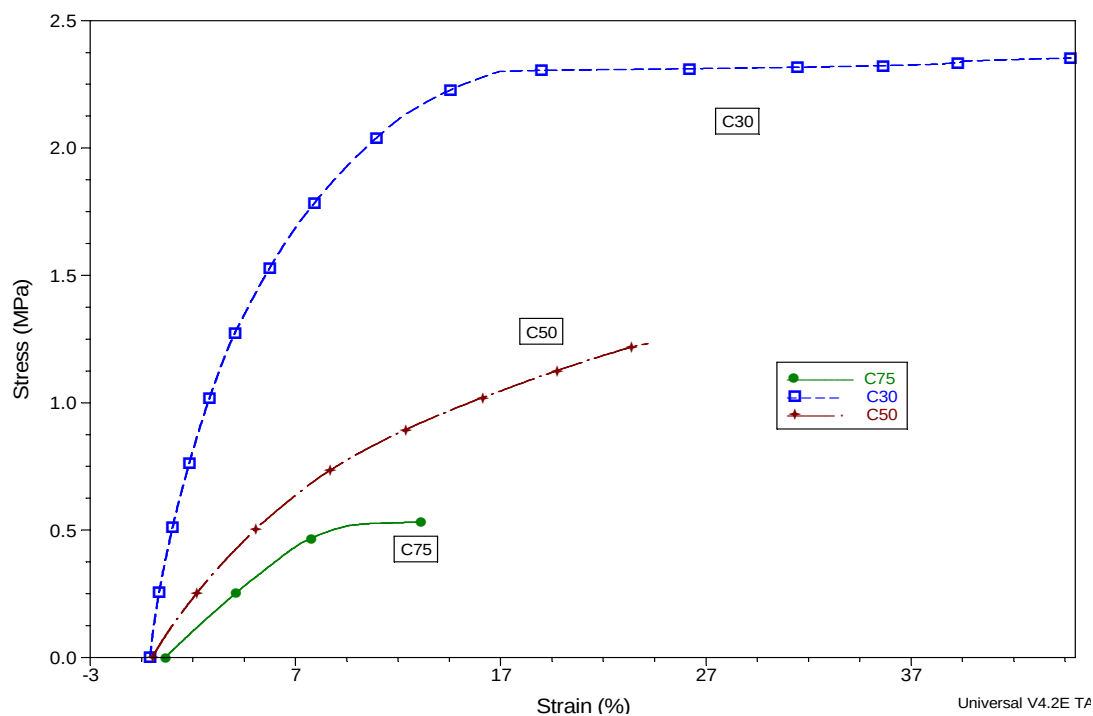


Figure 3.56: Stress strain curve of 5 g PU + Epoxy(5 g A + 2 g B)+0.015 g CF composite at different temperatures, after 5 days.

Maximum stress value of polymer system was decreased in respect to temperature. Polymer system at 30 °C has the highest strain value, also stress value. Its toughness has the biggest magnitude of under the curves at different temperatures.

Table 3.28: Young modulus of polymer systems at different temperatures (Hardener effect).

Content of Specimens	Young Modulus / MPa		
	30 °C	50 °C	75 °C
5 g PU+ Epoxy(5 g A+ 2 g B) + 0.015 g CF	25.14	10.25	6.893
5 g PU+ Epoxy(5 g A+ 3 g B) + 0.015 g CF	12.85	6.382	5.772
5 g PU+ Epoxy(5 g A+ 4 g B) + 0.015 g CF	5.123	4.360	2.365

SEM micrographs of all three specimens were taken for the investigation of surface morphology.

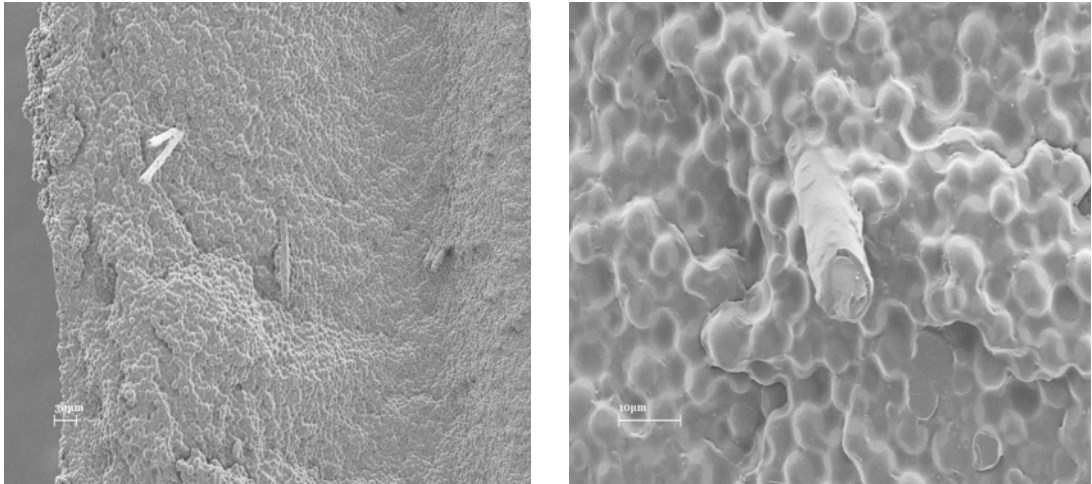


Figure 3.57: SEM micrographs of PU-[Epoxy/Hard:(5/2)] with 0.015 g carbon fiber

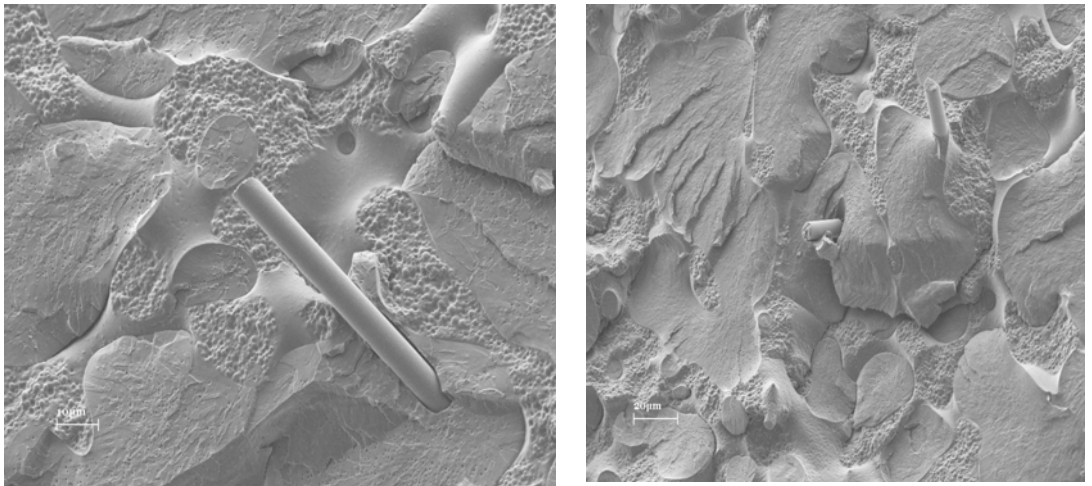


Figure 3.58: SEM micrographs of PU-[Epoxy/Hard:(5/3)] with 0.015 g carbon fiber

4. CONCLUSION

The aim of this thesis is to investigate the dynamic mechanical properties of reinforced carbon fiber composites which contain polyurethane and epoxy. Carbon fiber was used reinforcement agent in chopped and bundle form.

Two of procedures were applied to prepare composites or polymer systems. Firstly, curing method was used for epoxy composites and polymer systems. Curing temperature was 100 °C. The other method was “casting”, which is carried out at room temperature. This method was used to prepare polyurethane composites.

Thesis has presented in three sections. In the first part, epoxy has been reinforced with carbon fiber. Two kinds of carbon fibers were used for epoxy composites. In the second part, both polyurethane and epoxy has been reinforced with chopped carbon fiber. Here, the amount of carbon fiber and hardener were changed. In the last step, polyurethane and carbon fiber composite was presented.

An important part of this study was preparation of reinforced epoxy composites with carbon fiber. In the first part, chopped and bundle of carbon fiber were used to prepare epoxy composites. Two types of carbon fibers were used in the experiments, and mechanical properties of epoxy composites by using dynamic mechanical analysis were studied. Storage modulus, loss modulus, tan delta and complex modulus of epoxy-carbon fiber composites were investigated at different temperatures. The stress-strain relationship was studied for five and fifth day left day samples. There were significant developments obtained on modulus values after fifth days. In this part, rate of hardener in epoxy composite was changed, at the same time the amount of carbon fiber was changed, too. It was shown that the rate of hardener and amount of carbon fiber in epoxy-carbon fiber composites has played an important role in a final properties of composite. While increasing amount of carbon fiber in an epoxy composite, it affected the mechanical properties. In addition, young modulus, toughness, maximum stress and strain were developed with carbon fiber. On the other hand, investigation of surface morphology was studied for some specimens. The highest value of storage modulus belonged to [(epox/hard:10/5)] + % 0.5 g carbon fiber, 2427 MPa; after five days.

The second part was related to polyurethane-epoxy-carbon fiber polymer systems. The experiments were performed in variety of amount of carbon fiber and hardener in polymer systems. Besides, the amount of polyurethane was increased in polymer systems. The modulus values were obtained for all conditions of structure of polymer system. In this section, stress strain curves were taken at different temperatures, 30 °C, 50 °C and 75 °C. Increasing applied temperature resulted a decrease in young modulus and toughness of polymer systems which contain polyurethane, epoxy and carbon fiber. Here, all experiments were done after five days. The amount of carbon fiber was well proportional with the resulting modulus of polymer system. Hardener of epoxy was an important factor for mechanical behavior of polymer systems. The highest value of storage modulus belonged to polymer system of 5 g polyurethane + [(epox/hard:5/2)] + 0.015 g carbon fiber, 451.4 MPa; after five days. The highest value of young modulus belonged to 5 g polyurethane + [(epox/hard:5/2)] + 0.015 g carbon fiber, 25.14 MPa, after five days. Additionally, studies on surface morphology of polymer systems by using SEM micrographs were performed.

In the last part, polyurethane composites were also prepared with carbon fiber. Bundle of carbon fiber was used for polyurethane composites. Every sample was left at room temperature for one day. Here, all measurements were done after five days. These results indicated that using bundle of carbon fiber was resulted in significant increase for modulus of polyurethane composites. Polyurethane composite which has three bundles of carbon fiber has the highest storage modulus value for Torayca and Metyx carbon fiber. Polyurethane composite which has three bundles of carbon fiber (T) showed a storage modulus 880 times bigger than neat PU composite as well. Carbon fiber improved the mechanical properties of polyurethane-carbon fiber composites.

REFERENCES

- [1] **Park, J.H. and Jana, S.C.**, 2003. The Relationship Between Nano- and Micro-Structures and Mechanical Properties in PMMA-Epoxy-Nanoclay Composites, *Polymer*, **44**, 2091-2100.
- [2] **Wu, C.L., Zhang, M.Q., Rong, M.Z. and Friedrich, K.**, 2002. Tensile Performance Improvement of Low Nanoparticles Filled-Polypropylene Composites, *Composites Science and Technology*, **62**, 1327-1340.
- [3] **Jordan, J., Sharaf, M., Jacob, K., Tannenbaum, R. and Jasiuk, I.**, Experimental Trends in Polymer nanocomposites - A Review, *Georgia Institute of Technology*, p. 1-29.
- [4] **Gersappe, D.**, 2002. Molecular Mechanisms of Failure in Polymer Nanocomposites, *Physical Review Letters*, **89**(5),1-4.
- [5] **Kontou, E. and Kallimanis, A.**, 2006. Thermo-visco-plastic behaviour of fibre-reinforced polymer composites, *Composites Science and Technology*, **66**, 588–1596.
- [6] **Al-Haik, M.S., Garmestani H and Savran A.**, 2004. Explicit and implicit viscoplastic models for polymeric composites, *Int. J. Plasticity*, **20**, 1875–907.
- [7] **Megnis, M. and Varna, J.**, 2003. Micromechanics based modeling of nonlinear viscoplastic response of unidirectional composite, *Compos. Sci. Technol.*, **63**, 19–31.
- [8] **Borda, J., Keki, S., Rathy, I., Bodnar, I. and Zsuga, M.**, Novel Polyurethane Elastomer Continuous Carbon Fiber Composites: Preparation and Characterization, *Journal of Applied Polymer Science*, **103**, 287-292.
- [9] **Fokes MJ.**, 1982. Short fibre reinforced thermoplastics, Wiley, New York.
- [10] **Rosato, DV.**, 1999. Injection molding handbook 2nd ed. Boston/ London: Kluwer Academic Publishers.

- [11] **International Conference**, 1987. Reinforced injection processing. London: Plastic and Rubber Institute (PRI).
- [12] **Lee, SC., Yang, DY., Ko, J. and Yoon, JR.**, 1997. Effect of compressibility on flow field and fiber orientation during the filling stage of injection molding. *J. Mater. Process Technol.*, **70**, 83–92.
- [13] **Sahin, Y.**, 2000. Introduction of composite materials. Ankara: Gazi Book Publishing.
- [14] **Karaagac, R., Ozdemir, A. and Guldaz, A.**, 2002. An experimental investigation of the weld line on the plastic products molded. Institute of Science and Technology, Gazi University, **15**, 717–26.
- [15] **Ghosh, T., Grmela, M. and Carreau, P.J.**, 1995. Rheology of filled thermoplastics, *Polymer Composite*, **16**(2), 144–153.
- [16] **Clegg, DW. and Collyer, AA.**, 1986. Mechanical properties of reinforced thermoplastics. New York: AMI House.
- [17] **Titow, WV. and Lanham, B.J.**, 1974. Reinforced thermoplastics. London: Elsevier Applied Science Publishers.
- [18] **International Conference**, 1985. Short fibre reinforcement thermoplastics. London: Plastics and Rubber Institute (PRI).
- [19] **Yu, Z.**, 1994. Prediction of mechanical properties of short Kevlar fiber – Nylon 66, *Polymer Composites*, **16**, 64-73.
- [20] **Ozdemir, E.**, 2001. The experimental investigation of the physical properties of polypropilen (PP) and Nylon 6 (PA6) that unreinforced and fibre reinforced. *M.Sc. Thesis*, Institute of Science and Technology, Gazi University. Ankara.
- [21] **Shah, V.**, 1984. Handbook of plastics testing technology. Munich: Hanser Publishers.
- [22] **Botelho, E. C., Pardini, L.C. and Rezende, M.C.**, 2006. Hygrothermal effects on the shear properties of carbon fiber/epoxy composites, *J Mater Sci*, **41**, 7111–7118.

- [23] **Evora, V.M.F. and Shukla, A.,** 2003. Fabrication, Characterization, and Dynamic Behavior of Polyester/TiO₂ Nanocomposites, *Mater. Sci. Eng. A*, **361**, 358–366.
- [24] **Iwahori, Y., Ishiwata, S., Sumizawa, T. and Ishikawa, T.,** 2005. Mechanical properties improvements in two-phase and three-phase composites using carbon nano-fiber dispersed resin, *Compos. A*, **36**, 1430-1439.
- [25] **Zhang, M.Q., Rong, M.Z. and Friedrich, K.,** 2005. Application of non-layered nanoparticles in polymer modification, in: K. Friedrich, S. Fakirov, Z. Zhang (Eds.), *Polymer Composites: From Nano- to Macro-scale*, Springer, New York, pp. 25–44.
- [26] **Saliba, C.C., Orefice, R.L., Carneiro, J.R.G., Duarte, A.K., Schneider, W.T. and Fernandes, M.R.F.,** 2005. Effect of the incorporation of a novel natural inorganic short fiber on the properties of polyurethane composites, *Polymer Testing*, **24**, 819–824
- [27] **Chowdhury, F. H., Hosur, M. V. and Jeelani, S.,** 2007. Investigations on the thermal and flexural properties of plain weave carbon/epoxy-nanoclay composites by hand-layup technique, *J. Mater. Sci.*, **42**, 2690–2700.
- [28] **Zhou, Y., Pervin, F., Rangari, K. and Jeelani, S.,** 2007. Influence of montmorillonite clay on the thermal and mechanical properties of conventional carbon fiber reinforced composites, *Journal of Materials Processing Technology*, **191**, 347–351.
- [29] **Xu, Z., Huang, Y., Zhang, C. and Chen, G.,** 2007. Influence of rare earth treatment on interfacial properties of carbon fiber/epoxy composites, *Materials Science and Engineering A*, **444**, 70–177.
- [30] **Kim, JK. and Mai, Y-W.,** 1991. High strength, high fracture toughness fibre, composites with interface control—a review, *Compos Sci Technol.*, **41**, 333–378.
- [31] **Xu, Z., Pervin, F., Lewis, L. and Jeelani, S.,** 2007. Experimental study on the thermal and mechanical properties of multi-walled carbon nanotube-reinforced epoxy, *Materials Science and Engineering A*, **452–453**, 657–664.

- [32] **Lai, Y.H., Kuo, M.C., Huang, J.C. and Chen, M.,** 2007. On the PEEK composites reinforced by surface-modified nano-silica, *Materials Science and Engineering A*, **458**, 158–169.
- [33] **Mascia L.,** 1974. The role of additives in plastics. Interscience Publisher.
- [34] **Mader, E., Gao, S. and Plonka, R.,** 2007. Static and dynamic properties of single and multi-fiber/epoxy composites modified by sizings, *Composites Science and Technology*, **67**, 1105–1115.
- [35] **Tarantılı, P.A., and Andreodopoulos, A.G.,** Mechanical Properties of Epoxies Reinforced with Chloride-Treated Aramid Fibers, *Journal of Applied Polymer Science*, **65**(2): 267-276.
- [36] **Akbar, AK. and Maib, YW.,** 2002. Characterisation of fibre/matrix interfacial degradation under cyclic fatigue loading using dynamic mechanical analysis, *Composites: Part A*, **33**, 1585–1592.
- [37] **Li, JX., Wu, J. and Chan, CM.,** 2000. Thermoplastic nanocomposites, *Polymer*, **41**, 6935–6937.
- [38] **Varga, J. J.,** 1992. Supramolecular structure of isotactic polypropylene, *Material Sci.*, **27**(10), 2557-2579.
- [39] **Costa, H.M., Ramos, V.D. and Marisa, C.G.,** 2006. Rocha Analysis of thermal properties and impact strength of PP/SRT, PP/EPDM and PP/SRT/EPDM mixtures in single screw extruder, *Polymer Testing*, **25**, 498–503.
- [40] **Modesti, M., Lorenzetti, A., Bon, D. and Besco, S.,** 2006. Thermal behaviour of compatibilised polypropylene nanocomposite: Effect of processing conditions, *Polymer Degradation and Stability*, **91**, 672-680.
- [41] **Costa, HM., Ramos, VD. and Oliveira, MG.,** 2007. Degradation of polypropylene (PP) during multiple extrusions: Thermal analysis, mechanical properties and analysis of variance, *Polymer Testing*, **26**, 676–684.
- [42] **Wal, AVD., Mulder, J.J., Oderkerk, J. and Gaymans, R.J.,** 1998. Polypropylene-rubber blends: 1. The effect of the matrix properties on the impact behaviour, *Polymer*, **39**, 6781-6787.

- [43] **Hines, P. J., Tsuib, S.-W., Coatesb, P. D., Warda, I. M. and Ducketta, R. A.,** 1997. Measuring the development of fibre orientation during the melt extrusion of short glass fiber reinforced polypropylene, *Composites Part A*, **28A**, 949-958.
- [44] **Fu, S.Y., Lauke, B., Mader, E., Hu, X. and Yue, C.Y.,** 1999. Fracture resistance of short-glass-fiber-reinforced and short-carbon-fiber-reinforced polypropylene under Charpy impact load and its dependence on processing, *Journal of Materials Processing Technology*, **89-90**, 501-507.
- [45] **Gullu, A., Ozdemir, A. and Ozdemir, E.,** 2006. Experimental investigation of the effect of glass fibres on the mechanical properties of polypropylene (PP) and polyamide 6 (PA6) plastics, *Materials and Design*, **27**, 316–323.
- [47] **Turach, H.,** 2000. Manufacturing of injection molds. Istanbul: PAGEV Publishing.
- [48] **Fakirov, S., Bhattacharyya, D. and Shields, R.J.,** 2007. Nanofibril reinforced composites from polymer blends, *Colloids and Surfaces A: Physicochem. Eng. Aspects* xxx (2007) xxx–xxx; Received 20 October 2006; accepted 8 May 2007.
- [49] **Zilg, C., Mulhaupt, R. and Finter, J.,** 1999. Morphology and toughness/stiffness balance of nanocomposites based upon anhydride-cured epoxy resins and layered silicates, *Macromol. Chem. Phys.*, **200**, 661-670.
- [50] **Zilg, C., Thomann, R., Finter, J. and Mulhaupt, R.,** 2000. The influence of silicate modification and compatibilizers on mechanical properties and morphology of anhydride-cured epoxy nanocomposites, *Macromol. Mater. Eng.*, **280-281**, 41-46.
- [51] **Lan, T. and Pinnavaia, T.J.,** 1994. Clay-reinforced epoxy composites, *Chem. Mater.*, **6**, 2216-2219.
- [52] **Wei ZG, Tang CY and Lee WB.,** 1997. Design and fabrication of intelligent composites based on shape memory alloys, *J. Mater. Process Technol.*, **69**, 68–74.

- [53] **Ma N and Song G.,** 2002. Control of memory alloy actuator using pulse width (PW) modulation. Smart structures and materials 2002: modeling, signal processing, and control. In: Rao, *Journal of Intelligent Material Systems and Structures*, **14**(1), 15-22.
- [54] **Edited by B. Ellis,** 1993. Chemistry and technology of Epoxy Resins, Blackie Academic&Professional, p. 1- 4.
- [55] **Edited by B. Ellis,** 1993. Chemistry and technology of Epoxy Resins, Blackie Academic&Professional, p. 203, 252-253.
- [56] **Gonçalez, V., Barcia, FL. and Soares and BG.,** 2006. Composite Materials Based on Modified Epoxy Resin and Carbon Fiber, *J. Braz. Chem. Soc.*, **17**(6), 1117-1123.
- [57] **Sue H-J, Puckett PM, Bertram JL, Walker LL,** 2000. The network structure of epoxy systems and its relationship to toughness and toughenability. In: Pearson, Sue, Yee, editors. Toughening of plastics: Advances in modelling and experiments. ACS Symposium Series 2000. p. 759.
- [58] **Maity, T., Samanta, BC., Dalai, S. and Banthia, AK.,** 2007. Curing study of epoxy resin by new aromatic amine functional curing agents along with mechanical and thermal evaluation;; *Materials Science and Engineering A*, **464**, 38–46.
- [59] **Potter, W.G.,** 1970. Epoxide Resins, Springer-Verlag, New York.
- [60] **Lee, H. and Neville, K.,** 1976. Handbook of Epoxy Resin, McGraw-Hill Book Co.,New York,
- [61] **Wetzel, B., Rosso,P., Hauptert, F. and Friedrich, K.,** 2006. Epoxy nanocomposites–fracture and toughening mechanisms, *Engineering Fracture Mechanics*, **73**, 2375–2398.
- [62] **Zhou, Y., Pervin, F., Rangari, V.K. and Jeelani, S.,** 2006. Fabrication and evaluation of carbon nano fiber filled carbon/epoxy composite, *Materials Science and Engineering A*, **426**, 221–228.
- [63] **Menard, K.P.,** 1999. Dynamic Mechanical Analysis: A Practical Introduction, CRC Press LLC.

- [64] **Donnet, J.B.**, 2003. Nano and microcomposites of polymers elastomers and their reinforcement, *Compos. Sci. Technol.*, **63**, 1085–1088.
- [65] **Chen, W., Tao, X. and Liu, Y.**, 2006. Carbon nanotube-reinforced polyurethane composite fibers, *Composites Science and Technology*, **66**, 3029–3034.
- [66] **Borda, J., Teslery, B., Zsuga, M. and Muanyages Gumi**, 1999. Biologically Degradable Fibre-resin Urethane Composites from Wheat Straw, **1**, 28.
- [67] **Xin, L., Xu, G., Hofstra, P. and Bajcar, R. C.**, 1998. Moisture-absorption, dielectric relaxation, and thermal conductivity studies of polymer composites, *J. Polym. Sci. Part B: Polym Phys.*, **36**, 2259-2265.
- [68] **Chou, P. and Ding, D.**, 2000. Characterization of Moisture Absorption and Its Influence on Composite Structures, *J Thermoplast Compos Mat*, **13**, 207-225.
- [69] **Correa, R. A., Nunes, R. C. R. and Filho W. Z. F.**, 1996. Effect of Injections on the Orientation of Short Fibre Composites. An Optical Microscopic Analysis, *Polym. Test.* , **15**, 467-475.
- [70] **Chen, C. H., Ma and C. C. M.**, 1992. Pultruded Fiber Reinforced Blocked Polyurethane (PU) Composites. I. Processability and Mechanical Properties, *J. Appl. Polym. Sci.*, **46**, 937-947.
- [71] **Poynting, J. H.**, 1909. On pressure perpendicular to the shear planes in finite pure shears, and on the lengthening of loaded wires when twisted, *Proceedings of the Royal Society, Series A*, **82**, 546.
- [72] **Dealy, J.**, 1992. *Rheometers for Molten Plastics*, Van Nostrand Reinhold, New York, p136–137 and 234–236.
- [73] **Ferry, J.**, 1980. *Viscoelastic Properties of Polymers*, 3rd ed., Wiley, New York.
- [74] **Gilham, J. and Enns, J.**, 1994. On the cure properties of thermosetting polymers using torsional braid analysis, *Trends in Polymer Science*, **2**(12), 406-419.

- [75] **Murayama, T.**, 1977. *Dynamic Mechanical Analysis of Polymeric Materials*, Elsevier, New York. (This book is the ultimate reference on the Rheovibron.)
- [76] **Read, B. E. and Brown, G. D.**, 1978. *The Determination of the Dynamic Properties of Polymers and Composites*, Wiley, New York.
- [77] **Ray D, Sarkar BK, Das S and Rana AK.**, 2002. Dynamic mechanical and thermal analysis of vinylester-resin-matrix composites reinforced with untreated jute fibres, *Compos Sci Technol*, **62**, 911–917.
- [78] **Saha AK, Das S, Bata D and Mitra BC.**, 1999. Study of jute fiber reinforced polyester composites by dynamic mechanical analysis. *J Appl Polym Sci*, **71**, 1505–1513.
- [79] **Sepe MP.**, 1998. *Dynamic mechanical analysis*. Norwich, New York, USA: Plastics Design Library.
- [80] **Manikandan-Nair KC, Thomas S and Groeninckx G.**, 2001. Thermal and dynamic mechanical analysis of polystyrene composites reinforced with short sisal fibres, *Compos. Sci. Technol.*, **61**, 2519–2529.

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