

**UNIVERSITY OF TURKISH AERNAUTICAL ASSOCIATION
INSTITUTE OF SCIENCE AND TECHNOLOGY**

**VISCOELASTIC BEHAVIOUR OF THERMOSETS - THERMOPLASTIC
POLYMER COMPOSITE**

MASTER THESIS

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Institute of Science and Technology

Mechanical and Aeronautical Engineering Department

Master Thesis Program

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**IN PARTIAL FULFILLMENT OF THE REQUIREMENT FOR THE
DEGREE OF MASTER OF SCIENCE IN MECHANICAL AND
AERONAUTICAL ENGINEERING**

**Supervisor: Assist. Prof. Dr. Munir ElFarra
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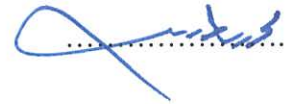
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AYAD A. MUHMMED

27.01.2017

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ABSTRACT

VISCOELASTIC BEHAVIOUR OF THERMOSETS - THERMOPLASTIC POLYMER COMPOSITE

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Thesis Supervisor: Assist. Prof. Dr. Munir Elfarrar

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Polyester has gained a considerable importance as a thermoset polymer in structural, marine and electrical applications. To expand their usage and overcome their brittleness, many of the polymers were blended with it. An example is the addition of polystyrene which helps in increasing the impact resistance of polyester; but on the other hand, it reduces the tensile strength, hardness, elastic modulus and creep resistance, which leads to use another material to compensate the reduction in these properties. The aim of the current study is to modify the properties of Unsaturated Polyester (UP) by adding Polystyrene (PS) in different weight percentages; 5%, 7.5%, 10%, 12.5%, 15% and 20% to prepare binary polymer blends. With weight percentage 7.5% of the polystyrene, the results show that the tensile strength decreases (42%), while the addition of fiber glass has helped in compensating the reduction in tensile strength. In other words, the rate of increase in tensile strength of the blend (7.5 wt. %) with two layers of fiber compared with polyester is 26%. This is correct also for the other mechanical properties, like toughness which increase with two layers of fibers in a percentage of 55% more than toughness compared with of polyester alone. The Scanning Electron Microscopy (SEM) examination showed that the polystyrene particles have uniform distribution through the Unsaturated Polyester; this distribution may be responsible for the lower

crack growth. Also reveals that these composites materials do not fail by fiber fracture, but either by matrix failure or interfacial de-bonding. The creep test was conducted under different applied stresses (5, 6, and 7 MPa) at room temperature. Burger model was used for modeling and analyzing the viscoelastic behavior of the materials. The creep results showed a good concordance with the Burger's model. It was found that the composite materials reinforced with two layers of fiber gave better results than the composites reinforced with one layer. The blend (7.5 wt. %) with two layers of fiber glass was provided good viscoelastic properties. It was also found that polystyrene additives led to greater creep and creep recovery. This result confirmed that polystyrene increases chain mobility. In addition, the creep resistance of blend reinforced with two layers of fiber was significantly enhanced.

Keywords: Unsaturated Polyester, Polystyrene, Fiber, Composites, Mechanical properties.

ÖZET

TERMOSETLERİN - TERMOPLASTİK POLİMER KOMPOZİT VİSKOELASTİK DAVRANIŞI

AL-MASHHADANI, AYAD A.

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Polyester yapı, deniz ve elektriksel uygulamalarda termoset polimer olarak büyük önem kazanmıştır. Kullanımını genişletmek ve kırılma direncini artırmaya yardımcı olan polisitiren ilavesi örnek olarak verilebilir. Ama diğer yandan çekme mukavemetini, sertliğini, elastik modülünü ve sürtünme direncini de azaltmaktadır. Bu durum, yapısal özelliklerin azalmasını telafi etmek için başka bir malzemenin kullanılmasına yol açar. Bu çalışmanın amacı, doymamış polyesterin (UP) özelliklerini, farklı ağırlık yüzdelerindeki % 5, % 7.5, % 10, % 12.5, % 15 ve % 20, polistiren (PS) ilave ederek değiştirmek ve ikili polimer karışımlarını hazırlamaktır. Polistirenin yüzde arttıkça gerilme mukavemetinin azaldığını gösterirken (%42), cam elyafın eklenmesi çekme mukavemetindeki azalmayı telafi etmeye yardımcı olmuştur. Diğer bir deyişle, iki katmandan polyesterle karşılaştırıldığında karışımın çekme mukavemetinde artış oranı (ağırlıkça %7,5) % 26'dır. Diğer bir deyişle iki tabaka cam elyafı karışımın çekme mukavemet artış oranı (ağırlıkça %7.5) polyester ile karşılaştırıldığında %26'dır. Tek başına polyester ile karşılaştırıldığında (ağırlıkça %55) daha fazla toklukta olan iki kat elyafı karışımındaki artan tokluk gibi diğer mekanik özellikler için de bu artış doğrudur. Taramalı Elektron Mikroskobu (SEM)

incelemesi, polistiren parçacıklarının doymamış polyester yoluyla düzgün dağılıma sahip olduğunu gösterdi; daha küçük parçacıkların bu dağılımı, daha düşük çatlak büyümesinden sorumlu olabilmektedir. Ayrıca, bu kompozit materyallerin elyaf kırığı nedeniyle değil, matris hatası veya ara yüzey bağlaşmasıyla koptuklarını ortaya koymaktadır. Kesilmiş cam matris ve elyaf tabakaları ile ağırlıkça % 7.5 oranında karıştırılarak takviye edilmiş numunelerin gösterdiği gibi, elyaflar yük-gerilim merkezi olarak davranış gösterir ve doymamış polyester karışımından daha yüksek darbe mukavemeti sergileyerek matris boyunca homojen olarak dağıtılır. Sürünme testi oda sıcaklığında (5, 6, ve 7 MPa) farklı uygulanan gerilmeler altında yürütülmüştür. Burger modeli, malzemelerin viskoelastik davranışını modellemek ve analiz etmek için kullanılmıştır. Sürünme sonuçları burger modeliyle iyi bir uyum gösterdi. İki kat elyaf takviyeli kompozit materyallerin bir kat ile takviye edilmiş kompozitlerden daha iyi sonuç verdiği bulunmuştur. Ağırlıkça % 7.5 iki tabakalı cam elyaf karışım ile iyi bir viskoelastik özellikler sağladı. Ayrıca, polistiren ilavesinin daha büyük sürünme ve sürünme iyileşmesine yol açtığı bulundu. Bu sonuç, polistirenin zincir hareketliliğini arttırdığını doğrulamıştır. Buna ek olarak, iki kat elyaf takviyeli karışımın sürünme direnci önemli ölçüde arttırılmıştır.

Anahtar Kelimeleri: Doygun Olmayan Polister, Polisterine, Fiber, Bileşik, Mekanik Özellikleri

LIST OF ABBREVIATIONS

UP	: Unsaturated Polyester
PS	: Polystyrene
UV	: Radiation Ultraviolet
CGFM	: Chopped Glass- Fiber Mats
ASTM	: American Society for Testing and Materials
SEM	: Scanning Electron Microscope
SFRPCs	: Short Fiber Reinforced Polymer cComposites
T_g	: Glass Transition Temperature
V_f	: volume fraction
PVC	: Poly Vinyl Chloride
MEKP	: Methyl Ethyl Keton Peroxide
MCR	: Minimum Creep Rate
PMMA	: Poly Methyl Methacrylate
PET	: Polyethylene Terephthalate
RT	: Room Temperature
TR	: Retardation Time

LIST OF ACRONYMS

D	:	Creep Compliance (1/MPa)
D(t)	:	Creep Compliance at Any Time (1/MPa)
E	:	Modulus of Elasticity (MPa)
E_t	:	Relaxation of Modulus (MPa)
G	:	Modulus of Rigidity (MPa)
q	:	Distributed load (N)
s	:	Transform Laplace Variable
P	:	Linear Deferential Operator
ε	:	Normal Strain
ε₀	:	Initial Strain
ε_t	:	Strain at Any Time
T	:	Temperature (°C)
min.	:	Time (sec.)
G_t	:	Modulus of Rigidity at Any Time (MPa)
σ	:	Normal Stress (MPa)
σ₀	:	Initial Stress (MPa)
σ_t	:	Stress at Any Time (MPa)
δ	:	Deformation (mm)
δ₀	:	Initial Deformation (mm)
δ_t	:	Deformation at any time (mm)
η	:	Viscosity of Material (Dashpot Constant) (N. sec/mm ²)

CHAPTER ONE

GENERAL INTRODUCTION

1.1 Introduction

Over the last 50 years, fiber-reinforced plastics have been used in many products, applications and industries. "Composite" refers to a combination of any individual materials [1]. Fiber-reinforced composites are very widely used, especially glass fibers, that are impregnated in a plastic resin matrix. These fiber-reinforced composites have desired properties (e.g. high compressive strength and specific tensile, tremendous strength-to-weight, corrosion-resistant, low coefficient of thermal expansion, design flexibility, good fatigue resistance and electrical conductivity) rendered them ideal to be used in a growing number of industries. Therefore they became the alternatives of conventional structural materials such as metals, steel and wood in a lot of industries. Ideal industry fields for composites are automotive industries, marine industries e.g. boats, ships, aircraft fabrication, ... etc [1, 2].

Although some plastics are relatively weak, but they are quite versatile and tough. Also glass fibers are strong, yet liable to be damaged. Those two components if combined together will result in a composite that is more ideal than either of them, and meets the final product requirements that are not provided by other materials, especially when using the suitable resin, fiber and manufacturing process. Polymers which are time dependent materials, nowadays play an important role in many industry applications, particularly in aerospace industry. This is due to their viscoelastic behavior [1-3], which affect the functionality of these materials after a while. When using time dependent materials for designing of structural elements it is essential to know how the mechanical properties of the materials might change throughout the useful life of a product. In polymers, the extent of change in mechanical properties depends on many factors, e.g. pressure, humidity, temperature and stress conditions applied on it, during its

manufacturing, and later during its application. The quality of polymeric products is defined by their functionality and durability (i.e., behavior over a long period of time).

During their use, polymeric products are frequently exposed to different loading in the form of force. Such loading might result in material creep, a change in geometry, which eventually might cause product failure [1, 4].

In 2005, Smith K. J. investigated the behavior of E-glass fiber-reinforced polymer (FRP) composite under certain conditions; load and temperature. This study included testing compression and creep of material coupons conducted at a constant stress level of 33% of ultimate strength and variable temperature levels; 23.3°C , 37.7°C and 54.4°C. Experimental results obtained were used to plot a curve reflecting the creep behavior of these sections [5].

More experiments were conducted to test the effects of changing temperature in order to better simulate service conditions. In 2007, Adnan H., [6] investigated the effect of temperature on Creep of unsaturated reinforced Polyester (UP) with continuous fiber E-glass type, the result showed that creep rates increase when the temperature increases and creep decreases with reinforcement E-glass fiber.

Also in 2011, M. K. Jawad, et. al, [7], studied the tensile strength of Unsaturated Polyester (UP) / Poly Vinyl Chloride (PVC) composites reinforced with glass fibers. Hand layup method was used to prepare sheets of UP/PVC blends in different weight percentages of PVC 10%, 20% and 30%. Results showed that the increase in values of tensile strength, yield strength and modulus of elasticity of UP/PVC blends with increasing the weight percentage of the PVC. UP/PVC blends reinforced with one layer of glass fiber detected good mechanical properties for the composite which contains 20% PVC. Also the results showed that composites reinforced with two layers of glass fiber randomly had increased in the values of tensile strength with increasing PVC percentage.

Salman. Ali, in 2012, [8], studied tensile and impact properties of a composite formed from polystyrene matrix reinforced by 50% palm natural fibers and 50% carbon fibers. The weight fractions of carbon fiber and palm fiber are 10, 20, 30, 40, 50, and 60% by weight (wt). To determine the tensile and impact properties of these composites,

tensile and impact tests were done, and the maximum impact strength was $175\text{Kg}/\text{m}^2$ and fiber weight fraction of 60 % , while $19\text{KJ}/\text{m}^2$ for virgin polystyrene material and a maximum tensile strength of 358MPa at a weight fraction of fibers 60 % , and 59Mpa for virgin polystyrene material.

In 2013, Dr. Oleiwi J. et. al, [9], investigated the tensile properties of unsaturated polyester reinforced with glass fiber in different volume fraction V_f , the reinforcement with coarse and fine woven fibers in different angles. When VF for fine woven fibers was 11%, and fibers angle $0^\circ/90^\circ$, the maximum value of the modulus of elasticity was 11.5 GPa and the maximum value of ultimate tensile strength was 240 MPa .

In 2015, Eftekhari and Fatemi, [10], studied the mechanical properties of short fiber reinforced polymer composites (SFRPCs) including polystyrene reinforced with short fibers at elevated temperatures. The study focuses on creep, tensile, thermal fatigue, creep-fatigue and thermo-mechanical fatigue interaction. Also environmental factors e.g. moisture and ageing at elevated temperatures are involved. This study compared between modeling works and the experimental works, and studied failure mechanisms.

In 2016, Kohl. J et al. [11], investigated the characterization and the effect of micro-indentation test methods and frictional behavior on the viscoelastic properties of fiberglass-reinforced polyester composite. Results showed that these glass fibers significantly caused an increase in both the viscoelastic indentation modulus parameters, E_0 and E_1 , of the composite. Also scratch tests were applied on these composites, the coefficient of friction for a diamond stylus in contact with a fiberglass-reinforced polyester composite was higher than in case of contact with a polyester. Also, it was noted that as the diamond stylus's sliding speed increased, for the composite, the coefficient of friction was decreasing.

The preceding researches enlarge our scope of studying the same field of work, This thesis discusses the influences of combining polystyrene (PS) and one type of fibers, chopped glass-fiber mats (CGFM) to the polyester, so as to obtain a matrix material with improved mechanical properties e.g. good toughness without compromising the other desired properties of the polyester resin, also studying the

influence of changing PS and fibers concentration on the UP mechanical properties, viscoelastic behavior, (creep resistance) to improve the viscoelastic behavior of the unsaturated polyester (UP) and polystyrene (PS) blend using Maxwell and Kelvin models and Burger's models. Molecular structural changes, and morphological analysis, all will be discussed and tried to correlate between them.

1.2 The Objective of the Thesis

The goal of this research is to:

- 1- Studying the effect of adding thermoplastic polymer (polystyrene PS) to thermoset (unsaturated polyester PU) on the mechanical properties (tensile, hardness and creep behaviour).
- 2- Assessing the impact of reinforcing the polyester/polystyrene blend with fibre glass.
- 3- Evaluating the viscoelastic behaviour of the polyester and the blend reinforced with E. glass fibre (chopped glass-fiber mats CGFM) under different stresses in room temperature.

CHAPTER TWO

THEORETICAL PART

2.1 Introduction

In this chapter, the study of viscoelastic behavior of composites materials is presented. The polymer and composite structure and types were shown. The mechanical tests that are used for evaluating the viscoelastic properties of composite also proposed as the importance of such types of materials used nowadays.

2.2 Polymers

2.2.1 Polymers Properties

Polymers are much more complex than ceramics and metals and they are low cost and easily addressed. Polymers can be addressed in many ways to produce very intricate parts or thin fibers. Polymers have module, lower strengths, lower temperature use limits than do metals and ceramics. Because of their predominantly covalent bonding, they are usually very resistant to chemicals than metals, and but prolonged exposure to some solvents and ultraviolet light can cause degradation of a polymer's properties. polymers are generally a good insulators of the electricity and heat. With varying degrees of strength, the polymers are very light [12]. Fig. 2.1 shows the types of polymers.

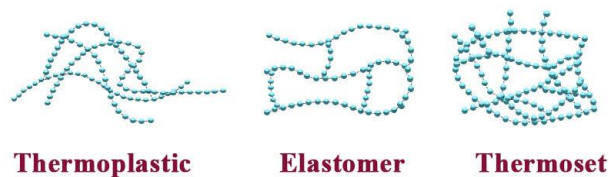


Figure 2.1: Types of Polymers [12].

2.2.2 Polymers Structure

The distinction in the behavior of polymers stems from their molecular structure, shape, weight, molecular size, and type of bond. The various chain configurations are shown in Fig. 2.2. The linear polymers consisting of a long chain of atoms with attached side groups as shown in Fig. 2.2a. Branched polymers have branches connected with to the main chain as shown in Fig. 2.2b. Branched can happen with linear, or crosslinked, or any other types of polymers. A crosslinked polymer has molecules of one chain confided with those of another as shown in Fig. 2.2c. It is simple to observe that crosslinking makes sliding of molecules past one any more difficult, resulting in rigid and strong polymers. Ladder polymers have two linear polymers linked in a regular manner Fig. 2.2d. Note, the type ladder polymers are more rigid than the type linear polymers [13, 14].

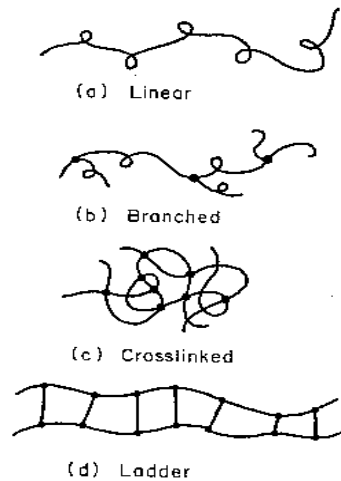


Figure 2.2: Different type of molecular chain configurations [13].

Another classification of polymers is based on the type of the repeated units. Fig. 2.3 when we have one type repeat unit for example, A-A forming the polymer chain, can we call it a Copolymers or homopolymer. Another side is polymer chain having two different monomers. Can we are distributing randomly along the chain, If the two different monomers for example, (B) and (A), then we have a random or regular

copolymer. However, along sequence of one monomer (A) is followed by along sequence of another type (B), we have a block copolymer. If we have a chain of one type of monomer (A) and monomer (B). We have a graft copolymer as shown in Fig. 2.3 describe some important chain formations in a planning manner [13, 15].

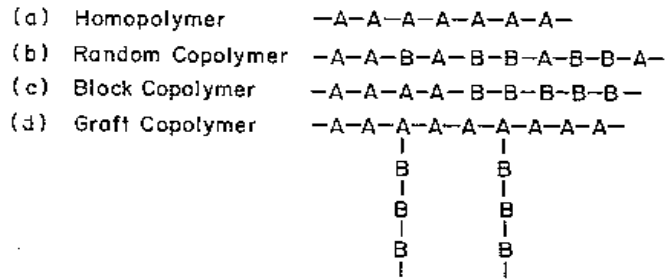


Figure 2.3: Classifications of polymers is on the type of the repeating unit [13].

2.3 Composites

A composite is a multiphase material that is unnaturally made, as opposed to that occur forms naturally. In addition, must be chemically dissimilar the constituent phases and separated by a distinct interface. But do not fit this definition with many ceramics and some metallic alloys. Because their multiphase are consisted as a consequence of natural phenomena. The engineer and scientists in designing composite material, have combined various polymers, ceramics and metals to produce a new generation of extraordinary materials. Most composite material has been formed to improve group of mechanical characteristics such as toughness, stiffness, strength and high temperature [16, 17].

Many composite materials are consisted of only two phases, one it is called matrix, which is constant, and besetment the other phase, which is termed the reinforcement phase. The properties of the constituent phases are a function of the properties of composite. The planner for the classification of composite material is shown in Fig. 2.4, [18, 19].

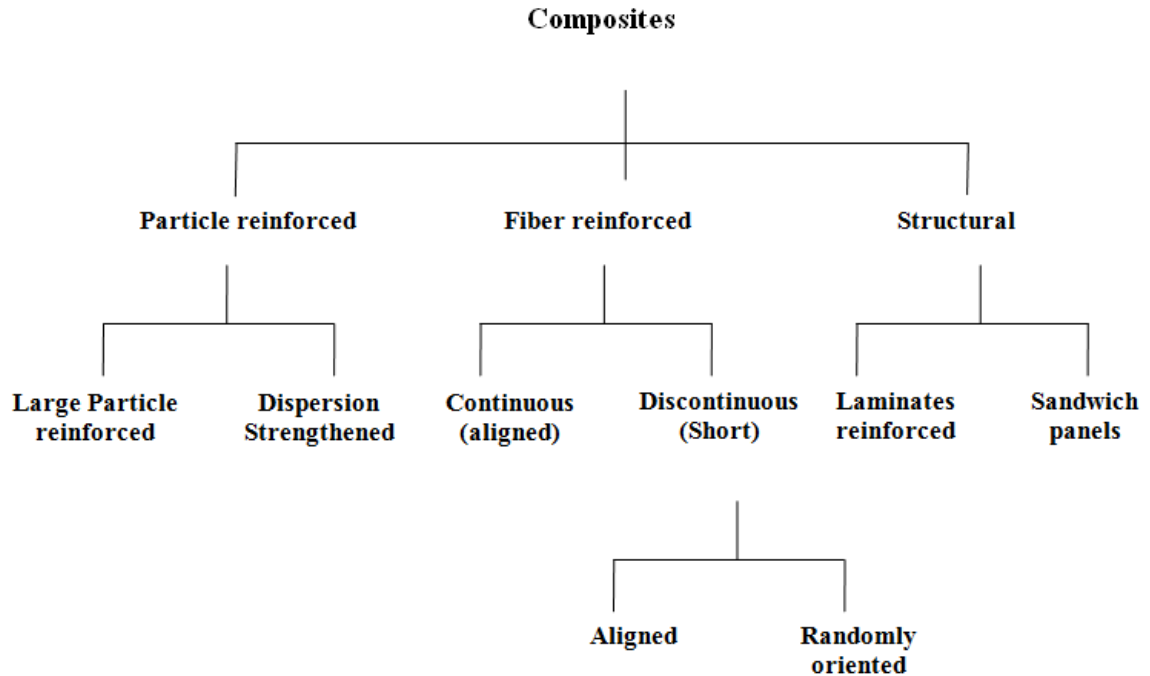


Figure 2.4: A classification scheme for the various composite types [19].

2. 4 Matrix Material

Matrix resins link glass-reinforcing fibers together, shielding them from effect and the environment. Resin properties prevail reinforced by the glass when glass is used as a intermittent reinforcement. Glass fiber properties, for example, strength prevail in persistently enhanced composites. Polymer matrix resins divided into two categories: thermoplastic and thermoset as above. The distinction is in their chemistry. Thermoset resin is chemically includes of molecular chains that crosslink during the cure reaction (catalyst, set off by heat, or both) and “set” into a final rigid form. At higher temperatures, molecular chains in thermoplastic resin are processed and stay on “plastic,” or capable of being reshaped and reheated [18, 19].

2.4.1 Unsaturated Polyester Materials

Polyester resins are an unsaturated, thermosetting resin produced by a chemical reaction between many polyhydric alcohols and organic acids, see Fig. 2.5.

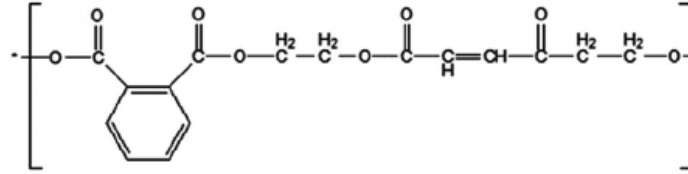


Figure 2.5: Chemical structure of unsaturated polyester resin [20].

The unsaturated polyester resins used in many casting applications are a viscous liquid which requires the catalysts addition and accelerators to complete the process of curing. Unsaturated polyester resin is a contact product which does not require pressure to cure and can be cured by a solid state or fluid. Although this product has many disadvantages when compared with other very often used composite resins, it still shows an attractive balance of low cost, ease of use also positive physical characteristics [20, 21].

2.4.2 Polystyrene Material

Polystyrene is amorphous thermoplastic, rigid, hard, and free of odor and taste. It is characterized by thermal stability, low specific gravity, ease of heat fabrication, excellent electrical and thermal properties for insulating purposes and low cost. It is solid at room heat, but it flows if warmed over the glass transition heat (for extrusion or molding), and when chill outside it becomes strong again. Pure polystyrene is a hard plastic, colorless, with limited flexibility. It can be made on various colors or transparent. The chemical structure of polystyrene is a long hydrocarbon chain with each other carbon linked to a phenyl set as shown in Fig. 2.6, [22, 23].

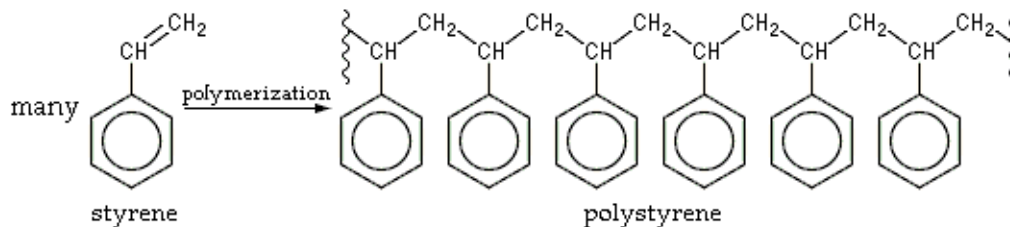


Figure 2.6: Chemical structure of Polystyrene resin [23].

Polystyrene contains hydrogen and carbon. Because polystyrene is aromatic hydrocarbon, it burns with a yellow flame with soot, as opposite to polymers with non-aromatic hydrocarbon such as polyethylene [24].

2.5 Polymer Blends

There is no doubt that the main reason for blending, compounding and reinforcing is economy. If a material can be developed at a lower cost with properties meeting manufacturing specifications, it must remain competitive. In general the following economy related reasons can be listed [25, 26]:

- Extend engineering resin performance by diluting it with a low cost polymer.
- Improvement a group of desired properties
- Formulation a high performance blend from synergistically interacting polymer.
- Adjusting the composition of the blend to customer specification.
- Recycling industrial or municipal plastics waste

2.6 Fiber Reinforced Composite Materials

Reinforced fiber composite materials comprised of fibers of high strength and embedded in to a matrix with distinct boundaries between them. In this form, both matrix and fiber retain their chemical and physical identities, yet they produce a combination of properties that cannot be achieved using either of constituents acting alone [27]. Generally, fibers are the principal load-carrying member, while the surrounding matrix keeps them in the desired orientation and location, for example, acts as load transfer component between them, and protects them from environmental damages due to elevated and heat humidity. Thus, even though the fibers provide reinforcement for the matrix, the latter also serves a many of useful functions in a fiber reinforced composite material [28, 29].

As Cs. Varga et al studied that the reinforced fiber and the chemical structure has its effect on the mechanical properties of composite. They found the influence of addition

two types of reinforcing materials (chopped glass-fiber mat and glass woven) on mechanical properties of polyester as shown in Fig. 2.7 and Fig. 2.8 [2].

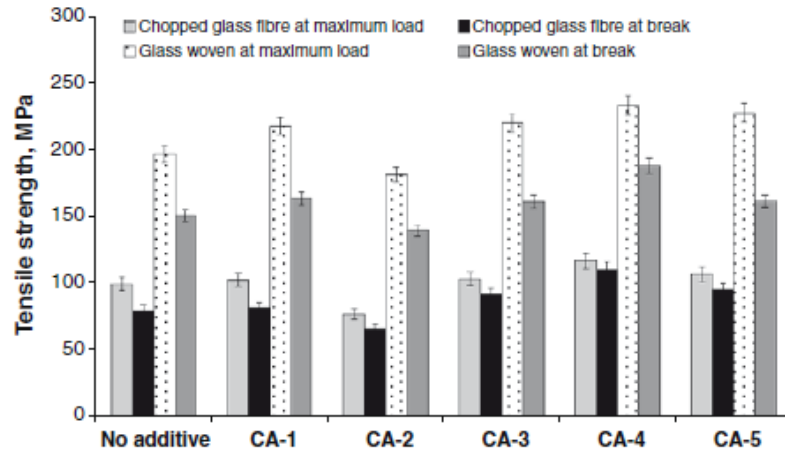


Figure 2.7: The tensile Strength of composites [2].

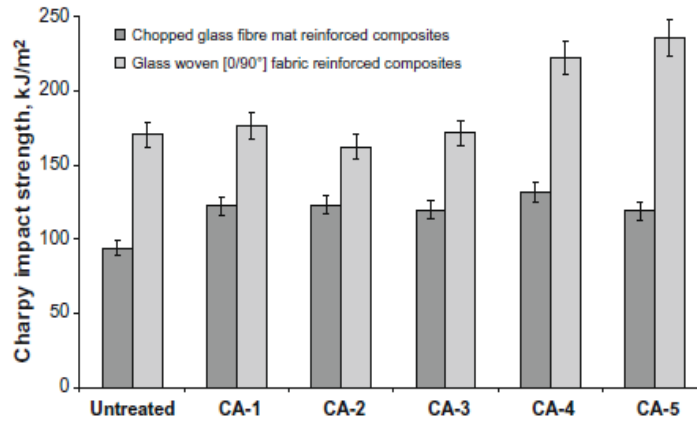


Figure 2.8: Charpy Impact Strength of reinforced polyester composites in case of different coupling additives [2].

Also, S. Srinivasa Moorthy et al. studied the tensile strength on the composites increase with increase in weight ratio percentage of fiber. They found properties TiO₂ of particulate glass fiber reinforced polymer composite with different percentage, as shown in Fig. 2.9 [30].

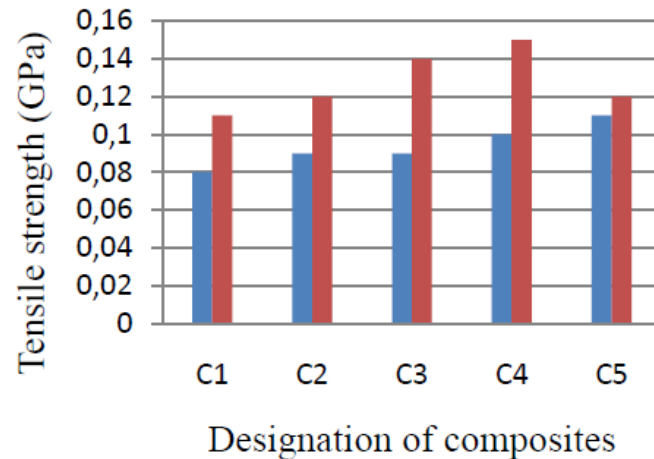


Figure 2.9: Tensile strength for different fiber length composite [30].

Chevali et al. [31], concluded that the nonlinear experimental power model could represent experimental data very well for all the materials at different temperatures. used to represent the empirical data of nylon-6.6, polypropylene and HDPE discontinuous glass fiber composites in the temperature range of (23 to 120)°C in flexural creep tests. In a study by Hugo et al. Was used as a parameter to investigate the minimum creep rate (MCR), which is the slope of (strain–time curve) in the secondary or steady state region of creep, and was used as a parameter to investigate different influences including temperature, fiber content and fiber/matrix adhesion on creep behavior of short glass fiber reinforced polypropylene [32].

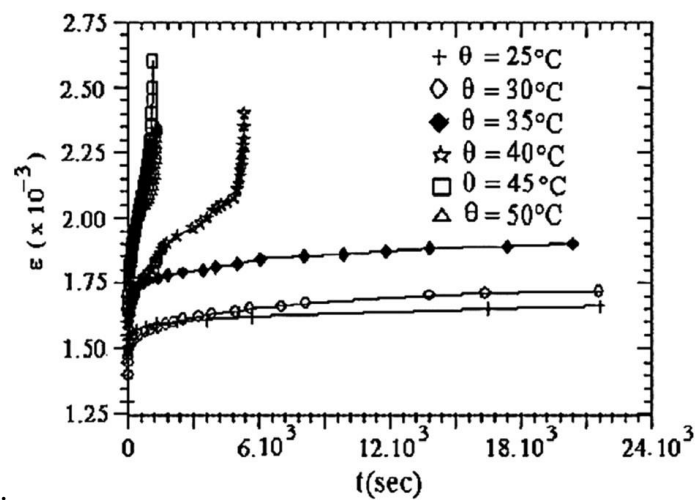


Figure 2.10: Effect of temperature on creep behavior of glass fiber reinforced polyester composites under a load [32].

2.7 Fiber

Glass-reinforced composites gain their strength from thin glass fibers group within their resin matrix. These stiff and strong fibers carry the load while the resin matrix diffusions the load imposed on the composite. Can be produced fibers made primarily from silica-based glass containing several metal oxides, fibers can also be produced from carbon, boron and aramid. While these materials offer higher tensile strength and are stiffer than glass, they cost significantly more. Can we use a hybrid fiber (combining an expensive fiber with a glass fiber), which improves overall performance yet costs less than using premium fibers alone [33-35].

E-glass is a popular fiber made primarily of silica oxide, along with oxides of aluminum, boron, calcium and other compounds. Characterized by for its good electrical resistance, E-glass is strong yet low in cost, and accounts for over 90% of all glass fiber reinforcements, especially in aircraft [36, 37].

2.7.1 Types of Fiber - Mats Fabric

Available in chopped and continuous-strand, Chopped-strand mat contains randomly distributed fibers held together with a chemical binder as shown in Fig. 2.11. Since the binder dissolves in styrene (a material contained in polyester and polystyrene resins), chopped-strand mats contains randomly conform easily to complex shapes. Providing low-cost plastic reinforcement, chopped-strand mat is primarily used in hand lay-up [38].

Stronger than chopped-strand, continuous-strand mat is formed by swirling continuous strands of fiber onto a moving belt, and then finished with a chemical binder to hold fibers in place, resulting in a thick, smooth, resin-rich type [39].



Figure 2.11: Chopped-strand mat contains randomly distributed fibers.

2.8 Interfaces and Bonding Composite

The interface region in a particular composite has a great deal in determining the ultimate properties of the composite, essentially because the interface occupies a very large area per unit volume in a composite, and in general the reinforcement and the matrix form a system that is not in thermodynamic equilibrium [40].

It can be define an interface as a boundary surface between two phases in which a discontinuity in one more material parameters occurs. An important parameter in regard to the interface is the wet-ability of reinforcement by the matrix. Wet-ability refers to the ability of a liquid to spread on a solid substrate .Good wetting is a necessary ,but not sufficient condition for strong bounding, the other important factors such as chemical, mechanical, thermal and structural factors, affect the nature of the bounding between reinforcement and matrix materials [41, 42].

As Mocko et al. [43] studied the effect of strain rate and temperature on compression test results. Fig. 2.12 presents the stress-strain characteristics of each orientation. It showed the differences in the tensile curves, and hence differences in such mechanical parameters as ultimate tensile stress and Young's modulus. Increase of the strain rate resulted in an increase of the elastic modulus and the generation of maximum stress leading to material damage. This behavior may be caused by the viscoelastic properties of the composite matrix. In addition, the type of loading affects the mechanism of material damage. In addition a temperature increase led to material softening behavior.

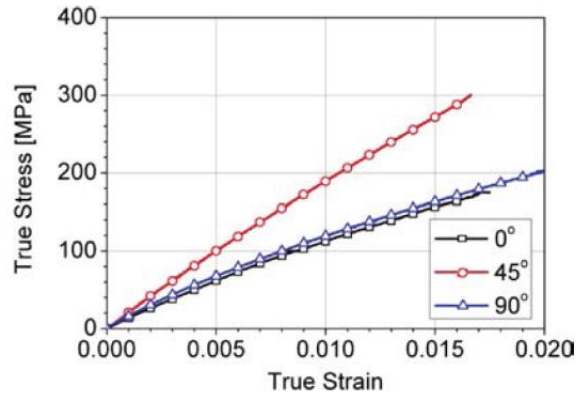


Figure 2.12: Stress-Strain characteristics of the composites selected along the three directions considered [43].

The effect of varying loading rate dependence on the tensile, compressive, shear, and flexural properties of fiber-reinforced composite materials has been investigated by a number of workers and a variety of contradictory observations and conclusions have resulted. Hence, more work must be done in the pursuit of eliminating all disagreements that currently exist regarding the effect of loading rate on the tensile, compressive, shear, and flexural properties of fiber-reinforced polymer composite material. There was also not much literature available on the effect of strain rate on the tensile, compressive, and shear properties of random chopped fiber reinforced composite materials that can find extensive use in a wide range of load-bearing engineering and industrial process applications primarily due to the low costs involved in their manufacture in addition to the ease of manufacture. Hence, the need for investigating and characterizing in detail the strain rate effects on the tensile, compressive, shear, and other mechanical properties of random chopped fiber composites is suggested, [44, 45].

2.9 Stress-Strain Behavior of Polymers

The mechanical properties of polymers are most often obtained using a uniaxial tensile test. Schematic (stress-strain) diagrams characteristic of those found for the indicated types of solid polymers are shown in Fig. 2.13. Curve-1 represents a linear elastic and brittle material like an epoxy, polystyrene, etc. Curve-2 is similar to that of a

semi-ductile material like Poly (methyl methacrylate) PMMA. Curve-3, is similar to that of a ductile material like Polyethylene Terephthalate (PET) or polycarbonate. Curve-4 is similar to that of a typical elastomer such as a flexible urethane. The 0.2% offset method to determine yield stress cannot be used as strains in polymers are quite large compared to structural metals such as steel and aluminum. The yield stress of a ductile material is often assumed to be equal to the proportional limit stress or the first peak in the stress strain diagram (termed the intrinsic yield point) as indicated in Fig. 2.14. It is to be noted that many approaches to determining the yield point are used, although the intrinsic yield point is the most common. The extrinsic yield point is the point of tangency of a line drawn from a point on the strain axis of (-1.0%) to the stress-strain diagram [46-47].

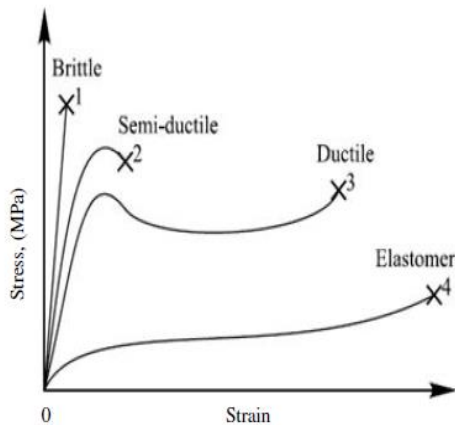


Figure 2.13: Typical Stress-Strain (Load-Elongation).

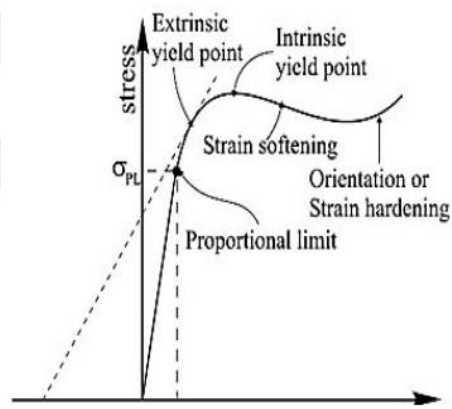


Figure 2.14: Considerer's definition of diagrams yielding for polymers [47].

In 2015, M. Eftechari et al, [10], investigated and evaluated the mechanical behavior of short fiber reinforced polymer material composites SFRPCs at elevated temperature (tensile, creep, fatigue and viscoelastic behavior of short fiber reinforced composite at elevated temperature and the significant effect of elevated temperatures on increased mobility of polymer chains, and in turn, on mechanical behavior and failure mechanisms of short fiber reinforced composites. Several studies consider the effect of temperature on tensile properties of these materials. Increasing temperature has been observed to significantly affect the tensile behavior of SFRPCs. For instance, stress-strain curves in tension tests of 35 wt% short glass fiber reinforced polyamide-6.6 at room temperature RT and 130 °C for samples with different fiber orientations with

respect to the loading axis are shown in Fig. 2.15. At RT, the material behaved in a brittle way with a linear elastic behavior for most of the stress–strain curve, while at 130 °C significant plastic deformation is observed. This results from increased mobility of polymer chains at 130 °C, which is much higher than Tera gram Tg at 65°C

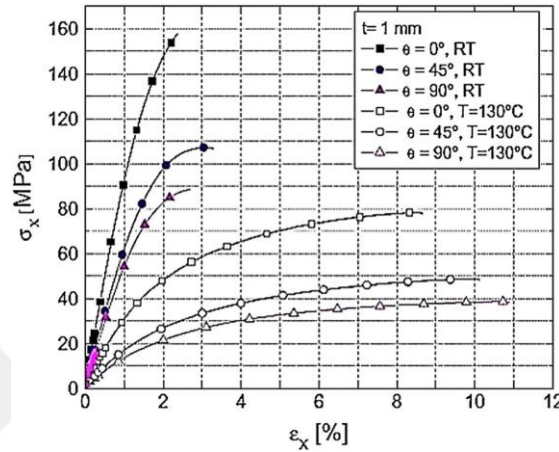


Figure 2.15: Comparison of stress–strain curves [10].

Comparison of stress–strain curves for 1mm thick specimens of short glass fibre reinforced polyamide-6.6 in different directions at RT and 130 °C. θ is the fiber orientation with respect to the loading axis [48].

2.10 Creep of Polymers

When plastic materials are subjected to a constant load, they deform continuously, Fig. 2.16. The initial strain is predicted by its stress-strain modulus. The material will continue to deform slowly with times indefinitely or until rupture or yielding causes failure.

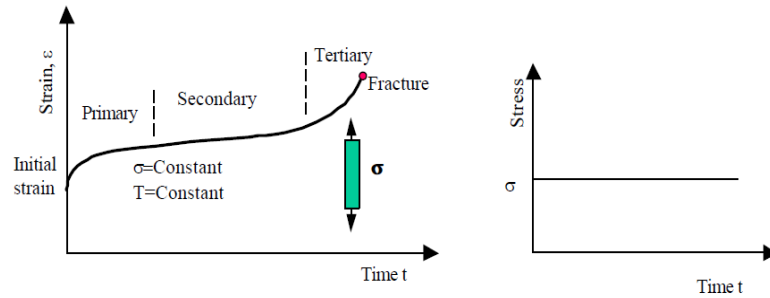


Figure 2.16: Creep curve for plastics, a constant load is applied [49].

The primary region is the early stage of loading when the creep rate decreases rapidly with time. Then it reaches a steady state which is called the secondary creep stage followed by a rapid increase (tertiary stage) [48-49].

2.11 Polymer Viscoelastic Behavior

Viscoelasticity is the property of materials that exhibit both viscous and elastic characteristics when undergoing deformation. Viscous materials resist shear flow and strain linearly with time when a stress is applied. Elastic materials strain when stretched and quickly return to their original state once the stress is removed, shown in Fig. 2-17a. Viscoelastic materials have elements of both of these properties and, as such, exhibit time-dependent strain. Whereas elasticity is usually the result of bond stretching along crystallographic planes in an ordered solid, viscosity is the result of the diffusion of atoms or molecules inside an amorphous material [49] as seen in Fig. 2.17 b.

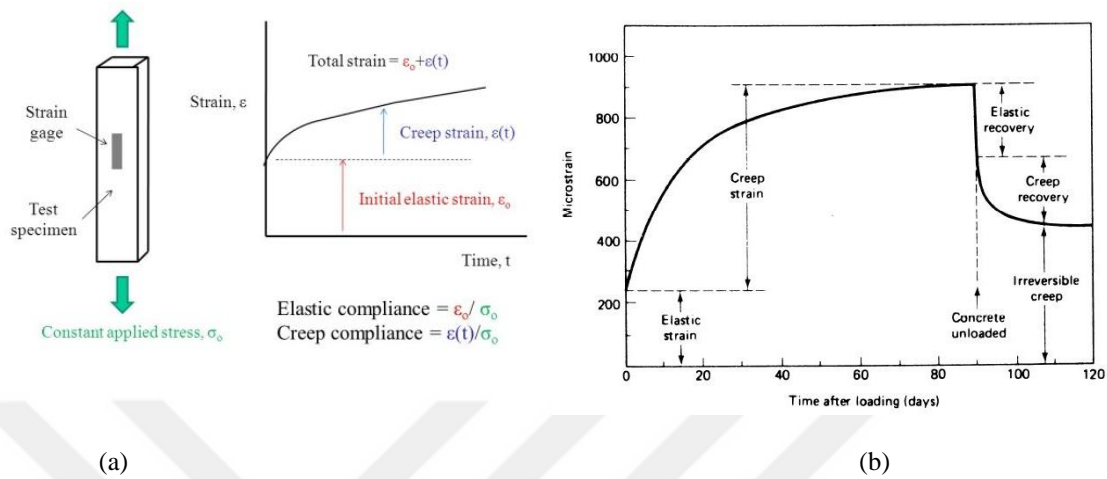


Figure 2.17: (a) Viscoelastic behavior curve of material, (b) the creep test parameters

2.11.1 Polymer Viscoelastic Properties and Characterization

The word viscoelastic is derived from the words "viscous" + "elastic"; a viscoelastic material exhibits both viscous and elastic behavior – a bit like a fluid and a bit like a solid. One can build up a model of linear viscoelasticity by considering combinations of the linear elastic spring and the linear viscous dash-pot. These are known as rheological models or mechanical models [50].

Viscoelasticity or Rheology is the study of materials whose mechanical properties have characteristics of both solid and fluid materials. Viscoelasticity is a term often used by those whose primary interest is solid mechanics while rheology is a term often used by those whose primary interest is fluid mechanics. The term also implies that mechanical properties are a function of time due to the intrinsic nature of a material and that the material possesses a memory (fading) of past events [48, 51].

2.11.2 Viscoelastic Material Properties

- Hysteresis is seen in the stress–strain curve
- Stress relaxation occurs: step constant strain causes decreasing stress

- Creep occurs: step constant stress causes increasing strain

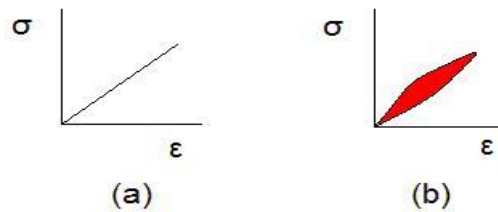


Figure 2.18: stress-strain curve (a) plastic and (b) viscoelastic.

The red area is a hysteresis loop and shows the amount of energy lost (as heat) in a loading and unloading cycle. Specifically, viscoelasticity is a molecular rearrangement. When a stress is applied to a viscoelastic material such as a polymer, parts of the long polymer chain change positions. This movement or rearrangement is called creep. Polymers remain a solid material even when these parts of their chains are rearranging in order to accompany the stress, and as this occurs, it creates a back stress in the material. When the back stress is the same magnitude as the applied stress, the material no longer creeps. When the original stress is taken away, the accumulated back stresses will cause the polymer to return to its original form [47, 49].

2.11.3 Creep and Relaxation Tests

One of the fundamental methods used to characterize the viscoelastic time dependent behavior of a polymer is the relaxation test. In a relaxation test, a constant strain is applied quasi-statically to a uniaxial tensile (or compression or torsion) bar at zero time. That is, the bar is suddenly stretched to a new position and rigidly fixed such that the strain remains constant for the duration of the test. The sudden strain must not induce any dynamic or inertia effects. When the polymer loaded in the described manner, the stress needed to maintain the constant strain will decrease with time. Eventually, the stress will go to zero for an ideal thermoplastic polymer but will decrease to a constant value for a cross linked polymer. The strain input and the stress output for typical thermoset and thermoplastic materials in a relaxation test are shown in Fig. 2.19. Obviously, if the stress is a function of time and the strain is constant, the modulus will

also vary with time. The modulus so obtained is defined as the relaxation modulus of the polymer and is given by [47, 51]:

$$E(t) = \frac{\sigma(t)}{\epsilon_0} = \text{Relaxation Modulus} \quad (2.1)$$

or;

$$\sigma(t) = E(t) * \epsilon_0 \quad (2.2)$$

The latter equation is the uniaxial stress-strain relation for a polymer analogous to Hooke's law for a material that is time independent but is valid only for the case of a constant input of strain. The relaxation test provides the defining equation for the material property identified as the relaxation modulus.

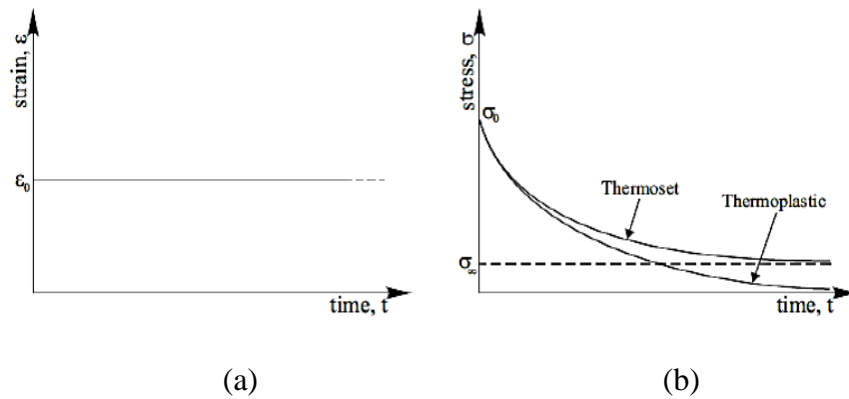


Figure 2.19: Relaxation test: (a) strain input and (b) qualitative stress output [49].

The limiting modulus at $t = 0$ and at $t \infty$ for a cross linked material are defined as:

$$E(t = 0) = \frac{\sigma(t=0)}{\epsilon_0} = E_0 = \text{Initial Modulus} \quad (2.3)$$

$$E(t = \infty) = \frac{\sigma(t=\infty)}{\epsilon_0} = E_\infty = \text{Equilibrium Modulus} \quad (2.4)$$

In addition to the relaxation test, another fundamental characterization test for viscoelastic materials is the creep test in which a uniaxial tensile (or compression or torsion) bar is loaded with a constant stress at zero time as shown in Fig. 2.20. Again, the load is applied quasi-statically or in such a manner as to avoid inertia effects and the

material is assumed to have no prior history. In this case, the strain under the constant load increases with time and the test defines a new quantity called the creep compliance:

$$D(t) = \frac{\varepsilon(t)}{\sigma_0} = \text{Creep compliance} \quad (2.5)$$

In this case,

$$\varepsilon(t) = \sigma_0 D(t) \quad (2.6)$$

An equally important fact of a constant stress test is to understand the resulting strain variation if the stress is removed. This is referred to as a creep-recovery test and is also shown in Fig. 2.20. For an ideal thermoset material, the strain will decay to zero after a sufficient time interval which may be quite long is compared to the time of loading. For an ideal thermoplastic material, a residual deformation or permanent strain (ε_p) will remain even after a very long (or infinite) time [49, 52].

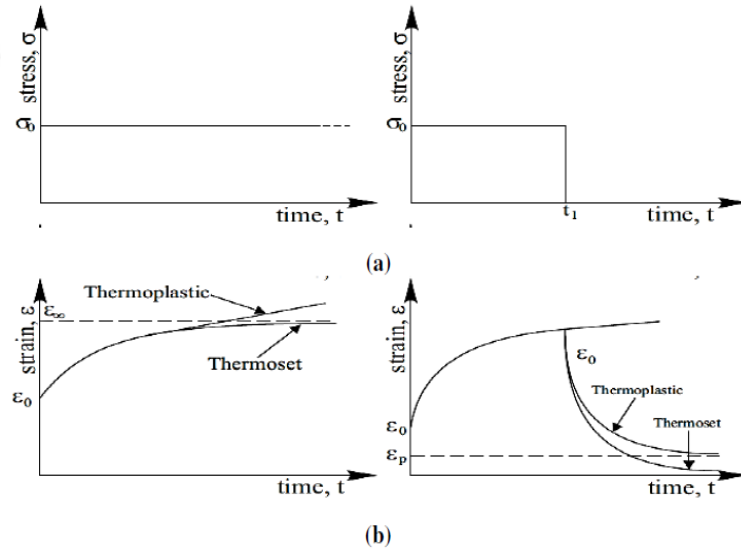


Figure 2.20: Creep and creep recovery tests: (a) stress input and (b) qualitative material Strain response [49].

The deformation mechanisms associated with relaxation and creep are related to the long chain molecular structure of the polymer. Continuous loading gradually induces strain accumulation in creep as the polymer molecules rotate and unwind to accommodate the load. Similarly, in relaxation, the initial sudden strain occurs more rapidly than can be accommodated by the molecular structure. However, with time the

molecules will again rotate and unwind so that less stress is needed to maintain the same strain level. It is also clear from these tests that polymers have some characteristics of a solid and some characteristics of a fluid. In a relaxation test, the ratio of the initial stress and strain is,

$$E(t = 0) = \frac{\sigma_0}{\varepsilon_0} \quad (2.7)$$

$$D(t = 0) = \frac{\varepsilon_0}{\sigma_0} \quad (2.8)$$

This is analogous to the behavior of an elastic solid. On the other hand in a creep test the rate of change of strain (or slope) for a thermoplastic material is,

$$\frac{d\varepsilon(t=0)}{dt} = \text{Constant} \quad (2.9)$$

After a sufficiently long period of time which is characteristic of a fluid, the flow characteristics of a thermoplastic are due to the lack of primary bonds between molecular chains and the solid characteristics of a thermoset are due to entanglements and the primary bonds between individual chains. In both thermosets and thermoplastics, creep (which is also viscous like), is related to the motion of molecules between entanglements, while the mechanisms for creep are further limited to motion between cross linking sites for thermosets. The initial and equilibrium modules of a thermoset are solid like with the former being due to both entanglements and cross links and the latter being principally due to cross links [53, 54].

2.11.4 Viscoelastic Behavior / Mathematical Models

The most successful of the mathematical models have been based on spring and dashpot elements to represent, respectively, the elastic and viscous responses of plastic materials.

1) The linear elastic spring:

The constitutive equation for a material which responds as a linear elastic spring of stiffness δ is:

$$\varepsilon = \frac{\sigma}{\delta} \quad (2.10)$$

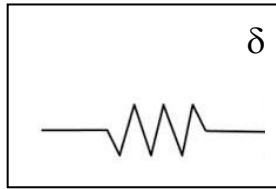


Figure 2.21: The linear elastic spring [51].

The response of this material to a creep-recovery test is to undergo an instantaneous elastic strain upon loading, to maintain that strain so long as the load is applied, and then to undergo an instantaneous destraining upon removal of the load, Fig. 2.21 illustrate elastic spring element [54, 55].

2) The linear viscous dash-pot

Imagine next a material which responds like a viscous dash-pot; the dash-pot is a piston cylinder arrangement, filled with a viscous fluid, Fig. 2.22 shows a strain is achieved by dragging the piston through the fluid. By definition, the dash-pot responds with a strain rate proportional to stress:

$$\dot{\varepsilon} = \frac{\sigma}{\eta} \quad (2.11)$$

Where:

η : is the viscosity of the material.

This is the typical response of many fluids; the larger the stress, the faster the straining [51, 55].

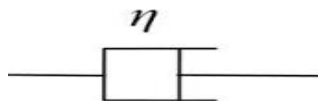


Figure 2.22: The linear dash-pot [51].

2.11.5. Maxwell Model

The Maxwell Model consists of spring and dashpot in series as shown in Fig. 2.23.

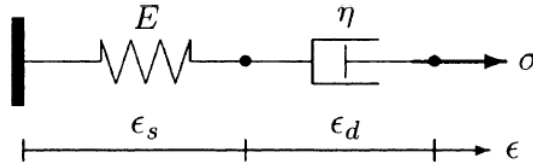


Figure 2.23: Maxwell model [51].

A) Stress -Strain Relations:

The spring is the elastic component of the response and obeys the relation [55].

$$\sigma_1 = \delta_1 * \epsilon_1 \quad (2.12)$$

Where:

δ_1 : is spring constant.

ϵ_1 : The strain in dashpot.

The dashpot is the viscous component of the response and in this case (σ) and (ϵ) are the stress and strain respectively and (σ) is a constant, as shown in Fig. 2.24.

$$\sigma_2 = \eta_1 * \dot{\epsilon} \quad (2.13)$$

Where:

$\dot{\epsilon}$: the strain in spring

η_1 : is a material constant (coefficient of viscoelasticity).

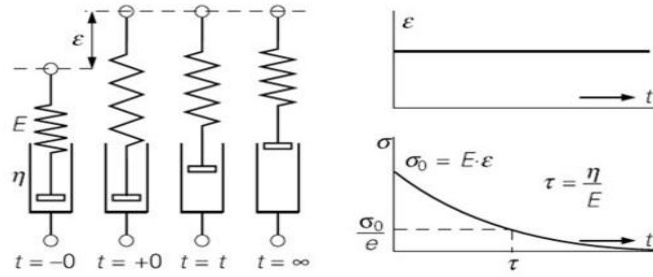


Figure 2.24: Stress Relaxation of a Maxwell element [55].

B) Equilibrium Equation:

For equilibrium of forces, assuming constant area of applied stress,

$$\sigma = \sigma_1 = \sigma_2 \quad (2.14)$$

Since both elements are connected in series, the total or the rate of strain is:

$$\dot{\varepsilon} = \dot{\varepsilon}_1 + \dot{\varepsilon}_2 \quad (2.15)$$

1) Creep

If a constant stress, σ , is applied then the equation becomes,

$$\dot{\varepsilon} = \frac{1}{\eta} * \sigma \quad (2.16)$$

This indicates a constant rate of increase in strain with time. From Fig. 2.25 it may be seen that for the Maxwell model, the strain at any time, t , after the application of a constant stress, σ , is given by [56]:

$$\varepsilon(t) = \frac{\sigma}{E} + \frac{\sigma}{\eta} t \quad (2.17)$$

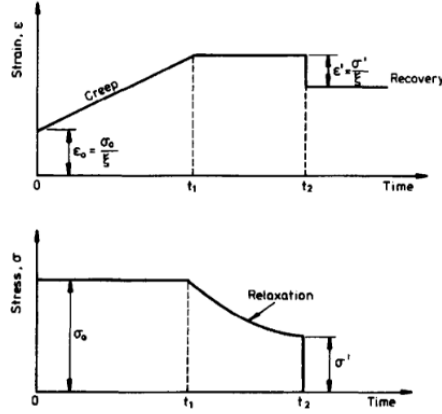


Figure 2.25: Response of Maxwell model [49].

Hence, the creep modulus, $E(t)$ is given by

$$E(t) = \frac{\sigma}{\varepsilon(t)} = \frac{\delta_1 \eta_1}{\eta_1 + \delta_1 t} \quad (2.18)$$

2) Relaxation:

If the strain is held constant then the equation becomes,

$$\sigma(t) = \sigma * e^{-\frac{\delta t}{\eta}} \quad (2.19)$$

3) Recovery:

When the stress is removed there is an instantaneous recovery of the elastic strain, δ , and then, the strain rate is zero so that there is no further recovery. It can be seen therefore that although the relaxation behavior of this model is acceptable as first approximations to the actual material response, it is inadequate in its prediction for creep and recovery behavior.

2.11.6. Kelvin or Voigt Model

Consists of a Newtonian damper and Hooke elastic spring connected in parallel, as shown in Fig. 2.26 [57].

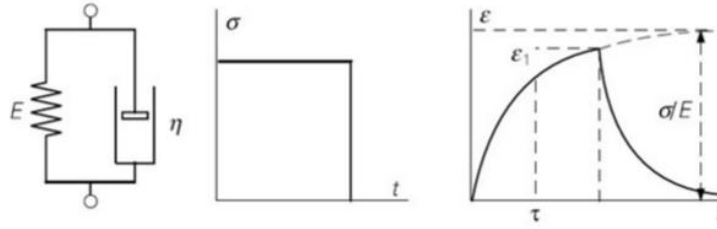


Figure 2.26: Kelvin or Voigt model [57].

A) Stress-Strain Relations

These are the same as the Maxwell Model and are given by Equations 2.12 and 2.13.

B) Equilibrium Equation

Jointly by the spring and the dashpot, so for equilibrium of forces it can be seen that the applied load is supported.

$$\sigma = \sigma_1 + \sigma_2 \quad (2.20)$$

C) Geometry of Deformation Equation

In this case the total strain is equal to the strain in each of the elements, i.e.;

$$\varepsilon = \varepsilon_1 = \varepsilon_2 \quad (2.21)$$

Or using equation,

$$\sigma = \delta_2 \varepsilon_2 + \eta_1 \dot{\varepsilon} \quad (2.22)$$

This is the governing equation for the Kelvin (or Voigt) Model and it is interesting to consider its predictions for the common time dependent deformation [51], [56] and [57].

1) Creep

If a constant stress, is applied then Eq. 2.20 becomes [54], [56] and [57]:

$$\sigma_0 = \delta_2 \varepsilon_2 + \eta_2 \dot{\varepsilon} \quad (2.23)$$

And this differential equation may be solved for the total strain, to give

$$\varepsilon(t) = \frac{\sigma_0}{\delta} [1 - e^{-\frac{\delta t}{\eta}}] \quad (2.24)$$

where the ratio $\frac{\eta}{\delta}$ is referred to as the retardation time, TR. This indicates an exponential increase in strain from zero up to the value that the spring would have reached if the dashpot had not been present. This is shown in Fig. 2.13 as for the Maxwell Model, the creep modulus may be determined as,

$$E(t) = \frac{\sigma_0}{\varepsilon(t)} = \delta [1 - e^{-\frac{t}{TR}}]^{-1} \quad (2.25)$$

2) Relaxation

If the strain is held constant then Eq. 2.22 becomes [54, 56]:

$$\sigma = \delta_2 * \varepsilon_2 \quad (2.26)$$

That is, the stress is constant and supported by the spring element so that the Predicted response is that of an elastic material, i.e. no relaxation, Fig. 2.26.

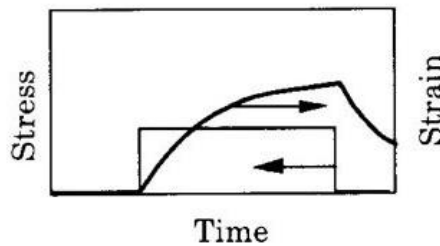


Figure 2.27: Response of Kelvin or Voigt model [49].

3) Recovery

If the stress is removed, then Eq. 2.22 becomes [54, 57]:

$$0 = \delta_2 \varepsilon_2 + \eta_1 \dot{\varepsilon} \quad (2.27)$$

This differential equation is solved with initial conditions $\varepsilon = \varepsilon_0$ at the time of stress removal, then

$$\varepsilon(t) = \varepsilon_0 * e^{-\frac{\delta_2 t}{\eta_1}} \quad (2.28)$$

This represents an exponential recovery of strain which is a reversal of the predicted creep.

2.12.7 Burger's Four – Element Model

Maxwell viscoelastic model represents the series connection of spring and dashpot, while the kelvin model represents the parallel connection of spring and dashpot. The simplest model, which describes the stress relaxation is Maxwell's two-element model,[3,22] but its usage for mechanical deformation in polymer and composite systems interpretation leads to significant discrepancies with experimental results. For description of the experimental creep and stress relaxation curves the Burger's four-element viscoelastic model has been used. This model is a combination of elastic elements (springs) with the Hookean deformation and dampers, which are deformed according to Newton's law. Theoretical creep and stress relaxation curves of Burger's model. The Burger's model is shown in Fig. 2.28 a and 2.28 b where the Maxwell and kelvin models are connected in series [3, 53]:

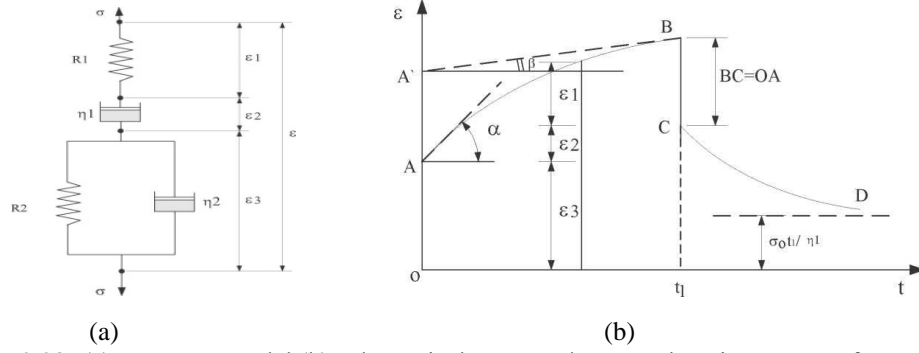


Figure 2.28: (a); Burger's model (b); Theoretical creep and stress relaxation curves of Burger's model [53].

The total strain can be written as follows:

$$\varepsilon = \varepsilon_1 + \varepsilon_2 + \varepsilon_3 \quad (2.29)$$

In which

ε : Total strain in Burgers four – element model .

ε_1 : The strain in spring for Maxwell model .

ε_2 : The strain in dashpot for Maxwell model .

ε_3 : The strain in Kelvin model .

That is :

$$\varepsilon_1 = \frac{\sigma}{R_1} \quad (2.30)$$

$$\dot{\varepsilon}_2 = \frac{\sigma}{\eta^1} \quad (2.31)$$

$$\dot{\varepsilon}_3 + \frac{R_2}{\eta_2} + \varepsilon_3 = \frac{\sigma}{\eta_2} \quad (2.32)$$

From Equations 2.29-2.32, the following second order differential equation between stress and strain can express as :

$$\sigma + \left(\frac{\eta_1}{R_1} + \frac{\eta_1}{R_2} + \frac{\eta_2}{R_2} \right) \dot{\sigma} + \frac{\eta_1 \eta_2}{R_1 R_2} \ddot{\sigma} = \eta_1 \dot{\varepsilon} + \frac{\eta_1 \eta_2}{R_2} \ddot{\varepsilon} \quad (2.33)$$

The creep behavior of Burgers four – element model under constant stress ($\sigma = \sigma_0$) can be obtained from Eq. 2.33 by solving this second order differential equation with two initial conditions [53, 58] :

$$\text{At } t = 0 ; \varepsilon = \varepsilon_1 = \frac{\sigma_0}{R_1} , \quad \varepsilon_2 = \varepsilon_3 = 0 \quad (2.34)$$

$$\dot{\varepsilon} = \frac{\sigma_0}{\eta_1} + \frac{\sigma_0}{\eta_2} \quad (2.35)$$

The Laplace transformation method of solving differential equations is used to solve Eq. 2.33 as follows:

$$\varepsilon(t) = \frac{\sigma_0}{R_1} + \frac{\sigma_0}{\eta_1} t + \frac{\sigma_0}{R_2} (1 - e^{-\frac{R_2 t}{\eta_2}}) \quad (2.36)$$

The material constant (R_1, R_2, η_1, η_2) may be determined from the experimental data for creep test in linear viscoelasticity behavior.

Appendix (A1) shows this method.

The stress relaxation resulting from a step strain to (ε_0 at $t = 0.0$) result:

$$\varepsilon = \varepsilon_0 H(t) \quad (2.37)$$

$$\dot{\varepsilon} = \varepsilon_0 \delta(t) \quad (2.38)$$

$$\ddot{\varepsilon} = \varepsilon_0 \frac{d\delta(t)}{dt} \quad (2.39)$$

Where:

H (t): Heaviside function .

$\delta(t)$: Dirac delta function .

Thus, Eq. 2.25 becomes:

$$\sigma + P_1 \sigma + P_2 \sigma = q_1 \varepsilon_0 \delta(t) + q_2 \varepsilon_0 \frac{d\delta(t)}{dt} \quad (2.40)$$

Where:

$$P_1 = \left(\frac{\eta_1}{R_1} + \frac{\eta_1}{R_2} + \frac{\eta_2}{R_2} \right)$$

$$P_2 = \frac{\eta_1 \eta_2}{R_1 R_2}$$

$$q_1 = \eta_1$$

$$q_2 = \frac{\eta_1 \eta_2}{R_2}$$

Taking the Laplace transformation of Eq. 3.40 yields:

$$\sigma(s) + q_1 s \sigma(s) + q_2 s^2 \sigma(s) = q_1 \delta_0 + q_2 \delta_0 s \quad (2.41)$$

Solving for $\sigma(s)$:

$$\sigma(s) = \frac{\delta_o(q_1+q_1s)}{1+P_1s+P_2s^2} \quad (2.42)$$

Expanding Eq. 3.42 by partial fractions and performing the inverse Laplace Transformation yields the stress relaxation [59]:

$$\sigma(t) = \frac{\delta_o}{A} [(q_1 - q_2 r_2) e^{-2r}] \quad (2.43)$$

Where:

$$r_1 = \frac{P_1-A}{2P_2}$$

$$r_2 = \frac{P_1+A}{2P_2}$$

$$A = \sqrt{p_1^2 - 4P_2}$$

2.11.8 Creep Compliance:

In a creep test a step of constant stress $\sigma = \sigma_o H(t)$

is applied and the time dependent strain $\varepsilon(t)$ is measured . For linear material, the strain can be represented by [60, 61]:

$$\delta(t) = \sigma_o D(t) \quad (2.44)$$

$$D(t) = \frac{\varepsilon(t)}{\sigma_o} \quad (2.45)$$

Where:

D (t): creep compliance (creep strain per unit stress).

Creep compliance is a material property. Thus each material has its own function of creep compliance can be written from Eq. 2.36 as:

$$D(t) = \frac{\delta(t)}{\sigma_o} = \frac{1}{R_1} + \frac{1}{\eta_1} + \frac{1}{R_2} (1 - e^{-Rt/\eta_2}) \quad (2.46)$$

2.11.9 Relaxation Modulus

In a relaxation test a step of constant strain $\varepsilon = \varepsilon_0 H(t)$

is applied and the stress is measured . If the material behavior is linear, the stress can be represented by:

$$\sigma(t) = \varepsilon_0 E(t) \quad (2.47)$$

$$E(t) = \sigma(t)/\varepsilon_0 \quad (2.48)$$

The function E(t) is thus obtained and is called the relaxation modulus . It is the stress per unit applied strain which is different for each (material property).

For example in Burger model the equation of relaxation modulus can be written from Eq. 2.43 as:

$$E(t) = \sigma(t)/\varepsilon_0 \quad (2.49)$$

On the other hand, the shear relaxation modulus can be obtained from Eq. 2.49 according to the theory of elasticity law as follows [62]:

$$G = \frac{E}{2(1+\nu)} \quad (2.50)$$

Where:

G: modulus of rigidity

Or [61]:

$$G(t) = \frac{E(t)}{2(1+\nu)} \quad (2.51)$$

Substitute from Eq. 2.49 into Eq. 2.51 gives:

$$G(t) = \frac{1}{2A(1+\nu)} [(q_1 - q_2 r_1) e^{-r_1 t} - (q_1 - q_2 r_2) e^{-r_2 t}] \quad (2.52)$$

Eq. 2.52 represents shear relaxation modulus viscoelastic material according to Burger model.

2.12. Typical Creep Curve

A typical creep curve is shown in Fig. 2.29 depicting the three stages of creep after an initial strain upon loading. (ε_o) stage I: this stage show the creep rate as a diminishing function of creep strain and is a result of strain hardening, which dominates deformation in this stage. Stage II: corresponds to the steady - state creep - rate range. Where there is a balance between the strain hardening and softening process. The steady state creep rate is given the symbol ($\dot{\varepsilon}_o$). It is also often designated as the minimum creep rate. The third stage: indicated as III in the figure. It is also known as the tertiary stage: it shows the creep rate accelerating with strain, which is culminated by ultimate fracture of the material at (t_r), the time to rupture [13].

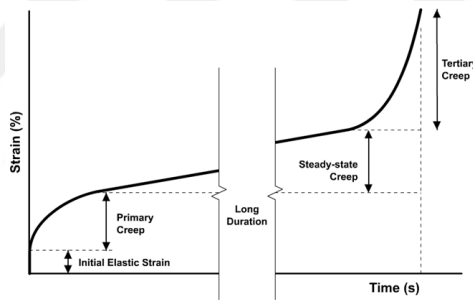


Figure 2.29: Schematic of a typical creep curve [13].

2.13 Creep Equation

We can represent behavior the polymer matrix composite. That we study it by the equation below:

$$\varepsilon = \varepsilon_o + a.t^b \quad (2.54)$$

Where:

t = time (Sec.), ε = stain (%), ε_o = initial strain

a, b = constant depend on the variable volume fraction

CHAPTER THREE

EXPERIMENTAL WORK

3.1 Introduction

The experimental work was divided into three stages, blend manufacturing, specimens preparation for mechanical testing to find the optimum ratio and final stage was carrying out creep test to study the viscoelastic properties of the chosen composite.

Fig. 3.1 depicts schematically flow chart of experimental work. Many variables were studied include processing the composite matrix (mechanical mixing, magnetic stirring time and temperature), and volume fraction of fibers. The preparation of the test specimens without flaws was the main difficulty that should be overcome, while other objectives were all within reach, though some adaptations should be made. So the approach that was concluded is as in the following:

- 1- Tensile test were conducted to investigate the relation between the strength and the toughness on polyester and composite materials, the effect of fibers on the blend, and compare between the different resulting composite materials.
- 2- Hardness test to evaluate the reduction in polyester hardness due to polystyrene addition.
- 3- Creep test was achieved to show the effect of the different fibers and loads on the creep behavior of polyester and its composites, and evaluating stress relaxation and viscoelastic behavior.
- 4- SEM were used in this research to study morphology process of the polyester, blend and their composite.

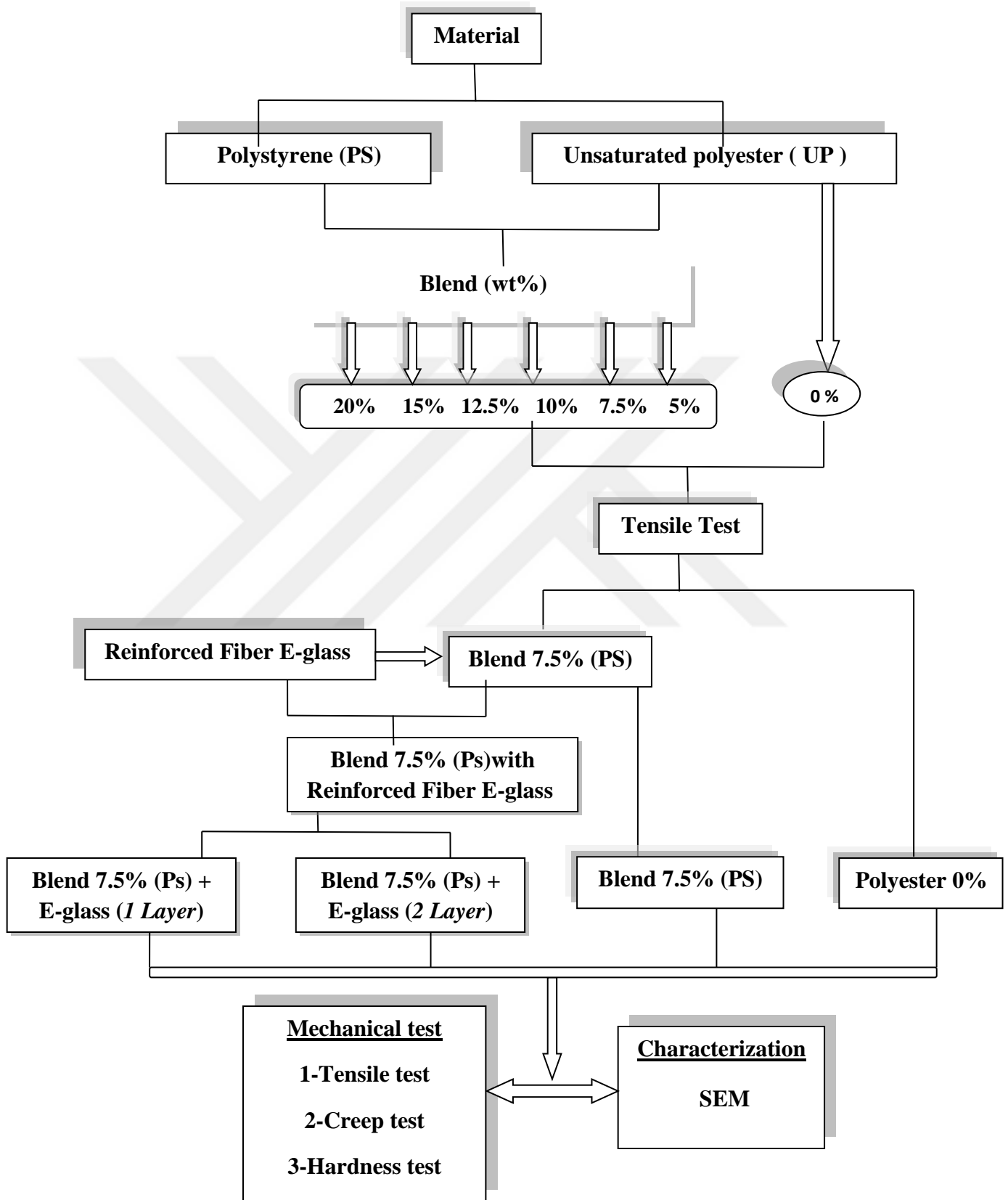


Figure 3.1: Flow chart of experimental work.

3.2 Materials Used for the Blend

3.2.1 Unsaturated Polyester (UP)

The used rein was supplied by Saudi Industrial Resins (SIR) Company and it is viscous transparent liquid at room temperature. The curing agent hardener was Methyl Ethyl Keton Peroxide (MEKP) which is obtained from the same company while the catalyst system was a liquid solution of cobalt octoate in dibutyl phthalate as accelerator of reaction. The hardener was added to the mixture with 2% weight ratio, while the ratio of the accelerator was (0.5%) [23, 63], used in this study are listed in Table 3.1.

Table 3.1: Unsaturated Polyester Mechanical and physical Properties [38].

Properties	Value	Condition
Tensile Strength (MPa)	57-65	at break & at yield
Elastic Modulus (MPa)	1932-3002	ASTM (D-638)
Elongation at break (%)	2-3	ASTM (D-638)
Hardness	68-78	Rockwell M
Thermal conductivity (W/m)	0.176 - 0.288	-
Density (kg/m ³)	1.2	-

3.2.2 Polystyrene (PS)

Polystyrene (PS) is produced from styrene monomer by continuous mass polymerization using chloroform CHCl₃. It is a crystal-like, brittle and hard polymer with high clearness and medium flow. It also has medium viscosity and makes it suitable for various applications. The properties of PS used in this study are listed in Table 3.2 [23].

Table 3.2: Polystyrene mechanical properties [23].

Properties	Value	ASTM method
Tensile strength	43 MPa	D-638
Tensile elongation	2 %	D-638
Tensile modulus	2598 MPa	D-638
Density at 23 °C	1050 kg/m ³	D-792
Hardness	95	(Rockwell L) D-785

3.2.3 Chloroform CHCl₃

It is a solvent used to dissolve PS. The properties of this solvent are given in Table 3.3.

Table 3.3: Chloroform CHCl₃ properties [67].

Molecular	weight 119.38 gm/mole
Density	1.4892 g/mL at 20°C
Viscosity	0.57 cP at 20°C
Boiling point	61.15°C
Solubility of water in chloroform	0.056% at 20°C
Solubility in water	0.815% at 20°C
Surface tension	27.16 dyn/cm at 20°C
DOT Hazard Class	6.1, Toxic

3.3 Fiber E-glass -Reinforcing Material

E-glass has been the predominant fiber for many mechanical engineering applications because of an economical balance of cost and specific tensile strength. Each of these fibers is available in chopped form and has low cost so they can be used industrially. The specifications fibers are given in Table 3.4 [39].

Table 3.4: E-glass fiber properties [37, 39]

Modulus of Elasticity (GPa)	Density (g/cm ³)	Elongation at break (%)	Max. Temp. In service °C	Length to diameter ratio
72-78	2.5-2.9	3-4	250	5

3.4 Specimens Preparation

Specimen's preparation process includes three stages:-

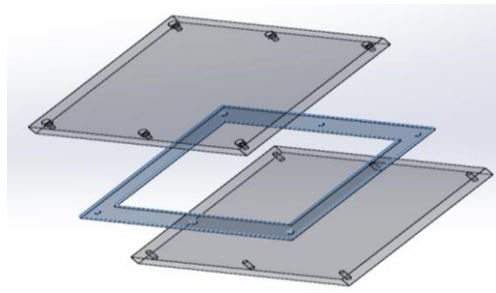
- a- Mold preparation.
- b- Preparation of unsaturated polyester – polystyrene blends.
- c- Preparation of fiber reinforced composites.

3.5 Mold Preparation

This stage includes preparing of special mold for each test. Two types of molds were used. The first type is rectangular plastic mold and the other type was sheet type mold.

3.5.1 Tensile Test Mold

Tensile tests as in the standard ASTM. (D-638) have been fulfilled using dog-bone specimens. A special mold has been used for the preparation of tensile test specimens as shown in Fig. 3.2a. The mold is a rectangular plastic shape with (360×245×4), Fig. 3.2b. It is manufactured according to ASTM standards; this technique was chosen to limit surface defects. Strain was measured using extensometer with free length 50mm. Loading rate was (1 mm/min.). Vaseline oil is used to prevent resin adhesion on mold.



(a)



(b)

Figure 3.2: Tensile test mold, (a) Mold Composites Specimen, (b) Mold Polyester Specimen.

3.5.2 Creep Test Mold

The other type of mold that is used for creep test was prepared using plastic sheet mold as shown in Fig. 3.3. By using laser cutting machine, two types of mold manufactured with three major parts fastened together by set of screws, the lower and upper plates are the same with (10 mm) thickness, and the mold (cavity) according to the required thickness (2 mm) which represents the resulting specimens' thickness.

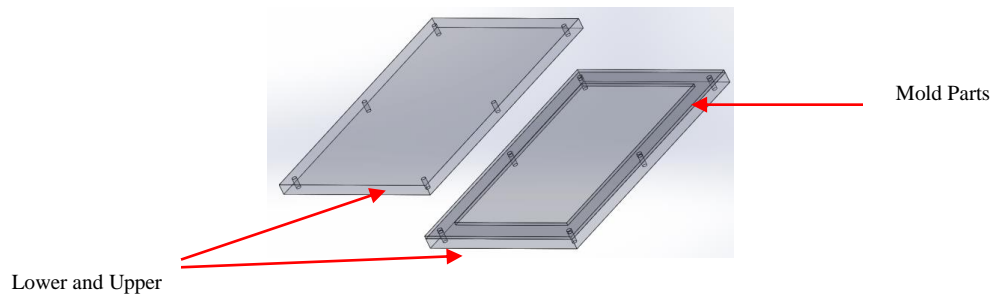


Figure 3.3: Creep test Mold.

3.6 Preparing Unsaturated Polyester - Polystyrene Blends

In this work, preparation of polymer blends was achieved by mixing Unsaturated polyester resin with different ratios of polystyrene, the ratios are shown in Table 3.5. The

above mentioned blends and composites were prepared according to the following procedure:

- 1- Dissolution of PS in chloroform solvent by using magnetic stirrer for a minimum of one hour, then it was put for 24 hours to obtain liquid polystyrene [23].
- 2- The required weight ratios of unsaturated polyester and polystyrene were obtained using sensitive balance with following ratios for each constituent of blend as in Table 3.5.

Table 3.5: weight ratios (wt%) of the required Unsaturated polyester and polystyrene

Polyester	100%	95%	92.5%	90%	87.5%	85%	80%
Polystyrene	-	5%	7.5%	10%	12.5%	15%	20%

- 3- The blend was mixed for 15 minutes of polyester and polystyrene resins blend with varying concentrations (wt%) using a mechanical mixer.
- 4- The blend mixing (Unsaturated polyester with Polystyrene) is put in magnetic stirrer for 3 hours period in room temperature to get good homogeneity.

3.7 Preparation of Fiber Reinforced Composites

The preparation of specimens was made as follows:

- 1- After preparing blend, it was subsequently put into vacuum desiccators at 0.017 bars for at least (15) minutes to remove bubbles from the mixture, see the Fig. 3.4.



Figure 3.4: Desiccators used for vacuuming

- 2- The hardener was then used for curing with mixing ratio of 2%.
- 3- The blend was poured into the tensile and creep molds.
- 4- Filling the mold with the mixture and leaving it for 24 hours in order to consolidate.
- 5- Sheets specimens were taken from the molds after curing for 24 hours and cut into tensile and creep specimens with the required dimensions as in Fig. 3.5 and Fig. 3.6. The mechanical properties were measured at room temperature.

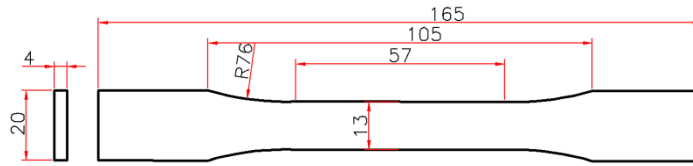


Figure 3.5: The dimensions specimen tensile test.

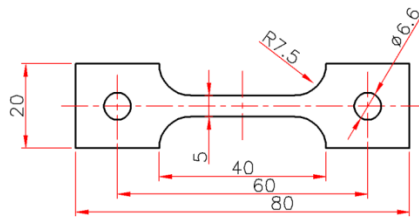


Figure 3.6: The dimensions specimen creep test.

- 6- The resulting plate was placed into an oven with a set temperature of 50 °C for one hour to ensure that full cure was achieved.
- 7- Tensile and creep specimens were cut using CNC machine to cut the specimens into the desired dimension
- 8- Composite specimens were prepared by adding E-glass (Chopped-strand mat contains randomly distributed fibers) using hand layup method in two types of reinforcing; one layer and two layers types.

3.8 Mechanical Test

To understand the effect of fibers on the behavior of polymeric materials, various mechanical tests have to be done. This evaluation enables the user to decide which is the most suitable combination of the three materials to be employed in the desired application. The mechanical tests done in this research include:-

3.9 Tensile Test

The tensile test was fulfilled using tensile test apparatus type (TENSION) that available in mechanical laboratory in the University of Aeronautical association / Institute of Science and Technology. The specimen dimensions were initially measured (width, length and thickness), as shown in Fig. 3.5. The surface of the specimens is cleaned from any dirt. The tensile test measured the tensile strength (σ), strain (ϵ), elongation, and Young modulus (E) were calculated. Strain was measured using extensometer with free length 50mm. Loading rate was (1 mm/minute). For all the tensile tests at least 5 specimens were tested per condition. Some case more than 5 specimens. Specimens before and after testing are shown in Fig. 3.7. The specimen it is manufactured according to ASTM D638 standards.

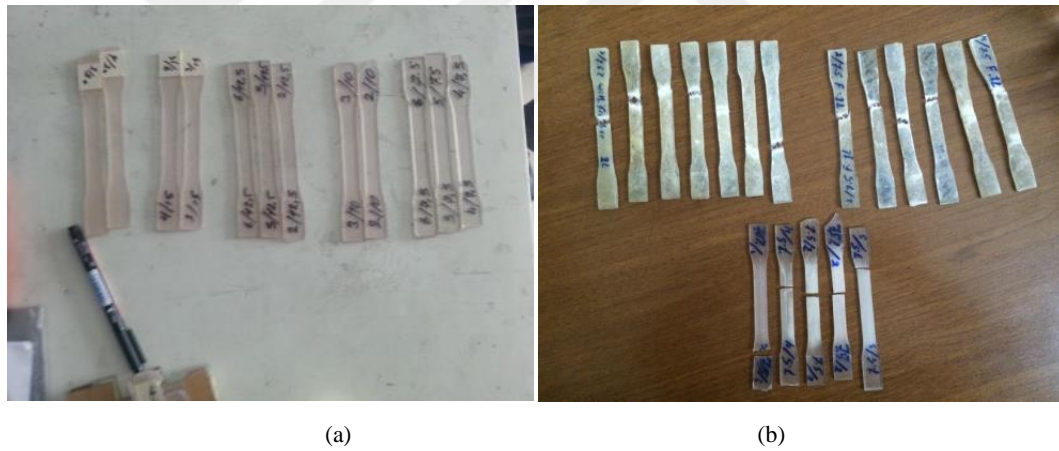


Figure 3.7: The broken specimens (a) after Tensile test (b) before testing.

3.10 Creep Test

By using Gunt WP600 creep testing machine, at Engineering Technical College- Baghdad, it is possible to demonstrate the typical phenomena of creep responses, such as periods of different time dependent creep rate, temperature dependent creep behavior or viscoelastic performance. The constant load was applied on the test specimen in a visible manner using sets of weights. The specimen it is manufactured according to ASTM standards.

Short-term creep tests of 120 minute creep testing followed by 120 minute recovery were conducted. Tests were performed at three load levels between (5-7) MPa in increment of 1N at room temperature. The test is conducted according to ASTM 2990 standard and fulfilled at room temperature Fig. 3.8.



Figure 3.8: Gunt WP600 Creep device.

3.11 Hardness Test

Hardness device used is shore D type according to ASTM D2240 as shown in Fig. 3.9.



Figure 3.9: Shore (D) Hardness instrument.

3.12 Microstructural Characterization

Characterization and/or morphology of the individual composite components and blend- blend with fibers composite were examined by:-

3.12.1 Scanning Electron Microscope (SEM)

A low voltage JEOL scanning electron microscope (JSM-5800, Japan), Fig. 3.10, was used to examine the fractured surfaces of the samples.

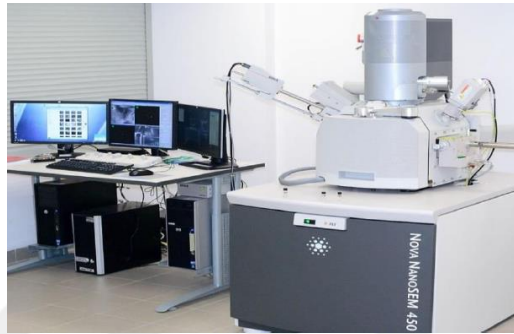


Figure 3.10: Scanning Electron Microscope (SEM) device.

CHAPTER FOUR

RESULTS AND DISCUSSION

4.1 Introduction

Blending of polystyrene with another polymer especially UP, and adding fiber to the blend is an important issue. This chapter explore and discuss the results obtained regarding tensile strength , hardness and creep tests for both PS and UP, and their composite which is reinforced with E-glass fiber . Morphologies of the fractured surfaces as examined by a scanning electron microscope are also discussed. Also the effect of loads on the viscoelastic behavior of materials, in terms of creep behavior, stress relaxation, at constant temperature (room temperature) was investigated.

4.2 Influence of Polystyrene and E-glass Fiber Additives on Tensile Behavior

4.2.1 Effect of Polystyrene Addition to UP on the Behavior of Mechanical Properties by the Tensile Test

The effect of polystyrene additive to polyester led to a decrease in tensile strength accompanied by an increase in strain, Fig. 4.1. This is because polystyrene is a thermoplastic material which increases elongation of the matrix when compared with polyester which is of thermosets group. Tensile strength of unsaturated polyester (UP) with different percentages of Polystyrene (PS) was investigated. The results showed that the addition of polystyrene to polyester in a weight percentage of 5% led to a decrease in tensile strength from (61MPa) to (45MPa), and with (7.5%) blend, the tensile strength decreased from (61MPa) to (35MPa) , as well as, changed the stress-strain curve behavior from brittle to ductile , depending on the polystyrene additive percentage.

Fig. 4.1 shows two different stages of deformation, the elastic region, and plastic region. The elastic region is characterized by a linear pattern with steep rise in stress at relatively small strains; this is clearly demonstrated for pure UP. The plastic region is characterized by an increase in elongation of blend when there is an increase in the

weight percentage of the PS, as 10%, 12.5%, 15%, and 20%. Worth to note that there is a varying behavior in the tensile test curves of the PS and the blends.

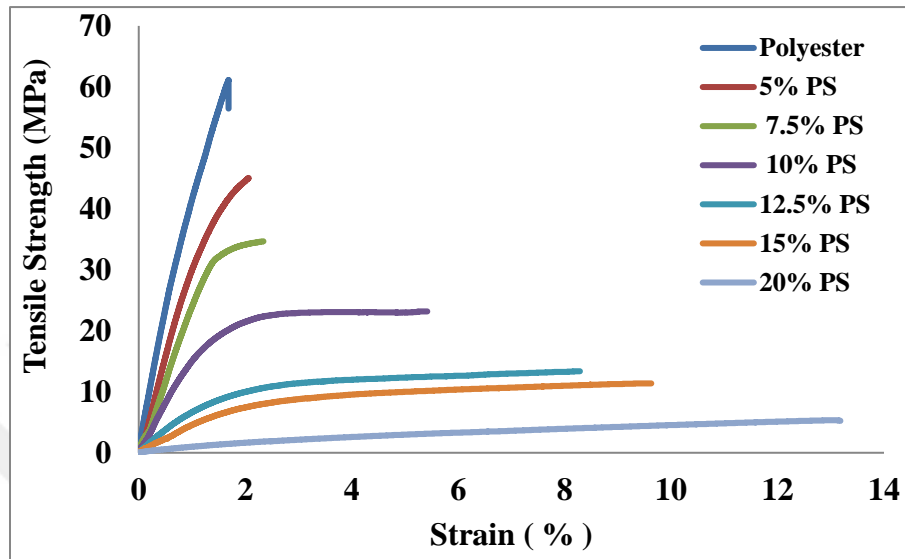


Figure 4.1: Stress - Strain curve of pure UP and blend (UP + PS)

The tensile strength of blend 7.5% polystyrene is 35 MPa which is higher than that of the other blends except 5%. The tensile strength of blend of 5%, 7.5%, 10%, 12.5%, 15% and 20% polystyrene is lower than that of UP matrix by (26%), (43%), (62%), (78%), (81%) and (91%) respectively. On the other hand, the strain at break increases with the increase in polystyrene, so the strain of blends of 5%, 7.5%, 10%, 12.5%, 15% and 20% polystyrene is more than the strain of unsaturated polyester matrix by (22%), (38%), (69%), (79%), (82%) and (87%) respectively Fig. 4.2. On the other hand, the Fig. 4.3 shows that the polystyrene-polyester blend has a lower elastic modulus and tensile strength. This is clearly demonstrated for blend with high PS percentage. The modulus of elasticity of UP is 4.33 GPa, while, the modulus of elasticity of blend 5%, 7.5%, 10%, 12.5%, 15% and 20%, is lower by 23%, 40%, 66%, 70%, 97% and 99% respectively. The drop is regular and it refers to the increasing softness of the blends. Moreover there is an increase in elongation with increase in PS percent associating with decreasing in the elastic modulus substantially.

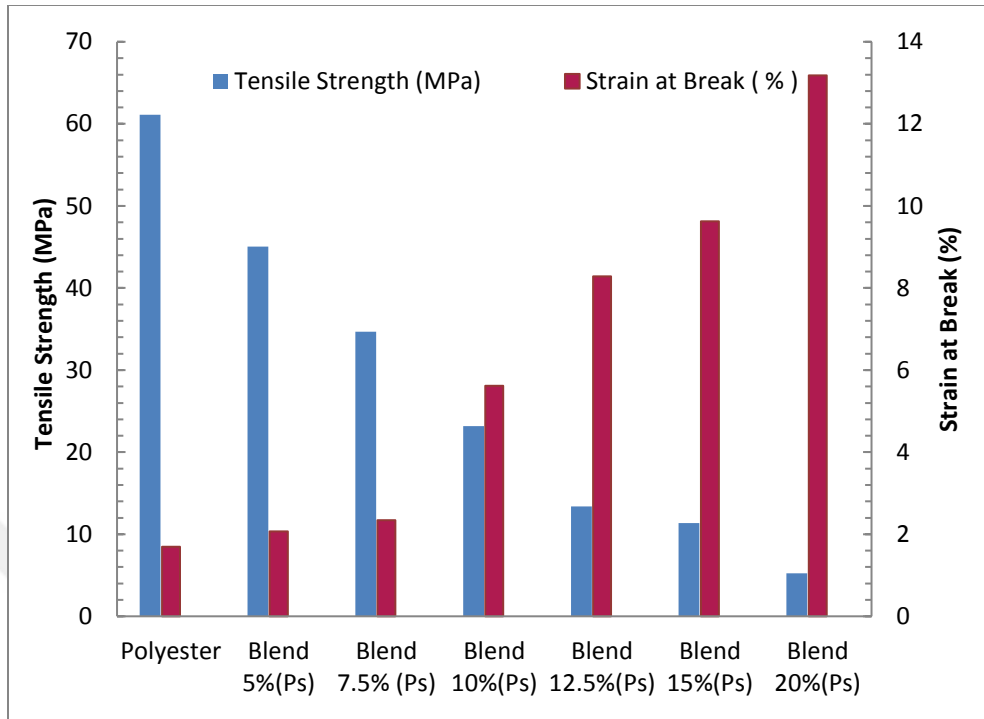


Figure 4.2: Strain at break and tensile strength of blends.

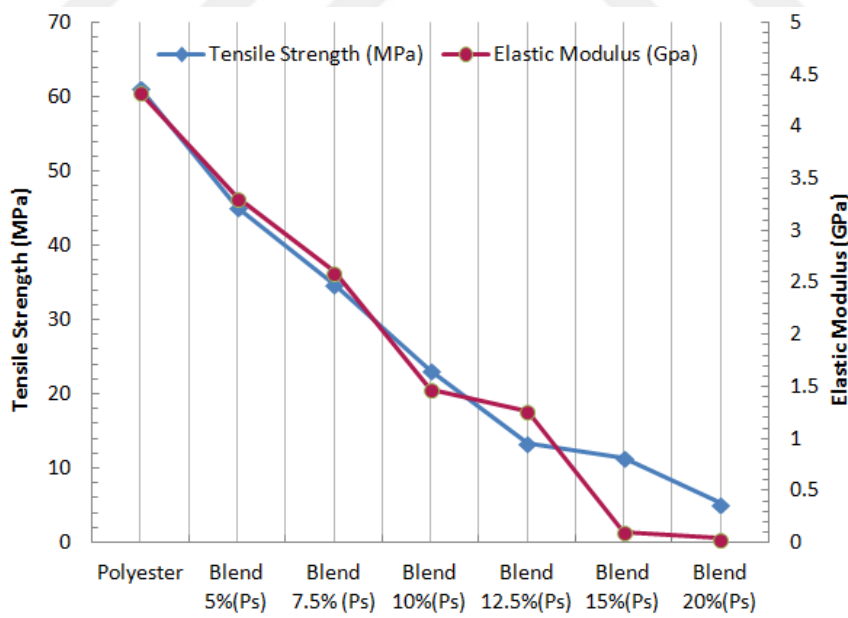


Figure 4.3: Effect of PS % on the elastic modulus and tensile strength.

The 7.5% of PS-blend was selected for the reinforcement because it achieved acceptable reduction in stiffness with suitable increase in elongation. An addition of more than 7.5% of PS reduces the stiffness of UP resin and makes it behave as a ductile material. When the PS content is increased above the selected ratio, the mechanical

properties severely decrease. This behavior may be related to the increase in fraction of thermoplastic in thermoset, which leads to an increase in dispersing phase of thermoplastic in the thermoset rich matrix, hindering the cross linking of the thermoset with hardener by their decreasing the total strength of the system, as shown in Fig. 4.4.

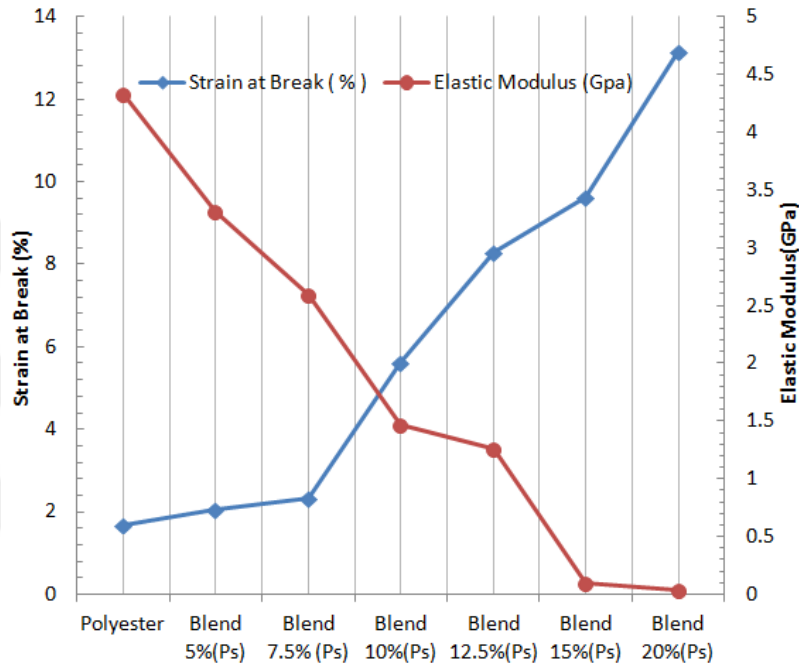


Figure 4.4: Effect of PS% on the strain at break and elastic modulus.

4.2.2 Influence of Fiber Addition on Mechanical Properties of Polyester and Blend (Polystyrene-Polyester)

The addition of chopped E-glass fiber to the blend shows a significant effect on tensile properties Fig. 4.5. The reason could be attributed to the ability of chopped glass-fiber mat to absorb more ester monomers that lead to increase plastic content [9]. The addition of polystyrene to polyester to form a blend 7.5% led to a decrease in tensile properties, toughness and hardness, and this is true because polystyrene is characterized by its high elongation property, if compared with UP, and this gives the blend the desired flexibility. This has been proved by the increase in strain percentage at break (38%), and reducing the modulus of elasticity (40%) as well as lowering toughness, which was calculated from the area under the curve (tensile – strain). The tensile strength of the UP

is 61 MPa, and when polystyrene is added with 7.5%, the tensile strength decreased to 35 MPa. When one layer of fiber is added to reinforce the blend, the tensile strength increased again to reach 59 MPa, and to 77 MPa when two layers is added. This means that the improvement in tensile strength in case of adding one layer of fiber is (68%) compared with the blend alone, while the reduction is approximately (3%) comparing with UP. In the case of blend reinforced with two layers of fiber, the tensile strength is improved by (120%) compared with the blend alone, and the total improvement in its tensile strength in relation to the UP is (26%). This difference in the rate of improvement between the two types of layers is due to weight ratio of fiber added to the blend 7.5% Fig. 4.6. With single layer, the ends of fiber act as stress concentrators and encourage the bond between fiber and matrix to break, while with two layers, the matrix is sufficiently restrained and the stress is more evenly distributed and overcomes the effect of the stress concentrations. The net result is the increase in tensile strength, toughness and elastic modulus. Similar observations are reported in other fiber reinforced polymer systems [64, 65].

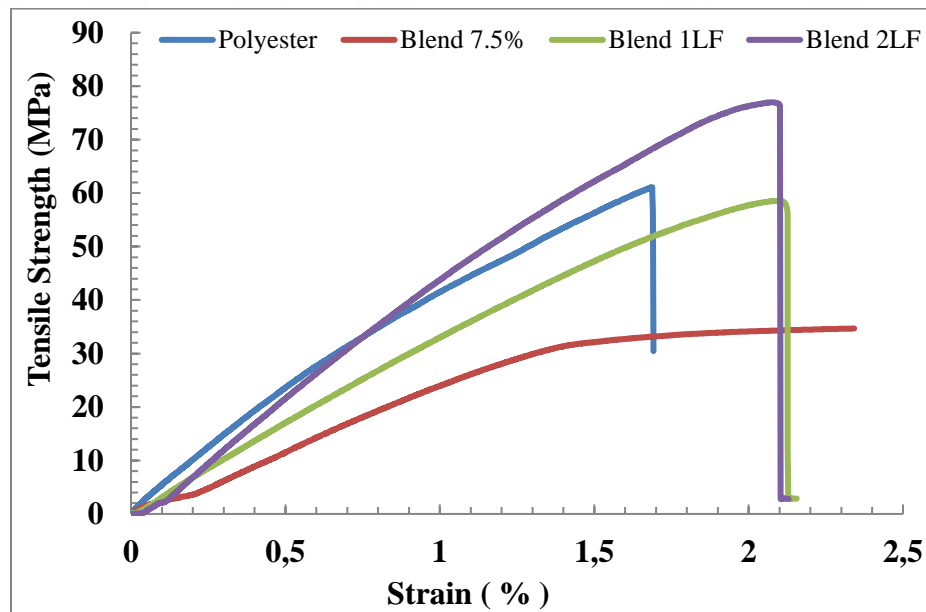


Figure 4.5: Effect of Fiber Addition on Stress-Strain curve of Blend and Composites.

The modulus of elasticity for the blend (7.5%) was decreased by 40% compared with the UP. While it is increased by 22% and 72% for blends with one layer and two layers respectively. But if it is compared with UP, the increment with two layers of fibers

is 3.2%. So the rate of decline between the UP and the blend is less than the rate of the increase between the blend and the fiber-reinforced blend Figures 4.7 and 4.8.

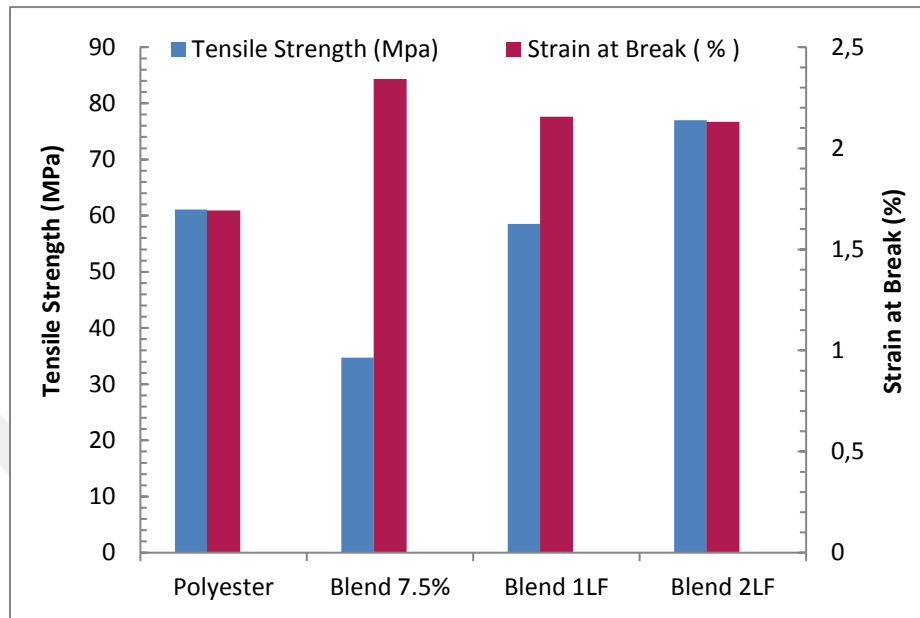


Figure 4.6: Effect of fiber addition on tensile strength and strain at break.

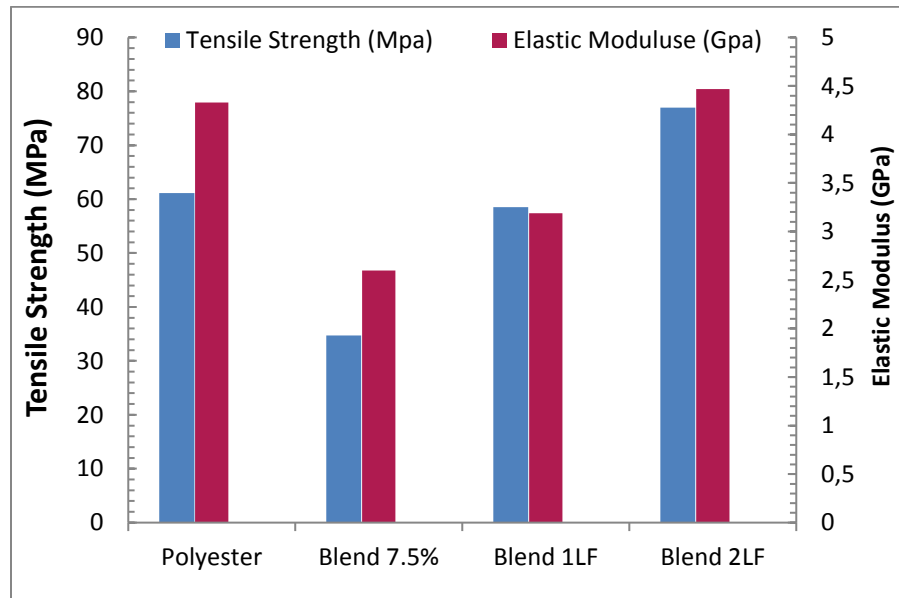


Figure 4.7: Influence of fiber additives on Modulus of elasticity and tensile strength

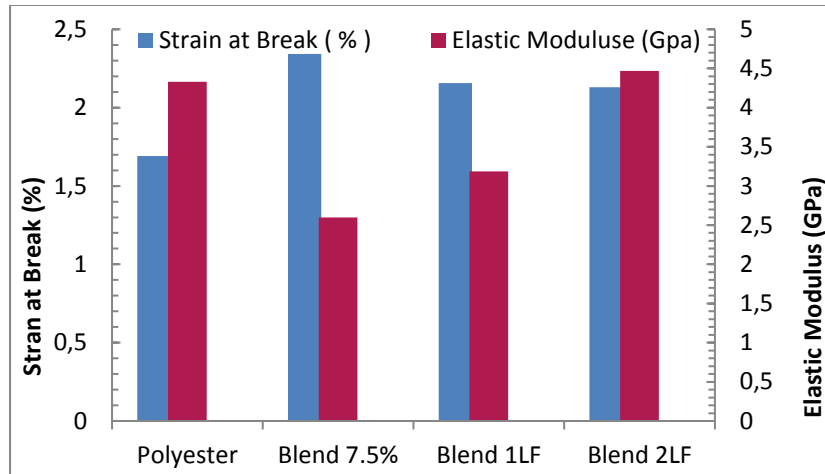


Figure 4.8: Effect of fiber addition on strain at break and modulus of elasticity.

4.3 Effect of E-glass Fiber Reinforced Addition

The modulus of toughness which was calculated from the area under the curve (tensile-strain) increased for two fibers layers by (66%) and (55%) when compared with 7.5% blend and polyester respectively, and by (30%) and (21%) for one layer compared with 7.5% mixture and polyester respectively in Figures 4.9 and 4.10.

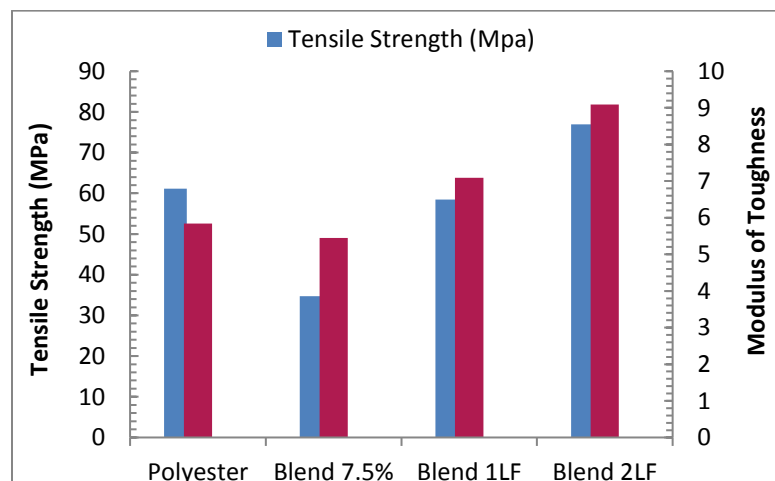


Figure 4.9: Effect of Fiber Addition on the Modulus of Toughness and Tensile Strength

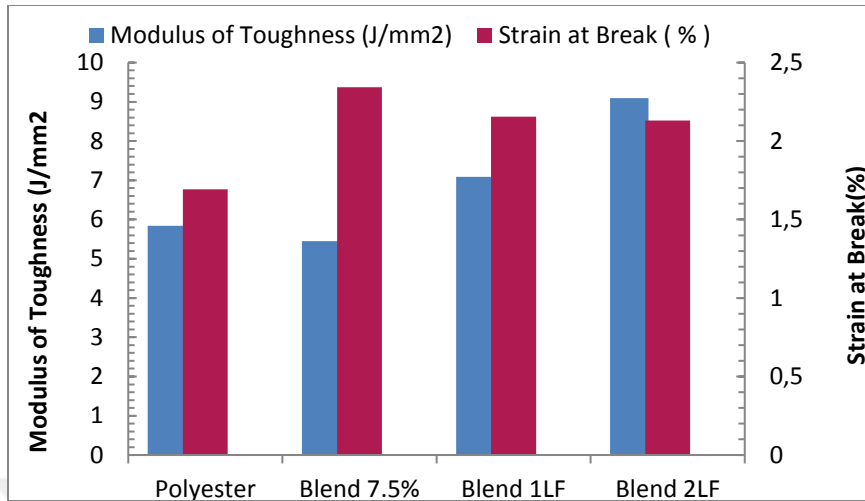


Figure 4.10: Effect of Fiber Addition on the Modulus of Toughness and Strain at Break.

The hardness of the unsaturated polyester decreases with an increase in polystyrene content, Fig. 4.11. This is due to some of the polystyrene being dissolved in the unsaturated polyester phase and plasticizing the system. Moreover, incorporation of polystyrene, which has little resistance to penetration, will create domains of polystyrene particles between the cross-links which causes a reduction in the cross linking density [66, 67]. So the addition of Polystyrene to Polyester affects the hardness property of the composite material, which does not depend only on the properties of components, but also depends on the nature of the interface between them and the weight percentage of additives [68].

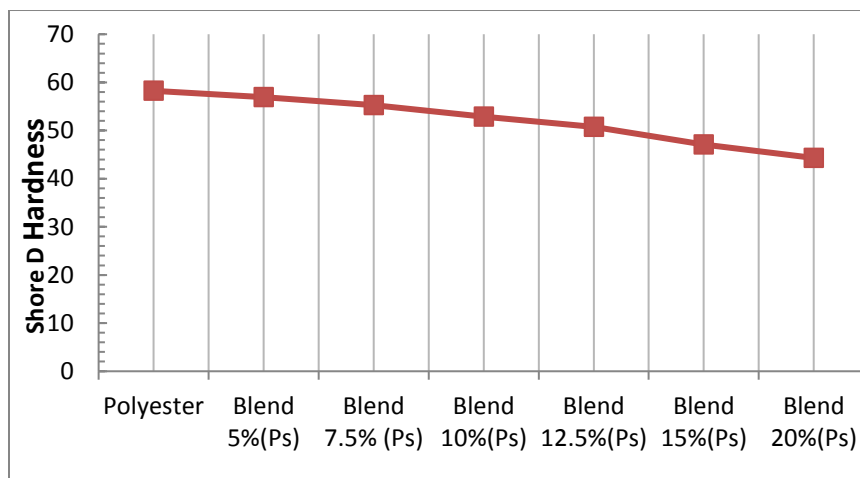


Figure 4.11: Polystyrene Effect on Hardness of Unsaturated Polyester.

The reinforcement of blend 7.5% with glass fiber has a great role on hardness. Fig. 4.12 illustrates that the hardness of blend (7.5%) increases with increasing glass fiber percentage; this is due to the increase in material resistance against the plastic deformation. Results reveal that the hardness of blend 7.5% reinforced with one and two fiber layers was 57 shore D and 60 shore D, respectively, which was higher than the hardness of blend 7.5% alone 55 shore D. The main reason of slight increment in hardness of composites that fiber is harder than the matrix. Generally speaking, the addition of fiber mat to polymer matrix causes a significant increase in the mechanical properties, especially modulus of elasticity and hardness and toughness of the composites if the reinforcing fibers are strongly bonded to the polymer matrix [69].

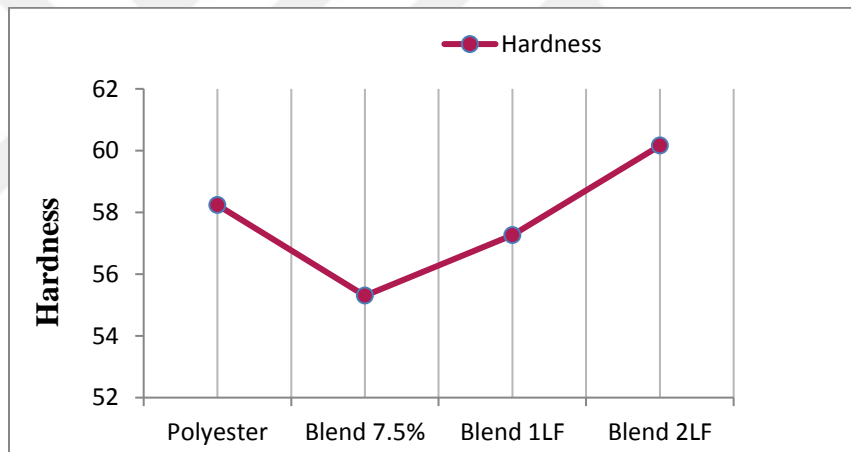


Figure 4.12: Effect of Fiber Reinforced on Hardness of the Blend Composite.

The decline of the hardness of blend (7.5%) is (5%). After added fiber layers , the hardness recording an increase for blend 7.5% reinforced with a single layer and two layers by (3.5%) and (8.8%) respectively as shown in Figures 4.13 and 4.14.

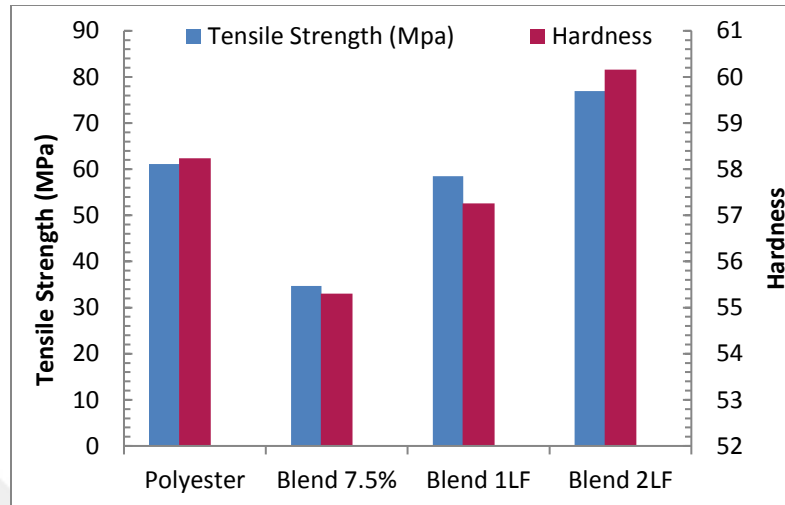


Figure 4.13: Influence of Fiber Addition on Shour D Hardness and Tensile Strength

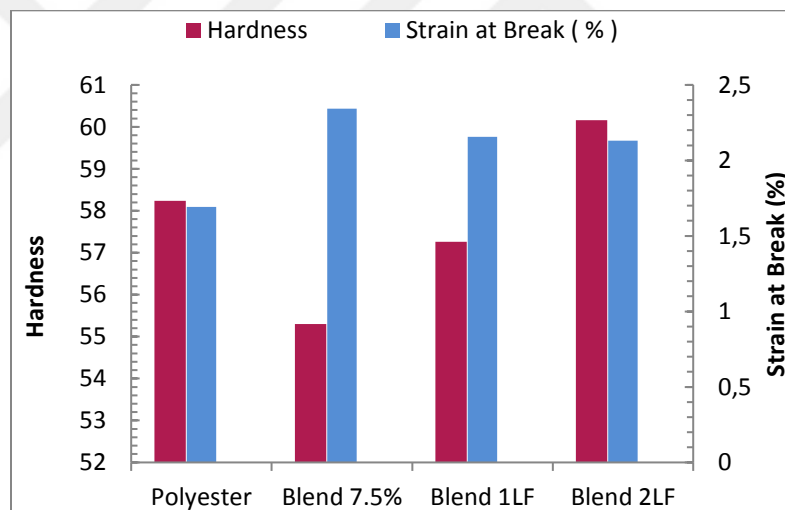


Figure 4.14: Effect of Fiber Addition on Strain at Break and Hardness.

4.4 Fracture Surfaces Morphology

The fractured surfaces of tensile specimens were examined using Scanning Electron Microscope (SEM). The fractured surface of unsaturated polyester is smooth, rippled and glassy Fig. 4.15. Irrespective of the few shear deformation lines in the fractured surface, the relative smoothness of this surface indicates that there is no significant plastic deformation had occurred. Also, the same behavior has been reported

by Cs. Varga et al [2]. The brittle fracture causes ripples, and accounts for a poor impact strength of the network, while there is no energy dissipation mechanism operating here, as mentioned by [70].

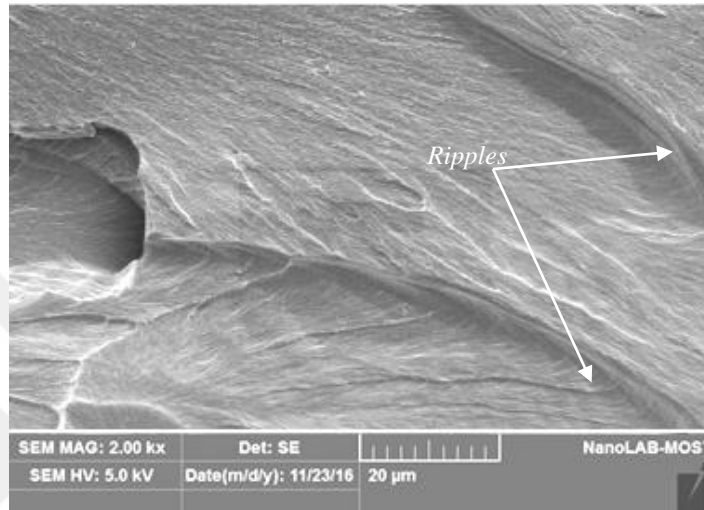


Figure 4.15: Fractured Surfaces of the UP Material

For unsaturated polyester blended with 7.5 wt% of polystyrene, Fig. 4.16, the particles are distributed in a homogenous pattern throughout the matrix, which is responsible for the lower rate of growth of the crack in these specimens that show the presence of relatively large number of deformation lines. The micrographs show broken polystyrene particles, specially the small size, this is due to the polystyrene particles has less resistance to the applied load, Fig. 4.17.

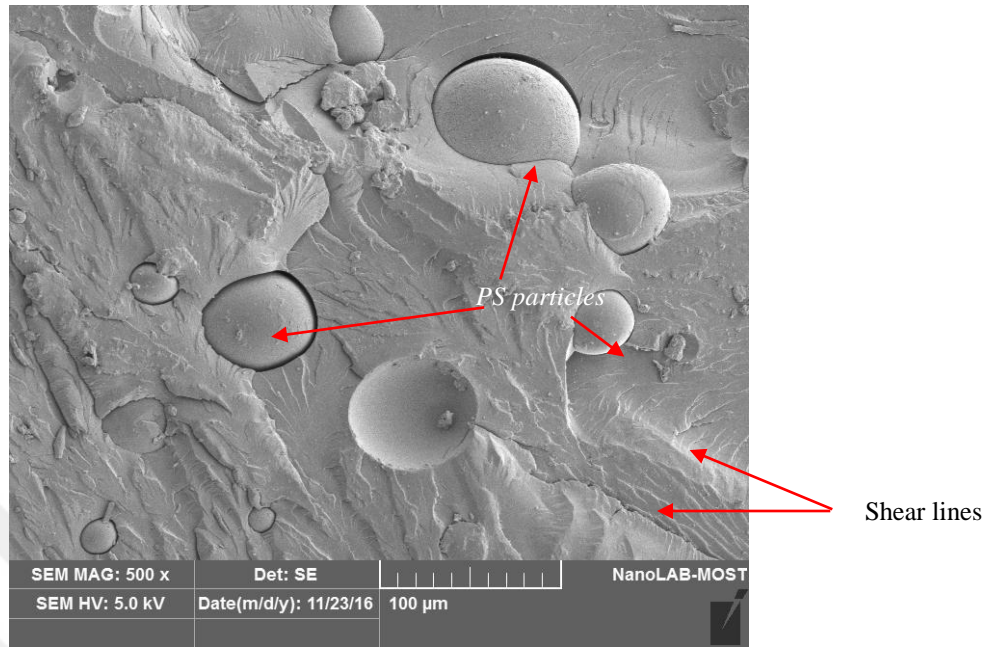


Figure 4.16: Shear lines, Fracture surface of Blend (UP- PS 7.5%).

Also, the fracture surfaces in these specimens, compared to pure unsaturated polyester, are not so smooth, indicating a ductile pattern of fracture. According to Yee and Pearson [71], the amount of deformation lines or the size of initiation crack zone is proportional to the increase in toughness of the material. The relative distortion in the shape of polystyrene domains in these cured resin matrices is thought to be due to more plastic deformation. Lines of deformation are propagated through polystyrene domains; promoting the transfer of stress between the polystyrene particles and unsaturated polyester matrix Fig. 4.17.

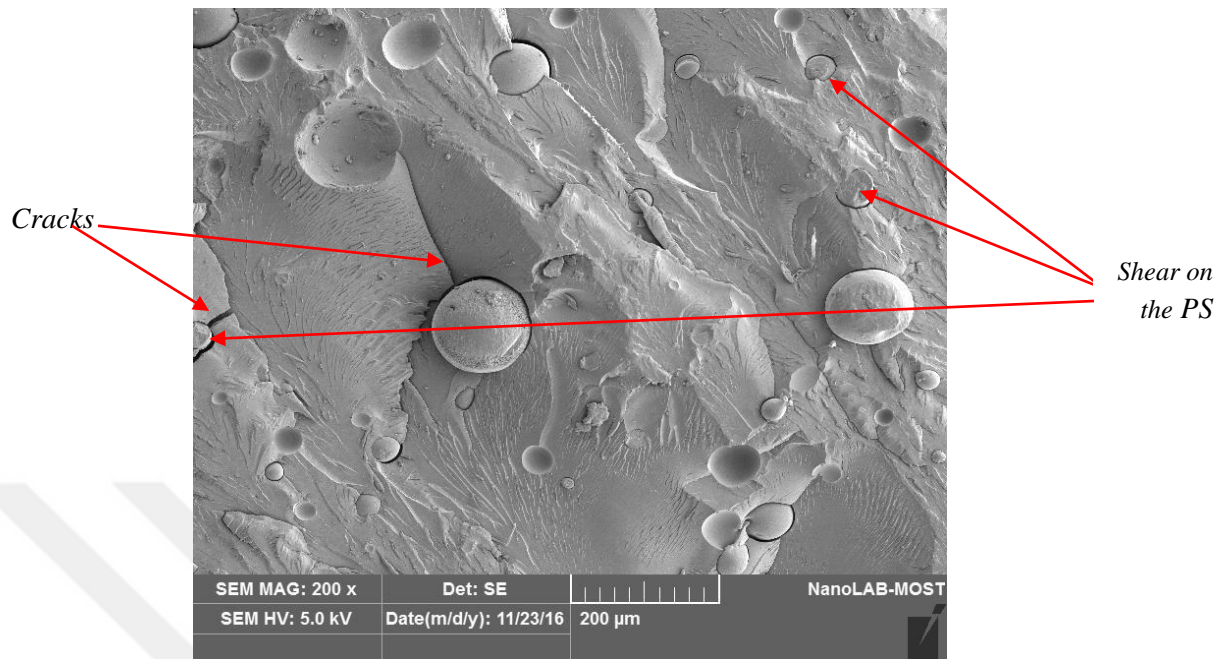


Figure 4.17: Fracture surface of Blend (UP- PS 7.5%).

The homogenous distribution of the smaller spherical shaped particles of polystyrene in the polyester resin matrix is necessary for operating the yielding process throughout the matrix, and explains the toughening mechanism of polyester. This morphological pattern is actually responsible for the enhanced damping nature and the highest impact strength of the samples [72]. These particles of polystyrene in such systems act as stress concentrators, and on applying force, during failure, energy will be transferred through these particles and dissipate in due course. Thus ultimate tensile stress of the matrix is protected from catastrophic failure.

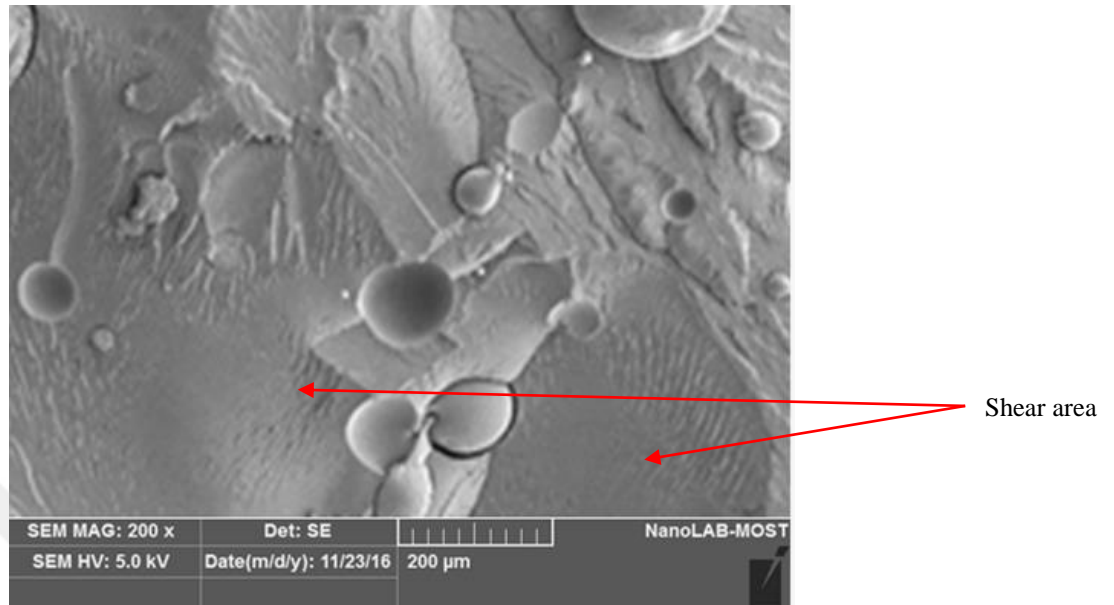


Figure 4.18: Fracture surface of Blend of 7.5 wt %.

Practically, short fiber-reinforced composites mostly do not fail by fiber fracture, but break by either matrix failure or interfacial de-bonding. When the interfacial bonds between the fiber and the polymer matrix are weak, the interface fails and the fibers will dislocate and pull out of the matrix. While, if interfacial bonds are strong enough, and the interaction of fiber ends can produce relatively high local stresses in the adjacent matrix, failure will occur within the polymer matrix, resulting in a matrix initiated fracture mode. In either of the two events, whether interfacial bond or matrix failure, premature failure of the composite will occur at relatively low strains.

Examining the fracture surfaces of the fiber-reinforced blend by SEM Fig. 4.19, reveals that these materials fail by some mechanism other than fiber fracture, either by matrix failure or interfacial bonds failure.

The more highly filled material fails at relatively low strains and exhibit brittle failure. By SEM examination of the fracture surface of these samples, it's evident that there is Extensive fiber dislocations and little matrix drawing , which indicate that the failure of the fiber-matrix interface preceded fracture of the composite matrix.

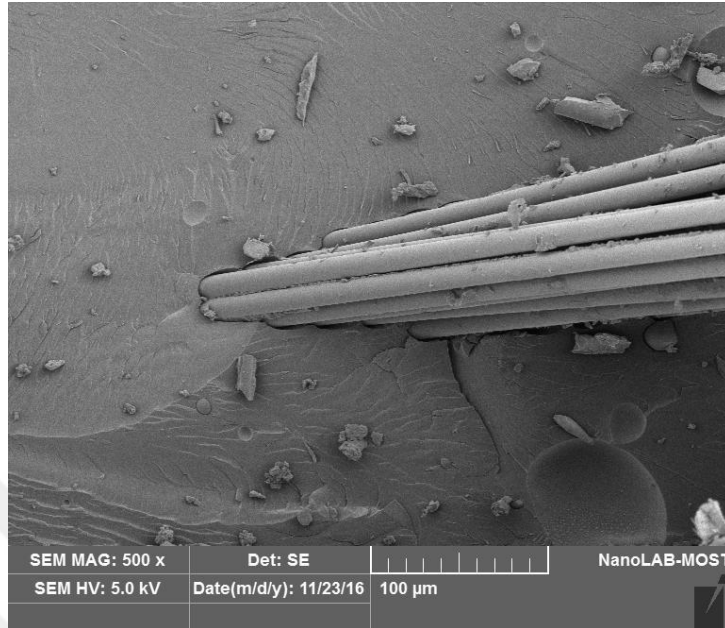


Figure 4.19: SEM image of affect Fracture surface showing Fiber/Blend.

From Fig. 4.20, the fibers are homogenously distributed throughout the matrix which act as stress centre and exhibit highest impact strength than a blend of unsaturated polyester , also we see that the fractured surface is smooth which indicate that the blend has suffered shear stress while the glass fibers were tolerating the applied load , therefore multiple holes on either side of the fracture surfaces of the specimen are obvious ,which represents dislocation sites of the glass fibers that are pulled out and remain intact in the other side of the specimen

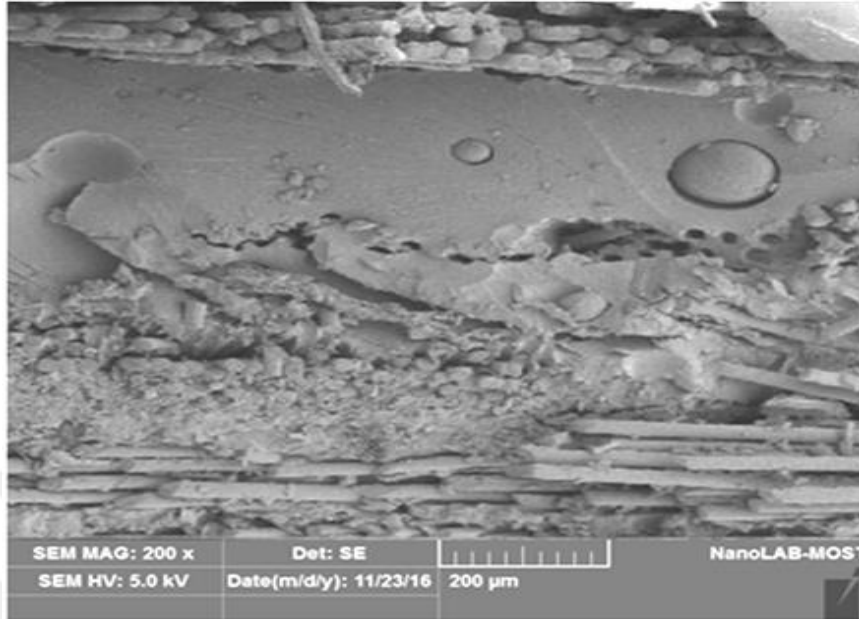


Figure 4.20: SEM image of affect Fracture surface showing Fiber two layers/Blend 7.5 wt %.

4.5 Viscoelastic Behavior of Blend and Composite

In this part, the results of the experimental work are analyzed and discussed according to the principle of the viscoelastic behavior in terms of creep behavior, creep compliance, stress relaxation.

The viscoelastic behavior was studied for (UP, 1LF and 2LF) at room temperature and different stresses (5, 6 and 7 MPa). The specimens are bonded by two materials; the first one is unsaturated polyester, the other is a composite of blend with chopped glass-fiber mats (one and two layers). The percentages of blend used are (7.5 wt%), All of these tests done under the same optimum conditions.

4.5.1 Unsaturated Polyester (UP)

The creep test results are shown in the Fig. 4.21, which shows the creep stages, as instantaneous deformation, primary creep, and unfinished secondary creep processes. The tertiary stage, however, is not shown here because the creep failure or rupture was not considered under the current condition under which it would take a very long period

of time to make the material failure, instead of that, a recovery stage was conducted to evaluate the retained strain, as shown in Fig. 4.21. Normally, increasing the stress gives an increase in the strain values through the time of test. The creep behavior of polyester increase proportionally when stress increases and temperature is constant.

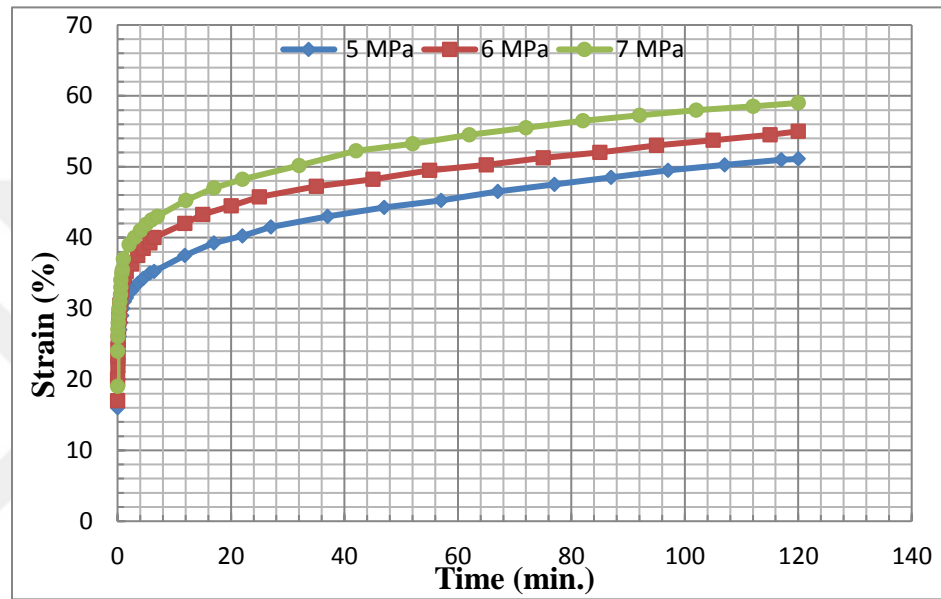


Figure 4.21: Creep behavior at temperature ($T=30^{\circ}\text{C}$) of standard specimen for material Unsaturated Polyester.

From the experimental results which are shown in Fig. 4.21, it's noted that the relaxation stress is similar to the creep resistance. It is clear from the curves of creep strain and the stress relaxation modulus versus creep time of the tested samples obtained at constant applied stress of 5, 6 or 7 MPa which gives linear behavior tested at room temperature. The increase in applied stress at room temperature causes increase in values of instantaneous extension (Maxwell element), creep Kelvin element, creep Maxwell element, as well as causes increase in total strain, as shown in Fig. 4.22.

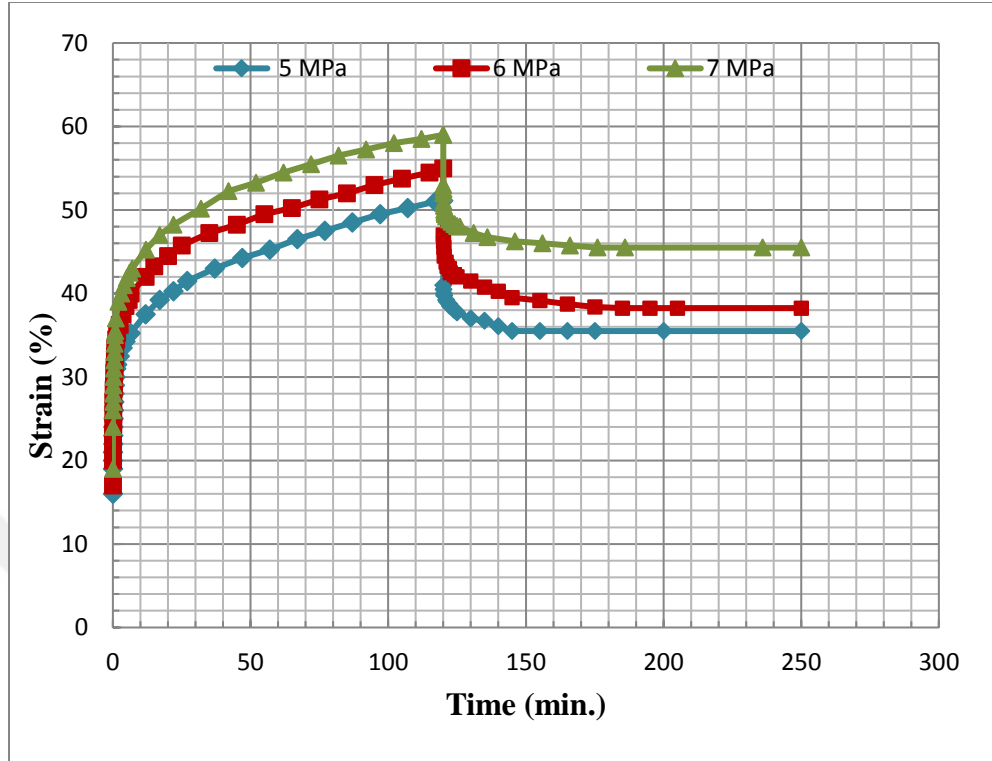


Figure 4.22: Creep and creep recovery behavior of UP different applied stress at 30 °C.

Creep stages are clearly shown in the figures as instantaneous deformation, primary creep, and unfinished secondary creep processes. The tertiary stage is, however not observed here because the creep rupture or failure is not considered under the current condition, under which it would take a very long period of time to make the material failure, instead of that, a recovery stage was conducted to evaluate the retained strain, as shown in Fig. 4.23 [73].

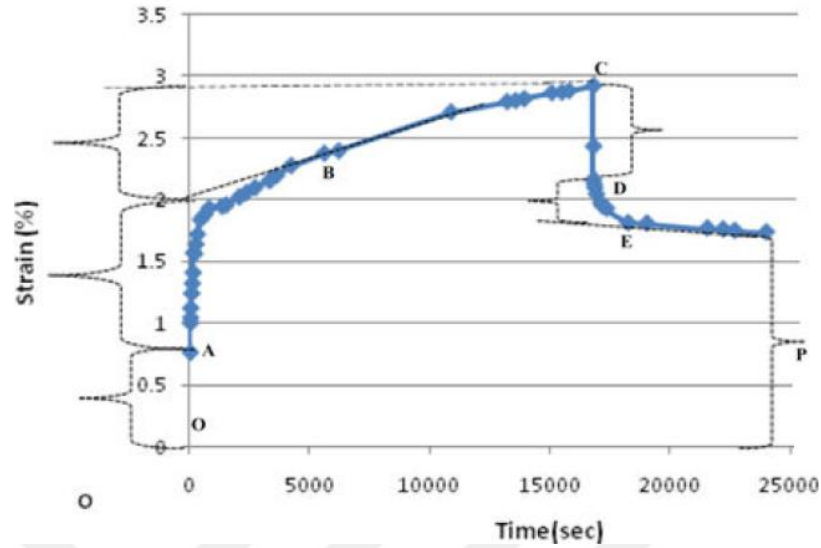


Figure 4.23: Creep Curve Parameters [73].

Where:

OA: Instantaneous extension (Maxwell element)

AB: Creep Kelvin element

BC: Creep Maxwell element

CD: Instantaneous recovery, Elastic relaxation (Maxwell element)

DE: Delayed recovery, Plastic relaxation (Kelvin element)

P: Permanent deformation

The resulting deformation tends to increase with time as entangled chain segments undergo conformational changes in response to external stress. Viscoelasticity refers to molecular rearrangement, when a viscoelastic material like a polymer is subjected to a stress, parts of the long chain of the polymer will change its position, even though, these polymers will remain solid, in order to accompany the stress, and as this occurs, they create a back stress in the composite. As this back stress has the same magnitude as the applied stress, the material will no longer creep. As soon as the original stress is taken away, the accumulated back stress will cause the polymer to return to its original form. The material creeps, given the prefix viscous, and when fully recovers, given the suffix elasticity [53,74]. The instantaneous recovery (Maxwell element) increases with increasing applied stress. The total strains at time 7200 second and permanent strains are

more pronounced and increase with increasing applied stress, compared with instantaneous extension and instantaneous recovery, Fig. 4.24.

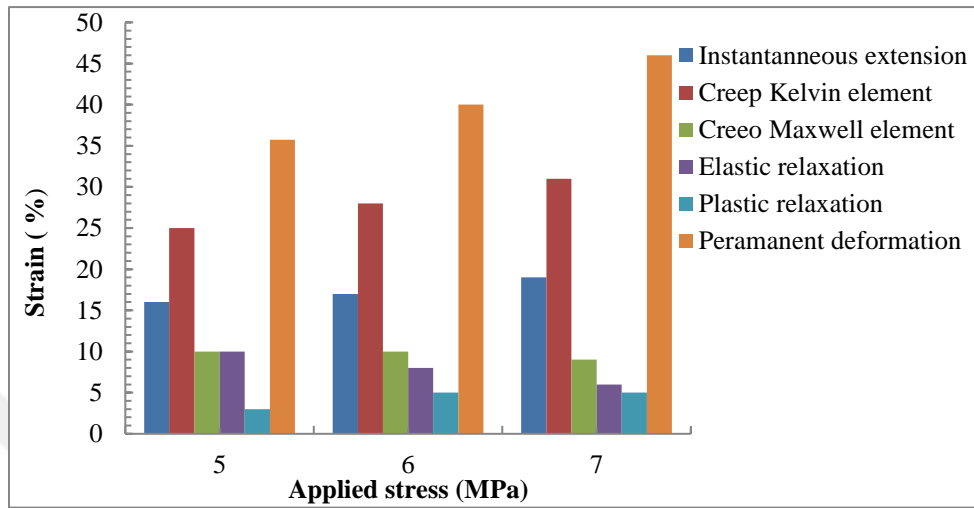


Figure 4.24: Variation of Creep Parameter of UP with Applied Stress, at room temperature.

Because polystyrene increase the ductility of the UP matrix this limits the application areas in which they can be used. The approach taken to overcome this problem is the addition of polystyrene and glass fiber to UP, aiming to develop an improved matrix material obtaining good ductility without reducing the other desired mechanical properties of the UP matrix, like toughness.

The major cause for creep of fiber reinforced polymer comes from creep of polymer matrix, creep of glass fiber considered insignificant [75]. UP and blend exhibits more amount of creep compared with E-glass reinforced blend. Fiber glass reinforced blend not only had lower instantaneous deformation, due to the high stiffness of glass fiber, but also shows a smaller creep rate than blend and UP Figures 4.25a and 4.25b. Creep strain increased with increasing stress. The permanent deformation in blend reinforced with two layers of fiber at (5, 6 and 7 MPa) applied stress is 41 strain (%) , which is lower than that of UP (51 strain %) . This implies that the creep behavior is greatly improved by the presence of glass fiber in a 23.6% of improvement.

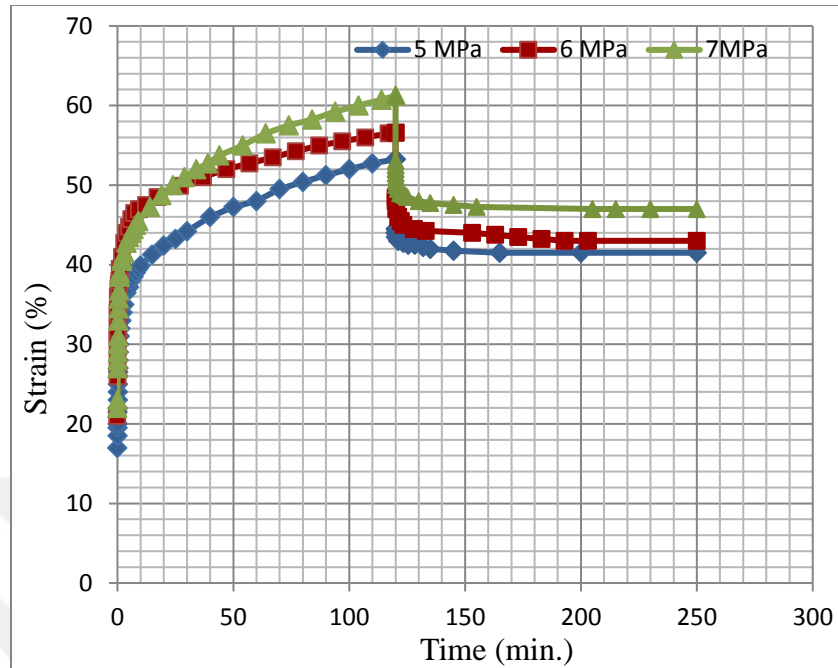


Figure 4.25 a: Creep and Creep recovery behavior of UP and composite reinforced with one layer of fiber, different applied stress at 30 °C.

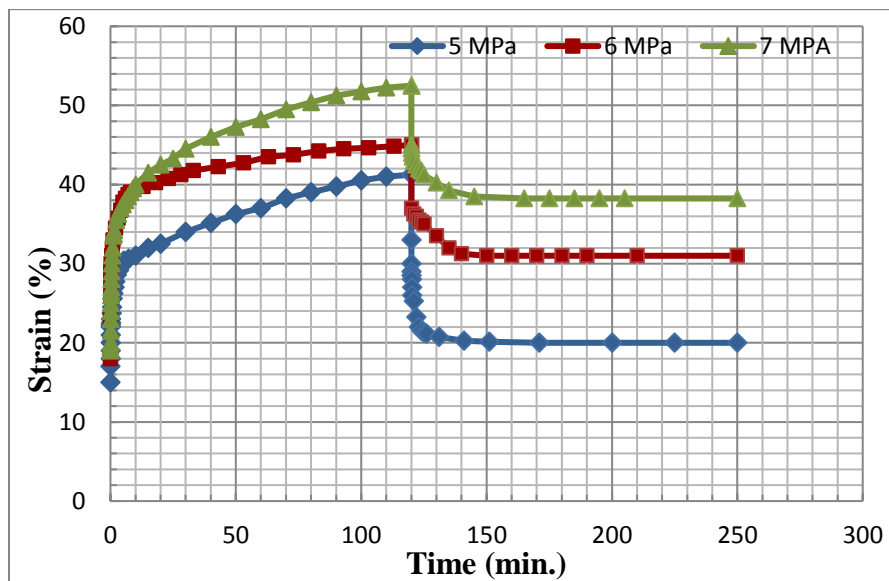


Figure 4.25 b: Creep and creep recovery behavior of UP and composite reinforced with two layers of fiber, different applied stress at 30 °C.

Figures 4.26 a and 4.26 b demonstrate that the creep behavior for blend reinforced with two layers and one layer of fiber is similar to that of the polyester, but the creep resistance is increased in case of blend with two layers in comparison with polyester. The

data in Fig. 4.23 shows that the results are very repeatable. Also there is some scattering in data because of the efficiency of Maxwell model and more scattering is due to the random distribution of fibers in case of the reinforced samples, that may occur due to some chain degradation in materials [76].

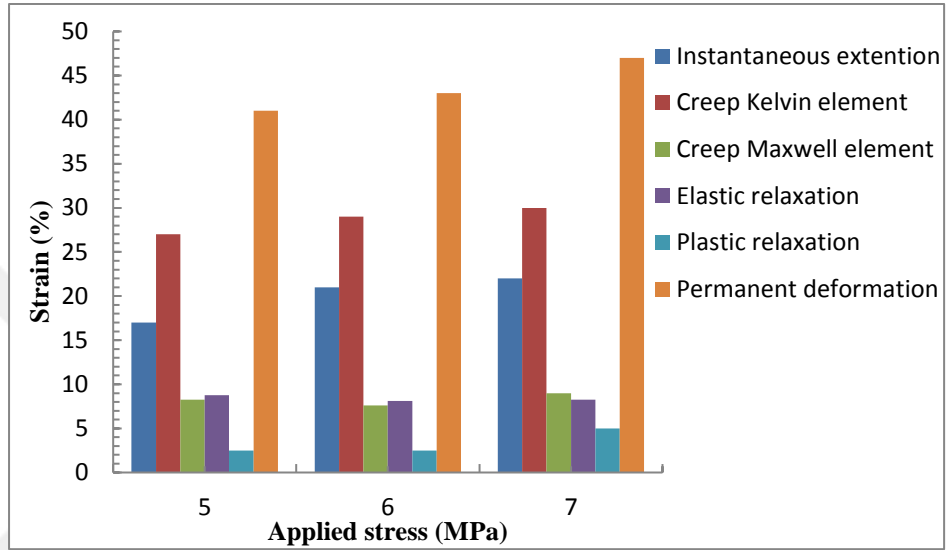


Figure 4.26 a: Variation of Creep Parameter of One Layer of fiber, different applied stress at 30 °C.

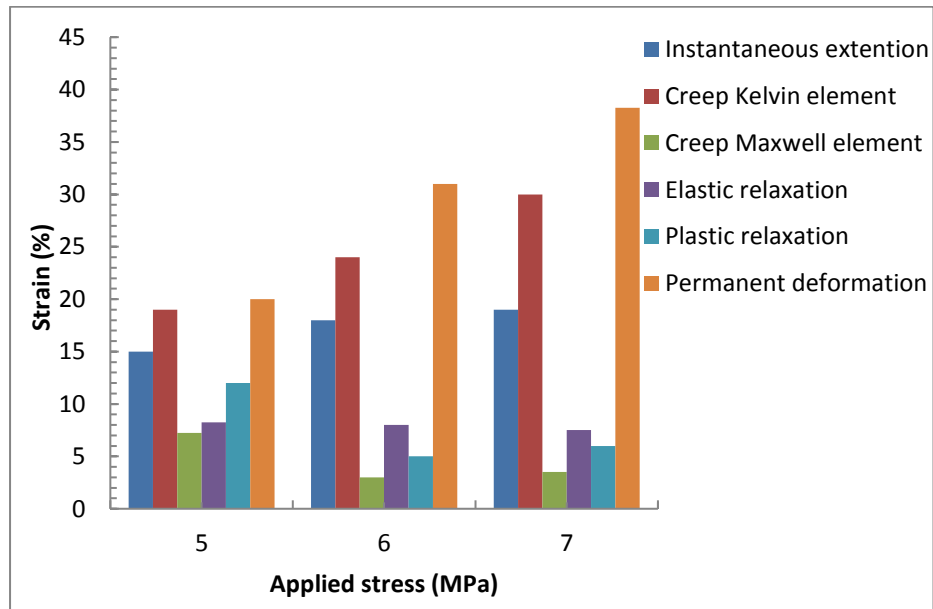


Figure 4.26 b: Variation of Creep Parameter of Two Layers of fiber, different applied stress at 30 °C.

4.6 Predicting Stress Relaxation and Creep Modulus

The theory can be used to estimate the stress relaxation response and creep modulus of real materials. The constants have been obtained from a creep test. The classic way to obtain viscoelastic constitutive model is by using the mechanical analogs. These simple mechanical models are blended together to produce viscoelastic effects. Both Kelvin and Maxwell will be used for predicting the results. Maxwell model is predicting a linear relationship between strain and time, which is most often not the case, while a Kelvin model is predicting creep more realistically than the Maxwell model.

Although Kelvin model is efficient in predicting creep, it is not suitable for estimating the relaxation behavior after removing the stress load. That's why Maxwell model will be used for stress relaxation prediction, and Kelvin model will be used for the prediction of creep modulus.

By this model, when a constant strain is applied on the material, stress will gradually relax. When the applied stress is constant, the strain will have two components. The first one is elastic component which occurs instantaneously, corresponding to the spring, and will relax as soon as stress is released. The other is a viscous component which is growing with time as long as the stress is applied. The Maxwell model predicts that the stress decays exponentially with time, and this is true for most polymers. The only limitation of the Maxwell model is that it cannot predict creep accurately. For creep or constant-stress conditions, this model postulates that the strain increases with time in a linear fashion. However, in polymers, the strain rate will decrease with time [77]. The stress can be estimated by modeling mathematically using:

$$\sigma(t) = \sigma_0 e^{-\frac{\delta_1 t}{\eta_1}} \quad (2.19)$$

Values of the variables (δ_1, η_1) were calculated in which the spring constant (δ_1) would determine the instantaneous reversible deformation whereas the dashpot (η_1) would account for non-reversible deformation. In order to demonstrate the method, a creep curve of UP under 5MPa stress and at room temperature is illustrated and from which the creep rate and initial strain are obtained in Fig. 4.27.

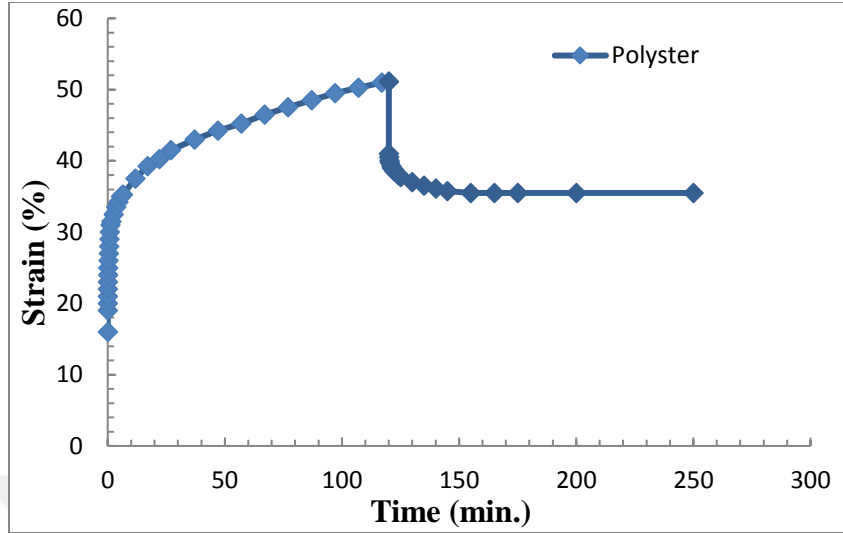


Figure 4.27: Strain Creep curve of Polyester under 30oC temperature and 5MPa stress.

$$\sigma_1 = \delta_1 \varepsilon_1. \quad \delta_1: \text{Spring constant}$$

$$\sigma_2 = \eta_1 \cdot \dot{\varepsilon} \quad \eta_1: \text{Dashpot constant} \quad \sigma = 5$$

$$\text{where: } \sigma = \sigma_1 = \sigma_2$$

$$\delta_1 = \frac{\sigma}{\varepsilon_1}, \text{ Eq. 2.12, } \varepsilon_1 = 0.16 \implies \delta_1 = 31.25 \text{ MPa}$$

$$\eta_1 = \frac{\sigma}{\dot{\varepsilon}} \quad \text{where } \dot{\varepsilon} = \frac{d\varepsilon}{dt} = \frac{0.041-0.355}{250-120} = 4.23 \times 10^{-4} \text{ min.}^{-1}$$

$$\eta_1 = 1.18 \times 10^4 \text{ MN} \cdot \text{sec}/\text{m}^2$$

By using Eq. 2.19, stress relaxation is obtained in Fig. 4.28.

$$\sigma(t) = 5 * e^{0.002644*t}$$

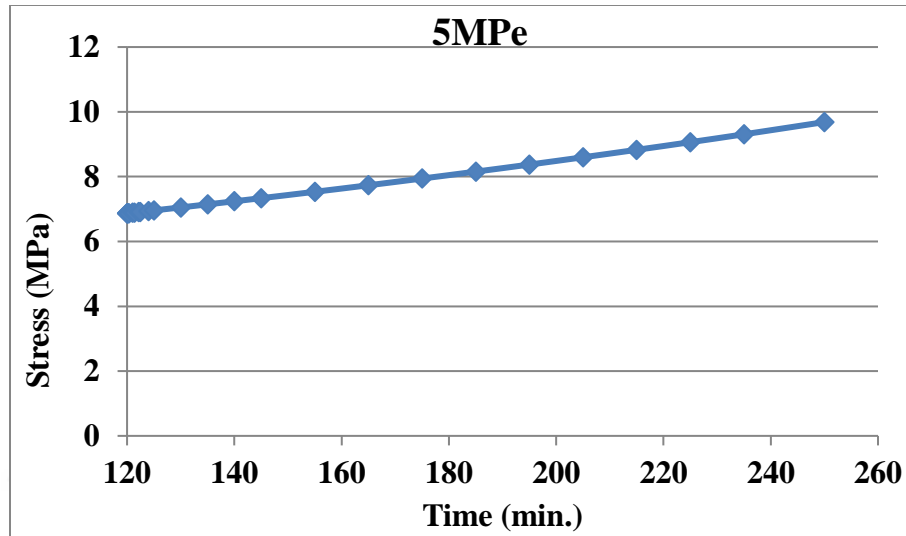


Figure 4.28: Stress relaxation data for UP predicted using Maxwell model

It is worth noting that these results are not considered accurate and for better results, more complex models should be selected (Burger model for example which we adopted in our study , and will be discussed later) and compared with experimental results. From Fig. 4.29, it's noted that when the initial stress increases, the rate and degree of relaxation increases too. While for fiber reinforced one and two layers of fiber material, Figures 4.30 a and 4.30 b, the mechanism of relaxation is more complex and varies with the initial stress applied. Failure mechanisms like crack formation will be initiated in the specimen at lower stress levels which is the case of 5, 6 MPa used in the test. Because the deformation involves a small amounts of fibers, the degree of stress relaxation is relatively low.

When higher levels of initial stress are applied , the failure process will involve the fibers in the composite , and the sample may undergo a crack propagation stage. In this case , rearrangement of the partially failed fibers and a gradual scission of the interfacial bonds may occur in a complicated mechanism [75]. This will cause a high stress relaxation rates, and a high relaxation level at the same relaxation time. Therefore, it is essential to consider the effect of initial stress when comparing the relaxation behavior of composites.

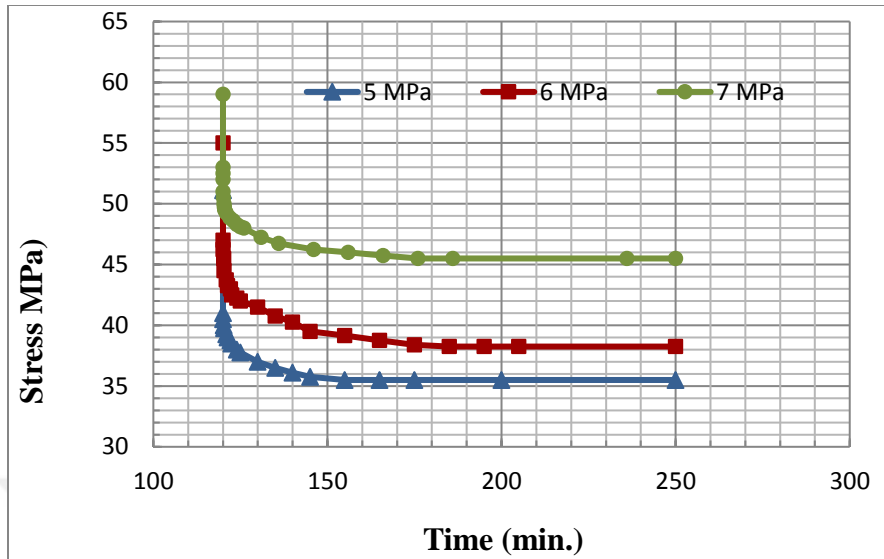


Figure 4.29: relaxation curves of with varying different applied stress for UP

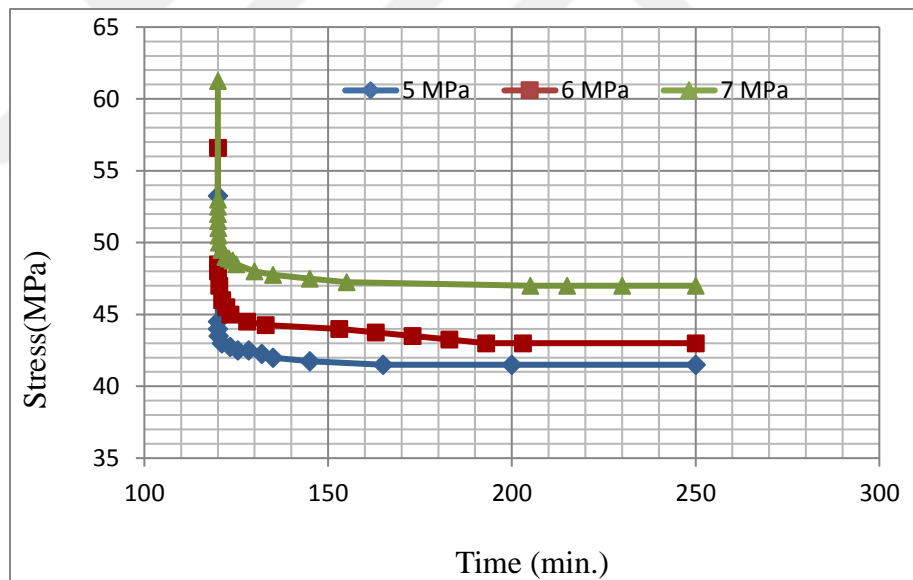


Figure 4.30 a: Relaxation curves of blend with One Layer of reinforced glass fiber with varying different applied stress.

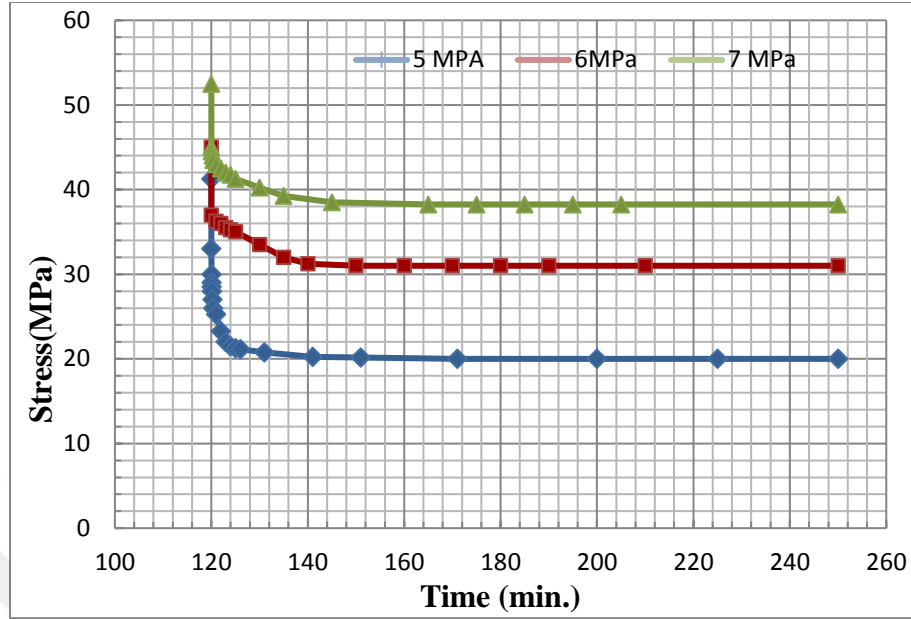


Figure 4.30 b: Relaxation curves of blend with Two Layers of reinforced glass fiber with varying different applied stress.

4.6.2 Creep Modulus Prediction by Kelvin Model

This model represents a solid undergoing a reversible, viscoelastic strain. When applying a constant stress, the material will deform asymptotically, at a decreasing rate, reaching the steady-state strain. Upon releasing the stress, the composite gradually relaxes to its undeformed state. Similar to the Maxwell model, the Kelvin–Voigt model also has limitations. This model is extremely good with modeling creep in materials, but regarding the relaxation, the model is much less accurate. It can be applied to organic polymers, rubber and wood when the load is not so high [77].

The creep modulus can be predicted by modeling mathematically using Eq. 2.21.

$$E(t) = \frac{\sigma}{\varepsilon(t)} = \frac{\delta_1 \eta_1}{\eta_1 + \delta_1 t}$$

Values of the variables (δ_1, η_1) are calculated in the same matter done previously, and by applying Eq. 2.19, creep modulus is obtained, Fig. 4.31.

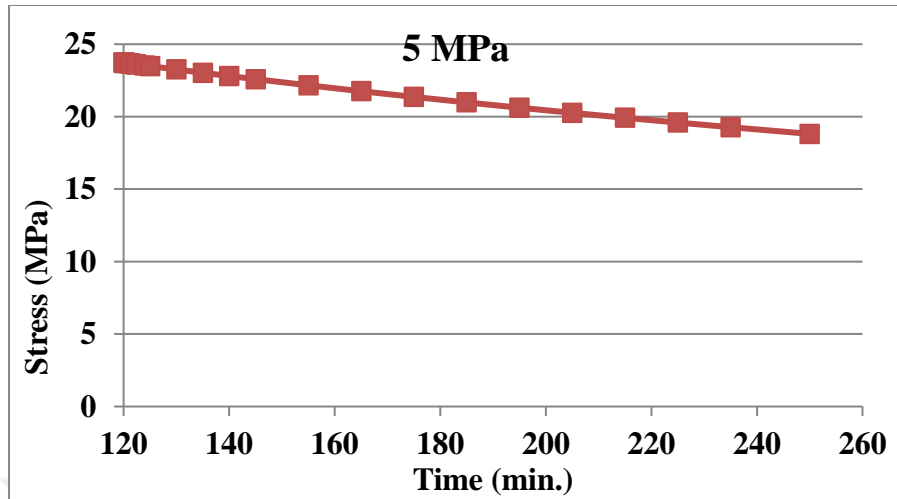


Figure 4.31: Creep Modulus data for UP predicted using Kelvin model.

Kelvin model gives an acceptable predicted values for creep modulus of UP and blend in which it has an inverse relation with the applied stress, as shown in Figures 4.32 a, b and c, while for fiber-reinforced samples, because of the complex mechanism of the deformation in these composites. The following figures show the creep behavior (creep resistance) as shown in the Figures 4.33 a, b and c.

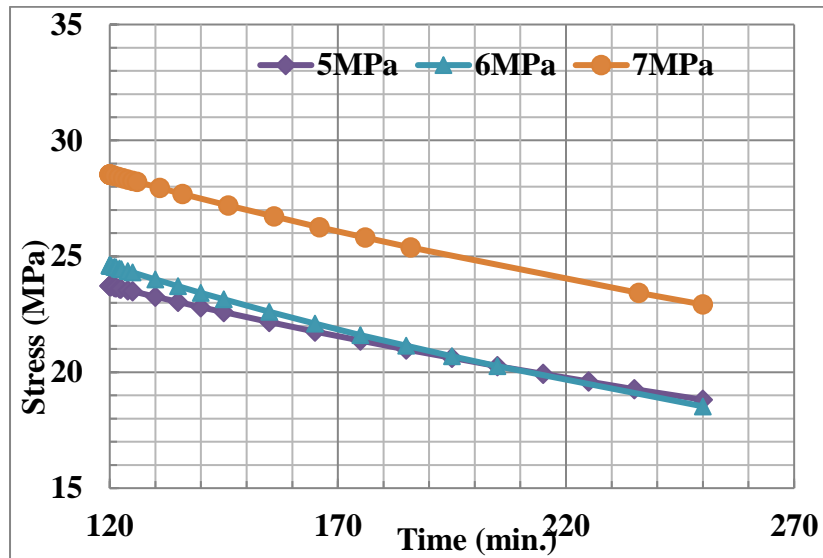


Figure 4.32 a: Creep Modulus curves of Polyester with varying different applied stress.

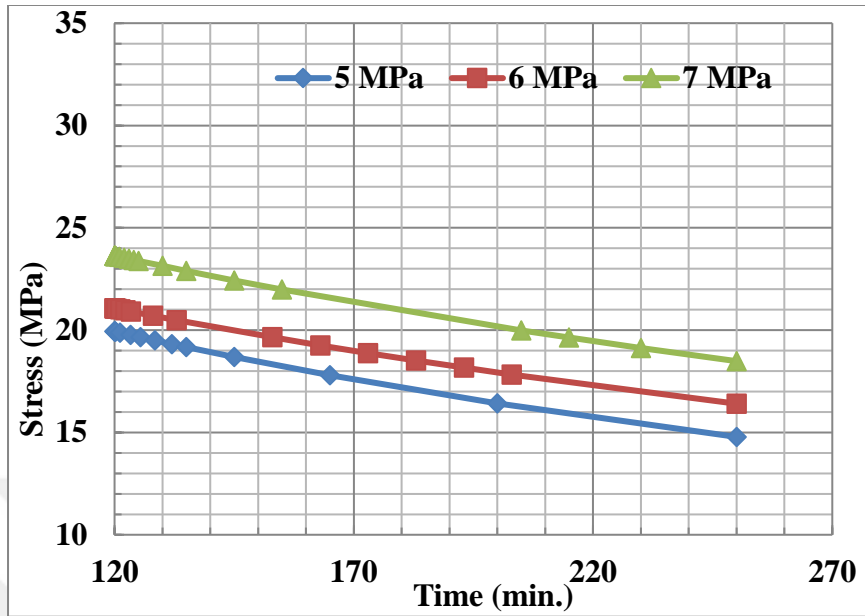


Figure 4.32 b: Creep Modulus curves of blend with One Layer of reinforced glass fiber with varying different applied stress.

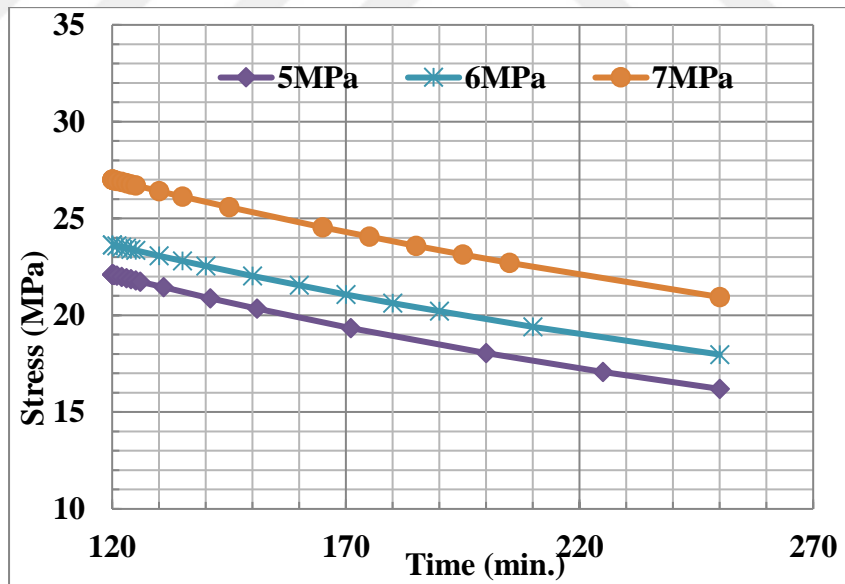


Figure 4.32 c: Creep Modulus curves of blend with Two Layers of reinforced glass fiber with varying different applied stress.

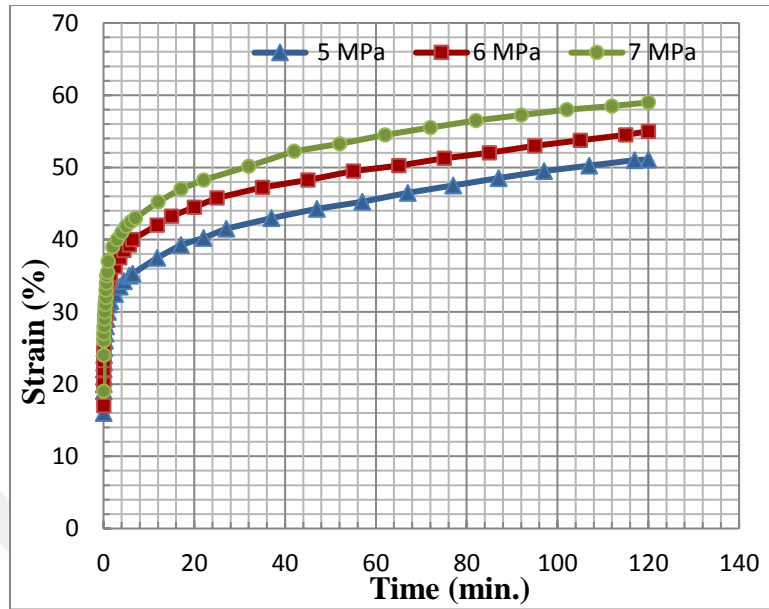


Figure 4.33 a: Creep resistance curves of Polyester with varying different applied stress.

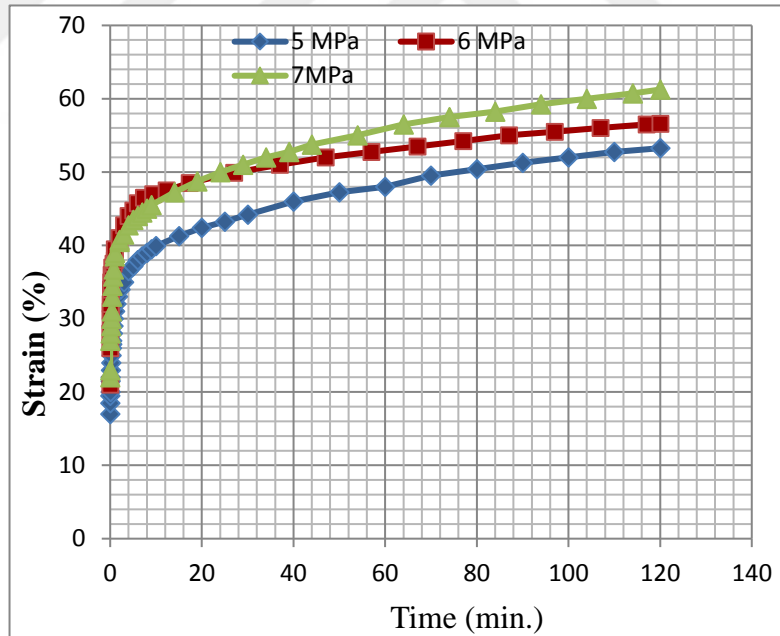


Figure 4.33 b: Creep resistance curves of blend with one Layer of reinforced glass fiber with varying different applied stress.

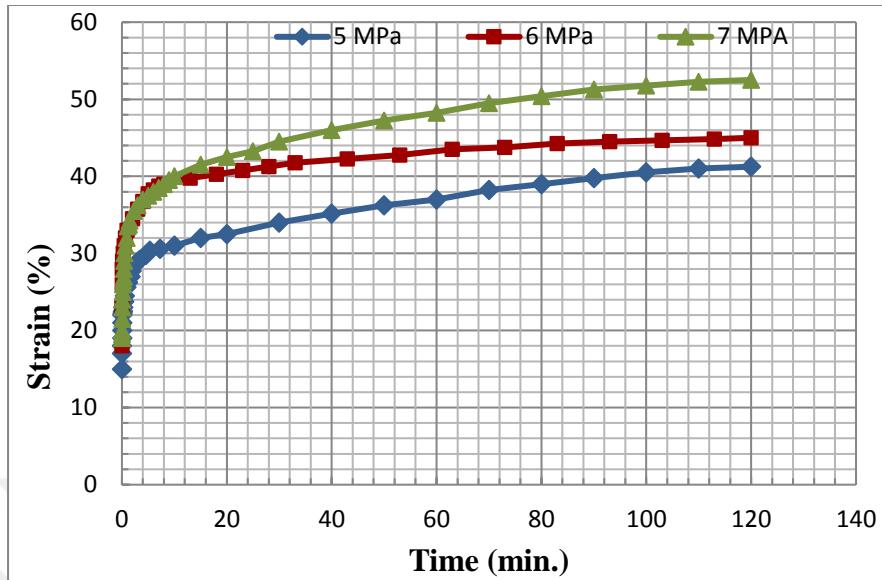


Figure 4.33 c: Creep resistance curves of blend with Two Layers of reinforced glass fiber with varying different applied stress.

4.7 Viscoelastic Behavior of Burger's

The resulting deformation tends to increase with time as entangled chain segments undergo conformational changes in response to external stress. Viscoelasticity is a molecular rearrangement. When a stress is induced in a viscoelastic material such as a polymer, parts of the long chain of the materials change position. In spite of this complex mechanism of rearrangement in order to accompany the stress, polymers remain a solid material. Upon releasing the original stress, the polymer will return to its original form by the effect of the accumulated back stresses. When the material creeps, it is given the prefix- viscous, and when fully recovers, it is given the suffix-elasticity. The theory can be used to predict the creep compliance, creep behavior, stress relaxation and shear stress relaxation response and creep modulus of real materials, the constants have been obtained from a creep test [53].

4.7.1 Linear Viscoelastic Test

When a constant strain is applied on the material, stress will gradually relax. When the applied stress is constant, the strain will have two components. The first one is

elastic component which occurs instantaneously, corresponding to the spring, and will relax as soon as stress is released. The other is a viscous component which is growing with time as long as the stress is applied [77].

4.7.2 Calculating the Creep Behavior of Burgers Four – Element Model

The tests carried out at room temperatures and the stress of different ranges 5, 6 and 7 (MPa), in order to reach the best results, so the creep behavior is calculated to predict the occurrence of change in this material.

The creep behavior equation for (UP) material is evaluated according to Burger four elements, where Eq. 2.36 is used here. The material constants (η_1, η_2, R_1, R_2) are calculated according to the method explained by Burger, shows the data of the material constants for the (UP) same process for all tests is used. The final creep behavior result for Polyester at ($T=30^\circ\text{C}$, stress= 7 MPa). Eq. 2.36 can be written as follows. Fig. 4.23 shows the creep behavior for this equation.

It is noted that Burger model gave a good concordance with the experimental results obtained for all specimens, and this indicates that the creep behavior in the test is of viscoelastic, and it's of linear type, as shows in the Fig. 4.35. See Appendix (A)

So by comparing between the experimental results and Burger model, and by calculating the error percentage, we can divide the results into two periods. During the time period ($0 < t < 120$), which represents the creep time we noticed that the error percentage between the Burger model curve and the experimental results was (1.29%), and this small percentage indicate that the creep behavior is viscoelastic of linear type. Therefore, the Burger model is suitable to predict the viscoelastic behavior for this type of polymers (polyester), as shown in the Fig. 4.34.

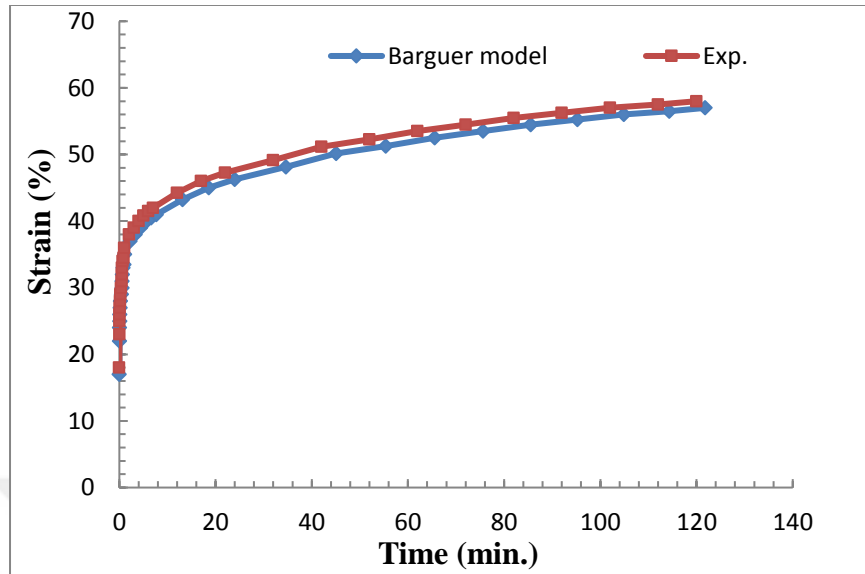


Figure 4.34: Creep Behavior Of Linear Viscoelastic UP applied 7 MPa.

While, during the experimental period ($130 < t < 250$) which represents the stress relaxation stage, the error percentage was approximately (2.537 %), which is very small, indicating that the results are approximately similar to burger model, as shown in the Fig. 4.35. It's noted that the results are repeatable for all specimens.

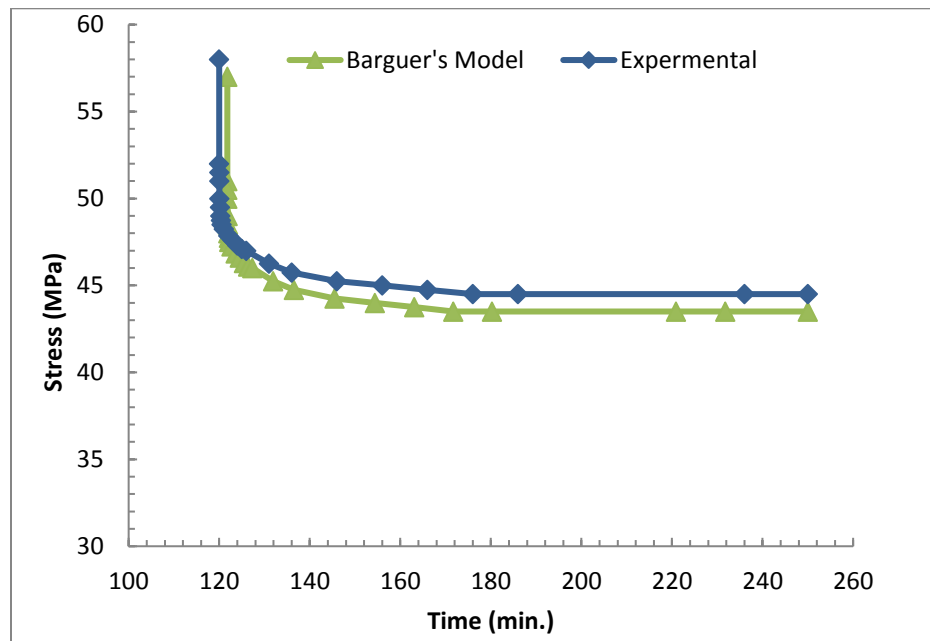


Figure 4.35: Stress Relaxation of Linear Viscoelastic UP applied 7 MPa

4.8 Effect of Adding Fiber on Creep Behavior

The following figures show the creep behavior, creep resistance and relaxation stress (5, 6 and 7 MPa) separately, at room temperature. In these figures, it's clearly noted that there is an improvement in creep resistance and relaxation stress of unsaturated polyester, after the addition of chopped E-glass fiber especially when two layers of fiber are added to the blend.

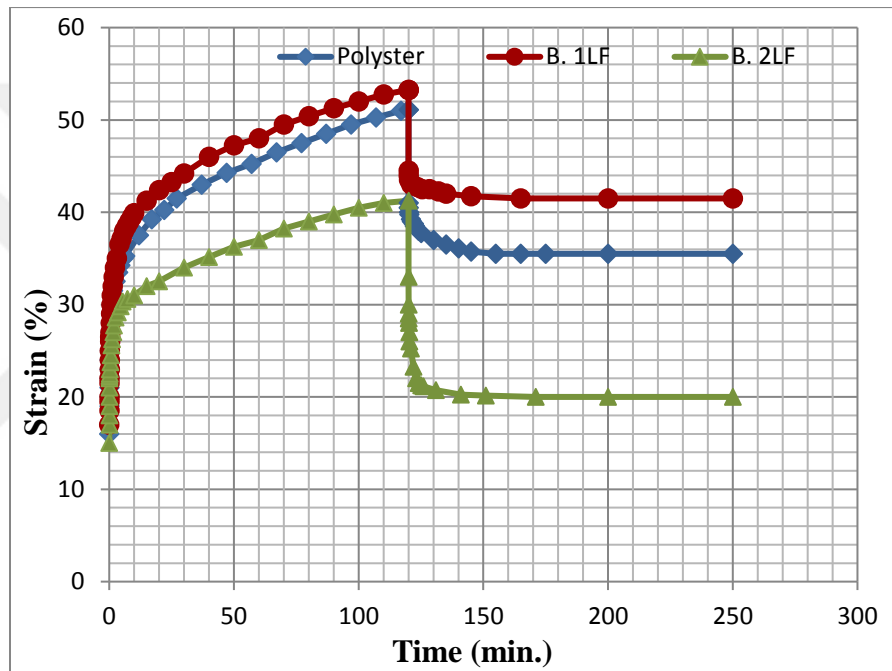


Figure 4.36: Viscoelastic Behavior, at Applied 5 MPa.

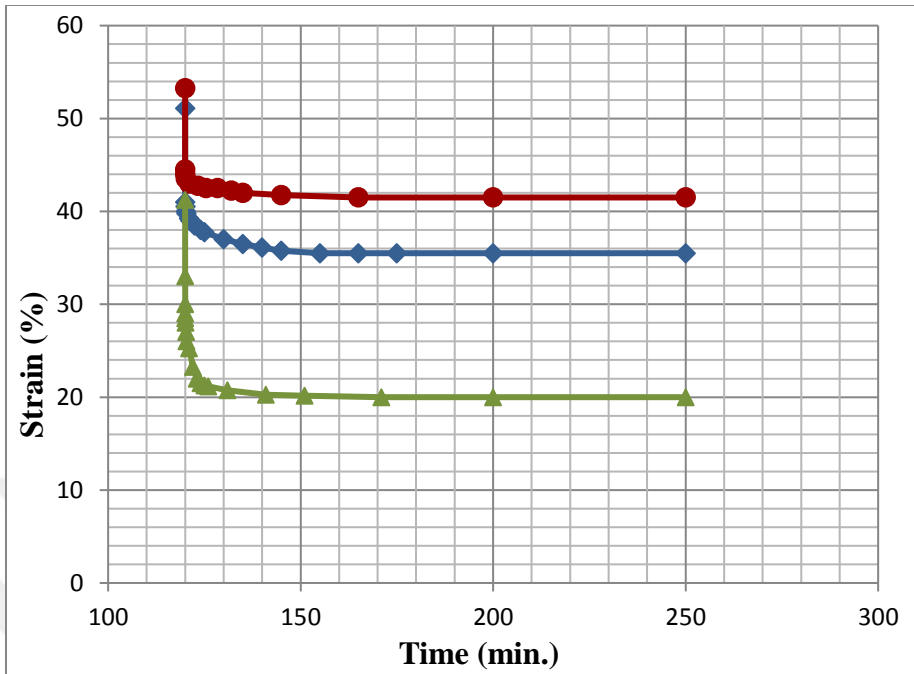


Figure 4.37: Viscoelastic Behavior, Stress relaxation, at Applied 5 MPa.

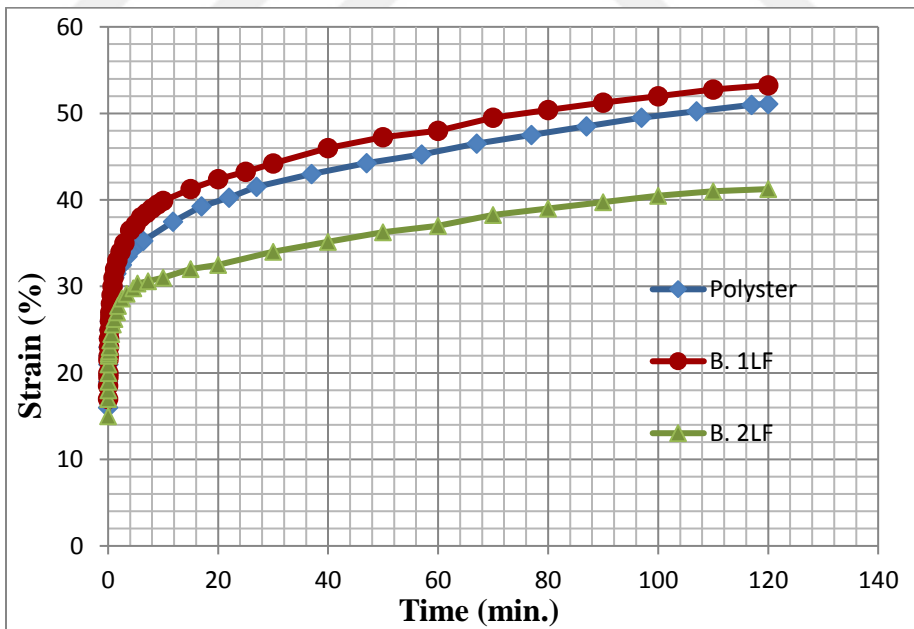


Figure 4.38: Viscoelastic Behavior, creep recovery, at Applied 5 MPa.

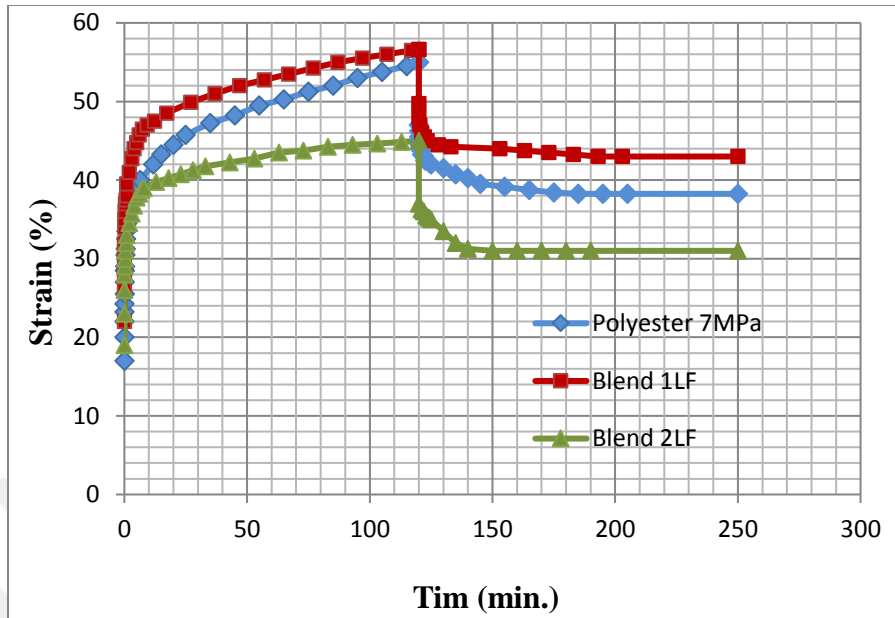


Figure 4.39: Viscoelastic Behavior, at Applied 6 MPa.

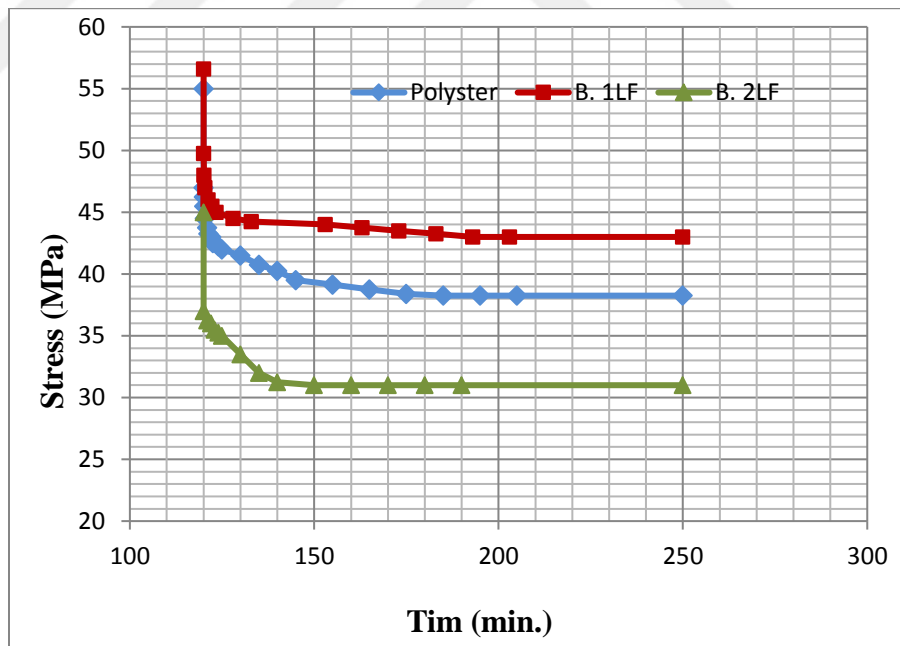


Figure 4.40: Viscoelastic Behavior, Stress relaxation, at Applied 6 MPa.

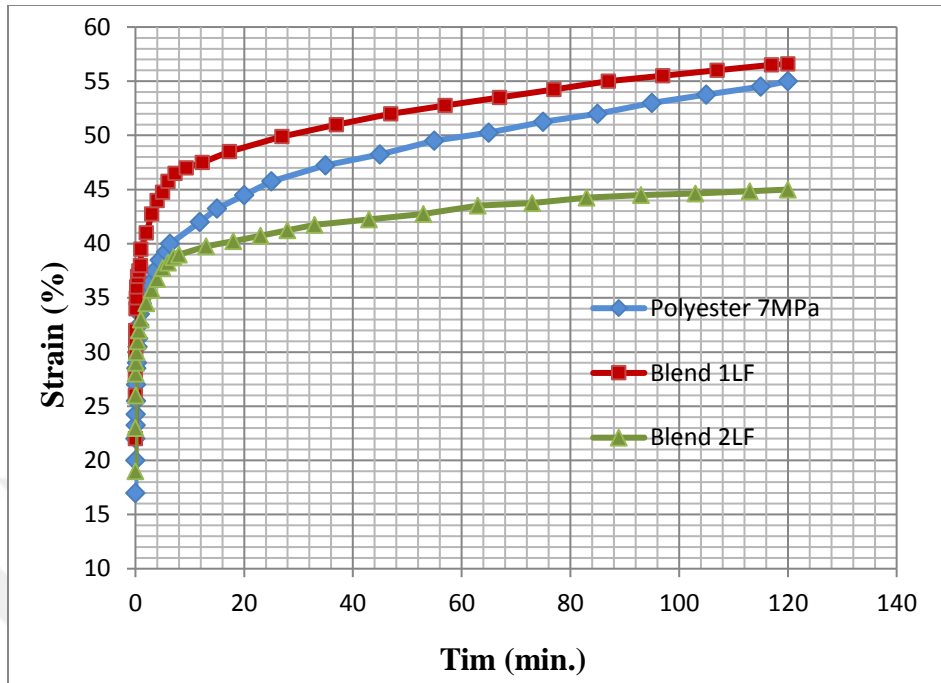


Figure 4.41: Viscoelastic Behavior, creep recovery, at Applied 6 MPa.

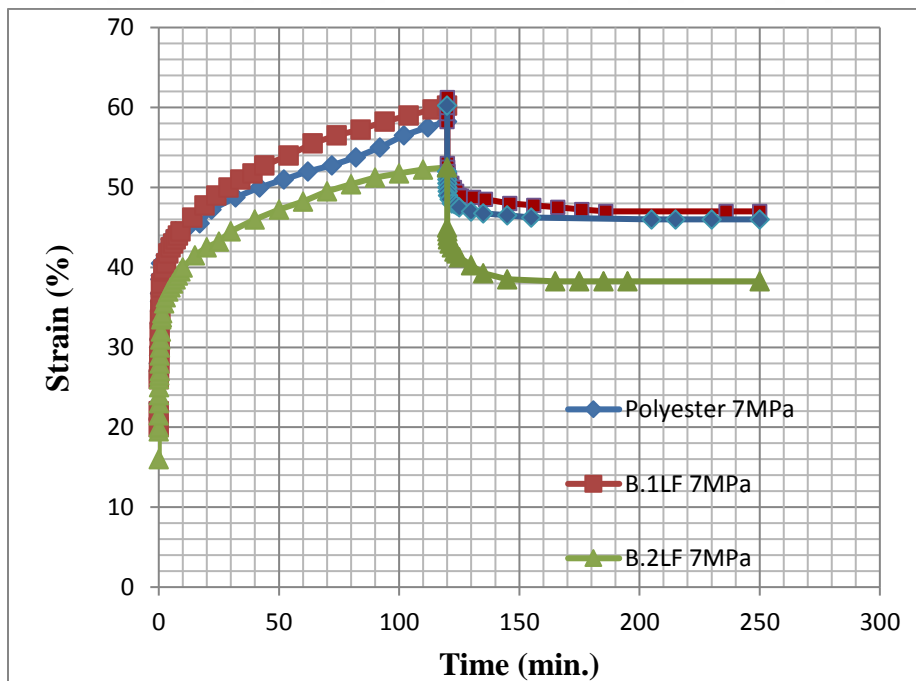


Figure 4.42: Viscoelastic Behavior, at Applied 7 MPa.

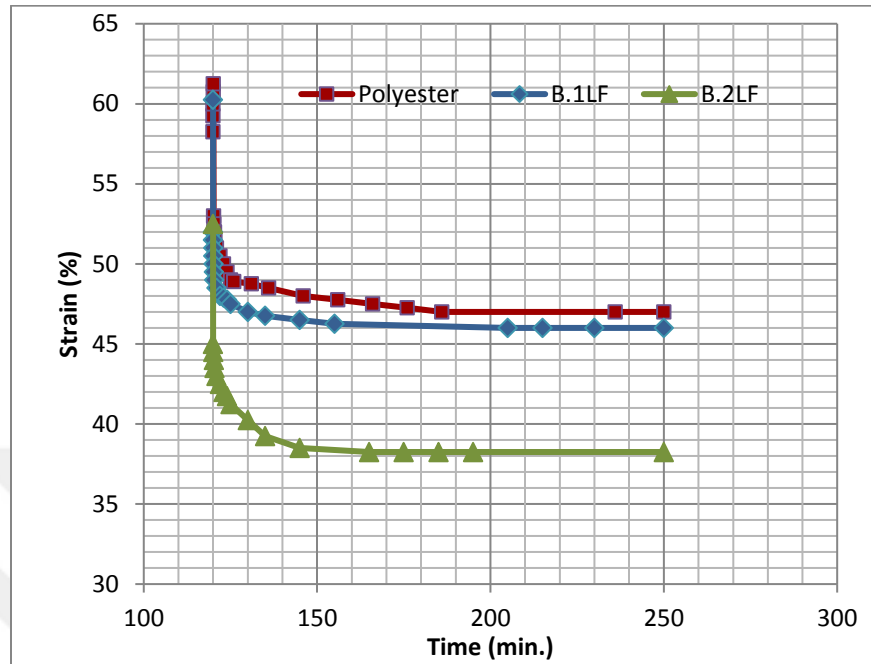


Figure 4.43: Viscoelastic Behavior, Stress relaxation, at Applied 6 MPa.

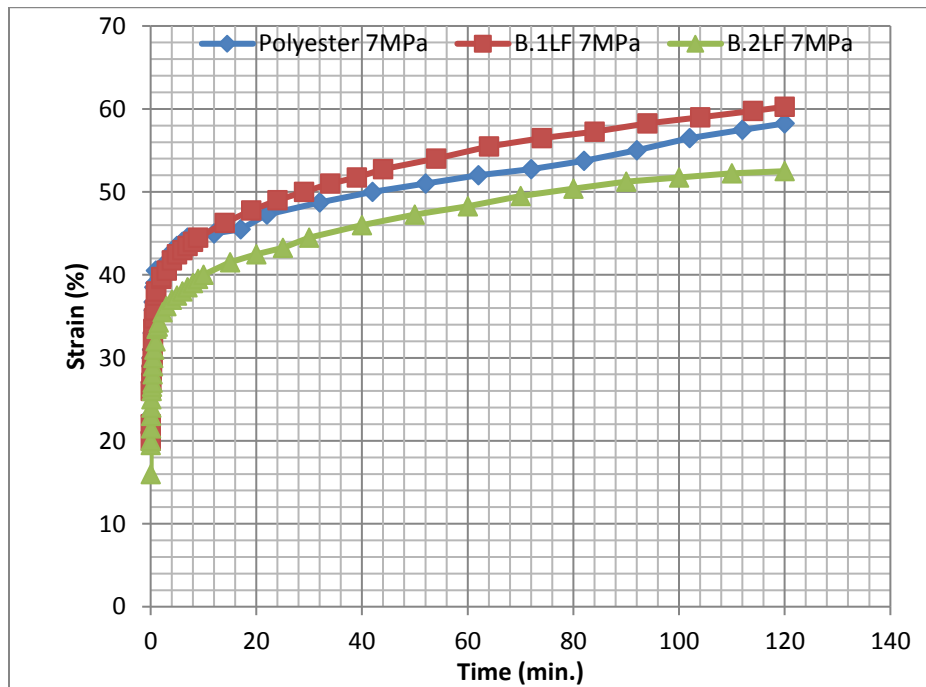


Figure 4.44: Viscoelastic Behavior, creep recovery, at Applied 7 MPa.

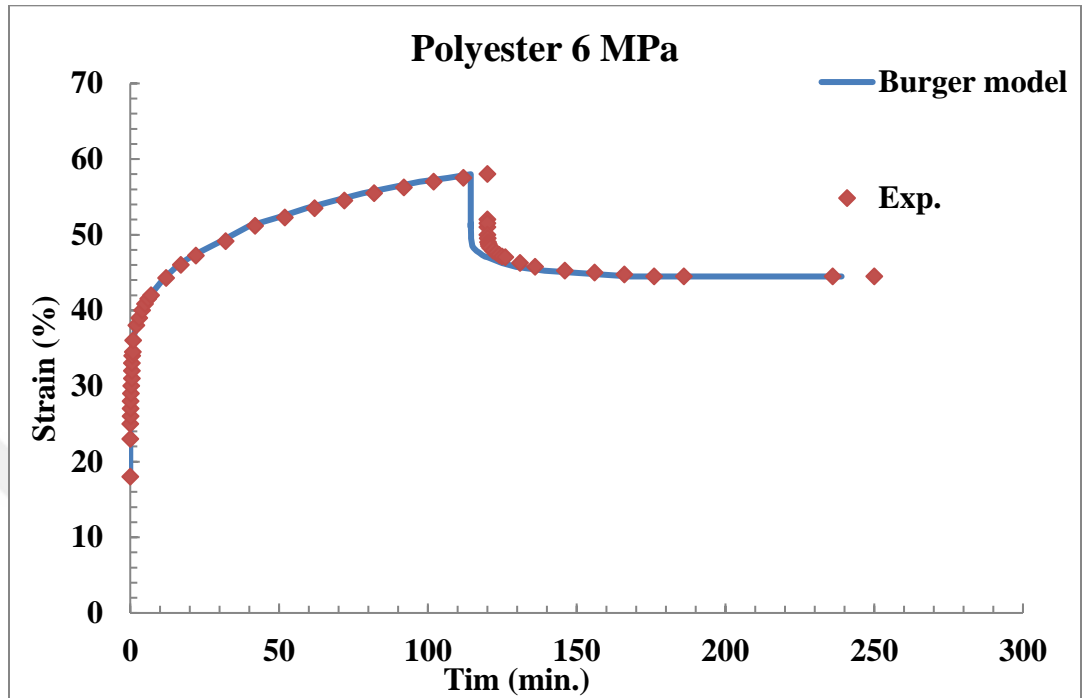


Figure 4.45: Viscoelastic Behavior, creep recovery and Stress relaxation of Burger's model.

CHAPTER FIVE

CONCLUSION AND SUGGESTIONS

5.1 Conclusions

Based on the results obtained and discussed previously, the following conclusions can be drawn:

- 1- Incorporation of the ductile polystyrene reduces the tensile strength to a high extent, but on the other hand improves the strain at break.
- 2- The mechanical performance of the polyester is changed with the incorporation of polystyrene. The elastomeric nature of the polystyrene caused reduction in tensile strength, hardness and creep resistance, but strain at break values increment and attained a maximum for 20 wt % of PS.
- 3- The combination of blend of 7.5 wt % fibers offers the possibility to formulate tough and stiff unsaturated polyester resin system.
- 4- Reinforcing blend of 7.5 wt % PS with chopped glass-fiber mats improves modulus of toughness, tensile strength, hardness, creep resistance and strain at break. The best mechanical properties obtained when two layers of glass fiber added to the blend. It has balancing effect to compensate the PS reduction in mechanical properties and gives the highest toughness.
- 5- SEM analysis showed that the distribution of the smaller particles of polystyrene in the blend is responsible for the low crack growth and consequently higher fracture toughness.
- 6- Creep resistance and relaxation stress increase proportionally when two layers of fiber are added to the blend.
- 7- Burger's model gave a good agreement with the experimental data of creep test.

5.2. Suggestions

- 1- Using another polymer, like natural rubber as a blender with Unsaturated Polyester and evaluate their effect on the mechanical properties.
- 2- Studying the thermal, rheological behavior of PU-PS blend and its composites.
- 3- Assess the effect of low temperatures on creep behavior of tested materials.
- 4- Investigate the relationship of instantaneous recovery and damping capacity of different solid polymer.
- 5- Proceeding relaxation test experimentally and comparing theoretical and experimental data.

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APPENDIX A1

1. Determine of the material constants for Burger's model

The behavior of creep for Burger's model has been shown in Fig. 2.28. Assuming the experimental data for creep test has been recorded for the region of time [53]:

$$0 < t < t_1 \quad (A.1)$$

A1.1 Determine η_1 :

The slope of creep function at an infinite time ($t = t_1$) represent $(\tan \beta)$. from curve fitting β can be measured and η_1 can be calculated from the following equation:

$$\dot{\varepsilon}(t_1) = \frac{\sigma_o}{\eta_1} = \tan \beta \quad (A.2)$$

$$\dot{\varepsilon}(t_1) = \frac{\sigma_o}{\eta_1} = \tan \beta \quad t\varepsilon(t) = \frac{\sigma}{\varepsilon_1} + \frac{1}{\eta_1} t$$

Where:

$\dot{\varepsilon}(t_1)$: the slope of Eq. 2.36 at ($t = t_1$)

A1.2 Calculation of η_2 :

The creep rate starts at $t=0$ with finite value. By measuring α , the value of η_2 can be evaluated from the following equation:

$$\dot{\varepsilon}(0) = \left[\frac{1}{\eta_1} + \frac{1}{\eta_2} \right] \sigma_o = \tan \alpha \quad (A.3)$$

A1.3 Determine R_1, R_2 :

From the above figure, initial creep strain is

$$\dot{\varepsilon}(0) = OA = \frac{\sigma_o}{R_1} \quad (A.4)$$

Eq. A2.3 gives the value of R_1 , also :

$$AA' = \frac{\sigma_0}{R_2} \quad (\text{A.5})$$

The final Eq. A.5 gives the value of R_2 by measuring the length AA' from the curve of creep test.



APPENDIX A2

1. Calculation of the materials coefficient (η_1, η_2, R_1, R_2) for linear UP at 7 MPa:

$$OA = 18 \cdot 10^{-2}$$

$$OA' = 50 \cdot 10^{-2}$$

$$\sigma_o = 7 \text{ Mpa}$$

It has been shown from fig.(a):

$$\tan \beta = \frac{8 \cdot 10^{-2}}{120} = 6.667 \times 10^{-4} (\text{min})^{-1}$$

$$\tan \beta = \frac{\sigma_o}{\eta_1} \Rightarrow \eta_1 = \frac{\sigma_o}{\tan \beta} \Rightarrow \eta_1 = 10500 (\text{MPa} \cdot \text{min.})$$

$$\tan \alpha = \frac{25 \cdot 10^{-2}}{20} = 1.25 \times 10^2 (\text{min})$$

$$\left[\frac{1}{\eta_1} + \frac{1}{\eta_2} \right] \sigma_o = \tan \alpha \Rightarrow \eta_2 = 591.549 (\text{MPa} \cdot \text{min.})$$

$$OA = \frac{\sigma_o}{R_1} \Rightarrow 18 \cdot 10^{-2} = \frac{7}{R_1} \Rightarrow R_1 = 38.888 (\text{MPa})$$

$$AA' = \frac{\sigma_o}{R_2} \Rightarrow 32 \cdot 10^{-2} = \frac{7}{R_2} \Rightarrow R_2 = 21.8775 (\text{MPa})$$

Where:

$$q_1 = \eta_1 = 591.549 (\text{MPa} \cdot \text{min.})$$

$$q_2 = \frac{\eta_1 \eta_2}{R_2} = 283943.662 (\text{MPa} \cdot \text{min.})$$

$$P_1 = \left(\frac{\eta_1}{R_1} + \frac{\eta_1}{R_2} + \frac{\eta_2}{R_2} \right) = \left(\frac{61325.8}{3.96} + \frac{61325.8}{2.23} + \frac{1522.4}{2.23} \right) = 777.04$$

$$P_2 = \frac{\eta_1 \eta_2}{R_1 R_2} = 7301.4$$

$$A = \sqrt{P_1^2 - 4P_2} = 758.016$$

$$r_1 = \frac{P_1 - A}{2P_2} = 1.3 * 10^{-3}$$

$$r_2 = \frac{P_1 + A}{2P_2} = 10.51 * 10^{-2}$$

$$\sigma(t) = \frac{\varepsilon_0}{A} [(q_1 - q_2 r_1) e^{-r_1 t} - (q_1 - q_2 r_2) e^{-r_2 t}]$$

$$\text{So: } \varepsilon(t) = \frac{\sigma_0}{R_1} + \frac{\sigma_0}{\eta_1} t + \frac{\sigma_0}{R_2} \left(1 - e^{-\frac{R_2 t}{\eta_2}}\right)$$

2. Calculation of the materials coefficient (η_1, η_2, R_1, R_2) for linear Blend with

1LF

$$OA = 0.22$$

$$OA' = 0.31$$

$$\sigma_0 = 7 \text{ MPa}$$

$$\tan \beta = 8.33 \times 10^{-2} (\text{min})^{-1} \implies \eta_1 = 8 * 10^6 \text{ MPa} \cdot \text{min}.$$

$$\tan \alpha = 1.333333 \text{ MPa} \cdot \text{Min}.$$

$$\eta_2 = 205128.2 \text{ MPa} \cdot \text{Min}.$$

$$OA = \sigma_0 / R_1 \implies 0.22 = 7 / R_1 \text{ MPa}$$

$$AA' = \sigma_0 / R_2 \implies 0.31 = 7 / R_2 \text{ MPa}$$

3. Calculation of the materials coefficient (η_1, η_2, R_1, R_2) for linear Blend with

2LF

$$OA = 0.17$$

$$OA' = 0.30$$

$$\sigma_0 = 7 \text{ MPa}$$

$$\tan\beta = 1.16 \cdot 10^{-5} \text{ MPa. Min.} = \sigma_o / \eta_1$$

$$\eta_1 = 601605.6849 \text{ MPa. min.}$$

$$\tan \alpha = 4.8 \cdot 10^{-4} \text{ MPa. Min.}$$

$$(1/\eta_1 + 1/\eta_2) \sigma_o = \tan \alpha$$

$$\eta_2 = 14946.72391 \text{ MPa. Min.}$$

$$OA = \sigma_o / R1 \quad \Longrightarrow \quad 0.17 = 7 / R1 \text{ MPa}$$

$$AA' = \sigma_o / R2 \quad \Longrightarrow \quad 0.30 = 7 / R2 \text{ MPa}$$

