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Study the Biaxial Creep of Continuous Fiber Reinforced Polymer

A Thesis
Submitted to the Council of the College of Materials Engineering/University of Babylon in Partial Fulfillment of the Requirements for the Master Degree in Materials Engineering / Polymer

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الملخص

الزحف هو التغيير البطيء المستمر في أبعاد القطعة الهندسية أثناء الخدمة. يعتبر الزحف سلوكًا مهمًا جدًا في المواد تحديدًا للبوليمر بسبب حدوث ذلك في درجة حرارة الغرفة. الزحف هو تغيرات في أبعاد المنتج ، ويعتبر سلوك الزحف من الامور المهمه في المركب بسبب التباين في المواد التي تتكون منها المادة المركبة و الذي يؤدي إلى زحف ثنائي المحور

يهدف العمل إلى دراسة الزحف ثنائي المحور في المواد المركبة من الأنواع التالية (البولي إيثيلين عالي الكثافة ، مصفوفة البولي إيثيلين عالية الكثافة المقواة بألياف الكربون) بطبقات مختلفة (roving ، woven ، الالياف أحادية الاتجاه). تم تنفيذ هذه الدراسة عن طريق تحوير جهاز الزحف الأحادي المحور إلى جهاز زحف يعمل بمحورين ودراسة سلوك اللزوجة المرنة (الزحف) تحت تاثير الحمل المسلط .

طرق التصنيع النموذجية

١- آلة البثق ذو اللولبين لبثق صفائح HDPE النقية

٢- مكبس هيدروليكي Electro-Thermal لتصنيع أربعة أنواع من الألواح وهي عبارة عن بولي إيثيلين نقي عالي الكثافة ومادة مركبة معززة بألياف الكربون من النوع ، woven و roving وأحادي الاتجاه.

بعد ذلك ، تقص العينات بالليزر CNC على شكل صليب. لتلائم تجربة الزحف ثنائي المحور بعد ذلك تجرى مجموعة من الاختبارات على العينات. لقد تم الخروج بنتيجه بأن المادة المركبة من النوع woven هي الأكثر مقاومة للزحف ثنائي المحور ، لأن محاذاة ألياف التعزيز مثالية في كلا الاتجاهين و نسبة الالياف عالية ، أما بقية الاختبارات فهي الاختبارات (اختبار الشد ، اختبار الصدمه ، اختبار الصلادة ، اختبار FTIR) .

وأظهرت النتائج ، تحسنًا واضحًا في أداء الزحف لجميع مركبات HDPE المقوات بالياف الكاربون ، حيث يتم توفير أفضل أداء لمقاومة الزحف من نوع ألياف الكربون woven (الألياف المنسوجة) الى المادة الاساس المصنوعة من البولي اثلين عالي الكثافه ، متفوقة على افضل أداء توفره المادة المركبة المقوات بالياف الكاربون من نوع roving (الألياف المتقاطعة بزاوية معينة) ، زادت قوة الشد بإضافة ألياف الكربون إلى مادة البولي اثلين عالي الكثافه ، و أعلى زيادة يتم الحصول عليها في النوع المنسوج woven أكثر من غيرها الأنواع المركبة مثل الالياف المتقاطعة roving و الالياف أحادي الاتجاه. تم تقدير أعلى نسبة زيادة تم الحصول عليها لمعامل يونغ في نوع woven. تم الحصول على أعلى مقاومة للصدمات في العينات من نوع woven كذلك ، يليها roving ثم الالياف أحادي الاتجاه ، حيث لعبت الألياف دورًا رئيسيًا في مقاومة الصدمات المفاجئة.

بسم الله الرحمن الرحيم

(يَرْفَعِ اللَّهُ الَّذِينَ آمَنُوا مِنْكُمْ وَالَّذِينَ
أُوتُوا الْعِلْمَ دَرَجَاتٍ وَاللَّهُ بِمَا تَعْمَلُونَ خَبِيرٌ)

صدق الله

العظيم

 سورة المجادلة: الآية ١١

This is dedicated to

my family,

&

Dr. Najm Abdel Amir Saeed

For their endless love, support and encouragement

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First of all, I would like to express my thanks and gratitude to Allah Almighty for granting and facilitating this research. Thank you befitting your Majesty and greatness.

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I also extend my heartfelt thanks to all my professors who supervised me during my undergraduate studies in the Faculty of Materials Engineering, Department of Polymers and Petrochemical Industries.

I also extend my sincere thanks to all the staff members in the Faculty of Materials Engineering, Department of Polymers and Petrochemical Industries at the University of Babylon, for their great role in overcoming the difficulties in front of the research students by assisting with examinations.

Committee Certification

I certify that this thesis entitled " **Study the Biaxial Creep of Continuous Fiber Reinforced Polymer** " had been carried out under my supervision at the University of Babylon/ College of Material's Engineering / Department of Polymer in partial fulfillment of the requirements for the degree of Master in Material's Engineering/Polymer.

Signature

Prof. Dr.

Najim Abd Al-Ameer Saad

Date: / / 2023

Examining Committee Certificate

We certify that we had read this dissertation entitled (**Study the Biaxial Creep of Continuous Fiber Reinforced Polymer**) and as an Examination Committee examined the student (Ali radha Kadhim) in its contents and that in our opinion it meets the standard of a dissertation and is adequate for the award of the Degree of Master in Materials Engineering / Polymer Engineering and Petrochemical Industries Department.

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Abstract

Creep is the slow continuous change in the dimensions of the engineering piece during the service . Creep is very important behavior in materials specifically of polymer because of this occurred in room temperature.Creep is changes of the dimensions of the product , specifically in the composite because of the anisotopic which lead to biaxial creep

The work aims to study the biaxial creep in different composite material (High-density polyethylene, high density polyethylene matrix reinforcement by carbon fibers) (woven, roving, unidirectional). These study implementated by modification the uniaxial creep in to biaxial creep and study the behavior of viscoelastic behavior (creep) under shedder load .

Sample manufacturing methods double screw extruder for pure HDPE sheet extruder and hydraulic press Electro-Thermal or the manufacture of four types of sheets, which are pure high-density polyethylene and a composite material reinforced with carbon fibers of woven, roving and unidirectional type.

After that, cut the samples by CNC laser in the form of a cross. The two-axis creep experiment after that will conduct a set of verification tests on the samples. prove the during which, the composite material of woven type is the most resistant to biaxial creep, because the reinforcement fiber alignment is ideal in both directions and high fiber content , and these tests (tensile test , impact test , hardness test , FTIR test).

The results are, a clear improvement in the creep performance of all HDPE composite reinforcement by carbon fibers, where the adding carbon fibers

type woven to the HDPE matrix is provided the best performance of creep resistance, superior to the best performance provided by the HDPE matrix when introducing of carbon fiber type roving, The sample that provided the best creep resistance performance , The tensile strength increased by added carbon fiber, which is the highest increase obtained in type woven of carbon fibers than other types such as roving and unidirectional . The highest percentage of increase obtained for Young's modulus was estimated at woven of carbon fiber . The highest shock resistance was obtained in the woven samples, followed by roving and then unidirectional, where the fibers played a major role in withstanding sudden shocks.

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List of Abbreviations

HDPE	High density polyethylene
Cf	Carbon fibers
HDPE + Cf	High density polyethylene reinforcement by carbon fibers , composite materials
min	Minutes
mm	Millie meter
N	Newton is unit of measure
/	Symbol indicating division

CNC	Computer Numerically Controlled, Machine
FTIR	Fourier Transforms Infrared

List of Symbols

Symbol	Description	Unit
A	Cross- sectional area of sample	m^2
$\varepsilon(t)$	Total creep strain	mm/mm
D(t)	Creep compliance function	Mpa^{-1}
E	Elastic modulus	N/m^2
Ϭ	Tensile strength	MPa
ε'	Creep rate	mm/min
ε	Strain of materials	mm/mm
F	Tension force	N
H	Viscosity	$kg \cdot m^{-1} \bullet s^{-1}$
I.S	Impact resistance of material	J/m^2
L	Final length	mm
Lo	Initial length	mm
Q	Activation energy	$J\ mol^{-1}$
R	The universal gas constant	J/mole K
Tg	Glass Transitions Temperature	^oC
Δt	Time change	mi
U_c	Impact energy	Joule
$\Delta\epsilon$	Strain change	mm/mm

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Chapter one

Introduction and literature review

1.1 Introduction

Creep in materials science, creep (sometimes called cold flow) is the tendency of a solid to slowly move or deform permanently under constant mechanical stress. It can occur as a result of long-term exposure to high levels of stress that are still below the material's yield strength.Creep is more intense in materials exposed to heat for long periods and generally increases as they approach the melting point. biaxial creep of continuous polymeric fibres composite one of the important matters in the practical aspect is that must know that there is no creep of materials in one axis (in one direction), as if it is in the vertical direction only or only horizontally, since in fact the creep is in two or more directions under the influence of loads placed under more than one direction see figure (1.1). [1,2].

Detectable problems by studying the biaxial creep of the continuous polymeric fiber composit ،study the biaxial creep in polymeric materials in order to predict the mechanical problems that the engineering part may encounter during the engineering service it performs, and thus allow to avoid errors or problems that may occur by choosing materials that have good features in terms of resistance to service conditions or choosing suitable designs[3].

The study of bi-axial creep is also involved in many important matters and designs that major countries and well-known institutions need, including the design of vehicles such as cars, trains, tractors and other basic means of transportation in people's lives in general, as well as the design of devices of various types, including air conditioning and machines that are used In land excavation and many important designs of devices used in people's daily lives[4].

Dangerous when the dimensions differ due to creep as the polymers are sensitive to work at high temperatures [5].

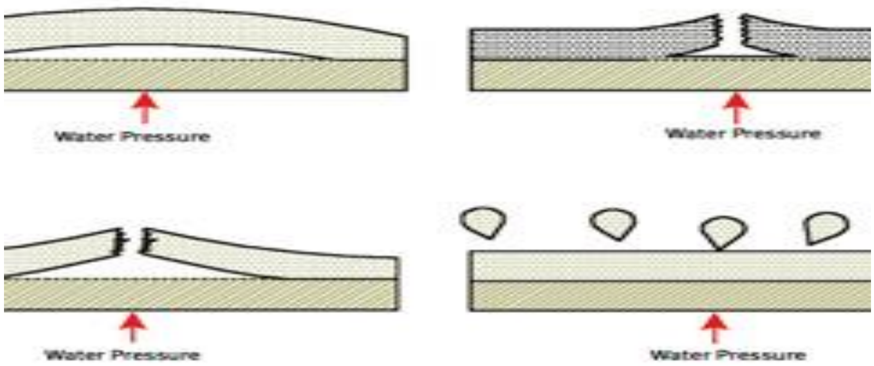


Figure 1.1 : Sample under effect creep bay water pressure [5]

As for polymers, creep occurs at room temperature and since it is not crystalline in structure, and at a temperature higher than (Tg) it begins to creep under the influence of a load (constant tensile stress) with the temperature remaining constant where the agitation with the time taken during the examination and the examination takes several hours or days if the test is below a temperature less than (Tg) because the material is hard and brittle, and the duration increases for weeks if the material is reinforced with reinforcing materials, and the creep curve consists of strains in terms of the time taken as the deformation (elongation) passes through creeping stages, the first of which is instantaneous distortion primary (transition) characterized by recovery when the load is removed, then followed by a secondary (steady) creep with a lower level that has a sticky or plastic behavior (a steady state) and this depends on the level of influential stress and temperature, and finally a third accelerated creep in fact represents a process characterized by the gradual deterioration that leads to the breaking of the material due to interfacial collapse of the crystal[6].

1.2 How is biaxial creep studied?

This phenomenon is known as viscoelastic creep .Where the composite material of the woven type showed greater resistance to creep than other types due to the interconnection of the fibers and their cohesion in both directions. It is known that the molecular weight increases with the increase in the percentage of added fibers, and since the woven material samples have the highest percentage of fibers, it causes an increase in the molecular weight as the ، It is known that the molecular weight of the polymer influences the creep behaviour. The effect of increasing the molecular weight tends to

1- Enhance the secondary bonding between the polymer chains and thus make the polymer more resistant to creep due to the added stiffness.
2- The increase in molecular weight adds thermal stability to the polymers, which increases the creep resistance of the polymer.

Accordingly, the uses of high-density polyethylene reinforced with carbon fibers have increased in high applications that require the material to be of high strength and hardness and withstand high temperatures, as the reinforcement fibers made of carbon, in turn, have high melting points, which raised the melting temperatures of the composite material. It is known that the higher the melting temperature of a substance, the greater the creep resistance [7].

1.3 Applications of High Density Polyethylene

1-Cover agricultural crops

2-It is used in some car parts in the struts of modern cars

3-For the production of tubes, films, shopping bags, containers, drums, cans, caps, packing supplies, toys, etc.

4-In door panels [8].

1.4 Literature Review

A group of previous studies dealing with topics similar to the topic that studied in experience, as well as the similarity at the level of the materials used

In 2003 , Joop Baltussen et al , The viscoelastic extension of polymer fibres: Complex loadings the response of oriented polymer fibres to complex loading patterns is investigated. It is shown that the creep and stress relaxation is non-linear with the applied stress . By analysing the results of the step-creep and the strain-relaxation-strain experiments performed on poly(p-phenylene terephthalamide) fibres, it is shown that the linear superposition principle does not apply to the tensile deformation of polymer fibres above the yield point. Finally the various components of the tensile deformation that should be covered by a constitutive equation for polymer fibres are discussed[9].

In 2005 , Evagelia Kontou **et al** , Tensile creep behavior of unidirectional glass-fiber polymer composites The tensile creep behavior of unidirectional glass-fiber polymer composites . The creep strain rate was negligible at room temperature, while it was considerable at the higher temperatures examined. The materials exhibit nonlinear viscoelastic behavior, and the creep response of the composites was treated as a thermally activated rate process. The creep strain was considered to include an elastic, a viscoelastic and a viscoplastic part. The viscoplastic part was calculated through a functional form, developed in a previous work, assuming that viscoplastic response of polymer composites arises mainly from the matrix viscoplasticity. The model predictions in terms of creep compliances were found to be satisfactory, compared with the experimental results. POLYM. COMPOS. 26:287-292, 2005. 2005 Society of Plastics Engineers[10].

In 2011 , P .Davies et al , Tensile and creep behaviour of polyethylene terephthalate and polyethylene naphthalate fibres ropes made of twisted polyester (PET) yarns have been replacing traditional steel ropes and chains as mooring lines for offshore platforms in deep-sea environments. Both fibres show similar non-linear tensile behaviour, with an evolution of modulus in four steps. The same microstructural model is proposed for both fibres, based on microfibrils aligned along the fibre axis and composed of an alternation of amorphous, mesamorphous and crystalline phases. Creep behaviour is also evaluated by considering the evolution of creep rate with applied load. This evolution is again similar for PET and PEN, both from a qualitative and quantitative point of view. The similarity in creep rate values for both fibres indicates that the microstructural mechanisms involved in creep may differ from those involved in short term tension loading[11].

In 2013, Martin M. Riara et al, study Polyethylene compounds with various cellulose loading ratios create by pressure molding.Creep performance deteriorated with a rise in temperature and improved with cellulose content, but with a rise in time and temperature declined, the creep modulus. Timing overlay has been used to predict the creeping behavior of the samples over long periods of time (Kontou 106 s). Use the William Land Ferry model to predict the long-term behavior of the material, use the free volume for the action of deformation[12].

In 2019, Xiaolong Hao et al, investigated the mechanical properties and creep values that are expected to be affected by the content of filled wood in the casing, the basic fill layers, and the dimensional stability of the model for high-density polyethylene extruded with wood particles . Test (SEM) revealed the existence of an association between the two basic layers and the shell, Impact resistance and flexing stress are greater than the basic control elements despite their lower values, creep stress was greatly reduced when hardwood flour (WF) was introduced into The shell and substrate, model (Co-WPCs) with 70% WF in the base layer and 20% WFin the shell had a change in creep values of an amount equal to those of the basic control only, but the impact resistance and bending stress were clearly better[13].

In 2019, Harun Sepet et al, of unfilled HDPE . The impact strength of filled HDPE composites decreased slightly with micro - and particle contents . microparticles were found to be more effective at low - stress levels (8 and 12 MPa) unfilled HDPE. It was found that the particle size has a profound effect on the thermal and physical properties of unfilled HDPE, such as density, melt flow index. The results showed that the size of filler particles has a significant effect on the mechanical and thermal properties of the unfilled HDPE. Therefore, the size selection of constituent materials of micro composites is an important consideration because directly affects the functional performance of particle - filled HDPE micro composites. Concentration[14,15].

In 2020, Kenechi Nwosu-Obieogu, et al, investigated the creep and stress relaxation behavior of rice husk reinforced low density polyethylene composite. The exponential and power model was used to study the creep while the stress relaxation assessed the time required for the composites to maintain a certain strain level. The creep strain increased with increase in time , at various temperatures , with its highest creep at 70 ° C while the lowest is at 30 ° C , the power model provided an excellent fit than other models with a coefficient of determination of 0.9977 at 30 ° C, the neat low density polyethylene had a good stress relaxation behavior with 4.95 seconds for it to decay and decreased with an increase in filler concentration[16].

As for the following study, it is the closest studies to our subject in terms of materials, experience, and examinations used

In 2023, Mostafa Katouzian et al, Modeling Study of the Creep Behavior of Carbon-Fiber-Reinforced Composites: The aim of this paper is to present some important practical cases in the analysis of the creep response of unidirectional fiber-reinforced composites. Some of the currently used models are described: the micromechanical model, homogenization technics, the Mori-Tanaka method, and the finite element method. (FEM). Each method was analyzed to determine its advantages and disadvantages. Regarding the accuracy of the obtained results, comparisons are made with experimental tests. The methods presented here are applied to carbon-fiber-reinforced composites, but these considerations can also be applied to other types of compositematerials [17].

1.5 Summary of scientific research

All the previous studies that we mentioned dealt with the creep test and the common characteristic between them is that they used polyethylene samples(PE), some of them used high-density polyethylene and others used low-density polyethylene and other material but similar to . Among the things that were observed through our study of these researches is that the resistance of polyethylene samples increases about adding other materials with it, the researchers used materials such as carbon fiber or other materials, some of them tended to strengthen their samples using fiber reinforcement. Strengthening and then allowing the material to solidify. In experience, used high density polyethylene and made a composite material consisting of high-density polyethylene reinforced with carbon fibers, where we made the composite material by thermal methods. And we conducted creep test, and then we conducted tests similar to those made by previous studies, such as FTIR, hardness test, tensile test and impact.

1.6 Aim of this study

Study the creep viscoelastic behavior of HDPE and it is composite materials (HDPE-CF) different directional of fibers (woven, roving, unidirectional) and different method fabrication shap , Where it is desirable that carbon fibers increase the mechanical properties of high-density polyethylene while preserving its chemical properties.

Chapter two

Theoretical part

2.1 Introduction

This chapter focuses on the viscoelastic properties and relationship of the fibers with polymer to the materials used in the research and their definitions, and it also shows mechanical, rheological, and morphological properties of polymers to the tests and their definitions.

2.2 Polymers

It is a high molecular weight compound composed of repeating subunits. These materials may be organic, inorganic, or mineral-organic, and may be natural or synthetic in origin. Polymers have become an integral and integral role in everyday life due to their unique properties. They are essential materials in everyday industrial sectors, such as adhesives, building materials, paper, clothing, fibers, plastics, ceramics, concrete, liquid crystals and photoresists, and coating[18]

2.3 Polymers Structure

In general, polymers consist of long chains that may be organic, inorganic, or semi-organic composed of a small unit, monomer molecules, which are linked by covalent connections with each other. The long chains, therefore, consist of repeating chemical compositions and are represented by the polymerization degree by the number of repeating units. These macromolecular chains are interconnected physically by Van der Waals force or coherent energy density (CED) which is responsible for the outstanding properties of the polymeric material such as physical, mechanical, thermal, and rheological properties. The strength of the Vander Waals depends largely on the chemical formula, the molecular weight of the substance, as well as the polarity, the space organization of the substance, the tactics of the repeating units of the structure, the presence of heterogeneous atoms in the structure of the repeating units. The units that are repeated within the basic polymer structure determine the identity of the polymer, as shown in the commercial polymers in Table (2.1) [19].

Table 2.1: Chemical Structure of Some Commercial Polymers[19].

Polymer	Chemical structure of the repeating units	Tg (°C)	Tm (°C)	Coherent Energy Density (CED)
Polyethylene	-CH2-CH2-	-125	141	7.9
Polypropylene	-CH2-CH- | CH3	-20	165	8
Polyvinylchloride	-CH2-CH- | Cl	87	190	9.5
Polymethyl methacrylate	CH3 | -CH2-C- | CH3O- C=O	105	160	9.1

2.4 Mechanical Behavior of Polymers

Polymers cannot be classed as certain types of substances such as glass-based solids or viscous liquids because their mechanical characteristics depend so much on the applied load rating, temperature, and strain value. They are usually described as a viscoelastic material, which has an intermediate position between viscous liquid and elastic solids see figure (2.1) B. A polymer can show all the features of glassy, brittle solid, elastic rubber, or a viscous liquid depending on the temperature and time scale of measurement, The law of Hook has been derived from the basic elasticity theory as in Equation No. (2.1), which deals with mechanical characteristics for elastic substances, see figure (2.1) A, where stress is directly proportional to strain when deformation is small. The classical theory of Newtonian rheology deals with the properties of viscous liquids from which Newton's Law is obtained, where the stress is always directly proportional to the rate of strain. In the theory of elasticity, the stress is independent of the rate of strain, and in the theory of hydrodynamics, independent of strain. These two theories do not describe the behavior of viscoelastic materials [20,21].

$$E = \frac{\sigma}{\epsilon} \qquad (2.1)$$

Where:

σ: stress applied.

ϵ: strain.

E: elastic modulus.

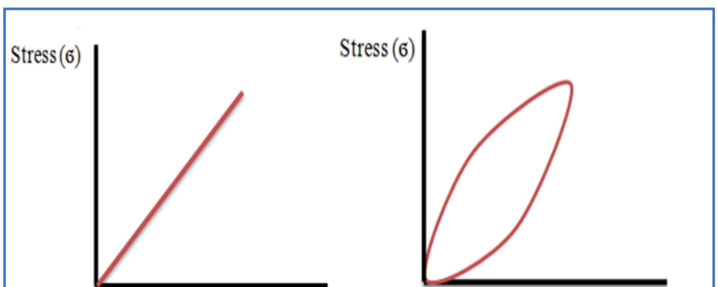


Figure 2.1: Stress-strain curves (A) for a purely elastic material, (B) a viscoelastic material. The area of the loop in curve B represents hysteresis loop[21].

$$\frac{\Delta\epsilon}{\Delta t} = Ae^{\frac{-Q}{RT}} \qquad (2.2)$$

Q : creep energy

A : constant

R : The general constant of gases

T : absolute temperature

2.5 Viscoelastic Behaviour of polymer

One of the most interesting features of polymers is that a given polymer can display all the intermediate range of properties between an elastic solid and a viscous liquid depending on the temperature and the experimentally chosen timescale. which has great commercial importance and great uses. This type of reaction that combines liquid and solid-like properties is called viscoelastic [22].

Viscoelastic materials have elements of both of these properties and, as such, exhibit time-dependent strain showing a 'fading memory'. Such behavior may be linear (stress and strain are proportional) or nonlinear. Whereas elasticity is usually the result of bond stretching along crystallographic planes in an ordered solid, viscoelasticity is the result of the diffusion of atoms or molecules inside an amorphous mmaterials [23].

The linear viscoelastic region, see Fig.(2.2), where the isochronous stress-strain curve follows a straight line. Thus the mechanical behavior of polymeric materials can, at least formally, be handled by the theory of linear viscoelasticity, if the stresses (or strains) are sufficiently small. This linear response region, however, is often small or negligible in comparison with the total range available before yielding or fracture. The advantage of the linear theory is that it is a simple and a good mathematical tool that makes it possible to deal with rather complicated deformation problems in time-dependent materials and to obtain at least approximate results and trends, provided that the stresses are not too large [24].

The nonlinear viscoelastic region where the isochronous stress-strain curves show a deviation from the straight line towards the strain axis with increasing stress, see Fig.(2.2). The deviation occurs beyond a certain strain level, which varies for different polymers depending on the nature of such polymers, time, and temperature. This lies between (0.2 -0.5 %). Thus for stresses of larger magnitudes, the linear theories fail, necessitating the use of nonlinear ones. The extent of creep in polymers can be significant depending on temperature, time, and applied load. To reduce creep, the application of polymers should be at a temperature below the glass transition. However using polymers only below Tg could prove uneconomical, for instance in the case of polypropylene (Tg= -20°C).

Therefore the use of polymers for engineering purposes necessitates further research into ways of measuring creep and reducing its extent [25].

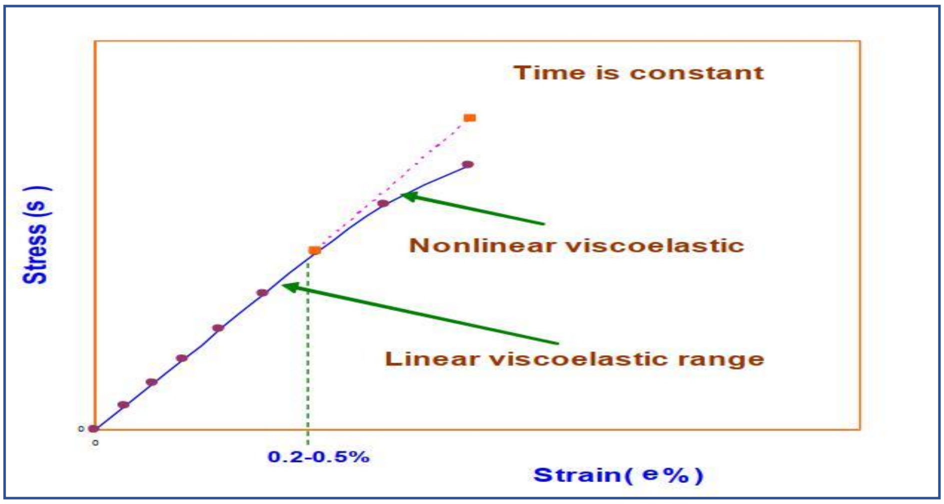


Figure 2.2: Isochronous stress-strain curve [25].

2.6 Linear Viscoelastic

Commonly, the LVE limit is defined as a level of stress or strain above which actual behavior deviates from the behavior predicted based on linearity assumption. However, stress and strain are interrelated time-dependent functions strongly influenced by test type and action of external factors. The concept of an energy threshold as a criterion of LVE limit has the advantage of combining the effects of both stress and strain into one physical function initiating nonlinear behavior[26].

Viscoelastic materials are characterized by their ability to display both viscous and elastic behavior. Most real-life solids demonstrate some viscoelastic properties, and these effects may be particularly important when considering synthetic polymers, The established theory for linear elasticity provides a sound starting point for the study of linear viscoelasticity, Theories of linear viscosity are inferred through the use of well-known models to characterize the behavior of viscoelastic [27].

However, most polymers exhibit nonlinear viscoelastic behavior and the use of known LVE models for the description of their deformability becomes invalid in this case. To choose an appropriate model for characterization of a material time-dependent behavior it is important to know when the contribution of nonlinear components to a general viscoelastic behavior becomes essential. Then, more complicated models of nonlinear viscoelasticity have to be used for further analysis [28].

A valid estimation of limits of LVE has an important role in the analysis of the long-term behavior of composites, in particular, in calculations of their effective characteristics of viscoelasticity and evaluation of stresses in their components. The use of nonlinear models for characterizing the behavior of the components often requires passage to more complicated numerical approaches. The action of external factors, such as temperature or moisture, accelerates relaxation processes and leads to a reduction of service life of polymers [29].

2.6.1 Creep

Creep is the progressive deformation of material at constant stress and temperature. It is used to describe the slow plastic deformation that occurs under prolonged loading, usually at high temperatures. Creep in its simplest form is the progressive accumulation of plastic strain in a specimen or machine part under stress at elevated temperatures over a period of time [30].

polymeric materials can exhibit the phenomenon known as a creep; following an initial, instantaneous linear elastic response the material continues to accumulate strain over time, even when the applied load is held constant. Figure (2.3) shows the stress and typical creep strain response to illustrate this concept[31].

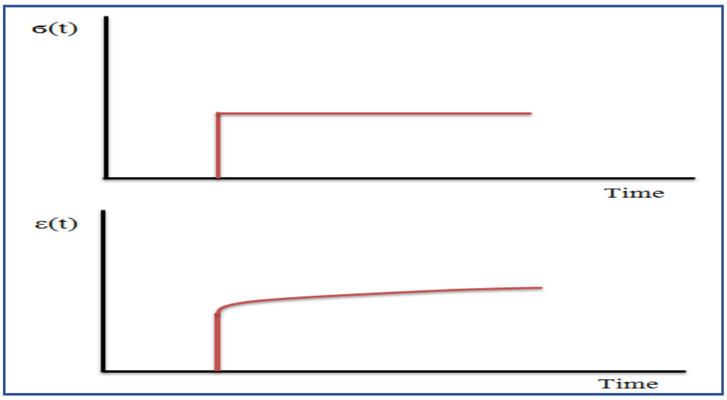


Figure 2.3: Typical creep behavior [31].

the creep strain response to constant applied stress For a viscoelastic material is given by:

$$\varepsilon(t) = D(t)\sigma \qquad (2.3)$$

where D(t) is the creep compliance function, ε(t) is the total creep strain, and σ is the constant applied stress. Understanding the creep behavior of materials is important in design and manufacturing because it can lead to dimensional instability of the final product and failure under constant stress that is significantly lower than the ultimate tensile strength [32].

In general, creep can be described in three stages: primary, secondary, and tertiary. In the first stage, the material undergoes deformation at a decreasing rate, followed by a region where it proceeds at a nearly constant rate. In the third or tertiary stage, it occurs at an increasing rate and ends with fracture see Figure(2.4) [33]

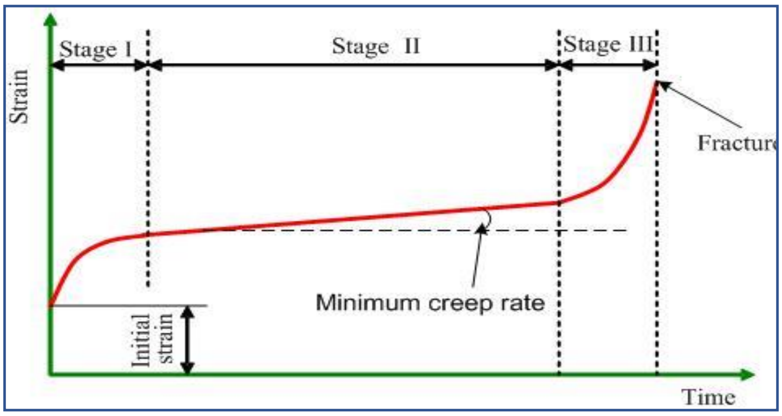


Figure 2.4: Creep stages [33].

The equation governing the rate of steady-state creep is:

$$Creep\ rate\ = \varepsilon' = A\sigma^n\ exp(\frac{-Q}{RT}) \qquad (2.4)$$

Q = activation energy (J/mol).

n = stress exponent.

A = constant.

σ = is the applied stress (MPa).

R= is the universal constant of gases (J/mole K).

T= the temperature at which creep test was performed (K)

The activation energy Q can be determined experimentally, by plotting the natural log of creep rate against the reciprocal of temperature[34].

2.6.2 Stress Relaxation

A typical material in terms of elasticity and rigidly may be subjected to an immediate strain that affects it, then fixed and limited stress is recorded. concerning elastic solid materials that are subjected to strain, The initial stress is directly proportional to the effective strain. Then gradually decreasing with time, see figure (2.5), this elapsed time is called the relaxation time. This behavior is called stress relaxation. The stress values may reach zero in some polymers that have linear behavior, especially random ones [35].

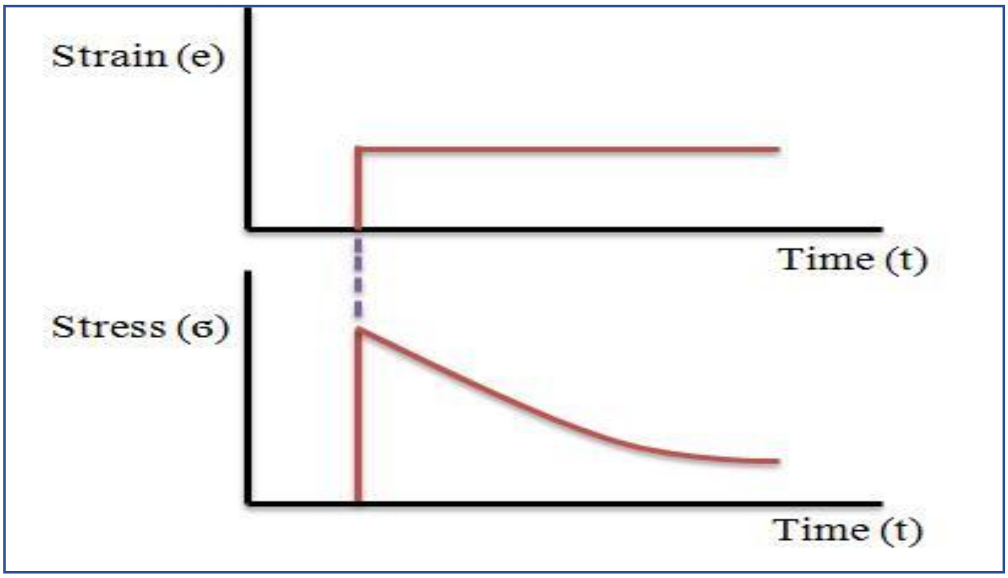


Figure 2.5: Stress relaxation (idealized) [35].

Rheological parameters directly affect the polymer manufacturing process and the properties of the final solid product. Therefore, understanding the rheology of molten polymers is very important in this field. The molten viscosity and relaxation time are among the most important rheological parameters. The amount of elastic energy stored by the molten polymer is related to the relaxation time. Within the backbone of the chain and also outside the chain occurs a variable spectrum of relaxation times related to the relaxation process. The multiplicity of relaxation times usually refers to the interactions that occur in the liquid state between the different polymer chains (i.e. repeats). When the molten polymer undergoes deformation, the entire chain relaxation time is slower than the sub-chain time, and thus the viscoelasticity can be quantified by the relaxation time lengths [36].

2.7 Composite Materials

A composite is a structural material that consists of two or more combined constituents that are combined at a macroscopic level and are not soluble in each other. One constituent is called the reinforcing phase and the one in which it is embedded is called the matrix. The reinforcing phase material may be in the form of fibers, particles, or flakes see figure (2.6). The matrix phase materials are generally continuous, in general, the reinforcements are much stronger and stiffer than the matrix. There are many factors to be considered when designing composite materials. The type of reinforcement and matrix, the geometric arrangement and volume fraction of each constituent, the anticipated mechanical loads, the operating environment for the composite, etc., must all be taken into account[37].

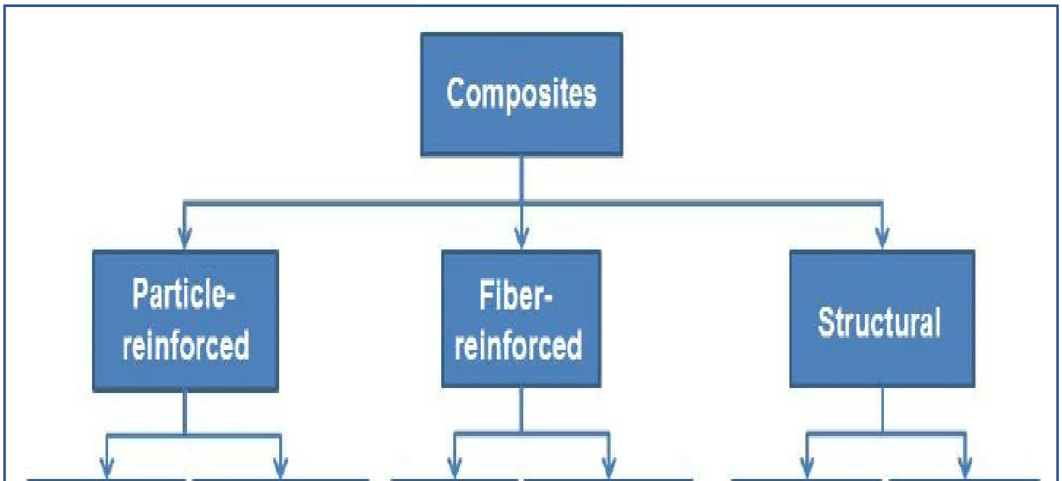


Figure 2.6 : A classification scheme for the various composite types [37].

Over the past few years, the development of new materials has been oriented towards polymer composites to obtain materials, which are characterized by specific properties and low manufacturing costs.

2.7.1 Polymer Matrix Composite

It is a polymeric material that is chosen to be the base or grounding of the composite material. It protects and contains fibers fically, composites based on polyethylene with inorganic fibers are of great research interest. Properties fibers composites are determined by properties of the polymer, properties of fiber, volume fractions of the constituent phase as well as polymer– fiber and fiber- fiber interactions. Interactions of polymer molecules with inorganic fibers affect their properties on the microscopic level (conformation, crystallinity, molecular dynamics) and macroscopic level (stiffness, hardness, toughness) [38].

2.7.2 Reinforcement Materials

Composite materials are usually classified according to the type of reinforcement used. Two broad classes of composites are fibrous. Each has unique properties and application potential and can be subdivided into specific categories as discussed below [39].

2.7.3 Fibrous

A fibrous composite consists of either continuous (long) or discontinuous fibers suspended in a matrix material. Both continuous fibers and discontinuous can be identified from a geometric viewpoint [40] .

2.7.4 Matrix

The matrix is the binder material that supports, separates, and protects the reinforcement materials. The matrix is typically ductile, elastic, or plastic. They can have either linear or nonlinear stress-strain behavior [41].

Thermoplastic polymers and especially polyolefins and polyethylene are produced and consumed today in vast quantities. However, they are seldom used as neat polymers and are usually compounded with mineral fillers. Initially, fibers were used as ‘extenders’ for polymers to reduce cost but as the polymer price decreased and the requirements of modern applications increased, attention has been more and more focused on functionality enhancement. Nowadays, ‘functional fibers ication in the polymer industry almost exclusively, e.g. to improve stiffness, toughness, dimensional stability, electric-insulation, or to decrease the dielectric loss. A prerequisite for functional fibers s full dispersion and uniform spatial distribution in the polymer matrix because agglomerates entrap air and act as sites for fracture initiation, thus leading to premature material failure [42,43].

To cope with the obvious limitations of polymers, for example, low stiffness and low strength, and to expand their applications in different sectors, inorpartic high density polyethylene , carbon fibers , which normally combine the advantages of their constituent phases. Fibers modify the physical and mechanical properties of polymers in many ways It has been shown that dramatic improvements in mechanical properties can be achieved by incorporation of fibers in polymer Stiffness or Young's modulus can be readily improved by adding microparticles since rigid inorganic particles generally have a much higher stiffness than polymer matrices. However, strength strongly depends on the stress transfer between the particles and the matrix. For well-bonded particles, the applied stress can be effectively transferred to thed fibers from the matrix as in figure (2.7) (2.8) [44,45].

The properties of the compound are greatly affected by the direction and nature of those fibers

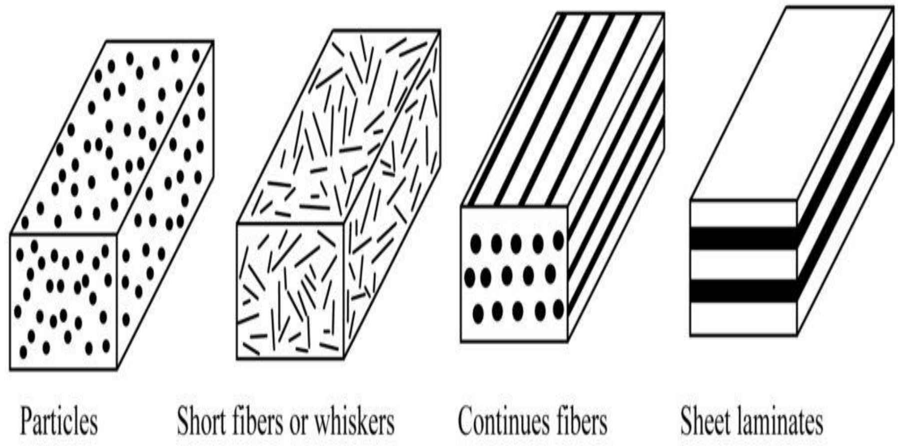
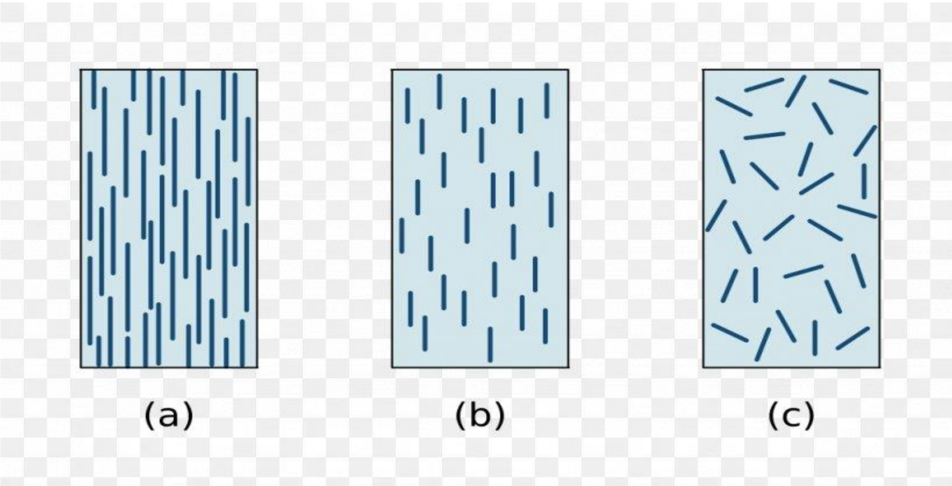


Figure 2.7 : Types of reinforcement in composite materials[44]

Figure 2.8 : (a) Reinforcement with long fibers continuous in one



direction

، (b) Reinforcement in short fibers، (c) Reinforcement in random fibers[45]

2.8 Materials

2.8.1 Polyethylene

Polyethylene of different density ranges can be obtained by chain polymerization of ethylene, the simplest olefin monomer. The observed chemical nature and physical properties of the polymer depend largely on the nature of the catalyst used and conditions employed during polymerization. Polyethylene obtained by polymerization of ethylene is mostly branched polymers, the chemical nature, and frequency of braches being largely dependent on reaction condition and the mechanism involved. Thus poly (ethylene) is the linkage of many ethylene units: $H2C=C2H \rightarrow$ - (CH2-CH2) n- [46].

Factors contributing to the widespread application of polyethylene are low cost, easy processability by a variety of techniques, several types of polyethylene depend on some characteristics in classification, such as density, molecular weight and branches in the polymeric chain .Table(2.2) shows some of these types and their properties [47].

Table 2.2: Some Properties of Various Grades of PE [47].

property	LDPE	LLDPE	MDPE	HDPE	UHMWPE
Tensile strength (MPa)	10–17	20	14	20-35	21
Glass transition temperature (°C)	-110	-110	-118	-110	-118
Melting temperature (°C)	105–120	220–260/105–120 (depends on Branching)	105–120	120–130	123-133
Water absorption, 24 h (%)	0.005–0.015	0.005–0.01	0.01	0.005–0.01	0.005–0.1
Density (g/cm3)	0.917–0.94	0.915–0.95	0.926-0.940	0.95	0.93–0.95

2.8.2 High Density Polyethylene (HDPE)

Is a type of high density plastic. High Density Polyethylene is the toughest of other types of polyethylene, and it is heavier and stiffer compared to LDPE. High Density Polyethylene is lighter than water. It is manufactured by casting or extrusion technique. It can be processed in machine tools, and connected using special welding methods. It is very difficult to be connected using adhesives. It is characterized by a natural appearance and a pompous color similar to beeswax. No harm to food[48].

These materials were classified according to this assessment as follows

1-It is resistant to aging and its life span has been calculated to be at least fifty (50) years but nevertheless its true performance is much mo re than that. They are great at resisting stress cracking and offer that cracking

2- Polyethylene is characterized by good flexibility that gives it ease and durability under the worst working conditions.

3- Light and easy to connect compared to steel, wood and aluminium. It provides ease of forming all types of structures.

4-Thanks to the anti-corrosion property of polyethylene, it maintains its resistance and performance in all working conditions.

5-It is characterized by high shock resistance, and resists breaking even at low temperatures.

6- It does not pollute easily, does not contain toxic substances and is very eas y to clean.

7- High anti-freeze resistance. It is not affected by the freezing of the liquid inside it.

8- In addition to all these characteristics, in terms of raw materials, it is more economical than steel, and wood[49].

2.8.3 Carbon fibers

Are very fine fibers with a diameter between 0.005 and 0.010 millimeters and are composed mostly of carbon atoms. Carbon atoms are bound together in microscopic crystals more or less parallel to the axis of the fibers. This parallel or orientation makes the fibers very strong compared to their size [50].

About carbon fibers are fibers consisting of at least 92% carbon in their composition. The fibers may be short or continuous, and their structure may be crystalline and shapeless, or partially amorphous. The crystal form has the shape of a graphite crystal. The carbon atoms in the layer are linked to each other by: chemical bonds resulting from the sharing of electrons. Metal bonds secured by repositioning their orbits This repositioning of the granite field is a good conductor of heat and electricity. The bond between the layers is the Van der Waals bond, so the layers of carbon can slide easily with respect to one layer to another. The high qualities of carbon fibers are due to the fact that the layers of carbon are not flat, but rather parallel to the axis of the fiber. This method of crystallization is preferred and is known as carbon weaving. For this reason, carbon fibers have many fibers parallel to the axis of the fiber more than perpendicular to it, as well as the conduct of electricity and heat, as they have a higher conductivity along the fiber. Also, the coefficient of volume change along the fiber is less than the perpendicular to the fiber axis. In graphite, the carbon atoms are stacked on top of each other in adjacent layers and when they crystallize in the form of a crystal (hexagonal prism)[51,52].

It is divided into a chemical section and a mechanical section using polyacrylonitrile. The raw material is a filament of fibers heated to high temperatures without any contact with oxygen so that the fibers [53].

This high temperature allows the atoms of the fiber to vibrate strongly until the majority of the non-carbon atoms are composed of a long fiber structure with closed edges between the chains of carbon atoms with a few remaining non-carbon atom [54].

2.9 The effect of temperature on creep

Heat responsive polymers are polymers that exhibit a drastic and discontinuous change in their physical properties with temperature. The term is commonly used when the property in question is the solubility of a particular solvent, but it may also be used when other properties are affected. Heat-responsive polymers belong to the class of materials responsive to stimuli, in contrast to thermosensitive materials (for short, heat sensitive), which constantly change their properties with environmental conditions. In a more stringent sense, heat-responsive polymers display a mixing gap in their temperature profile. Depending on whether the miscibility gap is present at high or low temperatures, there is a degree of shortening (high or low temperature of the critical solution, respectively) as in figure (2.9) [55].

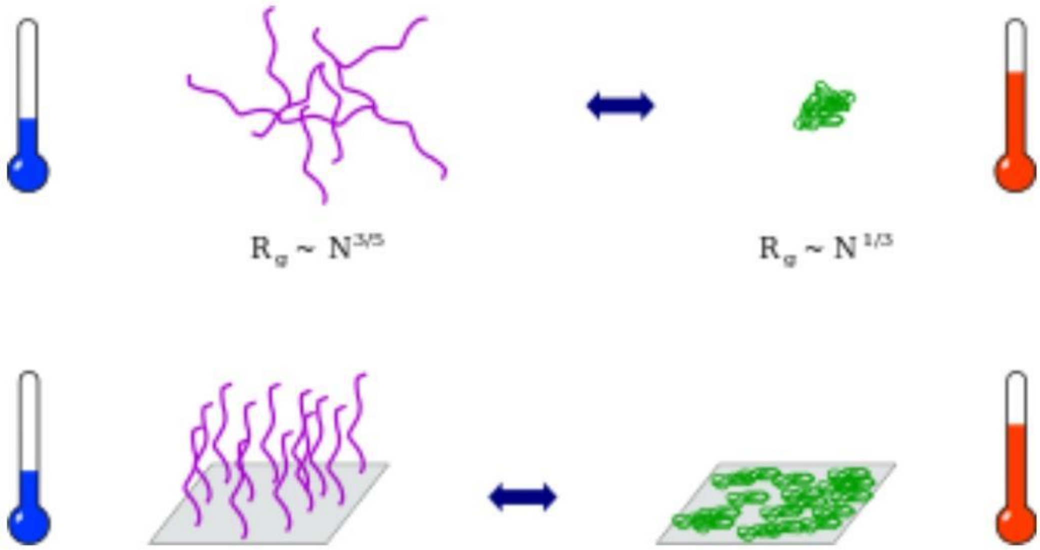


Figure 2.9 : Showing the effect of temperature on polymeric materials[55].

2.10 characteristics test

2.10.1 Creep Test

Creep is the progressive deformation of material at constant stress and temperature. It is used to describe the slow plastic deformation that occurs under prolonged loading, usually at high temperatures. Creep in its simplest form is the progressive accumulation of plastic strain in a specimen or machine part under stress at elevated temperatures over some time [59].

Deformation or strain is measured and plotted as a function of elapsed time up to rupture. Most tests are the constant load type, which yields information about the engineering nature; constant stress tests are employed to provide a better understanding of the mechanisms of creep. Creep often takes place in three stages. In the initial stage, strain occurs at a relatively rapid rate but the rate gradually decreases until it becomes approximately constant during the second stage. This constant creep rate is called the minimum creep rate or steady-state creep rate since it is the slowest creep rate during the test. The slope of the steady-state portion of the creep curve is the creep rate which is calculated from the equation below [60].

$$Creep\ rate\ = \frac{\Delta\epsilon}{\Delta t} \qquad (2.8)$$

$\Delta\epsilon$:is strain change (mm/mm)

Δt:is time change (min)

In the third stage, the strain rate increases, creep necking begins. The stress increases and the specimen deforms at an accelerated rate until failure occurs. The time required for failure to occur is the rupture time[61].

2.10.2 Tensil testing

In mechanical engineering and architecture is a standard test to determine the main properties of materials such as yield strength, yield strength (elongation strength), shear strength, and others.This test causes the sample piece to break[56].

Prepare for tensile testing a sample of the material having a specific section. It is attached at both ends to the tension device, and an increasing tension force is applied to it until the samples are cut. During the experiment, the tensile strength F and the elongation of the ΔL sample were continuously measured. The mechanical stress on the sample is calculated from the strength and cross-section of the sample before testing it as in figure(2.10) [57].

$$\sigma = \frac{F}{So} \qquad (2.5)$$

Change of the sample length of the ΔL can calculate the total elongation εt , relative to the original length of the sample Lo:

$$\varepsilon\tau = \frac{\Delta L}{Lo} \qquad (2.6)$$

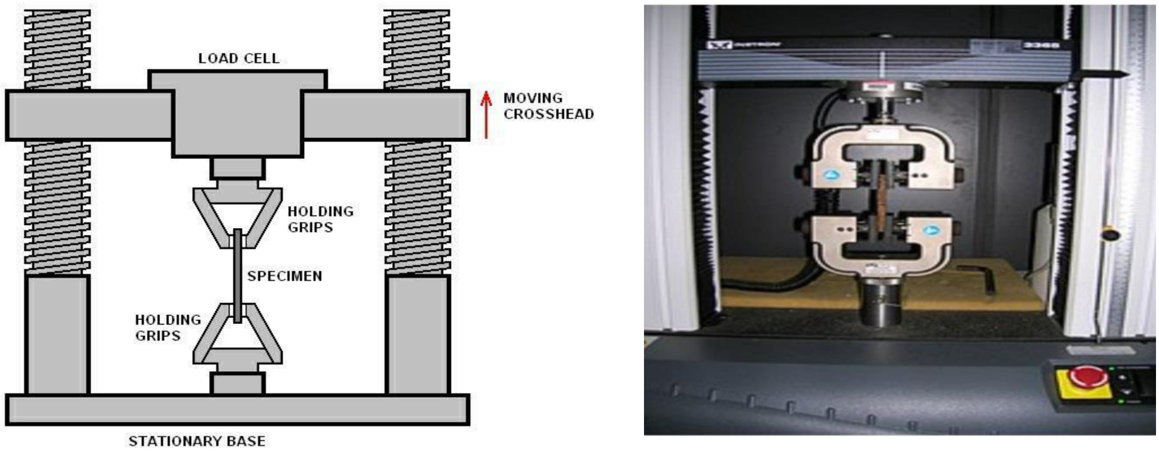


Figure 2.10 : Tensile experience[57]

2.10.3 Impact Test

When a material is subjected to a sudden load (dynamic load) i.e. high strain rate, it will follow behavior different from its behavior when subjected to static load. The material may follow ductile behavior when exposed to static load (tensile and bending test). But when you expose it to dynamic load(impact test), it will follow brittle behavior. Impact resistance can be obtained from the following relationship[58].

$$I.S = \frac{Uc}{A} \qquad (2.7)$$

Where:-

I.S: impact resistance of the material (J/m^2).

Uc: impact energy (J).

A: cross-sectional area of the sample (m2).

2.10.4 Hardness test

As for the hardness test, a Shore-D device (Shore Durometer) was used to measure the hardness of polymeric materials, which is a compass-

like device and contains a needle in the middle. The surface of the sample to be examined, and then pressure is applied to the sample to measure the hardness of the sample as in figure (2.11) [62].

Figure 2.11 : Shows the hardness measuring device

2.10.5 Infrared Spectroscopy Test

Infrared (IR) spectroscopy is a popular way to describe polymers, FT-IR standard for (Fourier Transform Infrared), the preferred method of infrared spectroscopy. In infrared spectroscopy, it is passed IR radiation through a sample. Some of the infrared radiation is absorbed by the sample and some of it is passed through (transmitted) [63].

The resulting spectrum represents the molecular absorption and transmission and creates a molecular fingerprint of the sample. Since a fingerprint is a unique property of each material and no two molecular structures produce the same infrared spectrum which makes infrared spectroscopy useful for many types of analysis. FTIR can identify unknown materials, the amount of components in a mixture, and the quality or consistency of a sample [64].

Chapter three
Experimental part

3.1 Introduction

The practical part empirically consists of

1- Samples of high-density polyethylene, which were obtained in the form of granules, and then were extruded by a double-screw extruder in the form of sheets, and then they were precisely cut by a CNC machine to fit the creep device.

2-Talk about how to manufacture the composite material, which was made in three types, namely woven, roving and unidirectional, using an hydraulic press, and then the samples were cut by a CNC device as well.

As for creep device ،creep device in college works with one axis. The device has been modified so that it works with two axes to suit the two-axis creep.

After that, about the creep experience in general, and since put a lot of heat on the samples in order to make them in the shapes want, so studied the effect of heat on the samples practically in this chapter, and finally also talked about mechanical and physical tests, which are (hardness,tensile, impact , creep ، FTIR) tests as they are one of the tests that required to perform on the samples to.

3.2 materials

The materials used in the preparation of the samples consist of a polymer matrix material (high-density polyethylene) and a reinforcing material consisting of fibers (carbon fiber) and they were used separately for each type as in figure (3.1) and figure(3.2) , figure(3.3) , figure(3.4) .

Figure 3.1 : Primary high density polyethylene granules

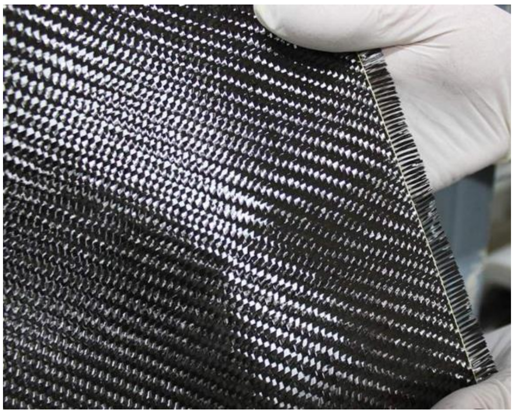


Figure 3.2 : Carbon fibers type woven

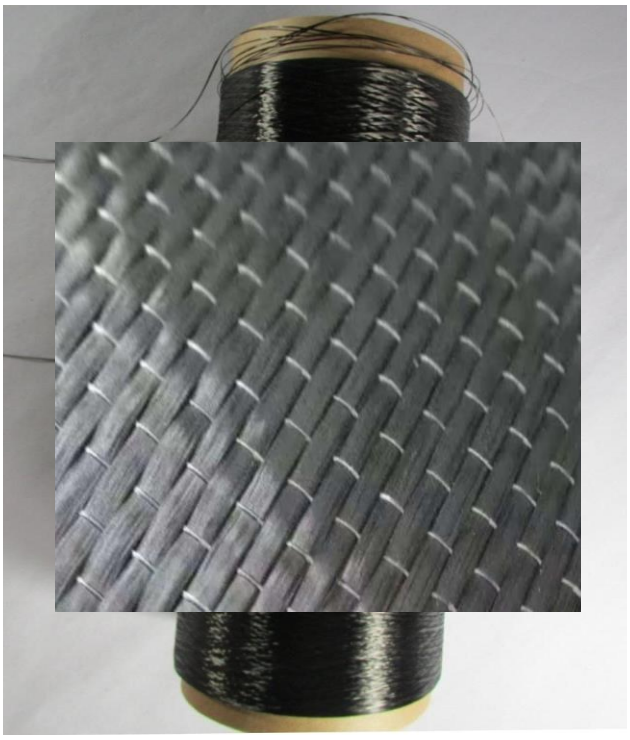


Figure 3.3 : Carbon fibers type roving

Figure 3.4 : Carbon fibers type unidirectional

3.2.1 Matrix material

HDPE was obtained as pellets from of From the laboratories of the Faculty of Materials Engineering Sone characteristics of HDPE are shown table (3.1) according to produced compony[66].

Table 3.1: Properties of HDPE

Properties	Units	Values
Density	g/cm^3	0.93
Hardness	Shore D	52

strength Tensile	MPa	10
Melting point	°C	135

3.2.2 Reinforced Materials

Type of fibers were used in this work as materials with the matrix (HDPE). The following is a summary of the materials used:

Carbon fibers

The reinforcing material is The carbon (C) fibers provided by the Thomas Baker Mumbai company, India,. Was used as fiber to obtain the new modified composite material (HDPE/ Cf), table (3.2) shows properties of carbon fibers according to produced company[77]:

Table 3.2: Properties of carbon fiber

The Properties	Units	Values
The Density	g/cm^3	1.8-2.1
Melting point	°C	3500
Solubility in water[%w]		Insoluble in water

3.3 Manufacture of HDPE samples

This process is done by taking amount of HDPE, taking into account the damage of some HDPE during the extrusion process, due to the difficulty of extrusion except with high temperatures and shrinkage.

1- HDPE is dried for 24 hours to get rid of moisture and also to avoid shrinkage during extrusion at room temperature which is 25 °C

2- The material is placed in the screw extruder in batches, each batch is about 10 grams .

3-These granules are placed in the upper funnel of the device, where they then pass to the heating unit and then to the melting unit in which temperatures reach 174 degrees Celsius or more, as in the case of HDPE,

as this material at temperatures that are less than 174 it suffers from shrinkage .

4 -The substance comes out in the form of a sticky molten substance, where the working person must take precautions, due to the high temperature.

5 -The fuse is pressed to seal the sheets of HDPE.

6 -HDPE sheets see figure (3.5) are then taken and cut by laser using CNC as in figure (3.6) and figure (3.7) technology to obtain samples of equal and consistent dimensions as well as to avoid cracks that occur when cutting samples by other methods.In the biaxial creep experiment of the continuous polymeric fiber composite, the samples are cut in the form of a cross in order to suit the experiment that depends on applying loads at the same time and the same quantity in two different directions, one in the vertical direction and the other in the horizontal direction, and then the creep of the sample is monitored and the results recorded.Samples are cut with the same dimensions for both directions The thickness of the high-density polyethylene sheets is 3.5 mm.

Figure 3.5 : High density polyethylene sheet extruded by twin screw extruder

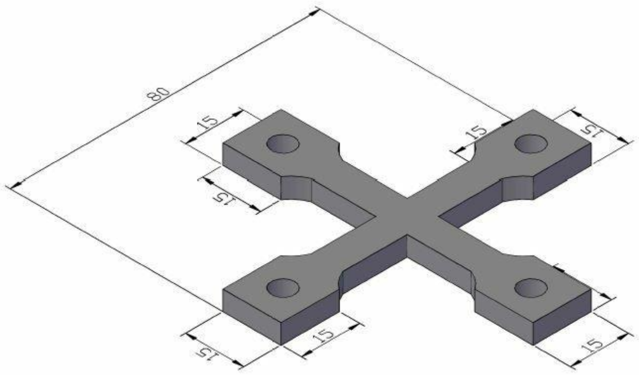


Figure 3.6 : showing the shape and dimensions of the samples used in the experiments.

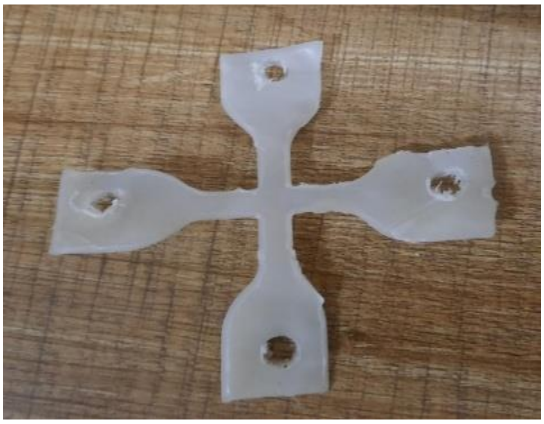


Figure 3.7 : sample of high-density polyethylene after being cut by a Computer Numerically Controlled CNC machine

3.4 Twin screw extruder

A double-screw extruder was used for the purpose of converting the dried granules of high-density polyethylene into sheets. The process was carried out by placing HDPE in batches of about 10 grams, and a high temperature of 174 was used to overcome the shrinkage it suffers from. HDPE at lower temperatures.

Extrusion operation was achieved by a twin extruder (SLJ-30A) located in the College of Materials Engineering - University of Babylon figure (3.8). The dry mixtures are entered into the cylindrical chamber of the extruder, which heats the polymeric material through heaters spread along the cavity. The screw inside the extruder is set at a rate of (40 rpm) revolutions per minute. The screw works to mix the resin and break up agglomerates. The material is pressed at the end of the screw through a narrow neck and then goes through a rolling process after leaving the extruder to form into sheets see figure (3.8).

Figure 3.8 : The twin-screw extruder

3.5 Composite material manufacturing.

A device (hydraulic electric-thermal press) was used, because it was not possible to manufacture a composite material by means of the double screw extruder, because the HDPE material cooled quickly and this was observed in practice as well. Special mold to withstand very high temperatures. Where HDPE is placed at the bottom of the mold in the form of a layer of granules, then carbon fibers are placed, and then another layer of HDPE is placed, and the mold is closed. The device presses with a high temperature of up to 200 degrees celsius. Note that the composite material is left to cool. Since the cooling rate has a role in its final properties, we then get sheets of the composite materials

3.6 Determine volume fraction of the fibers

To find volume fraction of the fiber the following equation will be applied obtained by calculation based on the rule of mixtures for the density, using the measured density of the composite and the known densities of the fiber and matrix. The equation is

$$\rho_c = v_f \rho_f + (1 - v_f)\rho_m$$

$$v_m = (1 - v_f)$$

where ρ_c is the density of the composite, ρ_f is the density of the fiber, ρ_m is the density of the matrix, and v_f is the volume fraction of the fiber , v_m is the volume fraction of the matrix .

ρ_f=1.8 g/cm³

ρ_m=0.93 g/cm³

Where the density will be calculated only for the central region of the composite sample because it is the one that is subjected to stress or the stress is concentrated on it, as the dimensions of this region are 40 mm in length, 7.5 mm in width, and 3 mm in thickness .

$v_f = 0.36$

$0.36 \times 100\%$

$36\ \%$ vol

For the manufacture of composite samples by electro-thermal hydraulic press devicesee figure (3.9)

Figure 3.9 : Electro-thermal hydraulic device

Molds made of stainless steel are used to withstand high heat and high pressure simplified by means of a hydraulic piston device. The molds are 30 cm long and about 12.5 cm wide, as shown in the figure (3.10) .

Figure 3.10 : Iron formwork

3.7 Type of composite sample (HDPE/Cf)

Three types of composite samples will be manufactured, which differ according to the direction and angle of the reinforcing fibers

1-Unidirectional in which the fibers are directed only in one direction, either vertically or horizontally, meaning that the angle is zero as shown in the figure (3.13).

2-Cloth (woven) In this case, the carbon fibers are in two directions crossed perpendicular at the same time, one of them is horizontal and the other is vertical, the angle between them is 90 or zero degrees as shown in the figure (3.14).

3-Roving, which is the third type of samples of composite materials, in which the carbon fibers are intermittently shaped like the letter (X) and at an angle about 30 degrees or more or less within this range which cannot be 90 as in the case of woven as shown in the figure (3.15).

For the manufacture of composite samples, high-density polyethylene granules are placed at the bottom of the mold with a calculated weight, then the fibers are placed according to the desired shape of the fibers, and then these fibers are covered with the same amount of high-density polyethylene granules that were placed at the bottom of the mold as in the figure (3.11).

Figure 3.11 : Composite samples before manufacturing by hydraulic press

Then the mold is closed and transferred to the electro-thermal hydraulic piston device in order to obtain a sheet of the composite material as shown in the figure (3.12) .

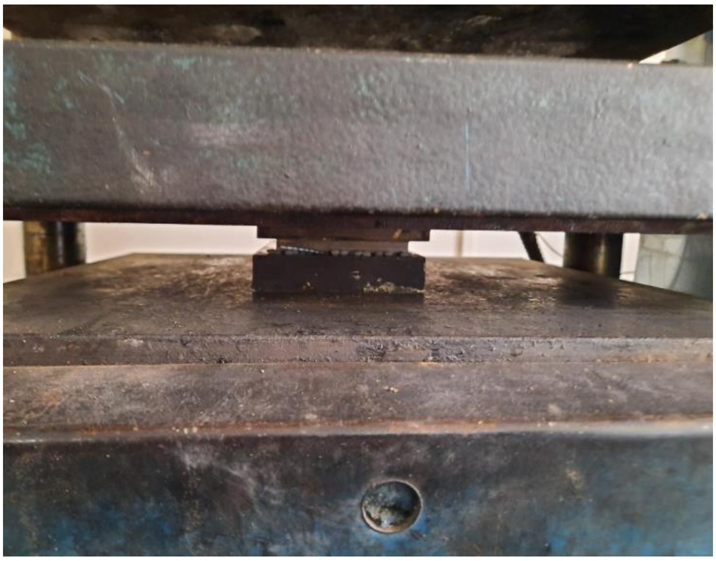


Figure 3.12 : mold during the pressing process

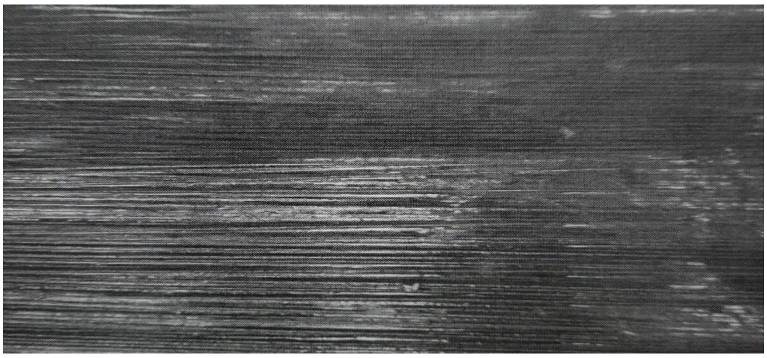


Figure 3.13 : Unidirectional sheet

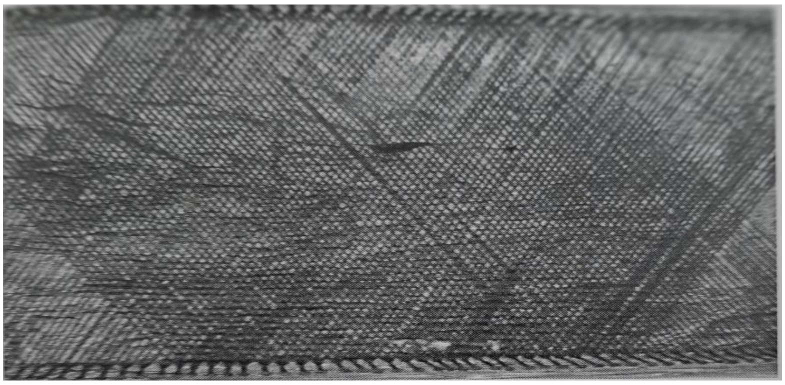


Figure 3.14 : Woven sheet

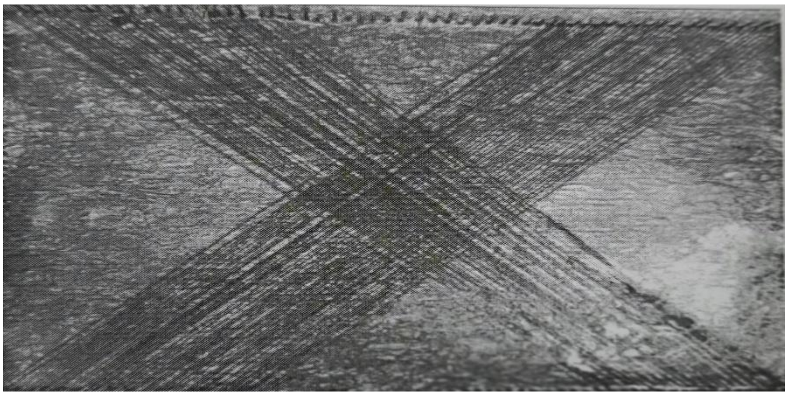


Figure 3.15 : Roving sheet

3.8 Creep test device

The WP 600 Kriechtester creep test machine was used to perform basic experiments demonstrating the behavior of polymeric materials under constant load and providing a clear idea of the suitability of polymeric materials for a particular application. Figure (3 .16) and figure (3.17) shows the creep test equipment found in the laboratories of the College of Materials Engineering at the University of Babylon to perform this test under constant load and different temperatures. The dimensions of the samples in this test are within specification ASTM D 2990, and this test method was used to know creep behavior plastics reinforced and non-reinforced.

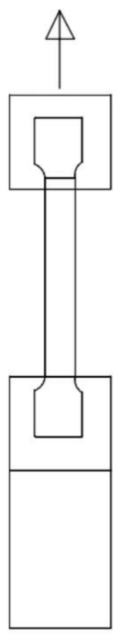


Figure 3.16 : Showing how the uniaxial creep works

Figure 3.17 : Uniaxial Creep Device

Note from the previous pictures a uniaxial creep measuring device for polymeric materials, as it works on the basis of material creep in one direction, which is the vertical direction, where creep rienced by placing weights, which is a set of weights as if 3.5N or 5N are in place on the arm of the device . These weights It will affect the polymeric materials and pull them in the direction of shedding the load, and since the samples will begin to gradually expand. and shorten in the middle until it reaches a point where the sample is cut into two parts, there is no creep in one direction, the modified the device and made it work in two directions, that is, it became bi-axial, through the manufacture of two arms , the first on which is placed the creep scale the weight and the weight to the side of the sample , and this arm consists of

1- base based The arm has

2- screw-shaped columns in order to be reduced or enlarged to suit the position of the sample .

3- A place to install the sample.

4- It contains a pulley to allow the porter's thread to move easily see figure(3.18).

On the other side of this arm , another arm was made for installation

As a result, two arms were designed, the one on the left that is used for fixation, and the one on the right, the load is shed from its side, and it contains a counter to measure the creep of the material when the load is shed.

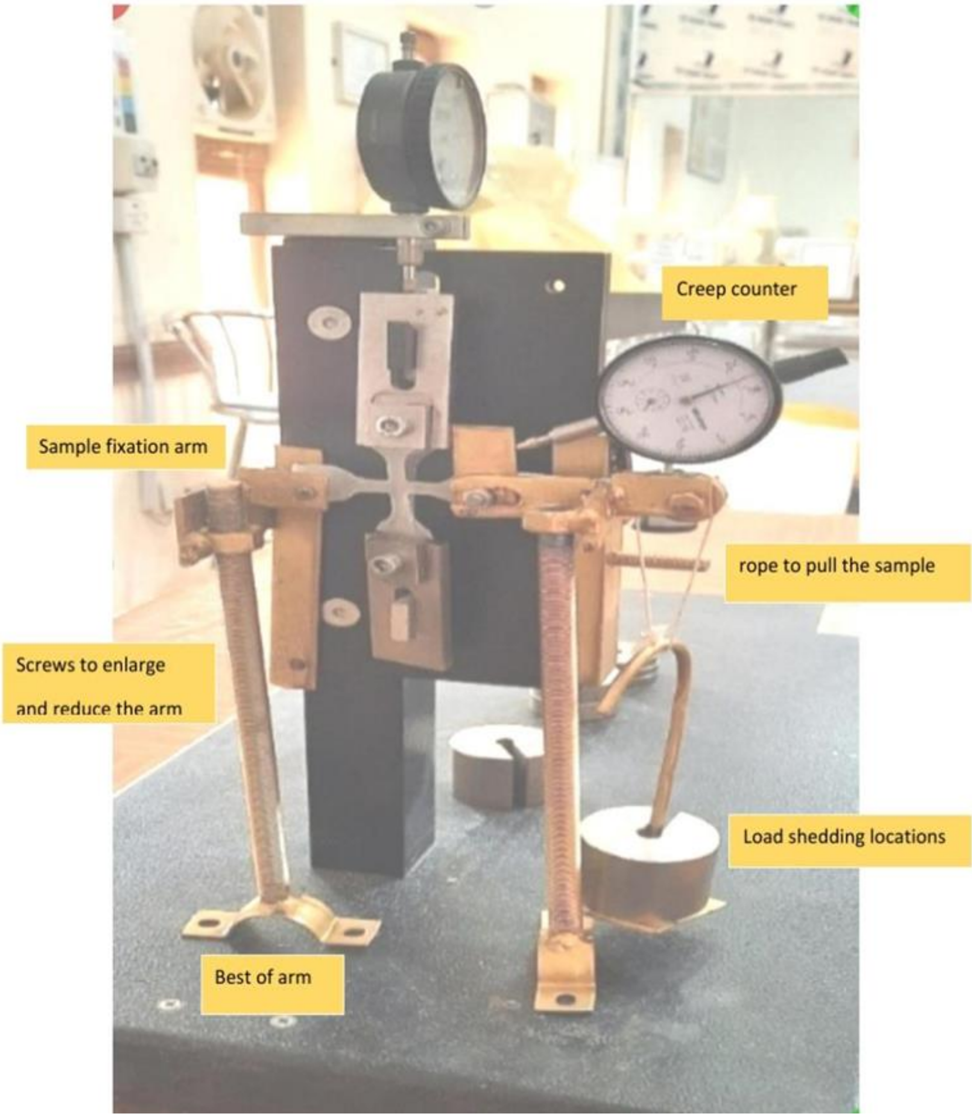


Figure 3.18 : Diagram showing the biaxial creep device

3.9 Tests

3.9.1 Tensile Test

Specimens were cut according to ASTM (D638-03), Microcomputer Controlled Universal Electronic Testing Machine Model is the used for Tensile Traits Testing (WDW-5E), Supplied from the People's Republic of China, Made in China, Found in Laboratory of College of the Materials Engineering / University of Babylon shown in Figure (3.19). The test was performed at room temperature with a load of 5 kN and a speed of 10 mm/min for all samples. The axial force is applied to the sample until the sample detaches, the computer attached to the machine records a stress-strain

Figure 3.19 : Tensile test machine.

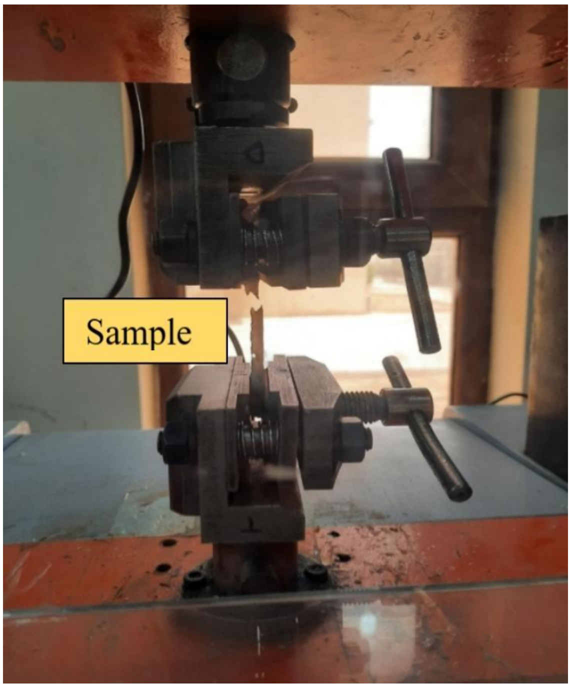


Figure 3.20: The breaking moment of the high-density polyethylene sample produced by the hydraulic piston device in the tensile test

3.9.2 Impact Test

As shown in Figure (3.20), the impact tests have been carried out employing the impact device (Type GUNT Hamburg), which available in the Polymer and Petrochemical Industries Department at the Materials Engineering College/ University of Babylon.

The impact test has been conducted employing an un-notched Charpy-Impact testing device following the standard (ASTM D256), using a rectangular specimen having dimensions (55 x 10 x 6) mm as in figure (3.20) and figure (3.21) . In this test each value of the energy represents the average of five samples.

This instrument is consisting from an energy gauge and a pendulum. The Charpy's impact test comprises a standard specimen that can be broken with a single blow from a swinging hammer. The procedure for examining in this instrument involves raising the pendulum to its maximum height and maintaining its rigid position.

In this test the specimen is fixed in a horizontal position. Then the energy gauge is reset to zero, and the pendulum was released, whereas its potential energy is converted to kinetic energy, and a portion of this kinetic energy is utilized for fracturing the specimen, while the energy gauge measures the tested model's fracture energy.

For determining the Charpy impact strength of an un-notched sample, the energy absorbed via the model's breaking is dividing to its original cross-sectional area [92]:

$G=W/A$

Where:

G: The material's impact strength (J/m^2)

W: The absorbed energy (J)

A: The sample's cross-sectional area (m^2)

Figure 3.21 : The machine of Impact test.

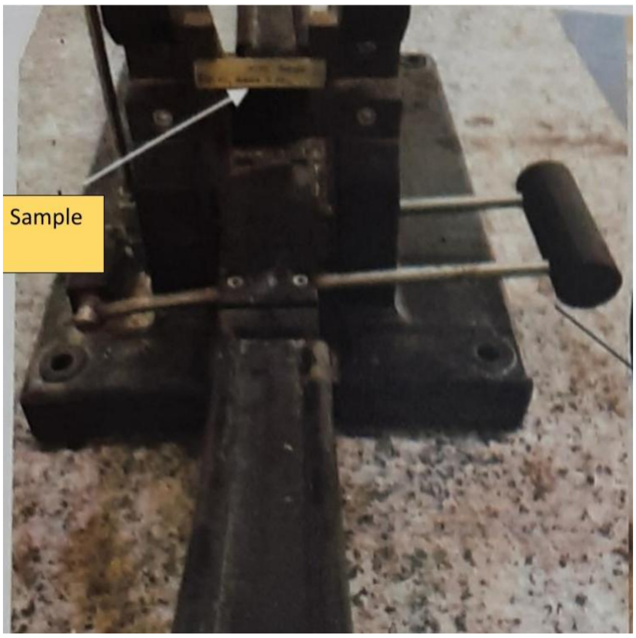


Figure 3.22 : Sample location

3.9.3 Hardness test

In this study, the hardness of the sample was measured by utilizing a hardness test digital device (Type TIME®5431), as depicted in figure (3.22). This device was available in the Polymer and Petrochemical Industries Department in the Materials Engineering College at the University of Babylon. The hardness test was done for pure HDPE samples, homogenies composites (HDPE+Cf), and both sides of all the functionally graded material models. The test was performed via maintaining the penetration of the tool of Shore D instrument which fixed upon the surface of the sample. At last, the hardness number is appearing upon the Electronic Display device.

The Shore D test has been used to measure the hardness of the surface. with the hardness device indenter connected to a digital scale that gradually increasing within (0-100 unit), capacity of (5±0.5) kg and a dwell time (10 sec) per standard (ASTM D2240). In shore D tests, the indenter is forced into the material's surface to be tested for a specified time. The hardness results were recorded directly by digital scale, and each measurement requires only a few seconds. The hardness test results are the average of (5) readings from different samples. For examining the gradation properties through thickness, a hardness test was performed; where the hardness property for each layer was determined by utilizing grinding and polishing machine type (Mopao-160E) to prepare the samples for hardness tests through remove layers via layers. The concept is to obtain the hardness profile through the model and compare the hardness of layers and the intermediate graded layers.

Figure 3.23 : Shows the hardness measuring device

3.9.4 Fourier Transforms Infrared Spectroscopy Analysis (FTIR)

Samples are characterized using the Fourier transform infrared spectra methodology. This was accomplished using an IR Affinity-1 (manufactured in Japan), which is an available instrument from the Materials Engineering college laboratory/University of Babylon's (ASTM D2412) .

Chapter four

Results and Discussion

4.1 Introduction

In this chapter, present the results obtained from various tests that were conducted on the samples, and then discuss these results, where initially review the results physical tests such as (FTIR) and discuss it, and then review the results of the mechanical tests such as (Hardness, Impact, Tensile) and discuss it, and finally review the results of the biaxial creep test with a drawing Curves and discuss for the following samples

4.2 Creep results

Figure(4.1) shows the relation between the creep strain for HDPE reinforced by varying carbon fibers (vertical) direction of Sample, The creep test was carried out under the following conditions, a temperature of 20 0c and an applied load of 5KN. from this chart can be seen the creep strain it very clear that the creep strain for HDPE pure is higher than the creep strain value for the HDPE composites and its behavior increasing with time, to clarify the effect the fibers behavior creep of polymer, need to know the mechanism by which creep performance is affected when fibers are inserted, the creep characteristics are modified by the presence of stiff fibers of Carbon, which in effect will deform throughout the test. It should also be noted that the same applied loads were used for all composite materials, therefore, the greater stiffness of the composites meant that the elastic strains were lower and so the true stresses were also lower. This effect will reduce the creep strain. This is consistent with the expected load transfer to the fibers (and also with the concept of the fibers remaining well-bonded to the matrix). On the other hand, the improvement is not a dramatic one when compared with the other HDPE composites, which might have been expected from the limited nature of the load transfer (and indeed of the stiffening effect) [60].

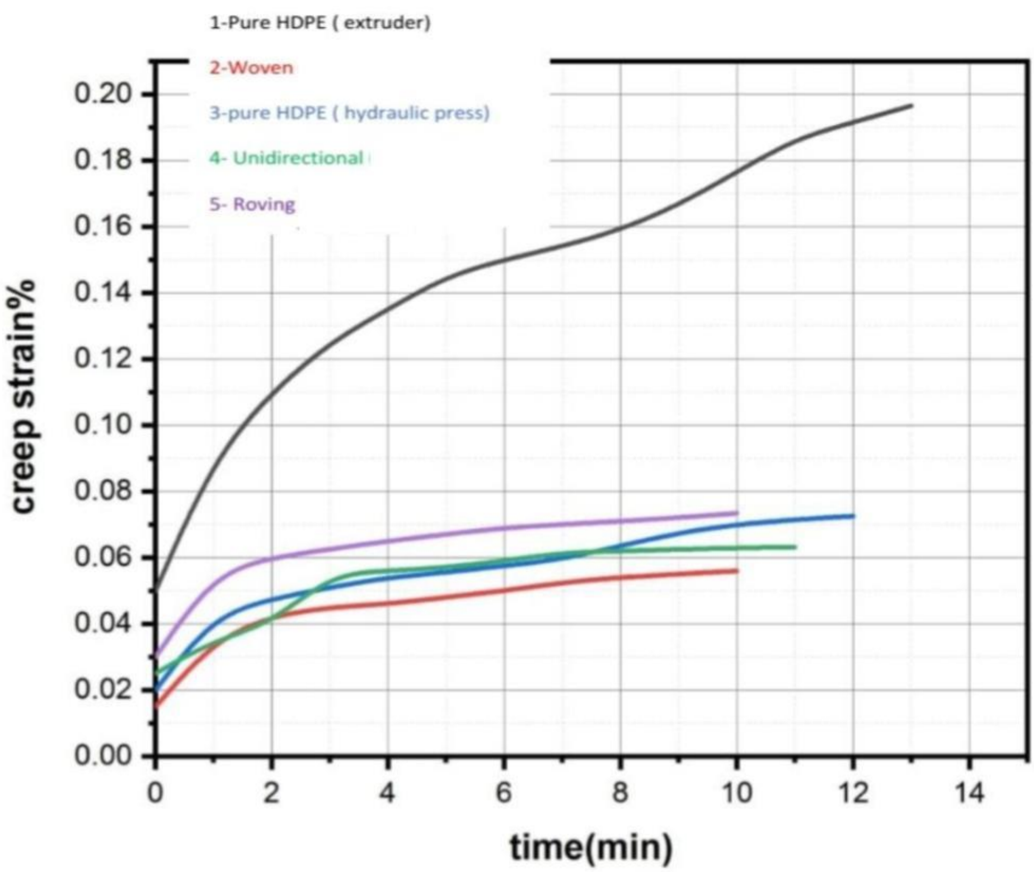


Figure 4.1 : Percentage creep strain for HDPE /Carbon fibers composite

.

This graph indicate that the creep strain for HDPE pure is higher than the creep strain value for the HDPE composites and its behavior increasing with time. With an increase in the fibers content, the creep performance with increased the fibers , were The sample was presented the good resistance in this creep test, It showed the best performance off all the other composites including the virgin HDPE matrix, When carbon fiber content is increased, the creep performance begins to decrease gradually over time. The creep performance depends on the physical properties of HDPE composites, where interference at moderate proportions of the fibers allows to provide support for the polymer matrix, which improves the performance of the creep strain., thus reducing the chances of the fibers dispersal and increasing the chances of, especially at high percentages fibers, which contributed to a relative decrease in the creep strength and on nevertheless, the values of HDPE composites remain close to each other in their creep resistance due to the interference of the fibers between the polymer chains.

From the previous creep schemes, as well as through previous tests, conclude that the strongest types of samples are the woven composite samples, followed by the Roving samples and then the unidirectional. The reason is due to the strengthening of these samples with fibers. We also note that the high-density polyethylene samples made by the electro-thermal hydraulic piston device are stronger. From samples of high density polyethylene made by twin screw extruder

One of the obvious questions is why did high-density polyethylene material expand to this degree . The answer to this question is that polymeric materials follow the method of processing them, since this material was made by extrusion process in the form of sheets as previously mentioned, so the extrusion process produced is continuous, and the end cannot be reached, because the mold It is open, as the pressure is low in extrusion and we cannot raise it, so we do not get a product with good properties. As a result, we resorted to composite materials and resorted to the manufacture of samples using an electro-thermal hydraulic press.

4.3 Tensile results

When the as-prepared samples are mounted in the tensile testing machine and the test is performed, the graph between the stress (σ) and the sample strain (ε) was obtained from the graph of the tensile testing machine. Through this scheme, the values of stress and strain were calculated using equations, respectively

$\sigma = F / So$

$\varepsilon t = \Delta L/Lo$

note from the diagram to pure HDPE that the elastic area is higher where the shed load was able to easily bypass it after a short period of time. The sample will expand and shorten in the middle area, where the width will gradually decrease with time, and note through the diagram that the sample expands a lot during the loading on it, where a very high strain is shown, and from this conclude that this material is ductile and has a low elastic modulus, if can through this diagram knowing the properties of this material, as it is not of high propertie.

While vertical roving samples gave more resistance to stress resulting from tensile strength, the reason is that it contains a high percentage of fibers directed towards shedding the load, where the fibers here play a major role in bearing the applied loads, as they played here the function of transferring the applied stress, as it can come out with a result as it increases. The amount of fibers increases the tensile strength.

The stress-strain curves were drawn for all the prepared samples and numbers showing the mechanical behavior of these curves were for different types es of materials, figure (4.2)

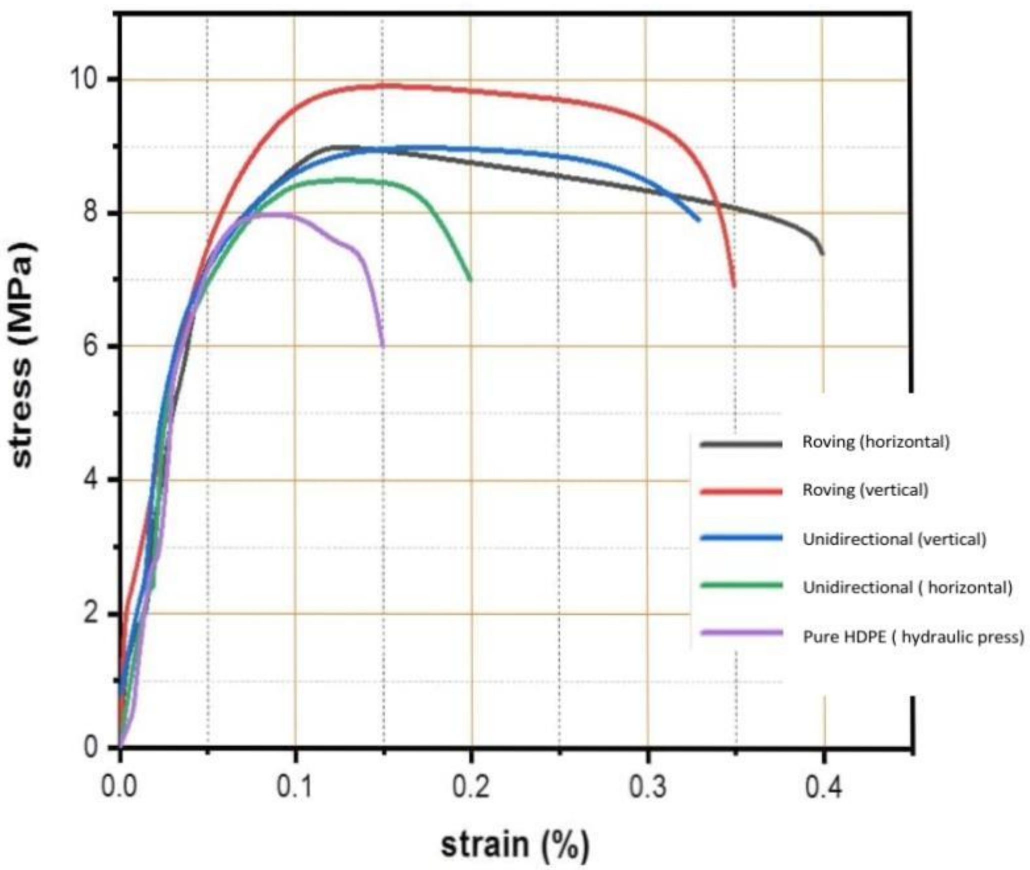


Figure 4.2 : Stress-strain curve for HDPE pure and reinforcement by carbon fibers

4.4 Impact results

Since the samples that made are in the form of a cross, the results obtained from the impact test, which is exposing both directions of the sample to impact, so the results are as follows see figure (4.3).

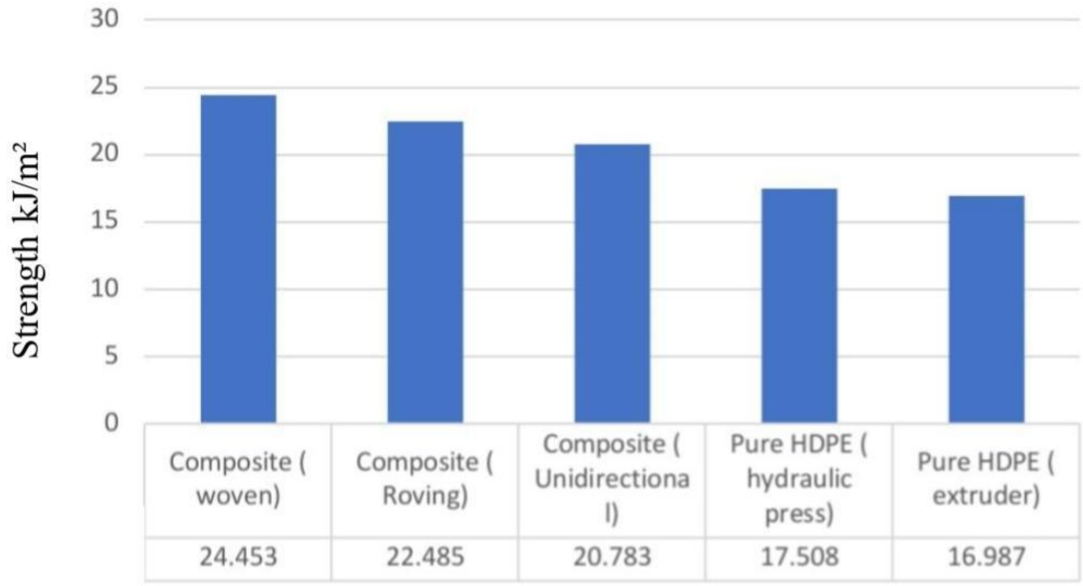


Figure 4.3 : Showing the results of the impact , from the vertical direction of the samples

When the reinforcement materials are appropriately acreep19to the correct combinations of the bonding materials, we will obtain exceptional properties and then the composite material is called according to the two natures of the base material and the reinforcing material. This is like the composite material: (polymer - metal), (polymer - polymer). If the

composite material combines in its properties the composition of the bonding material and the composition of the (reinforcing) fibers and the nature of the bonding between the base material and the reinforcing material, and the properties of the composite material are affected by the geometrical shape of the method of reinforcement with the material reinforcement [58].

4.5 Hardness result shore durometer

Note through the previous hardness measurement results that the hardness of the composite material consisting of high-density polyethylene reinforced with carbon fibers is more than that of high-density polyethylene alone, and this is due to two important reasons.

1-A key role in increasing the stiffness, as the main fiber’s function is to strengthen the mechanical properties, and when say mechanical properties, mean by it an increase in the modulus of elasticity and consequently an increase in the stiffness and thus an increase in dimensional stability. A composite is a mixture of more than one substance that is physically interconnected. Each substance will show its properties in a certain percentage. For example, we will notice that 60% of the properties of the composite material are the properties of the reinforcing fibers. 40% are the properties of the base material. As a result, the composite material has become more solid in the hardness test. know that these ratios cancontrolled, as an increase in one of them will be reflected in the other and therefore will decrease because the relationship between them is inverse .

2 - The manufacturing method plays a very important role in giving hardness to the samples. In experience, the composite material gained more rigidity than the high-density polyethylene material alone, because the composite material was manufactured in a closed mold by means of an electro-thermal hydraulic press, unlike the high-density polyethylene samples alone that were manufactured. With an open mold by extrusion method, as the closed mold has dimensional stability and the possibility of raising the temperature and pressure to high levels and thus obtaining high properties. In contrast to the open mold extrusion, the product is continuous

and its end cannot be reached. Also, the properties of the product are not at the required level where the hardness is low see Figure (4.4).

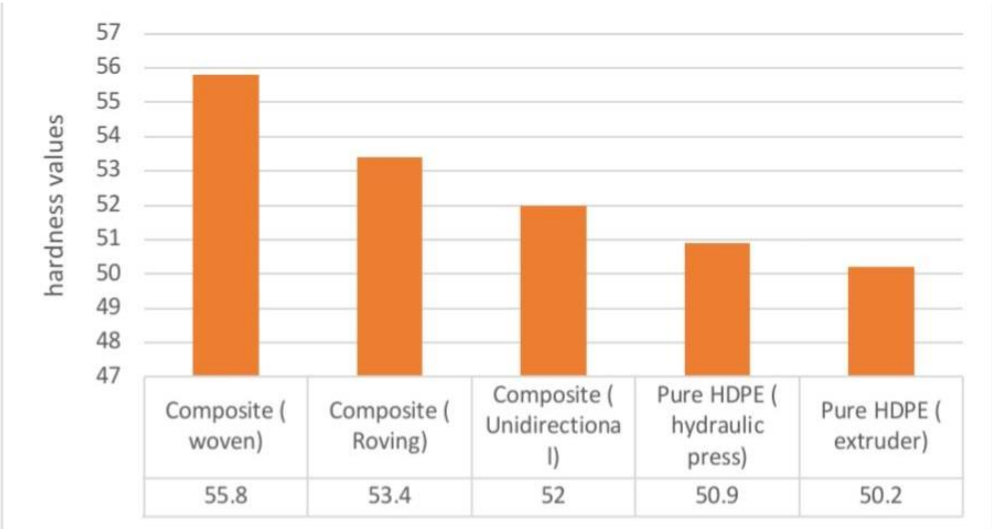


Figure 4.4 : Showing the results of the hardness .

4.6 Fourier Transform Infrared (FTIR) result

FTIR test was used for HDPE pure and HDPE Reinforced with Carbon fiber prepared by method extrusion to verify and occurrence of any reaction between reinforcement fiber and polymers matrix for HDPE composite, The band values and the changing in intensity or shifting of peaks for composites recorded by FTIR are shown in table (4-1), which were derived from the figures (4.5),(4.6),(4.7) respectively.

FTIR for HDPE virgin shows many bands at distinct peaks such as bands at 2916.37 and 2846.93 cm-1 for (C-H stretching), the band at 1465.90 cm-1 for(CH2 scissoring), The band at 717.52 cm-1 for (CH2 rocking)as in figure (4.1)(4.2).

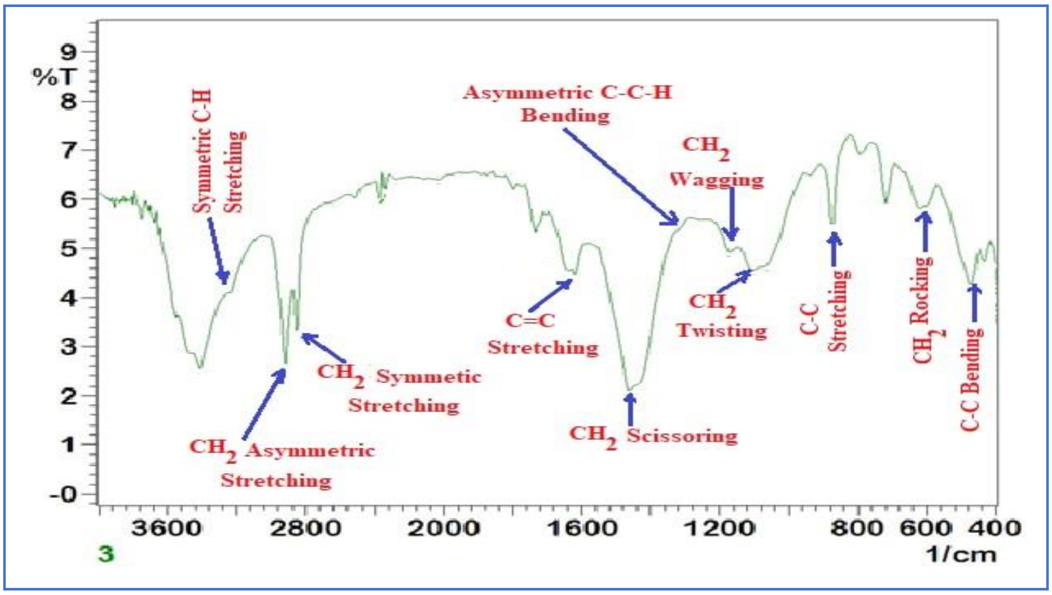
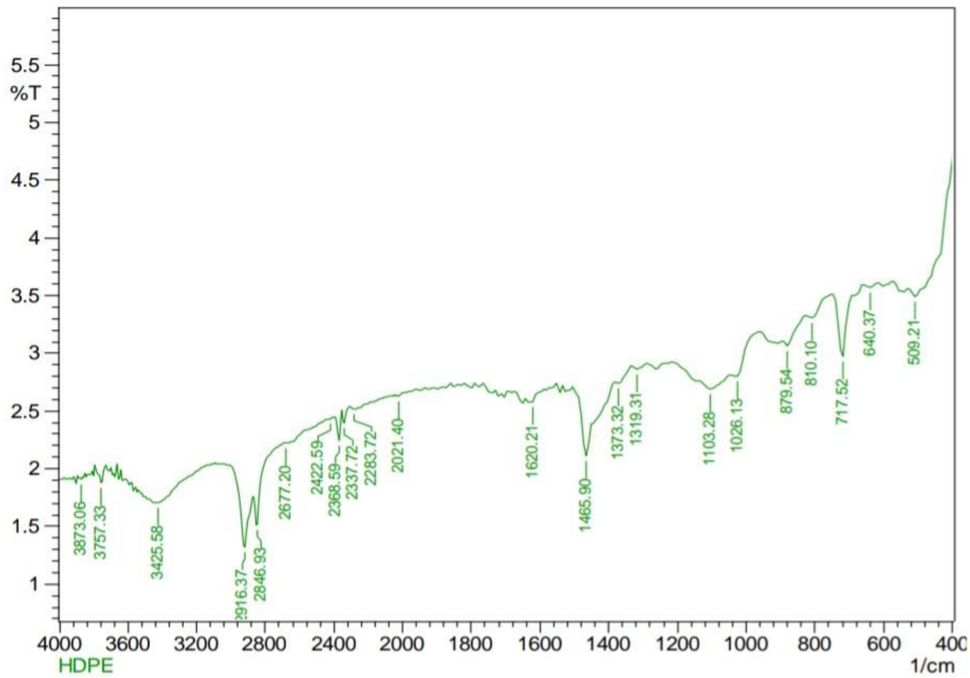


Figure 4.5 : The result of the FTIR test for samples of high density polyethylene produce by extruder

Figure 4.6 : The result of the FTIR test for samples of high density



polyethylene produce by hydraulic press

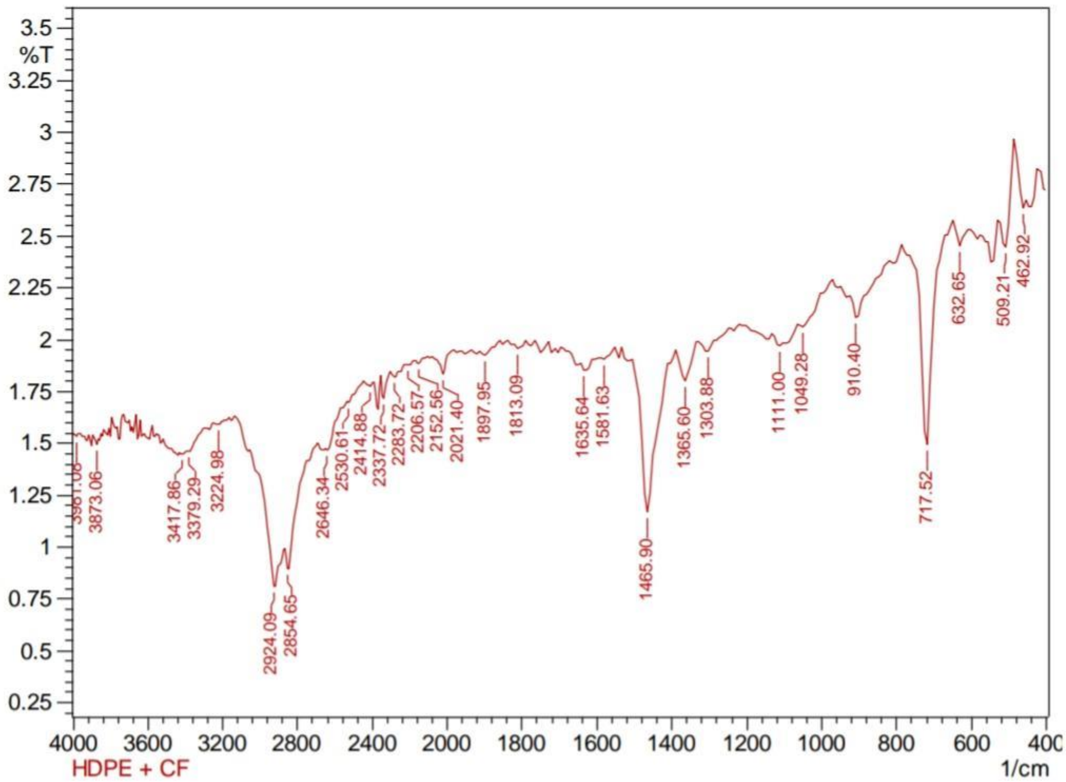


Figure 4.3 : The result of the FTIR examination of samples of high-density polyethylene reinforced with carbon fibers

for HDPE Reinforced Carbon fibers the bands at 2916.09 cm-1 shifted to 2924.09 cm-1 while the others band don't change, while HDPE Reinforced by Carbon fibers the bands at 2916 cm-1and 1465.90 cm-1 shifted to at 2924.09 cm-1and 1442 cm-1 respectively a for the other bonds, they remain unchanged as in figures (4.3) .

These results indicate that there is no change or chemical bonding in the structure of the polymer because there is no new bond that appears or disappears. also note that the permeability decreased after adding Carbon fibers, respectively,because these fibers increased the absorption of the polymer due to its optical properties, and also in some areas that the permeability decreased compared to the pure polymer which means that the polymer chain extends after adding carbon fibers where are these fibers the holes between the polymer structure.

Table 4.1: The Absorption Bands of IR Spectrum Characteristic of HDPE and HDPE Composite:

Type of bond	HDPE standard[100]	HDPE extruder	HDPE + CF
CH_2 stretching	2918 2849	2916.37 2846.93	2924.09 2854.65
CH_2 scissoring	1463	1465.90	1465.90
CH_2 rocking	719	717.52	717.52
C-H Symmetric Stretching	3425	3425.58	3417.86
C=C Stretching	1620	1620.21	1635.64
Asymmetric C-C-H Bending	1372	1373.32	1365.60
CH_2 Twisting	1026	1026.13	1049.28
C-C Stretching	879	879.54	910.40
C-C Bending	507	509.21	509.21

Chapter five

Conclusions and Recommendations

5.1 Conclusions

1- The addition carbon fiber to the HDPE matrix type woven provided the best performance of creep resistance, superior to the best performance provided by the HDPE matrix when introducing carbon fibers type roving.

2- The addition carbon fiber to the HDPE matrix type roving provided the best performance of creep resistance, superior to the best performance provided by the HDPE matrix when introducing carbon fibers type unidirectional

3- The highest tensile strength obtained when inserting carbon fibers for (HDPE/Cf) composites type woven then other types such as roving and unidirectional

4- The manufacturing method plays an important role in increasing the elastic modulus, as the samples produced by the hydraulic piston device are more solid than the samples produced by the two-screw extrusion method, due to the closed mold of the hydraulic piston device.

5- The highest impact resistance obtained was (23.825KJ / M2) when inserting carbon fibers (HDPE/Cf composites, while the highest for (HDPE) was (16.835KJ / M2)

5.2 Recommendations

1-propose to study the rheological and thermal properties of (HDPE) and (HDPE) reinforced by fibers.

2-Suggest the use of polypropylene (PP) or low-density polyethylene (LDPE) and the study of its mechanical and viscoelastic properties.

3-Suggest using fibers materials such as silicon dioxide (SiO2) or titanium dioxide (TiO_2) as reinforcing materials for (HDPE) and its studying their mechanical behaviour.

4-With regard to the extrusion process of high density polyethylene, a high temperature should be used, because this material suffers from shrinkage when extruded at low temperatures.

5.3 Reference

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