

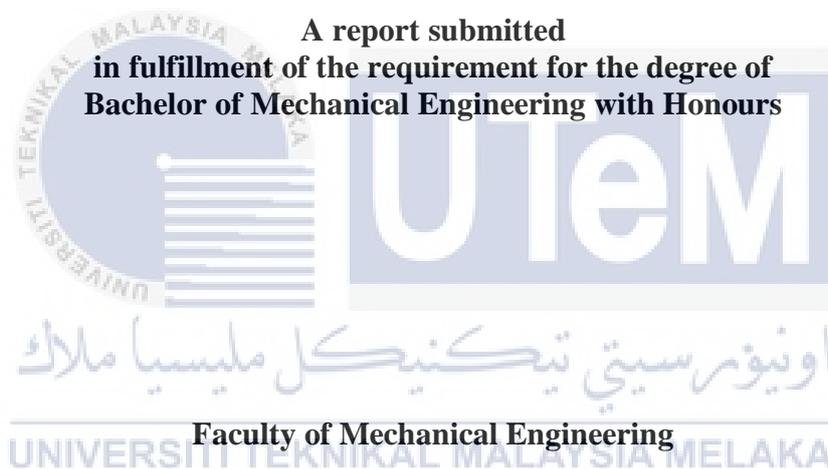
**TENSILE PROPERTIES OF GRAPHENE NANOPLETET REINFORCED
ULTRA-HIGH MOLECULAR WEIGHT POLYETHYLENE COMPOSITES**



UNIVERSITI TEKNIKAL MALAYSIA MELAKA

**TENSILE PROPERTIES OF GRAPHENE NANOPLETELET REINFORCED
ULTRA-HIGH MOLECULAR WEIGHT POLYETHYLENE COMPOSITES**

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UNIVERSITI TEKNIKAL MALAYSIA MELAKA

AUGUST 2020

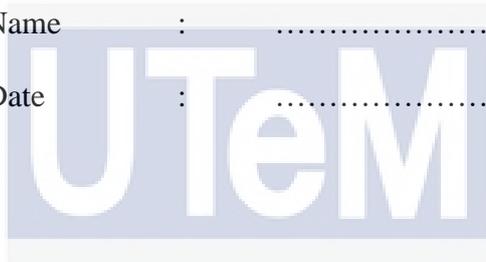
DECLARATION

I declare that this project report entitled “Tensile Properties of Graphene Nanoplatelet Reinforced Ultra-High Molecular Weight Polyethylene Composites” is the result of my own work except as cited in the references.

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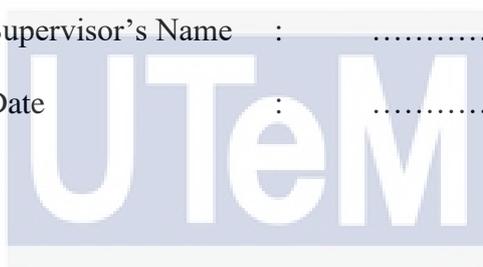
APPROVAL

I hereby declare that I have read this project report and in my opinion this report is sufficient in terms of scope and quality for the award of the degree of Bachelor of Mechanical Engineering with Honours.

Signature :

Supervisor's Name :

Date :



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DEDICATION

To my beloved mother and father.



ABSTRACT

Ultra-high Molecular Weight Polyethylene (UHMWPE) has been used extensively in orthopaedic implant fabrications as a component in total joint replacement especially for hip and knee replacement. A good reinforcing filler, Graphene Nanoplatelet (GNP) with different amounts (0.1, 0.3, 0.5, and 1.0 wt. %) were used to fabricate the GNP/UHMWPE composite. The composites were fabricated by the dry mixing process as a technique to mix these two materials followed by the hot-pressing with reference to ASTM D638 Type 1 standard. The process continued with several tests in order to fulfil the objective of the study. The objectives of this study are to fabricate the sample of Neat UHMWPE and GNP/UHMWPE composite, to investigate the effects of GNP on tensile properties of GNP/UHMWPE composite and to evaluate the effects of GNP composition in GNP/UHMWPE composite. From density analysis, the result shows that the density between theoretical and experimental have slightly different. The microhardness test shows that the GNP can increase the hardness of the composite with increasing amounts of GNP. The conductivity test shows that the small amount of GNP added do not conduct electric while at higher wt. % can conduct electric. The key findings from tensile test included the GNP with 0.1 wt. % can increase the tensile strength and tensile strain of the composite and decreased from 0.3 to 1.0 wt. %. The addition of GNP up to 0.3 wt. % also enhanced the Young's modulus of the composite. The GNP/UHMWPE displayed a remarkable combination of enhanced the tensile properties and others which making the composites safe to be used as material in arthroplasty implant in human body.

ABSTRAK

Ultra-high Molecular Weight Polyethylene (UHMWPE) telah digunakan secara meluas dalam fabrikasi implan ortopedik sebagai komponen dalam penggantian sendi terutama untuk penggantian pinggul dan lutut. Bahan penguat yang bagus seperti Graphene Nanoplatelet (GNP) dengan jumlah yang berbeza (0.1, 0.3, 0.5, dan 1.0 wt. %) telah digunakan untuk membuat komposit GNP/UHMWPE. Komposit dibuat melalui proses pencampuran kering sebagai teknik untuk mencampurkan kedua-dua bahan ini diikuti dengan proses penekanan panas dengan merujuk kepada piawai ASTM D638 Jenis 1. Proses ini diteruskan dengan beberapa ujian untuk memenuhi objektif kajian. Objektif kajian ini adalah untuk menghasilkan spesimen Neat UHMWPE dan komposit GNP/UHMWPE, mengkaji kesan GNP pada sifat tegangan komposit GNP/UHMWPE dan mengkaji kesan jumlah GNP dalam komposit GNP/UHMWPE. Dari analisis ketumpatan, hasilnya menunjukkan bahawa ketumpatan antara teori dan eksperimen sedikit berbeza. Ujian mikrokekerasan menunjukkan bahawa GNP dapat meningkatkan kekerasan komposit dengan peningkatan jumlah GNP. Ujian kekonduksian menunjukkan bahawa sejumlah kecil GNP yang ditambahkan tidak mengalirkan elektrik manakala pada jumlah wt. % yang tinggi boleh mengalirkan elektrik. Penemuan utama dari ujian ketegangan termasuk GNP dengan 0.1 wt. % dapat meningkatkan tegangan dan regangan komposit dan menurun dari 0.3 hingga 1.0 wt. %. Penambahan GNP hingga 0.3 wt. % juga meningkatkan modulus komposit Young. GNP/UHMWPE memperlihatkan kombinasi yang luar biasa untuk meningkatkan sifat tegangan dan lain-lain yang menjadikan komposit ini selamat digunakan sebagai bahan dalam implan artroplasti dalam tubuh manusia.

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LIST OF ABBREVIATIONS

| | | |
|--------|---|-----------------------------------------------------------------|
| ASTM | - | American Society for Testing and Materials |
| CMC | - | Ceramic Matrix Composite |
| CNT | - | Carbon Nanotubes |
| CVD | - | Chemical Vapor Deposition |
| GNP | - | Graphene Nanoplatelet |
| GO | - | Graphene Oxide |
| HDPE | - | High Density Polyethylene |
| LDPE | - | Low Density Polyethylene |
| MMC | - | Metal Matrix Composite |
| MTT | - | 3-(4, 5-di-methylthiazol-2-yl)-2, 5-diphenyltetrazolium bromide |
| MWCNT | - | Multi-Walled Carbon Nanotubes |
| PMC | - | Polymer Matrix Composite |
| PMMA | - | Poly-methyl-methacrylate |
| PSM | - | Projek Sarjana Muda |
| PTFE | - | Polytetrafluorethylene |
| SEM | - | Scanning Electron Microscope |
| SWCNT | - | Single-Walled Carbon Nanotubes |
| TEM | - | Transmission Electron Microscope |
| UHMWPE | - | Ultra-High Molecular Weight Polyethylene |

LIST OF SYMBOLS AND UNITS

| | | |
|--------------------|---|-----------------------|
| GPa | - | Giga Pascal |
| g | - | Gram |
| h | - | Hours |
| HV | - | Hardness Vickers |
| Hz | - | Hertz |
| kJ | - | Kilo Joule |
| M | - | Mass |
| m | - | Meter |
| min | - | Minutes |
| ml | - | Mililitre |
| mm | - | Milimeter |
| m/s | - | Meter per second |
| MPa | - | Mega Pascal |
| N | - | Newton |
| nA | - | Nanoampere |
| nm | - | Nanometer |
| psi | - | Pound square inch |
| rpm | - | Revolution per minute |
| S | - | Siemens |
| sqr | - | Square |
| TPa | - | Tera Pascal |
| wt. % | - | Weight percentage |
| ρ | - | Rho |
| Ω | - | Ohm |
| μm | - | Micrometer |
| $^{\circ}\text{C}$ | - | Degree Celsius |

CHAPTER 1

INTRODUCTION

1.1 Background

Composite is a combination of two or more materials (reinforcing elements, filler and composite matrix binder), differing in form or composition on a macro scale. Composite components are commonly used in many applications such as in sporting equipment, aerospace, medical field as well as military. The uses of composite over monolithic materials are due to its characteristic which are light in weight, high stiffness and strength, low thermal expansion and high fatigue resistance. Composite can be classified into two classes; classification of composite based on matrix material and classification of composite based on reinforcement geometry. Examples of classification of composite based on reinforcement geometry are Particle Reinforced, Fibre Reinforced and Structural. While classification of composite based on matrix material are Polymer Matrix Composite (PMC), Metal Matrix Composite (MMC) and Ceramic Matrix Composite (CMC) as shown in **Figure 1.1**.

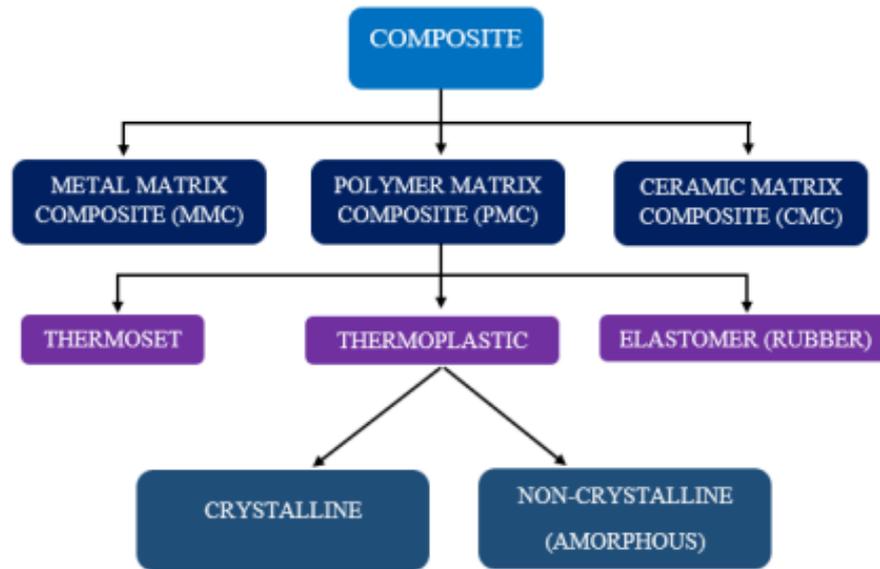


Figure 1.1: Classification of composite based on matrix material.

1.1.1 Polymer Matrix Composite

Polymers can be identified as long chain materials (macromolecules) consisting of many repeat units. Polymers can be either naturally produced or synthetically produced. Physical properties of the polymer are including molecular weight and molecular structure. Average molecular weights for most polymers are in the range of 10,000 up to more than 1,000,000 g/mol. There are four types of polymer molecular structures such as (a) linear, (b) branched, (c) crosslinked, and (d) network polymers as shown in **Figure 1.2** [1].

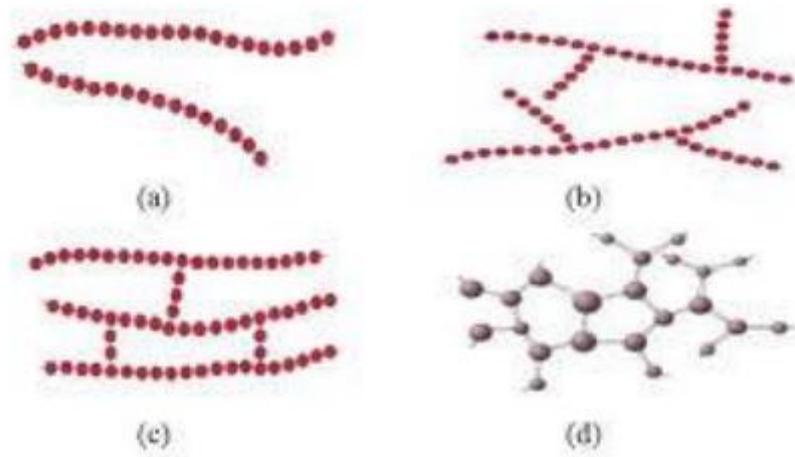


Figure 1.2: Type of polymer molecular structure [1].

The repeat units in linear polymers are covalently linearly bound in single chains and the molecule chains are flexible, as shown in **Figure 1.2(a)**. Van der Waals or hydrogen bonding are the bonding between the chains. Example of linear polymer is poly-methyl-methacrylate (PMMA) [1].

There is side branch chain connected to the main chains in branched polymer, as shown in **Figure 1.2(b)**. Polymer density is low due to formation of side branches that can reduce chain packing efficiency. A polymer that forms linear structure may also be branched. As example polyethylene. High density polyethylene is a linear polymer while low density polyethylene is a branched polymer [1].

The main linear chains are covalently bonded to each other in cross-linked polymers, as shown in **Figure 1.2(c)**. During synthesis or through a chemical reaction, the crosslinking process is achieved. Examples of cross-linked polymers are rubber-elastic materials [1].

Monomers form three-dimensional networks in network polymers, as shown in **Figure 1.2(d)**. It may also be possible to identify a strongly cross-linked polymer as a

network polymer. Mechanical and thermal properties of network polymers are relatively better. Polyurethane is an example of network polymers [1].

Polymers are commonly used in many applications because it is easy to process (do not require high temperature and pressure). Some applications of polymer that used in everyday life are electrical wire insulation, safety helmets, anti-adhesive coating and many more. However, in general polymer deteriorate due to physical, thermal and chemical factors. It has low stiffness and strength. Other disadvantages of this material are low working temperature, high coefficient of thermal expansion and it is sensitive to moisture and radiation. Polymer can be classified into thermoplastics, thermosets, and elastomers.

Thermoplastics

Thermoplastics are linear or branched polymers that can be either amorphous, crystalline or mixed and act in a ductile manner. When heat is applied, thermoplastics become soft and melt and solidify upon cooling [1]. Thermoplastics are reversible reaction which means recyclable. Some applications of thermoplastic in daily life are sterilisable bottles and film wrapping materials. Commonly known thermoplastics are polypropylene and polyethylene. Other examples of thermoplastic are shown in **Table 1.1**.

Thermosets

Thermosets are linear or branched molecules of polymers which can be cross-linked to form three-dimensional network structures. Generally, thermosets are stronger but more brittle in comparison with thermoplastics [1]. Thermosets cannot be recycled. Well known thermosets are polyester, epoxy resin and many more as shown in **Table 1.1**. Epoxy resins

used for application with superior performance and it is relatively costly compared to polyester.

Elastomers

Elastomers, also known as rubbers, are materials with more than 200 % elastic deformation. Elastomers may be thermoplastics, or thermosets that are lightly cross-linking. In elastomers, the polymer chains consist of coil-like molecules which can stretch reversibly when a force is applied [1]. Example of elastomers is natural rubbers. Other examples of elastomers are shown in **Table 1.1**.

Table 1.1: Example of Thermoplastics [1], Thermosets [1] and Elastomers [2].

| Thermoplastics | Thermosets | Elastomers |
|-------------------|--------------|---------------------------|
| Polyamide (nylon) | Phenolic | Polyisoprene |
| Polycarbonate | Polyurethane | Ethylene Propylene Rubber |
| Polystyrene | Vinyl Ester | Nitrile Rubbers |

1.1.2 Biomaterial

Biomaterial can be defined as any material used to make devices to replace a part or a function of the body in a safe, reliable, economic and physiologically acceptable manner. It is a synthetic material (man-made). The Clemson University Advisory Board for Biomaterials has formally defined a biomaterial to be *a systematically and pharmacologically inert substance designed for implantation within or incorporation with living systems.*

In recent times, the arena of nanoscience, nanotechnology and nanocomposites has flourished, and the importance of this topic has increased in other applications such as automotive, aerospace, packaging, biotechnology, biomedical, electronics, flexible sensors and many more. The innovations on polymer composite based on reinforcing materials has been an interested and become a significant addition especially in biomedical field. For example, some devices and implants are used to replace or improve the function of the original parts or organs in body, e.g. contact lenses, cardiac pacemaker, dental implants, and orthopaedic implants.

There four groups of synthetic material used for implantations in human body. As example, composites, metals, ceramics, and polymers. The important aspects of study on biomaterial are biological material, implant material, and interaction between the material added and the organs or parts in body. This interaction needs to be considered in order to know the biocompatibility of material in body.

Metallic implant materials such as stainless steel, titanium and titanium alloy can be used in dental roots implant and for bone plates and screw. The uses of metal because it is strong and ductile but there are some problems regarding this material which are metal tend to corrode in our body, heavy and difficult to fabricate. For ceramic implant materials such as alumina zirconia and hydroxyapatite also include in contributing to be as a material for orthopaedic and dental implants but need to be highlighted that these materials are brittle and weak in tension.

The success of a biomaterial or an implant is highly dependent on factors such as the properties and biocompatibility of the implant, and the health condition of the recipient. Polymeric implant materials such as ultra-high molecular weight polyethylene has been used

extensively for orthopaedic implant fabrications especially for such load-bearing surfaces as total hip and knee joints as shown in **Figure 1.3(a)** and **Figure 1.3(b)**, respectively.

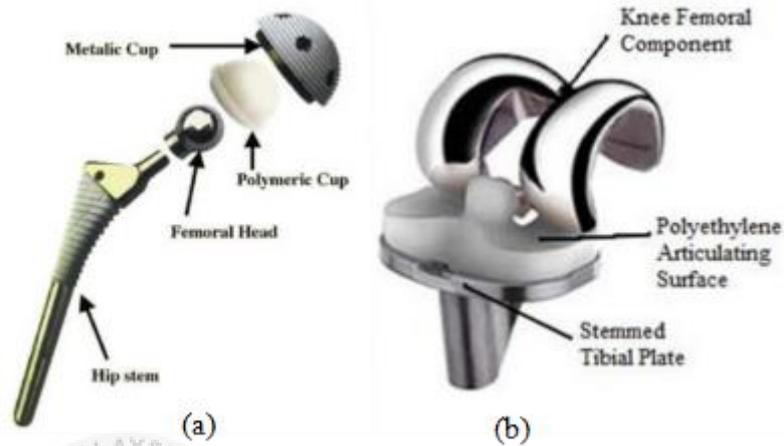


Figure 1.3: Application of polymer in (a) total hip and (b) knee replacement.

1.2 Ultra-High Molecular Weight Polyethylene

Polyethylene is under thermoplastic class. Polyethylene is available commercially in three major grades: Low Density Polyethylene (LDPE), High Density Polyethylene (HDPE) and Ultra-High Molecular Weight Polyethylene (UHMWPE). LDPE is a branched polymer and it is produced by high pressure Ziegler polymerization process. LDPE has an amorphous structure and low crystallinity. In the polyethylene group, LDPE is the largest volume production material. Nearly 50% of LDPE are transformed into thin films for the packaging industry. The rest is used for other applications. HDPE is a linear polymer produced by low pressure Ziegler process and it has high crystallinity. Blow-moulded containers for liquid product packaging are a major market area for HDPE [3].

UHMWPE is a subset of polyethylene thermoplastic. UHMWPE has high molecular weight usually around 2-6 million g/mole. The material is almost completely inert. The uses

of UHMWPE because it is low wear rate and high impact strength. Recently, cross-linked UHMWPE has been developed for the use of articulating joint materials such as the acetabular cup of a hip joint prosthesis. Other differences between these three types of polyethylene are shown in **Table 1.2** [3].

Table 1.2: Properties of LDPE, HDPE and UHMWPE [3].

| Properties | LDPE | HDPE | UHMWPE |
|------------------------|---------------|---------------|---------------|
| Specific gravity | 0.910 – 0.925 | 0.941 – 0.965 | 0.928 – 0.941 |
| Tensile strength, MPa | 4.1 – 15.8 | 21.9 – 38 | 38 – 48 |
| Elongation at break, % | 90 – 800 | 20 – 1000 | 200 – 500 |

1.3 Carbon-based Fillers

There are several types of carbon-based fillers such as graphite, graphene, carbon nanotube (CNT) and many more as shown in **Figure 1.4**. In order to synthesize the graphene and its derivatives, it has their own methods.

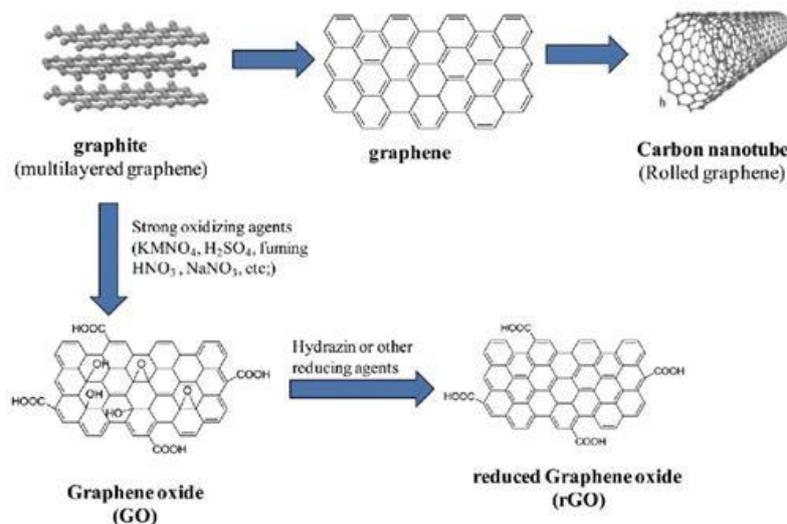


Figure 1.4: Type of carbon-based fillers.

Graphite

The lowest energy level in graphite is elemental carbon at atmospheric pressure and temperature. The graphite crystal lattice consists of two-dimensional parallel graphene sheet stacks of sp^2 hybridized carbon atoms tightly bound to hexagonal rings. The sp^2 hybridized carbon is shown in **Figure 1.5**.

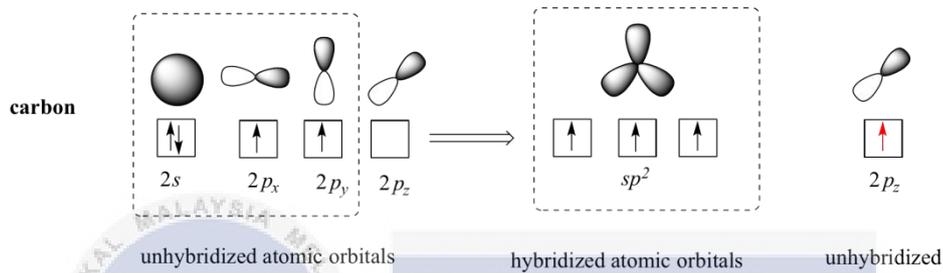


Figure 1.5: Carbon atom in orbital.

Since the $2p_z$ orbital carbon atoms easily can overlap if they are parallel, when it is completely flat, the graphene sheet has the lowest energy. Therefore, graphite is anisotropic due to the difference between the bonding of carbon atoms in-plane and out-of-plane. The elastic modulus is higher when parallel to the plane than that of perpendicular to plane. Therefore, graphite is stronger in the plane compared to diamond. The properties of graphite such as density, elastic modulus and others are shown in **Table 1.3** [4].

Table 1.3: Properties of graphite [4].

| Property | Unit | Graphite ^a | CNT ^a | VGCNF ^b |
|----------------------|-----------------------------------|-----------------------------------------------------------------|-----------------------------------------|-----------------------|
| Density | g cm ⁻³ | 2.26 | 0.8 for SWNT 1.8 for MWNT (theoretical) | 2.0 |
| Elastic modulus | TPa | 1 (in-plane) | ~1 for SWNT ~0.3-1 for MWNT | 0.5 |
| Strength | GPa | 130 | 50-500 for SWNT 10-60 for MWNT | ~3.0 |
| Resistivity | μΩ cm | 50 (in-plane) | ~5-50 | ~100 |
| Thermal conductivity | W m ⁻¹ K ⁻¹ | 3000 (in-plane) 6 (z-axis) | 3000 (theoretical) | 1950 |
| Thermal expansion | K ⁻¹ | ~1 × 10 ⁻⁶ (in-plane) 29 × 10 ⁻⁶ (z-axis) | Negligible (theoretical) | -1 × 10 ⁻⁶ |
| Thermal stability | °C | 450-650 (in air) | >700 (in air) 2800 (in vacuum) | ~600 (in air) |

Carbon Nanotube

There are two types of CNT: Single-Walled Carbon Nanotubes (SWCNT) and Multi-Walled Carbon Nanotubes (MWCNT). CNT possess high elastic modulus – 1TPa and strength – 200GPa [5]. CNT are unique because it has high stiffness and strength. The Young's Moduli of CNT is in the range of 1 – 2TPa and their fracture stresses is about 50GPa. CNT can be in the form of graphene sheets rolled up in some directions. There are some applications of SWCNT such as active channels in transistor devices and electrical interconnectors. The uses of SWCNT as active channels in transistor devices because it can be either semiconducting, metallic or semi-metallic. SWCNT have high mobility ($10000 \text{ cm}^2\text{Vs}^{-1}$). In electrical interconnectors, it is because of their low resistivity, high current-carrying capacities (10^9 Acm^{-2}) and high thermal conductivities ($3500 \text{ Wm}^{-1}\text{K}^{-1}$). CNT film produced from Chemical Vapour Deposition [6]. **Figure 1.6** and **Figure 1.7** show the schematic figure of SWCNT and MWCNT, respectively.

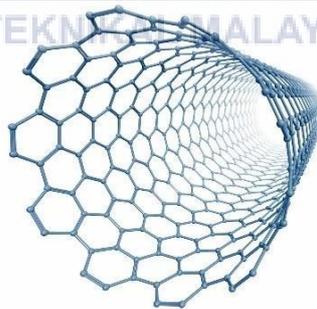


Figure 1.6: Single-Walled Carbon Nanotubes.

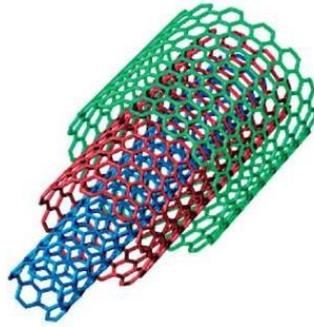


Figure 1.7: Multi-Walled Carbon Nanotubes.

Graphene

Graphene is a two-dimensional carbon atom structure constructed by crystalline hexagonal structure with sp^2 bonds as shown in **Figure 1.8**. It has a high surface area of $2630 \text{ m}^2/\text{g}$ (highest surface reactions and adsorption), high thermal conductivity ($5000 \text{ Wm}^{-1}\text{K}^{-1}$), mobility of electrons, and mechanical strength. For example, as compared to the other conventional fillers in composite material, graphene enhances the interaction between the sheets and polymer due to its high surface area. Moreover, graphene is the thinnest material in nature where it has a high Young's Modulus and an intrinsic strength of 1 TPa and 130 GPa , respectively [7].

Graphene is an exciting material. It has high intrinsic mobility ($20 \text{ m}^2\text{v}^{-1}\text{s}^{-1}$), high its optical transmittance (97.9%) and good electrical conductivity for applications such as transparent conductive electrodes [8].

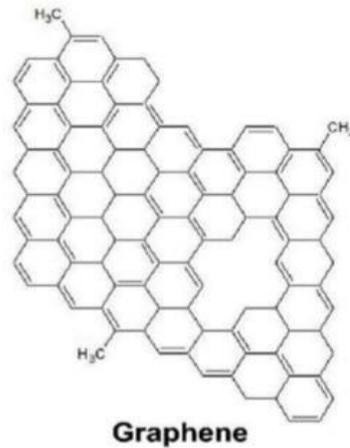


Figure 1.8: Graphene structure [7].

With the unique properties it has, graphene could potentially be used in biotechnology and bio-based applications. It finds promising applications in tissue engineering, drug delivery and DNA sequencing due to its large surface area and chemical stability as well as limpidity and functional viability. Nano-sized graphene or also known as Graphene Nanoplatelet (GNP) is used as filler in biomaterial implant. The high mechanical properties help regenerative medicine applications. Moreover, graphene can be used in imaging of biomolecules through transmission electron microscopy [7].

Graphene Oxide

Graphene oxide (GO) is a single-layer substance composed of molecules of carbon, hydrogen and oxygen by graphite crystals oxidation. The GO structure is shown in **Figure 1.9**. It is water-dispersible and quick to handle. Most notably, by eliminating the oxygen-containing groups and restoring a conjugated structure, the GO can be (partially) reduced to graphene-like plates. Due to the presence of oxygen functionalities, one of the advantages of GO is it is easy to disperse in water and other organic solvents as well as in different

matrixes. It remains a very important property when the material is combined with ceramic or polymer matrix to enhance their electrical and mechanical properties [9].

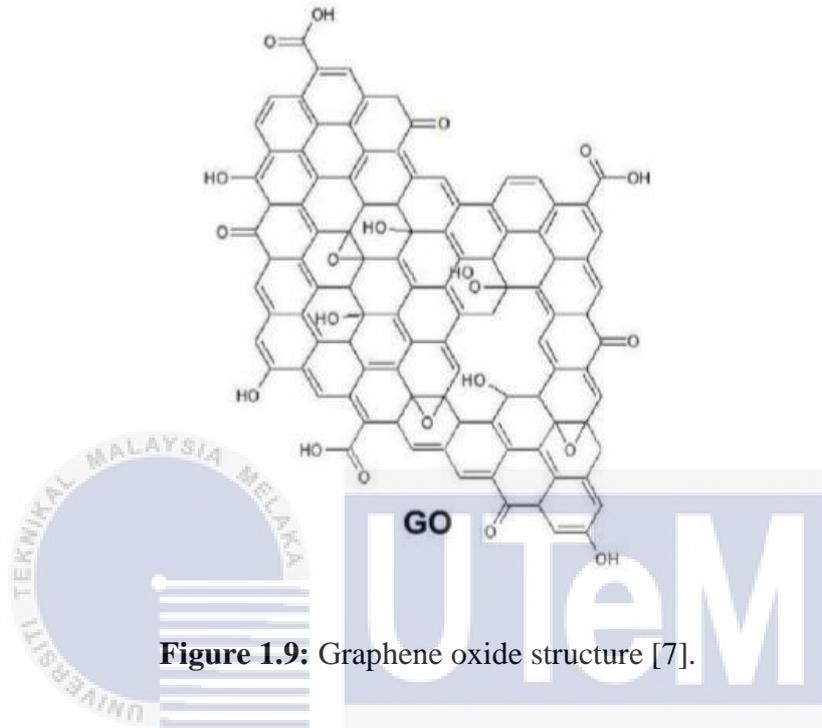


Figure 1.9: Graphene oxide structure [7].

GO is synthesized by either the Brodie, Staudenmaier or Hummer method. These three methods involve graphite oxidation at different levels. A combination between potassium chlorate and nitric acid were used by Brodie and Staudenmaier in order to oxidize graphite while Hummers method involves treatment of graphite with potassium permanganate and sulfuric acid [8].

The uses of graphene and its derivatives because of the advantage of graphene which is good in terms of mechanical properties. The application of graphene in a polymer matrix as a reinforcing agent has improved the overall performance and properties of these composites [7].

This graphene polymer composite has general aim to improve mechanical properties of UHMWPE. UHMWPE is widely used as a bearing pad in total knee replacement and acetabular cup component in total hip replacement. In this field, total joint arthroplasty, current UHMWPE are limited in their thickness due to concerns about elevated stresses and potential for fracture. Therefore, there is increased in UHMWPE composites to improve the strength of the material, without sacrificing its other excellent attributes such as biocompatibility, lubricity and wear resistance.

1.4 Problem Statement

Joint replacements have made it possible for thousands of people with joints damaged by disease or trauma to enjoy a more active lifestyle over the past century and continue their life. UHMWPE has been the choice of orthopaedic bearing material in total joint replacement for the past twenty years [10]. This is due to its characteristics which are high impact strength, good biocompatibility and low friction coefficient but the use of UHMWPE has been limited due to its low Young's modulus, low load bearing and anti-fatigue capacity [11]. Moreover, another study by Suner and Emami [12] stated that high amount of wear debris of the UHMWPE that contributes directly to the development of aseptic loosening is due to degradative oxidation behaviour of the UHMWPE.

Some excellent characteristics of UHMWPE from another study by Sui are high impact resistance, high wear resistance, good abrasion resistance, good chemical resistance and biocompatibility [13]. Therefore, the UHMWPE can be used as a biomaterial especially for component in arthroplasty application. The wear mechanism for hip and knee are different and wear debris produced from these two total joints also have differences. The acetabular component in total hip replacement produces wear debris that less than a micron

in size while in tibial component is larger thin flake-like [10]. In addition, the uses of UHMWPE are limited because of low yield strength which is approximately between 20-25 MPa [10]. Tensile strength of UHMWPE is 38–48 MPa [3]. In normal walking, for a total hip replacement the maximum principal stress of UHMWPE is less than 10 MPa while in total knee replacement it can reach up to 45 MPa.

Therefore, the improvement on the mechanical behaviour of UHMWPE is very important to be focused. Modern carbon-based composites have shown a potential advancement in UHMWPE biomaterials. In order to improve this, a good reinforcing material such as GNP need to be used in this study due to its superior mechanical properties. Different amounts of GNP will be used to fabricate different types of GNP/UHMWPE composite and investigate the tensile properties of GNP/UHMWPE composite.

1.5 Objective

The objectives of this study are as follow:

- To fabricate the sample of Neat UHMWPE and GNP/UHMWPE composite.
- To investigate the effects of GNP on tensile properties of GNP/UHMWPE composite.
- To evaluate the effects of GNP composition in GNP/UHMWPE composite.

1.6 Scope of Project

This study is focusing on evaluating the tensile properties of GNP/UHMWPE composites. The materials used are UHMWPE and GNP. The GNP/UHMWPE composite will be fabricated by the process of mixing and hot-pressing. The dry mixed technique will

be used to mix these two materials. The effect of GNP on tensile properties of the composites will be investigated and compared to the Neat UHMWPE. The amounts of GNP incorporated into UHMWPE is also important to be determined in order to identify the optimum amounts of GNP. The flowchart of the overall process for this project is shown in **Figure 1.10**.

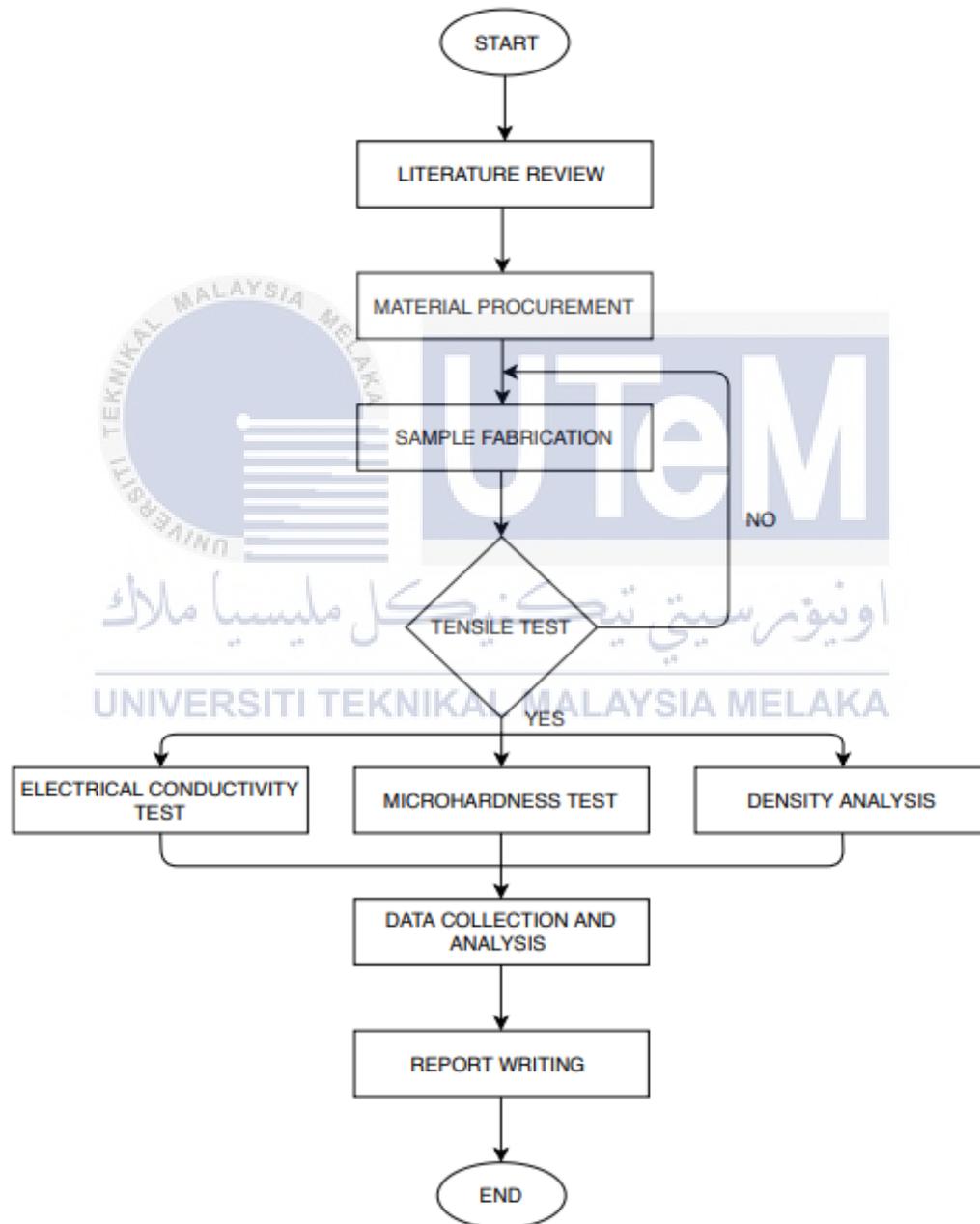


Figure 1.10: Flowchart of the Projek Sarjana Muda.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

This chapter will explain in detail about bio-composite for Arthroplasty Implant, biocompatibility of carbon-based filler composite and UHMWPE composite. Graphene is one of the carbon-based fillers that widely used as reinforcing element incorporate with polymer and other matrix in application such as joint replacement. The biocompatibility and mechanical properties of this filler and its composite will explain in detail in section below.

2.2 Biomaterial Composites for Arthroplasty Implant

Biomaterial developments for biomedical purposes have improved over the past few years, developing into new improved ceramic and polymeric materials to produce composite materials. Total hip replacement is now one of the world's most common operations. As the age of population is increase, the number of people will undergo this operation is expected to increase. Thus, the evolution of the composite based on ceramic and polymer materials need to be focused as these materials commonly used in arthroplasty implants. A biomaterial is a material that interacts with human tissue and blood fluids to treat, improve, or replace anatomical elements for the human body. Biomaterials that are used in medical devices for orthopaedic application are commonly alumina, UHMWPE, zirconia and titanium alloy [14].

The uses of polymer as implant material for acetabular component by Charnley in early 1960s is to replace the Cobalt-Chromium (Co-Cr) alloy implants. This is because to reduce friction causes by this material. The purpose also to eliminate metallic wear on particles of this implant material. He was the first to implant a femoral stainless-steel part with a Polytetrafluoroethylene (PTFE) as acetabular component. PTFE has a high thermal stability; it is hydrophobic, soluble in most chemicals and is generally considered inert in the body. However, clinical studies involving PTFE acetabular cups showed unacceptably high wear and distortion of total hip replacement prostheses. Thus, PTFE is no longer used for this application [14].

In November 1962, Charnley introduced an acetabular cup made of UHMWPE. UHMWPE consists of extremely long polyethylene chains and each individual molecule through its length adds strength to the entire structure. However, the UHMWPE socket debris can cause adverse biological reactions to the tissue resulting in bone loss or osteolysis. The scientific community always interested to improve this material. In the late 1990s, the radiation crosslinking technology combined with thermal treatment was developed in order to enhance the wear and oxidation resistance of UHMWPE implants [14].

2.3 Carbon-based Filler Composite: Biocompatibility

Biocompatibility is a key factor in the application of joint prosthesis due to degradation of material. The calculation of biocompatibility depends on the synthesis techniques and the choice of the study biological model system. Liu et al [15] study on the preparation, mechanical performance and biocompatibility effects of the graphene oxide/ultrahigh molecular weight polyethylene composites. [3-(4, 5-di-methylthiazol-2-yl)-2, 5-diphenyltetrazolium bromide] MTT assay is a cytotoxicity research tool by measuring

the number of living cells and the efficiency of the metabolism of living cells. MC3T3-E1 cells was used in this study. The cytotoxic effect of GO/UHMWPE and UHMWPE on cells was examined to determine the biocompatibility of as-prepared GO/UHMWPE. The GO/UHMWPE was significantly biocompatible based on the result shown in **Figure 2.1**. From that there is no obvious change in the adsorption on the GO/UHMWPE composites relative to that of the neat UHMWPE which can be indicated that there is no negative effect on the growth of cell when GO sheets was added to UHMWPE. This is due to functionalization of GO. Hence, the GO/UHMWPE is more compatible compared to neat UHMWPE.

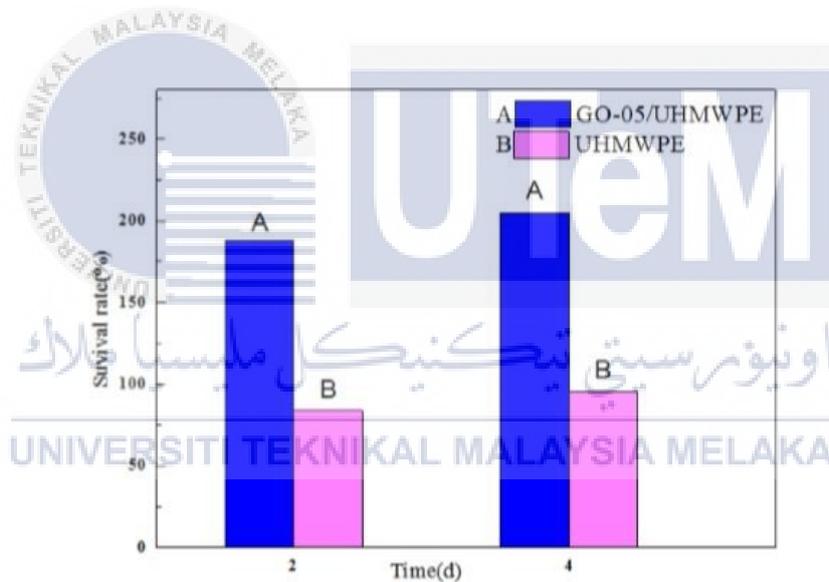


Figure 2.1: Viability of the MC3T3-E1 cells on the UHMWPE and GO/UHMWPE composites at different incubation times [15].

A study of graphene/chitosan composite by Fan et al [16] was done to explore the mechanical properties and biocompatibility this composite. Graphene/chitosan composite has potential to be used as scaffolds materials in tissue engineering. When graphene was added between 0.1-0.3 wt. %, the modulus of chitosan was increased. The graphene/chitosan

film was produced by solution casting method. In this study, L929 cell was used. In order to check the cytotoxicity of graphene/chitosan materials, MTT assays are performed. The cell viability was measured using [3-(4, 5-di-methylthiazol-2-yl)-2, 5- diphenyltetrazolium bromide] (MTT) assays. It is good biocompatibility. There are several steps need to be followed in order to check the biocompatibility of the composite. While graphene sheets are non-biodegradable materials, the low content of graphene in composites of graphene/chitosan can limit the possible negative effect of graphene on cells after decomposition of chitosan in the body.

The viability of the L929 cell that has been applied to graphene/chitosan composites with different graphene concentrations was shown in **Figure 2.2**. The tests of MTT did not reveal any significant reduction in viability between the 24 and 48 h negative control and experimental group. The composite showed strong biological protection and were almost non-cytotoxicity. As confirmed by in vitro MTT assays, the graphene/chitosan composite showed good biocompatibility for L929 cells.

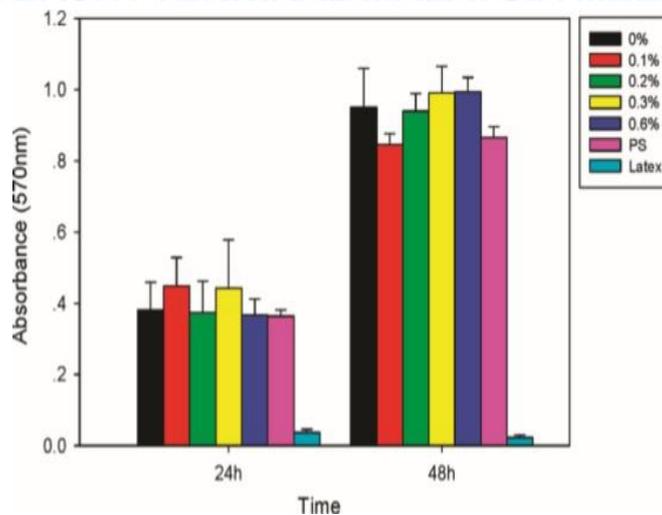


Figure 2.2: Result of MTT assay for L929 cells incubated with graphene/chitosan composite films at 24 and 48 hours [16].

As conclusion based on these two studies, it can be clearly said that the addition of graphene as filler into matrix that can be used in biomedical application, i.e. the incorporation of graphene would give no effect to the cell growth in human body.

2.4 Fabrication Method for UHMWPE Composite

Suner et al [12] study on thermal, mechanical, and wettability characterisation of ultra-high molecular weight polyethylene/graphene oxide nanocomposites. In this study, it is stated that the main aims of the study were to investigate the effect of adding GO nanoparticles to UHMWPE and to determine the optimal wt. % applied to improve the performance of nanocomposite. GO/UHMWPE nanocomposite has been prepared with specific GO wt. % material and mechanical, thermal, structural and wettability properties have been investigated and compared with neat UHMWPE. The amounts of GO used in this study were 0.1, 0.3, 0.5, 0.7, 1.0 and 2.0 wt. %. The ball milling technique was used to prepare GO/UHMWPE nanocomposite powders with specific GO wt. % content under optimized conditions. Some steps were involved in order to prepare the GO/UHMWPE mixture. Firstly, in 30 ml of ethanol, the required GO wt. % content was dispersed and mixed with the UHMWPE powder. Secondly, the slurry was then placed in a zirconium oxide grinding jar containing 5 mm diameter zirconium oxide balls. Thirdly, the planetary ball mill was used for the preparation of the GO/UHMWPE mixture at a mixing rate of 400 rpm for 2 hours. After that, ethanol was extracted in the oil bath under stirring at 60°C and the powder was kept in the oven at 60°C for 24 hours until it was completely dried. Finally, nanocomposite and neat UHMWPE powder were moulded into sheets with size of 65 x 25 x 2 mm³ and 115 x 17 x 2 mm³ using a 185°C-hot press at 15 MPa pressure.

The study by Chen et al [17] was done to investigate the effects of GO/UHMWPE composites on the mechanical properties and biocompatibility when added GO (0, 0.1,

0.3, 0.5 and 1.0 wt. %) on it. The liquid-phase ultra-sonication dispersion method was used in order to prepare a series of GO/UHMWPE composites followed by hot pressing method. Firstly, GO powder was dispersed in 50 ml of alcohol to form a well dispersed suspension using ultra-sonic for 30 minutes. In the suspension, UHMWPE powders were added, and the mixture was stirred for 30 minutes and then ultra-sonicated for 1 hour. After that, the alcohol was removed in an oil bath at 60°C and the solid product was dried in a 60°C oven completely. The resulting solid was eventually formed into 70 x 70 x 10 mm³ shape by 195°C hot pressing under 10 MPa pressure and 20 minutes remaining at that pressure.

Chang et al [18] stated that the study is about to investigate the different filler loading of zeolite on mechanical and tribology behaviour of zeolite/UHMWPE composite. The amount of filler used was 5-20 wt. %. The mechanical behaviour as tensile properties and impact strength were studied on the neat UHMWPE and zeolite/UHMWPE composite while for the tribology behaviour, the worn surface and transfer films of pure UHMWPE and zeolite/UHMWPE composite were observed under SEM. The pin-on-disc tester with different loads applied and sliding speeds were used in order to investigate the tribology properties. The load is applied at 10-30 N while sliding speeds used are 0.209, 0.419 and 0.838 m/s.

The method used to prepare zeolite/UHMWPE composites were dry mechanical ball mill. The zeolite was mixed homogenously with UHMWPE after four hours including two hours in clockwise rotation and two hours in counter-clockwise rotation. The rotational speed for the ball mill is 1800 rpm (30 Hz). Before the mixture was pressed using hot press at 1000 psi (~ 6.90 MPa) for 7 min, the mixture was pre-heated for 10 min at 160°C. To obtain the zeolite/UHMWPE composite, cold pressing was applied at 15°C for 5 min. For further testing analysis the zeolite/UHMWPE composite was cut according desired dimensions.

2.5 Mechanical Properties for UHMWPE Composite

Based on Suner et al [12] the mechanical characterisation such as Young's modulus, yield stress, fracture stress, fracture strain and fracture toughness of UHMWPE were studied. The additions of small amounts of GO content improve the mechanical properties of the nanocomposite but it is decreased after reaching the optimum amount of GO. The Young's modulus of composite increased approximately 15% from the neat UHMWPE as the GO is added as shown in **Figure 2.3**. With the higher concentration of GO, the Young's modulus of GO/UHMWPE nanocomposite decreased but at 2.0 wt. % of GO it is increased again. This trend almost similar with yield stress result, as shown in **Figure 2.4**.

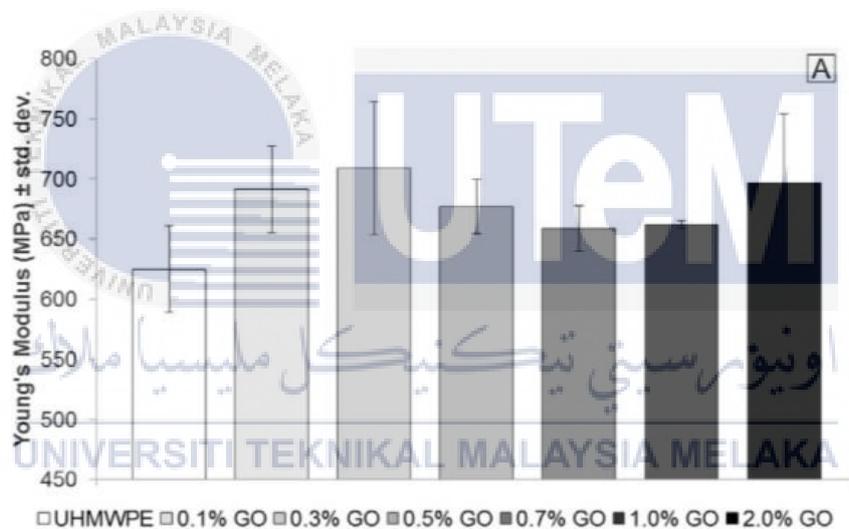


Figure 2.3: Young's modulus of GO/UHMWPE nanocomposites [12].

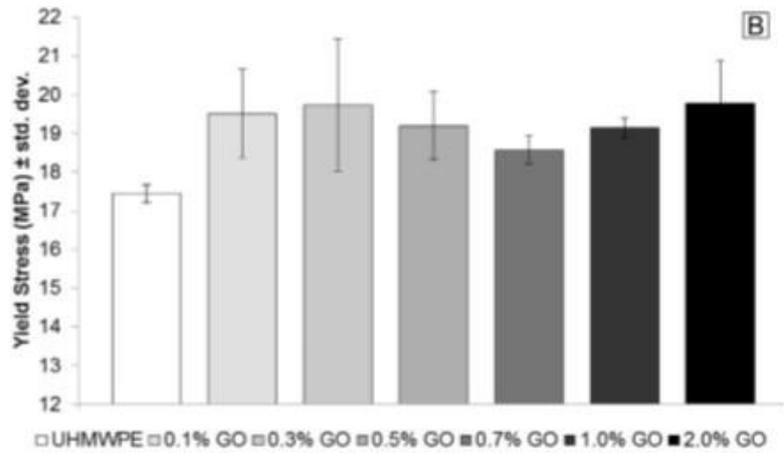


Figure 2.4: Yield stress of GO/UHMWPE nanocomposites [12].

Based on Chen et al [17], the stress-strain curve in **Figure 2.5** shows that the different amounts of GO added into UHMWPE had different effects on tension performance of this composite. The enhancement on yield strength was realized when the amount of GO added into UHMWPE even at low concentration.

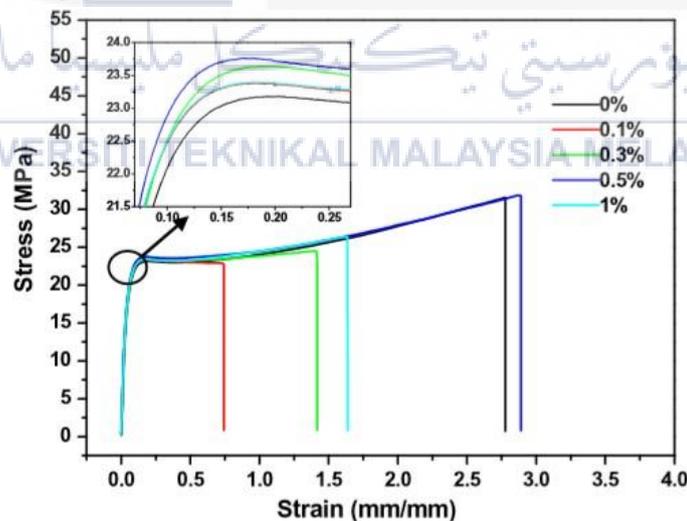


Figure 2.5: The stress-strain curve for the GO/UHMWPE composites with different concentration [17].

As shown in **Table 2.1**, when 0.1 wt. % of GO was added, it had increased the yield strength up to 23.03 MPa which is increased by 0.66% from the pure UHMWPE sample. The yield strength kept increased until it reached the optimum value for concentration which is 0.5 wt. % GO – 23.70 MPa. At 1 wt. % of GO the yield strength of this GO/UHMWPE composite was decreased 1.35% from the 0.5 wt. % GO concentration. The result for ultimate tensile strength and elongation at break with addition of 0.1 wt. % of GO was decreased but as further increase the concentration of GO up to 0.5 wt. %, the value of ultimate tensile strength and elongation at break also increased to 30.61 MPa and 2.76, respectively. To be concluded based on this study, the optimum amount of GO was 0.5 wt. %. This is because when further the concentration of GO, the mechanical properties of this GO/UHMWPE composites were decreased [17].

Table 2.1: Result for tensile properties of UHMWPE and GO/UHMWPE composite [17].

| Sample designation | Yield strength (MPa) | Ultimate tensile strength (MPa) | Elongation at break (%) |
|--------------------|----------------------|---------------------------------|-------------------------|
| UHMWPE | 22.88 ± 0.77 | 30.45 ± 1.27 | 2.71 ± 0.47 |
| GO/UHMWPE (0.1%) | 23.03 ± 0.46 | 22.82 ± 0.76 | 0.88 ± 0.32 |
| GO/UHMWPE (0.3%) | 23.42 ± 1.08 | 24.15 ± 1.56 | 1.20 ± 0.24 |
| GO/UHMWPE (0.5%) | 23.70 ± 1.16 | 30.61 ± 1.72 | 2.76 ± 0.53 |
| GO/UHMWPE (1%) | 23.38 ± 1.06 | 26.55 ± 0.80 | 1.53 ± 0.48 |

From Chang et al [18] the properties such as tensile strength, modulus and elongation at break were studied. Based on the result obtained, the uses of zeolite as reinforcing element into UHMWPE matrix has reduced the tensile strength and elongation at break but it increased the modulus of the zeolite/UHMWPE composite. As stated in this study, the tensile strength of pure UHMWPE was 52.27 MPa while elongation at break was 207.71. after the addition of the zeolite – 10 wt. %, the value for tensile strength has reduced to 46.80 MPa which is ~ 11%. This is same as elongation at break when 10 wt. % of zeolites added, the value reduced to 185.29 (~11 %). The modulus of 10 wt. % of zeolite/UHMWPE was 389.72

GPa. It is showed an increment of 92.4 GPa from pure UHMWPE. The result for other concentrations of filler are shown in **Table 2.2**. As conclusion, tensile strength and elongation at break of zeolite/UHMWPE composite decreased with the addition of higher concentration filler but it is increased in term of modulus of zeolite/UHMWPE composite.

Table 2.2: Tensile properties of pure UHMWPE and zeolite/UHMWPE composites [18].

| Sample | Density (g/cm ³) | Tensile Strength (MPa) | Modulus (GPa) | Elongation at break (%) | Impact Strength (kJ/m ²) |
|------------------------|------------------------------|------------------------|----------------|-------------------------|--------------------------------------|
| Pure UHMWPE | 0.93 | 52.57 ± 3.85 | 297.32 ± 16.83 | 207.71 ± 7.65 | 115.70 ± 9.47 |
| 10 wt.% Zeolite/UHMWPE | 0.96 | 46.80 ± 4.58 | 389.72 ± 46.66 | 185.29 ± 6.42 | 144.46 ± 12.91 |
| 20 wt.% Zeolite/UHMWPE | 1.01 | 31.01 ± 3.70 | 455.35 ± 30.51 | 129.02 ± 17.13 | 63.44 ± 12.53 |



CHAPTER 3

METHODOLOGY

3.1 Introduction

This chapter will show more details about the materials used to form the sample and methods used during the project starting from the beginning of the process until final process and the tests conducted on the samples. The mixing time of the two materials to form composite samples, the temperature and pressure for hot pressing also determined in this chapter.

3.2 Materials

In this study, Ultra-high Molecular Weight Polyethylene (UHMWPE) and Graphene Nanoplatelet (GNP) were used as the materials for the composite. These two materials were mixed in order to produce a GNP/UHMWPE powder mixture. The composition of these two materials are shown in **Table 3.1**. The total weight of Neat UHMWPE and GNP/UHMWPE composite sample are 8 g per sample. Nine samples were needed to be prepared for Neat UHMWPE and each type of GNP/UHMWPE composite to conduct test.

Table 3.1: Composition of UHMWPE and GNP in GNP/UHMWPE composite.

| Type of sample | No. of sample | UHMWPE (wt. %) | GNP (wt. %) |
|----------------|---------------|----------------|-------------|
| Neat UHMWPE | 9 | 100.0 | 0.0 |
| GNP/UHMWPE-0.1 | 9 | 99.9 | 0.1 |
| GNP/UHMWPE-0.3 | 9 | 99.7 | 0.3 |
| GNP/UHMWPE-0.5 | 9 | 99.5 | 0.5 |
| GNP/UHMWPE-1.0 | 9 | 99.0 | 1.0 |

Ultra-high Molecular Weight Polyethylene

UHMWPE in the form of white powder was used in this study as shown in **Figure 3.1**. The size of this powder is 150 μm . Other properties of UHMWPE are shown in **Table 3.2**.

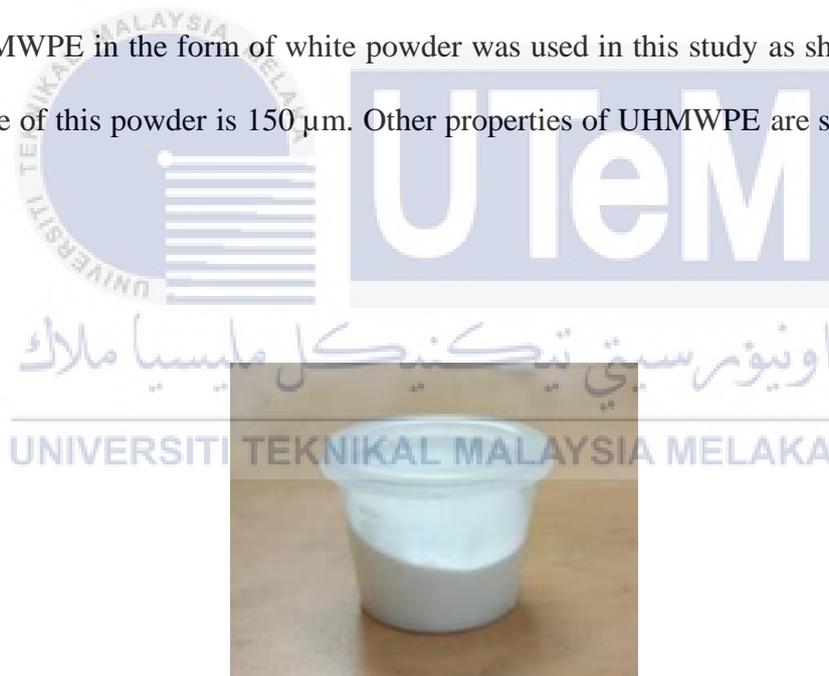


Figure 3.1: UHMWPE in the form of powder.

Table 3.2: Properties of UHMWPE.

| Properties | Value |
|---------------------------------------------------------|-----------|
| Density (g/cm ³) | 0.94 |
| Tensile strength at break (MPa) | 20 – 40 |
| Elongation at break (%) | 500 |
| Coefficient of thermal expansion $\times 10^{-6}K^{-1}$ | 130 – 200 |
| Coefficient of friction, P = 0.05 N/mm ² | 0.29 |
| Impact strength (kJ/m ²) | No break |
| Compressive stress at 10% deformation (psi) | 3000 |

Graphene Nanoplatelet

GNP powder, Grade C-500 was purchased from Merck. Sdn. Bhd. Malaysia with molecular weight of 12.01 g/mole and has surface area of 500 m²/g. The colour of GNP powder is black as shown in **Figure 3.2**.



Figure 3.2: GNP powder.

3.3 Mixing of Materials

The parameter such as time for mixing the UHMWPE and GNP need to be identified. In order to determine the suitable mixing time for these two materials, the rotational speed of Thinky mixer was fixed at 1500 rpm. The distribution of GNP in the UHMWPE matrix is important. In this case, it is difficult to check the distribution of GNP in UHMWPE due to their same particles which is Carbon (C). Therefore, nanoclay was utilised as an assumption has been made where nanoclay are same as GNP in terms of shape and size. The uses of nanoclay because it contains Magnesium (Mg), Aluminium (Al) and Silicon (Si) which the composition of nanoclay is different with GNP that contain Carbon (C). Then, it is easy to distinguish the particles between them.

For this purpose, four samples of nanoclay/UHMWPE mixture were prepared as shown in **Table 3.3**, where two samples (A and B) with 1.0 wt. % of nanoclay for 5 min and 10 min, respectively and two samples (C and D) with 0.5 wt. % of nanoclay for 5 min and 10 min, respectively.

The process started when the UHMWPE and the nanoclay were weighted according to the composition mentioned before using Mettler Toledo electronic balance (Model ME204E), as shown in **Figure 3.3**. For this purpose, the total weight for each sample is 5 g. After that, these two materials were placed together in a container. Then, the process was continued by dry mixing where the container was put inside a Thinky Mixer (Model ARE-310) as shown in **Figure 3.4**. The time and rotational speed for Thinky Mixer was set up. Thinky Mixer uses revolution and rotation to mix the material in a container. The placement of container is about 45° from rotational axis.

Table 3.3: Preparation of nanoclay/UHMWPE mixture.

| Mixing Time | nanoclay – 1.0 wt. % | nanoclay – 0.5 wt. % |
|-------------|------------------------------------------------------------------------------------------------|-------------------------------------------------------------------------------------------------|
| 5 min |  Sample A |  Sample C |
| 10 min |  Sample B |  Sample D |



Figure 3.3: Electronic balance.



Figure 3.4: Thinky mixer.

Once the mixing process was done, the mixture of nanoclay/UHMWPE was observed under Jeol scanning electron microscope (Model JSM-6010PLUS/LV). From the observation, nanoclay homogeneously dispersed into UHMWPE after 10 min of mixing. Thus, the time was applied on GNP and UHMWPE to produce composite sample.

3.4 Sample Preparation

GNP/UHMWPE Composite

The UHMWPE and GNP were weighted first based on the composition in **Table 3.1**. After that, these two materials were dry mixed using Thinky Mixer for 10 min and 1500 rpm. A rectangular mould consists of four segments of sample as shown in **Figure 3.5** was prepared. Wax was applied on both inside surfaces of the mould and the mixture of GNP and UHMWPE was placed into it. The uses of wax because it is easy to disassemble the sample from mould after cooling. The shape and dimension of the sample were followed ASTM D638 Type 1 standard as shown in **Figure 3.6**.

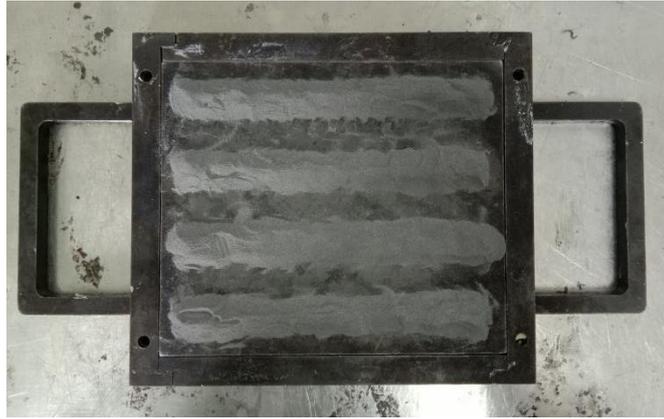


Figure 3.5: Mould for sample preparation.

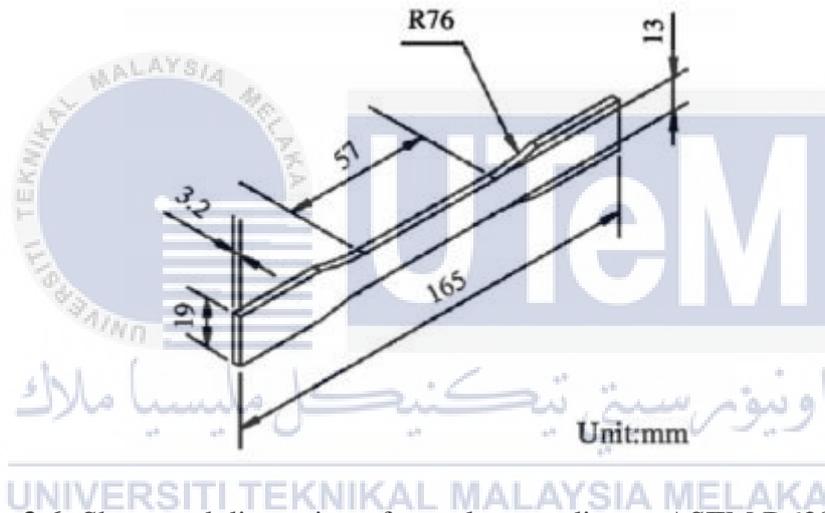


Figure 3.6: Shape and dimension of sample according to ASTM D638 Type 1.

Firstly, the mould with the GNP/UHMWPE mixture was preheated up to 180 °C before pressed using Motorise Hydraulic Test Press machine (Hot Press 30 Ton, Model GT-701 4-A) as shown in **Figure 3.7**. The hot press machine consists of two hot surfaces which are upper and lower surface. During preheating process, the mould was move towards upper surface with no pressure applied on it and left for 1 min. After that, the pressure was applied to the mould by pressing the button on machine for hot pressing process and it was followed the specifications as shown in **Figure 3.8**. Based on the graph of hot press condition, the

total time and maximum pressure for hot pressing process were 20 min and 1000 psi, respectively. The GNP/UHMWPE composite samples were obtained after cooling process for 10 min.



Figure 3.7: Preheating and hot-pressing process using Motorise Hydraulic Test Press Machine.

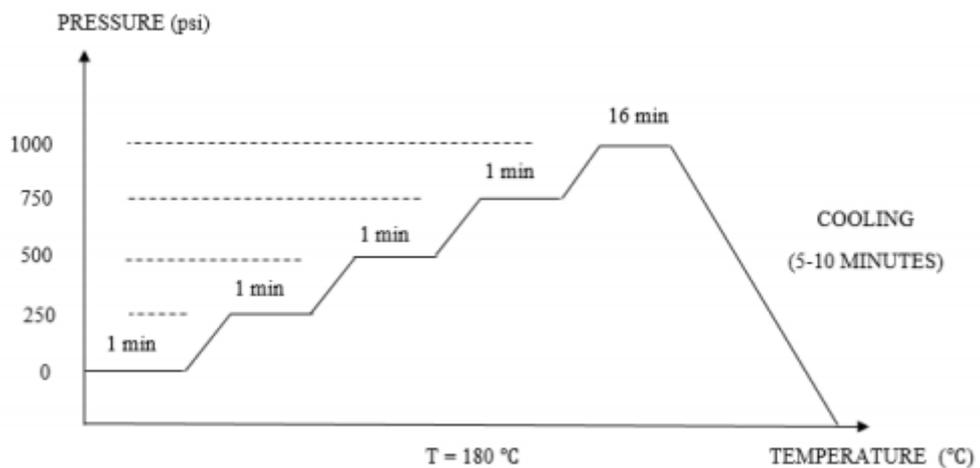


Figure 3.8: Graph of hot press condition.

Neat UHMWPE

For preparation of Neat UHMWPE sample, all the steps were same as to fabricate GNP/UHMWPE composite samples except for mixing process. No mixing process was involved during preparation of Neat UHMWPE sample. **Figure 3.9** shows the Neat UHMWPE was placed in mould for hot pressing process.



Figure 3.9: Preparation of Neat UHMWPE sample.

3.5 Density Analysis

Seven samples from neat and each type of composite were weighted using electronic balance to measure the mass. The mass was recorded, and the density of each sample was calculated using **Eq. (3.1)**.

$$\rho = \frac{m}{V} \quad (3.1)$$

Where ρ is the density of sample, m is the mass of sample and V is volume of sample which is 8004.63 mm^3 according to ASTM D638 Type 1.

3.6 Microhardness Test

The microhardness tester as shown in **Figure 3.10** was used. One sample from each type of samples were prepared for hardness test. The sample was placed on the test stage of the machine. The indentation force, 0.1 N and magnification lens, X40 were selected first before Vickers indentation was done on the sample. After the sample was indent by the Vickers, the indentation area was selected to know the hardness value. The selection for indentation area was done carefully by following the shape of the Vickers appeared on the surface of the sample.



Figure 3.10: Microhardness Tester.

3.7 Conductivity Test

The conductivity measurements of the samples were carried out using JANDEL Four-Point Probe Test as shown in **Figure 3.11**. Four-Point Probe worked by supplying current through two outer probes and measuring the voltage through two inner probes.



Figure 3.11: A JANDEL Four-Point Probe Test.

Nine samples from neat and each type of composite were prepared. The probe was placed on five different points on each sample to check the performance of the electrical and the average reading for resistivity were taken. The current used for this test is 10 nA as this can detect the resistivity in samples. The conductivity value of the samples was calculated using **Eq. (3.2)**.

$$\text{conductivity} = \frac{1}{R} \quad (3.2)$$

Where R is average resistivity.

3.8 Tensile Test

Five samples from neat and each type of composite were measured their thickness and width on three different points along gauge length of samples to calculate the average of thickness and width. The digital Vernier callipers was used as a tool to measure the thickness and width. These data were used during conducting tensile test. The Universal Testing Machine 50kN (INSTRON 5969) as shown in **Figure 3.12** was used. Tensile test was performed at room temperature with crosshead speed of 10 mm/min and followed

ASTM D638 Type 1. The extension used is 488 mm as it is the maximum allowable extension for this type of tensile machine. The tensile properties such as Young's modulus, tensile stress and tensile strain were recorded for further analysis.



Figure 3.12: Universal Testing Machine.

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CHAPTER 4

RESULT AND DISCUSSION

4.1 Introduction

This chapter will present the data and result for each test that was conducted during this project. The test started with determination parameter for mixing of material. The parameter was used to fabricate composite samples. Once samples were fabricated, the density analysis for each sample was carried out. After that, the test was continued to determine the microhardness of the sample. A conductivity test also done on each sample to measure the electrical performance passes through it. A tensile test was carried out to determine the tensile properties of each sample. In this chapter also will discuss in detail about the result.

4.2 Mixing of Material

As mentioned in previous chapter, nanoclay is used as substitute for GNP in order to identify the distribution of filler added in UHMWPE matrix. The uses of nanoclay to replace the GNP because generally similar in shape and size. The thickness and width are 1 nm and 100 nm, respectively. It can be assumed that the physical distribution of GNP in UHMWPE matrix is same as nanoclay in UHMWPE. Thus, the distribution of the filler is determined by the existence of the filler after mixing process. Mixing materials were undergo scanning electron microscopy to determine the existence of filler. By elemental analysis, the element

Silicon (Si) was detected as a component in nanoclay. For UHMWPE, the element Carbon (C) was detected as it is normal polymer.

The difference of Si existence after 5 and 10 min of mixing time at different weight percent (wt. %) of nanoclay is shown in **Figure 4.1**. The atomic percentage of Si is increase with the increasing of mixing time. At 0.5 wt. %, there is no significant to increase time since the difference of atomic percentage of Si between 5 and 10 min of mixing is 0.03%. At 1.0 wt. %, there is a significant to increase time. This is proved by the difference in the atomic percentage of Si in 5 and 10 min is bigger (0.31%). The increasing detection of Si is notified as distribution of filler within UHMWPE matrix. Thus, to be concluded that an appropriate mixing time for the dispersion of GNP in the UHMWPE matrix is 10 min. Thus, the decision was made where 10 min and 1500 rpm were used to produce samples for GNP/UHMWPE composite.

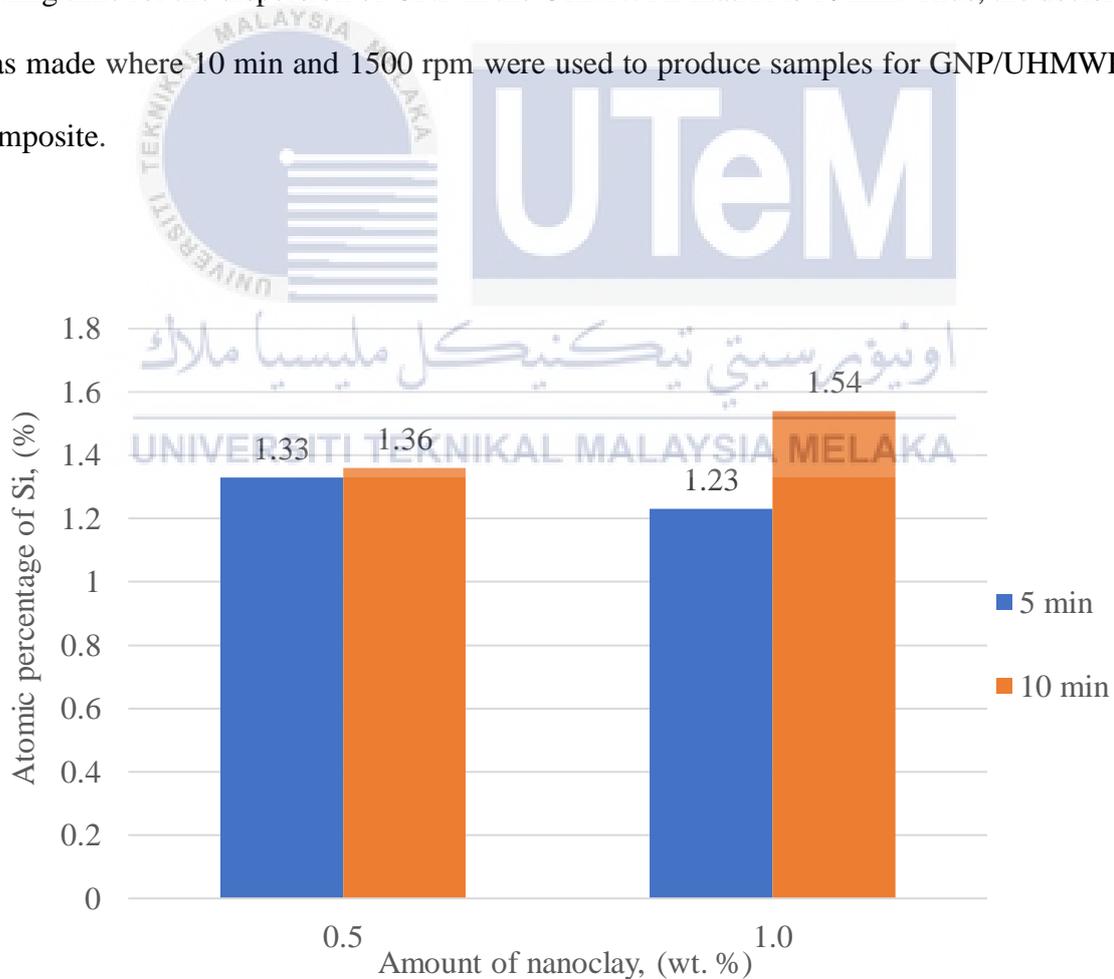


Figure 4.1: A graph of atomic percentage of Si with amount of nanoclay added.

Figure 4.2 – 4.5 show the elemental analysis of mixing materials at different mixing time and different wt. % of nanoclay.

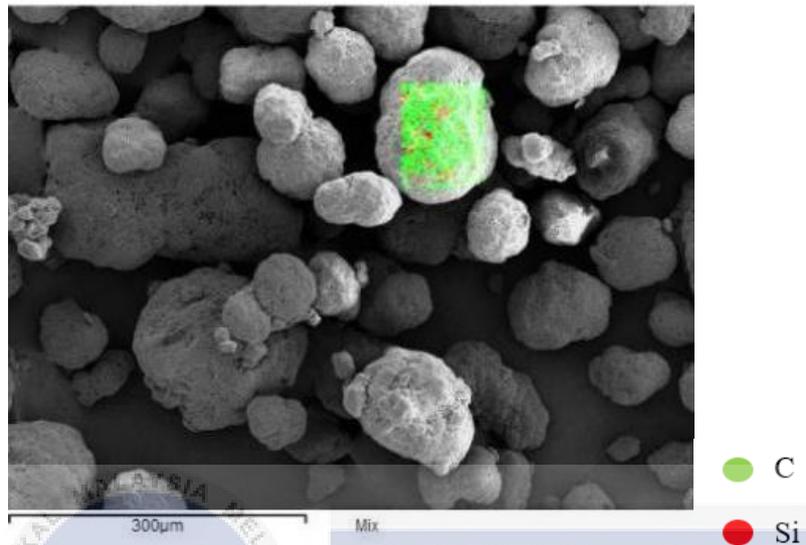


Figure 4.2: Mixing material with 1.0 wt. % of nanoclay at 5 min.

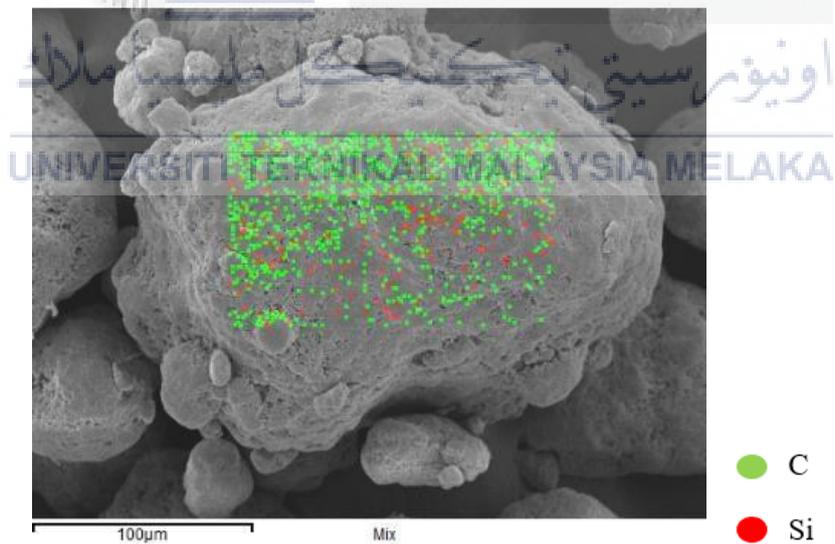


Figure 4.3: Mixing material with 1.0 wt. % of nanoclay at 10 min.

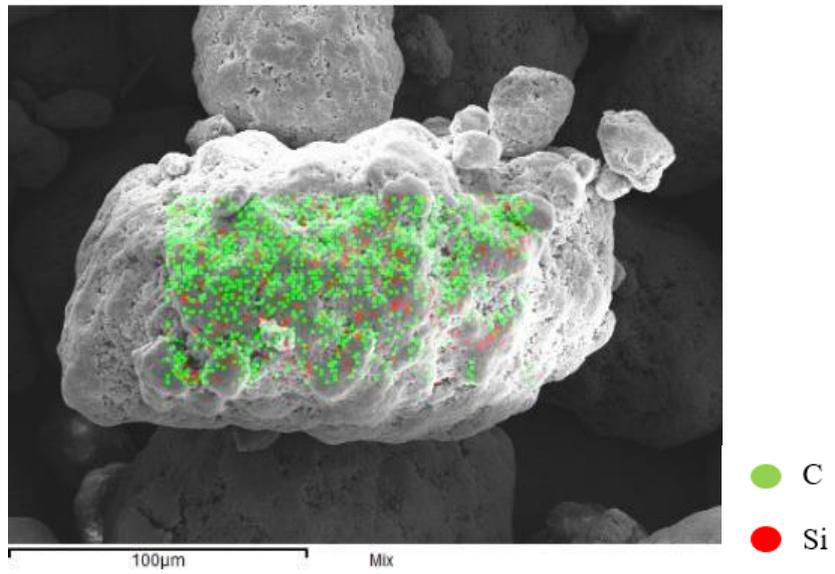


Figure 4.4: Mixing material with 0.5 wt. % of nanoclay at 5 min.



Figure 4.5: Mixing material with 0.5 wt. % of nanoclay at 10 min.

4.3 Density Analysis

The density of each neat and composite sample is calculated from Eq. (3.1) and summarize in Figure 4.6 – Figure 4.10.

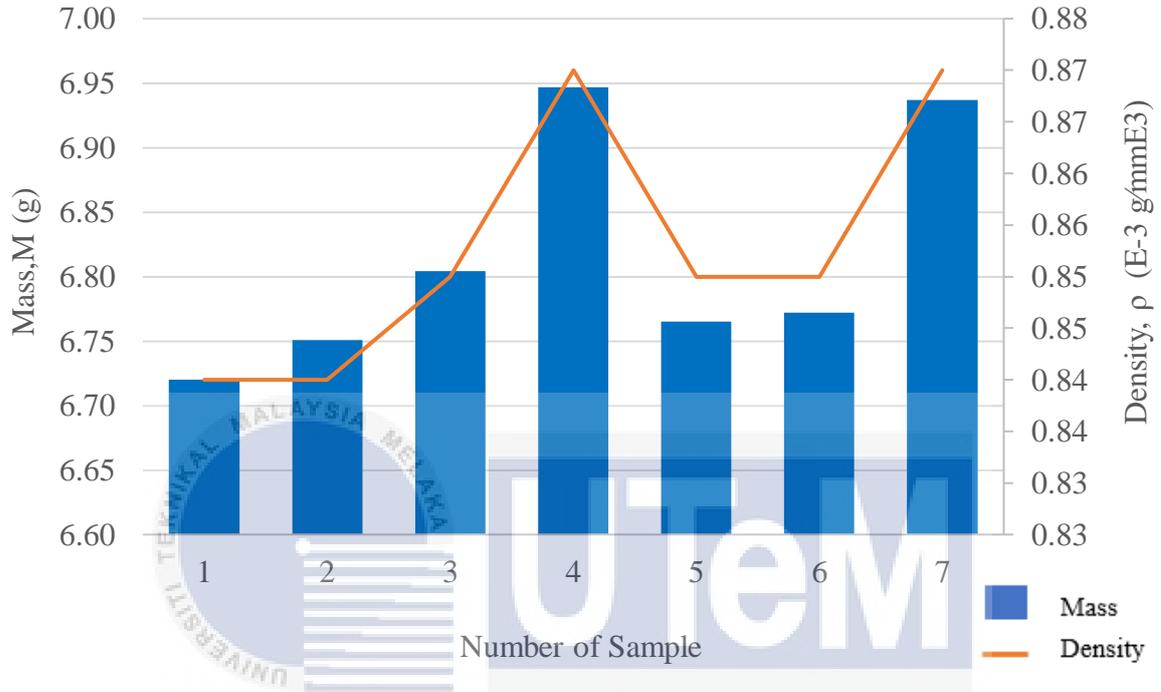


Figure 4.6: Graph of mass and density for Neat UHMWPE sample.

Based on Figure 4.6, the mass for sample number 1 is the lowest which is 6.72 g. The mass is increase up to sample number 4 which is the highest mass (6.95 g) for Neat UHMWPE sample. The mass for sample number 5 is decreased by 0.18 g from 6.95 g. After that, it is increase again up to sample number 7. For the density, it is directly proportional to the mass. The highest density is at sample number 4 and 7 which are $0.87 \times 10^{-3} \text{ g/mm}^3$.

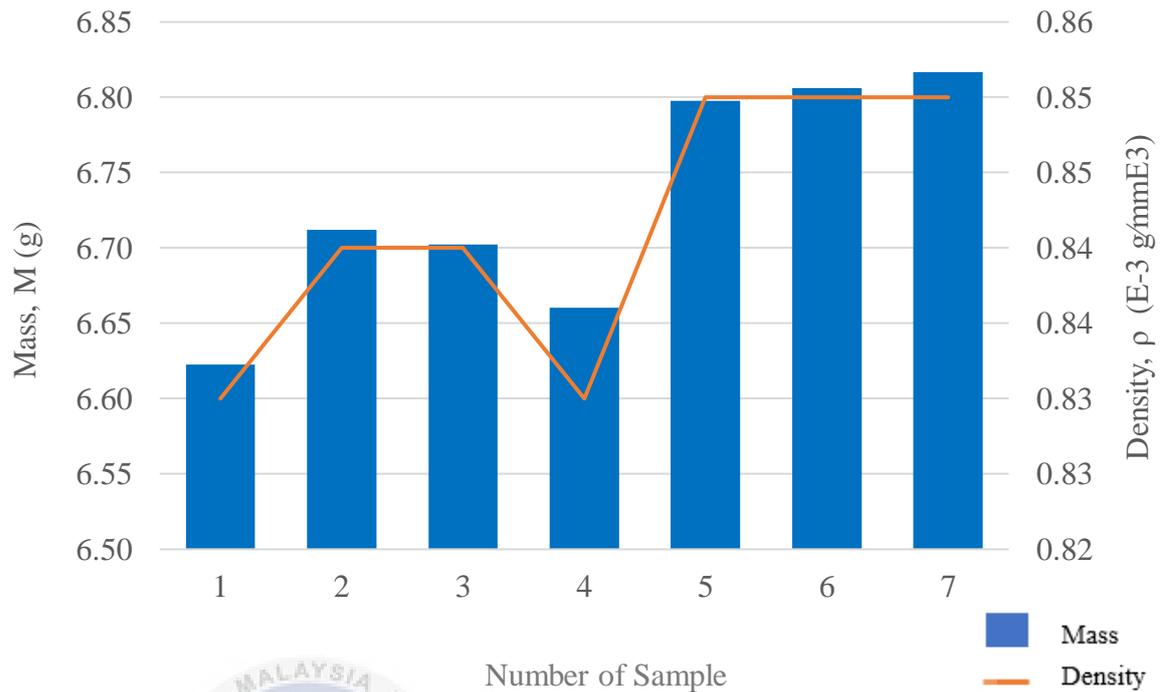


Figure 4.7: Graph of mass and density for GNP/UHMWPE-0.1 composite sample.

Based on **Figure 4.7**, the mass for sample number 1 is the lowest which is 6.62 g. From sample number 2 to 4, the mass is decreased. However, the reduction of mass for this sample is very small. Sample number 3 is reduced 0.01 g from 6.71 g in sample number 2. In sample number 4, the mass is 0.04 g less than mass for sample number 3. The mass is increased start from sample number 5 up to 7 but the density is constant which is $0.85 \times 10^{-3} \text{ g/mm}^3$. This is because the decimal places that considered during calculation. However, in overall sample, the density is directly proportional to the mass.



Figure 4.8: Graph of mass and density for GNP/UHMWPE-0.3 composite sample.

Based on **Figure 4.8**, mass for sample number 1 is 6.79 g. The mass is reduced 0.13 g from 6.79 g for sample number 2. After that, the mass is increased up to sample number 5 (the highest) which is 6.82 g. Then, the mass is decreased and increased again for sample number 6 and sample number 7, respectively. The maximum density for this type of composite is $0.85 \times 10^{-3} \text{ g/mm}^3$.

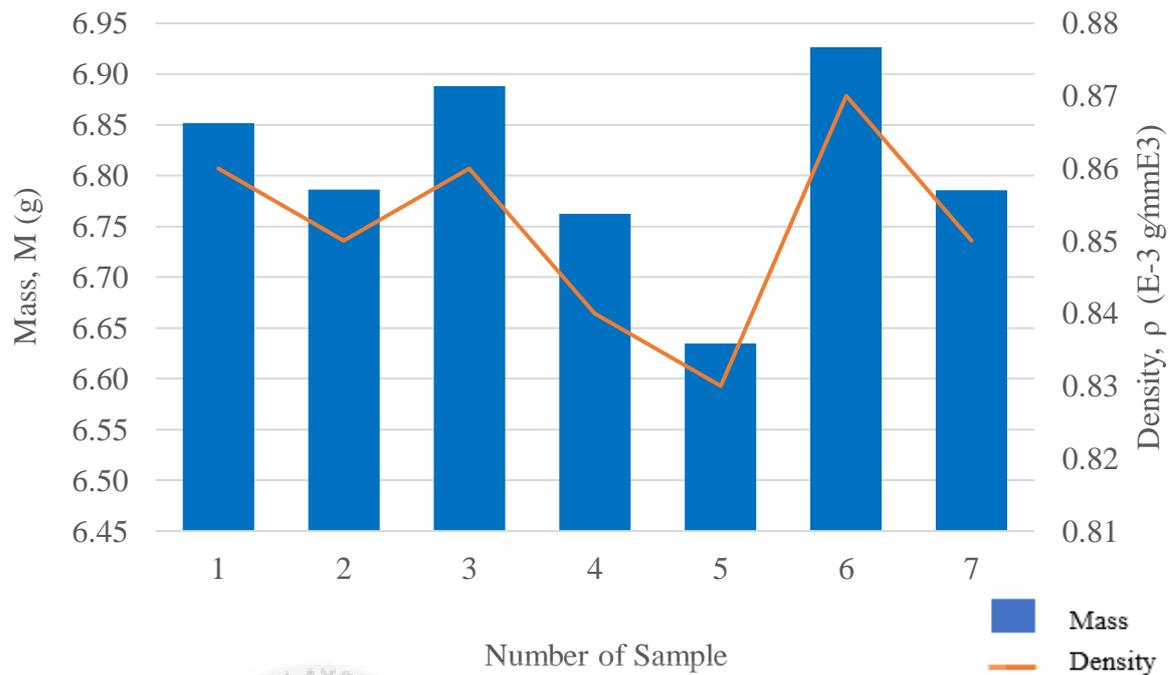


Figure 4.9: Graph of mass and density for GNP/UHMWPE-0.5 composite sample.

Based on **Figure 4.9**, the trend for the mass and density are not constant. The lowest mass is 6.63 g which is sample number 5 while the highest mass is 6.93 g which is sample number 6 in corresponding value of density is $0.87 \times 10^{-3} \text{ g/mm}^3$.



Figure 4.10: Graph of mass and density for GNP/UHMWPE-1.0 composite sample.

Based on the **Figure 4.10**, the lowest mass is sample number 2 which is 6.73 g while the highest mass is sample number 5 which is 6.95 g. The highest density is sample number 5 which is $0.87 \times 10^{-3} \text{ g/mm}^3$.

The average of mass and experimental density for each sample are shown in **Table 4.1**. The percentage error between theoretical density and experimental density also calculated.

Table 4.1: Average value of mass and density for all samples.

| Sample | Mass (g) | Theoretical Density ($\times 10^{-3}$ g/mm ³) | Experimental Density ($\times 10^{-3}$ g/mm ³) | Percentage Error (%) |
|----------------|-------------|------------------------------------------------------------------|-------------------------------------------------------------------|----------------------------|
| Neat UHMWPE | 6.81±0.09 | 0.940 | 0.853±0.013 | 9.3 |
| GNP/UHMWPE-0.1 | 6.73±0.08 | 0.939 | 0.841±0.009 | 10.4 |
| GNP/UHMWPE-0.3 | 6.75±0.06 | 0.936 | 0.843±0.008 | 9.9 |
| GNP/UHMWPE-0.5 | 6.81±0.10 | 0.934 | 0.851±0.013 | 8.9 |
| GNP/UHMWPE-1.0 | 6.83±0.08 | 0.927 | 0.853±0.011 | 8.0 |

The theoretical density of each sample is decreased as the GNP loading is increased as shown in **Table 4.1**. This is because the GNP is a type of filler with lower density. Thus, GNP contributed to produce light weight material which is important characteristic of composite. For the experimental density, the calculation only based on the volume of mold drawing which is 8004.63 mm³. However, the experimental density is dependent on mass and the average value is constant but not precise. Moreover, the percentage error between theoretical density and experimental density is might due to the percentage of porosity that exist within the samples and the percentage of shrinkage of the samples during fabrication that is not consider, i.e. fixed volume of sample that is used for the calculation of the experimental density.

4.4 Microhardness Test

A microhardness test was done on each neat and composite sample to determine the hardness value of the sample. As mentioned in previous chapter, the sample was indented by the Vickers as a type of indenter. Six readings were taken from different points on a sample and the average of hardness were calculated. Each reading was denoted as R_1 to R_6 .

Neat UHMWPE

Figure 4.11 shows the selected area after indentation. The hardness value in **Table 4.2** for Neat UHMWPE in R_1 is 4.82 HV and it decrease to 4.62 HV in R_2 . However, in overall point taken, the value for hardness is consistence. The average and standard deviation are 4.74 HV and 0.073, respectively.

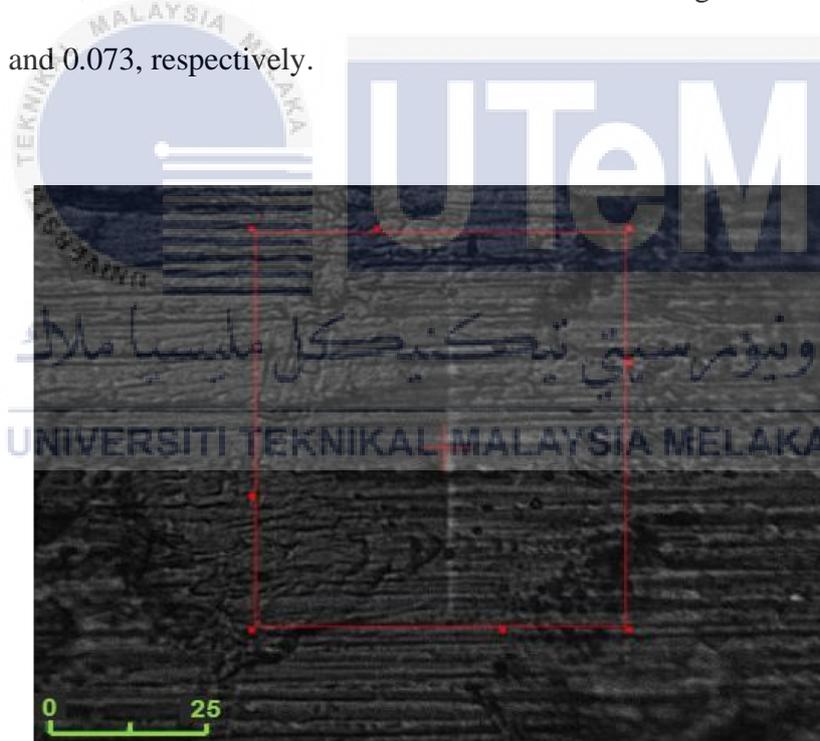


Figure 4.11: Indentation area on Neat UHMWPE sample.

Table 4.2: Hardness Value for Neat UHMWPE sample.

| | R_1 | R_2 | R_3 | R_4 | R_5 | R_6 | Average (HV) |
|----------|-------|-------|-------|-------|-------|-------|------------------|
| Sample 1 | 4.82 | 4.62 | 4.75 | 4.78 | 4.78 | 4.69 | 4.74 ± 0.073 |

GNP/UHMWPE-0.1 Composite

Figure 4.12 shows the indented area by the Vickers on the GNP/UHMWPE-0.1 composite sample. The hardness value for R_1 is 4.97 HV while in R_2 is 4.95 HV and other readings on sample are shown in **Table 4.3**. The average and standard deviation are 4.97 HV and 0.038, respectively.

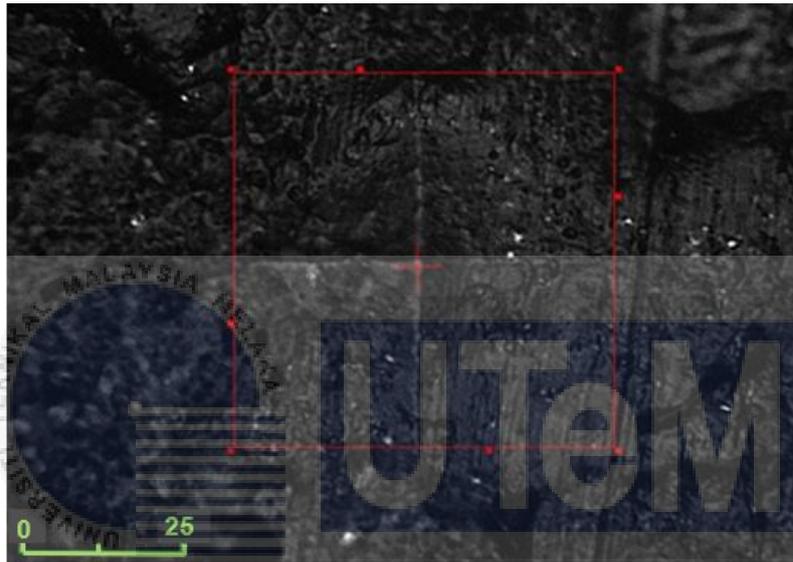


Figure 4.12: Indentation area on GNP/UHMWPE-0.1 composite sample.

Table 4.3: Hardness Value for GNP/UHMWPE-0.1 composite sample.

| | R_1 | R_2 | R_3 | R_4 | R_5 | R_6 | Average (HV) |
|----------|-------|-------|-------|-------|-------|-------|------------------|
| Sample 1 | 4.97 | 4.95 | 5.00 | 4.90 | 4.98 | 5.00 | 4.97 ± 0.038 |

GNP/UHMWPE-0.3 Composite

Figure 4.13 shows the indented area by the Vickers on the GNP/UHMWPE-0.3 composite sample. The hardness value for R_1 is 5.05 HV while in R_2 is 5.04 HV and other readings on sample are shown in **Table 4.4**. The average and standard deviation are 5.06 HV and 0.020, respectively.

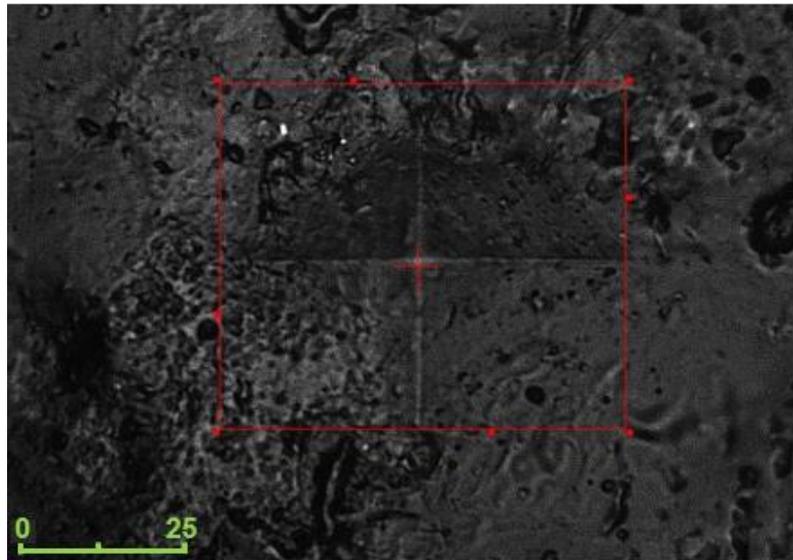


Figure 4.13: Indentation area on GNP/UHMWPE-0.3 composite sample.

Table 4.4: Hardness Value for GNP/UHMWPE-0.3 composite sample.

| | R_1 | R_2 | R_3 | R_4 | R_5 | R_6 | Average (HV) |
|----------|-------|-------|-------|-------|-------|-------|------------------|
| Sample 1 | 5.05 | 5.04 | 5.05 | 5.09 | 5.08 | 5.05 | 5.06 ± 0.020 |

GNP/UHMWPE-0.5 Composite

Figure 4.14 shows the indented area by the Vickers on the GNP/UHMWPE-0.5 composite sample. The hardness value for R_1 is 5.21 HV while in R_2 is 5.25 HV and other readings on sample are shown in **Table 4.5**. The average and standard deviation are 5.22 HV and 0.025, respectively.

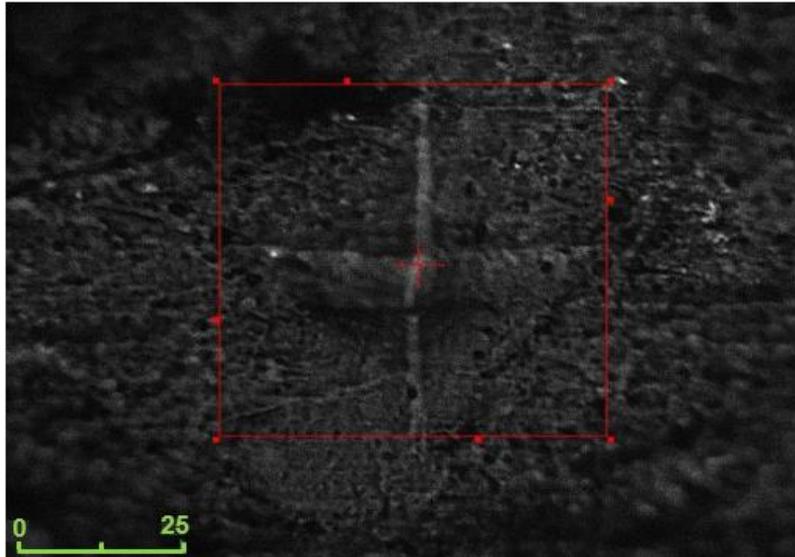


Figure 4.14: Indentation area on GNP/UHMWPE-0.5 composite sample.

Table 4.5: Hardness Value for GNP/UHMWPE-0.5 composite sample.

| | R_1 | R_2 | R_3 | R_4 | R_5 | R_6 | Average (HV) |
|----------|-------|-------|-------|-------|-------|-------|------------------|
| Sample 1 | 5.21 | 5.25 | 5.18 | 5.23 | 5.20 | 5.23 | 5.22 ± 0.025 |

GNP/UHMWPE-1.0 Composite

Figure 4.15 shows the indented area by the Vickers on the GNP/UHMWPE-1.0 composite sample. The hardness value for R_1 is 5.44 HV while in R_2 is 5.41 HV and other readings on sample are shown in **Table 4.6**. The average and standard deviation are 5.43 HV and 0.015, respectively.

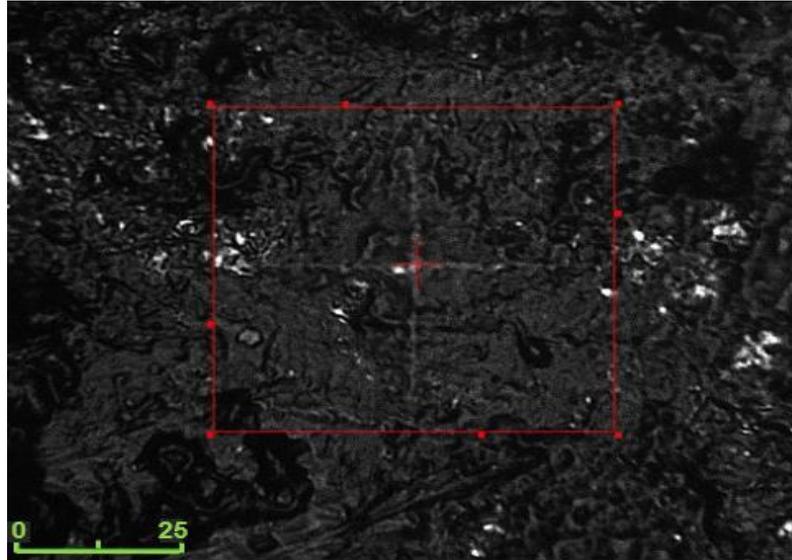


Figure 4.15: Indentation area on GNP/UHMWPE-1.0 composite sample.

Table 4.6: Hardness Value for GNP/UHMWPE-1.0 composite sample.

| | R_1 | R_2 | R_3 | R_4 | R_5 | R_6 | Average (HV) |
|----------|-------|-------|-------|-------|-------|-------|------------------|
| Sample 1 | 5.44 | 5.41 | 5.42 | 5.44 | 5.42 | 5.45 | 5.43 ± 0.015 |

As mentioned in Chapter 1, GNP has good mechanical strength. Thus, when GNP is added into UHMWPE matrix, it can increase the hardness value of the composite as shown in **Figure 4.16**. The addition of 1.0 wt. % of GNP increased the hardness of Neat UHMWPE from 4.74 to 5.43 HV in corresponding 15% of increasing from neat sample. The small amount of filler added also enhanced the hardness of the sample. As example, 0.1 wt. % of GNP increased 5% hardness value from neat sample. This was due to an excellent mechanical property of GNP as filler in UHMWPE matrix. This result met Chen et al [17] when the GO added in UHMWPE matrix increased the microhardness of the composite as shown in **Figure 4.17**.

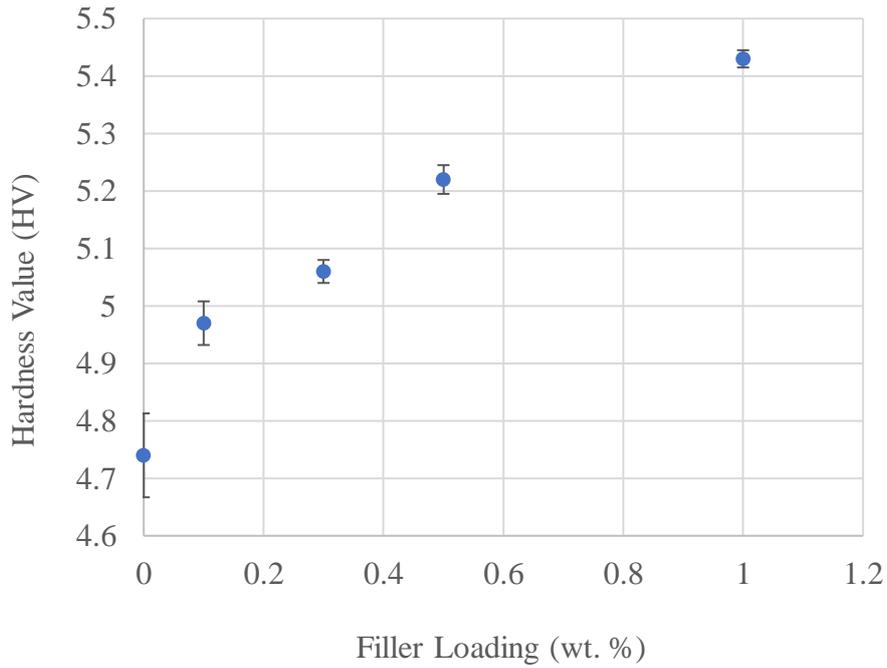


Figure 4.16: Graph of hardness value against filler loading.

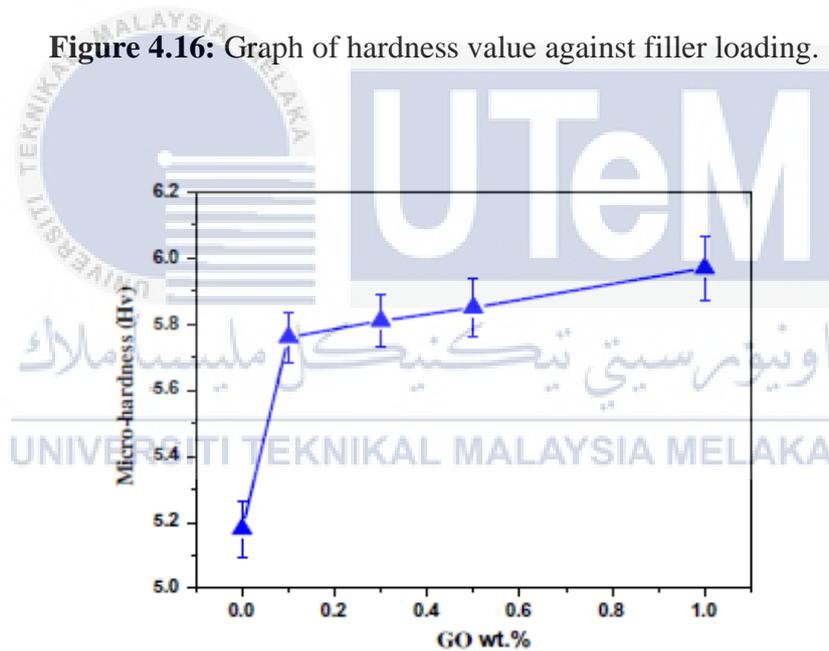


Figure 4.17: The microhardness of GO/UHMWPE composite with different amount of GO [17].

4.5 Conductivity Test

GNP is a type of filler that has properties of good electrical conductivity. As it is conductor, GNP is assumed to conduct electric when added in matrix material. For Neat UHMWPE, it is totally cannot conduct electric because UHMWPE is pure polymer.

However, the small amount of GNP (0.1 and 0.3 wt. %) added to the UHMWPE matrix cannot detect the conductivity in the samples. This is because the amount of GNP is insufficient to conduct electric even the GNP was distributed well within UHMWPE matrix as shown in **Figure 4.18**. The schematic figure shows that the 4-Point Probe was placed at any point along the samples. The 4-Point Probe cannot give the value of resistivity thus it means these types of samples were considered as non-conductive sample.

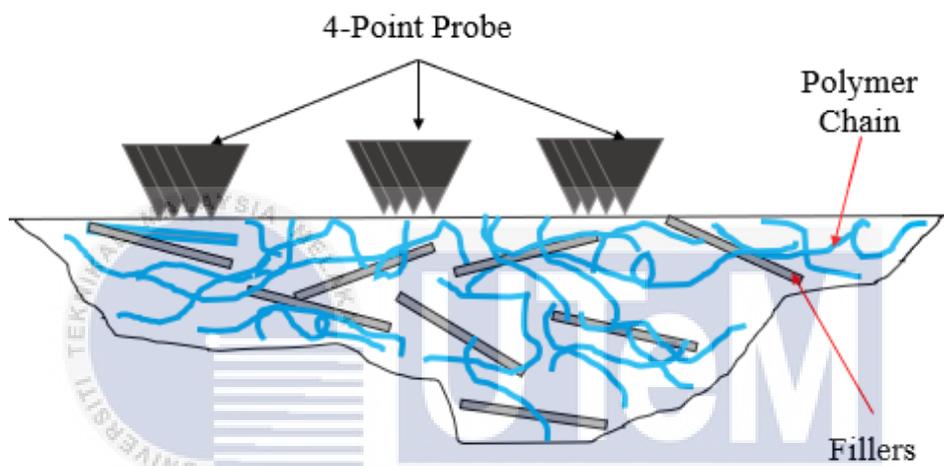


Figure 4.18: Schematic figure of non-conductive sample.

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GNP/UHMWPE-0.5 Composite

The resistivity of nine samples were measured and five readings were taken on each sample to calculate the average resistivity within the region as shown in **Table 4.7**. Each reading is denoted as the value of R_1 to R_5 . The readings of sample with negative value were not accounted during calculating the average resistivity. The lowest resistivity value is $79.38 \times 10^6 \Omega/\text{sqr}$ which is GNP/UHMWPE-0.5-8 while the highest resistivity value is $422.27 \times 10^6 \Omega/\text{sqr}$ which is GNP/UHMWPE-0.5-7. The average resistivity for GNP/UHMWPE-0.5 composite sample is $199.66 \times 10^6 \Omega/\text{sqr}$ with value of standard deviation is 112.12. The standard deviation is bigger because the average value of resistivity is varying within

samples. At some areas, the resistivity is high compared to other areas. For example, R_1 in GNP/UHMWPE-0.5-1 shows the highest value for resistivity which is $320.59 \times 10^6 \Omega/\text{sqr}$ compared to R_3 ($24.73 \times 10^6 \Omega/\text{sqr}$). This shows that it is highly conductive at certain area. The inconsistency of the resistivity value contributed to the large value of standard deviation in GNP/UHMWPE-0.5 composite sample.

Table 4.7: Resistivity value for 9 samples of GNP/UHMWPE-0.5 composite.

| | R_1 | R_2 | R_3 | R_4 | R_5 | Average ($\times 10^6 \Omega/\text{sqr}$) |
|------------------|---------|---------|---------|---------|---------|------------------------------------------------|
| GNP/UHMWPE-0.5-1 | 320.59 | -287.10 | 24.73 | 74.68 | -44.24 | 140.00 |
| GNP/UHMWPE-0.5-2 | 370.89 | 21.51 | 259.76 | 12.66 | -54.61 | 166.20 |
| GNP/UHMWPE-0.5-3 | 299.67 | 90.55 | 112.16 | -135.36 | 269.26 | 192.91 |
| GNP/UHMWPE-0.5-4 | 453.17 | 264.63 | 51.96 | 201.34 | -105.14 | 242.78 |
| GNP/UHMWPE-0.5-5 | 3.70 | 375.05 | 369.96 | 455.76 | 392.70 | 319.43 |
| GNP/UHMWPE-0.5-6 | -156.09 | -21.59 | 126.00 | 112.11 | 19.06 | 85.73 |
| GNP/UHMWPE-0.5-7 | -16.75 | 392.56 | 445.51 | -52.92 | 428.73 | 422.27 |
| GNP/UHMWPE-0.5-8 | 55.14 | 157.77 | -406.13 | -86.73 | 25.23 | 79.38 |
| GNP/UHMWPE-0.5-9 | 313.55 | 30.28 | 147.27 | 147.27 | 102.85 | 148.25 |
| | | | | | | 199.66 ± 112.12 |

GNP/UHMWPE-1.0 Composite

The method to find the resistivity of GNP/UHMWPE-1.0 composite samples are similar with the previous composite. **Table 4.8** shows the readings and the average value of resistivity of each sample and each reading is denoted as the value of R_1 to R_5 . The lowest

resistivity value is $29.46 \times 10^6 \Omega/\text{sqr}$ which is GNP/UHMWPE-1.0-9 while the highest resistivity value is $172.62 \times 10^6 \Omega/\text{sqr}$ which is GNP/UHMWPE-1.0-8. The average resistivity value for GNP/UHMWPE-1.0 composite sample is $89.55 \times 10^6 \Omega/\text{sqr}$ with value of standard deviation is 54.88.

Table 4.8: Resistivity value for 9 samples of GNP/UHMWPE-1.0 composite.

| | R_1 | R_2 | R_3 | R_4 | R_5 | Average ($\times 10^6 \Omega/\text{sqr}$) |
|------------------|---------|--------|--------|--------|--------|------------------------------------------------|
| GNP/UHMWPE-1.0-1 | 53.60 | 96.06 | 182.03 | -65.25 | -36.35 | 110.57 |
| GNP/UHMWPE-1.0-2 | 53.68 | 2.14 | 196.09 | -53.11 | 370.03 | 155.49 |
| GNP/UHMWPE-1.0-3 | 19.86 | 4.29 | 361.79 | 28.02 | 36.94 | 90.18 |
| GNP/UHMWPE-1.0-4 | 27.60 | 45.32 | 30.49 | -26.84 | 19.24 | 30.66 |
| GNP/UHMWPE-1.0-5 | 428.79 | -0.15 | 27.83 | 2.59 | 37.52 | 124.18 |
| GNP/UHMWPE-1.0-6 | 19.27 | 36.30 | 45.19 | 28.29 | -53.81 | 32.26 |
| GNP/UHMWPE-1.0-7 | 151.04 | 44.60 | 9.33 | 5.03 | 92.88 | 60.57 |
| GNP/UHMWPE-1.0-8 | 19.14 | 54.06 | 356.39 | 363.47 | 70.03 | 172.62 |
| GNP/UHMWPE-1.0-9 | -415.14 | -32.97 | 46.12 | 11.97 | 30.28 | 29.46 |
| | | | | | | 89.55±54.88 |

The summary from conductivity test is shown in **Table 4.9**. Since the resistivity of Neat UHMWPE, GNP/UHMWPE-0.1, and GNP/UHMWPE-0.3 are not available, thus the conductivity value of its also not available. The conductivity value for GNP/UHMWPE-0.5 and GNP/UHMWPE-1.0 were calculated using **Eq. (3.2)**.

Table 4.9: Average resistivity and conductivity values for all samples.

| Sample | Average resistivity ($\times 10^6 \Omega/\text{sqr}$) | Conductivity ($\times 10^{-9} \text{ S/sqr}$) |
|----------------|------------------------------------------------------------|----------------------------------------------------|
| Neat UHMWPE | N/A | N/A |
| GNP/UHMWPE-0.1 | N/A | N/A |
| GNP/UHMWPE-0.3 | N/A | N/A |
| GNP/UHMWPE-0.5 | 199.66 \pm 112.12 | 5.01 |
| GNP/UHMWPE-1.0 | 89.55 \pm 54.88 | 11.17 |

Based on the information in **Table 4.9**, the conductivity value for GNP/UHMWPE-0.5 is $5.01 \times 10^{-9} \text{ S/sqr}$ while GNP/UHMWPE-1.0 is $11.17 \times 10^{-9} \text{ S/sqr}$. The value for GNP/UHMWPE-1.0 is higher than GNP/UHMWPE-0.5 because as more GNP added, the samples were in conductive state even the value is measured as 10^{-9} S/sqr . At these amounts of filler added, the polymer chain and GNP are connected to each other as shown in **Figure 4.19**. When the 4-Point Probe was placed at any point on the sample, it can detect the resistivity in the sample. Thus, the schematic figure shows the conductive condition of the samples when tested with 4-Point Probe. The schematic figure also revealed the inconsistency value of the resistivity in GNP/UHMWPE-0.5 and GNP/UHMWPE-1.0. Case 1 in schematic figure defined the highly conductive region while Case 2 and Case 3 are partially conductive region.

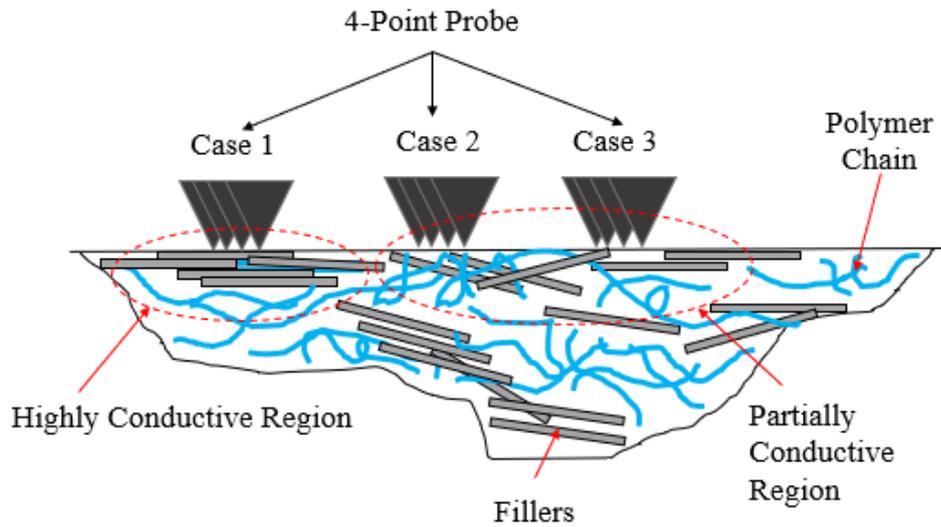


Figure 4.19: Schematic figure of conductive sample.

4.6 Tensile Test

As mentioned in previous chapter, the tensile test was done on each neat and composite sample to determine the tensile properties.

Neat UHMWPE

The **Figure 4.20** represents the stress-strain curve for each of Neat UHMWPE sample obtained from tensile test.

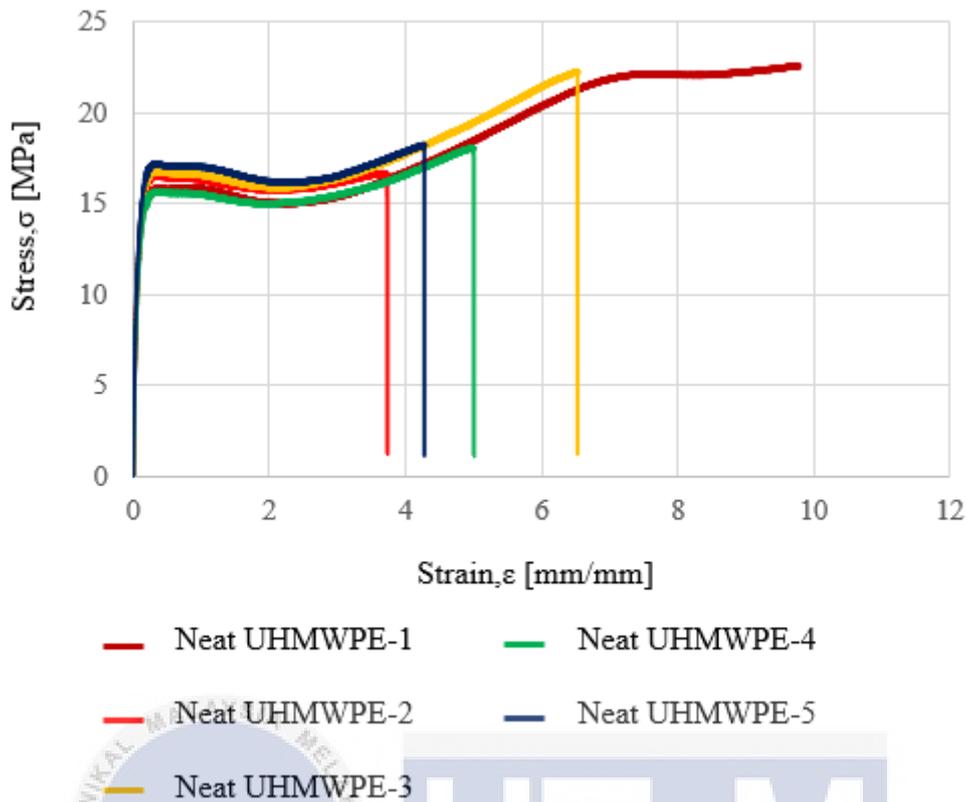


Figure 4.20: Stress-strain curves for Neat UHMWPE samples.

The initial linear region in the stress-strain curve is called elastic region where at this region the material will return to its original length when a force is released. Once the material deformed (past the elastic region and enter the plastic region), the material will not return to its original length. For pure polymer such as Neat UHMWPE sample, it is considered as ductile material. Thus, when it is tested under tensile, the material only elongates until reached the maximum extension that has been set up. In this case, it is different when there are four samples from Neat UHMWPE (2,3,4 and 5) that break during conducting the test as shown in **Figure 4.20**. Only Neat UHMWPE-1 was not break. The **Figure 4.21** shows the Neat UHMWPE samples after tensile test.



Figure 4.21: Neat UHMWPE sample after tensile test.

The tensile properties obtained from this test was summarize in **Table 4.10**. The Young's modulus was calculated based on slope at linear region of each stress-strain curve. Based on **Table 4.10**, Neat UHMWPE-1 shows the highest tensile strength which is 22.57 MPa and its tensile strain is 9.78 mm/mm. The lowest value of tensile strength is Neat UHMWPE-2 which is 16.38 MPa and its tensile strain is 3.73 mm/mm. In term of Young's modulus, the Neat UHMWPE-5 shows the highest value which is 0.317 GPa. However, the Young's modulus can be said as consistence for each sample.

Table 4.10: Tensile properties of Neat UHMWPE samples.

| Sample | Tensile Strength (MPa) | Tensile Strain (mm/mm) | Young's Modulus (GPa) |
|---------------|---------------------------|---------------------------|--------------------------|
| Neat UHMWPE-1 | 22.57 | 9.78 | 0.304 |
| Neat UHMWPE-2 | 16.38 | 3.73 | 0.313 |
| Neat UHMWPE-3 | 21.40 | 6.53 | 0.303 |
| Neat UHMWPE-4 | 17.24 | 5.00 | 0.305 |
| Neat UHMWPE-5 | 18.95 | 4.28 | 0.317 |
| Average | 19.31 | 5.86 | 0.308 |

GNP/UHMWPE-0.1 Composite

The **Figure 4.22** represents the stress-strain curve obtained from the tensile test conducted on GNP/UHMWPE-0.1 composite samples.

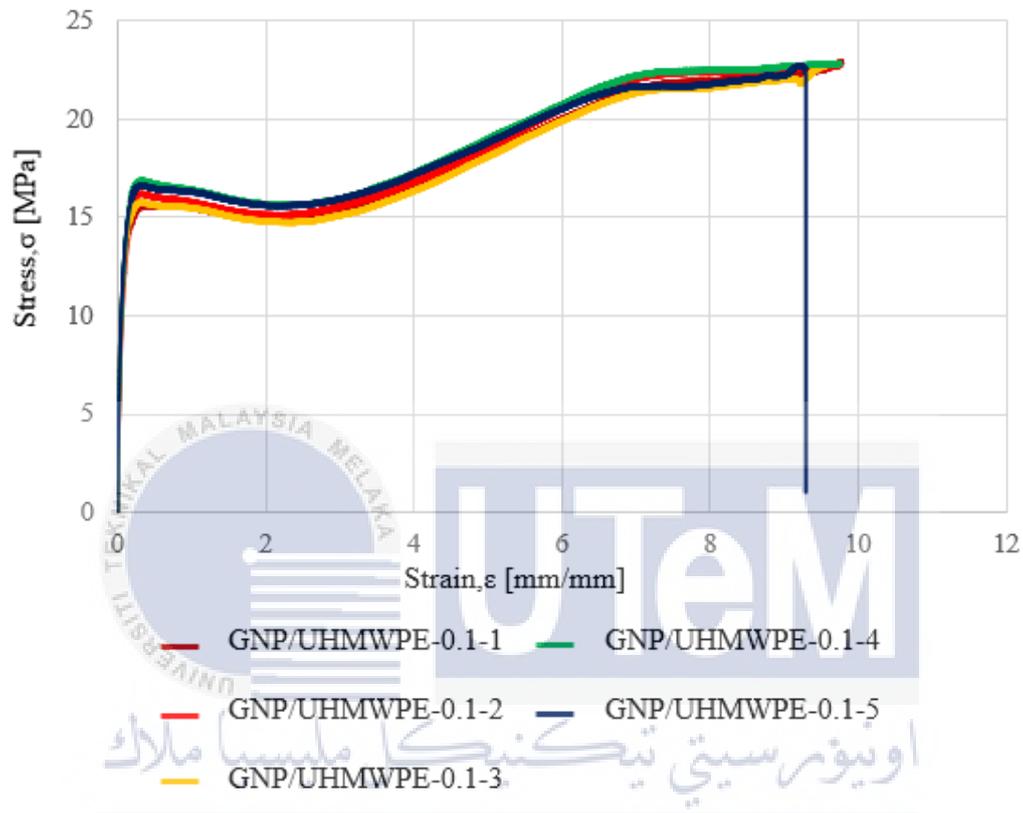


Figure 4.22: Stress-strain curves for GNP/UHMWPE-0.1 composite samples.

As shown in **Figure 4.22**, all the samples experienced similar pattern when tested under tensile except for the GNP/UHMWPE-0.1-5 where the sample was break before reached its maximum extension. The other 4 samples were elongated until reach the maximum extension available on tensile machine. The samples can be said as tough since the samples do not break. The **Figure 4.23** shows the GNP/UHMWPE-0.1 composite samples after tensile test.



Figure 4.23: GNP/UHMWPE-0.1 composite samples after tensile test.

The **Table 4.11** shows the tensile properties obtained from tensile test for this type of composite. Based on the information, the tensile strength for GNP/UHMWPE-0.1-1 is the highest among the other samples which is 22.93 MPa while the GNP/UHMWPE-0.1-4 is the lowest which is 22.40 MPa. Besides, the highest tensile strain is GNP/UHMWPE-0.1-1 which is 9.78 mm/mm while the lowest tensile strain is GNP/UHMWPE-0.1-5 which is 9.29 mm/mm. In addition, the Young's modulus of the samples is the highest for GNP/UHMWPE-0.1-5 which is 0.330 GPa. However, the Young's modulus for GNP/UHMWPE-0.1-1 is the lowest where the value is 0.288 GPa.

Table 4.11: Tensile properties of GNP/UHMWPE-0.1 composite samples.

| Sample | Tensile Strength (MPa) | Tensile Strain (mm/mm) | Young's Modulus (GPa) |
|------------------|---------------------------|---------------------------|--------------------------|
| GNP/UHMWPE-0.1-1 | 22.93 | 9.78 | 0.288 |
| GNP/UHMWPE-0.1-2 | 22.69 | 9.76 | 0.322 |
| GNP/UHMWPE-0.1-3 | 22.49 | 9.67 | 0.307 |
| GNP/UHMWPE-0.1-4 | 22.40 | 9.76 | 0.309 |
| GNP/UHMWPE-0.1-5 | 22.50 | 9.29 | 0.330 |
| Average | 22.60 | 9.65 | 0.311 |

GNP/UHMWPE-0.3 Composite

The **Figure 4.24** represents the stress-strain curve obtained from the tensile test conducted on GNP/UHMWPE-0.3 composite samples.

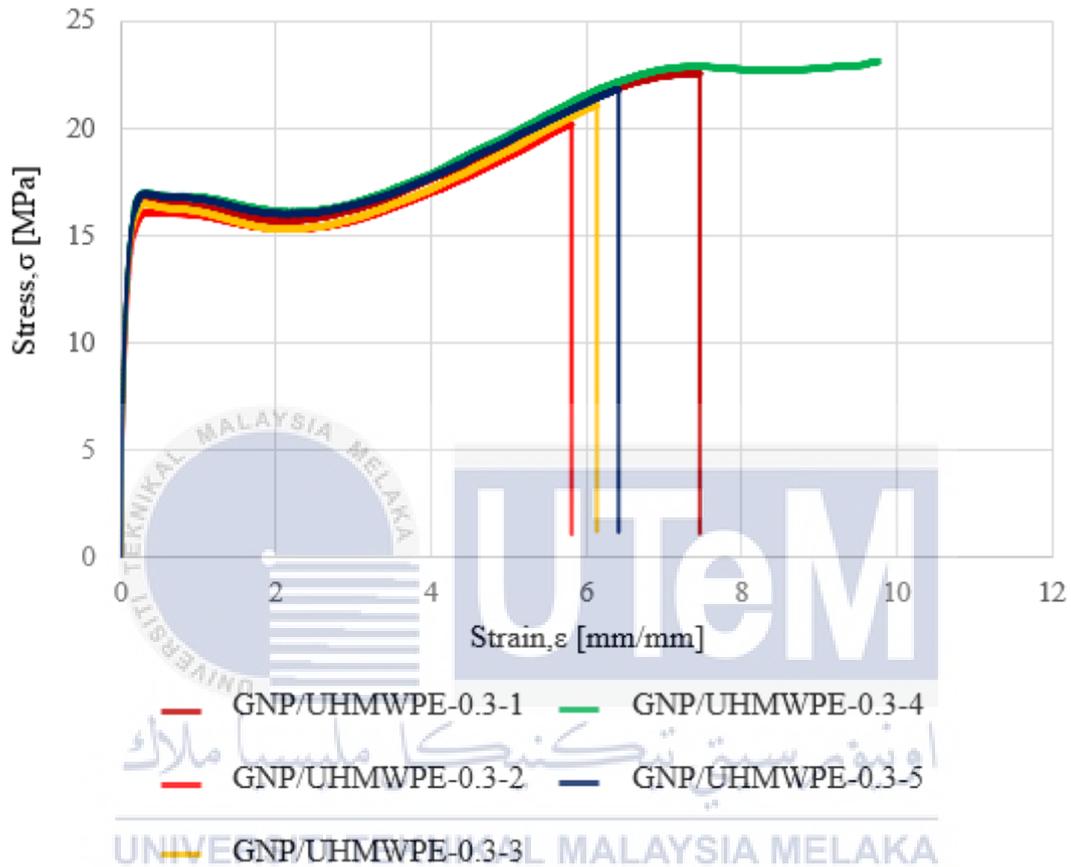


Figure 4.24: Stress-strain curve for GNP/UHMWPE-0.3 composite samples.

As shown in **Figure 4.24**, each sample shows the similar pattern for stress-strain curve even the samples break at different elongation. Moreover, for this type of composite there is 1 sample which is GNP/UHMWPE-0.3-4 that do not break during the test. The sample tends to elongate until reach the maximum allowable extension for the tensile machine used. The **Figure 4.25** shows the GNP/UHMWPE-0.3 composite samples after tensile test.



Figure 4.25: GNP/UHMWPE-0.3 composite samples after tensile test.

The result for tensile test on GNP/UHMWPE-0.3 were summarized in **Table 4.12**. Based on that, the GNP/UHMWPE-0.3-4 shows the highest value of tensile strength and tensile strain which are 23.10 MPa and 9.76 mm/mm, respectively. However, the lowest tensile strength and tensile strain were shown by GNP/UHMWPE-0.3-2 which are 18.95 MPa and 5.81 mm/mm, respectively. This pattern is similar for the Young's modulus where the highest (0.343 GPa) and the lowest (0.311 GPa) represented by GNP/UHMWPE-0.3-4 and GNP/UHMWPE-0.3-2, respectively.

Table 4.12: Tensile properties of GNP/UHMWPE-0.3 composite samples.

| Sample | Tensile Strength (MPa) | Tensile Strain (mm/mm) | Young's Modulus (GPa) |
|------------------|---------------------------|---------------------------|--------------------------|
| GNP/UHMWPE-0.3-1 | 19.38 | 7.46 | 0.338 |
| GNP/UHMWPE-0.3-2 | 18.95 | 5.81 | 0.311 |
| GNP/UHMWPE-0.3-3 | 20.99 | 6.13 | 0.325 |
| GNP/UHMWPE-0.3-4 | 23.10 | 9.76 | 0.343 |
| GNP/UHMWPE-0.3-5 | 21.72 | 6.41 | 0.337 |
| Average | 20.82 | 7.11 | 0.331 |

GNP/UHMWPE-0.5 Composite

The **Figure 4.26** represents the stress-strain curve obtained from the tensile test conducted on GNP/UHMWPE-0.5 composite samples.

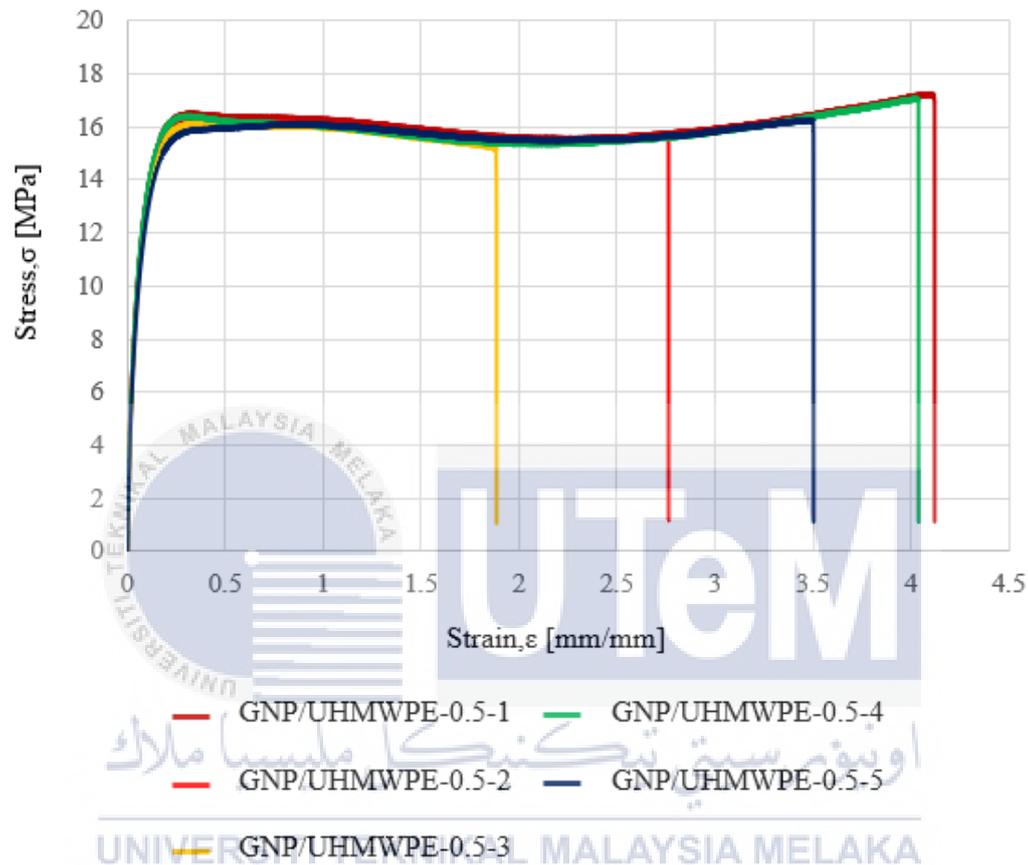


Figure 4.26: Stress-strain curve for GNP/UHMWPE-0.5 composite samples.

For GNP/UHMWPE-0.5, all the samples were break when tested under tensile as shown in **Figure 4.26**. The samples also started to have low elongation at break. The **Figure 4.27** shows the GNP/UHMWPE-0.5 composite samples after tensile test.

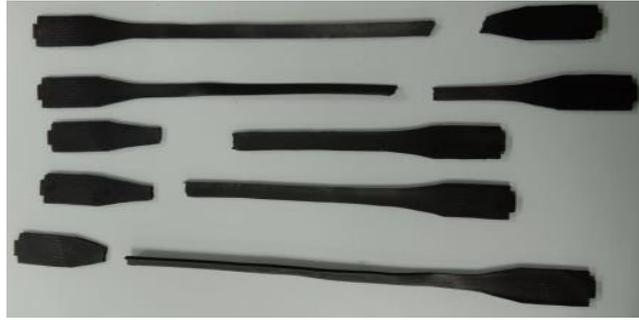


Figure 4.27: GNP/UHMWPE-0.5 composite samples after tensile test.

The **Table 4.13** shows the summary of the tensile test conducted on GNP/UHMWPE-0.5 composite samples. Based on the information, the highest tensile strength and tensile strain among the samples were shown by the GNP/UHMWPE-0.5-1 which are 17.21 MPa and 4.12 mm/mm, respectively. The lowest tensile strength and tensile strain were shown by GNP/UHMWPE-0.5-3 which are 15.91 MPa and 1.89 mm/mm, respectively. For the Young's modulus, the highest value represented by the GNP/UHMWPE-0.5-4 which is 0.342 GPa. Besides, the lowest value of Young's modulus is GNP/UHMWPE-0.5-5 which is 0.295 GPa.

Table 4.13: Tensile properties of GNP/UHMWPE-0.5 composite samples.

| Sample | Tensile Strength (MPa) | Tensile Strain (mm/mm) | Young's Modulus (GPa) |
|------------------|---------------------------|---------------------------|--------------------------|
| GNP/UHMWPE-0.5-1 | 17.21 | 4.12 | 0.333 |
| GNP/UHMWPE-0.5-2 | 16.28 | 2.76 | 0.331 |
| GNP/UHMWPE-0.5-3 | 15.91 | 1.89 | 0.315 |
| GNP/UHMWPE-0.5-4 | 17.04 | 4.04 | 0.342 |
| GNP/UHMWPE-0.5-5 | 16.09 | 3.50 | 0.295 |
| Average | 16.51 | 3.26 | 0.323 |

GNP/UHMWPE-1.0 Composite

The **Figure 4.28** represents the stress-strain curve obtained from the tensile test conducted on GNP/UHMWPE-1.0 composite samples.

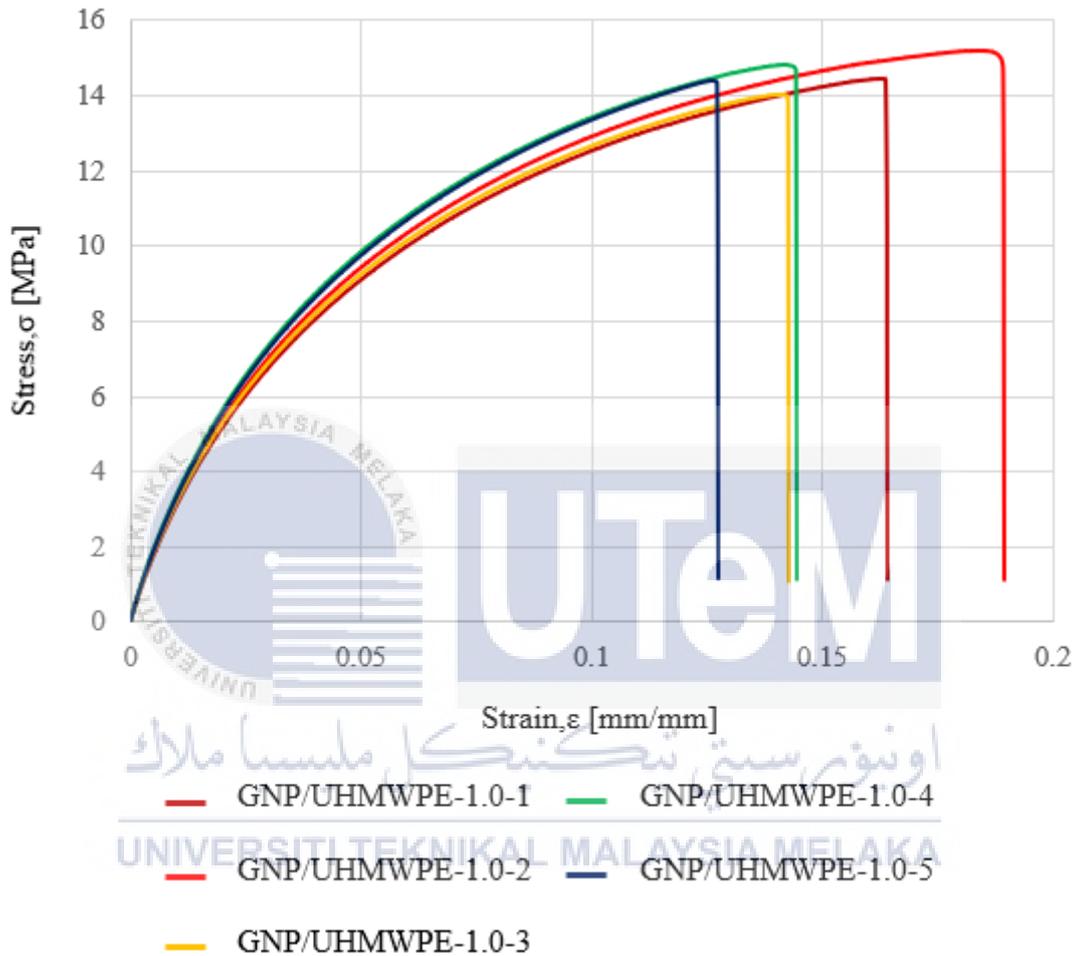


Figure 4.28: Stress-strain curve for GNP/UHMWPE-1.0 composite samples.

By referring to the **Figure 4.28**, the stress-strain curve for the GNP/UHMWPE-1.0 composite samples is different with the other types of composite since the samples start to have brittle properties as the samples have low tensile stress and shorter elongation. The **Figure 4.29** shows the GNP/UHMWPE-1.0 composite samples after tensile test.



Figure 4.29: GNP/UHMWPE-1.0 composite samples after tensile test.

The tensile properties of GNP/UHMWPE-1.0 composite samples were summarized in **Table 4.14**. The highest tensile strength and tensile strain within the samples were shown by GNP/UHMWPE-1.0-2, which are 15.21 MPa and 0.19 mm/mm, respectively. Besides, the lowest tensile strength and tensile strain were shown by the GNP/UHMWPE-1.0-5, which are 14.21 MPa and 0.13 mm/mm, respectively. The highest value for Young's modulus is GNP/UHMWPE-1.0-4 where the value is 0.340 GPa while the lowest Young's modulus is GNP/UHMWPE-1.0-1 which is 0.309 GPa.

Table 4.14: Tensile properties of GNP/UHMWPE-1.0 composite samples.

| Sample | Tensile Strength (MPa) | Tensile Strain (mm/mm) | Young's Modulus (GPa) |
|------------------|---------------------------|---------------------------|--------------------------|
| GNP/UHMWPE-1.0-1 | 14.38 | 0.16 | 0.309 |
| GNP/UHMWPE-1.0-2 | 15.21 | 0.19 | 0.321 |
| GNP/UHMWPE-1.0-3 | 14.27 | 0.14 | 0.328 |
| GNP/UHMWPE-1.0-4 | 14.72 | 0.14 | 0.340 |
| GNP/UHMWPE-1.0-5 | 14.21 | 0.13 | 0.334 |
| Average | 14.56 | 0.15 | 0.326 |

The **Figure 4.30** shows representative curves for each of neat and composite samples. The average of tensile properties of neat and each type of composite samples are shown in **Table 4.15**.

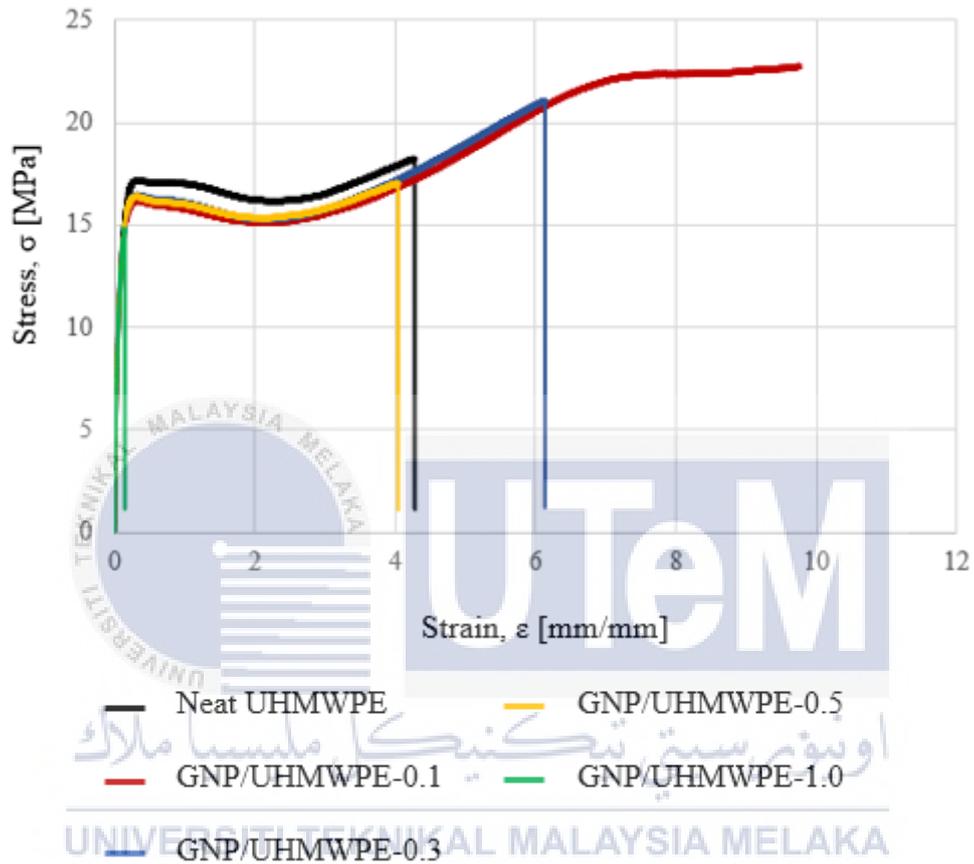


Figure 4.30: Representative curves for each of neat and composite samples.

Table 4.15: Average tensile properties for all samples.

| Sample | Tensile Strength (MPa) | Tensile Strain (mm/mm) | Young's Modulus (GPa) |
|----------------|---------------------------|---------------------------|--------------------------|
| Neat UHMWPE | 19.31 ± 2.646 | 5.86 ± 2.428 | 0.308 ± 0.006 |
| GNP/UHMWPE-0.1 | 22.60 ± 0.212 | 9.65 ± 0.207 | 0.311 ± 0.016 |
| GNP/UHMWPE-0.3 | 20.83 ± 1.703 | 7.11 ± 1.604 | 0.331 ± 0.013 |
| GNP/UHMWPE-0.5 | 16.51 ± 0.583 | 3.26 ± 0.940 | 0.323 ± 0.019 |
| GNP/UHMWPE-1.0 | 14.56 ± 0.414 | 0.15 ± 0.024 | 0.326 ± 0.012 |

Based on the information given, the tensile strength and tensile strain of the samples increased as the addition of GNP increased up to 0.1 wt. %. After that, the tensile strength and tensile strain of the samples experienced decreasing as the GNP content increased from 0.3 wt. % to 1.0 wt. %. The tensile strength of the GNP/UHMWPE-0.1 composite samples is increasing 17.04% from the Neat UHMWPE. This trend also similar for tensile strain where the GNP/UHMWPE-0.1 increased 64.68% from Neat UHMWPE. However, the pattern is slightly different for Young's modulus of the samples where the value is increased as the GNP content increased up to 0.3 wt. %. The addition of 0.1 wt. % of GNP can increase 0.97% of Young's modulus of GNP/UHMWPE-0.1 from Neat UHMWPE. However, 0.3 wt. % of GNP can be said as the optimum amount of fillers to be added as it shows the highest value of Young's modulus (increased 7.47% from Neat UHMWPE). While further added the GNP into UHMWPE matrix, the Young's modulus of the sample tends to decrease but only for 0.5 wt. %. As the GNP reached 1.0 wt. %, the Young's modulus of the composite increased again.

For further discussion, pure polymer like Neat UHMWPE sample can exhibit the maximum extent of elongation compared to others as it has more polymer chains. From the

result obtained, there are samples from Neat UHMWPE were broke when tested. This may be due to the weak bonding of the polymer chains. Although the sample can be considered as solid sample when see by naked eyes, the microstructure of the sample may be different as the sample have 9.3% error between experimental and theoretical density. In addition, the samples were fabricated manually using hot-press machine. The fractured part of Neat UHMWPE sample is assumed to be at the area with more pores as shown in **Figure 4.31**. Thus, the porosity assumed in the density analysis contributed to the such value of tensile stress as presented in the Neat UHMWPE tensile test result.

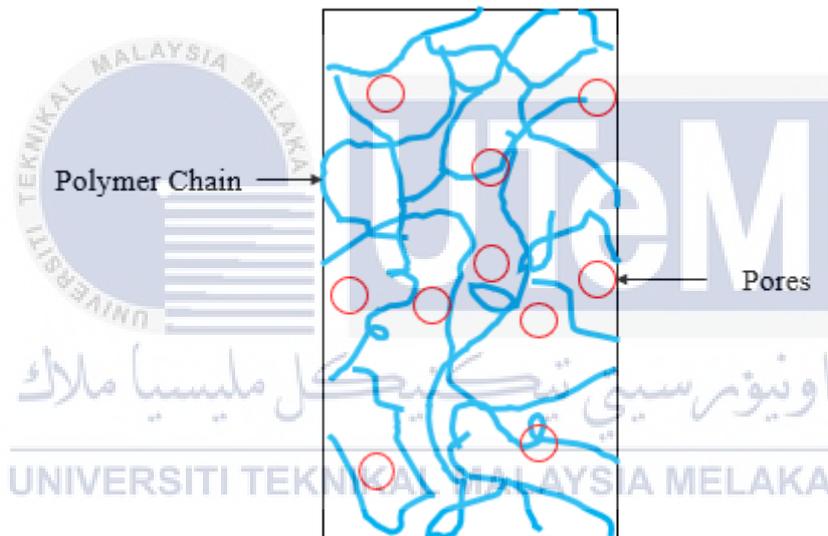


Figure 4.31: Schematic figure for Neat UHMWPE fractured part.

From the tensile test also, it is significant to add GNP as a filler in UHMWPE as GNP can increase the tensile stress of the UHMWPE matrix even with small amount like 0.1 wt. %. This can relate with the strong interaction between filler and matrix as GNP has high surface area with assumption that the interaction contributed to the changes of UHMWPE microstructure when added with stiff filler. Thus, the filler would affect the composite samples to become stronger compared to Neat UHMWPE sample. In addition,

the distribution of GNP in UHMWPE matrix also played an important role as it leads to an improvement in tensile properties of the composite. The 0.1 wt. % of GNP is distributed well within UHMWPE as shown in **Figure 4.32(a)** and even the amount is small, the fillers added is sufficient to hold and receive the stress from matrix. The small amount of filler can enhance the stress transfer from matrix to reinforcing particles. Although the porosity inside the sample is higher when consider the 10.4% error in term of density value as assumed before, the porosity was not giving too much effect on this type of composite sample. This is because the filler added has higher potential to affect the microstructure of the sample. This is proven by the value of tensile stress of this composite has increased 17.04% from Neat UHMWPE sample. For GNP/UHMWPE-0.3, it is assumed to have not much different with GNP/UHMWPE-0.1 in term of microstructure. This is because the different of density error between both samples only 1%. Thus, the **Figure 4.32(b)** represents the schematic figure of GNP/UHMWPE-0.3 composite sample.

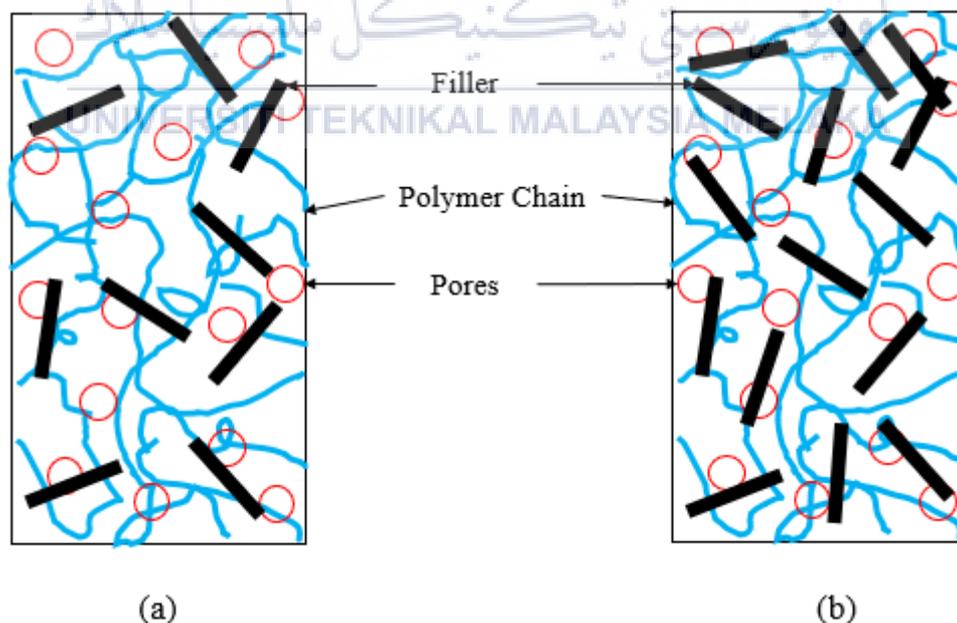


Figure 4.32: Schematic figure for (a) GNP/UHMWPE-0.1 and (b) GNP/UHMWPE-0.3 fractured part.

However, as higher amount of GNP added into UHMWPE matrix, the material tends to show the decreasing in tensile stress. At 0.5 and 1.0 wt. %, the material tends to be brittle. Thus, resulted in small value of tensile stress and tensile strain. This can be assumed that the GNP is tends to agglomerate at certain areas in UHMWPE matrix thus formed the cluster of GNP as the amount is too much even the fillers were dispersed well as shown in **Figure 4.33**. This assumption is met other research by Taromsari et al [19] where the sample of UHMWPE with 10 wt. % of Hydroxyapatite (HAp) and 1 wt. % of GNP namely UHG (1) was observed under SEM to see the cryogenic fracture surface. From the result, it shows that the GNP accumulated at some areas thus resulting in weak interface between the GNP and UHMWPE. The agglomeration of the fillers at certain areas in the samples can lead to weak stress transfer as the reinforcing particles cannot provide the desired mechanical properties. The weak interfaces also contributed to the reduction in Young's modulus at higher fraction of GNP (5 and 10 wt. %) as mentioned by Alam et al [20].

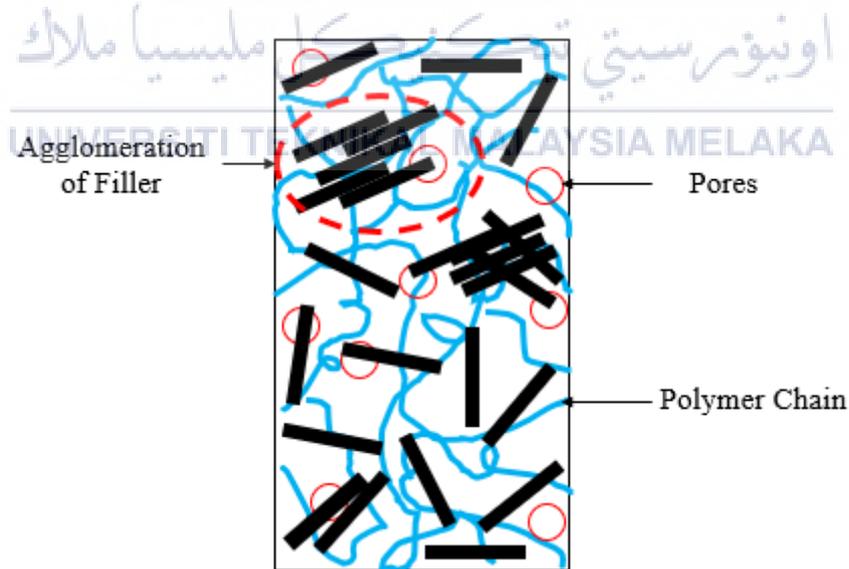


Figure 4.33: Schematic figure for GNP/UHMWPE-0.5 and GNP/UHMWPE-1.0 fractured part.

The analysis between Young's modulus and microhardness of the samples have been considered in order to see the correlation. This is because as mentioned in the microhardness test result, the hardness value of the samples is increased with increasing amount of filler loadings while the tensile test give the result for Young's modulus of the samples have a slightly different as it reduced after reached the optimum value (0.3 wt. %). By referring to the **Table 4.16**, it shows that all the samples have the correlation between the young's modulus and hardness even with small value. This indicate that the young's modulus of the samples is valid.

Table 4.16: Correlation between Young's modulus and hardness for all samples.

| Sample | Correlation |
|----------------|-------------|
| Neat UHMWPE | -0.39 |
| GNP/UHMWPE-0.1 | 6.10E-15 |
| GNP/UHMWPE-0.3 | 0.78 |
| GNP/UHMWPE-0.5 | 0.61 |
| GNP/UHMWPE-1.0 | -0.03 |

CHAPTER 5

CONCLUSION AND RECOMMENDATION

5.1 Conclusion

There are several steps were conducted in order to fulfil the objectives of the study starting from determination of material composition, mixing of material, sample preparation, density analysis, microhardness test, conductivity test and tensile test. The analysis and the tests conducted are to support the tensile test result. In this study, the investigation is focused on the effect of GNP on tensile properties of GNP/UHMWPE composite. The addition of GNP incorporate with UHMWPE matrix is significant as it can enhance the tensile properties of GNP/UHMWPE composite compared to Neat UHMWPE sample as discussed in previous chapter.

Moreover, the effects of GNP composition in GNP/UHMWPE composite also been studied. To compare between the amounts of filler added, 0.1 wt. % can increase the tensile strength and tensile strain while 0.3, 0.5 and 1.0 wt. % show the reduction of the tensile strength and tensile strain. However, in term of Young's modulus, the value is increased up to 0.3 wt. % and decreased for 0.5 wt. % and increased back for 1.0 wt. % of GNP. The effects of GNP composition in composite also seen by other tests. The 0.1 and 0.3 wt. % give no effect on conductivity test while the 0.5 and 1.0 wt. % can conduct electric. For the microhardness test, the amounts of filler added can increase the hardness of the GNP/UHMWPE composite samples. As conclusion, the most significant GNP amount to be added into UHMWPE matrix is 0.3 wt. % and below due to low density which is important

characteristics of composite as to produce lightweight materials, increasing in microhardness value, non-conductive samples which is good to implement in human body and increasing in tensile properties.

5.2 Recommendation

For future work in which may be an appropriate in studying the mechanical properties of UHMWPE composite may consider the following:

- The improvement on mixing of material should be considered as it plays an important aspect for sample preparation.
- The amount of GNP added which is 0.2 wt. % should be considered to see its effect in composite since it is in between the optimum amount (0.1 and 0.3 wt. %).
- The uses of TEM is more suitable to figure out the nanostructure since GNP is used in the study.
- The mechanical testing may be considered the bending and impact test.

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