

CHARACTERIZATION OF NANO CARBON MATERIAL: MORPHOLOGY
AND STRUCTURE ANALYSIS

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SUPERVISOR DECLARATION

“I hereby declare that I have read this thesis 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 (Thermal-Fluids)”

Signature:

Supervisor:

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AND STRUCTURE ANALYSIS**

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**This report is submitted in accordance with requirement for the
Bachelor of Mechanical Engineering (Thermal Fluid)**

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DECLARATION

“I hereby declare that the work in this report is my own except for summaries and quotations which have been duly acknowledge.”

Signature:

Author:

Date:

My love goes to

mother and father and my little brother

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Firstly, a big appreciation goes to Universiti Teknikal Malaysia Melaka (UTeM) especially to Faculty of Mechanical for giving me the opportunity to do my Final Year Project. It was a great opportunity and I gained a lot of great experience when completing this project. I learned how to manage my project and handling all the pressure. I also learned many skills that required from mechanical student especially thermal fluid student should know like doing the experiment, and most important thing is how to communicate and work with other people and show good attitude to supervisor and research assistant.

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Last but not least, I would also like to thank you my family and friends for their moral support and advice.

ABSTRACT

Nano carbon has huge potential in common day. Many scientists have discovered nano carbon and its potential in the various applications that we use every day. This research investigates about the characterization of nano carbon material which we can classify as carbon nano tubes (CNT) and carbon nanofibers (CNF). Three nano carbons which are MER, Nanoamor and Pyrograf were used as samples for this project. The method to investigate the morphology and structure of the nano carbon will be present in these papers which are Scanning Electron Microscope (SEM) and X-Ray Diffraction (XRD). From these two methods, we enabled to get the morphology and the structure of the CNTs and we enabled to analyze and characterized it so that we can identify the properties of this three nano carbon. The SEM test revealed the morphology of the CNT and its properties such as diameter or length of the CNT and the arrangement of the CNT. The graph shows that most the number of diameters of the pyrograf are in range 20-40nm. For MER, the highest number of diameter tubes are in range 80-120nm and the nanoamor are in range 120-160nm. Meanwhile from the XRD test, we revealed the graphite structure of the CNT as the CNT is usually identified as the arrangement of the graphite.

ABSTRAK

Penggunaan nano karbon dalam pelbagai bidang semakin berkembang dalam industri di dunia. Sebilangan saintis telah menemui nano karbon dan potensinya untuk pelbagai aplikasi yang kita gunakan setiap hari. Projek ini bertujuan mengkaji tiga jenis nano karbon dan dalam projek ini, nano karbon yang digunakan adalah tiub nano karbon (CNT) dan karbon nanofiber (CNF). Tiga nano karbon yang digunakan iaitu MER, Nanoamor dan Pyrograf akan digunakan sebagai sampel untuk kajian ini. Kaedah untuk mengkaji morfologi dan struktur daripada nano karbon adalah melalui kaedah SEM dan XRD. Daripada kedua-dua kaedah ini, kita boleh mendapatkan morfologi dan struktur CNT dan kemudiannya akan dianalisis dan disiasat tentang sifat tiga nano karbon dan penggunaannya dalam aplikasi seharian.

Daripada analisis SEM kita dapat mencari morfologi tiub yang terhasil dan sifat-sifatnya seperti diameter atau panjang CNT dan tatacara tiub tersebut. Manakala daripada ujian XRD, kita dapat mengetahui struktur grafit daripada CNT seperti yang di mana CNT adalah terhasil daripada susunan grafit. Setelah itu kita dapat mencirikan tiga nano karbon ini berdasarkan morfologi. Daripada graf yang kita perolehi, purata diameter bagi MER adalah 120-160nm, pyrograf 20-40nm dan nanoamor 80-120nm. Ujian XRD pula bertujuan untuk menganalisis sebatian yang terdapat dalam nano karbon tersebut sehingga membentuk tiub. Akhir sekali, perbandingan akan dibuat dan ciri-ciri nano karbon tersebut dalam dibandingkan dengan aplikasi yang terdapat dalam kehidupan seharian yang berkaitan dengan projek ini.

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LIST OF SYMBOL

| | | |
|-------|---|---|
| CNT | = | Carbon Nanotube |
| SEM | = | Scanning Electron Microscope |
| XRD | = | X-Ray Diffraction |
| DNA | = | Deoxyribonucleic acid |
| RHEED | = | Reflection high-energy electron diffraction |
| MWCNT | = | Multi wall carbon nano tube |
| NASA | = | National Aeronautics and Space Administration |
| NP | = | Nano particle |
| TEM | = | Transmission Electron Microscopy |
| TGA | = | Thermal gravimetric analysis |
| SWCNT | = | Single wall carbon nano tube |
| CVD | = | Chemical vapor deposition |
| PVD | = | Physical vapor deposition |

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CHAPTER I

INTRODUCTION

1.1. PROJECT BACKGROUND

Application using nano carbon is one direction of the modern nanotechnologies. One of the nano carbon materials is carbon nano tubes (CNT). Carbon nanotubes are the arrangement of graphite that form a structure of carbon that has a nano scale size. It is known that nano carbon has very large surface area and because of that it has high thermal conductivity, stability that can be use for many applications. This research will present the method to check about the nano carbon properties.

The objective of this project is to investigate the morphology and structure analysis of the nano carbon material using two methods which are Scanning Electron Microscope (SEM) and X-Ray Diffraction (XRD). While SEM test is for the morphology of the nano carbon, the XRD analysis is for structure of the graphite in the nano carbon. From all this test and variable type of nano carbon, we can investigate its morphology and structure and also compare which is the better nano carbon material based on the analysis.

Previous researches have showed that the nanotubes are 100 times stronger than steel and six times lighter. Nanotubes can be either semi-conductors or insulators, depending on how their carbon sheets are rolled up. Dozens of products containing

carbon nanotubes are commercially available (in order to increase strength without increasing weight) including tennis racquets, bicycle frames and auto body parts. Researchers are hoping that one day nanotubes will replace copper in wiring and silicon in computer chips.

1.2. PROJECT OBJECTIVE

The main objectives for this project are:

1.2.1. To investigate the physical properties of nano carbon materials which are carbon nano tubes (CNT) and carbon nano fiber (CNF).

1.2.2. To characterize the nano carbon using Scanning Electron Microscopes' and X-Ray diffraction.

1.3. PROBLEM STATEMENT

Nano carbon has been proven to have huge potential in many applications. Every nano carbon has its own properties based on its morphology and structure. So, this project is to investigate the nano carbon from its morphology and structure analysis using Scanning Electron Microscope (SEM) and X-Ray Diffraction (XRD) methods. From this research we can characterize it and its potential.

1.4. SCOPE OF PROJECT

This project is mainly focused on morphology and structure analysis for nano carbon material using analytical spectroscopy. The experiment will be conducted using nano carbon which are carbon nano tubes (CNT) and carbon nano fibers (CNF). The method to check the properties are Scanning Electron Microscopy (SEM) and X-Ray

Diffraction (XRD). Experiment will be using three types of nano carbon material from three suppliers that are Material and Electrochemical Research, Inc. (MER), Nanostructures and Amorphous Material, Inc (Nanoamor) and Pyrograf Inc. (Pyrograf).

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CHAPTER II

LITERATURE REVIEW

2.1. Nanotechnology

Nanotechnology can be defined as the science and engineering involved in the design, synthesis, characterization, and application of materials and devices whose smallest functional organization in at least one dimension is on the nanometer scale or one billionth of a meter (1×10^{-9} m) (e.g. a DNA molecule is about 2.5 nm) (V. Shanov et al. 2006). Nanotechnology provides the ability to engineer the properties of materials by controlling their size, and this has driven research toward a multitude of potential uses for nanomaterials. In the biological sciences, many applications for metal nanoparticles are being explored, including biosensors, labels for cells and biomolecular, and cancer therapeutics (A.S.Lobach et al. 2002).

According to A.G Rinzler et al. (1998), nanoscale studies are fascinating because it is on this scale the atoms and molecules interact and assemble into structures that possess unique properties. It is expected that nanotechnology will be developed at several levels: materials, devices and systems. In recent years, nanomaterials have been a core focus of nanoscience and nanotechnology, which is an ever growing multidisciplinary field of study attracting tremendous interest, investment and effort in research and development around the world. Nanomaterials level is the most advanced at present, both in scientific knowledge and in commercial applications.

According to Liu C (1999), nanomaterials can be designed and tailor made at the molecular level to have desired functionalities and properties through taking control over the manipulation of constituent molecules and atoms. Out of plethora of size dependant physical properties available to someone who is interested in the practical side of nanomaterials, optical, magnetic effects are the most used for biological applications.

Some of the important tools available at the moment to assist nanoscale studies are; (a) Highly focused (i.e., 1-2 μm) synchrotron X-ray sources for X-ray diffraction and related techniques that provide detailed molecular structural information by directly probing the atomic arrangement of atoms, (b) Scanning electron and scanning tunneling microscopy that allow three-dimensional type topographical views of nanoscale structures, (c) Monitoring techniques that allow the monitoring and evaluation of building block assembly and growth, such as reflection high-energy electron diffraction (RHEED). (Nuriel S et al 2005) Ultimately, these and other tools are required to probe not only the structure of the materials but also to study the interface between these materials and the cells and tissues they are designed to interact with.

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2.2. Carbon Nano tube

Carbon nano tubes (CNTs) are rolled up cylinders of graphene sheets. They were first observed as multi-walled carbon nanotubes (MWCNTs) by Dr. Iijima in 1991. He discovered microtubules of graphitic carbon with outer diameters of 4–30nm and a length of up to 1 mm. These tubules consisted of two or more seamless graphene cylinders concentrically arranged. The innermost tubes of the tubules had diameters as small as 2.2nm in some of my observations. Electron diffraction analysis showed that the crystal axis of the graphene sheets in some of the tubes had a helical arrangement relative to the tube axis. He called these tubes multi-wall carbon nano tubes (MWNT) because the tube diameters belonged to the nanometer order. The unique properties of these structures promised unimaginable applicability and thus attracted a great deal of interest that continues to this day. For example, super high quality MWNTs have

recently been produced by carbon evaporation using high-frequency plasma. These nano tubes had a purity of over 95% and were uniform in structure with an inside diameter of 0.4 nm, an outside diameter of about 5 nm, a cone angle at the tips of 19 degrees, and a length on the micrometer order.

Their walls are highly graphitized. These super-high-quality MWNTs are thus close to the ultimate in MWNTs. Single-wall carbon nano tube (SWNT), which are seamless cylinders each made of a single graphene sheet were first reported in 1993. Their diameters range from 0.4 to 2–3 nm, and their length is usually of the micrometer order. SWNTs usually come together to form bundles. In a bundle, the SWNTs are hexagonally arranged to form a crystal-like structure. These unique structures lead to unique SWNT properties—mechanical strength greater than that of Fe, density lower than that of Aluminum, and thermal stability at 14001 °C in a vacuum. The SWNTs emit electrons from their tips when exposed to a low electric field, enabling their application in flat-panel displays. Their thin needle-like structure allows them to be used as probe tips in scanning tunneling microscopy and atomic force microscopy. The nanometer-scale spaces inside and among the SWNTs should provide large gas adsorption capacities. Carbon nano tubes (CNT) are structures of nanometric dimension built up entirely by atoms of carbon and they have a ratio of length-to-diameter up to 10000:1 (Diaz E et. Al 2205). The carbon-carbon atomic bond is one of the strongest existing in nature and this leads to outstanding mechanical performances: very high specific strength and elastic modulus, high thermal and electrical conductivity, low density, high superficial area, almost free-defect structure and the opportunity to be functionalized with reactive chemical units that can form bonds with system in which they are dispersed.

Table 2.1: Properties of CNT confronted to steel and aluminum alloy

| | CNT | Steel | Aluminum |
|------------------------------|---------|---------|-----------|
| Density (g/cm ³) | 1,3-2,0 | 7,8 | 2,7 |
| Elastic Modulus (GPa) | 1000 | 200 | 70 |
| Tensile strength (GPa) | 10-60 | 0,3-0,5 | 0,22-0,25 |
| Thermal conductivity (W/mK) | 3000 | 10-100 | 100-250 |

(Source: SAATI Group (SEAL S.P.A.))

According to NASA Small Business Innovation Research Proposal (2003), Carbon nano tubes (CNT), graphene and their compounds exhibit extraordinary electrical properties for organic materials, and have a huge potential in electrical and electronic applications such as photovoltaic, sensors, semiconductor devices, displays, conductors, smart textiles and energy conversion devices (e.g., fuel cells, harvesters and batteries). It also has particularly attracted the attentions of many scientists due to its advantageous properties such as high Young's modulus, effective capability for the storage of a large amount of hydrogen. This updated report brings all of this together, covering the latest work from 100 organizations around the world to details of the latest progress applying the technologies.

CNT also are an important new class of technological materials that have numerous novel and useful properties. The forecast increase in manufacture makes it likely that increasing human exposure will occur, and as a result, CNT are beginning to come under toxicological scrutiny. This review seeks to set out the toxicological paradigms applicable to the toxicity of inhaled CNT, building on the toxicological database on nanoparticles (NP) and fibers. Relevant workplace regulation regarding exposure is also considered in the light of our knowledge of CNT. CNT could have features of both NP and conventional fibers, and so the current paradigm for fiber toxicology, which is based on mineral fibers and synthetic vitreous fibers, had been discussed (Deborah Berhanu et al. 2009). The available peer-reviewed literature suggests that CNT may have unusual toxicity properties. In particular, CNT seem to

have a special ability to stimulate mesenchymal cell growth and to cause granuloma formation and fibro genesis. (Peter Lackner et al. 2006) In several studies, CNT have more adverse effects than the same mass of NP carbon and quartz, the latter a commonly used benchmark of particle toxicity. There is, however, no definitive inhalation study available that would avoid the potential for artifactual effects due to large mats and aggregates forming during instillation exposure procedures. Researcher also showed that CNT may exhibit some of their effects through oxidative stress and inflammation. CNT represent a group of particles that are growing in production and use, and therefore, research into their toxicology and safe use is warranted. (Hadi N Yehia et al. 2007)

Applications of carbon nano tubes and graphene for electronics applications depending on their chemical structure, carbon nanotubes (CNTs) can be used as an alternative to organic or inorganic semiconductors as well as conductors, but the cost is currently the greatest restraint. However, that has the ability to rapidly fall as new, cheaper mass production processes are established. In electronics, other than electromagnetic shielding, one of the first large applications for CNTs will be conductors. In addition to their high conductance, they can be transparent, flexible and even stretchable. Today, applications are for displays, touch screens, photovoltaic and display bus bars and beyond (Wolf E.L, 2004).

In addition, interest is high as CNTs have demonstrated mobility which is magnitudes higher than silicon, meaning that fast switching transistors can be fabricated. In addition, CNTs can be solution processed, i.e. printed. In other words, CNTs will be able to provide high performing devices which can ultimately be made in low cost manufacturing processes such as printing, over large areas. They have application to super capacitors, which bridge the gap between batteries and capacitors, leveraging the energy density of batteries with the power density of capacitors and transistors (Isao Mochida et al. 2006).

Challenges are material purity, device fabrication, and the need for other device materials such as suitable dielectrics. However, the opportunity is large, given the high

performance, flexibility, transparency and printability. According to Bonnemann H, Richards (2001) graphene, a cheap organic material, is being enhanced by companies that are increasing its conductivity, to be used in some applications as a significantly cheaper printed conductor compared to silver ink. Graphene and its compounds are increasingly used to make transistors that show extremely good performance a progress that comes with new cheaper production processes for the raw material.

According to Deborah Berhanu (2009), electron microscopy provides detailed information at the smallest scales but larger scale investigation can provide complementary information and is as important. In this case, the use of optical microscopy was essential to identify graphite visually and therefore give support to the XRD data. The ability to acquire a large-scale view of the sample by zooming out can be very helpful even when the material for characterization is nanostructure. Optical microscopy imaging can be described as equivalent to TEM imaging, but at lower resolution. There, the MWCNTs are almost transparent to the light beam and the graphitic microstructures in the sample become obvious.

There are three principle techniques to produce high quality of SWCNT, laser ablation, electric arc discharge, and Chemical Vapor Deposition (CVD). Laser ablation and arc discharge are modified Physical Vapor Deposition (PVD) technique and involved the condensation of hot gaseous carbon atom generated from the evaporation of solid carbon (Evan Analytical Group LLC, 2007). The advantages of the electric arc-discharge technique lie in the fact that very uniform and almost defect-free nanotubes can be formed during the growing process.

The major drawback of this technique is that it is not efficient for large scale production. Both electric arc-discharge and laser ablation techniques are well developed to understand the mechanism of nanotube nucleation and growth. Other disadvantages of these two techniques are the high cost and difficulty to obtain individually separated CNTs. For example, high temperature is required for the CNT growth and undesired tangled-shape CNTs are commonly formed. Though arc-discharge and the laser ablation

methods are generally considered not as competitive as the CVD method in the long term for low-cost production, they are methods currently used to prepare CNTs for commercial products (C. M. Trottier et al. 2005). The CVD method is considered to be the most favorable method for mass production. Besides its economic advantage, it is the only method of the three that can produce MWCNTs with open end. CVD technique creates defects, providing lots of C dangling bonds. These dangling bonds are desirable for immobilization of biomolecules.

Commercially available CNT are often synthesized by CVD as this process is easily scaled up for industrial production. The annual global production of single-walled tubes was 9 tons in 2004 (Cientifica). Typically, a major producer of SWCNT produces ~ 450 kg/day. The high-pressure carbon monoxide method, which uses iron as the catalyst and carbon monoxide as the carbon source, is commonly employed. MWCNT are also in commercial production, and one company has been making plastic composites incorporating multiwalled nanotubes since 1983. The annual global production capacity for MWCNT was estimated at 100 tons in 2004 (P. Sarrazin et al. 2004).

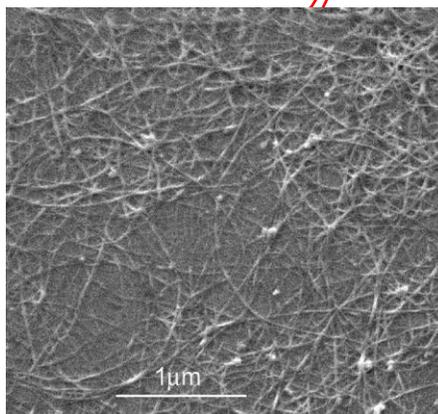


Figure 2.1: Image of carbon nanotubes (CNT) using SEM
(Source: A.S.Lobach et al. (2002))

To observe the morphology of CNT coatings, CNT ink was sprayed onto a 175- μm PET sheet to a sheet resistance of 250 Ω . The open network produced is shown in

Fig.2.0 using a Field Emission Amray 3600 scanning electron microscope (SEM) (A.S.Lobach et al. 2002).

2.3 Applications

Researchers anticipated nano tube applications in several important areas. One use is as field emitters in flat-panel display technologies, an application that will probably become available as products sooner than any other. SWCNT displays could eventually displace liquid-crystal and plasma displays in large flat panels. Their advantages over standard liquid crystal displays include lower power consumption, higher brightness, a wider viewing angle, faster response rate, and a wide operating temperature range. But nano tube displays are technically complex and require concurrent advances in electronic-addressing circuitry, low voltage phosphors, methods to maintain the required vacuum, and the elimination of faulty pixels. CNTs are very strong, thus there is also interest in them for their mechanical properties about 100 times stronger than steel at one-sixth the weight. Thus, SWCNTs may provide reinforcing elements for composite materials that would have exceptional mechanical and, possibly, superior thermal characteristics. Another potential application lies in ultra miniaturized electronics. Companies have active research programs for investigating usage carbon nano tubes for future generations of non silicon microchip circuitry, which could be 0.01% the size of today's most advanced versions, or smaller.

Nano tubes can also be used to make high-performance fibers with double the energy absorption and increased tensile strength, and for efficient, flexible, low-cost sensors for gas-leak detection, medical monitoring, and industrial process control. The new X-ray machine that does not require high temperature to generate the high energy electrons needed to produce X-rays has developed. It uses a thin layer of carbon nano tubes operating at room temperature instead of the usual metal filaments heated inside a vacuum chamber. Due to its high operating temperatures easily burn out the metal filaments, the new devices will last longer and because the devices are smaller and can

operate at room temperature, it should be possible to develop portable X-ray machines for use in ambulances, airport security, and customs operations. CNTs have valuable optoelectronic applications. The injected electrons and holes are confined in the nano tube structure, and when they meet, they are neutralized. If their net momentum is zero and they have opposite spin, they can recombine and give off the recombination energy in the form of light. The SWCNT light source is a three-terminal device that involves no doping and also allows control of the emission intensity and the position of the emitting spot along the length of the CNT. The diameter of the CNT defines the wavelength of the emitted light, typically in the infrared range. The reverse process of photocurrent generation with a significant yield by photo excitation of a CNT device has also been demonstrated. This single CNT device can function as an electrical switch, a light emitter, or a light detector, depending on the biasing.

The high thermal conductivity of nano tubes may be useful for a number of thermal management applications, such as heat sinking of silicon processors, or to increase the thermal conductivity of plastics in such areas as housing for electric motors. Although many groups have studied nano tube-epoxy composite materials for their mechanical properties, their possible thermal properties have only recently attracted attention.

Biercuk et al. (2002) have measured the thermal conductivity of epoxy resin loaded with SWNTs. Fig. 2.2 shows the enhancement in the thermal conductivity for loadings up to 1% SWNTs, and the enhancement for identical loadings of graphitic carbon fibers. Addition of 1% SWNTs doubles the thermal conductivity of the epoxy, while the same loading of carbon fibers provides only a 40% increase. This initial result is quite promising for the development of composites for thermal management.

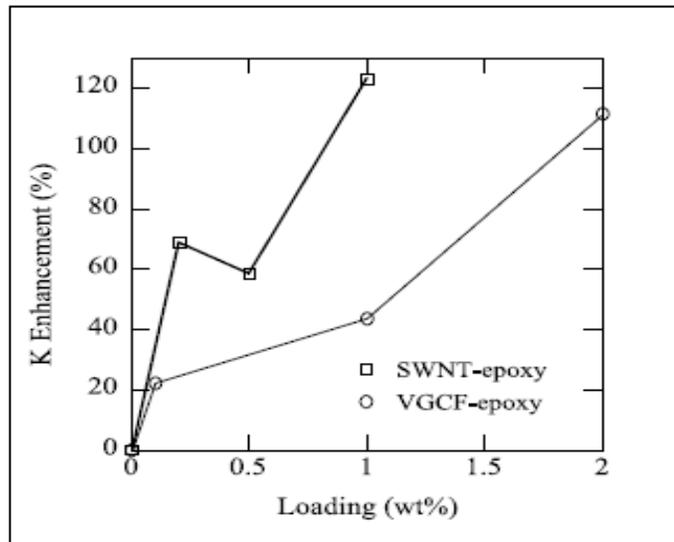


Figure 2.2 Enhancement of the thermal conductivity of epoxy resin as a function of loading by SWNTs and by carbon fibers.

(Source: Biercuk et al 2002)

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CHAPTER III

METHODOLOGY

3.1. FLOW CHART

Methodology is defined as the analysis of the principle of methods, rules and postulates employed by a discipline. It is also defined as a particular procedure or set of procedures that refers to more than a simple of methods. Due to requirement of accurate diameter of bundles, SEM method was used and to investigate about the chemical composition of the nano carbon, the XRD method also used in the research.

Three nano carbons were chose based on diameter which have different in diameter of tubes. Moreover, these nano carbons have different properties as we can classify the MER and Nanoamor are the carbon nanotubes while the pyrograf is the carbon nanofiber.

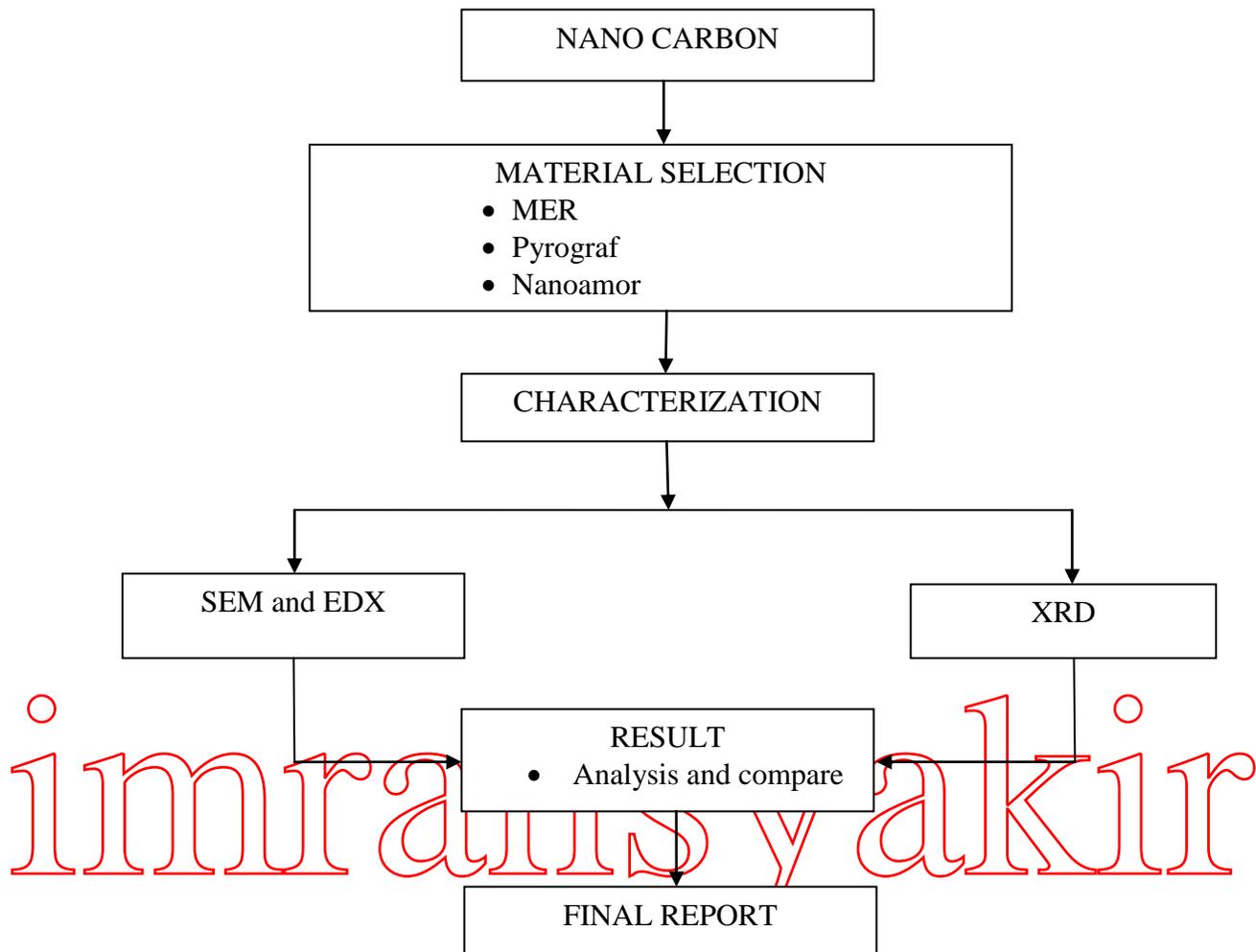


Figure 3.1: Methodology Flow

3.2. EXPERIMENTAL SETUP

The experimental were conducted using Scanning Electron Microscopy (SEM) and X-ray Diffraction (XRD) machine. For SEM test, we will take three images from three different magnifications for each sample using high voltage scanning. After that, we will take the EDX test for the elemental analysis. For XRD test, the sample will be test in the XRD machine and we will get the data after 7-8 hours.

3.2.1. Scanning Electron Microscope

The scanning electron microscope (SEM) is a type of electron microscope that images the sample surface by scanning it with a high-energy beam of electrons in a raster scan pattern. Conventional light microscopes use a series of glass lenses to bend light waves and create a magnified image. The Scanning Electron Microscope creates the magnified images by using electrons instead of light waves. The SEM shows very detailed 3-dimensional images at much higher magnifications than is possible with a light microscope. The images created without light waves are rendered black and white. From the image we will measure the diameter of the CNT from the 50 spot and then we can make a graph distribution from the data. The diameter can be measured from the SEM machine itself. We also can know the length of the CNT whether it is short or long and it doesn't have to measure enough to analyze by human eye only.



Figure 3.2.SEM Machine
(Source JEOL JSM 7500F-1)

3.2.2 Energy Dispersive X-ray Spectroscopy

Energy-dispersive X-ray spectroscopy (EDS or EDX) is an analytical technique used for the elemental analysis or chemical characterization of a sample. It is one of the variants of X-ray fluorescence spectroscopy which relies on the investigation of a sample through interactions between electromagnetic radiation and matter, analyzing X-rays emitted by the matter in response to being hit with charged particles. Its characterization capabilities are due in large part to the fundamental principle that each element has a unique atomic structure allowing X-rays that are characteristic of an element's atomic structure to be identified uniquely from one another.

3.2.3. X-Ray Diffraction

These techniques are based on observing the scattered intensity of an X-ray beam hitting a sample as a function of incident and scattered angle, polarization, and wavelength or energy. XRD is a technique used to characterize the crystallographic structure, crystallite size (grain size), and preferred orientation in polycrystalline or powdered solid samples. From the graph pattern we can identify the structure of the graphite of the CNT.



Figure 3.3.XRD Machine

(Source Perkin Elmer Diamond TG/DTA)

CHAPTER IV

RESULT AND DISCUSSION

4.1. RESULT

4.1.1. SEM images

The images from the SEM test show the different type of carbon nanotubes from the three samples. Figure 4.1 show the morphology of the nano carbon from three different samples. MER CNT (Figure 4.1a) in 20,000 magnification shows agglomerate nanocarbon with average diameter from 70-150nm. It shows mainly tubular fibers structure with polygonal cross section. Figure (4.1b) show image tubular fibers structure and straight graphene sheet of nanoamor CNT with average diameter 100-200nm in 120,000 magnifications. Pyrograf CNT Figure (4.1c) in 60,000 magnification shows long tubes and cylindrical graphite with smallest average diameter 20-40nm.

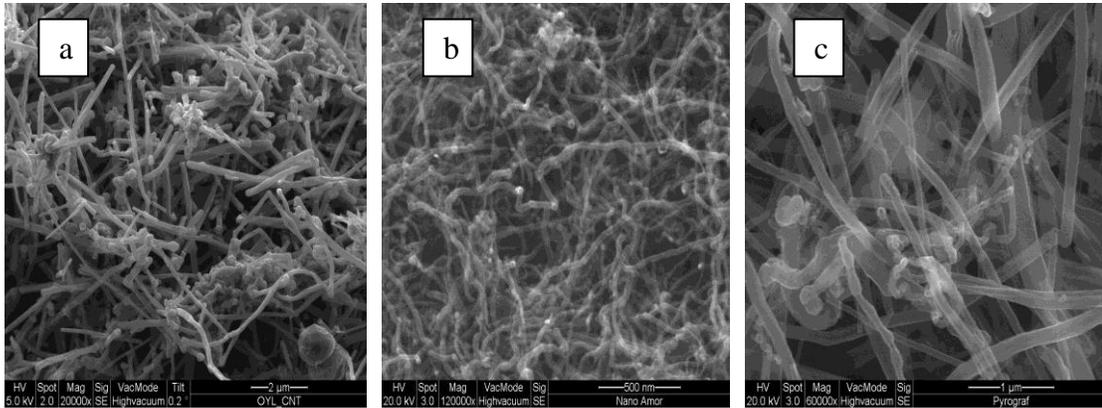


Figure 4.1: SEM images for MER CNT (a), Nanoamor CNT (b), and Pyrograf CNT(c)

4.1.2. RESULT

4.1.2.1. DATA

The graph below show the average diameter of the nanocarbon where it shows that the pyrograf have the smallest average diameter than the other two nanocarbon. This is because pyrograf is a nano fiber where it have small inner diameter unlike MER and nanoamor which is nanotubes. The graph also shows that most the number of diameter of the pyrograf are in range 20-40nm. For MER, the highest number of diameter tubes are in range 80-120nm and the nanoamor are in range 120-160nm. Comparing with this three nano carbon, pyrograf was proven to improve the efficiency based on its diameter and it was showed in the thermal conductivity test that pyrograf improve the efficiency of the test 30% than the other two nano carbon. These improve because of the small diameter which result high surface area. The other nano carbon also was proven to increase the efficiency. These three nano carbon possibly can be use to upgrade the efficiency in heat transfer applications.

Table 4.1: Diameter of Nanocarbon

| Diameter (nm) | Distribution | | |
|---------------|--------------|----------|----------|
| | MER | Pyrograf | Nanoamor |
| 0-20 | - | 9 | - |
| 20-40 | - | 36 | - |
| 40-80 | 7 | 5 | - |
| 80-120 | 35 | - | 7 |
| 120-160 | 8 | - | 29 |
| 160-200 | - | - | 14 |

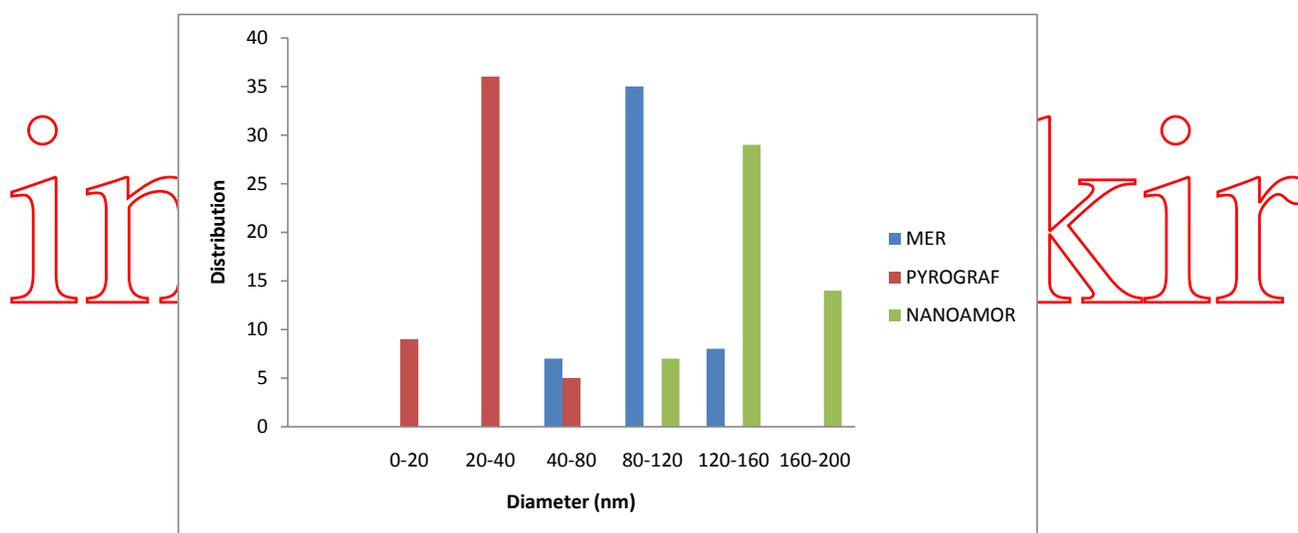


Figure 4.2: Graph distribution of diameter of nano carbon

4.1.3. EDX ANALYSIS

4.1.3.1. MER

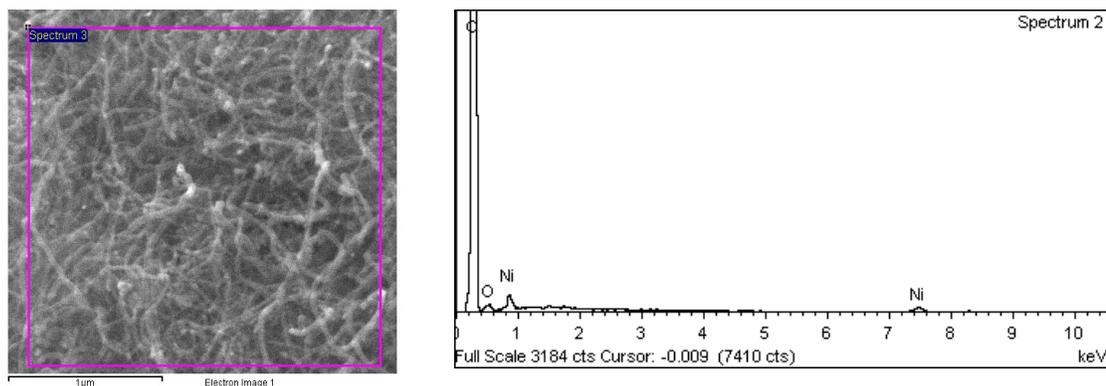


Figure 4.3 SEM image and EDX graph for MER

Table 4.3 EDX data for MER

| Element | Weight % | Atomic % |
|---------|----------|----------|
| C | 95.51 | 97.38 |
| O | 3.03 | 2.32 |
| Ni | 1.46 | 0.3 |
| Total | 100 | |

The EDX data for nanoamor show that most of the element consists of pure carbon where carbon weight percentage is 95.51% compared to oxygen 3.03% and nickel 1.46%. Atomic percentage for the nanoamor is carbon 97.38%, oxygen 2.32% and nickel 0.3%.

4.1.3.2. Nanoamor

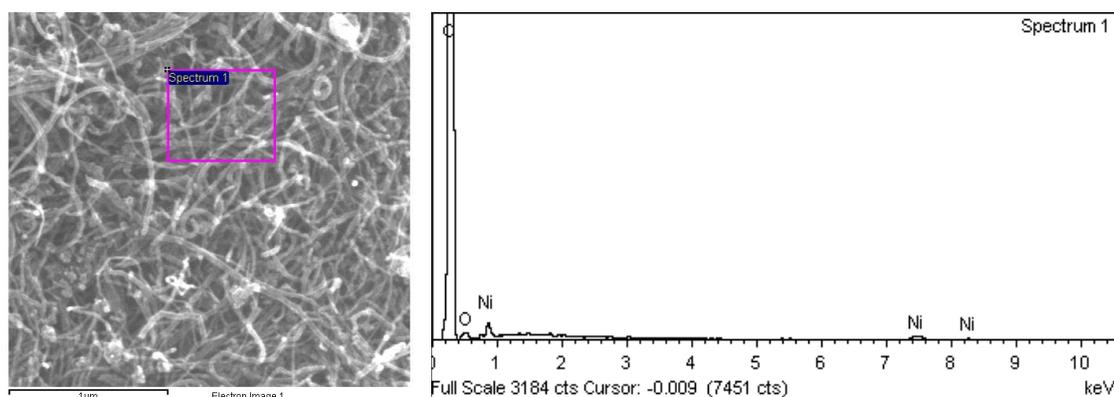


Figure 4.4: SEM image and EDX graph for Nanoamor

Table 4.4: EDX data for Nanoamor

| Element | Weight % | Atomic % |
|---------|----------|----------|
| C | 96.10 | 97.82 |
| O | 2.45 | 1.88 |
| Ni | 1.45 | 0.3 |
| Total | 100 | |

The EDX data for nanoamor show that most of the element consists of pure carbon where carbon weight percentage is 96.10% compared to oxygen 2.45% and nickel 1.45%. Atomic percentage for the nanoamor is carbon 97.82%, oxygen 1.88% and nickel 0.3%.

4.1.3.3. Pyrograf

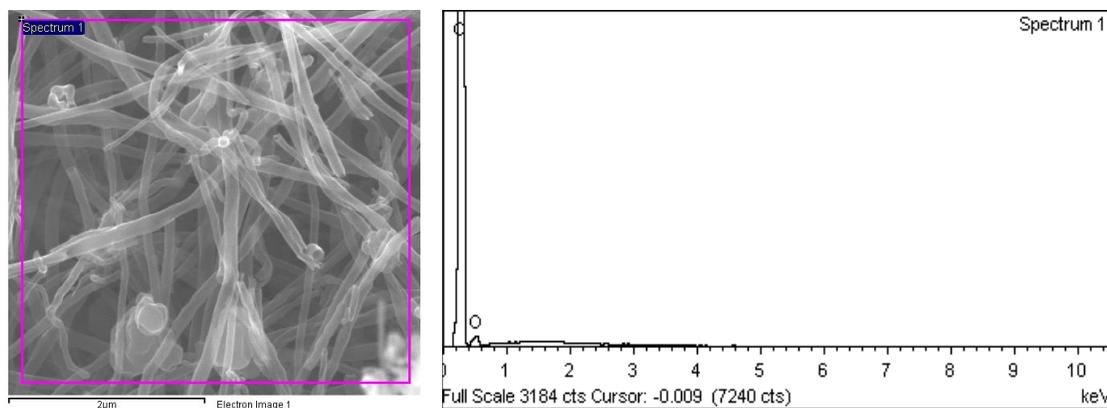


Figure 4.5: SEM image and EDX graph for Pyrograf

Table 4.5: EDX data for Pyrograf

| Element | Weight % | Atomic % |
|---------|----------|----------|
| C | 96.72 | 97.52 |
| O | 3.28 | 2.48 |
| Total | 100 | |

The EDX data for pyrograf show that most of the element consists of pure carbon where carbon weight percentage is 96.72% compared to oxygen 3.28%. Atomic percentage for the pyrograf is carbon 97.52%, oxygen 2.48%.

4.1.4. XRD Result

In this method, the Bragg's law is used to make this XRD graph. Bragg's law refers to the simple equation:

$$n\lambda = 2d \sin\theta$$

This equation derived to explain why the cleavage faces of nano carbon appear to reflect x-ray beam at certain angles of incident (θ , λ).

4.1.4.1. MER

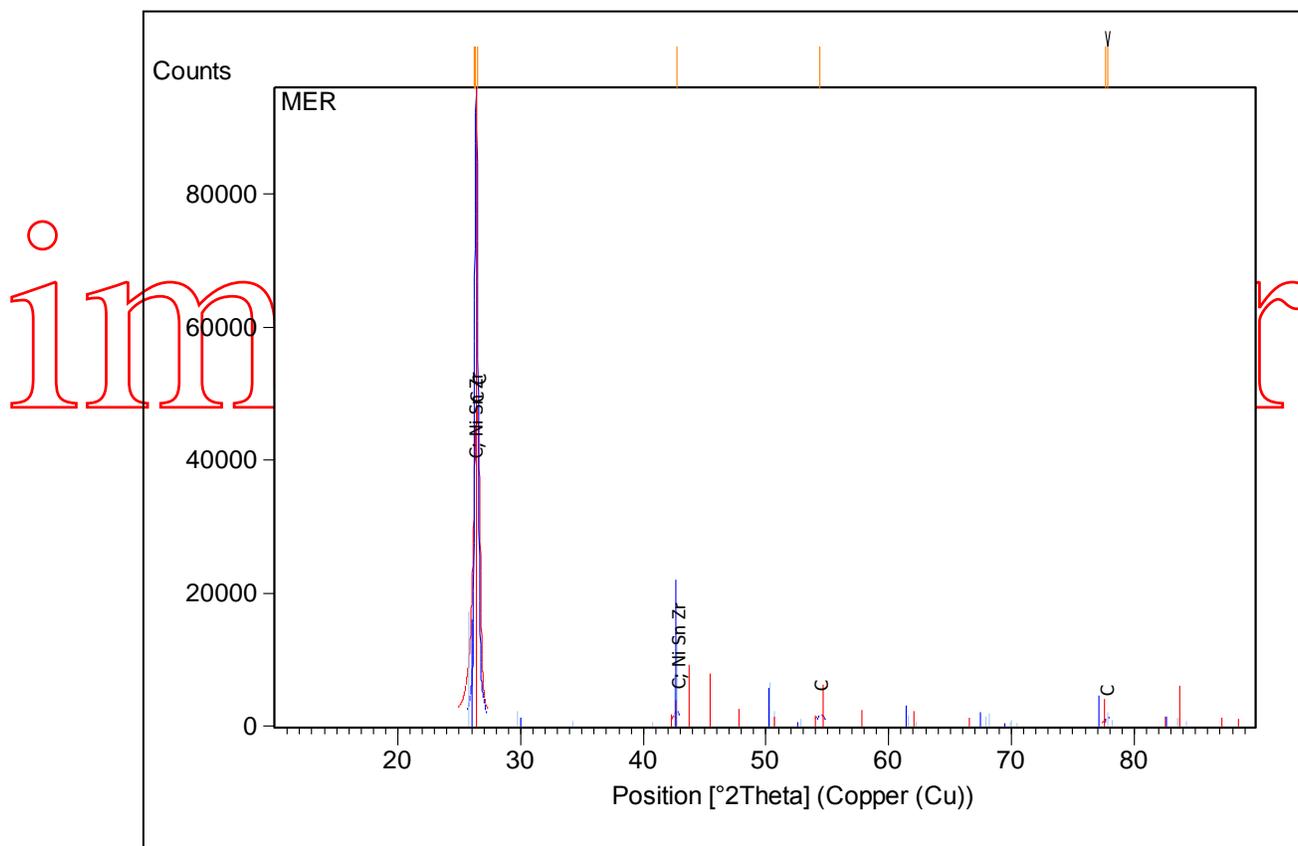


Figure 4.6.XRD graph for MER

Figure 4.6 show the graph for MER, the high intensity of carbon showed with the score is 49 compare with other compound and scale factor 0.986. This was proven that almost all of the nano carbon graphite that growth is carbon based.

4.1.4.2. Nanoarmor

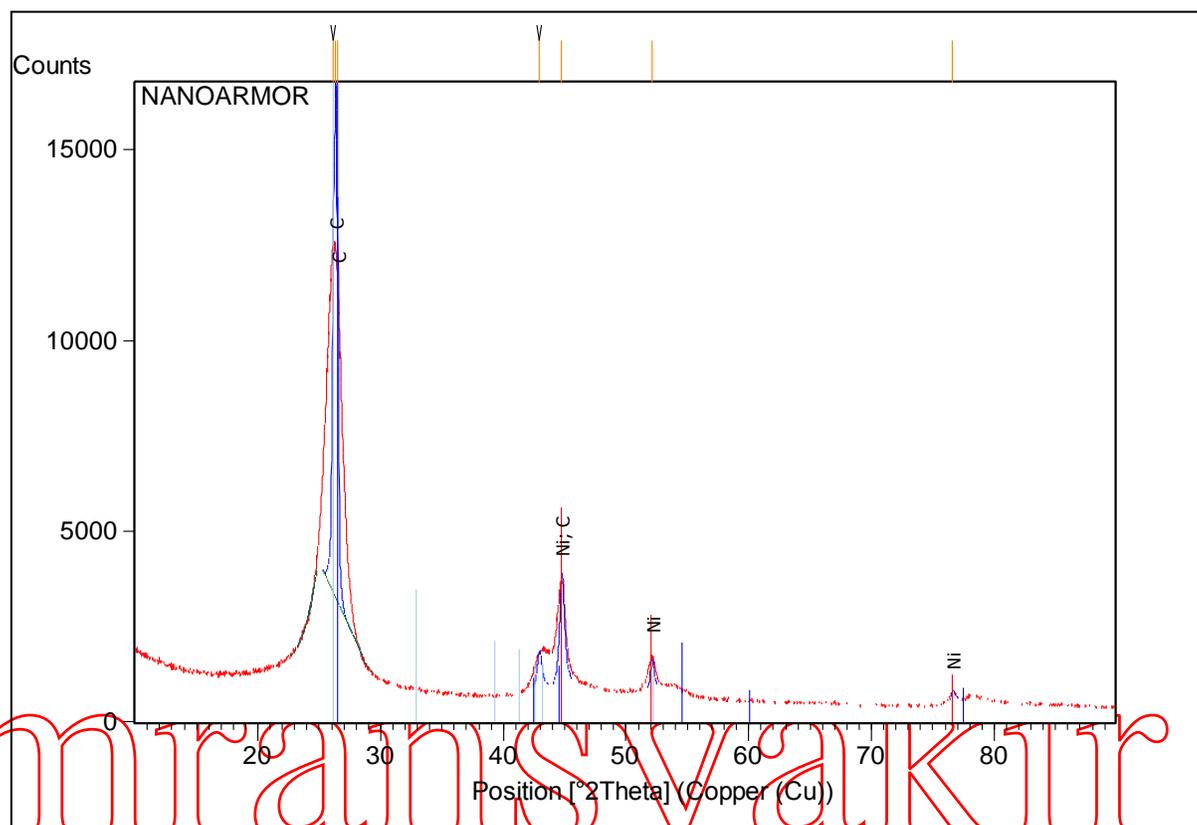


Figure 4.7 XRD graph for Nanoarmor

Figure 4.7 show the graph for nanoarmor. From the graph, the high intensity of nickel showed with the score is 78 compare with graphite compound but graphite have 0.918 scale factor with 50 score.

4.1.4.3. Pyrograf

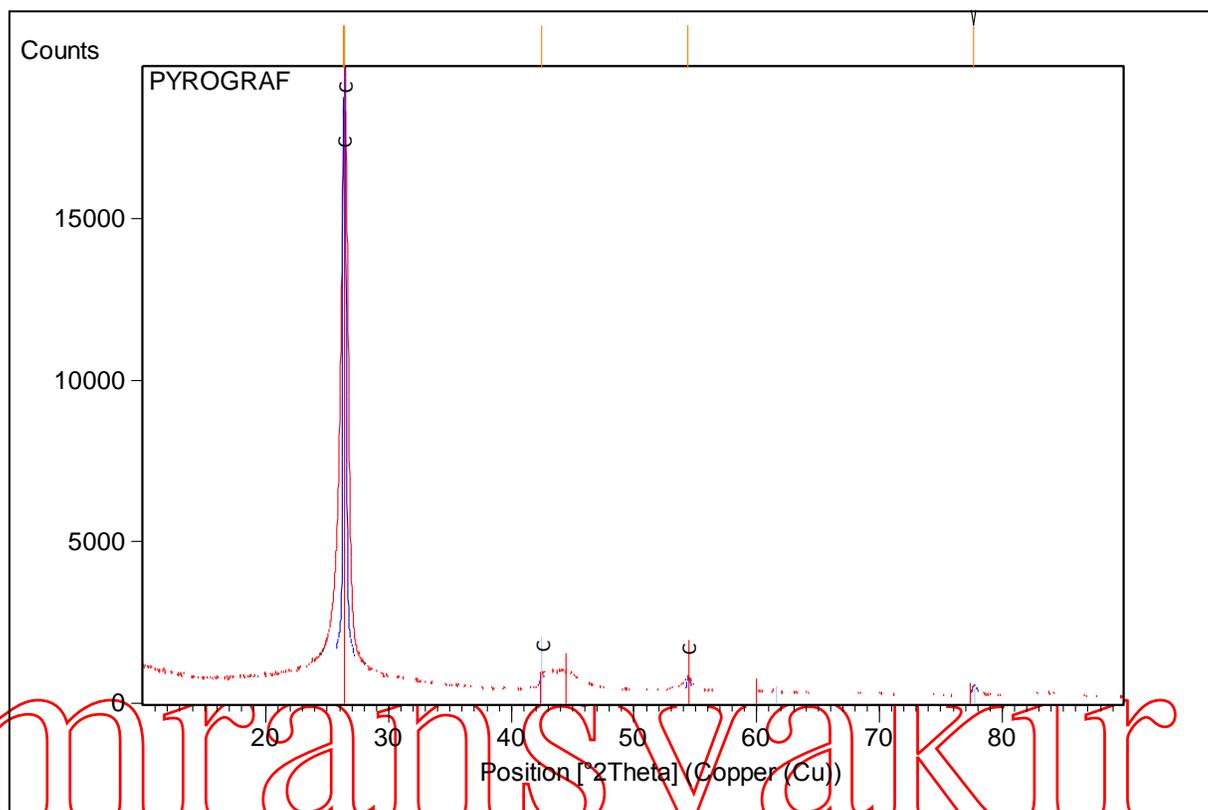


Figure 4.8 XRD graph for pyrograf

Figure 4.8 show the graph for pyrograf. From the graph, the high intensity of carbon showed with the score is 57 compare with almost zero other compound and scale factor 0.986. This was proven that almost all of the nano carbon graphite that growth is carbon based.

4.1.4.4. Overlapping graph

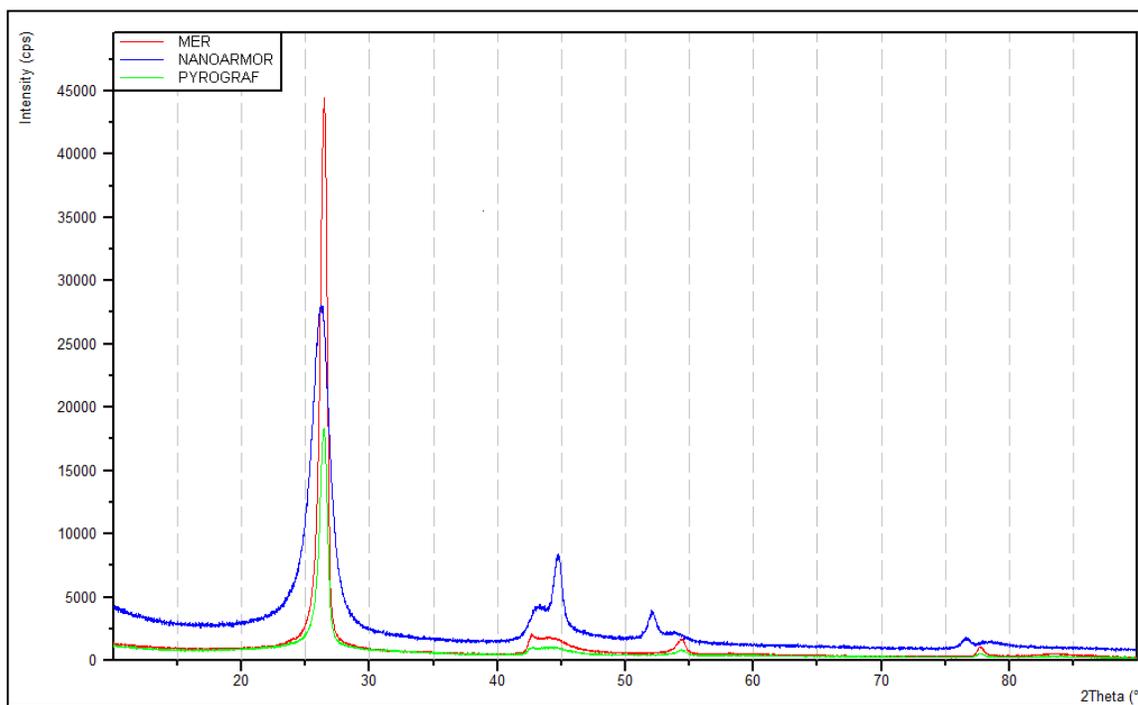


Figure 4.9 Combined graphs for XRD test

After combining those three graphs from the XRD test show that all three nano carbon has high intensity of carbon and also the present of other element like nickel. From the graph and data prove that nano carbon structure based on carbon compound and because of its high intensity, it can easily react with other elements.

4.2. DISCUSSION

We have using the commercial nano carbon or we called it as loose nano carbon. We called it 'loose' because this nano carbon did not support by any supporter such as silica, activated carbon and etc. SEM is one of the main methods to characterize the morphology of the nano carbon (CNT and CNF) and the best way is to take some images from different magnification. We also have to take the reading for the EDX analysis or elemental analysis.

From the images we can find the diameter of the CNT whether it is uniform or not by taking the measure from different magnification and from the data we can create the graph of the CNT's diameter distribution. We also can find the length of the CNT whether it is all long or short or mix. We don't have to measure the length but only from our observation from the images. We also can see the growth of the CNT that produced whether it is coil or straight like a bamboo order.

The result from XRD and EDX show the present of carbon with high intensity which we know that carbon is the element that can react with other element and also many of the applications today involve in using carbon to react with other element.

Comparing these three nano carbon prove that the pyrograf is better than the other two nano carbon but the morphology and the structure analysis prove that all nano carbon have improve other application. The small diameter with high intensity of carbon

based prove that nano carbon is a super compound that many researchers found it can improve many applications especially that related to carbon compound.

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CHAPTER V

CONCLUSION AND RECOMMENDATION

5.1. CONCLUSION AND RECOMMENDATION

Three nanocarbon that we characterize to find its morphology and structure prove that each of its have very small in diameter which result of high surface area and because it is carbon-based which is element that can react with any element, the efficiency of nano carbon to react and increase the efficiency in many applications are high. The diameter of the pyrograf are in range 20-40nm. For MER, the highest number of diameter tubes are in range 80-120nm and the nanoamor are in range 120-160nm. These three nano carbon can be use in many possible applications that use nano carbon such as heat transfer, and electronic applications.

This nano carbon may have other properties than can improve other application which we recommend to analyze and investigate its other properties to show that nano carbon is the new material that applicable in improves other applications this nano carbon also need more research on the devise on how to mass produce these nano carbon that can control the diameter and length and other properties.

Its special behavior that arise from single or multi walled grapheme sheet regarding its unique nanometer scale can improve many application possibilities than attract many researchers.

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