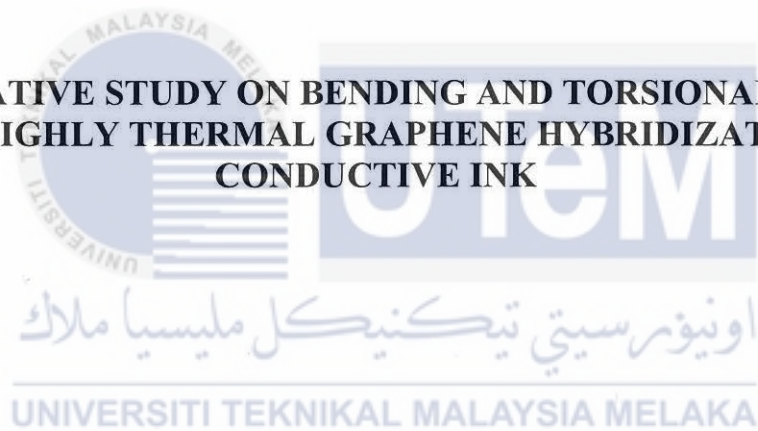




**Faculty of Mechanical Engineering**

**COMPARATIVE STUDY ON BENDING AND TORSIONAL EFFECT  
OF HIGHLY THERMAL GRAPHENE HYBRIDIZATION  
CONDUCTIVE INK**



**Ismaniza binti Ismail**

**Master in Mechanical Engineering (Energy)**

**2023**

**COMPARATIVE STUDY ON BENDING AND TORSIONAL EFFECT OF  
HIGHLY THERMAL GRAPHENE HYBRIDIZATION CONDUCTIVE INK**

**ISMANIZA BINTI ISMAIL**

**A thesis submitted  
in fulfilment of the requirements for the degree of Master in Mechanical Engineering  
(Energy)**



**UNIVERSITI TEKNIKAL MALAYSIA MELAKA**

**2023**

## DECLARATION

I declare that this thesis entitled “Comparative Study on Bending and Torsional Effect of Highly Thermal Graphene Hybridization Conductive Ink” is the result of my own research except as cited in the references. The thesis has not been accepted for any degree and is not concurrently submitted in candidature of any other degree.

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## APPROVAL

I hereby declare that I have read this thesis and in my opinion this thesis is sufficient in terms of scope and quality as a partial fulfilment of Master of in Mechanical Engineering (Energy)

Signature : .....  .....

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Date : ..... 1 Jun 2023 .....



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## DEDICATION

To my beloved mother and father and  
most importantly, my beloved husband and children.



## ABSTRACT

Electronic device printing on flexible and rigid substrates is advancing rapidly. Health, advertising, vehicles, transportation, energy, and electronics are among the sectors that employ this technology. Printing ink is an important component of printed electronics technology, with conductive inks being the most commonly used material. In recent years, researchers have investigated the combination of metal nanoparticles with graphene nanosheets. Hybrid conductive graphene inks for electronic applications have increased due to the addition of conductive polymers or metallic nanoparticles to improve graphene's original capabilities. Silver nanoparticle (AgNP) conductive ink dominates all metal-particle-based conductive inks for printed electronics. The objective of this study is to first formulate a new hybridization formulation between GNP and Ag. Besides, this new formulation of conductive ink has been investigated and characterised in terms of electrical and mechanical behaviour. The third was to evaluate the resistance and resistivity of the new conductive ink formulation using bending and torsion tests. Following that, research was carried out on the formulation and performance of GNP hybrids using GNP, silver flakes (Ag), and silver acetate (SA) as conductive fillers mixed with organic solvents to produce powder and paste. Then, the GNP hybrid paste was printed on a copper substrate using a mesh stencil method. The curing process involved in this experiment was 250°C per hour. The resistivity was evaluated at room temperature before the bending and torsional tests. After the test, the GNP hybrid formulation's reliability was evaluated in terms of electrical and mechanical resistivity. The resistivity value before performing the mechanical test was acceptable due to the lowest resistivity value in the range of  $0.963 \times 10^{-5}$  to  $1.293 \times 10^{-5} \Omega.m$  at room temperature. The finding exposed that the resistivity values for each of the three samples of bending and torsional tests significantly changed after 1000 cycles. However, the resistivity value of the torsional test at 5000 cycles was increased due to the damage impact of increasing the number of torsion cycles on the sample. Overall, the results revealed that this hybrid conductive ink has good resistivity and performs with acceptable reliability and durability. In future work, it is recommended that the conductive ink be printed on a more flexible substrate than copper, and the evaluation of temperature dependence can also be made more comprehensively.

# **KAJIAN PERBANDINGAN KE ATAS KESAN LENTURAN DAN KILASAN DAKWAT KONDUKTIF HIBRIDISASI GRAFEN TERMA TINGGI**

## **ABSTRAK**

*Percetakan peranti elektronik pada substrat yang fleksibel dan tegar sedang berkembang pesat. Kesihatan, pengiklanan, kenderaan, pengangkutan, tenaga dan elektronik menggunakan teknologi ini. Dakwat percetakan ialah komponen penting dalam teknologi elektronik bercetak, dengan dakwat konduktif adalah yang paling biasa digunakan. Dalam beberapa tahun kebelakangan ini, penyelidik telah menyiasat gabungan nanozarah logam dengan nanosheet graphene. Dakwat graphene konduktif hibrid untuk aplikasi elektronik telah meningkat disebabkan penambahan polimer konduktif atau nanopartikel logam untuk meningkatkan keupayaan asal graphene. Dakwat konduktif nanozarah perak (AgNP) mendominasi semua dakwat konduktif berasaskan zarah logam untuk elektronik bercetak. Objektif kajian ini adalah untuk terlebih dahulu merumuskan rumusan hibridisasi baharu antara GNP dan Ag. Selain itu, rumusan baharu dakwat konduktif ini telah disiasat dan dicirikan dari segi kelakuan elektrik dan mekanikal. Yang ketiga ialah menilai rintangan dan kerintangan formulasi dakwat konduktif baharu menggunakan ujian lenturan dan kilasan. Susulan daripada itu, penyelidikan telah dijalankan terhadap perumusan dan prestasi hibrid GNP menggunakan GNP, kepingan perak (Ag), dan perak asetat (SA) sebagai pengisi konduktif yang dicampur dengan pelarut organik untuk menghasilkan serbuk dan pes. Kemudian, pes hibrid GNP dicetak pada substrat kuprum menggunakan kaedah jaringan stensil. Proses pengawetan yang terlibat dalam eksperimen ini ialah 250°C sejam. Kerintangan dinilai pada suhu bilik sebelum ujian lenturan dan kilasan. Selepas ujian, kebolehpercayaan formulasi hibrid GNP dinilai dari segi kerintangan elektrik dan mekanikal. Nilai kerintangan sebelum melakukan ujian mekanikal boleh diterima kerana nilai kerintangan terendah dalam julat  $0.963 \times 10^{-5}$  hingga  $1.293 \times 10^{-5} \Omega.m$  pada suhu bilik. Dapatan ini mendedahkan bahawa nilai kerintangan bagi setiap tiga sampel ujian lentur dan kilasan berubah dengan ketara selepas 1000 kitaran. Walau bagaimanapun, nilai kerintangan ujian kilasan pada 5000 kitaran telah meningkat disebabkan oleh kesan kerosakkan peningkatan bilangan kitaran kilasan pada sampel. Secara keseluruhan, keputusan menunjukkan bahawa dakwat konduktif hibrid ini mempunyai kerintangan yang baik dan berfungsi dengan kebolehpercayaan dan kebolehtahanan yang boleh diterima. Dalam kerja akan datang, adalah disyorkan bahawa dakwat konduktif dicetak pada substrat yang lebih fleksibel daripada kuprum, dan penilaian kebergantungan suhu juga boleh dibuat dengan lebih komprehensif.*

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*In the name of Allah, The Beneficent, The Merciful*

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## LIST OF ABBREVIATIONS

PCB	Printed circuit boards
PE	Printed electronics
GNP	Graphene nanoplatelets
Ag	Silver flake
SA	Silver acetate
PI	Polyimide
SEM	Scanning Electron Microscopy
Au	Aurum (Gold)
AgNP	Silver nanoparticles
AuNP	Aurum nanoparticles
PET	Polyethylene terephthalate
PVA	Polyvinyl alcohol
TPU	Thermoplastic Polyurethane
GO	Graphene oxides
rGO	Reduced graphene oxides
OLED	Organic light-emitting diodes
RFID	Radio-frequency identification
CTE	Coefficient of thermal expansion

## LIST OF SYMBOLS

$\rho$	Volume of resistivity
$R, \Omega$	Resistance
$A$	Cross-sectional area
$\sigma(z)$	Conductivity
$\ell$	Length of the specimen
$w$	Width of conductive ink
$t$	Thickness of conductive ink
$I$	Current source to the sample
$V$	Voltmeter
$^{\circ}\text{C}$	Degree Celsius
$Q$	Amount of heat transferred
$k$	Thermal conductivity
$\Omega/\text{sq}$	Ohm square
$\Omega.\text{m}$	Ohm meter
$\gamma$	Surface tension

# CHAPTER 1

## INTRODUCTION

### 1.1 Background of Research

Printed electronics (PE) is an emerging technology that refers to a method in which printing technology is utilised to manufacture a variety of electronic devices, including electrical circuits, displays, sensors, and Radio-frequency identification (RFID) as in Figure 1.1. It is comprised of organic conducting and semiconducting materials in addition to printed inorganic components. The field of printed electronics encompasses a vast variety of materials and printing techniques (Gregor-Svetec 2018).

Today, the printing electronic devices on flexible and rigid substrate is advancing rapidly. This technology is used in a variety of industries, including health, advertising, automobile, transportation, energy and electronics (Ghadimi et al., 2021). Printed materials become thin, light, and flexible enough to be incorporated into current manufacturing processes as substrates get thinner. The printing ink is one of the key components of printed electronics technology, with conductive inks being the most often utilised. There are numerous types of conductive inks, such as metal-based (silver, gold, and copper) materials, conducting polymers, and other conductive organic materials, such as carbon-based inks (graphene, graphene oxide, and carbon nanotube), which have been utilised for a variety of printed, flexible, and wearable electronic applications (Khan et al., 2020). Companies and researchers continue to develop the most effective conductive inks for printed electronics and research for the ingredients that will make their printed electronics the best on the market.

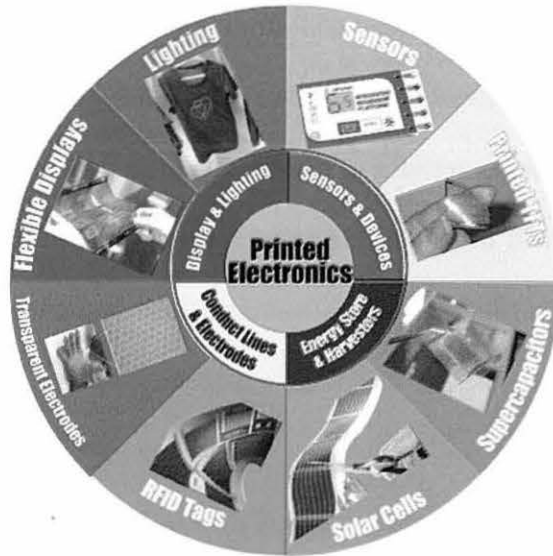


Figure 1.1: Various application of printed electronics technology (Wu, 2017).

In recent years, researchers have investigated the combination of metal nanoparticles with graphene nanosheets. The use of hybrid conductive graphene inks for various electronic applications has increased due to the incorporation of conductive polymers or metallic nanoparticles with the aim of enhancing the original capabilities of graphene. Due to its superior oxidation resistance, electrical conductivity, and other desirable physical qualities that give it exceptional substrate adherence, silver nanoparticles (AgNP) conductive ink is the dominating option for printed electronics among all metal-particle-based conductive inks that have been investigated (Lotya et al., 2009). Therefore, it is a possible alternative to silver for use in conductive inks (Song et al., 2018). The entire advantages of these two materials could be utilised to boost the graphene nanosheets' conductivity and decrease the metal particle concentration (Saidina et al., 2019).

In addition, there has been considerable interest in the use of graphene nanocomposites in high-tech applications. Graphene is used extensively in several electronic applications, notably as conductive inks for printed flexible electronics. Many previous researches concentrated to hybrid graphene and metal based composite materials such as

silver (Ag), copper (Cu) or gold (Au) of conductive inks. The research method of hybridization was developed, thus offering an effective strategy to improve the electrical characteristics and conductivity. Basically, conductive inks must possess excellent electrical, mechanical, and thermal qualities in order to utilize in printed flexible electronics devices. However, current conductive ink that is commercially accessible has a number of drawbacks, including poor printing quality, excessive electrical resistance, and weak mechanical strength.

According to Huang and Zhu (2019), printed electronics play a crucial role in the development of flexible electronics and, more recently, stretchable electronics. Flexible electronic devices are characterised by their portability, deformability, and reconfigurability (Lee et al., 2019; Shi et al., 2019). This means the flexible electronic devices retain their functionality even when bend, folded, compressed or stretched. Traditional electronics are built on hard substrates and stiff materials, while flexible electronics are not. It makes advancements in the morphology, structure, function, and application of electronic devices by constructing them using materials and structures that have physical bending capability and can endure specific deformations (Xiao et al., 2022; Nathan et al., 2012).

This study is concentrated on graphene nanoplatelets (GNP) and silver (Ag) which are employed as conductive fillers in formulation of hybrid conductive inks. In the synthesis of this hybrid conductive ink, the fabrication procedure is emphasized. The efficiency of conductive ink in obtaining better conductivity is determined on the fabrication process. This production method requires the optimal ink composition, printing technique, substrate contact, and curing procedure. Resistivity testing using a multimeter is used to explore the characteristics of conductive ink in order to get a deeper knowledge.

In order to examine the property of conductive ink, this study also focuses on the mechanical flexibility. Particularly in portable applications, the modules are subjected to the

most severe forms of mechanical deformation, including impact, shear, twisting, stretching, and bending (Krebs et al.,2010). For modules to accept these sorts of deformations, the substrates, encapsulants, and active materials must work appropriately to store or discharge mechanical energy without degrading the electrical performance (Fin et al., 2018). The investigation for this study is conducted after repeated cycles with two different modes of deformation, namely the bending and torsion tests. These modes were intended to simulate the sorts of deformations that may occur during the actual functioning of a device. The findings of this research study might serve as an essential resource for the industry, particularly in the sector of electronic interconnection.

## 1.2 Problem Statement

Flexible electronics are now being incorporated into a vast array of consumer and commercial items on the market. These technologies include mobile phone and display components, human and health performance tools, security tags, sensor components and many more. Flexibility is defined differently depending on the circumstances. From bending and rolling for easier handling of wide area photovoltaics to adapting to unconventional forms, folding, twisting, stretching, and deforming for electronic skin devices, all while retaining device function and reliability.

However, the challenges of this field are still a long way from delivering final products due to limitations such as durability, stability, and reliability in terms of device functionalities, as well as manufacturing barriers such as the adaptation of equipment to developed materials and inks. One major challenge is ensuring the durability, stability, and reliability of the devices themselves. This means that the devices need to be able to function properly over an extended period of time and withstand the wear and tear of everyday use. Achieving this requires developing materials that can withstand environmental factors like moisture, temperature changes, and exposure to light, as well as ensuring that the devices

are designed to prevent damage from normal usage. Another challenge is adapting existing manufacturing equipment and processes to accommodate the new materials and inks being used in these devices. This requires significant investment in research and development to create new manufacturing techniques that are compatible with the materials and inks, and may involve the development of entirely new equipment to handle these materials.

Therefore, this study aims to produce highly thermal conductive ink by synthesizing hybrid graphene nanoplatelets with silver and organic solvents such as 1-butanol and terpineol. The investigation is conducted in terms of the resistivity of a newly formulated GNP hybrid at room temperature. The effect of resistivity printed on a copper-type substrate is observed as a correlate to ink reliability in mechanical bending and torsional testing.

### **1.3 Research Objective**

This research focuses on the development of GNP hybrid as conductive ink materials. The objectives of this study are listed below:

- i. To formulate a new hybridization formulation of between Graphene nanoparticle (GNP) and silver (Ag)
- ii. To investigate and characterize the electrical and mechanical behaviour of the new formulation conductive ink
- iii. To evaluate the resistance and resistivity of the new conductive ink formulation using bending and torsion test.

### **1.4 Scope and Limitation**

In this study, the scopes are focused on:

- i. Formulation of conductive ink from graphene nanoplatelet (GNP) and silver (Ag), known as GNP hybrid.

- ii. Fabrication of samples by a printing method of mesh screen stencil on copper substrate.
- iii. Experimental work on GNP hybrid sample is carried out at room temperature.
- iv. Electrical conductivity is measured in term of resistance and resistivity using multimeter.

## **1.5 Outline of Research**

This thesis is structured appropriately, with five chapters in total. Chapter 1: Introduction, Chapter 2: Literature Review, Chapter 3: Methodology, Chapter 4: Results and Discussion, and Chapter 5: Conclusion and Future Works.

In Chapter 1, the introduction to the research is presented in general. The chapter begins with an overview of the background of the study involving the issue of printed electronics, flexible electronics and conductive inks. This chapter also states the problem statement, objectives, and scope and limitations of the study.

Chapter 2 provides a literature review of the research topic. This chapter includes the fundamental of conductive inks, mixing method, printing method, curing process, material of substrate and characterization of conductive ink along with a brief review on previous studies which related to hybrid conductive ink. Chapter 3 describes the methodology of experimental works. This includes the selection and preparation of materials, apparatus of experiment and procedures to conduct the tests.

Chapter 4 presents the results and discussion of the study. The discussion is about research hypothesis and objectives of the study. Assumptions from the discussion and limitations linked to this research were highlighted at the conclusion of this chapter. Finally, Chapter 5 contains a brief summary of the entire work, including methods, results and major conclusion and recommendations for future works.

## CHAPTER 2

### LITERATURE REVIEW

#### 2.1 Introduction

This chapter provides an overview of conductive ink particularly formulated from carbon and metal nanoparticles for flexible and stretchable electronics. Nowadays, graphene conductive inks have been the most applied material in flexible substrate. Environmentally friendly, low-cost, and easy manufacturing processes enable the creation of flexible electronics. Therefore, conductive inks are generally formulated with carbon particles like graphene with metallic particles mainly from silver material as to improve the original properties of graphene. This chapter also discuss on curing and printing process necessarily as complement to produce the applicable graphene hybrid.

#### 2.2 Printed Electronic

Printed electronic technology is developing as a feasible alternative to current lithographic-based manufacturing procedures for electronic device fabrication (Htwe and Mariati, 2022). In recent years, the increasing interest in the field of PEs has led to a rise in the number of efforts to develop and manufacture practical devices. These initiatives would give businesses with effective-cost applications and massive manufacturing capacity (Wu 2017). According to Avuthu et al. (2016), PE has various benefits, including high productivity, decreased resource consumption, and lower production temperature, and is much less difficult. Compared to traditional silicon-based technology, advanced photolithographic patterning methods and high-vacuum, high-temperature deposition procedures are used in silicon-based technology manufacture. Despite being a rapidly expanding area of technology, printed electronics is not without challenges. Companies and

researchers continue to develop the most effective conductive inks for printed electronics and research for the ingredients that will make their printed electronics the best on the market. As stated in Camargo et al. (2021), printed electronic fabrication typically requires three primary components: conductive ink, flexible substrate, and a printing equipment. PE is an interdisciplinary field of study since it combines ink chemistry and printing technology.

Electronic devices in printing technology are necessary for the development of good electrical contact among the different components (Wang 2015). Printing functional inks with dispersed or soluble conductive fillers enables the fabrication of stretchable and flexible electrical devices. The most significant printed electronics applications in flexible and wearable electronic systems include flexible light-emitting electrodes, antennas, photodetectors, solar cells, strain sensors, wearable skin sensors, and supercapacitors (Htwe and Mariati, 2022). Electronics that are both flexible and stretchable must have certain characteristics, including electrical conductivity, high flexibility, stretchability, high durability, biocompatibility, low weight, and environmental friendliness.

Flexible electronic devices with fashionable wearability, flexibility, and compatibility are attracting a great deal of interest for use in touch screens, healthcare monitoring, smart wound dressing, electronic skin, energy harvesting and storage devices, flexible displays, and human-machine interfacing. In addition to electrochemical performance, mechanical flexibility and even stretchability are also important factors to consider for electronic devices to guarantee the reliability of data collecting in applications (Gong et al., 2020). Figure 1.2 depicts the typical flexible electronics and its application.

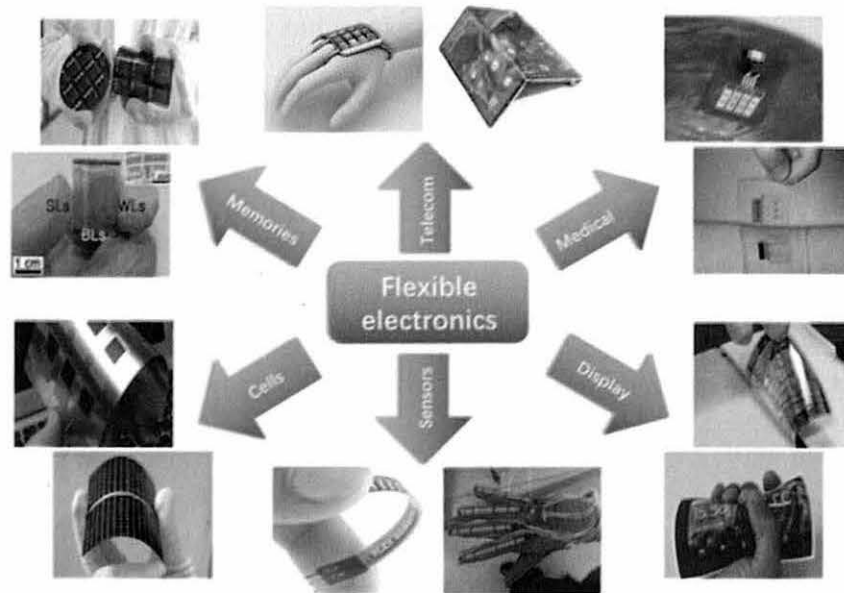


Figure 2.1: Flexible electronics and application (Gao et al., 2020)

The fast growth of electronic devices nowadays requires a more flexible and stretchable conductive matrix. Stretchable conductive ink has a focus on flexibility and expandability while keeping excellent conductivity values. Conductive ink is simple and cost effective thus ideal for flexible electronics, claim Xia et al., (2018).

### 2.3 Conductive Ink

Conductive inks are used to produce electrical conductors on printed items. The use of conductive inks has become a fresh and innovative method to advance electronics manufacturing processes. Printed electronics provide extra desirable qualities including flexibility, lightweight, the potential for downsizing, and sometimes optical transparency in addition to being more affordable and easier to create (Kamarudin et al., 2021). Printed circuits, wearable sensors for remote healthcare monitoring, flexible electrodes, photovoltaic cells, flexible displays, organic light-emitting diodes (OLEDs), thin-film transistors, and smart textiles, among others, have been manufactured using conductive inks due to their remarkable versatility (Li et al., 2020). Conductive ink consists of a conductive filler,