

THE EFFECT OF CARBON NANOTUBE ASPECT RATIO ON THE FUNCTIONAL
PROPERTIES OF ELECTRICALLY CONDUCTIVE ADHESIVES

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UNIVERSITI TEKNIKAL MALAYSIA MELAKA

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**A report submitted
in fulfillment of the requirement for the degree of
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UNIVERSITI TEKNIKAL MALAYSIA MELAKA

2018

DECLARATION

I declare that this project entitled “The Effect of Carbon Nanotube Aspect Ratio On the Functional Properties of Electrically Conductive Adhesives” is the result of my own work except as cited in the references.

Signature :.....

Name :.....

Date :.....

APPROVAL

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

Signature :.....

Name :.....

Date :.....

DEDICATION

This report is dedicated to my beloved parents.

ABSTRACT

Eliminating lead containing solder in electrical industries has driven the need to develop lead-free conducting material termed Electrically Conductive Adhesive (ECA), since the former interconnect material is high in toxic. Thus, ECA is seen as a substitute material with the advantage of being more environmental friendly interconnect material. ECA is made up of polymer as the binder and filler as the conductive material. Metal fillers such as silver, copper and gold are the most commonly used filler. More recently, non-metallic carbon-based material filler such as carbon nanotubes, carbon black and graphene are extensively studied and developed to be used as ECA conductive filler. In addition, ECA with multiwall carbon nanotubes (MWCNT) is developed to improve the strength and electrical conductivity. Current ECA used in industries has low electrical conductivity, poor impact strength and conductivity fatigue since the filler used are metal filler. This project investigates the effect of carbon nanotubes aspect ratio on the functional properties of electrically conductive adhesive. Generally, with higher aspect ratio of MWCNT filler in ECA, the electrical conductivity is better while as the filler loading increase, the shear strength decrease. Electrical and mechanical samples of ECA with different aspect ratio of the MWCNT filler were prepared and tested with reference to ASTM F390-11 and ASTM D1002 respectively. The ECA were prepared by using solution mixing process before curing in an oven at 100°C for 30 minutes. Here, the two aspect ratio of the MWCNT is 1750 while the second MWCNT aspect ratio is 112.5, with the range of MWCNT filler loading is used in this research, which is 5 wt.%, 6 wt.% and 7 wt.%. The specimens were subjected to electrical test using a four-point probe test unit and mechanical testing by using universal testing machine. By formulating ECA using high aspect ratio MWCNT, lower filler loading is needed to reach the percolation threshold which shows better conductivity compared to low aspect ratio MWCNT. As the filler loading increase, the result suggests lower shear strength of the MWCNT-filled ECA. MWCNT aspect ratio has significant effect on the electrical and mechanical properties of ECA which is caused by the agglomeration and dispersion of the MWCNT in the ECA.

ABSTRAK

Usaha menggantikan pateri berplumbum yang tinggi kandungan toksik di industri elektrikal telah membawa kepada pembangunan pateri bebas plumbum yang dinamakan pelekat konduktif elektrik. Kelebihan pelekat konduktif elektrik ini dilihat sebagai gentian yang lebih mesra alam. Pelekat konduktif elektrik ini diperbuat daripada polimer sebagai perekat dan pengisi sebagai bahan konduktif. Logam pengisi yang biasa digunakan adalah perak, tembaga, dan emas. Baru-baru ini, bahan pengisi seperti karbon nanotube, karbon hitam dan graphene telah dikaji dan dibangunkan secara meluas untuk digunakan sebagai pengisi konduktif didalam pelekat konduktif elektrik. Di samping itu, pelekat konduktif elektrik dengan karbon nanotube berlapis dibangunkan bagi meningkatkan kekuatan dan kekonduksian elektrik. Pelekat konduktif elektrik semasa yang digunakan dalam industri mempunyai kekonduksian elektrik yang rendah, kekuatan impak yang lemah dan kegagalan kekonduksian kerana pengisi yang digunakan adalah bersifat logam. Projek ini menyiasat kesan nisbah aspek karbon nanotube ke atas sifat kefungsiian pelekat konduktif elektrik. Pada umumnya, dengan nisbah aspek karbon nanotube yang lebih tinggi digunakan sebagai pengisi didalam pelekat konduktif elektrik, prestasi kekonduksiannya adalah lebih tinggi, manakala kekuatan ricihnya menurun dengan pemuatan pengisian yang menaik. Sampel bagi ujikaji elektrik dan mekanikal pelekat konduktif elektrik dengan nisbah aspek karbon nanotube berlapis yang berbeza disediakan dan diuji dengan merujuk kepada ASTM- F390-11 dan ASTM D1002 masing-masing. Pelekat konduktif elektrik disediakan dengan proses pencampuran sebelum diletakkan didalam ketuhar pada suhu 100 °C selama 30 minit. Nisbah aspek bagi karbon nanotube berlapis yang pertama adalah 1750 manakala nisbah aspek bagi yang kedua pula adalah 112.5 dan nilai pemuatan pengisian yang berbeza digunakan bagi kajian ini iaitu 5% berat, 6% berat dan 7% berat. Sampel elektrikal diuji menggunakan probe empat titik dan sampel mekanikal diuji menggunakan mesin ujian sejagat. Dengan merumuskan pelekat konduktif elektrik menggunakan nisbah aspek karbon nanotube yang lebih tinggi, ambang perkolasi dapat dicapai dengan pemuatan pengisian yang lebih rendah yang menunjuk pengaliran elektrik yang lebih baik. Keputusan kajian ini juga menunjuk semakin meningkat pemuatan pengisian, semakin rendah nilai kekuatan ricih bagi pelekat konduktif elektrik. Nisbah aspek mempunyai kesan terhadap sifat elektrik dan mekanik bagi pelekat konduktif elektrik yang disebabkan oleh pengaglomeratan dan penyebaran karbon nanotube berlapis didalam pelekat konduktif elektrik.

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LIST OF ABBREVIATIONS

3D	Three Dimensional
ACA	Anistropically Conductive Adhesive
CNT	Carbon nanotubes
CVD	Chemical vapor decomposition
ECA	Electrically conductive adhesive
ICA	Isotropically Conductive Adhesive
MWCNT	Multiwall carbon nanotubes
NCA	Non-Conductive Adhesive
PDMS	Polydimethylsiloxane
SEM	Scanning Electron Microscope
SWCNT	Single wall carbon nanotubes
TEM	Transmission electron microscopy
TPU	Thermoplastic polyurethane
UV	Ultra violet

LIST OF SYMBOLS

$^{\circ}\text{C}$	=	Degree Celcius
k	=	Kelvin
Ω	=	Ohm
sq	=	Square
T_g	=	Glass temperature
g	=	Gram
m	=	Meter
nm	=	Nanometer
μm	=	Micrometer
L	=	Length
OD	=	Outer diameter
V_m	=	Volume of matrix
V_f	=	Volume of fiber
V_c	=	Volume of composite
wt%	=	Weight percentage
mm	=	Milimeter
τ	=	Shear
F	=	Force
A	=	Area
R	=	Resistance
V	=	Voltage
I	=	Current
C	=	Lateral correction factor
Pa	=	Pascal
Mpa	=	Mega Pascal
Gpa	=	Giga Pascal
Tpa	=	Tera Pascal

CHAPTER 1

INTRODUCTION

1.1 Background

The use of lead (Pb) containing solder for assembly of electronic components has been introduced for a long time. Since lead (Pb) is a substance that is high in toxicity, the electronic industries are eliminating or minimizing the usage volume of lead containing solders in response to allow for a better environmental friendly and sustainability industry. It also one of the response toward the international restriction on using Hazardous Substance (RoHS) legislation [1].

The effort to eliminate lead in solder have lead the electrical industries to two alternatives, that is lead-free metal solder alloys and polymer-based electrically conductive adhesive (ECA) [2]. There are two components in ECA which consists of resin or polymer matrix which can be either thermoplastic or thermosetting and a conductive filler typically based from metallic materials. The polymer matrix in electrically conductive adhesive (ECA) provides the mechanical properties such as mechanical strength, adhesion, and impact strength while the conductive filler provides the electrical properties which are relatively different from metal solder, in which the mechanical and electrical properties are provided by only one component [3].

Due to higher capability compared to other materials, high electrical conductivity, and chemical stability, the most commonly used filler in ECA is silver. Other than lowering the processing condition temperature and stress on substrates, ECA also have fine pitch interconnect capability and environmental friendly [4]. Multiwall carbon nanotubes (MWCNT) is another type of filler that is currently under development.

Even though silver is currently the most useable metal filler in ECA, the industry requires a lower processing temperature material to replace silver. Furthermore, there is no current commercialized ECA that is able to overcome the limitations of such materials ; these being lower electrical conductivity, low reliability and capability and poor impact strength [4].

1.2 Problem Statement

Due to the requirements of law to decrease and eliminate the use of hazardous materials, the industries have stop using lead-containing solder. The most common problem with lead-free solder is that a thick layer of intermetallic compounds is formed between the substrate and solder which decrease the electronic components performance significantly [5]. The other problem with lead free metal solder are the processing temperature is high which is more than 180 °C.

There are few problems that limits the usage of ECA in electronic industries such as poor impact strength, low electrical conductivity, conductivity fatigue which means the conductivity of the ECA lowers under increasing temperature and humidity aging [6].

ECA with MWCNT is being developed to improve and overcome all the problems. Thus, the aim of this study is to develop a MWCNT ECA with different aspect ratio with good mechanical and electrical performance at much lower processing temperature.

1.3 Objective

The objectives of pursuing this research topic are:

1. To fabricate the electrically conductive adhesive (ECA) using multi-wall carbon nanotube (MWCNT) with different aspect ratio and filler loading.
2. To study the electrical properties of the MWCNT ECA with different aspect ratio and filler loading.
3. To study the mechanical strength of MWCNT-filled ECA with different aspect ratio and filler loading.

1.4 Scope of Project

The followings are the scope of this research projects:

- I. Fabrication of ECA.
- II. Electrical characterization using four-point probe test unit.
- III. Mechanical characterization using universal testing machine (lap shear test)
- IV. Surface morphology study.

1.5 Planning and Execution

Figure 1.1 below illustrates the research activities for PSM 1 that includes the process of research title selection, literature review, designing the experiment, formulation of samples, material characterization testing, data analysis and followed by report writing and report submission and lastly PSM 1 seminar. The material characterization includes electrical and mechanical testing. The research activities in PSM II started with the formulation of sample and followed by the material characterization for electrical and mechanical properties for all sample. Morphological study using Scanning Electron Microscope of each sample is done before all the data is analyzed. Finally, all the data and results are discussed in the report before submission and PSM II seminar. Figure 1.2 shows the Gantt chart of PSM II research activities.

Activities	Week													
	1	2	3	4	5	6	7	8	9	10	11	12	13	14
Research Title Selection	█	█												
Literature Review	█	█	█	█	█	█	█	█	█	█	█	█	█	█
Design of experiment	█	█	█	█										
Formulation of Sample				█	█	█	█	█						
Characterization Testing Electrical Mechanical							█	█	█	█				
Data Analysis									█	█	█			
PSM I Report Writing										█	█	█		
PSM I Report Submission													█	
PSM 1 Seminar														█

Figure 1.1: Gantt chart detailing research activities and time frame for PSM I

Activities	Week													
	1	2	3	4	5	6	7	8	9	10	11	12	13	14
Literature Review	█	█	█	█										
Formulation of Sample	█	█	█	█										
Characterization Testing Electrical Mechanical			█	█	█									
Morphological Study (SEM)				█	█	█	█							
Data Analysis					█	█	█	█	█					
Result and Discussion						█	█	█	█	█	█	█		
PSM II Report Writing									█	█	█	█		
PSM II Report Submission													█	
PSM II Seminar														█

Figure 1.2: Gantt chart detailing research activities and time frame for PSM II

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

In this chapter a review on interconnect material, electrically conductive adhesive, polymers, filler, carbon nanotubes, and the mechanical properties, electrical properties and thermal properties are reviewed from the previous studies on this area.

2.2 Interconnect Material

Interconnect material or commonly term as solder is a metal alloy that can fuse and used to bond electronic connections. In the semiconductor and electronics industries, it is common to use lead (Pb) containing solder as the interconnecting materials of power, signal or transmission. However, due to the amount of toxic produced by lead substance, industries are seeking for other alternative to change for a more environmental friendly interconnecting material [7].