# HYGROTHERMAL AGING EFFECT ON RELIABILITY PERFORMANCE OF ELECTRICALLY CONDUCTIVE ADHESIVES (ECA)

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# HYGROTHERMAL AGING EFFECT ON RELIABILITY PERFORMANCE OF ELECTRICALLY CONDUCTIVE ADHESIVES (ECA)

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**A report submitted**

**in fulfillment of the requirements for the degree of**

**Bachelor of Mechanical Engineering (Hons)**

**Faculty of Mechanical Engineering**

**UNIVERSITI TEKNIKAL MALAYSIA MELAKA**

**2018**

# DECLARATION

I declare that this project entitled "Hygrothermal aging effect on reliability performance of electrically conductive adhesive (ECA)" is the result of my own research 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 report is sufficient in terms of scope and quality for the award of degree in Bachelor of Mechanical Engineering (Hons)

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

Name :.....................................

Date :.....................................

# DEDICATION

To my beloved mother and father

**ABSTRACT**

This research investigates the hygrothermal aging effect on performance reliability of electrically conductive adhesives made from solution mixing process using epoxy matrix and MWCNT with filler loading of 5 wt.%, 6 wt.% and 7 wt.%. The test specimens which are the electrically conductive adhesives (ECA) were prepared in accordance with ASTM F390-11 using a four point probe for electrical conductivity measurement while the lap shear test was conducted with reference to ASTM D1002-10 using a universal testing machine. For the hygrothermal aging study, the ECA samples were conditioned in a humidity chamber at setting conditions of 85°C and 85 % of relative humidity (85 % RH) to assess the reliability performance of the ECA. The test specimens were subjected to 168 hours and 504 hours of hygrothermal aging and specified test specimens were characterized at normal condition as controlled specimens. Following hygrothermal aging period, the test specimens were characterized in terms of their electrical and mechanical performance. With presence of moisture attack, that is the water molecules, the electrical conductivity of the ECA increase with hygrothermal aging period. Meanwhile, lap shear results revealed contradicting trend. Regardless of the amount of MWCNT filler loading used (5-7 wt.%), due to moisture attack, voids are created in the epoxy matrix of the ECA, which results in a decrease in the shear strength of the ECA, when the samples were subjected to 168 hours and 504 hour of hygrothermal aging at 85°C and 85% RH. The trend changes at the ECA after being aged for 504 hours. The 6 wt. % and 7 wt. % of MWCNT filler loading shows an increase of shear strength while the 5 wt. % shows a decrease in shear strength. Nevertheless, all specimen shows the formation of voids in the epoxy matrix of the ECA, which contributes to the decrease in the shear strength of the ECA.

**ABSTRAK**

*Kajian ini mengkaji tentang kesan penuaan hygrothermal terhadap prestasi kebolehpercayaan pelekat pengalir elektrik dengan kaedah pencampuran cecair menggunakan epoksi matriks dan MWCNT dengan muatan sebanyak 5 wt. %, 6 wt. % dan 7 wt. %. Spesimen ujian iaitu pelekat elektrik (ECA) telah disediakan mengikut piawai ASTM F390-11 dan menggunakan alat "4 point probe" untuk mengukur aliran elektrik manakala ujian ricih telah dijalankan dengan merujuk kepada piawai ASTM D1002-10 dengan menggunakan mesin ujian universal. Untuk kajian penuaan hygrothermal, sampel ECA telah diletakkan di dalam mesin ruang kelembapan dalam keadaan suhu 85℃ dan 85% kelembapan relatif (85% RH) untuk menilai prestasi kebolehpercayaan ECA tersebut. Spesimen ini di uji selama 168 jam dan 504 jam di dalam ruang kelembapan manakala spesimen ujian yang berada dalam keadaan normal dikenali sebagai spesimen terkawal. Selepas penuaan selesai, spesimen ujian dicirikan dari segi prestasi elektrik dan mekanikal mereka. Kajian ini mendapati bahawa keupayaan ECA untuk mengalirkan arus elektrik semakin bagus. Ini kerana molekul – molekul air yang telah meresap hasil daripada tempoh penuaan hygrothermal. Akan tetapi, hasil daripada ujian ricih telah mendapatkan trend yang bertentangan. Tanpa mengira jumlah muatan MWCNT digunakan (5-7 wt.%), hasil keresapan air terhadap ECA tersebut telah menghasilkan ruang kaviti di dalam matriks epoksi ECA, di mana telah menyebabkan penurunan dalam kekuatan ricih ECA, apabila sampel tertakluk kepada 168 jam dan 504 jam penuaan hygrothermal pada 85 ° C dan 85% RH. Perubahan trend pada ECA 504 jam penuaan, spesimen dengan muatan MWCNT 6 wt. % dan 7 wt. % mereka telah menunjukkan peningkatan kekuatan ricih manakala muatan MWCNT 5 wt. % menunjukkan penurunan dalam kekuatan ricih. Walau begitu, kesemua specimen telah menunjukkan penghasilan kaviti didalam epoksi matriks pada setiap ECA, di mana ruang kaviti ini memberi kesan terhadap penurunan dari segi kekuatan ricih pada ECA tersebut.*

**ACKNOWLEDGEMENTS**

Foremost, I would like to thank all the people who made this report to be completed. I felt grateful for having a chance to learn knowledge and experience during the journey for completing this report. First of all, I would like to express my sincere gratitude to my supervisor, Dr. Siti Hajar Binti Sheikh Md Fadzullah, who offers me countless valuable knowledge and advice throughout the completion for this report by spending her time and energy to help me completing this project.

I would like to thanks all the technicians of Mechanical and Manufacturing Engineering Faculty where they also make their time for me to use the labs and apparatus especially Mr. Bahatiar for allowing me to use the apparatus at Manufacturing Engineering Faculty. A big thanks to Muhamad Muaz Bin Nasaruddin who helped me a lot by sharing his knowledge, time and experience even though he is busy with his works. Nevertheless, without his help I would not be able to complete the experimentation in time.

Last but not least, I would like to express my thanks to dearest family members and friends for their endless support, advice and guidance throughout my final year project journey. Without their precious support, it would not be possible for me to complete this project holding my head high.

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| | |
|---|---|
| ACA | Anistropic Conductive Adhesive |
| ACF | Adhesive Conductive Film |
| AFM | Atomic Force Microscopy |
| ASTM | American Society for Testing and Materials |
| C | Carbon |
| CNT | Carbon Nano – Tube |
| DWCNT | Double Wall Carbon Nano – Tube |
| ECA | Electrically Conductive Adhesive |
| EMI | Electromagnetic Interface |
| ICA | Isotropic Conductive Adhesive |
| MWCNT | Multi – Wall Carbon Nano – Tube |
| PDA | Polydopamine |
| RH | Relative Humidity |
| SEM | Scanning Electron Microscope |
| SWCNT | Single – Wall Carbon Nano – Tube |
| TEM | Transmission Electron Microscope |

## LIST OF SYMBOLS

| | | |
|---|---|---|
| $T_g$ | = | Glass Temperature (°C) |
| $W_t$ | = | Weight after environmental test |
| $W_i$ | = | Weight before environmental test |
| Xi | = | irritant |
| $W_{fraction}$ | = | Weight fraction of CNT |
| $W_{composite}$ | = | Total mass of composite |
| $W_{filler}$ | = | Mass of CNT (g) |
| $W_{matrix}$ | = | Mass of polymer matrix (g) |
| Wt% | = | Weight percentage |
| V | = | Potential difference (V) |
| I | = | Current (μA) |
| R | = | Sheet resistance (Ω/sq) |
| τ | = | Shearing stress ($N/mm^2$) |
| F | = | Force (N) |
| A | = | Area of contact ($mm^2$) |

# CHAPTER 1

# INTRODUCTION

## 1.1 BACKGROUND

Electrically conductive adhesives (ECA) is one of the replacements for the use of lead-bearing solders where it is known as one of the environmentally friendly solders in electronics (Li, Lu and Wong, 2010). The characteristic of the ECA is that it has a property to electrically conduct electricity as well as heat. The electrical conductivity in the ECA is important since the adhesive must be able to create an electric connection between electric components as well as providing electromagnetic interface (EMI) or radio frequency interference functions (Metal Finishing, 2008). The ECA also has a good adhesion, high mechanical strength, impact strength and have a conductive filler.

The ECA consists of conductive metallic properties and a polymer matrix. The most typically used conductive metal in the ECA are silver and epoxy resin (Amoli, 2015). This is due to the environment-friendly properties that silver has rather than using lead which is harmful towards humans. Also, silver has the highest electrical conductivity and the lowest resistivity compared to other conductive metal such as gold, nickel and copper. However, all the metallic conductor listed are expensive. The epoxy resin that is used in the ECA is one of the conducting polymers where it is possible to obtain high dielectric constant (Lu et al., 2007). Other than silver, gold, nickel and copper as the metallic filler in the ECA, carbon nanotube (CNT) are also used as the metallic filler in the ECA. The CNT is a tube-shaped material which is made from carbon where the diameter is measured in nanometre.

The CNT varies in length, thickness and number of layers. Its characteristic can be changed depending on the layer of graphene sheet that being rolled to create the tube and changes the property whether is metallic or semiconductor (Nanoscience Instruments, 2016). There are two types of CNT, the first one is called the single-walled CNT. The single-walled CNT or SWCNT shaped as a tube with only one layer. While the second type of CNT is called the multi-walled CNT or MWCNT. The MWCNT consists of several layers of CNT which it increases in diameter. The CNT is capable for lowering the cost for developing the ECA since it helps reduces the metal content in the ECA by constructing conductive networks hence reducing the cost of ECA (Luo et al., 2016).

## 1.2 PROBLEM STATEMENT

It is found that ECA is greatly affected when the ambient temperature and humidity are changed. The water gain in the polymer matrixes causes an internal hydrolysis of colloids which can result in an increase of electrical resistance and reduces the bonding strength which may lead to failure. In other words, hygrothermal aging of the adhesive bonds may result in decreasing of volume (Mach, Skvor and Szaraz, 2000).

In the previous study, the mechanical and the electrical properties of the ECA were tested under a constant humidity level of 85% at 85°C for about 500 hours. The results from the studies show that the contact resistance is increased with aging time. However, it is seen that the longer the duration of hygrothermal aging results in less effect on the contact resistant of the ECA (Cui et al., 2013).

Hence, this study aims to prolong the duration of the hygrothermal aging process for about 504 hours followed by performance reliability of the ECA in terms of the electrical conductivity and mechanical properties of the ECA using varying MWCNT filler loading.

## 1.3 OBJECTIVES

The objectives of this project are:

i. To investigate the effect of hygrothermal aging on the reliability performance of the ECA.
ii. To examine the shear strength of the ECA following hygrothermal aging.
iii. To study the effect of temperature and humidity on the electrical conductivity of the ECA.

## 1.4 SCOPE OF PROJECT

The scope covered in this project is as stated below:

i. Fabrication of ECA.
ii. Electrical characterization using a 4-point probe.
iii. Mechanical characterization using Universal Test Machine (Lap Shear Test).
iv. Surface morphology.
v. Reliability study

## 1.5 PLANNING

The research activities that were undertaken for final year project I or PSM I are summarized in Table 1.1. The progress for about the first four weeks includes background study, literature review and fabrication of ECA. In week five, the aging process is conducted by inserting the ECA into the humidity chamber for about four weeks. Following this, the thermally aged ECA is then tested for its conductivity and the mechanical properties, via lap shear test. Then, the experimental data are analysed from week 10 and initiation of the PSM I report writing began. Week 13 is the week for the draft to be submitted to the project supervisor for further advice and amendment. Finally, in week 14, the PSM I report will be submitted.

As for PSM II, the research activity are summarized in Table 1.2. The literature review are continued from the PSM I and being updated until week 12. Next is the hygrothermal aging process which takes about three weeks depends on the availability of the humidity chamber. Following the aging process is the electrical and mechanical characterization. The electrical characterization tested on its conductivity while the mechanical characterization is tested using the lap shear test. After the lap shear test, the specimen are subjected to SEM and EDS to determine the structure of the MWCNT and the fracture after the test. The EDS is done to verify the water content in the ECA after being subjected to hygrothermal aging.

Table 1.1: Gantt chart for the research activities for PSM1

| Progress | Week | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 | 11 | 12 | 13 | 14 |
| Background Study | | | | | | | | | | | | | | |
| Literature Review | | | | | | | | | | | | | | |
| Lab Visit | | | | | | | | | | | | | | |
| ECA Fabrication | | | | | | | | | | | | | | |
| Conduct Experiment: Hygrothermal Aging | | | | | | | | | | | | | | |
| Conduct Experiment: Electrical Conductivity | | | | | | | | | | | | | | |
| Conduct Experiment: Lap Shear Test | | | | | | | | | | | | | | |
| Data Analysis | | | | | | | | | | | | | | |
| Report Writing | | | | | | | | | | | | | | |
| Draft Submission | | | | | | | | | | | | | | |
| Report Submission | | | | | | | | | | | | | | |

Table 1.2: Gantt chart for the research activities for PSM II

| Progress | Week | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 | 11 | 12 | 13 | 14 |
| Literature Review | | | | | | | | | | | | | | |
| ECA Fabrication | | | | | | | | | | | | | | |
| Conduct Experiment: Hygrothermal Aging | | | | | | | | | | | | | | |
| Conduct Experiment: Electrical Conductivity | | | | | | | | | | | | | | |
| Conduct Experiment: Lap Shear Test | | | | | | | | | | | | | | |
| Conduct Experiment: SEM analysis | | | | | | | | | | | | | | |
| Data Analysis | | | | | | | | | | | | | | |
| Report Writing | | | | | | | | | | | | | | |
| Draft Submission | | | | | | | | | | | | | | |
| Report Submission | | | | | | | | | | | | | | |

# CHAPTER 2

# LITERATURE REVIEW

## 2.1 Introduction

In this chapter, reviews of electrically conductive adhesives (ECA) which include the type of ECA filler materials, types of Carbon Nanotube, mechanical and electrical properties, surface morphology and the reliability performance of ECA from the previous studies are presented.

## 2.2 Electrically Conductive Adhesives

Electrically conductive adhesives (ECA) is a form of adhesives that provides mechanical and electrical properties which interconnects between a device, lead or chip carrier onto a circuit board. The unique ability is obtained by the composites material resides in the ECA. It is mainly composed of a polymer matrix and metallic particles (Li and Morris, 1998). The ECA is used as a replacement for soldering where the ECA is lead-free (Gilleo, n. d.). It also offers some advantages like flexiblility, simple processing and at a lower cost. There are two categories of ECA. The first one the Isotropic Conductive Adhesive (ICA) and the second one is the Anisotropic Conductive Adhesive (ACA) (Melorose, Perroy and Careas, 2015).

ICA is usually made up of silver as their metallic particle and known to be one of the substitutes for lead-based solder. The ICA possess a low-temperature processing, flexible and is compatible with non-solderable materials (Yim and Kim, 2010). However, due to its low conductivity and unstable contact resistance, it does not satisfy the similarity with the solder.

For example, the contact between silver particles is supposed to increase when the density increases due to contraction in curing of the binder and the electrical connection which results in the contraction during curing is not necessarily (Kohinata et al., 2013). The matrix shrinkage during curing does not help in development of increasing the conductivity (Lu, Tong and Wong, 1999). ACA is known as a method of connecting high density electrodes on electric components (Dou, Chan and Liu, 2004). It has the advantages of having low processing temperature, high component density and compatible with non-solderable components (Dou, Chan and Liu, 2003). Due to the adhesion between the substrate and the adhesives is one of the major concern for this type of ECA, the volume fraction is about 0.5% to 5% are below the percolation threshold. This results in unstable electrical conductivity before bonding (Mantena, 2009).

### 2.2.1 Matrix

The polymer matrix used in the ECA belongs to two categories. The first one is called thermoplastics. Thermoplastics consists of linear polymer chains with or without branching or side groups (Epoxy Technology, 2016). Its materials are rigid materials when at below the glass transition temperature, $T_g$. If the temperature is above the $T_g$, its starts to develop flow characteristics (E.C. Adhesives, 2010). The disadvantages of the thermoplastics is the degradation at high temperatures, where cavity is formed during the solvent evaporation.