FABRICATION AND PROPERTIES OF SILICA-EPOXY THIN FILM COMPOSITE

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FABRICATION AND PROPERTIES OF SILICA-EPOXY THIN FILM COMPOSITE

by

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DECLARATION

I hereby declare that I have conducted, completed the research work and written the dissertation entitled **“Fabrication and Properties of Silica-Epoxy Thin Film Composite”**. I also declare that it has not been previously submitted for the award of any degree or diploma or other similar title of this for any other examining body or University.

Saya isytiharkan bahawa kandungan yang dibentangkan di dalam disertasi bertajuk ***“Fabrikasi dan Sifat-Sifat Komposit Filem Nipis Silika-Epoksi”*** *ini adalah hasil kerja saya sendiri dan telah dijalankan di Universiti Sains Malaysia kecuali dimaklumkan sebaliknya. Disertasi ini juga tidak pernah disertakan untuk ijazah yang lain sebelum ini.*

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LIST OF ABBREVIATIONS

mm	milimetre
SSA	specific surface area
CTE	Coefficient of thermal expansion
DMA	Dynamic mechanical analysis
TMA	Thermal mechanical analysis
SEM	Scanning Electron Microscope
TEM	Transmission Electron Microscope
FTIR	Fourier Transform InfraRed Spectroscopy
TGA	Thermo Gravimetric Analysis
DTG	Derivative Thermo Gravimetric
IDT	initial decomposition temperature
GPa	Giga pascal (10^9 Pa)
H	hour
ppm	Part per million
M	Molarity
ml	mililiter
min	minutes
MPa	Mega pascal (10^6 Pa)
mm	milimeter
s	second
T	Temperature

LIST OF SYMBOLS

^{o}C	Degree Celsius
%	Percentage
μm	Micrometer (10^{-6} m)
nm	Nanometer (10^{-9} m)
E’	Storage Modulus
E	Tensile Modulus
TS	Tensile Strength
ε_B	Strain at Break
T_g	Glass Transition Temperature
T_{max}	temperature at the maximum rate of weight loss
$Tan\ \delta$	Tan Delta
α_G	Coefficient of Thermal Expansion at glassy region
α_R	Coefficient of Thermal Expansion at rubbery region
D	Mean particle size
$I_{A/V}$	interface area between the epoxy and filler per volume unit

FABRIKASI DAN SIFAT-SIFAT KOMPOSIT FILEM NIPIS SILIKA-EPOKSI

ABSTRAK

Dengan peningkatan minat terhadap pembungkusan elektronik berketumpatan tinggi, penskalaan dimensi menjadi kunci terhadap evolusi ini. Dalam kajian ini, komposit filem nipis epoksi dengan pengisi silika dihasilkan menggunakan proses salutan putaran. Dalam bahagian pertama penyelidikan ini, perbandingan sifat-sifat mekanik dan terma bagi komposit filem nipis yang mengunakan pengisi mikro-silika (μ-Si) dan nano-silika (n-Si) telah dijalankan. Komposit filem nipis dengan pembebanan n-Si yang rendah didapati mencapai sifat-sifat tegangan dan terma yang setara dengan komposit filem nipis yang mengandungi pembebanan μ-Si yang tinggi. Dengan nisbah luas permukaan dan isipadu yang tinggi, n-Si dengan pecahan berat rendah menunjukkan kemampuannya dalam mencapai prestasi yang setara dengan μ-Si. Dari segi agen pematangan, komposit filem nipis yang dimatangkan dengan imidazol didapati mempunyai sifat-sifat mekanik dan terma yang baik hasil daripada kehadiran lima anggota gelang dalam struktur kimia imidazol. Penyerapan lembapan bagi sampel yang dimatangkan dengan imidazol didapati lebih tinggi daripada sampel yang dimatangkan oleh polieteramin kerana resin yang telah dimatangkan dengan imidazol menjadi resin yang lebih berkutub. Serakan bagi pengisi n-Si didapati bertambah baik melalui rawatan n-Si dengan agen gandingan (*3-Glycidyloxypropyl) trimethoxysilane*. Adalah didapati bahawa rawatan n-Si dengan agen gandingan *(3-Glycidyloxypropyl) trimethoxysilane* selama 8 jam menghasilkan sifat-sifat mekanik komposit filem nipis yang optimum. Masa rawatan yang panjang menyebabkan penurunan nilai modulus tegangan dan kekuatan tegangan tetapi menaikkan kebolehlenturan komposit filem nipis.

Kewujudan n-Si yang dirawat dalam sistem komposit menyebabkan peningkatan nilai pekali pengembangan terma (CTE) dan merendahkan kestabilan terma. Walau bagaimanapun, rawatan n-Si adalah didapati menyebabkan pengubahsuaian permukaan n-Si yang hidrofilik kepada permukaan yang hindar air, ini mengurangkan penyerapan lembapan pada komposit filem nipis.

FABRICATION AND PROPERTIES OF SILICA-EPOXY THIN FILM COMPOSITE

ABSTRACT

With the increasing interest in high density electronics packaging, dimensional scaling becomes the key to evolution. In this study, epoxy thin film composites with silica fillers were fabricated using spin coating process. In the first part of this research, the mechanical and thermal properties of the thin film composites using micron-silica (μ-Si) and nano-silica (n-Si) fillers were compared. Low loading of n-Si thin film composites was found to have the ability to achieve equivalent tensile and thermal properties compared to the highly loaded μ-Si thin film composite. With higher surface area to volume ratio, n-Si shows its capability to achieve a substantial performance with a lower weight fraction compared with μ-Si. In terms of curing agents, imidazole cured thin film was found to have better mechanical and thermal properties due to the presence of five membered ring in the imidazole chemical structure. The moisture absorption of the imidazole cured samples were slightly higher than the polyetheramine cured samples because imidazole cured resin is a polar resin. The dispersion of the n-Si filler was improved by the treatment of n-Si with (3-Glycidyloxypropyl) trimethoxysilane coupling agent. It was observed that 8 hours treatment time showed high mechanical properties of the thin film composites. Prolong treatment time caused the drop in tensile modulus and tensile strength but increase the flexibility of the thin film composites. The presence of treated n-Si in the composite system causes the increase in the coefficient of thermal expansion (CTE) value and lowers the thermal stability compared to the untreated n-Si thin film composites. However, treatment of the n-Si was found to cause

the modification of the hydrophilic n-Si surface into a hydrophobic surface, thus reduced the moisture absorption of the thin film composites.

CHAPTER 1

INTRODUCTION

1.1 Background

In line with the miniaturization trend, the evolution of the traditional integrated circuit (IC) packaging had taken place from wire-bonded silicon chip on lead frame into flip chip packaging. This high electronic packing density gave smaller, cheaper and lighter assemblies with improved microprocessor performance (He et al., 2000). With the continuous improvement of the microprocessor performance, dimensional scaling of the electronic packaging materials becomes the key of evolution other than the circuit design and materials selection. Hence, the trend of the electronics design has now shifted to flexible electronics technology, which are also known as “flex circuits”. Thickness and flexibility are the major concern in flexible electronic technology. Therefore, polymer materials are promoted as the main choice in the materials selection for thin film package fabrication.

Thin film is the size reduction of bulk materials in one dimension. According to ASTM D 882-02, a material is known as thin film when the thickness is less than 0.25 mm. Thin films of polymers are getting acceptance for technological applications ranging from multicolor photographic printing to paints, adhesives, index-matched optical coatings, photoresists and low dielectric electronic packaging (Ade et al., 1998). In comparison to bulk properties, much less is known about the properties of polymers when they are processed into thin films. Typically, the polymer-substrate and polymer-air interfacial energies or kinetic barriers play important roles in determining the morphology and dynamics in thin films.

1.2 Problem Statements

Polyester and polyimide are the most common materials used in flexible electronic (Coombs, 2001). In the late 1960’s polyimide was favorable as the solution to the temperature limits of polyester. The low coefficient of thermal expansion (CTE) and ability to retain its electrical and mechanical properties over wide range of temperature (as high as 400^{o}C) had made it become the favorable choice of materials used in electronic packaging (Minges & ASM International Handbook Committee, 1989). However, the cost ratio of polyimide to polyester is approximately 20:1 (Gilleo, 1997). Comparing with epoxy resins, polyimide cost twice or thrice higher. Besides, the fast moisture uptake of polyimide causes huge changes in the electrical properties that subsequently limit its development for the electronic industry (Kamei et al., 2000). In terms of fabrication, polyimides are difficult to work with, especially in multi-layer processing. Extra precaution steps have to be taken when drilling and routing which are required in the fabrication process. This is because polyimide materials have lower inter-laminar bond strength than epoxy systems (www.ami.ac.uk).

Epoxy resins are commonly used in electronic packaging due to their good chemical, mechanical and electrical properties. Although epoxy resin covers a range of materials with widely differing characteristics and costs, but a multifunctional materials/blends of epoxy resins can be developed to fill the niche between lower-cost regular epoxies and high-performance resins, and give an extended operational temperature range at lower cost than polyimide (Coombs, 2001). The properties of the epoxy film can be easily altered through the selection of fillers and curing agents. Understanding on the resin network formation through the application of different curing agents is necessary since the properties of cured epoxy are dependent on the chemical structure and the degree of crosslinking of resin network (Buist et al., 1994). However, the poor thermal properties of the epoxy resin such as high CTE causes the warpage failure in the electronic packaging. Table 1.1 shows the CTE of epoxies and other materials used in electronic packaging. The large differences in CTE raise the issue of thermal mismatch between the silicon chip and the flexible epoxy substrate. Residual warpage takes place with the presence of thermal cycling which leads to the mechanical constraints of solder joint attached on the substrate. Subsequently, this also causes the cracking and delamination problem on the package (Bruce, 2007; Valdevit et al., 2008). In order to enhance the thermal stability of the epoxy based packaging, many efforts such as incorporation of inorganic filler into the underfill were studied (He et al., 2000; Lee et al., 2007; Lee et al., 2005).

Table 1.1. Typical CTE of electronic packaging materials (Minges & ASM International Handbook Committee, 1989; Suhir et al., 2007; Hodgin & Estes, 1999)

Materials	**CTE (ppm/K)**
Epoxies	60-80*
Encapsulant	17*
Die-attach	75
Silicon	2-7
Copper	17
silver	19.7
aluminium	23

* below T_g for epoxies and encapsulant due to the polymer based system.

The effect of filler loading, filler shape and surface treatment of the silica filler towards the mechanical and thermal performance of the composite had been studied by previous researchers (Palaniandy et al, 2008; Xing & Li, 2004; Ahmad et al., 2008). However, upon addition to the polymer composite system, the micron- and submicron-sized silica requires a very high loading of up to 95 vol% in order to obtain a composite with a CTE value below 10 ppm/^{o}C (Teh et al., 2007a). This will weaken the mechanical performance of the composites especially those involving thin film composites. The high loading of micron and submicron silica leads to the increase in the viscosity of the system and causes difficulty to the fabrication process. Additionally, the high loading of large size silica fillers often result in rougher surface and lower film flexibility. Advancement in nanotechnology has attracted the study of polymer nanocomposites in the scientific research and engineering applications. Due to its extremely small size and large surface to volume ratio, nano-sized fillers induce different properties compared with micron-sized fillers of the same chemical composition (Sun et al., 2005a). This broadened the application of nanocomposites in the microelectronics field (Singha & Thomas, 2008; Imai et al., 2005). Moreover, the flexibility of thin film composites at a high loading of nano-sized filler was reported to remain the same with the low loading (Das et al., 2009). This results in the possibility of the fabrication of epoxy/silica thin film composites. In terms of thermal properties, the smaller nano-sized inorganic fillers show an improvement in CTE compared with the micron-sized fillers (Goyal et al., 2008). The high surface area per unit volume of this nano filler shows a better efficiency in reducing the CTE compared with the micron filler.

1.3 Objectives

1. To study the effect of silica size and loading on the tensile, thermal properties and moisture absorption of epoxy thin film composites.
2. To evaluate the behavior of different curing agents on the tensile, thermal properties and moisture absorption of epoxy thin film composites.
3. To optimize the tensile, thermal properties and moisture absorption of the epoxy thin film composites via treatment using silane coupling agent.

1.4 Summary of the Thesis

In this thesis, the fabrication of epoxy thin film composites and study on the properties of the thin film composites were reported. There are five chapters being covered in this thesis. Chapter 1 covers the introduction regarding the evolution of the miniaturization in the electronic packaging which leads to the invention of thin and flexible circuits, the importance of the proper materials selection and the parameters involved during the filler selection. Three objectives of this study were stated in this section.

While for the Chapter 2, the literature review on the electronic packaging was reported. It covers the types of polymer chosen to be used in electronic packaging, the current polymer composites that discovered to be applied in electronic application and also the researches on the polymer thin film done by the previous researchers. At the end of Chapter 2, the current research work done on the polymer thin film composites was reported as well.

The materials and methodology was reported in the Chapter 3. The testing and characterization was also reported in the same chapter. Chapter 4 covers the results and discussions of the planned objectives as indicated in Chapter 1. The results and discussions are separated into three parts, which is the effect of filler size and loading, the behavior of different curing agents, and the effect of filler surface treatment towards the properties of the epoxy thin film composites. In the end of the thesis (Chapter 5), the conclusion of the project and the proposal on the future work was reported.

CHAPTER 2

LITERATURE REVIEWS

2.1 Electronic Packaging

2.1.1 Introduction

Electronic packaging might sounds like the packaging involved for the marketing and shipment of the electronic devices. But in electronic industry, the definition of electronic packaging refers to the technology involved where the components in a system are electrical interconnected and interfaced to external devices (Minges & ASM International Handbook Committee, 1989). In the other words, electronic packaging playing an important role in protecting, providing mechanical and thermal support from the micro to macro scale circuit and enclosing of the electronic devices. Packaging technology also help to protect the IC from humidity and contaminant, and provide path for signal and electrical current to past through the silicon chip and electrical substrate (Pecht, 1999). Besides, a well-designed package must be able to dissipate the heat generated by the circuit and able to minimize the delay in the signal transmission within the system (Tong, 2010).

Figure 2.1 shows the typical hierarchy of electronic packaging where three packaging levels are involved. The electronic packaging process is started with the attachment of a single integrated circuit (IC) on the package. An IC chip is a single chip made from semiconductor material with integration of many circuits or components. Dependent on the application, IC can be a single component such as power transistor for simple application, while it can have many components such as integrated microprocessor in the high performance workstation. Hence, these ICs are classified by their composition, integration level, number of transistors, function of systems, equivalent gates and device type (Kwak & Hubing, 2007). In order to provide interconnection, power distribution, heat dissipation and protection to the IC chip, IC packaging, which is the first level packaging in the packaging hierarchy is involved. In this IC packaging stage, bare IC chip is mounted directly to the substrate in the direct chip attach (DCA) methods through the technologies such as wire bonding, flip-chip bonding and tape-automated bonding (Stout, 1994). The substrate in this first level packaging is normally the lead frames with specific shape which is ready for interconnection to the next level of packaging. However, for a complete system, number of different active and passive devices is required. Hence, second level packaging which is also known as system-level packaging is involved. All the required components are assembled on the system-level board and the interconnections between the components are done through the conductor wiring. While for the interconnection between the first level packaging and second level packaging, the connection is achieved through the bonding of solder from the first level package to the printed wired conductive board of the second level package (Tummala et al., 1997). For the high performance product such as supercomputers, several processors are required. Therefore, the total system become more complicated and several system level packages are required to provide extremely high transactional throughput. The assembly of the system level packages is referred as third level packaging in the hierarchy packaging. The system level packages are assembled on the motherboard or backplane, and the connections between the system level packages are completed with cables and connectors (Harper, 2005).

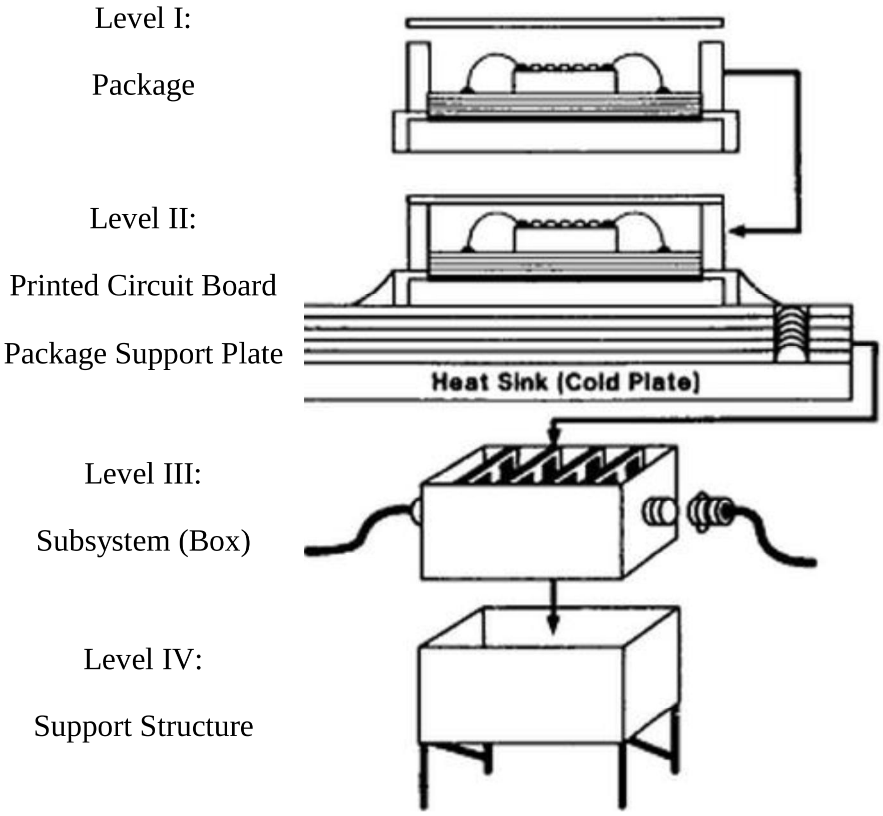

Level I:
Package
Level II:
Printed Circuit Board
Package Support Plate
Heat Sink (Cold Plate)
Level III:
Subsystem (Box)
Level IV:
Support Structure

Figure 2.1 Typical hierarchy of electronic packaging (Chung, 1995).

2.1.2 Polymer in Electronic Packaging

In electronic packaging, metals are used as conductor to transfer signal and power out from the IC to the outer world, while ceramics and polymers are commonly used as the insulator between the conductors and gives mechanical support. Due to the demand on smaller and lighter devices, adoption of the polymers as electronic packaging material has increased. Other than having lower density compared to the ceramic, the unique combination properties of the polymers such as low cost, good mechanical properties and eases of processing have widespread its use in electronic industries (Frear, 1999; Wong et al., 1999; Gilleo, 1996). The application of polymers no longer constrain as an insulator. Innovation of the technologies had made polymers an excellent semiconductors and conductors (Said, 2009). Varied application of the polymer can be seen these days ranging from dielectric, passivation layers, encapsulants, adhesives for die attach and underfill materials. (Rimdusit & Ishida, 2000; Shick et al., 1998)

Polymer can be divided into two groups which are thermoplastics and thermosets. Thermoplastics are the polymer that can repeatedly heat to soften for remolding while thermosets are the polymer that cannot be remelted due to the cross-linking formation during the conversion into the finished product (Rosato et al., 2000). Table 2.1 contains the common type of thermoset and thermoplastic materials used in electronic application, their characteristics, and applications descriptions. During the polymerization, thermoset polymers form a three-dimensional network which is very rigid in compared with the linear thermoplastic structure. Hence, thermoset materials with more cross-link densities are rigid, hard, brittle and less flexible compared to the thermoplastic materials (Gilleo, 2003).

Thermoplastics such as vinyls, polyethylene teraphthalate (polyester), fluorocarbons, polyimides and fluorocarbon-polyimides are commonly utilized for the application where flexibility is required, such as flexible circuit which will be discussed later. Among all of these materials, polyimide is the most expensive raw material to be used. However, it is still the best combination of cost and properties for high performance application such as military. Polyester is also one of the common materials for cost-sensitive flexible circuit but only to the low temperature application due to the poor thermal resistance. In the late 1960’s polyimide was favorable as the solution to

Table 2.1.Characteristic and applications for the common type of thermoset and thermoplastic materials used in electronic industry (Harper, 2005).

Materials		Characteristics	Applications
Thermoplastics	Flourocarbon	• Heat resistance • Superior chemical resistance • Low dielectric losses • Zero water absorption • Low friction coefficient	• Wire and cable insulation • Electrical components
	Liquid Crystal Polymer (LCP)	• High temperature and chemical resistance • High mechanical strength • Low thermal expansion	• Chip carriers • Sockets • Connectors • Relay cases
	Polyester (Polybutylene terephthalate, polyethylene terephthalate, polycyclohexylene dimethylene terephthalate)	• Good electrical properties • Chemical resistance • High-temperature resistance • Low moisture absorption	• Connectors • Sockets • Chip carriers
	Polyimides	• Superior high-temperature properties • Radiation resistance • Flame resistance • Good electrical properties	• Integrated-circuit applications • Insulation for electric motors
Thermosets	Epoxy Resin (Bisphenol A and bisphenol F)	• Excellent combination of physical, chemical, mechanical and electrical properties • Dimensional stability	• Printed-wiring-board substrates • Chip carrier • connectors
	Bismaleimides	• Superior elevated-temperature performace properties	• Printed-wiring-board substrates

the temperature limits of polyester due to its low coefficient of thermal expansion and good electrical and mechanical properties (Minges & ASM International Handbook Committee, 1989). However, the disadvantages of polyimide such as high cost, high moisture absorption had limited its mass application in electronic industry. Fluorocarbon is also commonly used in flexible circuit. Although it is an expensive materials and hard to handle, but its superior dielectric properties make it suitable to be applied in controlled impedance boards (Coombs, 1996).

In compared with thermoplastic materials, thermoset materials are generally higher strength than that of thermoplastic (Ritchie et al., 2003). Another advantange about the thermoset materials is they do not have a tendency to cold flow (creep) at room temperature. This is due to the formation of tightly cross-lined three dimensional network structure in the thermoset materials upon curing (National Research Council, 1985). These rigid thermosets are formed through the combination of two or more components and cured by applying sufficient heat. The cured thermoset is impossible to convert back into liquid form. The application of heat on the cured thermosets will not melt or flow, but it will start to lose its hardness or soften where the temperature to measure this deformation is known as glass transition temperature (Sheikh-Ahmad, 2008).

2.1.2.1 Epoxy

Epoxy resin is the most common thermoset materials used for electronic encapsulation and printed circuit board due to their good chemical, mechanical and electrical properties (Miao et al., 2010; Liu & Chen, 2004). Although epoxy resin covers a range of materials with widely differing characteristics and costs, but a multifunctional materials/blends of epoxy resins can be developed to fill the niche between lower-cost regular epoxies and high-performance resins, and give an extended operational temperature range at lower cost than polyimide (Coombs, 2001). The properties of the epoxy film such as thermal stability, electrical conductivity and thermal conductivity can be easily altered through the selection of fillers (Alapati & Thomas, 2008; Cheng et al., 2008; Liu et al., 2004; Teh et al., 2008). Apart from that, the simpler fabrication process of epoxy based thin film composite allows high volume production.

There are many types of epoxy resins in the market and Figure 2.2 shows the categories of these epoxy resins. The epoxy resins are categorized into two main categories which are glycidyl epoxy resins and non-glycidyl epoxy resins (Dunn, 2003). For glycidyl epoxy resins such as diglycidyl ether of bisphenol A (DGEBA), diglycidyl ether of bisphenol F (DGEBF) and epoxy novolac, they are prepared via condensation reaction of appropriate dihydroxy compound, dibasic acid or a diamine and epichlorohydrin. For non-glycidyl epoxy resins such as aliphatic and cycloaliphatic epoxy resins, they are formed by peroxidation of olefinic double bond (Dunn, 2003).

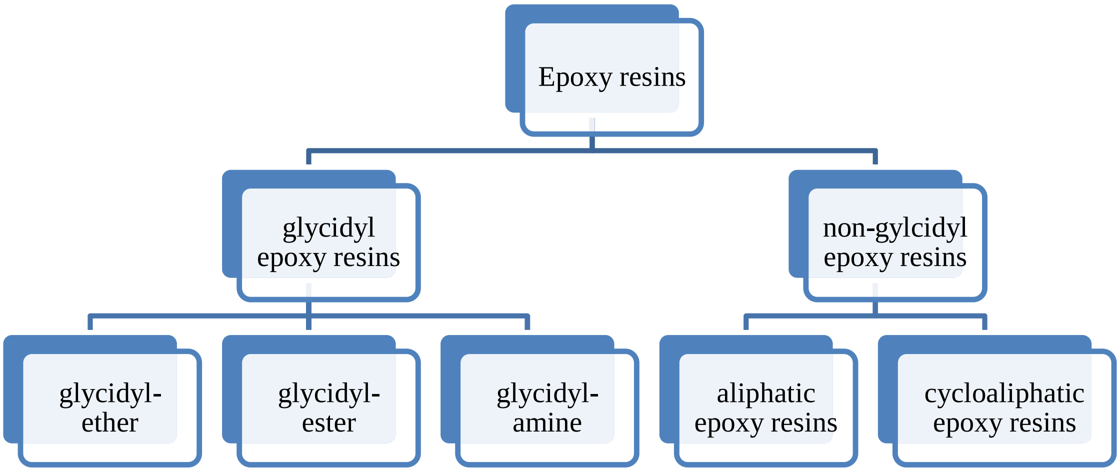

Epoxy resins
glycidyl epoxy resins
non-gylcidyl epoxy resins
glycidyl-ether
glycidyl-ester
glycidyl-amine
aliphatic epoxy resins
cycloaliphatic epoxy resins

Figure 2.2. Categories of epoxy resins (Dunn, 2003; Mays & Hutchinson, 1992).

DGEBA is the most common epoxy resins used not only in electronic packaging, it also have a well-established record in structural composites due to its fascinating performance with the reinforcement (Campbell, 2010). Figure 2.3(a) shows the chemical structure of DGEBA. They are a synthesis product through the condensation of bisphenol-A and epichlorohydrin with the presence of a basic catalyst. The properties of the DGEBA are depending on the value *n*, which is also known as degree of polymerization. This *n* value or the degree of polymerization depends on the stoichiometry of the synthesis reaction or the ratio of bisphenol A to epichlorohydrin (Dufton, 2000).

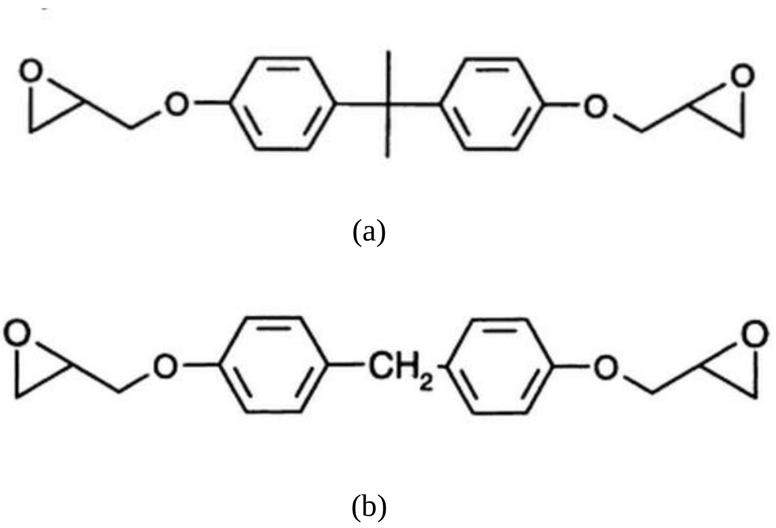


Figure 2.3. Chemical structure for (a) DGEBA and (b) DGEBF (Mays & Hutchinson, 1992; Balachandran, 2000)

DGEBF is the simplest novolac that prepared by the condensation of formaldehyde with phenolic substance under acidic conditions, and followed by epoxidation (Rabilloud, 1997). This type of epoxy resin has much attractive application that is paralleling with the DGEBA (Ardebili & Pecht, 2009). In compared with DGEBA, DGEBF posses lower viscosity and lower tendencies to crystallize. Figure 2.3(b) shows the chemical structure of DGEBF. It is noticed that the chemical structure of DGEBF has the same general structure as DGEBA. Therefore, the physical and chemical properties of these two epoxy resin are almost similar. The small difference in the chemical structure is actually contributed to the lower viscosity on DGEBF in compared with the DGEBA (Balachandran, 2000).

2.1.2.2 Curing Agents

The widespread usage of epoxy resins from large industry such as structural industry to small discrete industry such as electronic industry is due to their unique combination of properties when it cured (Tagami et al., 2008; Aziz, 2010; Kim et al., 2001). The characteristic of the cured epoxy resins are dependent on the curing system used for the formation of crosslinking in the epoxy resin. For the crosslinking reaction to occurs, two types of reaction are involved which are addition reaction and catalytic reaction (Craver & Carraher, 2000). For the addition reaction, the curing agent molecule which is also known as hardener molecule will chemically bonded to the epoxide group and serve as a bridge of crosslinking in the epoxy resins. Meanwhile for the catalytic reaction, the curing agent will serve as catalyst, encouraging the self-polymerization of the resin (Goosey, 1999).

There are hundreds of curing agents available in the market these days. These curing agents can be divided into four major categories which are amines, anhydrides, imidazoles and catalytic agents (as shown in Figure 2.4) (Lubin & Peters, 1998). Amines are the substitution of hydrogen molecule(s) of ammonia (NH_3) in the hydrocarbon and the structure of this organic compound is $R - NH_2$. They can be aliphatic (straight chain or branched), alicyclic (closed ring) or aromatic (closed ring and exhibit a number of substantial peculiarities of their own) depending on the types of hydrocarbon involved (Gooch, 2010). Figure 2.5 shows the curing reaction of the epoxy resin and amines. In the case of aliphatic amine, the R is an alkyl group while for aromatic amine, the R is aryl group. Alicyclic amine is similar to those simple alkyl amines, but it exhibit hazardous properties. Aliphatic is commonly used for room temperature curing due to its rapid reaction with epoxy resin. In the other words, aliphatic amine has the shortest pot life. While for aromatic amine, the steric hindrance of the aromatic ring allows slow curing in the room temperature. Heat is normally involved to accelerate the curing. As compared with aliphatic amine, aromatic amine gives an excellent heat resistance and chemical resistance due to its aromatic structure (Licari & Swanson, 2011).

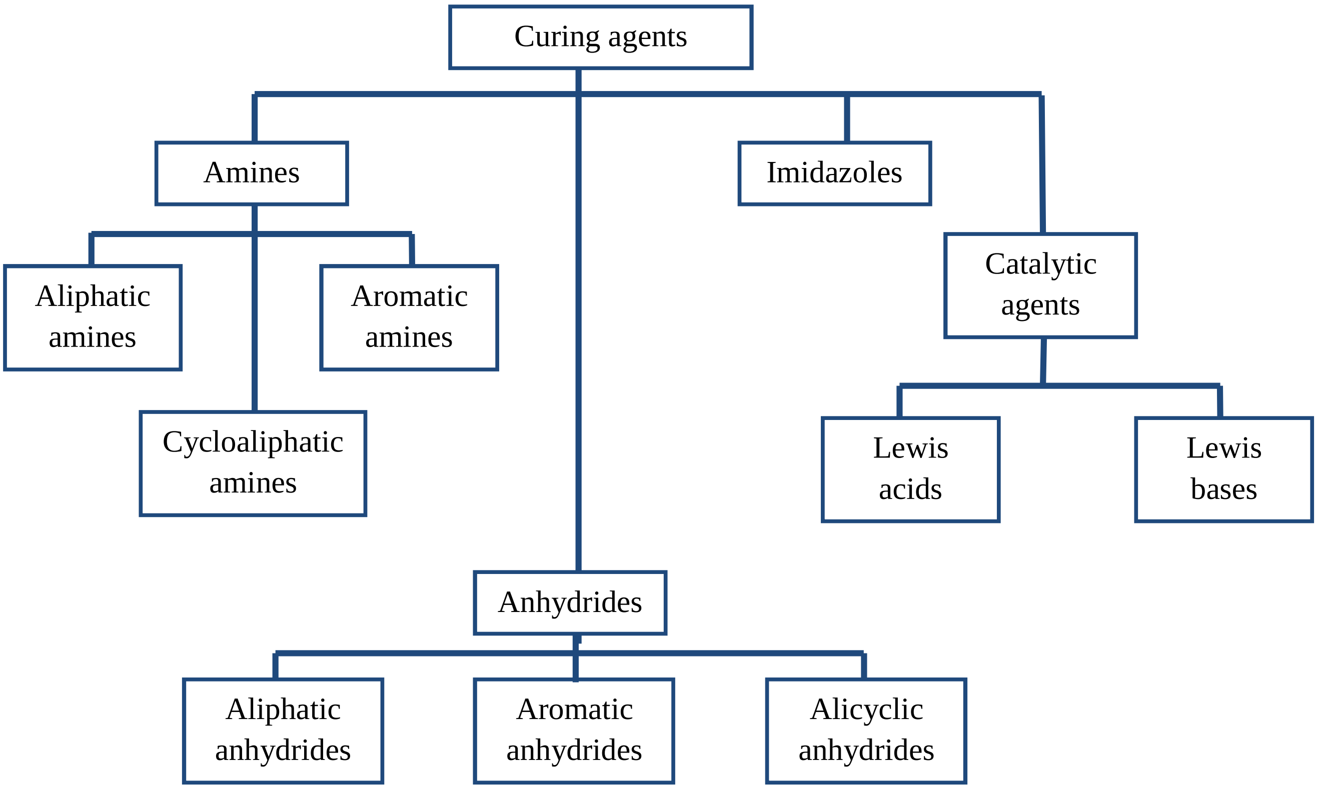
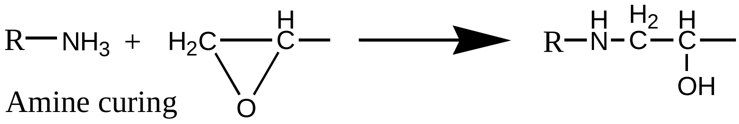

Curing agents
Amines
Imidazoles
Catalytic agents
Aliphatic amines
Aromatic amines
Cycloaliphatic amines
Lewis acids
Lewis bases
Anhydrides
Aliphatic anhydrides
Aromatic anhydrides
Alicyclic anhydrides

Figure 2.4. Categories of curing agents

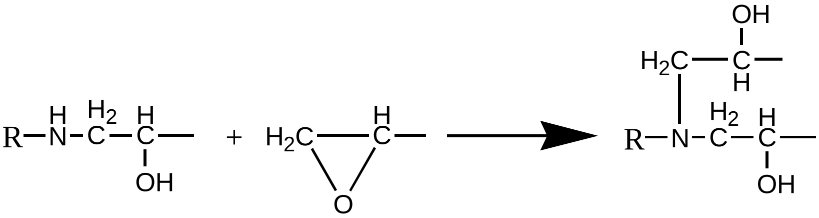
Anhydride curing agents which derived from a diacid by dehydration are also used extensively for the curing of epoxy resins (www.scribd.com). Figure 2.6 shows the curing mechanism of epoxy by anhydride curing agents. In contrast with amine, anhydrides are less reactive, therefore the curing of the epoxy by anhydrides is slow, and temperature of curing at around 150 – 170^{o}C is required for at least 2 hours

(Campbell, 2004). However, the curing rates can be accelerated with the addition of catalytic curing agents such as tertiary amine (Jin et al., 2006).



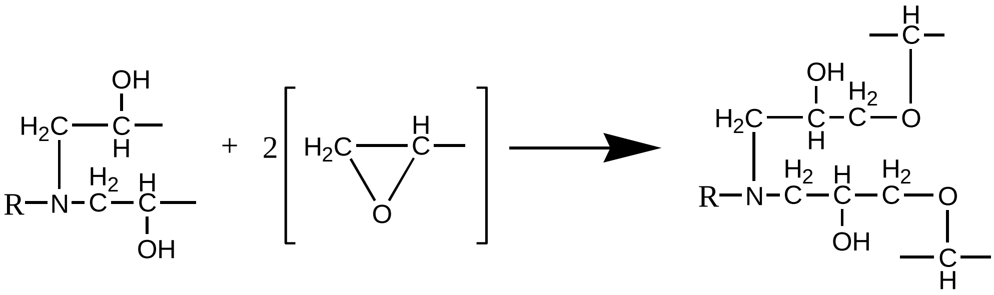
Epoxide group from
epoxy

Formation of secondary amine and more hydroxyl groups



Epoxide group from
epoxy

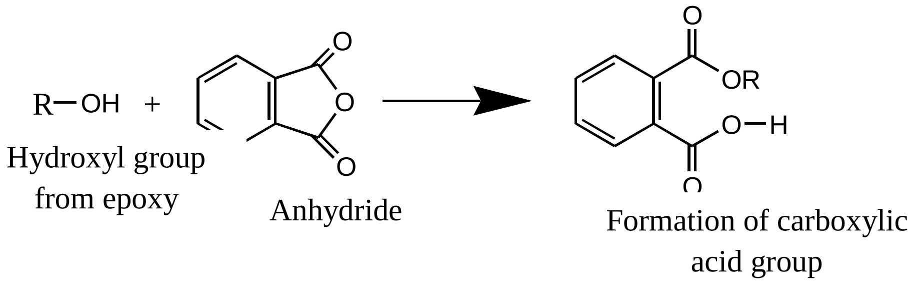
Formation of tertiary amine



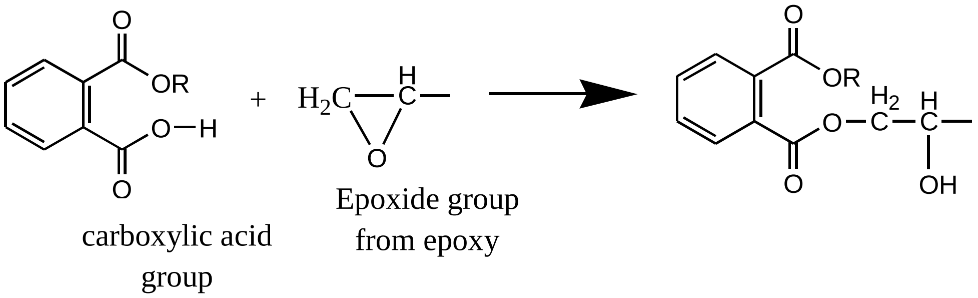
Epoxides group from
epoxy

Cross-linking through reaction of hydroxyl groups

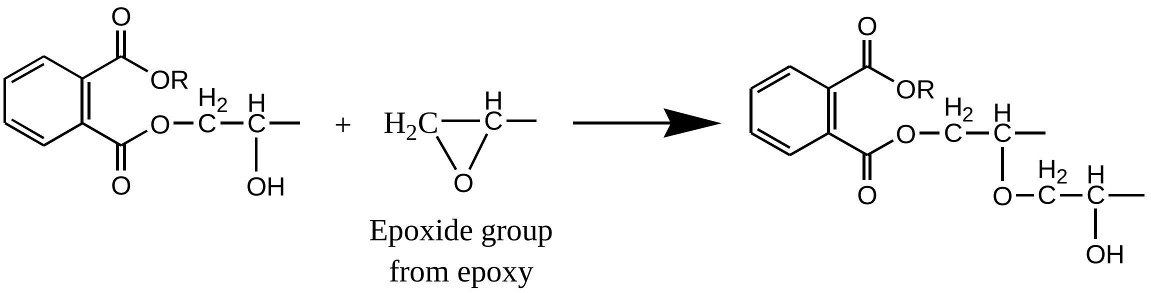
Figure 2.5. Curing mechanism of epoxy by amine curing agents (Licari & Swanson, 2011)


R—OH +
Hydroxyl group
from epoxy
Anhydride
O
O
O
OR
O—H
O
O
Formation of carboxylic
acid group

Opening of anhydride ring by hydroxyl group from epoxy to form carboxylic acid group


O
OR
O—H
O
carboxylic acid
group
+
H₂C—C—
H
O
Epoxide group
from epoxy
O
OR
O—C—C—
H₂ H
O
OH

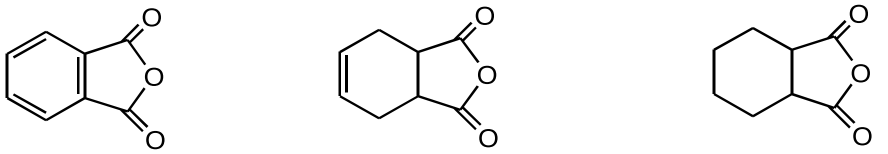
Etherification of hydroxyl group by epoxide group from epoxy


O
OR
O—C—C—
H₂ H
O
OH
+
H₂C—C—
H
O
Epoxide group
from epoxy
O
OR
O—C—C—
H₂ H
O
O—C—C—
H₂ H
OH

Reaction of hydroxyl group with epoxide group from epoxy

Figure 2.6. Curing mechanism of epoxy by anhydride curing agents (Licari & Swanson, 2011)

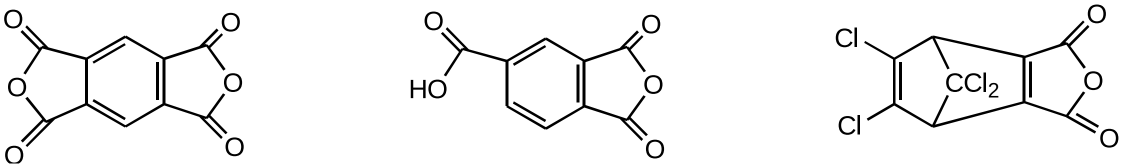
There are three types of anhydrides which are aromatic anhydrides, alicyclic anhydrides and aliphatic anhydrides. The solid form of aromatic anhydrides is commonly used in powder paints for powder molding, while the solutions in liquid anhydrides are used in insulating coating (Three Bond Technical News, 1990). For the application with epoxy resins, alicyclic anhydrides shown in Figure 2.7 are normally used. Polycarboxylic anhydrides are one of the example of aliphatic anhydrides. It is formed through the dehydration condensation reaction between aliphatic dibasic-acid molecules. The cured product by these anhydrides gives excellent flexibility and thermal shock resistance (Kulshreshtha & Vasile, 2002; Goosey, 1999).



Phthalic anhydride

Tetrahydrophthalic anhydride

Hexahydrophthalic anhydride



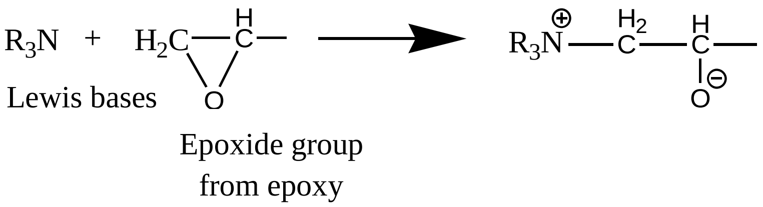
Pyromellitic dianhydride

Trimellitic anhydride

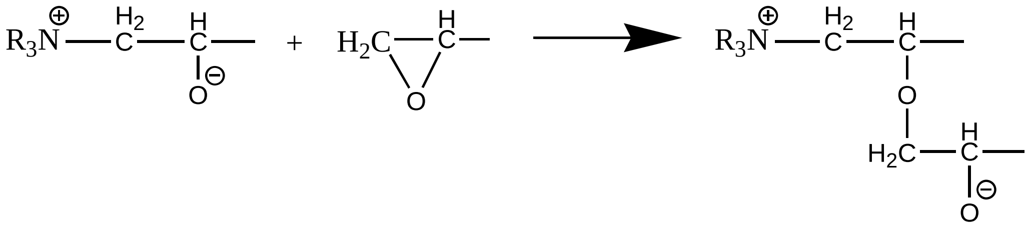
Chlorendic anhydride

Figure 2.7. Types of alicyclic anhydrides commonly used with epoxy resin (Lubin & Peters, 1998).

Catalytic curing agents are actually Lewis bases and Lewis acids (Craver & Carraher, 2000). Compounds that contain a free pair of electrons are Lewis bases, such as tertiary amines. While for Lewis acids, they are compounds that accept a pair of electrons, for example, boron trifluoride. Lewis acids is very reactive, highly volatile and toxic, this gives very short pot life. However, the complexes of Lewis acids and bases are very stable, until it been heated to certain temperature for the dissociation of complexes into reactive species for curing mechanism (Licari, 2003; Lubin & Peters, 1998). However, the polymerization reactions by the dissociation of these complexes are actually similar to those curing of epoxy resin by tertiary amine, as shown in Figure 2.8.



(a) Ring opening



(b) Cross-linking

Figure 2.8. Polymerization reaction of epoxy by Lewis bases (a) ring opening, (b) cross-linking (Licari & Swanson, 2011).

Imidazoles, the strong organic bases and good nucleophiles are the most common curing accelerator or co-curing agents that used in epoxy adhesives for electronic application. Today, imidazoles have become an optional curing agent for time constraint application (Hamerton, 1996). The curing mechanism of epoxy by imidazoles is shown in Figure 2.9. In the beginning of homopolymerization of epoxy resins by imidazoles, zwitterion is formed which rearranges to 1:1 adduct by internal proton transfer. This etherification reaction occurs through the first nucleophilic attack

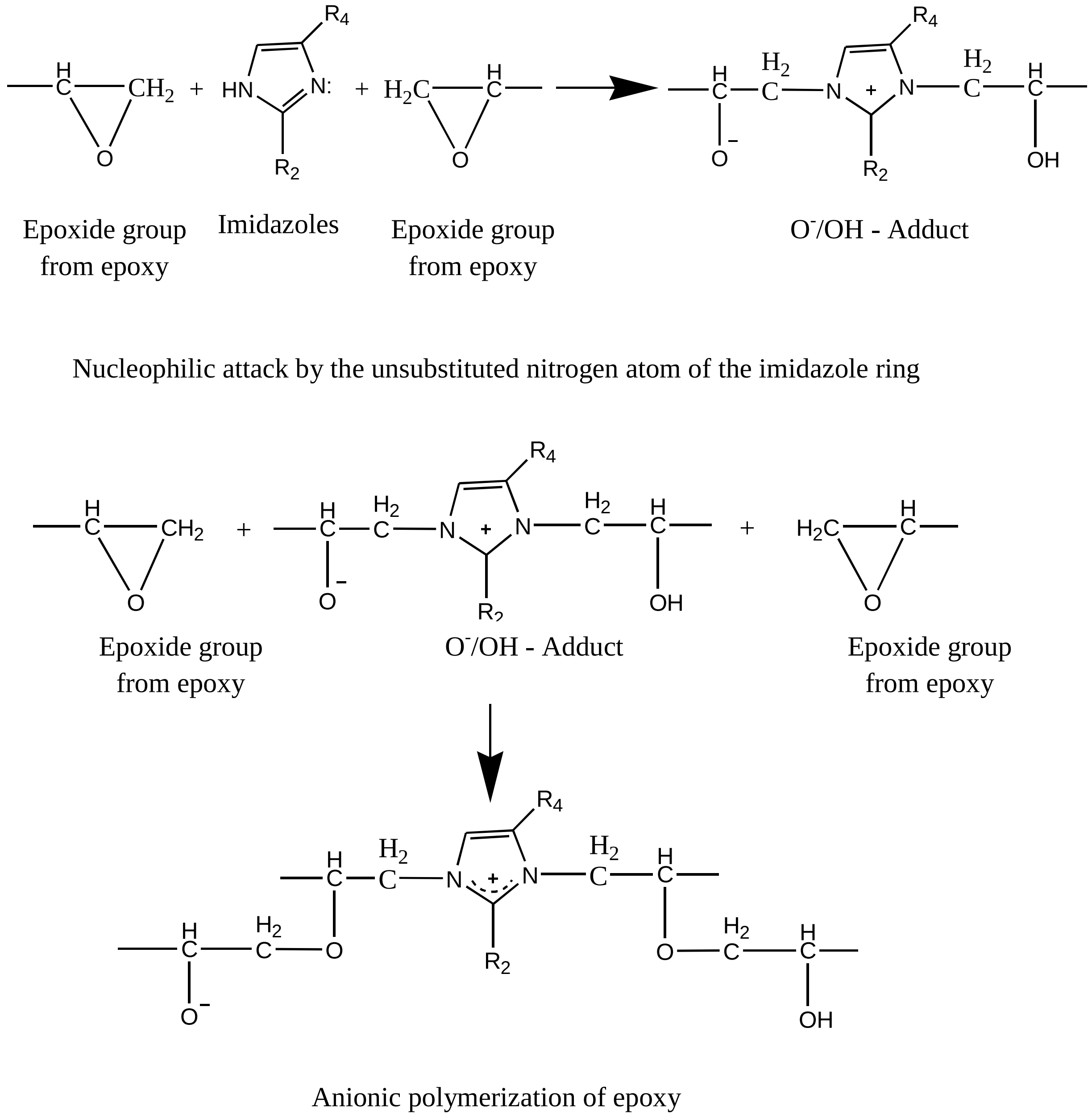

Epoxide group from epoxy
Imidazoles
Epoxide group from epoxy
O⁻/OH - Adduct
Nucleophilic attack by the unsubstituted nitrogen atom of the imidazole ring
Epoxide group from epoxy
O⁻/OH - Adduct
Epoxide group from epoxy
Anionic polymerization of epoxy

Figure 2.9. Curing mechanism of epoxy by imidazoles (Omrani et al., 2008).

by the unsubstituted nitrogen atom in imidazole ring. The newly formed unsubstituted nitrogen is then going through nucleophilic reaction, and a second epoxy group is opened to give the 2:1 adduct. These adducts generations are the slow initial rate of epoxy conversion. Polyetherification reaction are then initiated by the 2:1 adduct which are able to promote the anionic polymerization of the epoxy (Cognard, 2005). In the other words, the polymerization process are believed to be catalyst by the 2:1 adduct which contains the nucleophilic alkoxide ion (Heise & Martin, 1989; Rabilloud, 1997).

2.2 Polymer Composite in Electronic Packaging

2.2.1 Introduction

The demand trend on the small size and high performance electronics has driven changes in the electronic packaging requirements. This has raised issue such as reliability, weight-critical system, warpage of the IC chips and package, cost, and poor heat management which are the major failure factor of the electronic devices (Huang et al., 2006). Therefore, the innovation of the technologies had expanded the application of polymer other than used for insulation purpose, and brought further into the electronic packaging throughout the three packaging level (Lupinski & Moore, 1989; Thompson et al., 2004). However, properties of the polymer itself are still far to meet the packaging design requirements. Hence, polymer composite materials which have tailorable properties are created especially for this electronic packaging.

Glass fiber reinforced polymer (GFRP), the well established packaging materials has replaced the bakelite and masonites in printed circuit board. Application of GFRP has solved the weight and reliability issues in the electronic packaging (http://www.4pcb.com/pcb/). Nevertheless, this polymer composite is only utilized in the second and third level of packaging hierarchy. In order to continue solving the packaging issue arises in line with the miniaturization especially in the first level of packaging hierarchy, new composite materials which distinguish them from the GFRP is developed. This packaging material is known as advanced composites (Chung, 1995). The choice of the fillers, size and shape of the fillers are taking into the account when it's come to the manufacturing of advanced composite for first level of packaging hierarchy. Many research works have been done with the incorporation of suitable ceramic and metal fillers in this advanced composite to make the properties such as dielectric, electrical conductivity, thermal conductivity, coefficient of thermal expansion (CTE) and some mechanical properties tailorable to the required value (Bae et al., 2004; Ho et al., 2008; Hodgin & Estes, 1999). Moulding compound, thermal interface materials and underfills are the most common application of these advanced composites (Kim & Bae, 2004, Yu et al., 2007; Lee & Yu, 2005).

2.2.2 Factors Influence Polymer Composite

Polymer composites are the multifunctional materials which their properties can be tailored to meet the requirement of the electronic packaging. It is generally known that polymer matrices are weak, low stiffness and viscoelastic materials. They have very poor mechanical properties, low thermal and electrical conductivity (Harper, 2002). The properties of the polymer composites are contributed by the fillers or reinforcements that incorporated into it. Their properties are strongly dependent on the types of fillers or reinforcements, volume fraction or loading, dispersion as well as the microstructural of these fillers or reinforcements (Tong, 2010).

2.2.2.1 Types of Fillers

Depending on the required properties, types of fillers are normally the first thing to concern when it comes to the materials selection for the fabrication of polymer