Failure analysis of tamper resistant materials for IC packaging

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**NANYANG
TECHNOLOGICAL
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**Failure Analysis of Tamper Resistant Materials for IC
Packaging**

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SCHOOL OF MATERIALS SCIENCE AND ENGINEERING

2019

Failure Analysis of Tamper Resistant Materials for IC Packaging

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SCHOOL OF MATERIALS SCIENCE AND ENGINEERING

A thesis submitted to the Nanyang Technological
University in partial fulfilment of the requirement for
the degree of Master of Engineering

2019

Statement of Originality

I hereby certify that the work embodied in this thesis is the result of original research and has not been submitted for a higher degree to any other University or Institution.

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Abstract

In recent years, the surge in counterfeit products in the microelectronics industry has escalated to problems such as losses and damages of companies' reputation and consumers' safety and confidence. With the aim to protect products and devices from attackers to obtain their circuit design, intellectual property (IP) or even secured data, anti-tampering (security) features have been gaining great interest in companies as well as government bodies. With equipment technology advancement, attackers may utilize them to tamper with the devices to retrieve information in an unorthodox manner. Therefore in this report, the motivation for tamper resistant material for IC packaging is first elaborated, follow by literature review conducted from types of integrated circuits (IC) packaging available on the market to the packaging materials, steps taken to produce counterfeit IC and analysis of the internal structure by removal of packaging materials with different techniques (e.g. laser decapsulation). In addition, the rationale of focusing on laser utilization will be mentioned and lastly, the fundamentals of laser and the laser applications in microelectronics will be elaborated.

Experiments are conducted on commercial packages and in-house formulated epoxy resin, Glycidyl-POSS (GP), either with or without carbon black/surface treatment on fillers. The components that formed GP, sample preparation processes, laser parameters and the characterization tools utilized will be described. Different types of GP samples prepared are characterized. Images of these GP samples after laser ablation are captured and step profiler is used to quantify the etch depth for analysis.

The resistance to laser ablation is obtained, analyzed and discussed. From the data, it is postulated that due to the strong light absorptivity of carbon black, GP samples which do not contain carbon black but with fillers show better resistance to laser ablation in contrast to samples that contain both carbon black and fillers. In addition, the relationship between adhesion of the matrix and fillers to improve stress transfer

and its effect on the extent of laser ablation is also studied. Amino silane treated fillers added into GP matrix were used to fabricate good matrix-filler adhesion samples while Glycidyl silane treated fillers added into GP matrix produced samples with poor adhesion. More details of the results and analysis will be explained in this report.

Overall, samples without carbon black showed superior performance, being almost laser resistant. This indicates that the carbon black plays the key role as the light absorber and if the material is designed to not absorb any of the laser light, the damage incurred will be very minimal. In the case where colour pigment such as carbon black cannot be omitted (for blocking light transmission) in the device encapsulating process, it is important to reduce air gaps due to poor adhesion between matrix and fillers or pores/voids as all these defects act as the point of weakness which hinder stress transfer, or to find alternative colour pigments which would absorb the laser light less efficiently.

Lay Summary

As technology advances, machines usage becomes an essential part of humans' everyday life. Some examples include smartphones, credit cards and personal laptops. These machines, regardless big or small, require microelectronic devices to power and provide them with respective functionality. The microelectronic device in credit cards store pin or monetary information while the microelectronic devices in smartphones allow communication through messaging or phone calls, storing of captured images with the phone camera and etc. With information stored in the microelectronic devices, users are able to make purchases, retrieve messages or images instantly, at any time.

Methods to obtain sensitive information are often eyed by unauthorized personnel as they would bring about numerous monetary benefits. For instance, stealing credit cards' details and transferring victim's money unknowingly or to the extent of stealing the circuit design of the device, replicating it illegally and selling them as the official products to consumers. The attackers may make use of the techniques developed by the industries for failure analysis to open up the microelectronics devices and retrieve those data.

In this work, the main aim is to understand the factors affecting the method to decapsulate the microelectronic devices from electronic packages, so that possible material designs to make the opening process more tedious or difficult, in other words hindering any unauthorized person to easily access the data stored in the device, can be proposed.

This would aid in the future development of materials for protecting devices that require security features so as to reduce the threats that are currently faced by the community or economy.

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Table of Contents

Abstract	i
Lay Summary.....	iii
Acknowledgements	v
Table Captions	ix
Figure Captions.....	xi
Abbreviations	xv
Chapter 1 Introduction	1
1.1 Background	2
1.2 Problem Statement	3
1.3 Objectives and Scope	5
1.4 Dissertation Overview	6
1.5 Findings and Outcomes	7
References	8
Chapter 2 Literature Review	9
2.1 Integrated Circuits	10
2.2 Encapsulant Materials	13
2.3 Types of Threats and Tampering.....	17
2.4 Countermeasures against Tampering	20
2.5 Decapsulation Methodologies	22
2.5.1 Wet Chemical Etching.....	22
2.5.2 Mechanical Milling	23
2.5.3 Plasma Etching	24
2.5.4 Laser Decapsulation	25
2.6 Laser Fundamentals and Application in Microelectronics	26
2.6.1 Introduction	26
2.6.2 Types of Laser	28
2.6.3 Laser Selection for Decapsulation Application [19]	31
2.6.4 Laser's characteristics and ablation mechanisms	36
2.6.5 Conclusion.....	43
References	45

Chapter 3 Experimental Methodology	49
3.1 Materials.....	50
3.2 Sample preparation.....	52
3.3 Testing and Characterization Tools.....	55
3.2.1 Laser System	55
3.2.2 Surface Profiler	56
3.2.3 Thermogravimetric Analysis (TGA)	57
3.2.4 Fourier Transform Infrared spectroscopy (FTIR)	58
3.2.5 Ultraviolet Visible Near-Infra red (UV-Vis-NIR) spectroscopy	59
3.2.6 Scanning Electron Microscopy (SEM).....	61
References	65
Chapter 4 Results and Discussion	67
4.1 Study on GP encapsulant material.....	68
4.1.1 Sample characterizations	68
4.1.2 Laser Ablation on GP and commercial IC packages	74
4.2 Factors affecting Laser Ablation	88
4.2.1 Laser light absorption.....	90
4.2.2 Adhesion between Matrix and Fillers	91
4.3 Summary	94
Chapter 5 Conclusion and Future Work	97
5.1 Conclusion	98
5.2 Future work.....	98

Table Captions

Table 2.1: Functions of the Components of Epoxy Molding Compounds[5]	15
Table 2.2: Areas of laser application	28
Table 2.3: Types of laser and their applications [25]	30
Table 3.1: SiO ₂ fillers surface treatment.....	53
Table 3.2: Specifications of the SESAME 1000 laser system.....	56
Table 4.1: Selected samples for FTIR analysis	71
Table 4.2: Molding compound datasheet of XL package [6]	73
Table 4.3: Laser ablation parameters for position 1 to 10 (refer to XL Package above)	75
Table 4.4: Laser ablation parameters for position 1 to 10 (refer to LG Package above)	75
Table 4.5: Laser ablation parameters for position 1 and 2, a to j (GP samples T1 to T3)	77
Table 4.6: Average etched depth (μm) of 5 different samples (XL, LG, GP-T1, GP- T2 and GP-T3) under different frequency when subjected to 100% laser power and 900 mm/s marking speed for 1 pass	78
Table 4.7: Average etched depth (in μm) of 5 samples (XL, LG, GP-T1, GP-T2 and GP-T3) under different frequency when subjected to 100% laser power and 900 mm/s marking speed for 5 passes	79
Table 4.8: GP T1 (With carbon black and no fillers)	86
Table 4.9: GP T2 (With carbon black and fillers)	86
Table 4.10: GP T3 (With fillers but without carbon black)	86
Table 4.11: Average amount of material removed (mg) of respective samples	89
Table 4.12: UV-Vis-NIR of respective samples at 1064 nm.....	90
Table 4.13: Average die shear force (g) for respective samples	92

Figure Captions

Figure 2.1: Three levels of packaging architecture [2]	10
Figure 2.2: Schematic of a typical IC device [3]	11
Figure 2.3: Types of plastic and ceramic through-hole mount packages [4]	11
Figure 2.4: Types of plastic and ceramic surface mount packages [4]	12
Figure 2.5: Categories of packaging technology [5]	13
Figure 2.6: Contents of a typical epoxy molding compound by weight percent [5]	14
Figure 2.7: Graph of CTE vs fused silica filler content [7]	16
Figure 2.8: Viscosity as a function of silica filler loading [9]	16
Figure 2.9: X-ray images of (a) a device without XBS material protection and (b) same device with XBS material protection [15]	21
Figure 2.10: IC package with wire mesh/reactive membrane installed [12]	21
Figure 2.11: IC Package (a) before and (b) after manual chemical decapsulation [17]	23
Figure 2.12: Cross section schematic drawing of (a) milled package and (b) milled and jet etch package [18]	24
Figure 2.13: Schematic drawing of plasma decapsulation of package in plasma chamber [18]	25
Figure 2.14: IC package (left) after laser precavitated and (right) followed by jet etching [20]	26
Figure 2.15: Difference between ordinary light and laser light [24]	27
Figure 2.16: Absorption, transmission and reflection of a laser beam when it strikes onto a material [26]	28
Figure 2.17: Types of laser and their wavelengths [28]	29
Figure 2.18: Absorption of three common PCB materials for six types of lasers [31]	33
Figure 2.19: Recommended laser applications at different pulse duration (from fs to CW) and wavelength (IR to UV) [32]	34

Figure 2.20: The effects of a long-pulse laser compared to an ultrashort pulse laser during ablation [33].....	35
Figure 2.21: Features machined with nanosecond /picosecond pulse laser at different wavelength on Polyethylene terephthalate (PET) [32].....	36
Figure 2.22: Pulse emission for (a) CW laser and (b) Pulse laser [28].....	37
Figure 2.23: Schematic of the processes involved in ns laser ablation in air, with an approximate timeline of their occurrence [37].....	41
Figure 2.24: Laser ablation of holes drilled in a steel foil with (a) ns irradiation (b) fs irradiation [39]	43
Figure 3.1: Structure of Glycidyl-POSS EP0409 [1].....	50
Figure 3.2: Silane Coupling Agents Mechanism between inorganic and organic component [5]	51
Figure 3.3: Chart showing the effectiveness of silane on respective substrates [6].....	52
Figure 3.4: Molded sample (a) GP matrix and (b) GP matrix with carbon black...	53
Figure 3.5: Process flow of surface treatment using silane on silica fillers	54
Figure 3.6: Chemical structure of silica surface after amino-silane and glycidyl/epoxy-silane treatment [7]	54
Figure 3.7: FESEM image of (a) GP matrix + amino silane treated SiO ₂ and (b) GP matrix + glycidyl silane treated SiO ₂	55
Figure 3.8: Schematic of diode pumped Nd:YAG laser with Q-switch [8].....	56
Figure 3.9: Basic operating principle of a surface profiler	57
Figure 3.10: Schematic of TA Instrument Q500 TGA [10].....	58
Figure 3.11: Types of molecular vibrations [13]	59
Figure 3.12: Internal components of a UV-Vis-NIR Spectroscope [14]	60
Figure 3.13: UV to NIR wavelengths [16].....	60
Figure 3.14: Basic working principle of a UV-Vis-NIR Spectroscope [17].....	60
Figure 3.15: Schematic of a typical scanning electron microscope and its internal components [19].....	62
Figure 3.16: Interaction volume and signal from electron microscopy [19]	62
Figure 3.17: Relationship between aperture angle and depth of focus [20]	63

Figure 3.18: Schematic of bond shear test setup.....	64
Figure 4.1: (a) Setup for TGA test with GP sample, (b) GP sample retrieved after TGA test (c) Magnified optical image of GP sample after TGA test	68
Figure 4.2: SEM images of GP sample (a) before and (b) after thermal decomposition at 200x	69
Figure 4.3: TGA Graph of GP heating from ambient to 800°C	70
Figure 4.4: FTIR spectra of Package LG and XL	71
Figure 4.5: FTIR spectra of GP with carbon black and GP with carbon black & Al ₂ O ₃	72
Figure 4.6: FTIR spectrum of Al ₂ O ₃ fillers	73
Figure 4.7: XL and LG packages before and after laser ablation with respective parameters at locations 1 to 10	74
Figure 4.8: 3 types of GP samples before laser ablation (top row) and after laser ablation at specific locations (bottom row) and their respective compositions	76
Figure 4.9: Surface measurement profile for each ablated area	77
Figure 4.10: (a) Graph of etch depth against Q-switched frequency for 1 laser pass and (b) Optical IR image of GP T1 sample (left) and GP T2 sample (right) after 1 laser pass at 100% Power, 20 kHz frequency	79
Figure 4.11: (a) Graph of etch depth against Q-switched frequency for 5 laser passes and (b) SEM image of a localized region in one of GP T2 sample after 5 laser passes.....	80
Figure 4.12: (a) to (f) Laser ablation process (scanning from left to right) for GP (T1) sample in 1 laser pass at different interval.....	82
Figure 4.13: Laser ablation process (scanning from left to right) for GP (T1) sample at (a) 0 pass (b) 1st pass (c) 2nd pass (d) 3rd pass (e) 4th pass (f) 5th pass.....	83
Figure 4.14: (a) to (f) Laser ablation process (scanning from left to right) for XL sample in 1 laser pass at different interval.....	84
Figure 4.15: UV-Vis-NIR spectrum of GP samples T1, T2 and T3.....	85

Figure 4.16: Enlarged image of 2f region, after 5 passes of ceramic laser cut on (a) GP T2 sample and (b) GP T3 sample	87
Figure 4.17: Samples utilized in this part of work	88
Figure 4.18: Scatter graph on amount of material removed (mg) of different samples laser ablated at 1 x 30P and 30P	89
Figure 4.19: (a) Test samples were fixed onto the glass slide for die shear test and (b) schematic cross-section of an individual sample, (c) die shear test setup and (d) die shear process	92
Figure 4.20: Material properties of epoxy and common fillers [19]	94
Figure 4.21: SEM images of (a) and (b) glycidyl silane treated silica in GP matrix showing poor adhesion and, (c) and (d) amino silane treated silica in GP matrix showing good adhesion.....	95

Abbreviations

APTMS	3-aminopropyltrimethoxysilane
CTE	Coefficient of Thermal Expansion
CW	Continuous Wave
DSO	DSO National Laboratories
FA	Failure Analysis
fs	Femtosecond
FTIR	Fourier Transform Infrared Spectroscopy
GP	Glycidyl-POSS
GPTMS	3-glycidyloxypropyl)trimethoxysilane
IC	Integrated Circuit
IR	Infra-red
ns	Nanosecond
Nd:YAG	Neodymium: Yttrium-aluminum-garnet
PCB	Printed Circuit Board
ps	Picosecond
RE	Reverse Engineering
SEM	Scanning Electron Microscopy
TGA	Thermogravimetric Analysis
UV-Vis-Nir	Ultraviolet Visible Near-infrared spectroscopy

Chapter 1

Introduction

Anti-tamper encapsulation materials have been gaining much interest because of the increase in threats which include counterfeiting, reverse-engineering (RE) and theft (hardware theft and cloning). This leads to the need to implement anti-tampering feature(s) in the ICs to either provide a physical barrier to prevent unauthorized personnel from inspecting or tampering with the underlying circuitry and content. In this chapter, the background will first be elaborated, followed by problem statement, objectives and scope, dissertation overview and lastly, the summary of findings and outcomes in this work.

1.1 Background

Integrated circuits (IC) packages are widely utilized in daily lives, ranging from small scale applications such as personal mobile phones to large scale applications including communication systems (satellite), aerospace industries (airplanes) and in defence systems. Technology advancement raises the requirement of increasing sophisticated manufacturing facility to fabricate devices with decreasing dimensions, resulting in an exponential surge in the cost of equipment that reaches billions of dollars. Under such circumstances, chip manufacturers are forced to seek alternatives such as collaboration with external parties to produce their circuits, splitting the production system into two different entities, namely ‘designers’ and ‘foundries’. The firms who are mainly focusing on the circuits designing are usually known as ‘designers’, while ‘foundries’ are the manufacturing companies that own the means of production. Despite initial agreements on collaboration, the original terms of use may be disregarded and the design may end up being misused by the foundry or the system integrator. Over the decade, there have been increasing threats faced by the designers of IC over intellectual properties (IP), which include counterfeiting, reverse-engineering (RE) and theft (hardware theft and cloning). In between 2001 and 2011, the amount of counterfeit electronic circuits collected by U.S. Customs had increased over 700 times. Moreover, U.S. Customs confiscated 5.6 million counterfeit electronic products between 2007 and 2010. Overall, approximately around 7% of counterfeiting is circulating in the semiconductor market, resulting in a loss of about \$22 billion for the lawful industry in 2014.

Acts of products, components and designs piracy have induced enormous damage on the economy, constantly breaking new record levels and victimizing companies through pirating and illegal copying of their products. Without any expenditure on research and development processes, pirated goods can be produced and sold cheaply, creating distortion in markets. The adverse effects on the companies range from market shares loss, damage to a company’s reputation to job losses. As a

consequence, this put an instantaneous burden on the economy. However, not only firms are experiencing the impact, consumers are also affected by creating a fake impression of them purchasing a brand name product. These products, however, are unreliable counterfeits with questionable safety and reliability.

A system can be defined by several means, whether it is just a silicon chip to an entire electronic board or from simple logic gates to an IP core. There is always a potential access point that an attacker can enter to obtain the system design or information and tamper with the devices, especially those operating in defence and aerospace systems in ways that are undetectable and thereby manipulate or defeat the key defence systems. The strategy should, therefore, provide safeguarding of the most critical ICs used in intelligence, communication systems and most importantly, sensitive weapons. This leads to the need to implement anti-tampering feature(s) in the IC to either provide a physical barrier to prevent inspection or tampering with the underlying circuitry and content. It is especially significant for applications which require storing and processing of private or highly sensitive information. [1-3]

1.2 Problem Statement

There are various methodologies (e.g. chemical, laser, milling) developed mainly for usage in Failure Analysis (FA) which attackers may exploit to tamper the IC package and retrieve the circuitry information and its content. Thus, having an anti-tamper feature implemented onto the IC package that contains sensitive or critical data is extremely important. This has led to increasing interest in companies to develop tamper resistant methodologies on IC and one of these techniques includes anti-tamper encapsulating materials. [4] Hence understanding the package removal mechanism(s) is essential for future development of anti-tamper packaging material. In this work, the study of the interaction between the laser and encapsulation material during laser ablation (one of the widely used FA decapsulation techniques) will be carried out.

The ablation (removal) of material by laser is likely to be affected by two factors:

- (1) Laser light absorption – It refers to the ability of the material's system to absorb the energy introduced by the laser source. Good absorption of the laser (influenced by the carbon black pigment) allows energy to be converted into other forms of energy (e.g. heat) causing ablation to occur.
- (2) Adhesion between matrix and fillers for stress transfer – It refers to the stress dissipation through the bonds between matrix and fillers at the interface. Poor adhesion between matrix and fillers is likely to result in air gaps which would become the point of weakness. This weakness would hinder stress transfer within material and result in poorer stress tolerance/strength. Figure 1.1 shows the schematic on how to improve the stress transfer by having good adhesion between fillers and matrix. Figure 1.2 shows examples of matrix-fillers adhesion without treatment and with aminosilane surface treatment of fillers. Poor contact can be observed between matrix-fillers in Figure 1.2(a) while good adhesion (bonded) can be observed in Figure 1.2(b).

Figure 1.1: Schematic of matrix and filler compatibility [5]

Figure 1.2: FESEM image of the contact between matrix and glass sphere fillers (a) without silane surface treatment on fillers and (b) with aminosilane surface treatment on fillers [6]

This thesis will validate the above claims through varying the colour pigment (carbon black) content mainly for the light absorption, and fabricating samples with poor adhesion and good adhesion between the matrix-fillers and analyzing their performance.

1.3 Objectives and Scope

As anti-tamper encapsulation materials against attacker have been gaining much interest, the aim of this project is to understand the interaction mechanism of the tamper resistant materials, Glycidyl-POSS (GP) (one of the in-house synthesized tamper resistant material developed by another project team for chemical resistance), when subjected to one of the decapsulation methodologies, mainly focusing on laser ablation with Nd-YAG laser operating at the wavelength of 1064 nm (which is widely used in Failure Analysis industries).

The scopes of work include comparison of commercial IC packages and GP after laser ablation, performing characterizations of GP with different test designs (varying three different contents of carbon black and types of surface treatments on fillers) and analyzing the components/designs influence on the laser ablation results.

1.4 Dissertation Overview

This thesis consists of the following:

Chapter 1 provides a background on how the counterfeit products in microelectronics industry have affected the industries and consumers leading to the interest in applying anti-tamper features on integrated circuit (IC) and the motivation/aim of looking into one of the tampering techniques, which involve the use of laser to remove the IC encapsulation (packaging) materials.

Chapter 2 reviews the literature on what an IC is composed of and the different levels of packaging. The focus will be on the first level of packaging (single chip module), and the role and importance of the encapsulating materials in packaging will be described. The types of threats, tampering/decapsulation methods to remove the IC packaging and countermeasures developed so far against the removal of packaging material are elaborated. As mentioned earlier, laser decapsulation will be the area of focus hence the literature review on laser fundamentals and their applications in microelectronic will also be conducted.

Chapter 3 consists of the raw materials information, the purpose of selecting the materials, the samples fabrication processes for this work and lastly the principles of the testing/characterization techniques or tools employed and the reason for using them.

Chapter 4 presents the characterization work done on GP resin matrix, elaborates and discusses the set of results obtained from various tests done on both the commercial packages and in house fabricated samples. Moreover, the analysis of the two factors (light absorption and adhesion between matrix and fillers) affecting the laser ablation will also be addressed. After which, the material design to achieve laser resistant will be suggested.

Chapter 5 concludes the overall work done for this thesis and gives a summary of possible future work.

1.5 Findings and Outcomes

The main objective of this thesis is to first find out how the encapsulation materials performed when subjected to laser ablation followed by understanding the interaction mechanism between the laser and the encapsulating materials. The suggested factors affecting the laser ablation (light absorption and adhesion between matrix-fillers) are investigated. The results have shown that once carbon black pigment is omitted in the sample formulation, the light absorption (%A) is reduced extensively from $\sim 95\%$ to $\sim 15\%$. By keeping it to a minimal amount, it has shown superior performance of resisting the laser ablation. In addition, the shear test shows that the samples with good matrix-fillers adhesion have superior shear strength ($\sim 40\%$ increase) as compared to samples with poor matrix-fillers adhesion. This indicates that good adhesion between matrix and fillers does enhance the stress transfer.

As there is no laser resistant material applied in the area of microelectronic encapsulation currently, the novelty of this work includes evaluating a new matrix material (GP) with different engineering design modifications towards resisting laser ablation so as to propose/provide an insight on the anti-tamper encapsulant material's design for future development, targeting for microelectronics. For instance, the first level of design is to avoid carbon black pigment to eliminate laser light absorption. Secondly is to couple with compatible matrix and fillers to have good bonding so as to reduce defects generated in the material. In the case where carbon black is still required, further work on improving the thermal or stress relaxation may be looked into.

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Chapter 2

Literature Review

This chapter first introduces the types of packaging in integrated circuits, the importance of packaging materials and their roles, followed by the types of threats faced; tampering methods and countermeasures developed against tampering will be highlighted. The decapsulation methodologies that can remove the packaging materials and the reviews on the laser fundamentals and applications in microelectronics (types of laser, laser mechanisms, etc) will also be elaborated.

2.1 Integrated Circuits

Integrated Circuits, also known as ICs, have revolutionized the electronics industry ever since their invention. They are miniature electronic circuits fabricated to perform different functionality, made of active and passive components such as bipolar transistors, field-effect transistors, diodes, resistors, capacitors and etc. [1] In addition, at least three levels of packaging architecture depicted in Figure 2.1 are required to power, interconnect, distribute signals, and cool the IC chips in an electronic equipment.

Figure 2.1: Three levels of packaging architecture [2]

Chip-level interconnections may be deemed as zero-level packaging. The first level packaging is where individual dies are electrically connected with bonding wires or solder bumps encased with encapsulant commonly known as molding compound to form a single package. The structure of a typical IC device is schematically illustrated in Figure 2.2.

Figure 2.2: Schematic of a typical IC device [3]

The types of single-chip packages are widely categorized into ceramic and plastic packages, and whether through-hole mount or surface mount is preferred (Figures 2.3 and 2.4) depends on their application or operating environment. The details for the package abbreviation can be found in Appendix 1.

Package Types				Package Types			
Plastic	DIP	Standard		Ceramic	Standard DIP
		Skinny			CER-DIP
		Shrink			PGA

Figure 2.3: Types of plastic and ceramic through-hole mount packages [4]

Package Types		
Plastic	SOP
	TSOP
	QFP
	TQFP
	LQFP	
	BGA/FBGA
	FLGA
Package Types		
Ceramic	HQFP

Figure 2.4: Types of plastic and ceramic surface mount packages [4]

Ceramic packages are known for their high reliability as they are able to withstand high temperatures and operate in harsh conditions. On the other hand, plastic packages (when compared to ceramic packages) are more popular because of the lower manufacturing cost and improved reliability in recent years. As for the type of mounting, through-hole packages are mounted by inserting lead pins into holes of the printed circuit board (PCB) followed by soldering and they are widely used in electronic equipment where there is no limitation on the board space or cost constraint. In contrast, surface mount packages have solder balls attached underneath or lead pins attached at the sides which are mounted directly onto the PCB by soldering, and they find wide applications in situations where double-sided IC packing are required. IC types (analog, digital, RF, sensor, and etc.) differ, so do their packaging needs and requirements. The encapsulate materials serve several important purposes, to protect IC chips from external environment, to maintain the electrical characteristics of the IC, to facilitate the handling of the brittle IC chips

and to dissipate heat generated by the IC chips. Without the encapsulation to shield the IC when subjected to different operating conditions as well as external factors such as heat, moisture and physical stress, the chip's functionality would be lost. Since an IC carries the circuitry for logic and memory which allows the IC package to function, the focus will be on first level packaging.

2.2 Encapsulant Materials

The importance of the encapsulant for an IC had been highlighted in the previous section. With reference to the type of encapsulation technologies, they can be classified into five main groups: molding compound, glob-top, potting, underfill and printing encapsulation materials (Figure 2.5). The most common way to package ICs is by enclosing chips within polymer molding compounds and over 90% of all IC packages are produced of the plastic variety.

Figure 2.5: Categories of packaging technology [5]

Resins (e.g. epoxy), curing agents (hardeners) and accelerators are the three main constituents in a molding compound. Epoxies are thermosetting polymers formed by chemical linking (cross-linking network) of the liquid branched polymers. The process of cross-linking is denoted as the curing where the material changes from soft to a hardened state when subjected to heat or ultraviolet (UV) light. In a molding compound, more than ten kinds of additives may be present which include fillers, flame retardants, stress-relief additives, surface treatment additives, colouring agents and so on. As epoxy resins are reported to have a high coefficient of thermal expansion (CTE) and low thermal conductivity, it cannot be used as the only main encapsulation source. The list of compositions inside a molding compound system is shown in Figure 2.6. Table 2.1 further describes these components with their functions and the respective agents in molding compounds.

Figure 2.6: Contents of a typical epoxy molding compound by weight percent [5]

Table 2.1: Functions of the Components of Epoxy Molding Compounds [5]

Component	Concentration (phr)	Major Function	Typical Agents
Epoxy resins	Matrix	Matrix for mold compound	Epoxy cresol novolac, biphenyl, dicyclopentadiene, multifunctional
Curing agents (hardeners)	Up to 70	Linear/cross polymerization	Polyphenol, amine, acid anhydride
Accelerators	Very low (<3)	Enhance rate of polymerization, reduce cycle and cure time	Amines, imidazole, organophosphine, urea, Lewis acids and their organic salts
Inert fillers	550–1,200	Reduce cost, decrease CTE, increase TC, reduce moisture absorption, reduce resin bleed	Silica (widely used), aluminum nitride, alumina
Coupling agents	Very low (<5)	Improve adhesion between organic and inorganic materials	Epoxy silanes, amino silane, titanate, aluminum chelate
Flame retardants	10	Retard flammability	Brominated epoxy, antimony oxide, phosphate ester, solid phosphate, aluminum trihydrate
Stress-relief agents	Up to 25	Reduce stress in package, improve toughness, provide thermal shock and impact protection	Silicone, butyl rubber, aliphatic epoxide, polybutyl acrylate
Mold-release agents	Very low (<3)	Aid in release of package from mold	Stearic acid, natural wax, synthetic wax
Coloring agents	5	Reduce photonic activity, reduce device visibility	Carbon black

CTE: Coefficient of thermal expansion; phr: Parts per hundred epoxy resin; TC: Thermal conductivity.

The additives have their specific role to play during the encapsulation process, contributing to the usability of the package encapsulation. Fillers such as silica which has a typical loading of 65 - 85%, for instance, [6] are used to lower both shrinkage on curing and CTE (Figure 2.7) and enhance the thermal conductivity of the molding compound. By carrying out this process, the stress from the thermal mismatch will be lowered between the packaging material and die or other components embedded inside the IC package, and thus improving reliability of the electronic package.

Figure 2.7: Graph of CTE vs fused silica filler content [7]

However, it is important to note that adding more fillers to increase the filler content will have drawbacks such as degrading the flow (having high viscosity) (refer to Figure 2.8) and modulus of the material [8]. To address this issue, formulators have fine-tuned other ingredients to maintain flow and modulus, one example is to lower polymer melt viscosity. However, lowering the viscosity of polymer melt reduces process margin.

Figure 2.8: Viscosity as a function of silica filler loading [9]

Overall, no approach is ideal for all applications and formulation largely depends on the demands of each application [8]. The user has to consider the advantages and disadvantages of each constituent so that the most suitable formulation can be determined.

2.3 Types of Threats and Tampering

As mentioned in the earlier section, the common encapsulation materials help to maintain the functionality of the chip but they are prone to the security threats faced in the microelectronic industries such as counterfeiting, theft and reverse engineering (RE) where attackers could easily “open” the packages to access the chip information.

Counterfeiting exists in three categories, “Relabelling”, “Refurbishing” and “Repackaging”. [10] The intention of relabelling is to create a false impression of the device, leading consumers into believing the products to be from another supplier or of a better grade. Relabelling is achieved by removal of serial numbers or identifiers followed by replacing them with others. For instance, one application can be to make a chip that does not fulfil military specifications to act as it does, or advertise an incorrect computing frequency (e.g. giving a much higher value than the actual). Counterfeiting by ‘refurbishing’ refers to reintroducing abandoned products into the market. It includes reselling old parts as new parts by aesthetically improving them to look new. ‘Repackaging’, is the third form of counterfeiting where the IC is removed from its original package and inserted into a different one. The purpose may be to alter the pins quantity or to use packaging of smaller size. In the process of repackaging, the final circuit may be modified causing malfunction. The reason being is that repackaging is usually handled under poor handling conditions. For example, pins could be damaged or broken, or the top passivation layer could be scratched. All these practices mislead the end user into purchasing products that fall below their expectations.

‘Theft (hardware theft and cloning)’ comes in three forms. First, the attacker, who is the foundry or system integrator, uses ‘overbuilding’ to produce more of the devices than originally specified by the designer. Second, the attacker illegally clones the design data and this can happen when an untrustworthy or unauthorised person obtained a copy of the design. Third, hardware parts, such as masks or ICs, may be plagiarized to build more devices. A serious consequence of theft is building extra circuits without the knowledge of rightful owner and claiming the design as his own to gain profit when sold.

Reverse Engineering (RE) is a major issue for both IC designers and IP rights owners because it allows the investigation on the device working mechanism [11] and some companies specialize in providing such services. RE basically subjects the device to undergo all kinds of tests and extracts the components according to how the device responds to the tests. ‘Test’, is a general descriptor for all methods available in the investigation process. RE can be either ‘invasive’, where the device can no longer function after testing, or ‘non-invasive’ which allows the device to maintain its integrity after the tests. Invasive tests proceed with partial or full removal of the chip package for exposure of the silicon die. A range of decapsulation techniques is available for selection, depending on the package type and the requirements for further analysis. For microcontrollers and smartcards, partial decapsulation is commonly used so that the device can be tested in a standard programmer. Invasive RE can lead to two results: (1) recovering the register-transfer level (RTL) schematic of the IC, or (2) locate the design’s weaknesses and attack it. These can be attained using pattern recognition by first taking the circuit apart and micro-photographing it. The photographs obtained then run through the pattern recognition algorithm to obtain the RTL schematic of the IC. In addition, RE can take several forms [12-14]:

- Product teardown – Identify the product, package, internal boards and components

Product teardown or, in other words, identifying individual components, is to examine the package with non-destructive techniques such as X-ray imaging

to view the internal chip before carrying out subsequent depackaging or decapsulation procedures, is the first level of RE. The circuit components of interest are used for estimating materials costs. Images of the board are taken and the components are identified base on the printed labels on their housings.

- System level analysis – Analyze operations, signal paths and interconnections

The communication between components is analysed on top of component identification. There is a distinction between RE the system and functional analysis. During system RE, images of the circuit are acquired beforehand, similar to product teardown and the identified components and their connections are recorded. The board is further dismantled into its individual parts and analyzed. For multilayer boards, each individual layer is first removed, digitized and followed by reconstructing the system functionality using software tools. Functional analysis is another procedure where the focus is not on precise identification of the component but rather its functionality. This is usually done with a signal generator, a logic analyser and an oscilloscope. Utilizing certain test patterns, the system is stimulated by the signal generator to perform operations and the chip functionality can be evaluated with the logic analyzer and the oscilloscope. This practice can be used to act as substitution of components.

- Process analysis – Examine the structure and materials to see how it is manufactured, and what it is made of.

Details on the material, process variables and structure of a chip are obtained with the aid of the process analysis. The analysis methods and tools are commonly available in all semiconductor manufacturers as they are a necessity for process control and production error analysis. The initial step is depackaging (removal of the package) which is done by etching with different acids for plastic packages, as for ceramic packages, grinding will be carried out. Once the chip has been uncovered, various microscopes (scanning and transmission electron microscopes, scanning probe microscope and etc.) and chemicals can be used to expose material transitions,

structure dimensions, number of metallization and p/n-doped zones. There is no protection against this analysis.

- Circuit extraction – Delayer to transistor level, and then extract interconnections and components to create schematics and netlists.

After depackaging and identifying thicknesses of the individual layer using process analysis, layer by layer removal of the chip can be done using polishing or etching steps. Imaging is performed and the information obtained is reassembled by software. The imaging step is followed by the actual circuit analysis. The features (transistors, coils, resistors, capacitors and conductors) will be identified in the individual layers and connected to each other. This is done either manually or automatically with support tools with regards to the circuit complexity. Finally, a verification check will be conducted to verify that there are no open vias (Vertical Interconnect Access) and all components are connected.

2.4 Countermeasures against Tampering

Countermeasures can be either protective or destructive. Protective measures raise the difficulty level of the tampering process to make it more time consuming while destructive measures destroy the chip's functionality almost immediately upon tampering attempt to catch the attacker unaware. Several countermeasures developed thus far include: (1) removal of chip labels to increase difficulty in analysis, (2) utilization of nanocomposites for the packaging materials that effectively block and scatter incident X-ray radiation so as to prevent it from reaching the detector of the imaging equipment to produce sufficiently distorted image (Figure 2.9), and (3) install wire meshes and reactive membranes surrounding the chip or module to act as trigger sensors (Figure 2.10).

Figure 2.9: X-ray images of (a) a device without XBS material protection and (b) same device with XBS material protection [15]

Figure 2.10: IC package with wire mesh/reactive membrane installed [12]

In the process of exposing the chip package for circuit extraction, the wire mesh or membrane will be damaged, resulting in the loss of the entire chip's functionality. For instance, a sensor mesh will normally be incorporated on the top metal layer of smartcards, consisting of a serpentine pattern of sensor, ground and power lines. The EEPROM memory will reset or zero if any of the paths in this mesh are interrupted or circuits are shorted. EEPROM data memory bus encryption acts as an additional protection mechanism against micro-probing in some recently developed smartcards. With this protection system, information recovery is impossible despite the

attacker's ability to retrieve signals. The protection features of modern smartcard typically consist of the following:

- Voltage sensors that are able to detect internal voltage change used in power glitch attacks
- Clock frequency sensors to hinder static analysis and clock glitch attacks
- Top-metal sensor meshes to prevent easy accessing of chip
- Hardware encryption to increase difficulty in analysis of data
- Light sensors to restrict exposed chip from functioning
- Password protection to restrict software access to internal memory

The preliminary step of designing suitable protection is to find out the motivation of potential attackers so that the possible attack methods can be predicted. By also considering the attackers' background (whether it is an organization or outsider), it allows the approximation of their capabilities so that the protection level of the component(s) can be determined.

2.5 Decapsulation Methodologies

Physical attack of the IC typically starts with partial or full removal of the chip package (e.g. molding compound) to expose and allow close examination or to access the silicon die, and this is known as decapsulation or depackaging. In addition, decapsulation has been widely used in failure analysis (FA) of ICs and hence many different techniques have already been developed. Depending on the requirements, an IC package can be decapsulated with different type or a mixture of those approaches. The well-established techniques to decapsulate electronics packages consist of plasma etching, mechanical milling, wet chemical etching, and laser ablation.[16]

2.5.1 Wet Chemical Etching

Chemical etching is the most widely used method to decapsulate IC packages. It uses liquid chemicals such as Fuming (100% concentrated) Nitric Acid or Sulphuric

acid or a mixture of both to etch the encapsulant and it can be conducted either automatically or manually. Automatic acid decapsulation allows the user to preset the acid flow rate, temperature as well as the time required for the etching. When the process commences, the heated acid will be directed onto the package's surface to expose the die through a chemical reaction. Different kinds of polymer encapsulation will determine the acid needed for the decapsulation. For manual decapping, it can be performed by soaking the whole package in acid or dripping heated acid onto the molding compound surface until it dissolves. Decapping using automatic decapsulation will give a cleaner surface compared to manual decapping (drip). It is essential to note that the acid reacts with copper and thus making this technique unsuitable for prolonged decapsulation of packages with bonded copper wires. Furthermore, these acid fumes are hazardous to health, and ventilation apparatus (e.g. fume hood) is needed.

Figure 2.11: IC Package (a) before and (b) after manual chemical decapsulation [17]

2.5.2 Mechanical Milling

Mechanical milling or polishing can be used to partially open a cavity in devices, but cannot fully expose the die surface without risking damage to the passivation and bond wires. [18] The user will start with milling a hole on top of the package so that the acid used subsequently will affect only area of interest above the chip die (Figure 2.12). It is a blind process and requires patient, gradual increments of material removal until the top of the bond wires are exposed. It is often fast and easy

but can cause physical damage to the die or compromise the electrical integrity of the package. Milling is often used on thick packages to reduce etch time and acid exposure to the device and if the knowledge of die dimensions and plastic thickness are known, the device can be milled as deep as possible precisely over the die surface.

Figure 2.12: Cross section schematic drawing of (a) milled package and (b) milled and jet etch package [18]

2.5.3 Plasma Etching

Plasma with a combination of O_2 and CF_4 gases can also be employed for molding compound removal. Conventional Reactive Ion Etching (RIE) technique is carried out in vacuum where plasma is generated by radio wave power (Figure 2.13). This etching technique will not induce much damage on copper bond wires and the components due to the high etching selectivity of the plasma and absence usage of acid. However, the disadvantage of plasma etching is that the etch rate is extremely slow when compared to wet etching. To address this issue, the proposed solution is to first carry out laser decapsulation (or laser ablation) to get rid of the bulk and leave a thin layer of molding compound on top of the chip. Subsequently, plasma etching can be utilized as the cleaning step. Nevertheless, due to the shortcoming (low etch rate) of plasma, long hours may still be required.

Figure 2.13: Schematic drawing of plasma decapsulation of a package in plasma chamber [18]

2.5.4 Laser Decapsulation

Beside acid and plasma methods, laser decapsulation technique was developed mainly because copper (Cu) and aluminum (Al) are now widely used as the materials for bond wires in IC which are more susceptible to acid attack, and new epoxy materials nowadays have better resistance to acid and solvent. Laser decapsulation uses the energy from a laser beam to remove the mold compound from plastic encapsulated microcircuits. [19] Advantages of laser decapsulation through ablation include precision, selectivity (in terms of rate of ablation between different materials), cleanliness and safety from reduced plasma and chemical usage. The processing time for laser to remove EMCs all the way to wire bonds and die surface generally falls between 5 to 10 minutes, depending on the thickness of molding compounds. The encapsulating material typically does not affect the etch rate to a great extent (less than 10%). In comparison with milling process, this technique does not generate vibration and shearing forces which may result in damage to the specimen. [16] However, pure laser decapsulation cannot be employed to expose IC chip completely because it will create thermal stress. Therefore, a combination of wet etching and laser decapsulation are often carried out.

Figure 2.14: IC package (left) after laser pre-cavitated and (right) followed by jet etching [20]

As the application of laser ablation is increasingly used in IC package decapsulation process and offers many advantages, it will prompt the attackers to exploit this technique to access the die. Therefore it will be of great interest to study the interactions between laser and the polymeric encapsulant materials during ablation to assist future development of laser resistant material as a possible alternative to the current acid or solvent resistant epoxy (for instance Masterbond; Henkel). Despite much interest from the industries, its higher cost has deterred the replacement of common polymer encapsulants extensively. [21]

2.6 Laser Fundamentals and Application in Microelectronics

2.6.1 Introduction

LASER is an abbreviation for “Light Amplification by Stimulated Emission of Radiation”. A laser is created when the electrons in atoms in special glasses, crystals, or gases absorb energy from an electrical current or another laser and become “excited.” The excited electrons move from a lower-energy orbit to a higher-energy orbit around the atom’s nucleus. When they return to their normal or “ground” state, the electrons emit photons (particles of light). These photons are at the same wavelength and are “coherent”. On the other hand, ordinary visible light comprises

multiple wavelengths and is not coherent. Laser light is different from ordinary light in terms of (1) it contains only one wavelength (one specific color) and (2) it is directional (Figure 2.15) [22, 23].

	Directivity (light waves travel in straight line)	Monochromaticity	Coherence
Ordinary light	Light bulb	Many different wavelengths
Laser beam	Laser	Single wavelength	Peaks and troughs align

Figure 2.15: Difference between ordinary light and laser light [24]

It has been reported that when the laser strikes on the matter, the interaction between photons and matter include mainly Absorption (A), Transmission (T) and Reflection (R) [25] (shown in Figure 2.16). The total intensity of the incident light striking a surface is equal to the sum of the absorbed (I_A), reflected (I_R), and transmitted (I_T) intensities and can be expressed in terms of:

$$I_0 = I_A + I_T + I_R$$

If a material allows a laser beam to pass through (transmits), it is said to be transparent. If there is no transmission of the beam, the material is said to be opaque and the incident radiation is absorbed or reflected. The removal of matter from a surface or substrate through direct absorption of light energy is known as laser ablation.

Figure 2.16: Absorption, transmission and reflection of a laser beam when it strikes onto a material [26]

2.6.2 Types of Laser

The basic development of lasers started in the 1960s with numerous types of lasers including semiconductor lasers, Nd-YAG (Neodymium-doped Yttrium Aluminium Garnet; $\text{Nd:Y}_3\text{Al}_5\text{O}_{12}$) solid-state lasers, CO_2 gas lasers, dye lasers and other gas lasers being invented in this period. After 1968, the existing lasers were further redesigned and fabricated with improved reliability and durability. More reliable lasers were introduced for industrial applications by mid-1970s, such as cutting, welding, drilling and marking. After which, exploration of surface-related applications of laser such as heat treatment, cladding, alloying, glazing, and thin film deposition was conducted. Laser has such wide applications in various researches and practical fields can be attributed by its unique properties (i.e. coherence, monochromaticity and collimation). The areas of laser application (depending on the power) can be classified as follows (Table 2.2) [27]:

Table 2.2: Areas of laser application

Low Power (intensity) Lasers	High power (intensity) Lasers
Communications include:	Military include: Guide of weapons

<p>Optical fiber communications, Telecommunications, Optical data storage</p> <p>Metrology include: Distance and Velocity measurement, Alignment, Interferometry, Holography</p> <p>Reprography include: Scanning, Printing, Data storage</p> <p>Entertainment include: Laser beam performance, Audio-video recording, Pointers</p>	<p>Chemical include: Photochemical deposition, Spectroscopy, pollution control</p> <p>Medical field include: Tumor therapy, Dentistry, Surgical Operation, Dermatology</p> <p>Heating source include: Welding, Coating, Laser deposition, Laser ablation</p> <p>Scientific include: Coherent ultra-short X-ray beam for atomic structure analysis, Laser fusion, Particle acceleration</p>
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Over the past several decades, different kinds of lasers that are able to deliver a wide variety of wavelengths (covering the whole spectral range from the far-infrared to the soft X-ray), energy, temporal/spectral distribution and efficiency were developed. They are further categorized and named according to the form of optical active materials used and they are present in the form of gas, solid, or liquid (or dye) lasers (Figure 2.17). Table 2.3 further summarizes the different types of laser and their applications.

Figure 2.17: Types of laser and their wavelengths [28]

Table 2.3: Types of laser and their applications [27]

Laser	Year of discovery	Commercialised since	Applications
Ruby	1960	1963	Metrology, medical applications, inorganic material processing
Nd-Glass	1961	1968	Length and velocity measurement
Diode	1962	1965	Semiconductor processing, biomedical applications, welding
He-Ne	1962		Light-pointers, length/velocity measurement, alignment devices
CO ₂	1964	1966	Material processing-cutting/joining, atomic fusion
Nd-YAG	1964	1966	Material processing, joining, analytical technique
Ar+	1964	1966	Powerful light, medical applications, material processing
Dye	1966	1969	Pollution detection, isotope separation, scientific purposes due to wavelength tunability
Cu	1966	1989	Isotope separation
Excimer	1975	1976	Medical application, material processing, colouring
Ti-Sapphire	1982		Scientific purposes: tunable ultrashort laser pulses due to the large bandwidth

The most commonly used wavelengths for laser material processing (e.g. drilling, cutting, engraving) includes CO₂ laser in the far infra-red range at 10.6 μm , solid state Nd-YAG laser in the near infra-red range at 1.06 μm and excimer laser in the UV range below 400 nm. The characteristics of CO₂, Nd-YAG and excimer laser are described below. [22, 24, 29]

CO₂ Lasers

- Longest wavelength
- Good for processing non-metallic material such as wood and transparent materials such as glass or plastic
- Absorb poorly by metals, hence high power is required
- Due to long wavelength and transfer of heat, machining is a thermal process therefore melting and burning occur
- Simple operation and low maintenance cost

Solid state Nd-YAG Lasers

- Shorter wavelength
- Cannot process transparent objects like glass as the laser passes through such objects
- Wide range of processing applications from resins to metals (can cut high reflective materials, such as aluminium, copper and many other non-ferrous metal materials)
- High versatility (outputs can be frequency doubled, tripled or quadrupled through harmonic generation)
- Simple operation and low maintenance cost

Excimer Lasers

- Shortest wavelength
- High peak power of the laser beam permits ablation of the target material with little or no heat affected zone
- Good for processing metals, printed circuit board (PCB)
- The 193-351 nm wavelength permits generation of high resolution ($\sim 1 \mu\text{m}$) features on the target surface
- High cost of maintenance and sophisticated operation

As mentioned in 2.5.4, the objective of laser decapsulation is to remove the package encapsulation by laser ablation and the most commonly used laser for the ablation/decapsulation of microelectronics packages is Nd-YAG laser [30]. In the next section, the reasons Nd-YAG laser being widely employed for this application by the market will be further elaborated.

2.6.3 Laser Selection for Decapsulation Application [19]

Generally, two main factors are considered when selecting the most suitable laser based on user's application. The first is the wavelength and the second is the pulse duration. Choosing the right wavelength and pulse duration allows the user to have

the laser system to process the type of materials with high efficiency and optimum results.

(1): Wavelength

The laser systems of the three different wavelengths (CO₂, Nd-YAG, and Excimer) mentioned in the previous section is able to carry out mold compound removal. However, each wavelength has its advantages and disadvantages.

CO₂ laser system can be used to remove mold compound by melting and vaporization of the epoxy after absorbing the energy from the laser. However, due to the long wavelength, the laser is capable of penetrating deeper into the material which could damage the die surface. When the passivation layers absorb this energy and transfer it to the interconnect below, damage to the circuits would occur and rendering circuits non-functional.

One could also utilize an excimer laser (e.g. pulsed krypton-fluoride (KrF)) that operates at 248 nm to remove mold compound epoxies. As the wavelength is very short, the energy deposited is limited to the surface of the epoxy which leads to the removal process to be very slow, producing a lower throughput. Unless one requires very high precision and do not require the removal of large amounts of bulk material, it is not economical to use this option for decapsulation purpose.

Removal of the mold compound epoxies with Nd-YAG lasers operating at 1064 nm is the most common practice by the industries as the purchasing cost is relatively low, and the system is easy to operate and maintain. Moreover, at this wavelength, the dark-colored epoxy also absorbs the energy, causing it to melt and vaporize.

Figure 2.18 shows the common PCB materials with respect to their absorption wavelength. It can be seen that resin has good absorption at most of the wavelengths while copper only absorbs well in the UV range. If the main application is for drilling of micro-via holes in PCB where precision is necessary, it would be more suitable

to utilize lasers operating in the UV range instead of Nd-YAG or CO₂ lasers. If removal of molding compound with small precision is the main application, Nd-YAG laser would suffice.

Figure 2.18: Absorption of three common PCB materials for six types of lasers [31]

(2): Pulse duration

Besides selecting a suitable laser wavelength, the pulse duration should also be part of the consideration when purchasing a laser since there are nanosecond (ns) (from 10 to 100 ns), picosecond (ps) (≤ 20 ps), and femtosecond lasers (fs) operating at IR, visible or UV wavelength. In a typical manufacturing environment, only one wavelength is used because in this setting, the number of parts and materials to be processed is usually huge and consistent. For R&D or service providers, it is frequently useful to have access to more than one wavelength so that customization can be done to achieve a wider range of applications.

Figure 2.19 shows a graph of pulse duration (from fs to continuous-wave) and wavelength (IR to UV) and their recommended laser applications. Lasers with output characteristics near the bottom left are ideal for achieving the highest-quality features on target where they have both short wavelength and short pulse length. The lasers at the top right have long pulse lengths and IR wavelengths, so these lasers are generally not suited for micromachining applications, although they may be quite useful for other applications like welding, joining, surface treatment, and additive manufacturing. Note that there is no application for lasers with output characteristics shown at the top left (a long-pulse UV laser) because it is counterproductive. [32, 33]

Figure 2.19: Recommended laser applications at different pulse duration (from fs to CW) and wavelength (IR to UV) [32]

Figure 2.20 illustrates the effects during laser processing between a long pulse laser (ns and above) and an ultrashort/ultrafast pulse laser (ps and below). Material removal under ultrafast pulses is based on photoablation which involves the direct breaking of molecular or atomic bonds that hold the material together rather than heating it. When using ultrafast pulses, material exposure time to the laser energy is too short such that the energy cannot transfer beyond the target area, so the

surrounding area stays cold with minimal heat affected zone (HAZ). Energy left over after the bond-breaking process is usually carried away with the expelled particles. Together, these effects result in a cleaner process without leaving recast material and thereby eliminating the need for post-processing. Long pulse laser, in contrast, is photothermal where focused laser beam acts as an intense heat source rapidly heating targeted material eventually causing it to be vaporized. This allows quick removal of relatively large amounts of target material but comes with peripheral HAZ damage, formation of recast material and microcracking. [32-34]

Figure 2.20: The effects of a long-pulse laser compared to an ultrashort pulse laser during ablation [33]

Figure 2.21 shows examples of polyethylene terephthalate (PET) sample ablated under different pulse width and wavelength at approximately the same output power. The nanosecond IR and UV samples (c and d) show a lot of debris and charring on the edges. The picosecond samples (g to i) have cleaner marking over the nanosecond samples, but there is still a visual difference between the three wavelengths at the 12 ps pulse length. Overall, choosing one that has sufficient quality with the least cost is always preferred.

Figure 2.21: Features machined with nanosecond /picosecond pulse laser at different wavelength on Polyethylene terephthalate (PET) [32]

2.6.4 Laser's characteristics and ablation mechanisms

Laser ablation is generally discussed in the context of pulsed lasers; nevertheless, it is also possible to carry out ablation with intense continuous wave (CW) irradiation. A CW laser emits a steady laser beam over a period of time. On the other hand, a pulsed laser is a system which emits radiation pulses at regular intervals in ultra-short time duration. Figure 2.22 illustrates the difference between the CW laser and pulse laser during pulse emission.

Figure 2.22: Pulse emission for (a) CW laser and (b) Pulse laser [28]

In addition, “Q-switched laser” is generally classified as a pulsed laser, which operates in the kHz pulse rate. It makes use of pulsing to increase the effective power by concentrating the laser power into a short pulse, allowing each pulse’s peak power to increase while sustaining an average low power.

Knowing the amount of power delivered in each pulse is one of the key parameters and this is referred to peak power P_{peak} (or P_s), which is the maximum amount of power a pulse delivers. Power is commonly expressed as the energy with respect to the amount of time it requires to release the energy, and thus peak power is defined as:

$$P_{\text{peak}} = \frac{\text{Energy}}{\tau_p} \quad (1)$$

The above equation indicates the peak power (P_{peak}) is reliant on the pulse duration/width (τ_p). For a particular pulse energy it applies, the shorter the τ_p the larger the P_{peak} . The average power (P_{avg}) of a pulsed laser is defined as the amount of energy released over the period (T^L) of the cycle. The formula is expressed as the following:

$$\begin{aligned}
 P_{\text{avg}} &= \frac{\text{Energy}}{T^L} \\
 &= \text{Energy} \times \text{frequency}
 \end{aligned} \quad (2)$$

Where frequency (in Hz) = $1/T^L$. This equation reveals that the shorter the T^L (or higher pulse frequency), the higher the P_{avg} . Given the pulse energy, the energy density or laser fluence for a particular beam diameter can be determined by the equation (3) below. Unless the beam has other shapes than round, it is easiest to consider the area to be in the form of a circle: [35]

$$E_{density} = \frac{Energy}{\pi r^2} \quad (3) \quad \text{where the unit of } E_{density} \text{ is J/cm}^2$$

During laser ablation, the removal of material may involve a variety of mechanisms, depending on the specific system of material. The ablation usually begins above threshold fluence, which is dependent on several factors. These factors are namely (1) the way absorption occurs, (2) the material intrinsic properties, (3) the defects present in the material, (4) morphology, (5) microstructure and (6) the processing parameters of laser (fluence, wavelength (λ) and pulse duration) [7]. With a cumulative series of pulses, the threshold of ablation (minimum energy required to generate atomic and molecular separation or displacement due to incident intense laser irradiation) may drop with increasing defects created by the laser. According to Beer-Lambert law, for uniform material, the amount of material removed per pulse has a logarithmic relationship with fluence when exceeding the ablation threshold.

$$I(z) = I_0 e^{-\alpha z} \quad (4)$$

Where $I(z)$ represents the irradiance (W/cm^2) at depth z (in cm), I_0 represents the incident irradiance (W/cm^2), and α is the absorptivity (cm^{-1}) of the material. The absorption coefficient introduced by the Beer-Lambert's law accounts for the attenuation of the absorbed energy in the target.

The precise process of ablation under the short laser pulse is still under investigation at present as it occurs within a very short period of time. Pulsed solid-state lasers, like Nd-YAG, are widely used for ablation works related to nanosecond and picosecond lasers. In general concept, most of the literature describe the radiation-

matter interaction mainly under two major directions: photochemical and photo-thermal. A combination of these two mechanisms, although one usually dominates another, is involved in most of the laser removal of material. The dominating mechanism is generally decided by the type of materials and parameters of the laser beam (wavelength, pulse width, and pulse peak power).

Photochemical: In the photochemical interaction, theoretically, there is no modification in the temperature of the material's surface as light is absorbed to break the chemical bonds directly. For instance, the absorption of UV radiation by organic molecules is known to involve an electronic transition to a higher state. If the transition leads to a level above the dissociation limit, then the molecule can dissociate almost immediately [36] (e.g. C-H bond monomers breaking from the epoxy polymer chain). Chemical bonds are typically broken with high energy ultraviolet (UV) laser while resisting low energy near-IR or IR photons. A UV laser ($\lambda < 350$ nm) is required to break the high energy bonds of C-H (binding energy (BE) = 3.5 eV), [7]. From the application point of view, the most desirable mechanism would be that of the photochemical since it eliminates material damage caused by thermal effect and give a "cleaner" cut.

Photothermal: Photo-thermal interaction involves the transfer of photon energy to the targeted area of material removal which rapidly results in localized heating and temperature rise. The process results in sufficiently high temperature to cause melting and vaporization or sublimation, and this causes the removal of material. Photothermal ablation is often associated with surface swelling and delayed material ejection while photochemical ablation is considered to produce less swelling and instant etching. Since this process creates stresses, it is preferred to be kept at a minimum during laser decapping of the IC package as this process may damage the circuitry and die in it.

However, besides the two main mechanisms mentioned, Zhigilei *et al.* described another mechanism, *photomechanical*, which is also possible in laser ablation. In

absence of photochemical decomposition that breaks the molecular bonds upon absorption of light energy (mainly in the far UV), organic materials ablated at sufficiently high laser fluences is likely due to explosive vaporization. There are still plenty of experimental works claiming that initiation of ablation can occur at much reduced energy densities than the required boiling and vaporization values.

It has been proposed that the *photomechanical* ablation is possible as a result of stresses/pressure induced by laser. The relationship between the rate of energy deposition and the mechanical equilibration time for the absorbing volume, τ_s , influences the degree of the stresses caused by the laser. As the pulse width of the laser becomes lower or almost similar to the time required to start a series of movements of molecules within the absorbing volume, laser heating occurs at almost constant volumetric conditions. With constant volume but energetic vibration of molecules, pressure buildup happens and this can be typically referred to as inertial or stress confinement, defined as $\tau_p \leq \tau_s \sim L_p/C_s$ (L_p is the laser penetration depth and C_s is the speed of sound in the irradiated material).

There are many analytical simulations illustrating how the absorption area is affected by the compressive thermoelastic stress during the laser irradiation, which can lead to brittle material undergoing mechanical fracture (or spallation) or formation of cavities due to high tensile stresses. The cavities or coalescence of microcracks can result in the ejection of large to moderately sized cold pieces of material, decreasing the minimum energy density required for removal of material below the vaporization.

If one were to explore deeper, laser ablation can be very complex, as it involves many simultaneous processes during and following the laser pulse. These include heat transfer, electron-lattice energy exchange, material melting and evaporation, plasma plume formation and expansion, laser energy absorption, and etc. The series of event that happened during ns laser-matter interaction is depicted in Figure 2.23.

Figure 2.23: Schematic of the processes involved in ns laser ablation in air, with an approximate timeline of their occurrence [37]

If the selected laser is a femtosecond one, non-thermal processes will dominate the ionization. The pulse is too short to induce thermal effects; hence other effects would ionize the atoms, depending on the kind of sample. The pulse has a huge amount of energy and effects like multiphoton absorption and ionization, tunneling, and avalanche ionization will excite the sample. With this amount of energy, the electron-hole pairs created will induce emission of X-rays, hot electrons, and photoemission. This will create highly charged ions through a process called Coulomb explosion (explosive disintegration of the lattice). The absence of thermal

effect creates a crater with highly defined edges without melted or deposited materials.

In contrast, ns lasers induce thermal effects. The electron-lattice heating time is around 10^{-12} s, much shorter than the pulse time. This causes thermal effects to dominate the ionization process. The laser energy melts and vaporizes the sample, and the temperature increase ionizes the atoms. If the irradiance is high enough, non-thermal effects will be induced as well and both will ionize the sample. Between 10^{-9} s and 10^{-8} s, plasma becomes opaque for laser radiation, thus the last part of the laser pulse interacts with plasma surface and will be absorbed or reflected, and hence it will not ionize additional material. This effect is called plasma shielding and is strongly dependent on environmental conditions (surrounding gases or vacuum) and experimental conditions (laser irradiance and wavelength) [38]. This shielding reduces the ablation rate because the radiation does not reach the sample's surface. This induces a crater with melted and deposited material around it but at the same time, the plasma is reheated and the lifetime and size of plasma is higher. When the expelled liquid solidifies and the plume material condenses, it can modify the ablated region's topography at the edge and surrounding areas. Figure 2.24(a) shows the presence of a relatively large molten layer in ns laser irradiation of steel compared to the precise ablation with fs laser irradiation, where there is no trace of molten material. [39] At shorter pulse length, the ablation threshold fluence for the material reduces, and becomes more sharply defined. Nonetheless, at the end of the ultrashort pulses, the excess energy that remained in the material can still lead to thermal effects in the surrounding material.

Figure 2.24: Laser ablation of holes drilled in a steel foil with (a) ns irradiation (b) fs irradiation [39]

The dissimilarity between fs and ns laser ablation can be accounted by the laser–material interaction: (1) material response time and (2) ejection. The plume ejected during ablation by ns laser could shield the material surface, hence reducing the energy that can be taken in by the material. Material often displays a combination of the following: surface ablation, melting of material surface, and other reactions that are activated thermally. Such would cause morphology and texture of material surface to change, as well as the reaction chemistry. An example would be, after ablation, the residual heat that resides in the material can cause continuous melting process or heat initiated reactions on the material’s intact surface and its surrounding. A combination of these effects can modify the material to be used for a variety of laser processing applications.

2.6.5 Conclusion

The discussion above shows that for ablation of materials, it highly depends on the threshold fluence, the ejected plume, and the characteristics of the damage done to the target which consist of both the mechanical and thermal processes. Thus, having a better understanding of the physical processes and the conditions affecting the ablation transition at different regime condition is always desired. This would allow accurate fine-tuning of the laser fluence, pulse width, and wavelength during experiments to attain the desired results. So far, the thermal, stress confinement

conditions and some of the characteristics of laser ablation mechanisms are not well grasped.

Although there are papers reporting on the ablation of metals such as steel, Cu and semiconductor such as silicon, organic materials such as polymethyl methacrylate (PMMA), PTFE, and composite mixture such as glass/carbon fiber reinforce epoxy resin, the actual ablation behavior and mechanism have not been fully established because the details of the mechanisms happening at the microscopic scale and the reactions throughout the whole laser ablation process at the submicron scale are still not clear. By having a greater understanding on laser ablation of molding compound when conducting laser decapsulation will better benefit the development of laser resistant encapsulation materials for the IC packages.

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Chapter 3

Experimental Methodology

The first section of this chapter provides the details of the raw materials used in this research (consisting of Glycidyl-POSS matrix, fillers, silane surface treatment candidates), the reasons for selecting them and how the materials are utilized in the sample fabrication process. The second part of the chapter will elaborate on the principles of various characterization techniques and testing tools employed.

3.1 Materials

The developed anti-tamper material, GPOSS (GP) epoxy resin matrix will be used extensively in this study. Two commercially available constituents, Glycidyl-POSS (Hybrid plastics ep0409 from Hybrid Plastic Inc.) and EP62-1 (from Masterbond Inc.), are used in the formulation of GP. The Glycidyl-POSS ep0409 Cage Mixture structure is shown in Figure 3.1. It is a hybrid molecule with inorganic silsequioxane at the core and organic glycidyl groups attached at the corners of the cage. On the other hand, the EP62-1 is a two-component epoxy resin system comprising of bisphenol-A epoxy resin as part A and curing agents as part B.

Figure 3.1: Structure of Glycidyl-POSS EP0409 [1]

Besides the matrix, an encapsulant also requires inorganic fillers as mentioned in Section 2.2. Thus, inorganic fillers will be added into the samples. Two factors are hypothesized to affect the laser ablation rate as mentioned in Chapter 1 previously. For factor 1 (laser absorption), samples are fabricated with different amount of carbon black. For factor 2 (adhesion between matrix and fillers), samples are fabricated with a variation of fillers (with and without surface treatment).

Utilization of silane coupling agents for fillers' surface treatment is an established method widely reported for improving the mechanical and thermal properties of various polymers such as polypropylene (PP), through enhancing the adhesion between fillers and matrix [2]. Silane coupling agents are silicon-based compounds containing two reactive sites (inorganic and organic) in the same molecule, which

will react with the polymer and mineral to form durable covalent bonds across the interface. In other words, the alkoxy silane functionality can involve the hydroxyl groups on the substrate surface of e.g. silica to provide covalent bonds between the polymer and the filler, resulting in improved compatibility between the matrix and the filler. [3] It has been proposed that these bonds are both hydrolyzable and reformable, and therefore provide a means of stress relaxation at the organic/inorganic interface. [4]

A general structure of silane is $XY-Si(OR)_3$, where OR is a hydrolyzable group such as methoxy, ethoxy, or acetoxy, and XY is an organofunctional group such as amino, methacryloxy, epoxy, etc. A simplified picture of the coupling mechanism is shown in Figure 3.2.

Figure 3.2: Silane Coupling Agents Mechanism between inorganic and organic component [5]

A silane coupling agent will act as a bridge between an inorganic substrate (such as glass, metal, and mineral) and an organic material (such as an organic polymer, coating, adhesive) to bond, or couple, the two dissimilar materials together. Figure 3.3 shows the effectiveness of silane treatment on different range of materials. In this part of study (adhesion between matrix and fillers), silica is selected as the fillers material.

Figure 3.3: Chart showing the effectiveness of silane on respective substrates [6]

3.2 Sample preparation

The GP matrix is synthesized by mixing 85 wt% of EP0409 with 15 wt% of EP62-1. Additional components such as fillers or colour pigment (Alfa Aesar Carbon Black (CB)) can be added into the pure GP epoxy resin to formulate different types of the samples. For instance, to prepare the GP sample with carbon black (0.05 wt%), the oven is first preheated at 120°C and the preparation steps involve mixing at room temperature in centrifugal mixer in the following manner:

If inorganic fillers (e.g. silica SiO_2 or alumina Al_2O_3) are to be added, the addition will be carried out together in step 2. After which, the mixture will be poured into either silicone mold or Teflon sheet, transferred into an oven and cured for 24 hours at 120°C . Figure 3.4 shows an example of the samples after being cured and removed from the silicone mold.

Figure 3.4: Molded sample (a) GP matrix and (b) GP matrix with carbon black

Silane Treatment

For the surface treatment of fillers, two functional silane candidates, (3-glycidyloxypropyl)trimethoxysilane (GPTMS) and (3-aminopropyl)trimethoxysilane (APTMS) from Shin-Etsu Chemical Co will be utilized. The fillers used for the silane treatment in this work are fused spherical SiO_2 (Tecosphere A from IMERYS) with average particle size d_{50} of $20\text{ }\mu\text{m}$. Table 3.1 shows the information of the two selected silanes.

Table 3.1: SiO_2 fillers surface treatment

Name	Treatment	Remarks	Chemical Structure
Neat	No	N.A	N.A
Glycidyl	Yes	(3-Glycidyloxypropyl) trimethoxysilane (OCH_3) ₃ Si(CH ₂) ₃ OCH ₂ (OC ₂ H ₃)	
Amino	Yes	(3-Aminopropyl) trimethoxysilane (OCH_3) ₃ Si(CH ₂) ₃ NH ₂	

The silane surface treatment procedure [2] (e.g. on silica fillers) consists of the following steps as shown in Figure 3.5. Figure 3.6 shows the schematic of a silica surface after treatment with amino-silane and glycidyl/epoxy-silane.

Figure 3.5: Process flow of surface treatment using silane on silica fillers

Figure 3.6: Chemical structure of silica surface after amino-silane and glycidyl/epoxy-silane treatment [7]

After completing the surface treatments, respective treated SiO_2 fillers were added into the GP matrix to fabricate samples for examination under FESEM to briefly understand how the matrix and fillers adhere. Figure 3.7 shows the FESEM images of the amino/glycidyl silanes treated SiO_2 in the GP matrix.

Figure 3.7: FESEM image of (a) GP matrix + amino silane treated SiO_2 and (b) GP matrix + glycidyl silane treated SiO_2

Good adhesion between the amino silane treated SiO_2 fillers and GP matrix is observed in Figure 3.7(a), as compared to the glycidyl silane treated SiO_2 in GP matrix with poor adhesion and presence of air gaps. This indicates that amino silane is a better choice over glycidyl silane for surface treatment of SiO_2 fillers to bond with GP matrix. In addition, the glycidyl silane treated SiO_2 which yielded poor adhesion with GP matrix could also be used for the study to understand how it affects the laser ablation.

3.3 Testing and Characterization Tools

3.2.1 Laser System

The SESAME 1000 laser system (from Digit Concept) is employed to conduct the laser ablation experiments in this work. SESAME 1000 is a Q-switched Nd:YAG (1064 nm) pulse laser equipped with a diode pumping source. This laser system is mainly used for decapsulation of plastic packages. The specifications of the laser are

shown in Table 3.2. Figure 3.8 illustrates the schematic of a diode pump Nd:YAG laser.

Table 3.2: Specifications of the SESAME 1000 laser system

Laser type	Diode pumped solid state laser
Laser source	Nd:YAG
Wavelength	1064 nm
Beam diameter	50 μm
Power	8 W
Frequency	Max at 50 kHz

Figure 3.8: Schematic of diode pumped Nd:YAG laser with Q-switch [8]

The area and position of ablation are defined manually using the laser tool software. Several laser parameters such as power, frequency, and marking speed can also be tuned according to user's preference.

3.2.2 Surface Profiler

Surface profiler can be used for the measurements of step height, roughness, or obtaining some other information such as shape and form in 2D. The stylus is lowered vertically down onto the sample and moved horizontally across the sample surface (Figure 3.9) according to the distance and contact force specified by the user.

The KLA-Tencor profiler is used in the initial phase for the etch depth comparison between the commercial package and the GP material after laser ablation.

Figure 3.9: Basic operating principle of a surface profiler

3.2.3 Thermogravimetric Analysis (TGA)

TGA is one of the thermal analyses used for characterizing the thermal stability and degradation of materials under user-specified conditions. It measures the amount and rate of change in the mass of a sample as a function of temperature or time in a controlled atmosphere by examining the kinetics of the physicochemical processes occurring in the sample (using heat to force reactions and physical changes in materials). It is a useful technique for determining the absolute and relative thermal stability of material, filler content in polymers as well as the moisture content. [9] The TGA utilized for characterization in this work is a TA Instruments Q500, shown in Figure 3.10. The sample was first placed onto the platinum pan before loading it into the furnace. The test was carried out from ambient to 800°C at a heating rate of 10°C/min.

Figure 3.10: Schematic of TA Instrument Q500 TGA [10]

3.2.4 Fourier Transform Infrared spectroscopy (FTIR)

Infrared (IR) spectroscopy is used for the identification and structural analysis of chemical compounds (commonly organic or polymeric, and sometimes inorganic) [11]. When the IR radiation is shone onto the sample, some radiation is absorbed and some pass through. Absorption occurs when the radiant energy matches the energy of a specific molecular vibration. The peaks in the resulting IR spectrum represent the excitation of vibrational modes of the molecules in the sample and thus are associated with the various chemical bonds and functional groups present in the molecules. The different types of molecular vibrations are shown in Figure 3.11. A change in inter-atomic distance along the bond axis will give rise to stretching vibration while a change in angle between two bonds creates bending vibration. It is important to note that not all molecular vibrations give rise to infrared absorption bands. A fundamental vibrational mode will only be "IR active" when excitation of this vibration results in a change in the dipole moment of the molecule. [12]

Figure 3.11: Types of molecular vibrations [13]

In this report, the FTIR test is carried out using PerkinElmer Frontier FTIR-ATR which captures the signal ranging from 6000 to 400 cm^{-1} .

3.2.5 Ultraviolet Visible Near-Infra red (UV-Vis-NIR) spectroscopy

The UV-Vis-NIR spectroscopy utilizes a spectrophotometer to emit light ranging from UV to NIR wavelengths onto the sample (which can be in the form of solid, liquid or film). It then records the intensity of light absorbed or reflected off from a sample. This is a non-destructive method of evaluating optical characteristics of materials. The equipment consists of four main components: light source, monochromator, sample and reference port, and the detecting system (Figure 3.12).

Figure 3.12: Internal components of a UV-Vis-NIR Spectroscopy [14]

The light produced by the light source, for instance a tungsten halogen lamp, is focused and directed by a concave mirror towards the monochromator. The purpose of the monochromator is to split up the white light from the lamp into its component colors [15]. Figure 3.13 shows the range of wavelengths for UV to NIR and Figure 3.14 shows the basic working principle of a UV-Vis-NIR spectroscopy.

Figure 3.13: UV to NIR wavelengths [16]

Figure 3.14: Basic working principle of a UV-Vis-NIR Spectroscopy [17]

The selected system for analysis in this report covers the range of 175-3300 nm, allowing data collection at the wavelength of 1064 nm since it is the wavelength at which the laser system employed in this report operates. A baseline scan without a sample in the beam path must always be completed before a sample is loaded for analysis. After which, the graph obtained can be used to perform qualitative or quantitative analysis of the sample.

3.2.6 Scanning Electron Microscopy (SEM)

The scanning electron microscope (SEM) is a technique for observing fine details of a specimen such as the morphology or elemental composition. The working principle of a SEM consists of the electron gun releasing a focused beam of high-energy electrons (known as e-beam) which passes through the center hole of an anode. The hole in the anode allows a fraction of the electrons to continue down the column through the lenses to produce a smaller, more cohesive beam. [18] The beams continue to flow through the electromagnetic lens and land onto the specimen's surface and near-surface of mainly solid specimens to generate a variety of signals (secondary electrons, backscattered electrons etc.). Secondary electrons are emitted from the atoms occupying the top surface of the sample while backscattered electrons are primary beam electrons which are "reflected" by atoms in the solid. The signals that are being received by the different detectors from the electron-sample interactions will reveal sample information such as topography, morphology (texture) and elemental composition. The schematic of a SEM is shown in Figure 3.15 and the interaction volume of electron is shown in Figure 3.16.

Figure 3.15: Schematic of a typical scanning electron microscope and its internal components [19]

Figure 3.16: Interaction volume and signal from electron microscopy [19]

The advantages of SEM over light microscopy include (1) higher resolution (2) higher magnification (greater than 100,000 times) while OM is 500-1500 times, and (3) greater depth of field up to 100 times that of light microscopy. OM's resolution is lower, usually down to only sub-micron or a few hundreds of nanometers, due to the light diffraction limit. The magnification of SEM depends on the scan width of the e-beam. The bigger the scan width, the smaller the magnification will be and vice versa. Lastly, the depth of field refers to the range of height (distances) on the specimen surface for which the image is in focus. As the aperture angle of the electron beam in the SEM is much smaller than that of the objective lens in OM, SEM is able to achieve a larger depth of field (refer to Figure 3.17) .[20]

Figure 3.17: Relationship between aperture angle and depth of focus [20]

It is important that the sample examined in SEM is electrically conductive to minimize charge buildup on the sample caused by the electron beam. The charge buildup can degrade the sample and distort the image data. As the materials in this work are polymers based, the sample surface is coated with a thin layer of conductive film such as platinum (Pt) or carbon (C) before imaging to prevent this effect. [21]

3.2.7 Bond/Die shear test

Bond/Die shear testing is the process of determining the strength of adhesion of a die to the package's die attach substrate, by subjecting the die to a stress that's parallel to the plane of die attach substrate, resulting in a shearing stress between (1) the die-die attach material interface; and (2) the die attach material-substrate interface. [22] In this case, this test is done to quantify the samples' strength of adhesion between matrix and fillers with silane and without silane surface treatment. This test was performed using DAGE 4000 multipurpose bond tester with a 100 N load cell at a shearing rate of 50 $\mu\text{m/s}$ and a shear height of 150 μm at the start of every shear repeat.

Figure 3.18: Schematic of bond shear test setup

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Chapter 4

Results and Discussion

This chapter consists of two main sections. The first section elaborates on the studies carried out on the GP encapsulant through material characterizations and laser ablation tests in comparison with commercial packages and evaluating their performance. The second section discusses the work done to investigate the factors (light absorption and adhesion between matrix and fillers) affecting laser ablation. Possible phenomena are highlighted and the requirements in designing packaging materials to resist laser are also suggested.

4.1 Study on GP encapsulant material

4.1.1 Sample characterizations

TGA was carried out on GP sample to understand its thermal stability and decomposition profile. Upon completion of the test, the sample was retrieved and observed under an optical microscope. Figure 4.1 shows the TGA test setup and images of the sample after thermal decomposition.

Figure 4.1: (a) Setup for TGA test with GP sample, (b) GP sample retrieved after TGA test (c) Magnified optical image of GP sample after TGA test

FESEM was performed for a closer look at the morphology of the sample. The images of the sample before and after thermal decomposition were obtained as shown in Figure 4.2.

Figure 4.2: SEM images of GP sample (a) before and (b) after thermal decomposition at 200x

Crack lines were observed throughout the whole sample on its initial smooth surface after thermal decomposition. A probable reason for such a cracking structure is due to the shrinkage of material when moisture escapes and this results in the formation of gaps [1].

The TGA graph (Figure 4.3) obtained indicates a small ($\leq 2\%$) weight loss below 300°C , hence the GP matrix is thermally stable up to 300°C . After which, a larger weight loss is indicative of thermal decomposition around 357°C . With the thermal stability and degradation temperature of GP established, it allows subsequent characterizations to be carried out within the temperature limit for determining the heat capacity and thermal conductivity of GP sample to ensure accuracy of results obtained.

Figure 4.3: TGA Graph of GP heating from ambient to 800°C

In the initial phase of this work, two commercial IC packages (XL and LG) were randomly selected to carry out various comparison tests with GP samples that were prepared for preliminary study, mainly to find out how they perform when subjected to laser ablation.

Before carrying out laser ablation on the samples, FTIR was conducted on the samples listed in Table 4.1 to identify the chemical bonds present in the materials. After obtaining the spectra of the respective samples (Figures 4.4 and 4.5), the peaks were referenced to the literature [2-4] and summarized in Appendix 2.

Table 4.1: Selected samples for FTIR analysis

Sample		Remarks
XL		Commercial Package 19 x 19 mm ²
LG		Commercial Package 19 x 13 mm ²
GP with carbon black		In-house synthesized 20 x 20 mm ²
GP with carbon black & Al ₂ O ₃ fillers		In-house synthesized 23 x 23 mm ²

Figure 4.4: FTIR spectra of Package LG and XL

Figure 4.5: FTIR spectra of GP with carbon black and GP with carbon black & Al_2O_3

It can be observed that the position of the peaks in the spectra of package XL and LG are similar, hence they are likely to contain common functional groups. In comparison, GP samples also contain similar bonds, except for the difference at below 1000 cm^{-1} (see yellow box in Figures 4.4 and 4.5). While the entire IR spectrum can be used as an identification for molecules, the $600\text{ to }1400\text{ cm}^{-1}$ range is known as the fingerprint region. With a complicated series of absorption that frequently overlap each other in this region, usage of this region for accurate identification of chemical bonds has imposed a limitation for analysis [5].

XL's packaging was found to be using Nitto GE100 series' mold compound. Its datasheet (Table 4.2) presents the available chemical components and content information, which allows the understanding of the components present in the commercial package. With that, it helps to relate the FTIR results and chemical bonds present in Package XL's molding compound.

Table 4.2: Molding compound datasheet of XL package [6]

Composition / information on ingredients

Substance or Mixture:

Mixture

The common chemical name or the generic name	Solid Epoxy Resin	Phenol Resin	Fused Silica ※1	Carbon Black	Metal Hydroxide
Other name	-	-	-	-	-
Chemical formula or structural formula	-	-	SiO ₂	C	-
Content (mass%)	2-20	2-20	85-95	0.1-1.0	0.1-1.0

By comparing XL's FTIR spectrum with the above datasheet, the functional groups (e.g. O-H stretching at $3200 - 3550\text{ cm}^{-1}$, C-H stretching at $2700 - 3000\text{ cm}^{-1}$) captured do represent some of the chemical bonds present in the molding compound. In addition, according to P.J. Launer [2], all forms of SiO₂ show strong band at $1080 - 1110\text{ cm}^{-1}$. On top of that, the absorption band of amorphous silica (such as silica glass, fused quartz and etc.) in the range of $800 - 810\text{ cm}^{-1}$ is weak and broad. Due to the complex absorption in the fingerprint region, the amorphous silica SiO₂ could not be easily identified. Moreover, from the FTIR spectra of GP samples without and with fillers (Figure 4.5), it is also difficult to accurately assign peaks of Al₂O₃ because the peaks of Al₂O₃ (generally found in the range of $400 - 1000\text{ cm}^{-1}$ [7] and Figure 4.6) might not fall in the working range of the FTIR equipment ($600 - 3000\text{ cm}^{-1}$) as well as having overlaps with the Si-O functional group in GPOSS which hinders overall qualitative analysis.

Figure 4.6: FTIR spectrum of Al₂O₃ fillers

4.1.2 Laser Ablation on GP and commercial IC packages

Laser ablation experiments were conducted on XL, LG and GP samples. Figures 4.7 and 4.8 show the overall view of the samples before and after laser ablation respectively. The laser ablation parameters are shown in Tables 4.3 – 4.4. The ablated area on all samples was kept constant at approximately 4 mm² (2 mm x 2 mm) while varying other parameters. These parameters include frequency, power and marking speed, so as to understand the effects of these parameters in laser ablation on the material tested.

Figure 4.7: XL and LG packages before and after laser ablation with respective parameters at locations 1 to 10

Table 4.3: Laser ablation parameters for position 1 to 10 of XL Package (in Figure 4.7)

XL Package				
Position	Frequency (kHz)	Laser Power (%)	Marking speed (mm/s)	No of Laser Pass
1	5	100	900	1
2	5	100	900	5
3	10	100	900	5
4	15	100	900	5
5	20	100	900	5
6	25	100	900	5
7	25	100	900	1
8	20	100	900	1
9	15	100	900	1
10	10	100	900	1

Table 4.4: Laser ablation parameters for position 1 to 10 of LG Package (in Figure 4.7)

LG Package				
Position	Frequency (kHz)	Laser Power (%)	Marking speed (mm/s)	No of Laser Pass
1	5	100	900	1
2	10	100	900	1
3	15	100	900	1
4	20	100	900	1
5	25	100	900	1
6	25	100	900	5
7	20	100	900	5
8	15	100	900	5
9	10	100	900	5
10	5	100	900	5

For the proof of concept that laser ablation occurs is due to absorption of laser light energy by carbon black in material (factor 1), an additional GP sample was fabricated without adding carbon black colour pigment. Figure 4.8 shows the 3 types of GP samples before and after laser ablation with their respective compositions. In addition, Table 4.5 lists the laser parameters executed on the GP samples.

	GP w carbon black Type 1 (T1)	GP w carbon black and Al ₂ O ₃ Type 2 (T2)	GP w Al ₂ O ₃ & no carbon black Type 3 (T3)
Before Laser Ablation
After Laser Ablation
Component in wt%			
	T1	T2*	T3*
GP matrix	84.95	21.2	21.2
EP62-1	15	3.75	3.8
Carbon black	0.05	0.05	-
Fillers	-	75 (Al ₂ O ₃)	75 (Al ₂ O ₃)

*wt% of components in T2 and T3 are normalized to 100%

Figure 4.8: 3 types of GP samples before laser ablation (top row) and after laser ablation at specific locations (bottom row) and their respective compositions

Table 4.5: Laser ablation parameters for position 1 and 2, a to j (GP samples T1 to T3)

Position	Frequency (kHz)	Laser Power (%)	Marking speed (mm/s)	No. of Laser pass	Remark
1a	5	100	900	1	
1b	10	100	900	1	
1c	15	100	900	1	
1d	20	100	900	1	
1e	25	100	900	1	
2a	5	100	900	5	
2b	10	100	900	5	
2c	15	100	900	5	
2d	20	100	900	5	
2e	25	100	900	5	
1f	18	100	900	5	Package Cut
1g	1	25	100	1	No Ablation
1h	1	50	100	1	
1i	1	75	100	1	
1j	10	75	100	1	
2f	1	100	50	5	Ceramic Cut
2g	1	25	100	5	No Ablation
2h	1	50	100	5	
2i	1	75	100	5	
2j	10	75	100	5	

The “No. of Laser pass” indicates the number of laser scans executed on the specific position. Package cut refers to laser decapsulation parameters for normal plastic package while ceramic cut refers to laser decapsulation parameters for ceramic package. After performing the laser ablations, the etch depths were measured using a surface profiler. 3 measurements were taken for each area shown by the yellow dotted lines in Figure 4.9.

Figure 4.9: Surface measurement profile for each ablated area

The average etched depth (μm) was then calculated using the 3 readings obtained and shown in Tables 4.6 and 4.7.

Table 4.6: Average etched depth (μm) of 5 different samples (XL, LG, GP-T1, GP-T2 and GP-T3) under different frequency when subjected to 100% laser power and 900 mm/s marking speed for 1 pass

Frequency	XL	LG	GP(T1)	GP(T2)	GP(T3)
5 kHz	-7.83	-7.97	0	0	0
10 kHz	-27.13	-29.17	0	-13.04	0
15 kHz	-39.07	-34.83	0	-1.43	0
20 kHz	-36.4	-36.53	-0.59	-6.19	0
25 kHz	-49.07	-44.73	0	-9.23	0

The zero reading for GP samples does not necessarily indicate an absence of reaction or ablation (unless stated), but an indication of no significant etching depth was detected. The result plotted in Figure 4.10(a) shows the Etch depth against Q-switched frequency for 1 laser pass on the 5 samples and Figure 4.10(b) shows the optical IR image of GP T1 sample (left) and GP T2 sample (right) after 1 laser pass at 100% Power, 20 kHz frequency.

Figure 4.10: (a) Graph of etch depth against Q-switched frequency for 1 laser pass and (b) Optical IR image of GP T1 sample (left) and GP T2 sample (right) after 1 laser pass at 100% Power, 20 kHz frequency

From Figure 4.10(a), it can be noticed that when packages XL and LG were ablated with 1 laser pass, the etched depths were both significant, while the GP samples experienced minute effect. It is observed from the graph that in the absence of fillers (GP T1) and when the sample does not have carbon black (GP T3), there is none or almost no etching after 1 laser pass of ablation. For GP T2 sample, there was slight etching but not as much when compared to commercial packages XL and LG.

In Table 4.7 and Figure 4.11, the etched depths for the 5 samples after 5 laser passes were measured and shown.

Table 4.7: Average etched depth (in μm) of 5 samples (XL, LG, GP-T1, GP-T2 and GP-T3) under different frequency when subjected to 100% laser power and 900 mm/s marking speed for 5 passes

Frequency	XL	LG	GP(T1)	GP(T2)	GP(T3)
5 kHz	-33.43	-17.03	0	-54	0
10 kHz	-176.67	-205	-4.77	-53.97	-1.47
15 kHz	-195.67	-220	-10.42	-40.07	-3.01
20 kHz	-204.33	-218.67	-10.78	-37	0
25 kHz	-208	-217.67	-16.22	-46.03	0

Figure 4.11: (a) Graph of etch depth against Q-switched frequency for 5 laser passes and (b) SEM image of a localized region in one of GP T2 sample after 5 laser passes

When the laser pass was increased to 5, both packages XL and LG again showed a similar trend where the etched depths increased significantly and deepened ($\geq 150 \mu\text{m}$) from 10 kHz onwards to higher frequencies whereas samples GP T1 and T3 were only mildly altered. GP T2 was etched but the depth was only in the range of 37 to 54 μm , considerably less significant than XL and LG packages.

Overall, if the sample is able to absorb laser light, the addition of fillers to GP will lead to some degree of ablation to occur (comparing GP T1 and T2). From Figure 4.10(b), numerous crack lines and ablated regions were observed on the surface of T2 sample as compared to T1. This indicates that 1 laser pass had weakened T2, making it more prone to removal upon further laser ablation. Therefore, the combination of T2 experienced some etching because the addition of fillers introduced more interface which resulted in structural defects (e.g. air gap/pores/poor wetting, refer to Figure 4.11(b) for the observations). Moreover, with high wt% fillers, the fillers might not be evenly distributed, leading to aggregation of filler particles within the matrix which is difficult to be eliminated during the fabrication process. Aggregation of fillers may also lead to gaps formed within the fillers if the matrix is unable to fill up the space in between. The presence of these defects will hinder the fillers from effectively dissipating the thermal stress.

In addition, to further understand the absence of significant etching on GP when compared to commercial packages, the images of GP T1 sample and XL package captured during laser ablation under the high speed imaging CMOS camera can be used to observe the phenomena. (Note: At the time during product demonstration of Phantom camera, only GP T1 and XL package were available). Figure 4.12(a) to (f) show the *in-situ* ablation process on GP T1 sample for 1 pass using 20 kHz, 100% power and taken at 370,000 frames per second (fps). Notice that after 1 pass of laser, small cracks started to appear on the surface (circled in yellow) without obvious etching. The cracks were probably caused by the heating and stress built-up after absorbing the laser energy, leading to expansion within the material defects such as micro-pores when the laser pulses continuously bombarded the surface.

Figure 4.12: (a) to (f) Laser ablation process (scanning from left to right) for GP (T1) sample in 1 laser pass at different interval

Changes at the ablated area can be monitored directly in Figure 4.13(a) to (f) where it shows the *in-situ* laser ablation process from beginning (0 pass) till completion (5th pass) using the same parameters as previous (1 pass) experiment. During the 4th pass, much of the GP T1 started to eject and dents were observed throughout the sample. At the fifth pass, the surface was etched as indicated by the dark contrast. With reference to [8, 9], it is likely that when the laser supplies sufficient energy to heat up the sites in pre-existing pores, expansion would occur and thus crack the encapsulation, releasing materials into the surrounding either in the form of solid or liquid. It is believed that a mixture of photomechanical and photothermal mechanisms are taking place since the laser used in the ablation is not in the UV range, and surface deformation with delay in material ejection was observed. [10]

Figure 4.13: Laser ablation process (scanning from left to right) for GP (T1) sample at (a) 0 pass (b) 1st pass (c) 2nd pass (d) 3rd pass (e) 4th pass (f) 5th pass

Figure 4.14 shows the laser ablation process for 1 pass carried out on XL package. The laser parameters were again kept constant (20 kHz, 100% power). It can be observed that every pulse of the laser seems to “punch” into the sample, causing the materials to be expelled with a blast (see Figure 4.14(b) and (e)), resulting in deep ablation. Thus, it is likely that the pulse of the laser possesses sufficient energy to damage XL package during each shot.

Figure 4.14: (a) to (f) Laser ablation process (scanning from left to right) for XL sample in 1 laser pass at different interval

To understand why laser ablation has different effects on the GP samples T1 to T3, UV-Vis-NIR spectroscopy was carried out. In the UV-Vis-NIR graph (Figure 4.15), it is plotted in %R (amount of reflectance) against wavelength (nm). The data can be analyzed in the following manner assumingly:

- At 0% R $\rightarrow$ There is no reflectance, hence 100% full absorption
- 100% R $\rightarrow$ There is full reflectance, hence no absorption

Figure 4.15: UV-Vis-NIR spectrum of GP samples T1, T2 and T3

It can be observed that at 1064 nm (marked with grey dotted line), GP T1 and T2 samples have about 10% Reflectance (R) which indicates that most of the light ($\approx 90\%$) are being absorbed. On the other hand, GP T3 sample (opaque white in colour) has 100% R, meaning no light was absorbed at that wavelength.

The next set of tables, Table 4.8 – 4.10, show the microscopic images of each of the laser ablated areas on sample GP T1, T2 and T3 at 50x under different parameters with respect to Table 4.5.

Table 4.8: GP T1 (With carbon black and no fillers) **Table 4.9: GP T2 (With carbon black and fillers)** **Table 4.10: GP T3 (With fillers but without carbon black)**

By observing the above images, it is deduced that among the 3 GP samples, GP T2 (with fillers and carbon black) sample sustained the most damage from the laser ablation test whereas GP T3 (with fillers but without carbon black) has the least degree of damage. A ceramic cut of 5 passes has resulted in a deep trench as shown in position 2f in GP T2 (Table 4.9). However, it was not observed on GP T3 sample. Its surface only showed several crack lines, refer to Figure 4.16 (scale bar: 100 μm) for the enlarged image.

Figure 4.16: Enlarged image of 2f region, after 5 passes of ceramic laser cut on (a) GP T2 sample and (b) GP T3 sample

With the aid of the data collected from UV-Vis-NIR spectroscopy, it indicates that the absorption of the laser light by carbon black in the sample contributed to one of the main factors to ablation. This can be considered as one of the tamper resistance material design rules. For a direct comparison, GP T2 and GP T3 contain almost similar components, however the difference between them is that one contained carbon black pigment and the latter did not. Thus, if the sample is prone to absorb laser, the photon energy will be transferred easily to the material or pore(s), leading to heating effect and stress buildup. Spontaneous reactions then lead to ablation of the encapsulation. In contrast, if the sample does not have any component that will absorb the laser light, negligible energy will be transferred to the material or pores. Without sufficient energy, the accumulation of heat and stress will then require a longer time, and the increased laser ablation duration will lead to heat dissipation.

This, in turn, results in the minimal ablation or no ablation of the material.

4.2 Factors affecting Laser Ablation

From the study in the previous section, when carbon black pigment is added into the sample, its laser light absorption is enhanced. This indicates that light absorption by the colour pigments in the material will affect laser ablation. In this part of the work, samples were fabricated with carbon black varied at 0 wt%, 0.01 wt% and 0.1 wt% to further investigate how the amount of carbon black influences laser ablation. In addition, SiO₂ fillers with different surface treatments were added into the samples to explore how adhesion between the matrix and fillers affects laser ablation, which was mentioned in chapter 1.4 (factor 2). The fabricated samples that were studied in this section are shown in Figure 4.17.

Figure 4.17: Samples utilized in this part of work

The area of laser ablation and laser parameters for all samples were fixed at 5 x 10 mm² and using package cut (refer to table 4.5), respectively. Furthermore, to better represent the amount of materials removed after laser ablation, weight loss was

measured and expressed in mg (samples' density is constant at 1.7 g/cm³). The GP with neat SiO₂ serves as a reference sample.

Table 4.11: Average amount of material removed (mg) of respective samples

Sample Type	Without Carbon Black		With 0.01wt% Carbon Black		With 0.1wt% Carbon Black	
	1x30Pass	30Passes	1x30Pass	30Passes	1x30Pass	30Passes
	weight loss (mg)					
GP w Neat SiO ₂	0.05	0.14	9.31	13.74	8.91	13.20
GP w Glycidyl Treated SiO ₂	0.06	0.12	8.70	11.66	13.89	21.37
GP w Amino Treated SiO ₂	0.06	0.11	8.20	11.59	8.90	13.74

Table 4.11 shows the data collected after the samples were subjected to 30 laser passes with interval break of 5 seconds (1x30Pass) and without interval break (continuous scanning 30Passes) and the result is plotted in Figure 4.18.

Figure 4.18: Scatter graph on amount of material removed (mg) of different samples laser ablated at 1 x 30P and 30P

From the results shown in Figure 4.18, some observations can be drawn:

1. One obvious trend is, without adding any carbon black into the 3 sample types (with different surface treatment of SiO₂), it is close to being laser resistant where the amount of material removed is almost negligible regardless of going through 1x30 or 30 laser passes.
2. Once carbon black is added, the removal of materials by the laser would occur instantaneously and significantly.
3. Within the batch of samples that were added 0.1 wt% carbon black, GP with glycidyl silane treated SiO₂ had the worst performance
4. The material removed at 30 laser passes without interval break is always more than that removed at 30 passes with interval break.

4.2.1 Laser light absorption

Table 4.12: UV-Vis-NIR of respective samples at 1064 nm

Sample	Without Carbon Black			0.01wt% Carbon Black			0.1 wt% Carbon Black		
	%T	%R	%A	%T	%R	%A	%T	%R	%A
GP + Neat SiO ₂	45.52	39.32	15.16	0.12	4.76	95.12	0.08	4.66	95.26
GP + Glycidyl SiO ₂	49.04	36.58	14.38	1.2	4.68	94.12	0.07	4.55	95.38
GP + Amino SiO ₂	42.7	40.79	16.51	0.58	4.78	94.64	0.14	4.69	95.17

T: Transmission, R: Reflectance, A: Absorption

To understand how carbon black influences the light absorptivity during laser ablation, UV-Vis-Nir spectroscopy was carried out again on the samples. The percentage values of transmission, reflectance and absorption listed in Table 4.12 were extracted at 1064 nm. The absence of carbon black caused the samples to have low light absorption (e.g. $\approx 15\%$). For damage to occur during laser irradiation of polymer, the material must absorb at the wavelength of the incident radiation [11]. When the sample does not absorb much of the energy from the laser, it reduces the probability of energy transformation into other forms of energy such as heat. On the other hand, once carbon black is involved in the sample fabrication, whether the amount is small at 0.01 wt% or 0.1wt%, they will absorb the light substantially ($\approx$

95%). Since one of the carbon black main properties is to absorb infrared radiation/light rays and transform the energy of the light rays into heat and dissipate them throughout the product [12], this heating phenomena causes vibrational excitation in molecule chains of the material. These vibrational motions induce stress that may lead to mechanical fracture or vaporization depending on the intensity and eventually material removal.

With this information, the first rule of design for the anti-tamper encapsulant to be laser resistant is to minimize light absorption as much as possible to prevent a series of events from occurring. Although it is a common practice for industry to add carbon black (usually not more than 0.5 wt%) into their package encapsulation process, their main purpose is simply to reduce device visibility for cosmetic purpose or to protect device that is light sensitive (for reducing photonic activity). [13] It is still possible to divert from the carbon black addition practice since the encapsulant that contains matrix and fillers is opaque white and if the device that requires anti-tamper feature is deemed to be not light sensitive. Under the circumstance where the device that requires anti-tamper is light sensitive, omitting carbon black may not be that suitable because a certain amount of light is still able to transmit through ($\approx 40+$ %) in the matrix-silica fillers encapsulant. Thus, an alternative colour pigment that does not absorb light at 1064 nm may be required. In the case where carbon black is required, improving the adhesion between matrix and fillers for better stress transfer may provide an alternative solution.

4.2.2 Adhesion between Matrix and Fillers

It has been widely reported in literature that the interfacial adhesion between fillers and polymer matrix greatly affects the mechanical properties. Good adhesion results in efficient stress transfer from the continuous polymer matrix to the dispersed fillers, leading to improved mechanical performance of composites [14-17]. Die shearing test is conducted to determine the shear strength of the GP matrix with respective treated SiO₂. In this case, the surface of the silicon die and the glass substrate is

treated with the amino/glycidyl silane and the GP matrix is used as the “sandwich” material to mimic the bonding between GP matrix and the SiO_2 fillers. The die is subjected to a stress parallel to the glass substrate. This causes stress at the interface between the die and GP matrix material. The force needed to break the bond is the die shear strength and die shear strength that is greater than 3400 psi (10 kg) is considered high. [18] 10 samples were sheared for each test (Figure 4.19) and the average shear force (kg) was calculated and shown in Table 4.13. The test parameters have been previously mentioned in Section 3.2.7.

Figure 4.19: (a) Test samples were fixed onto the glass slide for die shear test and (b) schematic cross-section of an individual sample, (c) die shear test setup and (d) die shear process

Table 4.13: Average die shear force (kg) for respective samples

Die Shear Test			
Simulate →	GP + Neat SiO₂	GP + Glycidyl SiO₂	GP + Amino SiO₂
Silicon chip	No treatment	Glycidyl silane treated	Amino silane treated
Material	GP Matrix	GP Matrix	GP Matrix
Glass slide substrate	No treatment	Glycidyl silane treated	Amino silane treated
Avg shear force (kg)	8.1	6.8	9.8

From Table 4.13, the silicon chip and glass slide surface that were treated with amino silane (mimic GP + Amino SiO₂) has the highest shear force (~ 9.8 kg) while the glycidyl silane treated silicon chip and glass slide (mimic GP + Glycidyl SiO₂) required a lower shear force (~ 6.8 kg). This indicates that a composite material with good adhesion between the matrix and fillers would improve the stress transfer (~40 %) as compared to poor adhesion samples. To relate this to the laser ablation result, the GP + glycidyl SiO₂ which has the lowest shear strength also had the worst performance when added with 0.1 wt % carbon black. For GP + amino SiO₂, although it has quite a high shear strength (9.8 kg), it did not show a superior performance against reference sample during laser ablation. Plausible factors that could prevent GP + amino SiO₂ material system from showing a better performance are (1) adhesion/contact between the fillers and the matrix may be imperfect, (2) CTE mismatch between the matrix and fillers which may affect the transfer of stress, or (3) presence of air gap/voids/micro-pores generated during fabrication process could also act as point of failure. [14]

Lastly, carrying out laser scanning at 1 x 30P and continuous 30 P (with interval breaks vs without interval break) is to explore how time given for heat dissipation or stress relaxation in material would reduce laser ablation. This information could be useful in the future in fine-tuning of the anti-tamper encapsulant design such as adding thermal conductive fillers or stress relaxation additive to further retard the process.

4.3 Summary

In summary, the carbon black pigment when added into a package encapsulant is prominently the source that causes laser ablation to occur due to having excellent light absorbing property. Every single carbon black dispersed in the material serves as a site that absorbs laser light energy and transforming them into heat. The matrix/filler surrounding the carbon black gain this heat energy vibrates and leads to thermal expansion. CTE of resin matrix is usually ten times greater than fillers (from Figure 4.20, epoxy resin's CTE is 150 times more than silica). With this difference, stress would be generated during the initial phase of heating.

Filler	Epoxy	Silica	BN	Alumina	Diamond
Density (g/cc)	1.19	2.2	2.25	3.98	3.5
Thermal conductivity (W/mK)	0.23	1.5	300	36	2000
CTE (ppm/°C)	75.18	0.5	<0.5	6.6	0.8
Modulus (GPa)	2.3	74	43	385	1050
Poisson's ratio	0.4	0.19	0.25	0.21	0.068
Filler shape	—	Spherical	Platelet	Polygon	Polygon
Filler particle mean diameter size (μm)	—	3	1	0.3	1

Figure 4.20: Material properties of epoxy and common fillers [19]

In the situation where the adhesion between matrix and fillers is poor (see Figure 4.21(a) and (b)), the space in between them will act as a point of weakness. The heat energy transformed from the laser light source will heat up the sample and cause expansion of air pockets. With different expansion rate of matrix, fillers and air gaps, crack lines are likely to form within the sample. During continual scanning of the laser on the sample, the cracks will propagate and ablation occurs. In contrast, when there is perfect adhesion between matrix and fillers, theoretically, no space should be present and this reduces localized stress build up caused by the expansion of air.

Figure 4.21: SEM images of (a) and (b) glycidyl silane treated silica in GP matrix showing poor adhesion and, (c) and (d) amino silane treated silica in GP matrix showing good adhesion

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Chapter 5

Conclusion and Future Work

This chapter consists of two sections, conclusion and future work. Conclusion addresses the factors affecting laser ablation according to the results obtained and relate the knowledge towards the future development of laser resistant encapsulate material for microelectronic devices. Future work provides suggestions to explore on further optimization of the encapsulate materials' property so that it can be applied into real-life application of microelectronic devices with anti-tamper features.

5.1 Conclusion

In summary, the two factors mentioned in chapter 1 that will affect the laser ablation, (1) laser light absorption and (2) adhesion between matrix and fillers to enhance stress transfer, had been investigated and addressed. The results have shown that when no carbon black pigment is added in the sample formulation, the light absorption (%A) reduces extensively from 95% to 15% and the amount of materials removed after laser ablation is about 90 times lesser. This indicates that by keeping the laser light absorption on the material to a minimum, it would have a superior performance of resisting the laser ablation. In addition, the shear test result shows that the samples with good matrix-fillers adhesion (better interfacial bond) have higher shear strength (about 40% increase) as compared to samples with poor matrix-fillers adhesion. This indicates that good adhesion between matrix and fillers enhances the stress transfer between them, thus allowing the encapsulation material to be more resistant to removal when subjected to laser ablation.

In conclusion, minimizing laser absorption through omitting carbon black pigments and pairing this with good matrix-filler adhesion in the encapsulant material is likely to promote laser resistant property in the material. With this knowledge, it will contribute to the future development of laser resistant encapsulant material for secure microelectronic devices.

5.2 Future work

In order to be able to apply the material studied in this work to actual application on microelectronic devices, other properties such as thermal conductivity, reliability that are required in molding compound also needs to be looked into so that it can suit the manufacturing process. So far, surface treatment done on silica fillers to promote adhesion between matrix and fillers have shown interesting findings. Exploring other surface treatment methods or candidates besides aminopropyltrimethoxysilane may be useful for further optimizing the stress

relaxation property. Moreover, the study of adding additives that can conduct heat away from the device to reduce thermal effects and stress build up can be considered as well.

If the microelectronic device is light sensitive and requires colour pigments filler (in most cases carbon black is used) to block off any light transmission, alternative colour pigment (i.e white in colour such as Teflon™ PTFE (polytetrafluoroethylene) powder or coating with TiO₂) that would not absorb the laser light at 1064 nm may be looked into since this wavelength is the most common one in laser decapsulation equipment of molding compound in the market today. After changing to suggested white pigments, the series of characterization tests in this work can be repeated on the new samples to firstly find out if GP would be able to resist laser ablation, and secondly, finding the appropriate amount of the white pigment to prevent light from transmitting through (with the aid of UV-Vis-NIR spectroscopy) and at the same time without causing any detrimental change in other physical properties can be further studied.

APPENDIX

Table A.1: List of package types in the market

PACKAGE INFORMATION
1. PACKAGE CLASSIFICATIONS

Package Name	Characteristics
Dual In-line Package	DIP packages are hermetic ceramic package. The lead pitch is 2.54 mm (100 mil) and the package body is made of ceramics. Metal or glass may be used as a sealing material.
Dual In-line Package (Glass Sealed)	Dual In-line packages are called "CER-DIP" package. The lead pitch is 2.54 mm (100 mil), and the package body is molded with powder ceramics. The sealing material is glass.
Pin Grid Array	PGA packages are featured by the leads which are drawn out vertically from each package body and arranged on the specified grid. The package body is made of ceramics, and the standard lead pitch is 2.54 mm (100 mil). PGA packages are suited to multipin packaging.

Package Name	Characteristics
Small Out-line L-Leaded Package	SOP packages are characterized by gull-wing type leads which are drawn out from each package body in two directions, and can be mounted flat. The standard lead pitch is 1.27 mm (50 mil).
Shrink Small Out-line L-Leaded Package	SOP packages with a lead pitch of less than 1.27 mm (50 mil) are called SSOP.
Thin Small Out-line L-Leaded Package	TSOP packages are ultra thin SOPs with a package L-Leaded mounting height of less than 1.27 mm, and are suited to ultra-thin electronic equipment such as smart cards.
Quad Flat L-Leaded Package	QFP packages are characterized by gull-wing type leads which are drawn out from each package body in four directions, and can be mounted flat. There are various QFPs available because the lead pitch is variable and the dimensions of the package body fixed.
Quad Flat L-Leaded Package with Heat Sink	QFP packages with a heat sink. (for high power device)
Quad Flat L-Leaded Package with Heat Spreader	QFP packages with built-in heat spreader. (for high power device)
Thin Quad Flat L-Leaded Package	Thin QFP packages have the package body heights (mold thickness) of 1.0 mm and of 1.27 mm.
Low Profile Quad Flat L-Leaded Package	LQFP packages have the package body height (mold thickness) of 1.4 mm.
Ball Grid Array/Fine Pitch BGA	BGA packages are surface mounting package with solder ball arrays on backside surface of printed circuit board (PCB).
Fine Pitch Land Grid Array	FLGA packages are surface mounting packages with solder land arrays on backside of printed circuit board (PCB).

Table A.2: List of infrared spectroscopy absorptions by frequency regions [1]

4000-3000 cm ⁻¹							
3700-3584	medium	sharp	O-H	stretching	alcohol	free	
3550-3200	strong	broad	O-H	stretching	alcohol	intermolecular bonded	
3500 3400	medium		N-H	stretching	primary amine		
3400-3300 3330-3250	medium		N-H	stretching	aliphatic primary amine		
3350-3310	medium		N-H	stretching	secondary amine		
3300-2500	strong	broad	O-H	stretching	carboxylic acid	usually centered on 3000 cm ⁻¹	
3200-2700	weak	broad	O-H	stretching	alcohol	intramolecular bonded	
3000-2800	strong	broad	N-H	stretching	amine salt		
3000-2500 cm ⁻¹							
3333-3267	strong	sharp	C-H	stretching	alkyne		
3100-3000	medium		C-H	stretching	alkene		
3000-2840	medium		C-H	stretching	alkane		
2830-2695	medium		C-H	stretching	aldehyde	doublet	
2600-2550	weak		S-H	stretching	thiol		
2400-2000 cm ⁻¹							
2349	strong		O=C=O	stretching	carbon dioxide		
2275-2250	strong	broad	N=C=O	stretching	isocyanate		
2260-2222	weak		C≡N	stretching	nitrile		
2260-2190	weak		C≡C	stretching	alkyne	disubstituted	
2175-2140	strong		S-C≡N	stretching	thiocyanate		
2160-2120	strong		N=N=N	stretching	azide		
2150			C=C=O	stretching	ketene		
2145-2120	strong		N=C=N	stretching	carbodiimide		
2140-2100	weak		C≡C	stretching	alkyne	monosubstituted	
2140-1990	strong		N=C=S	stretching	isothiocyanate		
2000-1900	medium		C=C=C	stretching	allene		
2000			C=C=N	stretching	ketenimine		

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