SYNTHESIS AND CHARACTERIZATION OF GRAPHENE OXIDE-SILICON POLYMER VIA LIQUID-PHASED LASER ABLATION

By

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The growing demand for efficient thermal management in electronic devices necessitates the exploration of advanced materials for thermal interface applications. Graphene oxide (GO) combined with tetraethyl orthosilicate (TEOS) forms a promising mixture for thermal interface materials (TIMs). Pulsed laser ablation (PLA) is a physical deposition technique to modify material structures, offering potential enhancements in thermal conductivity and other relevant properties. However, a comprehensive understanding of the specific effects of laser ablation on the structural characteristics of the GO-TEOS mixture remains limited. In this work, both GO that immersed in TEOS and ethanol solvent were used to fabricate graphene-silica polymer using liquid phase pulsed laser ablation (LP-PLA) technique. Different GO concentration for GO-TEOS mixture were ablated with different fluence of laser. The GO-TEOS solution was ablated with different fluence of laser. The ablated GO-TEOS solution was characterized by Fourier Transform Infrared (FTIR) Spectroscopy, Raman spectroscopy and Ultra-Violet Visible (UV-Vis) Spectroscopy to study the structural and optical properties of graphene-silicate polymer. The electrical and thermal properties were also characterized by using Electrochemical Impedance Spectroscopy (ELS), Differential Scanning Calorimetry (DSC) and Thermal Conductivity Analyzer to study the specific heat capacity, electrical and thermal conductivity of GO-TEOS mixture. The FTIR result showed that the laser ablation process has provided sufficient laser energy to create or break the chemical bond of GO and TEOS compounds as observed on Si-O and C-O bonds. The Raman result showed that intensity change in D band which suggesting the carbon atom of the GO has been functionalized with other compounds. The UV-VIS result showed a broad absorption band with center at 492 nm and 532nm with increasing absorbance at low fluence then saturated and decreased at maximum laser fluence. The EIS measurement showed the electrical conductivity is the highest in 10-min laser fluence (10-min), thus observing an increment of conductivity. This high laser fluence have supplied sufficient energy to GO, resulting the transformation of benzene on GO basal plane to

cyclohexane thus increased the nucleation and agglomeration of GO particles that finally contributed to lower phonon scattering and improvement of the thermal conductivity. From the results above, several chemical interactions between GO and TEOS were observed, and the data suggested laser fluence as the major source to cause both photothermal and photochemical reaction on the samples. In short, both laser ablations provide sufficient energy to induce the chemical bonding, which further allow the structural modification on materials.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Master Sains

SINTESIS AND CIRI-CIRI GRAFIN OKSIDA-POLIMER SILIKON MELALUI TEKNIK ABLASI LASER DENYUTAN FASA CECAIR

Oleh

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Permintaan yang semakin meningkat untuk pengurusan haba yang cekap dalam peranti elektronik memerlukan penerokaan bahan termaju untuk aplikasi antara muka terma. Grafin-oksida (GO) digabungkan dengan tetraetil-ortosilikat (TEOS) membentuk campuran yang menjanjikan untuk bahan antara muka terma (TIM). Ablasi laser denyutan (PLA) ialah teknik pemendapan fizikal untuk mengubah suai struktur bahan, menawarkan potensi peningkatan dalam kekonduksian terma dan sifat lain yang berkaitan. Walau bagaimanapun, pemahaman menyeluruh tentang kesan khusus ablasi laser pada ciri-ciri struktur campuran GO-TEOS masih terhad. Dalam kajian ini, GO yang terendam di dalam TEOS dan pelarut etanol telah digunakan untuk menghasilkan polimer grafin-silika dengan menggunakan teknik ablasi laser denyutan fasa cecair (LP-PLA). Kepekatan GO yang berbeza telah diablasi dengan kelancaran laser yang berbeza untuk campuran GO-TEOS. Campuran GO-TEOS yang diablasi telah dicirikan oleh spektroskopi transformasi Fourier infrared (FTIR), spektroskopi Raman dan juga spektroskopi ultraungu tampak (UV-Vis) untuk mengkaji sifat struktur dan optic bagi polimer grafin-silikat. Sifat electrik dan haba juga dicirikan dengan spektroskopi impendans electrokimia (EIS), kalorimetri pengimbasasn beza (DSC) dan penganalisis konduksi terma untuk mengkaji muatan haba tentu, kekonduksian electrik dan terma bagi campuran GO-TEOS. Keputusan FTIR telah menunjukkan bahawa proses ablasi laser telah memberikan tenaga yang mencukupi untuk mencipta atau memecahkan ikatan kimia dari sebatian GO dengan TEOS seperti yang dicerap pada ikatan Si-O and C-O. Keputusan Raman menunjukkan bahawa perubahan keamatan dalam jalur D yang menunjukkan atom karbon daripada GO telah difungsikan dengan sebatian lain. Keputusan UV-VIS telah menunjukkan jalur penyerapan yang luas pada pertengahan 492nm dan 532nm dengan peningkatan penyerapan pada kelancaran rendah, kemudian tepu dan menurun pada kelancaran maksima. Pengukuran EIS telah menunjukkan pengurangan dalam rintangan pukal apabila pemalar dielektrik meningkat. Kenaikan dalam kekonduksian telah ditunjukkan. Kelancaran laser yang tinggi ini telah membekalkan tenaga yang

mencukupi untuk GO, mengakibatkan transformasi benzena pada satah basal GO kepada sikloheksana sekali gus meningkatkan nukleasi dan aglomerasi zarah GO yang akhirnya menyumbang kepada penyerakan fonon yang lebih rendah dan peningkatan kekonduksian terma. Berdasarkan keputusan-keputusan di atas, beberapa interaksi kimia antara GO dan TEOS telah diperhatikan. Data yang berkaitan telah mencadangkan kelancaran laser sebagai sumber utama yang menyebabkan kedua-kedua reaksi fototerma da fotokimia pada sampel. Konklusinya, kedua-dua ablasi laser telah memberikan tenaga yang mencukupi untuk mengaruh ikatan kimia dan mengubahsuai struktur pada campuran GO-TEOS.

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Date: 14 March 2024

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μm	micrometer
D	Dimensional
ASTM	American Society for Testing and Material
Cp	Specific Heat Capacity
CNTs	Carbon Nanotubes
CQDs	Carbon Quantum Dots
CVD	Chemical Vapour Deposition
DC	Direct Current
DLS	Dynamic Light Scattering
DSC	Differential Scanning Calorimetry
EIS	Electrochemical Impedance Spectroscopy
fGO	Functionalized Graphene Oxide
FTIR	Fourier Transform Infrared
UV-VIS	UltraViolet-Visible
FWHM	Full Wave Half Maximum
GO	Graphene Oxide
K	Kelvin
LO	Longitudinal Optic
LP-PLA	Liquid Phased-Pulsed Laser Ablation
MWCNTs	Multiwall Carbon Nanotubes
NA	Avogadro's constant
Nd:YAG	Neodymium-doped Yttrium Aluminium Garnet
nm	nanometer
NSMs	Nanostructures Materials
^{o}C	degree celsius

PC	Polycarbonate
PDI	Polydispersity Index
PE	Polyethene
PI-ODA-GO	Polyimide-4,4'-Diaminodiphenyl ether-graphene oxide
PLA	Pulsed Laser Ablation
PMMA	Poly(methyl methacrylate)
PP	Polypropylene
PS	Polystyrene
PSA	Particle Size Analyzer
PU	Polyurethane
PVA	Polyvinyl Alcohol
PVDF	Polyvinylidene Fluoride
QDs	Quantum Dots
rGO	Reduced Graphene Oxide
SAED	Selected Area Electron Diffraction
SWCNTs	Single-Wall Carbon Nanotubes
TEM	Transmission Electron
TEOS	Tetraethyl orthosilicate
TIM	Thermal Interface Material
TIR	Thermal Interfacial Resistance
TMOS	Tetramethylorthosilicate
TO	Transverse Optic
TPU	Thermoplastic Polyurethane
VTMS	Vinyl Trimethoxy Silane

CHAPTER 1

INTRODUCTION

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1.1 Introduction

This chapter presents the overview of thermal management in various industries such as automotive, electronics and more. Besides that, the materials that used in this work are also introduced in this chapter. This chapter also presents the motivation of the research study, problem statements related to the thermal applications for the industry, the objectives of this work and the outlines of this thesis.

1.2 Overview of Thermal Management of Technologies

According to Thermal News, the thermal management market is estimated to grow from $8.8 billion in 2020 to $12.8 billion by 2025 with a growth rate of 8.2%. The market has a promising growth potential due to several factors, including the rising demand for effective thermal management solutions and systems in consumer electronics, increasing demand for electric and hybrid vehicles, increasing use of electronic devices in different end-use industries, and ongoing radical miniaturization of electronic devices.

Another good example of the company having detailed thermal management is the Apple Computer, Inc.. The optimum temperature for an iPhone is between 0ºC to 35ºC regarding the Apple documentation. The temperature beyond 35ºC will surge the risk of permanent damage such as shortened battery life. There have also been reports of screens cracking due to excessive temperatures. Excessive heat caused by a poor heat dissipation design has become serious problem lately due to the increased high thermal output of high-end electronic components and devices. In power electronic components and devices, the thermal management system is a crucial to control and regulate the heat dispersion, storage and conversion to ensure the operation of the equipment. As the electronic devices is indispensable to our life from communication, energy storage, electric vehicles, battery and so many others appliance, effective thermal management system is urgently necessitated (Renteria *et al, 2014*; Wu *et al*., 2019; Marshall *et al.*, 2019; Wang *et al.*, 2020). An inefficient thermal management can cause overheating of the components become a challenging problem accelerate with the demand of smaller footprint, more capable, and more efficient electronic devices for future market (Feng *et al*., 2019). Thus, the development for advanced thermal management materials and technologies has been recognized as a challenge in thermal sciences research, thus demanding continuous improvements in device and system design performance.

Hannemann (2003) stated that the main approach in thermal management of electronic design can be viewed in terms of three different aspects but non-separable problems. Firstly, the temperature of semiconductor chips must be maintained at a relatively low level from high local heat density. Next, the heat loads must be controlled and maintained low at the whole assembly or module level. Lastly, the thermal capacity of an electronic system must be contained to avoid a catastrophic failure. Thus, in order to avoid devices failure, advanced cooling solutions and pragmatic design for electronic package are required.

The electronic packaging represents the physical barrier between electronic parts with the environment. In general, physically attached thermal interface material is used between the semiconductor and the lead frame to conduct heat to environmental. Although this method is effective and low cost, frequently it can produce a mechanical stress that may cause localized structural damage and the growth of cracks to the semiconductor chip die (Edwards *et al.*, 1987).

Additionally, thermal problem related to electric and electronic component can prompt to humidity complication. A high temperature environment has higher surrounding humidity that cause corrosion to the packaging materials and damaging the inner parts, leading to electrical failure (Einspruch, 2014). High environmental temperature and humidity can cause overstressing the wire bonds, thus tearing the connections loose and cracking the semiconductor dies, or causing packaging cracks.

In the automotive industry, advanced automotive systems contribute greater quality to automobile safety, reliability, emission/noise performance and comfort. The automotive industry is slowly shifting from traditional vehicles powered by internal combustion engine to the electric vehicles. Energy efficiency refers to the amount of energy from the fuel source that is converted into actual energy for powering the wheels of a vehicle. From the study of Albatayneh *et al.* (2020), the traditional vehicles convert about 79% to 83% of the heat while electric vehicles convert about 10% to 15% of the heat energy for powering the wheels of a vehicle (Albatayneh *et al.*, 2020).

Thermal management and proper insulation are very important to maintain the engine durability and passenger comfort. Notedly, the heat is produced by the combustion engine in conventional vehicles or by the electric vehicle components such as battery, motor, power electronics. Materials with poor heat transfer coefficient will reduce the performance and engine lifetime of the conventional vehicles or components in electric vehicles. For an example, heat shield can be used to shield and dissipate heat from engine component when a large amount of heat given off by internal combustion engines. It also protects internal electronic components that could become damaged by extreme temperatures. (Fortunato *et al.*, 2007; Chinchole *et al.*, 2018).

Thermal management required a good thermal interface material (TIM). There are two categories of TIMs, first is the ability to conduct heat and second is the ability to store heat. Basically, TIM is a material placed between two interfaces of module to transfer or insulate the heat. The heat energy will be stored in the system or dissipated to environment. It is important for the TIM to have good contact between the surfaces so that it able to reduces or increases the thermal interfacial resistance (TIR) between two interfaces (Lewis *et al*., 2021). Hence, the need of advanced TIM is crucial to improve the performance and reliability of the advanced devices.

Regarding to these critical needs, advanced materials and fabrication process improvements in packaging and cooling technology are required to provide efficient thermal transfer's material, longer usage lifetime, good thermal transient behavior, environmentally friendly, low weight and fabrication cost.

1.3 Graphene Oxide (GO)

Historically, in the year of 1859, graphene oxide (GO) was first and successfully synthesized by oxidation and exfoliation of graphite (Brodie, 1859). GO is a chemically modified graphite that contains chemically reactive oxygen-functional groups. These oxygen functional groups, such as epoxides, carboxylic acid and alcohol shown in Figure 1.1 are covalently bonded to the basal plane and edges of graphene, making it contain a mixture of sp^2 and sp^3 hybridized carbon atoms (Dreyer *et al.,* 2010). The oxygen-functional groups in GO leads to the good electrical and optical properties (Loh *et al*., 2010; Ekiz *et al*., 2011) which provides potential applications in the several fields, such as energy (Li *et al*., 2010; Zhao *et al*., 2012), electronics (Eda *et al*., 2008: Eda & Chhowalla, 2010), optics (Loh *et al*., 2010) and biosensors (Balapanuru *et al*., 2010). Nowadays, GO has attracted a lot of attention from the researchers because it's simple, scalable, and can be synthesized at low-cost (Smith *et al*., 2019). It can easily be mixed with different types of polymers and other materials, and enhance several properties of composite materials like tensile strength, elasticity, electrical and thermal conductivity (Tang *et al*., 2014; Chee *et al*., 2015; Smith *et al*., 2019).

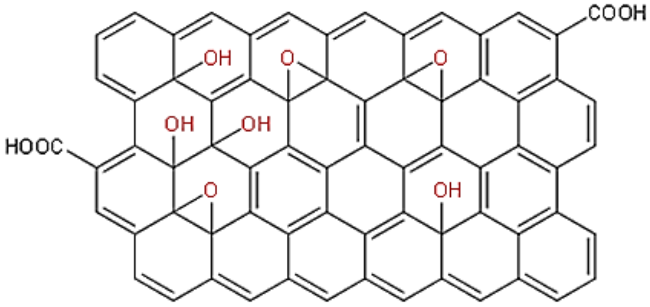

Figure 1.1: Chemical structure of Graphene Oxide (GO)

1.4 Tetraethyl Orthosilicate (TEOS)

Tetraethyl orthosilicate (TEOS) is a silicone polymer which has chemical formula of $Si(OC_2H_5)_4$. It is a colorless liquid that easily degrades in SiO_2. TEOS is a glass precursor in the semiconductor industry. When an alcohol is added to the TEOS, TEOS will easily convert to silicon hydroxide. This process is known as alcoholysis (Brinker & Scherer, 2013). The reaction proceeds through the condensation reactions causing the formation of Si-O-Si linkages in TEOS molecules. It can be acted as coupling agent which formed a covalent bond between organic and inorganic compounds (Arklas, 2006). Figure 1.2 shows the chemical structure of the TEOS which often exploited as one of the silanes coupling agent materials.

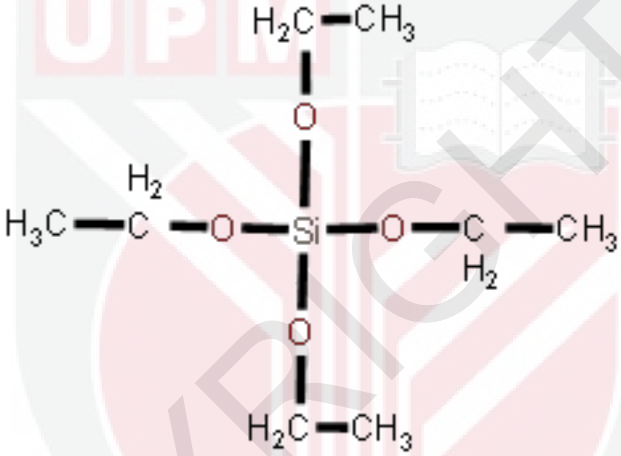

Figure 1.2: Chemical structure of Tetraethyl Orthosilicate (TEOS) which is used as coupling agent.

1.5 Pulsed Laser Ablation (PLA)

Pulsed laser ablation (PLA) is a process related to the ejection of material from the surface using high energy photon (Smith & Turner, 1965; Dell'Aglio *et al.*, 2015). In the year of 1965, the first PLA system for the growth of thin films was developed after the invention of pulsed ruby laser (Smith & Turner, 1965). From the study of Smith & Turner (1965), several types of thin films, such as copper (Cu), Selenium (Se), Tellurium (Te) and Germanium (Ge) were synthesized on the various target materials under different types of background gases and laser parameters such as the laser wavelength, fluence, and pulse duration. In the year of 1987, Patil and co-workers are the first group that conducted the experiment involving laser ablation in liquid (Patil *et al.*, 1987). They used pulsed ruby laser to ablate an iron target in water to form metastable phase of iron oxide. This method is called as Liquid Phase Pulsed Laser Ablation (LP-PLA). The solid iron target was immersed in the liquid and laser beam is focused through the liquid onto the surface of solid target (Patil *et al.*, 1987). Although Patil and co-workers developed a new laser ablation technique in liquid, they unable to attract attention from other researchers. But the situation was changed after the work from Ogale (1988). The potential of LP-PLA was highlighted to modify metal surface, such as metallic oxidation, nitriding, and carbiding. This pioneering work successfully opened new era for materials processing based on the PLA of solids in various liquids. Comparing with other traditional physical methods, such as chemical vapour deposition (CVD), hydrothermal and so on (Kempa *et al.*, 2003; Yang & Zeng, 2004), LP-PLA provided a simple and clean synthesis technique. LP-PLA provided a lot of advantages when compared to others traditional physical deposition methods. For examples, LP-PLA does not produce any byproducts that contained in the final product. Other than that, LP-PLA is a cost-effective approach and is considered easier to implement compared to the more conventional use of controlled vacuum or gaseous media environments (Amans *at al*., 2009; Al-Hamaoy *et al*., 2014).

1.6 Research Motivation

The current market and demands for the smaller, more capable, and more efficient power devices are increasing along with the new technology advancement. However, the biggest issue of the power and electronic devices is excessive heat that caused the overheating problem. Any devices that operate above the optimal condition will experience a performance reduction, shorter lifetime and importantly affect the reliability of devices (Almubarak, 2017; Bi *et al.*, 2017). To overcome the heat problem, TIMs have been used and play a very important role to dissipate the heat in these devices.

Nowadays, silicone polymer is widely used as the polymer binder for high performance TIMs (Zhou *et al*., 2020; Chowdhury *et al*., 2021; Hu *et al.*, 2022). Silicone polymer is comprised by silicone backbones (Si-O) with attaching monovalent organic radicals (RSi-O). Furthermore, the silicone polymers shown excellent characteristics such as low glass transition temperature, high thermal stability and good electrical insulation (Goodman, 1989; Chowdhury *et al*., 2021) mainly relies on the addition of functional fillers (Zhou *et al*., 2020; Chowdhury *et al*., 2021). Thus, it is important to choose suitable fillers to cater to the specific application requirements of silicone polymer.

In order to develop a new silicone-based TIMs with higher thermal conductivity, several types of filler material have been examined such as alumina (Al_2O_3), carbon nanotubes (CNTs), silver (Ag) and graphite (Wong *et al*., 1999; Sim *et al*., 2005; Cola *et al*., 2007; Zeng *et al*., 2010; Theerthagiri *et al*., 2019). The filler materials such as CNTs and metal are preferred due to high thermal conductivity coefficients. The electrical and thermal conductivities of filler materials are showed in the Figure 1.3 (Lewis *et al.* 2021).

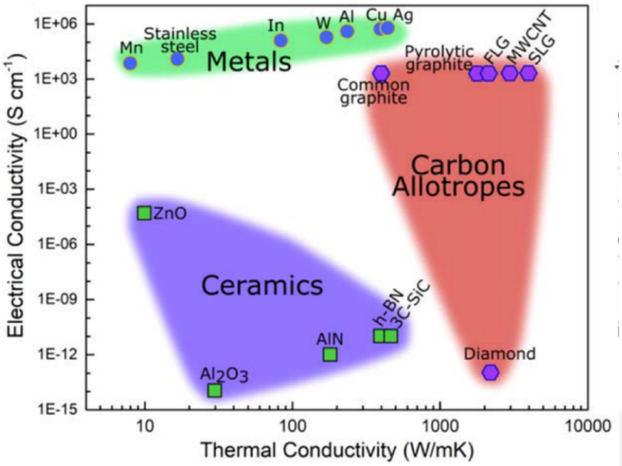

Figure 1.3: Electrical and thermal conductivities of filler materials.
(Source: Lewis *et al*., 2021)

Among many of fillers for polymers, carbon allotropes are new type of materials has drawn an extensive attention and interest for thermal related-application. Graphene and CNTs are two most recognize materials that used for the thermal related-application. Based on the several studies, graphene-based TIM have achieved better thermal conductivity and low TIR when compared to CNTs-based TIM (Shahil *et al*., 2011; Mohamed *et al*., 2020). Graphene has higher surface-to-volume ratios than CNTs because of the inner nanotube surface of CNT is inaccessible to the TIM's polymer molecules (Saifuddin *et al*., 2013; Bhattacharya, 2016). This caused the polymer molecules difficult to enter the inner nanotube surface of CNTs, thus increased the overall TIR of the polymer. This makes graphene sheets is more favorable for altering the thermal, electrical, physical and chemical properties of TIM's polymer molecules. However, it is known that the carbon atoms in the two-dimensional (2-D) honeycomb structure of graphene is very stable due to tightly packed carbon atoms and sp^2 orbital hybridization (Pareek & Mohan, 2019). Thus, the functionalized of graphene must be carried out to maximize the bonding with polymer molecules. Specifically, the chemically bond affinity of the graphene with other elements/materials is due to the oxygen functional groups in GO (Fraga *et al.*, 2020). Therefore, when the number of oxygen functional groups in GO increased, the reactivity between GO and polymer molecules also increases.

Photoreduction method is environmentally friendly approach that can be used to reduce/remove the oxygen functional groups in GO (Naik & Krishnaswamy, 2017). From their studies, Naik and co-workers (2017) found that the carbon-oxygen ratio in GO increased when the surface temperature of GO increased to 200ºC. They stated that the observation can be due to the laser heating and the heat generated due to the reduction reaction within the GO. In this method, the light/photon is used to provides the energy required for the chemical reactions, which can breakdown or create new bonds of chemical species. Basically, photoreduction can be classified into three categories, such as photochemical, photothermal and laser reduction. The photochemical method occurs when the chemical molecule absorbs the photon energy. The photothermal method occurs when the chemical molecule absorbs the heat energy, generally by an increase of the temperature of the reaction medium. In general, the photochemical process occurs when the wavelength of laser light is approaching the ultraviolet (UV) region while the photothermal process occur when the wavelength of laser light is reached above the UV region. For the laser reduction, both photothermal and photochemical were involved when the chemical molecules absorb the photon and heat energy at the same time. Thus, the photoreduction method is a cost-effective, simple and efficient when compared with compared with conventional thermal and chemical routes (Zhang *et al*., 2010).

1.7 Problem Statements

Recently, GO/reduced graphene oxide (rGO)-based polymer composites are widely used in the thermal management applications due to higher thermal conductivity coefficient (Teng *et al.*, 2011; Li *et al.*, 2017; Pan *et al.*, 2021). Teng and co-worker (2011) reported that the thermal conductivity of GO-based polymer composites is higher when compared to the single-walled carbon-nanotube (SWCNT)/epoxy composites. They also found that the thermal conductivity of composites increased when the GO content increases. Besides, Li *et al.* (2017) reported that the rGO-based thermoplastic polyurethane (TPU) composite exhibits a high thermal conductivity (0.8 W/mK) at an ultralow graphene concentration of 1.04wt.%. They found that the increment of the rGO improved the heat conduction between rGO and TPU due to higher heat flux in rGO. Pan *et al.* (2021) have prepared GO-based polymer composites by using water-soluble polyvinyl alcohol (PVA) and GO dispersion. They found that the intermolecular reaction between PVA and GO contribute to an improved thermal conductivity (~25 W/mK). Their results also indicated the thermal conductivity increases with increasing concentration of GO. From many studies above, the concentration of GO becomes an important factor to enhance the thermal conductivity of the composites.

GO as in pure form has very limited usage. Thus, the functionalization of GO is very important to enhance the physical and chemical properties of GO. GO can be functionalized by using either covalent or non-covalent approaches. Covalent functionalization of GO is the formation of covalent bond between the unsaturated carbon π-bond with oxygen-functional groups such as carbonyl, hydroxyl, and epoxides groups. This oxygen-functional groups are the most peculiar site studies for covalent functionalization of GO. There are several common reactions for covalent functionalization of GO, such as acylation, esterification, salinization, nucleophilic addition, amide formation, diazotization and cycloaddition (Innocenzi, 2014; Lei *et al.*, 2017; Wang *et al.*, 2010; Du & Cheng, 2012; Xu *et al.*, 2009; Chua & Pumera, 2012; Burress *et al.*, 2010; Wang *et al.*, 2008; Xu *et al.*, 2012; Li *et al.*, 2011; Lomeda *et al.*, 2008). While for the non-covalent functionalization, the carbon atoms in the GO planar or oxygen-functionalized GO planar interact with other molecules via hydrogen bonds, ionic interaction, π-π interaction, hydrophobic interaction, etc (Tarajeshwar & Kim, 2001; Dreyer *et al.*, 2010; Vijayakumar *et al.*, 2014). This functionalization is particularly interest as it does not affect the π-conjugation of the GO sheet and therefore retain their intrinsic thermal and electric conductivities. There are several researchers studied shown the covalent bond GO with siloxane.

The oxygen-functional group of GO affect the physical and optical properties of polymer composites. The oxygen functional groups have better interactions with various polymers as reported Wang *et al*. (2010), Du & Cheng (2012); Xu *et al.* (2009). Several numbers of GO-based polymer nanocomposites have been studied from different research groups (Wang *et al*., 2010; Du & Cheng, 2012; Xu et al., 2009) Based on the study from Wang *et al*. (2011), they prepared the polyimide-4,4'-Diaminodiphenyl ether-graphene oxide (PI-ODA-GO) composites via in situ polymerization. They functionalized the GO with the ODA. Their SEM and XRD results show that the ODA monomer successfully attached to the epoxy groups on the GO surface via nucleophilic attack by amine. Xu *et al*. (2009) modified the surface of graphene via the covalent attachment of a porphyrin ring on the GO surfaces. They used thionyl chloride to activate the carboxylic acid group in the presence of porphyrin using N,N-dimethylformamide (DMF) as a solvent. Unfortunately, the chemical interaction of GO with polymer is a complex process and some possible interactions have been proposed to explain the interaction (Bhattacharya, 2016) based on their unique influences on the physical, thermal, electrical and optical properties of polymer nanocomposites.

Based on the studies of electrical behavior of GO-based polymer composites, the electrical conductivity was found to highly depending on filler type, shape, size, and filler dispersion and distribution in the composite. The electrical conductivity coefficient of GO-based polymer composites was found to enhance when the concentration of GO is higher (Campbell, 2010; Cassinese, 2011). The conductive path is formed which make the free electrons in the composite to travel easily, and eventually raised up the electrical conductivity of the composite.

The thermal properties study performed by Luo and Lloyd (2012) show the influences of graphene size, interfacial bonding strength, and polymer density on the interfacial thermal transport. They found the thermal conductivity of graphene/graphite-polymer composites was improved by increasing the size of graphene particles which led to extra covalent bonds between the graphite edges and polymer molecules. However, the effect of geometries of the graphene/graphite also affects the overall thermal conductivity of the composites (Balandin, 2011; Pop *et al.*, 2012). Basically, thermal conductivity is the intrinsic property of a material which relies on the transport of electrons and phonons. The thermal conduction is dominated by phonons when few free electrons are observed in the 2-D materials limitedly. It is also noticed that thermal conductivity in 2-D materials varies significantly in different planes due to their anisotropic atomic structures. Based on the literature studies, the in-plane thermal conductivity of graphene is the highest (2000-4000 W/mK) at room temperature when compared with other materials, such as CNTs and graphite (Dresselhaus *et al.*, 1996; Balandin, 2011; Pop *et al.*, 2012). This is because of the strong covalent sp^2 bonding resulting in high in-plane group velocities of phonons and low crystal lattice anharmonicity for in-plane vibrations. By contrast, heat flow in the out-of-plane direction of 2-D material is strongly limited by weak Van der Waals force, thus, the thermal conductivity is very low (6 W/mK) at room temperature (Dresselhaus *et al.*, 1996; Balandin, 2011; Pop *et al.*, 2012).

The laser-ablated GO exhibits strategic enhancements in dispersion and interaction within the polymer matrix, leading to the optimization of thermal transport efficiency across the interface (Altuwirai, 2022). This advancement not only elucidates the effectiveness of laser ablation in modifying the material's structure but also opens up a promising avenue for crafting high-performance polymer-based TIMs with tailored thermal properties. This development suggests significant progress in thermal management applications. Besides, composite materials were fabricated with varying concentrations of GO, revealing a clear and substantial correlation between GO concentration and improved thermal conductivity (Lewis *et al.*, 2021). These results underscore the critical role of GO in refining heat transfer pathways within the TIM, emphasizing that elevated concentrations of GO contribute to superior thermal performance. This insight not only adds to our fundamental understanding of heat dissipation mechanisms in GO-based TIMs but also carries practical implications for the design of advanced thermal management solutions across diverse applications, including electronics and microelectronics.

1.8 Objectives of the Research

The main objective of the research project is to synthesis GO with silicon polymer composite using laser ablation method and to investigate the thermal properties of the GO-silicon polymer for thermal application. To achieve this purpose, the research work objective is constituted and deliberated further. Therefore, the objectives of the research are divided into three small objectives to facilitate the research work.

The research objectives for this present study will be stated as follow:

- To study the effect of laser ablation on the structure of GO-TEOS mixture for thermal interface materials.
- To characterize the chemical composition, physical and optical properties of GO-TEOS mixture solution with different GO concentration.
- To investigate electrical and thermal conductivities of GO-TEOS mixture with different GO concentration.

1.9 Research Scope

This new light-induced functionalization of GO in solutions has been considered to emulate a great deal of attention as an effective way for obtaining an acceptable degree of exfoliation and improvement of silicone polymer performance with GO. When a high-power pulsed laser beam irradiates the sample, the extremely high-energy from the laser breaks down the chemical bonds thus functionalizing the GO with TEOS polymer. The synthesized GO-silicone polymer is a new type of materials which drawn an extensive attention and interest for thermal related-application.

Fourier Transform Infrared (FTIR) is used to determine the chemical composition and the vibration modes in the mixture solution. Raman spectroscopy is used to distinguish the number of graphene layers and quantify the effect of disorder on its properties of the GO. Besides that, surface topography and lattice spacing of samples will be determined by using Transmission Electron Microscopy (TEM). The particle size distribution of the materials is determined by using Particle Size Analyzer (PSA) utilizing dynamic light scattering (DLS) technique in the particle size range of 0.2μm to 1000μm.

The conductivity and dielectric measurements of the GO-TEOS mixture ranging between 10 Hz to 1 MHz will be performed to determine the electrical properties of the laser ablated GO-TEOS mixture. In addition, the phase transition and specific heat capacity of samples is determined by using Differential Scanning Calorimetry (DSC) in the range of 30°C to 125°C with sapphire as the reference material. Moreover, thermal conductivity of the samples is determined by using TEMPOS thermal analyzer.

1.10 Thesis Outline

This thesis will be separated into 5 chapters.

Chapter 1 contains the brief overview of the research. This chapter provides a short introduction of the materials and relevant technologies used in the research. Besides, the motivation and problem related to current thermal management system in various industries also discussed. Lastly, the objectives of the research are listed in this chapter.

Chapter 2 discusses the mechanism of the thermal conductivity of the materials. The related fabrication methods are mentioned in this chapter and used as guidance to determine the most effective fabrication method. Other than that, the types of carbon allotropes are also discussed in this chapter. Furthermore, this chapter also deliberates the carbon-family nanocomposites fabrication methods. Besides that, current research and findings related to the laser ablation process are also discussed in this chapter.

Chapter 3 presents the methodology of the sample preparation and characterization techniques that used to measure the physical, optical, thermal and electrical properties of the samples. In addition, the data analyzing techniques for samples are also discussed in this chapter.

Chapter 4 reports the experimental result of physical, optical, electrical and thermal characterization of the samples. The analysis of the experimental results and discussion of the findings of the chemical composition, physical, optical, electrical and thermal properties for the fabricated samples are presented.

Chapter 5 presents the conclusion of the research work. All the finding from analyzed results in Chapter 4 are summarized. A brief summary of the main results is presented together with possible future research directions.

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