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Liquid Silicone Rubber as Secondary Matrix in Epoxy/Graphene Nano-Platelets (GNP) Conductive Materials

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ABSTRACT

Graphene nanoplatelets (GNPs) were employed as conductive fillers in an epoxy/liquid silicone rubber (LSR) system, with a range of filler loadings from 5 vol.% to 20 vol.%. An investigation was carried out to assess the mechanical, electrical, and thermal stability properties. At low GNP loading (0.2 vol.%), a notable enhancement in the toughness of epoxy/LSR/GNP system was observed. At high GNP loading (0.8 vol.%), the toughness and flexural properties were slightly reduced, but there was a dramatic increase in electrical conductivity. The epoxy system with 5 vol.% of LSR and 0.8 vol.% of GNP had better mechanical properties but lower electrical conductivity compared to the epoxy system with 20 vol.% of LSR and 0.8 vol.% of GNP. Moreover, the epoxy/LSR/GNP system exhibited superior thermal stability compared to the pure epoxy. The incorporation of GNP nano-fillers in the epoxy/LSR system resulted in an improvement in mechanical, thermal, and electrical properties.

Keywords: *Graphene nano-platelets, epoxy, liquid silicone rubber, conductive polymer composites*

1. INTRODUCTION

In recent times, conductive materials that offer high strength, toughness, wide accessibility, and affordability are required in various fields of application, such as antistatic coatings, conducting adhesives, sensors, and electromagnetic interference shielding materials [1]. Polymer composites have emerged as exceptionally versatile conductive materials capable of fulfilling these requirements. Transforming from insulators to conductive materials can be done by mixing [2], doping [3], and coating [4].

Epoxy resins are widely utilized as thermosetting matrices in composites and nanocomposites due to their impressive mechanical strength, thermal stability, and low fatigue [5]. However, epoxy resins are characterized by their intrinsic brittle nature due to extensive cross-linking density in the cured state, which results in limitations in mechanical properties, including weak resistance to crack propagation, and they are prone to fracture [6], [7], [8].

To intentionally address this issue, a common strategy for increasing the toughness of epoxy resins is introducing a small proportion of liquid silicone rubber into the epoxy matrix [9]. Instead of using other types of rubber, liquid silicone rubber is employed due to its unique properties, such as superior thermal stability, oxidation resistance, and environmental stability over a broad temperature spectrum from -50°C to +75°C, attributed to the presence of Si-O bonds within its chemical structure [10], [11].

The incorporation of conductive nano-fillers into epoxy resins is seen as a promising strategy to create multifunctional thermosetting materials with exceptional mechanical and electrical properties, thus expanding their potential applications in electronic devices [1], [7]. Among conductive nanofillers, graphene nanoplatelets (GNP) have become increasingly appealing as nanofillers for conductive polymeric materials due to their remarkable combination of multifunctional properties, such as excellent thermal, mechanical, and electrical properties, and cost-effectiveness [1], [7].

The objective of this study was to explore the impact of graphene nanoplatelet loading and liquid silicone rubber content on the mechanical and electrical conductivity properties of epoxy/liquid silicone rubber (LSR)/graphene nanoplatelets (GNP) conductive materials. This study specifically focuses on investigating the optimum LSR content based on fracture toughness and flexural performance and the influence of GNP loading (in vol.%) in

epoxy resins to identify the percolation threshold of these conductive materials, which depends on their electrical performance.

2. EXPERIMENTAL PROCEDURE

2.1. Materials

Crystal clear epoxy resin (DGEB-A) has an epoxide equivalent weight of 182–192 g/Eq and a viscosity of 11,000–14,000 MPa s at 25 °C. Crystal clear epoxy hardener (isophorone diamine) with amine value 260–284 (mg KOH gm^{-1}) and an equivalent weight per active-H of 110. Crystal clear epoxy resin and epoxy hardener were purchased from Euro Chemo-Pharma Sdn. Bhd. (Malaysia). Liquid silicone rubber G450 was purchased from Euro Chemo-Pharma Sdn. Bhd. Graphene nano-platelets were purchased from SkySpring Nanomaterials (USA) with a thickness of 11–15 nm and in-plane bonding of 0.3408 nm.

2.2. Sample Preparation

The epoxy/LSR system with 5 vol.% LSR and 20 vol.% LSR and varied content of GNP, which was 0.2, 0.4, 0.6, 0.8, and 1.0 vol.% filler loading, was prepared. This part was carried out to determine the percolation threshold of GNP.
To prepare the epoxy/LSR/GNP system, GNP nanofiller was incorporated into the epoxy/LSR mixture through magnetic stirring for 15 min. The GNP nano-fillers were dispersed in the epoxy/LSR system with the aid of an ultrasonic water bath for 30 min at a frequency of 53 kHz and 60 °C. The mixture was stirred well with epoxy hardener and was cured in an oven at 90 °C for 2 h.

2.3. Sample Characterization

Flexural strength and modulus were measured using a Universal Instron testing machine (Model 5569). The testing was performed according to ASTM D790, under a three-point bending method with a span length of 50 mm at a crosshead speed of 2.38 mm min^{-1}. The rectangular samples had dimensions of 60 mm in length, 12.7 mm in width, and 3mm in thickness. Fracture toughness (KIC) was evaluated in accordance with the standard of ISO 13586 under Mode I (tensile opening) by using a Universal Instron testing machine (Model 5569) at a crosshead speed of 1 mm min^{-1}. The rectangular samples had a notch created at a point one-third of the sample's width, and their dimensions were 60 mm in length, 12.7 mm in width, and 3mm in thickness. The fractured surfaces of the samples were examined using scanning electron microscopy (SEM), specifically the JEOL JSM-6460 LA model, at an accelerating voltage of 10 kV. The fractured surfaces of samples were coated with a thin layer of palladium by using an auto fine coater machine with a model of JEOL JFC 1600 since the specimen must be electrically conductive to avoid electrostatic charging. The electrical parameters were measured at room temperature using a fully automated high-precision LCR meter (HIOKI-IM3536) and a four-terminal probe (HIOKI-L2000) over a frequency range of 10 Hz to 1 MHz. The circular-shaped samples, which had a diameter of 10 mm and an optimum thickness of 1.0 mm, were subjected to this measurement. The capacitance (c), bulk resistivity, and bulk conductivity were recorded directly.

3. RESULTS AND DISCUSSION

The mechanical properties, electrical properties, and thermal properties of the epoxy/LSR/GNP system were discussed with 5 vol.% LSR and 20 vol.% LSR, and GNP loading varied from 0, 0.2, 0.4, 0.6, 0.8, and 1.0 vol.% GNP.

3.1. Mechanical Properties

Figure 1 shows the impact of GNP content (vol.%) on the flexural strength of pure epoxy and epoxy/LSR/GNP composites with 5 vol.% and 20 vol.% LSR. The epoxy with 5 vol.% and 20 vol.% of LSR has lower flexural strength compared to the pure epoxy. Introducing 0.2 vol.% GNP filler reduced flexural strength, while increasing GNP loading from 0.2 vol.% to 1.0 vol.% led to further reductions in flexural strength compared to the epoxy/LSR system without GNP.

Higher GNP content resulted in decreased flexural performance, a typical behavior in composites with nano-scale fillers, likely due to poor GNP dispersion [12], [13]. Poor dispersion is caused by GNP re-stacking and aggregation due to strong π–π bonding and intermolecular forces, which create stress concentrators that reduce flexural strength [14].

At the same GNP loading, composites with 20 vol.% LSR showed lower flexural strength than those with 5 vol.% LSR and pure epoxy. Higher LSR content results in larger particles and less contact area with the epoxy, weakening interfacial interactions. Additionally, more LSR occupies space in the epoxy matrix, leading to higher GNP packing density and poorer dispersion.

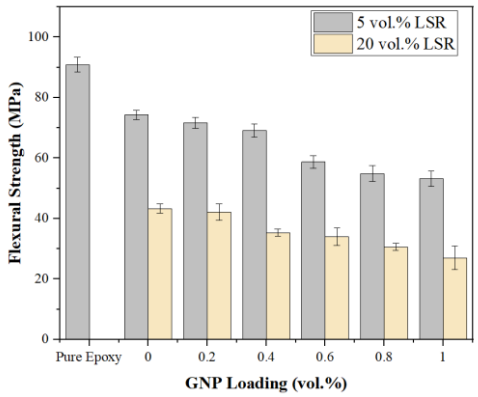

Figure 1. Effect of GNP loading on flexural strength of epoxy system with 5 vol.% LSR and 20 vol.% LSR.

Figure 2 shows the flexural modulus of GNP-filled epoxy resin with 5 vol.% and 20 vol.% LSR at various GNP loadings. The flexural modulus of epoxy/LSR/GNP composites with both 5 vol.% and 20 vol.% LSR is higher than that of epoxy resin with only LSR. This increase is mainly due to the high modulus and stiffness of GNP [15].
For composites with 5 vol.% LSR, the flexural modulus increases as GNP loading rises from 0.2 vol.% to 1.0 vol.%, benefiting from the mechanical properties of graphene, which enhance stiffness and modulus. Higher GNP loading also restricts epoxy chain movement, further improving stiffness and modulus [16].

In composites with 20 vol.% LSR, the flexural modulus increases up to 0.2 vol.% GNP loading but decreases with further loading up to 1.0 vol.%. High LSR content hinders GNP dispersion, leading to agglomeration and local stress concentrations, which reduce the overall flexural modulus [17].

At the same GNP loading, composites with 5 vol.% LSR have a higher flexural modulus than those with 20 vol.% LSR. The flexibility and elastomeric properties of LSR counteract the reinforcing effects of graphene, decreasing the flexural modulus as LSR content increases [18], [19].

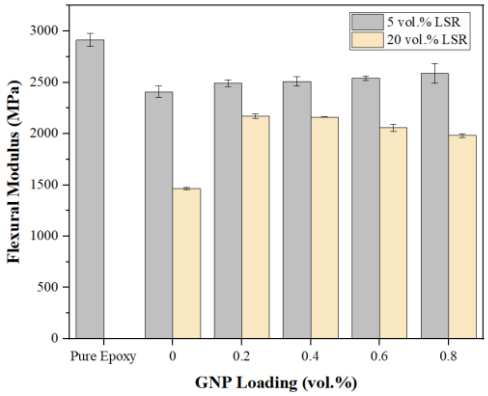

Figure 2. Effect of GNP loading on flexural modulus of epoxy system with 5 vol.% LSR and 20 vol.% LSR.

Figure 3 illustrates the fracture toughness of epoxy resin filled with GNP at 5 vol.% and 20 vol.% LSR with varying GNP loadings. For epoxy with 5 vol.% LSR and 0.2 vol.% GNP, enhanced fracture toughness is shown compared to the pure epoxy and epoxy/LSR system without GNP, but increasing GNP to 1.0 vol.% reduces it. This reduction is likely due to poor cross-linking and GNP dispersion at higher loadings [20].

In composites with 20 vol.% LSR, fracture toughness decreases as GNP loading increases. Higher LSR content leads to larger particle sizes and less contact area with the epoxy, weakening interfacial bonding [9].

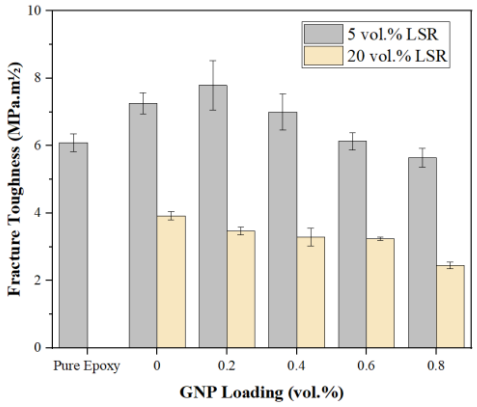

Figure 3. Effect of GNP loading on fracture toughness of epoxy system with 5 vol.% LSR and 20 vol.% LSR.

Figure 4 shows SEM images indicating that as LSR content increases, the particle sizes of LSR phases grow due to the coalescence of rubber particles during curing, leading to larger and fewer rubber inclusions. These enlarged rubber particles tend to have poor interfacial bonding with the surrounding epoxy matrix. This results in cracks propagating through the poorly adhered interface between the epoxy matrix and larger LSR phases, limiting crack energy dissipation and causing detachment of LSR phases from the epoxy matrix. Consequently, mechanical properties are reduced when LSR content increases.

At lower LSR content (5 vol.%), SEM images reveal more evenly distributed rubber particles and more tortuous crack paths. These features indicate improved energy dissipation during fracture, as the cracks are deflected or absorbed by the well-dispersed LSR domains. This leads to enhanced toughness under mechanical loading.

In contrast, at higher LSR content (20 vol.%), the SEM images show smoother, more continuous crack propagation through the matrix, often along the interface between the epoxy and larger rubber particles. This indicates weak interfacial adhesion and limited crack resistance, which in turn reduces the composite's mechanical strength and toughness. At 500x magnification, the epoxy composite with 5 vol.% LSR exhibits denser crack lines compared to the 20 vol.% LSR system, indicating higher energy dissipation.

The LSR content also affects the distribution of GNP in the epoxy/LSR/GNP composite system, which in turn influences the mechanical failure behavior. In the system containing 5 vol.% LSR and 0.8 vol.% GNP, the microstructure shows that the GNPs are spread out fairly evenly. Features such as crack bridging and pull-out can be observed, indicating good load transfer between the GNPs and the surrounding matrix. This improves the fracture toughness of the material and helps slow down crack growth.

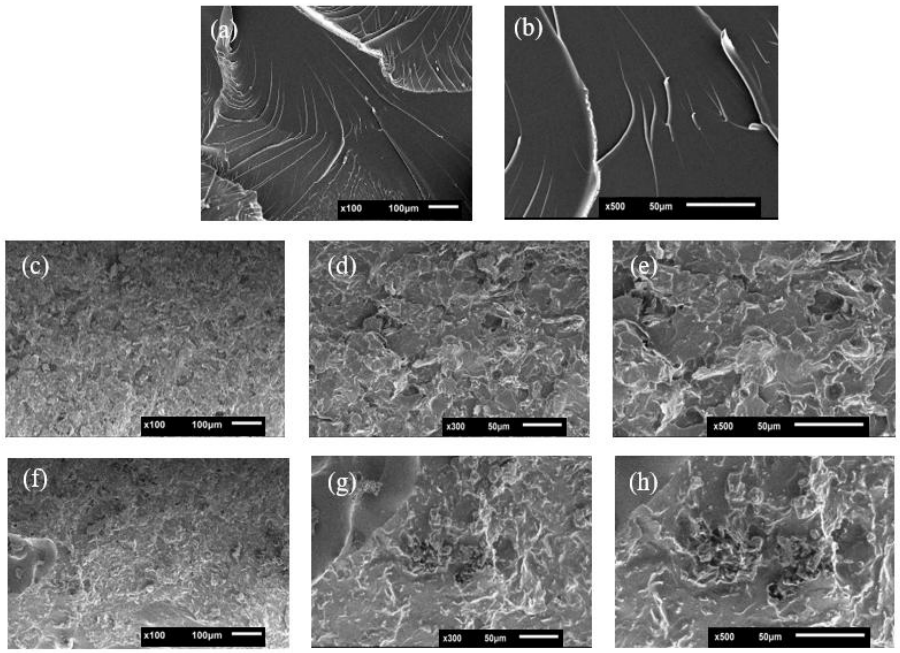

Figure 4. SEM micrographs of the fractured surfaces of the epoxy/LSR/0.8 vol.% GNP system with (a), (b) pure epoxy, (c), (d), (e) 5 vol.% of LSR contents, and (f), (g), (h) 20 vol.% of LSR contents.

On the other hand, the system containing 20 vol.% LSR exhibits noticeable agglomeration of GNPs and an uneven matrix structure, caused by increased phase separation. These GNP clusters are often weakly bonded to the surrounding epoxy, forming weak spots and stress concentration areas that make it easier for cracks to start and spread. As a result, these zones are ineffective at stopping or bridging cracks, leading to early mechanical failure. Therefore, while a moderate amount of LSR helps achieve better GNP distribution and enhances toughness, too much LSR disrupts the structural integrity and results in more brittle failure of the composite.

3.2. Electrical Properties

Figure 5 shows the impact of GNP loading on AC electrical conductivity, impedance, dielectric constant, and dielectric loss in epoxy/LSR/GNP systems with 5 vol.% and 20 vol.% LSR. At low GNP levels, the system shows low electrical conductivity and high resistivity. Increasing GNP loading to 0.8 vol.% significantly boosts conductivity and decreases resistivity. Beyond this, at 1.0 vol.% GNP, these properties stabilize.

According to percolation theory, at low GNP loading (0.2 vol.%), GNP disperses evenly in the epoxy with minimal contact between fillers [21]. As GNP loading rises, more nanofillers connect, forming conductive pathways. At 0.8 vol.% GNP, a percolation network forms, allowing electron flow. Further increasing GNP to 1.0 vol.% does not create more conductive pathways due to GNP agglomeration [22].

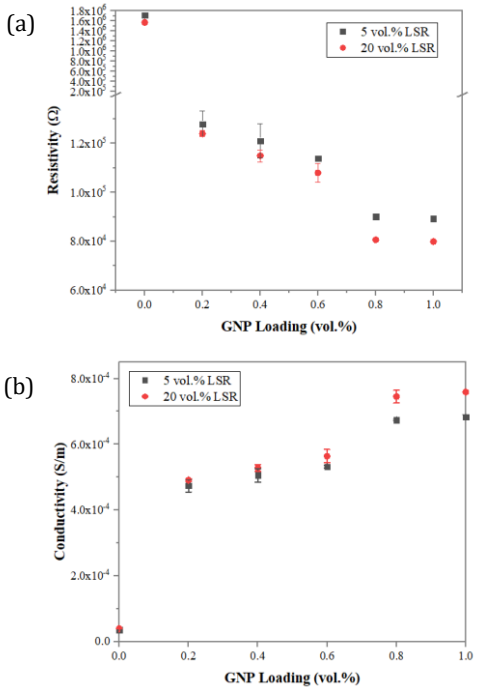

Figure 5. Effect of GNP loading on (a) resistivity, (b) conductivity, epoxy/LSR/GNP system.

At the same GNP loading level, the epoxy/LSR/GNP system with 20 vol.% LSR exhibits slightly higher conductivity and slightly lower resistivity compared to the system with 5 vol.% LSR. The presence of LSR acts as an elastomer spacer, embedding the conductive filler more closely within the epoxy matrix, thus facilitating the formation of a conductive network. Consequently, a higher LSR content (20 vol.%) enhances the system's conductivity.

3.3. Thermal Properties

Figures 6(a) and (b) depict the TGA and DTG curves of pure epoxy and epoxy/LSR/GNP systems. Thermal resistance was evaluated by determining the temperature at which a 5% weight loss occurred (T_5) and T_{max}. Results in Table 1 show that both epoxy/LSR/GNP systems, with 5 vol.% and 20 vol.% LSR, exhibit higher T_5 and T_{max} compared to pure epoxy. The addition of GNP enhances thermal stability due to its excellent inherent thermal conductivity, preventing heat buildup within the system. Improved stability may also result from reduced polymer chain mobility induced by epoxy-GNP interaction at the interface [2].

Table 1. The T_5, T_{50}, T_{d1}, and T_{max} of the pure epoxy and epoxy/LSR/GNP systems

Samples	T_5 (°C)	T_{50} (°C)	T_{d1} (°C)	T_{max} (°C)
Pure Epoxy	183.20	355.52	336.55	381.40
Epoxy/LSR/0.8 vol.% GNP (5 vol.%)	200.70	368.45	345.88	399.11
Epoxy/LSR/0.8 vol.% GNP (20 vol.%)	201.50	375.95	345.12	398.95

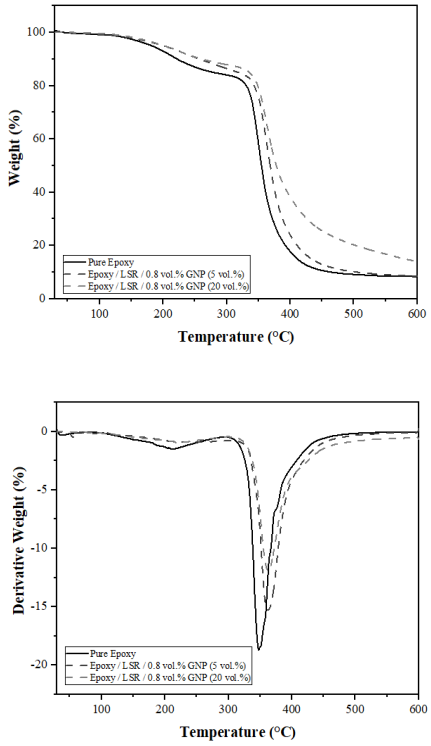

Figure 6. (a) TGA and (b) DTG curves of pure epoxy and epoxy/LSR/GNP systems.

At the same GNP loading, the epoxy/LSR/GNP system with 20 vol.% LSR demonstrates higher T_5 compared to the 5 vol.% system. The Si-O functional group in LSR has higher bond energy, requiring more energy to break LSR-LSR interactions at higher LSR content [18]. However, the Tmax of the 20 vol.% LSR system is slightly lower than the 5 vol.% system. Elevated LSR content increases GNP packing density, leading to agglomeration and reduced interfacial interactions between epoxy and GNP, requiring less heat to break these bonds [23].

4. CONCLUSION

In summary, the percolation threshold of GNP on both epoxy/LSR/GNP systems (5 vol.% and 20 vol.% of LSR) was 0.8 vol.%. The increase in a small amount of GNP loading (0.2 vol.%) in epoxy/LSR/GNP composite with 5 vol.% of LSR improved the fracture toughness of the system but did not significantly enhance the system with 20 vol.% of LSR. The flexural strength of both systems decreased as the GNP loading increased. As anticipated, ~~the~~ incorporation of GNP likewise enhanced the thermal stability and conductivity of both systems. The introduction of LSR further improved the thermal stability and conductivity of both systems.

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