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Review

Laser-Induced Graphitization of Thermosetting Polymer Substrate and its Application—A Review

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Abstract. Laser-induced graphene (LIG) production and application is currently receiving tremendous attention of the research community, perhaps because of its facile, clean, and sustainable nature. Thus, any critical review about the subject matter cannot be over-emphasized. Herein, the stage-wise procedure of producing the LIG that involves preparation of polymer substrate, specification of laser machine parameters/condition, laser-induced irradiation on the prepared substrate, and characterization/confirmation of the LIG have been explained, and most importantly the various applications of the LIG in the areas of energy and power, electromagnetic interference (EMI) shielding, environmental, and sensor development have also been reviewed. It is worthy of nothing that polyimide film is predominantly employed by the different independent researches as substrate in the LIG production and applications, and fascinating findings were found. Furthermore, future perspective of LIG production and applications have been suggested. Overall, information summarized herein will no doubt provide basis for research and development in the area of LIG production and application.

Keywords: Graphene, laser irradiation, LIG, LIG production, LIG applications.

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1. Introduction

Graphene is an allotrope of carbon exhibiting extraordinary properties such as mechanical strength, surface area, conductivity, and optical and chemical properties [1]–[3]. The extraordinary properties are being exploited since its discovery and put to further investigation by the research communities across the fields of engineering, medicine, and life and physical sciences. It is traditionally synthesized by mechanical, chemical deposition, liquid exfoliation, and flash joule heating methods [4]. But overtime, the aforementioned traditional methods are classified to have hectic fabrication process, high energy expenditure, and very low throughput [4],[5].

In the year 2014, laser-assisted graphene (LIG) was accidently established, and thereafter the technique has emerged as a versatile and efficient alternative method that can be easily up scaled fabrication of graphene on thermosetting polymer substrates with desired specific designs. The technique involves the utilization of substrates which are high temperature resistant thermoplastic materials. It was found to have advantages of; synthesis of larger surface area graphene and desired designs with a single production step, not needing expensive cleanroom equipment or/and wet chemical steps, reducing agents, and solvents, not needing any subsequent treatments [4],[6]. Recently, studies established significant performance of LIG in energy and power [7],[8],[9], electromagnetic shielding [10],[11], environmental [12], and other applications [2],[13],[14],[15],[16]. LIG has becomes a very interesting research direction and that could be corroborated from the data obtained from a literature survey conducted in the web of science data base where a total of 2,964 and 424 research and review articles were found published in the last six years (Fig. 1(a)).

However, as regards to the published research articles, different research groups contributed differently by using different polymer substrates and laser irradiation condition in the production of LIG, and putting the LIG produced to unique application. While on the other hand, the published review articles differs in terms of scope and perspectives. So far, there is little attention towards providing a comprehensive review strictly on the thermosetting polymer based LIG production process, its recent applications, and future perspectives.

Thus, this review focused on establishing a mini but comprehensive, and up-to-date information regarding the LIG production process, LIG applications, and future perspective of the thermosetting polymer based- LIG. Certainly, the information contained herein will provide basis for student and researchers working on the subject matter.

2. LIG Production from Thermosetting Polymer Substrate

The LIG production is a stage-wise process that involves preparation of polymer substrate, specification of laser machine parameters/condition, laser-induced irradiation on the prepared substrate, and characterization/confirmation of the LIG as described in Fig. 1(b). Firstly a substrate is produced as a precursor on which the LIG will be formed. Mostly, polymer films formed from phenolic resin, polyimide, polysulfone, and polybenzoxazine. Among the polymer substrates used, polyimide substrate was found to outperform other polymer substrates. Perhaps due it it's significantly higher thermal and char yielding characteristics [17]. Such characteristic shown relative to polybenzoxazine in Fig. 1(c) could be attributed to the strong intermolecular forces between the polymer chains and the stacked aromatic heterocyclic structure of the polyimide. Thus, alloying polybenzoxazine and polyimide could produce polybenzoxazine-based substrate with improved characteristics. As polybenzoxazine also offers some exceptional properties such as easy polymerization process, near-zero shrinkage and non-release of toxic products due to polymerization, and molecular design flexibility [18],[19].

Secondly, after placing the substrate on the working table of the laser machine, laser parameters such as power, wavelength, scanning speed, pulse density, image density, and focusing are adjusted to optimum magnitudes based on the size and microstructural properties of the polymer used in the production of the substrate film. For instance, it has been established that microstructure, surface morphology, thickness, and sheet resistance of the LIG is significantly affected by the laser power, scanning speed, image density, and focusing [9]. The authors explained that irradiation dose could be regulated by majorly controlling the laser power and /or the writing speed. Application of high laser power directly means application of high irradiation dose to the substrate material, while lower writing speed allow high spot overlap and thus high irradiation dose.

Therefore, it is suggested that parameters should be adjusted to the optimum based on preliminary experiments, to ensure that optimum irradiation is applied to the substrate through the focusing head of the laser machine.

Lastly, the obtained LIG can then be subjected to some classical characterization techniques (Raman spectroscopy, Scanning electron microscopy (SEM), and x-ray diffraction analysis (XRD)) for the LIG confirmation.

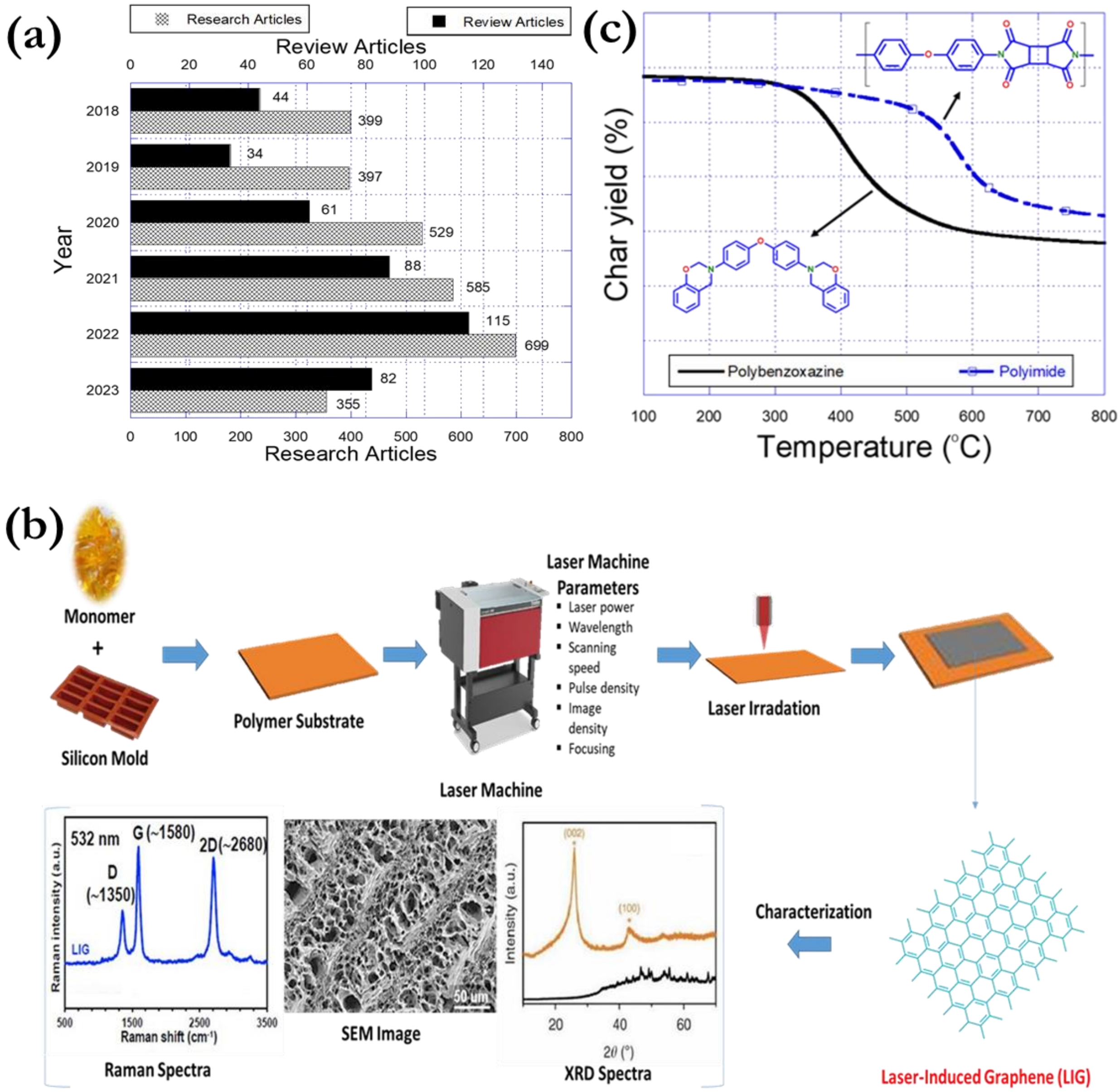

Fig. 1. (a) Data on LIG articles published on the web of science in the last six years; (b) Production of LIG; (c) Chemical structure and thermogravimetry of polyimide and polybenzoxazine.

3. Applications of Laser-induced Graphene

Progress in research and developments related to LIG have identified and explored different applications of the thermosetting polymer based LIG, the areas of such applications includes; energy and power, electromagnetic interference shielding, environmental protection and management, sensors, and others (Fig. 2). The following sub-sections presents some important explanations regarding such applications.

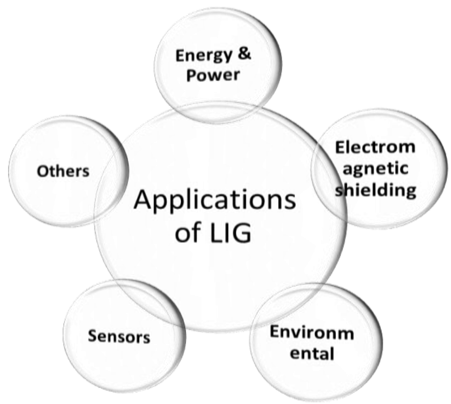

Fig. 2. Applications of LIG.

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Table 1 Characteristics of LIG based super-capacitors.

Substrate used	Configuration	Specific capacitance (m/Fcm^2)	Power density (W/cm^3)	Reference
Polyimide	Sandwich	9	4	Peng et al [23]
Polyimide	In-plane	16.5	4	Peng et al[24]
Polyimide/CMC	In-plane	60.6	-	Yuan et al [21]
Polyimide	In-plane	20-25	11.8	Li et al [25]
N & P doped Polyimide	In-plane	70.1	9.9	Parthiban et al [20]
B & F co-doped Polyimide	-	49.81	-	Yuan et al[22]
Polyimide	In-plane	4	9	Lin et al[26]
Phenolic	In-plane	0.78	-	Zang et al[27]
Polybenzoxazine	In-plane	4.8-22.2	-	Cao et al[28]
Polethersulfone/lignin	In-plane	22	1.53	Sun et al [29]

CMC: Sodium carboxymethyl cellulose, N: Nitrogen, P: Phosphorous, B: Boron, F: Fluorine.

3.1. Application of LIG in Energy and Power

Different independent research groups have established excellent applications of the LIG in the areas of energy storage, storage batteries, proton exchange membranes, and electro-catalysis. LIG based energy storage is a concept where an LIG is used to form electrodes, the electrodes formed were used with an insulation layer to fabricate a super-capacitor that stores charge in a non-Faradaic form [9]. The super-capacitors could be made into two different categories, i.e. either of in-plane or sandwich configuration. In in-plane configuration, the LIG electrodes are arranged in an interdigital manner with a portion between the electrodes filled with electrolyte. While on the other hand, the sandwich configuration is made to have the LIG layers arranged in alternate with the portion occupied by electrolyte [9]. The prominent parameters measured to characterize the LIG based super-capacitor used in energy storage are specific capacitance (m/Fcm^2) and power density (W/cm^3). Table 1 presents a summary of some findings established by some independent research groups. Overall, 4-361 m/Fcm^2 and 1.53-11.8 W/cm^3 ranges of specific capacitance and the power density have been reported respectively using the thermosetting polymer based LIG.

Findings shown in Table 1 suggests that polyimide is the most commonly used polymer as the substrate in the production of LIG based super-capacitors, and although inert environment is reported to relatively facilitate formation of thinner LIG films with higher conductivity, almost all the studies reported, ambient air is the most used environment in the production of the polymer based LIG. Also, as it could be seen, the specific capacitance and the power densities of the super-capacitors are roughly within the range irrespective of whether it was made to have in-plane or sandwich configuration. However, it is obvious that doping a polymer substrate with some active dopants could impact a highly significant improvement in both the specific capacitance and the power density relative to lonely polyimide substrates as achieved by Parthiban et al [20], Yuan et al [21] and Yuan et al [22] (Table 1). Improvements in the performance of the super-capacitor is also possible by blending polymers with desirable properties in the synthesis of the substrate to be used in the production of the LIG , so far there is limited or no study towards such direction to the best of our knowledge.

The typical layouts of the in-plane and sandwich super-capacitors are depicted in Fig. 3.

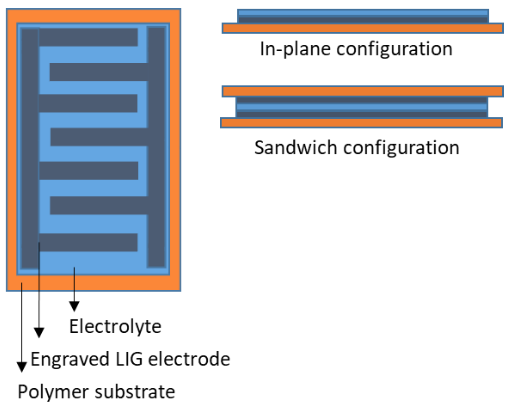

Fig. 3. LIG-based super-capacitor.

Recently, polymer based LIG have also been applied in solving problem that has to do with energy storage batteries. Storage batteries are no doubt the most essential and efficient energy storing device so far, but challenges that has to do instability of the its components (electrodes and electrolytes) during its active service leads to deposition of lithium metal which results to a serious decline in columbic efficiency and life span of the batteries [30]. To solve the aforementioned challenge, Yie at al [30] fabricated polyimide film based LIG and used it as walls of a polyimide film. The inclusion of LIG therein significantly improves stability of the components especially the anode of the lithium battery which reduced nucleation and deposition of metals thereby significantly improving columbic efficiency of the battery, and its life span by about hundred percent. The authors attributed the improvements achieved to the high defects and heteroatoms present in the LIG. Similar intervention has also been reported by Xiao et al [31] were a combination of LIG and lithiophilic MnO_X nanoparticles were used to improve columbic efficiency and life span of the energy storage battery.

Application of LIG in proton-exchange membrane fuel cell (PEMFC) has also been explored. It could be known that PEMFC technology have entered the early stages of commercialization, and traditionally, porous nano-carbides, pyrolyzed carbon black powder, carbon aerogels and graphene materials are usually used in making a vital component called the microporous layers (MPLs) of the PEMFC. However, findings reveal that the achieved mechanical and chemical stability of the MPLs with the aforementioned traditional materials is inferior and problematic. Consequently, Tiliakos et al [32] provided a useful intervention where polyimide based LIG was used instead of the aforementioned traditional materials to make the MPLs, and results obtained suggests that the power density got increased by 20% relative to MPLs made from carbon black.

In another development, polyimide based LIG was also found applicable as catalyst on the air electrode of a zinc-air batteries to improve the electro-catalysis performance [33]. The research group (Ren *et al.*[33]) prepared metal oxide/LIG hybrid catalyst which was reported to exhibit good performance, suggesting the hybrid catalyst as an excellent cathode catalyst for rechargeable Zn-air batteries, where 98.9 mW/cm^2, 842 Wh/kg, and 500 were recorded as peak discharge power density, energy density, and cycles of charge and discharge, respectively.

3.2. Application of LIG in Electromagnetic Shielding

Yu et al [11] demonstrated the application of LIG in the electromagnetic interference (EMI) shielding exploring the concept depicted in Fig. 4. LIG was produced from the synthesized polybenzoxazine substrate and defocusing method was used to improve the pore formation and electrical conductivity of the LIG, where sheet resistance of as low as 2.55 'Ω/sq was achieved. More so, shielding effectiveness of 24.8 dB at 68 μm thickness and 32.7 dB at 53 μm thickness were achieved at both 8-12.5 GHz frequency for the LIG and LIG embedded with Fe_3O_4 (LIG/Fe_3O_4), respectively. This result suggests that the introduction of Fe_3O_4 nanoparticles in LIG have significantly improved the EMI shielding effectiveness of the LIG, perhaps due synergistic effects of the conductivity and magnetic property offers by the LIG and the Fe_3O_4 respectively.

3.3. Application of LIG in Environment

LIG was also found applicable in adsorption of polluted water. For instance, Jiang et al [12] employed polybenzoxazine based LIG anchored with Fe_3O_4 nanoparticles in adsorption of ethylene blue (MB) from an aqueous solution, where maximum adsorption capacity of 350.9 mg/g was recorded. The adsorption kinetics and isotherms were also studied and findings shows that the result trend suits the Langmuir pseudo-second-order kinetic model. Rathinan et al [34] has also reported a similar study, where LIG fabricated from polyimide film was used as adsorbent for water treatment. From their result, it was observed that the LIG was effective in the removal of MB and methylene orange (MO) from aqueous solution. Maximum adsorption capacity of 926mg/g and 132 mg/g were achieved against MB and MO respectively, and the adsorption processes have also followed the Langmuir pseudo-second-order kinetic model. The concept used by both Jiang et al and Rathinan et al are described in Fig. 5.

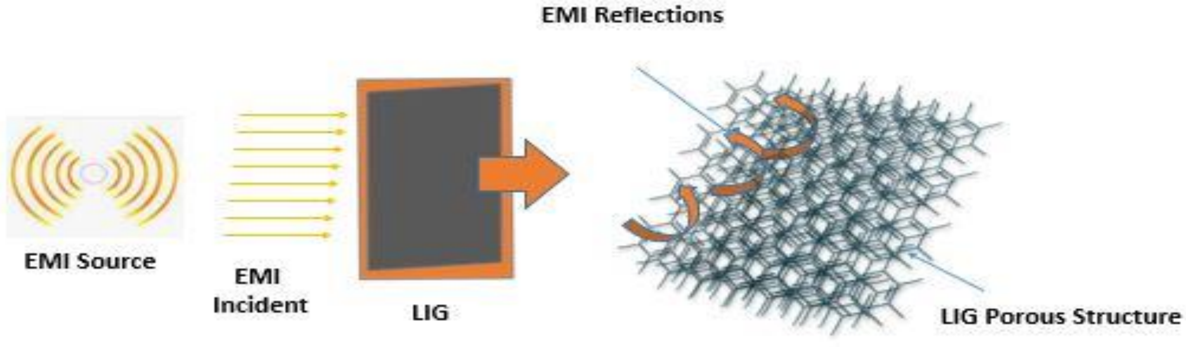

Fig. 4. Application of LIG in EMI shielding.

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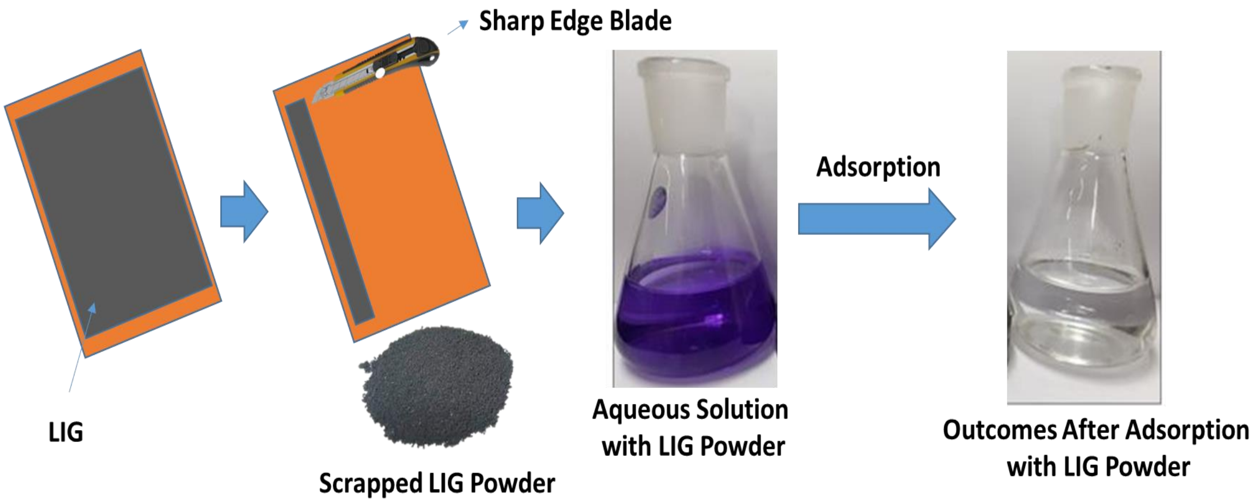

Fig. 5. Application of LIG in adsorption of contaminated water.

3.4. Application of LIG in Sensors

Application of LIG in the area of sensors have been reported. The application are into three major categories; biomedical sensors, wearable devices, and smart materials/structures sensors. Under the biomedical sensors, Hui et al [35] demonstrated an excellent performance of an LIG sensor for the detection of dopamine (DA). DA is an essential information transmitter in the mammalian central nervous system, excessive secreation of the DA may result to irritability, rapid nerve reflex, and extreme hyperactivity. Thus, group [35] fabricated a Pt-Au nanoparticles functionalized polydimethylsiloxane based LIG for the detection of the DA in human urine. The sensor was found to exhibit excellent sensitivity of about 865.80 mA/The LIG-based sensor exhibited an excellent sensitivity of approximately 865.80 mA/mMcm^{-2} and a Limit detection of 75 Nm.

Wan et al [36] reported the utilization of a nitrogen atom (N) incorporated polyimide based LIG sensor for miRNA detection. The N doping within the substrate was to improve conductivity and sensitivity of the sensor to the nucleic acids. Their findings reveal that the sensor performs excellently and a limit of detection of as low as 10 fM was recorded.

Applications of LIG wearable sensors has also been reported recently [37]. Typical of such studies is a report published by Li and Bo [37] where a polyimide based LIG sensing system that is finger wearable was investigated and its feasibility and potential in qualitative detection of chloramphenicol (CAP), clenbuterol (CLB) and ractopamine (RAC) in meat was extensively done (Fig. 6). The electrochemical response of CAP, CLB and RAC was found to be 2.70, 1.29 and 7.81 µM within the linear range of 10–200, 5–80 and 25–250 µM in phosphate buffer saline (PBS) and 7.0 pH, correspondingly. Also, minimum detection concentrations of CAP, CLB and RAC found were 20, 10 and 30 µM, respectively, in the actual pork samples, whereas the minimum detection concentrations of CAP, CLB and RAC were found to be 10, 5 and 25 µM in milk, respectively. Such an integrated sensing platform enriches application of wearable finger sensors that provides information that useful in preventing food contamination.

Fig. 6. Application of LIG in wearable sensor.

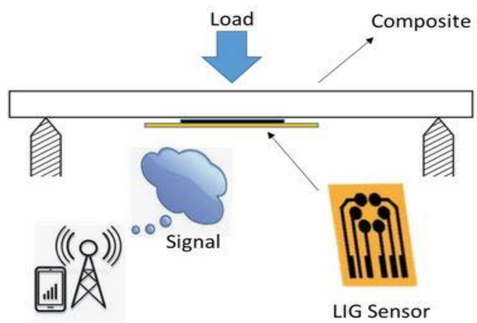

Fig. 7. Description of self-sensing composite Using LIG sensor.

Liu et al [38] investigated the application of a polyimide based LIG in a multifunctional composite in an effort towards achieving a smart devices/structures. Firstly, the LIG was patterned on the polyimide film before attaching it to the composite as shown in Fig. 7 for *in situ* structural performance and deformation sensing. Results obtained reveal that the self-sensing capability of LIG hybridized composites was finally achieved by deploying a 5 × 5 matrix array for mapping strain distribution. Where gauge factor of 0.67 to 648.8, and stability of more than 10000 cycles of the sensor performance in detecting large-scale deformation and mapping of strain distribution were achieved.

3.5. Other Applications of LIG

Other applications of the LIG includes but not limited to; face masks, air and water filters, antiviral surfaces, and sludge treatment as discussed extensively by Dixit and Singh [39]. It was found that the hydrophilic nature, negatively charged surface, and nano-porous structure of the LIG are exploited for the aforementioned applications.

4. Conclusion and Future Perspective

The recent researches on the production and application of thermosetting polymer based LIG have been reviewed. The stage wise methodology of producing the LIG has been dissected and effects of varying the laser machine parameters on the LIG characteristics have been discussed. Based on the published reports, the LIG was found to perform excellently in the areas of energy and power, EMI shielding, and environmental management. More so, LIG was found to also satisfactorily perform in the aspects of sensors, air filters, sludge treatment, face masks, and antimicrobial surfaces. However, applications in the aspect of sensors seems to be receiving tremendous attention of the research community, where developments of sensors that targeted the area of biomedical sciences and engineering, wearable devices, and smart composites and structures were obvious. Overall, it has been observed that different independent researches carried out mostly utilized polyimide polymer as substrates in the production of the LIG, perhaps due to its high char yield. Thus, it be concluded that the utilization of other thermosetting polymers like the polybenzoxazine or/and their alloys with polyimide in the production substrate for the production of the LIG is not well studied. Also, results obtained from different studies reveal that substrate modification by doping the polymer matrix with certain nanoparticles or/and atoms that could impact some desired properties proved to be a reliable idea. Therefore it could be suggested that substrate modification and transformation irrespective of the type of thermosetting polymer used and area of LIG application presents a very huge perspective in the area of LIG application.

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Declaration of Competing Interest

The authors would like to declare that there is no known competing interest of any kind between them and/or their universities.

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