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Laser-Induced Flexible Electronics (LIFE) for Resistive, Capacitive and Electrochemical Sensing Applications

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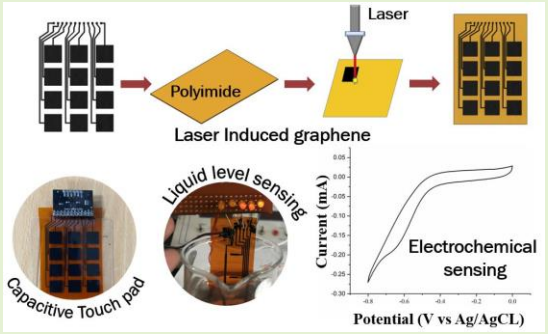
***Abstract*—Engineering a cost-effective, flexible electronic device in a one-step fabrication process is quite challenging to perform. Herein, we have introduced a simple, low-cost, solid-state process for producing and printing of complex circuits using Laser-Induced Graphene (LIG). In the present work, LIG has been effectively and selectively formed from direct CO_2 laser ablation on a polyimide sheet. Varying CO_2 laser power and speed, the electrical conductivity of the LIG has shown a linear increment in the conductivity measurement. The laser-induced samples were structurally characterized using Scanning Electron Microscopy (SEM), EDX, X-ray Photoelectron Spectroscopy (XPS), Raman spectroscopy. The results show a one-step method to create Graphene-derived structures on the polyimide sheet surface. This method of generating LIG on a flexible substrate (polyimide sheet) offers an easy way to fabricate Laser-Induced Flexible electronics (LIFE) circuits. Using this, the feasibility and the realization of a capacitive touch sensor and liquid level sensor has been successfully demonstrated. Further, as a prototype system, the LIG was examined for the H_2O_2 electrochemical sensing application. It gives an appreciable response for the detection of H_2O_2 in comparison to other carbon-based electrodes with limit-of-detection (LOD) as 0.3 µM in a linear range from 1 µM to 10 µM.**



***Index Terms*— Laser-Induced Flexible Electronics (LIFE), Polyimide, electrochemical sensing, Touchpad, water-level monitoring.**

I. INTRODUCTION

Electronics has become so dominant and dependable that it is continuously harnessed in many ongoing research domains to realize cost-effective, user-friendly and robust devices with better accuracy and precision. Since their inception, the electronic circuits were bulky as they were realized on the dotted circuit boards, with a manual and time-consuming process. Later on, a thin copper sheet based Printed Circuit Boards (PCB) were developed, which has brought remarkable changes to modern electronics. On PCB, the copper conductive traces were obtained by a well-established subtractive chemical wet-etching process. Even though such a PCB fabrication process is well-established, but there is a huge scope to make it automated, additive, and cost-and-time efficient. Further, the PCB fabrication process involves complex fabrication method that includes carcinogenic chemicals, and hot soldering is necessarily leading to another significant concern. In few cases, such as packaging of the circuit, stacking of Integrated Circuits (IC), electrical connection between the stacks of circuit boards, rigid PCB have disadvantages, leading to the requirement of the flexible boards [1]–[3]. Hence, an alternative technique is necessary to create electronic circuits on a flexible surface, which reduces the area and increases the application potential of the device.

The flexible electronics has been reported for many applications in day-to-day life, includes connectors for flexible displays, contacts in keypads, keyboards, health monitoring devices and much more[4][5][6][7][8]. Such flexible electronic devices have been fabricated by popular approaches such as screen-printing and inkjet printing, where suitable conductive ink, developed using Carbon or Silver, was employed to get the conductive traces. These traces are formed on many substrates such as polymeric film, polyethylene terephthalate (PET), polyimide (PI) and can be easily connected on an onboard PCB. This approach is shown in stacking of PCBs, resulting in the reduction of the area to realize the necessary circuit. These traces can be connected to the PCB using Flexible Printed Electronic Circuit connector, or card edge connector[9]. These genres of flexible circuits play a prominent role to develop sensors, Radio-frequency

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identification (RFID), flexible displays and antennas. In recent times, the compositions of conductive inks were achieved using many novel materials, such as nanoparticle, nanowires and graphene sheets, to increase the conductivity. The conductive inks were prepared by mixing conductive material with solvents to maintain the consistency based on the requirement. However, the main challenge in developing such inks is to properly synthesize the nanomaterials itself and the bonding process after solidification[10]. Other drawbacks of these methods are clogging of nozzles of the inkjet printers, preparation of stencils for screen-printing, complicated and tedious process for obtaining nanoparticles. These challenges increase the time to obtain the flexible circuits, an increase in cost and non-uniformity of the conductance of the circuit.

Various nanomaterials fabrication techniques are proposed during recent years, which includes nanoparticles, Nano-rods of materials such as copper, silver, gold, platinum that have high conductive properties. The CO_2 laser method is being used widely for obtaining the carbon on polyimide sheet[11]. An alternative process is synthesizing of Graphene, an allotrope of carbon, which has immense advantages due to its physical, chemical, mechanical and electrical properties. The graphene is a three-dimensional porous structure with very few layers (3-5 layers); this ensures flexibility, high surface area and mechanical endurance[12]. However, the major challenge exists in obtaining a few layers of graphene . As the number of layers increases, the properties change which may result in different carbon allotropes such as Graphene oxide, reduced Graphene oxide, and Graphite. Due to the advantages as mentioned above, the applications of graphene are seen in various domains including storage of electrical energy as a supercapacitor, on electrode surface reversibility of absorption and desorption of electrolytic ions. The well-established procedures to fabricate crystalline graphene structures are oxidative acid synthesis route, chemical vapour deposition on thin films of Cu and Ni at elevated temperatures leading to porous material deposition[13]. This film of graphene can be transferred from one surface to the other transfer methods involving either a wet or dry process[14]. One of the reported drawbacks with the processes of forming reduced GO is that the quality of the graphene obtained does not remain the same as of native graphene[15].

One of the innovative and straightforward process to obtain graphene or graphene oxide has been demonstrated by focusing high energy density laser beam on to the surface of the substrates. This was achieved by ablating substrates under CO_2 laser in the desired pattern, designed in software as per the requirement, and fed to the CO_2 laser equipment[16]. The CO_2 laser ablation was been reportedly performed on different non-flexible substrates such as bread slice, coconut shell, various kinds of wood and much more. A similar process has been repeated on a flexible substrate, like polyimide (PI), whereby the traces of carbon was observed [17][18]. This process is famously known as Laser-Induced Graphene (LIG)[19]. The obtained samples were subjected to physic-chemical characterization techniques, such as XPS spectra, Raman Spectroscopy[20] and Scanning Electron Microscope (SEM)[21], and provided sufficient justification to conclude that the obtained carbon traces were of graphene oxide.

Since the present approach has been carried out on a hydrophobic polyimide substrate, therefore, it will be easily compatible with subsequent material processing. In this work, an optimized process has been demonstrated to create LIGs on a polyimide sheet using a low-power CO_2 laser. Rigorous characterizations were carried out to support the feasibility to employ such LIGs to create LIFE components for sensing applications. T. han et al reported in 2019, with speed and power of CO_2 laser the polyimide sheet was ablated and used as real time strain sensing[22]. To realize the potential and scope of such LIFE devices, varied applications like capacitive touchpad, electrochemical sensing and resistive liquid level sensor is demonstrated.

II. Materials and Methods

Polyimide sheet of 125 μm thickness was purchased from Dali Electronics, and a 30 W CO_2 Laser (Universal Laser Systems, VLS 3.60) was utilized to obtain the Laser-Induced Graphene (LIG) conductive traces. Hydrogen peroxide (30%) was procured from Sigma Aldrich and other basic chemicals of analytical grade were used. The topographical morphology and elemental analysis of the obtained CO2 laser-induced graphene (LIG) were characterized by Scanning Electron Microscope (SEM) and Energy Dispersive Spectroscopy (EDS) techniques using Apreo Scanning Electron Microscope (SEM) from Thermo Fisher Scientific. Fourier transform infrared spectrometer instrument (FTIR-4200 from Jasco) was used to examine the samples for identifying the functional groups (organic or inorganic). In further, Witec Alpha 500 confocal Raman microscopy was used for the characterization. A Thermo scientific K-Alpha X-ray Photoelectron Spectrometer (XPS) instrument was utilized to examine the samples for surface analysis. For conductivity, resistance, resistivity measurement, Ossila machine, based on the Four-point probe mechanism was used. All the cyclic voltammetry (CV) measurements were performed using Bio-logic (SP-150) electrochemical workstation in a standard three-electrode configuration.

III. Exerimental Procedure

A. Formation of Laser-Induced Graphene(LIG) on Polyimide sheet

A basic design was drawn using AutoCAD Fusion 360, and the file was saved in .dxf format. The .dxf file was transferred in CorelDraw X7, and the outline width of the design was changed according to the preference of the CO_2 laser. As shown in Fig 1, by varying the speed and power, of the CO_2 laser, Laser-Induced Graphene (LIG) was obtained on polyimide (PI) [23], LIG reaches its highest crystallinity by laser power amplification[24][25].Literature reports fabrication of LIG using CO_2 laser machine by varying the power and speed on PI, wherein conductivity is obtained and calculated. For instance, Lin et. al. induced 60W CO_2 laser on PI by varying power from 2.4 W to 5.4 W, showing the high conductivity of 25 S/cm at a power of 5.4 W. In present work, for 5.4 W laser power has 91.3% of carbon, 7.7% of oxygen

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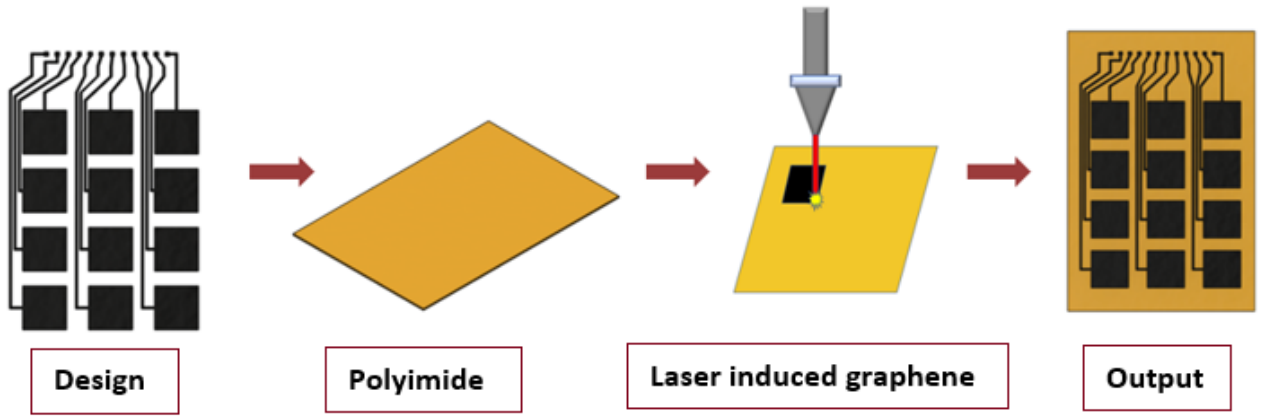

Fig 1 Schematic process to create Laser-Induced Graphene (LIG) structure on Polyimide

and 1.0% of nitrogen was observed. In the present study, above 95% of carbon was observed by varying 30 W CO_2 laser power from 1.35 W to 1.95 W as shown in Table I.

In general, by optimizing the laser power, the LIG thickness increased improving its conductivity.

B. Capacitive Touchpad

By touching a capacitive touchpad, an amount of charge is drawn to the contact point which becomes functional. The change in the electrostatic field is calculated to determine the location. In this work, touchpads were designed with different approaches such as resistive and capacitive, whereby the capacitance-based touchpad has seen the upper hand in terms of extensive usage. The electrode patterns were created to act as touchpads with a particular pre-defined pattern. These patterns obtained on the PI sheet with the help of CO_2 laser was integrated along with the ICs, meant to detect the change in the capacitance, which was used along with a microcontroller and a display unit.

C. Liquid level sensor

These sensors are used to detect liquid levels or interfaces between liquids such as water and oil. Level sensors calculate within the range. Level sensor shows whether the fluids are above or below the point of sensation. Level detection sensors can detect the fluid level in a container, and therefore, they have ample scope in real-life applications. Herein, an electronic-based approach was explored. LEDs were used to show the level, which was one of the natural and visual approaches to exhibit.

D. Electrochemical sensing

A conventional three-electrode system consisting of a working electrode, a counter electrode, and a reference electrode is used for electrochemical sensing. Herein, the laser-induced graphene (LIG) sample was used as a working electrode, Ag/AgCl as reference and platinum as a counter. Cyclic voltammetry (CV) experiments were performed using 0.2 M phosphate buffer solution (PBS) pH 7 and 500 µM H_2O_2 sample.

IV. Material characterization

In order to interpret the surface morphology and structure of the LIG, samples were exposed to various physico-chemical and microscopic characterization like SEM, EDX, XRD analysis, XPS and Raman Spectroscopy[26].

A. Scanning Electron Microscopy (SEM) and Dispersive X-ray Spectroscopy (EDX)

The Surface morphology study of the formed LIG on polyimide sheet was explored using scanning electron microscopy (SEM). Fig 2 exhibits SEM images of PI sheet before and after exposing to the CO_2 laser machine. Fig 2. (a - c) Clearly depicts the bare polyimide sheet without laser ablation, whereas, in Fig 2 (b-d), represents post exposure to CO_2 laser. An evident deposition of graphitic like framework was observed authenticating the LIG formation. Fig 2 (e) shows the EDX of a LIG sample. From the EDX, the carbon content in the LIG is above 95%, and oxygen content is near to 4%. Table 1 depicts the elemental analysis of the LIG [27].

TABLE I
CARBON & OXYGEN CONTENT IN EACH SAMPLES

Elements	Bare PI	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5
Carbon (%)	79.97	96.25	96.20	96.22	96.50	96.55
Oxygen (%)	20.03	3.75	3.8	3.78	3.5	3.45

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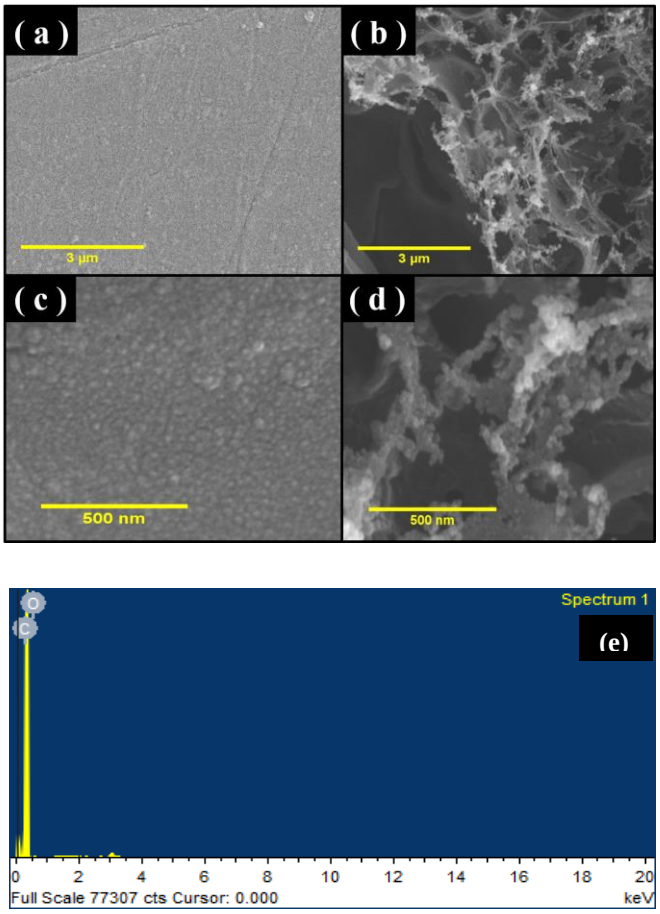

Fig 2. SEM images of (a-c) bare polyimide, (b-d)LIG sample after exposing to the CO_2 laser, (e) EDX of LIG sample showing carbon and oxygen peak

B. X-ray Photoelectron Spectroscopy (XPS)

Varying the speed and power of the CO_2 laser, various samples were prepared. XPS C 1s spectrum analysis for all the samples was done. The peak values for the five samples are represented in Table 2. Fig 3. shows the XPS spectrum of the prepared Graphene Oxide (GO). From the literature [28][29], the deconvolution of GO peak happens at 284.6, 286.6 and 288.5eV. The number of counts per second depends on the concentration of the elements present in the compound. For the LIG sample, the binding energies of the corresponding functional group gives different counts per second with respect to the concentration present in the sample.

TABLE II
XPS C 1 S SPECTRUM OF LIG SAMPLE

Samples	Counts/s	Binding energy (eV)
1	265713	284.38
2	138620	284.78
3	170895	284.78
4	248508	284.68
5	241663	284.58

For the LIG sample, the C 1s XPS spectrum peaks after the deconvolution, are at 284.67, 286.55 and 288.48 eV, similar to the reported values. The peak values represent a functional group of C-C, O-C=O, and sp2. Here, without deconvolution, some samples show a peak at 284.78 that is near to sp3 carbon. The C 1s XPS spectrum intensity peaks manifest that it contained only C and O elements indicating the absence of impurities.

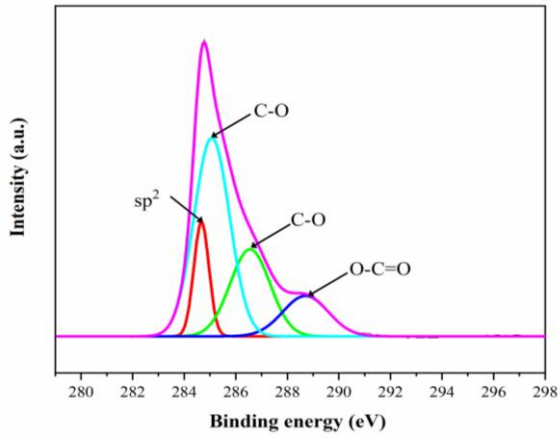

Fig 3. XPS of graphene oxide

C. Raman Spectroscopy

Investigation of the obtained LIG was carried out using Raman Spectroscopy to make sure that the material was carbon allotropes. Five LIG samples were characterized using Raman spectroscopy (varying speed and power), and the Raman peaks are illustrated in Fig 4. and Table 3. The results for the intensity of G bands, D bands and 2D bands are shown in Table 3.

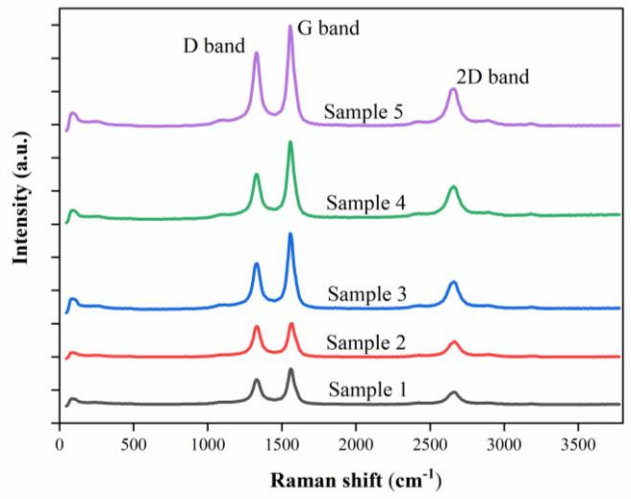

Fig 4. Raman spectra of Graphene oxide for five samples varying speed and power

The disordered carbon structure can be represented by the intensity ratio of the G band to the D band (I_D/I_G)[23][30]. The I_D/I_G ratio of the as prepared LIG samples varied between 0.668 and 0.942, indicating the varying carbon structural defects in the lattice structure of various LIG samples. Raman results for five samples show the same D and G band wavelength, i.e. 1330.039 cm^{-1} and 1557.68 cm^{-1}. Therefore, based on the values obtained it can be concluded that the

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samples had a formation of graphene oxide. As the ratio, I_{2D}/I_G, manifest the number of graphene layers, the variation of the number of layers should be one of the reasons behind the variation in electrical conductivity for various LIG samples.

TABLE III
D AND G BANDS FOR THE LIG SAMPLES

S.no	D Band	G Band	2D Band	I_D/I_G	I_{2D}/I_G
1	658.33	818.30	468.36	0.805	0.572
2	743.58	789.55	513.94	0.942	0.651
3	1007.61	1462.30	727.80	0.689	0.498
4	995.83	1490.11	810.22	0.668	0.544
5	1446.41	1849.08	898.80	0.782	0.486

D. Conductivity Test (Four-Point Probe System)

The prepared LIG samples were tested for conductivity by placing them at the center of the four-probe system. To measure the conductivity, the input of a maximum 5 V with 0.01 V increment and 25 repetitions were carried out. Fig 5. (a) shows the plot between applied voltage (V) and measured current (A), and Fig 5 (b) shows the plot between applied current (A) and measured voltage (V). As can be seen, the conductivity of the sample changes with varying speed and power of laser on the polyimide sheet.

TABLE IV
CONDUCTIVITY RESULTS FOR VARIOUS LIG SAMPLES

Samples	Power (W)	Speed (m/min)	Conductivity (S/m)
1	1.35	0.45	250.64
2	1.5	0.54	276.70
3	1.65	0.63	293.08
4	1.8	0.72	319.95
5	1.95	0.82	367.78

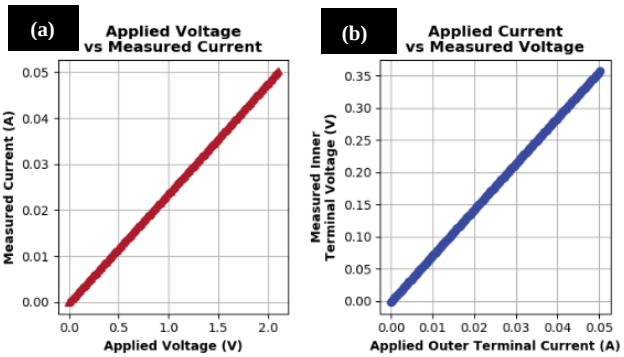


Fig 5. Measured Current vs voltage for the LIG samples

V. Results and discussion

A. Capacitive Touchpad

By using AutoCAD Fusion 360, a touchpad was designed, as shown in Fig 6. The designed file was then induced with LIG on the polyimide sheet using the CO_2 laser machine to obtain the conductive traces. The values of the capacitance and resistance have been measured using an LCR meter. The observed values for capacitance was constant i.e. 1 pF. The resistance values for five samples are as follows 138.3 Ω, 138.7 Ω, 140.2 Ω, 520 k Ω, 490 k Ω. The obtained LIG was then used as a capacitive sensor with mounted MPR121[31], which acted as a charge transfer sensing using single wire electrodes. On touching the electrode, both the capacitance and the charge transfer time increased and the microcontroller, in turn, received the information from the MPR 121, and the result for the capacitive touch sensor was displayed on the computer.

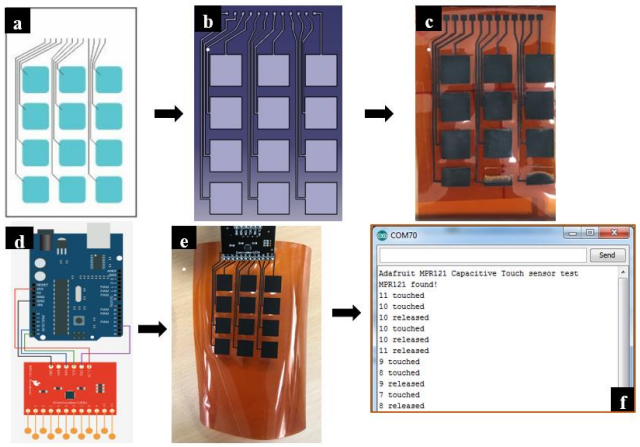


Fig 6. (a). Schematic of a touch sensor (b). CAD model of Capacitive touch sensor (c). CO_2 Laser Induced Graphene touch sensor on polyimide sheet (d). Schematic of Microcontroller integrated with MPR121 (e). MPR 121 mounted on LIG (f). Displaying results for Capacitive touch sensor.

B. Liquid-Level Sensor

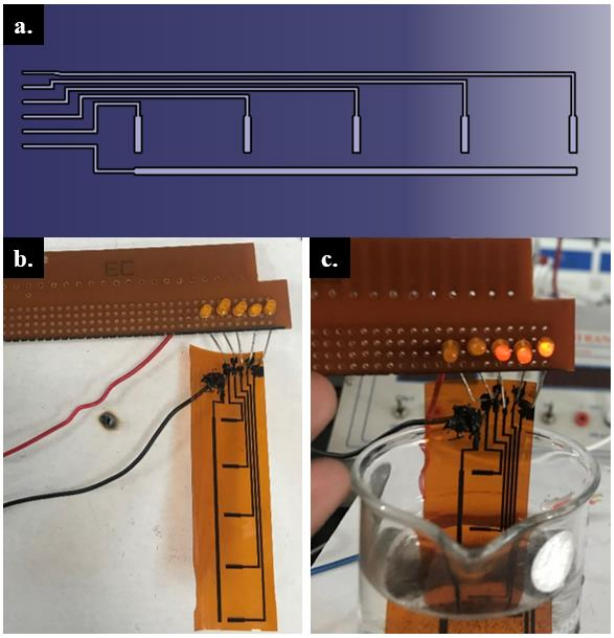


Fig 7. (a). CAD model of liquid level sensor (b). LIG arranged with LED lights on PCB (c). Testing of Liquid Level sensor with water.

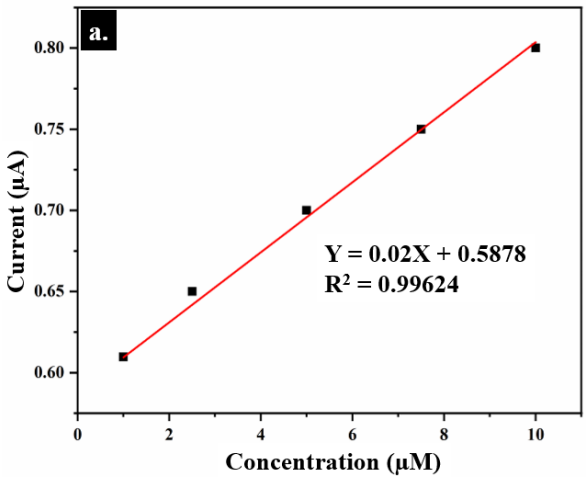
A liquid level sensor has been designed in AutoCAD fusion 360, where the positive terminal/trace are in straight length, and the negative terminals/trace are joined with five intervals of a horizontal line as shown in Fig 7. Then after laser ablation on the polyimide sheet [32][33], the positive terminal and the negative terminals were arranged with five LED lights, and a

9 volts battery with 7805 regulator was incorporated to observe the function of the device.

C. Electrochemical Detection of Hydrogen Peroxide

For analyzing the hydrogen peroxide sensing with bare LIG, as a prototype, a 3-electrode system was created with the prepared LIG as a working electrode while Ag/AgCl and platinum wire as a reference and counter electrodes, respectively. Here, a rectangular shaped pattern with dimension 50 mm x 03 mm has been fabricated. Unmodified LIG is used for electrochemical sensing of Hydrogen peroxide. Further, 500 µM of H_2O_2 was prepared in 5 mL of pH 7 phosphate buffer solution. As stated, a three-electrode system with Ag/AgCl as working, Platinum as counter is used along with LIG as working for carrying out the cyclic voltammetry experiments. The experiments were carried out at 10 mV s^{-1} in a potential window of -1 to +1 V. Aparicio-Martinez et al. in 2019 reported silver modified Laser Scribed Graphene (LSG) for H_2O_2 sensing using a commercial GO and a 7.9 µM LOD was reported[34]. In the present case, GO was directly induced by laser and without any further modification, it was used for H_2O_2 sensing to obtain an appreciable detection limit of 0. 3 µM. Before experiments, all the solutions were purged with high-purity nitrogen. The Cyclic Voltammetry[35] measurements were carried out in 0.2 M phosphate buffer solution (PBS, pH 7.0) at the ambient temperature in the potential window 0.0 V to -1.0 V (vs. Ag/AgCl) at a scan rate of 10 mV s^{-1}. Fig 8 (a) depicts the calibration plot (b.) depicts the CV of the LIG electrode in 0.2 M PBS (pH 7.0) and with various H_2O_2 concentrations (from 1 µM to 10 µM).

With the successive addition of H_2O_2, the reduction current gradually increases, showing excellent electro-catalytic behavior of the fabricated LIG sensor towards hydrogen peroxide sensing. Based on the literature[36], the detection limit is theoretically calculated by using a standard formula, 3×Standard deviation/ Slope. Wherein, the standard deviation of the triplicated samples with the lowest concentration current was taken. The slope was derived from the calibration plot of the concentration effect. The detection limit of the sensor was estimated to be 0.3 µM within a linear range from 1 µM to 10 µM.



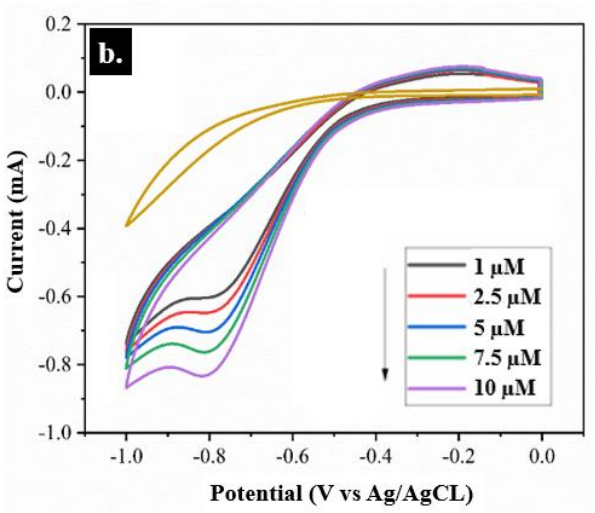


Fig 8. (a) Calibration plot of [H_2O_2] µM vs I/µA (b) Cyclic Voltammetry response for different concentrations of hydrogen peroxide (H202).

VI. Conclusion

Herein, a single-step, cost-effective approach to realize Laser-Induced Flexible Electronic (LIFE) circuits, using low power and user-friendly CO_2 laser, has been presented. Based on the design, graphene oxide (GO) has been observed to be formed on polyimide by CO_2 ablation. To confirm the type of carbon allotrope, various physico-chemical characterizations, such as SEM, EDX, , XPS and Raman Spectra, have been carried out. The CO_2 laser parameter, such as speed and power, has been varied to optimize the conductivity of LIFE circuits. After rigorous characterizations and optimizations, experiments were carried out to explore the feasibility to test various LIFE circuits for resistive, capacitive and electrochemical sensing. With such sensing applications, the LIFE circuits have been utilized as resistive touchpads, capacitive liquid-level sensor, and electrochemical H_2O_2 sensor. The Capacitive sensor, integrated with a mini-microprocessor, the functioning of the device, is proven by showing the result of glowing the LED and displaying the result of the touch sensor on the computer. For electrochemical H_2O_2 sensing, the device showed a good response for various concentrations and appreciable limit of detection without any further modifications, i.e. 0.3 µM. Overall, the LIFE devices have a strong potential to be harnessed for diverse sensing applications.

VII. ACKNOWLEDGMENT

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