Graphitic surface layer formation on organic substrates for electronics using a concentrated solar simulator

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Abstract
Heat spreading is an important attribute that improves thermal management and operation of high-performance packages, such as those for solid-state power amplifiers. This attribute can be enhanced through direct transformation of polymers into graphitic films. The present work reports a custom vacuum deposition process that synthesizes thin graphitic layers on organic substrates through direct pyrolysis via concentrated irradiation from a xenon lamp to produce a peak heated zone of approximately 3 cm diameter with a maximum heat flux of 3.2 MW/m². The light source, replaceable with concentrated terrestrial solar power, provides ultrafast heating of substrates through rapid flashes (< 0.5 s) and improves growth rates over relatively larger areas compared to laser processing, mitigating the issue of porosity in graphitic layers that deteriorate heat spreading capabilities. The enhanced organic substrates are analyzed using Raman and energy-dispersive X-ray spectroscopy, scanning electron microscopy, and thermal diffusivity measurements, indicating graphitic conversion.

Introduction
Graphene-based structures have gained wide interest due to their outstanding thermal, electrical, and mechanical properties [1]. The conventional approach to producing graphene structures is through chemical vapor deposition (CVD) processes [2, 3]. However, such processes can be time-consuming, expensive, or require high temperature conditions. Additionally, they require the physical transfer of graphene product from catalytic metal substrates (when present) to receiving substrates [4], which can be challenging [5]. Such conventional approaches may be unsatisfactory for enhancing heat spreading capabilities of organic substrates used for power amplifiers. In contrast, prior work has shown that organic polymers can be directly processed and converted to graphene-based structures [6]. In addition to eliminating the need for graphene transfer, starting with a common organic polymer such as epoxy resin provides an inexpensive raw carbon source in comparison with CVD processes.

One approach to convert polymers to graphene structures is through direct pyrolysis in a heating furnace [7]. Such an approach is energy intensive and generally lacks controllability of the local material portion to be converted, where the entire polymer is heated to high temperatures. However, the latter can be overcome by lithography techniques that deposit organic polymers on selected portions of a substrate [8]. In prior work [6], three different polymer films, which consist of polymethyl methacrylate (PMMA), polystyrene (PS), and polyacrylonitrile (PAN), were converted into graphene. The polymers were initially formed into thin layers onto SiO₂/Si substrates (or catalytic nickel) in a furnace at 1000 °C and Ar/H₂ flow at 4 Torr to study graphene conversion. Results indicate that PAN converts to amorphous carbon during pyrolysis at 800 °C but forms graphene at 1000 °C, based on the absence or presence of a 2D Raman peak \( \approx 2700 \text{ cm}^{-1} \) [9]. In another study [10], pyrolysis of SU-8 (epoxy-based photoresist) into a graphene-rich elemental carbon form was investigated in the temperature range of 500–1200 °C. The authors divided
the pyrolysis process into three general categories: (a) < 550 °C, formation of a carbonaceous backbone; (b) 550–700 °C, formation of pyrolytic carbon with high composition of dangling bonds; (c) > 700 °C, formation of short-range order and continuing graphitization of polymers. Therefore, the carbon product’s structure is very sensitive to the pyrolysis temperature.

Another approach to forming graphene structures from polymers utilizes CO$_2$ infrared (IR) lasers [11, 12]. In prior work [13], a 3.6 W CO$_2$ IR laser was used in ambient conditions (localized temperature > 2500 °C) to convert a commercial polyimide (PI) film into porous graphene (laser-induced graphene—LIG). This process does not require a graphene oxide precursor as in laser-reduced graphene methods, significantly simplifying the conversion process [14]. A total of 15 different polymers were studied, including PS, PMMA, and PAN, where only PI and polyetherimide films were formed into LIG; imide and aromatic repeating units strongly correlate to formation of LIG. In a similar work [15], a CO$_2$ IR laser was used to transform PI to 3D porous LIG under ambient conditions through a single-step process. Despite the high quality of LIG products, the relatively high porosity/discontinuity of the graphene structure leads to additional barriers for heat spreading. In a different work [16], plastic waste was used as a carbon source and directly converted into graphene using a flash joule heating process. Such an approach brings the sample to very high temperatures locally (maximum ≈ 2900 K), removing all non-carbon volatiles and leading to significant energy savings as opposed to pyrolysis in a furnace.

In the present work, a different approach to obtaining graphene structures from polymers is reported. A concentrated light source (i.e., high flux solar simulator) is used to locally heat a polymer sample with a significantly larger spot size compared to IR lasers, to avoid forming discontinuous graphene structures. This light source is normally used as a platform for various experiments [17] ranging from solar fuels and commodities production [18], photovoltaic device testing [19], and high-temperature material processing [20] and characterization [21]. For photovoltaic device testing, the concentrated light source is usually operated in a controllable flash mode with flashes lasting from few to a hundred milliseconds [19]. In this study, we utilize the light source to convert FR4 (glass fiber epoxy laminate) into graphene structures under short (<1 s), high irradiation flashes as illustrated in Fig. 1a. This approach starts from a safe and

![Fig. 1](image_url)
inexpensive carbon source, is energy efficient (localized heating only), produces very fast heating and cooling rates, and can ultimately be scaled up as a green manufacturing technique using solar energy.

Materials and methods

Experimental setup

The experimental setup consists of three main components: (1) a custom-built and designed concentrated light source, (2) a vacuum chamber, and (3) a circular FR4 substrate.

The concentrated light source has a 10 kW_{e} xenon short arc lamp placed at the truncated ellipsoidal reflector’s first focal point to concentrate the output radiation from the lamp to its second focal point. The protected silver-coated aluminum reflector’s focal points are at 7.49 and 102.3 cm from the ellipsoid’s vertex, with truncated diameters of 10.0 and 38.7 cm. Additionally, a motor-controlled douser is installed to allow further control of the lamp’s output via direct blocking and for the application of cyclic pulsing of output irradiation. Other auxiliary components include intake and exhaust blowers for proper cooling, monitored using an IR temperature sensor, as shown in Fig. 1b.

The output irradiance at the focal plane of a single-lamp concentrated light source strongly resembles a Lorentzian distribution [17]. This heat flux distribution has been directly measured at discrete points in prior work [22] outside the vacuum chamber and at the focal plane using a heat flux gauge. Heat flux measurements were also complemented and verified with numerical simulations using a validated Monte Carlo ray tracing in-house code [23]. A variable DC power supply is used to control the lamp’s ignition and its power output by varying the current supply within 100–200 A. The experimental steady-state heat flux distribution \( q''(r) \) along with a Lorentzian fit and cumulative power at the focal plane with a maximum current supply \( I \) of 200 A is shown in Fig. 1c. This Lorentzian fit is represented by Eq. 1, where \( A_{L} \) and \( \sigma_{L} \) are the Lorentzian parameters with values of 124.9 kW/m and 0.00864 m. These parameters provide a maximum center peak heat flux value of approximately 4.6 MW/m² and a concentrated beam diameter of 1.7 cm as presented by the distribution’s full width at half maximum (FWHM).

\[
q''(r) = \frac{A_{L}(I)}{\pi} \left( \frac{\sigma_{L}}{r^2 + \sigma_{L}^2} \right)
\]  

(1)

The cylindrical vacuum chamber reactor has an inner diameter of 9.7 cm with a 6.4 mm thick fused quartz aperture window to allow radiation from the concentrated light source to heat FR4 substrates directly. The chamber is equipped with a capacitance manometer for pressure monitoring and a zinc selenide viewport with 4.8 cm viewing diameter to allow for temperature monitoring of the sample’s back side using a high-resolution longwave IR camera (FLIR, A655sc). Additionally, the chamber is connected to various mass flow control lines (for Ar, CH\(_4\), and H\(_2\) flow), gas manifold, valves, and vacuum pump to allow testing under various controlled environments. The experimental setup and components are shown in Fig. 1d.

Methodology

A readily available commercial FR4 substrate (Curbell, G10) with a thickness of 1.5 mm was cut into 6.9 cm in diameter and directly supported on a flange within the processing chamber at the focal plane of the concentrated light source. The light source was operated in flash mode, where the lamp can be ignited for any time duration longer than 50 ms. For the present study and results, the light source’s DC current supply was maintained constant at 200 A during processing while varying other parameters. These parameters include the duration and number of flashes, pressure level \( P \), and gas composition/flow rate (when applicable), where the duration between each flash was maintained constant at 25 s. Importantly, the flash mode’s heat flux distribution varies linearly from its steady-state distribution, where the flux values start at approximately 70% of their steady-state values represented by Eq. 1 [17, 22]. Therefore, the peak heat flux for this study is estimated to be 3.2 MW/m².

In evaluating the form and quality of the carbonaceous product within the processed FR4 substrates, Raman spectra were acquired using an in-house Raman spectrometer with a 532 nm laser and a 40× achromatic objective lens that connects to a visible-light detector of a Horiba iHR 550 imaging spectrometer. To estimate the average inter-defect distance, \( L_{D} \), and crystallinity of the carbonaceous product from Raman spectra, a laser-dependent relationship is used [9]:

\[
L_{D}^2[nm^2] = \frac{4.3 \times 10^3}{E_{Las}^{4}[eV^4]} \left( \frac{I_{D}}{I_{G}} \right)^{-1}
\]

(2)

where the excitation energy \( E_{Las} \) = 2.33 eV. Additionally, Scanning electron microscopy (SEM) images and energy-dispersive X-ray spectra (EDS) were collected using a ZEISS Supra 40VP field emission SEM and its secondary electrons and EDS detectors. Furthermore, thermal diffusivity measurements were obtained using a modified Ångström’s method and an in-house setup described in prior work [24] on rectangular strip samples with length, width, and thickness of approximately 25, 7, and 0.2 mm. Briefly, a thermoelectric module was used to modulate temperatures sinusoidally across an edge of tested samples, where...
amplitude ratios and phase shifts of temperatures at different locations along the material measured using an IR camera provide accurate estimates of the material’s thermal diffusivity.

Results

FR4 processing was performed under various operating conditions to examine the type and quality of carbonaceous product formed under light flashes with a peak heat flux of approx. 3.2 MW/m² (light source current supply of 200 A). Although testing was performed using significantly lower peak heat fluxes (1.2–1.8 MW/m²) and light source currents under various operating conditions, no or little carbonaceous conversion was observed due to relatively lower heating rates that likely vaporize products without re-adsorption, consistent with results from a prior study [6]. For testing with peak flux of approx. 3.2 MW/m², operating conditions and Raman observations at the substrate’s center (maximum heat flux and temperature) of selected experiments are summarized in Table 1. Results indicate successful conversion of epoxy resin within the original FR4 substrate into relatively amorphous carbon or graphitic product as concluded from the presence of D and G Raman peaks. The additional presence of a 2D Raman peak accompanied with a lower D/G peak ratio ($I_D/I_G$) indicates formation of graphitic product, where the 2D peak signifies the presence of long-range graphitic structures [25]. As summarized in Table 1, four of the 11 conditions yielded graphitic products.

The present heat flux distribution (Eq. 1) allows evaluating carbonaceous conversion at different operating conditions under irradiation fluxes that vary radially in a single test, without requiring additional experiments at different light source powers. This distribution can be readily modified to produce a uniform distribution through appropriate optical alignment (e.g., moving out of focus) [23] and/or by incorporating additional optical components [26]. The quality of the carbonaceous product is represented in the results of Fig. 2 for processed sample 8, which shows (a) a photo of the processed substrate and (b) the Raman shift spectra obtained at different radial positions ranging from 0 to 2.5 cm in diameter. Sample 8 exhibited the most prominent conversion, with its product quality varying from graphitic at the center (maximum temperature) to low-quality amorphous carbon at the edges of the converted region. Therefore, its results are used to compare Raman spectra of other samples; samples 1 and 6 exhibit Raman spectra similar to sample 8 at position 5, samples 2 and 7 are similar to position 4, samples 3, 5, and 11 are similar to position 3, and samples 4, 9, and 10 are similar to position 1. Figure 2 also shows SEM images of original FR4 substrates (c and d) and processed samples 4 (e) and 8 (f), showing the formation of continuous, 3D porous graphitic networks. Representative EDS spectra of the original and processed FR4 substrates were obtained as shown in Fig. 2g. The original substrate exhibits several major and minor elemental peaks reflecting its chemical composition, which includes carbon, oxygen, and chlorine from the epoxy resin, in addition to other peaks corresponding to oxides of boron, magnesium, aluminum, silicon, and calcium present within the glass fibers. After processing, several of the elemental peaks are no longer present, indicating removal of volatiles originally present in the organic polymer of the FR4 substrate to leave behind predominantly carbonaceous product on the surface.

To estimate local temperatures that lead to epoxy conversion into carbonaceous products, representative experiments were duplicated in another experimental configuration that allows measuring front-side temperatures under light flashes using an IR camera positioned at a 45° viewing angle. The maximum temperature contour of a FR4 substrate subjected to 0.3 s light flashes is shown in Fig. 2h, demonstrating the large temperature gradient that forms as a result of the heat flux distribution (Eq. 1). Once again, this temperature gradient allows investigating the carbonaceous product quality at different temperatures in a single experiment and can be readily adjusted via additional optics to produce a more uniform heat flux. From IR camera measurements of light flashes with different exposure durations ranging from 0.1 to 0.5 s with a 0.1 s increment, the resulting maximum temperatures were 720 K, 990 K, 1250 K, 1310 K, and 1350 K ± 4.5%.

### Table 1
Summary of FR4 sample processing operating conditions and Raman spectroscopy results at substrate’s center under maximum irradiation

| Sample | $P$ [Torr] | Flow [sccm] | Flashes #/duration [s] | $I_D/I_G$ | $I_{2D}/I_G$ |
|--------|------------|-------------|------------------------|-----------|-------------|
| 1      | Vac        | N/A         | 1/0.3                  | 0.81$^a$  | –           |
| 2      | Vac        | N/A         | 1/0.5                  | 0.88      | –           |
| 3      | 20         | Ar:H$_2$ = 80:20 | 5/0.3                  | 0.94      | 0.11$^b$   |
| 4      | Vac        | N/A         | 10/0.3                 | 0.72      | 0.41        |
| 5      | 20         | Ar:H$_2$ = 80:20 | 15/0.25                | 0.97      | 0.18$^b$   |
| 6      | Vac        | N/A         | 20/0.1                 | 0.83      | –           |
| 7      | Vac        | N/A         | 20/0.2                 | 0.86      | –           |
| 8      | Vac        | N/A         | 20/0.3                 | 0.68      | 0.43        |
| 9      | Vac        | N/A         | 20/0.4                 | 0.62$^a$  | 0.44$^c$   |
| 10     | 20         | Ar:H$_2$ = 80:20 | 20/0.3                 | 0.78      | 0.35        |
| 11     | 750        | Ar = 100    | 20/0.3                 | 0.89      | 0.13$^b$   |

$^a$Weak Raman D and G peaks, indicating limited carbonaceous conversion

$^b$Weak Raman 2D peak, indicating onset of graphitic conversion

$^c$Sample displayed deterioration and regions of discontinuity
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To assess enhancement in the effective heat spreading capability of processed FR4 substrates, the thermal diffusivity of the original substrate and processed substrates with graphitic surfaces were measured using a modified Ångström’s method [24], where testing oscillation frequency was set to 0.025 Hz. Figure 3 shows (a) a photo of experimental setup, (b) a representative temperature contour map of tested substrates indicating the region of analysis, and (c) amplitude ratio and (d) phase shift measurements of a tested FR4 substrate. The measured thermal diffusivity of original FR4 is $0.476 \pm 0.023 \text{ mm}^2/\text{s}$. In contrast, processed FR4 samples 4, 8, and 10 exhibited effective thermal diffusivity values of $0.946 \pm 0.053 \text{ mm}^2/\text{s}$, $0.949 \pm 0.062 \text{ mm}^2/\text{s}$, and $0.913 \pm 0.048 \text{ mm}^2/\text{s}$, respectively. These results demonstrate approximately twofold increases in the effective thermal diffusivity of the entire substrate material upon processing to form a graphitic carbon surface layer.

**Discussion**

Based on the results in Table 1, processed samples 4, 8, and 10 demonstrate conversion to surface graphitic product with reasonably high quality as indicated by a strong 2D peak and a relatively low D/G peak ratio of approximately 0.69 (Fig. 2b). This ratio corresponds to an average inter-defect distance of approximately 14.6 nm, where $D_0$ increases with

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![Fig. 2](image_url)
a lower $I_D/I_G$ ratio. Although processed sample 9 demonstrated lower $I_D/I_G$ ratio and a prominent 2D Raman peak, it displayed deterioration and regions of discontinuity that hinder its thermal spreading capability, which is undesirable. Additionally, testing with different gas compositions at different pressure levels was not observed to yield significant improvement, potentially due to the fast heating rates. Therefore, the effects of pressure and gas flow composition were not thoroughly investigated in this study.

From the presented results, optimum graphitic conversion was achieved at relatively high temperatures ($\approx 1250$ K), but more importantly under multiple flashes and at very fast heating rates with a peak heat flux of approximately $3.2$ MW/m$^2$. Samples tested under these conditions exhibited relatively low $I_D/I_G$ with distinct D and G peaks that have significantly lower FWHMs than others tested under less optimum conditions, which is a direct indication of enhancement in the crystallinity of the graphitic structure. A single light flash even for a relatively long duration and high temperature (sample 2) did not yield a detectable graphitic product. In contrast, multiple light flashes induced graphitic conversion, which may be due to propagating graphitic conversion under consecutive flashes with limited time for sudden excessive vaporization. In comparison with experiments performed in a heating furnace [6], the present fast heating rate results in carbonaceous conversion in situ rather than polymer vaporization without re-adsorption. For sample 8, graphitic conversion was detected up to a diameter of 1.25 cm around the center (positions 1 and 2) with heat flux decreasing from 3.2 to 2.6 MW/m$^2$ (and temperature from 1250 to 1120 K), which is consistent with other samples’ observations. Between 1.25 and 2.5 cm, epoxy in FR4 converts into a more amorphous carbon product based on the higher FWHMs and ratio of the disorder-induced D peak to the G peak and disappearance of the 2D peak. Conversion was not detected beyond 2.5 cm in diameter with heat flux values below 1.5 MW/m$^2$ (and temperatures well below 700 K). At the periphery of the converted region (position 5), the Raman spectrum exhibits significant fluctuations in signal due to the co-presence of carbonaceous product with the original epoxy resin as a result of low surface temperatures.

Effective thermal diffusivity measurements indicate significant improvement (by twofold) in the processed substrate’s overall heat spreading capability, which is expected to enhance the performance of power amplifiers. Hence, the local enhancement within the converted film is expected to be even higher. However, we postulate that the improvement in thermal diffusivity, while reasonably high, is limited due to porosity and induced surface cracks/defects that can create barriers to heat spreading. Present results are promising for substituting an energy intensive production process with a potentially green manufacturing technique that uses solar energy, and further work is required to optimize the process and approach. Future work should focus on complementing the present process and substrate with assisted conversion via methane pyrolysis (or pyrolysis of other carbon sources).
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