

# Graphene-on-Polymer Flexible Vaporizable Sensor

CNF Project Number: 1121-03

Principal Investigator(s): Amit Lal

User(s): Ved Gund

Affiliation(s): School of Electrical and Computer Engineering, Cornell University

Primary Source(s) of Research Funding: NSF-EFRI

Contact(s): amit.lal@cornell.edu, vvg3@cornell.edu

Website: <http://www.sonicmems.ece.cornell.edu/>

Primary CNF Tools Used: GSI PECVD, CVC SC4500 odd-hour evaporator, Oxford 81, Unaxis DRIE, spinners, SÜSS MA-6, Zeiss SEM

## Abstract:

Wireless sensor nodes integrated on flexible substrates are critical to meet the demands of a burgeoning market of wearable sensors, fieldable environmental sensors, and consumer electronics for Internet-of-Things (IoT) applications. While silicon-based circuits are pervasive, they face critical challenges for the aforementioned new and emerging applications. Wearable sensors necessitate conformality and stretchability to curved surfaces, in contrast to silicon-based sensors which are non-compliant. The use of silicon substrates for sensor nodes in a large, distributed network of sensors, whether for diagnostic and therapeutic biomedical devices or for soil and crop monitoring, also poses a significant concern from a sustainability standpoint since these sensors cannot be reacquired or resorbed easily back into the natural environment which can lead to non-degradable waste accumulation. Hence, it is critical to design sensors and electronics on flexible substrates that can also incorporate mechanisms for vaporization or disintegration which would minimize electronic waste. With this perspective, we have developed a graphene-on-polymer vaporizable and flexible piezoresistive sensor platform. This sensor technology can be used for wearable microsystems and disposable environmental sensors. A proof-of-concept pressure sensor with a high sensitivity of  $68 \times 10^{-3} \text{ kPa}^{-1}$  has been demonstrated. The device needs 22 mW to achieve a temperature of  $220^\circ\text{C}$  for the partial vaporization of a  $1 \text{ mm}^2$  membrane made of  $1.8 \mu\text{m}$  thick polypropylene carbonate (PPC), thus demonstrating a pathway to sensor self-destruction.

## Summary of Research:

It is essential to design flexible sensors with materials that have high mechanical strength for stretchability and conformality as well as excellent electronic properties. Atomically thin two-dimensional graphene is an outstanding candidate for such sensors because of its high Young's modulus of 1 TPa and as zero-bandgap semi-metal with out-of-plane p-orbital electrons for conduction modulation [1]. These properties make it suitable for piezoresistive sensing. Flexible graphene-based piezoresistive sensors with record high sensitivities have previously been reported [2]. Our work (schematic in Figure 1) demonstrates a graphene-on-polymer flexible piezoresistive pressure sensor that uses lithographically defined metal electrodes and membranes while maintaining a high quality of graphene as the piezoresistive sensor. The PPC is a highly compliant and flexible structural layer (Young's modulus of 1 GPa) for the sensor with low-temperature vaporization for potential self-destruction on environmental resorption [3].

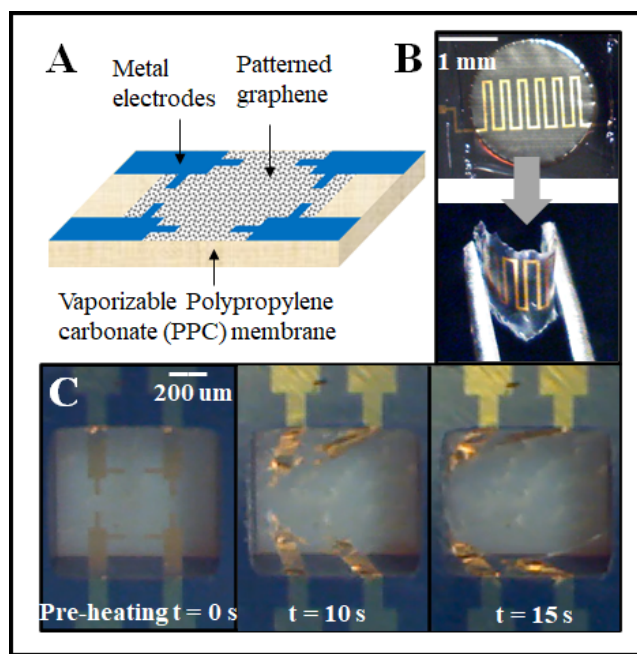


Figure 1: A) Flexible vaporizable pressure sensor architecture with lithographically defined graphene and metal electrodes on PPC. B) Flexibility: Metal-on-PPC piezoresistor on Si wafer (top) and after scribing out and bending with tweezers (bottom) C) Vaporization: Time series of sensor transience by vaporization of PPC in 15 seconds.

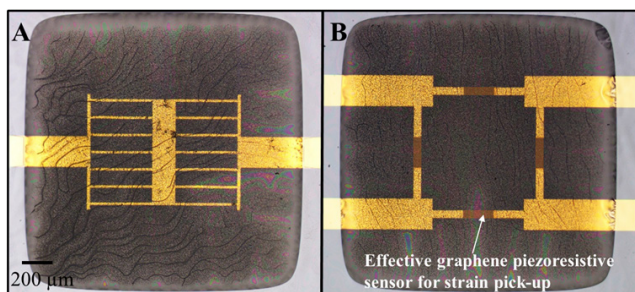


Figure 2: Optical micrographs of (A) Ti/Au metal-on-PPC flexible sensor and (B) graphene-on-PPC with false color between metal electrodes to show the effective graphene pressure sensing region for strain pick-up.

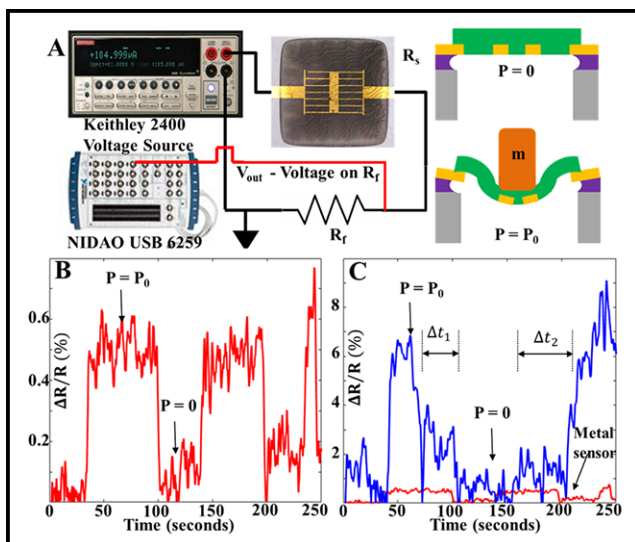


Figure 3: A) Piezoresistive pressure sensor test-setup. B) Fractional change in resistance ( $\Delta R/R$ ) for the metal sensor and C) Graphene sensor for loading and unloading of pressure  $P_0$ . The graphene sensor shows delays in response to unloading and loading after the first cycle.

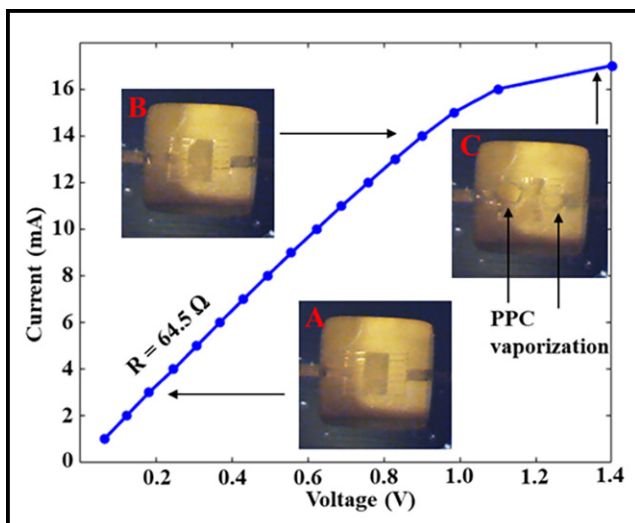


Figure 4: Voltage generated on the sense resistor vs. input current. A) For low input power  $< 15$  mW, the sensor remains intact. B) As the input is increased further, the sensor starts to vaporize. C) Partial vaporization at sensor end-of-life occurs before the resistor breaks, producing an open circuit.

## Fabrication:

$0.5 \mu\text{m}$  and  $1 \mu\text{m}$  PECVD oxide ( $\text{SiO}_2$ ) films were deposited on the device side and backside respectively of a  $500 \mu\text{m}$  silicon wafer with the GSI PECVD tool. PECVD oxide on the backside of the wafer was then patterned by reactive etching in the Oxford 81 tool followed by deep silicon etching in the Unaxis DRIE tool to produce oxide membranes on the device side.  $10\text{nm}/40\text{nm}$  Ti/Au was evaporated on the  $0.5 \mu\text{m}$  device side and patterned with lift-off to realize metal contacts to the piezoresistive graphene layer using the odd-hour evaporator. Commercially purchased CVD-grown single layer graphene (SLG) on copper foil was then transferred on the device side with PMMA as a handle layer and patterned with oxygen plasma in the Oxford 81 to confine it to the membrane region. A 5 wt. % PPC solution in dichloromethane (DCM) was then spin-coated on the device side to produce a  $1.8 \mu\text{m}$  PPC film, which eventually serves as the sensor membrane — details of the PPC solution preparation are available in [4]. The graphene-on-polymer and metal-on-polymer devices are shown in Figure 2. Figure 3 shows the electrical configuration for testing the metal-on-polymer and graphene-on-polymer devices with sensitivities of  $6.12 \times 10^{-3}$  and  $68 \times 10^{-3}$  kPa $^{-1}$  respectively, the graphene sensor showing a more than 10x improvement in performance over the metal strain gauge. Figure 4 shows the DC current vs voltage (IV) sweep and corresponding sensor self-destruction with optical verification.

## Conclusions and Future Steps:

Our work demonstrates a graphene-on-polymer flexible sensor architecture for self-destructing electronics with low-voltage operation and high pressure-sensitivity using a representative sensor device. The architecture can be extended other self-destructing sensors such as accelerometers, gyroscopes, and gas sensors for a more ubiquitous self-destructing sensor platform.

## References:

- [1] H. Tian, et al., "A graphene-based resistive pressure sensor with record-high sensitivity in a wide pressure range," *Sci. Rep.*, vol. 5, no. 1, pp. 1-6, Feb. 2015.
- [2] P. Miao, J. Wang, C. Zhang, M. Sun, S. Cheng, and H. Liu, "Graphene Nanostructure-Based Tactile Sensors for Electronic Skin Applications," *Nano-Micro Letters*, vol. 11, no. 1. SpringerOpen, Sep. 01, 2019.
- [3] V. Gund and A. Lal, "Graphene-On-Polymer Flexible Vaporizable Sensor," in *Proceedings of the IEEE International Conference on Micro Electro Mechanical Systems (MEMS)*, Jan. 2021, vol. 2021-January, pp. 521-524.
- [4] V. Gund, A. Ruyack, A. Leonardi, K. B. Vinayakumar, C. Ober, and A. Lal, "Spatially Controlled Transience of Graphene-Polymer Electronics with Silicon Singulation," *Adv. Funct. Mater.*, vol. 29, no. 20, p. 1900592, May 2019.