Interlayer bonding characterization of rGO-based electronics

Maysam Rezaee[1], Li Chih Tsai[2], Azam Elyasigorji[3], Nathan Salowitz[1]

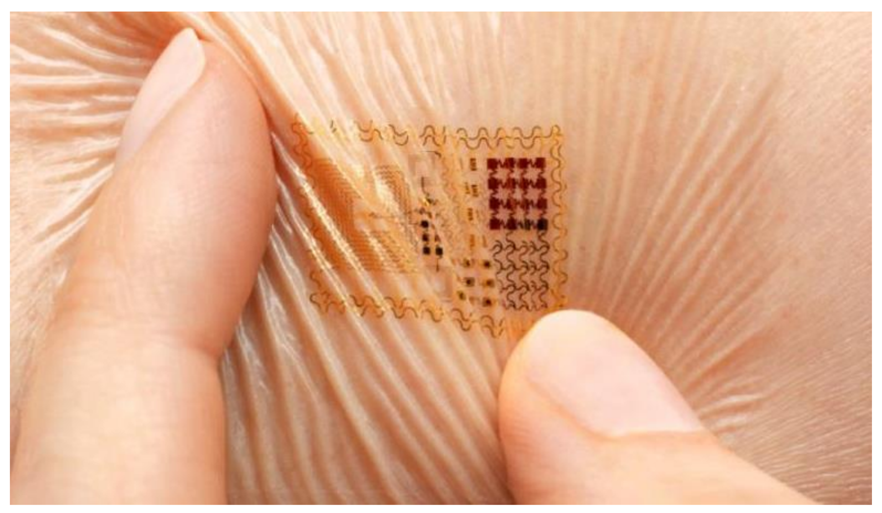
[1]Department of Mechanical Engineering, University of Wisconsin-Milwaukee

[2]Department of Biomedical & Health Informatics, University of Wisconsin-Milwaukee

[3]Department of Civil and Environmental Engineering, University of Wisconsin-Milwaukee

Introduction

Adhesive and cohesive properties of layered structures are critical to their strength. Analysis of mechanical properties of micro/nano-fabricated devices, which are usually synthesized in a layer-by layer process is challenging due to their small scale and associated low strength, especially when the devices are created on a flexible substrate.



Credit: Christopher Mims; qz.com

This poster presents a novel technique and results evaluating the adhesive and cohesive failure of reduced graphene oxide (rGO) films deposited on flexible polyimide substrates. rGO was drop casted on a thin polyimide substrate and then thermally reduced. Parameters of the synthesis process were varied to evaluate their effects on their strength. An image processing technique was employed on each sample to characterize the adhesive vs. cohesive failure modes. The failure energy released by each failure mode were characterized by superimposing of the record of measured force and image processing data It was found that adhesive and cohesive failure energies were highly dependent to the actual amount of rGO particles peeled off of the substrate.

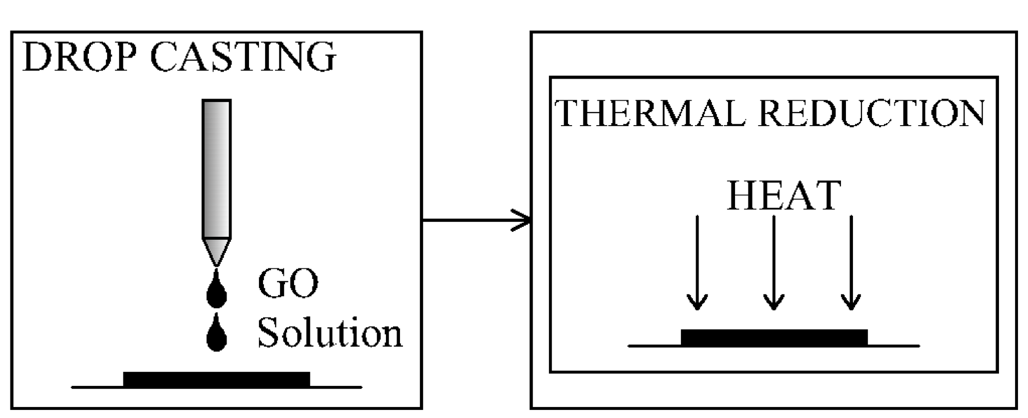
Methods

•**Drop casting:**

- Kapton was selected as a flexible substrate and high thermal chemical stability.
- Substrates were treated by O2 for 5 min to activate the surface for bonding.

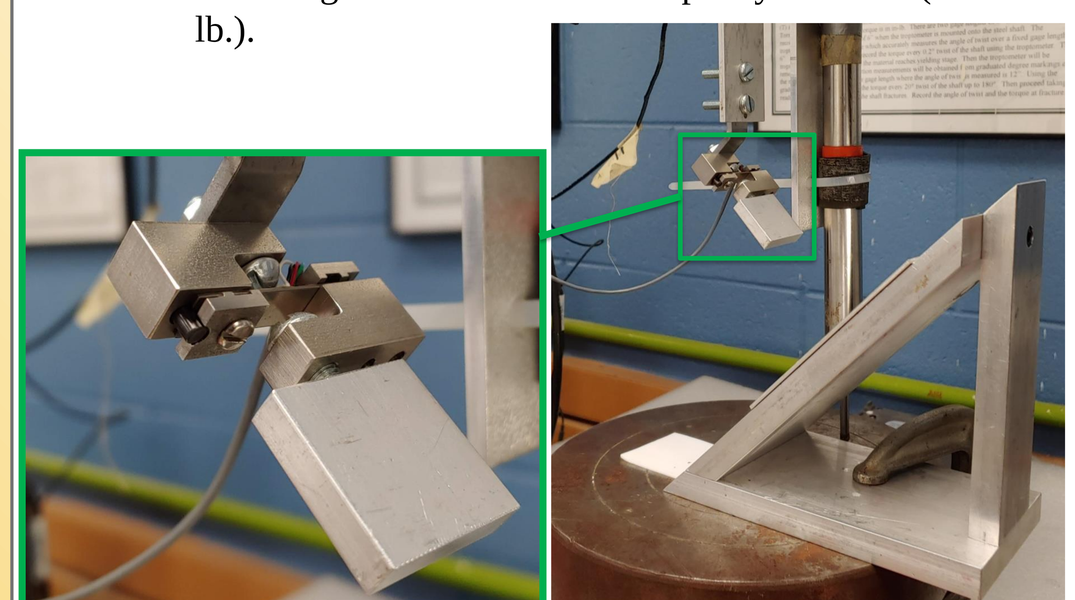
•**Thermal reduction:**

- Sonication bath for one hour
- Drying in oven with temperature of 36°C overnight
- Reduction in argon environment with temperature ramped to 350°C in 10 minutes



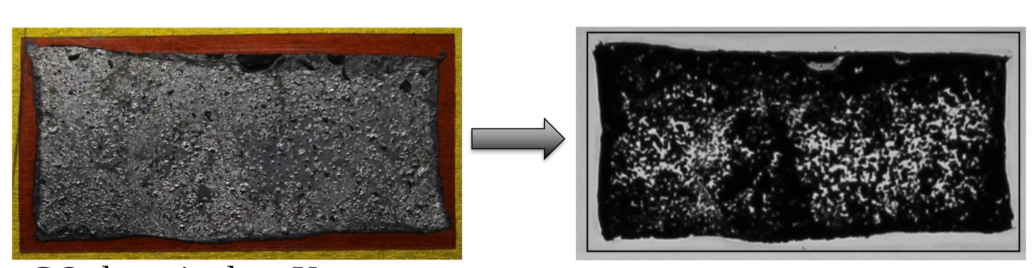
• **Peel test:**

- Peel tests were performed using the peel test method proposed by Rezaee et al [2019] to control significant factors on peel test measurement. In this method, a fixed peel rate and peel angle of 90° were consistently generated using the Instron MTS and a 45° titled test fixture. 3M Scotch® Magic™ 810 tape (with the width and thickness of 19 mm and 0.060 mm respectively) was used in testing. To prevent the Kapton film from lifting or deforming during testing, it was attached to an aluminum testbed with a thin double sided Kapton tape. The tape was pulled up in a direction parallel to the normal vector of the substrate surface by extension rate of 1 mm/sec using the Instron MTS. Measurements were recorded by Omega LCL-010 full bridge load cell with the capacity of 45 N (10 lb.).



• **Image processing:**

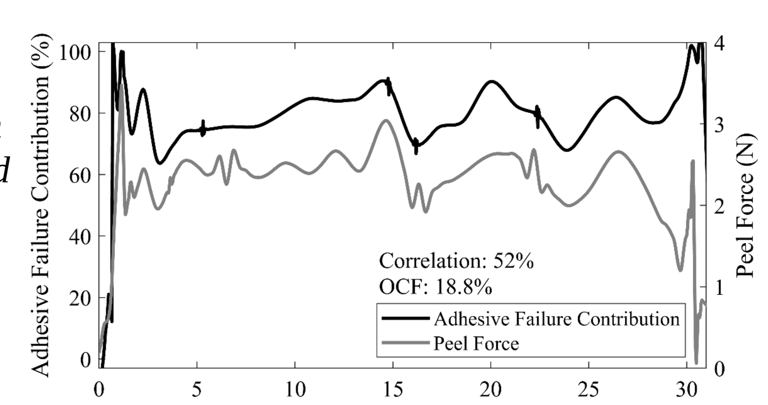
- After peeling, the tape with remnants of rGO was transferred onto an overhead transparency film and a light pad was employed to take images of visible rGO particles stuck on the tape after peeling using a Canon EOS Rebel T6 18 megapixel DSLR camera with a 55mm lens at a distance of 300 mm from samples. After converting images to grayscale, a threshold-base image processing technique was used to identify the profile of visible rGO particles along the tape. In addition to the total amount of peeled rGO, measurements of actual width of peeled rGO were done across the width of the tape at every pixel which is in average corresponding to 1/20th of millimeter.



rGO deposited on Kapton before peel test

Image of rGO particles peeled off and stacked on tape back

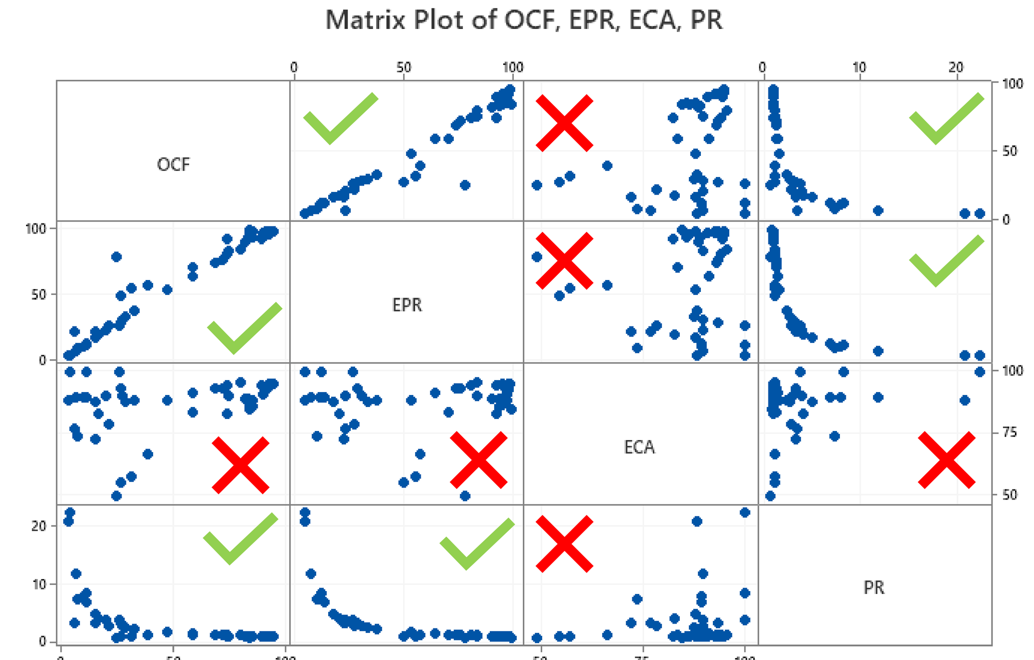
High correlation between unpeeled width variation and peel force variation



Result

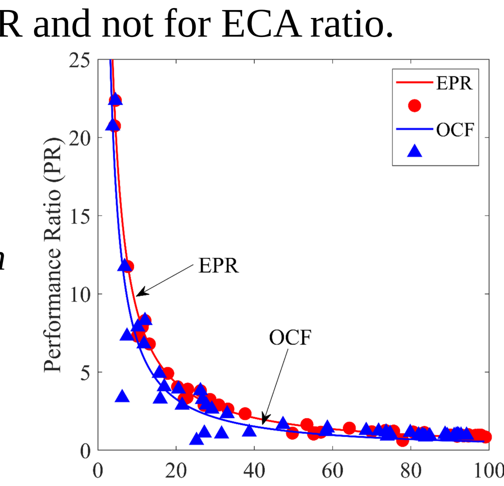
- Thermal reduction stability evaluation:
  - Overall Cohesive Failure (**OCF**)
    The percentage of peeled rGo particles with respect to the whole area of sample.
  - Effective Covered Area (**ECA**)
    A percentage whole area of sample which is covered by rGo particles after thermal reduction process
  - Effective Peeled Region (**EPR**)
    A percentage of effective covered area in which the rGO particles were peeled off from sample
  - ❑ ECA and EPR has a combinatorial effects on the peeled OCF which is not desirable
  - Performance Ratio (**PR**)
    The ration of ECA over EPR representing an index to evaluate the stability of thermal reduction process.

Matrix Plot of OCF, EPR, ECA, PR



- The PR ratio shows a meaningful variation with OCF and EPR and not for ECA ratio.

Strong dependence of performance ration (PR) on variation of OCF and EPR



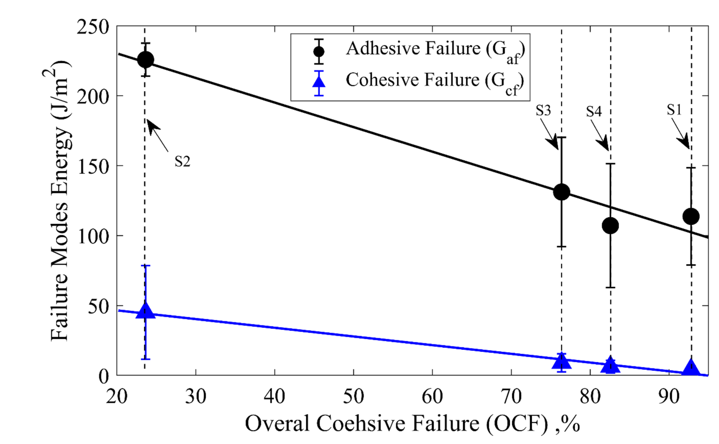
Result

- Bonding force calculation:
- Correspondence of measured peel force and unpeeled regions profile

$$F_i = G_{af} w_i + G_{cf} b_i$$

G_{af}: equivalent adhesive failure energy
w_i: width of unpeeled region
G_{cf}: equivalent cohesive failure energy
b_i: width of peeled region

- Equivalent adhesive failure G_{af}
  tape & bare substrate
  tape & rGO (visible and invisible)
  cohesive failure of invisible rGO layers
- Equivalent cohesive failure G_{cf}
  interlayer rGO
  Adhesive failure rGO & substrate



- Obtained G_{af} and G_{cf} are significantly affected by overall cohesive failure (OCF)
- Constant values of G_{af} and G_{cf} are obtained.

Conclusions

- A controlled peel tests produced repeatable result so that the compatibility with image processing output and bonding force model has been approached.
- High correlation between variations of peel force and unpeeled width was promising fact which led to mathematical model to calculate the release energy associated with failure modes of bonding.
- Cohesive failure energy is highly dependent to the amount of peeled rGO particle from substrate which was expected.
- Adhesive failure energy was found with less correlation with linear trend in comparison to cohesive failure.
- The magnitudes of adhesive and cohesive failure energies were determined only using a single test which a powerful tool to predict the cohesive failure in near future.

Selected References:

- M. Rezaee, L.-C. Tsai, M. I. Haider, A. Yazdi, E. Sanatizadeh, N. P. Salowitz, *Sci. Rep.* **2019**, *9*, 19805.
- L.-C. Tsai, M. Rezaee, M. I. Haider, A. Yazdi, N. P. Salowitz, in *ASME 2019 Conf. Smart Mater. Adapt. Struct. Intell. Syst.*, American Society Of Mechanical Engineers, **2019**
- G. Eda, M. Chhowalla, *Adv. Mater.* **2010**, *22*, 2392
- Z. Gu, S. Li, F. Zhang, S. Wang, *Adv. Sci.* **2016**, *3*, 1500327

Acknowledgement

This work was supported by The National Science Foundation (NSF) under Grant No 1727846, titled "SNM: Customized Inkjet Printing of Graphene-Based Real-time Water Sensors". CS Hyde Company provided samples of PTFE and PEEK. DuPont Company provided Kapton samples.