

Capturing subtle changes during plant growth using wearable mechanical sensors fabricated through liquid-phase fusion

Hussam Ibrahim, Satyanarayana Moru, Sung-Min Kang, Shihao Yin, Yuncong Chen, Liang Dong

Abstract— This paper reports the development of a novel mechanical sensing elastomer that leverages the advantages of graphene, and multi-walled carbon nanotubes (MWCNTs) into Ecoflex and integrated using a unique liquid phase fusion process. The graphene-MWCNT elastomer is used to form a stretchable sensor for monitoring subtle changes of the plant growth over multiple days in a field condition.

I. INTRODUCTION

Wearable sensors have been vastly studied and utilized for consumer electronics and biomedical applications, including but not limited to flexible electronics [1][2], on-skin sensors [3][4][5], and energy harvesting [6]. In particular, carbon-based elastomers have been extensively developed for low-cost flexible mechanical sensors [7][8][9], but yet are not in the commercial phase.

The selection of high yielding and stress-tolerant plants is necessary to ensure that crop production [10][11][12][13], biomass, expansion rates, and leaf movement are important phenotypic traits. Plants show different growth rates at different growth stages, biotic and abiotic stresses, and environmental conditions. While cameras have been widely used to image overall growth of plants even in field conditions, it is a challenging task to provide enough details or subtle changes of the growth at different parts of plant. In addition, imaging the growth at night using a camera is not easy due to poor light condition and high power needs.

Here we report the development of a novel mechanical sensing elastomer that integrates graphene [14], multi-walled carbon nanotubes (MWCNTs), and Ecoflex silicone together a liquid phase fusion process [15]. The graphene-MWCNT elastomer is used to realize a stretchable sensor for monitoring subtle changes of plant growth over multiple days under field conditions.

II. EXPERIMENTAL

A. Fabrication

Figure 1 shows the fabrication process for the graphene-MWCNTs Ecoflex sensor. First, 6-ml precursor solution of Ecoflex was placed on a hotplate at 65°C for 5 min to obtain partially cured Ecoflex. Second, 2 ml graphene-dispersed solution was poured onto the top of the partially cured Ecoflex and then left on the hotplate till the elastomer was fully cured. The graphene-dispersed solution used both ethanol and DI water in a 7:3 volume ratio [16], where the DI water allows graphene to experience a hydrophobic behavior, and transfer from solution of water to elastomer and form a graphene layer

at the top surface of Ecoflex. Consequently, bonding between graphene and Ecoflex at the top interface is established.

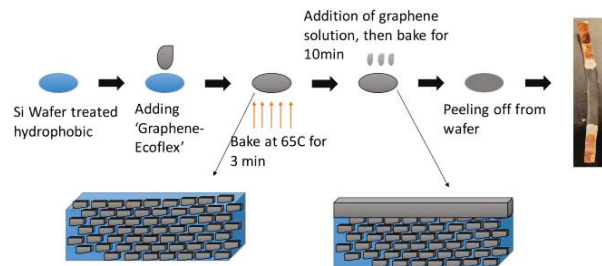


Figure 1. Fabrication processes for the sensor. A Si wafer is first treated with silane to render a hydrophobic surface. Ecoflex parts A and B are mixed, stirred, and poured over the wafer, followed by being heated on a 65°C hotplate for 3 min. The Ecoflex layer is thus partially cured. After that, a pre-made mold is placed in a bath of graphene dissolved in ethanol:DI water. The graphene undergoes liquid-phase fusion due to the use of DI water, and graphene particles are transferred into the porous Ecoflex, thus producing a graphene-fused Ecoflex device. Finally, a suspension solution of MWCNTs is added onto the top surface, and the MWCNTs are further rolled into the graphene-fused Ecoflex after the solvent evaporates.

Figure 2 shows the scanning electron microscope (SEM) images to highlight the difference between the fully cured pure Ecoflex (Fig 2A), and the fully cured Ecoflex with graphene fused at the top layer (Fig 2B). After that, a suspension solution of MWCNTs was added at the top of graphene and uniformly distributed inside the fully cured Ecoflex-graphene layer to enhance conductivity. On both ends of the graphene-MWCNT sensor, copper wires were integrated through adding silver ink. Following that, a thin layer (~150 μm) of Ecoflex is spin coated and fully cured to minimize the effect of humidity.

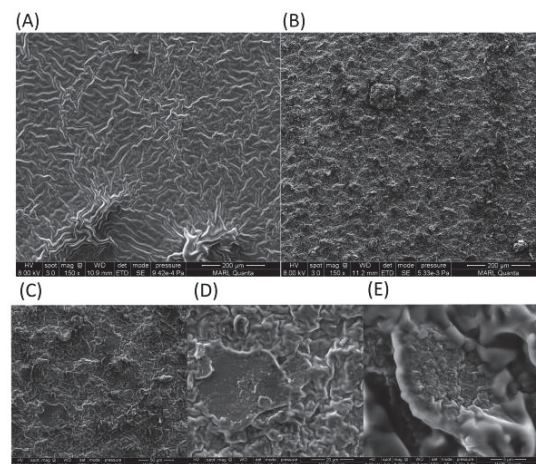


Figure 2. SEM images for the fully cured Ecoflex (A) and fully cured Ecoflex with graphene fused into the surface (B). SEM images for the sensor at different magnifications. 500x (A), 1500x (B), and 5000x (C). MWCNTs were not shown since it was rolled onto the surface.

A motorized linear stage was powered by a stepper motor, and commands were sent to the linear stage through a custom-

All authors are with the Department of Electrical and Computer Engineering, Iowa State University, Ames, Iowa, USA 50011.
*Contacting Author: Liang Dong (phone: +1 515-708-0525; email: ldong@iastate.edu).

made Arduino code to characterize the response of the graphene-MWCNT sensor to different strain%, as well as the stability and life-span of the sensor. Figure 3A shows the response of the sensor to different large strains from 10 to 150 %. At each strain%, the resistance of the sensor was found to shift to a higher level with minimal noise. Figures 3B and 3C compare the surface morphologies of the sensor in the relaxed and stretched state (150% strain), where no changes in graphene distribution were observed compared to the relaxed state of the sensor.

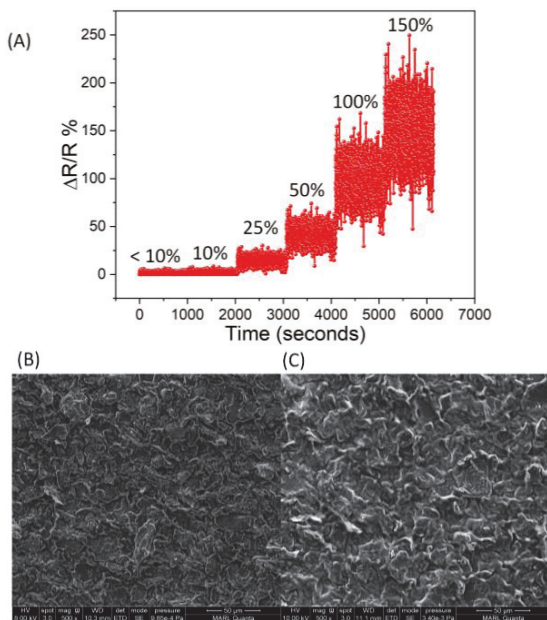


Figure 3. Response of the graphene-MWCNT to different strain% from 0 to 150% (A) SEM images of the graphene-MWCNT sensor in relax state (B), and after 150% stretch (C).

Figure 3A shows the response of the sensor to large strains from 10 to 150 %. At each strain%, the sensor's resistance was found to shift to a higher level with minimal noise. Figures 3B and 3C compare the surface morphologies of the sensor in the relaxed and stretched state (150% strain), where no change in graphene distribution was observed compared to the relaxed state. Figure 4 shows that the sensor is sensitive to micrometer scale stretching within 10% strain.

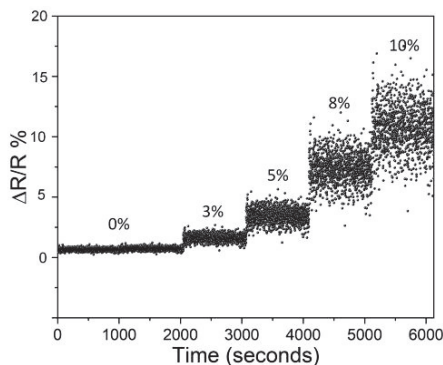


Figure 4. Response of the graphene-MWCNT to different strain% from 0 to 10%.

Figure 5A shows that after more than 10000 cycles of 45% stretch-release processes, the data shows periodic square waves that are synchronized with stretching (resistance increases) and relaxing (resistance goes back to initial baseline value). Figure 5B confirm that the graphene dispersion hasn't been affected by the multiple stretch-release cycles applied on the sensor.

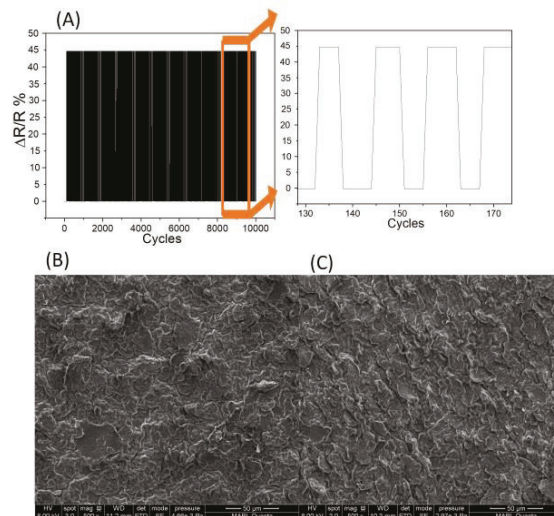


Figure 5. Response of the graphene-MWCNT to 10000 cycles of 45% strain (A) SEM images of the graphene-MWCNT sensor in relaxed state (B), and after 10000 cycles of 45% strain (C).

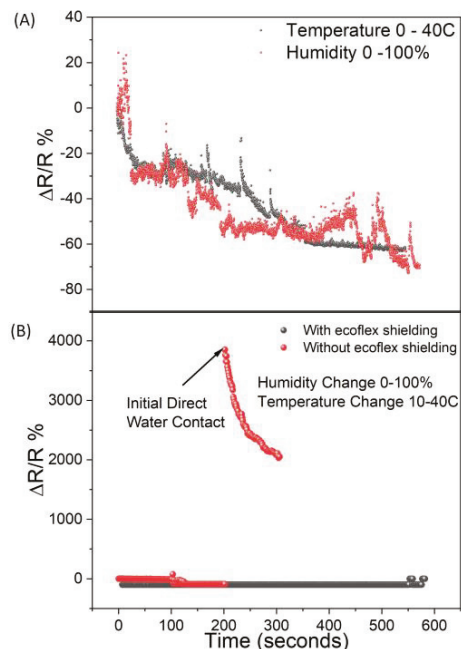


Figure 6. Response of the graphene-MWCNT sensor (without shielding) to different temperature and humidity settings. (A) Response of graphene-MWCNT sensor (with and without shielding) to temperature, humidity, and direct water contact (B).

To deploy the sensor in the field, the effects of humidity, temperature and water contact on the sensor's measurement were characterized. Figure 6 highlights the importance of integrating a shielding layer to eliminate outside effects on the

sensor performance; the performance of the sensor without a shielding Ecoflex layer was significantly influenced by the temperature and humidity (Fig. 6A), but those variations are minimized when the thin Ecoflex shielding layer ($\sim 130\mu\text{m}$) is added on top of the graphene-MWCNT layer. The large variations in temperature and humidity are used to resemble different climate conditions in the summer, while the direct water contact is used to resemble the event of rain fall on the crops.

III. RESULTS AND DISCUSSION

In the corn field, two sensors were installed in the lateral direction, while two other sensors were installed in the radial direction. Pure Ecoflex was used as an adhesive material to attach the sensors to the plants as described before, and left to cure overnight. The sensors contact pads were connected to data loggers to collect resistance values for the plants at different locations. As a reference, images for the plants were acquired by a phone camera to confirm the growth of the plants on a daily basis, while the data loggers were used to continuously measure resistance values for a week.



Figure 7. Installation of graphene-MWCNT sensor in lateral and radial directions of plant stock at different heights (lower stock in left picture, and higher stock in right picture).

Figure 8 shows the growth of the plant using camera photos taken discretely (Fig 8A), and continuous measurement of resistance values (Fig 8B) at different positions of the stem. From a visual perspective, plant 1 has grown by 4.4 cm. However, the plot from Fig 8B has shown fluctuations in the resistance values owing to a plant's growth and behavior during the course of the day. Plant 1 showed growth patterns of $327\Omega/\text{cm}$ in lower stem part, and $400\Omega/\text{cm}$ in upper stem part.

The same type of data was taken for plant 2 as displayed in figure 9. The visual images show that plant 2 has grown by 7.4 cm; it grow at a slower rate than plant 1 since it was an older plant. After 7 days, plant 2 showed growth patterns of $300\Omega/\text{cm}$ in lower stem part, and $207\Omega/\text{cm}$ in upper stem part which were lower rates when compared to plant 1. Both plants 1 and 2 had the same treatments, and were present in the same location), the faster growth of plant 1 was evident in both the visual pictures, as well as rate of growth obtained from lower stem.

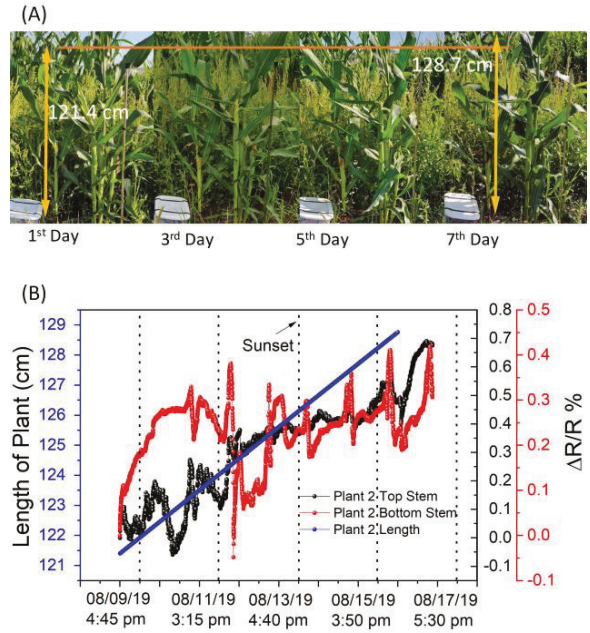


Figure 8. Growth monitoring of maize plants using two different methods. Camera in (A), and graphene-MWCNT sensors in (B).

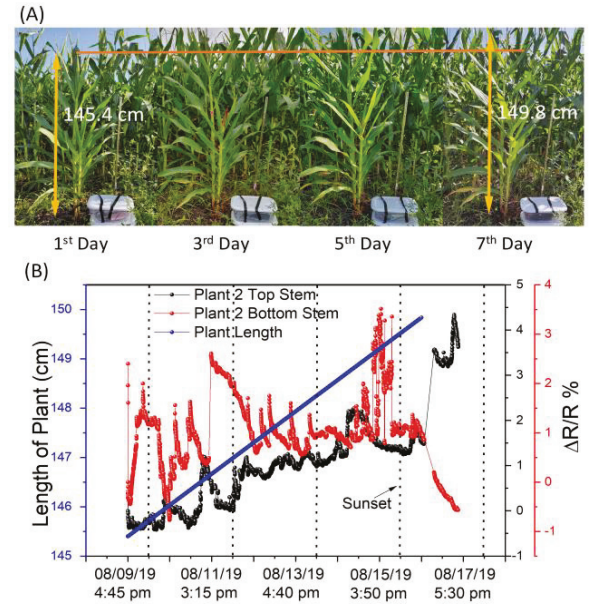


Figure 9. Images of the plot's growth over the course of 7 days taken discretely (A), and continuous measurements of the resistance using graphene-MWCNT sensors (B).

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