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Electrodes fabrication on Kapton film with NEJE mini laser - L-Cu1.1 V.1

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We use this protocol and it's working

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Disclaimer

Abstract

This protocol describes the fabrication of laser inscribed graphene (LIG) electrodes on polyimide film (i.e., Kapton film) using the NEJE mini laser with copper electrodeposition.

Figure 1: Process flow for LIG fabrication using NEJE mini laser.

Guidelines

The protocol is based on numerous published manuscripts that use similar methods with the, but is not a comprehensive guide.

Materials

General

- Polyimide Film - Electrical-Grade, 0.0050" thick, 12" x 12" sheet
- Scissors
- Isopropyl Alcohol
- Kim Wipes
- Tape
- Metallic Tape
- Two Copper Rods
- Ethanol
- Phosphoric Acid
- Copper (II) Sulfate
- Silver/Silver Chloride Paste
- Sodium Sulfate
- Stirring Bar
- Beakers

Hardware and Safety

- Mini Laser Engraver Machine - NEJE, 1500mW
- Power Supply - Potential Range: 0 - 30 V; Current Range: 0 - 5 A
- Arduino System
- Stirring Plate

Software

- NEJE_V4.7.2
- Arduino IDE 1.8.13

Troubleshooting

Safety warnings

General Laser Safety

- Wear protective safety goggles when operating the laser.
- Exposure to the laser material processing beam can result in burns to the skin and can cause severe eye damage.

a. The laser will burn skin so avoid direction contact.

b. Do not look directly into the laser beam of use a reflective surface to redirect or view the beam. Do not view directly with optical instruments such as binoculars or microscopes.

- There is not information in the MSDS for polyimide covering the harmful effects of inhaling fumes, but safety should be taken to avoid exposure through inhalation.
- The backing material plays a major role in the amount of vapor/smoke produced, and this should be carefully monitored when changing materials.
- Never operate the laser system without constant supervision of all cutting, marking, and engraving processes. Exposure to the laser beam may cause ignition of combustable materials which can lead to a fire. A properly maintained fire extinguisher should always be kept on hand.
- A remote connection to the system across a network does not negotiate the constant need for constant supervision of all cutting, marking, and engraving processes.
- Never leave materials in the laser system after processing. Always remove all materials, including scrape metal, from the machine after use.

Before start

- Wear proper PPE (personal protective equipment).
- Ensure the workspace is clean and ready for use. Make a note of the location of the fire extinguisher, safety shower, and first aid kit. Ensure there are not stools or bags blocking isles.
- Ensure all materials needed are easily accessible.
- Thoroughly read and understand the steps of the procedure

Procedure

1h 17m

1 Prepare Substrate

5m

a. Cut a 2" by 5" (5cm x 5cm) sample of kapton film.
b. Place a few drops of isopropyl alcohol on a Kimwipe and clean the surface.

> Note
>
> Make sure to not apply the alcohol directly to the sample. The alcohol should be sprayed onto the Kimwipe, not the sample.

c. Place the sample onto the center of the laser platform (as shown in *figure 2A*)

d. Ensure that the workpiece is flat. If the workpiece is not flat, use the heat press.
e. Before turning on the laser, confirm that the power and the USB cables (shown in *figure 2B*) are connected to the electricity supply and the computer, respectively.

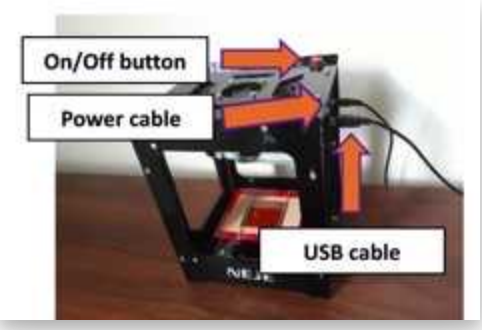

Figure 2B

f. Press the red button at the top of the laser (shown in *figure 2B*) to turn it on and verify that the laser beam is set in the center of the kapton film. If not centered, turn off the laser, move the substrate and verify again by turning the laser on (repeat until centered).

> Note
>
> Do not move the laser beam with the software as this can damage the sample.

g. Open the software NEJE - V4.7.2.

h. Open the folder containing the picture "Three electrodes design x 2" and drag it to the NEJE software. Double click on the picture to make sure that the picture will be scribed completely (shown in *figure 2C*). Then press "Ok." Press "Ok" in step 2.

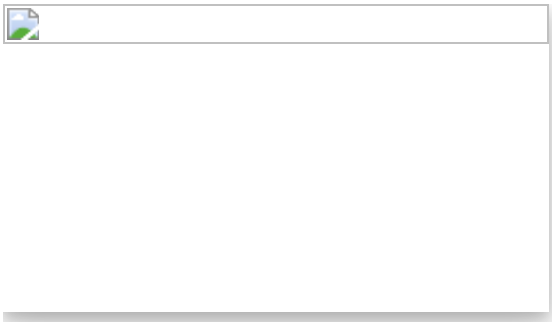

Figure 2C

i. Next, choose the first option (top, left corner shown in *figure 2D*). In step 4, click "ok."

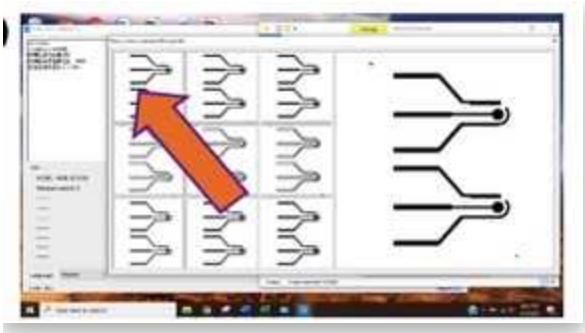

Figure 2D

j. Click on the square with red dots in the menu to the right (shown in *figure 2E*) to confirm that the laser will not scribe outside the kapton film and turn the laser on again. If needed, turn off the laser, accommodate the kapton film and turn the laser on again.

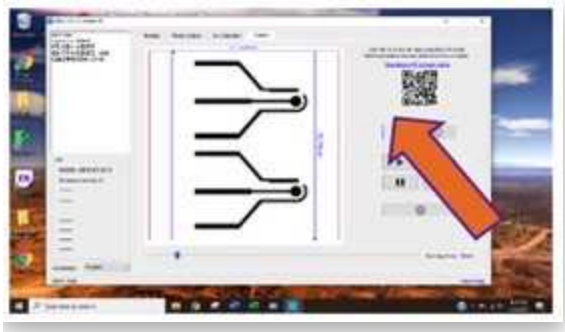

Figure 2E

k. Place the green protective screen on the laser (shown in *figure 2F*).

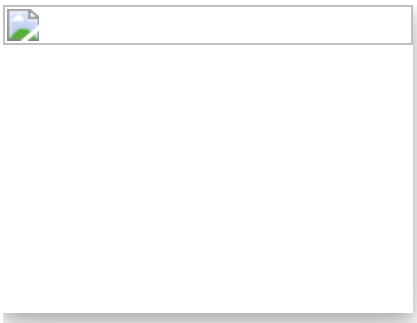

Figure 2F

2 Enter Laser Settings

2m

a. Set the laser to burn 2 times each at 15 minutes (shown in *figure 3A*).

Figure 3A

b. In the settings, verify that brightness is on 3 (shown in *figure 3B)*.

Figure 3B

3 Process Job; Pause if Necessary

10m

a. Press the "go" button to start the job (shown in *figure 4A*).

Figure 4A

b. The time left to complete each burn is displayed in the bottom, right area of the screen. The software also shows if the first or second carving is in process displayed on the top, left area (shown in *figure 4B*).

Figure 4B

Note

Do not look directly at the laser beam during the process. *Figure 4C* provides an example if you are curious.

Figure 4C

c. The job can be paused using the software (shown in *figure 4D*). The "go" button will resume the process. Turn off the laser using the red button at the top of it during an emergency.

Figure 4D

4 **Turn off Laser & Inspect LIG Electrode** 10m

a. Turn the laser off by pushing the red button at the top of the laser.
b. Remove the green, protective screen and take the sample.
c. Inspect the image with a UV pen (shown in *figure 5*).

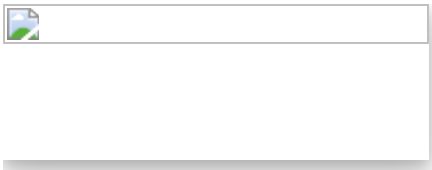

Figure 5

5 **Incorporation of Metallic Tape & Nail Polish** 10m

a. Cut each electrode and incorporate a small piece of metallic tape in the connectors of each electrode (shown in *figure 6A*).

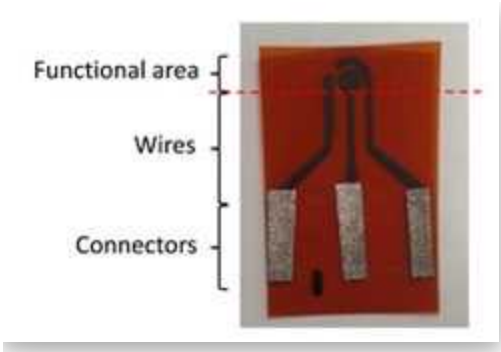

Figure 6A

b. Apply sufficient nail polish on the wires of the working electrode and counter electrode (shown in *figure 6B*).

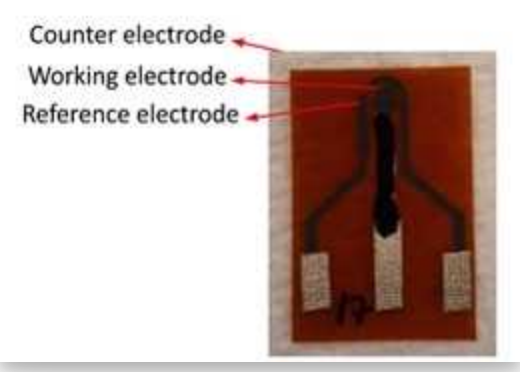

Figure 6B

Note

Do not cover the functional area of the electrodes. Only apply nail polish in the wires of the electrodes.

c. Let the nail polish dry completely.

6 **Electropolishing of Copper Rod** 10m

a. Prepare a 30mL solution of 25% v/v phosphoric acid and 25% v/v ethanol.
b. Connect the Arduino system to the DC power supply and computer.
c. Open the "Electroplating_code" file code on the Arduino software.
d. Set the long onTime to 30000 to perform the electropolishing for 30 seconds, and the long offTime to 5000, and click upload (arrow found in the top, left area of the screen shown in *figure 7A*). The software should indicate "Done uploading" at the bottom of the screen (shown in *figure 7B*).

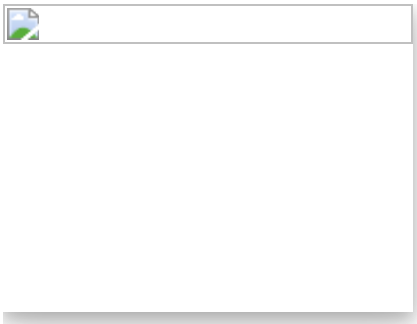

Figure 7B

e. Connect the copper rod that will be polished to the cathode (negative terminal) and the other copper rod to the anode (positive terminal) of the DC power supply.

f. Immerse the copper rods into the phosphoric acid solution.

Note

Be careful to keep the alligator clips separate from the solution.

g. Set a constant voltage of power supply to 9 V by turning the gray nob (shown in *figure 7C*).

Figure 7C

h. Turn on the power supply by pushing the big orange button. Then, push the small button (shown in *figure 7C*).
i. Push the black button on the breadboard. If nothing happens, push the black button again. The red light indicates that the board is on. The green light indicates that the current is flowing (shown in *figure 7D*). Bubbles will form around the copper rods.

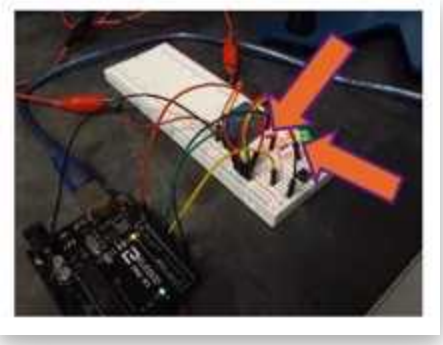

Figure 7D

j. When the time is complete, push the black button on the board to disable the system.
Note: The polished copper rod should look brighter than before (as shown in *figure 7E*).

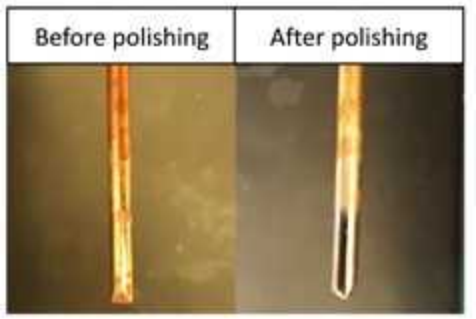

Figure 7E

k. Press the small button on the power supply to keep it on stand-by.
l. Remove the copper rods and rinse them with water.

Note

Hazardous waste management: The rinse water cannot be poured down the drain. Collect it in a beaker, and dispose of it in a hazardous waste container.

7 **Copper Electrodeposition** 10m

a. Prepare a 20mL solution of 250mM copper (II) sulfate $CuSO_4$ and 2.50mM sodium sulfate Na_2SO_4.
b. In the Arduino software, set the long onTime to 1000 to perform the electrodeposition for 1 second, and the long offTime to 5000, and click "upload."
c. Connect the polished copper rod to the cathode (negative terminal) and the working electrode to the anode (positive terminal) of the DC power supply.
d. Insert a stirring bar into the solution, place the beaker containing the solution over a stirring plate, and turn it on.
e. Immerse only the copper rod to the electroplating solution
f. Ensure that the voltage of the power supply is 9 V. Turn the gray nob to change the potential if necessary.
g. Turn on the power supply by pushing the small button.
h. Push the black button on the breadboard. If nothing happens, push the black button again.
i. When the circuit is off (only the green light is on), immerse the electrodes into the electroplating solution. Keep the electrodes in the solution until 1 cycle is completed (the red light turns on for 1 second and then turns off) and remove the electrode (shown in *figure 8*). During the electrodeposition, verify that there is a change of color on the surface of the working electrode due to the dispersion of bubbles. If this does not happen, discard the electrode and verify that all the connections are correct.

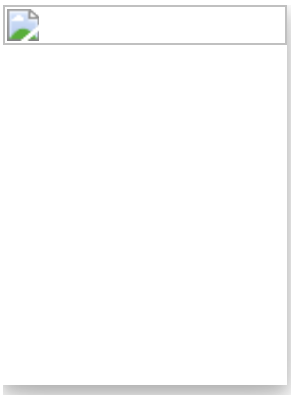

Figure 8

> Note
>
> Be careful in making sure the electrodes in the solution only sit for one cycle. If necessary, increase the long offTime in Arduino.

j. Push the black button on the board to disable the system.
k. Rinse the electrodes with DI water and let them dry off at room temperature for about 10 minutes.

> Note
>
> Hazardous waste management: The rinse water cannot be poured down the drain. Collect it in a beaker and dispose of it in a hazardous waste container.

l. Turn off the DC power supply and computer.
m. Properly store the materials and reagents for future experiments.
n. Clean the working station.

8 Silver/Silver Chloride Paste Application 20m

a. Apply a layer of silver/silver chloride (Ag/AgCl) paste over the functional area of the reference electrode and let it dry (shown in *figure 9*).

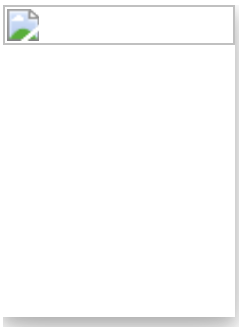

Figure 9

b. Cover the wires with nail polish and let them dry (shown in *figure 9)*.

Supplemental Information

9 Arduino Code:

```
/* Electroplating
 *
 * Toggle relay with set on and off durations
 * Power switch interrupt to toggle cycling of relay
 *
 */

const int powerSwitch = 2;
const int bathLed = 6;
const int relay = 7;
const int powerLed = 13;

long onTime = 30000;
long offTime = 5000;

volatile bool isActive = false;

void turnOn() {
  digitalWrite(relay, HIGH);
  digitalWrite(bathLed, HIGH);
}

void turnOff() {
  digitalWrite(relay, LOW);
  digitalWrite(bathLed, LOW);
}

void toggleState() {
```

```
  isActive = !isActive;
  digitalWrite(powerLed, isActive);
}

// Delay with escape logic
void await(long timeToWait) {
  for(int j=0; j<timeToWait; j++) {
    delay(1);

    if(isActive == LOW) return;
  }
}

void setup() {
 // Set both relay and powerLed pins to OUTPUT
  pinMode(relay, OUTPUT);
  pinMode(powerLed, OUTPUT);

  // Set power switch pin to INPUT
  pinMode(powerSwitch, INPUT);

  // Default power switch to HIGH state
  // Pressing switch brings it LOW
  digitalWrite(powerSwitch, HIGH);

  // Attach an interrupt to the power switch pin
  attachInterrupt(digitalPinToInterrupt(powerSwitch), toggleState, FALLING);

}

void loop() {

  while(isActive) {
    // Turn on relay for on duration
    turnOn();
    await(onTime);

    // Turn off relay for off duration
    turnOff();
```

Protocol references

Tehrani & Bavarian, 2016. https://10.1038/srep27975
Vanegas et al., 2018. https://doi:10.3390/bios8020042