

Submission ID #: 68230

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Title: Optimized Fabrication Procedure for High-Quality Graphene-Based Moiré Superlattice Devices

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Author Questionnaire

1. Microscopy: Does your protocol require the use of a dissecting or stereomicroscope for performing a complex dissection, microinjection technique, or something similar? **No**

2. Software: Does the part of your protocol being filmed include step-by-step descriptions of software usage? **Yes, all done**

3. Filming location: Will the filming need to take place in multiple locations? **No**

Current Protocol Length

Number of Steps: 32

Number of Shots: 60

Introduction

- 1.1. **Pablo Jarillo-Herrero:** Our research is focused on the quantum electronic properties of two-dimensional materials and their heterostructures, aiming to discover and investigate new quantum phenomena and develop novel electronic devices.

1.1.1. INTERVIEW: Named Talent says the statement above in an interview-style shot, looking slightly off-camera.

What significant findings have you established in your field?

- 1.2. **Pablo Jarillo-Herrero:** We pioneered the field of “twistronics”, where the properties of 2D materials heterostructures change as a function of the angle between the layers. The most impactful discovery was superconductivity in magic-angle graphene.

1.2.1. INTERVIEW: Named Talent says the statement above in an interview-style shot, looking slightly off-camera. *Suggested B.roll: 2.8*

What are the current experimental challenges?

- 1.3. **Shuwen Sun:** Achieving a precise twist angle remains challenging due to heterostrain, disorder, and lattice relaxation introduced during the nanofabrication process, which often lead to device inhomogeneity and limit reproducibility across samples.

1.3.1. INTERVIEW: Named Talent says the statement above in an interview-style shot, looking slightly off-camera.

How will your findings advance research in your field?

- 1.4. **Shuwen Sun:** This protocol reflects our experience optimizing each fabrication step to improve uniformity and yield, enabling more researchers to reliably build high-quality graphene moiré devices and accelerate progress in this field.

1.4.1. INTERVIEW: Named Talent says the statement above in an interview-style shot, looking slightly off-camera. *Suggested B.roll:3.4*

Videographer: Obtain headshots for all authors available at the filming location.

Protocol

2. Laser Patterning and Pickup of Top Hexagonal Boron Nitride Layer

Demonstrator: Shuwen Sun

2.1. To begin, place a graphene wafer on the sample stage of the transfer setup integrated with a supercontinuum laser [1]. Use **Peek Through** to make the **Inkscape** window semi-transparent while floating above the camera window [2]. Use the **Draw Bezier curves and straight lines** function in **Inkscape** to outline the graphene boundary and the planned laser-cutting lines [3].

2.1.1. WIDE: Talent places the graphene wafer on the sample stage.

Added Shot: SCREEN: 68230_2.1-2.3_3.x.mp4 00:11-00:18

2.1.2. SCREEN: 68230_2.1-2.3_3.x.mp4 01:54-02:21 .

2.2. Now power on the laser and increase the intensity until the beam spot is visible [1]. Adjust the sample stage to align the beam spot with the start of a planned laser-cutting line [2-TXT].

2.2.1. Talent switching on the laser and gradually increasing intensity.

AND

SCREEN: 68230_2.1-2.3_3.x.mp4 02:58-03:08

Video Editor: Please play both shots side by side

2.2.2. Talent adjusting the stage so the laser spot aligns with the beginning of a cutting line. **TXT: Adjust laser intensity to suitable level**

AND

SCREEN: [68230_2.1-2.3_3.x.mp4](#) 03:26-03:29

Video Editor: Please play both shots side by side

2.3. Next, move the sample stage to guide the beam spot along the planned laser-cutting line [1]. After zeroing the laser intensity, inspect the laser-cutting line [2-TXT].

2.3.1. Talent guiding the stage to trace along the laser-cutting path.

AUTHOR'S NOTE: Taken as 2 shots

2.3.2. SCREEN: [68230_2.1-2.3_3.x.mp4](#) 03:37-03:51 **TXT: Repeat steps until all planned lines are laser cut**

2.4. To pickup the top hexagonal boron nitride flake, adjust the orientation of the flake until the straight edge is vertical and positioned on the right side [1-TXT].

- 2.4.1. SCREEN : 68230_2.4-2.10.mp4 00:00-00:19. **TXT: Outline flake boundary in Inkscape**
- 2.5. Gently clamp the glass slide with the PC/PDMS (*P-C-and-P-D-M-S*) stamp inside the socket at a downward tilt angle of 2 to 3 degrees [1]. Slide the socket to the left using the sliding tray until the glass slide is positioned above the sample wafer then manually lock it in place [2].
- 2.5.1. Talent clamping the glass slide into the socket at a tilt. **TXT: PC/PDMS: polycarbonate/Polydimethylsiloxane**
- 2.5.2. Shot of the socket being slide to the left and locked.
- 2.6. After setting the sample stage temperature to 50 degrees Celsius, engage the glass slide downward using the Z direction actuator [1-TXT]. Focus the microscope on the polycarbonate film and inspect the surface cleanness [2].
- 2.6.1. Talent engaging the actuator to lower the stamp. **TXT: Speed: 1 mm/s until stamp is 2 mm above wafer**
- 2.6.2. SCREEN: 68230_2.4-2.10.mp4 01:27-01:31
- 2.7. Move the hexagonal boron nitride flake to the selected clean region and engage the slide further downward [1-TXT]. ~~When the polycarbonate film and wafer are nearly in the same focal plane, reduce the speed to 5 micrometers per second [2].~~
- 2.7.1. SCREEN: 68230_2.4-2.10.mp4 02:06-02:30 **TXT: Slide speed: 0.1 mm/s. Reduce to 5 μ m/s when the polycarbonate film and wafer are nearly in the same focal plane**
- ~~2.7.2. SCOPE/SCREEN: Talent adjusting the speed and continuing to engage slowly.~~
NOTE: Shot deleted by authors
- 2.8. Now engage the stamp onto the wafer with a speed of 5 micrometers per second until it completely covers the hexagonal boron nitride flake [1]. Then set the sample stage temperature to 80 degrees Celsius [2].
- 2.8.1. SCREEN: 68230_2.4-2.10.mp4 03:15-03:32.
- 2.8.2. Talent adjusting the stage temperature to 80 degrees Celsius.
- 2.9. After the sample stage temperature reaches 80 degrees Celsius, disengage the stamp with 5 micrometers per second until the wavefront is close to the hexagonal boron nitride straight edge then pick up the flake with a reduced speed of 2 micrometers per second [1].

2.9.1. Shot of the sample stage temperature reaching 80 degrees Celsius

AND

SCREEN: 68230_2.4-2.10.mp4 04:02-05:00.

Video Editor: Please play both shots side by side

2.10. Once picked up, increase the speed to 5 micrometers per second to detach the polycarbonate film from the wafer [1]. Switch off the stage heater and open the water-cooling system [2].

2.10.1. SCREEN: 68230_2.4-2.10.mp4 05:10-05:20.

2.10.2. Talent turning off the heater and turning on the water cooling system.

2.11. When the temperature drops below 45 degrees Celsius, close the water-cooling system [1]. Disengage the glass slide all the way up at 1 millimeter per second [2].

2.11.1. Talent closing the water-cooling system.

2.11.2. Shot of the glass slide being disengaged.

3. Picking up the Graphene Moiré Superlattice

3.1. Place the wafer with the freshly laser-cut graphene on the sample stage and locate the graphene using the 10X objective [1]. Adjust the flake orientation so the laser cutting lines are parallel to the hexagonal boron nitride straight edge [2].

3.1.1. SCOPE/SCREEN: 68230_2.1-2.3_3.x.mp4 06:28-06:47.

3.1.2. SCOPE/SCREEN: 68230_2.1-2.3_3.x.mp4 06:47-07:01.

3.2. Outline the graphene boundary in Inkscape, including the laser-cutting lines [1]. ~~Use Peek Through to make the drawing semi-transparent while floating above the camera window [2].~~

3.2.1. SCREEN: 68230_2.1-2.3_3.x.mp4 07:05

Video Editor: Please freeze frame here .

~~3.2.2. SCREEN: Peek Through is being used to make the drawing semi-transparent.~~

NOTE: Shot deleted by Authors

3.3. Now, move the glass slide with the top hexagonal boron nitride flake to the pre-engage position with the PC/PDMS stamp 2 millimeters above the graphene wafer [1]. Align the flake's straight edge with the center of the laser-cut line to match the hexagonal boron nitride with the graphene drawing [2]. Tune the microscope to focus on the graphene and align it to its Inkscape drawing [3].

- 3.3.1. Shot of the glass slide being moved to the pre-engage position with the top flake above the wafer.
- 3.3.2. SCREEN: 68230_2.1-2.3_3.x.mp4 08:22-08:35 .
- 3.3.3. SCREEN: 68230_2.1-2.3_3.x.mp4 08:45-08:54.
- 3.4. Then, focus on the top flake and move the glass slide in XY (X-Y) direction to align it to its drawing [1]. After refocusing on a focal plane slightly higher than the pre-cut graphene, engage the glass slide downward at 0.1 millimeter per second until the top flake comes into focus [2].
 - 3.4.1. SCREEN: 68230_2.1-2.3_3.x.mp4 09:07-09:19.
 - 3.4.2. SCREEN: 68230_2.1-2.3_3.x.mp4 09:38-09:55
- 3.5. Refocus on the graphene and set the speed to 5 micrometers per second [1]. Engage the stamp slowly until the flake becomes roughly visible while ensuring no contact with the wafer [2-TXT]. Continue to engage the stamp downward until it contacts the wafer [3].
 - 3.5.1. SCOPE/SCREEN: 68230_2.1-2.3_3.x.mp4 10:13-10:25
 - 3.5.2. SCOPE/SCREEN: 68230_2.1-2.3_3.x.mp4 10:30-10:54. **TXT: Realign both the graphene and hBN to their drawings**
 - 3.5.3. SCOPE/SCREEN: 68230_2.1-2.3_3.x.mp4 11:21-11:38.
- 3.6. Engage the stamp at 0.5 micrometers per second until the wavefront is close to the left edge of the flake [1]. Reduce the speed to 0.02 micrometers per second and continue engagement to contact the first graphene piece [2].
 - 3.6.1. SCOPE/SCREEN: 68230_2.1-2.3_3.x.mp4 11:47-12:09.
 - 3.6.2. SCOPE/SCREEN: 68230_2.1-2.3_3.x.mp4 12:09-12:20,15:58-16:14 .
- 3.7. Once the flake fully covers the first graphene piece and is pinned at the straight edge, stop the engagement [1]. Clear hysteresis by moving the stage upward at 5 micrometers per second until the wavefront starts retracting [2].
 - 3.7.1. SCOPE/SCREEN: 68230_2.1-2.3_3.x.mp4 20:40-21:03
 - 3.7.2. SCOPE/SCREEN: 68230_2.1-2.3_3.x.mp4 21:03-21:16
- 3.8. Set a disengaging speed of 0.02 micrometers per second to pick up the graphene gently [1]. When the wavefront moved away from the flake, increase the speed to 5 micrometers per second to fully detach the stamp from the wafer [2].
 - 3.8.1. SCOPE/SCREEN: 68230_2.1-2.3_3.x.mp4 21:22-21:45 .

3.8.2. SCOPE/SCREEN: 68230_2.1-2.3_3.x.mp4 41:07-41:32

3.9. Now copy the entire graphene drawing in Inkscape and rotate it by the desired twist angle [1].

3.9.1. SCREEN: 68230_2.1-2.3_3.x.mp4 41:53-42:15

Video Editor: Please speed up the video .

3.10. Align the second graphene piece in the copied drawing with the first piece in the original drawing to maximize overlap [1-TXT]. Align the remaining graphene on the wafer to the copied drawing [2].

3.10.1. SCREEN: 68230_2.1-2.3_3.x.mp4 42:25-42:38 **TXT: Rotate sample by desired twist angle .**

3.10.2. SCOPE/SCREEN: 68230_2.1-2.3_3.x.mp4 43:12-43:22.

3.11. Focus on the top hexagonal boron nitride flake on the glass slide and align it to the drawing [1-TXT].

3.11.1. SCOPE/SCREEN: 68230_2.1-2.3_3.x.mp4 43:28-43:50 . **TXT: Examine quality of top stack under microscope then repeat steps with remaining graphene**

3.12. ~~After aligning the flakes to the drawings, repeat the demonstrated steps to pick up the remaining graphene [1]. Once picked up, examine the quality of top stack under microscope [2].~~

NOTE: Step is a repetition of demonstrated procedure. It has been converted to on-screen text

~~3.12.1. SCOPE/SCREEN: 68230_2.1-2.3_3.x.mp4 44:42-1:12:03.~~

~~3.12.2. SCOPE/SCREEN: 68230_2.1-2.3_3.x.mp4 .~~

4. Encapsulation of the Graphene Moiré Superlattice

4.1. Place the wafer with the pre-cleaned bubble-free bottom gate on the sample stage [1]. Outline the boundary of the bottom gate using Inkscape [2]. Align the drawing of the top stack to that of the bottom gate to place the twisted bilayer graphene on the graphite finger [3].

4.1.1. Talent positioning the wafer with bottom gate on the stage.

4.1.2. SCREEN: 68230_4.x.mp4 00:18-00:40, 01:12-01:17

Video Editor: Please speed up the video .

Added shot SCREEN: 68230_4.x.mp4

01:32-01:50

4.2. Set the sample stage temperature to 160 degrees Celsius to tear off the polycarbonate film after encapsulation [1]. Then load the glass slide with the top stack and move it to the pre-engage position [2]. Roughly align both the bottom gate and the top stack to the drawings [3].

4.2.1. Talent setting the stage heater to 160 degrees Celsius.

4.2.2. Talent loading the glass slide with the top stack and moving it to the pre-engage position.

4.2.3. SCREEN: 68230_4.x.mp4 03:41-03:56, 04:02-04:12 .

4.3. Focus on the bottom gate, then slightly raise the focal plane [1-TXT].

4.3.1. SCREEN: 68230_4.x.mp4 04:26-04:43 TXT: Engage slide downward at 0.1 mm/s until top stack becomes visible

4.4. Identify another middle focal plane between the bottom gate and top stack [1]. Then set the speed to 5 micrometers per second and engage the stamp until the top stack is visible [2]. Align both to the drawings accurately [3-TXT].

4.4.1. SCREEN: 68230_4.x.mp4 05:03-05:08 .

4.4.2. SCREEN: 68230_4.x.mp4 05:09-05:49
Video Editor: Please speed up the video .

SCREEN: 68230_4.x.mp4 05:50-06:08 . TXT: Repeat until bottom gate and top stack are almost in the same focal plane, and the sample stage temperature > 150 °C

4.5. When the sample stage is above 150 degrees Celsius, engage the stamp at 5 micrometers per second until the wave front is close to the bottom gate, then reduce the speed to 0.5 micrometers per second [1].

4.5.1. SCREEN: 68230_4.x.mp4 08:09-08:21.

4.6. Slowly engage the top stack onto the bottom gate [1]. Once the wavefront passes the top stack, increase the speed back to 5 micrometers per second to fully engage the stamp onto the wafer [2-TXT]. Then disengage the stamp at 5 micrometers per second [3-TXT].

4.6.1. SCREEN: 68230_4.x.mp4 08:43-08:55.

- 4.6.2. SCREEN: 68230_4.x.mp4 09:07-09:40 **TXT: Wait for 1 min to soften PC film**
- 4.6.3. SCREEN: 68230_4.x.mp4 10:53-11:09. **TXT: The retracting wavefront denotes the detachment between the PC film and PDMS stamp**

- 4.7. After fully separating the stamp from the wafer, lift the stamp upward for 3 seconds at 5 micrometers per second [1]. Begin moving it in the X-Y directions to tear off the polycarbonate film [2].
 - 4.7.1. SCREEN: 68230_4.x.mp4 11:11-11:16
 - 4.7.2. SCREEN: 68230_4.x.mp4 11:23-11:48.

- 4.8. Fully disengage the glass slide at 1 millimeter per second and remove it from the socket [1]. Switch off the stage heater and activate the water-cooling system [2].
 - 4.8.1. Talent lifting and removing glass slide.
 - 4.8.2. Talent switching off heater and activating cooling.

- 4.9. Once the temperature drops to room temperature, remove the wafer and inspect under an optical microscope [1].
 - 4.9.1. Talent transferring the wafer to a microscope.

Results

5. Results

- 5.1. Four-fold degenerate Landau fans emerged from both the charge neutrality point and superlattice gaps in the high-quality magic-angle twisted bilayer graphene device [1], breaking down into two-fold or one-fold degeneracies at higher magnetic fields [2].
 - 5.1.1. LAB MEDIA: Figure 8A. *Video editor: Highlight the symmetrical red-and-blue striped patterns radiating between 0 to 3*
 - 5.1.2. LAB MEDIA: Figure 8A. *Video editor: please highlight the area of the graph at 3 and 4*
- 5.2. Symmetry-broken two-fold degenerate Landau fans were identified at half-filling states indicating correlated insulating behavior [1]. The superconducting dome was located underneath the Landau fan around the half-filling state [2].
 - 5.2.1. LAB MEDIA: Figure 8A. *Video editor: please highlight the area at ± 2*
 - 5.2.2. LAB MEDIA: Figure 8A. *Video editor: please highlight the low-field area at -2*
- 5.3. Temperature-dependent resistance measurements revealed two superconducting domes at both sides of the half-filling state with the maximum critical temperature reaching approximately 1.7 Kelvin [1].
 - 5.3.1. LAB MEDIA: Figure 8B. *Video editor: please highlight the dotted area*
- 5.4. Among 14 measured devices, the optimal critical temperature peaked at a twist angle near 1.08 degrees, consistent with theoretical predictions [1].
 - 5.4.1. LAB MEDIA: Figure 8C. *Video editor: Focus on the tallest data point in the plot*
- 5.5. Disordered devices near the magic angle exhibited significantly reduced critical temperatures compared to high-quality devices [1], as seen in their resistance versus temperature profiles [2].
 - 5.5.1. LAB MEDIA: Figure 8C. *Video editor: Please highlight the two green data points*
 - 5.5.2. LAB MEDIA: Figure 8D. *Video editor: Please highlight the orange and pink curves*

1. Pronunciation Guide:

Moiré

- Pronunciation link: <https://www.merriam-webster.com/dictionary/moire>
- IPA: /mwɑː'reɪ/
- Phonetic Spelling: mwah-ray

2. **Superlattice**

- Pronunciation link: <https://www.howtopronounce.com/superlattice>
- IPA: /ˌsuː.pəˈlæt.ɪs/
- Phonetic Spelling: soo-per-lat-iss

3. **Graphene**

- Pronunciation link: <https://www.merriam-webster.com/dictionary/graphene>
- IPA: /ˈgræ.fi.n/
- Phonetic Spelling: gra-feen

4. **Heterostructure**

- Pronunciation link: <https://www.howtopronounce.com/heterostructure>
- IPA: /ˈhɛt̬.ə.roʊˌstrʌk.tʃə/
- Phonetic Spelling: heh-tuh-roh-struhk-chur

5. **Twistronics**

- Pronunciation link: <https://www.howtopronounce.com/twistronics>
- IPA: /twɪsˈtrɑː.nɪks/
- Phonetic Spelling: twis-trah-niks

6. **Hexagonal Boron Nitride**

- Pronunciation links:
 - Hexagonal: <https://www.merriam-webster.com/dictionary/hexagonal>
 - Boron: <https://www.merriam-webster.com/dictionary/boron>
 - Nitride: <https://www.merriam-webster.com/dictionary/nitride>
- IPA: /hɛkˈsæɡənəl ˈbɔːrən ˈnaɪˌtraɪd/
- Phonetic Spelling: hek-sag-uh-nuhl bore-on nye-tride

7. **Polydimethylsiloxane (PDMS)**

- Pronunciation link: <https://www.howtopronounce.com/polydimethylsiloxane>
- IPA: /ˌpɑː.li.daɪ.mɛ.θəlˈsɪ.lə:k.seɪn/
- Phonetic Spelling: pah-lee-dye-meth-uhl-sih-lock-sayn

8. **Polycarbonate**

- Pronunciation link: <https://www.merriam-webster.com/dictionary/polycarbonate>
- IPA: /ˌpɑː.liˈkɑːr.bə.neɪt/
- Phonetic Spelling: pah-lee-kar-buh-nayt

9. **Landau**

- Pronunciation link: <https://www.howtopronounce.com/landau>
- IPA: /ˈlændəʊ/
- Phonetic Spelling: lan-dow

10. **Inkscape**

- Pronunciation link: <https://www.howtopronounce.com/inkscape>

- IPA: /'ɪŋk.skeɪp/
- Phonetic Spelling: ing-skayp

11. Degenerate

- Pronunciation link: <https://www.merriam-webster.com/dictionary/degenerate>
- IPA: /dɪ'dʒenərət/
- Phonetic Spelling: dih-jen-uh-ruht

12. Heterostrain

- Pronunciation link: No confirmed link found
- IPA: /'hɛt̬.ə.roʊ.streɪn/
- Phonetic Spelling: heh-tuh-roh-strayn

13. Nanofabrication

- Pronunciation link: <https://www.howtopronounce.com/nanofabrication>
- IPA: /,næn.oʊ.fæb.rɪ'keɪ.jən/
- Phonetic Spelling: nan-oh-fab-ri-kay-shun

14. Cryogenic

- Pronunciation link: <https://www.merriam-webster.com/dictionary/cryogenic>
- IPA: /,kraɪ.oʊ'dʒen.ɪk/
- Phonetic Spelling: kry-oh-jen-ik

15. Superconductivity

- Pronunciation link: <https://www.merriam-webster.com/dictionary/superconductivity>
- IPA: /,suː.pə.kən'dʌk'tɪ.və.ti/
- Phonetic Spelling: soo-per-kuhn-duhk-tiv-uh-tee