Submission ID #: 61374
Scriptwriter Name: Bridget Colvin
Project Page Link: https://www.jove.com/account/file-uploader?src=18719118

Title: In Situ Grazing Incidence Small Angle X-Ray Scattering on Roll-To-Roll Coating of Organic Solar Cells with Laboratory X-Ray Instrumentation

Authors and Affiliations: Michael Korning Sørensen, Moises Espindola Rodriguez, Marcial Fernández Castro, Ashwin Nambi, Luise Theil Kuhn, and Jens Wenzel Andreasen

Department of Energy Conversion and Storage, Technical University of Denmark

Corresponding Author:
Jens W. Andreasen
jewa@dtu.dk

Co-authors:
mkso@dtu.dk
mesro@dtu.dk
marfca@dtu.dk
s181252@student.dtu.dk
luku@dtu.dk

Author Questionnaire

1. Microscopy: Does your protocol involve video microscopy, such as filming a complex dissection or microinjection technique? **N**

2. Software: Does the part of your protocol being filmed demonstrate software usage? **Y**

Videographer: Screen captures not provided, please film

3. Filming location: Will the filming need to take place in multiple locations (greater than walking distance)? **N**

Protocol Length
Number of Shots: **49**

Introduction

1. Introductory Interview Statements

REQUIRED:

1.1. **Michael Korning Sørensen**: This protocol enables us to perform in situ studies of the photoactive layer of organic solar cells, which would otherwise only be possible at synchrotrons, in our home laboratory **[1]**.

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

REQUIRED:

1.2. **Jens Wenzel Andreasen** or **Moises Espindola Rodriguez**: Using in situ grazing incidence small angle x-ray scattering, we can study the structure development of the donor and acceptor intermixed state under conditions similar to actual large-scale coating conditions **[1]**.

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

Introduction of Demonstrator on Camera

1.3. **Marcial Fernández Castro** : The roll-to-roll slot-die coating procedure is going to be performed outside of the GISAXS set up for the purpose of this demonstration. Later it will be installed in the to show the full experiment **[1][2]**.

1.3.1. INTERVIEW: Author saying the above
1.3.2. The named demonstrator(s) looks up from workbench or desk or microscope and acknowledges the camera

Protocol

2. Roll-to-Roll Slot Die Coating

2.1. For slot die coating, wind 18 meters of PET (P-E-T) substrate foil onto a feeder roll **[1-TXT]** and attach the free end of the substrate to the winder roll **[2]**.

2.1.1. WIDE: Talent winding foil onto feeder roll **TEXT: PET: polyethylene terephthalate**

2.1.2. Talent attaching substrate to winder roll

2.2. Run the foil 0.2 meters to tighten the substrate **[1]** and set the first hot plate of the roll-to-roll setup to 60 degrees Celsius **[2]** and the second hot plate to 80 degrees Celsius to ensure that the film is dried when wound onto the winder roll **[3]**.

2.2.1. Motor being started/foil being run

2.2.2. Talent setting hot plate temperature

2.2.3. Talent setting timer, with both hot plates visible in frame

2.3. When the hot plates have stabilized for about 15 minutes, mount a 3-milliliter syringe loaded with 2.2. milliliters of roll-to-roll coating ink onto a syringe pump **[1-TXT]** and attach a tube from the syringe to the slot-die coating head **[2]**.

2.3.1. Talent loading syringe onto pump *Videographer: Important step* **TEXT: See text for roll-to-roll coating ink preparation details**

2.3.2. Talent attaching tube to head *Videographer: Important step*

2.4. Adjust the horizontal translation stage so the coating head is positioned close to the end of the first hot plate **[1]** and place the meniscus guide approximately 5 millimeters above the substrate **[2]**.

2.4.1. Talent adjusting stage

2.4.2. Guide being placed 5 mm above substrate

2.5. Then set the syringe pump to a 0.08 milliliter/minute flow rate and a 12.7-millimeter syringe diameter **[1]**.

2.5.1. Talent setting pump

2.6. To control the thickness of the active layer, adjust the flow rate and the speed of the moving substrate according to the formula, in which *w* is the width of the film and *p* is the density of the materials in the ink **[1-TXT]**.

2.6.1. BLACK TEXT ON WHITE BACKGROUND: $d = \frac{cf}{\rho v w}$ *Video Editor: please emphasize "d" with thickness of the active layer, "f" with flow rate and "v" with speed of the moving substrate, "w" with width of the film, and "p" with density of the materials in the ink*

2.7. When the pump parameters have been set, manually dispense the ink from the syringe and through the hose **[1]**, stopping 1 centimeter before the ink reaches the coating head **[2]**.

2.7.1. Ink being pressed through syringe and hose *Videographer: Important step; Videographer/Video Editor: shot will be used again*

2.7.2. Ink stopping 1 cm before coating head *Videographer: Important step*

2.8. With the meniscus guide is 5 millimeters above the substrate, start the syringe pump **[1]**.

2.8.1. Talent starting pump

2.9. When a droplet has wet the entire width of the meniscus-guide **[1]**, immediately lower the coating head to wet the substrate with the ink **[2]** and raise the meniscus guide to the coating position 2 millimeters above the substrate **[3]**.

2.9.1. Shot of guide wet with droplet/droplet wetting guide NOTE: 2.9.1 – 2.11.2 all in one shot, 2.10.1 and 2.11.1 were deleted *Videographer: Important step*

2.9.2. Coating head being lowered/substrate being wet *Videographer: Important step*

2.9.3. Guide being raised 2 mm above substrate *Videographer: Important step*

2.10. Then start the motor that winds up the substrate ~~[1]~~ and start coating with the ink **[2]**.

~~2.10.1. Talent starting motor~~

2.10.2. Substrate being coated *Videographer: Important step*

2.11. To stop the coating, stop the pump and the moving substrate ~~[1]~~ and raise the coating head approximately 20 millimeters above the substrate **[2-TXT]**.

~~2.11.1. Talent stopping pump and/or substrate~~

2.11.2. Talent raising coating head **TEXT: Clean head and hose with tetrahydrofuran**

3. In Situ Roll-to-Roll Grazing Incidence Small Angle X-Ray Scattering (GISAXS)

3.1. To perform a GISAXS (djee-sacks) experiment, fasten the mini roll-to-roll coater to the goniometer **[1]** and mount the goniometer with the roll-to-roll coater on the optical bench at the sample position **[2]**.

3.1.1. WIDE: Talent fastening mini roll-to-roll coater to goniometer

3.1.2. Talent mounting goniometer and coater to bench

3.2. Fasten the three motor cables and the goniometer stage to the bench **[1]** and position the flight tube as close to the mini roll-to-roll coater as possible **[2]**.

3.2.1. Cable(s) and/or stage being fastened

3.2.2. Tube being placed close to coater

3.3. Align the sample with the coater **[1]** and coat 10 centimeters of the ink onto the sample **[2]**.

3.3.1. Sample being aligned

3.3.2. Ink being coated

3.4. Then roll the film into the beam **[1]**.

~~3.4.1. Film being rolled~~ NOTE: Use 3.3.2. here

3.5. To align the sample parallel to the beam, scan the summed intensity of the direct beam as a function of the vertical sample position and incidence angle **[1 a and b]** and use the formula to calculate the angle reflected beam on the detector **[2]** to allow the sample to be aligned to a 0.2-degree incidence angle **[3]**.

3.5.1. LAB MEDIA: 3.5.1.a.jpg. Talent scanning summed intensity the vertical sample position NOTE: Shot split in 2

LAB MEDIA: 3.5.1 b.jpg : Talent scanning the incidence angle

3.5.2. BLACK TEXT WHITE BACKGROUND:

$$\alpha_i = \frac{1}{2} arctan\left(\frac{RB[cm] - DB[cm]}{SDD\ [cm]}\right)$$

3.5.3. SCREEN: To be provided by Authors: Sample being aligned NOTE: 3.5.3 and 3.6.1 still not uploaded at postshoot stage

3.6. To optimize the intensity in the reflected beam, scan the height of the sample position using an incidence angle of 0.2 degrees **[1]**.

3.6.1. SCREEN: To be provided by Authors: Beam intensity being optimized

3.7. Install the beam-stop just before the detector to extend the lifetime of the detector **[1]** and use a circular beam stop for the direct beam **[2]**.

3.7.1. Talent installing beam-stop

~~3.7.2. Talent placing circular beam stop~~ NOTE: Action is in the previous shot

3.8. Place a point-suction to remove all of the gases from the evaporating solvents **[2]**.

~~3.8.1. Rectangular beam stop being placed,~~

3.8.2. Point suction being placed

3.9. Mount a 3-milliliter syringe loaded with 2.2 milliliters of ink onto the syringe pump **[2]**.

~~3.9.1. Talent fastening point suction~~

3.9.2. Talent mounting syringe

3.10. **[1]**

~~3.10.1. Use 2.7.1. Ink being dispensed through tubing~~

3.11. Place the coating head 120 millimeters from the x-ray beam along the moving direction of the foil to ensure a drying time of 12 seconds **[1-TXT]**. NOTE: Authors deleted shot but not VO here.

~~3.11.1. Coating head being placed 120 mm from beam **TEXT: For 3-s drying, place coating head 30 mm from x-ray beam**~~

3.12. When the coating head is in place, position the meniscus-guide 5 millimeters above the substrate **[1]** and start the syringe pump **[2]**.

3.12.1. Guide being positioned

3.12.2. Talent starting pump

3.13. When the entire width of the meniscus-guide has been wet, immediately lower the coating head to wet the substrate with ink **[1]** before raising the meniscus guide to the coating position 2 millimeters above the substrate **[2]**.

3.13.1. Shot of wet guide, then head being lowered NOTE: 3.13.1. – 3.14.1. in one shot

3.13.2. Guide being raised

3.14. When the guide is in place, start the motor that winds up the substrate to begin coating the ink **[1]**.

3.14.1. Substrate winding up/being coated

3.15. Use a camera to monitor the quality of the coated film **[1]**, looking for de-wetting effects of the film on the substrate and meniscus misalignments **[2-TXT]**.

3.15.1. LAB MEDIA: 3.15.2 Shot of film.mp4. Talent monitoring film with camera

3.15.2. Shot of film *Videographer: Important step; Video Editor: please emphasize de-wetting effect(s) and/or meniscus misalignment(s) when mentioned* **TEXT: Stop measurements and redo experiment as necessary**

3.16. **[1]**, **[2]**,**[3]**.

~~3.16.1. Talent stopping pump~~

~~3.16.2. Talent raising coating head~~

~~3.16.3. Talent unwinding foil~~

Protocol Script Questions

A. Which steps from the protocol are the most important for viewers to see?
2.3., 2.7., 2.9., 2.10., 3.15.

B. What is the single most difficult aspect of this procedure and what do you do to ensure success?
3.5., Align the sample is one of the most difficult steps, to succeed we used the formula in 3.5.2 and scan the height of the sample as in 3.6.

Results

4. Results: Representative Optoelectronic Organic Solar Cell Characteristics

4.1. Based on the fitting, it can be deduced that the Teubner-Strey model **[1]** successfully describes the data for P3HT:EH-IDTBR (P-three-H-T-O-eye-D-T-B-R) **[2]** and P3HT-O-IDTBR for both 12 and 3 seconds of drying **[3]**.

4.1.1. LAB MEDIA: Figure 10
4.1.2. LAB MEDIA: Figure 10 *Video Editor: please emphasize top and then bottom black data line (or both at the same time)*
4.1.3. LAB MEDIA: Figure 10 *Video Editor: please emphasize top and then bottom blue data line (or both at the same time)*

4.2. In these Tables, the characteristic length scales **[1]** based on the Teubner-Strey model and their corresponding errors can be observed **[2]**.

4.2.1. LAB MEDIA: Tables 3 and 4 *Video Editor: please emphasize Table 3*
4.2.2. LAB MEDIA: Tables 3 and 4 *Video Editor: please emphasize Table 4*

4.3. For all four fits, the domain size and correlation length **[1]** for the highest scattering vector are close to the same value **[2]**.

4.3.1. LAB MEDIA: Tables 3 and 4
4.3.2. LAB MEDIA: Tables 3 and 4 *Video Editor: please emphasize first two data columns of Table 3*

4.4. For the large structures, there is a clear tendency for the structures to become larger as they dry **[1]**.

4.4.1. LAB MEDIA: Tables 3 and 4 *Video Editor: please emphasize 562 and 489 data cells in Table 3*

4.5. Noticeably, the correlation length is more pronounced after 3 seconds of drying **[1]** than after 12 seconds of drying for P3HT-O-IDTBR **[2]**, while for P3HT-EH-IDTBR, the correlation length is more pronounced after 12 seconds of drying **[3]** than after 3 seconds of drying **[4]**.

4.5.1. LAB MEDIA: Table 3 *Video Editor: please emphasize 30 data cell in Table 3*
4.5.2. LAB MEDIA: Table 3 *Video Editor: please emphasize 34 data cell in Table 3*
4.5.3. LAB MEDIA: Table 3 *Video Editor: please emphasize top 41 data cell in Table 3*

4.5.4. LAB MEDIA: Table 3 *Video Editor: please emphasize bottom 41 data cell in Table 3*

4.6. For the large structures, there is a clear tendency for the structures to become larger as they dry **[1]**.

4.6.1. LAB MEDIA: Table 3 *Video Editor: please emphasize d3 and zeta3 data columns*

Conclusion

5. Conclusion Interview Statements

5.1. **Michael Korning Sørensen**: With this experiment, we have shown that the drying process of EH- and O-IDTBR differs on the nano-scale. [**1**].

5.1.1. INTERVIEW: Named talent says the statement above in an interview-style shot, looking slightly off-camera (2.6., 3.5.)

5.2. **Marcial Fernandez Castro**: This protocol can be used to study new non-fullerene polymers and to identify the coating conditions that can help us to improve the power conversion efficiency of our flexible solar cells **[1]**.

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

5.3. **Jens Wenzel Andreasen** or **Moises Espindola Rodriguez**: In situ X-ray scattering may become an indispensable tool for optimizing industrial processes, from the semiconductor to the biomedical industries **[1]**.

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