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Deposition of Organic Crystals in Large Scale by Bar Coating Method

--Manuscript Draft--

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Abstract:	High-quality organic semiconductor layers are critical for achieving high-performance organic transistors. Other than the quality of the active layer, the compatibility of the fabrication method for mass production and low fabrication cost are also important investigation directions. In the current study, a solution processed bar coating method is investigated for its availability in depositing small molecule organic crystals by a homemade experimental setup. The bar coating approach shows advantages of saving materials, suitability for large-scale deposition, and availability to guide the crystal orientations of the deposited organic layers. It also has particular advantages over other shearing methods that the confinement of solution is low near the meniscus area by using the bar, which assists in forming larger crystal domain size. The coated 2,7-Dioctyl[1]benzothieno[3,2-b][1]benzothiophene (C8-BTBT) layers are highly crystalline films with a thickness of a few monolayers and single crystal domains can achieve millimeter-scale with uniform orientations. The structural and electrical properties of the C8-BTBT layers are studied by atomic force microscopy (AFM), polarized microscopy, X-ray diffraction (XRD) and electronic measurements. The bar coating method can be further developed into manufacturing platform to deposit high-performance organic crystals for industrial applications.
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Cover Letter

The University of Hong Kong
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27^{8h} March 2017

Dear Editor,

We are submitting a manuscript entitled "Deposition of Organic Crystals in Large Scale by Bar Coating Method" for your kind consideration for publication in *Journal of Visualized Experiments (JOVE).*

High-quality organic semiconductor layers are critical for achieving high-performance organic transistors. Other than the quality of the active layer, the compatibility of the fabrication method for mass production and low fabrication cost are also important investigation directions. In the current study, a solution processed bar coating method is investigated for its availability in depositing small molecule organic crystals by a homemade experimental setup. The bar coating approach shows advantages of saving materials, suitability for large-scale deposition, and availability to guide the crystal orientations of the deposited organic layers. It also has particular advantages over other shearing methods that the confinement of solution is low near the meniscus area by using the bar, which assists in forming larger crystal domain size. The coated 2,7-Dioctyl[1]benzothieno[3,2-b][1]benzothiophene (C8-BTBT) layers are highly crystalline films with a thickness of a few monolayers and single crystal domains can achieve millimeter-scale with uniform orientations. The structural and electrical properties of the C8-BTBT layers are studied by atomic force microscopy (AFM), polarized microscopy, X-ray diffraction (XRD) and electronic measurements. The bar coating method can be further developed into manufacturing platform to deposit high-performance organic crystals for industrial applications. Our investigation about the bar coating deposition method provides the fundamental knowledge to fabricate high performance organic field effect transistors over a large area.

Thanks for your review and consideration.

Sincerely,

Paddy K. L. Chan
Assistant Professor
Mechanical Engineering
The University of Hong Kong

TITLE:

Deposition of Organic Crystals in Large Scale by Bar Coating Method

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KEYWORDS:

Organic semiconductor, solution process, single crystal, bar coating, large area, field-effect transistor

SHORT ABSTRACT:

A low-cost solution shearing method, bar coating, is used to deposit highly crystallized and uniformly oriented organic crystals with a domain size on the millimeter scale. The organic semiconductor films show very good performance as active layers in organic field-effect transistors (OFETs).

LONG ABSTRACT:

High-quality organic semiconductor layers are critical for achieving high-performance organic transistors. Other than the quality of the active layer, the compatibility of the fabrication method for mass production and low fabrication cost are also important research directions. In the current study, a solution processed bar coating method is investigated for its ability to deposit small molecule organic crystals by a homemade experimental setup. The bar coating approach shows advantages in saving material usage, suitability for large-scale deposition, and the ability to guide the crystal orientations of the deposited organic layers. It also has particular advantages over other shearing methods such as the low confinement of solution near the meniscus area by using the bar, which assists in forming a larger crystal domain size. The coated 2,7-dioctyl[1]benzothieno[3,2-b][1]benzothiophene (C8-BTBT) layers are highly crystalline films with a thickness of a few monolayers and single crystal domains can achieve millimeter-scales with uniform orientations. The structural and electrical properties of the C8-BTBT layers are studied by atomic force microscopy (AFM), polarized microscopy, X-ray diffraction (XRD) and electronic measurements. The bar coating method can be further developed into manufacturing platform to deposit high-performance organic crystals for industrial applications.

INTRODUCTION:

Organic electronics have rapidly developed in the last decade. As an essential layer of these devices, the properties of the organic semiconductor active layers have experienced significant improvements due to the emergence of novel deposition technologies and a better understanding of their molecular structures[1-4]. High mobility organic crystals have been achieved by a variety of low-cost solution methods including inkjet printing[5], spin coating[6], droplet pinning[7] and blade shearing[8,9] *etc*. Among these solution processes, shearing methods have great potential for applications in industrial manufacturing because only small amounts of materials are needed for deposition. Besides, the fast deposition rate and the compatibility with roll-to-roll processes for large area deposition are also the advantages of the solution shearing approach. The guided meniscus line in the solution shearing can attain good uniformity and regulate the orientations of the deposited crystals[10,11]. Compared with other shearing methods, bar coating has less confinement for the solutions near the meniscus line, which favors the crystal deposition[12]. Previously bar coating has been used to deposit a smooth and uniform layer of polymer or oxide dielectrics, but the deposited layers are usually in an amorphous form[13-17]. Here, we optimized the deposition parameters and modified the meniscus of this method to deposit small molecule organic crystals with good crystallinity and aligned orientations.

As a representative small molecule semiconductor, 2,7-dioctyl[1]benzothieno[3,2-b][1]benzothiophene (C8-BTBT) has attracted worldwide investigations because of its high intrinsic mobility and good solubility in organic solvents[18,19]. The unit cell of C8-BTBT single crystal is monoclinic with herringbone-packed in-plane structure, and the self-assemble of this material can be easily achieved from solutions or vapor phase. Here, we use the bar coating method to deposit highly crystallized C8-BTBT thin layers with large domain size and guided orientations from solutions.

PROTOCOL:

1. Substrate Cleaning and Self-Assembled Monolayer (SAM) Treatment

1.1. Put the pre-cut silicon substrates (1.5 cm x 2 cm) with 300 nm silicon dioxide (SiO_2) into a glass beaker with substrate holders and clean by ultra-sonication in detergent solution, DI water, acetone and 2-propanol separately for 10 min each.

1.2. Heat up the substrates in 2-propanol on a 200 °C hot plate for around 10 min until the solvent boils. Then dry the substrates with nitrogen gas.

1.3. Put the substrates into oxygen plasma for one h.

1.4. Immediately immerse the cleaned substrates into the phenyl trichlorosilane (PTS) solution (0.2 %wt in toluene), and seal the glass vial.

1.5. Put the vial onto a 90 °C hot plate for 1.5 h.

1.6. Wait for 0.5 h until the vial cools down, take out the samples and clean in chloroform and 2-propanol by sonication for two times (5 min each time). Then blow dry with nitrogen gas.

2. Organic Crystal Deposition by Bar Coating Method

2.1. Treatment of metal bar surface

2.1.1. Clean the steel bar with a smooth surface by the similar procedure as in 1.1-1.3.

2.1.2. Immerse the bar into 1H,1H,2H,2H-perfluorooctanephosphonic acid (PFPA) solution (1 mM in 2-propanol) at 70 °C overnight in ambient air.

Note: The PFPA with fluorine atoms as terminal groups can reduce the surface energy and adhesion force for organic molecules, thus reduce the organic residues on the bar during shearing.

2.1.3. Clean with 2-propanol by sonication for 10 min. Then dry it with 99.99% pure nitrogen gas.

2.2. Growth of C8-BTBT crystals

2.2.1. Prepare organic semiconductor solution by dissolving 5 mg C8-BTBT into 0.5 mL m-xylene solvent.

2.2.2. Turn on the Peltier heater to preheat the metal stage to 60 °C.

2.2.3. Stick the substrates onto the metal stage and wait for around 5 min until the surface temperatures reach equilibrium.

2.2.4. Tune the height of the bar to the 70 µm higher than the substrate surface.

2.2.5. Drop 18 µL of C8-BTBT solution into the gap between the bar and substrate. The solution will be pinned between the bar and substrate by capillary force.

2.2.6. Move the bottom stage horizontally towards certain direction at a constant speed 200 µm/s by a movement controller.

2.2.7. Collect the samples after shearing. Put them into the vacuum chamber for more than 5 h to get rid of the residual solvents.

Note: The experiment can be paused here.

3. Organic Field-Effect Transistors (OFETs) Fabrication

3.1. Put the Si/SiO_2 substrate with coated crystals into the evaporation chamber.

3.2. Pump down the system to a vacuum of 2 x 10^{-6} Torr and then deposit the charge injection layer 2,3,5,6-tetrafluoro-7,7,8,8-tetracyanoquinodimethane (F4-TCNQ) at 1 Å/s to 3 nm and silver (Ag) electrode at 0.2 Å/s to 50 nm through the same shadow mask with patterns.

Note: The F4-TCNQ layer can help the injection of charge carriers[20] and work as a buffer layer to reduce the damage of organic semiconductors during metal deposition, the slow rate of Ag deposition can also reduce the damage of crystals. All these parameters help to minimize the contact resistances.

4. Characterizations

4.1. Polarized microscopy

4.1.1. View the crystals under a polarized microscope with a rotation stage. The crystal size and crystallinity can be indicated.

4.2. Atomic-force microscopy (AFM)

4.2.1. Measure the thickness and surface morphology by AFM system with tapping mode.

Note: For thickness measurement, the crystals are scratched by a sharp needle, the substrate surface is exposed after scratching, and the height difference can be measured.

4.3. X-ray diffraction (XRD)

4.3.1. Put the crystals into the XRD system (with the highest power of 9 kW) and quantify the lattice structure and crystal orientation by using out-of-plane and in-plane XRD mode.

4.3.2. For the out-of-plane test, use $2\vartheta/\omega$ mode.

4.3.3. For the in-plane test, use $2\vartheta/\chi$ mode and set the incident angle of X-ray to be 0.15°. Place the sample parallel or perpendicular with the incident X-ray.

4.4. Electronic measurement of OFETs

4.4.1. Put the fabricated OFET devices into the glove box, connect the source electrode to ground, the drain and gate electrodes with two channels of a source meter.

4.4.2. Scan the transfer curves by fixing the V_{DS} to -80 V and V_G is set from 20 V to -

80 V. Plot the output curve by scanning the V_{DS} from 0 to -80 V under a constant V_G.

REPRESENTATIVE RESULTS:

The homemade setup for bar coating deposition is illustrated in **Figure 1**, which consists a lift stage, a smooth bar, a metal stage, a Peltier temperature controller and a translation stage. The stage can tune the height of the bar and change the gap between the bar and substrate. The metal stage was used for temperature test and the feedback was transmitted to a temperature controller. The temperature controller can control the power supply for Peltier heater and adjust the deposition temperature. The translation stage was controlled by an electronic movement controller. When C8-BTBT solution is dropped into the gap, and the substrate begins to move, the organic crystals will be deposited at the air-liquid-solid three phase contact line. Large size crystals will be grown along the shearing direction as indicated in **Figure 2**. The length of the crystals can be up to centimeter scale, and the width of the single domain can reach a few millimeters. After rotating the samples by 45°, the bright regions will become totally dark under POM, which indicates uniform crystallinity of the deposited crystals. The surface morphology and the thickness of the deposited layers are measured by AFM and the results are shown in **Figure 3**. It can be indicated from the morphology image of area 10 µm by 10 µm that a very smooth crystal surface was formed, and the root-mean-square (RMS) roughness is only 0.24 nm, quite comparable with underneath SiO_2. The thickness of the film is around 24 nm, *i.e.* eight monolayers of C8-BTBT. The crystallinity and lattice structure is further characterized by X-ray diffraction. Layer-by-layer growth of C8-BTBT is observed in coated crystals due to the multiple (00*l*) peaks in out-of-plane results as shown in **Figure 4A**. For in-plane XRD test, when the sample was put in the way as the schematic in **Figure 4B**, only (020) peak can be clearly observed, which indicates that the b-axis is dominated in the shearing direction. When the sample rotates, the intensity of (020) becomes much smaller, and other peaks begin to form. (200) Peak shows its highest value when the sample rotates 90°. Those in-plane results clearly indicate a good crystallization with uniform orientations of the formed crystals and the lattice constants are calculated to be a = 5.925 Å, b = 7.905 Å and c = 29.1 Å (comparable with the values of bulk single crystals[18]). The uniform orientation and good coverage of crystals on substrate surface also induce a very good performance and uniformity of OFETs. The OFET device structure is presented In **Figure 5A**, a charge injection layer F4-TCNQ was introduced to reduce the contact resistance, and a clear linear region can be observed in the output curve (**Figure 5B**). The field-effect mobility is calculated using transfer curve in **Figure 5C** in saturation regime by the equation:

$$\mu = \frac{2LI_{DS}}{WC_i(V_G - V_{th})^2}$$

Where L and W are channel length (250 µm) and width (500 µm) respectively. I_{DS} is the on current value at certain V_G; C_i represents the capacitance of the dielectric layer (300 nm SiO_2) and V_{th} is the threshold voltage. The mobility of the OFETs can reach as high as ten $cm^2V^{-1}s^{-1}$ with the averaged value of 8.7 $cm^2V^{-1}s^{-1}$. These mobility values are comparable with the devices based on single-crystallized C8-BTBT[21].

Figure 1. Experimental setup of bar coating method for organic crystals deposition.

Figure 2. POM images of deposited C8-BTBT crystals.
Crystals are grown along the shearing direction. The sample in right figure is the same region as in left figure after rotating by 45°.

Figure 3. Surface morphology and thickness measurement by AFM.
The upper figure shows the smooth surface of deposited C8-BTBT crystals. The lower figures are the thickness of the coated crystal layer.

Figure 4. X-ray diffraction patterns of deposited C8-BTBT crystal.
The schematics of XRD test is indicated above the line graphs. (A) Out-of-plane measurement for a layer-by-layer growth of C8-BTBT. (B) and (C) are in-plane XRD measurements, by changing the position of the sample, different peaks can be observed, which indicate good crystallinity and orientations of the crystals.

Figure 5. Electronic measurement of OFETs.
(A) The device structure of field-effect transistors, W/L = 2. (B) Output curves. (C) Transfer curve and leakage current.

DISCUSSION:

The quality of crystals deposited by the bar coating method depends on some important processing factors. Suitable surface energy and roughness of the substrates are the primary requests. The surface energy should be high enough to wet the solution and a smooth surface with fewer defects is always favorable for a large and continuous crystal layer deposition. Also, a smooth surface with fewer charge traps can favors the performance of the electronic devices. Here we treated the smooth SiO_2 surface with PTS monolayer as reported in other literatures[8,22].

During the shearing process, the deposition of the organic semiconductor is dominated by evaporation of solvent and supersaturation of organic semiconductors at the end of the meniscus (three phase contact line). Parameters including substrate temperature, shearing speed and the properties of solvent should all influence the crystals deposition. Higher temperature can help the solutions to reach supersaturation regimes and enhance the deposition rate/shearing speed. But if the temperature is too high, cracks may form inside crystals due to the differences in coefficients of thermal expansion (CTE) of the substrate and semiconductors. 60 °C is found to be suitable for our case to deposit a continuous layer without cracks. As another important parameter, the shearing speed can influence the amount of deposited semiconductor and the thickness of the semiconductor. Higher shearing speed will induce thinner layer (the speeds we used in current work are still inside the evaporation regime, which means we do not need to consider the elongation of meniscus due to the viscous force and the increase of thickness with speed in Landau-Levich regime[23]). Reduction of the layer thickness was found to have better performance of bottom gate top contact OFETs, and it is attributed to decrease of the accessary resistance between the electrode and conductive channel[6]. However, if the shearing speed is too high, voids would begin to form and the coverage of organic semiconductors drops. An optimized shearing speed of 200 µm/s was applied in this study. At last, the boiling point, volatility, surface tension and molecular weight[11] of solvent will also determine the deposition, since they are not within the scope of this manuscript, we just focus on the m-xylene as a solvent. Other physical parameters like the volume of the solvent should also affect the crystals. In our experiments, it was found that very small amount of solution dropped into the gap may reduce the meniscus area, thus reduce the amount of semiconductors deposited and form voids inside the crystals. The change of surface tension due to temperature gradient inside the solution is also found to influence the deposited crystals, and it is investigated in another paper from our group.

This bar coating method provides another way of depositing small molecule organic crystals with large grain size and uniform orientations. It can be a good supplement for current technologies and provide more options to fabricate organic electronics by low-cost solution process.

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DISCLOSURES:

The authors have nothing to disclose.

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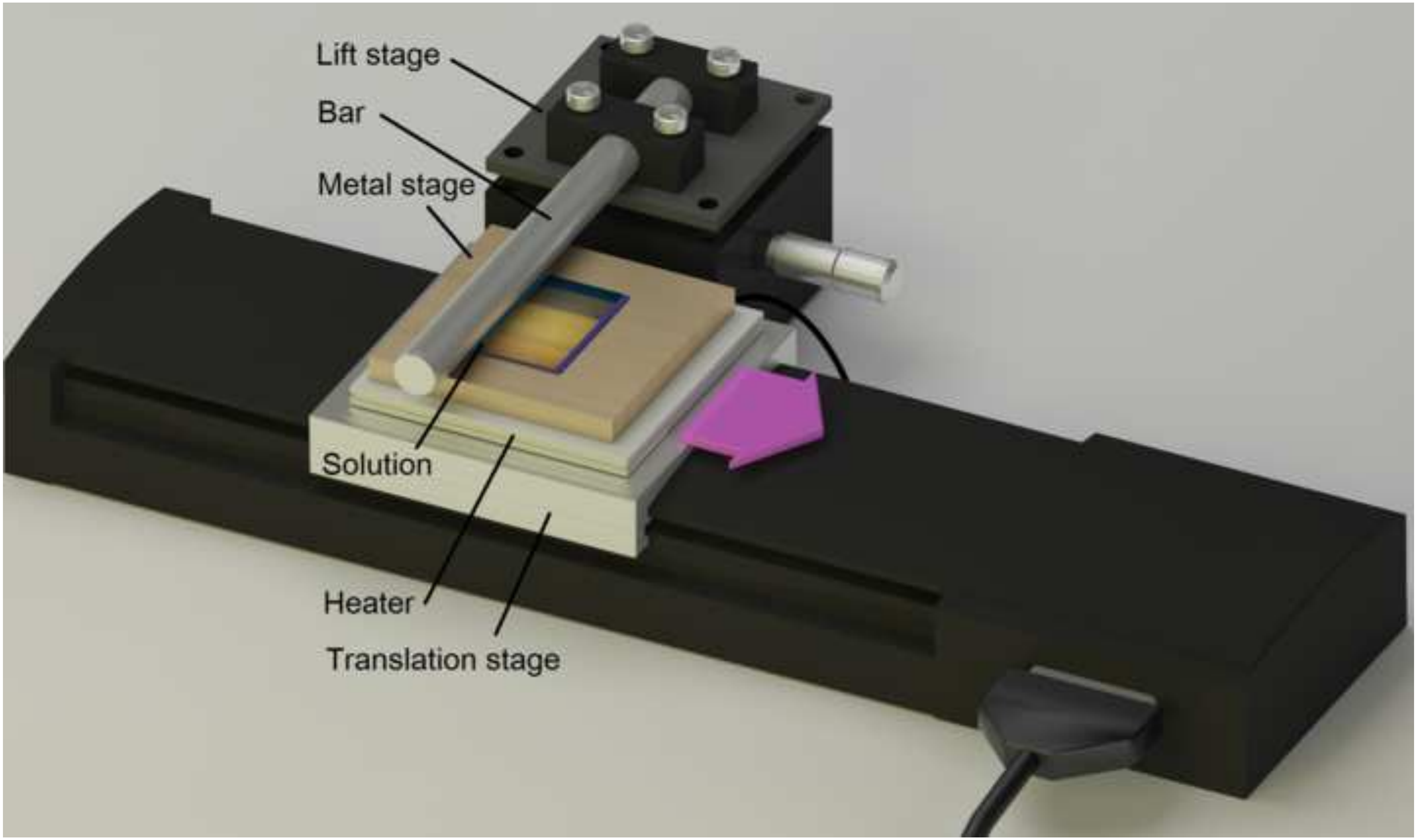
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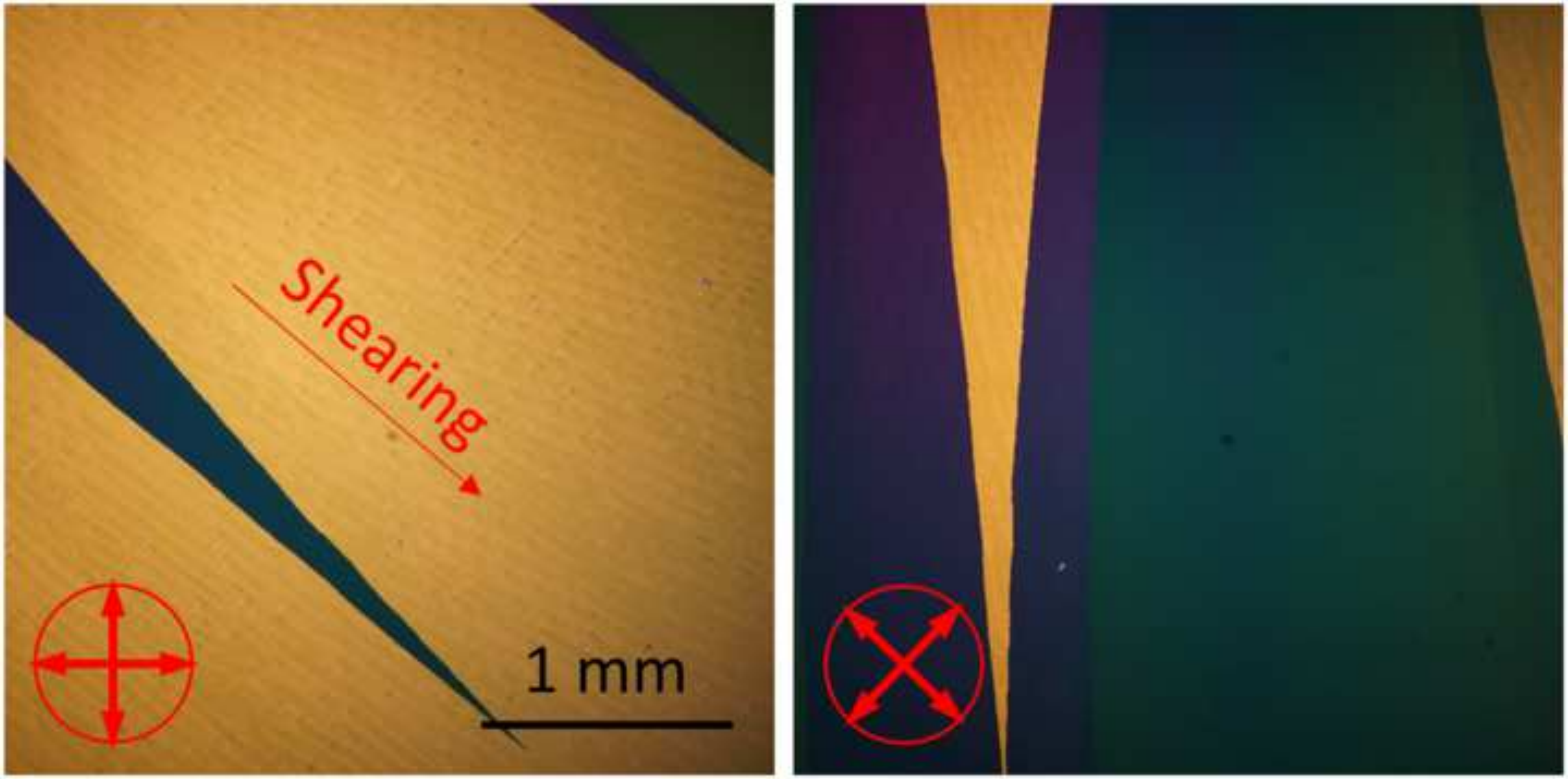
Shearing
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Figure 3

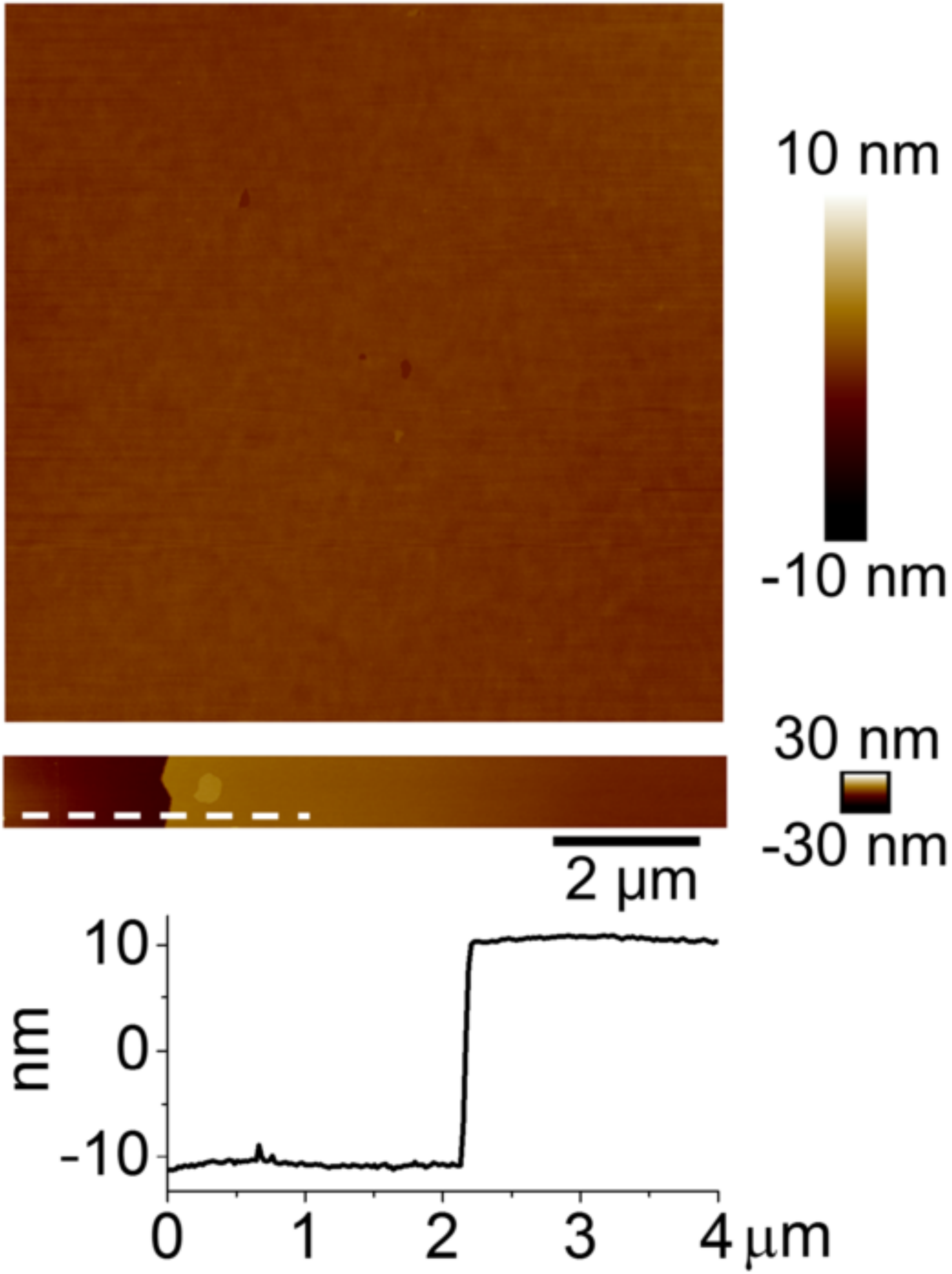
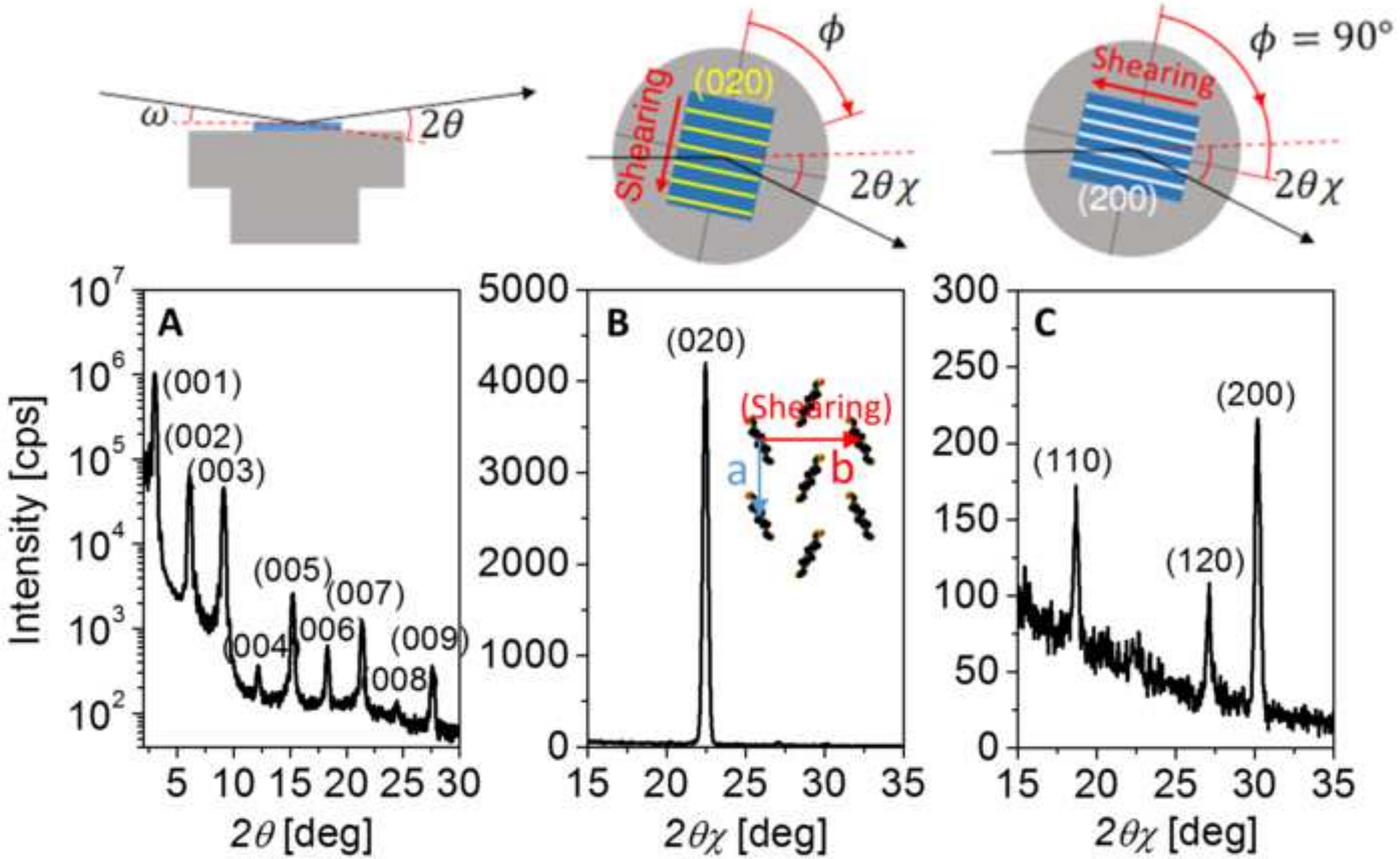


Figure 4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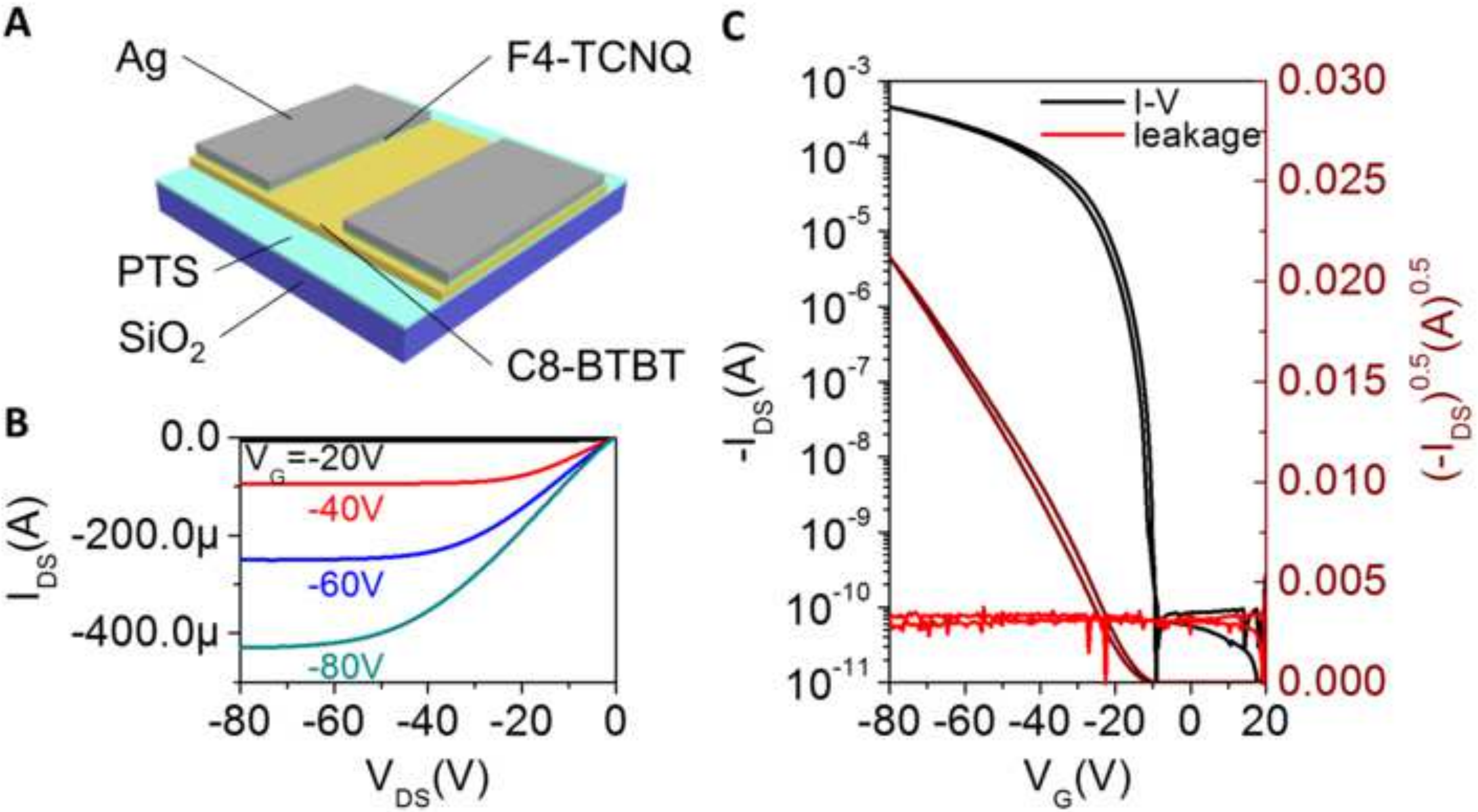
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$-I_{DS}$(A)
I-V
leakage
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0.005
0.000
$(-I_{DS})^{0.5}(A)^{0.5}$
-80
-60
-40
-20
0
20
V_G(V)

Name of Material/ Equipment	Company
2,7-Dioctyl[1]benzothieno[3,2-b][1]benzothiophene (C8-BTBT 99.5%)	Luminescence Technology Corp.
phenyl trichlorosilane (PTS 98%)	J&K Scientific
Silicon with 300 nm SiO_2	University Wafer
1H,1H,2H,2H-perfluorooctanephosphonic acid (PFPA 95%	Sigma-Aldrich
m-xylene 99%	International Laboratory USA
6-tetrafluoro-7,7,8,8-tetracyanoquinodimethane (F4-TCNQ	Luminescence Technology Corp.
Temperature Controller	Newport
Motion Controller	Newport
2636 System SourceMeter	Keithley
Eclipse LV100N polarizing	Nikon
MultiMode 8	Bruker
SmartLab X-ray Diffraction system	Rigaku

Catalog Number	Comments/Description
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580706	
D7194.01	
737461	
2110991	
LT-E208	
Model 350B	
Model ESP301	

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