

Advanced Optical Metrology

Electronics







Contents

- B Electronic Materials for Sustainable Energy Conversion and Storage
 Markus Fabich
- 8 Nanodiamond-Based Separators for Supercapacitors Realized on Paper Substrates Giuseppina Polino, Alessandro Scaramella, Valerio Manca, Elena Palmieri, Emanuela Tamburri, Silvia Orlanducci, and Francesca Brunetti
- Native Oxide Barrier Layer for Selective Electroplated Metallization of Silicon Heterojunction Solar Cells
 Thibaud Hatt, Sven Kluska, Mananchaya Yamin, Jonas Bartsch, and Markus Glatthaar
- Spray-Coated Polymer Solar Cells Based on Low-Band-Gap Donors Processed with ortho-Xylene
 Luca La Notte, Giuseppina Polino, Claudio Ciceroni,
 Francesca Brunetti, Thomas M. Brown, Aldo Di Carlo, and Andrea Reale
- 25 Scientific Publishing5 tips for writing better science papers
- 28 Scientific Publishing The Core of Your Research ArticleP. Trevorrow, G. E. Martin

Imprint

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Electronic Materials for Sustainable Energy Conversion and Storage

Markus Fabich

Given the present environmental crises there is a pressing need for sustainable methods to convert and store energy. Consequently, strenuous efforts are made to develop high-performance materials for photovoltaics, batteries, gas reservoirs, just to name a few. This chapter outlines the research that is done in Dr Manuela Schiek's group in Oldenburg, Germany, on sustainable manufacture of photovoltaics. The group has discovered how the latest technology in confocal laser scanning microscopy is enhancing both accuracy and efficiency of their research into organic semiconductors and transparent electrodes. Following this, you can find current studies on the fabrication of environmentally friendly supercapacitors (pp. 8-13), the metallization of silicon heterojunction solar cells by electroplating of copper onto a multifunctional patterned metal layer stack (pp. 14-18), and the manufacturing of spray-coated polymer solar cells based on low-band-gap donors (pp. 19-23).

Harvesting energy directly from the sun in order to generate electricity, solar cells appear to represent the epitome of green energy. Unfortunately, the manufacturing process of common starting materials, like crystalline silicon as the main component of conventional solar cells, requires a huge amount of energy. Firstly, transforming silica ore into its crystallized form requires temperatures of above 2,000°C. Not only is this an incredibly energy-hungry process in itself, but obtaining ultra-pure silicon also involves several hazardous chemicals and a potent greenhouse gas. Other main offenders in the modern manufacture of many inorganic thin-film solar cells include components comprising finite resources such as indium, or the toxic elements selenium and cadmium.

With the increasing strain on the global power grid and the drive towards sustainable energy production, we must therefore ensure that methods of manufacturing green-energy devices are equally sustainable – and this is the goal of Dr Manuela Schiek's research group at the University of Oldenburg, Germany. Their research focuses on alternative materials for solar cell manufacture that are both non-hazardous and readily available. This includes the use of organic semiconductors within the energy-capturing active layer, and a transparent electrode system formed from a silver nanowire mesh embedded in an organic polymer matrix (see text box "organic solar cell architecture").

With its complex multi-layered structure, surface analysis techniques provide vital insights into the workings of a solar cell. While tactile profilometry and Atomic Force Microscopy (AFM) have been the mainstays of surface metrology for a number of years, 3D confocal laser scanning microscopy (CLSM) is becoming an ever more popular tool.

Combining the ability to generate detailed, true-color optical images with the non-contact capabilities of laser scanning technology, the confocal laser scanning microscope really comes into its own as an optical profilometer. Faster and more efficient than stylus-based techniques, 3D CLSM is able to measure soft or adhesive surfaces and offers a resolution of 0.2 µm.

ORGANIC SOLAR CELL ARCHITECTURE

The most popular organic solar cell architecture is based on a photon-harvesting active layer, sandwiched between two electrodes – one of which must be transparent in order to allow light to penetrate (Figure 1).



Fig. 1: Schematic view of the most popular organic solar cell architecture.

Photons hitting the organic semiconductor generate the charge-carrying excitons, which by the use of two materials – an electron donor and an electron acceptor – are then separated into their separate electron and holes. Driven by an electrical field, the electrons and holes travel toward their respective electrodes, creating the charge separation necessary to form an electrical circuit.

Dr Schiek's research looks at using a transparent silver nanowire (AgNW) mesh electrode to replace the brittle and rare ITO, in addition to forming the active layer from organic materials as an alternative to environmentally damaging chemicals – creating flexible, sustainable and affordable thin-film solar cells for consumer applications.

ORGANIC MATERIALS IN THE ACTIVE LAYER

The active layer is where energy is captured from photons, and within organic solar cells this is often formed from a discontinuous blend of two materials – a polymer and a fullerene. With the polymer acting as an electron donor and the fullerene as an electron acceptor, this bulk heterojunction structure leads to enhanced charge separation of electrons and holes, and hence improved solar cell function. But polymers are often roughly defined mixtures of material with differing chain lengths, and with properties that are highly batch-specific. Molecular semiconductors, on the other hand, are defined building blocks with properties that can be adjusted by small changes to their structure, which can therefore be optimized for improved solar cell function. An interesting class of such molecules are the squaraine dyes (Figure 2), whose structure gives a broad absorption in the red region of the light spectrum. Dr Schiek's research is investigating a bulk heterojunction active layer formed from squaraines mixed with a fullerene acceptor (for more information, see reference 1). The thickness of the active layer is crucial for this application: too thin and the mobility of charge carriers is restricted, but too thick and both light absorbance and flexibility are significantly reduced.

Accurate measurement of layer thickness is therefore equally important. Within Dr Schiek's laboratory, once a scratch is made through the active layer surface with a fine needle, the step edges of this 'valley' are subsequently mea-



Fig. 2A: single crystals exhibit a golden metallic luster.



Fig. 2B: a spincoated layer of neat squaraine, with spherulite aggregates forming through crystallization upon thermal annealing

sured using profilometry. Tactile profilometry was previously relied upon, but the softness of the organic material hampered accurate measurement. In fact, a height discrepancy of around 20 nm was frequently observed between the two step edges, which is significant considering the average thickness of the active layer is 100 nm. As the needle steps up from the valley, it scratches into the surface and results in the false lower height reading.

With 3D confocal laser scanning microscopy, it is the laser that scans the surface, and such a

non-contact approach achieves far greater accuracy of surface profilometry (Figure 3). Moreover, providing a visual image of the sample is more intuitive, and with the LEXT OLS4100 this information is easily compiled into a report, presenting the image in support of numerical data (Figure 2B).

SQUARAINES UNDER POLARIZED LIGHT

These molecular dyes present a sustainable alternative for the active layer, here visualized between crossed polars.

Accurate metrology of soft material

Formed from soft organic material, the active layer is typically 100 nm thick and easily damaged by contact.

Transparent electrodes

Combining optical transparency with conduction, transparent electrodes form the anode of the solar cell while also allowing light to pass through to the active layer. The rapidly diminishing resource of ITO is currently the industrial standard for transparent electrodes. Moreover, ITO is a brittle material, restricting its use in mechanically flexible devices, and the hunt is on for a lightweight, cheap, flexible alternative that is also compatible with large-scale processing. Graphene presents a potential alterna-



Fig. 3A: Non-contact profilometry.

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Fig. 3B: Data compiled into a report.



Fig. 4: A flake of multilayer graphene coated with a light emitting organic semiconductor.

tive, for example (Figure 3), but the flakes are quite small, which limits its use for larger areas.

One promising alternative is a mesh of silver nanowires (AgNWs) embedded in a polymer matrix, and a second project within Dr Schiek's laboratory focuses on the production of the AgNWs, the subsequent pro-

cessing to form the electrodes and ultimately the integration into organic solar cells.

For optimum conductivity, a uniform connection must exist between the active later and electrode, demanding a homogenous AgNW mesh. Since the diameter of AgNWs at 100 nm is equivalent to the thickness of the active layer, it is also important to avoid these regions of aggregation and to prevent puncturing the active layer. However, in practice this is challenging to achieve across the whole solar cell using current spin-coating production techniques, and surface roughness evaluation plays a central role in optimizing the synthesis protocol.

AFM has been the main technique employed for the surface roughness evaluation of the AgNW mesh, but CLSM (using the LEXT OLS4100) has vastly improved the efficiency of this. Firstly, Dr Schiek found that expanding the field of view using the image stitching function allowed her to view a more representative sample of the electrode surface. The AgNW mesh may appear regular on a smaller scale, but by creating high-resolution images of one mm² (ten times greater than possible with AFM), regions of aggregation are easily identified that would otherwise have been missed (Figure 4 A,B). As can be seen in Figure 4C, the software also allows the height profile to be visualized in 3D, useful for both analysis and documentation. Moreover, the ability to increase the low-pass filter from a height of 80 µm to 800 µm allows more insightful analysis of the elevated regions of the silver nanowires.

It is also the case that AFM is time consuming. Not only does the scan itself take up to an hour, after setting up the instrument and adjusting for tip-related artefacts, it can often take a whole day to acquire a single useful image. With 3D CLSM, image acquisition is rapid and also highly intuitive owing to the dedicated software, even for students lacking microscopy experience. In terms of performance, the researchers found that AFM and CLSM produced comparable results, and have benefited from the improved efficiency of surface roughness evaluation of transparent electrodes.

Another interesting aspect of this project is the far-reaching potential of transparent electrodes throughout opto-electronic applications, including LEDs and touch screens, where developing alternatives to ITO is also a focus of intense investigation. In the



Fig. 5A: facilitating analysis of nanowire distribution on a larger scale in brightfield.



Fig. 5B: Height colour plot.



Fig. 5C: Organic solar cell architecture.

future, opto-electronic interfaces might even enable sight restoration, with retinal implants using light to generate electrical output and stimulating neuronal activity.

This structure is currently under investigation for organic light emitting diodes, while graphene also has potential for use as a transparent electrode – although flakes are too small for larger photovoltaic surfaces. Visualized with crossed.

Surface roughness evaluation of silver nanowire mesh electrodes

Image stitching function expands the field of view, Low-pass cut-off filter set to 800 µm.

SUMMARY

Feeding the global power grid in a sustainable fashion remains one of the biggest challenges faced by the modern world, and it's an exciting time for research into innovative solutions. The use of widely available materials holds future promise in providing a green alternative to conventional solar cell manufacture, with such investigations driven by the latest technological developments.

From enabling accurate step measurements of the active layer with non-contact profilometry, to improving the efficiency of analysis with speed and advanced software functions, the 3C confocal laser scanning microscope has provided Dr Manuela Schiek's research group with many advantages over stylus-based methods. As alternative energy becomes a greater focus in the coming years, the evolving light microscope technologies are likely to play a central role in the solar revolution.

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O 1 Nanodiamond-Based Separators for Supercapacitors Realized on Paper Substrates

Giuseppina Polino, Alessandro Scaramella, Valerio Manca, Elena Palmieri, Emanuela Tamburri, Silvia Orlanducci, and Francesca Brunetti

ABSTRACT

Significant interest is focused on developing environmentally friendly supercapacitors. In this context, cellulose-based substrates for energy storage devices can be well-engineered, lightweight, safe, thin, and flexible. Herein, a scalable, low-cost, and easy-to-process approach for the preparation of supercapacitors using large area techniques like spray and blade coating is presented. Symmetric supercapacitors using common copy paper and electronic paper as the substrate and poly(3,4-ethylenedioxythiophene)- poly(styrenesulfonate) (PEDOT:PSS) as electrodes are realized and investigated. This work uses composites based on detonation nanodiamonds (DNDs) and hydroxypropyl cellulose (HPC) as solid-state electrolyte and separator, achieving performance comparable to when in the presence of a liquid electrolyte.

INTRODUCTION

Cellulose-based substrates are considered among the most appealing solutions for green recyclable flexible electronics due to their compatibility with large-scale printing techniques. In that context, cellulose nanofibrils networks ensure a high surface area and a large amount of hydroxyl groups that can be functionalized, tuned, and are compatible with hydrophilic environment.^[1]

In the current work, a route for the realization and characterization of a biocompatible paper-based symmetric supercapacitor using electronic paper and common copy paper as substrates and poly(3,4-ethylenedioxythiophene)-poly(styrenesulfonate) (PEDOT:PSS) spray-coated electrodes is proposed. Moreover, we proposed a composite material based on hydroxypropyl cellulose (HPC) and detonation nanodiamond (DND) as separator and as solid electrolyte. DNDs are obtained by the purification of the soot generated through the detonation of explosives and present outstanding properties, such as thermal conductivity, thermal and chemical stability, low electrical conductivity, and mechanical properties (abrasiveness and hardness).^[2] Of particular interest, it has been shown that porous aggregates of DND and of DND dispersed in a polyelectrolyte (Nafion) are capable of increasing the ionic conductivity in the electrochemical system.^[3, 4]

We also compared the performance of devices based on solid electrolyte (HPC + DNDs) and liquid solution electrolyte (HPC + ND + Na₂SO₄) realized on both substrates (copy and electronic paper) using PEDOT:PSS spray-coated electrodes. The performances obtained are comparable with those found in the literature for devices realized on polyethylene terephthalate substrate using spray-coated PEDOT:PSS electrodes.^[5]

METHODS

To investigate the effects of different paper morphologies on the supercapacitor, we used a common acid-free copy paper with alkaline reserve (copy paper) and an electronic ultra-smooth paper designed for high-definition patterning and electroplating (electronic paper). The negative electrode was deposited on paper substrates by spray-coating. A thickness of 400 nm of PEDOT:PSS was obtained. After the deposition of the separator and the solid electrolyte, the positive electrode was spray-coated. The thickness of 300 nm of PEDOT:PSS for the positive electrode was chosen to avoid the melting of the separator, but still maintaining a sheet resistance comparable with the negative electrode. The deposition of the separator and the solid electrolyte was conducted by an automatized blade/ slot-die coater with drying step (hot air).

Morphology characterization of the devices' layers was performed using an Olympus[®] LEXT[™] OLS4000 laser microscope and a field emission SEM. The thickness of the layers was evaluated using a feeler gauge (digital thickness gauge).

Electrical and electrochemical characterizations were made using a four-probe setup connected to a source meter (Keithley 2420), and a potentiostat PGSTAT302N (EcoChemie Autolab B.V.).

RESULTS AND DISCUSSION

The steps of fabrication and the final layout of the device can be seen in Figure 1.

Figure 2 shows the morphology of the spraycoated PEDOT:PSS negative electrode deposited on electronic and copy paper. Here, it is possible to observe that the porous and



Figure 1: Process steps of fabrication; a) final layout (dry device); and b) immersion in liquid electrolyte (wet device).

(a) PEDOT:PSS ON E-PAPER (b) PEDOT:PSS ON COPY PAPER <u>160 µm</u> (c) HPC+ ND ON PEDOT:PSS (E-PAPER) <u>160 µm</u> <u>160 µm</u> <u>160 µm</u> <u>160 µm</u>

Figure 2: Microscope images of morphology: a) spray-coated PEDOT:PSS electrode on electronic paper substrate; b) spraycoated PEDOT:PSS electrode common copy paper substrate; c) HPC + ND separator on PE-DOT:PSS electrode deposited on electronic paper substrate; and d) HPC + ND separator on PE-DOT:PSS electrode deposited on copy paper substrate.



Figure 3: Layer-by-layer SEM images of the device on copy paper. a) Morphology of spray-coated PEDOT:PSS (negative electrode); b) morphology of the negative electrode blade coated with HPC; c) morphology of the negative electrode blade coated with HPC + DND; d) morphology of spray-coated PE-DOT:PSS (positive electrode) (on HPC layer); and e) morphology of spray-coated PEDOT:PSS positive electrode (on HPC + DND layer). rough nature of the copy paper substrate affects the deposition of the PEDOT:PSS electrode. Comparing the morphology of the HPC + DND separator deposited on the PEDOT:PSS electrodes, it can be noted that a smoother surface can be obtained on electronic paper, whereas for copy paper, the roughness of the starting PEDOT:PSS electrodes affects the separator morphology.

Figures 3 and 4 show layer-by-layer scanning electron microscopy (SEM) images, from the negative electrode to the subsequent upper layers of the final wet device on copy and electronic paper, respectively. On copy paper, the presence of DND seems to strongly affect the substrate roughness, filling the concavities and voids in the paper structure (Figure 3c), thus allowing a better deposition of the positive electrode (Figure 3e), which results in a more continuous and homogenous appearance with respect to the device prepared without DND (Figure 3d). On the contrary, as expected, on electronic paper, a smoother surface is always observed. PEDOT:PSS deposition results a high amount of homogeneity for both electrodes (Figure 4a,d). Conversely to what we observed with copy paper, the presence of DND leads to a rougher surface (Figure 4c,e), without, however, affecting the homogeneity of the deposition. A few Na_2SO_4 electrolyte crystals were observed, especially in the case of electronic paper, where the presence of DND seems to increase the quantity of the electrolyte retained in the separator.

Electrochemical tests of the assembled devices with both the typologies of electrolyte and electrode substrate show only an electric double-layer capacitor (EDLC) behavior. A stronger resistive behavior is produced for solution electrolyte and was observed in charge-discharge tests. We also identified a better charge propagation at lower scan rates. In fact, we observed an increasing shape deviation with an increase in the scan rate, probably due to higher internal resistance. In all the cases, we did not record rectangular-shaped cyclic voltammograms due to the use of PEDOT as electrode material.^[6]



Figure 4: Layer-by-layer SEM images of the device on electronic paper. a) Morphology of spray-coated PEDOT:PSS (negative electrode); b) morphology of the negative electrode blade coated with HPC; c) morphology of the negative electrode blade coated with HPC + DND; d) morphology of spray coated PE-DOT:PSS (positive electrode) (on HPC layer); e) morphology of spray coated PEDOT:PSS positive electrode (on HPC + DND layer).

	C [μF cm ⁻²]	C_{sp} [F g ⁻¹]	Q [μC cm ⁻²]	Ε [μWh cm ⁻²]	Ρ [μW cm ⁻²]
HPC + ND 1% solid electrolyte					
Copy paper	319.14	0.13	255.31	0.056	22.69
E-paper	1248.09	0.52	998.47	0.221	19.48
HPC + ND 1% + Na_2SO_4					
Copy paper	907.37	0.38	725.89	0.161	24.19
E-paper	1957.29	0.82	1565.83	0.347	22.77

Table 1: Electrochemical parameters extracted from charge-discharge cycles.

Supercapacitor parameters were calculated from charge–discharge cycles, are shown in Table 1, and are graphically represented in Figure 5. It is possible to observe that the devices produced with electronic paper show higher values of specific capacitance for both solid and solution electrolytes. Conversely, comparable power densities were achieved for both papers in dry and wet supercapacitors.

Although still an object of study, it is reasonable to assume that the hygroscopic environment guaranteed by the cellulose derivative-based separator allows the deprotonation/protonation of carboxylates and amphoteric functional groups present on the DND surface. As hypothesized by Postnov et al. (2017) and Gareeva et al. (2014),^[3, 4] the presence of DND could also ensure a structural arrangement of ionic channels and pores of DND aggregates and HPC chains organization crucial to charge transfers.

In Figure 8, the normalized capacitance values decay obtained from several charge-discharge cycles for both solid (Figure 8a) and solution electrolytes (Figure 8b) are reported. Devices on electronic paper present much higher capacitance values, and the capacitors realized with a solid electrolyte exhibit higher capaci-



Figure 5: Radar plot comparison of the performance of all the devices realized.

Figure 8: Capacitance evaluated over time obtained for: a) devices on copy paper and electronic paper with solid electrolyte; b) devices on copy paper and electronic paper with solution electrolyte.



tance retention. From this experimental data, we assume that in absence of DND, it is not possible to have significant measures due to observed short circuit of the device. This is ascribable to the deterioration (due to hydrolysis processes) of the thin separator layer, mainly caused by the high acidity of PEDOT electrodes above and below it. The better performance of the device with the solid electrolyte (DND + HPC) if compared with the one with solution electrolyte (DND + HPC + Na_2SO_4) could also be related to the deterioration of the HPC layer that occurs when the device is immersed in the electrolyte solution. Therefore, it is clear that DND manages to preserve the integrity of the separator; however, a significant drop in potential was still observed in the chargedischarge tests of the wet device if compared with the solid-state electrolyte curves.

CONCLUSION

We fabricated symmetric supercapacitors using upscalable techniques and green materials. Our devices are based on conducting PEDOT:PSS polymer electrode sprayed on two different types of papers (i.e., copy paper and electronic paper). Moreover, a new nanocomposite material based on HPC charged with DNDs was used as both separator and solid electrolyte and compared with the same composite wet with a Na₂SO₄ solution electrolyte. This work confirmed DND electrolytic properties due to both its special surface chemistry and its capability to modify and organize the structure of the matrix in which it is dispersed. Moreover, DND has once again proven to be an efficient consolidating material for cellulose derivatives, as we demonstrated that the device does not work in its absence due to the chemical nature of the separator.

The performance of our devices has been evaluated, and maximum power densities of ~24 μ W cm⁻² and specific capacitance of ~0.8 F g⁻¹ were achieved. Our results demonstrate that it is possible to obtain comparable performances on paper substrates with respect to analogous super-capacitors fabricated on plastic substrates with spray-coated PEDOT:PSS electrodes.

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02 Native Oxide Barrier Layer for Selective Electroplated Metallization of Silicon Heterojunction Solar Cells

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ABSTRACT

The metallization of silicon heterojunction (SHJ) solar cells by electroplating of highly conductive copper onto a multifunctional patterned metal layer stack is demonstrated. The approach features several advantages: low temperature processing, high metal conductivity of plated copper, no organic making, and low material costs (almost silver-free). A PVD layer stack of copper and aluminum is deposited onto the cell subsequently to TCO deposition. The aluminum layer is patterned with a printed etchant, and its native oxide on the remaining areas inhibits plating. During electroplating, the full-area aluminum layer supports plating current distribution and allows homogeneous plating height distribution over the cell. The NOBLE (native oxide barrier layer for selective electroplating) approach allows reaching a first encouraging SHJ solar cell efficiency of 20.2% with low contact resistivity.

INTRODUCTION

Electroplated copper contacts are particularly attractive for crystalline silicon heterojunction (SHJ) solar cells, which require a low-temperature metallization. Current trends in the silicon photovoltaic field point toward passivated contact architectures for high-efficiency solar cells.^[1, 2]

The symmetrical SHJ solar cell architecture is created by first depositing a thin intrin-

sic amorphous hydrogenated silicon layer – i.e., a-Si:H(i) onto the cleaned and textured c-Si wafer, followed by the doped a-Si:H (n or p), which act as carrier-selective junction. Both ultra-thin a-Si layers are quite sensitive to process temperatures above 200 °C (392 °F).^[3] Typically, a transparent conductive oxide (TCO) is sputtered onto the a-Si:H layers to contact the silicon, promote the lateral conductivity in the solar cell, and to serve as an antireflective coating. The cell architectures then need to be contacted by metalli-



Figure 1: Overview of the low-temperature routes to metallize bifacial SHJ solar cells covered by TCOs.



Figure 2: Sequence and tools used for the low-temperature NOBLE metallization on TCO for bifacial silicon heterojunction solar cells.

zation approaches, where metal paste printing is the most common and metal plating offers promising alternatives. Figure 1 presents an overview of the low-temperature routes to metallize SHJ solar cells covered by TCO.

Our work demonstrates an alternative plating approach containing one step less than the "Resist Masking route" and uses very low amount of silver (Ag). Our novel native oxide barrier layer for selective electroplating ("NOBLE route") aims at improving the process flow with fewer steps and by avoiding the use of any costly organic masks. In this concept, the native oxide of a thin aluminum (Al) layer enables performing selective copper (Cu) plating onto a patterned metal seed. An etchant is inkjet-printed to structure the contact positions in the Al layer. This grid-patterning method is even cheaper than the deposition of a reactive silver ink onto the aluminum.^[4, 5]

The NOBLE metallization route depicted in Figure 2 allows fast and homogeneous electrodeposition of copper onto a multifunctional patterned metal layer.

After vacuum deposition of the PVD layer stack (Figure 2(1)), the grid patterning is achieved by printing a low concentrated alkaline ink or paste to etch the aluminum-aluminium oxide (Al-Al₂O₃) selectively (Figure 2(2)). In the present work, inkjet printing was employed. This selective etching removes the Al layer only in grid positions and does not impact the underlying metal-seed (on top of the TCO). This patterning step allows plating Cu selectively onto the metal seed (Figure 2(3)). At this state, the less noble metal Al and the Al₂O₃ are acting as barrier to inhibit Cu plating in the non-grid positions as described by Hatt et al.^[5] A thin silver capping (≈200 nm) follows to protect the Cu-contacts against oxidation. The remaining PVD layers (Al and metal seed) in nongrid positions are then etched-back by chemical processing without damaging the underlying TCO (Figure 2(4)). On the contact flank, only a few nanometers of Cu are not protected by Ag in comparison to the "Resist route," which leaves several micrometers un-capped.

METHODS

Investigations on metal etching were realized on planar glass (soda-lime glass) with different sputtered metals (Al, Cu, Ag, NiV (93:7), and Ti) Electron-gun evaporation was also used to deposit Al onto glass to observe the impact of the PVD technique on the etching rate for this metal. The metal thicknesses were measured before and after etching using an Olympus[®] LEXT[™] OLS4000 confocal microscope, a profilometer (Dektak - STYLUS PROFILER), or an X-ray fluorescence tool.

After sputtering the metallic layers onto the SHJ solar cells, a PIXDRO LP50 printer from Meyer Burger Technology AG was used to print the grid positions with NaOH_{aq}, thus completely etching the AI. A complete description of the cell fabrication process can be found in the original work. The contact metallization was characterized by the Olympus LEXT OLS4000 confocal microscope after each processing step.

RESULTS AND DISCUSSIONS

The etching selectivity of different metals was studied to enable the selective etching steps mentioned above. There are many solutions that may etch metals deposited by PVD. The investigation was focused on PVD AI, Ti, NiV, Cu, and Ag, which are promising for either low-contact resistance or plating selectivity. The etch rates in nm s⁻¹ are presented in Figure 3 for metals sputtered on planar glass. Although etching rates might be different on different surfaces, general trends were confirmed to be similar on TCOs and on the textured surface of the SHJ solar cells.

In summary, it was observed that Al can be selectively removed versus Cu or Ag with dilute alkaline solutions and at high etching rates. On the other hand, Cu can be etched with $FeCI_3$ or $(NH_4)_2S_2O_8$, while Ag and Al will remain unaffected.

For the NOBLE processing, both sides of the textured SHJ precursor with ITO are entirely covered by thin layer stacks of either PVD-Cu/ Al (50/100 nm) or Ag/Al (20/100 nm). The selective etching of Al against the metals below (Cu or Ag) enables easy patterning of the grid positions. In the present work, inkjet-printing of NaOH_{aq} was employed to structure lines in the Al-Al₂O₃ layer. Narrow lines in the PVD Al of around 25 μ m wide without spreading or interruption were created. The underlying PVD Cu-seed can be observed in line positions as demonstrated in Figure 4a.

The selective Cu electrodeposition is then performed onto the Cu-seed as observed in Figure 4b. The native oxide, covering the Al surface, acts as inhibitor for Cu deposition out-

Figure 3: Etching rates of thin ($\leq 1\mu$ m) sputtered metal layers removed from planar glass at room temperature in different solutions (*lower NH₃/H₂O₂ concentration)





Figure 4: Microscopic pictures of a finger on the ITO from a SHJ solar cells along the NOBLE metallization after: a) inkjet-printing of NaOH_{aq}, b) Cu-Ag plating, and c) etching-back PVD layers in non-contacted positions.



Figure 5: SEM pictures in the cross-section of the SHJ solar cell covered by ITO after NOBLE metallization: a) finger plated on Cu-seed, b) area on a pyramid flank in non-finger position, and c) finger plated on Ag-seed.

side of the designated contact area. The Cu grows isotropically onto the metal seed, which widens the contact a bit. The Cu deposition is fine-granular, which gives high contact conductivity near the value of the Cu-bulk. A thin silver capping is also deposited to prevent Cu from any oxidation, as seen on the inset.

After plating, the PVD layers are selectively etched back (Figure 4c) in non-grid positions with a solution of H_3PO_4/HNO_3 . SEM images of the fingers' cross-sections (Figure 5) confirms that ITO was not significantly damaged through the etch-back step, and the layers presented good adhesion even with non-optimized conditions. No voids or uncontacted area could be detected, which would increase the resistivity at the interface. Good adhesion was also confirmed by tape testing.

Sheet resistances (R_{sh}) of ITO and contact resistivities (ρ_c) measured on both sides of different stacks after NOBLE metallization are summarized in Table 1. The measured ρ_c shows little difference between PVD metal seed composed by Cu or Ag, which means the use of Cu-seeds can be an efficient way of cost reduction, getting closer to Ag-free metallization. Moreover, our contact stack after NOBLE metallization present similar results to the one used by CSEM to reach a very high efficiency up to 24.1% on a bifacial plated SHJ solar cell.^[6]

SHJ solar cell made with NOBLE metallization presented efficiency (η) of 20.2% and an encouraging fill-factor (FF) of 78.0% was obtained. This is despite the fact that the pseudo fill factor (pFF) of this cell is already limited to only 80.5%, due to the small cell size. Table 2 presents the SHJ solar cell properties under 1-sun illumination.

The open-circuit voltage V_{oc} and pseudo fill-factor pFF limitations result from the non-optimized metal sputtering process on ITO and from the small cell size. The fill factor is impacted by the non-optimal grid design. The short-circuit current J_{sc} might be reduced by the lab-scale etch-back procedure of the PVD layers. Optimizations of the process might exploit further the full potential of the solar cell in combination with the apparently well performing metallization. **Table 1:** Sheet resistances (R_{sh}) of ITO and contact resistivities (ρ_c) of different stacks measured by TLM after NOBLE metallization on commercial SHJ solar cells.

Metal stack	Rsh [Ω sq⁻¹]	$\rho_{contact}$ [m Ω cm ²]
c-Si (n)/a-Si:H(i/n)/ITO/Cu _{PVD} /Cu-Ag _{plated}	82.7 ± 0.2	4.0 ± 0.4
c-Si (n)/a-Si:H(i/n)/ITO/Ag _{PVD} /Cu-Ag _{plated}	80.6 ± 0.2	3.5 ± 0.8
c-Si (n)/a-Si:H(i/p)/ITO/Cu _{PVD} /Cu-Ag _{plated}	169.7 ± 0.5	2.8 ± 0.5 Not influenced
c-Si (n)/a-Si:H(i/p)/ITO/Ag _{PVD} /Cu-Ag _{plated}	120.3 ± 0.4	1.8 ± 0.3 by the bulk

Table 2: SHJ solar cell properties after NOBLE metallization.

Area [cm ²]	V _{oc} [mV]	pFF [%]	FF [%]	J _{sc} [mA cm ⁻²]	η [%]
6.25	718	80.5	78.0	36.1	20.2

CONCLUSION

An alternative approach for low-temperature metallization of solar cells with TCO layer (e.g., SHJ solar cells), which is currently in the early development stage, was demonstrated. It takes advantage of the selectivity of etchants and plating processes toward different metals.

A thin stack of two PVD metal layers were sputtered onto the cell right after the TCO in the same tool without breaking the vacuum. Thus, sputtering costs are kept low, especially for these thin layers and materials such as Al and Cu. These metal layers allow plating the patterned contacts on both sides of the cell at the same time quickly and homogeneously. The selective etching of metals was investigated in a study of etching rates for Al, Cu, Ag, Ti, and Ni in different acidic and alkaline solutions. This selectivity enabled grid patterning of a thin PVD-Al layer by inkjet printing of an alkaline solution. Copper could then be plated in the printed areas, without parasitic deposition, onto the underlying PVD metal-seed due to the presence of native oxide on the Al surface. After electroplating, the thin PVD layers were removed by etching in non-grid positions selectively. Contact resistivity below 3 mΩcm² were achieved on stack systems of ITO-Cu_{PVD}-Cu_{plated}-Ag_{plated}, and the improvement of the sputtering might allow reaching 0.4mΩcm², as reported in the literature. A promising efficiency of 20.2% with a FF of 78.0% was presented on a commercial SHJ solar cell precursor. This approach saves processing steps and consumables as compared to the electroplating into openings in an organic mask.

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ORIGINAL PUBLICATION

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O3 Spray-Coated Polymer Solar Cells Based on Low-Band-Gap Donors Processed with *ortho*-Xylene

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ABSTRACT

Low-band-gap polymer donors in the field of polymer solar cells (PSCs) allow high conversion efficiencies to be achieved. Unfortunately, optimal performance is strongly correlated to the use of chlorinated solvents, known to be toxic in working conditions. For this reason, high-performing PSCs have been only deposited via small-area techniques. We report the use of a PBDTTTC-T:PC₇₀BM blend dissolved in the non-chlorinated solvent *ortho*-xylene for the deposition of spin-coated and spray-coated active layers in direct PSCs. The results in this work open the way to the use of low-band-gap materials in spray-coating, a nonwasteful technique compatible with coating large areas.

INTRODUCTION

Polymer solar cells (PSCs) have attracted the attention of the scientific community because they offer the possibility to fabricate photovoltaic devices by solution processing, enabling a reduction in cost and increase in their potential for application in fields such as consumer electronics and building-integrated photovoltaics (BIPVs).^[1, 2] In recent years, marked improvement was made possible by the use of photoactive layers based on low-bandgap polymers, which allow better harvesting of solar energy compared to the conventionally used material poly(3-hexyl-thiophene) (P3HT). In particular, poly{[4,8-bis-(2-ethyl-hexyl-thiophene-5-yl)-benzo[1,2-b:4,5-b']dithiophene-2,6-diyl]-alt-[2-(20-ethyl-hexanoyl)-

thieno[3,4-b]thiophen-4,6-diyl]} (PBDTTT-C-T) in combination with $PC_{70}BM$ is an effective blend system that reaches high efficiency.^[3-5]

In spite of this remarkable progress, a strong limitation that hinders the scaling-up of PSC fabrication is the use of chlorinated solvents (toxic toward human health and the environment), which serve to effectively dissolve the polymer/fullerene blend to obtain a suitable morphology. Hence, blends dissolved in more eco-friendly solvents have attracted significant interest.

We report on the use of a PBDTTT-C-T: PC₇₀BM composite dissolved in *ortho*-xylene, and deposited both via spin-coating and spray deposition, in direct PSCs. A record conversion efficiency of 8% is achieved by devices with the spin-coated blend, whereas the devices with the spray-coated blend reach an efficiency of 4.4% after proper optimization of the spray parameters.

METHODS

The photovoltaic devices were fabricated on ITO-coated glass substrates. The electron transport layer was PEDOT:PSS VPAI 4083. The PBDTTT-C-T and PC₇₀BM blend (1:1.5) were dissolved in *ortho*-xylene. Undiluted and diluted blends with and without di-iodooctane (DIO) were prepared before deposition.

Glass/ITO substrates were patterned by wet etching. PEDOT:PSS was spin-coated in a glove box and then dried by thermal annealing at 150 °C (302 °F) for 10 minutes in a nitrogen atmosphere. PBDTTT-CT:PC₇₀BM was either spin-coated or sprayed by using a dual-action commercial airbrush supplied by compressed air. After drying, the samples were completed by thermal evaporation of 10 nm of calcium and 100 nm of aluminum in high vacuum.

Optical images of the deposited photoactive layers were acquired by using an Olympus[®] LEXT[™] OLS4000 microscope. The thicknesses of the as-deposited films were measured with a profilometer (Dektak 150). The surface topography was monitored using an A.P.E. Research AFM. Absorption curves were obtained by a UV-VIS-NIR spectrophotometer (Shimadzu UV2550). A custom-made tool comprising a sourcemeter (Keithley, mod.2612) and a monochromator (Newport, Mod.74000) was used to measure the external quantum efficiency (EQE) values of the photovoltaic cells. J–V curves of the devices were evaluated by using a class A solar simulator (ABET Technologies, Sun2000). Before each measurement, the irradiation level at the height and position of the solar cell was verified by a means of a calibrated pyranometer (SkyeSKS1110).

RESULTS AND DISCUSSION

A preliminary investigation was carried out to choose the different solution formulations for spin and spray deposition in order to obtain suitable layers of the PBDTTT-C-T:PC₇₀BM blend dissolved in *ortho*-xylene. We found that the solution used for spin-coating was also applicable for the spray technique; in fact, the pure solution was not viscous and easy to spray. However, we also investigated a diluted solution (1:4) to determine the effect of dilution on film-forming properties of the spray-coated samples. Blend deposition was performed by using blends both with and without di-iodooc-



Figure 1: Optical images of the active layers obtained with various blend formulations and deposition techniques.

Active Layer	Deposition Technique	Thickness [nm]	RMS [nm]
PBDTTT-C-T:PC ₇₀ BM	spin	100	1.52
PBDTTT-C-T:PC ₇₀ BM + DIO	spin	130	3.85
PBDTTT-C-T:PC ₇₀ BM + DIO	spray	122	2.90
PBDTTT-C-T:PC ₇₀ BM (1:4 dilution)	spray	126	1.76
PBDTTT-C-T:PC ₇₀ BM + DIO (1:4 dilution)	spray	120	18.8

Table 1: Thickness and root-mean-square (RMS) roughness of the active layers with various blend formulations and deposition techniques.

tane (DIO) in order to understand the influence of the additive on the morphology of PBDTTT-CT:PC₇₀BM dissolved in *ortho*-xylene.

Figure 1 shows an optical analysis of the active layers. The DIO additive evidently enhances the surface morphology of the photoactive layer, which should result in a better performance compared to the cases without DIO. In fact, when DIO is not added, large agglomerates of non-dissolved fullerene are visible in the spincoated films, whereas in the spray-coated ones, there is no coalescence among the droplets, with most of them surrounded by coffee rings. Focusing on the diluted blend, DIO improves the appearance of the surface morphology of



Figure 2: UV/Vis absorption spectra of the active layers obtained with various blend formulations and deposition techniques.

the film but coalescence is not at an advanced stage, leading to the deposition of visible droplets that are homogenous in composition but still different in size. Some uncovered areas are noticeable in the layer obtained using the undiluted blend with DIO, suggesting that it is difficult to obtain perfectly uniform coverage by spraying the blend for a few seconds.

A deeper investigation of the surface topography was performed by atomic force microscopy (AFM). The corresponding root-meansquare (RMS) roughness values and layer thicknesses were measured by profilometry and are reported in Table 1. Topographies of spin- and spray-coated active layers without DIO are similar and present low roughness. The addition of DIO increases the RMS roughness of both spin and spray-coated films; in particular, for sprayed layers, the diluted blend with DIO exhibits larger, differently sized domains, showing higher roughness than the diluted blend without DIO. Notably, the RMS roughness is very low in spray-coated PBDTTT-C-T:PC₇₀BM (1:4 dilutions) because the AFM measurement covers only a small area within the large (tens of micrometers) droplets shown in Figure 1 and does not consider the overall topography, which includes the solid content of droplet boundaries, and thereby underestimates the measurement. Regarding thickness, spin-coated active thickness increased with addition of DIO, which can be attributed to the higher viscosity of the modified solution.^[6] Meanwhile, the spray-coated blends with and without DIO have similar thicknesses.

Absorbance spectra (Figure 2) show a greater absorption when DIO is added to the solutions; also, the spectra for the undiluted solutions with DIO used both for spin- and spray-coating match very closely. Regions of lower absorption are apparent in the 1:4

Active Layer	Deposition Technique	Voc [V]	Jsc [mA cm ⁻²]	FF	PCE [%]
PBDTTT-C-T:PC ₇₀ BM	spin	0.57 (0.49±0.14)	5.6 (5.03±0.05)	0.35 (0.33±0.02)	1.1 (0.84±0.33)
PBDTTT-C-T:PC ₇₀ BM	spin	0.78	16.2	0.65	8.0
+ DIO		(0.77±0.005)	(15.1±0.05)	(0.63±0.01)	(7.39±0.34)
PBDTTT-C-T:PC ₇₀ BM	spray	0.72	12.2	0.50	4.4
+ DIO		(0.71±0.02)	(11.1±0.11)	(0.48±0.01)	(3.8±0.55)
PBDTTT-C-T:PC ₇₀ BM	spray	0.39	4.8	0.32	0.5
(1:4 dilution)		(0.34±0.04)	(4.6±0.02)	(0.31±0.005)	(0.48±0.04)
PBDTTT-C-T:PC ₇₀ BM +	spray	0.7	9.6	0.44	2.7
DIO (1:4 dilution)		(0.65±0.03)	(8.1±0.1)	(0.38±0.03)	(2.03±0.47)

Table 2: Electrical performance of the best fabricated devices from various blend formulations and deposition techniques. Average values and standard deviations over eight devices are reported in parentheses.

diluted solution with DIO. Because the thicknesses are approximately the same as in the undiluted spray-coated layer, this decrease may be due to the use of a non-optimized DIO concentration. The lower intensities of the sprayed diluted active layer without DIO suggest a poor morphology of the phase-separated bulk heterojunction (BHJ) materials.

In Table 2 we summarize the photovoltaic performance of the described blend formulations and deposition techniques in terms of short-circuit current density (J_{SC}), open-circuit voltage (V_{OC}), fill factor (FF), and power conversion efficiency (PCE). The film-forming properties of the various solutions deposited by spin and spray-coating, with and without DIO, are clearly correlated to the observed photovoltage in the finished cells. Voc is high only in the presence of DIO, revealing that such an additive is essential in achieving a favorable morphology and proper coverage of the surface, in agreement with the optical analysis in Figure 1. An abrupt decrease of fill factor occurred in the case of spin and spray-coated active layers without DIO, and this may be caused by the poor bulk nanometer-scale morphology. In addition, in the spray-coated layer the droplet boundaries (as evident in Figure 1) hinder charge transport, leading to a high series resistance that heavily affects the J_{SC} and the FF values.

Comparing the performance obtained using the additive in PBDTTT-C-T:PC₇₀BM both for spin and spray-coating, an enhancement of the photo-generated current is observed. This enhancement can be ascribed to improved charge transport at the interface, corresponding to a larger surface roughness (as shown by the RMS values in Table 1) that implies a more uniform distribution of the domains on the active layer's surface.^[6] The different values of J_{SC} observed in the diluted and undiluted cases suggest that the dilution does not achieve the same uniformity in the thickness of the active film, as confirmed by the very large value of roughness in the diluted film. Moreover, the photocurrent is affected by resistive losses (high series resistance).

Remarkable electrical performance is obtained from the device based on the undiluted blend deposited by spray-coating, but it is lower than those from a device using the same blend applied by spin-coating. We ascribe this difference to the spray environment (air) and to non-optimal coverage due to short spraying time. Focusing on the data in the Table 2, a satisfactory reproducibility is observed both for spin- and spray-coating in the case of the undiluted blend, and for spray-coating in the case of the diluted blend. This demonstrates the good control over the process and the ability to generate good coverage by using solutions with different formulations in two different techniques (see Figure 1), providing reliable electrical results.

Figure 3 shows the external quantum efficiency (EQE) spectra of the best-performing devices fabricated with the spin- and spray-coated active layers. Although the solution used to deposited both the layers is the same (PBDTTT-C-T:PC₇₀BM + DIO), the EQE for the spray-coated layer is lower than that

of the spin-coated layer, explaining the lower J_{SC} values; moreover, at short wavelengths the peak associated to PC₇₀BM absorption is not so distinct and at longer wavelengths the profile is decreased, probably due to the lack of a uniform coverage which influences the blend/evaporated contact interface.

The difference between the devices with spin-coated and spray-coated layers is still large, but this work opens the way to the use of low-band-gap materials in spray-coating, a non-wasteful technique that is compatible with large areas and potentially effective for the fabrication of organic photovoltaic devices.

CONCLUSION

We report the results of a study on the behavior of spin- and spray-coated PBCDTTT-C-T:PC₇₀BM active layers, both pristine and with an amount of additive (DIO), to fabricate bulk heterojunction polymer solar cells. This work highlights the critical issues of the spray-coating technique in relation to the use of a low-band-gap polymer. However, we achieve promisingly high efficiencies of 8% for a spin-coated PBCDTTT-C-T:PC₇₀BM layer and 4.4% for a spray deposited layer, both processed in a non-chlorinated solvent in ambient atmosphere. We also demonstrate the importance of the solution used in the improvement of the film quality and the compatibility with large-area processes. Those results are promising to produce organic solar cells with non-chlorinated solvents at low temperatures and low costs.

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© 2014 WILEY-VCH Verlag GmbH & Co. KGaA, Weinheim **Figure 3:** EQE spectra of the best performing devices with spin- and spray-coated active layers (undiluted PBDTTT-C-T:PC₇₀BM with DIO).

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