Flexible and Printed Electronics

PAPER

Regenerated cellulose fiber solar cell

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Abstract

Wearable electronics and smart textiles are growing fields in the cause to integrate modern communication and computing tools into clothing instead of carrying around smart phones and tablets. Naturally, this also requires power sources to be integrated in textiles. In this paper, a proof-of-concept is presented in the form of a photovoltaic cell based on a commercially available viscose fiber. This was realized using a silver nanowire network around the viscose fiber to establish electrical contact and a photoactive coating using the standard workhorse among organic thin film solar cells, a blend of poly(3-hexylthiophene) and phenyl-C₆₁-butyric acid methyl ester. The structure and performance of single fiber devices demonstrate their feasibility and functionality. The applied materials and methods are compatible with solution processing, and therefore qualify for potential roll-to-roll large-scale production.

Introduction

Nowadays, people increasingly rely on portable electronics, from mobile phones to tablets and a whole range of other gadgets. Manufacturers are now trying to integrate the technology directly into clothing. This concept is known as smart textiles (Service 2003, Weng et al 2016). However, all these modern helpers require electrical energy to work. At present, rechargeable batteries are the main solution to providing the power needed. However, these have two main disadvantages that limit their practicability for wearable electronics. First, their storage capacity is limited, and second their weight increases with capacity. One possible solution to this problem is energy conversion. Therein an entire field of research is dedicated to the transformation of different human daily life energy sources (e.g. light, body heat, movement) into usable electrical power (Weng et al 2016).

Light, as an abundant source of energy, motivated various reported concepts for photovoltaic devices that are compatible with textiles (Weng et al 2016). In one approach, a textile-based solar cell was realized by stacking textile electrodes of woven metal, carbon and dye-decorated titania wires, finalized by adding an electrolyte to make a dye-sensitized solar cell (e.g. Pan et al (2014)). However, the practical handling of the electrolyte in the final fabric was problematic. In another report, a standard inverted organic photovoltaic device structure was deposited onto a gold-decorated textile electrode and stitched onto a fabric, to be connected by conducting fibers to potential appliances (Lee et al 2014). There, the durability of a stitched-on device during wear is the limiting factor. There have been also attempts to fabricate photovoltaic fibers to enable direct integration of the solar cell into the fabric. For this latter approach, versatile concepts have been presented. For example Qiu et al (2014) reported a perovskite solar cell assembled onto a flexible stainless steel wire. A dye-sensitized solar cell was even transferred into a fiber design by Chen et al (2012) by using intertwined strands of carbon nanotube (CNT) fiber with and without titania/dye coating. An organic solar cell fiber using the common conjugated polymer poly(3-hexylthiophene) (P3HT) and fullerene derivative phenyl-C₆₁-butyric acid methyl ester (PCBM) was presented by (Zhang et al 2014) built on a titanium wire electrode, stranded with a CNT fiber counter electrode. One thing these approaches have in common is a metal- or carbon-based conductive fiber as center electrode. Another approach was chosen by Greulich-Weber et al (2009),
who built an organic–inorganic hybrid solar cell based on differently doped single-crystalline silicon carbide (SiC) fibers, which are optically transparent and dependent on the dopant concentration conductor or semiconductor. As the latter, SiC was used as the acceptor in combination with the conjugated polymer donor P3HT deposited on the fiber.

The main drawback of most of the above-mentioned fiber-based photovoltaic device designs is the limited mechanical durability of the metallic, spun-CNT, ceramic or ceramic-decorated core-fibers to bending and stretching, compared to common textile fibers. Therefore, this paper presents a photovoltaic fiber based on a regenerated cellulose (viscose) fiber, which is readily found in common fabrics. This viscose fiber is decorated with a network of silver nanowires (AgNWs) to form the conductive electrode, followed by a coating of organic semiconductors for the photoactive layer, finalized by a one-sided metal counter electrode. In this paper, we focus on materials and methods enabling scalability, e.g. adaptable to roll-to-roll (R2R) processing, as outlined by Bedeloglou et al. (2009).

In the following, the architecture, production and electronic properties of these viscose fiber-based solar cells will be presented and discussed.

Experimental

Materials

Hollow viscose fibers with a linear mass density of 2.1 dtex (equivalent to a diameter of approximately 20 μm) and a length of about 40 mm were used in this study and provided by Kelheim Fibres GmbH (Kelheim, Germany). A AgNW suspension was supplied by Sigma-Aldrich (0.5 wt% in isopropanol) and the dispersed AgNWs had a nominal average diameter of 115 nm and lengths between 20 and 50 μm. Further, anhydrous chlorobenzene, as solvent for the organic semiconductors, was purchased from Sigma-Aldrich.

A PEDOT:PSS (Clevios™ PVP CH 8000), was obtained from Heraeus-Clevios. Regioregular P3HT (EE-4002, RR 93%, Mw 70 000 g mol⁻¹) and PCBM (99%) were bought from Rieke Metals Inc. (US) and Ossila Ltd (UK), respectively. All substances mentioned were used without further purification.

Preparation of photovoltaic fibers

For better handling under laboratory conditions, four viscose fibers at a time were fixed equidistantly with their two ends to two separate glass plates, as shown in figure 1(a), allowing access to the fiber without the influence of a foreign contact surface. Nail polish was used as adhesive between the viscose fiber and glass, which, in contrast to other regular glues, does not tend to creep and spread along cellulosic fibers, forming an unwanted coating (Fischer et al. 2014).

The following manufacturing procedure is based on Kopeinik et al. (2015). To form a conductive surface for an electrode, the AgNWs were deposited on the viscose fibers via dip coating (SDI company, Nanodip ND-0407) at withdrawal speeds between 11 and 66 μm s⁻¹ from isopropanol suspension and allowed to dry for 1 h at room temperature. The viscose–AgNW fibers were treated with oxygen plasma (Diener Electronics, FEMTO Low Pressure Plasma System) at 100 W for 10 min and 80 cm³ min⁻¹ O₂ flow, to facilitate wetting for follow-on coatings. For deposition of the hole-conduction interlayer, a PEDOT:PSS suspension was sonicated for 30 min and 100 μl was drop-casted onto the freshly plasma-treated fiber, followed by drying in flowing argon at an elevated temperature (200 °C, 30 min).

The active layer was applied in the argon atmosphere by repeated drop-casting from a hot (70 °C) solution of 1:2 P3HT:PCBM in chlorobenzene (17 mg ml⁻¹ + 34 mg ml⁻¹). The layer was applied in three steps of 70 μl, 100 μl and 100 μl, with 15 min drying at 70 °C after each step and final drying at room temperature for at least 1 h.

Finally, the outer cathode of aluminum (~100 nm) was deposited via thermal evaporation on one side of the fiber.

Characterization

Visual investigation of the fibers was performed using an Olympus BX 51 optical microscope with integrated digital camera. Electrical characterization of the AgNW-coated viscose fiber and photovoltaic device characterization were carried out with electrical microprobes in argon atmosphere, using a Keithley 2636A source-measure unit. For photocurrent measurements, the current–voltage characteristics were acquired under white light illumination. In the absence of standard AM1.5G (100 mW cm⁻² light source, solar simulator) access in the glovebox, an LED white light source of color temperature of 7000 K (Osram LED ‘cool daylight’, arrangement of 3 LEDs) with a light intensity of 0.54 mW cm⁻², was used.

The surface of the AgNW-coated viscose fibers was investigated by scanning electron microscopy (SEM) imaging with the Everhart–Thornley detector of the high resolution SEM Zeiss Ultra 55. For transmission electron microscopic (TEM) evaluation of cross-sections of completed photovoltaic fibers, the hollow viscose fiber with their coatings of AgNWs, PEDOT:PSS and photoactive composite were sputter-coated with a thin gold layer and then embedded in cyanoacrylate between acrylic glass platelets and sliced using an ultra-microtome (UC6, Leica Microsystems) equipped with an 35° diamond knife (Diatome AG, Switzerland). The clearance angle was 4°, the nominal feed was generally set at 70 nm and the cutting speed was chosen as 0.6 mm s⁻¹. These sections were then transferred (Perfect Loop, Diatome) to a 75 mesh grid.
coated with formvar. The sliced cross-sections of the photovoltaic fiber were examined using a Tecnai T12 (FEI Europe) TEM with a LaB6 field emission cathode, operated at 120 kV in bright-field mode, with a 7.5 mrad objective aperture.

Results and discussion

The principal design of the suggested photovoltaic viscose fibers is as follows (see figure 1(b)): First, the viscose fiber is coated with a continuous but flexible conductive material to create the flexible inner electrode.

A follow-on hole-conductor coating acts as a buffer to prevent short circuits, leading to selectivity of the electrode and diminishing charge extraction barriers. This is followed by a photoactive (absorbing) layer consisting of solution-processable organic semiconductors. The final layer is the external transparent electrode. Suitable solution-processable conductive materials for the outer electrode are sol–gel derived transparent conducting oxides, as found in the literature (Hilgendorff et al 1998, Kisailus et al 2006, Granqvist 2007, Morgenstern et al 2011) or graphene (Wei et al 2010, Zhu et al 2010).

In the present proof-of-concept, the devices have the following configuration: viscose/AgNW/PEDOT:PSS/P3HT:PCBM/Al. The choice of AgNWs for the inner electrode has multiple reasons: first, their convenient solution-processability; second, good adhesion and network-building properties on cellulose surfaces, as has been shown earlier by Kopeinik et al (2015); and third the immense benefit of the resulting large metallic surface area, which is considerably higher than if merely a smooth continuous coating of silver was applied, generating a larger contact area for charge-transfer from the semiconductor, thus enabling a larger current-output per area (of the fabric). PEDOT:PSS as hole-conductor and photoactive blends of P3HT as donor with PCBM as acceptor are standard materials used in conventional organic thin film solar cells (Dang et al 2011). For the outer electrode in our fiber devices, a one-sided evaporated aluminum cathode was chosen for simplicity reasons. This means that here a common model system from planar organic photovoltaic devices (Dang et al 2011) is adapted for application on a 1D-device based on a regenerated cellulose fiber, decorated with AgNWs, as the electrode. For planar devices with these materials and architecture, power conversion efficiencies of up to 5% were reported (Dang et al 2011).

In the SEM image of the viscose fibers used in this study (figure 2) a hollow tubular structure and corrugated surface can be clearly seen. In principle, any other type of viscose fiber with a more or less smooth outer surface could be used, too.

To the bare eye the viscose fibers coated with AgNWs appear white to light gray, depending on the coating conditions affecting their coverage. Under an optical microscope (figure 3(a)) the transparent viscose fiber and the attached AgNWs are already well distinguishable by the darker appearing fiber and the bright lines on the surface, which are reflections of the
AgNWs. However, a much more detailed picture of the adhered AgNWs on the viscose fiber surface can be seen in the SEM image in figure 3(b). Though the hollow fiber structure has obviously collapsed (due to capillary forces), therefore appearing flat and deformed instead of tubular, the dense network of tight-fitting, closely adsorbed NWs is clearly visible.

AgNWs are known to enable very small bending radii, with bending angles as low as 7° to 20°, observed for comparable AgNWs (Wu et al 2006). This fact explains the observation that hardly any AgNWs stick out at the edges of the viscose fiber, as the adhesion force to the cellulose is stronger than the elasticity of the NW (McDowell, Leach, Gall 2008). However, even a few upstanding AgNWs can easily lead to considerable leakage currents or even short circuits in the device if they cannot be sufficiently covered by the thin organic semiconductor coating. For this reason, the yield of functioning photovoltaic fibers under non-optimized laboratory conditions was at 4% (out of 80 devices). This problem could be solved by using viscose fibers that do not collapse during processing, as they would provide higher probability of achieving homogeneous semiconductor surface coatings. The conductivity of the AgNW-decorated viscose fiber depends on its surface coverage. This amount can be tuned by the solid content of the AgNW suspension and the withdrawal speed in the dip-coating process. The lowest resistivity of about 30 Ω was achieved with a concentration of 0.5 wt% and a withdrawal speed of 33 μm s⁻¹. Faster speeds lead to lower coverage, thus less conductivity, e.g. at 66 μm s⁻¹ the resistivity increases to about 200 Ω. Speeds lower than 33 μm s⁻¹ do not increase the conductivity of the sample any further, indicating some sort of saturation in AgNW coverage. It has previously been shown on wood-pulp fiber networks (paper) that the AgNW coating is proportional to the AgNW concentration in the suspension and the dip-coating parameters (Kopeinik et al 2015). An approximately linear relationship between withdrawal speed and conductivity was observed. The minimum coverage threshold value for noticeable conductivity of the viscose fiber is given by the fact that the AgNW needs to form an interconnected network along the fiber. The actual amount of AgNW at this threshold value was not determined in this study. The electrical properties of the viscose/AgNW electrode fibers show perfectly ohmic behavior, as demonstrated in an exemplary current–voltage diagram of a measured fiber in figure 4. In total, 11 fibers were compared regarding their conductivity and an average resistivity of 45 ± 21 Ω was found.

The good adherence of AgNWs to cellulotic surfaces has already been reported by Kopeinik et al (2015) on AgNW-decorated wood-pulp fibers. There, based on evidence from infrared spectroscopy it was suggested that the cellulose surface strongly interacts attractively with the AgNW surface. In addition, electron microscopy images also showed a close alignment of the AgNWs along the wood-pulp fiber surface (Kopeinik et al 2015) similar to the effect seen for viscose fibers in the SEM image in figure 3.
Therefore, we assume a similarly strong interaction for the AgNW adsorption to viscose fibers in the present case.

Due to practical reasons for electrical measurement (avoiding short-circuiting between the core and outer electrode during contact) the precise design of the present viscose-based photovoltaic fibers was engineered in an overlapping structure, as shown in a schematic picture in figure 5, and described as follows. The viscose fiber, indicated by the white region, is not covered with AgNWs for its entire length, but only one third of it (in the laboratory test device about 1 cm), represented by the light gray area surrounding the right-hand part of the fiber in the sketch. The PEDOT:PSS hole-conductor coating, indicated in yellow, leaves some part (2 mm) of the AgNW area free (on the right of the sketch) but covers a (2 mm) wider part of the viscose fiber beyond the extent of the AgNW-coated area on the other end (on the left of the sketch). The subsequently applied organic semiconductor coating, indicated by the green area, is again shifted (1 mm) to the left, starting behind the PEDOT:PSS layer on one end, but extending beyond it on the other. Finally, the outer aluminum electrode is again deposited shifted (1 mm) to the left, indicated by light gray on the left side of the sketch. With this design, the aluminum coating only partially overlaps with the AgNW region underneath the semiconductor/hole-conductor layer of the photovoltaic fiber. Safe electrical contact to the NW network can thus be established on the bare AgNW end of the viscose fiber (indicated by the dark gray spot and the microprobe needle on the right side of the sketch). Safe contact to the outer aluminum electrode is made on that part of the fiber that is not overlapping with the AgNW region (indicated by the dark gray spot and the microprobe needle on the left side of the sketch). This leads to a total device length of about 15 mm. The overlap has to be chosen wisely, because if the distance for the charges generated in the semiconductor to the respective electrodes is too far, this is disadvantageous for the performance of the photovoltaic cell, due to the higher recombination probability of electron–hole pairs leading to low efficiencies.

The thickness of the organic hole-conductor/semiconductor coating on the photovoltaic fiber device is, at about 1 μm, relatively large compared to planar devices with usually 100–200 nm, but was required here to effectively cover the AgNW network. The TEM images in figure 6 show exemplarily cross-sections of a sliced fiber with the coating configuration viscose/AgNW/P3HT:PCBM/gold. PEDOT:PSS and Al have been disregarded for facilitated TEM preparation.

In figure 6(a) the collapsed viscose fiber is clearly visible in the lower half of the image. Above one can see the detached coating, which has been lifted off from the fiber by vacuum. The AgNWs are visible in the cross-section as dark spots within the brighter organic semiconductor matrix and on top a thin dark layer originating from the TEM gold coating. The detached coating reflects clearly the outer surface structure of the original viscose surface, like a fingerprint. It is also visible from figure 6(a) that the coating is not homogeneous, but varies in thickness between 0.7 and 5 μm due to the folds of the collapsed viscose fiber, leading to higher deposition in ‘valleys’ than on higher regions. Such a thicker coating from a trench of a viscose fiber is seen as magnified cross-section in figure 6(b). In addition, here the surface topology of the viscose fiber is still preserved, visible in the former viscose–organic contact area at the bottom of the coating. Further, one can see that the distribution of AgNWs in the coating, visible as dark spots, is restricted to a certain height in the mantle, indicating that they do not stick out or float when follow-on liquid materials are applied. This suggests that a homogeneous coating of a specific height of around 1 μm
should be sufficient to reliably cover the AgNWs and achieve reproducible fiber devices, even more on non-collapsing viscose fibers.

To determine the electrical device properties, current–voltage (I–V) characteristics measurements of the photovoltaic fibers were recorded in the dark and under 0.54 mW cm⁻² LED white light illumination in an inert atmosphere.

Exemplarily, the dark and photocurrent characteristics obtained for one photovoltaic fiber are shown in figure 7 in one semi-logarithmic plot to enhance visibility. On first sight, both the dark current (represented by the black squares) and photocurrent (represented by the red circles) curves look quite symmetric, indicating ohmic behavior caused by a considerable contribution of leakage currents of the order of 2 × 10⁻³A at zero bias. These are most likely caused by thickness inhomogeneities in the semiconductor coating on the collapsed viscose fiber surface, as mentioned above. However, a detailed analysis reveals that the devices still exhibit reasonable contributions of diode behavior, visible as a subtle difference between forward- and reverse-bias current, seen in the dark, but even more pronounced in the photo- I–V plot. But even more important is the fact that the photovoltaic fiber shows signs of a light-induced current, visible as an open-circuit voltage (V_OC) different from zero, which would not be observed in a shortened cell, because of a total voltage drop at the shorts. Effectively, the presented exemplary photovoltaic fiber shows a V_OC of 0.08 V and a short-circuit current (J_SC) of −0.5 mA cm⁻², leading to a value of 0.29 for the fill factor (FF). Taking the LEDs’ light intensity into consideration, this results in a power conversion efficiency η of 0.023%. For calculation of current density, the photoactive device area was estimated with 0.025 cm², which is half of the viscose fiber’s surface area (using the mean fiber diameter and the length of the device). Obtaining such solar cell key values for a not yet optimized photovoltaic viscose fiber device and at relatively low light intensity is quite satisfactory. For comparison, optimized planar devices of this configuration deposited on ITO glass usually deliver V_OC of a maximum 0.66 V, J_SC between 8 and 12 mA cm⁻² and FF between 0.50 and 0.65, under simulated solar conditions (AM1.5G and 100 mW cm⁻² (Dang et al 2011), leading to a power conversion efficiency η of around 5%. We note that in the present case direct comparison of the dark current and photocurrent of the fiber devices is difficult, due to instabilities of electrical contact when touching the quite delicate fiber with the comparatively macroscopic microprobes. In consequence there are inevitable contact resistance changes; accordingly the expected shift in reverse-bias current due to photogenerated charges is not apparent in the plot in figure 7.

For a single cellulose fiber photovoltaic cell as a proof-of concept, the results are remarkable.
However, one clear culprit is the inhomogeneity of the semiconductor coating due to the collapse of the viscose fiber structure, leading to losses by not insignificant dark current contributions. A stable viscose core fiber should solve that problem and make way for advancement of the suggested concept for R2R production of photovoltaic fibers based on a common industrial cellulose fiber.

Conclusions

In this paper, a proof-of-concept is given, showing that the fabrication of functional photovoltaic fibers based on common industrial cellulose fibers is feasible. This was demonstrated by preparation of short few centimeter long prototype photovoltaic fibers, built on commercially available hollow viscose fibers. Thereby the application of a AgNW surface network on the fiber forms the core electrode. This is followed by coatings of PEDOT:PSS for the hole-conductor and P3HT:PCBM for the photoactive semiconductor layer, all materials that are frequently used in common planar organic solar cells. The presented photovoltaic fibers show quite remarkable characteristics with fill factors and short-circuit current densities of comparable dimensions to planar devices of this configuration, taking the relatively low light intensity during testing into account. Only the open-circuit voltage shows considerable weakness: losses due to considerable amounts of leakage current as a consequence of inhomogeneous semiconductor surface coverage, caused by the collapse of the chosen hollow viscose fiber. However, this obstacle can be solved by the choice of a viscose fiber with a flat, compact or reinforced structure. The stable dense structure of closely adhered AgNWs on the viscose fiber surface enable reproducible core electrodes, which enable reproducible uniform follow-on coatings with a semiconductor mantle of limited thickness. The presented concept can be adapted easily to large-scale R2R processing because all the materials used are solution-processable and can be applied by dip-coating, spraying or printing. Fabrics made of such photovoltaic fibers could be integrated into smart clothing to generate the power for many wearable electronic devices.

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