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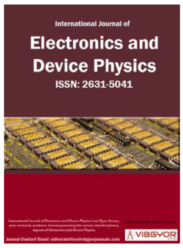

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Design and Construction of Home-Made Spin Coater for OLED Production

Zafer Gültekin, Mürsel Alper*, Cengiz Akay and M Cüneyt Hacıismailoğlu

Department of Physics, Science and Art Faculty, Bursa Uludag University, Turkey

Abstract

This study devotes to the design and fabrication of a spin coating equipment using locally available materials for the deposition of uniform thin films on several substrates and also the production of organic light emitting diodes (OLEDs) using this home-made device. The equipment consists of a brushed direct current (BDC) motor with high speed, chuck (substrate holder) and regulated power supply. The system has manual control, wide spin speed ranging from 500 to 6500 revolutions per minute (rpm), spin speed stability and compact size. The spin speed stability has been determined by a tachometer against the spinning time. The performance of our equipment was tested for coating Poly (3,4-ethylene dioxythiophene)-poly (styrene sulfonate) (PEDOT:PSS) polymer. Three different PEDOT:PSS thin films were successfully deposited on glass substrates that are standard products, using the spin speeds of 1000, 2000 and 3000 revolutions per minutes (rpm). Also, under similar experimental conditions, PEDOT:PSS films were prepared by a commercial spin coater. These two group samples have been characterized by optical and atomic force microscope (AFM) measurements and compared with each other. Furthermore, in order make an OLED, a multilayer system consisting of, in sequence, PEDOT:PSS, poly[2-methoxy-5-(2`-ethylhexyloxy)-1,4-phenylenevinylene] (MEH-PPV) and Tris (8-hydroxyquinolinato) Aluminium (Alq3) layers was fabricated on commercial indium tin oxide (ITO)/glass substrates using both our home-made and a commercial spin coater. The top layers of all samples were covered by the Al using thermal evaporation. The current-voltage (I-V) characteristics of OLED samples prepared by two different devices were examined. The obtained data showed that OLEDs could successfully be produced by our home-made spin coater.

Keywords

Spin coating, Organic synthesis, PEDOT:PSS film, Semiconducting polymer, OLEDs

Introduction

Today, thin films such as metallic, semiconductor, on organic layers, are fabricated using several deposition methods such as physical vapour deposition (sputtering), chemical vapour deposition, atomic layer deposition, laser ablation deposition, electrochemical deposition, spin coating and sol-gel. Among them, one of the most important methods is the spin-coating technique, which is a simple, safe, inexpensive and easy to

***Corresponding author:** *Mürsel Alper, Department of Physics, Science and Art Faculty, Bursa Uludag University, 16059 Görükle, Bursa, Turkey*

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use at room temperature [1-4]. Also, this method offers homogeneous and smooth thin film layers on substrates such as single crystal, glass, polymer and semiconductor wafers and are successfully used to obtain the films containing metallic, metal-oxide, magnetic, polymeric materials or biomaterials [5,6]. Therefore, the technique has attracted much attention in the fabrication of the layers for micro- and nano-devices, optoelectronics, magnetic disk, sensors etc., [7,8].

The spin coating technique is simply based on coating and fabricating uniform thin films by rotating substrate and thin film solution with a certain angular velocity. The solution containing the film material is dropped on the centre of a substrate, which is rotated at high speed such as 5000 revolutions per minute (rpm) by a DC motor. During the rotation of the substrate, centrifugal force causes the solution to spread to the edge of the substrate, and thus a thin film layer of the solution is formed on the surface of the substrate. Many properties of the films including the film thickness may changes depending on the factors such as the nature of the solution (viscosity, drying rate, composition etc.), the spin speed and the spin time.

Nowadays, there have been many efforts to build up the low cost spin coater [9-15] in order to produce several thin films. A simple way of making a home-made spin coater is to use an Arduino microcontroller and a DC motor. The Arduino is a platform that is often preferred, due to both inexpensive and easy to find itself and its components. The DC motor is used to rotate the substrate. In spin coating process, the film deposition is basically achieved at the four main stages: Firstly, the liquid containing materials to be deposited is dropped on the substrate, secondly the substrate is accelerated to the desired speed. At the third step, the steady spinning of the substrate is provided at this speed. The fourth is the evaporation of the solution on the substrate surface, which occurs in all these stages. The substrate is placed in a holder called a chuck, which is used to keep stable the substrate. After dropping the coating liquid, the substrate is rotated at the desired speed. During the rotation of the substrate, centrifugal force acts on the coating liquid depending on the increasing speed. By the action of this force, a wave that moves out from the centre is formed. As the wave progresses, the coating liquid becomes thinner [4], its viscosity increases and the solution fluidity decreases [10]. In the spinning phase at a constant speed, excess solvent continues evaporating from the solution on the substrate. Viscosity increases further, and centrifugal force cannot move the solution over the surface any further. Ultimately, a homogeneous, dry and thin-film layer is formed from the centre to the edges [11]. In the spin coating, the solvent evaporation is an important, so the solvent should be selected from materials that evaporate rapidly at room temperatures, such as acetone, toluene, chlorobenzene, and chloroform. The drying rate of the liquid on the substrate is related to environmental conditions, like air, as well as the evaporation of the volatile liquids used in the solution. While coating, airflow and turbulence on the substrate should be minimized or at least kept constant [12]. The film thickness is determined by the spinning speed and the coating time according to the evaporation rate of the liquid [13-15].

An organic light emitting diode (OLED), which is a solid state semiconductor device, is made of organic electroluminescent materials and emits light when an electric current flows through it. Since OLEDs have more efficient semiconducting light sources than all conventional lightning sources they are of an importance economic potential for display and lightning applications. Furthermore, they have some advantages such as low voltage, low power consumption, more flexible, thinner, lighter, compared to semiconductor light emitting diodes (LEDs). Basically, OLEDs are made using an optically transparent substrate material such as glass and organic semiconductor materials such as poly[2-methoxy-5-(2-ethylhexyloxy)-1,4-phenylenevinylene] (MEH-PPV), polyethylenimine ethoxylated (PEIE) and Poly (3,4-ethylene dioxythiophene) -poly (styrene sulfonate) (PEDOT:PSS) or semiconducting metal oxide films (CuO, NiO CoO). In the fabrication of OLEDs, these organic films and/or metal oxide films are sandwiched between a transparent indium thin oxide layer (anode) and a non-transparent reflective metal such as aluminium Al (cathode) and provide transporting holes from the anode and electrons from the cathode. The conduction in the OLED is provided by the delocalization of π electron. When a voltage is applied to OLED, the conduction level in the organic films changes from insulator to conductor. Holes are injected

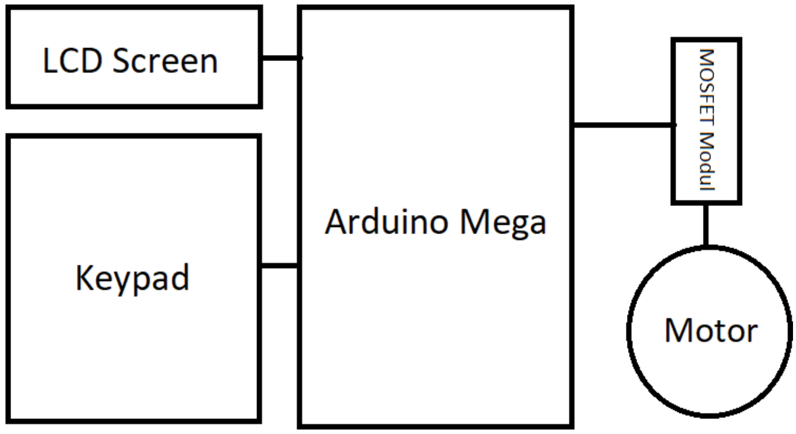

Figure 1: Schematic diagram of the spin coater.

from the anode into the conductive layer through highest occupied molecular orbital (HOMO), while electrons flow from the cathode into the emissive layer (EML) through lowest unoccupied molecular orbital (LUMO). Therefore, the electron-hole pair will recombine, form exciton (a bound electro-hole pair) and give rise to the photon emission, which produce light in the form of electroluminescence [16,17].

In this study, firstly we have developed a novel programmable, low cost, spin coating device using Arduino and a brushed DC motor (BDC) and tested by measuring spin speeds. The system has manual control, wide speed range (from 500 to 6500 rpm), spin speed stability and compact size. To confirm the performance of our device, it was used to produced three PEDOT:PSS films on glass substrates and investigated the effects of spin speed on the optical transmittance and surface morphology of these films. Similar films were also produced by a commercial spin coater. The results obtained from both devices were compared. Secondly, OLEDs consisting of multiple layers have been fabricated using both our own and commercial device. The OLED layers have been arranged in sequence, commercial ITO/glass as a substrate, where indium tin oxide (ITO) serves as anode (+), PEDOT:PSS as hole transport layer HTL), MEH-PPV as active layer, Tris (8-hydroxyquinolinato) Aluminium (Alq3) as electron transport layer (ETL), and aluminium as cathode (-). Al contacts (layers) were made by vacuum thermal evaporation (VTE). The I-V (current-voltage) characteristics of the OLEDs produced by both commercial and home-made spin coater have been investigated and compared with each other. The obtained results revealed that the OLEDs with good quality could be fabricated by our spin coater.

Design and Construction of the Spin Coater

In this work, one of our main purposes was to construct a home-made spin coater on the based on brushed DC (BDC) motor. The components used in the spin coater unit are a DC motor of 12-18V (Mabuchi C9000-60005), AOD4184A 40 V N-Channel Metal Oxide Semiconductor Field Effect Transistor (MOSFET) module to drive this motor, a liquid crystal display (LCD 16 × 2), a power supply (12 V, 3 A) and Arduino-mega microcontroller, which controls the whole system by our own software. All these components are commercial products from www.direnc.net, except for the BDC motor, which was removed from an HP brand printer. The schematic diagram of the whole system is shown in Figure 1.

The substrate is placed on the chuck mounted on the axle of the BDC motor. While the BDC motor rotates, the substrate also rotates along with the chuck. The coating solution is dropped on the substrate by a syringe. The centrifugal force on the solution droplets leads to their homogenous distribution on the surface of the substrate.

The desired spin speed (rpm) and the coating time (s) are entered by keypad. The microcontroller simultaneously provides the motor to be driven and controls the spin speed of the motor using the pulse-width modulation technique, which is known as pulse-width modulation (PWM) in practice. The picture of the developed spin coater is given in Figure 2.

The coating unit of our spin coater consists of

Figure 2: Home-made spin coater.

Figure 3: a) Chuck; b) Tachometer; c) Home-made tachometer.

a chuck (Figure 3a) and a pulley arrangement that connects it to the shaft of the motor. In our system, the substrate to be coated is directly placed on the chuck, while in some studies, either a vacuum substrate holder or a double-sided tape to fix the substrate to the DC motor is used. The use of double-sided tapes cannot be considered as a very reliable way of producing uniform thin films. On the other hand, vacuum substrate holders cause bending, especially in the middle parts of flexible substrates [18].

During spinning, in order to avoid the throw of the substrate, a 26 mm × 26 mm × 2 mm cavity on the chuck was made. Also, the escape channels at the corners of the cavity were opened to go out the excess solution on the substrate. This design can provide better homogeneous coatings on flexible substrates and improve the film quality.

Performance Measurements of Spin Coater

The performance of our device were tested by both a commercial (Figure 3b) and home-made tachometer (Figure 3c). As seen from the Figure 4, the values measured by both tachometers are

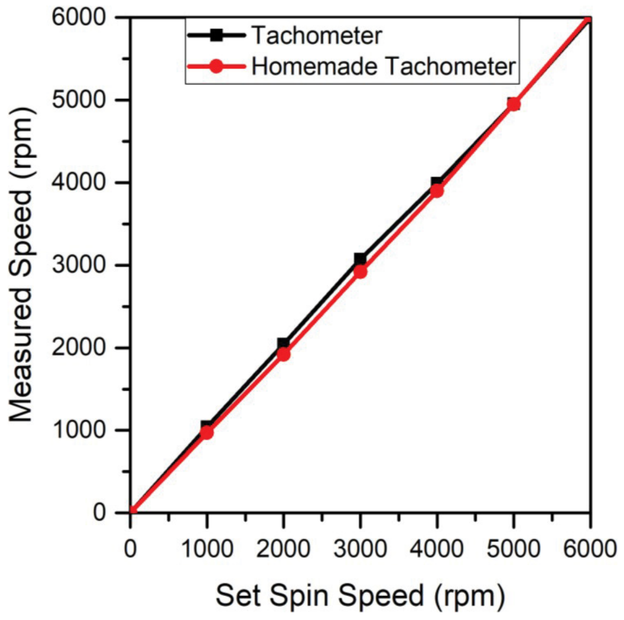

Figure 4: Comparison of the measured spin speed with the set spin speed.

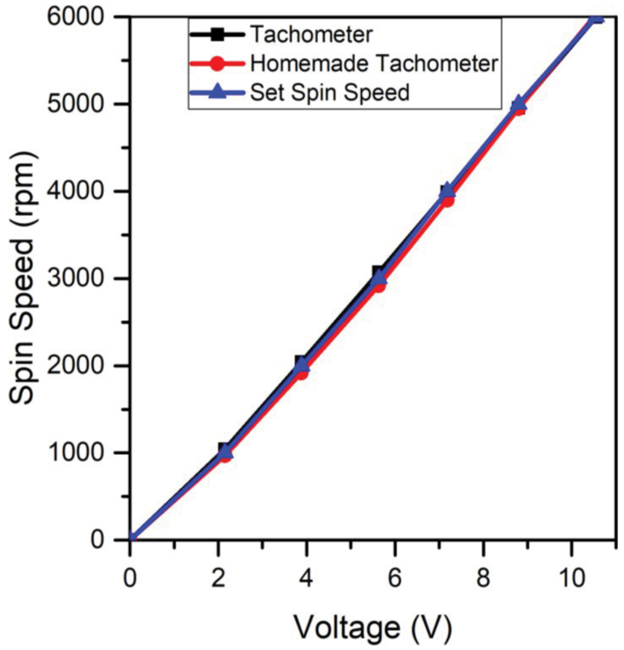

Figure 5: Variation of spin speed with DC voltage.

directly proportional with the spin speeds set in our device and in agreement very well with each other. Furthermore, the variation of the set and measured spin speeds with the applied DC voltage is given in Figure 5. It is clearly seen from the Figure 5 that as expected, all set and measured spin speeds increase linearly as the voltage increases. This is an indication that our device has a good performance and calibration.

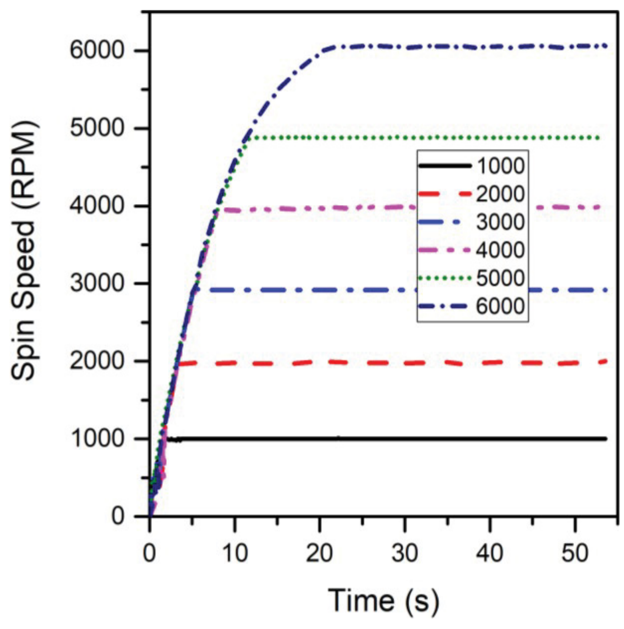

Figure 6: The change of spin speed with time.

The change of the spin speed with time for different spin speeds is given in Figure 6. At the beginning, the spin speed increases with time and then becomes stable. Note that the reaching to a stable speed value takes longer time as the applied voltage, and hence the spin speed increase. For example, for a spin speed of 1000 rpm, the speed stability achieves in ~ 2 seconds, while for a spin speed of 6000 rpm it takes ~ 20 seconds.

In addition, our spin coater offers one more useful feature. When the motor starts to spin suddenly, the occurring high centrifugal force causes immediately the dispersion of the solution dropped on the substrate. Such a situation makes it difficult the formation of a thin and uniform film layer on the substrate. When the substrate starts to spin, the solution on it will also begin to dry, during this time the acceleration must be controlled accurately. It was reported that 50% of the solvent in the solution evaporates in the first few seconds of the process [19]. To avoid such a situation, the speed must be provided to reach the set speed value gradually with a variable acceleration and after reaching a stable spin speed, the substrate must be rotated at this constant speed. The microcontroller monitors the entered spin time, starts counting down and stops the engine calmly at the end. Figure 6 and Figure 7 show the time that is required to reach the speed values in the range of 1000-6000 rpm and the square wave pulse widths that provide this acceleration, respectively.

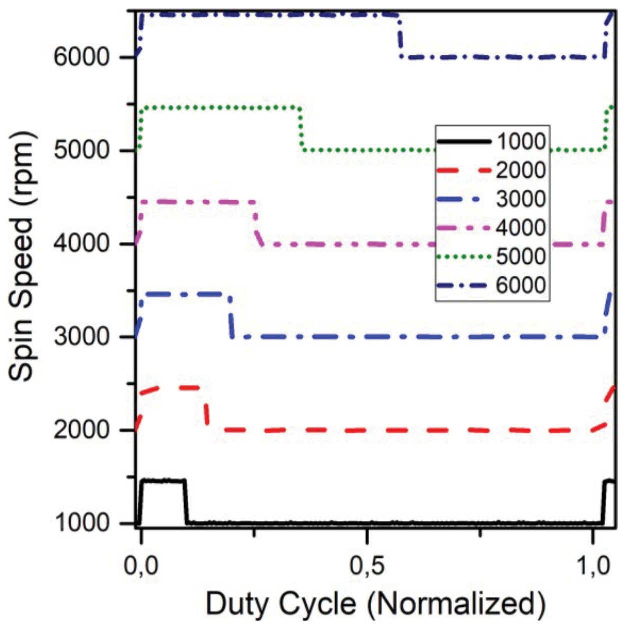

Figure 7: Variation of spin speed with the normalized duty cycle.

The common feature of both graphs is that an exponential behaviour indicates according to the time. This is a good behaviour, because the MOSFET switching time is in order of 10-9 s, which causes very sudden acceleration. For this reason, driving the motor with low on/off rates first will increase the coating efficiency. However, in spin coaters that do not have this feature, it is waited for a while to stabilize the spin speed before dropping, and then the solution is dropped. If the contact time of the drop that suddenly touches the surface becomes too short to stick, the quality of the coating may reduce. According to the results in Figure 6 and Figure 7, the spin speeds stays stable after a certain time, indicating that our spin coater is suitable to obtain thin homogeneous films.

PEDOT:PSS Thin Films Prepared by Spin Coater

In OLED fabrication, selection of material is a critical parameter for both their efficiencies and lifetimes. The one of the commonly used materials is organic PEDOT:PSS layers. So, firstly, PEDOT:PSS thin films were produced by both the developed and commercial spin coating unit and characterized. In the OLED fabrication, PEDOT:PSS thin films is generally used to improve the contact properties of ITO. They efficiently accumulate and transmit the holes. Before deposition of PEDOT:PSS layers, glass substrates were cleaned chemically using

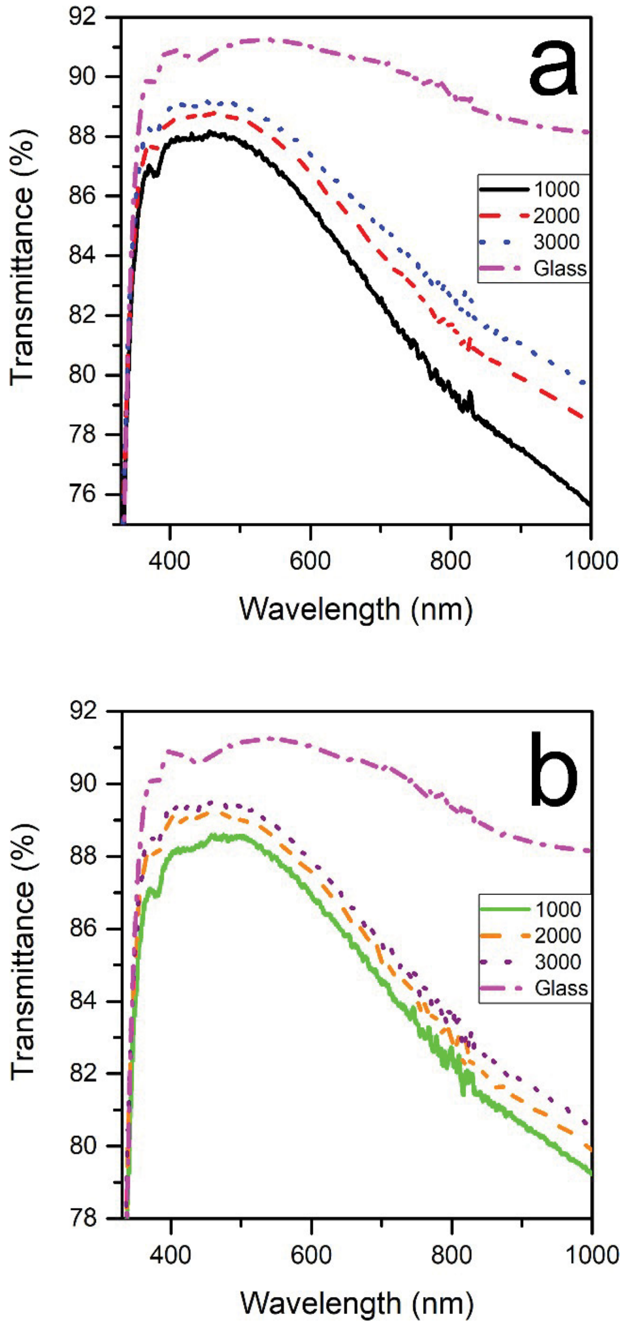

Figure 8: Transmittance curves for glass and PEDOT:PSS films produced on glass by: a) Home-made and; b) Commercial spin coater.

acid and distilled water. PEDOT:PSS solution was filtered through a 0.45 µm aperture Polyvinylidene Fluoride (PVDF) filter. Then, a group of PEDOT:PSS thin films were deposited on glass substrate using our spin coater equipment. These samples were produced at the spin speeds of 1000, 2000 and 3000 rpm for a 60-second spin period. Also, under similar experimental conditions, the second group samples were prepared using a commercial spin coater (Laurell Technologies WS-650-8B).

The transmittances of the PEDOT:PSS films in each group and a blank sample without coating (glass) were measured with a UV spectrophotometer (SHIMADZU UV-2600). Figure 8a and Figure 8b shows the variation of the transmittance with the wavelength for the films produced by our own and commercial device, respectively. As seen from the figure, for all PEDOT:PSS samples, the transmittance values increases rapidly to 400 nm, remains almost constant between 400-500 nm and then the decrease monotonically. Note that all samples have similar transmittance curves. As expected, the blank sample (glass) indicates the highest value, for which the transmittance value is over 90%. On the other hand, it is clearly seen that as the spin speed increases, the transmittance increases, for example, the transmittance values of the films prepared by an 1000 rpm of the spin speed are less than those of the films by 3000 rpm. This can be attributed to the decreases of the film thickness at the high spin speeds, because as the spin speed increases the film thickness decreases, as reported in many studies [4,10].

In spin coater technique, in general the film thickness is inversely proportional to the square root of the spinning speed of the chuck [20]. For PEDOT:PSS films prepared by our spin coater, the variation of the film thickness with the spin speed of the disk is drawn in Figure 9. As seen, the thickness decreases with increasing the spin speed. This agrees with the reported curves [21,22] and confirms also by the increase of transmittance values with increasing spin speed in Figure 8.

Figure 10a shows the photographs of the PEDOT:PSS films coated on glass substrate at 1000, 2000 and 3000 rpm and uncoated glass. As seen from the figure, the colour contrast of the samples changes on depending the spin speed, due to different thickness. The colour difference between the samples is very clear. On the other hand, in order to more clearly see the colour/thickness difference between the samples, a ImageJ software was used [13]. The software calculates the black and white balance by assigning contrasts to images loaded in memory in grayscale mode, in which each pixel corresponds to a contrast between 0 for black colour and 255 for white colour. Using this software, the contrast of each pixel in each photograph was calculated for these samples. Figure 10b shows the grayscale contrast values versus the pixel numbers.

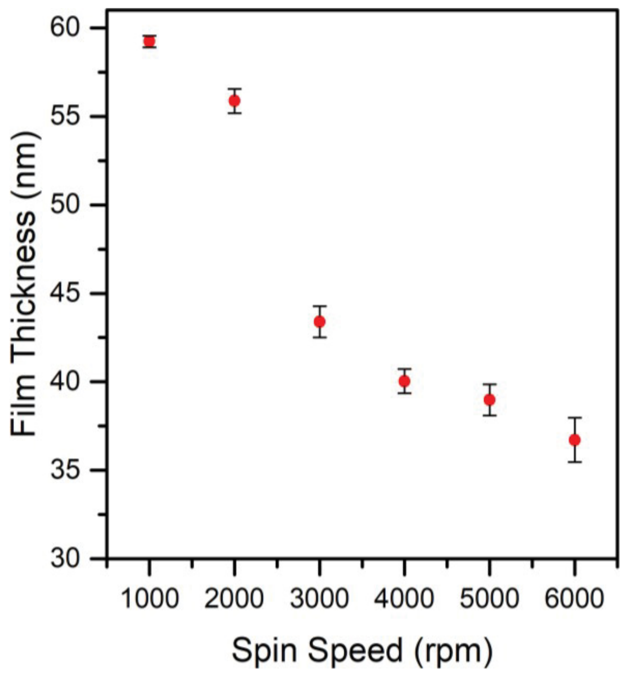

Figure 9: Variation of the film thickness with spin speed.

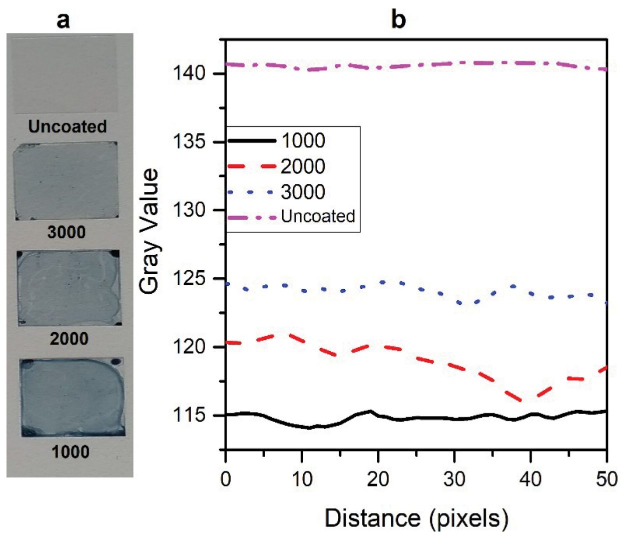

Figure 10: a) Photographs of samples; b) Grayscale values vs. distance (pixels) for the samples at 1000, 2000 and 3000 rpm, and uncoated glass.

It is clearly seen from the figure that the thicknesses of all the samples could easily be distinguished by the values of the grayscale contrast.

The surface morphology and surface roughness of the PEDOT:PSS films deposited on glass substrate using both home-made and commercial spin coating unit were examined by an atomic force microscope (AFM). Figure 11(a-c) for home-made and Figure 11(d-f) for commercial spin coater show

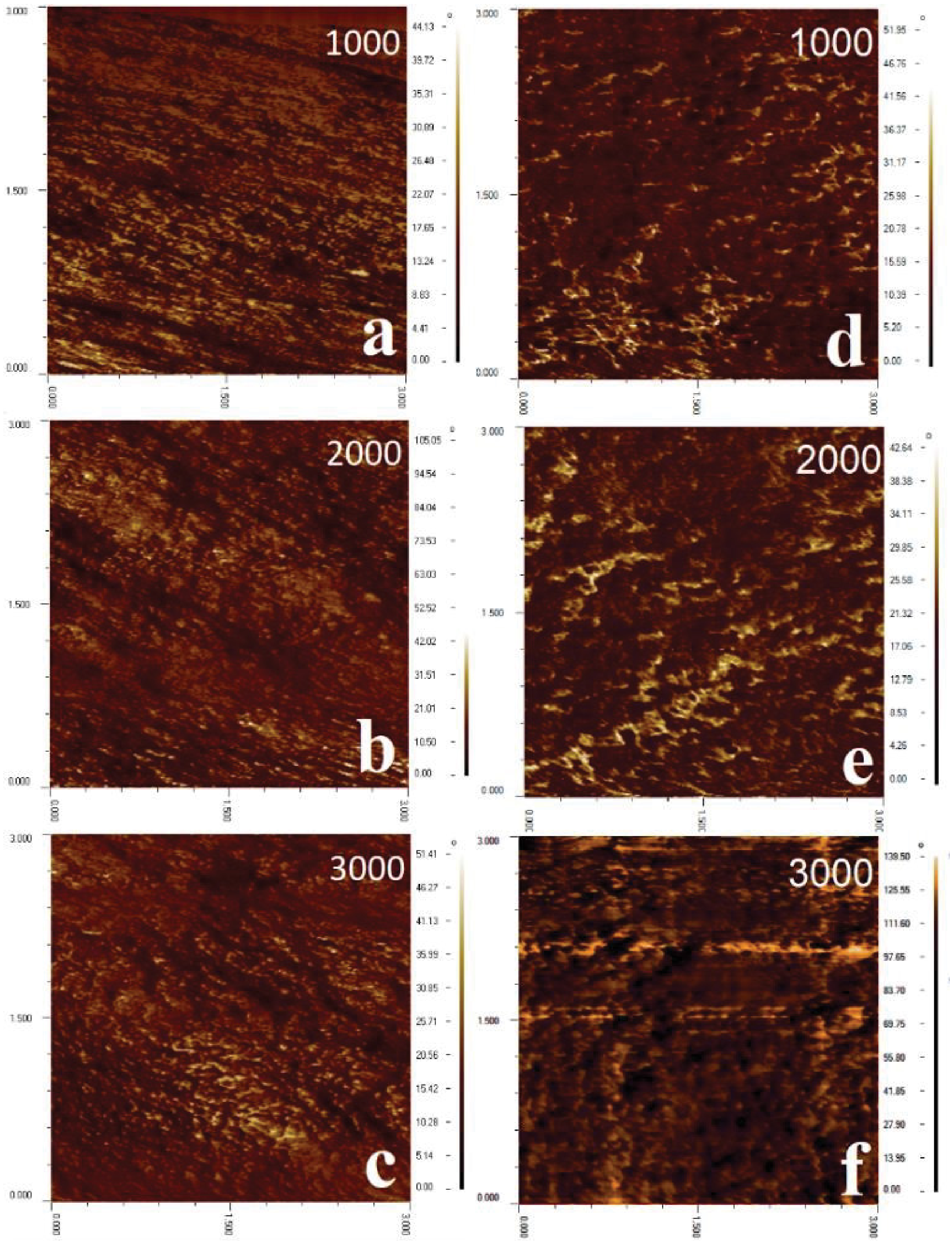

Figure 11: AFM images of PEDOT:PSS films produced by (a-c) Home-made (d-f) Commercial spin coater.

the AFM pictures of these films deposited at 1000, 2000 and 3000 rpm, respectively. The images were obtained by scanning an area of 3 × 3 μm^2 from the middle parts of all samples. From the figure, it is obviously seen that all samples have similar surface morphology. However, as the spin speed increased, the root mean square (RMS) values that is a measure of the surface roughness increase, because for home-made spin coater, the RMS values was determined to be about 12 nm, 15 nm and 21 nm for 1000, 2000 and 3000 rpm, respectively, while for commercial device, these values become about 13 nm, 16 nm and 21 nm, respectively. This suggests that the film roughness increases with increasing spin speed for both spin coater and is agreement with each other. The surface roughness is one of the important parameters for the production of OLED, because it affects the conduction of current within the layers of OLED [23,24]. By considering these results, for the OLED production, the coating speed and time were selected as 1500 rpm and 60 s, respectively.

Use of Our Spin Coater to Produce OLED

After obtaining the desired performance from our spin coater unit and fabricating successfully the PEDOT:PSS films on glass substrates, our spin coater was tested to produce a OLED structure.

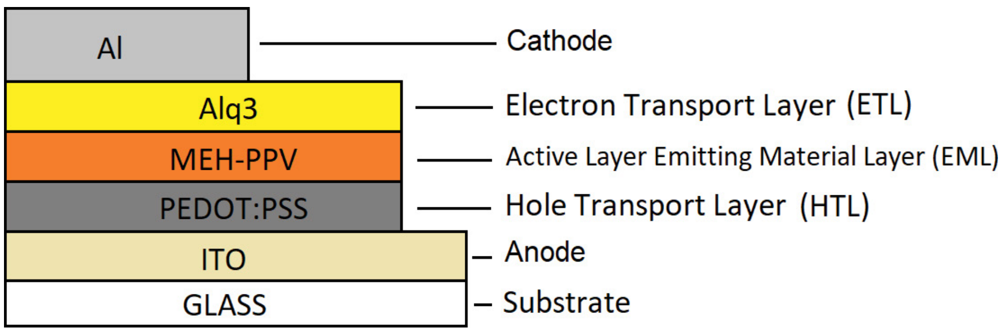

Figure 12: Structure of the produced OLEDs.

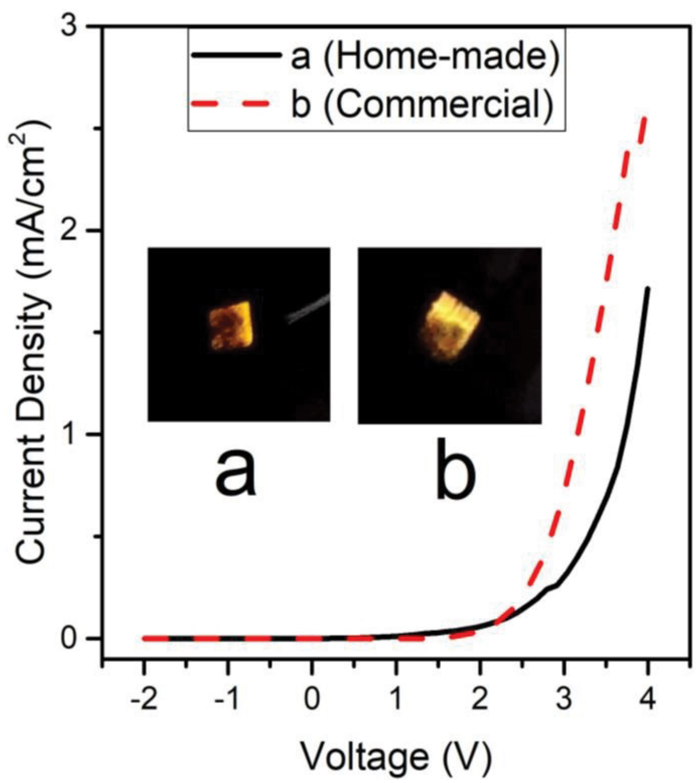

Figure 13: I-V characteristics of OLEDs produced by home-made and commercial devices. The inset is the photographs of OLED samples: a) Home-made device; b) Commercial device.

Two OLED samples were prepared using both our own device and commercial spin coater. The OLEDs consist of the layers with PEDOT:PSS as HTL on ITO/glass, MEH-PPV as EML, Alq3 as ETL and Al layers as cathode. A schematic structure of the produced OLEDs is given in Figure 12. All organic layers were made by spin coaters and Al contact by vacuum thermal evaporation.

For prepared OLED samples, DC current vs. voltage measurements were performed using a Keithley 2400 source meter. The voltage across the OLED was applied from -2.0V to +4.0V by a step of 0.1V. Figure 13 shows the current density (J)-voltage (V) curves of the OLEDs produced by our (a) and commercial (b) spin coater. As seen from the figure, two different regions for two samples are observed from the J-V measurements. The first region ranges from -2V to +2 V. In this region, the current is almost constant. After this point, the current begins to increase rapidly. Note that the turn-on voltage for both OLED is about 2.5-3 V, but the current density value for the OLED produced by commercial spin coater is larger than that produced by our own device. The inset in the Figure 13 shows the illumination from OLEDs produced by our device (a) and commercial device (b). Samples produced with the commercial device have both more homogeneous and more bright light emitting, probably due to more efficient recombination. In the pictures, while the bright spots correspond to photon emitting, the dark spots represent phonons that arises from heating of OLEDs.

Conclusions

We have designed and constructed a home-made spin coater with low cost. For the fabrication of the spin coating unit, a BDC motor with high speed, chuck and regulated power supply which are locally available have been used. The equipment presents a wide range of speed (from 500 to 6500 rpm) and has high spin speed stability. The performance of the equipment was tested by making the spin speed and time measurements and also compared with the results of a commercial spin coater. Both our own device and the commercial spin coater were successfully used to fabricate PEDOT:PSS thin films on glass substrates. The PEDOT:PSS films produced by both of them were examined optically and morphologically. It was observed that two group samples have similar properties. Furthermore, the suitability of our device was tried to fabricate OLED structures. It was observed that the OLED samples could successfully be prepared by our device. Also, similar OLED samples were produced under the same experimental conditions by the commercial device. The current-voltage measurements for two group samples were made at room temperature. It was found that the OLEDs produced by both our and commercial device have similar J-V curves and a turn-on voltage of about 2.5 V. The results revealed that our home-made spin coater could be used to fabricate OLEDs.

Conflict of Interest

The authors declare that we have no financial and personal relationships with other people or organisations that can inappropriately influence our work, there is no professional or other personal interest of any nature or kind in any product.

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