Semi-Transparent Organic Photovoltaic Cells with Dielectric/Metal/Dielectric Top Electrode: Influence of the Metal on Their Performances

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Abstract: In order to grow semi-transparent organic photovoltaic cells (OPVs), multilayer dielectric/metal/dielectric (D/M/D) structures are used as a transparent top electrode in inverted OPVs. Two different electrodes are probed, MoO$_3$/Ag/MoO$_3$ and MoO$_3$/Ag/Cu:Ag/ZnS. Both of them exhibit high transmission in visible and small sheet resistance. Semi-transparent inverted OPVs using these electrodes as the top anode are probed. The active organic layers consist in the SubPc/C$_{60}$ couple. The dependence of the OPV performances on the top electrode was investigated. The results show that far better results are achieved when the top anode MoO$_3$/Ag/MoO$_3$ is used. The OPV efficiency obtained was only 20% smaller in comparison with the opaque OPV, but with a transparency of nearly 50% in a broad range of the visible light (400–600 nm). In the case of MoO$_3$/Ag/Cu:Ag/ZnS top anode, the small efficiency obtained is due to the presence of some Cu diffusion in the MoO$_3$ layer, which degrades the contact anode/organic material.

Keywords: semi-transparent organic photovoltaic cells; dielectric/metal/dielectric transparent electrode; top transparent anode; MoO$_3$/Ag/MoO$_3$; inverted planar heterojunction; SubPc/C$_{60}$ active layers

1. Introduction

The growing interest in organic photovoltaic cells (OPVs) is due to the fact that they possess some specific advantages such as light weight, intrinsic flexibility, and possible semi-transparency of organic thin films [1]. More specifically, semi-transparent OPVs attract strong interest due to the efforts currently directed toward building integrated photovoltaics (BIPVs). Recently, much research has focused on improving OPV efficiency [1–4], but some applications, such as BIPV, induce the use of semi-transparent OPVs, which are less efficient than the usual opaque OPVs because they have to transmit a significant amount of visible light [5,6]. Therefore, for semi-transparent OPVs, it is not appropriate to use optimal organic layer thickness. It is known that, the diffusion length of excitons being smaller than the organic layer thickness necessary to absorb light, the geometry of bulk heterojunction (BHJ) was used with success [7–11], while in the case of planar heterojunctions (PHJs), the excitons created too far from the electron donor/electron acceptor interface are lost, which, contrary to the case of BHJs, limits the thickness of PHJs and, therefore, their efficiency [12]. However, as said above, in the case of semi-transparent OPVs, we must not use the optimum thickness to preserve some OPV transparency, so we chose the PHJ–OPV configuration to grow semi-transparent devices. Such configuration allows for using small molecules and deposition under vacuum. Small molecules have a number...
of benefits such as easy preparation and purification and a unique final molecule that prevents the problem of batch-to-batch reproducibility [13–15]. Furthermore, the thermal deposition technique allows for easy fabrication of multilayer devices with precise control of the thickness of each layer [16,17]. Usually, the electrodes of opaque OPVs consist of a transparent conductive electrode (TCE), often an ITO (Indium Tin Oxide) thin film deposited onto a glass substrate, and a metal electrode, often aluminum. The high reflectivity of Al allows light to be reinserted into the organic layers. To achieve semi-transparent OPVs with acceptable performances, the main challenge is the substitution of a transparent electrode with high conductivity with a metal electrode [18]. Different solutions are possible, among them, dielectric/metal/dielectric (DMD) structures [19,20]. They possess quite equilibrated optical and electrical properties; moreover, they are easy to realize given the technique we have chosen to use via successive thermal sublimation/evaporation. In DMD, M allows obtaining small sheet resistance, but its high reflectivity strongly penalizes the light transmission. Thus, to obtain the transmission of the visible light, it is necessary to sandwich the M layer between two high refractive index dielectrics. MoO$_3$ is one of the possible dielectrics; it is well known that it is very efficient as a hole-transporting layer (HTL) at the anode/electron donor interface [21,22]. Therefore, in the present work, MoO$_3$ was chosen as the dielectric. As for the metal, Ag is commonly selected because it exhibits the highest metal conductivity coupled with the lowest absorption in the visible range among the metals [23]. Nevertheless, Ag is quite expensive, and it would be of high interest to use a cheaper metal. Therefore, we also used Cu, whose optical and electrical properties are very close to those of Ag. Unfortunately, Cu tends to diffuse into transition metal oxides such as MoO$_3$ [24], so we tried to improve DCuD stability through the use of ZnS dielectric and Cu:Ag alloy [25]. After optimization, these DMD electrodes are used as top electrodes in inverted OPVs, such as ITO/Alq$_3$/C$_{60}$/SubPc/DMD. We chose the inverted OPV configuration due to its better stability [26]. The study shows that, even after the DMD structure optimization, the best results are obtained with DagD top electrodes. In comparison with the reference OPV, i.e., with Al as the top electrode, the efficiencies of the semi-transparent OPVs are only 20% smaller, but with a transparency of nearly 50% in a broad range of the visible light (400–600 nm).

2. Materials and Methods

The deposition and characterization techniques have already been described, and they are recalled in Supporting Information S1 and S2.

The inverted OPVs were deposited under vacuum (see Supporting Information S1), and they were as follows: ITO/Alq$_3$/C$_{60}$/SubPc/DMD (Figure 1). They are based on the electron acceptor/electron donor couple (EA/ED): C$_{60}$/SubPc. SubPc is known for its high absorption coefficient and C$_{60}$ for its high efficiency as an electron acceptor [27]. The thickness of these layers in inverted OPVs was earlier optimized: 40 nm for C$_{60}$ and 16 nm for SubPc [28]. The buffer layer inserted between the cathode and the electron acceptor, which is called the exciton blocking layer (EBL) in the case of PHJ-OPVs [29], is a thin layer (9 nm) of Alq$_3$. Often, the EBL consists in a bathocuproine layer [26]; however, it was shown that Alq$_3$ allows obtaining more stable OPVs, which justifies our choice [30].

Regarding the DMD top electrode, the first dielectric layer also serves as a hole-transporting layer (HTL), so, as evocated above, it consists in a MoO$_3$ layer. For the other layers of the MoO$_3$/M/D structure, we used two metals: either Ag or Cu:Ag alloy and ZnS as the dielectric. The atomic concentration of Ag in the alloy was 5 at%. In the case of the reference opaque electrode, the top electrode was MoO$_3$/Al. We have already optimized the DMD structures, either as transparent conductive structures or as bottom electrodes in opaque OPVs [23,25]. Here, due to the fact that the DMD structures were deposited onto stacked ITO/Alq$_3$/C$_{60}$/SubPc layers, we had to check the optimum thickness of the MoO$_3$ interfacial layer.

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Figure 1. Typical inverted semi–transparent organic photovoltaic cell (OPV).

The different devices were characterized using the following techniques: optical transmission and absorption measurements, electrical conductivity measurements, X-ray photoelectron spectroscopy, J-V characteristics of OPV in the dark, and under AM1.5 solar spectrum.

These different techniques are described in Supporting Information S2.

3. Results and Discussion

Regarding the DMD structures, when Ag was used as metal and MoO$_3$ as dielectric, reproducible results were obtained. The performances of the structures Glass/MoO$_3$ (20 nm)/Ag(10 nm)/MoO$_3$ (35 nm) are summarized in Table 1, and the visible optical transmission is shown in Figure 2.

Table 1. Main parameters of the transparent electrodes.

| Transparent Electrode | Sheet Resistance (Ω/sq) | Maximum Transmission (%) | Figure of Merit $10^{-3}$ (Ω)$^{-1}$ |
|-----------------------|-------------------------|---------------------------|--------------------------------------|
| ITO                   | 15                      | 93.5                      | 34                                   |
| MoO$_3$/Ag/MoO$_3$    | 5                       | 90.1                      | 70                                   |
| MoO$_3$/Ag (1 nm)/Cu:Ag (16 nm)/ZnS | 33                     | 85.2                      | 6.1                                  |
| MoO$_3$/Ag (2 nm)/Cu:Ag (15 nm)/ZnS | 29                     | 83.9                      | 6.0                                  |

From optical and electrical measurements, we have calculated $\Phi_M$, the figure of merit proposed by Haack using the empiric formulae [31]:

$$\Phi_M = T^{10}/\sigma_{sh}$$

(1)

where $\Phi_M$ is the figure of merit, $T$ is the transmission of light, and $\sigma_{sh}$ is the sheet resistance.

It allows for comparing the “opto-electrical” performances of the different electrodes.

In the case of MoO$_3$/Ag/MoO$_3$ structures, the optimum thickness of the layers has been determined in previous publications to be 20 nm/10 nm/35 nm, respectively [23].

The thickness of the Ag layer, 10 nm, corresponds to the percolation threshold of the metal layer; for less thickness, the metal film is discontinuous; for thicker films, the reflectivity and absorption of the metal film increase.
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ready shown that Cu diffuses easily and spontaneously into MoO3 [24]; therefore, in the
However, we cannot substitute ZnS for the other MoO3 layer because, while MoO3 is an
table layer; for less thickness, the metal film is discontinuous and the structure is insulating; for
Moreover, 2 nm of Ag permits obtaining structures more stable than those with 1 nm of
Cu:Ag metal layer is slightly better with 2 nm of Ag than a structure with 1 nm of Ag.

Figure 2. Transmission spectra of (——) glass/MoO3 (20 nm)/Ag (10 nm)/MoO3 (35 nm), (——)
In the case of copper, it is not so easy to grow performing TCE. In fact, we have already shown that Cu diffuses easily and spontaneously into MoO3 [24]; therefore, in
structures MoO3/Cu/MoO3, we inserted ZnS in the MoO3 top layer in such a way
that we used MoO3/Cu/ZnS structures, the diffusion of Cu in ZnS being far smaller than in
MoO3. However, we cannot substitute ZnS for the other MoO3 layer because, while
MoO3 is an excellent hole-extracting layer, ZnS is not, and the performances of the OPVs
in MoO3/M/ZnS structure with acceptable

In Figure 2, we introduce the absorption curve of SubPc. It can be seen that the
maximum of absorption of SubPc corresponds to the transmission maximum of the DMD
structures, which can be favorable for a compromise between the absorption and transmission of semi-transparent OPVs. On the other hand, it can be seen that, on the ultraviolet side of the spectrum, the transmission is limited due to the optical band gap of the dielectrics, while in the near-infrared domain, the transmission decreases due to the plasma effect resulting from the high concentration of mobile electrons in the IR region, which allows having conductive electrodes.

Therefore, at the beginning of the study of the semi-transparent OPVs, we used MoO$_3$ (20 nm)/Ag (10 nm)/MoO$_3$ (35 nm) and MoO$_3$ (20 nm)/Ag (2 nm)/Cu:Ag (15 nm)/ZnS (45 nm) top electrodes. Unfortunately, the efficiency of the OPVs was quite low, with high-series resistances. MoO$_3$ being resistive—a disappointing result—was attributed to the thickness of the interfacial MoO$_3$ layer, 20 nm. Therefore, it was necessary to decrease this thickness. The experimental study shows that a thickness of 10 nm allows obtaining optimum results.

Figure 3 displays the spectral transmission of the complete OPVs: ITO/Alq$_3$/C$_{60}$/SubPc/DMD, the DMD top electrode being either MoO$_3$ (10 nm)/Ag (10 nm)/MoO$_3$ (35 nm) or MoO$_3$ (10 nm)/Ag (2 nm)/Cu:Ag (15 nm)/ZnS (45 nm).

![Figure 3. Transmission spectra of OPVs with different top electrodes: (---) MoO$_3$ (10 nm)/Ag (10 nm)/MoO$_3$ (35 nm) and (-- --) MoO$_3$ (10 nm)/Ag (2 nm)/Cu:Ag (15 nm)/ZnS (45 nm) and (· · · · · ·) absorption spectrum of the OPV without top electrode: Glass/ITO/Alq$_3$/C$_{60}$/SubPc.](image)

Obviously, the transmission spectrum of the opaque OPV, with a 100 nm thick Al top electrode, is null. In the case of semi-transparent OPVs, i.e., when the top electrode is either MoO$_3$ (10 nm)/Ag (10 nm)/MoO$_3$ (35 nm) or MoO$_3$ (10 nm)/Ag (2 nm)/Cu:Ag (15 nm)/ZnS (45 nm), the transmission is higher for wavelengths exceeding $\lambda = 615$ nm, due to the absorption spectrum shape of SubPc (Figure 3). After reaching a maximum value, the transmission gradually decreases, following the decreasing transmission curve of the DMD structures. On the whole spectrum, the transmission difference between the two types of OPVs follows the difference in transmission of the DMDs themselves. On the other hand, as expected, their minimum transmission, situated between 550 nm and 615 nm, corresponds to the domain of maximum absorption of SubPc. More globally, the transmission curve of the OPVs is the inverted image of the absorption curve, which confirms the relatively good transmission of light by the anodes used.

The J-V characteristics of the inverted OPVs are shown in Figures 4 and 5 and summarized in Table 2.
Figure 4. J-V characteristics inverted OPV with MoO$_3$/Ag/MoO$_3$ as top anode: in the dark (■), under light from the ITO side (●), and from the MoO$_3$/Ag/MoO$_3$ side (▲). Classical MoO$_3$/Al top anode (▼).

Figure 5. J-V characteristics inverted OPV with MoO$_3$/Ag/Cu:Ag/ZnS as top anode: in the dark (■), under light from the ITO side (●) and from the MoO$_3$/Ag/Cu:Ag/ZnS side (▲). Classical MoO$_3$/Al top anode (▼).

Table 2. Parameters of the inverted OPVs using different top anodes.

| Anode               | Light Side | Voc  | Jsc  | FF  | η   | Rs  | Rsh  |
|---------------------|------------|------|------|-----|-----|-----|------|
| Al/MoO$_3$          | ITO        | 0.94 | 5.60 | 57  | 3.00| 20  | 1800 |
| MoO$_3$/Ag/MoO$_3$  | ITO        | 0.94 | 5.04 | 48  | 2.34| 30  | 1200 |
| MoO$_3$/Ag/MoO$_3$  | DMD        | 0.92 | 3.85 | 45  | 1.61| 35  | 1000 |
| MoO$_3$/Ag/Cu:Ag/ZnS| ITO        | 0.84 | 4.77 | 40  | 1.55| 40  | 700  |
| MoO$_3$/Ag/Cu:Ag/ZnS| DMD        | 0.43 | 3.71 | 39  | 1.10| 40  | 500  |
The reference OPV with the opaque Al top electrode gives the best results, which is not unexpected. Nevertheless, in the case of the MoO$_3$/Ag/MoO$_3$ top anode, when the OPV is illuminated on the ITO side, the efficiency reached is not too far from that obtained with the Al top electrode.

While the open circuit voltage $V_{oc}$ is stable, the efficiency decrease is due to the short circuit-current—$J_{sc}$—and the Fill Factor (FF). The reflectivity of the Al top anode is far higher than that of MoO$_3$/Ag/MoO$_3$, which justifies the decrease in $J_{sc}$.

Regarding FF, the Ag film thickness being only 10 nm, its homogeneity must be less than that of the 100 nm thick Al layer, which should induce interface traps and a higher sheet resistance as shown by the increase in series resistance. When deposited onto organic layers, the sheet resistance of the MoO$_3$/Ag/MoO$_3$ electrode is probably not as low as when deposited onto a polished glass substrate.

This effect is reinforced when the OPV is illuminated on the MoO$_3$/Ag/MoO$_3$ side. To the effect of the low reflectivity of the electrode and higher sheet resistance, we must add the smaller light transmission; all these losses result in a deterioration of the OPV performances, mainly in a significant decrease in $J_{sc}$.

In the case of MoO$_3$/Ag/Cu:Ag/ZnS as the top anode, the performances obtained are far smaller than those of the reference OPV. In order to understand such poor results, we have proceeded to do some more characterization. We have already shown that the presence of Cu at the organic material/anode interface induces severe degradation of the OPVs, so we have studied the profile of the MoO$_3$/Ag/Cu:Ag/ZnS structure. In order to reproduce the experimental conditions of the OPVs, we deposited onto a glass substrate the layer sequence MoO$_3$/Ag/Cu:Ag/ZnS. Generally, when thin layers are superimposed, the higher the number of layers superimposed, the rougher the sample surface is. This makes it more difficult to interpret an XPS profile because following this roughness, we lose resolution. Subsequently, we chose to make the profile on a sample deposited on glass.

The XPS profile obtained on such glass/MoO$_3$/Ag/Cu:Ag/ZnS multilayer structure is reported in Figure 6. To check the influence of the thin Ag layer introduced between the Cu and MoO$_3$ layers, we also present the profile of a glass/MoO$_3$/Cu:Ag/ZnS multilayer structure in Figure 6b. It can be seen that even if Ag limits the Cu diffusion into MoO$_3$, without Ag, the profile Cu/Mo is flat, and there is a significant diffusion of Cu into Mo, with 40 at.% of Cu present at the interfacing electrode/organic material. Nevertheless, in comparison with Figure 6b, at the center of the structure, when the thin layer of Ag is present, the atomic concentration of Cu is higher, while, on the other hand, it is lower in the MoO$_3$ layer. This shows the effectiveness of this thin Ag layer in limiting the diffusion of Cu. However, the presence of a relatively high concentration of Cu at the interface has a negative effect on the OPV performances. We have already shown that if an ultra-thin layer of 0.6 nm of Cu is an efficient anode buffer, this positive effect is destroyed when Cu is present in a thicker layer [33], which is the case at hand. The diffusion of Cu induces a decrease of the shunt resistance, resulting in poor rectifying properties. Moreover, the Cu diffusion results in an increase of the sheet resistance of the electrode. All that results in a decrease in the values of $V_{oc}$, $J_{sc}$, FF, and efficiency. As in the case of MoO$_3$/Ag/MoO$_3$, the performances are smaller when the OPV is illuminated from the top anode side, for the same reasons.

The partial diffusion of Cu into MoO$_3$ in the MoO$_3$/Ag/Cu:Ag/ZnS structures justifies the fact that to obtain acceptable sheet resistance it is necessary to use thicker (17 nm) metal films in the case of these TCE, than in the case of MoO$_3$/Ag/MoO$_3$.

Nevertheless, it must be underlined that, as regards results obtained with the MoO$_3$/Ag/MoO$_3$ anodes, the results obtained are at the level of results already published [34–37], but using here the simple PHJ–OPV configuration and based on well-known and inexpensive molecules.
4. Conclusions

It can be said that, up to now, if DMD structures using Ag as metal and MoO$_3$ as dielectric have been demonstrated to be highly efficient transparent electrode on top of OPVs, the results obtained with MoO$_3$/Ag/Cu:Ag/ZnS electrodes are not as convincing. Such a disappointing result is attributed to the fact that copper tends to diffuse in MoO$_3$, even if the use of Cu:Ag alloy permits limiting the Cu diffusion. That said, the results obtained with MoO$_3$/Ag/MoO$_3$ top electrode are far more promising. An optimized MoO$_3$/Ag/MoO$_3$ top electrode allows achieving semi-transparent OPVs with quite high transparency. Of course, this transparency penalizes the short circuit current and, therefore, the OPV efficiency; nevertheless, the performances of these OPVs put them among the most transparent OPVs for a yield of the same order of magnitude as those made with cells based on BHJ. In fact, since, in order to obtain semi-transparent OPVs, it is necessary to limit the thickness of the organic materials, the present work shows that the benefit of using BHJ, which is very efficient in classical OPV, is lost here. In the case of semi-transparent OPVs,
planar heterojunction appears to be a promising solution, paving the way toward building integrated photovoltaics (BIPVs) for their ease of production and high reproducibility.

In the case of MoO$_3$/Ag/Cu:Ag/ZnS electrodes, the performances of the DMD are diminished due to partial Cu diffusion into the dielectrics, mainly into MoO$_3$. In order to improve the power conversion of OPV using a top electrode containing Cu, we are now probing new DMD configurations:

- Since Cu diffusion in ZnS is far smaller than in MoO$_3$, we are going to probe MoO$_3$/ZnS/Ag/Cu:Ag/ZnS structures. The bilayer of dielectric MoO$_3$/ZnS has a dual goal:
  - MoO$_3$ allows an efficient collection of holes;
  - ZnS minimizes the diffusion of Cu.

  This should improve the efficiency of the cells.

Another possibility is to use another Cu alloy; we aim to replace Cu:Ag with other alloys such as Cu:Cr, as Cr is well-known as a diffusion barrier.

Supplementary Materials: The following are available online at https://www.mdpi.com/2079-4991/11/2/393/s1, S1: Preparation of the substrates and deposition conditions; S2: Characterization techniques.

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