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Interface and Electrode Engineering for Next-Generation Organic Photovoltaic Cells

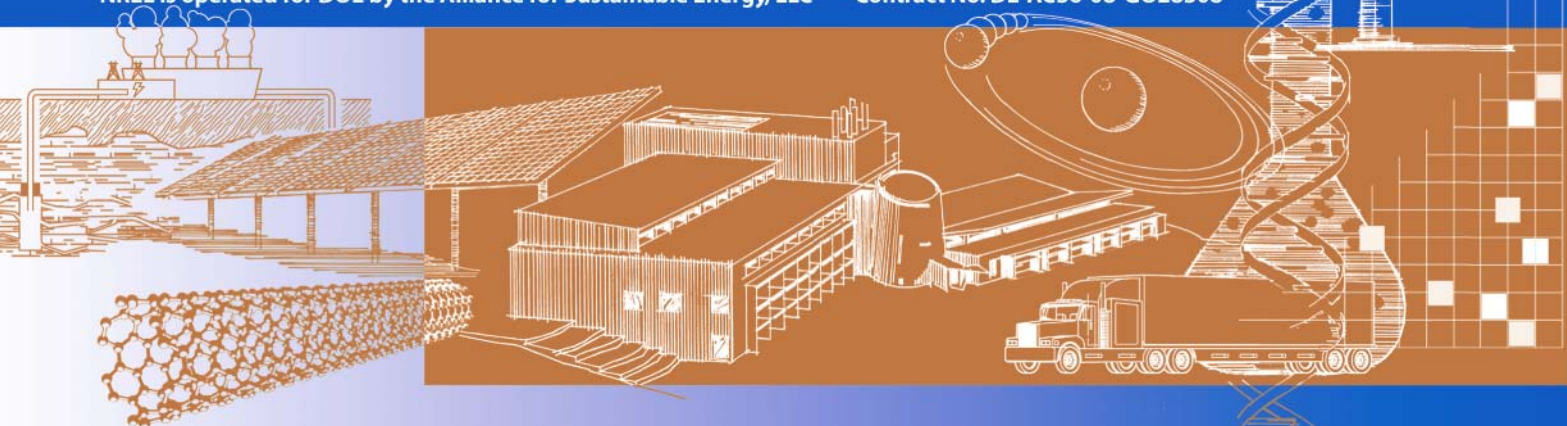
Final Technical Report March 2005 – August 2008

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*Northwestern University
Evanston, Illinois*

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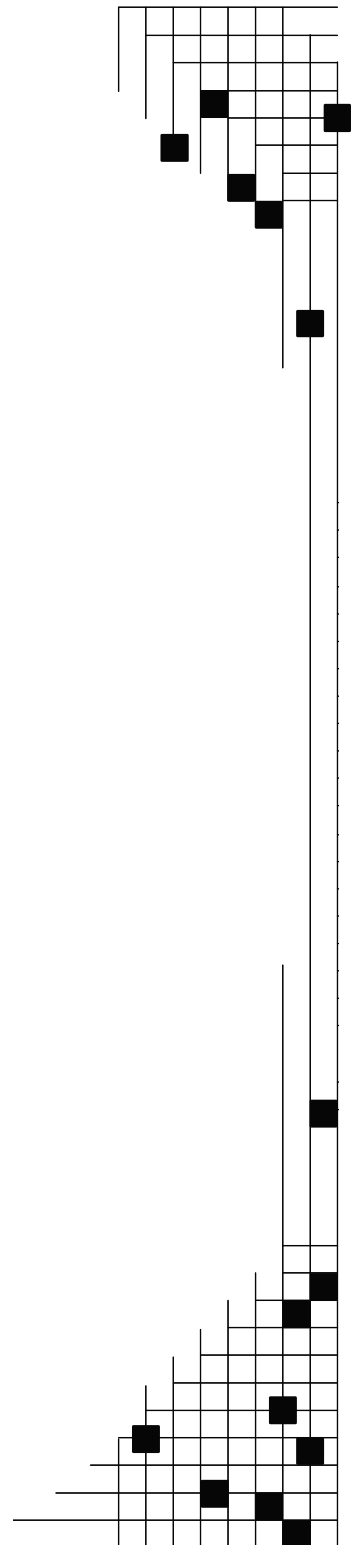
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NREL Technical Monitor: Fannie Posey Eddy
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Objective and Approach

The objective of this project was to enable next-generation, efficient, easily manufacturable, and durable organic photovoltaics (OPVs) through interface and electrode engineering. The Northwestern University (NU) group combines expertise in first-principles electronic structure theory, exploratory synthesis, thin-film deposition, device fabrication, and characterization. Building on success at NU in developing improved organic light-emitting diodes (OLEDs), the program addressed two crucial aspects of OPV technology: 1) improved electrode-organic interfaces, and 2) improved transparent electrode materials. The NU group has extensive expertise in the area of unconventional transparent electrodes (or transparent conducting oxides, TCOs), including novel “nanostructured” TCO morphologies. The electronic properties of TCO surfaces (e.g., band offsets/work functions) are believed to play a crucial role in OPV performance. New TCOs were examined that better match (chemically and electronically) the organic components of OPV cells. The group also developed high-efficiency organic adhesion/current-collection layers for improved electrode-organic interfaces. The ultimate goal was to develop and test a high-efficiency prototype organic solar cell incorporating NU-developed interfacial and electrode materials.

Research Highlights

This final report details the most recent results, as well as the most significant results emerging from our project over its lifetime. We proceed from A) theoretical developments, to B) TCO synthesis and characterization, to C) OPC interface engineering and OPV cell fabrication/testing.

A. Theoretical Developments

A major premise of our work is that transparent electrode alternatives to indium tin oxide (ITO) need to be developed, exhibiting: 1) better band alignment with organics in OPVs, and 2) improved stability during OPV processing and for long-term durability. Toward the goal of better band alignment, the Freeman group has pioneered methods for predicting surface electronic structure, including work-function predictive capabilities. They employed a highly precise, thin-film (single-slab) FLAPW (full-potential linearized augmented plane wave) band-structure method to determine, for the first time, the electronic properties at oxide surfaces. Work functions were calculated for the polar $\{0001\}$ and non-polar $((10\bar{1}0)$ and $(11\bar{2}0))$ of ZnO films, and results are in good agreement with available experiments.

For example, the polar $\{0001\}$ surfaces of ZnO show a significant difference (about 4 eV) in work function between the cation (0001) and anion $(000\bar{1})$ surface layers, as did the previous calculations for InN. The cation surface of the polar ZnO films shows 3.11 eV (GGA) and 3.34 eV (LDA) for the work function, when the anion surface shows 7.28 eV (GGA) and 7.43 eV (LDA). However, it is known that the polar surfaces of ZnO with a small number of bilayers (less than 10) show the structural phase transition from wurtzite to graphitic structure to remove the destabilizing dipoles as in other wurtzite materials. In this graphitic structure, we have found that the ZnO surface shows about 5.5 eV for the work function. The non-polar $((10\bar{1}0)$ and $(11\bar{2}0))$ surfaces show 4.50 eV

~ 4.78 eV (GGA) and 5.06 eV ~ 5.31 eV (LDA). For the non-polar surfaces, the work functions were calculated from the conduction band minimum because ZnO has a Fermi-level stabilization energy just 0.2 eV below the conduction-band edge, and thus, it shows a propensity for n-type conductivity (Walukiewicz, *Physica B* 302, 123 (2001)). These work-function calculations for ZnO surfaces give comparable results with experiments (4.53 eV, Park et al., *Jpn. J. Appl. Phys.* 44 L242 (2005)), (4.23 eV, Forsythe et al., *Journal of Vacuum Science & Technology A*, 17(4), 1761(1999)), (3.6 eV, Rao et al., *APL* 87 (3) (2005)). Preliminary results on bulk ceramic Al-doped ZnO by the Mason group (with Klein at TU-Darmstadt) yielded work functions of ~4.5 eV. These experimental work functions are, in most cases, for polycrystalline materials (thin films or bulk). In addition, the calculation for the anion (000 $\bar{1}$) surface of the polar films yields the very high work-function value, but it is envisioned that this anion surface is not stable. For instance, the literature (Gan et al., *PRB* 74 115319 (2006)) shows that the stable structure of the InN (000 $\bar{1}$) surface is a monolayer of In atoms directly above the surface N atoms, whereas the relaxed, unreconstructed InN (0001) surface is stable under N-rich conditions.

The Freeman group has also investigated the electronic structures and work functions of (001) antiferromagnetic type-II NiO (110) films using their highly precise thin-film (single-slab) FLAPW method. Acceptor-doped NiO is a potential p-type TCO candidate material for OPVs and also an effective “electron blocking layer” or EBL at OPV anode surfaces (see below).

To treat strong correlations (i.e., the Coulomb repulsion of the 3d electrons of Ni ions), the LSDA+U method with $U=8.0$ eV and $J=0.95$ eV was applied to treat the Ni atoms. The calculations with LSDA+U for bulk NiO yield a charge-transfer-type bandgap (~3.4 eV) between the predominantly O p -states at the valence-band maximum (VBM) and the Ni e_g states at the conduction-band minimum (CBM), whereas the LSDA shows a significantly smaller bandgap (~0.4 eV) between the mainly d -states of Ni. The LSDA+U for the bulk NiO yields $1.69 \mu_B$ for the Ni spin-magnetic moments, which agrees well with experimental values, $1.64 \sim 1.90 \mu_B$, whereas the LSDA-only calculations yield a significantly smaller value, $1.1 \mu_B$. The O site, on the other hand, has zero spin-magnetic moment due to the symmetry in the bulk.

These bulk features of the spin magnetic moments are also well reproduced in the central layers of Ni (001) films. The geometry optimization of a 9-layer NiO (001) film results in a very small rumpling of the surface layer, 1.8% of the interlayer spacing (inward displacement of O by 0.026 \AA and outward displacement of Ni by 0.0097 \AA).

The same value of U was employed for the NiO films as that for the NiO bulk system. The existence of the surface, with its weaker screening of electron-electron interactions due to the vacuum, may affect this parameter, but the dominant screening mainly comes from electrons of the same ion. Hence, the effect of the surface on the U parameter is assumed to be negligible. The 9-layer (001) NiO film calculations yield an approximately 2.9-eV bandgap, which is smaller than the calculated bulk bandgap, whereas the LSDA calculations show a nearly zero bandgap (~ 0.007 eV). Compared to the NiO bulk system, the reduction of the bandgap can be attributed to the surface states with sharp peaks near the band edges, that is, by mainly the weak surface potential and the broken symmetry. Considering the broken symmetry, O- p_z states and Ni- $d(3z^2-r^2)$ states are responsible for these surface states and also for the non-zero magnetic moments

of O atoms at the surface layer. At the surface, the in-plane p-d hybridization is the same as in the bulk, but the broken symmetry in (001) causes asymmetric inter-plane hybridization between O-pz and Ni-d ($3z^2-r^2$) states. The spin magnetic moments of the O ions at the surface layer are $0.7 \mu_B$ with LSDA and $0.4 \mu_B$ with LSDA+U. This difference can be explained by the stronger localization of the Ni *d*-electrons in the LSDA+U calculations.

The work function for the (001) surface of a 9-layer NiO film is predicted to be about 4.76 eV with LSDA, which is calculated from the VBM. It is known that NiO is a p-type semiconductor due to intrinsic metal vacancies. However, the LSDA+U calculations yield a significantly larger work-function value, 5.62 eV, due to the down-shifting of the valence bands. Experimentally, NiO deposition on ITO for OLED applications has been shown to increase the work function of ITO (~ 4.8 eV) dramatically to 5.4 eV, which is closer to the optimum level of the HOMO (highest occupied molecular orbital) in typical organic layers of both OLEDs and OPVs. The use of a NiO-based EBL in an OPV cell, and the corresponding cell performance, is described later in this report.

B. TCO Materials Synthesis and Characterization

The Marks group has explored double-layer TCO structures for OPV anode applications. For example, CdO-based TCOs are of great interest owing to their high conductivity and low cost. In-doped CdO (CIO) films grown by metal-organic chemical vapor deposition (MOCVD) on amorphous glass substrates have conductivities as high as $\sim 12,000$ S/cm. However, they possess a low work function (4.2 eV) and exhibit relatively rough surface morphology. By employing a double-layer strategy, we can achieve lower sheet resistance, smoother surface morphology, and tunable work function. The double-layer ITO/CIO thin-film approach was successfully implemented by the Marks group, using MOCVD of CdO combined with ion-assisted deposition (IAD) of ITO, which has lower overall In content and higher conductivity than commercial ITO films.

$\text{Cd}(\text{hfa})_2(\text{N,N-DE-N}',\text{N}'\text{-DMEDA})(\text{hfa}=\text{hexafluoroacetylacetonate}, \text{N,N}'\text{-DE-N,N}'\text{-DMEDA} = \text{N,N}'\text{-diethyl-N,N}'\text{-dimethylethylenediamine})$ and $\text{In}(\text{dpm})_3$ (dpm = dipivaloymethanate) were synthesized according to published methods and purified by sublimation. These were then used as the precursors for the growth of CIO films in a home-made, low-pressure, cold-wall MOCVD reactor. CIO thin films with thickness of ~ 180 nm were then deposited on glass substrates at a growth rate of ~ 1.5 nm/min for 2h. The In doping level is around 4.0 atom%. The as-deposited CIO films were then transferred to an IAD chamber for ITO over-coating using an ITO target ($\text{In}_2\text{O}_3:\text{SnO}_2 = 9:1$) purchased from Sputtering Materials, Inc. The double-layer films exhibited average optical transmittance over 85% in the 400–700-nm region and typical sheet resistances of about $6 \Omega/\square$ were obtained.

The Poeppelmeier, Mason, and Chang groups made significant progress understanding the “self-doping” character of ZITO (Zn-In-Sn-O) alternatives to ITO for potential OPV anode applications. The first step was to complete the subsolidus phase diagram for the $\text{ZnO-In}_2\text{O}_3\text{-SnO}_2$ system (by Poeppelmeier and Mason). This is an important TCO development, because there are a number of TCO phases in the system—

e.g., all the unary compounds are TCOs (ZnO, In₂O₃, SnO₂), when appropriately doped, and the Zn,Sn codoped In₂O₃ “bixbyite” solid solution is also an exceptionally good TCO system. One noteworthy aspect of Zn,Sn codoped In₂O₃ is the ability to replace up to 40% of indium by less expensive zinc and tin.

A second step by Mason and Chang was to establish that the ZITO bixbyites are “self-doped,” i.e., there is an inherent cation off-stoichiometry in favor of Sn donors over Zn acceptors, as shown in Fig. 1a. This is seen in the equilibrium phase field existing to the right (Sn-rich) side of nominal stoichiometry. The significance of this finding is that ZITO-based OPV anodes, unlike ITO, do not require intentional donor-doping to achieve excellent TCO properties.

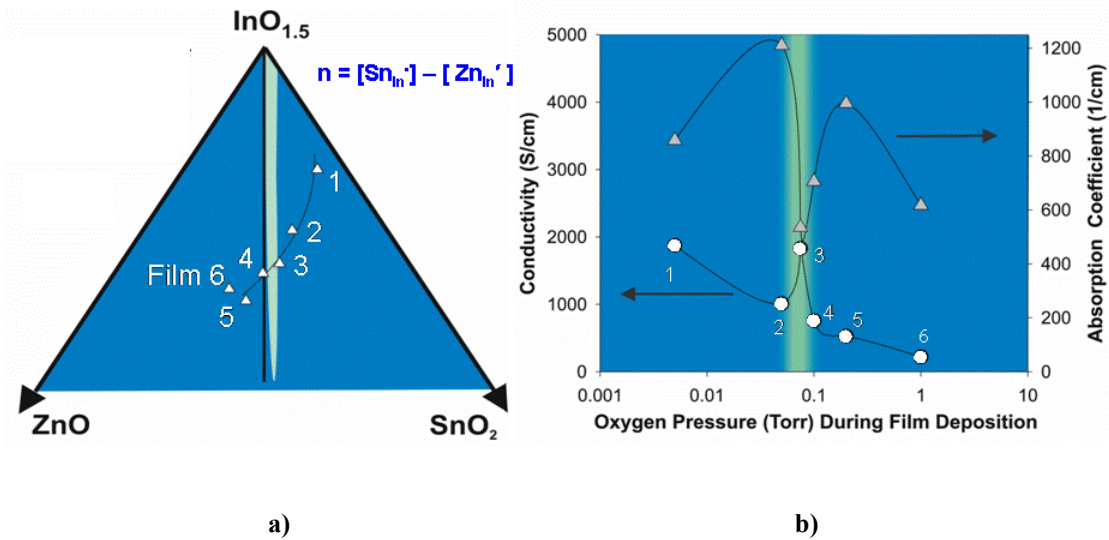


Fig. 1a) The In-end of the Zn-In-Sn-O (ZITO) phase diagram showing the bixbyite phase field and the curved trajectory of PLD films grown from a 30% co-substituted target (variable pO₂ during growth). b) Thin-film conductivity and optical absorption vs. composition along this trajectory.

The third step was to demonstrate that thin films made by pulsed-laser deposition (PLD) exhibit a sharp dip in optical absorption and a peak in electrical conductivity at “special compositions” (see Fig. 1b) corresponding, to the equilibrium phase field in Fig. 1a. Crystalline ZITO films have been grown, which exhibit conductivities as high as 4000 S/cm without sacrificing transparency. Low sheet resistance (30Ω/□), high transparency (>90%@400-700 nm), 30% zinc-tin co-substituted, *amorphous*, indium oxide (ZITO) films have also been grown at room temperature by PLD, which is a major improvement over the standard ITO. This work shows the power of combined bulk and thin-film approaches to understanding/tailoring novel TCOs.

It is well-established that one can independently control TCO Fermi level (by doping) and external work function (Fermi level to vacuum level) by surface dipole modification (SDM). By SDM, we are referring to changes in surface terminations and/or oxygen content/structure of the topmost surface. In collaboration with the group of Klein at TU-Darmstadt, the Mason group has obtained the work function vs. Fermi level data (relative to the VBM) in Fig. 2. An important first step is to grow highly

doped/conductive ZITO films under proper (reducing) conditions (step 1 in Fig. 2). It is then possible to increase the work function (by as much as 1 eV from ~ 4 eV to ~ 5 eV!) by a post-synthesis oxidation treatment (step 2 in Fig. 2). By subjecting the ZITO film surfaces to brief intermediate-temperature (400° - 500°C) oxidation treatments, the surface Fermi level remains high (~ 3 eV), but the work function increases dramatically. The correlation between such post-treated films and bulk ceramics (treated at much higher temperatures) argues for a similarity of surface dipoles in the two materials. The origin of this SDM requires additional study to elucidate the mechanism(s). However, this work illustrates an important two-step process for degenerately doping, and then enhancing the work function of ZITO OPV anodes.

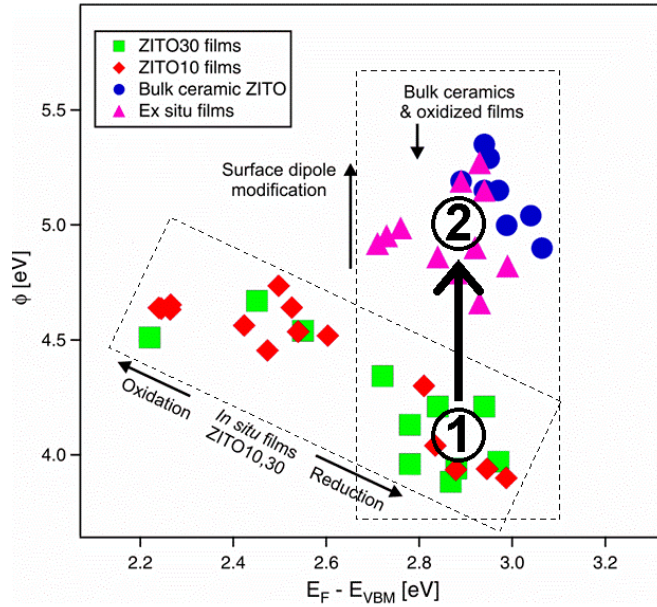


Fig. 2. Work function vs. Fermi level (vs. VBM) by XPS/UPS for in situ sputtered ZITO thin films (lower set) and degenerate films subjected to brief post-synthesis O_2 treatments (vertical set), which also agree with bulk values.

The Poeppelmeier group has made significant progress toward development of novel p-type TCO materials. All the known copper CuBO_2 ($B = \text{Al, Sc, Cr, Mn, Fe, Co, Ga}$ and Rh) and silver AgBO_2 ($B = \text{Al, Sc, Fe, Ga, Co, Ni, Rh, In}$ and Tl) delafossite oxides were produced in moderate to high yields by a low-temperature ($< 210^\circ\text{C}$) and low-pressure (< 20 atm) hydrothermal synthesis (HTS) technique. In contrast to two-step ion-exchange syntheses, or very high-pressure syntheses, all the known silver delafossites were successfully synthesized by a convenient, practical, and direct reaction for the first time, including several key compositions (AgAlO_2 , AgGaO_2 , and AgScO_2) never synthesized in any practical (good yield) reaction before.

These synthetic methodology advances are extremely important in the search for new and interesting transparent conductors (TCs), in particular those that can support oxidation (p-type). The synthesis of any mixed metal oxide containing silver and another metal never occurs in open systems because Ag_2O decomposes before any appreciable reaction can occur. The HTS process represents the first practical method to generate the

silver delafossite oxides AgAlO_2 , AgGaO_2 , and AgScO_2 on a gram scale. The diffuse reflectance of AgGaO_2 , AgAlO_2 , AgScO_2 , and AgInO_2 polycrystalline powders were measured and their optical bandgaps were estimated from these measurements. AgAlO_2 and AgScO_2 , which have distinct absorption edges, are about 3.4 eV and 3.8 eV, respectively, ample for most opto-electronic applications. AgGaO_2 and AgInO_2 , which are green and orange in color, respectively, exhibit additional absorption in the visible region, but their optical bandgaps were estimated to be 3.5 eV and 4.2 eV.

Therefore, the same increase in the bandgap of silver delafossite oxides is observed as was calculated by NREL for the copper delafossite oxides (CuBO_2 , B = Al, Ga, and In) with an increase in the B-site cation radius (*Phys. Rev. Lett.* **2002**, *88*, 066405-1). Indeed, because the *a* lattice parameter and unitcell volume of copper and silver delafossite oxides with the same B-site cation (e.g., CuAlO_2 and AgAlO_2) increase in a similar fashion, it should not be surprising that their optical bandgaps respond similarly with a change in B-site cation radius. Moreover, the observed color of the silver delafossite oxides AgGaO_2 (green) and AgInO_2 (orange) can also be explained by the NREL calculations, owing to absorption from a “forbidden,” low-energy direct-gap transition.

The synthesis of all the silver delafossite oxides in gram quantities enabled the comparison of delafossite oxides with the same B-site cation, but different A-site cations. When compared to copper delafossite oxides, the onset of the optical band-gaps of the silver delafossite oxides are a few tenths of an electron volt greater, as predicted by calculations (*Solid State Sci.* **2002**, *4*, 1045). The authors conclude that while the density of states (DOS) of copper *3d* states is sharply peaked near the top of the valence band, the DOS for silver *4d* states is broader, tails to lower energies, and traces the oxygen *p*-character. The transition of a valence electron from hybridized silver *4d* and oxygen *2p* states to silver *5p* states in the conduction band thus requires more energy owing to the scarcity of silver *4d* states just below the VBM of silver delafossite oxides. As a result, silver delafossite oxides have a wider optical bandgap than copper delafossite oxides with the same B-site cation. All things considered, the larger bandgap and lower absorption coefficients of silver delafossite oxides should translate into more transparent thin films. Ongoing work is aimed at the intentional acceptor-doping of these materials by HTS.

C. OPC Interface Engineering/OPV Cell Fabrication/Testing

Building on the abovementioned theoretical work on NiO-based TCOs, an organic P3HT:PCBM bulk-heterojunction (BHJ) solar cell was fabricated employing a thin p-type NiO electron-blocking layer (EBL)/hole-transporting layer (HTL). To accomplish this, thin (5-80-nm) layers of NiO were deposited by PLD onto cleaned ITO surfaces. Ordinarily, a thin semiconducting PEDOT:PSS EBL is spin-cast as an aqueous dispersion onto ITO before active-layer deposition. Unfortunately, aqueous PEDOT:PSS dispersions are at pH~1 and are corrosive to the underlying ITO. Prior OLED work in the Marks group has also shown that PEDOT:PSS is an inefficient EBL. The use of an inorganic EBL/HTL eliminates the corrosion problem, while providing outstanding EBL/HTL performance. To prepare the active BHJ layer, an o-dichlorobenzene (ODCB) solution of P3HT and PCBM was spin-coated in a glove box onto the ITO/NiO surface. Within the glove box, the film was annealed and device fabrication was completed by

vapor-depositing LiF and then the Al cathode. The optimum NiO interlayer thickness was found to be 5-10 nm (see Fig. 3, below). The resulting glass/ITO/NiO/P3HT:PCBM/LiF/Al solar cell exhibited cell power efficiencies as high as 5.2%. The fill factor was enhanced by ~70% over a control device lacking the NiO EBL/HTL.

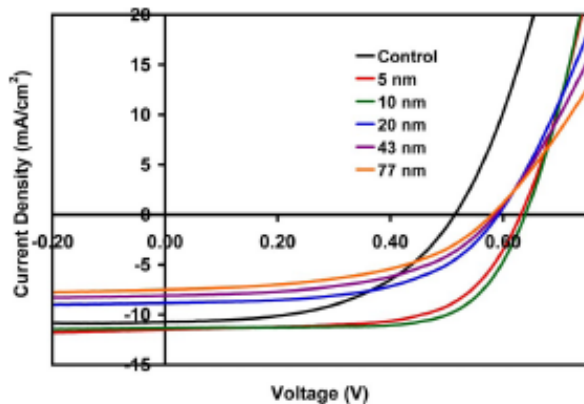


Fig. 3. Current density-voltage plots for BHJ solar cells fabricated with varying layer thicknesses of NiO on the ITO anode. The control device lacked the NiO EBL/HTL.

These promising results point the way to further increases in OPV cell performance, by both improved TCO development and by engineering the organic/inorganic interfaces in OPV designs.

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