Iron pyrite nanocrystal film serves as a copper-free back contact for polycrystalline CdTe thin film solar cells

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A B S T R A C T

We report the application of thin film nanocrystalline (NC) FeS₂ as the copper-free back contact for CdTe solar cells. The FeS₂-NC layer is prepared from solution directly on the CdTe surface using drop-casting coupled with a hydrazine treatment at ambient temperature and pressure, and requires no thermal treatment. Copper-free solar cells based on the CdS/CdTe/FeS₂-NC/Au architecture exhibit device efficiencies > 90% that of a standard Cu/Au back contact devices. The FeS₂-NC back contact solar cells show good thermal stability under initial tests. Devices prepared with untreated FeS₂-NC back contacts display a strong “S-kink” behavior which we correlate with a high hole-transport barrier arising from inter-NC organic surfactant molecules.

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1. Introduction

Following significant focus on FeS₂ solar cells in the early years of thin film photovoltaics (PV) [1–7], FeS₂ has in recent years attracted renewed attention as a potential light-absorbing layer in thin film PV cells based on its suitable band gap energy, high absorption coefficient, low toxicity, and elemental abundance [8–16]. However, FeS₂-based PV devices have thus far performed poorly compared with devices based on other recently-developed absorber materials (e.g. CZTS, perovskites such as methylammonium lead iodide, and PbS quantum dots) [17–21]. Tributsch et al.’s extensive efforts initiated in the 1980s led ultimately to a 2.8% efficient photo-electrochemical cell based on single crystal FeS₂ electrodes using an iodide/tri-iodide (I⁻/I₃⁻) redox couple [1–4]. This result stands as the efficiency record for FeS₂-based solar cells, owing in part to the well-recognized challenges presented by high defect densities resulting from poor phase and stoichiometry control [22,23]. Specifically, phase purity has been an expressed concern for effective FeS₂ photovoltaic devices because the different iron sulfide phases of FeₓSᵧ exhibit a wide range of optoelectronic properties [24].

Here we report on the discovery that the properties of FeS₂ nanocrystal (NC)-based thin films yield excellent performance as a low barrier back contact to CdS/CdTe solar cells, without copper pre-treatment of the CdTe p-type film. Cadmium telluride thin film modules represent ~7% of the 32 GW of PV modules produced in 2012, and CdTe manufacturing produces the lowest cost on a per-Watt basis ($0.49/Wp) [25]. The high work function of CdTe (~5.7 eV) [26,27] presents a long-studied challenge to creating a low-barrier, pseudo-ohmic back contact for use in the CdS/CdTe architecture [28]. Standard back contact preparation involves the introduction of copper, as a thin evaporated layer or through a CuCl₂ solution deposition, followed by thermal diffusion to produce a highly-doped near-contact CdTe layer that narrows the barrier at the interface with a metal, such as Au [29,30]. However, several studies have shown that Cu diffuses readily, and over time reaches and crosses the CdS/CdTe interface, reducing the operating voltage of the device [30,31]. Copper diffusion therefore serves as a degradation pathway, influencing device performance over the life of a PV system and ultimately degrading the economic performance of the technology [32].

As-deposited FeS₂-NC films studied at room-temperature using four-point probe and Hall measurements showed low resistivity (~100 Ω cm), high free carrier concentrations (~10¹⁸ cm⁻³), and low mobility (~10⁻¹ cm² V⁻¹ s⁻¹). In addition, FeS₂ NCs clearly show sub-bandgap optical absorption, indicating a significant density of sub-bandgap electronic states arising from core and/or surface defects. Consequently, these films are not expected to be effective absorber layers in photovoltaic devices. Indeed, the highest...
open circuit voltage ($V_{OC}$) attained in Tributsch’s work was $\sim 0.28$ V, much lower than the theoretically attainable value of $\sim 0.5$ V [33]; the authors attributed the low photovoltage to both strong Fermi level pinning associated with surface states and to bulk defects caused by sulfur deficiency [34]. For purposes of comparison, note that CdTe, which has achieved a conversion efficiency of 21.5% under standard test conditions [35], typically exhibits a resistivity of $\sim 10^4 \Omega$ cm, a free carrier concentration of $\sim 10^{14}$ cm$^{-3}$, and a hole mobility of $\sim 10^2$ cm$^2$/V·s [36,37].

Although FeS$_2$ has not served well as a solar cell absorber layer, the high free carrier concentration and low resistivity observed in our FeS$_2$-NC films suggest promise as a conductive low barrier contact to CdTe. The work function of FeS$_2$ ($\phi \approx 5.0$ eV [4], 5.45 eV [38]) compares favorably with that of Au ($\phi \approx 4.9$–5.3 eV) [39–41] and indicates the possibility of a barrier-less or low-barrier interface at the CdTe/FeS$_2$ junction. As shown in Fig. 1, the non-interacting band diagram [10,42,43] indicates the possibility of a low-barrier interface at the CdTe/FeS$_2$ junction. For CdS/CdTe solar cells, conventional back contacts are commonly made with Cu/Au or Cu/graphite [44]. Graphene was employed as a back contact for thin film CdTe solar cells, but to narrow the barrier at the CdTe/graphene interface, either Cu or B was incorporated [45,46]. Phillips et al. recently reported that thin films of single wall carbon nanotubes (SWNTs) make stable, high-performance back contacts to CdTe without introduction of Cu [47]. Their analysis suggests that semiconducting SWNTs make ohmic contacts with p-CdTe while metallic SWNTs make rectifying contacts to p-CdTe. Our initial results applying FeS$_2$ NCs as the back contact to CdTe solar cells show substantial promise for this Cu-free and potentially low-cost approach.

Solution-based synthesis and deposition offers a potentially low-cost and scalable photovoltaic manufacturing method for large glass substrate processes and roll-to-roll processing on flexible substrates. Numerous routes have been followed to synthesize FeS$_2$ NCs [8–10], and we follow a solution-based approach based on thermal injection reaction, in the presence of trioctylphosphine oxide (TOPO) or 1,2-hexanediol, of an iron salt solution with an elemental sulfur source. While others have utilized FeCl$_2$ as the iron source, we have found that FeBr$_2$ yields improved results regarding crystal structure and infrared absorption [48]. We prepared drop-cast FeS$_2$ NC films using a layer-by-layer (LbL) method [20,49–51] in a nitrogen environment, with and without a process consisting of layer treatment by dipping into 1 M hydrazine in ethanol. The hydrazine-treated LbL films were more conducting due to the removal of the long insulating capping ligands.

2. Experimental details

2.1. Synthesis of FeS$_2$ NCs

As described elsewhere [48], synthesis of iron pyrite nanocrystals is performed in a Schlenk line under N$_2$ environment. In a typical synthesis, about 0.49 mmol of FeBr$_2$ and 1 mmol of trioctylphosphine oxide (or 135 μL of 1.2-hexanediol) are mixed in 10 mL of oleylamine (OLA) in a three neck flask under constant stirring. The FeBr$_2$ mixture is heated to 170 °C for ~2.5 h using a heating mantle; during this time, the sulfur precursor solution is prepared by dissolving 3 mmol of elemental sulfur in 5 mL of OLA. After 2.5 h of heating at 170 °C, the temperature of the FeBr$_2$ solution is raised toward 220 °C, and once it exceeds 216 °C, the sulfur solution is rapidly injected. Following two hours stirring at 220 °C, the NC solution is allowed to cool to room temperature, with continued stirring. Nanocrystals so obtained are washed a minimum of three times using methanol as a non-solvent and toluene as solvent. Finally NCs so obtained are dried under nitrogen gas flow.

2.2. FeS$_2$ film fabrication

To fabricate FeS$_2$-NC films, we utilize a drop-cast method in a layer-by-layer (LbL) process. We prepare an FeS$_2$ NC solution in chloroform at a concentration of ~6 mg/mL, and proceed with film formation, in an N$_2$ environment, as follows. A layer of drop-cast NCs is deposited onto the chosen substrate, and allowed to dry. At this point, the film can optionally be treated with hydrazine for ligand removal (vide infra). In the case of an untreated film, the film thickness may be increased by simply repeating the drop-cast process followed by the drying process; preparation of a ~1 μm film typically requires 2 layers.

Preparation of an FeS$_2$-NC film treated with hydrazine to remove the surfactant is accomplished as follows. Subsequent to the first drop-cast layer deposition, the film is allowed to dry in the N$_2$ environment. The film is subsequently submerged in a 1 M hydrazine solution in ethanol for ~2 min. The film is withdrawn from the hydrazine solution and immediately submerged into a pure ethanol solution to remove any residual surfactant or hydrazine – i.e., as a rinse. The film is then allowed to dry. To attain a thicker film, the drop-cast/dry/hydrazine/rinse/dry process may be repeated as necessary.

2.3. CdS/CdTe device fabrication

The CdS and CdTe layers were deposited by RF magnetron sputtering, or by closed spaced sublimation (CSS), onto NSG TEC™15 TCO-coated glass (Pilkington N.A.), which consists of a dielectric film stack including SiO$_2$, SnO$_2$, and SnO$_2$:F deposited on soda lime glass [52]. As compared with sputtered CdS/CdTe films, CSS yields films with larger initial CdTe grain size (2–5 μm vs. 200–300 nm) due to the higher substrate temperature (~600 °C for CSS vs. ~275 °C for sputtered films) [53,54]. Following the CdTe deposition, a CdCl$_2$ treatment was carried out by applying a saturated solution of CdCl$_2$–methanol and subsequently annealing at 387 °C for 30 min in dry air to advance grain growth, release interfacial strain, and facilitate sulfur and tellurium mixing at the CdS/CdTe interface [55]. The thickness of CdS films in both methods was ~80 nm whereas the sputtered CdTe was ~2.0 μm and the CSS CdTe was ~4 μm. Our sputtered CdS/CdTe films enable best device efficiency of ~12%, and our CSS CdS/CdTe films enable best device efficiency of ~14.0%; all device efficiency measurements are made under AM1.5G simulated solar spectrum at ambient laboratory temperature. Following CdS/CdTe deposition, the standard back contact consists of a Cu/Au sequential deposition in which ~3 nm
of Cu followed by ~30 nm of Au is evaporated onto the CdTe, and the film is then heated to 150 °C for 45 min in air to drive Cu diffusion (Fig. S1, Supporting information). Similarly for FeS$_2$/Au back contact deposition, 30 nm Au is thermally evaporated onto FeS$_2$ NC film deposited at room temperature. The device area of 0.08 cm$^2$ is defined by using a shadow metal mask while depositing Cu/Au back contact. Since FeS$_2$ NC film is conductive, shadow mask cannot be used while making Au layer on top of NC film. In this case, FeS$_2$/Au or Cu/FeS$_2$/Au composite layers are scribed manually or using laser scriber to define an area equal to 0.085 cm$^2$.

3. Results and discussion

The cross-sectional SEM image shown in Fig. 2 was obtained for a CdS/CdTe device prepared in a stepwise process on TEC™15 glass using RF magnetron sputtering deposition of CdS (~80 nm) and CdTe (~2 μm), standard CdCl$_2$ treatment, LBL drop-cast deposition of NC FeS$_2$, and evaporation of ~30 nm Au for improved electrical contact for $J$–$V$ and QE characterization. The treated and untreated films appear physically essentially indistinguishable in cross-sectional SEM as shown in Fig. S2. Current density/voltage ($J$–$V$) characteristics for best-performing sputtered CdTe devices prepared with different back contacts are shown in Fig. 3a, and average device parameters are presented in Table 1. These solar cells were prepared on 1" × 1" TEC™15-coated glass substrates.

Each sample set consists of multiple cells prepared on a single substrate, based on either shadow masking or scribing. Table 1 demonstrates average and standard deviations of 15 cells of each contact type. Each $J$–$V$ curve in Fig. 3a represents a highest efficiency curve from among the 15 cells, with solid curves representing measurements under simulated AM1.5 illumination, and dashed curves represent device measurements in the dark. The red curve represents the $J$–$V$ behavior when 30 nm Au was evaporated as a back contact, without any Cu diffusion layer. Although $J_{SC}$ does not drop significantly compared to the other back contact types, $V_{OC}$ and efficiency ($\eta$) are poor when Au alone is used as the CdTe contact. Because of CdTe’s electron affinity (~4.4 eV) [56], a high work function material is required to form a low barrier height contact with p-type CdTe. To attain an ohmic junction with p-type CdTe, a metal should exhibit a minimum work function of ~5.9 eV [29]. Since no metals have a sufficiently high work function, any metal forms a Schottky junction with non-zero barrier height when contacting CdTe at its typical free hole concentration. Because the CdTe/Au Schottky junction opposes the main diode

![Fig. 2. Cross-sectional SEM image, obtained by secondary electron detector, of drop-cast hydrazine-treated FeS$_2$-NC thin film contact formed on a sputtered CdS/CdTe thin film.](image)

formed at the CdS/CdTe interface [57], hole transport is limited at the back contact. The current–voltage curve “rolls over” at forward bias, decreasing both the fill factor and the open-circuit voltage [58]. The problem associated with using a metal-only (e.g. Au) back contact is alleviated by depositing ~3 nm Cu prior to deposition of ~30 nm of Au, and annealing the film at 150 °C for ~45 min. The Cu-rich layer reduces the width of the space charge region, thus narrowing the barrier width sufficiently to allow efficient carrier tunneling to the Au; the result is a pseudo-ohmic contact. Compared to the Au-only contact, the Cu/Au contact (Fig. 3a) improved the $V_{OC}$ from 0.68 V to 0.79 V, and efficiency improved from 8.7% to 11.4%.

When the Cu deposition and interdiffusion was replaced by a ~1.0 μm thick FeS$_2$ NC layer at room temperature and 30 nm thick Au atop of it (green curve, Fig. 3a), performance of the device approached that of the Cu/Au contact. The hydrazine-treated FeS$_2$ NC back contact showed a $V_{OC}=0.77$ V, $J_{SC}=21.7$ mA cm$^{-2}$, and 10.6% efficiency. In contrast with the Cu/Au contact, the device was never heated following the CdCl$_2$ activation step. The fill factor did decrease, as is evident in Fig. 3a, in accord with a residual barrier and a slight increase in the series resistance at the maximum power point. The increase in the $J_{SC}$ has been frequently observed for the FeS$_2$-NC contact, and tests are ongoing to confirm and understand the effect. One possible source of the increased $J_{SC}$ is a reduced interfacial recombination velocity at the back contact. We have also considered the possibility of a systematic error in the device area, but integration of the spectral response at short-
Table 1
Average parameters for three sets of 15 sputtered CdTe solar cells, with each set based on a different back contact. Each tested device was defined by either a shadow mask (device area=0.080 cm$^2$) in the case of the Au-only and Cu–Au contacts, or by mechanical or laser scribing (device area=0.085 cm$^2$) in the case of the FeS$_2$-NC/Au contact.

<table>
<thead>
<tr>
<th>Back contact</th>
<th>$V_{OC}$ (V)</th>
<th>$J_{SC}$ (mA cm$^{-2}$)</th>
<th>Fill factor (%)</th>
<th>Efficiency (%)</th>
<th>Series resistance ($\Omega$ cm$^2$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Au</td>
<td>0.65 ± 0.04</td>
<td>20.2 ± 0.9</td>
<td>59.3 ± 2.4</td>
<td>7.7 ± 0.8</td>
<td>7.7 ± 0.6</td>
</tr>
<tr>
<td>Cu/Au</td>
<td>0.79 ± 0.02</td>
<td>21.8 ± 0.7</td>
<td>65.4 ± 1.9</td>
<td>11.3 ± 0.9</td>
<td>5.4 ± 0.9</td>
</tr>
<tr>
<td>FeS$_2$-NC/Au</td>
<td>0.77 ± 0.01</td>
<td>22.4 ± 1.0</td>
<td>61.5 ± 1.0</td>
<td>10.6 ± 0.4</td>
<td>6.9 ± 0.4</td>
</tr>
</tbody>
</table>

We investigated the dependence of device performance on the FeS$_2$ NC thickness as a back contact, using CSS-deposited CdS/CdTe device structures [60]. The type of CdS/CdTe CSS depositions utilized in our studies showed a champion cell efficiency of 14.3% under STC for the standard Cu/Au back contact. We studied the effect of replacing the Cu with varying thicknesses of FeS$_2$ NC layers (0.35 μm to 1.4 μm), fixing the other device architecture and processing parameters. As above, a ~30 nm Au layer was deposited following fabrication of the hydrazine-treated FeS$_2$-NC film. The performance parameters of these devices were compared with a standard device using Cu/Au as a back contact. Current density vs. voltage curves for all four device designs along with their performance parameters for the best cells are shown in Fig. 5. Detailed performance parameters and statistics are provided in Table S2 in S.I. Current density vs. voltage tests show that thin FeS$_2$ layers (0.35 μm) yield poor performance due to lower $V_{OC}$, perhaps due to an increased density of pinholes through which Au may contact the CdTe directly. The thickest FeS$_2$-NC film (1.4 μm) showed an increased effective series resistance and a slightly decreased $V_{OC}$. Comparatively low photo-conversion efficiency of the device using a back contact of 0.35 μm thick FeS$_2$ with 30 nm of Au is primarily due to the low $V_{OC}$; in contrast, the thickest FeS$_2$ contact layer showed a good $V_{OC}$ but $J_{SC}$ and FF were reduced, as was the overall device efficiency. Our measurements showed an optimal device performance for FeS$_2$ NC layer thickness of ~0.7 μm, with an efficiency ~13% below that of the champion Cu/Au back contact.
Au back contact device (the champion device resulted from separate studies, but under nominally identical processing conditions).

To determine whether hydrazine treatment alone affects the CdS/CdTe material and influences device performance, two standard solar cell device samples (~15 individual devices each) were prepared using sputtered CdS/CdTe, without the presence of the FeS$_2$ layer. Before back contact deposition, one of the devices was treated by soaking in 1 M hydrazine solution for two minutes followed by an ethanol rinse and drying in N$_2$. The Cu/Au back contact deposition was then completed using the normal procedure. Fig. S5 displays representative $J$–$V$ characterizations of these completed devices. We observe statistically identical performance between the data sets, indicating that hydrazine alone does not measurably influence the PV properties of the CdTe. Although during FeS$_2$ NC deposition the hydrazine treatment likely does reach the CdTe (e.g., through pinholes following the first layer of NC FeS$_2$), the degree of contact is reduced with each subsequent drop-cast layer of FeS$_2$.

In order to understand the contact and device behavior, we have characterized the electrical properties of FeS$_2$ NC films using hot probe measurements as well as four point probe and Hall measurement methods [48]. All hot probe measurements indicated clearly that the films were p-type in agreement with reports from other polycrystalline and NC-based FeS$_2$ films [8,10,61], indicating that the majority charge carriers in our pyrite films are holes. Four point probe measurement of untreated NC films revealed a typical sheet resistance of $\sim 10^5 \, \Omega$/□ and resistivity of $\sim 100$–200 $\Omega$ cm. For hydrazine treated films, sheet resistance decreased to $\sim 10^4 \, \Omega$/□ and resistivity to $\sim 10$–20 $\Omega$ cm. It is understood that Hall measurements made on polycrystalline films reveal the carrier concentration of the individual grains and the conductivity as determined by grain boundary scattering [62]. Our sheet resistance values measured by Hall effect and by four point probe generally agree quite well. The improved conductivity of the hydrazine treated films correlates with the removal of the surfactant molecules from the surface of the NCs which insulate neighboring NCs and inhibit efficient carrier transport. Though the overall FeS$_2$ NC stoichiometry is nearly ideal, we believe that the surfaces of the nanocubes are sulfur-rich and largely responsible for the p-type behavior we observe. The surface area to volume ratio is relatively high for these samples, and the significant surface-related contribution to the defect-based carrier density yields the near-degenerate p-type doping that supports formation of low-barrier contact to p-CdTe.

Comparing the performance of devices based on FeS$_2$ NC contacts prepared with and without hydrazine treatment reveals additional insights into the role played by the surfactant molecules, and the improvements resulting from their removal. During the synthesis of these NCs, TOPO controls the growth rate, influences the size and shape of the NCs, and caps the NC surface. The ~2 nm length of the TOPO molecules sheathe the NCs and maintain isolation between neighboring NCs within the film [63]. Cube-shaped FeS$_2$ NCs capped with TOPO molecules are displayed in Fig. 6a. As a strong reducing agent, hydrazine scavenges oxygen which breaks the linkage between the TOPO and the NC (Fig. S6). As TOPO is removed from the film, the NC films densify and the NC–NC electrical transport improves. Devices prepared with FeS$_2$ NC layers omitting hydrazine treatment exhibit dramatically different current–voltage behavior. Fig. 6b presents $J$–$V$ curves for sputtered CdTe solar cells prepared with back contacts of untreated FeS$_2$–NC layers with Au. The untreated film shows a clear S–shape signature, indicative of a transport barrier formed at the back contact. Similar “S-kink” shapes have been previously observed under illumination in CIS and also in organic solar cells (OSCs) when a charge buildup occurs at one contact [64–66], and recent work has sought to explain and suppress the effect in OSCs [67]. Inflection points in the $J$–$V$ curves observed in some organic solar cells are attributed to charge transport layers energetically misaligned to the energy levels of the active materials in planar heterojunction solar cells. In our case, the proposed charging behavior is attributed to the residual barrier at the CdTe/FeS$_2$–NC interface (possibly exacerbated by the low-mobility of the FeS$_2$ NC film) resulting in the S-kink behavior.
We note that the S-kink shape also appears, albeit subtly, in the illuminated J–V curves for the two thickest FeS2–NC contact devices in Fig. 5. The quantity d2J/dV2 dips briefly to negative values in the power generating quadrant for nearly all FeS2–NC/Au back contact devices (Figs. S7 and S8).

Within the power-generating quadrant, electron–hole pairs photogenerated within the CdTe separate under the built-in field and move toward their respective electrodes. The TOPO-capped FeS2 NC film presents a significant barrier to hole transport at the interface with CdTe, such that photogenerated carriers near the CdS/CdTe interface or within the CdTe layer experience impeded transport once reaching the FeS2 NC layer. This is due to the insulating inter-NC surfactant layer which persists when the FeS2–NC layer is untreated, or incompletely treated, with hydrazine. In such a case, holes may accumulate at the CdTe/FeS2–NC interface and produce a barrier which is overcome only at sufficiently high forward bias for which the dark current dominates total current. Consequently, as the bias increases in the power-generating quadrant and the built-in field decreases, the device current drops due to the effective back contact diode established at the CdTe/FeS2–NC interface. The hole transfer barrier and charge accumulation increase recombination and decrease current, resulting in an S-kink. For such devices to function optimally, one must sufficiently lower the effective resistance at the CdTe/FeS2–NC interface through a deep valence band edge and a high free hole concentration; these conditions are achieved with the FeS2 NC film after the hydrazine treatment.

We have also investigated the role played by Cu in an FeS2–NC/Au back contact design: Two sets of devices were fabricated where three-layer back contacts were deposited: 3 nm Cu/1 μm FeS2/45 nm Au. This experiment was done for both sputtered and CSS CdTe devices. In this case, as shown in Fig. 7, the presence of the FeS2 NC layer improved the performance of the devices compared with that of the standard Cu/Au back contact devices (statistics of 20 cells in each case are provided in S.I.). The thin layer of Cu utilized in the standard Cu/Au back contact improves performance by increasing the CdTe free hole concentration, one effect of which is a narrowing of the residual back barrier and improved hole tunneling efficiency, however, this does not completely make the barrier ohmic. When the FeS2–NC layer is deposited onto it and the device is completed with Au, the performance of the device increases. Copper narrows the barrier width, and FeS2 reduces the shunt resistance and increases Voc, perhaps because the FeS2 NC layer serves as a buffer layer with a relatively high work function. The device efficiency improves by ~5–9% when incorporating a FeS2 NC layer between the Cu-treated CdTe and the Au external contact layer.

4. Conclusion

We have achieved a high degree of control over the crystallinity and stoichiometry of iron pyrite nanocubes, and recognition that the high work function of FeS2 and the high free hole concentration of our FeS2 NC films have enabled their implementation as a low-barrier back contact to CdTe within a Cu-free architecture. This work illustrates the application of FeS2 NC as a low barrier electrical contact to thin film solar cells without the intentional preparation of a p+ highly-doped CdTe or Cu2+Te layer that would serve to narrow any barrier width and facilitate tunneling. Our results show promise for the development of an iron-based contact material, and suggests the investigation of other related pyrites such as FeTe2 and FeSe2 as potential electronic materials relevant to the PV industry. Additional optimization of transport through the FeS2 NC contact layer may further improve performance via removal of any mobility-induced resistance. The FeS2 layer is easily prepared using a room-temperature and solution-based approach, and initial accelerated lifetime tests indicated stability comparable to that of the laboratory standard Cu/Au contact.

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Appendix A. Supplementary material

Supplementary data associated with this article can be found in the online version at http://dx.doi.org/10.1016/j.solmat.2015.03.032.

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