Herein, the scalable chemical bath deposited NiO$_x$-NiSO$_4$ heterostructured films are reported as the efficient hole transport layers (HTLs) in perovskite solar cells. The NiO$_x$-NiSO$_4$ films show excellent hole extraction ability and reduce interfacial charge recombination in solar cell devices. By using NiO$_x$-NiSO$_4$ HTLs, a high power conversion efficiency of 20.55% is obtained, which is about 12.23% greater than that of the pure NiO$_x$ transport layer. This study provides a simple solution-processing route toward the large-area production and fabrication of full inorganic transport layers for perovskite photovoltaics.

1. Introduction

Lead halide hybrid perovskites are strong competitors for next-generation solar cells, benefiting from their broadband light absorption,[1] high absorption coefficient,[2] long carrier lifetime,[3] and feasible solution deposition.[4] Perovskite solar cells (PSCs) have advanced the efficiency space, which from 3.8% in 2009 to 25.7% within the last few years.[5] The typical PSCs comprise of perovskite absorbers, charge transport materials, and metallic contacts, in which the charge transport materials selectively extract the charge carriers and block the counter charges.[6] Recent progress in power conversion efficiency (PCE) heavily depended on the advancement of electron transport layers, such as the synthesis of n-type SnO$_2$[7] or TiO$_2$.[8] In contrast, the inorganic hole transport layers (HTLs) have made very limited success, whose efficiencies were generally lower than that of the organic ones.

The current high-efficiency solar cells are mostly employing 2,2',7,7'-tetakis[N,N-di[4-methoxyphenyl]amino]-9,9'-spirobifluorene (spiro-OMeTAD)[9] or poly[bis(4-phenyl)][2,4,6-trimethylphenyl]amine (PTAA)[10] as HTLs, which are expensive and intrinsically instable. Respectable p-type semiconductors, for example, Cu$_2$O,[11] CuSCN,[12] CuI,[13] and V$_2$O$_5$[14] with both chemical stability and cost-effectiveness, have been used as HTLs in inverted heterojunction PSCs. In these HTLs, NiO$_x$ has wide band gap,[15] acceptable carrier mobility,[16] and appropriate valance band edges.[17] Nowadays, NiO$_x$ films can be prepared by a great diversity of deposition techniques, e.g., magnetron sputtering,[18] spin coating,[19] atomic layer deposition,[20] sol–gel deposition,[21] pulsed laser deposition,[22] and physical vapor deposition.[23] However, a big challenge still remains to fabricate large-area NiO$_x$ films by using cost-effective solution process.[24] Another critical issue is the defective surface of NiO$_x$ films, which causes intense interfacial charge recombination.[25] Recent studies have shown the superior passivation effect of oxysalt in perovskite devices,[26] which offers a new option for modulating the electronic structure of HTLs.

Here, we report a NiO$_x$-NiSO$_4$ heterostructured layer by a chemical bath deposition (CBD) as efficient HTLs in perovskite photovoltaics. Such method provides a low-cost solution and low-temperature process for the preparation of NiO$_x$-based HTLs, which also gain scalable and compact films without the inherent weakness in spin-coating. The NiO$_x$-NiSO$_4$ HTLs showed excellent hole extraction ability and reduced interfacial charge recombination in PSCs. As a result, perovskite devices based on NiO$_x$-NiSO$_4$ HTLs achieved a high PCE of 20.55%, which was about 1.2 times higher than the solar cells based on pure NiO$_x$ HTLs.

2. Results and Discussion

The NiO$_x$-NiSO$_4$ heterostructured layer was deposited by gradual hydrolysis of nickel chloride with the presence of thiourea and ammonia water at 75 °C. After thermal annealing, the product shows uniform morphologies that comprises nanosized particles as depicted by the images of scanning electron microscopy (SEM) in Figure 1a. The grain size of NiO$_x$-NiSO$_4$ film is estimated to be 10–30 nm. By prolonging the reaction time, small particles join together and form large aggregates at 60 min (Figure S1,
Supporting Information). The crystalline phase of the films is illustrated by X-Ray diffraction (XRD) in Figure 1b. The (111), (200), and (220) planes of NiO can be confirmed, when the characteristic diffraction peaks at 37.2°, 43.3°, and 62.9° are observed (space group: Fm-3m, JCPDS file No.47-1049), while the diffraction peaks at 20.6°, 22.7°, 25.0°, 26.7°, 34.8°, and 35.1°, matching well with the (110), (020), (111), (021), (200), and (112) lattice planes of NiSO4 (space group: Cmcm, JCPDS file No.13-0435). The measurement of X-Ray photoelectron spectroscopy (XPS) was then conducted to probe the chemical components of the NiO\x-NiSO4 layer and the valence states of Ni element. The Ni 2p core electron in XPS spectrum displays binding energies at about 854.4 and 852.2 eV (Figure 1c), which correspond to Ni2+ and Ni3+ species for the NiO\x-NiSO4 films.

The presence of S is also confirmed by the S 2p XPS spectrum in Figure 1d, in which the peak of S—O bond at about 168.2 eV can be clearly observed. The structural characterizations have proved the successful synthesis of NiO\x-NiSO4 heterostructured HTLs by our CBD methods. We subsequently fabricated solar cell device structured as fluorine-doped tin oxide (FTO)/NiO\x or NiO\x-NiSO4/CH3NH3PbI3 (MAPbI3)/[6,6]-phenyl-C61-butyric acid methyl ester (PC61BM)/bathocuproine (BCP)/Ag. The spin-coated NiO\x HTLs were used as standard samples for comparison (see more information in Experimental Section). The effect of CBD reaction time was first investigated. Figure S2, Supporting Information, shows similar morphology of MAPbI3 films with the CBD reaction time of 20, 40, and 60 min. Current density–voltage (J–V) curves of devices based on the HTLs with multiple CBD reaction times are represented in Figure 2a tested with a 0.15 V s\(^{-1}\) scan speed in reverse direction under AM 1.5G illumination. It was found that the longer CBD reaction time of over 40 min was required for high fill factor (FF) and open-circuit voltage (VOC), which could be ascribed to the fully coverage on the FTO substrates. The champion cell based on NiO\x-NiSO4 HTLs delivers excellent photovoltaic parameters than the solar cell with NiO\x HTLs, especially in FF (Figure 2b and Table 1). The short-circuit photocurrent density (JSC) values of both devices also match well with the integrated photocurrent from the spectra of external quantum efficiency (EQE) (Figure 2c). The devices employing NiO\x and NiO\x-NiSO4 HTLs obtain a stabilized photocurrent (21.12 and 19.09 mA cm\(^{-2}\)) under simulated one sun illumination at the maximum power point (MPP), which correspond to the stable PCEs of 19.85% and 17.31%, respectively (Figure 2d). The increased JSC can be associated with the improvement in light absorption and interfacial carrier extraction. The light absorption spectra of NiO\x and NiO\x-NiSO4 films are very close (Figure S3a, Supporting Information). However, there is an evident promotion in light absorption from MAPbI3 film, which is deposited on NiO\x-NiSO4 HTLs. This can be interpreted as the enhanced quality of perovskite on NiO\x-NiSO4 HTLs (Figure S3b, Supporting Information). Therefore, such HTL not only improved the quality of perovskite but also optimized the interface between NiO\x-NiSO4 HTLs and perovskite with optimized charge collection.

To scrutinize the carrier transfer at perovskite/HTLs interface, we carried out the measurements of photoluminescence (PL) and time-resolved photoluminescence (TRPL). As shown in Figure 3a, the PL intensity of MAPbI3 film deposited on NiO\x-NiSO4 film is mildly stronger than that on NiO\x film, which indicates the NiSO4 can neutralize the interfacial defects and
reduce the nonradiative recombination velocity.\[^{27}\] In fact, the sulfate anions can coordinate with surface cationic Pb centers of perovskite to passivate corresponding trap states.\[^{1}\] We also operated thermal admittance spectroscopy (TAS) measurement to prove this. The PSC with NiO\(_x\)-NiSO\(_4\) HTL has much lower trap density of state (tDOS) than the NiO\(_x\) ones at the range from 0.33 to 0.46 eV (Figure S4, Supporting Information). TRPL curves of different HTLs in Figure 3b can be fit by a biexponential function,\[^{22}\] which can obtain the slow and fast delay lifetimes. The fast decay lifetimes of NiO\(_x\)-NiSO\(_4\) HTLs samples (2.4 ns) were shorter than the NiO\(_x\) ones (4.9 ns), revealing a shorter decay time and a faster decay rate for the perovskite films on NiO\(_x\)-NiSO\(_4\) HTLs. We also calculate the average carrier lifetimes, which indicate its good ability of carrier extraction. The average carrier lifetimes of NiO\(_x\)-NiSO\(_4\) and NiO\(_x\) HTLs were 40.1 and 43.2 ns, respectively. To explain why the charge extraction was accelerated, we prepared lateral devices with structures of FTO/HTLs/Au to obtain the conductivity data of HTLs (Figure S5, Supporting Information). We found that the bilayer of NiO\(_x\) and NiSO\(_4\) has a poor electrical conductivity than pure NiO\(_x\) HTL. However, the current density of NiO\(_x\)-NiSO\(_4\) HTLs shows a rising tendency at the same forward bias, revealing higher conductivity than others. This phenomenon can be explained by the formation of NiO\(_x\)-NiSO\(_4\) heterostructured...

| HTLs       | \(V_{OC} \) [V] | \(J_{SC} \) [mA cm\(^{-2}\)] | FF [%] | PCE [%] |
|------------|-----------------|-------------------------------|--------|---------|
| NiO\(_x\)-NiSO\(_4\) | 1.12            | 23.08                         | 79.50  | 20.55   |
| NiO\(_x\)   | 1.11            | 22.30                         | 74.30  | 18.31   |

Table 1. Photovoltaic parameters of solar cells with the NiO\(_x\)-NiSO\(_4\) and NiO\(_x\) HTLs.

Figure 2. a) \(J-V\) measurements of PSCs based on different CBD reaction times with NiO\(_x\)-NiSO\(_4\) HTLs tested under AM 1.5G illumination. b) The \(J-V\) measurements of the champion NiO\(_x\)-NiSO\(_4\) and NiO\(_x\) PSCs. c) EQE spectra and integrated photocurrent of the NiO\(_x\)-NiSO\(_4\) and NiO\(_x\) PSCs. d) Stabilized power output of the photocurrent with the champion NiO\(_x\)-NiSO\(_4\) and NiO\(_x\) PSCs at the MPP voltage of 0.94 and 0.91 V, respectively.

Figure 3. a) Steady-state PL spectra and b) TRPL decays of perovskite films deposited on NiO\(_x\) and NiO\(_x\)-NiSO\(_4\) films.
HTLs. In this structure, NiSO₄ species are likely to play an important role in confining the charge carriers within NiO network and shortening the transmission path of charge carriers. The increased conductivity resulted in an improved charge extraction rate and a suppressed nonradiative recombination.

To further explore the behavior of carrier recombination in the as-fabricated PSCs, we then collected the data of dependence between incident light intensity and $V_{OC}$. By fitting log-scaled light intensity versus $V_{OC}$ values linearly (Figure 4a), a slope of $1.837k_B/\tau_0$ for the device with NiO$_x$ and a flatter one of $1.382k_B/\tau_0$ for the device with NiO$_x$-NiSO$_4$ are obtained, where $k_B$ is defined as the Boltzmann constant, $\tau_0$ denotes the absolute temperature, and $q$ is elementary charge. The slope (ideality factor, $n_{id}$) of 1 normally denotes a radiative recombination of free holes and electrons in solar cells, whereas $n_{id} = 2$ indicates a trap-assisted Shockley–Read–Hall recombination process. The results here pinpoint the dominating role of bimolecular recombination channel in NiO$_x$-NiSO$_4$ HTLs based devices. Figure 4b shows the electrochemical impedance spectra (EIS) spectra of as-fabricated devices. We found that PSCs with NiO$_x$-NiSO$_4$ HTLs at the low-frequency region exhibited a larger semicircle than NiO$_x$ based devices, which is a signature of large recombination resistance ($R_{rec}$). The dramatically suppressed charge recombination was also revealed by transient photovoltage decay (TPV) measurements. As shown in Figure 4c, the average carrier lifetime is fitted to be 2.8 and 4.8 $\mu$s, respectively. The device with NiO$_x$-NiSO$_4$ HTLs exhibited large recombination resistance and long carrier lifetime, which can be attributed to the sulfate anions in NiO$_x$-NiSO$_4$ HTLs. The effect of defect passivation was achieved by the coordination between sulfate anions and Pb cations. Carrier lifetime was improved and carrier recombination was suppressed, which resulted in the improved $V_{OC}$.

3. Conclusion

In summary, a CBD method for facile, large-area deposition of efficient NiO$_x$-NiSO$_4$ heterostructured HTLs has been developed. The PSCs device based on NiO$_x$-NiSO$_4$ HTLs obtained a high PCE of 20.55%, which was much higher than the device based on pure NiO$_x$ HTLs (18.31%). Benefiting from the CBD process, the prepared devices exhibited enhanced stability, repeatability and should be compatible with large-area production. Our preliminary results have verified the compatibility of these HTLs with large-area solar cells (Figure S6, Supporting Information). The device may be further increased by optimizing the morphology of the heterostructures, such as the phase distribution, crystalline grain size, and aggregate structure. Other nickel-based oxysalts are also promising in the application of transport layers. Our study provides a new option for the large area and reliable production of inorganic HTLs for perovskite-based photovoltaics.

4. Experimental Section

Materials: The solution of methylamine (33 wt% in absolute ethanol), dimethyl sulfoxide (DMSO, 99.9%), and iso-propanol (IPA, 99.5%) was obtained from Sigma-Aldrich. N,N-dimethyleformamide (DMF, 99%), 2-methoxyethanol (99%), and lead iodide (PbI$_2$, 99.9985%) were received from Alfa Aesar. Hydroiodic acid (HI, 57 wt% aqueous solution) was purchased from J&K Scientific Ltd. Hydrochloric acid (HCl, 36–38%) and ethanol (AR, 99.7%) were purchased from Shanghai Titan Technology Co., Ltd. Zinc powder and nickel (II) acetate trihydrate were obtained from Sinopharm Chemical Reagent Co., Ltd. The power of BCP (99%) and PC$_{60}$BM (99.5%) was received from Nichem chemicals. FTO (8 $\Omega$ per square) was purchased from Nippon Sheet Glass Co., Ltd. Ammonia water was purchased from Shanghai Lingfeng Technology Co., Ltd. Thiourea was purchased from Fluka. Chlorobenzene (CB, 99.5%) was obtained from Aladdin.

Preparation of HTL Films: Nickel chloride (0.714 g) and thiourea (0.516 g) were dissolved in deionized (DI, 30 mL) water. Then, ammonia (2 mL) water was added, in order to adjust the value of pH (>7) after the powder complete dissolving. The cleaned FTO substrates (cleaned by the solution of detergent, acetone, and ethanol) were immersed into the mixed solution and subsequently placed on the hot plate at 70 °C with different processing time (20, 40, and 60 min). The treated FTO substrates were rinsed with DI water and ethanol. Finally, the substrates were heated at different temperature (150, 300, and 500 °C) for 60 min. NiO$_x$ films were prepared on the basis of the reported method.

Device Fabrication: PbI$_2$ powders (1.1986 g) were added into DMSO (185 $\mu$L) and DMF (2 mL). CH$_3$NH$_3$I (80 mg MAI synthesized as the reported method) powders were dissolved in IPA (2 mL). Before using, they are all stirred at 45 °C for 12 h. The precursor of PbI$_2$ was coated on the NiO$_x$ or NiO$_x$-NiSO$_4$ films (3000 rpm, 30 s), then spinning coating MAI solution on the PbI$_2$ films (5000 rpm, 30 s), after that the films annealing at 115 °C for 10 min. The charge-transport layer (ETL) was prepared by spin-coating PC$_{60}$BM solution (20 mg mL$^{-1}$, dissolved in CB) at 2000 rpm for 45 s and BCP solution (0.5 mg mL$^{-1}$, dissolved in ethanol) at 4000 rpm for 45 s, then heated at 70 °C for 15 min. Finally, a metal electrode
(Ag, 100 nm) was used in the method of thermal evaporation to deposit on the ETL.

**Characterization:** Field-emission SEM characterized the morphology of hole transfer layer films and perovskite samples using Hitachi S4800. Powder XRD patterns were used to analyze the crystallographic information of hole transfer layer films by Bruker Advance D8 X-Ray diffractometer (Cu Kα radiation, 40 kV). XPS was conducted to explore the elemental valence states and composition of the hole transfer layer films using PHI5300 (Mg anode, 250 W, 14 kV). Cary 500 UV–Vis–NIR spectrophotometer was equipped to collect the absorption spectra of HTL films and MAPbI₃ films. The PL measurement was acquired at room temperature using the spectrophotometer (Ocean optics QE pro) with a light source (365 nm). TRPL spectra were obtained by the high-resolution photoluminescence spectrophotometer.

J–V measurements were conducted under AM 1.5G irradiation (calibrate by a standard Si cell) by a Keithley 2400 digital source meter with a scan speed of 0.15 V s⁻¹. The active areas (0.0625 and 1 cm²) of the devices were defined using the metal masked. Newport-74 125 system was used to measure the EQE. The steady-state photocurrent output was measured at MPP for 300 s. The electrochemical workstation (Parstat 2273, Princeton) was used to measure EIS under different positive bias voltages from 100 000 to 1 Hz at dark conditions. An attenuated laser pulse was used to obtain the data of TPV. I–V measurements for NiO and NiO₂·NiSO₄ films were based on the FTO/HTLs/Au structures. TAS was performed to detect the tDOS.

**Supporting Information**

Supporting Information is available from the Wiley Online Library or from the author.

**Acknowledgements**

B.S. and M.C. contributed equally to this work. This work was financially supported by National Ten Thousand Talent Program for Young Top-notch Talent, National Natural Science Fund for Excellent Young Scholars (52020203), National Natural Science Fund for Distinguished Young Scholars (51725201), National Natural Science Foundation of China (51972111, 51902185), International (Regional) Cooperation and Exchange Projects of the National Natural Science Foundation of China (51920105005), Innovation Program of Shanghai Municipal Education Commission (E00014), the Fundamental Research Funds for the Central Universities (JKM01221621), the Fundamental Research Funds of Free Exploration of Science, Technology and Innovation Commission of Shenzhen Municipality (2021szzdp039), Shanghai Municipal Natural Science Foundation (22ZR1418000), and Shanghai Engineering Research Center of Hierarchical Nanomaterials (18DZZ252400).

**Conflict of Interest**

The authors declare no conflict of interest.

**Data Availability Statement**

The data that support the findings of this study are available from the corresponding author upon reasonable request.

**Keywords**

chemical bath depositions, hole transport layers, perovskites, solar cells

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