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# Solution-processed Sr-doped $NiO_x$ as hole transport layer for efficient and stable perovskite solar cells



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Jiankai Zhang<sup>a</sup>, Wujian Mao<sup>a</sup>, Xian Hou<sup>a</sup>, Jiaji Duan<sup>a</sup>, Jianping Zhou<sup>b</sup>, Sumei Huang<sup>a</sup>, Wei Ou-Yang<sup>a</sup>, Xuehua Zhang<sup>c</sup>, Zhuo Sun<sup>a</sup>, Xiaohong Chen<sup>a,\*</sup>

<sup>a</sup> Engineering Research Center for Nanophotonics & Advanced Instrument, Ministry of Education, School of Physics and Materials Science, East China Normal University, Shanghai 200062, China

<sup>b</sup> School of Automation Engineering, Shanghai University of Electric Power, Shanghai 200090, China

<sup>c</sup> Department of Chemical & Material Engineering, University of Alberta, Edmonton, Alberta T6G 1H9, Canada

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### ABSTRACT

Nickel oxide (NiO<sub>x</sub>) hole transport layer (HTL)-based planar perovskite solar cells (PSCs) have attracted much attention due to high power conversion efficiency (PCE) and simple processing. In this work, smooth and compact Sr-doped NiO<sub>x</sub> films with different Sr doping concentration were successfully prepared through a simple low temperature sol-gel method. The 1 at.% Sr-doped NiO<sub>x</sub> HTL-based PSCs exhibited the best performance with PCE of 20.07%, which is greatly higher than PCE of reference NiO<sub>x</sub> based PSCs (15.73%). Furthermore, the unencapsulated PSCs based on Sr:NiO<sub>x</sub> HTL still retains over 60% of the original PCE value aging for 100 days under ambient air, showing better stability. The superior performance of Sr-doped NiO<sub>x</sub> based PSCs is attributed to better electrical conductivity, crystallinity of perovskite film and energy level matching with perovskite layer, which can greatly improve hole transport and extraction abilities and reduce carrier recombination, resulting in high PCE and better stability.

#### 1. Introduction

Perovskite solar cells (PSCs) have attracted much attention because of their easy solution preparation and superior photovoltaic performance (Chen et al., 2016; Lin et al., 2016; Stranks et al., 2013; Xing et al., 2013). The power conversion efficiencies (PCEs) of PSCs have increased from 3.8% to 23.3% within several years, which makes it possible to be the main force in the next generation photovoltaic for sustainable energy (Cai et al., 2018; Kojima et al., 2009; Peng et al., 2018; Wang and Yang, 2016). Among all device architectures have been explored, the p-i-n configuration planar PSCs are more suitable to fabricate in low temperature process because the mesoscopic PSCs usually need sinter temperature over 400 °C to fabricate TiO<sub>2</sub> films (Liu et al., 2018; Zhang et al., 2018). The lower preparation temperature helps to reduce cost and fabricate flexible PSCs. Therefore, finding HTL materials right for low annealing temperature is quite important to obtain high performance planar PSCs of p-i-n configuration.

To date, many conducting polymers such as poly(3,4-ethylene dioxythiophene):poly(4-styrenesulfonate) (PEDOT:PSS) and poly(triarylamine) (PTAA), annealing at low temperature of less 150 °C, were widely used as hole transport layers (HTLs) in p-i-n configuration

planar perovskite devices due to their decent light transmittance and excellent hole transport capability (Hou et al., 2017; Lin et al., 2016; Shen et al., 2017; Wu et al., 2017). But their natural instability and relative high cost of synthesis limit their extensive use for commercialization (Jørgensen et al., 2008). For comparison, the inorganic material such as nickel oxide (NiO<sub>x</sub>) is an ideal HTL for p-i-n configuration planar PSCs due to its low temperature process, easy fabrication and chemical stability (Manders et al., 2013; Wang, C. et al., 2018a; Zhang et al., 2018). But regrettably, previous reports show that the pristine NiO<sub>x</sub> HTL is not satisfactory to fabricate high performance PSCs because of its relative low conductivity and surface defects (Chen et al., 2015a; Kim Yong et al., 2011), which would limit the hole transport and extraction, and increase carriers recombination (Corani et al., 2016). Thus, the element doping strategies have been developed to improve the electrical conductivity and reduce trap states of NiOx by some research groups (Chen et al., 2017; Jung et al., 2015; Kim et al., 2015; Liu et al., 2016). The NiO<sub>x</sub> co-doped with Lithium (Li) and Copper (Cu) have been demonstrated to greatly improve the electrical conductivity of NiO<sub>x</sub> layer under annealing at 500 °C (Liu et al., 2016). Jen et al. prepared p-i-n configuration planar PSCs based on copper-doped NiO<sub>x</sub> HTL with PCE up to 15.4% due to its increased electronic conductivity

\* Corresponding author.

E-mail address: xhchen@phy.ecnu.edu.cn (X. Chen).

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**Fig. 1.** (a) Current density-voltage (J-V) characteristics of PSCs with different Sr content for Sr:NiO<sub>x</sub> HTL under standard test conditions (AM 1.5G, 100 mW/cm<sup>2</sup>). All devices were unencapsulated and measured in ambient air. (b) UPS valence band edge and secondary electron emission cut off measured for Sr:NiO<sub>x</sub> film. (c) Optical band gap of Sr:NiO<sub>x</sub> film. (d) Energy diagram of each layer of PSCs.

and hole extraction ability (Kim et al., 2015). Han et al. developed  $Li_{0.05}Mg_{0.15}Ni_{0.85}O$  HTL annealing at 500 °C for p-i-n configuration planar PSCs, which greatly reduced the series resistance ( $R_s$ ) and increased the shunt resistance ( $R_{sh}$ ), resulting in an excellent fill factor over 0.8 and PCE of 18.3% (Chen et al., 2015b). All PSCs with these doping NiO<sub>x</sub> HTL achieved impressive PCEs due to the improved conductivity and reduced trap states. However, the high temperature annealing over 400 °C are usually required to achieve those high crystallinity doped NiO<sub>x</sub> films, which would increase the cost of fabrication and flexible devices. He et al used Cesium (Cs) doped NiO<sub>x</sub> with annealing at 275 °C as HTL for PSCs, which got impressive PCE of 19.35% and high stability (Chen et al., 2017), indicating that the superior doped NiO<sub>x</sub> HTL can be achieved by doping right element under the relative low annealing temperature.

Here, we further developed Sr-doped NiO<sub>x</sub> layers as HTL with lower temperature at 235 °C, which show better electrical conductivity and energy level matching with perovskite layer. Therefore, PSCs using 1 at. % Sr-doped NiO<sub>x</sub> as HTL exhibit the higher V<sub>oc</sub> of 1.118 V, FF of 79.1%, resulting in PCE of 20.07%, which is greatly higher than that of the reference cells. Furthermore, PSCs with 1 at.% Sr-doped NiO<sub>x</sub> HTL show better stability because it boosts the crystallinity of the perovskite layer.

#### 2. Experimental section

All solvents and reagents were used as received. Fluorine-doped tin oxide (FTO) glass (thickness: 2.2 mm, sheet resistance:  $15 \Omega \Box^{-1}$ ) were purchased from Shanghai MaterWin New Materials Co., Ltd. Formamidine iodide (FAI), Methylamine bromide (MABr) were

purchased from Borun New Material Technology Ltd. Methylamine iodide (MAI) was purchased from 1-Material-Organic-Nano Electronic. Lead (II) iodide (PbI<sub>2</sub>, 99%) [6,6]-phenyl-C<sub>61</sub>-butyric acid methyl ester (PCBM, 99%), dimethyl sulphoxide (DMSO, AR 99%) and  $\gamma$ -Butyrolactone (GBL, AR 99%) were purchased from Aladdin company (Shanghai, China). Nickel acetate tetra hydrate (98%), strontium chloride (SrCl<sub>2</sub>, 99%), chlorobenzene (CB, 99.5%), 2-methoxyethanol (99%) and ethanolamine (EA, 99%) were purchased from Sinopharm Chemical Reagent Co., Ltd.

Perovskite (FA<sub>0.15</sub>MA<sub>0.85</sub>Pb(I<sub>0.95</sub>Br<sub>0.05</sub>)<sub>3</sub>) precursor solution was prepared by dissolving FAI, MABr, MAI and lead (II) iodide in a mixture of DMSO and GBL (3:7 v/v) with concentration of 1.3 mol per liter. NiO<sub>x</sub> solution was prepared by dissolving nickel acetate tetra hydrate in 2-methoxyethanol and ethanolamine (EA) with concentration of 0.1 mol per liter, the molar ratio of  $Ni^{2+}$ /EA was maintained at 1:1. For the Sr-doped NiOx, SrCl<sub>2</sub> dissolved in 2-methoxyethanol are mixed with the NiO<sub>x</sub> solution at different molar ratios (from 0.5 at.% to 3 at.%). The pre-cleaned FTO coated glass was treated with ultraviolet ozone for 20 min before used. NiOx solutions were spin-coated onto FTO at 4500 rpm for 1 min to form 20 nm thick film (measured by the DEKTAK XT stylus profiler), followed by annealing at 235 °C for 45 min. The perovskite film was spin-coated on NiO<sub>x</sub> or Sr-doped NiO<sub>x</sub> layer by a consecutive two-step spin-coating process at 2000 rpm and 4000 rpm for 15 s and 60 s, respectively. During the second spin-coating step, chlorobenzene drop-casting was employed as antisolvent. The samples were then heated at 100 °C for 60 min to form black perovskite films. After cooling down, a 40 nm thick PCBM layer was formed onto the perovskite film by spin-coating PCBM dissolved in chlorobenzene (10 mg/ml) at 1200 rpm for 45 s. Finally, a 100 nm thick AgAl alloy



**Fig. 2.** (a) J–V curves of hole only devices with NiO<sub>x</sub> or Sr:NiO<sub>x</sub> layers, where the device structure is FTO/NiO<sub>x</sub> or Sr:NiO<sub>x</sub>/MoO<sub>3</sub>/AgAl. (b) J-V curves of pristine NiO<sub>x</sub> and Sr:NiO<sub>x</sub> films from vertical direction and the inset is the schematic diagram of testing structure. (c) XPS measurement of NiO<sub>x</sub> and Sr:NiO<sub>x</sub> films. (d) Steady-state photoluminescence (PL) spectra of FTO/perovskite, FTO/NiO<sub>x</sub>/perovskite and FTO/Sr:NiO<sub>x</sub>/perovskite films, respectively.

electrode was thermally evaporated onto the PCBM layer under the base pressure of  $5 \times 10^{-4}$  Pa. The weight ratio (wt %) of Al to Ag in the AgAl alloy, purchased from Trillion Metals Co., Ltd. (Beijing, China), is 3 wt%.

The surface morphologies of samples were investigated by fieldemission scanning electron microscopy (SEM, Hitachi S-4800) and Atomic Force Microscope (AFM, JPK Nano Wizard II), respectively. Xray diffraction (XRD) patterns of perovskite films were measured with PW3040/60 instrument (Holland Panalytical PRO) using a Cu Ka radiation source (V = 30 kV, I = 25 mA). Photoluminescence (PL) spectra were measured using a fluorescence spectrophotometer (HORIBA Jobin Yvon Fluoromax-4). The transmission and absorption spectra of samples were recorded using a UV-Vis spectrophotometer (Hitachi U-3900). The current density-voltage (J-V) characteristics were measured using a Keithley model 2440 source meter under a Newport solar simulator system with AM1.5G filter and 100 mW/cm<sup>2</sup> illumination calibrated by a standard silicon reference cell (Newport Oriel Instruments 91150 V). The incident photon to current conversion efficiency (IPCE) spectra were measured using a Newport Optical Power Meter 2936-R by illuminating the device with monochromatic light supplied from a xenon arc lamp in combination with a dual-grating monochromator. The reference numbers of photons incident on the sample were calibrated for each wavelength using a silicon photodiode (Newport Oriel Instruments 71675-71580). EIS measurements were recorded in the dark and 100 mW/cm<sup>2</sup> illumination in the frequency range from 0.1 to  $1 \times 10^{6}$  Hz using an Auto lab PGSTAT 302 N electrochemical workstation.

#### 3. Results and discussions

Fig. 1a shows the current density-voltage (J-V) curves of p-i-n configuration planar PSCs based on pristine NiOx or Sr-doped NiOx layers. The structure of p-i-n configuration planar PSCs is shown as inset in Fig. 1a, where the pristine NiO<sub>x</sub> or Sr-doped NiO<sub>x</sub> and (PCBM) are selected as HTL and electron transport layer (ETL), respectively. The perovskite films were spin coated onto pristine NiO<sub>x</sub> or Sr-doped NiO<sub>x</sub> layer by one-step solvent engineering. The statistic photovoltaic parameters including open-circuit voltage (Voc), short-current density (Jsc), fill factor (FF) and power conversion efficiency (PCE) of PSCs with different Sr doping concentration in NiOx HTL are summarized in Figs. S1a-d. PSCs prepared with Sr-doped NiOx HTL show higher open-circuit voltage (Voc) and short-current density (Jsc) than reference PSCs prepared with pristine NiOx HTL, leading to higher PCEs. Specifically, PSCs prepared with 1 at.% Sr-doped NiOx HTL show the highest opencircuit voltage ( $V_{oc}$ ) and short-current density ( $J_{sc}$ ), resulting in the highest average PCE of 18.87%. Thus, the 1 at.% Sr-doped NiOx was selected for deeply study and labeled as Sr:NiOx below.

To illustrate the higher  $V_{oc}$  for PSCs with Sr:NiO<sub>x</sub>, the energy level of Sr:NiO<sub>x</sub> film was calculated by Ultraviolet Photoelectron Spectroscopy (UPS) measurement. The secondary electron emission cut off, valence band edge and optical band gap measured for Sr-doped NiO<sub>x</sub> film are shown in Fig. 1b and c. Fig. 1d reveals the energy level arrangements of various functional layers in the PSCs. The valance band (VB) of Sr-doped NiO<sub>x</sub> film shifts to -5.4 eV from -5.2 eV of pristine NiO<sub>x</sub> film published elsewhere (Chen et al., 2015a), which can boost holes transport from perovskite layer to Sr:NiO<sub>x</sub> HTL due to the reduced contact barrier, leading to the higher  $V_{oc}$  (Wang, Y. et al., 2018b).



Fig. 3. SEM images of perovskite layers grown on NiO<sub>x</sub> (a) and Sr:NiO<sub>x</sub> (b) films; Absorption spectra (c) and XRD patterns (d) of perovskite films on NiO<sub>x</sub> and Sr:NiO<sub>x</sub> films.

The X-ray diffraction (XRD) patterns were measured to explore the crystallization of NiOx and Sr:NiOx films annealed at 235 °C, as shown in Fig. S2a. The characteristic diffraction peaks at 37.1°, 43.3° and 62.7° are corresponding to the (111), (200) and (220) planes of NiO<sub>x</sub> (JCPDS card no. 47-1409). However, the diffraction peaks of Sr:NiO<sub>x</sub> films slightly shift toward lower angle, indicating the slight increment of lattice constant (Chang et al., 2011). This means that strontium ions have successfully partly replaced Ni ions of crystal lattice, because the ionic radius of strontium (140 pm) is larger than that (78 pm) of nickel. Furthermore, the increased intensity of peak at 43.3° can be observed, indicating that Sr:NiO<sub>x</sub> films can easily grow along (200) plane and show better crystallinity. The enhanced film crystallinity can decrease trap centers and carrier scattering events, helping to improve carrier transport ability (Nam et al., 2012). The similar transmittance of pristine NiO<sub>x</sub> and Sr:NiO<sub>x</sub> films are over 90% in the wavelength range from 400 nm to 800 nm, as shown in Fig. S2b, indicating that Sr dopant won't obviously affect the transmittance of NiOx film. Therefore, the efficiency difference of PSCs based on pristine NiO<sub>x</sub> and Sr:NiO<sub>x</sub> layers is not related to their light transmittance. The doped metal oxide usually shows better conductivity compared to the pristine metal oxide (Chen et al., 2017; Chen et al., 2015b). Here, the hole only device with the structure FTO/NiOx or Sr:NiOx/MoO3/AgAl were fabricated and their J-V curves are shown in Fig. 2a. The results show that Sr:NiO<sub>x</sub> thin film has higher current density than the pristine  $\text{NiO}_{\boldsymbol{x}}$  film, indicating the better capability of hole extraction and transport for Sr:NiOx film. The hole mobility was calculated using the classical Mott-Gurney spacecharge-limited -current (SCLC) equation (Park et al., 2013):

 $\mathbf{J} = (9/8)\mu_{\rm h}\varepsilon_0\varepsilon_r(\mathbf{V}^2/\mathbf{L}^3) \tag{1}$ 

where J is the current density,  $\mu_h$  is the hole mobility,  $\epsilon_0$  is the dielectric

constant of the free space (=8.85  $\times 10^{-12}$  F/m),  $\epsilon_r$  is the relative dielectric constant, V is the applied voltage, and L is the thickness of charge transport layers. The hole mobility was increased from  $8.73 \times 10^{-3} \, cm^2 V^{-1} \, s^{-1}$  of the pristine NiO<sub>x</sub> film to  $1.44 \times 10^{-2} \, cm^2 V^{-1} \, s^{-1}$  of Sr:NiO<sub>x</sub> film.

To further investigate the conductivity of pristine NiOx and Srdoped NiO<sub>x</sub> films, the J-V curves from vertical direction are shown in Fig. 2b and S2c. The current density of NiO<sub>v</sub> doped with different Sr concentration compared to pristine NiO<sub>x</sub> films can greatly increase, indicating that Sr-doped NiO<sub>x</sub> films can obviously improve hole carriers transport ability and show better conductivity. The optimized Sr doping concentration is 1 at.%, which is obviously superior to the doping concentration of 0.5 at.%, 2 at.% and 3 at.%. The less doping concentration such as 0.5 at.% Sr compared to 1 at.% possibly results in the related lack of hole carriers and thus reduces its conductivity. While the excessive Sr doping concentration such as 2 at.% and 3 at.% may induce the lattice deformation and deteriorate its conductivity. Although the energy level difference between FTO and Sr:NiO<sub>x</sub> compared to pristine NiOx was slightly increased, as shown in Fig. 1d, the high concentration of hole carriers for Sr:NiO<sub>x</sub> can greatly reduce the tunneling width of carriers, which helps to reduce the contact barrier between FTO and Sr:NiO<sub>x</sub> film. The enhanced current density of Sr:NiO<sub>x</sub> film from vertical direction strongly supports that Sr:NiOx films have superior contact properties, better hole carriers transport and extraction abilities.

The incorporation of Sr element was confirmed by X-ray photoelectron spectroscopy (XPS) measurement, as shown in Fig. S2d. The decomposition of Ni  $2p_{3/2}$  spectrum can be well fitted by two different oxidation states (Ni<sup>2+</sup> and Ni<sup>3+</sup>) using Gaussian function, the typical peaks at 853.8 eV and 855.6 eV are corresponding to Ni<sup>2+</sup> and Ni<sup>3+</sup> (Kwon et al., 2016; Seo et al., 2016; Yin et al., 2016), as shown in



**Fig. 4.** (a) Current density-voltage (J-V) characteristics of PSCs based on pristine  $NiO_x$  and Sr: $NiO_x$  HTLs in different scan directions with scan rate of 100 mV/s. (b) Incident photon to current conversion efficiency (IPCE) curves of PSCs. (c) Dark J-V curves of PSCs based on pristine  $NiO_x$  and Sr: $NiO_x$  HTLs. (d) Stability of unencapsulated PSCs with aging time under the relative humidity (RH) of 18%.

#### Table 1

Photovoltaic parameters of champion PSCs based on NiOx and Sr:NiOx HTLs measured with different scan directions.

Device	Scan direction	J <sub>sc</sub> (mA/cm <sup>2</sup> )	V <sub>oc</sub> (V)	FF (%)	PCE (%)	Average PCE (%)
NiO <sub>x</sub>	Forward Reverse	20.99 21.19	1.054 1.040	68.8 71.4	15.22 15.73	15.47
Sr:NiO <sub>x</sub>	Forward Reverse	22.73 22.68	1.111 1.118	79.4 79.1	20.05 20.07	20.06

Fig. 2c. The Ni<sup>3+</sup>/Ni<sup>2+</sup> ratio of Sr:NiO<sub>x</sub> film is 2.10, which is obviously higher than the ratio (1.61) of pristine  $NiO_x$  film. The higher  $Ni^{3+}$ concentration means the existence of massive oxygen deficiencies and Ni vacancies in pristine NiO<sub>x</sub> and Sr:NiO<sub>x</sub> films. Compared to the Ni<sup>2+</sup>, the higher valence Ni<sup>3+</sup> is comparable to lack of one electron and would produce a hole carrier in NiO<sub>x</sub> films. Therefore, the obviously increased Ni<sup>3+</sup> concentration of Sr:NiO<sub>x</sub> film can greatly produce high concentration of hole carriers, which would contribute to better hole transport ability (Kelvin et al., 2016). Fig. 2d shows the steady-state photoluminescence (PL) spectra of perovskite films onto different HTL. PL intensity of perovskite film is related to carrier recombination and carrier transfer transport and extraction abilities at the interface of perovskite and carrier transport layers (Li et al., 2016; Sun et al., 2018). The significantly reduced PL intensity for perovskite/Sr:NiOx further indicates great improvement of Sr:NiOx hole transport and extraction capacity. Besides, the PL peak shows slightly blue-shifted for perovskite/Sr:NiO<sub>x</sub> film, meaning better crystallinity and the reduced trap states for perovskite layer (Shen et al., 2016; Zhou et al., 2018).

The surface morphologies of pristine  $\text{NiO}_{x}$  and  $\text{Sr:NiO}_{x}$  films were

measured using AFM method. The root-mean-square roughness (Rms) of Sr:NiOx film was decreased to 5.99 nm from 8.01 nm of pristine NiOx film, as shown in Figs. S3a and b. In the meantime, the contact angle of Sr:NiOx surface compared to pristine NiOx surface was increased to 48° from 32°, as shown in Figs. S3c and d. The smoother surface and increased contact angle of surface are beneficial to reduce the perovskite nuclei density, helping to form bigger perovskite grains and improve the crystallinity of perovskite layer (Mahmud et al., 2018; Wang et al., 2017). Fig. 3a and b show the surface SEM images of perovskite films grown on NiO<sub>x</sub> and Sr:NiO<sub>x</sub> layers. The perovskite grains onto Sr:NiO<sub>x</sub> film become obviously bigger, which can increase carrier transport ability and light absorption of perovskite layer (Seok Sang et al., 2018). The stronger absorbance for perovskite film onto Sr:NiOx around 450 nm, as shown in Fig. 3c, supports this result of its better crystallinity. This was further confirmed by XRD patterns of perovskite layers grown on NiO<sub>x</sub> and Sr:NiO<sub>x</sub> films, as shown in Fig. 3d. The characteristic peaks at 14.12°, 28.48° and 31.86° are corresponding to the (110), (220) and (310) planes of perovskite film, respectively. The obviously increased intensities of three peaks indicate better crystallinity for perovskite grown on Sr:NiOx film.

Fig. 4a shows the J-V curves of the champion PSCs prepared with pristine NiO<sub>x</sub> and Sr:NiO<sub>x</sub> HTLs under different scan direction with scan rate of 100 mV/S. The corresponding photovoltaic parameters are listed in Table 1. PSCs with pristine NiO<sub>x</sub> HTL got PCE = 15.22% with  $J_{sc} = 20.99 \text{ mA/cm}^2$ ,  $V_{oc} = 1.054 \text{ V}$ , FF = 68.8% under the forward scan, and 15.73% of PCE with 21.19 mA/cm<sup>2</sup> of  $J_{sc}$ , 1.040 V of  $V_{oc}$  and 71.4% of FF under the reverse scan. PSCs with Sr:NiO<sub>x</sub> HTL showed 20.05% of PCE with  $J_{sc} = 22.73 \text{ mA/cm}^2$ ,  $V_{oc} = 1.111 \text{ V}$ , and FF = 79.4% at a forward scan, and 20.07% of PCE with  $J_{sc} = 22.68 \text{ mA/cm}^2$ ,  $V_{oc} = 1.118 \text{ V}$  and FF = 79.1% at a reverse scan, showing negligible hysteresis. The hysteresis effects are related to many



Fig. 5.  $J_{sc}$  (a) and  $V_{oc}$  (b) as a function of light intensity for PSCs based on NiO<sub>x</sub> and Sr:NiO<sub>x</sub>. (c) Dark current-voltage curves from hole-only devices with the structure shown in the inset. (d) Mott-Schottky fitting to the C-V data for PSCs based on NiO<sub>x</sub> and Sr:NiO<sub>x</sub> HTLs.

factors, such as perovskite ion migration, carrier transport ability, interfacial carrier accumulation and etc. (Cao et al., 2018). Negligible hysteresis observed for PSCs prepared with Sr:NiO<sub>x</sub> HTL is primarily attributed to better hole transport and extraction abilities for Sr:NiOx layer, which can greatly reduce the carrier accumulation at perovskite/ HTL interface. The obtained PCE over 20% for PSCs with Sr:NiOx is impressive compared to those previous reported PSCs prepared with 2 at.% Li, 3 at.% Cu-codoped NiO<sub>x</sub> (14.53%) (Liu et al., 2016), Cu:NiO<sub>x</sub> (15.4%) (Kim et al., 2015), Li<sub>0.05</sub>Mg<sub>0.15</sub>Ni<sub>0.85</sub>O (18.3%) (Chen et al., 2015b), and Cs:NiO<sub>x</sub> (19.35%) (Chen et al., 2017). The incident photon to current conversion efficiency (IPCE) curve in the wavelength range from 400 nm to 750 nm for PSCs based on Sr:NiOx HTL is obviously higher than that of the pristine NiO<sub>x</sub> HTL, as shown in Fig. 4b, which is primarily attributed to the better hole transport and extraction capacities of Sr:NiOx layer. The integrated Jsc values from IPCE spectra of PSCs based on NiO<sub>x</sub> and Sr:NiO<sub>x</sub> are respectively 20.4 mA/cm<sup>2</sup> and 21.9 mA/cm<sup>2</sup>, which are slightly lower than the values obtain from J-V measurements. This is probably attributed to the defects of cells and part deviation of IPCE measurement (Jiang et al., 2016; Li et al., 2016). The dark J-V curves of PSCs based on NiO<sub>x</sub> and Sr:NiO<sub>x</sub> HTLs are shown in Fig. 4c. The reverse saturation current is obviously decreased and the rectifying property is improved for PSCs based on Sr:NiO<sub>x</sub> HTL, indicating that the carrier recombination and charge accumulation phenomena are decreased at the interface of Sr:NiO<sub>x</sub>/perovskite layers. The reduced leakage current and carrier recombination are helpful to improve the FF and J<sub>sc</sub> of PSCs (Chen et al., 2013; Jiang et al., 2016). Owing to better crystallinity of perovskite film, Sr:NiOx based PSCs also show better stability, as shown in Fig. 4d. The unencapsulated PSCs with Sr:NiO<sub>x</sub> still maintain over 60% of the original PCE value aging for 100 days with relative humidity (RH) of 18%.

In order to understand the photo-generated carrier recombination mechanism while the devices are in operation, the photovoltaic parameters of devices under different light intensity are measured. Fig. 5a shows the double logarithmic plot curves of J<sub>sc</sub> versus light intensity for PSCs with a fit to the power law (I  $\propto J_{sc}^{\alpha}$ ). The slopes of devices based on NiO<sub>x</sub> and Sr:NiO<sub>x</sub> HTLs are 0.99 and 1.01, respectively. Both slopes are close to 1, which suggests the bimolecular recombination, space charge effects are weak for PSCs based on both HTLs (Singh and Miyasaka, 2017; Yao et al., 2018; Zhou et al., 2018). Fig. 5b shows the semi logarithmic plot curves of  $V_{\rm oc}$  versus light intensity for both kinds of PSCs and the linear fit follows Voc  $\propto \frac{nkT}{n} \log_{10} I$ , where n is the ideal factor, k is the Boltzmann's constant, T is absolute temperature, q is elementary charge and I is light intensity. The carriers recombination condition between PSCs based on NiOx and Sr:NiOx can be effectively distinguished. Relatively lower ideal factor of 1.3 for PSCs with Sr:NiO<sub>x</sub> compared to that of 1.9 for PSCs with pure NiOx layers indicates the reduced trap-assisted recombination and interface charge recombination, which are related to better perovskite crystallinity and interface contact properties between perovskite and Sr:NiOx interface (Gil-Escrig et al., 2018; Guo et al., 2018; Tress et al., 2018).

Fig. 5c shows the space charge limited current (SCLC) curves of hole only devices. Here, the hole transport materials MoO<sub>3</sub> was used instead of ETL PCBM to fabricate hole-only devices. The trap density of perovskite can be calculated by the trap-filled limit voltage (V<sub>TFL</sub>) using  $V_{TFL} = \frac{entrapL^2}{2\epsilon_0 \epsilon}$ , where e is the elementary charge, L is the thickness of the perovskite film,  $\epsilon_0$  is the vacuum permittivity (8.854 × 10<sup>-12</sup> F/m), and  $\epsilon$  is the relative dielectric constant of perovskite. The V<sub>TFL</sub> values of NiO<sub>x</sub> and Sr:NiO<sub>x</sub> based devices are 0.71 V and 0.61 V, respectively. The trap density n<sub>trap</sub> of NiO<sub>x</sub> and Sr:NiO<sub>x</sub> HTLs based devices are calculated to be respectively 1.98 × 10<sup>16</sup> cm<sup>-3</sup> and 1.70 × 10<sup>16</sup> cm<sup>-3</sup>, indicating



Fig. 6. Nyquist plots (a) and Bode-phase plots (b) of PSCs based on NiO<sub>x</sub> and Sr:NiO<sub>x</sub> HTLs at the bias voltage of 0.5 V under dark condition. (c) Nyquist plots of PSCs based on NiO<sub>x</sub> and Sr:NiO<sub>x</sub> HTLs at the bias voltage of 0.5 V under 100 mW/cm<sup>2</sup> illumination.

that the trap density decreased by replacing NiO<sub>x</sub> with Sr:NiO<sub>x</sub> film. To deeply understand the origin of the improved V<sub>oc</sub>, the classical Mott-Schottky capacitance-voltage (C-V) measurement in the dark condition was applied, as shown in Fig. 5d. Herein, the relationship of C and V can be described as: (Cai et al., 2018)

$$\frac{1}{C^2} = \frac{-2}{\varepsilon_0 \epsilon q A^2 N} (V - V_{bi})$$
(2)

where C is the measured capacitance under dark, A is the active area, V is the applied bias,  $\varepsilon_0$  is the vacuum permittivity,  $\varepsilon$  is the relative permittivity, q is the elementary charge and N is the free carrier concentration. Thus, the built-in potentials (V<sub>bi</sub>) can be calculated using the intercepts of the linear region with the x-axis of the Mott-Schottky plots, which are 0.68 V and 0.74 V for PSCs prepared with NiO<sub>x</sub> and Sr:NiO<sub>x</sub>. The results show the similar trend with the V<sub>oc</sub> attained from J-V measurements. The built-in field helps the separation of photo-generated carriers and suppresses the electron-hole recombination, the higher V<sub>bi</sub> of Sr:NiO<sub>x</sub> HTL based PSC reveals less charge recombination due to better crystallinity of perovskite film (Cai et al., 2018).

To further investigate the interfacial charge transport and recombination behaviors of devices, electrochemical impedance spectra of PSCs based on NiO<sub>x</sub> and Sr:NiO<sub>x</sub> HTLs were measured. Fig. 6a presents the Nyquist plots measured in a frequency of  $0.1-10^6$  Hz at the bias voltage of 0.5 V under dark condition. A clear semicircle can be distinguished in the intermediate-frequency region for both PSCs, which is related to the charge transfer at FTO/HTL/Perovskite/ETL interfaces, mainly owing to the electron recombination. It is obvious that the recombination resistance (R<sub>ct</sub>) of PSCs with Sr:NiO<sub>x</sub> is much bigger than that of PSCs with NiO<sub>x</sub>. Fig. 6b shows the Bode-phase plots of PSCs with NiO<sub>x</sub> and Sr:NiO<sub>x</sub> at the bias voltage of 0.5 V. The frequency response peaks of different PSCs can be divided into two curves by curve fitting. The characteristic frequencies of low-frequency and high-frequency sides are related to carrier recombination and carrier diffusion, respectively (Choi et al., 2012; Perrier et al., 2012; Zhang et al., 2018). The frequency peak of low-frequency side is shifted to a lower region for PSC with Sr:NiOx, indicating a reduced carrier recombination due to better interfacial contact and crystallization of perovskite layer (Perrier et al., 2012; Zhang et al., 2018). Fig. 6c shows Nyquist plots measured in a frequency of 0.1–10<sup>6</sup> Hz under illumination (AM1.5, 100 mW/cm<sup>2</sup>) at the bias voltage of 0.5 V. The semicircle in high-frequency range is mainly ascribed to the charge transfer resistance (R<sub>CT</sub>) at the hole transport layer (Gonzalez-Pedro et al., 2014). The lower R<sub>CT</sub> value of PSCs with Sr:NiO<sub>x</sub> compared to pristine NiO<sub>x</sub> indicates that Sr:NiOx based PSC can more quickly transport carriers and show better hole transport ability (Sun et al., 2017).

#### 4. Conclusion

In summary, we have demonstrated a simple sol-gel method to prepare high quality Sr-doped NiO<sub>x</sub> films annealing at 235 °C. The optimized 1 at.% Sr-doped NiO<sub>x</sub> (Sr:NiO<sub>x</sub>) film compared to pristine NiO<sub>x</sub> shows better conductivity and superior contact properties between FTO and perovskite layers, which help to improve hole carrier transport and extraction. In the meantime, Sr:NiOx films can boost perovskite crystallization, which help to reduce trap density and inhibit carrier recombination. Therefore, PSCs with Sr:NiO<sub>x</sub> layer show highest PCE of 20.07% with negligible hysteresis, which is greatly higher than that (15.73%) of the reference PSCs with pristine NiO<sub>x</sub>. Furthermore, PSCs with Sr:NiO<sub>x</sub> still remained 60% of the original PCE values aging for 100 days, obviously superior to that (35%) of pristine NiO<sub>x</sub> based PSCs. Therefore, Sr doped NiO<sub>x</sub> as a hole transport layer is a good strategy to fabricate high efficient and stability of PSCs using low annealing temperature procedure.

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#### Appendix A. Supplementary material

Supplementary data to this article can be found online at https://doi.org/10.1016/j.solener.2018.10.004.

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