Review on NiO thin film as hole transport layer in perovskite solar cell

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The saturation in increasing the power conversion efficiency (PCE) of silicon-based solar cells made researchers around world to look for the alternatives. An alternative solar cell would possess some basic requirements like cost effectiveness, reproducible, durable (stability), non-toxicity and higher efficiency. Perovskite solar cell (PSC) opened the new realm of hope for this alternative, consisting of perovskite absorber sandwiched between the hole transport layer (HTL) and the electron transport layer (ETL). Good performance of PSCs can be achieved by optimizing many parameters of the components of PSC for obtaining the highest PCE. Among them, the HTL also plays a very vital role. Previously, organic poly (3,4-ethylenedioxythiophene):poly (styrene sulfonic acid) PEDOT:PSS was being widely used as the HTL in PSCs, but due to its hygroscopic nature and acidic properties, it lowered the stability and the life time of the PSCs. Later it was replaced mostly by NiO, a p-type transparent conducting oxide (TCO) enhancing the PCE of PSCs. Its excellent stability and electrical/optical properties attracted the interest of many researchers. Different types of PSCs used NiO thin films prepared from different synthesis routes and obtained variation in efficiency of PSCs. Different parameters of NiO thin films like thickness, annealing temperature (AT) and duration, precursor combinations and more in synthesis processes, have a significant role in optimizing the PCE. Though there are many routes for obtaining NiO thin film, here we are trying to focus more on sol-gel method, as this route is very cost effective and employs basic equipment. Its evolution, present status and the future perspectives will also be discussed.

Keywords: power conversion efficiency, organic photovoltaic device, annealing temperature, sol-gel method.

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1. Introduction

In human civilization, energy has played an important role in societal evolution. The trend of social development and many more dimensions of human civilization depend upon the availability of the energy resources and its rational utilization. The limited sources of energy materials and the ever-increasing demand for energy create a situation of scarcity over the resources. Sources of energy materials directly affect the global economy. So, the whole world is concerned about the permanent solution of the energy crisis.

Ultimately researchers are interested in solar energy. Solar energy can be converted into electrical energy and can be used as alternative to conventional carbon-based sources of energy. Gradual development took place from the initial discovery of the solar photovoltaic effect in 1839 by French Scientist Edmond Becquerel [1]. Since the world’s first Photovoltaic solar cell by Chapin et al. [3], solar cells have been based on silicon, selenium, cadmium, copper, dye-sensitized photochemical cell, thin film solar cell based on different materials with solar storage [2], to perovskite solar cells at present. Perovskite based solar cell have recently reached an efficiency > 25 % [4].

Many elements and compounds have photovoltaic properties which allow conversion of solar energy into electrical energy. Historically, the silicon-based solar cells have gained the popularity, but due to their limited efficiency, scientists are finding alternatives. The perovskite solar cell is currently popular. Lead-based perovskite solar cells have almost attained the efficiency of the silicon-based solar cells. As Pb is toxic to the environment, its commercialization became the hurdle in the development of high efficiency solar cell. So, now an alternative to the Pb-PSCs is currently a topic of research interest. Pb-free PSCs seem to be promising alternatives for replacing the conventional Si-based solar cells [5].

2. Perovskite and NiO in the solar cell

Perovskite is a crystal named after the Russian mineralogist Lev Perovski. Its crystal structure generalized as ABX3 form, in which A is a 2+ metal cation, B is a 4+ metal cation and X is a 2− oxygen anion or halogens. Common examples are CaTiO3, BaTiO3, and CsSnI3, etc. Gradually, many more perovskite materials were discovered and halide perovskite, organic perovskite, mixed organic-inorganic perovskite, double and triple perovskite materials were also introduced. In 2009, the first perovskite based Dye Sensitized Solar Cell (DSSC) was reported by Kojima.
et al. [6] and first solid state solar cell based on perovskite was reported by Kim et al. [7]. Currently, the organo-lead-halide perovskite has achieved the highest efficiency in comparison to the Si-Solar Cells. Pb is mostly replaced by Sn and achieved the comparable efficiency. Since, the oxide of Sn is unstable in ambient environment; it has become a serious issue to be resolved. Inorganic and double perovskites are also an other possibility for research.

The general architecture of the thin film based PSCs has a multi-layer structure of its components like electrodes, ETL, perovskite absorbers, HTL and back contact. Perovskite absorbers are mainly sandwiched between the HTL and ETL (Fig. 1) [42]. The variation in the ETL and HTL has shown improvement in increasing the efficiency of the PSCs. ETLs are the n-type semiconductors viz phenyl-C61-butyric acid methyl ester (PCBM), ZnO, SnO2, TiO2, etc. and HTLs are the p-type semiconductors namely, 2,2′,7,7′-tetrakis(N,N-pdimethoxyphenylamino)-9,9′-spirobifluorene (Spiro-OMeTAD), poly(3,4-ethylenedioxythiophene)/poly(styrenesulfonicacid) (PEDOT:PSS), NiOx, CuSCN, etc. Previously the organic HTL PEDOT:PSS was very common in PSCs, but due to its hygroscopic and poor electron blocking nature, significant degradation in PCE occurred. If the electrons are not blocked properly, then the recombination at the interface layer will be increased, leading to low cell efficiency [8]. PEDOT:PSS also has an acidic nature. Transport materials need to be transparent, able to block the electron, should possess Ohmic contact with the electrodes and good stability. NiO thin film was the next candidate with all these properties. It was found that the inorganic materials provide good stability and carrier mobility [10].

![Fig. 1](https://example.com/fig1.png)

Electron-hole pairs (E–H) are generated when light is incident upon the absorber. To extract the E–H and to transport to the corresponding electrodes, ETL and HTL are required respectively. In general, PSCs, ETL is deposited on the TCO (mainly FTO/ITO) followed by the perovskite absorber. HTL is deposited above the absorber to extract the holes efficiently. On the top of the hole, metal layer of silver (Ag) or gold (Au) is deposited (TCO/ETL/Perovskite/HTL/Ag) and in inverted configuration, just the position of HTL and ETL are interchanged (TCO/HTL/Perovskite/ETL/Ag). Previously, the ETL and HTL were incorporated with some meso-porous layers also.

This review mainly concentrates on development in synthesis process of NiO thin film for the application as HTL in PSCs. With the introduction of NiO as HTL in PSCs, many possibilities have been seen in improving the efficiency of the PSCs. NiO thin films have been synthesized varying many synthesis routes and parameters like thickness, different precursors or solvents [9], annealing time and temperature and influenced the properties of the solar cell. There are many routes for depositing the NiO thin film, including: sol-gel route (Fig. 2) [10, 11], direct spin coating from pre-synthesized nanocrystals [12], combustion method [13], electrodeposition [14], sputtering [15], pulsed laser deposition (PLD) [16], atomic layer deposition (ALD) [17], spray pyrolysis [18], chemical precipitation methods [19] and many more have been used for the thin film deposition.

Nickel oxide (NiO) has a basic cubic structure with the space group Fm3M which is similar to that of NaCl. Its lattice parameter is 0.4173 nm [20], NiO is a wide bandgap material having the band gap in the range of 3.6 – 4.0 eV with the work function of 5.4 eV. It is a highly transparent nano thin film enabling it to be a good p-type TCO [21,22].

3. Literature survey

Jimenez et al. synthesized NiO thin film via a dip-coating sol-gel route using Ni-acetate as precursor. They studied the effect of AT on crystal structure and found the films to be amorphous when annealed between 175 – 250 °C and above 250 °C it was crystalline with a good surface homogeneity. They found the dependency of electrical conductivity with the thickness in the range of $2 \times 10^{-5}$ to $6.5 \times 10^{-3}$ (Ω·cm)$^{-1}$ and similarly the energy band gap $E_g$ was from
3.9 to 3.8 eV for the corresponding film thickness varying from 49 to 191.8 nm. The transmittance of films decreased with increased thickness and the films with thickness > 100 nm had very low optical transmittance. Slower growth rate resulted in good morphology and uniform homogeneity with increasing electrical conductivity [23]. The effect of precursor on the grain size of the thin film was observed as 2 – 3 nm in case of Ni-sulfate precursor than Ni-acetate (average 5 nm) via SG route [24]. The electrical resistivity decreased with an increase in temperature (semiconductor behavior) and thickness was calibrated graphically for chemical bath-deposited films [25]. AT changes the surface roughness and the crystal structure. At an AT up to 300 °C good surface morphology was not observed, and it was found to be amorphous in nature. However, above 300 °C, the surface roughness also started improving with the proper crystal structure. The correlation between the deposition parameters and optical properties of the NiO thin film was also established. The AT and dipping speed also affected the refractive index and the extinction coefficient of the films [26]. Ghamadi et al. prepared thin film by the SGSC method using Ni-Ac as precursor with an optimum AT of 600 °C. Their study showed that the volume energy loss is greater than the surface energy loss at all incident photon energies. The optical band gap was 3.44 eV. They used single electronic oscillator mode to obtain the values for the oscillator strength, oscillator energy. They also calculated dielectric and refractive index of the film with values of 4.04 and 2.01 respectively. The value of third order susceptibility was estimated using Frumer model as \( 1.62 \times 10^{-13} \) esu [27]. Patil et al. synthesized NiO nanopowder via chemical precipitation route using nickel acetate as a precursor. Further, the NiO powder was mixed with m-cresol and prepared as a thin film using a spin-coater. They observed the decrease in band gap energy from 3.86 to 3.47 eV and increase in electrical conductivity from \( 10^{-4} \) to \( 10^{-2} \) (\( \Omega \cdot \text{cm} \)) after annealing the NiO films from 400 to 700 °C [28]. With increasing AT from 400 – 700 °C there was an increase in mean grain size. Increasing the grain size (40 – 60 nm) caused a decrease in the grain boundary density of a film and decrease in scattering of carriers at grain boundaries. With an increase in AT, the absorption coefficient of the film also increased. This could be assumed due to increase in the density of states of holes with increase in AT [29]. Annealing (600 °C) in different environments (air and \( \text{N}_2 \)) showed that grain size increased with the increased in number of layers for annealing at aerobic environment but in \( \text{N}_2 \) environment there was a decrease in grain size. Even the optical band gap energy decreased from 3.76 to 3.52 eV and 3.75 to 3.49 eV annealed in air and \( \text{N}_2 \) respectively. The resistivity of air annealed film was more than that of \( \text{N}_2 \) as \( 900 \times 10^2 \) and \( 40 \ \Omega \cdot \text{cm} \) respectively. Aerobic annealing leads to production of larger grains and better crystalline structure with lower oxygen vacancies, which can be the predominant factor of conductivity with higher resistivity [10]. Thermal analysis showed that below an AT of 400 °C there was continuous decomposition and weight loss and after reaching 400 °C, the weight loss became constant, leading to the formation of NiO crystal [30]. Annealing increased the surface roughness and it might be due to the fact that the annealing process would enlarge the NiO particle sizes [31]. Sahoo et al. showed the influence of AT on morphological, optical and electrical properties of SGSC synthesized NiO thin film. AT affected the current at light and dark mode from 9.9 to 3.3 mA and 7.9 to 3.6 mA respectively [11]. Kayani et al. synthesized NiO powder via SG route varying the AT from 400 to 1000 °C. The crystalline sizes increased with increased AT from 12 to 32 nm from 400 – 1000 °C respectively. The dislocation density decreased from \( 6.5 \times 10^{-3} \) to \( 2.3 \times 10^{-5} \) nm\(^2\) with an increase in AT. The transmittance decreased with increasing AT, but the band gap increased from 3.02 to 3.14 eV. This increase in the band gap may be due to an increase in the defect levels. When magnetic field was applied on NiO nanoparticles the magnetization showed a more or less linear dependence. Therefore, NiO nanoparticles showed paramagnetic behavior. NiO also showed ferromagnetic behavior above the critical diameter of 55 nm, below which, the nanoparticles become single magnetic domains [32]. The transmittance decreased with increasing the thickness but couldn’t find the proper correlation of reflectance with the thickness. Band gap energy increased from 3.87 to 3.94 eV as thickness increased from 124 to 166 nm. Electrical conductivity which was increased from \( 7.94 \times 10^{-3} \) to \( 27.89 \times 10^{-3} \) (\( \Omega \cdot \text{cm} \))\(^{-1}\) when film thickness increased from 124 to 137 nm but again decreased at 145 nm and at 166 nm showed a good electrical conductivity with \( 84 \times 10^{-3} \) (\( \Omega \cdot \text{cm} \))\(^{-1}\) [33]. Average crystalline size increased with
increasing AT whereas dislocation density decreased. Annealing reduced the lattice defects of the film and increased the crystalline quality by modifying the periodic arrangement of atoms in the crystal lattice [34].

4. NiO as hole transport layer

HTL serves as an electron blocking layer in bulk heterojunction (BHJ) solar cell. Previously used HTL PEDOT:PSS degraded so early and had a limited performance. Aqueous solution of PEDOT:PSS was highly acidic in nature with pH 1.2. Due to its hygroscopic nature, moisture may get absorbed leading to proton release and anode corrosion [8]. As a result, it leads to the degradation of device, lowering lifetime and overall performance of the device. For an ideal candidate to be used as HTL, it should have $E_g > 3$ eV, p-type transparent conducting material, it should be chemically stable and possess good conductivity.

Steirer et al. introduced NiO thin film replacing the PEDOT:PSS in OPV. The NiO thin film was deposited by a solution based Ni-organic inkjet technique. Although the work function of NiO was tailored by O$_2$-plasma treatment but the real effect was not understood properly. Thicker films produced uniform devices with lower open-circuit voltage ($V_{oc}$), lower short-circuit current density ($J_{sc}$) and higher shunt resistance ($R_s$). Thinner films failed to produce unique device on a substrate causing inconsistent performance, perhaps due to incomplete surface coverage. But the optimum thickness varied from device to device. The device ITO/NiO/P3HT:PCBM/Ca/Al, attained the PCE of 3.6 %.

In comparison, the performance of NiO was as good as that of PEDOT:PSS [16]. Films synthesized via PLD technique was applied in BHJ solar cell improved the performance with work function of $\sim 5.3$ eV, optical transmittance of $> 80$ %. The problem in forming good Ohmic contact caused lowering of efficiency to some extent [35]. Hsu et al. demonstrated, for the first time, the fabrication of uniform, defect-free, and conformal NiO ultra-thin films for use as HTL in BHJ polymer solar cell via ALD optimizing its processing parameters. ALD offers unparalleled advantages owing to its unique capabilities, including defect-free deposition, complete and uniform coverage over large-area/high-aspect-ratio surface features, precise control over deposited thickness, and low deposition temperatures. Thickness was optimized and 4 nm seemed to be the optimum one giving comparable PCE of 3.38 % comparable to PEDOT:PSS [17]. You et al. improved the air stability of the PSC using solution processed NiO as HTL. The maximum PCE obtained was 14.6±1.5% with device architecture glass/ITO/NiO/P3HT:PCBM/Ca/Al, attained the PCE of 3.6 %. In comparison, the performance of NiO was as good as that of PEDOT:PSS [16]. Films synthesized via PLD technique was applied in BHJ solar cell improved the performance with work function of $\sim 5.3$ eV, optical transmittance of $> 80$ %. The problem in forming good Ohmic contact caused lowering of efficiency to some extent [35].

Kim used polyethylene glycol (PEG)-assisted sol-gel synthesis of the compact NiO as HTL. There was a significant improvement in the uniformity and smoothness of the film morphology of the compact NiO, which modified the interfacial properties between the layers. It helped in the good extraction of charge and suppression of charge recombination. He also incorporated PEDOT:PSS onto NiO layer as a hybrid HTL, which facilitated efficient cascade charge migration and passivation of the tiny pinholes. The introduction of compact NiO HTL improved the PCE from 5.68 to 6.91 % and for the hybrid PEDOT:PSS and NiO HTL, PCE improved from 7.26 to 7.93 %. Thus, optimizing the interfacial properties also improved the overall PCE of the solar cells [39]. Akhtaruzzaman et al. used the EBVD technique to synthesize NiO thin film and incorporated in PSC as HTL with structure glass/FTO/NiO$_x$/CH$_3$NH$_3$PbI$_3$/PC$_{61}$BM/BCP/Ag. There was a difference in PCE for the HTLs which was normal NiO and annealed NiO thin film as 13.20 and 13.24 % respectively. Their optimized AT was 500 °C with a thickness of 18 nm. There was a difference in degradation in efficiency with time for normal and annealed device. In ambient environment normal device retained 72.2 % and annealed device retained 76.96 % of the initial efficiency after 28 days of fabrication of the PSCs [40]. The comparative performance of the PSCs with NiO as HTL is given in Table 1.
Table 1. Overall Performance of the PSCs with NiO as HTL based on SGSC method

| Device Configuration | $V_{oc}$ [V] | $J_{sc}$ [mA/cm$^2$] | FF | PCE | Ref. |
|----------------------|--------------|----------------------|----|-----|------|
| FTO/NiO$_x$/CH$_3$NH$_3$PbI$_3$/C$_60$/SnO$_2$ NCs/Ag | 1.12 | 23.7 | 0.76 | 18.8 % | [41] |
| ITO/NiO/C$_{0.05}$(MA$_{0.17}$FA$_{0.83}$)$_{0.95}$Pb(I$_{0.9}$Br$_{0.1}$)$_3$/PCBM/ZnO/Al | 1.023 | 22/2 | 0/82 | 18.6 % | [42] |
| ITO/NiO/CH$_3$NH$_3$PbI$_3$/PCBM/Ti(Nb)O$_y$/Ag | 1.07 | 21.88 | 0.79 | 18.49 % | [43] |
| FTO/NiO/CH$_3$NH$_3$PbI$_3$/PCBM/BCP/Ag | 0.99 | 22.92 | 0.803 | 18.15 % | [44] |
| ITO/NiO/CH$_3$NH$_3$PbI$_3$/C$_60$/Bis-C$_60$/Ag | 1.03 | 21.80 | 0.784 | 17.70 % | [45] |
| FTO/NiO/Perovskite/PCBM/Bis-C$_60$/Ag | 1.10 | 21.67 | 0.75 | 17.64 % | [46] |
| ITO/NiO/Perovskite/PCBM/PDINO/Ag | 1.11 | 20.57 | 0.765 | 17.5 % | [47] |
| ITO/NiO/CH$_3$NH$_3$PbI$_3$/PCBM/Ag | 1.04 | 23.51 | 0.69 | 16.91 % | [48] |
| NiO$_x$/MAPbI$_{3-x}$Cl$_x$/PCBM/C$_{60}$/C/Ag | 1.08 | 24.25 | 0.66 | 16.76 % | [12] |
| ITO/NiO/CH$_3$NH$_3$PbI$_3$/PCBM/Ag | 1.103 | 21.28 | 0.714 | 16.74 % | [49] |
| TO/NiO/CH$_3$NH$_3$PbI$_3$/PCBM/Ag | 1.09 | 19.9 | 0.769 | 16.68 % | [50] |
| TO/NiO/CH$_3$NH$_3$PbI$_3$/PCBM/Ag | 1.07 | 20.58 | 0.748 | 16.47 % | [51] |
| ITO/NiO$_x$/CH$_3$NH$_3$PbI$_3$/PCBM/Al | 1.01 | 21.0 | 0.76 | 14.5 ± 1.6 % | [36] |
| FTO/NiO$_x$/CH$_3$NH$_3$PbI$_3$/PCBM/BCP/Ag | 0.99 | 17.16 | 0.78 | 13.24 % | [40] |
| ITO/NiO/CH$_3$NH$_3$PbI$_{3-x}$Cl$_x$/PCBM/BCP/Al | 0.92 | 12.43 | 0.68 | 7.80 % | [52] |
| ITO/NiO/P3HT:PCBM/Ca/Al | 0.58 | 8.6 | 0.66 | 3.60 % | [16] |
| ITO/NiO/P3HT/PCBM/Ca/Al | 0.58 | 10 | 0.58 | 3.38 % | [17] |
| ITO/NiO(IJ)/P3HT:PC$_{60}$BM/LiF/Al | 0.60 | 8.57 | 0.50 | 2.60 | [37] |

5. Doping

Improvement in the efficiency of the cell is the main concern, so doping of some metals into NiO was also applied to enhance the HTL property. Jung et al. incorporated Cu doped NiO in PSCs consisting of methylammonium lead halide perovskite. Synthesis via low temperature combustion method ($150^\circ$C) and conventional solution based high temperature method ($500^\circ$C) achieved the highest PCE of 17.8 and 15.52 % respectively. ITO-free Cu-NiO based PSC showed PCE of 13.42 % [13]. Li doping in NiO thin film affected the grain size to decrease and variation in transmittance. Li doping also decreased the band gap but there was no such difference in crystallinity due to AT [53]. Addition of Cu-dopant increased the grain size influencing the surface morphology and decrease in transmittance. The optical band gap value of the Cu-doped NiO film (3.69 eV) was lower than that of the undoped NiO film (3.73 eV) and the resistivity of the Cu-doped NiO film was 23 Ω·m, which was significantly lower than that of the undoped NiO film (320 Ω·m) [54]. The doping effect has also been observed in conductivity at lower AT of 700 °C as increasing in conductivity [55]. Cs:NiO$_x$ HTL achieved 16 – 19 % PCE [56] and molecule F6TCNNQ doping achieved PCE of 20.86 % [57]. So, doping also enhanced the PCE of the PSCs. The comparative performance of the PSCs of doped NiO as HTL is given in Table 2.

6. Conclusion

The exploration of different properties of NiO nanoparticles and thin films led to the potential use in various applications. Incorporation of inorganic HTL showed a sound improvement in the overall performance of the PSCs. Different synthetic routes had its own advantages and disadvantages. The best route will be the one which is very cost effective and provides higher efficiency while leaving the other properties intact. Although the sol-gel method has its own limitations, it is very accessible for everyone. So, more improvement can be met using this method. The
doping has also showed good improvement. In most of the findings, variation in annealing temperature was done for optimization. The SGSC method is very facile for low cost synthesis and can sustain good method for higher PCE of the PSCs in future also.

**Conflicts of interest**

The authors declare there is no conflict of interest.

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