Spinel zinc ferrite nanostructured thin-films for enhanced light-harvesting in polycrystalline solar cells

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Spinel zinc ferrite (ZnFe_2O_4) nanocrystallites are applied as an anti-reflection coating (ARC) for the enhanced light harvesting in polycrystalline silicon solar cells (PCSSC) and its effect were studied. Spinel zinc ferrite nanocrystallites were prepared using precursors of zinc and ferric chloride by co-precipitation method. The morphological, optical, electrical characterizations are comprehensively used to establish the performance of spinel ZnFe_2O_4 Nanostructured Thin Films (NTF) covered and uncovered PCSSC. Further, X-ray diffraction and fluorescence analysis have been performed to demonstrate the crystallographic patterns and elemental compositions of ZnFe_2O_4 nanocrystallites. The developed spinel ZnFe_2O_4 NTF on PCSSC shows the reduction in reflectivity (20.3%), improvement in light trapping efficiency (17.5%) and transmittance of the fabricated spinel ZnFe_2O_4 NTF was validated with optical and electrical observations.

Keywords: *Spinel zinc ferrite nanocrystallites, nanostructured thin films, light-harvesting and anti-reflection coating, polycrystalline silicon solar cells*

1. Introduction

Global energy consumption is increasing rapidly day by day and the energy produced from fossil fuels may not be sufficient for future requirements [1]. Solar energy is a significant source of renewable energy and an extremely successful renewable resource due to its economic efficiency, enhanced energy performance and ability to be used in a variety of places. Polycrystalline silicon solar cells show less power conversion efficiency (PCE) due to reflection loss and light to heat conversion. One of the main aspects involved in order to achieve high-performance silicon solar cells is to reduce optical losses and increase light transmittance and trapping characteristics. Several techniques were used to increase the PCE and one such effective technique is the use of anti-reflection coating (ARC) [2, 3].

Recent studies reveal that magnetic nanoparticles can significantly improve the PCE of solar cells [4]. Magnetic oxide nanoparticles $[\text{M}(\text{II})$

$\text{Fe}(\text{III})_2\text{O}_4$ where M represents Co, Mn, Ni, Zn or Fe] have attracted considerable interest due to its wide range of applications [5]. Spinel ferrites possessing the cubic structure are described by the formula AB_2X_4 , where A and B indicate tetrahedral and octahedral cation sites, respectively, in an face centred cubic (FCC) anion (oxygen) sublattice. The zinc ferrite (ZnFe_2O_4) has a regular spinel structure with Zn^{2+} ions in the A-site and Fe^{3+} ions in the B-sites [6]. Among the spinel ferrites, ZnFe_2O_4 attracts great attention as it is a promising n-type semiconductor photocatalyst with 1.9 eV bandgap [7]. Spinel ferrites are widely used in different applications such as magnetic storage media [7], microwave devices [8], gas sensors [9], wastewater treatment [10], biomedical applications [11, 12], nanoelectronics [13], ferrofluids [14], catalysts [15], batteries [16], but only a few works have been carried out for PCSSC solar cells [17].

Properties of the ferrites are affected by particle size, cation distribution, morphology, synthesis and fabrication methods. ZnFe_2O_4 can be synthesized using different methods such as sol-gel

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technique [18–20] co-precipitation [21], hydrothermal [22], solvothermal [23], ball milling [24], and ceramic route [25]. Among them, co-precipitation is a promising technique for ferrite processing due to low production cost, controllable particle size, reactivity, enhanced uniformity and purity [26]. Spin-coating is a commonly used technique for optical coatings to manufacture extremely reproducible film thickness [27]. Single-layer coating on solar cells would not be sufficient to reduce reflection. Therefore, multilayer coating of ARC is often preferred for further reduction of reflection loss [28–30].

This present research work focuses on using the spinel ZnFe_2O_4 nanocrystallites as a light harvesting ARC material for enhancing the PCE of polycrystalline silicon solar cells (PCSSC). The ZnFe_2O_4 spinel nanocrystallites were synthesized using co-precipitation method, which are deposited on the PCSSC by using spin coating technique. The structural, optical, electrical behaviour of spinel ZnFe_2O_4 NTF covered and uncovered PCSSC were investigated under controlled atmospheric condition.

2. Materials and methods

2.1. Materials

Zinc chloride (ZnCl_2), ferric chloride ($\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$) and sodium hydroxide (NaOH) with 98% purity was purchased from Merck Life Science Private Limited (India). Ethanol ($\text{C}_2\text{H}_5\text{OH}$), 99.9% purity was purchased from Loba Chemical Private Limited (India). PCSSCs with dimensions of 52×38 mm were purchased from Eco-Worthy (China).

2.2. Preparation of ZnFe_2O_4 spinel nanoparticles

The ZnFe_2O_4 spinel nanoparticles were prepared using ZnCl_2 and $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$. The dual precursors of 1 g were dissolved in 100 ml of distilled water and added with 75 ml of 3M NaOH solution to act as a co-precipitating agent. After drop by drop addition of NaOH solution, the obtained solution was magnetically stirred at 750 rpm

for two hours, then it was further heated at 75°C for one hour. The resultant product was allowed to settle down at room temperature, and its pH was constantly maintained at 7.5 by subsequent washing with deionised water, causing the removal of chloride (Cl^-) ions. The obtained wet precipitate was placed in a hot air oven at 110°C for 12 hours, followed by calcination at 510°C for five hours in a muffle furnace. Finally, the obtained calcined ZnFe_2O_4 was finely grained for 3 hours using agate mortar and pestle to obtain fine ZnFe_2O_4 nanoparticles.

2.3. Deposition of ZnFe_2O_4 spinel ferrites on polycrystalline solar cells

The solution was prepared by dissolving 0.1 g of ZnFe_2O_4 spinel nanoparticles in 10 ml of ethanol. This solution was stirred by a magnetic stirrer at room temperature for 2 hours to obtain a homogeneous solution. Afterwards, this solution was sonicated for 10 minutes, and again the solution was stirred for 15 minutes at room temperature. Prior to deposition, the PCSSCs were cleaned with ethanol. The spinel ZnFe_2O_4 nanostructures were deposited on PCSSC by spin coating technique with a constant speed of 3000 rpm for a time period of 30 sec. The spinel ZnFe_2O_4 nanostructures were deposited as layer by layer (LBL) assembly, 1 to 4 layers (L1, L2, L3, and L4) were deposited. After each layer of deposition, PCSSC was dried at 80°C for 20 min in a hot air oven. The pictorial representation for the synthesis of spinel ZnFe_2O_4 nanoparticles and deposition of spinel ZnFe_2O_4 nanostructures was shown in Fig. 1.

2.4. Characterization techniques

The crystallographic features, crystalline size and phase of the synthesized samples were found using X-ray diffraction (XRD) on Smartlab SE, Rigaku, Canada. The $\text{CuK}\alpha$ radiation with a wavelength of: $1.541 \text{ \AA}$ and scanning rate of $0.5^\circ \text{ min}^{-1}$ by varying step size of 0.02° were used for measurements. XRD measurements were recorded in the range of $2\theta = 20\text{--}80^\circ$. The chemical composition of the synthesized sample calcinated at 510°C was identified using energy dispersive X-Ray

Fig. 1. Pictorial representation for the synthesis of spinel ZnFe_2O_4 nanoparticles and deposition of spinel ZnFe_2O_4 nanostructures

Fluorescence (XRF), Bruker, USA. The elemental composition of spinel ZnFe_2O_4 nanocrystallites coated PCSSC were analysed by Energy Dispersed X-ray Analysis (EDAX). The thickness of spinel ZnFe_2O_4 nanocrystalline films coated PCSSC were measured using Atomic Force Microscopy (AFM, NX 10, PARK systems). The transmittance and reflectivity of spinel ZnFe_2O_4 nanocrystallites coated samples were examined by UV Spectrophotometer, Shimadzu PC 1650 model. The surface morphology of the spinel ZnFe_2O_4 nanocrystallites coated PCSSC was characterized by Field Emission Scanning Electron Microscopy (FE-SEM, MIRA 3, TESCAN, USA). The I-V characteristics of spinel ZnFe_2O_4 nanocrystallites coated and uncoated PCSSC were determined using Keithley I-V meter (Model - 4240). The resistivity of pure and spinel ZnFe_2O_4 nanocrystallites coated PCSSC was determined using the Four Probe technique.

3. Results and discussion

The XRD pattern of the as synthesized ZnFe_2O_4 sample calcinated at 510°C is shown in Fig. 2. From the XRD pattern, it is observed that the obtained diffraction peaks can be clearly indexed with the spinel face centred cubic symmetry and the crystallographic peaks are well matched to the standard powder diffraction data, JCPDS Card No. 82-1042. The obtained Miller indices (220), (311), (400), (422), (511), (440) are substantially

indexed with the spinel ZnFe_2O_4 structure. The most significant peak at the plane (311) was used to measure the approximate size of crystallite using Scherrer formula. The Miller indices and position of diffraction peaks of as synthesized spinel ZnFe_2O_4 are in accordance with the earlier report [31].

Fig. 2. XRD pattern of as synthesized spinel ZnFe_2O_4 nanocrystallites.

The crystallite size D was calculated using the Debye-Scherrer equation as follows:

$$D = \frac{0.9\lambda}{\beta \cos \theta} \quad (1)$$

where, λ is the wavelength of X-ray, β is the Full Width at Half Maximum (FWHM) value of diffraction peaks and θ is the Bragg diffraction angle. It can be seen from Table 1 that all the diffraction peaks typically imitates the presence of nanocrystallites with size range of 40 to 104 nm. The efficiency of the PCSSC were increased for the nanocrystallites having higher size. The increase in output was followed by a major shift in short circuit current density (J_{sc}), with the difference in size.

Table 2 displays the XRF analysis of as synthesized spinel ZnFe_2O_4 nanocrystallites and shows their chemical compositions. As seen in Table 2, the chemical composition in the sample is well agreed with the elemental compositions of spinel ZnFe_2O_4 . The atomic and molecular percentage of zinc and iron oxide confirms the formation of spinel zinc ferrite with chemical composition of

Table 1. Average crystallite sizes of ZnFe_2O_4 sample determined from various diffraction lines

2 Theta	FWHM	Crystallite Size D (nm)
32.8560	0.1407	58.86575
34.6856	0.2047	40.65779
36.4940	0.1919	43.58997
43.0009	0.1791	47.67414
47.7918	0.1023	84.93736
56.4434	0.1279	70.49320
63.0784	0.0895	104.1472
68.3362	0.1023	93.85715

$\text{Zn}_1\text{Fe}_2\text{O}_4$. Further, it can be seen from XRF analysis that the obtained spinel ZnFe_2O_4 nanocrystallites has 99.5% purity with very low quantity of other metal oxides including MnO , CaO , CuO , NiO , Sc_2O_3 and Cr_2O_3 .

FTIR spectra is used to analyse the presence of metal oxides lattice vibrations in high energy fingerprint region as well as the organic functional bonding vibrations at low energy region. FTIR spectra as a function of transmittance (%) versus wavenumber (cm^{-1}) is displayed in Fig. 3. The transmittance bands at around 559 and 696 cm^{-1} indicates the lattice vibrations of the metal oxide ion composed of tetrahedrally coordinated Fe-O bond of the regular spinel ferrites whereas the band at around 400 cm^{-1} is associated with the octahedrally coordinated Fe-O bond lattice vibrations [32].

The characteristic FTIR band at 3385 cm^{-1} indicates the presence of water (O-H stretching frequency) molecules adsorbed on the surface of ZnFe_2O_4 nanocrystallites. The band at 2917 cm^{-1} is due to the stretching vibration of C-H bond and the characteristic bands present at 1631 cm^{-1} is due to the stretching frequency of C=C bonds. It can be revealed from FTIR analysis that the synthesized spinel ZnFe_2O_4 nanocrystallites has adsorbed carbon residue after calcinations and it is due to the usage of precursor ethanolic medium.

The surface topography, morphology and coating thickness of multilayer spinel ZnFe_2O_4 nanocrystallites coated PCSSCs were demonstrated using three dimensional (3D) AFM topog-

 Fig. 3. FTIR spectra of as synthesized spinel ZnFe_2O_4 nanocrystallites.

 Fig. 4. AFM images of spinel ZnFe_2O_4 nanocrystallites coated PSSCs: a) single layer coating, b) double layers coating, c) triple layers coating and d) quarter layers coating

Table 2. XRF analysis of as synthesized spinel ZnFe_2O_4 nanocrystallites

Elements	ZnO	Fe_2O_3	MnO	CaO	CuO	NiO	Sc_2O_3	Cr_2O_3
Chemical Composition (%)	33.377	66.157	0.280	0.072	0.071	0.025	0.014	0.002

raphy images. Fig. 4 exhibits the 3D AFM images of the layer by layer assembly of spinel ZnFe_2O_4 nanocrystallites modified PCSSC: a) single layer coating, b) double layers coating, c) triple layers coating and d) quarter layers coating. It can be seen from AFM analysis that the surface thickness of the L1, L2, L3 and L4 are found to be 103 nm, 205 nm, 260 nm and 431 nm respectively. The thickness and average particle size of the spinel ZnFe_2O_4 nanocrystallites assembly is increased due to the formation of multiple layer coating as well as the presence of Zn^{2+} ions in ferrite (Fe_2O_3) matrix influences the growth of particle size during the curing process [3]. AFM observation clearly indicates the formation of thin film growth processes with more aggregation of hard segments. Nano-micro phase separation can be observed in ZnFe_2O_4 nanocrystallites coated PCSSC with higher contents of ZnFe_2O_4 groups.

Fig. 5. (a) FE-SEM, (b) EDAX spectra and (c) percentage of the elemental composition of ZnFe_2O_4 three layer (L3) coated PSSC.

The morphology and elemental composition of spinel ZnFe_2O_4 nanocrystallites coated (L3 Layer) PCSSC were measured using FE-SEM coupled

with EDAX. The FE-SEM and EDAX spectra of the three layer (L3) coated PCSSC were displayed in Fig. 5(a) and Fig. 5(b). It can be observed from FE-SEM analysis that the spinel ZnFe_2O_4 nanocrystallites are uniformly assembled as cylindrical structure on PCSSC. The elemental composition of the layer three (L3) coated PCSSC was measured using EDAX at room temperature. Fig. 5(c) presents the elemental composition of spinel ZnFe_2O_4 nanocrystallites coated (L3 layer) PCSSC and confirms the presence of elemental Zn, Fe and O with trace amount of Ni. The percentage values obtained for Zn, Fe, Si, Ni and O elements are shown in Fig. 5(c). It is noted that the corresponding atomic mass ratio of the spinel ferrites are well aligned with the prepared stoichiometric ratio. The EDAX spectra and elemental composition (Fig. 5b & c) supports the presence of ZnFe_2O_4 nanocrystallites in surface coating on PCSSC.

Fig. 6. UV transmittance spectra of spinel ZnFe_2O_4 nanocrystallites coated and uncoated solar cells

Fig. 6 and 7 depicts the transmittance and reflectance of ZnFe_2O_4 samples with various film thicknesses. All the spinel ZnFe_2O_4 nanocrystallites coated samples rendered better transmittance, higher than 85%. The transparency gradually in-

creases and reflection decreases from L1 to L3 deposition while reverse effect on reflection was observed in L4 layer deposition due to the aggregation of crystallites.

Fig. 7. Reflectance of spinel ZnFe_2O_4 nanocrystallites coated and uncoated solar cells.

Fig. 8. Electrical resistivity of spinel ZnFe_2O_4 nanocrystallites coated PSSCs.

This also demonstrates the importance of thickness on reflection characteristics of spinel ZnFe_2O_4 nanocrystallites coating. It can be confirmed from Fig. 6 and 7 that the layer thickness influences the optical transparency as well as anti-reflection capacity of spinel ZnFe_2O_4 nanocrystallites functionalized silicon solar cells. It can be noted that

L4 not only lost its transparency, but it also increased its reflection properties and reduced the anti-reflection behaviour due to the aggregation of spinel ZnFe_2O_4 nanocrystallites in coating microstructure.

Fig. 8 shows the resistivity of the spinel ZnFe_2O_4 nanocrystallites, which is measured by using Four Probe technique. The L3 layer coating on the PCSSC shows minimum resistivity of $3.0 \times 10^{-3} \Omega \text{ cm}$ compared to the reference (uncoated) PCSSC resistivity of $6.8 \times 10^{-3} \Omega \text{ cm}$ (Fig. 8). Further, the slight increase in the electrical resistivity of the L4 layer may be due to the change in crystalline size without any change in crystal orientation based on the thickness effect [3, 4].

Fig. 9. I-V characteristics of spinel ZnFe_2O_4 nanocrystallites coated and uncoated PSSCs.

Fig. 9 presents the I-V characteristics of the spinel ZnFe_2O_4 nanocrystallites coated and uncoated PCSSC with the dimensions of $52 \text{ mm} \times 32 \text{ mm}$. The I-V characteristics were performed using a solar simulator under a closed-loop light stabilization system with the simulated 1.5 AM global spectrum. It clearly shows that the increased layers of spinel ZnFe_2O_4 nanocrystallites coating increases the open circuit voltage (V_{oc}), short circuit current (I_{sc}), fill factor and the efficiency η (%), as shown in the Table 3. Three layers of spinel ZnFe_2O_4 nanocrystallites deposited PCSSC demonstrates relatively higher I_{sc} and V_{oc} with en-

Table 3. PCE of spinel ZnFe₂O₄ nanocrystallites coated and uncoated PSSCs.

Solar cell	J_{sc} (mA/cm ²)	V_{oc} (V)	Fill factor (%)	Efficiency η (%)
Ref. cell	32.1	0.625	76	15.24
L1	33.6	0.636	76.4	16.37
L2	37.71	0.641	77.1	18.63
L3	39.30	0.652	78.2	20.03
L4	38.5	0.651	77.5	19.39

hanced power conversion efficiency (PCE) than the reference (uncoated) and other coated PCSSC.

4. Conclusion

Nanocrystalline spinel ZnFe₂O₄ was prepared from precursors (ZnCl₂ and FeCl₃·6H₂O) through simple co-precipitation method. The prepared spinel ZnFe₂O₄ nanocrystallites were comprehensively characterized by XRD, XRF and FTIR spectroscopy. Spin coating technique was used to fabricate multilayer spinel ZnFe₂O₄ nanocrystallites coatings on PCSSC. The microstructure, morphology, composition of deposited thin films was characterized by AFM, FE-SEM and EDAX analysis. The transmittance, reflectance, electrical, light harvesting and solar energy conversion properties of developed thin film coatings on PCSSC was demonstrated using UV-visible spectroscopy, four probe technique and I-V characterization respectively. The present research outputs confirm that the developed spinel ZnFe₂O₄ nanocrystallites thin films accommodate the transmittance of 90.1% and the reduction in reflectivity of 20.3% with improved light trapping and harvesting efficiency of 17.5%. Remarkably, 20% enhanced PCE of PCSSC has been achieved by deposition of L3 layer coated (260 nm thickness) spinel ZnFe₂O₄ nanocrystallites thin films on PCSSC. Hence, it is evident that the spinel ZnFe₂O₄ nanocrystallites act as a suitable light harvesting and anti-reflection material for PCSSC.

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