



# N-acetylcysteine assisted synthesis of core–shell Ag<sub>2</sub>S with enhanced light transmission and diminished reflectance: Surface modifier for c-SiN<sub>x</sub> solar cells



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## ABSTRACT

We report the synthesis of an amino acid viz. N-acetylcysteine (NAC) assisted silver sulphide (Ag<sub>2</sub>S) nanoparticles by in situ reduction technique of silver nitrate (AgNO<sub>3</sub>) with sodium borohydride (NaBH<sub>4</sub>) in presence of NAC, where NAC acted as the sulphur donor as well as encapsulating agent. The prepared nanoparticles were characterized by transmission electron microscopy, scanning electron microscopy, atomic force microscopy, X-ray diffraction technique and dynamic light scattering technique. An enhancement was noticed in the current–voltage characteristics, with major reduction in reflectance when c-SiN<sub>x</sub> solar cells were coated with such nanoparticles. Broadband enhancement of external quantum efficiency (EQE) was also observed.

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## Introduction

Silver sulphide (Ag<sub>2</sub>S) nanoparticles have received great attentions due to its fascinating and exceptional properties [1–6] as well as numerous applications in different fields of modern science and technology since last couple of decades. Ag<sub>2</sub>S, a compound semiconductor, appears in group I–VI with band gap [1] energy between 1.0 and 2.0 eV, is a favorable material in case of photovoltaic applications [4] due to its absorption mostly in the visible region. Several methods are available for the synthesis of shape and size controlled silver sulphide nanoparticles (NPs) using different stabilizing and protecting agents like polymers/organic molecules. With well controlled size and shape, Ag<sub>2</sub>S nanoparticles have great impact on emerging applications as they show diverse, fine and individual properties depending on their shape and size. Among different techniques of silver sulphide NP

preparation [1–6] like, chemical bath deposition, spray pyrolysis, successive ionic layer absorbance and reaction, thermal evaporation, sol–gel reduction, chemical deposition; we have opted an in situ chemical reduction pathway due to its low cost, easily controllable and large scale production facilities. Depending upon the preparation technique, silver sulphide nanoparticles can show different shapes and sizes, which lead to diversified optical and electronic/electrical properties [1–6]. Making a complex of the precursor metal ion (here Ag<sup>+</sup>) by a large organic molecule may result in some specific shape of the product semiconductor, as such large molecules can also act as the structure orienting or caging agents. Such complex molecules also help in preventing the agglomeration of the semiconductor. As there is high affinity of silver atoms toward sulphur due to soft–soft interaction; ligands with sulphhydryl group can be used to prepare stabilized and capped silver sulphide nanoparticles [1]. N-acetyl cysteine (NAC) is an α-amino acid where cysteine acted as sulphur (S) donor [2]. The accurate concentration of NAC and the temperature of the reaction bath matters a lot to acquire stable and monodispersed Ag<sub>2</sub>S NPs in the solution/colloid; high amount

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of NAC may cause aggregation of the nanoparticles due to faster reaction rate [3].

Our present study reports the synthesis of N-acetyl cysteine assisted worm-like silver sulphide nanoparticles by in situ chemical technique, where NAC acted as a structure orienting as well as complexing agent with sulphur donor. Extensive studies on the prepared nanoparticles were carried out. The prepared material showed significant application as a good antireflection coating for c-Si solar cells leading to its overall efficiency enhancement.

## Materials and methods

### Materials preparation

To synthesize the cysteine protected Ag<sub>2</sub>S NPs, chemicals used were: AgNO<sub>3</sub> (99.9% Sigma–Aldrich), NaBH<sub>4</sub> (99.99% Sigma–Aldrich) and N-acetyl cysteine (99.9% Sigma–Aldrich). All chemicals were of Analytical Reagent (AR) grade and used without further purification. Redistilled water was used to prepare the solutions. For the in situ synthesis of Ag<sub>2</sub>S, chemical reduction method using the following scheme was employed:



In a typical process, 45 mg of N-acetyl cysteine (NAC) was dissolved in 10 ml of de-ionized (DI) water (Millipore, ~18 MΩ) and stirred at 300 rpm for 5 min, and then 109 mg of AgNO<sub>3</sub> (solid) was added to this solution. After 5 min, 10 ml of 0.1 M of freshly prepared sodium borohydride (NaBH<sub>4</sub>) solution was added to it under continuous stirring. Before the addition of NaBH<sub>4</sub> the pH of the solution was cited as 4.5, however, after addition the pH was found to be 12. Temperature of the reaction bath was maintained 4–6 °C to slow down the overall process. The color of the final solution slowly turned in to deep brown, representing the color of Ag<sub>2</sub>S.

To study the optical properties of the coatings made from the prepared Ag<sub>2</sub>S, the stock solution was diluted in several proportions viz. 50%, 65%, 80% and 99% using redistilled water and spin coated (1 μL each) over a properly cleaned (cleaned successively with de-ionized water and boiling methanol) clinical glass slide with an active area of 1 cm × 2 cm at 150 rpm and then dried in a hot air oven at 30 °C for 10 min. Among the lot, the best optical transmittance and reflectance were obtained for the 99% diluted solution. So, the 99% diluted stock solution was taken to study its influence on the performance of a c-SiN<sub>x</sub> solar cell and other structural characterization.

### Characterization techniques

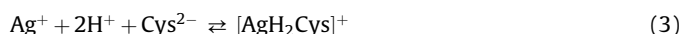
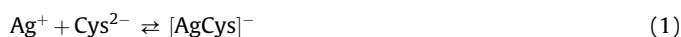
Particle size was determined using Malvern Laser Particle Size Analyzer (Zetasizer 1000 HS). X-ray diffractometer (XRD) with Bragg–Brentano goniometer and Cu–Kα radiation (λ = 1.54056 Å) was used to carryout XRD analysis. X-ray photo-electron spectroscopy (XPS, PHI 5000 VersaProbell) was carried out to confirm the composition of the prepared material. JEOL JEM 2100F transmission electron microscope (TEM, acceleration voltage 200 kV) was used to acquire the TEM images. A Bruker (D8) Field emission scanning electron microscopy (FESEM) was done with a Carl Zeiss ΣIGMA scanning electron microscope. Topography of the deposited films were analyzed using atomic force microscopy (NT-MDT Solver next) operated in semi-contact mode. To reveal the reflectance and transmittance, Shimadzu Solid Spec – 3700 UV-Vis-NIR spectrophotometer was used. Fourier transform infrared (FTIR) spectroscopic analyses were carried out using Shimadzu IRA

Infinity–1 FTIR spectrophotometer. External quantum efficiency (EQE) and current–voltage (I–V) characteristics were measured using Bentham PVE300 and Oriel Sol 3A Solar Simulator, respectively.

## Results and discussions

### Proposed mechanism

Before the addition of NaBH<sub>4</sub>, to the bath, the reactions mainly deals with the coordination of the sulfur atom of the Cys<sup>2–</sup> anion to Ag<sup>+</sup> ions followed by the protonation of the amine and carboxylate groups of the Cys<sup>2–</sup> moiety as described in detail by Alekseev et al. [7]. This reveals that silver has a tendency to form bi-metallic complexes with the NAC moiety, which can be explained with the help of the following reactions:



After the addition of NaBH<sub>4</sub>, the complex compound [Ag<sub>2</sub>H<sub>2</sub>Cys]<sup>+</sup> gets reduced to form [Ag<sub>2</sub>Cys] sol which may be presented as:



Podstawka et al. [8] studied different amino-acid stabilized silver nanoparticles and proved that cysteine molecules can interact with the Ag NPs mainly through the –CS groups due to strong soft-soft interaction between Ag and cysteinyl-S. Rao et al. [9] also discussed the capping phenomenon of noble metals with L-cysteine molecules to form stable covalent bonds via the sulfur atom. The polymerization of the bi-metallic protonated complex results in the formation of a colloid. The NAC modified colloidal silver NPs slowly transform to Ag<sub>2</sub>S nanoparticles by the cleavage of the C–S bonds [10,11]. On the basis of previous references [10,11] on C–S bond cleavage, we propose the following plausible mechanism as summarized in Fig. 1 for the NAC assisted synthesis of Ag<sub>2</sub>S NPs in our system. There are two plausible pathways for the formation of the encapsulated Ag<sub>2</sub>S NPs, Path 1: via the formation of a straight chain Ag–Cysteine complex and Path 2: via the formation of a zig-zag Ag–Cysteine complex. From the mechanism, it is evident that the metallic silver also influences the cleavage of the C–S bonds of the bi-metallic polymeric complex [12,13]. The strong soft-soft interaction between the soft acid Ag and soft base cysteinyl-S might be the reason behind this. The formed Ag<sub>2</sub>S NPs subsequently get encapsulated by the polymeric side product (Compound A) and retain the colloidal stability for about 100 days. The presence of the polymeric encapsulation is also evident from transmission electron microscopy which would be discussed in the characterization section.

### Dynamic light scattering

Particle size determination of the prepared cysteine assisted Ag<sub>2</sub>S colloid was performed by dynamic light scattering measurements with successive and increasing dilution by de-ionized water of the stock solution with the initial concentration as obtained by the chemical reactions mentioned in Materials Preparation Section. After 6th half-dilution followed by ultrasonication for 15 min, mainly one large peak centered ~40 nm was observed as shown in Fig. 2. This indicates the presence of

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