

Thermal Annealing of Magnesium Sulphide (MgS) Thin Films: Surface Interface Studies for Energy Applications

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Abstract

The surface morphology of materials can be changed/modulated by the application of temperature. sulphides of various alkaline earth metals like beryllium, calcium, magnesium, strontium, and barium are receiving interest because of the various advanced energy, sensors, and photochemical applications. Magnesium and nano magnesium are also receiving great interest in the last few years due to their Hydrogen storage capacity. Thermal annealing is a very important technique for the modification of surface properties. This can also change the modulated nano-interface of the materials. Hence it is important to determine the effect of temperature on sulfides of various alkaline earth metals like beryllium, calcium, magnesium, calcium, strontium, and barium. The study of thermal response to MgS thin films is complex and important to fully understand, as a highly challenging task to use in energy applications and water purification. Recently, authors have invested in the effect of annealing/hardening on magnesium sulphide (MgS) films. Thin films of MgS (Magnesium Sulphide) were prepared/ synthesized using spin coating system techniques. Then the films were annealed at various temperatures and times. The characterization of the films was done by SEM and AFM. The results indicate a change in the surface morphology of the films. The order of the change in surface parameters also depends on temperature, duration of temperature exposure, critical environment, presence of various impurities, the thickness of thin films, solution concentrations, etc. Various sizes of Nano-particles also significantly affect the UV-Vis and optical response of nanomaterial and are markedly different from materials.

Keywords: Thin film, annealing, energy application, surface properties, AFM

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Received Date: December 07, 2022

Accepted Date: March 23, 2023

Published Date: March 30, 2023

Citation: Narendra Kumar Agrawal, Dhvani Gangal, Ravi Agarwal, Narendra Jhakar, H.S. Palsania. Thermal Annealing of Magnesium Sulphide (MgS) Thin Films: Surface-Interface Studies for Energy Applications. Research & Reviews: Journal of Physics. 2022; 11(3): 26–30p.

INTRODUCTION

Magnesium sulphide (MgS) has gained immense interest because of its various advanced energy applications as well as in field effect transistors, light emitting diodes (LED), environmental, biological sensors, photocatalysis, water purification systems, nanomedicine, photo-electrochemical activity, photocatalysis, and mycobacterium activity [1–14], etc. MgS has an energy band gap of 2.42 eV at 300 K with cubic, hexagonal, or mixed crystal structures depending on growth and deposition [15]. Its size-dependent properties and real-stage applications are enhanced as thin films for the synthesis of light-emitting diodes (LED), flame retardant, dye-sensitized solar cells, gas sensors, solar energy conversion, and ultrasonic sensors [11–18].

The solution is prepared in a solvent that evaporates fast at room temperature, like ethanol, acetone, etc. [8]. The solution is poured in large amounts over the substrate and then rotated at high speed (~6000 rpm) to spread the fluid uniformly due to centrifugal force [18–21]. Thermal annealing can modulate surface morphology and structural properties that are useful for improving surface-interface properties [5]. To study the effect of annealing, recently we have synthesized MgS nanoparticles using chemical routes and characterized them in terms of nano range using various techniques. MgS NPs thus obtained were re-disperse acetone and used for the synthesis of thin film using the spin coating method. Low-temperature thermal annealing was performed at atmospheric pressure to modify the surface morphology and structural properties of thin films.

METHODOLOGY

0.1 M magnesium acetate (10 ml), 0.1 M sodium sulphide (10 ml), and 0.1 M ethylene diamine tetra acetate (EDTA) (20 ml) solutions were prepared in distilled water, and allowed to stir for 1 hour. Magnesium acetate and sodium sulphide solutions were then simultaneously poured into the EDTA solution dropwise with continuous stirring. The mixture was stirred overnight, which was then centrifuged using distilled water, and the final solution was prepared in acetone. The size of MgS NPs was determined by TEM. Technika TEM instrument operating at 200 kV was used for performing imaging. The thin films, from the so obtained solution, were prepared over a glass substrate using the spin coating technique at 6000 rpm. Prior to this, the substrate was thoroughly cleaned by immersing it in detergent for 15 minutes and then washing them with acetone and ethanol solution sequentially. Atmospheric thermal annealing of the thin films was done by keeping them in the oven at different temperatures (100°C, 200°C, and 300°C) for a constant time period (30 minutes). UV-Vis spectroscopy (by spectrophotometer Shimadzu 1800) generates absorption data corresponding to absorption wavelength, from which the energy band gap can be calculated, to ascertain the formation of thin films and modification band gap. The surface structure of the films was determined by an optical microscope and AFM (Nanosurf easyScan 2).

RESULT AND DISCUSSIONS

Synthesized MgS NPs are first dried and then re-dispersed in acetone by ultra-sonication. TEM image of Magnesium Sulphide MgS NPs having size 10–25 nm (Figure 1) shows individual particles. Each particle is easily identifiable. Hence, no aggregations in MgS NPs particles are observed in the image.

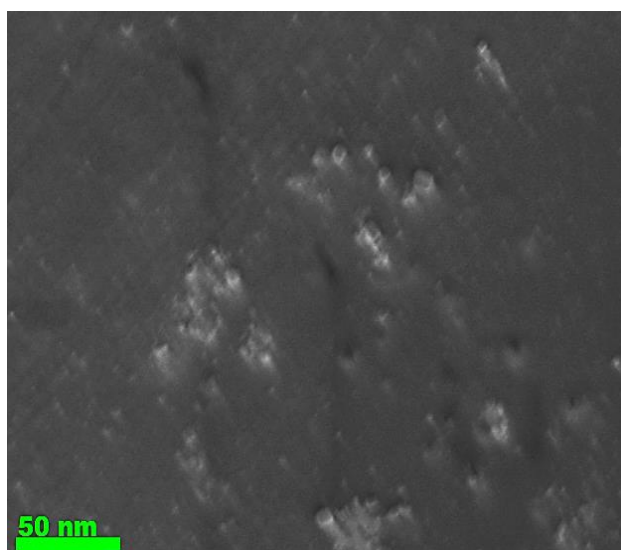


Figure 1. Transmission electron micrographs (TEM) of MgS NPs synthesized, having a spherical shape and particle size ranging from 10–25 nm.

Optical microscope is used to investigate/probe the effect of annealing (Figure 2). Atmospheric thermal annealing at low temperatures has increased the surface morphology and surface roughness of the films. Thin films were further investigated at lower dimensions (higher magnifications) by SEM or Scanning Electron micrographs (Figure 3).

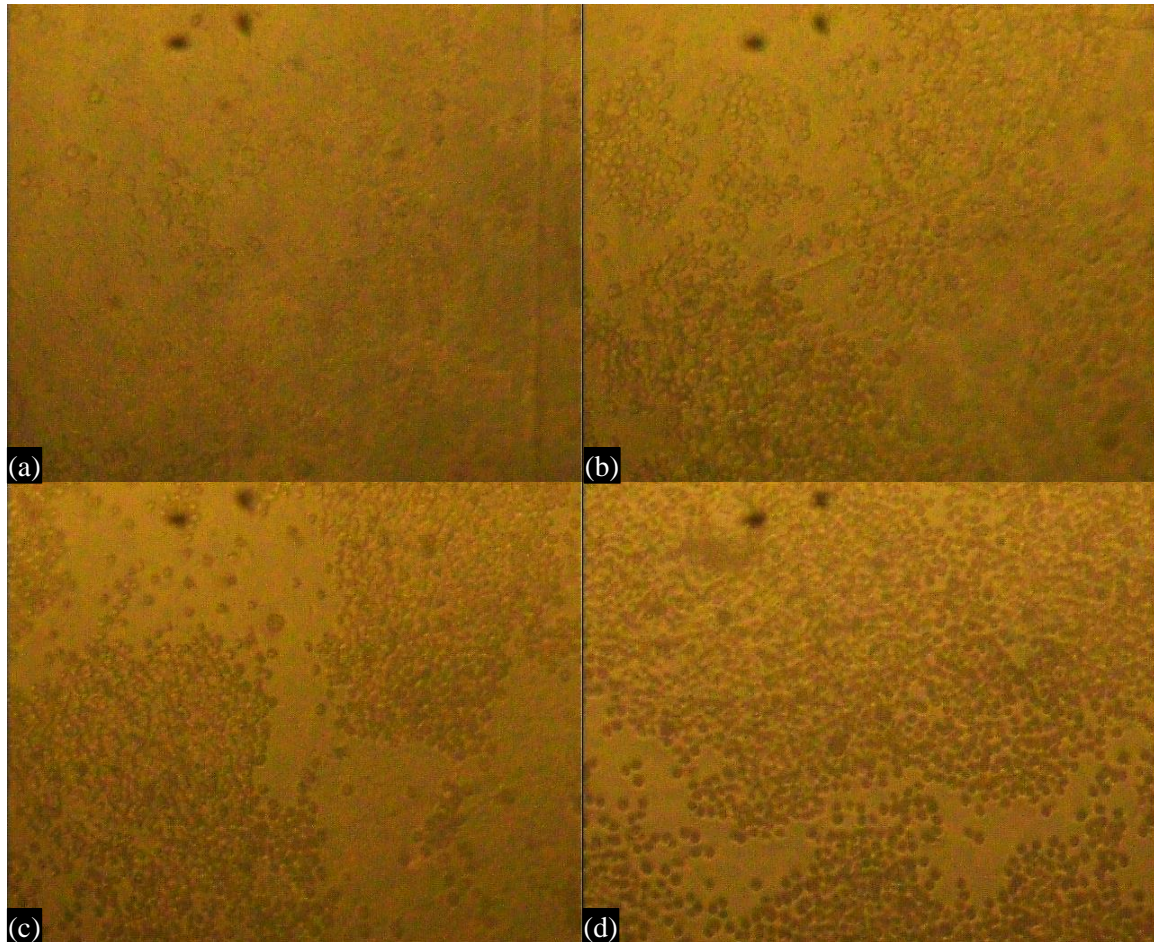


Figure 2. Optical microscope images of MgS Thin Films (a) MgS thin films before atmospheric thermal annealing (b) MgS thin films after atmospheric thermal annealing 100°C, (c) MgS thin films after atmospheric thermal annealing 200°C, (d) MgS thin films after atmospheric thermal annealing 300°C.

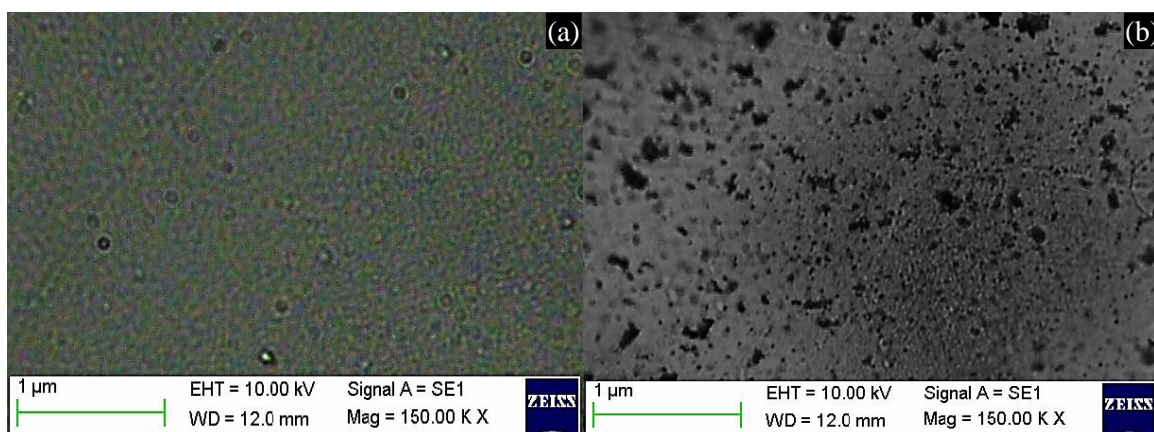


Figure 3. SEM images of MgS thin films (a) MgS thin films before atmospheric thermal annealing, (b) MgS thin films after atmospheric thermal annealing 300°C.

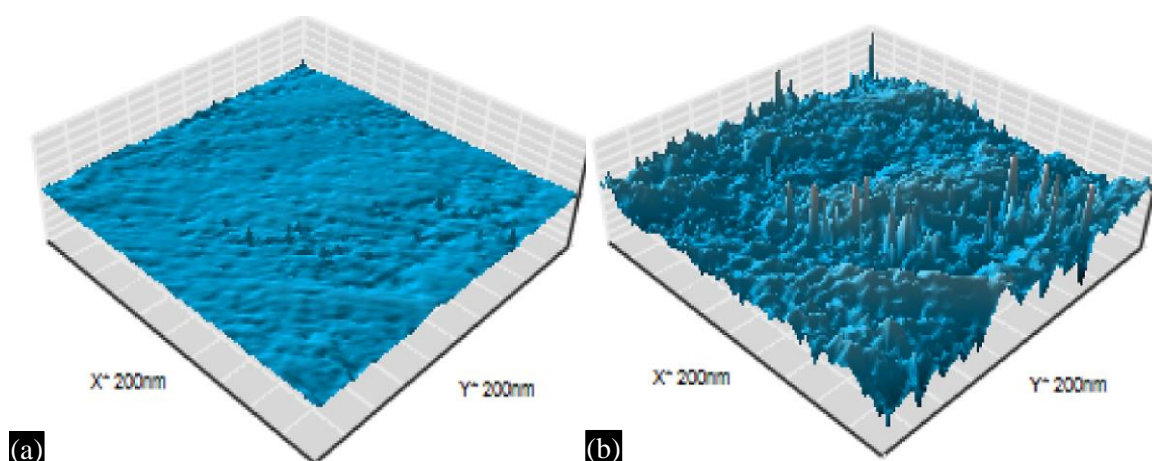


Figure 4. AFM images of MgS thin films (a) MgS PS Nanocomposite before plasma treatment, (b) MgS PS Nanocomposite after plasma treatment 200°C.

Higher surface roughness was observed after atmospheric thermal annealing at low temperatures. These results show the effectiveness of thermal annealing in the modulation of surface properties of thin films. Topography and Surface morphologies of MgS thin films determined using AFM (Figure 4) show high surface roughness.

CONCLUSIONS

Nanoparticles of MgS having sizes 10–25 nm and spherical are synthesized using a chemical method. MgS thin films are prepared by the spin coating method. The films are then exposed to atmospheric thermal annealing at low temperatures for improving the surface properties and activation. An increase in roughness and topography was observed by SEM and AFM.

Acknowledgment

We acknowledge CSIR-HRDG (Council of Scientific and Industrial Research-Human Resource Development Group) for providing fellowship/funding to Dr. Narendra Kumar Agrawal to carry out the work. We are also thankful to the Department of Physics, University of Rajasthan, and Malaviya National Institute of Technology for providing facilities for XRD, SEM, and TEM analysis. We are also thankful to the Department of Physics, MNIT, and UOR for providing the facility for SEM analysis.

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