



Review

Electrochemical synthesis of molybdenum sulfide semiconductor



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ARTICLE INFO

Keywords:

Electrodeposition
Molybdenum Disulfide
Ionic liquids
Aqueous solutions
Non-aqueous
Molten salts

ABSTRACT

The electrochemical deposition of MoS₂ from different media; aqueous, non-aqueous, molten salts and ionic liquids have been reviewed and discussed in this review. Electrochemical methods and substrates used for the electrodeposition of MoS₂ have been also discussed. All of the parameters affecting the electrodeposition process including temperature, pH and solution composition which lead to suitable shape of MoS₂ for using in different applications as photoelectrochemistry have been showed and discussed. The reaction mechanisms of the electrodeposition process of MoS₂ have been explored and discussed.

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1. Introduction

MoS₂ is one of an important IV–VI semiconductor type and diamagnetic, indirect bandgap semiconductor similar

to silicon, with a band gap of 1.7 eV. MoS₂ with particle size in the range of 1–100 μm is a common dry lubricant. Few alternatives exist that confer high lubricity and stability at up to 350 °C in oxidizing environments [1,2].

When added to plastics, MoS₂ forms a composite with improved strength as well as reduced friction. Polymers that have been filled with MoS₂ include nylon (with the trade name Nylatron), Teflon, and Vespel. Self-lubricating composite coatings for high-temperature applications have

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been developed consisting of molybdenum disulfide and titanium nitride by chemical vapor deposition.

MoS₂ is employed as a co-catalyst for desulfurization in Petrochemistry; e.g., hydro-desulfurization [3]. The effectiveness of the MoS₂ catalysts is enhanced by doping with small amounts of cobalt or nickel and the intimate mixture is supported on alumina. Such catalysts are generated in situ by treating molybdate/cobalt or nickel-impregnated alumina with H₂S or an equivalent reagent.

Nanotubes and buckyball-like molecules composed of MoS₂ exhibit unusual tribology and electronic properties [4]. MoS₂ has been investigated as a component of photoelectrochemical (e.g. for photocatalytic hydrogen production) applications and for microelectronics applications [5]. MoS₂ and other transition metal dichalcogenides form bulk crystals composed of two-dimensional layers stacked in the vertical direction. Such two-dimensional layers are similar in form to graphene and express diverse electronic and optical properties [6] that can differ from those in the bulk. Whereas bulk MoS₂ has an indirect band gap of 1.2 eV, single layers of MoS₂ have a direct 1.8 eV electronic bandgap [7] allowing the production of switchable transistors [5] and sensitive photodetectors [8]. The sulfur group on the surface of MoS₂ interacts with noble metals, including gold. The bond between MoS₂ and gold nanostructures was found to act as a highly coupled gate capacitor with a reduced carrier-transport thermal-barrier and increased thermal conductivity [9].

The main advantages of such semiconductors (MoS₂) is that they meet the requirements in order to function as a lubricant and efficient photoelectrode, (i) they exhibit intercrystalline slip and they adhere to the substrate well [10], and (ii) their inherently resistive nature to photocorrosion [11], because the photo transitions involve non-bonding d–d orbitals of Mo atoms [12].

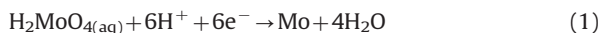
The usual thin film preparation techniques, such as several methods already reported for preparation of MoS₂ thin layers, including scotch tape assisted micromechanical exfoliation [13,14], intercalation assisted exfoliation [15,16], solution exfoliation [17,18], physical vapor deposition [19,20], hydrothermal synthesis [21] and sulfurization of molybdenum oxides [22,23] are cost intensive and sometimes present special problems for the preparation of transition metal chalcogenides films.

Electrochemical deposition is an attractive method for preparation of thin films in commercial quantities because it uses relatively cheap equipment, enables the deposition in large area and easy control of growth parameters through applied potential, current, pH and temperature of the bath. Many researchers have studied the mechanism of an electrochemical deposition of MoS₂ from aqueous and little from non-aqueous solutions. A variety of electrodeposition techniques have been reported for the preparation of MoS₂ films, using anodic oxidation or cathodic reduction. Good results and observations were obtained and reported in this review.

2. Electrodeposition of MoS₂ thin films from aqueous solutions

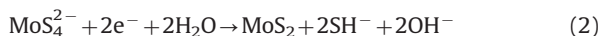
It is known that, the electrodeposition potential of molybdenum is more negative than the electroreduction

potential of hydrogen, so difficulties may be found for the cathodic electroreduction of molybdenum chalcogenide films from aqueous solutions. Theoretically, the deposition of pure molybdenum by electrolytic reduction of molybdates in acidic aqueous solutions is possible according to the reaction equation as follows:



It should be noted that, the MoS₂ electroreduction from solutions of molybdate and thiosulfate under various conditions of pH and temperature has some difficulties, which may be attributed to the big difference between the electroreduction potential of Mo and the electroreduction of thiosulfate. But in other work it was reported that, thin films of molybdenum disulfide have been prepared by electroreduction method under galvanostatic conditions for about ten minutes from an aqueous bath by using molybdenum trioxide dissolved in ammonia and sodium thiosulfate as Mo⁺ ion and S⁻² ion sources, respectively. The films deposited onto stainless steel and fluorine doped tin oxide (FTO) glass substrates were adherent to the substrate. The estimated deposition potentials for molybdenum disulfides is 1.32 V. X-ray diffraction studies show that films are polycrystalline in nature and orientation preferentially along the (116) plane. SEM shows films are continuous and uniform. Optical absorption gives bandgap energy of 1.7 eV [24].

Molybdenum sulfide thin films can be cathodically electroreduced under potentiostatic conditions at a potential of –1.4 V vs. SCE from aqueous solutions of sodium tetrathiomolybdate Na₂MoS₄ anion on quartz substrate covered by a thin Ni–Cr layer. It was carried out by a two-electron reduction of tetrathiomolybdate ions (MoS₄²⁻) as follows:



Annealing of these films in inert atmosphere at high-temperature in presence of sulfur resulted in the formation of highly-textured films of MoS₂ with the van der Waals planes parallel to the substrate and crystal size in the micrometer range. The annealed films are photoactive and their photoconductivity measurements reveal a direct band gap of about 1.71 eV [25]. But when the Mo and conductive glass were used as substrates, the electroreduced annealed films have an optical spectrum characteristic of MoS₂, with a direct bandgap of 1.78 eV [26].

Polycrystalline thin films of MoS₂ are electroreduced cathodically by using molybdic acid (H₂MoO₄) and sodium thiosulfate as initial solution on tin oxide (SnO₂) coated conducting glass substrates. The deposited films are characterized by various techniques includes the X-ray diffraction analysis, where the structure of the films is identified as hexagonal. From optical analysis, the bandgap value is calculated as 1.68 eV with indirect bandgap nature. From scanning electron micrographs, the surface appears to be comparatively granular with grains in irregularly shaped. From Mott–Schottky plots the films are found to be n-type semiconductor [27].

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