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Synthesis of copper sulphide thin film on Indium Tin Oxide glass plate by SILAR method and its characterization

ABSTRACT

Thin film of copper sulphide has been deposited on Indium Tin Oxide (ITO) glass plate. This film has been characterized by UV-Visible reflectance spectroscopy, FTIR spectroscopy, EDAX, and SEM. The film has been subjected to polarization study by immersing in sea water. The above studies on the thin film has been compared with copper sulphide prepared by chemical method; that is by mixing a solution of copper sulphate and sodium sulphide solution. For comparison study methods such as UV-Visible reflectance and FTIR have been employed. The UV-Visible reflectance spectrum reveals that the band gap of the copper sulphide film is 1.823eV. This indicates that the film functions as semi conductor. The UV- Visible absorption study of the film indicates that the λ_{max} *appears at 310 nm. The FTIR study of the copper sulphide film confirms the presence of CuS. The polarization study reveals that the linear polarization resistance (LPR) value decreases, when compared to ITO plate immersed in sea water.This indicates that the current flowing through the thin film increases.Such a finding can be used in solar cells. This is supported by the fact that the current flowing through the thin film, when it is immersed in sea water increases, when compared to the current flowing through the empty glass plate (without black coating) is immersed in sea water. This is further supported by the fact that for black thin film, the band gap decreases after coating.*

The EDAX study confirms the presence of elements Cu and S.The SEM study reveals the presence of thin film of copper sulphide on the ITO glass plate. The particle size of the copper sulphide is in the range of 101.1nm, 107.8nm and 114.5nm. Thus it is encouraging to note that copper sulphide nano particles have been prepared by SILAR method.

Keywords: *synthesis of thin film, copper sulphide, SILAR method, characterization, FTIR, EDAX.*

1. INTRODUCTION

A successive ionic layer adsorption and reaction (SILAR) method is one of the chemical methods for making uniform and large area thin films, which is based on immersion of the substrate into separately placed cations and anions.Compared to other methods, the successive ionic layer adsorption and reaction (SILAR) method is a simple, less expensive, and less time-consuming method for the deposition of binary semiconducting thin films. It is also applicable in the deposition of large-area thin films.

i) Initial adsorption of cationic precursor with its corresponding anion to form the Helmholtz double layer;

ii) Rinse-off to form a hypothetical monolayer in an ideal scenario;

iii) Reaction of cationic and anionic precursors to form the product;

iv) Rinse-off to remove excess ions and products.

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Several research works have been reported in this field [1-10].

Thin film materials have been used in semiconductor devices, wireless communications, telecommunications, integrated circuits, rectifiers, transistors, solar cells, lightemitting diodes, photoconductors and light crystal displays, lithography, micro- electromechanical systems (MEMS) and multifunctional emerging coatings, as well as other emerging cutting technologies. The ZnO, CdO and CdZnO thin films have been deposited by chemical routes such as chemical bath deposition, spray pyrolysis, Sol-Gel spin coating, electrodeposition, SILAR, chemical vapour deposition, atomic layer deposition, and pulsed vapour deposition have been discussed by Amudhavalli et al.[1].

CO_2 photoconversion to solar fuels requires materials with a high affinity to the acidic CO_2, and MgO and $Mg(OH)_2$ films represent good candidates due to their basic sites are highly active for CO_2 capture in a wide interval of temperatures. However, the deposition of MgO and $Mg(OH)_2$ as thin film is difficult to obtain by traditional methods. As an alternative , the successive ionic layer adsorption and reaction (SILAR) method is proposed by Cruz et al. to obtain $MgO/Mg(OH)_2$ mixtures over glass substrates at significantly lower temperatures (200–400°C). The films were tested as photocatalysts in the CO_2 photoconversion to solar fuels (HCOOH and CH_3OH) under UV–visible-NIR irradiation[2].

Yücel and Yücel have designed a reactor to coat the PbS thin film on glass substrate by continuous flow. A methodology is proposed to deposit PbS films based on reducing the roughness via a laminar and continuous flow in the reactor without being affected by the chaotic properties of the turbulent flow. The modified coating method was named flow-through chemical deposition (FTCD). The morphological analysis demonstrated that the surface roughness of PbS films deposited by the FTCD method was lower than the other films[3].

Cobalt doped CdS–TiO_2 nanocomposite thin films were coated by Jostar et al.[4] on glass substrates by bottom up approaching methods. ZnS nanolayer over cobalt doped CdS–TiO_2 films act as a protective layer of CdS nanocrystal which enhances the photo stability. The structural, optical, electrical and surface morphological properties of ZnS-cobalt CdS–TiO_2 composite thin film were systematically evaluated. Structural analysis indicates that Co^{2+} ions have been successfully incorporated in CdS site. The doping of cobalt in ZnS–CdS–TiO_2 film gives rise to an increase in the lattice parameter while the band gap decreased with increasing cobalt concentration. The bandgap of doped film decreases indicating the easy way of electron transfer from valence band to conduction band of CdS and TiO_2[4].

Tocreate new flexible thermoelectric and photosensitive thin-film material for in-plane thermoelectric generator (TEG) and broadband photodetector (PD) Klochko et al. [5] deposited nanostructured indium-doped zinc oxide (ZnO:In) thin films on polyimide (PI) substrates by using the inexpensive and scalable method Successive Ionic Layer Adsorption and Reaction (SILAR). To reduce the resistivity of the ZnO:In film in the ZnO:In/PI composition to about 0.02 Ω·m, it was annealed in a vacuum at 300°C[5].

Using SILAR thin film technique, which is low cost and is easy to control, the lead oxide (PbO) thin film was grown by Erdem et al. [6]onto both microscope glass and inorganic semiconductor silicon (Si) wafer after appropriate chemical cleaning processes. The coating stage was performed by keeping the solution at 80℃. After forming a PbO thin film on the silicon semiconductor, Pb metal was evaporated onto its upper surface. Optical, morphological and structural properties of the PbO thin film formed on glass were investigated[6].

Indium-doped zinc oxide (ZnO) thin films were coated by Adaikalam et al. [7]onto glass and silicon substrates at different indium concentrations (0%, 2.5%, 5%, and 7%) using successive ionic layer adsorption and reaction (SILAR) method. Furthermore, crystalline structural, morphological, and elemental properties of indium-doped ZnO thin films were characterized using X-ray diffraction (XRD), scanning electron microscopy (SEM), and EDAX techniques. Moreover, the optical properties were analyzed using UV–vis optical absorption, photoluminescence (PL), and Raman spectroscopy techniques. XRD analysis confirmed the growth of ZnO films with perfect inclusion of indium ions into the ZnO lattice of the hexagonal wurtzite structure [7].

Pure and tungsten (W) doped ZnO thin films have been successfully deposited by Sales Amalraj et al.[8] on glass substrates by successive ionic layer adsorption and reaction (SILAR) method. Effect of W doping causes the change of strained stress in ZnO films, which subsequently affected its structural and optical properties[8].

Wet chemical syntheses of nanomaterials and especially of thin-film coatings with controllable morphology, structure and composition are very promising for industrial applications due to their inherent simplicity and low cost, provided that the performance of the synthesized nanomaterials can match those achieved with vacuum-based depositions. Ratnayake et al.[9] have shownthe fabrication of bismuth vanadate ($BiVO_4$) thin films using a simple and scalable method called SILAR (Successive Ionic Layer Adsorption and Reaction), which is based on a series of immersion cycles in different precursor solutions. A comprehensive study on the growth of $BiVO_4$ layers, assessing the role of the deposition parameters on the structural, morphological, optical and electronic properties of the deposited films has been presented [9].

Metal–organic frameworks (MOFs) as cocatalyst for photoelectrochemical (PEC) water splitting is promising but still in its infancy due to the challenge to fabricate high-quality MOFs coated photoelectrodes. A successive ionic layer adsorption and reaction (SILAR) method is used by Xie et al.[10] to fabricate high-quality conformal and ultrathin MOFs overlayer with preferred orientation on nanoporous $BiVO_4$ films. 20 molecular monolayers of Co-MOF coated $BiVO_4$ photoanodes exhibit 3.1-folds of photocurrent density in comparison with pristine $BiVO_4$. The onset potential shows a negative shift of 320 mV. To further demonstrate the extensibility of SILAR method, the alternative Co-MOF and Ni-MOF coated $BiVO_4$ photoanodes deliver an enhanced photocurrent density of 2.97 $mAcm^{-2}$. The PEC performance achieved rivals the reported results of $BiVO_4$ and MOF hybrid photoanodes[11].

2. MATERIALS AND METHOD

SILAR METHOD

A successive ionic layer adsorption and reaction (SILAR) method is one of the chemical methods for making uniform and large area thin films, which is based on immersion of the substrate into separately placed cations and anions. Compared to other methods, the successive ionic layer adsorption and reaction (SILAR) method is a simple, less expensive, and less time-consuming method for the deposition of binary semiconducting thin films. It is also applicable in the deposition of large-area thin films.

In the present study, copper sulphide (CuS) thin film on ITO glass plate is prepared by SILAR method, by successive dipping of the ITO glass plate in copper sulphate solution, distilled water, sodium sulphide solution, distilled water, copper sulphate solutio (Figure1).

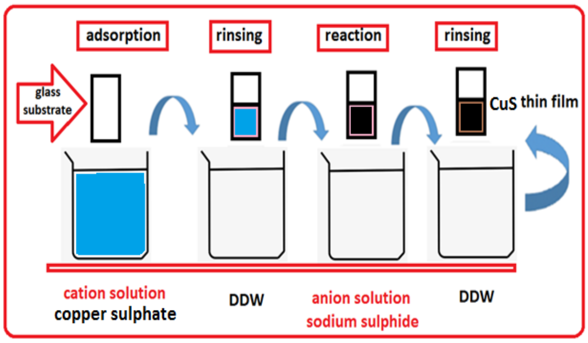

Figure 1. CuS thin film on ITO glass plate prepared by SILAR method

Slika 1. CuS tanak film na ITO staklenoj ploči pripremljen SILAR metodom

UV-Visible Spectroscopy

The UV-Visble spectra were recorded in Agilent diode array spectrometer (Agilent 8453).

FTIR Spectroscopy

FTIR Spectra were recorded in a Perkin Elmer 1600 series model, with resolving power $4cm^{-1}$.

EDAX –Analysis

EDAX analysis was done in Bruker Nano GmbH Berlin workstation.

SEM-Analysis

SEM image of various surfaces were recorded in a CAREL ZEISS make model EVO-18.

Polarization study

Polarization studies were carried out in a CHI Electrochemical work station/ analyzer, model 660A. It was provided with automatic iR compensation facility. A three electrode cell assembly was used.

The working electrode was ITO glass plate before coating and after coating. A (Saturated Calomel Electrode) SCE was the reference electrode. Platinum was the counter electrode. A time interval of 5 to 10 min was given for the system to attain a steady state open circuit potential.From polarization study, corrosion parameters such as corrosion potential (E_{corr}), corrosion current (I_{corr}), Tafel slopes anodic = b_a and cathodic = b_c and LPR (linear polarisation resistance) value were calculated. The scan rate (V/S) was 0.01.

3. RESULTS AND DISCUSSION

Copper sulphide was prepared on the ITO glass plate by SILAR method. The thin film was characterized and analyzed by various techniques.

Analysis of FTIR

Copper sulphide was prepared by mixing copper sulphate solution and sodium sulphide solution.It was dried. Its FTIR spectrum (KBr). It is shown in Figure 2. The CuS peaks appear at 618.26 cm^{-1}, 1105 cm^{-1}, 1627 cm^{-1} and 3466 cm^{-1}[11].

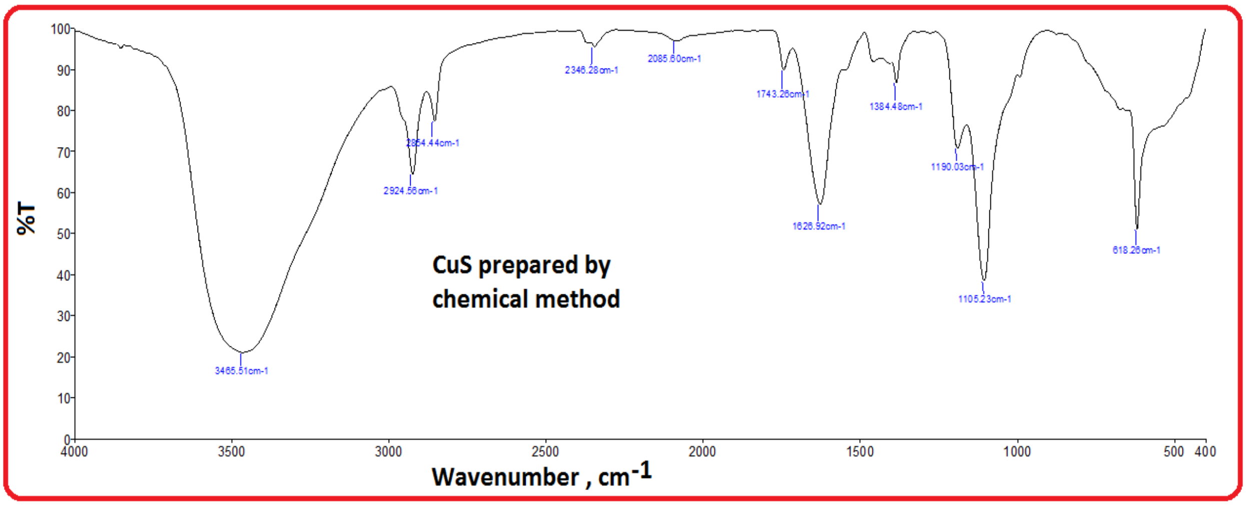

Figure 2. FTIR Spectrum of copper sulphide prepared bymixing copper sulphate solution and sodium sulphide solution (chemical method)

Slika 2. FTIR spektar bakar-sulfida pripremljen mešanjem rastvora bakar-sulfata i rastvora natrijum-sulfida (hemijska metoda)

The FTIR spectrum of copper sulphide formed on ITO glass plate by SILAR method is shown in Figure 3. The peaks due to CuS group appear at 617 cm^{-1}, 1101 cm^{-1}, 1645 cm^{-1} and 3405 cm^{-1}. Thus formation of black thin film on ITO glass plate is confirmed by FTIR spectroscopy.

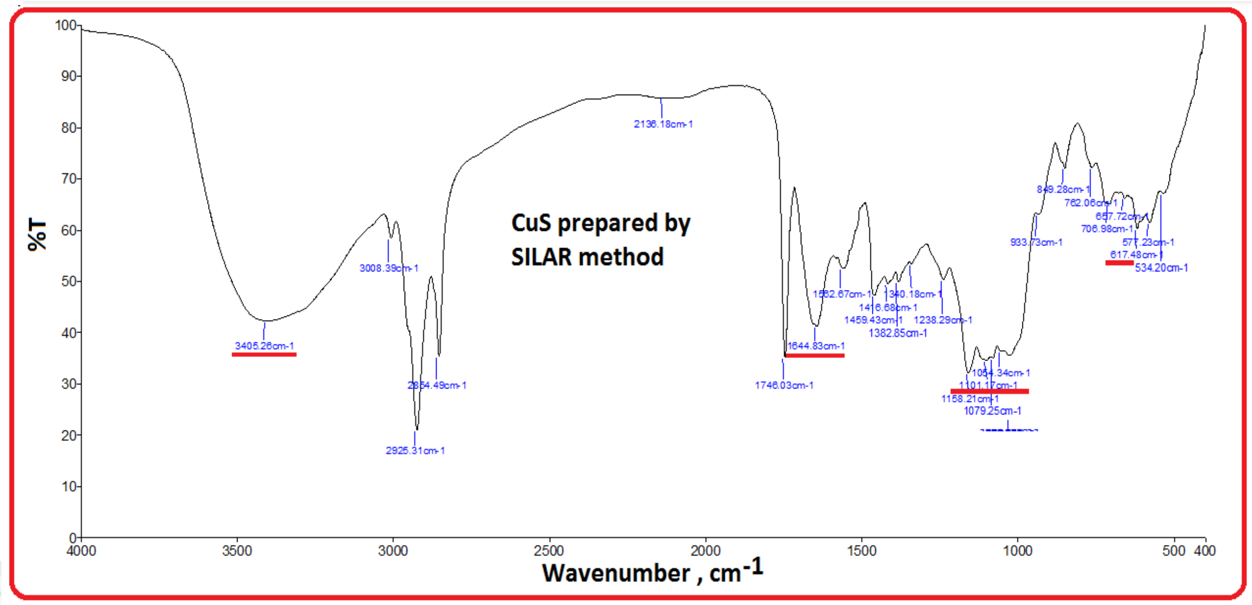

Figure 3. FTIR spectrum of copper sulphide formed on ITO glass plate by SILAR method

Slika 3. FTIR spektar bakar-sulfida formiranog na ITO staklenoj ploči SILAR metodom

Analysis of EDAX

The EDAX of copper sulphide(CuS) film prepared on ITO glass plate by SILAR method is shown in Figure 4. The formula of copper sulphide is CuS. It is observed from Figure 4 that the Cu peaks appears at 0.9 K_{eV} and 8.1 K_{eV}. The peak due to S appears at 0.2 K_{eV} and 2.3 K_{eV}. Thus the formation of Copper sulphide on the ITO glass plate is confirmed by EDAX.

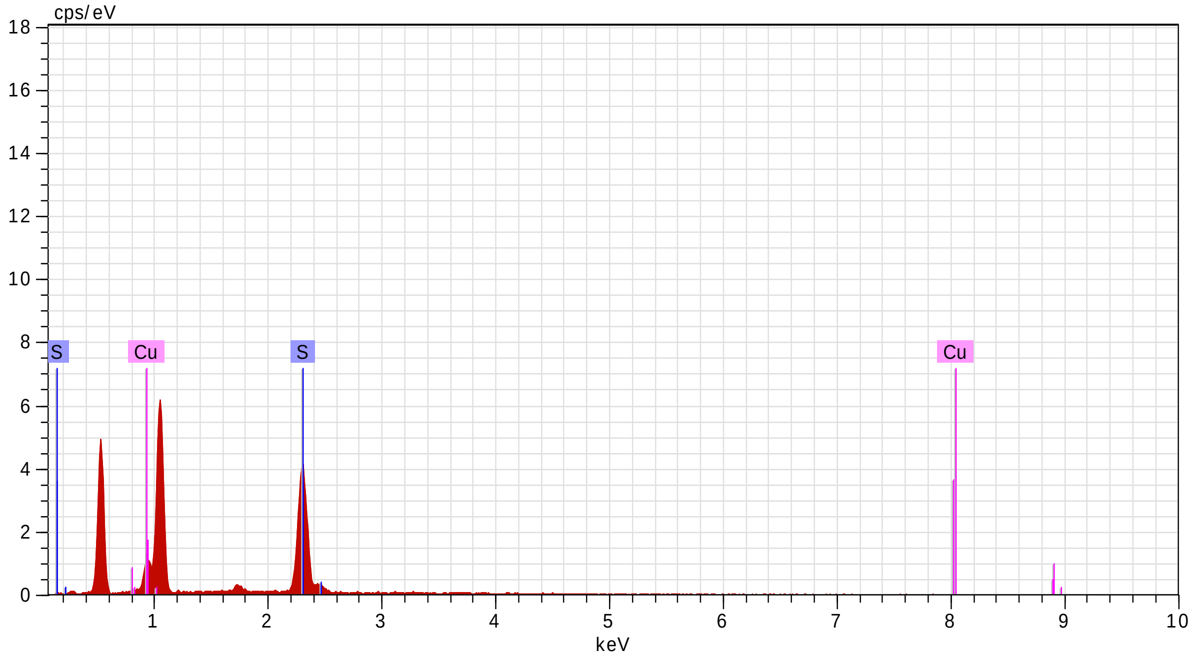

Figure 4.EDAX spectrum of CuS film prepared on ITO glass plate by SILAR method

Slika 4. EDAX spektar CuS filma pripremljenog na ITO staklenoj ploči SILAR metodom

Analysis of Scanning Electron Microscopy

The SEM images of various surfaces were recorded at different magnifications.The SEM image of ITO glass plate is shown in Figure 5. The SEM image of Copper sulphide formed on ITO glass plate by SILAR method is shown in Figure 6. It is observed from the Figure 6,that the sizes of the CuS nano particles are in the range 101.1 nm,107.8 nm and 114.5 nm. Thus it is encouraging to note that copper sulphide nano particles have been prepared by SILAR method.

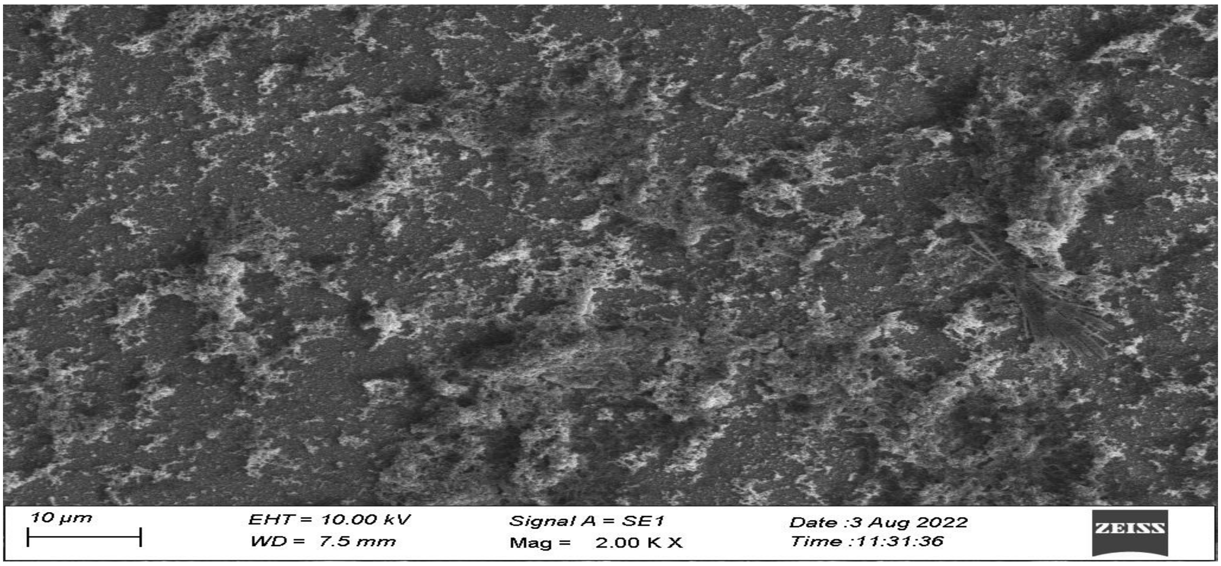

Figure 5. SEM image of ITO glass plate

Slika 5. SEM slika ITO staklene ploče

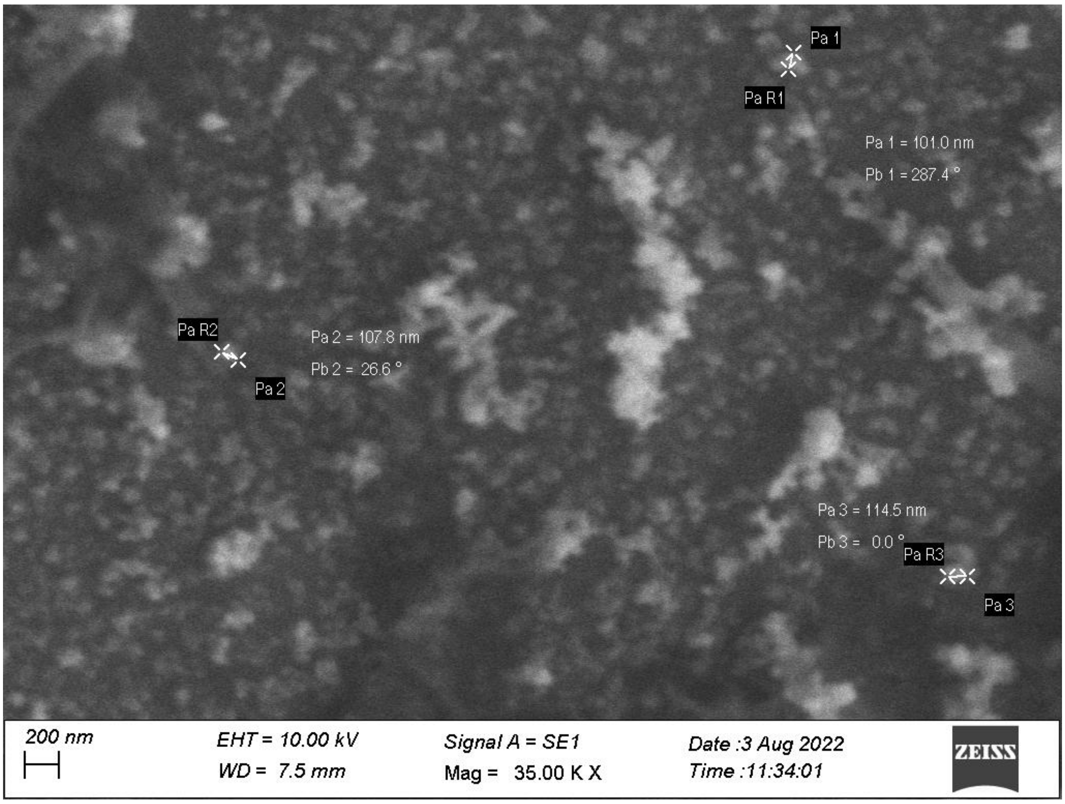

Figure 6. SEM image of CuS nano particles prepared by SILAR method

Slika 6. SEM slika CuS nano čestica pripremljenih SILAR metodom

Analysis of results of Polarisation technique

A thin film (black) of copper sulphide (CuS) was coated on indium tin oxide (ITO) glass plate by SILAR method. Electro chemical study, namely, polarisation study was carried out on the ITO glass plate,before coating and after coating.The glass plates were immersed in sea water.This study will be useful to know the influence of seawater vapour(marine environment) on the conductivity of glass plates in the marine environment,before coating after coating. The findings will be useful in solar cells.

Polarisation Study

The polarisation curves of ITO glass plates immersed in sea water before coating and after coating (CuS-black) are shown in Figure 7.

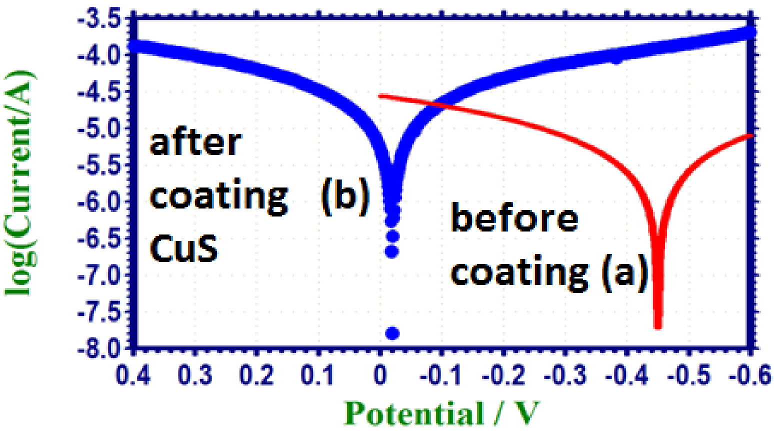

Figure 7. Polarisation curves of ITO glass plates immersed in sea water before coating (a) and aftercoating (b)

Slika 7. Polarizacione krive ITO staklenih ploča potopljenih u morsku vodu pre premaza (a) i posle premaza (b)

Table 1. Corrosion parameters of ITO glass plates in sea waterbefore and after black (CuS) coating

Tabela 1. Parametri korozije ITO staklenih ploča u morskoj vodi pre i posle crnog (CuS) premaza

System	E_{corr} mV vs SCE	b_c mV/decade	b_a mV/decade	LPR Ohmcm^2	I_{corr} A/cm^2
Glass plate before coating	-449	194	198	19685	2.164 x 10^{-6}
Glass plate(black) after coating	-20	205	199	3730	11.77x 10^{-6}

The polarisation parameters namely,corrosion potential(E_{corr}),Tafel slopes (b_c = cathodic,b_a = anodic) Linear polarisation resistance (LPR) and Corrosion current (I_{corr}) are given in the Table 1.

It is observed from Table 1 that when ITO glass plate is immersed in sea water the corrosion potential is -449 mV vs SCE.The current density flowing through the system is 2.164x10^{-6}A/cm^2.The LPR value is 19685 Ohmcm^2.When black film coated ITO glass plate is immersed in sea water the corrosion potential is shifted to anodic side (-20mV vs SCE). The LPR value decreases from 19685 to 3730 Ohmcm^2. The current density flowing through the system increases from 2.164x10^{-6} A/cm^2 to11.77x10^{-6} A/cm^2.

It is inferred that in the presence of film coating, the current flowing through the system increases in the marine environment. So,such glass plates can be used in solar panels used in the marine environment.

Analysis of UV-Visible spectra

The UV-Visible absorption spectrum of copper sulphide film formed on ITO glass plate by SILAR method is shown in Figure 8. Peaks appear at 300.90nm,345.45nm,361.65nm and 385.05nm.

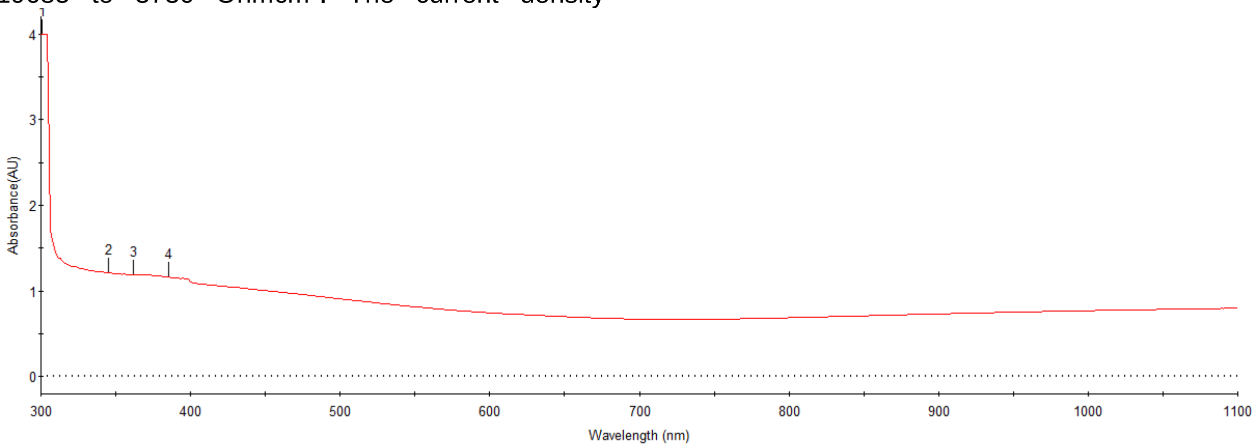

Figure 8. UV-visible absorption spectrum of copper sulphide film formed on ITO glass plate by SILAR method

Slika 8. UV-vidljivi apsorpcioni spektar filma bakar sulfida formiranog na ITO staklenoj ploči SILAR metodom

ITO glass plate

The UV-visible absorption spectrum of ITO glass plate is shown in Figure 9. Peaks appear at 293.50 nm, 299.70 nm, 304.65 nm, 308.35 nm and 532.95nm.

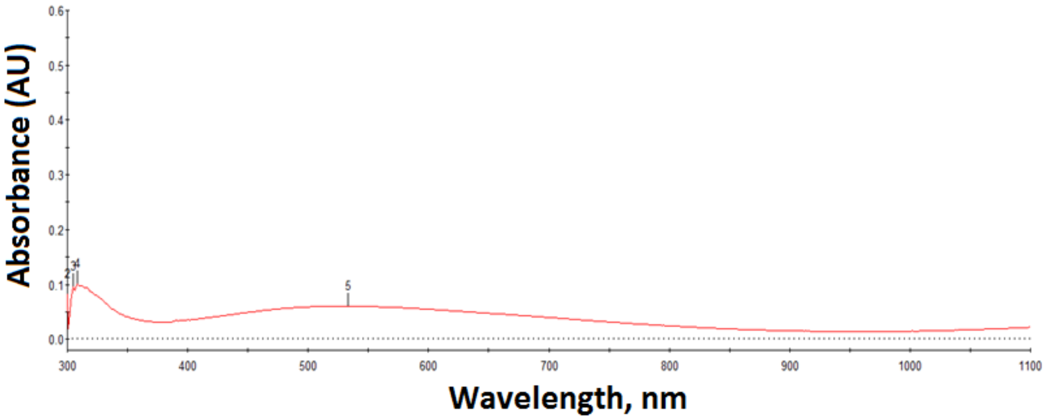

Figure 9.UV-visible absorption spectrumof UV- visible reflectance spectrum

Slika 9. UV-vidljivi apsorpcioni spektar UV-vidljivog spektra refleksije

The UV-Visible reflectance spectrum of the film (copper sulphide) formed on ITO glass plate by SILAR method is shown in Figure 10. Wavelength transition takes place at 680 nm. So the band gap of the film is E_g=1.240/0.680 = 1.823eV. This indicates that the film functions as semiconductor. The absorption band appears at 310nm.

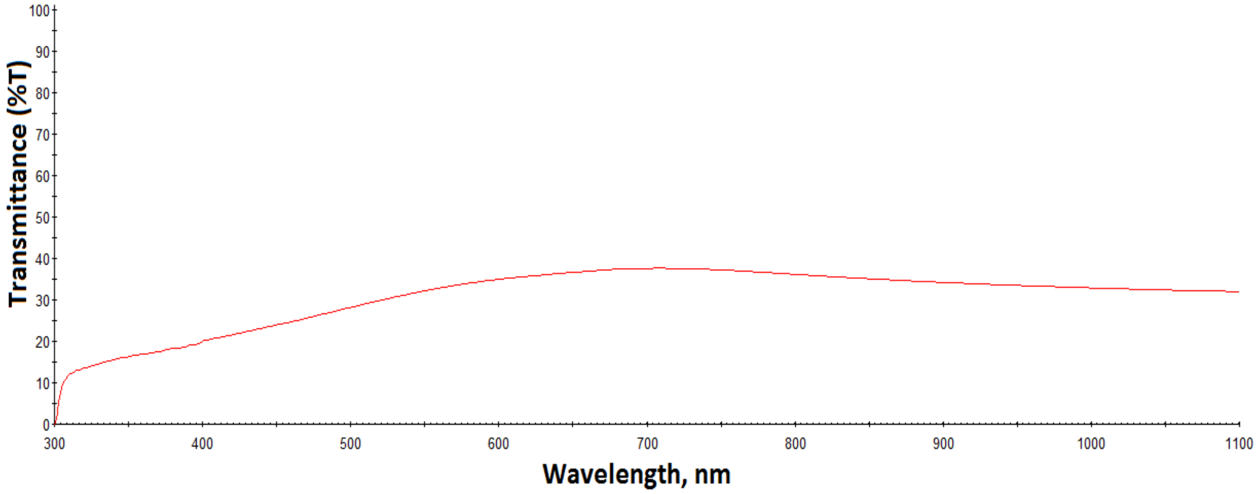

Figure 10. UV- visible reflectance spectrum of copper sulphide film formed onITO glass plate by SILAR method

Slika 10. UV-vidljivi spektar refleksije filma bakar sulfida formiranog na ITO staklenoj ploči SILAR metodom

The UV-Visible reflectance spectrum of ITO glass plate is shown in Figure 11. Wavelength transition takes place at 650 nm. So the band gap of the film is given E_g = 1.9061eV. This indicates that the film function as semiconductor. The absorption band appears at 300 nm.

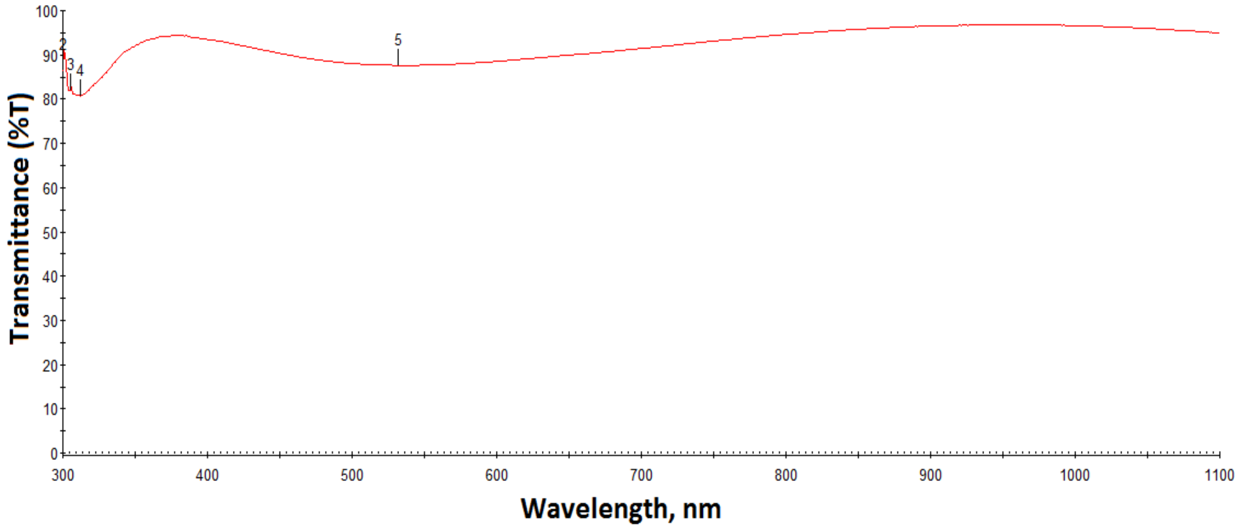

Figure 11.UV- visible reflectance spectrum of ITO glass plate

Slika 11. UV-vidljivi spektar refleksije ITO staklene ploče

It is interesting to note that after thin film coating, the band gap of ITO glass plate decre-ases. Hence electron flow will be easy. Current flowing through the system will be high.

Implication

The thin film of CuS deposited on ITO glass plate by SILAR method function as a semiconductor with a band gap of 1.823eV.

4. SUMMARY AND CONCLUSION

- A thin film of copper sulphide has been deposited on Indium Tin Oxide (ITO) glass plate.
- This film has been characterized by UV-Visible reflectance spectroscopy, FTIR spectroscopy, EDAX and SEM.
- The film has been subjected to electrochemical study, namely, polarization study.
- UV-Visible reflectance reveals that the band gap of the copper sulphide film is 1.823eV.
- This indicates that the film function as semi conductor.
- The UV- Visible absorption study of the film indicates the λmax appears at 310 nm.
- The FTIR study of the copper sulphide film confirms the presence of CuS.The polarization study reveals that the linear polarization resistance (LPR) value decreases, when the film is immersed in sea water.
- This indicates the current flowing through the thin film is increases. Such a finding can be used in solar cells. This is supported by the fact that the current flowing through the thin film, when it is immersed in sea water increases.
- When compared to the current flowing through the empty glass plate (without black coating) is immersed in sea water. This is further supported by the fact that for black thin film. The band gap decreases after coating 1.823eV.For ITO glass plate without coating the band gap Eg is 1.906eV.
- The EDAX study confirms the presence of elements Cu and S.
- The SEM study reveals the presence of thin film of copper sulphide on the ITO glass plate.
- Particle size of the copper sulphide is in the range of 101.1nm,107.8nm and114.5nm.
- Thus it is encouraging to note that copper sulphide nanoparticles have been prepared by SILAR method.

SCOPE FOR FURTHER STUDY

- CuS thin film has been deposited on ITO glass plate by SILAR method and the film has been characterized by various methods such as FTIR,SEM,EDAX,etc.
- Similarly CuS thin film can be prepared by chemical method and electrochemical method and it can be characterized.
- Instead of CuS thin films such as NiS, CoS, CdS, Prussian blue, etc.. can be deposited on ITO glass plate and then they can be characterized by the above mentioned methods.

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IZVOD

SINTEZA TANKOG FILMA BAKAR SULFIDA NA STAKLENOJ PLOČI OD INDIJUM-KALAJ OKSIDA METODOM SILAR I NJENA KARAKTERIZACIJA

Tanak film bakar-sulfida nanesen je na staklenu ploču indijum-kalaj oksida (ITO). Ovaj film je okarakterisan UV-vidljivom refleksivnom spektroskopijom, FTIR spektroskopijom, EDAKS i SEM. Film je podvrgnut proučavanju polarizacije uranjanjem u morsku vodu. Gore navedene studije o tankom filmu su upoređene sa bakar-sulfidom pripremljenim hemijskom metodom; odnosno mešanjem rastvora bakar-sulfata i rastvora natrijum-sulfida. Za poređenje korišćene su metode istraživanja kao što su UV-vidljiva refleksija i FTIR. Spektar UV-vidljive refleksije otkriva da je deo u okviru bakar-sulfidnog filma 1,823eV. Ovo ukazuje da film funkcioniše kao poluprovodnik. Studija UV-vidljive apsorpcije filma pokazuje da se λ max pojavljuje na 310 nm. FTIR studija filma bakar-sulfida potvrđuje prisustvo CuS. Studija polarizacije otkriva da se vrednost linearnog polarizacionog otpora (LPR) smanjuje u poređenju sa ITO pločom uronjenom u morsku vodu. Ovo ukazuje da se struja koja teče kroz tanki film povećava. Takav nalaz se može koristiti u solarnim ćelijama. Tome u prilog govori i činjenica da se struja koja teče kroz tanki film, kada je uronjen u morsku vodu, povećava, u poređenju sa strujom koja teče kroz praznu staklenu ploču (bez crnog premaza) uronjena u morsku vodu. Ovo je dodatno potkrepljeno činjenicom da se za crni tanki film, deo u pojasu smanjuje nakon nanošenja premaza. EDAKS studija potvrđuje prisustvo elemenata Cu i S. SEM studija otkriva prisustvo tankog filma bakar-sulfida na ITO staklenoj ploči. Veličina čestica bakarnog sulfida je u opsegu od 101,1 nm, 107,8 nm i 114,5 nm. Stoga je ohrabrujuće primetiti da su nanočestice bakar sulfida pripremljene SILAR metodom.

Ključne reči: *Sinteza tankog filma, bakar sulfid, SILAR metoda, karakterizacija, FTIR, EDAKS.*

Naučni rad

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