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MEASURING THE EFFECT OF VARYING MICROWAVE POWER ON AGO NANO THIN FILM

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Abstract

In this work, pure silver Ag (99.99 %) thin film deposited on glass substrate using radio frequency (RF) sputtering technique and exposed to microwave assisted oxygen plasma produced by microwave plasma Chemical Vapour Deposition (CVD). In this process Ag gets oxidized into AgO thin film. This AgO thin film was studied using varying microwave powers such as 300 W600 W900 W. The effect of varying microwave powers on morphology and size of oxide film was investigated. The, morphology, crystal size, crystal structure chemical composition, and optical properties of AgO thin film were studied and characterized using X-ray diffraction (XRD), field emission scanning electron microscopy (FESEM) and Raman spectroscopy. Morphology of these films exhibits a systematic change from metallic silver (Ag) to silver oxide (AgO). The size of AgO thin film was calculated using Scherrer equation and was observed to be 10 nm, 11.5 nm and 13.5 nm at 300 W, 600 W and 900 W respectively.
Keywords: AgO; Thin Film; RF; CVD ; oxygen plasma

1. Introduction

Silver Oxide (AgO) thin film has wide applications in fabrication of nano particles, battery technology and chemical technology etc. The thin films of metal-oxide are found to be highly promising in electronic, biothechnology and paints industry which require a uniform size nanoparticles distribution. There are various methods and techniques for the synthesis of thin film such as Chemical Vapour Deposition (CVD) Physical Vapour Deposition (PVD), and Electro-Chemical Deposition (ECD) [1]. The microwave-assisted method is energy-saving, rapid and promising route for the synthesis of nanomaterials. Microwaves have proven its role particularly in the synthesis of metal and metal oxide nano particles [2,3]. The microwave is an electro-magnetic wave having e frequency range 300 MHz - 300 GHz and the wavelength 1 m to 1 mm in electromagnetic spectrum. It has been analysed that the synthesis with microwave enhances structural, morphological, and mechanical properties [4]. With he interaction of microwaves with the material in microwave oven, the heat is generated within the sample itself while in conventional heating , the heat is generated by heating elements and then transferred to sample surface by the process of conduction or convection or both. Thus the heat produced by the method of interaction of microwave with materials is energy efficient and eco-friendly [5]. Under the effect of electromagnetic field, the particles of materials undergo polarisation such as atom polarization, electron polarisation, , orientation polarisation and space charge polarisation. The microwave-assisted method appreciably shortens the preparation time in a closed reaction system [6].

Silver oxide is an intrinsic p-type semiconductor with a band gap of 1.46 eV, which makes it as potential material for silver-oxide batteries, photo catalysis, gas sensing and antibacterial applications [7-9]. They can exhibit the growth of various micro-organisms such as yeasts, virus, bacteria and moulds [10-12]. This silver oxides in its different states like AgO, Ag_2O and Ag_2O_2 has potential applications in electrochemical capacitor (EC) due to of its high conductive nature as compared to other transition metal oxides. Thus having applications in fabricating electrochemical capacitors, silver oxide behaves as a strong candidate in the field of electrode materials. The nano structured silver oxides are used in the development of high voltage electrochemical super capacitors and thus optimizing their energy storage and power capability [13-15].

2. Experimental

Pure Ag (99.99%) thin films were deposited on glass substrate using Radio Frequency (Rf) sputtering technique. Before deposition the glass substrates were cleaned using acetone and deionised water. Then these glass substrates were dried under nitrogen gas. Now those deposited Ag thin films on glass substrates were oxidised by using microwave based plasma deposition unit (HPMS-2020 microwave plasma CVD equipment) connected with (HMG-2020S) 2KW microwave power source [16].The pure Ag thin films on glass substrate were placed onto the substrate holder inside the chamber. The chamber roughing of 1 Pa (base vacuum) by rotary pump and the working vacuum of 4 kPa inside the chamber by diffusion pump was created. Oxygen gas was purged at the flow rate of 70 sccm and working vacuum was maintained with the help of needle valve. Different microwave power of 300 W,600 W and 900 W were applied to generate the oxygen plasma. Each sample was exposed for about 20 minutes in this plasma for oxidation process at different microwave power. Oxygen plasma reacts with pure Ag thin films on glass substrate and resulting in the formation of silver oxide (AgO). Oxidation of Ag to AgO is dependent of the annealing temperature. Thus, the microwave power 300 W, 600 W and 900 W were chosen that generates temperature up to 320°C, 390°C and 460°C respectively to ensure complete oxidation of the films.

3. Result And Discussion

XRD analysis of pur Ag and AgO at varying microwave power (300 W, 600 W and 900 W) was carried was carried out by A Regaku Ultima IV X-ray Diffractometre. The pure Ag thin film having high intensity x-ray diffraction peak at angle 37.29^0 and one weak peak at 59.61^0 suggest a preferred orientation of the crystallites along the (111) and (220) direction [17-19]. These diffraction peaks (111) and (220) are related to the cubic structure of silver. It also shows that the grown Ag film on the substrate were of polycrystalline in nature. The average crystalline size was calculated and found to be ~ 7.5 nm by using Scherrer equation.

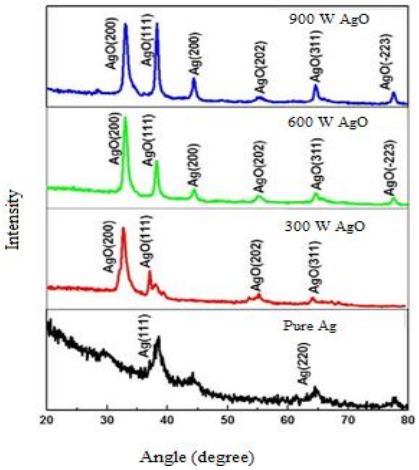

Fig. 1. XRD pattern of pure Ag and AgO thin films on glass substrate with varying microwave power 300W,600Wand1,900W

The oxidization of pure Ag film into AgO with at 300W shows intensified XRD peak at 29.37 and three weak peaks at 35.41, 49.23,59.51 .The crystallite size at intensed peak found to be ~09 nm. These diffraction peaks (200) (111) (202) and (311) are related to the monoclinic structure of AgO This shows that the pure Ag thin film turned into AgO thin film after oxidation and was polycrystalline in nature [20].

At 600W the intensity peaks of AgO (200), (202), (111) and (311) increase with diffraction 29.23, 37.03, 49.39, 59.71. The average crystalline size found at about 11 nm. At 900W At 600W the intensity peaks of AgO (200), (202), (111) and (311) increase with diffraction 31.43, 35.71, 49.39, 61.81. The average crystalline size found at about 12.9 nm. This reflects significant increase in diffraction and crystalline size as wll.

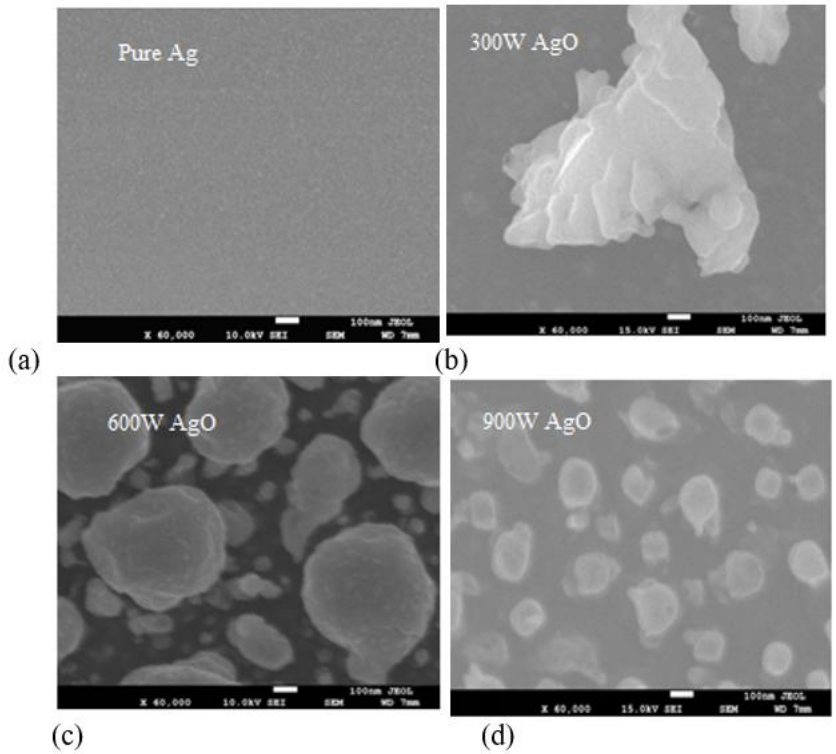

Fig. 2. SEM images of pure Ag and AgO thin films on glass substrate with varying microwave power 300W,600Wand1,900W

The SEM images are shown shown in Fig.2 . In pure Ag thin film, uniform morphology is observed whereas a significant changes are observed for AgO thin films at 300W, 600W and 900W. The significant changes in shape and size of the AgO results in the increase of microwave power, which is directly related to the annealing temperature. Therefore, with the increase in temperature, morphology of the thin films changes which results in smaller and uniform dispersion of the AgO.

In general the phenomena at nano-scale is relatively opposite to that normal metal as noble metals are chemically inert under normal conditions which results in relatively stable and inactive towards oxidation [21]. At nan-oscale chemically inert metals interacts and begin reacting due to the increase in surface to volume ratio and hence oxidized immediately.

The phenomena at nanoscale is relatively opposite as noble metals are chemically inert under ambient conditions which mean they are relatively stable and inactive towards oxidation [22-25]. At nanoscale chemically inert metals start interacting and reacting because of the increase in surface to volume ratio and thus oxidises immediately [26-29]. The oxidation at the surface depends mainly on oxidation rate which varies with the temperature. The controlled oxidation affects upto a few nm on the surface of Ag thin film by annealing below 300°C, which is in accordance with optical absorption results. Raman modes exhibits at 217, 290, 340, 395, 415 and 455 cm^{-1} in bulk silver oxide (AgO) and silver (I) oxide (Ag_2O) exhibits a single Raman mode at 470 cm^{-1}. Raman modes exhibits at 225 and 229 cm^{-1} of a thin film of 230 nm thickness, synthesised by thermal evaporation followed by annealing at above 300°C .Figure 3 represents Raman spectra of AgO thin films at 300 W, 600 W and 9000 W which is correspond to 290°C, 320°C and 405°C respectively[30-33].

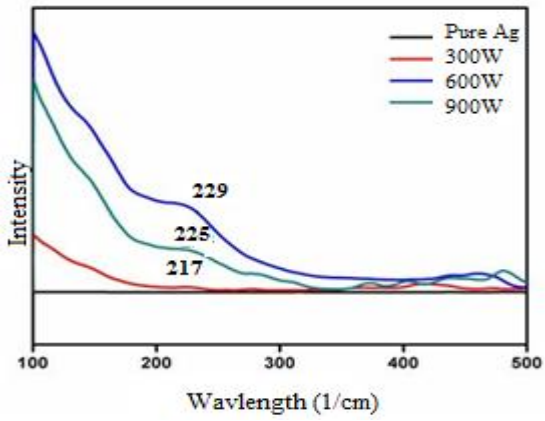

Fig. 3. Raman Spectra of pure Ag and AgO thin films on glass substrate with varying microwave power 300W,600W and1, 900W

4. Conclusion

The change in morphology of the silver oxide thin films with varying microwave powers was observed. The nano films of AgO grown on glass substrate using Radiofrequency (RF) sputtering was measured to be 8.3 nm thick.The size of nano particles increases with increasing annealing temperature being virtue of potential increasing concentration of the nano particles on the glass substrate which results in increased thin film thickness. XRD patterns confirm the formation of AgO structure. The Raman spectra shows the oxidation of AgO thin films. The SEM images shows that at 300 W the silver oxide forms leaf like or connected bone like structures of 9 nm in size is at 600 W, cauliflower like structure and size is found to be about 10.5 nm and at 900W, oval shaped like structure are formed with the increase in the size of the particles, i.e., 12.9 nm.

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