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Influence of Bath pH values on the Structural and Optical Properties of Electrodeposited MgO Thin Films for Optoelectronic applications

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Abstract

This paper investigates the influence of bath pH values on the structural and optical properties of magnesium oxide (MgO) thin films synthesized using electrodeposition technique. The films were deposited on conductive fluorine tin oxide (FTO) substrates using magnesium nitrate hexahydrate as a precursor material. The structural, morphological and optical properties of the electrodeposited films were examined by scanning electron microscopy (SEM), X-ray diffraction and UV-Vis spectrophotometer. The morphology and optical properties of the films were found to vary with bath pH values. The band gap decreased as the bath pH values increased. The deposited MgO films exhibited average transmittance of 80%, 50%, and 25% with thicknesses 400 nm, 480 nm, and 540 nm for bath pH values of 2.0, 5.0, and 9.0, respectively. The results obtained indicate that bath pH values play significant role in the formation of MgO films and can be used to tune the material into useful optoelectronic applications.

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1. Introduction

Magnesium oxide (MgO) has received considerable attention due to their unique properties such as chemical inertness, electrical insulation, optical transparency, high temperature stability, high thermal conductivity, high dielectric constant and secondary electron emission [1, 2, 3]. These excellent characteristics make MgO nanomaterial a promising material for several industrial applications. Magnesium oxide exhibits the cubic crystalline structure and has interesting properties because of its thermal and chemical stability properties [4]. It is non-toxic and has been employed as a layer material for the fabrication of thin film transistors (TFTs) for next-generation of electronic devices owing to its large dielectric constant (9.8)[5, 6, 7, 8].

Magnesium oxide materials are the best candidates for dielectric layer applications because of its high dielectric constant, large band gap energy, and breakdown voltage (12 mV/cm) when compared to the commonly used silicon dioxide (SiO_2) [9]. Magnesium oxide possess a direct large band gap energy in the range of 3.5-5.7eV, large exciton binding energy

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(80mev) [10], and high transmission value above 80% [11]. MgO thin films have been utilized as buffer layer for superconducting and ferroelectric thin film fabrications; however, its wide band gap, low optical loss and relatively low refractive index have reported improved the optical modes in many ferroelectric materials [12]. Moreover, due to their biocompatibility, MgO nano-materials find several bio-applications in the areas of antibacterial and anticancer agents, biosensors, reactive oxygen species, and bone regeneration [13, 14, 15]. Magnesium oxide films continue to find numerous applications in various technologies such as reflecting and anti-reflecting coatings [16, 17], light emitting diodes (LEDs) [18], solar cells [19], laser diodes [20] and even as a protective layer to shield electrodes from ionizing radiations especially in secondary electron emission by minimizing the discharge voltage layer in AC plasma display panels. Magnesium oxide (MgO) exists in many nanostructures such as nanotube, nanocrystals, nanowires, nanoparticles, nanofloweres, and even broken-floor like structures [21, 22]. These nanostructures are heavily influenced by the type of deposition technique.

The MgO thin films have been synthesized by various techniques such as pulsed laser deposition [23], chemical vapour deposition, CVD [24], RF magnetron sputtering [25], sol-gel synthesis [26], spray pyrolysis [27], and electrodepostion [28]-[31]. Among these deposition techniques, electrodeposition has become the most fascinating deposition techniques due to the controllable morphological and structural growth of nanomaterial with optimization of deposition parameters; the technique also allows the fabrication of thin films with high surface area to volume ratio, it is simple, does not required any sophisticated equipment and films can be deposited at low temperatures [32]-[39]. Generally, bath conditions have been noted to play vital role in the optimum deposition of thin films. In particular, less attention is usually placed on the bath pH values which to a large extent enhance transport of charges within the medium of deposition. In addition, the knowledge of the variation of properties of MgO with preparation conditions would assist in the fabrication process for optimum deposition, characterization and application.

In this paper we electrodeposited MgO thin films at different bath pH values and investigated the effect of the bath pH values (2, 5 and 9) on the morphological, structural and optical properties of MgO thin films.

2. Materials and Method

The materials used in this work are magnesium nitrate hexahydrate $Mg(NO_3)_2.6H_2O$ of purity 99% which was purchased from SIGMA-ALDRICH, Sodium hydroxide pellets (NaOH) and hydrochloric acid (HCL) purity over 98% were purchased from Thomas Baker, were used to adjust the bath pH values; deionized water was used as the solvent throughout the experiments. All chemicals and salts were analytical reagent grade and were used as received without further purifications. FTO coated glass substrates of sheet resistance 13-15 Ohm/square and dimension 20x40x2.2 mm were purchased from Brotain Hong Kong Co, Ltd, China. Before the electrodeposition, the FTO coated glass substrates were degreased and cleaned ultrasonically in acetone solution to remove any contaminants from its surface. The MgO thin films were potentiostatically electrodeposited onto well cleaned FTO coated glass substrates from a bath solution containing 40mM magnesium nitrate hexahydrate $Mg(NO_3)_2.6H_2O$. The bath solution of 40mM was prepared by dissolving 2.6 g of $Mg(NO_3)_2.6H_2O$ in 250 ml of deionized water in 300 ml beaker, the solution was stirred magnetically for 2hr before deposition. The bath pH values was adjusted to 2.0, 5.0, and 9.0 using NaOH or/and HCL.

A Metrohm AUTOLAB PGSTAT302N of three electrodes system configuration comprising FTO, platinum plate wires and Ag/AgCl as working (WE), counter (CE) and reference (RE) respectively was employed to perform the electrodeposition. The MgO thin films were deposited at cathodic potential of -1.2 V, temperature 80°C in 120 minutes, without stirring the solution during deposition. Immediately after electrodeposition, the MgO films were rinsed in deionized water to remove loosely bound particles. The electrodeposited MgO films were annealed at 500° for 1hr and allowed to cool down gradually in the annealing chamber before characterization. The annealed MgO films were characterized for morphological, structural and optical properties using Tescan Vega3 microscope at an accelerated voltage of 20kv, X-ray diffractometer D2 Phaser-e and Agilent Cary 60 UV-Vis spectrophotometer (Tshwane University of Technology, Pretoria, South Africa) respectively. The film's crystallite size of the films was calculated using the equation:

$$D = \frac{K\lambda}{\beta \cos\theta}, \tag{1}$$

where D is the crystallite size (in nm), K is a Scherrers constant 9 ($K = 0.9$), λ is the wavelength of X-ray radiation $\lambda = 1.5418$ Å, β is the full width at half maximum (FWHM), and θ is the Bragging angle. The thickness of the electrodeposited MgO films was determined using gravimetric method given as:

$$t = \frac{\Delta m}{\rho A}, \tag{2}$$

where Δm is the mass of deposited MgO thin film, the mass of the film was calculated as the difference between the bare FTO and electrodeposited MgO thin films, ρ is the density of bulk MgO, and A is the surface area of deposited film. The optical conductivity was calculated using the equation:

$$\sigma = \frac{\alpha n c}{4\pi}, \tag{3}$$

where α is the absorption coefficient, n is refractive index, c is the speed light. The band gap energy (E_g) is estimated from the Tauc plots of $(\alpha h\nu)^{1/n}$ versus photon energy ($h\nu$) by extrapolating the linear portion where $(\alpha h\nu)^n$ approaches zero on the horizontal axis in the equation:

$$\alpha h\nu = A\left(h\nu - E_g\right)^{1/n} \tag{4}$$

where A is a constant, the value of n is dependent on the type of electronic transition; it is $1/2$ for direct allowed transition, $n = 2$ for indirect allowed transition, $n = 3$ for direct forbidden transition, and $n = 3/2$ for indirect forbidden transition. Here the value of n is taken as $1/2$.

The refractive index (n) of the deposited ZnO-MgO was calculated using the equation:

$$n = \frac{(1 + \sqrt{R})}{(1 - \sqrt{R})}, \tag{5}$$

where R is the reflectance of the film. The optical conservation of energy is given as

$$T + R + A = 1, \tag{6}$$

where T = Transmittance, R = Reflectance, and A = Absorbance.

The voltammetry study was conducted on the $Mg(OH)_2.6H_2O$ at different bath pH values (2, 5 and 9) on a bare FTO to determine the suitable deposition potentials for the fabrication of MgO thin films . It was observed from the voltammogram (Figure 1) that the possible region of potentials lies within -1.0 to -1.5 V. Therefore, the MgO thin films in this study were deposited at -1.2 V being the potential of the optimum deposition for all the bath pH values considered.

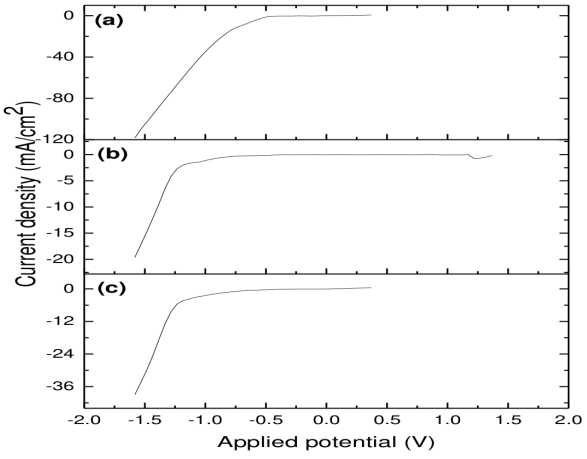

Figure 1: Cyclic voltammograms recorded on FTO coated glass substrate in a 40mM magnesium nitrate solution at various bath pH (a) 2.0, (b) 5.0 and (c) 9.0 at scan rate was of 20mV/s. The plots indicate a reverse (cathodic) scan.

3. Results and Discussion

3.1. Structural characterization of electrodeposited MgO Thin Films

Figure 2 shows the X-ray diffraction spectra of MgO thin films grown at different bath pH values 2.0, 5.0 and 9.0. The result revealed three basic peaks of MgO films which are localized at $2\theta = 42.87^o$, 62.1^o, and 78.34^o corresponding to (200), (220) and (222) respectively [40]. With increase in the bath pH values, the peaks became more intense, especially at bath pH value 9.0, and were found to shift towards the lower diffraction angles. The broadness of the peaks decreased which depicts improvement in the crystal quality of the films [1]. The films showed preferred orientation along (220) plane with high intensity. Furthermore, secondary phases relating to Mg $(OH)_2$ and FTO glass substrate were observed in the XRD spectra. Interestingly, aside these known phases no other peaks relating to impurities were found [8]. The presence of Mg $(OH)_2$ was localised at angle $(2\theta) = 37.89°$ and $51.49°$ which correspond to 101 and 102 planes. The presence of FTO substrate in the XRD spectra could be due to the thinness of the films deposited [18]. The calculated crystallite sizes are 22.18 nm, 21.11 nm, and 23.31 nm corresponding to thickness of 400 nm, 480 nm and 540 nm for MgO films deposited at bath pH values 2.0, 5.0 and 9.0, respectively.

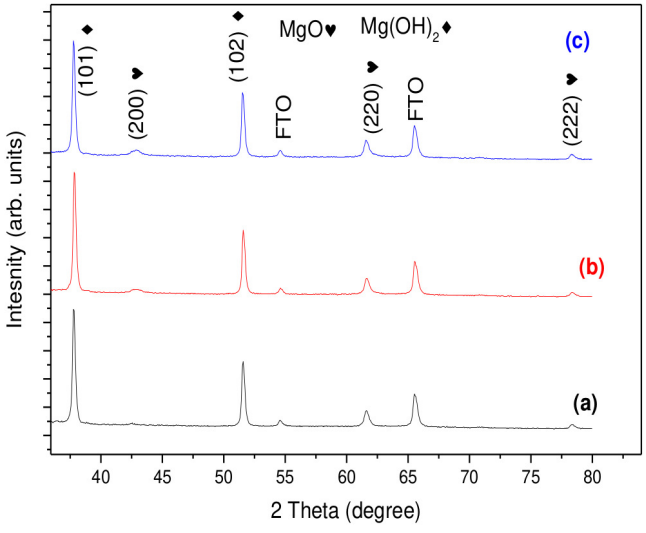

Figure 2: XRD diffraction patterns of the electrodeposited MgO thin films grown at bath pH (a) 2.0, (b) 5.0, and (c) 9.0

3.2. Morphology and energy-dispersive X-ray spectroscopy MgO Thin Films

Table 1: EDXS analysis of electrodeposited MgO thin films at different bath pH values

Sample	Weight percentage of the element (at. %)		
	O	Mg	Mg/O
pH: 2.0	55.9	28.2	0.50
pH: 5.0	56.2	24.8	0.44
pH: 9.0	55.9	29.5	0.52

Figure 3 (a-c) presents the SEM images of electrodeposited MgO thin films at different bath a pH values 2.0, 5.0 and 9.0. The films deposited at bath pH value of 2.0 revealed net-like structures with plenty of white dots (Figure 3a). As the bath pH value increased to 5.0, the morphology of film significantly

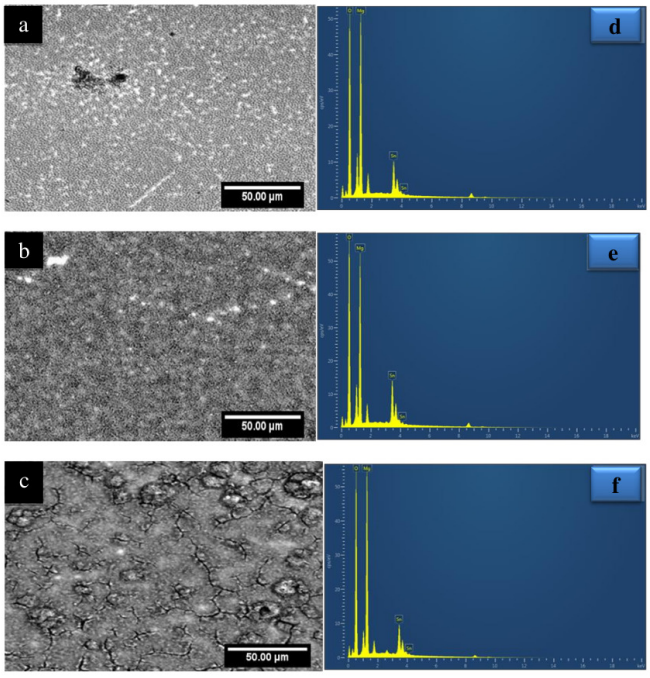

Figure 3: SEM with the energy-dispersive X-ray spectroscopy spectra of electrodeposited MgO thin films; films grown at various bath pH (a, d) 2.0, (b, e) 5.0 and (c, f) 9.0.

Table 2: Average results of absorbance, reflectance and transmittance of electrodeposited MgO at different bath pH values

Bath pH values	Absorbance (A) (arb. unit)	Reflectance (R) (arb. unit)	Transmittance (T) (arb.unit)	$A+R+T$
2.0	0.038	0.111	0.851	1
5.0	0.375	0.087	0.538	1
9.0	0.614	0.056	0.330	1

changed to sheet-like structures (Figure 3b). These sheet-like structures were agglomerated in high density and developed into orderly thick flower-like shaped structures. Furthermore, when the bath pH was raised to higher value of 9.0, the films drastically changed entirely into broken floor-like structures with plenty of heavy dark dots (Figure 3c). This result therefore demonstrates that the bath pH values can be used to tune the morphological properties of the MgO thin films. The pattern of this morphology had previously been reported [1, 3, 18]. The peaks of Mg and O elements in MgO films are displayed in energy dispersive spectra shown in Figure 3(d-f). For accuracy, the EDXS spectrum of each sample was recorded at two different locations on the MgO film surface and the average value of element concentration recorded as shown in Table 1. It was observed that the EDXS spectrum of MgO films consists of only Mg, O and peaks relating to the FTO substrate. This indicates that the electrodeposited MgO film is of high purity. Additionally, the quantitative EDXS data indicated that the (Mg/O at. %) ratio for all the samples has values less than one as seen in Table 1. The result therefore reveal inhomogeneous distribution of Mg and O atoms on the film surface with a deficiency of Mg atoms (that is high percentages of magnesium vacancies).

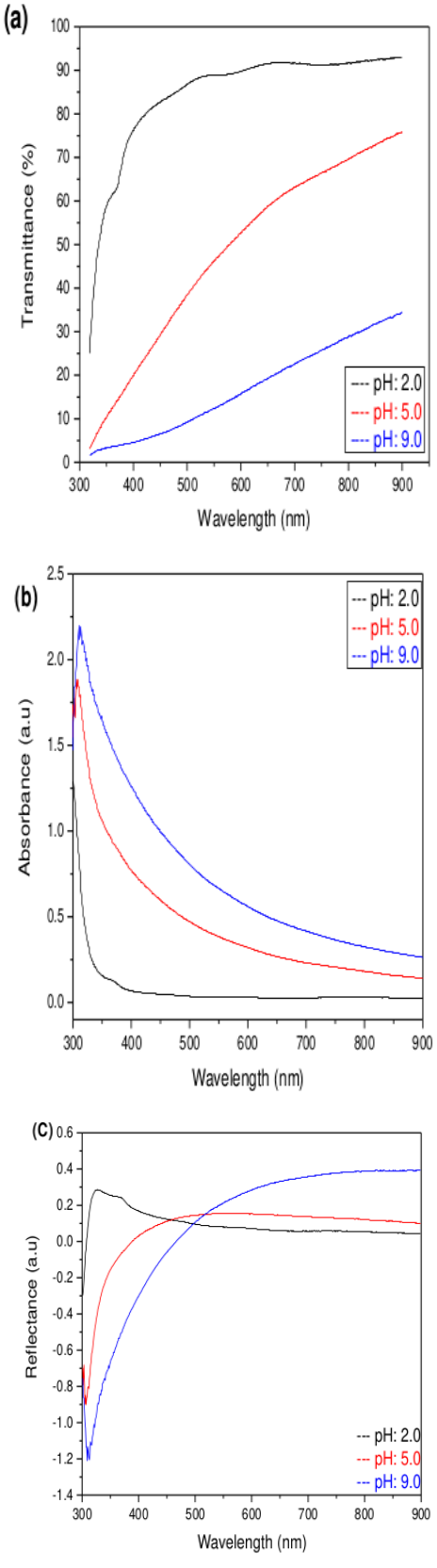

Figure 4: Transmittance (a), Absorbance (b), and Reflectance (c) versus wavelength of electrodeposited MgO thin films.

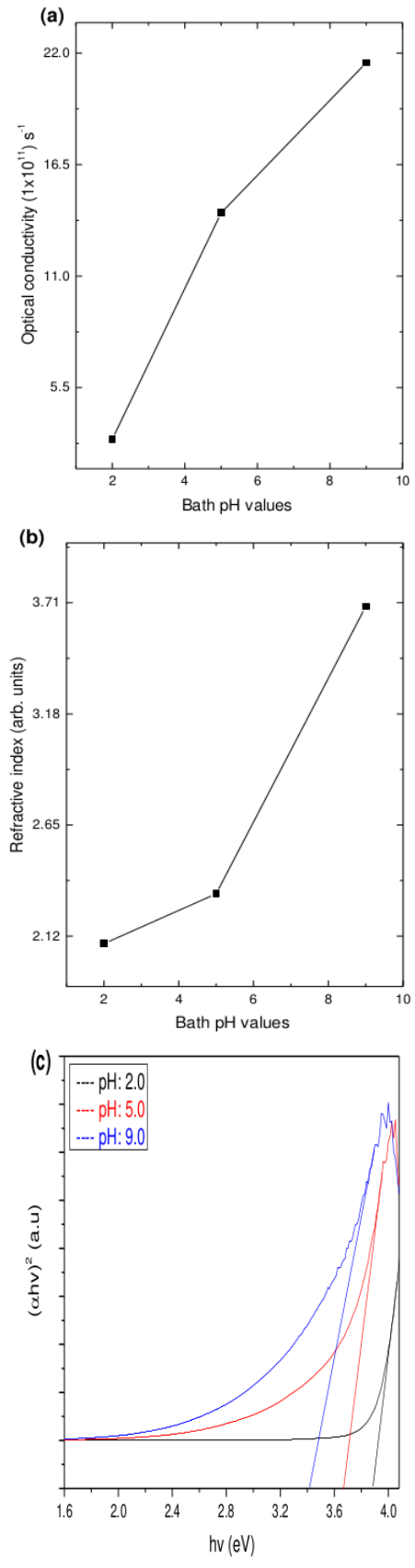

Figure 5: Plots of optical conductivity (a), refractive index (b) against bath ph values and (c) Energy band-gap estimation of MgO thin films.

3.3. Optical characterization of electrodeposited MgO Thin films

Table 2 shows the absorbance, reflectance and transmittance of the electrodeposited MgO thin films. The summation of these three basic optical properties agrees with the theoretical conservation of energy (i.e., unity) for all the bath pH values considered.

The optical properties were examined using UV-Vis spectrophotometry; the result showed that the absorbance increased with increase in bath pH values while the reflectance and transmittance decreased for all the bath pH values. Research has shown that high absorbance films are useful materials for absorber layers in solar cell device material applications [49]. Consequently, the thin films in the research can find useful applications as absorber layer in solar cell fabrication when deposited at high bath pH values.

The increase in optical absorption permits more charge carriers to be transited from the valence band into the conduction band, thereby enhancing the band gap energy of the materials (this was observed in the values of the band gap).

The plots of Transmittance, absorbance and reflectance against wavelength for MgO films at different bath pH values are shown in Figure 4. The transmittance spectra (Figure 4a) indicate that the film's transmittance values vary significantly with increase in the wavelength of radiation as the bath pH value increased. The MgO films deposited at bath pH value of 2.0 has transmittance value from 24% to 75% in the ultra violet (UV) region of the electromagnetic spectrum, increased from 75% to 91% in visible (Vis) region and maintains the same value in the near infra-red (NIR) region. As the bath pH value increased to 5.0, the films exhibited transmittance value from 3% to 19% in the UV region, 19% to 63% in Vis region, and 63% to 68% in NIR region. Furthermore, The MgO films deposited at 9.0 bath pH showed least optical transmittance in all the spectra regions i.e., the films had a transmittance value of 4% in UV region, 4% to 22% in Vis region, and 22% to 28% in NIR region.

These results in Table 2 showed that the transmittance values decreased with increase in bath pH values; this could be due to higher defect concentration occasioned by increased bath pH value [41]. However, it can be observed that the films maintained transmittance above 70 % in the Vis and NIR regions [18] suggesting that the films can find application as an absorbent of visible and NIR radiations making it fit as a protective coating to replace the extensive ITO [42].

The UV-Vis absorbance spectra of MgO film deposited at different bath pH values 2.0, 5.0 and 9.0 and annealed at 500^{o}C for 1hr is presented in Figure 4(b). The spectra were recorded within a wavelength range of 300-900 nm. The absorption spectra were classified into three regions depending on the amount of absorptions. In the highest absorption region

where $\lambda \leq 400$ nm the electron of the material absorbs higher energy from the incident photon to migrate into the conduction band, creating a hole (positive charge carrier) behind in the valence band thereby forming an exciton; a peculiarity of MgO material. At moderate absorption region, where 400 nm $\leq \lambda \geq$ 600 nm, the electron tends to cross over to shallow states below the conduction band. Finally the least absorption region occurs beyond 600 nm wavelength [18]. From the Figure 4(b), it obvious that the film deposited at bath pH value of 2.0 exhibited almost zero absorbance in the wavelength range 500-900 nm [43, 44, 45].
The UV-Vis reflectance spectra of MgO films deposited at different bath pH values is presented in Figure 4(c). The film showed high reflectance in UV region, while the films deposited at 5.0 and 9.0 bath pH values showed almost zero reflectance in the UV region. As the bath pH value increased to 9.0, the reflectance attained maximum value above 30% in the NIR region [46].

The plots of optical conductivity, refractive index against the bath pH values and band gap energy are shown in Figure 5 (a), (b) and (c) respectively. Figure 5(a) presents the optical conductivity of electrodeposited MgO films plotted against the bath pH values. It can be seen from this figure that the optical conductivity increased in great magnitude with increase in bath pH values. The increase in optical conductivity could be as a result of the increase in absorption coefficient of the films. The values of the optical conductivity calculated are 2.952 x10^{11}, 14.120 x10^{11} and 21.527 x10^{11} S^{-1} corresponding to bath pH values 2.0, 5.0 and 9.0 respectively. Figure 5(b) shows that the refractive index increased as the bath pH values increased. The calculated values of refractive index are 2.08, 2.32 and 3.69 for MgO thin films deposited at 2.0, 5.0 and 9.0 bath pH values respectively. Increase in refractive index with increase in deposition condition is in agreement with period research [47].

Figure 5(c) presents the graph of $(\alpha h\nu)^2$ versus photon energy ($h\nu$) for the MgO thin films. The band gap energy was estimated by extrapolating the linear region of the absorption curve to the point where $(\alpha h\nu)^2$ tends to zero [48, 49, 50, 51]. From the graph, the band gap values of the MgO thin films were calculated to be 3.88 eV, 3.67 eV, 3.42 eV for the films deposited at bath pH values 2.0, 5.0, and 9.0 respectively. It was observed that with increase in bath pH value, the band gap decreased gradually. The variation in the values of band gap of MgO nanomaterial could be attributed to recrystallization of atoms into the crystal lattice sites of the material [48] as the bath pH values increased. However, the remarkable band gap of 3.42 eV achieved at bath pH value 9.0 indicates that the films can serve as a suitable alternative to SiO_2 for device fabrication and utilization especially in capacitor applications [30].

4. Conclusion

MgO thin films have been successfully fabricated on FTO glass substrate by electrodeposition technique at different bath pH values. The results of the SEM, XRD, and optical properties showed good agreement with previous reports. The bath pH values contributed significantly to the growth, structural, morphology and optical properties of the electrodeposited MgO thin films. The thickness of the film increased from 400 to 540 nm as the bath pH values increased from 2 to 9. The morphology of the films transited from flower-like structure to a broken floor-like shaped structure with increase in the bath pH. The film deposited at bath pH value 9.0 was found to exhibit better morphological, structural, and optical properties. The band gap energy was found to decrease from 3.67 to 3.42 eV while the optical transmittance values decreased from 85% to 33%, and the optical conductivity increased significantly as the bath pH value increased from 2.0 to 9.0 respectively. The improvement in the optical properties with respect to bath pH values indicates that the electrodeposited MgO films can be employed as a suitable material for solar cells and protective layer applications.

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