Original Research

Structural Evolution and Optical Properties of Cobalt Oxide–Chromium Oxide Thin Films as a Function of Annealing Time

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Abstract: Cobalt–chromium oxide thin films were deposited on glass substrates using the spray pyrolysis technique and subsequently annealed at 773 K for 60, 90, and 120 minutes. The influence of annealing time on the structural, morphological, and optical properties of the films was systematically investigated. X-ray diffraction analysis confirmed the formation of single-phase cubic spinel cobalt oxide (Co_3O_4) with Fd-3m symmetry. The crystallinity of the films improved with increasing annealing time, as indicated by enhanced peak intensity and reduced full width at half maximum. The crystallite size increased from 41.82 nm to 48.66 nm, accompanied by a decrease in microstrain and dislocation density, suggesting reduced structural defects. The absence of secondary chromium oxide phases indicates the possible incorporation of Cr^{3+} ions into the spinel lattice. Atomic force microscopy revealed grain growth and improved surface uniformity with increasing annealing time, consistent with XRD results. Optical analysis showed that the band gap varied from 2.0 eV (as-deposited) to 2.6 eV at 90 minutes, followed by a decrease to 2.2 eV at 120 minutes, attributed to defect reduction and structural evolution. These results demonstrate that annealing time plays a significant role in tailoring the physical properties of cobalt–chromium oxide thin films. The improvements in structural properties, defect reduction, and surface morphology contributed to enhanced material performance.

Keywords: Cobalt–chromium oxide; Co_3O_4 spinel; Spray pyrolysis; Thermal treatment; Annealing time; Thin films.

1. Introduction

Semiconducting transition metal oxide materials have been extensively explored owing to their variable electronic structure, chemical stability, and strong chemical interactions with gaseous molecules[1, 2]. As a result, these materials have been recognized as highly promising candidates for gas sensing, catalysis, and optoelectronics[3, 4]. Among the transition metal oxide materials, cobalt oxide (Co_3O_4) has emerged as one of the most prominent candidates owing to its cubic spinel structure, mixed valency (Co^{2+}/Co^{3+}), and high surface reactivity[5, 6]. In addition, the p-type conductivity of Co_3O_4 is highly desirable for charge transport, as it offers high flexibility in modulating the adsorption/desorption process on the surface[7]. These processes play a vital role in the functional performance of gas sensing materials[8, 9]. The functional performance of Co_3O_4 thin films is highly dependent on their microstructural features, including crystallite size, defect density, lattice strain, and surface morphology[10]. Although defects play a crucial role in adsorption/desorption processes, excessive defects in the lattice structure might be detrimental[11]. Therefore, it is highly desirable to achieve the optimal microstructural features in the material for enhanced performance[12, 13]. A highly promising method of modifying the microstructural features of transition metal oxide materials is by incorporating trivalent chromium (Cr^{3+}) into the lattice structure[14, 15]. This is highly desirable owing to the similar ionic radius and valency of Cr^{3+} and Co_3+, which allows Cr^{3+} to occupy octahedral sites in the lattice structure without disrupting the lattice[16, 17]. The impact of chromium incorporation on the structural evolution of Co_3O_4 thin films following different durations of thermal treatment is not well explored[18]. Spray pyrolysis is a widely used technique for thin film deposition, offering many benefits such as cost-effectiveness and the ability to produce homogeneous and compositionally controlled thin films[19, 20]. Despite the above, the structural quality of thin films prepared by spray pyrolysis is often found to be limited, and structural disorders are frequently observed[21]. To improve the structural quality, it is often necessary to perform annealing treatment[7, 22, 23]. Although many works have explored the impact of annealing temperature on the structural quality of Co_3O_4 thin films, few works have explored the impact of annealing time, which is an independent variable and can be controlled during annealing treatment[24-26]. Understanding the impact of annealing time on the structural quality, crystallographic improvement, and optical properties, including the impact of chromium incorporation, is crucial for optimizing the structural quality and electronic properties of cobalt-chromium oxide thin films[4, 27, 28], particularly for applications where surface-controlled phenomena are dominant. In this work, the impact of annealing time at 773 K on the structural, morphological, and optical properties of spray pyrolysis-derived cobalt-chromium oxide thin films is systematically explored. The rest of this paper is organized as follows: Section 2 describes the experimental procedure; Section 3 presents and discusses the structural, morphological, and optical results; and Section 4 concludes the study.

2. Experimental

Cobalt chromium oxide thin films were deposited on glass substrates using the spray pyrolysis method. The chemical precursors used were $Co(NO_3)_2{\cdot}6H_2O$ (cobalt nitrate hexahydrate; molecular weight: 291.03 g/mol; purity: 97.5%, supplied by Oxford Lab Chemical), and the second compound was $Cr(NO_3)3{\cdot}9H_2O$ (chromium nitrate nonahydrate; molecular weight: 400.15 g/mol; purity: 98%, supplied by Loba Chemie). The reagents were used as received without further purification. Distilled water was used as the solvent for the solution preparation. Solutions of Co and Cr nitrates were prepared separately in distilled water at a molarity of 0.08 M each and then mixed in the ratio 85:15 to obtain the final solution for the film deposition. The solution was stirred magnetically for 30 minutes at room temperature. For the film deposition, the glass substrates were cleaned carefully by ultrasonic treatment in deionized water and ethanol to remove any impurities from the surface. After the ultrasonic treatment, the glass substrates were dried and then subjected to a temperature of 400 °C for the film adhesion during the spraying. The spraying was performed with a nozzle-to-substrate height of 24 cm. The spraying time was 3 seconds, and a total of ten cycles were performed. The intermittent spraying was used for the film formation[29]. After the spraying, the films were subjected to the annealing treatment at 500 °C for 60, 90 and 120 minutes in air atmosphere using a programmable furnace (KSL-1100X). The temperature was increased at a rate of 10 °C/min. After the annealing treatment, the power was turned off, and the samples were allowed to cool naturally inside the furnace overnight. A schematic illustration of the spray pyrolysis process used for the preparation of cobalt–chromium oxide thin films is presented in Figure 1. It highlights the precursor preparation, spraying conditions, and annealing treatment, along with the resulting film structure on the glass substrate.

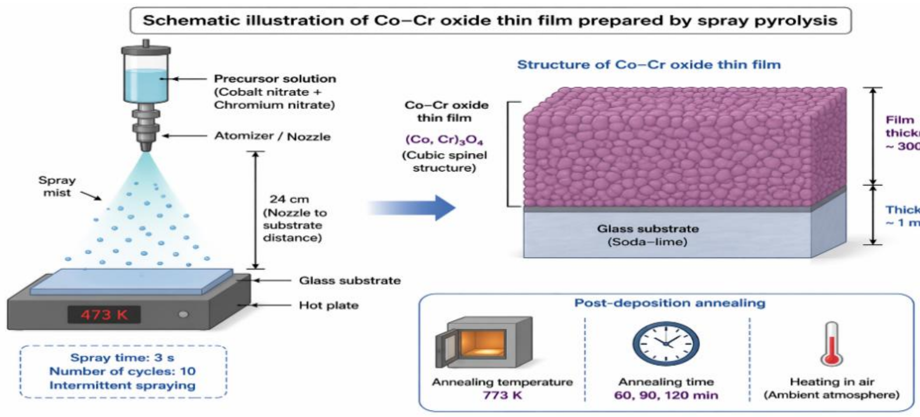

Figure 1. Schematic representation of the spray pyrolysis process used for the deposition of cobalt–chromium oxide thin films, including precursor preparation, spraying parameters (nozzle-to-substrate distance, spray time, and cycles), and post-deposition annealing conditions, along with the resulting film structure on the glass substrate.

3. Results and discussion

3.1. Structural properties

Figure 2 illustrates the structural properties of the spray pyrolyzed Co-Cr oxide thin films as observed from the X-ray diffraction patterns within the 2-theta range of 20 to 80 degrees [30]. The X-ray diffraction patterns for the as-deposited film and the films subjected to thermal annealing at 773 K for 1 h, 1.5 h, and 2 h demonstrate significant structural evolution with increasing annealing time. The as-deposited film exhibits weak and poorly resolved peaks, indicating low crystallinity and the presence of amorphous regions. In contrast, annealed films show well-defined diffraction peaks, confirming improved crystallinity. The major peaks at 2-theta values of 31°, 36°, and 65° correspond to the (220), (311), and (440) planes of cubic Co_3O_4, respectively. These peaks are consistent with the standard JCPDS card No. 00-042-1467, confirming a single-phase cubic structure with space group Fd-3m (No. 227). No secondary phases related to chromium oxide are detected within the resolution limit of the instrument. With increasing annealing time, peak intensity increases while the full width at half maximum (FWHM) decreases, indicating enhanced crystallinity. The crystallite size, calculated using the Scherrer equation, increases from 41.82 nm (1 h) to 46.56 nm (1.5 h) and reaches 48.66 nm after 2 h, which can be attributed to enhanced atomic diffusion and grain coalescence during annealing. Meanwhile, microstrain decreases with annealing time due to the relaxation of lattice distortions, accompanied by a reduction in dislocation density ($\delta = 1/D^2$), which decreases from 5.72×10^{14} m^{-2} to 4.22×10^{14} m^{-2}. This behavior further confirms the improvement in structural quality. Although chromium is used in the synthesis process, no distinct peaks corresponding to Cr_2O_3 are observed. This suggests that Cr^{3+} ions may be incorporated into the Co_3O_4 lattice by substituting Co^{3+} ions or remain below the detection limit. Due to the similarity in ionic radius and valency between Cr^{3+} and Co^{3+}, such substitution is plausible and may introduce slight lattice distortions without altering the cubic structure. Although complementary techniques such as XPS or EDS were not available to directly confirm chromium incorporation, indirect evidence is supported by the observed peak shifts, lattice parameter variations, and changes in optical properties, suggesting successful incorporation of chromium into the cobalt oxide structure.

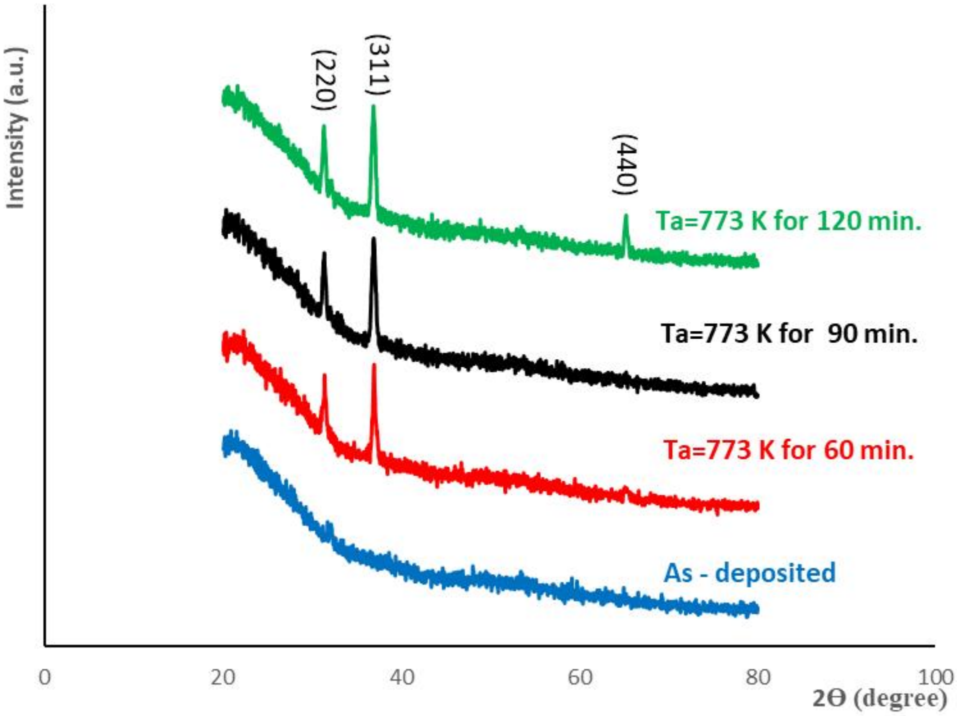

Figure 2. Structural and phase analysis by X-ray diffraction.

Table 1. XRD-derived structural parameters of the dominant peaks.

Annealing Time (hour)	2Θ(deg)	FWHM (deg)	d (Å)	Planes (hkl)	C.S (nm)	δ*10^14 (lines/m^2)	με
1	31.422	0.206	2.8445	220	41.83	5.72	0.865
	36.914	0.179	2.4329	311	48.85	4.19	0.741
1.5	31.386	0.185	2.8476	220	46.57	4.61	0.777
	36.891	0.171	2.4344	311	51.13	3.83	0.708
2	31.317	0.177	2.8538	220	48.67	4.22	0.744
	36.773	0.168	2.4419	311	52.03	3.69	0.696
	65.334	0.191	1.4270	440	51.58	3.76	0.702

Figure 3 In order to examine the surface morphology of spray pyrolyzed cobalt chromium oxide thin films, atomic force microscopy was used, with the aim of determining the effect of heat treatment time on the surface characteristics of these thin films[31]. As can be seen from Figure 3, it is clear that the surface morphology of the as-deposited thin film is relatively fine grained, with randomly arranged nanometric features on its surface. The grain boundaries are less clear, which is an indication of low crystallinity, as was also indicated in XRD analysis results. When subjected to heat treatment for 60 minutes at 773 K, it is clear that there is an observable change in the morphology of the thin film, with grains being more distinguishable and having started to aggregate, thus increasing their size. This is also an indication that heat treatment enhances the size of the grains in thin films, as was also indicated in XRD results. Further increasing heat treatment time to 90 minutes resulted in an even more uniform thin film surface, with grains continuing to grow in size. Grains appear more compact and clearly defined, indicating that there is an improvement in structural ordering. The reduction in structural defects, as indicated by XRD results, is also an indication that the thin film has become more homogeneous in terms of surface morphology. Notably, after 120 minutes of annealing, more pronounced grain enlargement and even possible agglomeration of neighboring grains are observed. It can thus be inferred that this prolonged annealing may lead to grain coarsening, resulting in increased surface roughness. As observed, the increase in grain sizes with prolonged annealing times corresponds to the

continuous increase in crystallite sizes observed from the XRD results. The changes observed in surface roughness parameters are directly related to changes in film structure, which are attributed to annealing. Grain growth and defect healing affect the surface energy, thus affecting adsorption sites. Therefore, from AFM results, it can be concluded that annealing time significantly affects film surface morphology. Grain growth and surface changes are consistent with XRD results and play a crucial role in film electronic transport and gas-sensing properties.

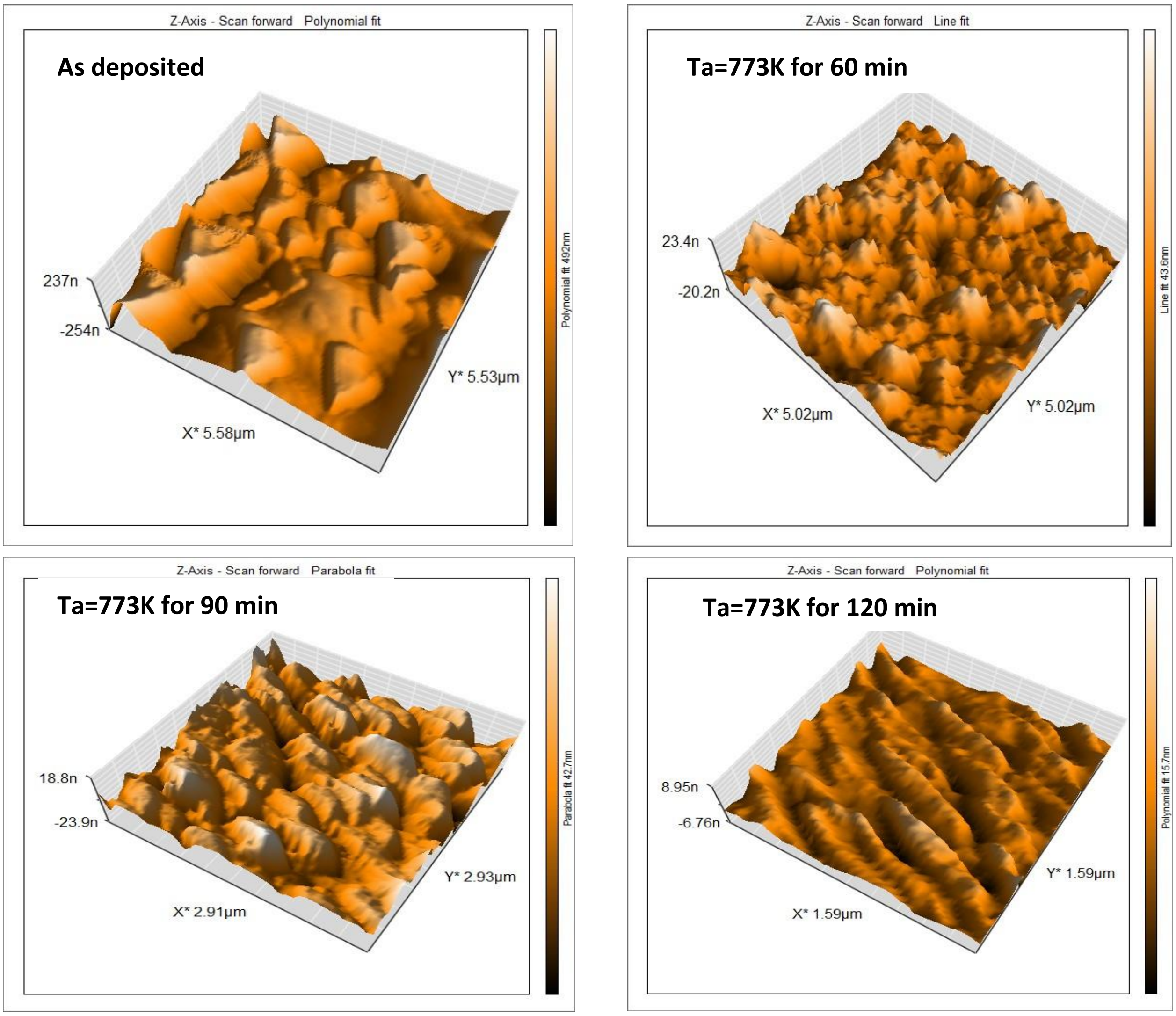

Figure 3. The AFM images in all the films were produced in three dimensions.

Table 2. AFM data for every films was produced.

Sample	Size of grain (nm)	Roughness. (nm)	Root mean square. (nm)
As deposited	**91.42**	**90.23**	**94.37**
Ta=773K for 60 min	**67.54**	**108.0**	**129.5**
Ta=773K for 90 min	**86.31**	**119.1**	**147.1**
Ta=773K for 120 min	**97.19**	**122.5**	**170.6**

3.2. Optical properties

Figure 4 displays the UV-Vis spectroscopic analysis of spray pyrolysis-derived cobalt-chromium oxide thin films within the wavelength range of 300-1100 nm. Figure 3 compares the absorption spectra of the unannealed sample and samples annealed at 773 K for 60, 90, and 120 minutes[32]. A high absorption level is observed within the ultraviolet region, followed by a gradual decrease within the visible and near-infrared regions. This phenomenon is common for transition metal oxides, where electronic and structural states are responsible for absorption. After annealing at 773 K, it is observed that the absorption spectra show significant variations in both intensity and morphology, confirming that the structural and electronic configuration of the samples have changed considerably. For samples annealed at 773 K for 60 minutes, it is observed that the absorption spectra are smoother and more defined, particularly within the visible region. For the sample annealed for 60 minutes, increased absorption is observed within the ultraviolet region compared to the unannealed sample, which may be due to increased crystallinity and reduced structural defects. For samples annealed at 773 K for 90 and 120 minutes, it is observed that the absorption intensity gradually changes within the visible region. This phenomenon can be correlated with grain growth and defect density reduction, as also supported by XRD results. The changes in absorption values with increased thermal treatment time are also due to changes in the mechanisms of light-matter interactions. The increased crystallinity reduces the defect states and structural irregularities, resulting in more stable electronic states. The presence of Cr^{3+} ions in the Co_3O_4 spinel structure also plays a role in the optical properties, as these ions can replace Co^{2+} ions in the Co_3O_4 crystalline structure without affecting the structural properties. In the near-infrared region, all the samples have low absorption values, and the absorption values depend moderately on the time of thermal treatment. The reduced absorption values for the samples subjected to increased thermal treatment time are due to the reduced defect scattering in the film, as also supported by the XRD results, showing the growth of crystallites with increased thermal treatment time. The thermal treatment at 773 K has a significant effect on the optical properties of the film by modifying the structural properties, defect density, and cation distribution in the Co_3O_4 crystalline structure.

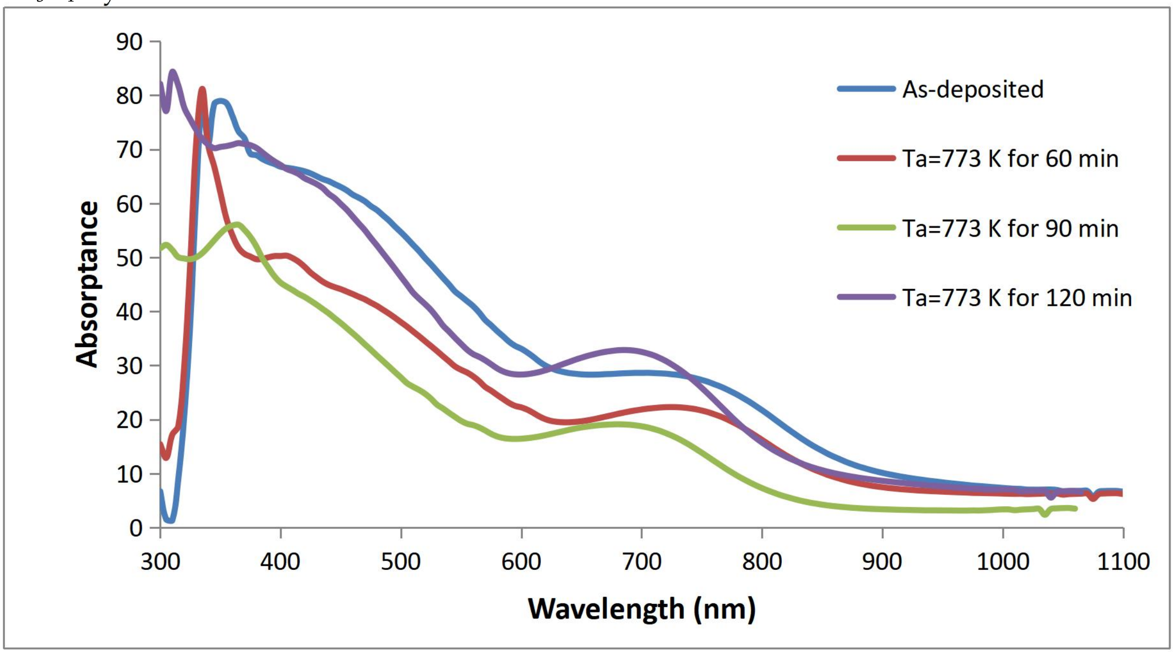

Figure 4. Optical absorptance each film's VS wavelength was created.

Figure 5 To understand the optical response of spray pyrolyzed cobalt-chromium oxide thin films, the absorption coefficient of these thin films was determined over a wavelength range of 300-1100 nm[33]. Figure 4 shows the change in absorption coefficient with wavelength for as-deposited thin film as well as thin films annealed at 773 K for 60, 90, and 120 minutes. It can clearly be seen that all thin films exhibit high absorption coefficients over the ultraviolet range of 300-400 nm. These results confirm that cobalt-chromium oxide thin films strongly absorb photons within this wavelength range. The as-deposited thin film exhibits broad absorption, which can be attributed to structural disorder. Upon annealing, changes in absorption coefficient are observed. The thin film annealed for 60 minutes exhibits enhanced absorption in the ultraviolet region compared to that of the as-deposited thin film. These changes may be attributed to enhanced crystallinity as well as structural ordering. With increasing annealing times of 90 and 120 minutes, changes in absorption coefficient are observed. These changes are attributed to grain growth as well as microstrain, as supported by XRD results. With increasing annealing times, smoothing of absorption coefficient curves can clearly be seen. These changes may be attributed to a decrease in defect-related scattering as well as localized energy levels within the lattice. With increasing annealing times, a decrease in absorption coefficient at longer wavelengths, corresponding to the visible as well as near-infrared regions, can clearly be seen. These changes may be attributed to improvements in structural ordering as well as defect density. In addition, the incorporation of Cr3+ ions into the cobalt oxide lattice may also affect optical transitions. These changes are critical in tailoring the optical properties of cobalt-chromium oxide thin films. In addition, improvements in crystalline ordering as well as defect density are critical in tailoring the optical properties of cobalt-chromium oxide thin films.

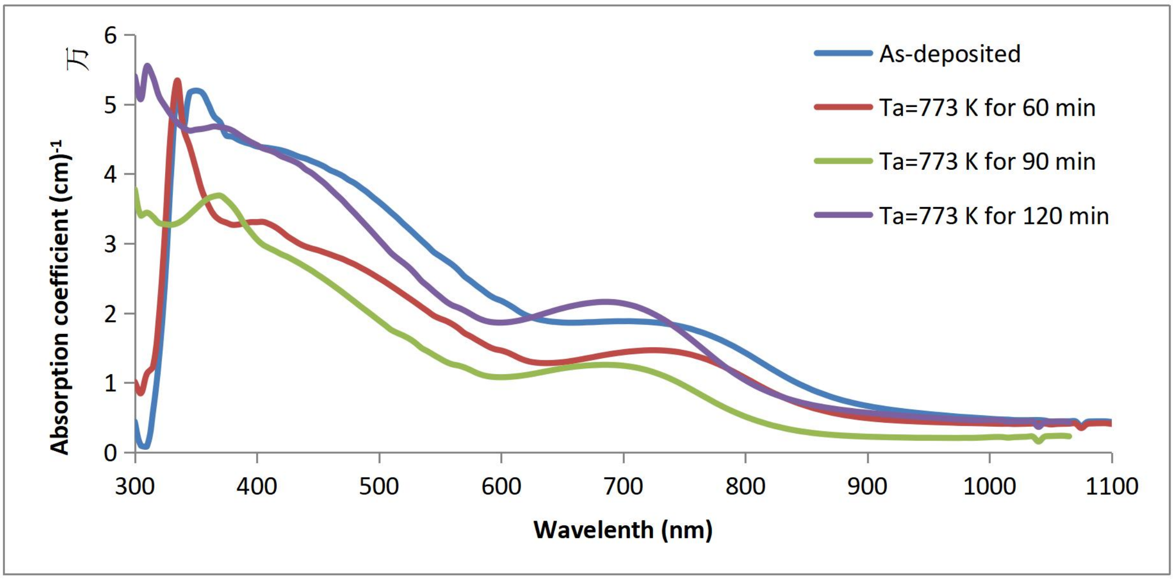

Figure 5. All films' absorption coefficients were calculated.

The band gap energy of the cobalt-chromium oxide thin films was calculated using Tauc plots of $(\alpha h\nu)^2$ vs. photon energy ($h\nu$), where the direct allowed transition was assumed. By extrapolating the linear region of the plots, the energy axis was intersected, and the band gap energies for the different samples were determined, as presented in Figure 6. For the as-deposited film, the calculated band gap energy was found to be approximately 2.0 eV. By annealing the sample at 773 K for 60 min, the calculated band gap energy was found to increase to approximately 2.4 eV, and it increased even more for longer annealing time, i.e., up to 2.6 eV. By increasing the annealing time up to 120 min, the calculated band gap energy was found to decrease to approximately 2.2 eV. By considering the increase in band gap energy from 2.0 eV for the as-deposited film up to 2.6 eV for the sample annealed for 90 min, it may be noted that improvements in crystallinity and structural defects are responsible for the increase in the band gap energy, which was corroborated using XRD analysis. By considering the decrease in microstrain and dislocation density, the localized defect states within the forbidden region are reduced, and hence the band gap energy increases. On the other hand, the reduction in the band gap at 120 minutes might be associated with increased grain growth and possible changes in the cation arrangement in the spinel lattice. As the annealing time is increased, there is a possibility that the Cr^{3+} ions will be more incorporated into the lattice structure of Co_3O_4. This might lead to changes in the electronic structure of the material. Moreover, excessive growth in the grains might lead to interactions within the lattice, resulting in a narrow optical band gap. It is also important to note that the non-linear behavior in the band gap values as a function of annealing time might be attributed to the competition between healing effects and compositional effects. It is also important to highlight that the modification in the band gap values is strongly related to the changes in the structure of the material, as demonstrated by the XRD analysis. It is also important to highlight that the annealing time is a significant factor in determining the optical band structure in the films. The obtained optical band gap (~2.6 eV) is consistent with previously reported values for cobalt-based oxide thin films. Variations in band gap values reported in the literature are often attributed to differences in crystallite size, defect density, and annealing conditions. In the present work, the increase in band gap with annealing time may be related to improved crystallinity and reduced defect states.

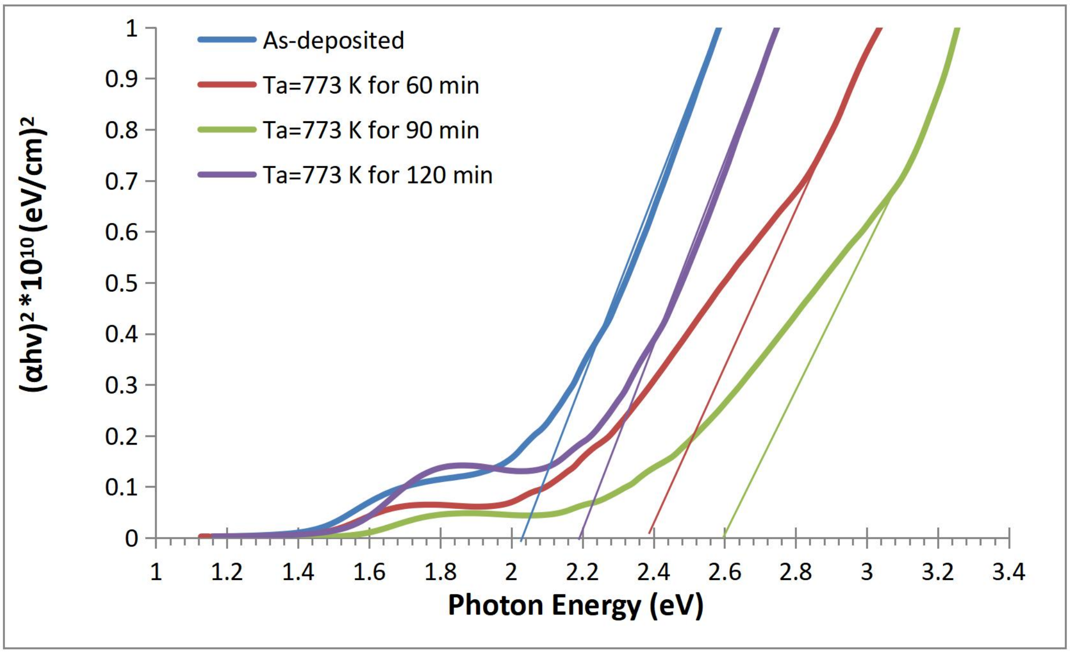

Figure 6. A Plot of Photon energy (hν) vs. $(\alpha h\nu)^2$ for as prepared thin films.

4. Conclusion

Thin films of cobalt chromium oxide have been synthesized using the technique of spray pyrolysis, followed by annealing at 500 °C for different durations. The study investigates the effect of the duration of the thermal treatment on the structural, surface, and optical properties of the films. The crystalline structure of the films has been confirmed to be a single-phase cubic spinel structure of Co3O4 using the XRD technique. The crystallinity of the films increases upon thermal treatment. The intensity of the diffraction peaks increases, the crystallite size increases, while the microstrain and dislocation density decrease upon increasing the duration of the thermal treatment from 60 to 120 minutes. The absence of the secondary peak of chromium oxide suggests the possible substitution of Cr3+ into the spinel lattice without affecting the symmetry of the crystal lattice. The surface morphology of the films has been found to change significantly upon thermal treatment. The optical properties of the films have been found to be significantly affected by the thermal treatment. The optical band gap of the films increases from 2.0 to 2.6 eV for 90 minutes of thermal treatment, followed by a decrease to 2.2 eV for 120 minutes. The results of the study indicate the importance of the thermal treatment time in optimizing the surface properties of cobalt chromium oxide films. The structural properties of the films become more stable upon thermal treatment. The defects in the films decrease significantly upon thermal treatment. The results of the study indicate the possible optimization of the surface properties of cobalt chromium oxide films using thermal treatment. The optimized films have the potential to be used for the detection of different gases.

Availability of Data and Materials: The datasets used and analyzed during the current study are available from the corresponding author on reasonable request.

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Author Contributions A.A.S. conceived and designed the research framework; S.K.A. carried out the experimental and computational work; L.S.A. provided critical guidance on the analytical methods and interpretation of results; M.H.M. performed the data analysis; L.S.A. prepared the initial draft of the manuscript. All authors have read and approved the final manuscript. All authors contributed to editorial changes in the manuscript. All authors have participated sufficiently in the work and agreed to be accountable for all aspects of the work.

Conflicts of Interest: The authors declare no conflict of interest.

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