



## Characterizing Crystalline Chromium oxide Thin Film Growth by Sol-gel method on Glass Substrates

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### ABSTRACT

In this work, nano-crystalline chromium oxide ( $\text{Cr}_2\text{O}_3$ ) thin film was synthesized using the Sol-gel method. The thin film was deposited on a glass substrate at  $50^\circ\text{C}$  by using chromium nitrate, methanol, ethanol, polyvinyl alcohol with a magnetic stirrer device and a hot plate. Each deposited thin film annealed in the microwave oven at  $200^\circ\text{C}$ . The structure of the prepared thin film was examined by means of X-ray diffraction (XRD). The Shearer formula was used to calculate the size of the film granules. The scanning electron microscopy (SEM) was used to study the surface morphology. UV-Vis measurement was also carried out to study optical properties of a sample by recording transmittance and reflectance data in the range 341-800 nm wavelength. All the tests confirmed the existence of a thin layer of  $\text{Cr}_2\text{O}_3$  on the glass substrates.

**Keywords:** Substrates, Sol-Gel Method, Thin Film, Chromium oxide, Crystallites, Dip coating, XRD, SEM, Optical properties, UV.

### INTRODUCTION

Alkali transition metal oxides have been attracted great attention due to their scientific and technological importance<sup>1-3</sup>. Among these oxides, chromium oxide exhibits an interesting type of magnetic ordering which has been studied by various methods<sup>4,5</sup>. Chromium oxides can be deposited by thermal spraying<sup>6</sup>, CVD method, Rf and DC reactive sputtering and vacuum evaporation<sup>7</sup>. The metal-organic CVD technique allows to produce

thin films with uniform surface and thickness at relatively low temperatures<sup>8</sup>. Deposition of chromium oxide has been reported in literature by various techniques<sup>9-11</sup>, chromium nitrate and chromium oxide are suitable candidates for protection of steels and decorative applications due to their distinct colors<sup>12-14</sup> and can also be used in optical applications as Electrochromic material<sup>15</sup>. High solar absorbance and low thermal remittance properties of  $\text{Cr}_2\text{O}_3$  are valuable in solar absorber coatings<sup>16-17</sup>. The chromium oxides have been extensively studied as



potential cathode material for lithium batteries<sup>18</sup>. In this paper, we report on the deposition, structure and optical characterization of chromium oxide films obtained by sol-gel method from chromium nitrate as a precursor and at low substrate temperature of 50°C. The thin films were characterized by XRD measurements, and Scanning Electron Microscopy (SEM). The thin films were optically analyzed by UV-Vis spectroscopy.

## EXPERIMENTAL

Initially, the chronic nitrate solution was prepared by dissolving (4.0015 g) in pure distilled

water (pH  $\approx$  7.0, Conductivity  $\approx$  36.00  $\mu$ S/m), then prepared polyvinyl alcohol solution was mixed with chromic nitrate solution in equal volume. The mixed solution was stirred on a hot plate for 3 h at a room temperature (30°C) under normal atmospheric pressure. After that, the temperature was raised to 50°C with continuous stirring for more than 5 hours. Detailed step by step experimental procedure is illustrated in Fig. 1. The glass substrate with dimension (24x40 mm) was dipped in the solution for 60 minutes. Deposited thin film onto glass substrate was dried, and then heated for 10 min. in a microwave oven (65 watts, 200°C and at frequency 7 (Hz)).

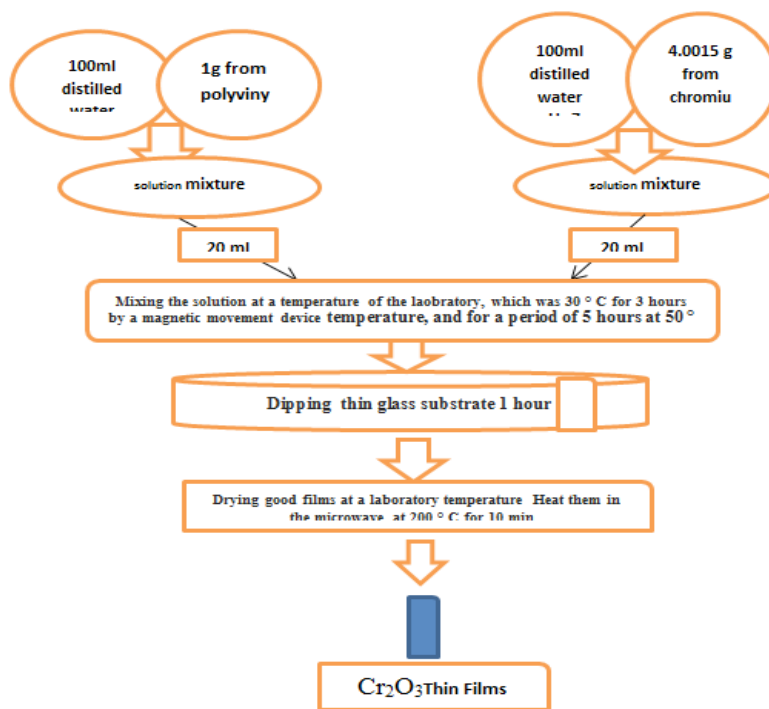


Fig. 1. Showing the process steps for the synthesis of a thin film of Nickel oxide

The thin film was characterized and the morphology of the CrO thin film was examined using (SEM). The structure and the grain size of the CrO thin film were studied using X-Ray diffraction. The UV-Visible spectrum of the sample was obtained by using an SL 210 UV-Vis Spectrophotometer-1800, in the wavelength range of 341 to 800 nm.

## RESULTS AND DISCUSSION

### Structural Analysis

The crystal structure and the orientation

of the  $\text{Cr}_2\text{O}_3$  thin films were investigated by X-ray diffraction. Fig. 2 shows XRD patterns of  $\text{Cr}_2\text{O}_3$  film annealed at 200°C. Some small and narrow peaks superimposed on the large and broad background indicates the amorphous component of the films and the glass substrate (Fig. 2). These peaks were contributed by crystallites in the films, and thus these films consisted of micro crystallites embedded in an amorphous matrix. The peaks occurred at  $2\theta = 24.6^\circ, 40.9^\circ, 41.5^\circ, 44^\circ, 51^\circ, 53.1^\circ, 57^\circ, 58.9^\circ, 63.4^\circ, 65.1^\circ, 71.8^\circ, 73.4^\circ, 77^\circ$  and  $78.5^\circ$ , are corrosspond to (012), (006), (113), (202), (024), (116), (211),

(122), (214), (300), (1010), (119), (220) and (306) planes (JCPDS card 38–1479)<sup>18</sup>. These peaks are attributed to a rhombohedral structure with preferred orientations, and the peaks (012), (113), (024), (116), (214), and (300), respectively are similar with the peaks obtained by<sup>19,20</sup>, and the peaks (012), (113), (006), (202), (024), (116), (211), (122), (214), (300), (119), (220) and (306) respectively are observed similar with the peaks obtained by<sup>21-23</sup>.

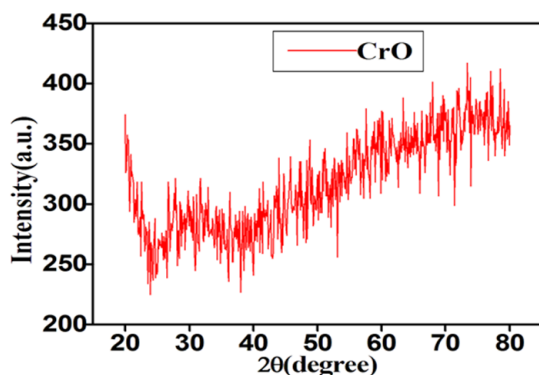


Fig. 2. The XRD pattern of the obtained Cr<sub>2</sub>O<sub>3</sub> thin film grown on glass substrate

In order to obtain detailed information of the structure of the film, the size of grain along the z-axis, and the Scherrer equation was used to calculate the average size of the crystalline based. It was observed about 77.728 nm.

$$D = 0.9\lambda/\beta\cos\theta \quad (1)$$

Where  $\lambda$  - wavelength of x-ray ( $0.154 \times 10^{-9}$ m).

$\theta$  - Bragg angle of peaks.

$\beta$  - Full width at half maximum value (FWHM).

### Morphological Analysis

To obtain insight information about the surface morphology and particle size of the Cr<sub>2</sub>O<sub>3</sub> thin film, SEM was used to get a micrograph of Cr<sub>2</sub>O<sub>3</sub> thin film, which was calcined at 200°C for 10 min. as shown in Figure 3 (A and B).

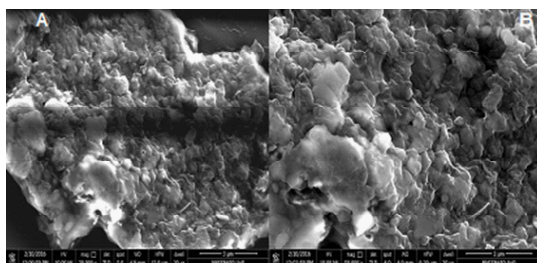


Fig. 3. SEM images of Cr<sub>2</sub>O<sub>3</sub> thin films in the form of particles spherical deposited on a glass substrate

The Cr<sub>2</sub>O<sub>3</sub> micrograph shows a compact structure composed of a single type of spherical granules which are coherent with each other with cavities and of a variable size. The mean crystallite size is found to be about ~77.7 nm.

### Optical Study

The optical properties of the synthesized chromic oxide nanostructures were examined via Spectrum of UV by falling light on the sample. Optical absorption spectra is shown in Fig. 4. It is observed from Figure that the sample absorbs the radiation in the ultraviolet region of 341 nm to 800 nm. The UV-light spectrophotometry analysis showed that the absorption peak at 392 nm. The energy gap of the thin layer of the nickel oxide was calculated by curve between  $(h\nu)$  and  $(\alpha h\nu)^2$ . The energy band gap obtained from the extrapolation curve is observed 3.16eV as shown in Fig. 5. These values are in good agreement with the reported data for CVD chromium oxide films<sup>24</sup> and close to the results obtained from those thin films with impurities prepared by the pyrolysis spraying technique and electron-beam evaporation technique<sup>25,26</sup>.

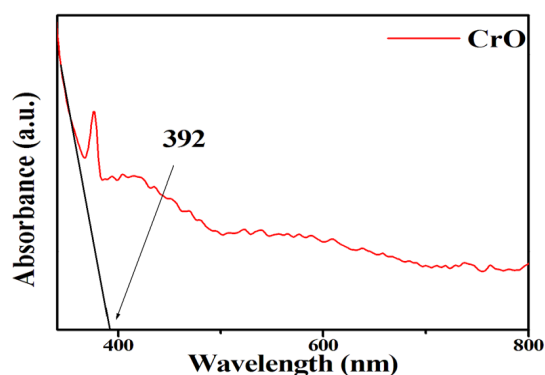


Fig. 4. Optical absorption spectra of Cr<sub>2</sub>O<sub>3</sub> thin films and energy band gap annealed at 200°C

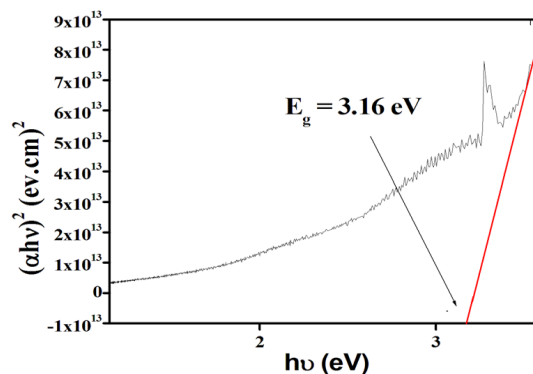


Fig. 5.  $(\alpha h\nu)^2$  vs.  $h\nu$  curves of the Cr<sub>2</sub>O<sub>3</sub> thin films annealed at 200°C

### CONCLUSION

The chromium oxide thin film was successfully synthesized by Sol-gel method. The XRD results revealed that the Cr<sub>2</sub>O<sub>3</sub> thin film is a nano crystalline with hexagonal structure. SEM

results showed that the Cr<sub>2</sub>O<sub>3</sub> micrograph has a compact structure composed of a single type of spherical granules with cavities and variable size. The mean crystallite size 77.728 nm is observed. UV study showed low absorption of optical absorption in the infrared and visible region with a energy gap of 3.16 eV.

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