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Structural, Surface and Optical Analysis of Al_2O_3 Thin Film on Al Substrates Prepared by Chemical Vapor Deposition Method

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Authors' contributions

This work was carried out in collaboration between all authors. Author SS designed the study, wrote the protocol. Author ASS wrote the first draft of the manuscript. Author JNJA managed the literature searches, analyses of the study performed the optical analysis and author DM managed the experimental process and author SS verified and corrected all the content of the manuscript. All authors read and approved the final manuscript.

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ABSTRACT

Aims: Synthesis of Aluminium oxide (Al_2O_3) thin films on Al substrates by CVD method and study their structural parameters and surface properties for various processing time.
Study Design: Synthesized thin film samples were post processed with various temperature. The structural and surface properties were analyzed by XRD technique and FESEM method to understand the influence of processing time and also the temperature.
Place and Duration of Study: *Nano Optoelectronic Research Laboratory, School of Physics, Universiti Sains Malaysia, Penang 11800, Malaysia*, between December 2013 and July 2014.
Methodology: Aluminium oxide (Al_2O_3) thin films were synthesized on Al substrates by CVD method and manipulating the deposition time for their structural evaluation using XRD spectra. The surface and optical properties were analyzed by FESEM and FTIR spectra respectively.

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Results: Polycrystalline structure was noticed with (025) phase as dominated in Al_2O_3 structure. From the XRD spectra, the observed crystallite size was increased with process time for (025) oriented phase and also noticed the average crystallite size was under 100 nm. The observed dislocation density was high for the film deposited at 20 mins duration for both (201) and (025) phases. The CVD synthesized Al_2O_3 thin film had bubble type structure on their surface irrespective to the process times and also annealing.
Conclusion: Al_2O_3 thin film was successfully deposited by CVD at low operating temperature. Post processing (annealing) was not supported to improve Al_2O_3 growth on Al substrates and also their structural properties.

Keywords: CVD; Al2O3; structural properties; surface properties.

1. INTRODUCTION

Thin films are extensively used in many industrial applications and can be prepared for various application. Aluminum oxide, commonly referred to as alumina, is a material of choice wherever hardness, wear resistance and thermal and chemical stability are desired. Alumina thin films in various forms are used in semiconductors devices as protective coatings and insulating layer, as wear resistant coatings and as sensors. [1-7]. A variety of processes such as chemical vapor deposition [8], spray pyrolysis [9], thermal evaporation, electron beam evaporation [10], magnetron sputtering [11], anodization [12], plasma enhanced chemical vapor deposition [13], the sol–gel process [14], pulsed laser deposition [15], aerosol-jet deposition [16] and atomic layer deposition [17] have been tried to synthesis Al_2O_3 thin film on various substrates. Plasma-enhanced chemical vapor deposition (PECVD) process satisfies the requirements, even though it has some limitations [8].

Among these, CVD process is a technique in which gases or vapors of chemical compounds of the elements which shall be forming the film are introduced into a reaction chamber; a solid deposit film is obtained via chemical reactions on a substrate. High-temperature CVD processes often cause unwanted thermal strain and diffusion problems. PVD processes, such as evaporation or sputtering, do not provide good coverage of surface steps, and are therefore not suitable for multilevel devices having complicated surfaces morphology. A considerable interest has been taken in low-temperature deposition process having good step coverage. Several researches adopted the low-temperature PECVD method for the preparation of aluminum oxide films using trimethylaluminum (TMA) source [18]. There are several reports with notable and useful results concerning the optical properties of Al_2O_3 films [8,19,20] and stated the behavior of the film is based on the microstructure. In order to understand, a detailed study has to be conducted on structural and surface properties of chemical vapor deposited Al_2O_3 thin film to extend their application. In this paper, Al_2O_3 thin film is synthesized on Al substrates by CVD method and studied their structural, optical and surface properties. A detailed structural parameter analysis is carried out here by changed the process time and also post processing such as annealing.

2. EXPERIMENTAL DETAILS

2.1 Synthesis of Al_2O_3 Thin Film

Al_2O_3 thin film was deposited on Al substrates by chemical vapor deposition method using 3 zones CVD furnace. In order to obtain a various elemental compositions and also optimization of the thin films, time taken for the deposition process was manipulated from 10 to 30 mins. The parameters of the process used in this study are provided in Table 1.

Table 1. Deposition parameters of Al_2O_3 thin film coating using CVD

Parameter	Condition
Precursor	Aluminium acetylacetonate
Precursor temperature	300℃
Substrate	Aluminium 2.5 cmx25 cm
Substrate temperature	450℃
N_2 flow rate	10 sccm
Deposition time	10 mins, 20 mins & 30 mins
Annealing condition	100℃, 200℃ & 300℃

Aluminium acetylacetonate [Al(acac)$_3$] was used as precursor for thin film deposition. Approximately 4 g of Al(acac)$_3$ powder was filled in a boat type crucible and placed at the left zone of the furnace while the Al substrates were in the middle zone. At the outset, CVD tube was evacuated initially by rotary vacuum pump followed by filling inert gas (Argon) to maintain the inert atmosphere inside the tube. The precursor temperature was set to be at 300℃. N_2 gas with a fixed flow rate of 10 sccm was used as carrier gas in which its flow rate was controlled by digital mass flow controller. N_2 gas was released into the tube from left zone, pushing in the Al(acac)$_3$ into the middle zone where the Al substrates were located with a fixed temperature of 450℃. The coating process time was varied (10, 20 and 30 mins) and coated Al_2O_3 thin film under atmospheric pressure. After the synthesis completed, the coated substrates underwent annealing process at 3 different temperatures (100℃, 200℃ and 300℃) for about 1 hour in separate tube furnace at ambient condition.

2.2 Characterization of Thin Film

The structural parameters of CVD processed Al_2O_3 thin film was evaluated from the X-ray diffraction analysis which was carried out using X-ray diffractometry (XRD) (X'pert-PRO, Philips, Netherlands) machine with Cu target working at 40 kV, 40 mA and a scanning 2θ range between 30° to 80°. From the XRD Spectra, the structural parameters such as crystallite size, dislocation density, residual stress, lattice strain etc were calculated and discussed in this analysis. The surface morphology of Al_2O_3 thin film was examined using Field Emission Scanning Electron Microscope (FESEM). The optical behavior was also tested using Fourier Transform Infrared (FTIR) (Model: Perkin Elmer Spectrum GX series) and also it has been used to identify the presence of Al and O bonding nature in the thin film.

3. RESULTS AND DISCUSSION

3.1 XRD Analysis

The XRD spectra of all CVD processed Al_2O_3 thin films were recorded as shown in Fig. 1 (a), (b) and (c) and indexed by using xrd software and JCPDS file. Diffraction occurs when each object in the periodic array scatters radiation coherently, producing concerted constructive interference at specific angles. From the Fig. 1, it is observed that the synthesized Al_2O_3 phases are polycrystalline and also noticed with intense peaks of (201) and (025) oriented phase for all samples. Additionally, (040) and (119) oriented phase were also noticed in the samples with low intensity. Using the JCPDS data, all samples were indexed and confirmed the formation of orthorhombic phase for the first two major peaks, followed by monoclinic and rhombohedra crystal system for the two other minor peaks consequently. It reveals the combination of polycrystalline phases of Al_2O_3 is possible in this process condition using CVD method. Moreover, Fig. 1 also clearly states the influence of annealing on the crystalline behavior of synthesized Al_2O_3 film as expected. On considering the effects of annealing temperature on the samples deposited at 20 mins and 30 mins, the crystalline quality of the Al_2O_3 thin film is high for the non-annealed sample than that of the annealed one. It clearly indicates that the annealing process doesn't show much influence on improving the crystalline quality of Al_2O_3 thin film for this process timings. It shows that the intensity of the peaks decreases on higher annealing temperature. But the intensity of (201) phase increases with annealing temperature increases for the samples prepared at 10 mins processing time. On considering the 30 mins processing time, the samples annealed at 200℃ shows improvement in peak intensity of (201) plane. It is the indication of reduction on the crystalline quality of the processed film at 100℃ for the film prepared for about 30 mins deposition time. Overall, the annealing temperature was influencing on the degradation of the crystalline quality of all phases other than (201).

As observed from Fig. 1, the peak analysis was done on (201) and (025) peaks since it is dominated among the other phases. By using the XRD software, two major peaks of (201) and (025) were zoomed in as shown in Fig. 2(a-c) and used for peak shifting and intensity analysis. Fig. 2 reveals that the observed peaks are doublet nature which is originated from the instrumental error i.e., unable to separate $K_{\alpha1}$ and $K_{\alpha2}$ radiation. This experimental error could be rectified by selecting the proper source with accuracy. The separation between the two peaks increases with increasing diffraction angle. As a result, the distance between the peak position of the doublet peak can be minimized and may be removed [21]. As seen from the Fig. 2 that the peak was shifted to higher 2θ as the annealing temperature increases for the film synthesized at 10 mins duration. A drastic reduction in intensity

of both phases was also noticed for the samples annealed at 100℃. As we know, the intensity of the diffraction pattern is directly proportional to the concentration of the component producing it.

The decrease in the concentration of the 30 mins process time sample annealed at 100℃ may be due to higher deposition time as the structure may not withstand longer time than 20 mins. For the film prepared at 10 mins process time, the one annealed at 200℃ shows the highest intensity as compared to all other peaks observed and the intensity started to decrease a little when the annealing temperature increase up to 300℃.

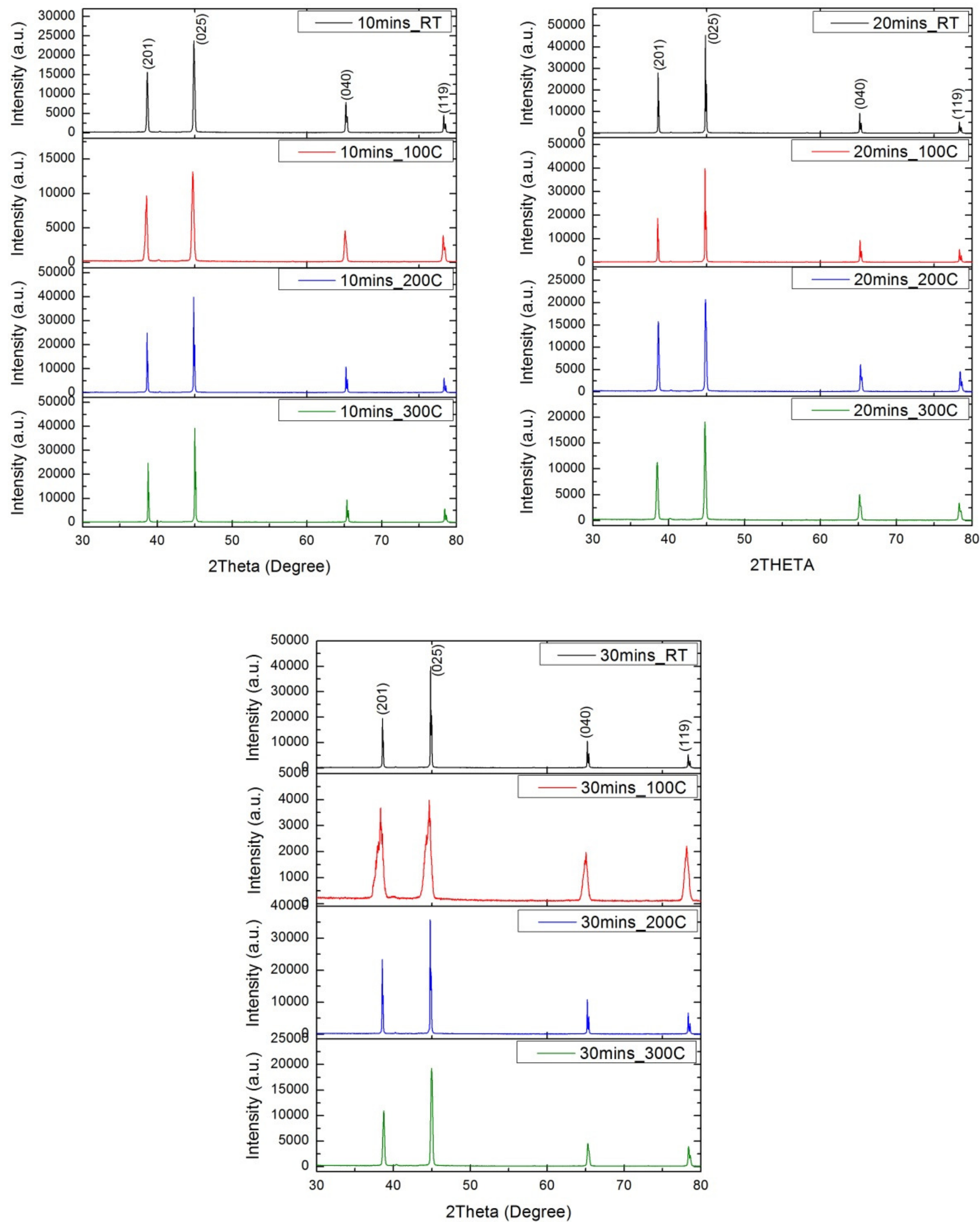

Fig. 1. XRD spectra of the Al_2O_3 thin film samples deposited with: (a) 10 mins process time, (b) 20 mins process time, and (c) 30 mins process time

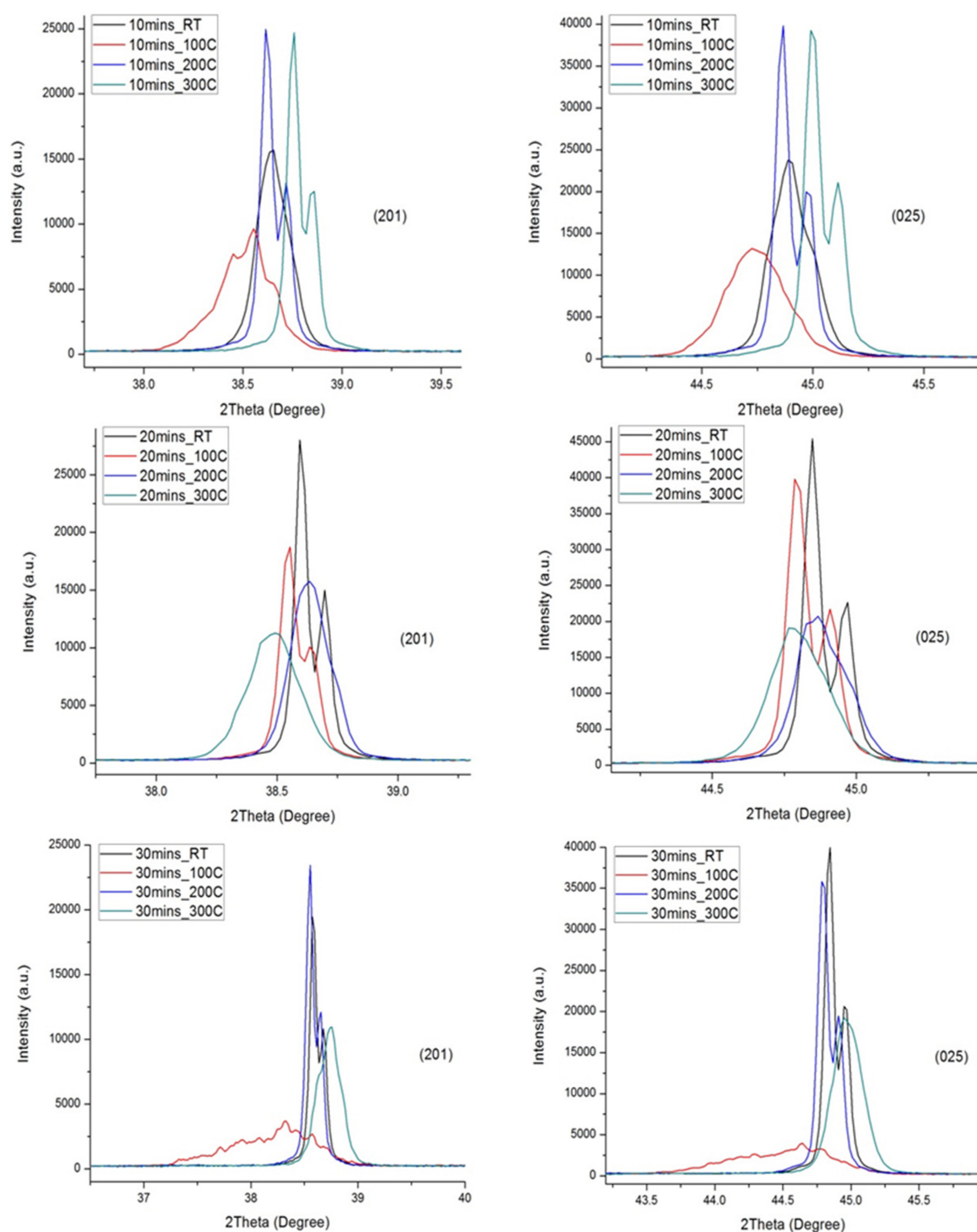

Fig. 2. XRD spectra of (201) and (025) for: (a) 10 mins process time, (b) 20 mins process time, and (c)30 mins process time

For the 20 mins process time, the highest intensity is shown for the non-annealed sample. Increase in the annealing temperature makes the intensity drops. It is also observed from the Fig. 2(b) and (c) that the processing time is influencing the change in intensity and also the position of the peak with respect to the phases observed in this study.

On considering the samples prepared at 30 mins duration, the annealing temperature of 100℃ is not preferred to grow such phases (201) & (025) and observed the optimum annealing temperature is about 200℃ mins for Al_2O_3 phases. In order to understand the detailed properties of CVD synthesized Al_2O_3 thin film, the structural parameters should be addressed in

detail and consequently, the mean crystallite sizes (*D*) of the two dominant peaks which are (201) and (025) were calculated by using the Debye Scherrer formula [22] using XRD data, and the values were given in Table 2.

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

where λ is the wavelength of the X-ray radiation used, in which in this case is 1.5406Å, β is the measured broadening of the diffraction line peak at 2θ, at half its maximum intensity (FWHM) in radian and θ is the Bragg diffraction angle of the peak. As we know, the annealing temperature supports to increase the crystallite size of sample. This applies on the 10 mins process time samples but not on the 20 mins and 30 mins process time samples. It is because of some structural defects during the crystal growth for long process timings > 20 mins.

The residual stress (σ) in the deposited film is the key parameter and decide the application of Al_2O_3 thin film at different conditions. Hence it should be addressed and is calculated using the relation:

$$\sigma = -E(d_a - d_o)/(2d_oY) \quad (2)$$

where d_a and d_o are the *d* spacing of the thin film form respectively obtained from the JCPDS data. *E* is the Young's modulus and *Y* is the Poisson's ratio of Al_2O_3. The internal stress developed during the growth of Al_2O_3 thin film was calculated for the two dominant peaks and the observed results are given in Table 2. The positive value of the stress indicates tensile stress while the negative values indicate compressive stress. As can be seen, the 20 mins process time shows the lowest residual stress for (025) phase as compared to all followed by the 30 mins process time for the same oriented phase. Overall, high and low values of tensile stress are noticed with (201) orientation of 30 mins process time annealed at 300℃ and (025) orientation of 10mins process time annealed at 100℃. Moreover, the stress applied during the film growth for the (025) phase shows compressive nature and increases with annealing temperature increases.

In addition to this, dislocation density (δ) which is the length of dislocation lines per unit volume of crystal was evaluated from the relation:

$$\delta = \frac{1}{D^2} \quad (3)$$

The dislocation density is mainly based on the crystallite size (*D*) and hence, we can say that it is the results of changes in crystallite size. The obtained results are tabulated in Table 2. Since the dislocation density is measured based on the crystallite size, the observed results are obeyed the behavior of changing the crystallite size for different process conditions. The lattice strain (ε) is calculated using the tangent formula and the values are summarized in Table 2 as well.

Table 2. Structural parameters of CVD synthesized Al_2O_3 thin film for various process conditions during and after synthesis

Conditions process time/annealing temperature		Crystallite size, D (nm)		Residual stress, σ (MPa)		Dislocation density (lines/m^{-2})		Lattice strain, ε	
		(201)	(025)	(201)	(025)	(201)	(025)	(201)	(025)
10 mins	RT	53.5	44.8	1.798	0.740	3.5x10^{14}	4.9x10^{14}	0.00034	0.00041
	100℃	116.9	25.6	0.813	-3.322	7.3x10^{13}	1.5x10^{15}	0.00014	0.00055
	200℃	116.9	119.4	2.228	0.029	7.3x10^{13}	7.0x10^{13}	0.00015	0.00014
	300℃	117.0	119.4	5.441	2.829	7.3x10^{13}	7.0x10^{13}	0.00018	0.00017
20 mins	RT	116.9	119.4	1.727	-0.292	7.3x10^{13}	7.0x10^{13}	0.00015	0.00014
	100℃	87.7	119.3	0.400	-1.412	1.3x10^{14}	7.0x10^{13}	0.00019	0.00013
	200℃	39.0	44.8	2.400	-0.404	6.6x10^{14}	5.0x10^{14}	0.00047	0.00038
	300℃	42.8	43.7	-1.074	-2.489	5.5x10^{14}	5.2x10^{14}	0.00037	0.00036
30 mins	RT	116.9	119.4	1.280	-0.487	7.3x10^{13}	7.0x10^{13}	0.00067	0.00069
	100℃	70.1	119.3	-5.037	-4.383	2.0x10^{14}	7.0x10^{13}	0.00163	0.00092
	200℃	116.9	119.3	0.493	-1.422	7.3x10^{13}	7.0x10^{13}	0.00014	0.00013
	300℃	116.9	22.4	5.454	1.714	7.3x10^{13}	2.0x10^{15}	0.00015	0.00087

$$\varepsilon = \frac{\beta}{4\tan\theta} \quad (4)$$

For (201) orientation of the 30 mins process time sample annealed at 100°C, the lattice strain shoot up, showing a high value than others. The strain developed during the synthesis of the thin film at room temperature showed a high value for both 10 mins and 30 mins process time. Overall, the average lattice strain was low for 10 mins synthesized Al_2O_3 thin film than other process time.

3.2 FESEM Analysis

FESEM was used in order to visualize the topographical nature of the surface of the CVD prepared Al_2O_3 samples. In order to obtain the thickness details, a reference Si samples were kept during the deposition process and used for thickness analysis. Consequently, a cross sectional images of each sample was captured and the average thickness was measured as presented in Table 3. As we expected, the thickness of the film increases as with deposition time increases. Additionally, the surface morphology of the prepared samples was also recorded with a magnification of 10 kx and presented in Figs. 3, 4 and 5.

As can be seen in the figures, bubbles-type structure are noticed on all film surfaces. As the annealing time increases for all deposition time, a noticeable surface modification occurred. Form the FESEM images, the surface is looking very rough since we used unpolished Al substrates. In order to confirm the bubbles-type structure, an elemental identification analysis has been done using energy dispersive X-ray analyzer (EDX) on the bubbles. The results showed the existence of Si other than Al and O out of the bubble.

Table 3. Average thickness of Al_2O_3 thin film prepared for different time of deposition

Time of deposition (mins)	Average thickness (nm)
10	310.8
20	951.9
30	1820.7

This may due to the surrounding effect towards the thin film during the annealing process as well as synthesis process. All the figures indicate that the annealing process induced the morphological structure of the samples. From Fig. 3, as the annealed temperature increased to 300°C, the bubble-type structure became smaller and lesser. Wavy surfaces were also noticed on Al_2O_3 thin film samples synthesized at 20 mins duration. As can be observed in Fig. 5, the sample surfaces became more flat along with the temperature rising, and only affects the bubble structure on the 200°C annealed samples.

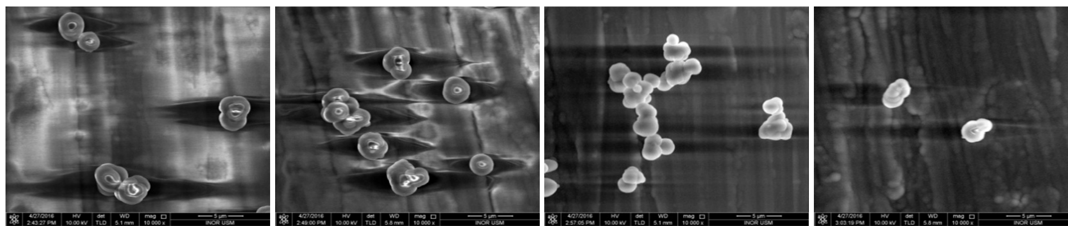

Fig. 3. FESEM images of Al_2O_3 thin film samples deposited in 10 mins at (a) room temperature, (b) annealed at 100°C, (c) annealed at 200°C and (d) annealed at 300°C

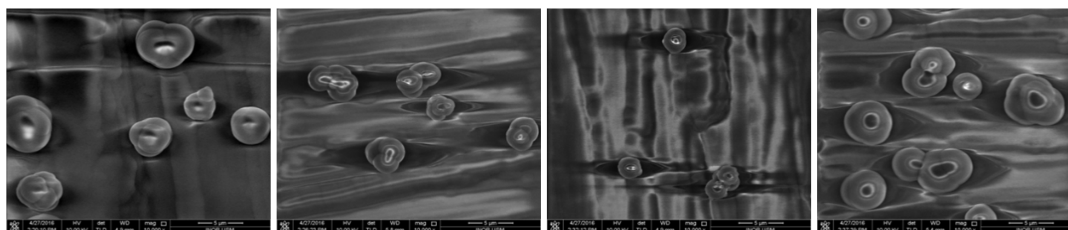

Fig. 4. FESEM images of Al_2O_3 thin film samples deposited in 20 mins at (a) room temperature, (b) annealed at 100°C, (c) annealed at 200°C and (d) annealed at 300°C

3.3 FTIR Analysis

FTIR is the preferred method of infrared spectroscopy. In infrared spectroscopy, IR radiation is passed through the CVD Al_2O_3 thin film sample. In this study, Al substrate was used which is an opaque material, and so some of the infrared radiation is absorbed by the sample while some of it is reflected. Fig. 6 (a), (b) and (c) shows the IR spectrum of the samples with various process times including different annealing condition. At wavelengths where the sample exhibits a strong IR absorption, the reflectivity of the sample increases. The superposition of the extinction coefficient spectrum with the refractive index dispersion results in a spectrum with derivative shaped bands as in Fig. 6. The frequencies at which reflectance occurs indicate the type of functional groups present in the thin film. It reveals that the different deposition time does influence the optical behavior of the Al_2O_3 thin film.

A number of reflection peaks can be identified as 700-750 cm^{-1}, 960-1000 cm^{-1}, 1200-1230 cm^{-1}, 1490-1520 cm^{-1}, 1720-1790 cm^{-1}, and 3670-3730 cm^{-1}. The noticed band in between 700-750 cm^{-1} indicates the Al-O transversal optic (TO) stretching [23] for the thin film samples synthesized in 20 mins and 30 mins duration. The peak around 960-1000 cm^{-1} is attributed to the Al-O longitudinal optic (LO) stretching [23]. The AlOH surface species were observed by the AlO-H stretching vibrations between 3670-3730 [24].

The increased in the annealing temperature in Fig. 6 (a) gives a huge effect to the dispersion spectrum. As the annealing temperature increased to 200℃ and 300℃, most of the peaks could not be identified. A slight difference in the intensity of the peaks for 20 mins and 30 mins deposition time may be due to the qualitative results of the chemical interaction present during the annealing process. Common band exists in all cases, such as the broad OH bond, C-H stretching vibrations and others, and they are not considered here for further discussion.

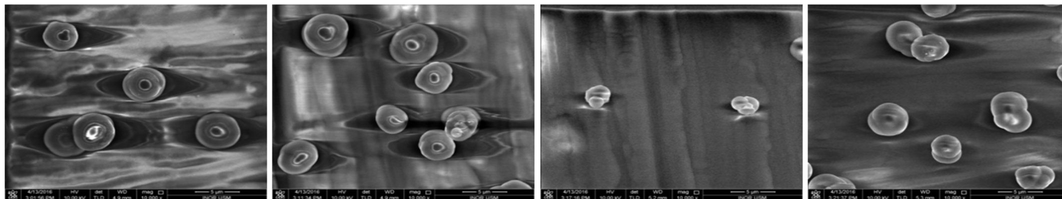

Fig. 5. FESEM images of Al_2O_3 thin film samples deposited in 30 mins at (a) room temperature, (b) annealed at 100℃, (c) annealed at 200℃ and (d) annealed at 300℃

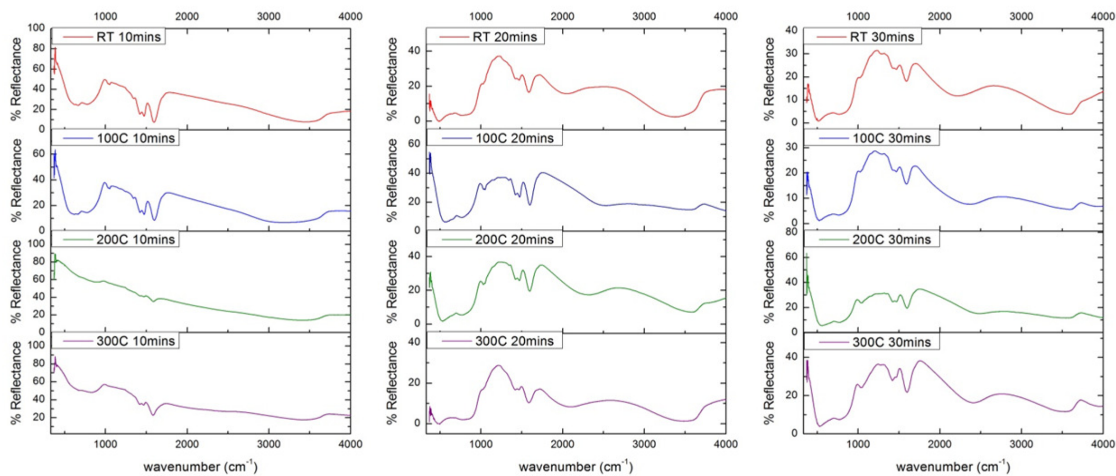

Fig. 6. FTIR spectrum of samples prepared with: (a) 10 mins process time, (b) 20 mins process time, and (c) 30 mins process time

4. CONCLUSION

CVD was used to synthesize Al_2O_3 thin film on Al substrate and tested their structural and surface properties. (025) phase was observed as preferred growth for Al_2O_3 thin film on Al substrates by CVD process irrespective to the process time. The structural studies showed the influence of process time and also temperature on crystallite size and other parameters. Polycrystalline Al_2O_3 was observed by CVD method with tensile stress as dominated stress. Bubbles-type structure was the key observation for CVD processed Al_2O_3. The prepared samples for about 10 mins showed less lattice strain than other samples for (201) orientation. The FTIR analysis confirmed the presence of transversal optic (TO) and longitudinal optic (LO) stretching modes in all prepared samples.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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