

## Structural and Optical Properties of Graphene Oxide:Zinc Oxide on Different Types of Substrates

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### ABSTRACT

The structural and optical properties of ZnO-based nanostructure exhibit substrate-dependent variations. A comprehensive understanding of how different substrates influence the crystalline structure, absorbance and surface morphology of the deposited films remains a great challenge. This study was done to investigate the structural and optical properties of zinc oxide (ZnO) that alter with graphene oxide (GO) deposited on various substrates; glass, silicon, indium tin oxide (ITO) and polyethylene terephthalate (PET) by solution immersion method. The structural properties were studied using X-ray diffraction (XRD) and Fourier-transform infrared (FTIR). The optical properties of samples were determined using ultraviolet-visible (UV-VIS) spectroscopy and Raman spectroscopy. The XRD result revealed that all GO/ZnO samples have polycrystalline structures while FTIR showed the presence of C-O, C=O and C-H compounds on all substrates. The highest absorbance of 3.40 was shown by the GO/ZnO sample which was grown on a glass substrate while the lowest absorbance of 1.50 was exhibited by the sample that was grown on PET substrate. Based on the Raman spectrum all samples showed variety in phonon energy when ZnO was grown on different substrates.

## 1. Introduction

Zinc Oxide (ZnO) is natively a n-type semiconductor material with a wide band gap energy of 3.37 eV and a high exciton binding energy of 60 MeV [1]. ZnO is the most popular chemical compound that has impressive properties such as low-cost material, non-toxicity, material stability, wide availability and high transparency [2]. ZnO can be applied to growth the formation of various

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nanostructures because of its well-balanced polar surface. ZnO nanostructures such as nanorods, nanowires and nanoflowers have been widely advertised for various applications, such as sensors, photocatalysts, solar cells and magnetic compounds [3]. The synthesis of ZnO nanostructures was successfully carried out with a variety of methods including laser ablation, electrochemical deposition, chemical vapour deposition, common thermal evaporation method, solvothermal method and many more. ZnO had formerly received relatively some attention within the very active studies that aim to produce electronic devices in the visible and UV wavelength region. Moreover, ZnO also can act as a catalyst for the capabilities and performance of these applications. Investigations on the preparation of ZnO nanostructures focused not only on changing morphological and optical properties but also on improving electrical properties to meet the demands of electronic applications. The properties of ZnO can be improved by coating and doping [4]. Mamat *et al.*, reported that Al doping increased charge carriers in ZnO films due to the extra ion charge of Al element might effectively enhance the optical and electrical properties of ZnO films [5]. Other studies were also done by adding carbon materials such as graphene oxide to improve the properties of metal oxide materials [6].

GO is a popular carbon material used in research due to its large surface area, amphiphilicity, and electronic properties that can be tuned through surface modification. It consists of an oxygen functional group covalently bonded to a carbon atom. GO has a chemically active surface that allows organic molecules or other metal oxides to attach to it and can change its electrical properties [7]. Graphene oxide (GO) is mostly used because of its highly functional 2-D conducting support material. The properties of graphene such as excellent physiochemical properties, greater electrochemical window and good conductivity make this material selected to be added to ZnO [8]. According to the previous study, Darina Schelonka *et al.*, reported that the preparation of GO/ZnO proved that the GO could enhance the photocatalytic performance [9]. Therefore, improving the charge separation which can lead to the growth in photocatalytic activity can be done by adding GO to ZnO. Moreover, K. S. Lee *et al.*, successfully fabricated GO/ZnO nanocomposites by precipitation for supercapacitor electrodes. The result shows that the maximum specific capacitance of  $97 \text{ Fg}^{-1}$  at a current density of  $0.5 \text{ Ag}^{-1}$  by galvanostatic charging-discharging was achieved on 1M of  $\text{Na}_2\text{SO}_4$  electrolyte [10]. Therefore, the results suggest that GO/ZnO nanocomposites can be used for supercapacitor material.

The study on the fabrication of ZnO with GO has been conducted by many researchers around the world. The combination of these two materials is expected to enhance the photo response due to the unique properties of GO [11]. However, the study of ZnO with GO on various substrates is rarely being studied. Even though the solution immersion method has been used by many researchers, this method still can be manipulated to be used on different kinds of substrates. The growth mechanisms of ZnO vary significantly across different substrates, leading to uncertainties in the robustness and reliability of the deposited layer for specific applications. Therefore, in this study, the growth of ZnO added with GO on various types of substrates was investigated. The selection of substrate material is one way for the fabrication of ZnO thin films based on the lattice parameters and thermal mismatching among substrates and also the film because of the increasing stress in the deposited films. The solution immersion method was chosen to grow GO/ZnO structure on various substrates due to better structural control and better homogeneity due to mixing at the molecular level. In addition, this method uses less energy because the network structure can be produced at a relatively low temperature. It also has distinct superiority over the other techniques due to accurate compositional control and homogeneity at the molecular level [4]. The four types of substrates chosen in the study are glass, silicon (Si), indium tin oxide (ITO) and polyethylene terephthalate (PET). Particularly in this study, the structural and optical properties of GO/ZnO nanostructures grown on different substrates were investigated. The outcomes of the study are the understanding of how

different substrates influence the properties of GO/ZnO and shows potential range of technological applications.

## 2. Methodology

### 2.1 Seeded Layer Preparation of ZnO Thin Films

Sol-gel spin coating method was used to prepare the ZnO seeded layer on various substrates. The Si, glass, ITO and PET were used as a substrate. Firstly, all the substrate needs to be cleaned about 10 minutes by using methanol, acetone and deionized (DI) water. Next, 0.4M zinc acetate dihydrate ( $\text{Zn}(\text{CH}_3\text{COO})_2 \cdot 2\text{H}_2\text{O}$ , 99.5% Merck) and 0.4M of monoethanolamine ( $\text{C}_2\text{H}_7\text{N}$ , 99.5% Aldrich) was dissolved in a 2-methoxyethanol solvent. Then, the mixed solution was heated and stirred at 80°C for 60 minutes. The solution was kept at ambient temperature for 24 hours to get a homogenous solution. Next, the solution was coated onto a substrate using the spin coating method. The solution was dropped onto the substrate at a speed of 3000 rpm for 30 seconds. The samples were preheated in an atmosphere ambient at 150°C to remove solvent. Finally, the samples were annealed in a furnace at 300°C for an hour.

### 2.2 Preparation of GO/ZnO Thin Films

GO/ZnO were grown on various types of substrates using the aqueous solution immersion method. Firstly, the solution was prepared using 0.1M zinc nitrate hexahydrate ( $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ , 98.5% Reindemann Schimdt) and 0.1M hexamethylenetetramine (HMTA,  $\text{C}_6\text{H}_{12}\text{N}_4$ , 99% Aldrich) as a precursor and a stabilizer [12], respectively. Then, 0.005M of GO was added to the mixture and sonicated for 30 minutes at 50°C. Next, the mixture was stirred and aged for 3 hours at ambient temperature and then the solution was poured into a vessel that had been placed with a seeded substrate at the bottom of the vessel. The immersion process was performed by immersing the sealed vessels into a hot water bath at 95°C for 1 hour. After immersion, the GO/ZnO thin films were rinsed with deionizing (DI) water and annealed using a furnace at 500°C for 30 minutes.

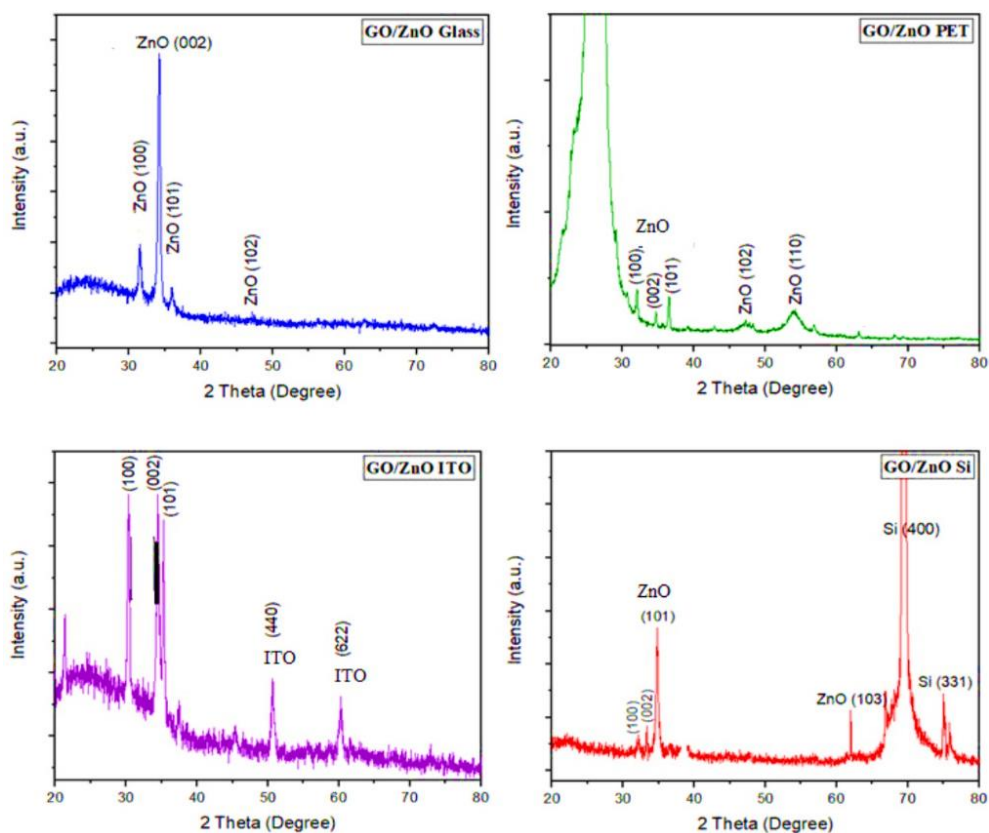
### 2.3 Characterization

Finally, The GO/ZnO prepared on different types of substrates were characterized using x-ray diffraction (XRD, Bruker, D-8 advance, Billerica, MA, USA) with radiation source of  $\text{CuK}\alpha$  X-ray ( $\lambda = 1.54 \text{ \AA}$ ), UV-VIS spectroscopy (Cary 5000, Varian) within wavelength range 300–800 nm at room temperature, Fourier transform infrared (FTIR, Perkin Elmer, Waltham, MA, USA) within wavenumber range 400–4000  $\text{cm}^{-1}$  and Raman spectroscopy (HR-800, Jobin Yvon, Edison, NJ, USA) with  $\text{Ar}^+$  ion laser line ( $\lambda =$  approximately 514.532 nm) wavelength source at room temperature.

## 3. Results

The crystal structure of the GO/ZnO thin films was characterized using X-ray diffraction (XRD) measurements. The diffraction pattern of ZnO/GO prepared with different types of substrate at  $2\theta$  in the range from 20° to 80° is shown in Figure 1. Three dominant ZnO peaks appeared at (100), (002), (101) and (102) planes when GO/ZnO grown on glass and ITO substrate correspond to the hexagonal wurtzite structure of ZnO (JCPDS 36-1451). XRD spectra show the highest peak intensity on a glass substrate along (002) planes. It is observed the existence of (100), (002), (101), (102) and (110) planes when GO/ZnO is grown on PET substrate. The broad peak and high intensity were observed at an

angle of 26° indicated to PET substrate [13]. Four dominant ZnO peaks are present at (100), (002), (101) and (103) planes when GO/ZnO is grown on a silicon substrate. Two peak intensities are seen which are on the (400) and (331) planes of the GO/ZnO thin film formed on the silicon substrate belonging to Si [14]. Various phases were obtained when GO/ZnO were grown on different substrates. It also observed in the XRD pattern the peaks corresponding to the substrate. The peak positions of the (002) plane for GO/ZnO thin films grown on different substrates were compared. Based on the observation, the degree of (002) plane for GO/ZnO thin films grown on glass, ITO, PET and silicon substrate were around 34.50, 34.42, 34.90 and 33.50 °, respectively. The obtained results show that the (002) peak position slightly changed. The shift occurs due to the change in lattice parameters contributed by strain or stress of different substrates. The variation of peak position may be due to the different strain levels in the samples that are attributed to the lattice mismatch between the ZnO thin films and substrates [15].



**Fig. 1.** XRD spectra of ZnO:GO thin films deposited on various substrates

The Debye-Scherrer equation is used to estimate the average size of crystallites  $D$ , in a sample. The Debye-Scherrer equation is shown as follows [16]

$$D = \frac{0.94\lambda}{\beta \cos \theta}$$

where  $\lambda$  is the wavelength of the X-ray,  $\beta$  is the full width at half maximum (FWHM) of the diffraction peak in radians, and  $\theta$  is the Bragg's angle in degrees. The dislocation density,  $\delta$  [17] is calculated using

$$\delta = \frac{1}{D^2}$$

The lattice constants, a and c are calculated using the following equation [18]

$$a = \frac{\lambda}{\sqrt{3} \sin \theta}$$

$$c = \frac{\lambda}{\sin \theta}$$

The formula to calculate the atomic packing fraction (APF) of GO/ZnO thin films is [18]

$$APF = \frac{2\pi a}{3\sqrt{(3)c}}$$

The average crystallite size and dislocation density of GO/ZnO thin films for three planes (100), (002) and (101) are shown in Table 1. It was found that the average crystallite size of GO/ZnO thin films on glass, PET, ITO and silicon substrates were 34, 31, 27 and 24 nm, respectively. The dislocation density of the GO/ZnO thin films grown on glass substrate has a minimum value, while GO/ZnO thin films grown on silicon substrate have a maximum value. Samples of a thin film with a small crystal size that has high dislocation density values show more ordered grains with higher micro-defects in the grains of the GO/ZnO sample [18]. The APF values are listed in Table 2. The bulk hexagonal ZnO materials are about 74%. In this study, the APF of GO/ZnO thin films grown on glass, PET and ITO substrates larger APF than pure ZnO. It seems the APF value of GO/ZnO thin films grown on a silicon substrate has a lower APF value compared to the bulk ZnO value. It means that APF in nanocrystals for thin films grown on glass, PET and ITO substrates are slightly larger than that of bulk materials.

**Table 1**  
 Parameters of XRD analysis for different substrates

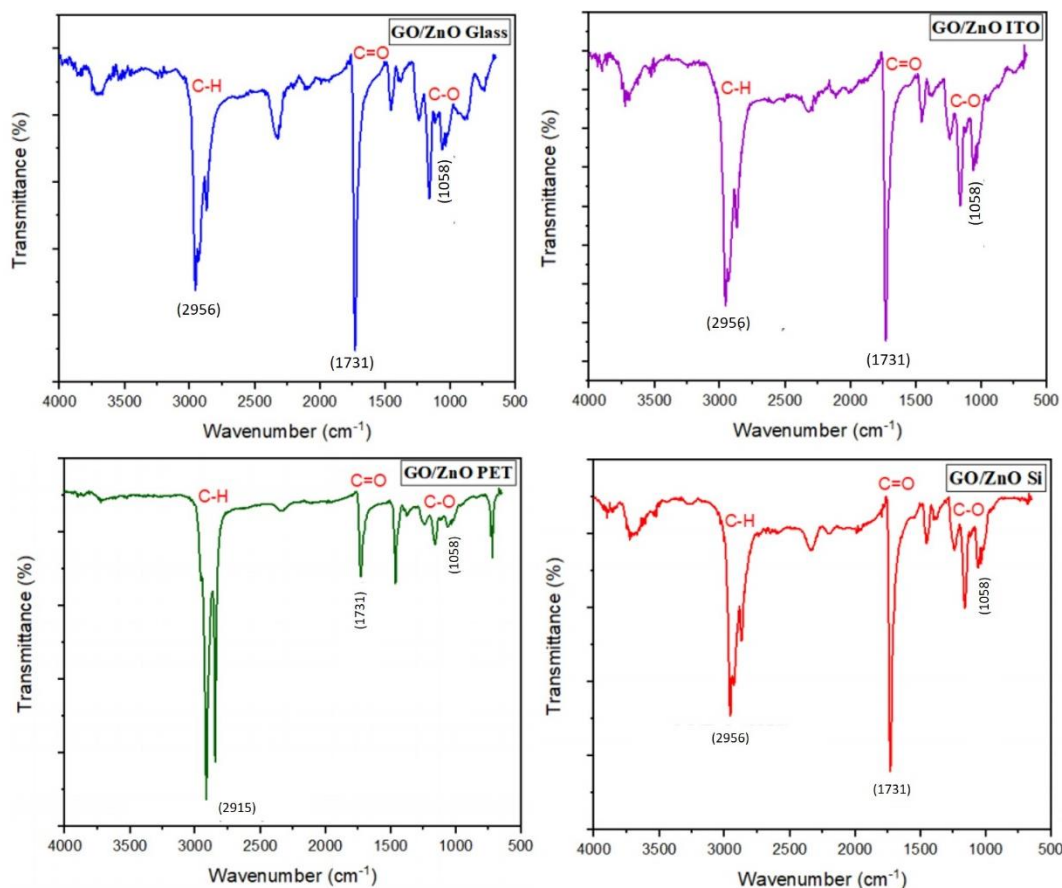
Substrate	Peak (hkl)	FWHM, $\beta$ (rad)	Angle diffraction peak, $\theta$ (rad)	Crystalline sizes, D (nm)	Average crystalline size of samples, D (nm)	Dislocation density, $\delta \times 10^{-4}(\text{nm})^{-2}$
Glass	(100)	0.3648	15.8953	23.64	33.75	8.78
	(002)	0.9260	17.3234	9.38		
	(101)	0.1279	18.1623	68.23		
PET	(100)	0.3230	16.1641	26.74	30.96	10.43
	(002)	0.2609	17.5362	33.33		
	(101)	0.2664	18.3996	32.82		
ITO	(100)	0.4721	15.3698	18.22	27.30	13.42
	(002)	0.8787	17.3394	9.89		
	(101)	0.1620	17.8312	53.78		
Si	(100)	0.6248	16.2730	13.83	24.21	17.06
	(002)	0.2338	16.8148	37.06		
	(101)	0.4001	17.5590	21.74		

**Table 2**

Lattice parameter and atomic packing factor of ZnO on different substrates

Substrate	Lattice parameter, (Å)			Atomic packing factor, APF (%)
	a (100)	c (002)	c/a	
Glass	3.2464	5.1719	1.5931	76
PET	3.1938	5.1111	1.6003	76
ITO	3.3546	5.1673	1.5404	78
Silicon	3.1730	5.3236	1.6778	72

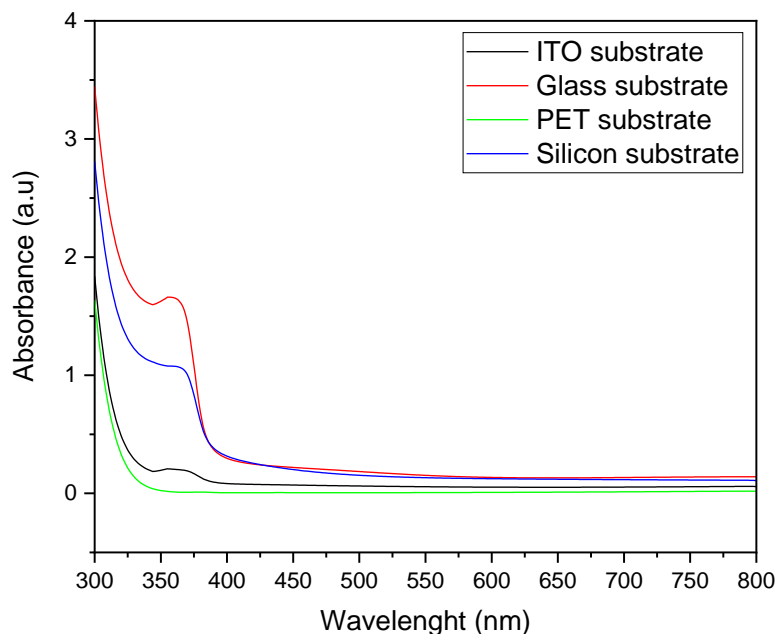
Figure 2 shows the FTIR spectra of the GO/ZnO thin films grown on four different substrates. Various stretching vibrations of the Zn–O band appear in the range of 600-900  $\text{cm}^{-1}$  for GO/ZnO thin films due to different substrates with strong absorption peaks grown on glass and PET substrates [19,20]. The broad peaks centred at 1058 and 1731  $\text{cm}^{-1}$  are features of GO corresponding to C-O bending and C=O stretching vibration, respectively [21,22]. The peak centred at 2956  $\text{cm}^{-1}$  is compatible with C-H stretching vibrations of various types of chemisorbed hydrogen present in all carbon films. The large peak centred at 2915  $\text{cm}^{-1}$  indicates the peak and was assigned to C-H symmetrical stretching for GO/ZnO growth on PET substrates [23].



**Fig. 2.** FTIR spectrum of ZnO:GO thin films deposited on various substrates

The optical properties of GO/ZnO thin films are determined using UV-VIS spectroscopy. Figure 3 shows the absorbance spectrum of GO/ZnO thin films in the wavelength range between 300 to 800 nm for samples grown on glass, ITO, silicon and PET substrates. The GO/ZnO thin films grown on a glass substrate have greater absorption edges (3.30) while GO/ZnO thin films grown on PET show lower absorption properties (1.50) in the ultraviolet range. The greater absorption of ZnO/GO thin

films is attributed to direct electron transitions between the conduction and valence bands [24]. We can see that this absorption edge is located around 379, 385 and 395 nm for GO/ZnO grown on ITO, glass and silicon/PET substrates, respectively. The absorption band edge of GO/ZnO on ITO has shifted to shorter wavelengths. A shift to low wavelengths identified at the absorption edge may be due to band filling called the Burstein–Moss shift and it means higher energy violet light that is good for optoelectronic applications [25].



**Fig. 3.** UV-VIS absorbance of GO/ZnO thin films deposited on various substrates

The Raman spectrum of GO/ZnO thin films deposited on various substrates is shown in Figure 4. The spectrum of GO/ZnO thin films on the glass substrate shows a peak at  $332\text{ cm}^{-1}$  that corresponds to zone-boundary phonons of hexagonal ZnO and a broad peak spectrum at  $437\text{ cm}^{-1}$  that belongs to the typical E<sub>2</sub> mode (High) or wurtzite type phase of ZnO. The peak at  $583\text{ cm}^{-1}$  appears due to E<sub>1</sub> mode (Low) mainly due to defects (oxygen deficiency) present in ZnO. The GO/ZnO thin films grown on ITO substrate exhibit a high peak at about  $437\text{ cm}^{-1}$ , which is attributed to the E<sub>2</sub> mode (High) due to the vibration of the oxygen atoms. Moreover, the GO/ZnO grown on PET substrate was found in E<sub>1</sub> mode (Low) at  $630.17\text{ cm}^{-1}$ , but there is no value was found in E<sub>2</sub> mode (High). Raman spectra are unstable to structural disorder, crystallisation, and defects in micro and nanostructures due to these phenomena [26]. Next, the value of the GO/ZnO thin films on Si found a broad peak at  $520.48\text{ cm}^{-1}$  indicating E<sub>1</sub> mode (Low).

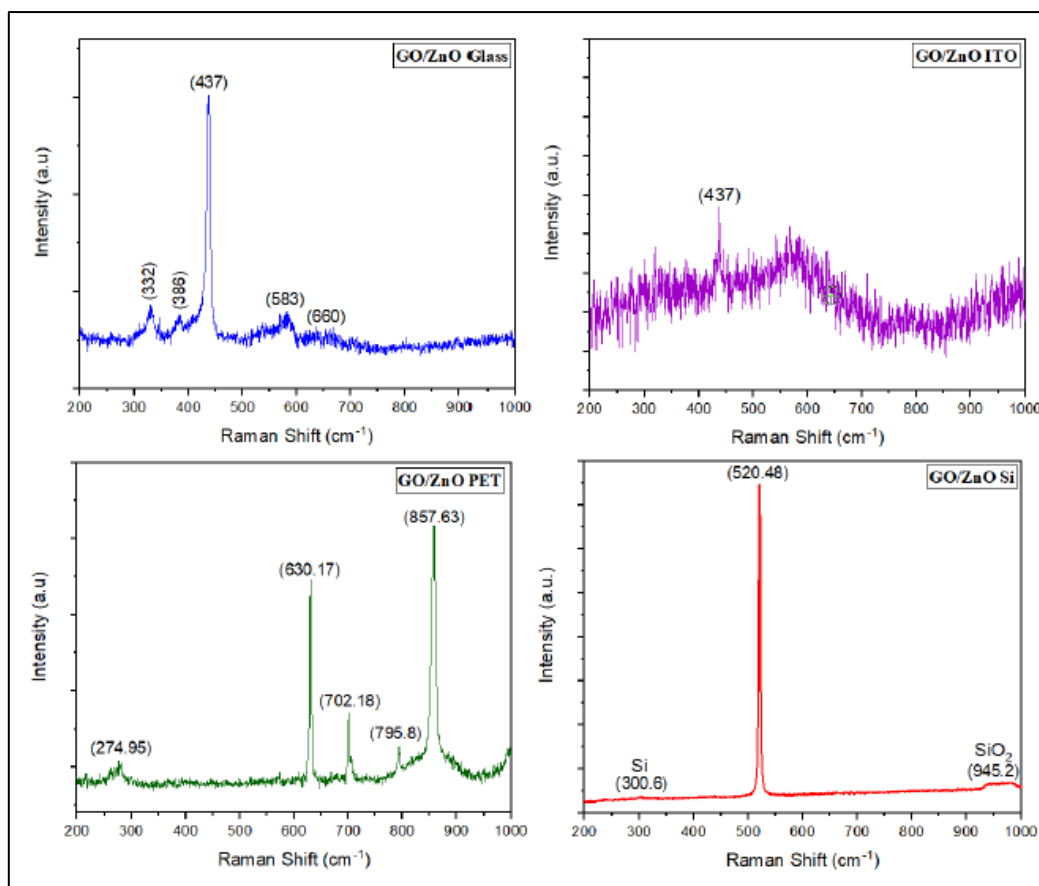


Fig. 4. Raman spectrum of ZnO:GO thin films deposited on various substrates

#### 4. Conclusions

In summary, the GO/ZnO thin films have successfully synthesized on various substrates by solution immersion method. The XRD pattern shows three dominant peaks (100), (002) and (101) planes appeared for GO/ZnO thin films grown on all substrates. Based on the XRD pattern of the (002) plane, the peak position shift maybe caused by lattice parameter change that contributes by the strain and stress of different substrates. The absorption properties in the UV range show the highest absorbance of GO/ZnO thin film that grew on the glass substrate, while the lowest absorbance sample that grew on the PET substrate. The FTIR results showed that GO/ZnO deposited on glass, silicon, ITO substrates deliver the same broad peak centred at  $1731\text{ cm}^{-1}$  which is known as the GO peak that is attributed to the C=O stretching vibrations while PET substrate, the broad peak was found to be at  $2915\text{ cm}^{-1}$  that indicates the GO/ZnO peak and was assigned to C-H symmetrical stretching. These findings indicate that all samples successfully showed C-O, C=O and C-H stretching vibrations that are known as GO peaks. Raman scattering findings observed that all the samples have a high crystalline quality of GO/ZnO thin films. Raman spectra also detected the existence of Raman-active modes for all samples. These findings indicate that the addition of GO will affect the structural and optical properties of ZnO structures.

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