Three-photon Absorption in Nanocrystalline ZnO under Low Intensity Laser

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Abstract

The three-photon absorption in nanostructure wide-band ZnO semiconductor material is observed under low intensity nanosecond Nd-YAG laser of 1.06 μm wavelength excitation. The measured nonlinear absorption coefficient was $0.058cm^3/Gwatt^2$ which is about two order of magnitude higher than its value in solid. The Z-scan measurements show that the threshold laser pumping intensity required to generate the 3photon absorption is very low compared to that for solid. The developed mathematical relation helps in the determination of the nonlinear coefficients from the direct measurements of the emitted fluorescent. The observation of the three-photon absorption under low intensity in nanocrystalline material attributed to the resonance between the $3\hbar\omega_{Yag}$ and $\hbar\omega_{flu.}$ which is simply achieved in nanostructure.

Keywords: Multiphoton processes, ZnO nanocrystalline, Nonlinear optics, Three photon absorption

1. Introduction

The ZnO nano structures have many applications in gas sensors, UV detector and solar cells Yanwu Zhu et.al (2006). Ramanathan. et.al (2005), Crissy et.al. (2008). This material has some additional advantages compared to other large band-gap semiconductors; for example, its large exciton binding energy (about 60 meV) which is three times the binding energies of ZnSe and GaN Hua (2008). This allows a stable exciton distribution and achieves efficient excitonic emission at room temperature. The optical and electrical properties of ZnO nanostructures are studied at different preparation techniques and at different substrate materials Sato et.al. (2005), Shubra et.al. (2007).

The pumping of ZnO crystalline film with near-infrared femtosecond radiation pulses, enhances the nonlinear interaction between the ultrahigh intensity applied optical field and the ZnO nanostructures Özgür et.al (2005), Ja-Hon et.al (2005). The nonlinear interaction leads to the simultaneous absorption of two or more photons of subbandgap energy. The absorption is through a virtual-state that assists the inter band transitions. This transition produces electron-hole pairs in the excited states and, subsequently, the band-edge emission via their radiative

recombination Bentley et.al (2007).The two photon absorption (2PA) in semiconductor nanocrystals (NCs) has been widely investigated Van Stryland (1993), Ja-Hon et.al. (2005) while the research effort on their three – photon absorption (3PA) is limited Jun et.al (2006).The three photon absorption was observed in ZnO and ZnS crystals at laser of ultra excitation irradiance which was more than 40 GW/cm^2 Bing et.al (2008), Shaul et.al (2008), Penzkofer. and Falkenstein (1976).

In this work the three photon absorption in ZnO nanostructure illuminated by low power (<5 GWatt/cm^2) Q-switch Nd:YAG laser is observed. The work concentrates on the effect of the nanostructure on the nonlinear parameters through the studying of the 3PA coefficient and the laser threshold pumping power. Simple mathematical relations are developed to describe the dependence of the fluorescent emission on the pumping laser intensity. The three photon absorption coefficient was estimated from the experimental measurements.

2. Experimental work

The ZnO nanofilms were prepared by chemical spray pyrolysis technique. The films were deposited on quartz substrates heated to 400C^o. The spray solution is prepared by mixing Zinc acetate {Zn $(CH3COO)_2.2H_2O$}, isopropyl alcohol with purity 95% and distillated water in a volume ratio 3: 1 at (0.2 M).The above mixture solution was placed in the flask of the atomizer and spread by controlled nitrogen gas flow on the heated substrates. The chemical spray pyrolysis experimental setup is similar to the standard one Islam and Podder (2009). The spraying time was controlled by adjustable solenoid valve. The heated substrate was left for 12sec after each spraying run to give time for the deposited (ZnO) layer to be dry. In order to get a film of proper thickness many layers deposited of ZnO are required. The optimum experimental condition for obtaining homogeneous ZnO thin film at 400C^o are determined by the spraying time, the drying time and the flashing gas pressure.

The crystalline structure of the ZnO film was studied by X-Ray diffraction using XRD-6000-Shemadzu system. The absorption and transmission spectra of the sample are recorded using Shemadzu UV-160/UV-Visible recorder spectrophotometer. The photoluminescence spectrum (PL) was obtained by SL 174 spectrofluorometer covering a rangy of (300-900) nm. The Hall measurements were carried at using ECOPIA type (HMS-3000VER3.5) system with Magnetic field of 0.55 Tasla.

Passively Q-switch Nd: YAG laser of variable energy up to 50 mJ and 12 ns pulse duration was used to pump the ZnO nano film. The beam was focused by a lens to (0.2mm) focal spot size. This lead to a laser power density of about 4 Gwatt/cm^2 on the ZnO film. The experimental setup for measuring the nonlinear coefficient is shown in Fig. 1.

In this setup the power of the laser was adjusted by a control unit, D_1 is a detection unit to measure the laser input power, L_1 is 10X objective len`s, L_2 is plane convex lens of 10 cm focal length and F is a low pass filter of cut – off frequency at 650nm. The Digital CCD camera was used to image the visible florescence radiation emitted from the pumped ZnO film, whereas the detection unit D_2 was used to detect the visible florescence pulses power.

3. Results and discussion

3.1 Structure and optical properties

The X-ray diffraction pattern of a ZnO film of 500nm thickness is illustrated in Fig. 2. The spectrum indicates that the ZnO film is a polycrystalline structure. The grains are highly oriented along the (002) direction implying that the crystal orientation is mostly along the c-axis perpendicular to the substrate surface.

The optical properties of ZnO films which were prepared on quartz substrates have been studied in this work. The absorption, transmission, and the photoluminescence (PL) Spectra of the ZnO film in the spectral range (200-1100 nm) are shown in Figs. 3, 4 and 5. The absorption spectrum shows low absorbance in the visible and infrared regions; however, the absorption in the ultraviolet region is high. Referring to the data extracted from the absorption spectrum in Fig. 3, the absorption coefficient (α) was calculated as a function of wavelength. Assuming allowed transition; the dependence of $(\alpha h\upsilon)^2$ on (hυ) is plotted as in Fig. 5. The extrapolation of the linear part of the plot $(\alpha h\upsilon)^2=0$, gives rise on estimation of the energy gap value of the ZnO film. The value of the energy gap was found to be about 3.28eV. This value was in a good agreement with values mentioned by other works Suhail et.al (2009).

It was noticed through this work that the energy gap of the ZnO films decreases with the increasing of the film thickness from 400 nm – 1200 nm. The decease of the band gap with increasing the film thickness implies that the ZnO sample is an n-type semiconductor. This decrease of the band gap may be related to the presence of

unstructured defects. The existence of the this defect increases the density of localized states in the band gap and consequently decrease the energy gap Islam and Podder (2009).

The optical transmittance spectrum of the ZnO film is shown in Fig. 4. It can be noticed from this Fig. that the transmittance is high in the visible and infrared regions. The oscillation appears at the saturation region of the spectrum is attributed to the interference between the reflected light from the film surface and the substrate. This oscillation was used in calculation of the refractive index which was found to be about (1.95) using the envelope theory. The optical fluorescence spectrum of the ZnO film illuminated by 320nm UV line is shown in Fig. 6.

The spectrum displays two major luminance peaks at 380 nm and around 556nm. The first peak is due to the energy gap transmission which corresponds to 3.28eV. The second peak is due to the excitonic emission; and it is in a good agreement with the results measured by many other authors Yim and Lee (2006), Xu et.al (2008).

The broad green emission peak that is dominated at 556nm (≈2.23 eV) is a good evidence for the exciton formation in ZnO with a binding energy of 60meV. The high binding energy enables the finding of the exciton at room temperature. The intensity at the 556nm peak is higher than that found around 380nm peak. This is because the band – to – band transition was quenched by the defect states. The same behavior was observed by Wang et.al (2008). The quenching of the band – to –band transition by the surface states was observed in CdS samples illuminated by 320nm UV line Suhail (2010).

The morphology of the ZnO film surface was imaged by an AFM, and illustrated in Fig. 7. This figure shows the formation of the nanostructure which helps in lowering the threshold power required to inducing the nonlinear effect in the ZnO material. The surface roughness may attribute to the clustering of the grains in the films prepared under high ambient pressure Antaryami and Thareja (2008), Mitra et.al. (2001), Anirban and Thareja (2001),

3.2 Nonlinear dynamics

The illuminated ZnO nanofilm by the short pulse Nd: YAG laser of 1.06 μm wavelength emits bluish purples pulses. The blush purple colored pulses were registered by a CCD camera and detected by filtered detection unit. The registered photos of the CCD camera were processed by special software similar to LIMO software to simulate the profile of the emitted pulse and its spectral distribution Preasanth (2006). The real spectrum of the emitted fluorescence was plotted by Jarral- Ash grating 75- 150 one meter Czerny-Turner spectrometer as shown in Fig. 8.

The emitting of this colored pulses are attributed to the three photon absorption. It can be noticed from this figure that the peak of the fluorescent radiation is around 395nm and spread by 20nm around the maximum wavelength, whereas the second excitonic peak was not registered. The appearance of the only band-to band photoluminescence emission when the ZnO nano film is illuminated by the high intensity short pulse Nd:YAG laser of wavelength 1.06μm, may be attributed to the nonlinear interaction between the high intensity radiation and the ZnO nano material. This interaction may alter the transition probability which make some of the allowed transition to be forbidden in the material under the influence of high intensity radiation. Thus the transition between the excited surface states (S.S) and the valance band may become forbidden. Whereas the two transitions (band- to band (b-b.T) and the excitonic photoluminescence) were appear under the low power UV line pumping. The schematic diagram for the photodynamic under one and three photon excitation is shown in Fig. 9.

The energy gap of the ZnO sample which was about 3.28eV, whereas the 3ħω of Nd: YAG laser is around 3.51eV. This difference in the energy shows that the energy gap and the energy of the three absorbed infrared photons are not in resonance. However, the pumping of ZnO by ultra intense laser beam makes the transition between the sub band below the top of the valance band and the bottom of the conduction band is allowed. This give rise to the resonance between the $3h\upsilon_{Nd:\ YAG}$ and the $h\upsilon_{ZnO}$. This resonance is highly increasing the absorption cross section of the Nd: YAG laser by the ZnO nanofilm. The transition from the deep- lying valance states was observed in the 2PA in ZnO Balltrameyunas et al. (1983). This resonance enhances the observation of 3PA in nanocrystalline ZnO films illuminated by low power density Nd-Yag laser of 1.06 μm wavelength.

3.3 The rate equations model

The rate equations describing the transitions between the valance band and the conduction band in our semiconductor sample can be written as:

$$\frac{dN_h}{dt} = -\frac{\alpha I^3}{3\hbar\omega} + \frac{N_e}{\tau_i} \tag{1}$$

$$\frac{dN_e}{dt} = \frac{\alpha I^3}{3\hbar\omega} - \frac{N_e}{\tau_i} \tag{2}$$

Where N_h and N_e are the population in the valance band and conduction band respectively. α is the three photon absorption coefficient, ħω is the energy of the IR pump photon Nd-YAG laser of 1.06 μm, I is the laser intensity at the sample position and τ_i is the life time of the excited level in the conduction band. The Gaussian laser beam intensity is given by:

$$I = I_0\left[\frac{\omega_0^2}{\omega^2(Z)}\right]\exp\left[-\frac{2r^2}{\omega^2}\right]\exp\left[-\frac{t^2}{\tau_p^2}\right] \tag{3}$$

where I_0 is the laser intensity at the west of the beam and τ_p is the laser pulse duration. Since the measuring intensity was at the focal point, in the Z-coordinate, thus the intensity in Eq 3 reduced to $I=I_0$.

In order to find the relation between the power of emitted fluorescent pulse and the intensity of the pumped laser (Nd-YAG), Eq 2 can be arranged as:

$$\frac{dN_e}{dt} + \frac{N_e}{\tau_i} = \frac{\alpha I_0^3}{3\hbar\omega} \tag{4}$$

The solution of Eq 4 can be written as:

$$N_e = \frac{\alpha.\tau_i.I_0^3}{3\hbar\omega}\left[1 - e^{-\frac{\tau_p}{\tau_i}}\right] \tag{5}$$

Since the pulse duration of the pump laser (t_p) is in nanosecond and the life time of the excited state (τ_i) in picoseconds Jun et.al (2005), the Eq 4 can be simplified as:

$$N_e = \frac{\alpha\tau_p}{3\hbar\omega}I_0^3 \tag{6}$$

Multiply both sides of the above equation by the volume (V) of the illuminated part of the ZnO sample and arranged the terms, Eq 6 can be written as:

$$\frac{3\hbar\omega.N_e.V}{\tau_p} = p_f = \alpha.V.I_0^3 \tag{7}$$

where P_f is the power of the emitted fluorescent (blush purple pulses) from the ZnO sample pumped by the Q-switch Nd:YAG laser. Introducing the quantum yield which include the probability of the exciton self-trapping and the efficiency of the detection unit, Eq 7 can be written as:

$$p_f = \eta\alpha V I_0^3 \tag{8}$$

In our experimental setting described before, the focal spot area was about (0.2 mm^2) and the ZnO film has 500nm thickness, thus the illuminated volume is about 5×10^{-7} cm^3 and n estimated to by 10^{-5} in our experimental setup.

3.4 Nonlinear absorption

The bluish purple florescence emission was observed by human eye and it was register by CCD camera when the intense Nd-YAG laser of 1.06 μm wavelength radiation was incident on the sample. The characteristic feature of

the 3PA induced fluorescence is the cubic dependence of the fluorescent intensity on incident laser radiation as described by equation (8). The relation between the pumping laser intensity I_0 and the emitted fluorescence power P_f is shown in Fig. 10.

The up conversion fluorescence power measured at maximum emission wavelength at 390nm, exhibits a cubic dependence on the incident laser intensity. The best –fitted exponent of the graph in Fig. 9 is n=2.6 through the relation $y=mx^n$ as is characteristic of three photon processes. The inset figure shows the logarithmic relation between the fluorescence emission and the IR incident radiation. The dependence is linear which give more evidence to the power relation between P_f and the IR incident radiation.

The value of (α) is found from the measurements of P_f at different values of I_0, and by applying equation (8) it was found to by about of 0.058cm3/ $GWatt^2$. This value of α is tow order of magnitude higher than that registered in the bulk Bing et.al (2008), Jun et.al (2005).

The two photon absorption was observed in ZnO nanowire pumped by low power density infrared laser Chunfeng et.al (2009), whereas the observation of the three photon absorption in ZnO at low pumping irradiance (≤5 Gwatt/cm2) was not mentioned before. The observation of 3PA at such low power density may by attributed to the high density nano spikes formation on the surface of the ZnO film prepared by this techniques. The variation of the 3PA coefficient with the Q-dot diameter of CdS was observed by Bentley et.al (2007)

The dependence of the 3PA coefficient on the nanostructure dimensions and the threshold of the laser pump intensity can be observed from the open aperture OA Z- scan measurements. The typical OA Z- scan curves for both the ZnO nanofilm and a bulk sample are illustrated in Fig. 11. The surface roughness mean square (rms) of the nanofilm estimated by AFM measurements was in the range of (50 – 100) nm as shown in figure (AFM), whereas the bulk sample was a disc of 5mm diameter and 0.5 mm thickness. It can be noticed from this figure that the three – photon absorption and the laser pump intensity required to generate the nonlinearity are nanostructure dependence.

In nanotechnology the film surface playing the role in the determination of the optical properties of the material. This is because the surface state localization in the energy gap is a function of the nano crystallite dimension in nanofilm Suhail et.al (2010) The existences of the surface states in nanostructure enhance the three photon absorption by simplify the transitions of the carrier through the energy gap. Thus the observation of 2PAor 3PA in nanostructure at low power laser irradiance is possible. Whereas in solid it required a high laser power density. Wang et al explain the observation of the 3PA in ZnO samples to the two – photon absorption followed by an additional one photon absorption by the help of the deep donor levels formed in ZnO as a surface states Vivas et.al (2010).

Another reason for the observation of the 3PA at low pumping power is the optical properties of the ZnO material. The ZnO has high optical nonlinearity which is due to the unequal atomic size of Zn and O atoms Preasanth et.al (2006) The eccentrically located bond change contributed to the homopolar energy gap. Since the nonlinear susceptibility is more sensitive to the crystal potential, ZnO is focused to be higher nonlinear.

Referring to the high optical nonlinearity of the ZnO material and the high density nanostructures on the prepared film surface. The nonlinear absorption coefficient increased leading to the appearance of the 3PA at low laser pumping intensity.

4. Conclusion

The 3PA has been observed in ZnO nanofilm prepared by chemical spray pyrolysis technique, illuminated by low intensity nanosecond laser excitation of 1.21eV photon energy. The 3PA absorption coefficient found to be 1-2 order of magnitude higher than the value in solid, this indicate that the nanostructure enhance the nonlinear interaction in the ZnO material. The simple developed relation between the fluorescent power and the intensity of the incident light was efficient in determination of the nonlinear absorption coefficient.

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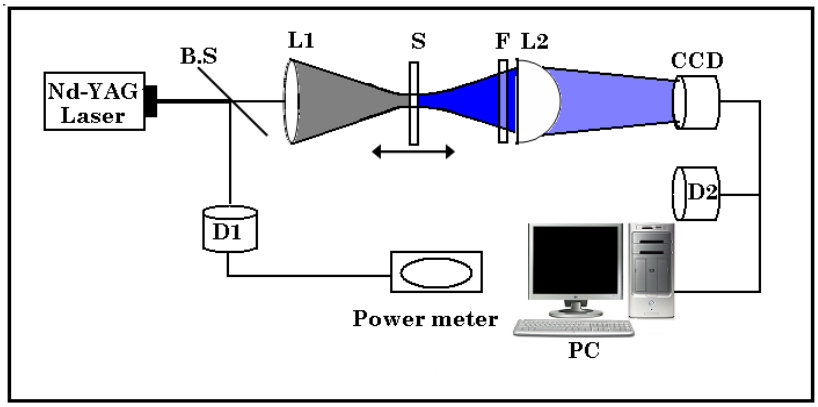

Figure 1. The experimental setup to measure the nonlinear absorption coefficient; $L_1$and L_2 are lances. S is the sample, F is the low pass filter, $D_1$and D_2 are detectors

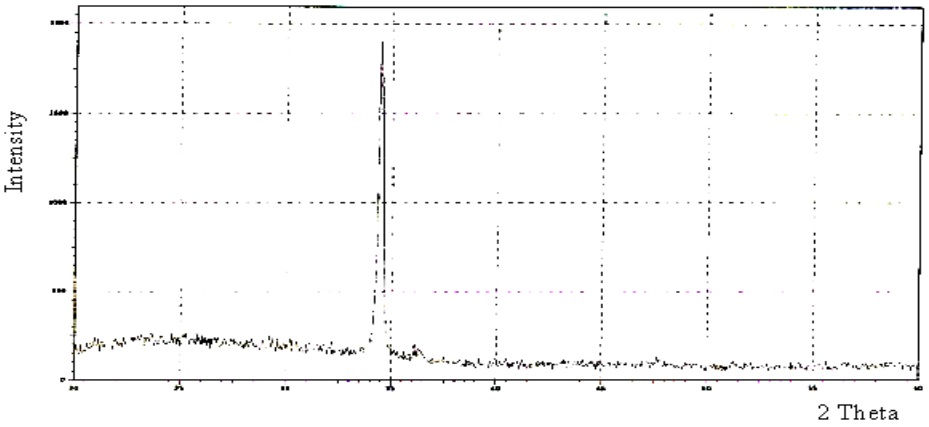

Figure 2. X-ray diffraction for ZnO thin film on a quartz substrate

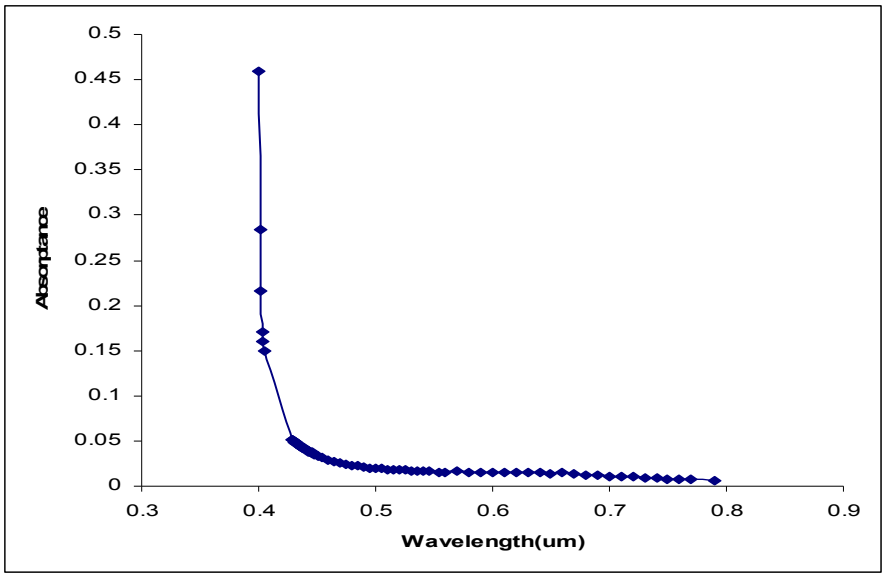

Figure 3. Absorption spectrum of ZnO thin film

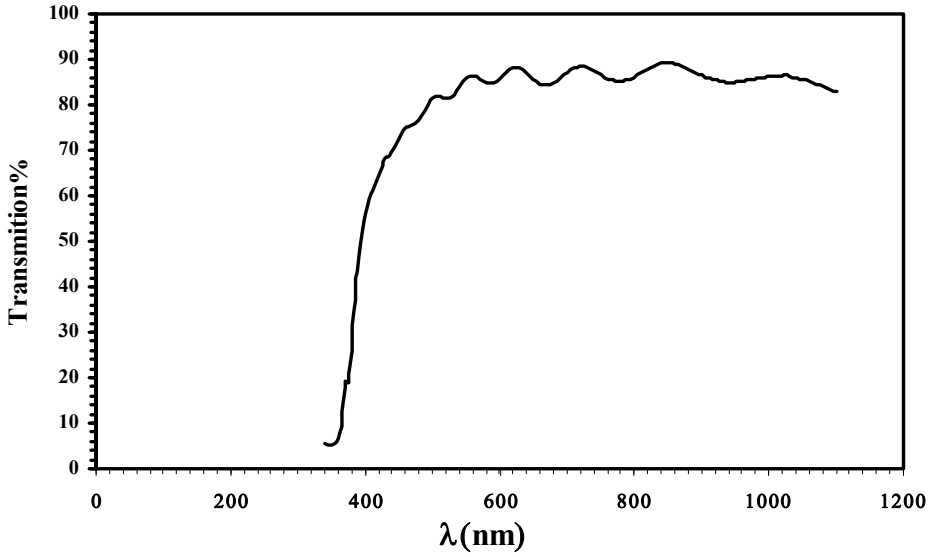

Figure 4. Transmission spectrum of ZnO thin film

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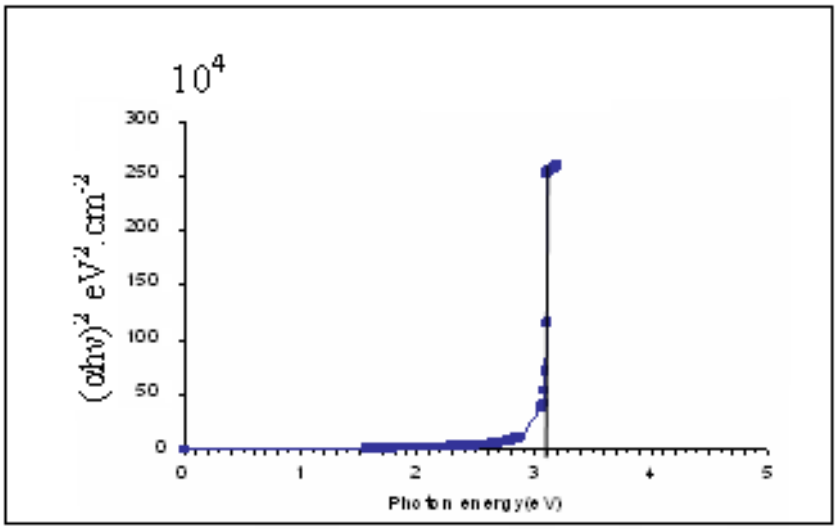

Figure 5. plot of $(\alpha h\upsilon)^{1/2}$ vs. Photon energy ($h\upsilon$) for ZnO thin film

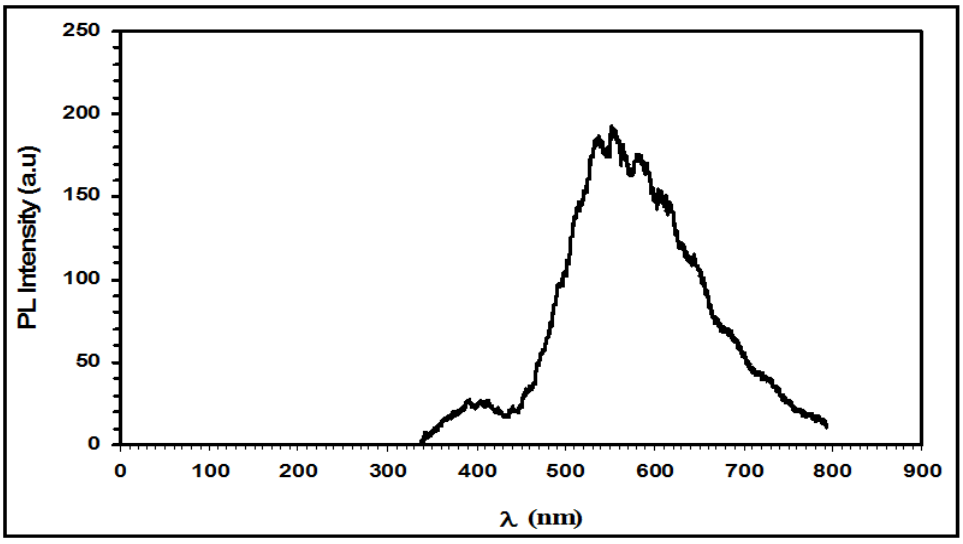

Figure 6. Photoluminescence spectrum of ZnO thin film on quartz substrate

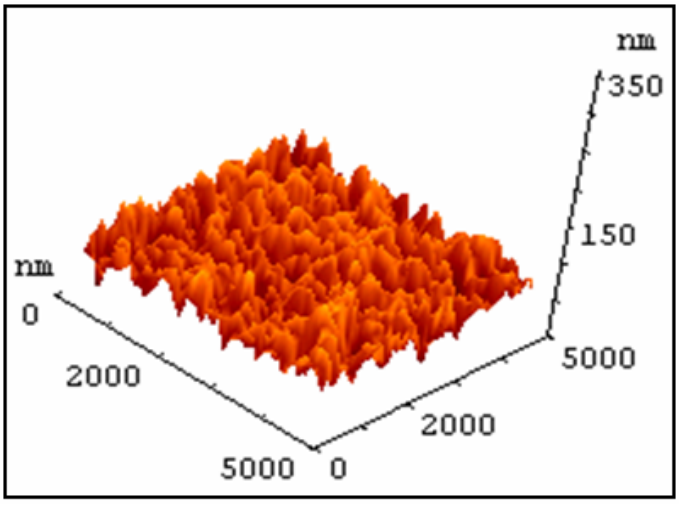

Figure 7. AFM image of ZnO nanofilm shows the surface morphology of the film deposited on glass substrate

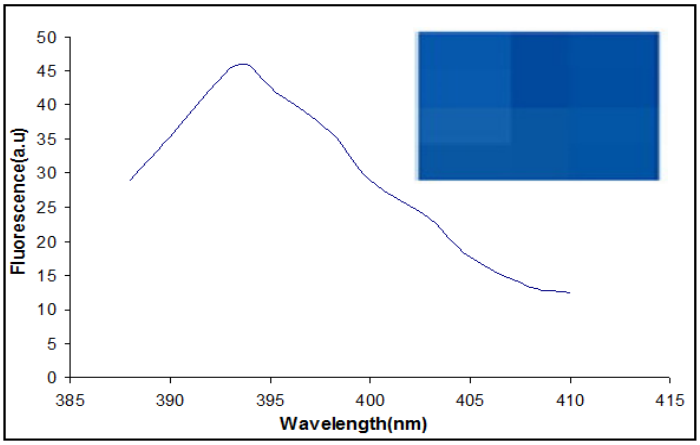

Figure 8. Spectral distribution of the emitted fluorescence pulse by Jaral-Ash spectrometer the inset figure represents the CCD image of the emitted fluorescence

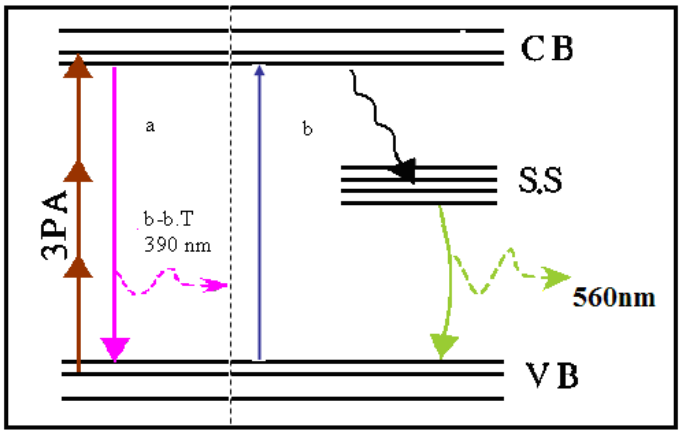

Figure 9. Schematic diagram for photodynamic of one and three photon absorption
(a) high intensity short pulse IR pumping (3PA), (b)low intensity CW ultraviolet pumping (1PA)

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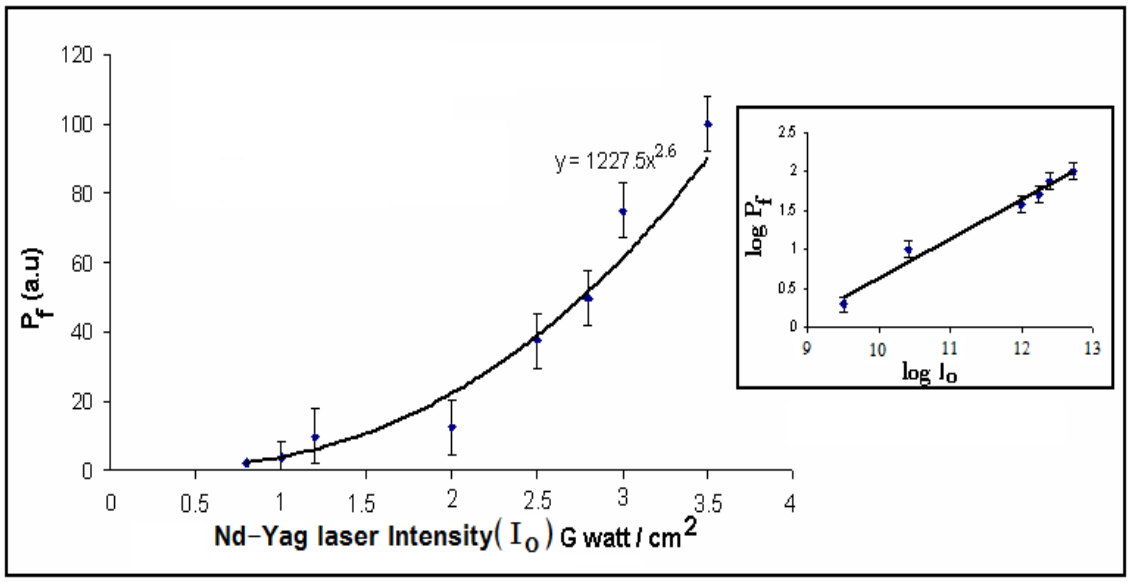

Figure 10. The relation between the laser pumping intensity and the out put fluorescence emitted power; the inset shows the logarithmic dependence of the fluorescence intensity on the incident IR radiation

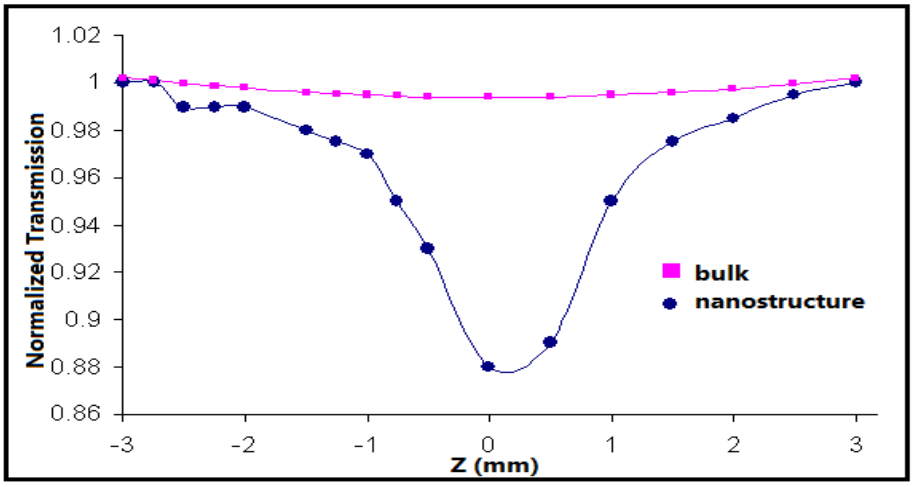

Figure 11. Open- aperture Z- scan with 1064 nm laser pulses of 4 G.Watt/ cm^2 irradiance (I_o) for bulk ZnO sample and nanofilm sample