A review of Pure and Doped ZnO Nanostructure Production and its Optical Properties Using Pulsed Laser Deposition Technique
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Abstract: In this review, the theoretical and experimental aspects of ZnO nanostructures production using pulsed laser deposition techniques were presented. It reviewed the work principles of pulse laser deposition technique (PLD) method, physical procedures such as ablation, and plasma plume creation accompanying the deposition of pure and doped ZnO from target to substrate material. Many ways of deposition and elements that affecting on the properties of thin films like the temperature of substrate, laser fluence (laser energy density), pulse repetition rate, pressure of oxygen in chamber, time of deposition process and post growth annealing which modify the deposition active factors like nucleation, and crystallization.

Keywords: Nanostructures, Zinc oxide, Pulsed laser deposition, Wurtzite, Nanocrystalline

1. Introduction
The term or word "nano" derived from the Greek word "dwarf", and applied at the molecular level in the field of engineering and industry. A comprehensive definition of the word nano where the preparation, modification and measurement represent values of less than 100 nanometers. Nanotechnology is involved in various disciplines that rely mainly on basic sciences, such as physics, chemistry, materials science, and all kinds of engineering disciplines and molecular analytical methodologies[1]. The genesis of most material properties like optical, mechanical, magnetic, and chemical lies in the states of electrons in the constituting atoms, their conformation in the structure of atomic and the manner of these electrons form bonds with the adjacent atoms in a molecular system. These electrons experience ‘confinement effects’ when those are trapped in a nanometer sized structure of the material. It is thanks to these ‘confinement effects’ that the material characteristics are unusual and generally size dependent.

A large number of books [2-5], review articles [5-8], and research papers [8-12] published on the subject of nanostructured semiconductors and related materials. Most of the reported papers are devoted to confinement effect, applications in the area of biotechnology, lasers, electronic devices and solar energy [13-17]. Some of these papers are also devoted to growth and characterization of nanostructured semiconductor. A semiconductor structure having at least one dimension in the range of one to few hundred nanometers defined as a nanostructured semiconductor. Semiconductor structure with reduced dimensionality has, amongst others, Optical characteristics that are completely different from them of crystalline bulk matter. For example, the numerical value of a silicon element's band gap was about 1.12 eV in the bulk crystalline state whereas nanocrystalline dots of silicon have “band gaps” of ≈2eV or more depending on the size. Thus, semiconductor nanostructure showed size dependent properties different from those of a macroscopic semiconductor, if one or more dimensions of the structure are comparable to wavelength of light or wavelength of electrons and holes. The quantum effects arise in systems, which confine electrons to regions comparable to their de Broglie
wavelength. In other words if the physical size of a material structure becomes smaller, the quantum mechanical effects become observable. In the following sections, the confinement effect and its implications on allowed energy bands and densities of states will be discussed [18-23].

In this review, the work will be limited to prepared thin films nanostructure semiconductors using a very specific methodology of Pulsed Laser Deposition (PLD). Some of the novel physical optical, structural, morphology and photo-luminance properties of these PLD grown nanostructures films also present and discussed in this review.

1.1. Fundamental of Pulsed Laser Deposition
Although the technology of laser evaporation employed in the 1960s for the first time to prepare various types of thin films [24], pulsed laser deposition (PLD) uses high-power laser pulses with an energy density of more than \(10^{8}\text{W.cm}^{-2}\) to melt, evaporate, excite, and ionize material from a single target. When using an energy level laser sufficient to ablate the target, the material will evaporate and a plasma plume will form. This plume of plasma spreads along the direction of the target surface.

Resulting a thin layer to form through vaporizing this material on the substrate sited in front of the target. The described PLD procedure can principally be divided into four regimes [25]:

- The evaporation regime or the interaction of the laser beam with the target resulting in evaporation of the surface layers.
- The isothermal regime or interaction of the laser beam with the evaporated materials causing the formation of isothermal expanding plasma.
- The anisotropic three-dimensional adiabatic expansion of the laser-induced plasma with a rapid transfer of thermal energy of the species in the plasma into kinetic energy.
- Thin film growth.

The absence of a theoretical model to describe the entire PLD process was due to the close association between the physical mechanisms involved in this technique, which made the full process description not possible. Yet, there was a possibility to analyze this process into parts (evaporation, plasma formation, plasma expansion and its growth in vacuum or in existence of an ambient gas). Where, the details analyzed each part accurately and then connects all the parts together to produce a complete process description[26]. So far, the mechanism of plasma expansion had not been understood (when it spreads in the vacuum and when it spreads in the presence of gas in deposition environment) as the plasma expansion process is considered one of the most important processes in the technique of pulsed laser deposition. The expansion of plasma in the presence of gas is a complex process as it used in the deposition of complex oxides. The physical processes affect the behavior of the plasma plume expansion in terms of its spread and heating speed and the shock waves and other additional processes that have emerged as a result of the interaction of the plasma with the gas environment [27].

1.2. Historical Review
Despite an early successful demonstration of efficacy of the PLD, the initial progress was rather slow and even stagnant till 1980s. During this period, the deposition of semiconductor thin films was carried out using PLD only with limited success.

In 1995 Craciun et al. [28], formed transparent, high electric conductive, and c-axis oriented ZnO films by PLD (pulsed laser deposition) technique on silicon wafer and glass substrates work either a KrF excimer laser with wavelength of 248 nm or a frequency doubled Neodymium YAG laser (Nd:YAG laser) with wavelength about 532 nm. They observed that FWHM value about 0.16° for C-axis of (002) orientation films, surface structure was flat while fractured surface was periodically columnar structures. The optical transmission showed a significant value in visible region which is higher than 85%.
In 1999 Sun et al. [29], prepared undoped Zinc oxide (ZnO) films via laser ablation technique on different substrates. The studies showed that in X-Ray Diffraction analysis and (SEM) scanning electron microscopy pictures, the oxygen pressure that used for the thin film deposition led to increases level of samples crystallization. All samples showed a rise in luminescence spectra at UV and green - yellow regions.

In 2000 Jin et al. [30], thin-film zinc oxide precipitation on a sapphire layer with a pulsed (001) laser precipitation with oxygen pressures ranging from 50 to 500 metric tons. From increasing the temperature, a strong ultraviolet radiation obtained. Also, a good quality of crystallization was obtained of ZnO films under temperatures above 300 °C of substrate and oxygen pressure of 200 mTorr.

In 2002 Zeng et al. [31], ZnO oriented C-axis films have been deposited on quartz and (001) Si substrates by PLD technology. Several parameters such as laser fluency, substrate temperature, and oxygen pressure studied. Laser fluency and oxygen pressure act as very active factors that cause film defects to disappear. While the increase in substrate temperature improved the quality of ZnO films.

In 2004 Liu et al. [32], ZnO (GZO) films that oriented heavily on the C-axis on sapphire substrates (0001) were grown by a pulsed laser deposition method (PLD). The photo luminosity spectra (PL) proved that Ga atoms have a significant influence on the luminance properties of ZnO deposited films. PL spectra show GZO films include near-band edge emissions (NBE) and deep orange emissions. The NBE emission converts to a higher energy region and its intensity decrease with increasing concentration. Ga.

In 2006 Rusop et al. [33], using PLD technology, researchers set up ZnO films on a silicon substrate across varying annealing temperatures. At 600 °C annealing temperature resulted in a high transmittance value of approximately 85% with a sharp absorption edge. At the same annealing temperature, there is a decrease in the value of electrical resistance.

In 2007 Raid et al. [34], authors used the Nd: YAG laser ablation process to prepare ZnO films (that are doesn’t doped) on glass substrates in the presence of oxygen but without any agents or heat treatment. The electrical resistivity values were about 0.27 Ω.cm, the mean optical transmittance values, and the optical band gap reached 80%, 3.25 volts respectively.

In 2007 Ramamoorthy et al. [35], various films were used as a substrates to grown by a thin and very transparent zinc oxide, without adding any dopants and annealing via pulsed laser deposition (PLD) technique and used ZnO pellets (Johnson Matthey). The optical transmission window showed transmission [32] ratio of 95% which found it is broader than another conducting transparent oxides. Prepared films acts as a coating surfaces of a highly antireflective.

In 2009 McLoughlin et al. [36], fundamental frequency of Nd-YAG laser and its frequency tripled was used with a fluence on target of 7.7 J/cm² with vacuum 10⁻⁴ Pa. The results showed that the preferential cauterization of the material into certain mass and there exists a correspondence among mass existence in the plume and the deposition of nano-structures.

In 2012 Youssif et al. [37], studied and investigated the polycrystalline ZnO:Co films which deposited on glass substrates by PLD technique employ Nd-YAG laser (pulsed mode) and wavelength about 532 nm, duration 7 ns, and energy fluence of1.4 J/cm² with different doping value. The pure ZnO and ZnO: Co films sensors offer a perfect commensurable response to methanol. The sensors with higher resistance give a higher response to the gas.

In 2014 Serhani et al. [38] c-axis oriented ZnO were grown-up on silicon aluminum and platinum substrates by PLD technique. The improved growing temperature is 300 °C for ZnO on Al and Pt, and 400 °C on Silicon. The piezoelectric ZnO film displays a fair electro mechanical coupling factor.

In 2016 Al-Douri et al. [39], nanostructures of Co-doped ZnO were deposited on a glass substrate employed PLD technique. The morphological properties were strongly dependent on the influence of the laser. SEM study shows that the grain size grows with increasing doping concentrations of Co the studied bulk modulus for Co-doped and undoped ZnO films of various doping concentrations are the hardest at 3% in parallel with the larger grain size of the same values of concentration.
In 2017 Nasser et al. [40], ZnO doped with silver nanoparticles (AgNPS) were deposited onto polypropylene carbonate (PPC) substrate with various concentrations of silver with employed the pulsed-laser deposition technique. AFM analysis appears that the surface of films turned out rougher when the ZnO concentration value was rising.

In 2018 Haidar et al. [41], used Pulsed Laser Deposition to prepared ZnO film mixed with 0-0.3 wt% Mg doping and deposited on Glass substrate. The parameters used are 2nd- Harmonic Nd:YAG laser, wavelength 532 nm. The optical properties were studied by examining absorption and transmittance measurements. Transmission tests showed value of 85% while absorption showed a shift toward a blue wavelengths when the magnesium concentration increased.

In 2019 Novotný et al. [42], ZnO:Eu thin films well deposited by PLD technique at room temperature. Films showed a wurtzite structure. Emission of Eu$^{2+}$ and Eu$^{3+}$ detected by indirect excitation. XPS exposed the proportion of Eu$^{2+}$/Eu$^{3+}$ various in the range of 27–35 % / 65–73%.

In 2020 Suhail et al. [43], Rhombohedral structure of thin-film chromium oxide (Cr$_2$O$_3$) doped hexagonal zinc oxide (ZnO) nanoparticles have been prepared via pulsed laser deposition technique at a various weight percent of ZnO from 0 to 9 wt%. An X-ray diffraction study showed that the films prepared were polycrystalline. The value of optical band gap ranged between 2.45 and 2.68 eV. I.e. Red-shift in wavelength was occurred. It has been observed that with an increase in the concentration of dopants, sensitivity have also increased.

2. Experimental Set-up and Methodology

Any Physical vapor deposition method must have three essential constituents: a source material, a substrate and an energy fund to transport material from the source to the substrate for the duration of the deposition of a material structure. Pulse laser technology requirements for evaporation considered as the short duration and high-energy pulse laser source. The reaction takes place in a very short period, but it is a series of complex and sequential operations in order to ablation the target material. The materials used for vaporization in the laser evaporation technique contain energetic neutral, electrons and ions. The materials that mentioned before will represent by the plume that will expand with a high rate and condense on the substrate whose temperature was high. Thus, thin films will grow on this substrate. Figure 1 displayed the PLD diagram set up. The deposition chamber was cylindrical in shape, and this chamber designed in a completely free, as the laser deposition process system does not need a very high vacuum.

Then, the Chamber pumped down to a base vacuum of more than 10$^{-3}$ Torr using a suitable rotary Pump with an oil free backing pump and checking the pressure in the cylindrical chamber by the use of Leybold-Heraeus the Pirani gauge. During the deposition process, the vertical direction of the target material carrier is constant throughout the deposition period. The target material was prepared from the powder (the consistent type), by pressing on consistent mixes to form the powder target. A pressed powder represent the targets that successfully used in the deposition process, in addition to sintered pellets, single crystals and metal foils. Another significant factor to be well-thought-out when selecting a powder was the powder purity. Selecting a powder with good purity will lead to a target with slighter impurities. The Glass substrates and quartz substrates both used to be ready for depositing metal oxides, as these substrates cleaned with distilled water to get rid of the residues and impurities suspended in them. The initial cleaning process must followed by a second cleaning process with ultrasound for a period of several seconds, followed by the drying stage of the substrate to be ready for the film to be deposited on it. The pulsed laser is used from outside the chamber to ablate the target material placed in front of the substrate at a set distance utilize lens with converging type of either Quartz or Glass to achieve required energy density i.e. fluency for ablation.
A higher laser energy density obtained either by increasing laser energy or by reducing laser spot area. The growth temperature used in this technique, which is suitable for the substrate loaded on the electric heater, is around 400°C. To ensure regular ablation of the deposition process, the target material must continuously rotated. The distance between the target and the substrate can be a very influencing factor on the energy of the deposited particles and the rate of their precipitation on the substrate. During the film growth phase inside the settling chamber, reactive gases such as O$_2$ and N$_2$O introduced, where the deposition process in such a state was reactive. One of the great advantages of PLD over other thin film fabrication techniques lies in the ease with which the in-situ multilayer structures can be grown. Target carousal capable of holding four or six targets can be used for this purpose and individual targets are ablated sequentially for a predefined time to grow multilayer structures. A schematic diagram of the set-up of laser deposition chamber, given in Figure 1, showed the arrangement of the target and substrate holders inside the chamber with respect to the laser beam.

3. Laser Ablation Mechanisms
In the pulsed laser deposition technique, a high-energy pulsed laser source is used. When the laser beam focused on the target material, the material will be ablated and a layer of dense vapor will form directly in front of the target material at the early phase during the subsequent phase of the laser pulse. The absorption of the target material by the laser energy will lead to a very large increase in the temperature and pressure of the vapor and thus will result in a partial ionization process, then the plasma column will form[44, 45]. Then, the thermal energy of ablated particles changed to kinetic energy about several hundreds of eV. When the expansion of the low-pressure gas occurs and due to the multiple collisions of the ablated particles, that will cause induce in the attenuation of the kinetic energy of the particles. In general, the process of laser removal is divided according to time into two stages [46]:
- Target material evaporation and plasma formation
- Plasma expansion
At higher laser fluence plasma contains a significant amount of highly energetic species. The high energy plum species after colliding with substrate may penetrate the substrate surface and get embedded in it. These immobile atoms then act as additional nucleation centers and promote an island type of growth. These islands along with naturally formed nucleation centers then grow in size and coalesce to form a continuous film. Very high laser plasma fluence can induce explosive boiling of the target material leading to phase explosion. The electric field produced by the laser beam in an absorbing medium under can be valued from the following relation [46]:

\[ E = \left( \frac{2P}{C \varepsilon_0 \eta} \right)^{1/2} \]  

Where \( E \) is the electric field, \( \Phi \) the power density, \( \varepsilon_0 \) the dielectric constant in vacuum, \( n \) the refractive index of the medium and \( C \) the speed of light.

### 3.1. Influence of Laser Fluence

When laser beam hits the target, the density of the laser energy will totally depend on its pulse energy and the area of the target where the laser beam fell. Laser fluence calculated using equation (2):

\[ \text{Laser fluence (J/cm}^2\text{)} = \frac{\text{Laser energy (J)}}{\text{Beam area (cm}^2\text{)}} \]  

To remove an atom from a target surface by a laser pulse, the laser fluence should override the binding energy of target material constituents. Therefore, the ablation rate from the target is a function of laser fluence. Laser fluence will affect surface morphology, optical, transmittance, and electrical properties of the ZnO films by immediately affecting species which created in the plasma plume. The size and the density of particles on the surface of deposited film have a tendency to increase with increasing laser wavelength and laser fluence [31]. Studies approved out by Naszalyi et al. [47] display increased O/Zn ratio as result of the increasing in optical band gap, grain size, and surface roughness with an increase in laser energy density for ZnO films deposited on silicon and glass substrates. At low laser fluence, films deposited have impurities and defects of Zn atoms whose concentration reductions with a rise in laser energy density value. The increase in laser energy level will caused in increasing of crystallite size, and Thickness of ZnO films ,while the structure appeared as amorphous when laser fluence more increase reach to 2.4J/cm\(^2\). From the results of X-ray diffraction study, it noticed an increase in the peak intensity of different films that deposited under the influence of different laser fluences as seen in the figure (2).
3.2 Effect of Pulse Repetition Rate
The quantity of removed species received to a substrate per laser shot rises as repetition rate of laser pulse increased. King et al.[48] Proved that the film crystallinity is to be sensitive to the pulse repetition rate and produced a film with thicknesses of about 272 nm, 289 nm and 255 nm respectively, as characterized by ellipsometry. The grain size of ZnO film growthed at 5 Hz was greater than that of 10 Hz. The repetition rates differences did not show effect on the optical properties of ZnO films as UV emission intensity/visible emission intensity ratios of ZnO thin films deposited at 5 Hz and 10 Hz are almost identical. Lee have reported the regression of the crystalline quality and surface morphology in ZnO film deposited at 10 Hz results from super saturation effect by shrinkage of time interval between a ZnO particle incoming on a substrate by laser shot and a ZnO particle arriving on a substrate by next laser shot[49]. During the study of pulse repetition rate effect other experimental parameters, such as substrate temperature, energy density of the laser, ambient gas pressure and the distance between a target and substrate kept constant.

4. Characteristics of PLD films
The laser ablation process generally forward-directed in nature and preferable to deposit the material on the substrate vertically at the point of hitting. The variation in the thickness of the film that is deposited on the substrate, and that is depending on the energy density of the laser beam and the other factors involved in the deposition process will be following this relationship: \((\cos^2\theta - 12 \theta)\) where \(\theta\) represents the angle between the substrate normal and radial vector. This thickness variation is much steeper than the \(\cos \theta\) variation in case of thermal evaporation process. However, this problem can be overcome by restring the substrate during the deposition.

Fig (2) XRD patterns of ZnO films grown on glass at various laser fluence [45].
constantly. This approach allows growth of the thin films over large substrate areas without much thickness variation. One of the greatest advantages of the PLD growth technique lies in growing meta-stable alloy phases, which are difficult to grow by other vapor deposition techniques operating under equilibrium conditions. Highly energetic plasma plume produced during the laser ablation allows one to grow material phases which are much beyond the thermodynamic solubility limits of the constituents using PLD [50]. The high cohesive energy of ZnO lead to a highly steady and perhaps the greatest radiation solid material between the direct band gap semiconductor group, which certify a long life and a great degradation threshold of ZnO based opto-electronic devices. The rising of melting and boiling points of ZnO permit one to explore a range of heat treatments that is necessary for alloying process and device construction. One of the most stimulating features of ZnO is the stability and its large exciton binding energy, which is about of ~60 meV [51]. This high bonding energy achieves high exciton absorption and recombination that can occur at room temperature. Using pulsed laser deposition technique, a stimulated emission achieved because of excitonic recombination at very low values of the threshold by the pulsed laser beam of a thin layer of ZnO that grows on a Sapphire substrate. The observed low threshold of pumping for efficient stimulated emission attributed to the excitonic recombination. Quantum confinement of excitons can further enhance the excitonic binding energy and may result in other interesting properties including extremely low threshold of lasing. In recent years, ZnO alloys have become the focus of the attention of researchers due to the excitonic effects at room temperature, which has opened the way for applications that fall in the spectral range of ultraviolet rays. The band gap has been improved using ZnO alloys with MgO or CdO to obtain band gap for lower or higher energies.

5. Band-gap Engineering of ZnO by Alloying

Band gap engineered adjustment of ZnO is necessary for low-dimensional structures (such as super-lattices and Quantum wells) to be realized. Sharma et al [52] have reported earlier a study on the metastable alloying of MgO and ZnO to fabricate Mg\textsubscript{x}Zn\textsubscript{1-x}O ternary alloy films by PLD. They used sintered targets of MgO mixed in different concentrations of ZnO to grow single wurtzite phase Mg\textsubscript{x}Zn\textsubscript{1-x}O films with high value of Mg concentration. Because of the low thermodynamic solid solubility limit of MgO in ZnO, therefore the PLD grown single phase Mg\textsubscript{x}Zn\textsubscript{1-x}O films with high Mg concentration could be considered as metastable phase as shown in figure (3).[41].

![Figure 3](image)

**Figure 3.** Optical transmittance spectra of the Mg\textsubscript{x}Zn\textsubscript{1-x}O films grow with different Mg content with different [41].
We have grown Mg$_x$Zn$_{1-x}$O films by PLD at different concentration and the optical transmittance spectra of the Mg$_x$Zn$_{1-x}$O films were grown on a sapphire substrate at suitable substrate temperature, oxygen pressure, laser fluence, and with different Mg-content, $x=0.0$ to $x=1$. Spectra displays a clear continuous shift in the transmission edge[41], Co-doped ZnO is being explored as another possibility to realize DMS based on ZnO. Various groups have reported ZnO or CoZnO thin films grown by PLD wherein magnetic, medical, antibacterial and magneto optical properties of the alloy films have been studied [54-57]. Kim et al have reported PLD of Co doped ZnO thin films grown under different conditions. Table (1) Shows energy gaps for different films at varying temperatures [53].

| Film          | Eg (eV) at T=200°C | Eg (eV) at T=400°C |
|--------------|--------------------|--------------------|
|              | E=400mJ            | E=400mJ            |
| ZnO pure     | 3.3 eV             | 3.32 eV            |
| Co-doped ZnO (1%) | 3.18 eV         | 3.2 eV             |
| Co-doped ZnO (3%) | 3.38 eV         | 3.52 eV            |
| Co-doped ZnO (5%) | 3.25 eV         | 3.27 eV            |

6. Conclusion
In conclusion, the pure and doped ZnO films which deposited on different substrates, temperatures, and different laser energy. We found that when increasing the laser energy, the energy gap increased and it found that the XRD scans of the samples indicated that the ZnO (100) crystals and the peak intensity increasing with increasing laser energy at high temperatures and slightly shift. The increase in Mg concentration and the change in thickness values resulted in an increase in the band gap values, also caused in the shifting toward the lower wavelength of the absorption edge. It has been verified that the doped ZnO films that have good morphological and structural properties possible deposited under suitable conditions. Where the film specifications using an alumina moderate concentration were denser than the others, moderately rough, and the increasing of Al$_2$O$_3$ concentration led to increasing in band gap. In addition, the increase in the addition of doping led to an increase in the roughness values of film. We also found that the substrate temperature has a very active role on the structural and morphological of ZnO thin films. Increasing the temperature in the pulsed laser deposition technique improves the quality and structure of the ZnO crystalline film, but it gets worse if the temperature rises to more than this value. From morphological studies, it found that the particle size is usually small, within several tens of nanometers and the film has slight defects and its surface is rougher. The structural and optical properties found to be dependent on the laser fluence. The effect of the laser fluence used in the deposition process on the grain size of the prepared film verified and thus the effect of the latter on the energy band gap. The strongest UV emission with a narrow full width at half maximum (FWHM) of 19.12 nm is observed from the films deposited at (1.6) J/cm$^2$. The proportions of Co-doped ZnO thin films were also strongly dependent on substrate temperature, when deposited on glass substrates using PLD method. Studies also have shown that the doping with Co has a great role in shifting the edge of absorption towards short wavelengths. The adjusted rate of growth factors like substrate temperature, background gas pressure, laser energy density (laser fluence), target to substrate
distance, repetition rate, oxygen pressure in the chamber, deposition time and post growth annealing which governed deposition parameters like surface diffusion, adsorption, desorption, nucleation, crystallization and recrystallization. In addition to the properties: morphology, roughness of the film surface, film thickness, grain size, optical transmittance, sensitivity, electrical conductivity, uniformity, and electrical resistivity, all mentioned above can be extracted from this review to develop sensors, optoelectronic devices, solar cells, photovoltaic inverters and transistor structures based on transparent conductive films of un-doped ZnO.

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