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Effect of Laser Pulses and Energy on the Structural Properties of ZnO Thin Film Prepared using PLA Technique

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Abstract

In this work, zinc oxide (ZnO) thin film has been fabricated on glass substrate using pulse laser ablation (PLA) technique. The effect of laser pulses of 1000, 1500, 2000 pulses at laser energy 700 mJ as well as, laser energy of 600, 700, and 800 mJ at fixed laser pulses of 1500 pulse, with methanol as a solvent on the structural properties of prepared films using XRD, SEM and EDX. XRD results revealed that the ZnO thin films have hexagonal structure with polycrystalline in nature with preferred orientation of (002). Crystalline size was increased to be 2.6, 4.4, and 5.8 nm with the increasing of the pulses of 1000, 1500, and 2000 respectively, and at lowest crystalline size of 2.86 nm with nanostructure like tree leaf have been seemed at energy of 700 mJ and laser pulses of 1500 pulse. In addition, narrow FWHM and no phase change have been observed in all cases. SEM images showed that for all cases the films were homogenous with some island and cluster then cracking started to obtain with the increasing of increase the pulse number. EDX analysis showed that the prepared films were free of defects and contaminations.

Keywords: ZnO; pulse laser ablation (PLA); laser pulses; laser energy; nanoparticle

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1. Introduction

Zinc oxide (ZnO) has many promising properties for optoelectronics, sensor applications, and transparent electronics and so on, since it has a wide band gap energy and large exciting binding energy of 60 mV at room temperature. There are two routes to produce a nanomaterials are: a top-down approach and a bottom-up approach. The idea behind the top-down approach is as following: An operator first designs and controls a macroscale machine shop to produce an exact copy of itself, but smaller in size such as laser ablation in liquid and chemical etching. While, the bottom-up approach is that one starts with atoms or molecules, which build up to form larger structures such as pulse laser deposition (PLD) and physical vapor deposition (PVD). However, the top-down approach is not expensive and rapid way of producing nanostructures [1]. The literatures on ZnO thin films started early and many researchers studied its physical properties, however, In 2001 Singh et al. prepared highly conductive and transparent Al-doped zinc oxide (ZnO:Al) thin films by (PLA) technique with irradiation time of 10-60 min (6000-36000 laser shots) [2]. Then, In 2006 Yoshie Ishikawa, et al. successfully, produced zinc oxide nanorods using (PLA) in deionized water media at high temperature and at room temperature [3]. In addition, Chen and Zhang Prepared Nano-ZnO thin film by PLA and studied different factors that effect on the prepared films. Besides[4], In 2007 Thareja and Shukla formatted colloidal suspension of zinc oxide nanoparticles by (PLA) of a zinc metal target at room temperature in different liquid environment[5]. In 2008 Ajimsha and his colleagues prepared ZnO quantum dots (QDs) in various liquid media using LP-PLA without using any surfactant. They concluded that, the emission wavelength was tuned due to the defect and varying in the laser fluency. After that [6], In 2009 Cho et al. produced ZnO Nanoparticles by (PLA) in deionized water. They revealed that there are strong in fluency of parameters such as, laser power, ablation time, and aging [7]. In 2010 Virtand and his colleagues studied the properties of ZnO and ZnMnO thin films prepared by (PLA) that have polycrystalline behavior with average sizes of particles of ZnMnO film at 300 K and at 473 K were 50 Å and 400 Å, respectively [8]. In 2011 Raidand and his colleagues synthesized ZnO by (PLA) in double distilled water with various laser fluencies at RT. They founded that, the optical properties, size, and the morphology of prepared ZnO have influenced by laser fluency and wavelength [9]. In 2012 Atanasovaand and his colleagues prepared of ZnO nanostructure by (PLA) and some changes occurring on the surface of the films after continued exposure in air [10]. In 2013 Nakamuraand and his colleagues succeeded in synthesized ZnO nanoparticle by (PLA) method by Q-switched Nd:YAG laser in the air with spheres shape of diameters 10-20 μm [11]. In 2015 Fadhil and Hadi prepared zinc oxide nanoparticles by (PLA) in isopropanol solvent at room temperature and studied effect of laser fluency [12]. In 2016 Salim et al. fabricated ZnO Nanoparticles using LP-PLA system at a different laser wavelength (1.06 and 0.532μm laser fluency and number of the laser pulse. They studied the effect of these parameters on surface morphology and found that the grain size of the films increased with laser fluency and decreased with number of laser pulses [13]. In 2016 Farahanand and his colleagues prepared ZnO nanoparticles by (PLA) and studied the effect of solvents (methanol and distilled water) on the characterization of ZnO. They found that the ZnO nanoparticles have a hexagonal crystal structure and different size was formed due to the change of environment of laser pulse. Additionally [14], Ismailand and his colleagues synthesized of pure and Er+3 doped ZnO nanoparticles by using (PLA) in ethanol at room temperature and found that the surface in granular morphology [15]. Late, In 2017 Khashan and Mahdi. Synthesized ZnO: Mg nanocomposite by (PLA) in liquid and they found that, the sample has hexagonal wurtzite [16].To fabricate zinc oxide nanostructures, pulsed laser ablation (PLA) has been used in many works .In this work PLA of a solid target in liquid media is becoming an increasingly popular approach for controlled synthesis of noble metal and metal oxide nanoparticle. It is a chemically simple and clean synthesis method, due to reduced by-product formation, simpler starting materials and there is no need for a catalyst [17]. Thus, the objectives of this work are to fabricate a nanostructure ZnO thin film on glass using PLA technique. The prepared films have been characterized to study the structural and optical properties using XRD, SEM, EDX and UV-Vis spectroscopy. As well as, the effect of pulse, types of solvent, types of substrate and energy on the structural and optical properties have been studied. The fabrication of ZnO nanostructure and the results from study the major parameters effects on its structural and optical properties are the first studied over the world as the best of our knowledge.

2. Materials and Methods

2.1 Laser Ablation System

The experimental setup for laser ablation, which includes Nd:YAG laser of 1064 nm were used for laser ablation process have been shown in Figure 1. The ablation process is done at room temperature. The target (Zn) (purity of 99.99%) has been immersed in water or aqueous solution, and fixed at bottom of glass vessel container.

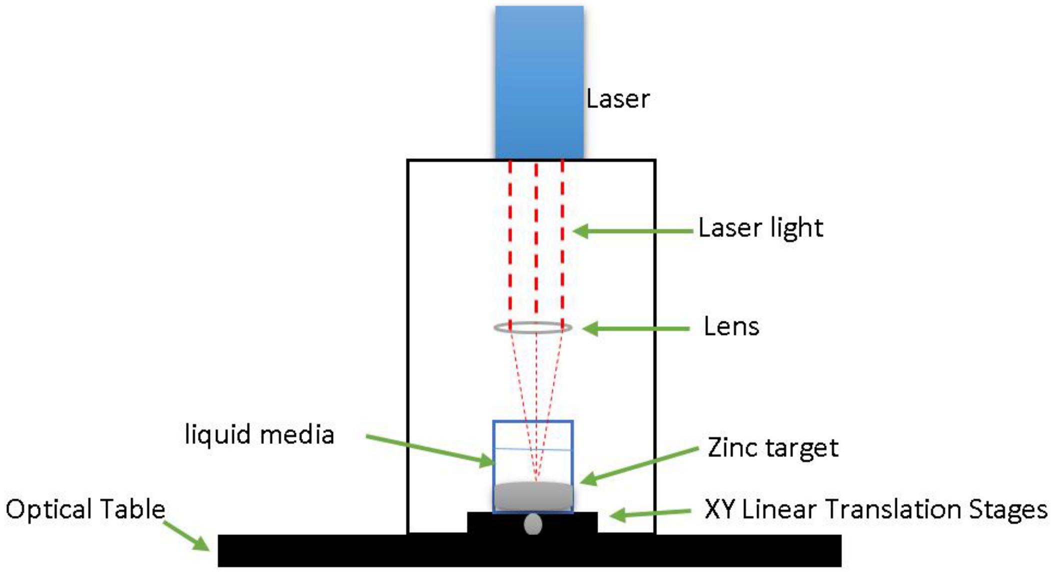


Figure 1: Experimental setup for nanoparticles synthesis by PLA process [12]

2.1.1 Laser Ablation Source

Nd:YAG laser system type HUAFEI providing pulses at 1064 nm wavelength in the visible was used for ablation of target with pulse duration is 10 ns, repetition rate of 1Hz and effective beam diameter of 4.8 mm, were used for laser ablation. The number of laser pulse ranged 1000, 1500, and 2000 pulses at energy of 700 mJ and laser energy of 600, 700, and 800 mJ at 1500 pulse have been used in this work. Glass container was applied as a cell. The Zn target was placed on the bottom of glass container loaded with 3 ml of distilled water. The solution profundity was around 3 mm during laser ablation. The cell holder that round at 9 rpm was used to round the cell and its contents during ablation process to provide uniform ablation in order to ensure identical irradiation conditions for the subsequent pulses during laser exposure as well as, to avoid a deep ablation trace and craters formation onto the target surface.

2.1.2 Solvents Types

Absolute ethanol with purity 96% was used as ablation medium, compared to other solvents, ethanol provides a relatively inert environment for the production of nanoparticles. Also it has special property to exclude the oxide layer and to prevent the nanoparticles from oxidation.

2.1.3 Substrate Preparation

The substrate used for deposits ZnO thin films in this work is a glass slides with dimensions (2.5×2.5) cm2.The cleaning of the substrate is very important because it has a great effect on the properties of the films. Firstly, cleaning the glass slides by using tap water to remove any dust that might be on the surface of the substrate and then they are placed in distilled water for 60 minutes. Then, Immersing the substrates in a pure acetone solution which reacts with contamination such as grease and some oxides, this process is fulfilled accurately by rewashing the substrates in the ultrasonic bath for 10 minutes and drying the slides by wiping them with soft paper.

3.3 Preparation of Target

The solid Zn target was taken from Fluka Company, united-states, product in 2015 with high purity (99.999%). The sheet of tin firstly cut into small piece with dimensions of 1×1 cm2 then polished and cleaned using HF-water (ration 1-50) before the ablation operation.

2.4 ZnO Nanoparticles Formation

In order to combine of ZnO NPs, a rigid Zn target was immersed at room temperature in glass container loaded with 3 ml of liquid was placed on top cell holder and was irradiated by Nd-YAG laser operating at 1064 nm wavelength. The rapid reactive quenching and aqueous oxidation of highly active Zn bunches in fluid media is in charge of the development of ZnO nanoparticles. The colloidal was vibrated for 15 minute before deposition by ultrasonic vibrator in order to get homogeneity of the product. It is simply based on covering the substrate with three of drops or more of the solution and leaving it for a while to dry which used the hot plate (magnetic stirrer) at 80 C° temperatures.

3- Results and Discussion

3.1 Structural properties of ZnO thin films

Figure (2) shows the XRD pattern of ZnO nanoparticle on glass substrate with laser pulses number of 1000, 1500, and 2000 pulse at energy of 700 mJ in methanol, which revealed that the ZnO films have hexagonal polycrystalline structure nature with lattice parameter c = 5.130, 5.159 and 5.162 respectively. Figure show that the diffraction peaks of ZnO nanoparticle were at 34.420, 36.250 and 47.530, which corresponding to the orientations of (0 0 2 (1 0 1) and (1 0 3). These results are agreement with those given in JCPD data card and it is clear that there is no change in phase.

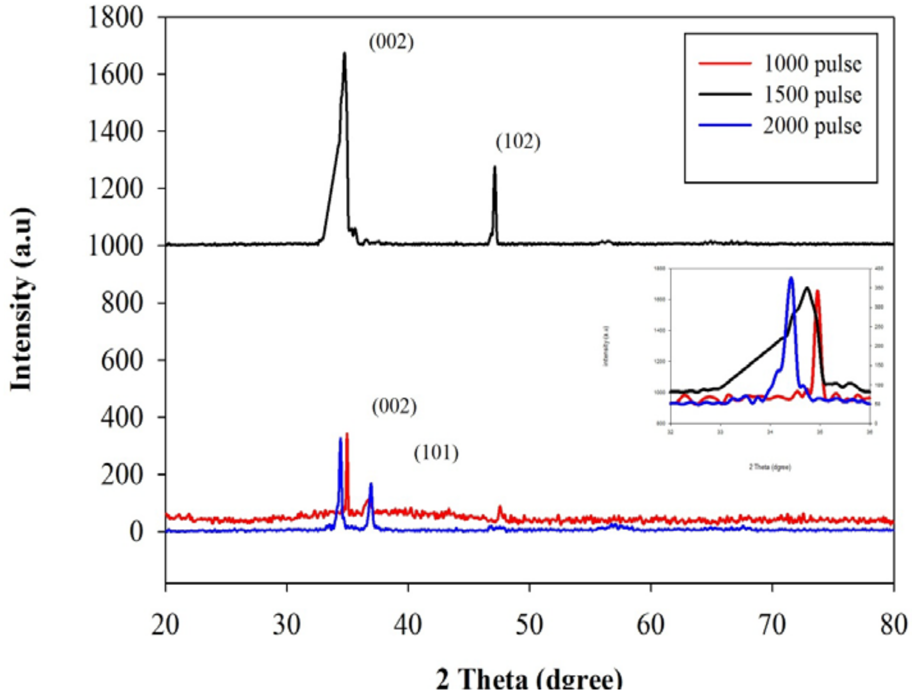


Figure 2: XRD pattern of ZnO nanoparticle on glass substrate with laser pulses number of 1000, 1500, and 2000 pulse at energy 700 mJ methanol solvent

Figure (3) shows the XRD pattern of ZnO nanoparticle on glass substrate with laser pulse number of 1500 pulse at energy of 600, 700 and 800 mJ dissolved in methanol. This Figure revealed that the ZnO films have hexagonal polycrystalline structure nature with lattice parameter c=5.130°, 5.159° and 5.130° respectively. Figure show that the diffraction peaks of ZnO nanoparticle on glass substrate were at 34.77° and 36.17°, which corresponding to the orientations of (0 0 2 and (1 0 1). These results are agreement with those given in JCPD data card and it is clear that there is no change in phase.

Figure (4) show SEM images of ZnO nanoparticle prepared using PLA with pulse laser ablation, at fixed energy of 700 mj and different Pulses of 1000, 1500 and 2000 dissolved in methanol solution and deposition on glass substrate. It clear that the structure became as island and then cracking started with increase the pulse number. The partials size was in nanoscale and it is seem like (cluster) at pulse number of 1500 which confirmed the XRD results.

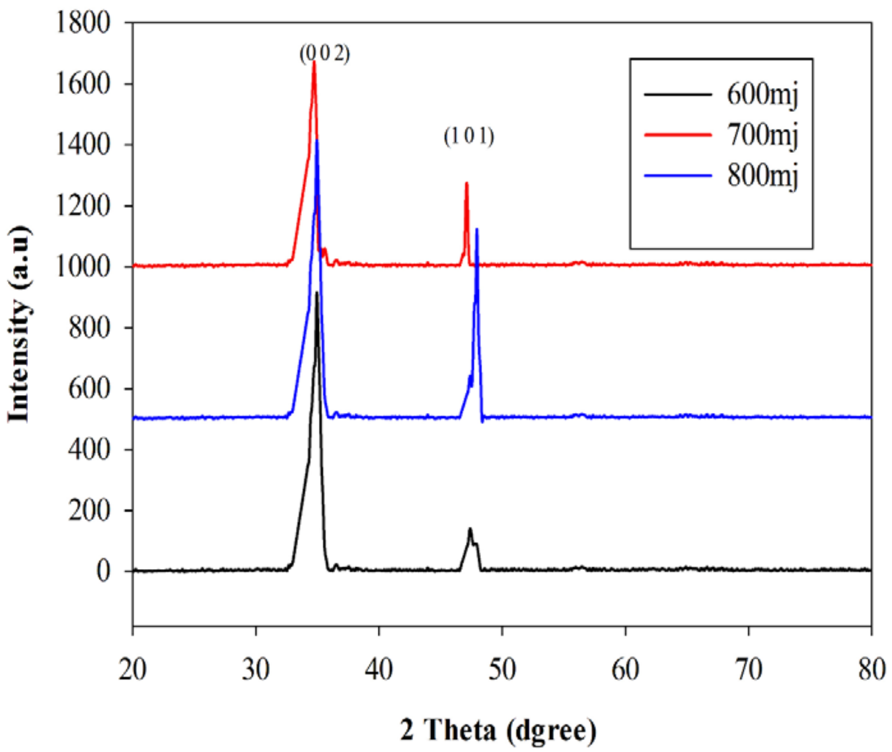
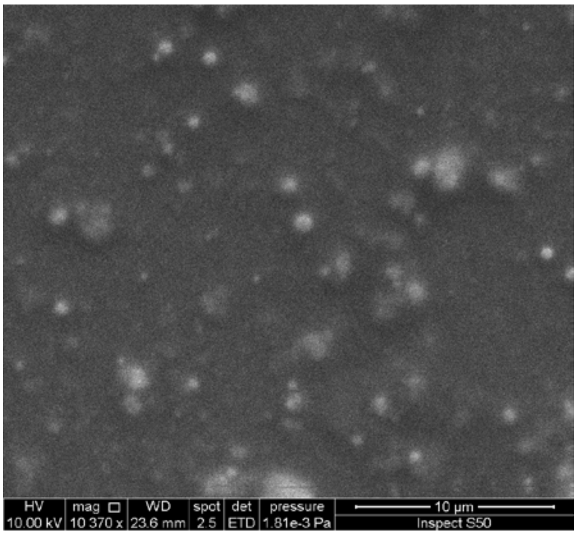


Figure 3: XRD pattern of ZnO nanoparticle on glass substrate with laser pulses number of 1500 pulse at energy 600 mj ,700 mJ and 800mj .

Table 1: Structure parameters of ZnO nanoparticle on glass substrate with laser pulses number of 1000, 1500, and 2000 pulse at energy 700 mJ methanol solvent

Pulses	Position (2 theta)	Lattice constant (c) (nm)	FWHM	Crystalline size (nm)	Strain	Dislocation density
at laser energy of 700 mJ						
1000	34.95	5.130902	0.055721	2.607609	0.01329	0.147067
1500	34.75	5.159515	0.03263	4.450524	0.00779	0.050487
2000	34.45	5.200524	0.041883	5.87444	0.00059	0.00029
at laser pulses of 1500 pulse						
600	34.95	5.12838	0.055721	2.97642	0.01329	0.147067
700	34.75	5.15697	0.03263	2.86873	0.00779	0.050487
800	34.95	5.12838	0.055721	2.97642	0.01329	0.147067



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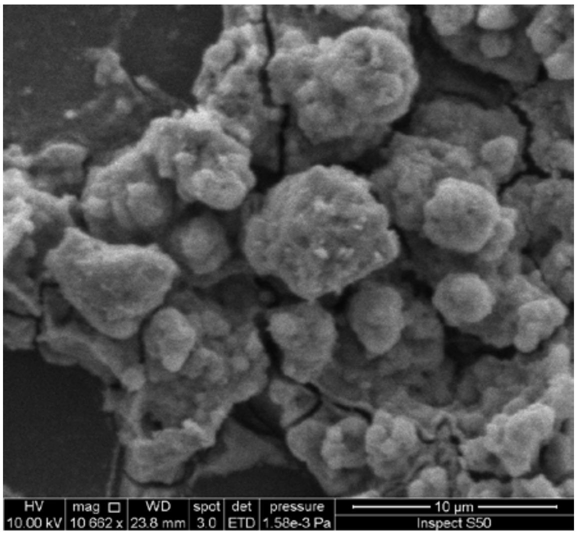

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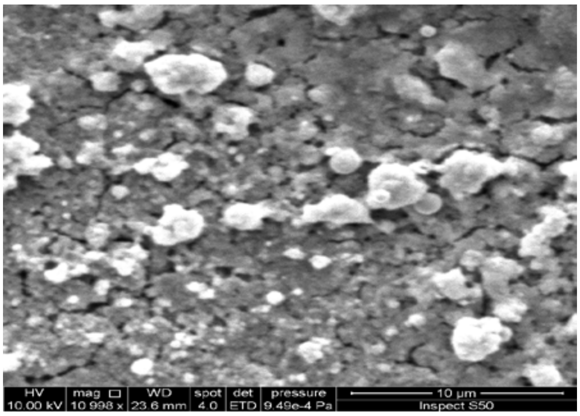

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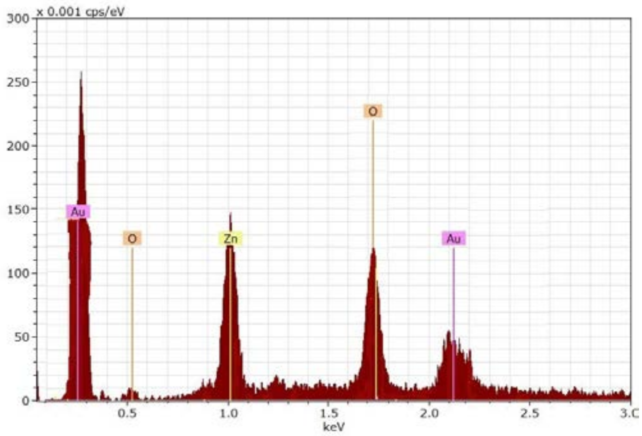
Figure 4: SEM images of ZnO thin film on glass substrate   prepared using PLA method in methanol solvent with energy 700 mj and pulses of a- 1000, b-1500, and c-2000 pulses

Figure (5) show the EDX analysis which confirmed that the Zn and O elements is including in the prepared films on glass substrate using PLA method with energy of 700 mj and at a- 1000 , b- 1500 , c-2000 pulses in methanol solvent .



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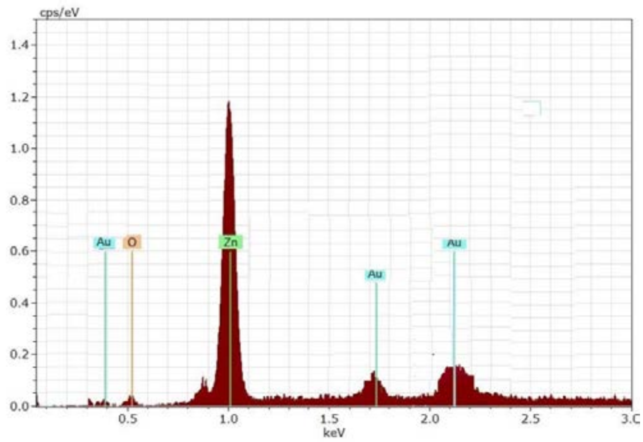

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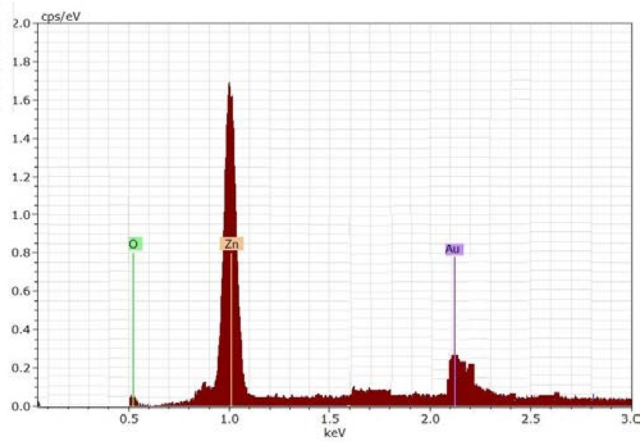

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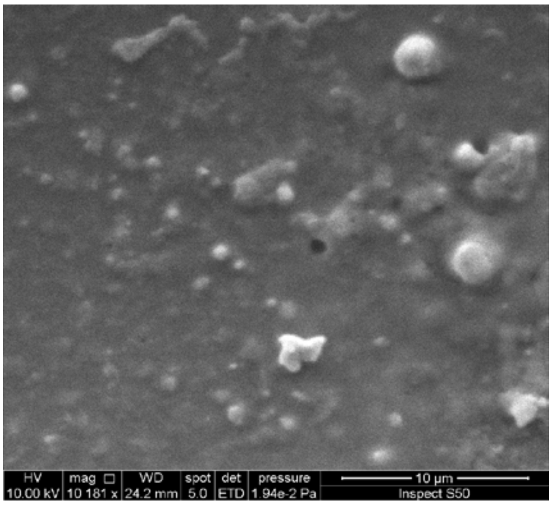
Figure 5: EDX analysis of ZnO thin films prepared by PLA method on glass substrate with energy 700 mj at pulses a-1000, b-1500 and c-2000 pulses in methanol solvent

Figure (6) show SEM images of ZnO nanoparticle that prepared on glass substrate using PLA with energy of 600, 700 and 800 mj at pulse number of 1500 pulse, dissolved in methanol solution. It clear that the partials size is formed in nanoscale especially at energy 700 mj which seem like (flower) structure, while, at 600 mj the surface look smooth with various crystalline with different size and at 800 mj the surface became more sponge with pore size in range of 100-1000 nm .This result is agreement with XRD results and open the door for more studies on this behavior.



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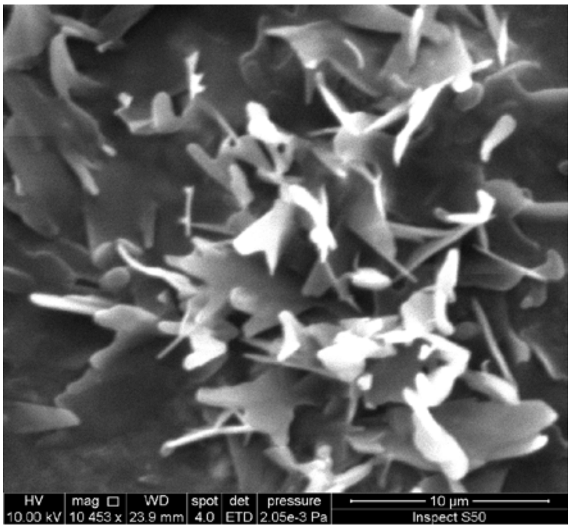

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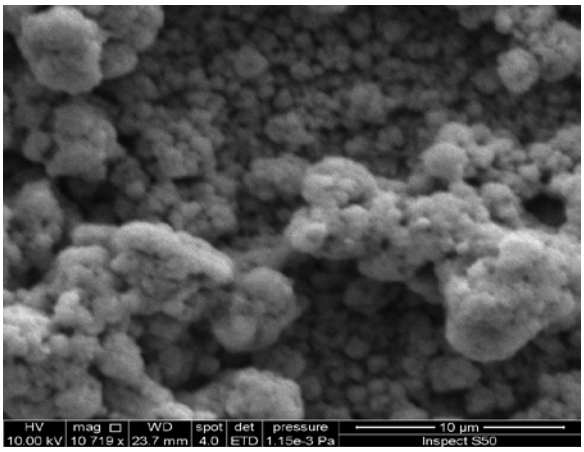

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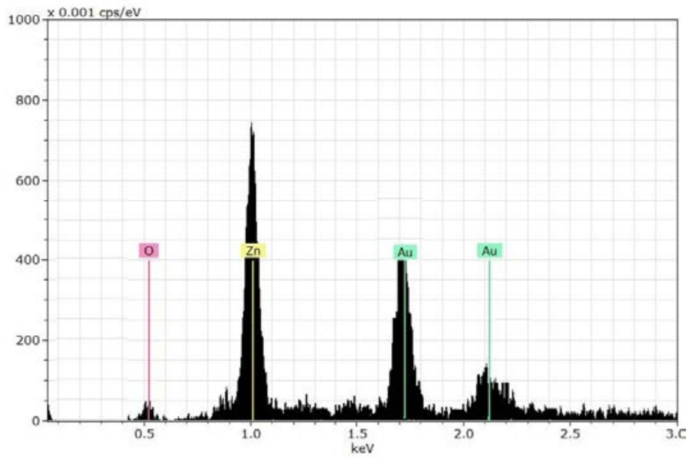
Figure 6: SEM images of ZnO thin film on glass substrate prepared using PLA at pulse number of 1500 and with energy of a-600, b-700 and c-800 mj

Figure (7) show the EDX analysis which confirmed that the Zn and O elements is including in the prepared films on glass substrate using PLA method with energy of 700 mj and at a- 1000, b- 1500 and c- 2000 pulses in distill water solvent.



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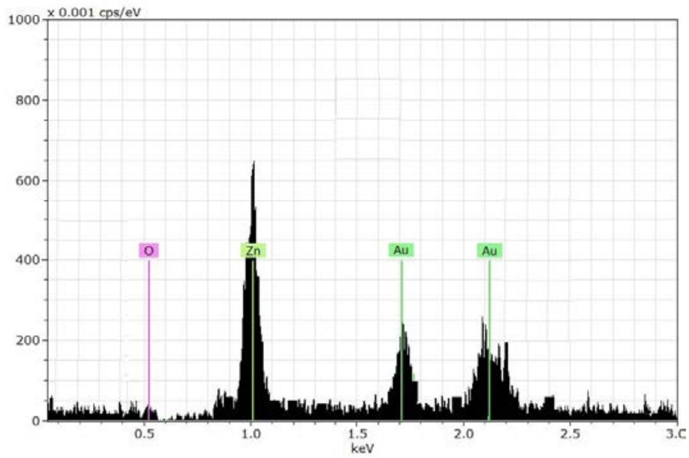

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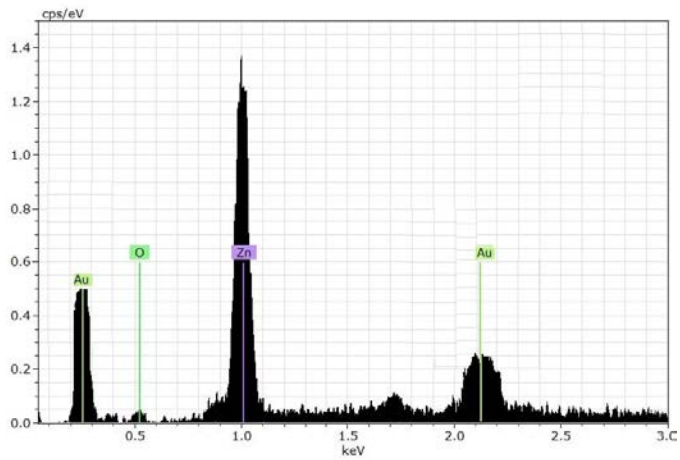

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Figure 7: EDX analysis of ZnO thin films prepared by PLA method on glass substrate with energy of a-600 ,b-700, and c-800 mj in methanol solvent.

Figure (4-13) show SEM images of ZnO thin film prepared using PLA with pulse laser ablation, with fixed energy of 700 mj and at Pulse 2000 dissolved in methanol solution, porous silicon substrate. It clear that the surface have pores with different size and different wall in surround and partials size was in nanoscale.this result is agreement with XRD results

4. Conclusion

ZnO thin films have been successfully fabricated on glass substrate using pulse laser ablation (PLA) technique and have hexagonal structure with polycrystalline in nature with preferred orientation of (002). Crystalline size was increased with the increasing of the pulses and at energy of 700 mJ and pulse of 1500 pulse seemed nanostructure like tree leaf. In addition, in all cases, narrow FWHM and no phase change and the films were homogenous and free of defects and contaminations with some island and cluster then cracking started to obtain with the increasing of increase the pulse number.

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