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CHARACTERIZATION OF PULSED LASER DEPOSITED ZNO FILMS: INFLUENCE OF LASER AND BEAM PROFILE AT 532 AND 1064 NM

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ABSTRACT

ZnO thin films have been elaborated using a pulsed laser deposition (PLD) technique onto glass substrate at room temperature. The PLD process is developed in oxygen atmosphere ($1*10^{-1}$ mbar). The morphology, chemical composition and optical characteristics were studied as function of laser wavelength and laser profile (532 and 1064 nm). Film properties are strongly influenced by the Gaussian profile to flat top shaped laser beam at 532 nm and 1064 nm. At regardless of laser wavelength, films prepared with flat top profile exhibit smooth surface and preferential growth direction (101), it is detected reduction of the density defects like interstitial or vacancies atoms. The optical band gap, the ratio intensity visible/UV fluorescence and peak position are modified in agree with the degradation of film stoichiometry. At regardless of the laser wavelength, the use of Gaussian beam stimulates the highest deposition rate; the surface roughness and clusters density are incremented. Films show a polycrystalline structure (100, 002 and 101). The optical band gap is modified, film stoichiometry is higher than flat top films, in agree with the fluorescence measurements. We demonstrated a simple, fast and low cost setup to elaborate ZnO films with tailored properties. These films could be used to applications in short wavelength optoelectronic devices, optical or electric sensors, also for the elaboration of nanowires using different types of substrates.

KEYWORDS: ZnO film; Pulsed laser deposition; Room temperature films; Film characterization; Laser beam shape

CARACTERIZACIÓN DE PELÍCULAS DE ZNO ELABORADAS POR DEPOSICIÓN CON LÁSER PULSADO: INFLUENCIA DE LA LONGITUD DE ONDA Y EL PERFIL DEL HAZ A 532 Y 1064 NM

RESUMEN

Las películas de ZnO se fabricaron por medio de Deposición por Láser Pulsado en atmósfera de oxígeno (0,1 mbar) sobre sustratos de vidrio, a temperatura ambiente. La morfología, composición química y características ópticas fueron evaluadas como función de la longitud de onda y el perfil del haz láser, a 532 y 1064 nm. Independientemente de la longitud de onda del láser; con el haz de perfil plano se preparan películas con superficies de baja rugosidad y dirección preferencial de crecimiento (101), se observa una reducción de defectos cristalinos como átomos intersticiales o vacancias; el *band-gap* óptico es desplazado y la posición e intensidad relativa de las bandas de fluorescencia son modificadas, en concordancia con la degradación de la estequiometría en la película. Con el uso del perfil gaussiano se obtienen tasas más

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altas de deposición, superficies más rugosas y con incremento en la densidad de agregados; son películas policristalinas, se detectan al menos los picos 100, 002 y 101; la estequiometría es mayor, en concordancia con desplazamiento del band-gap óptico y las medidas de fluorescencia. Se demuestra que con un sistema simple, rápido y de bajo costo se pueden elaborar películas de ZnO con propiedades ajustables, que podrían ser utilizadas para dispositivos optoelectrónicos, sensores ópticos y/o eléctricos, o para la elaboración de nanoestructuras sobre diferentes tipos de sustratos.

PALABRAS CLAVE: Deposición por láser pulsado; películas elaboradas a temperatura ambiente; caracterización de películas; perfil haz láser.

CARACTERIZAÇÃO DE PELÍCULAS DE ZNO ELABORADAS POR DEPOSIÇÃO COM LASER PULSADO: A INFLUÊNCIA DO COMPRIMENTO DE ONDA E O PERFIL DO FEIXE A 532 E 1064 NM

RESUMO

Os Filmes de ZnO se fabricaram por médio de deposição por laser pulsado em atmosfera de oxigénio (0,1 mbar) sobre substratos de vidro a temperatura ambiente. A morfologia, composição química e as características ópticas foram avaliadas como uma função do comprimento de onda e o perfil do feixe de laser a 532 e 1064 nm. Independentemente do comprimento de onda do laser; com feixe de perfil plano se preparam filmes com superfícies com baixa rugosidade e direção preferencial de crescimento (101) se observa uma redução de defeitos cristalinos, tais como átomos ou vagas; o band-gap óptico é deslocado e a posição e a intensidade relativa das bandas de fluorescência são modificadas, em concordância com a degradação da estequiometria no filme. Com o uso do perfil gaussiano, se obtém maiores taxas mais altas de deposição, superfícies mais ásperas e um aumento na densidade de agregados; são películas policristalinas, se detectam, pelo menos, picos 100, 002 e 101; a estequiometria é mais elevada, de acordo com o deslocamento do band-gapópticoe as medidas de fluorescência. Demostra-se que com um sistema simples, rápido e de baixo custo podem ser produzidos filmes de ZnO com propriedades ajustáveis, que poderiam ser usados para dispositivos opto-electrónicos, sensores ópticos e / ou eléctricos ou para a elaboração de nanoestruturas sobre diferentes tipos de substratos.

PALAVRA-CHAVE: A deposição por laser pulsado; Fitas feitas a temperatura ambiente; Caracterização de filmes; Perfil do feixe de laser.

1. INTRODUCTION

ZnO is an interesting and versatile material due to its attractive physical properties and its wide range of applications: films, nanostructures and nanowires systems. Pulsed Laser Deposition (PLD) technique allows the elaboration of films with tailored properties at lowest substrate temperatures than other techniques. Several studies on the influence of ablation laser wavelength on ZnO film properties have been elaborated (Ashfold, M N R, et. al., 2004; Craciun, V, *et al.*, 1995; Ianno, N J, *et al.*, 1992). UV (KrF, XeCl, ArF) are laser wavelength most usually reported in ZnO PLD films (Suchea, M, *et al.*, 2009; Hong, J.I, *et al.*, 2009); although the second armonic of Nd:YAG (532 nm) and IR laser (1064 nm) are used (Liu, M. *et al.*, 2006; Fan, X.M, *et al.*, 2007; Prenkumar, P. *et al.*, 2007; Padilla-Rueda, D. *et al.*, 2012). Film properties are strongest defined by the laser wavelength, the optical penetration length and the interactions matter-radiation are the explanation, the interactions target-laser (IR or Visible) are thermal interactions, mainly (Ashfold, M N R, et. al., 2004; Craciun, V, *et al.*, 1995; Ianno, N J, *et al.*, 1992; Hahn, D.W.; Omenetto, N., 2012; Tognoni, E.; *et al.* 2002). The influence of laser beam shape in ablation process is commented (Hahn, D.W.; Omenetto, N., 2012; Tognoni, E.; *et al.* 2002; Mateo, M.P., *et al.*, 2003), although in our knowledge, there are not recent papers over the influence laser beam shape on ZnO PLD films properties. We studied the influence of laser wavelength and the beam shape (at 532 and 1064 nm) onto ZnO films prepared with PLD on glass substrates at room temperature on oxygen atmosphere.

2. METHODS AND MATERIALS

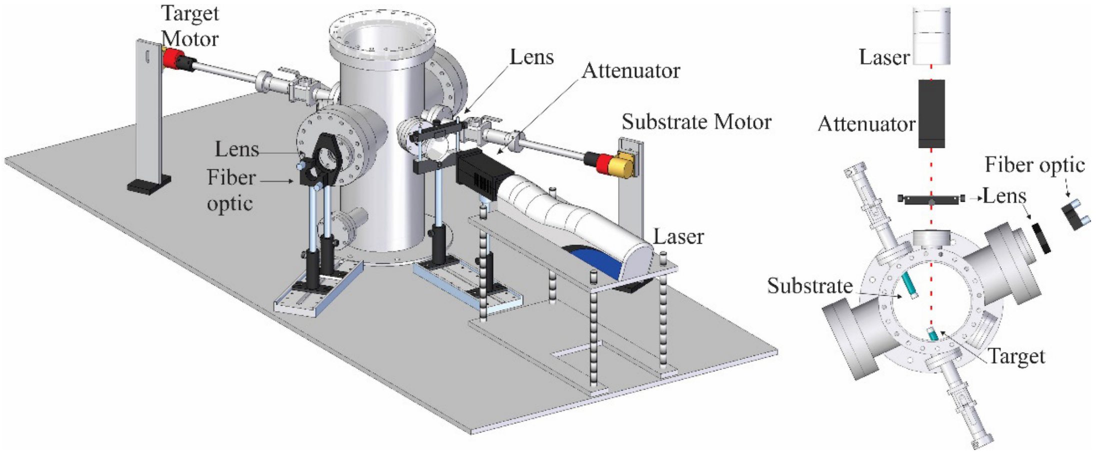
The deposition system and film growth parameters have been described previously (Padilla-Rueda, D. *et al.*, 2012). The deposition system consisted of a stainless steel chamber, a 10^{-3} mbar of oxygen atmosphere (Fig 1). ZnO targets was home made. Optical control of the process was made with a lateral port with quarz window. The targets are placed in a rotating automatized holder (15 rpm) and ablated using a Litron NANO-T-250-10 (τ_{fwhm}=7 ns, 10 Hz). Gaussian beam and flat-top beam was evaluated at 532 and 1064 nm. Laser beam is focused by a 30 cm focal length onto a ZnO target at an incident angle of ≈ 45°. Fluence was constant for all wavelengths and laser beam profiles (10 J/cm^2) and controlled with a home-made attenuator. Substrates were hydrolytic class D263M1 glass slides and placed in a rotating automatic holder (3 rpm) oriented parallel to the target surface at 4 cm. All films have been elaborated with 24000 laser pulses. All the measurements have been at room temperature. Photoluminescence data were acquired using a Cary Eclipse fluorometer, home-adapted to film measurements (λ_{exc}: 310 nm; λ_{emi}: 330-750 nm, spectral resolution 2 nm). Before test measurements, films were exposed for 1 h to dry air at room temperature. UV-Vis absorption spectroscopy was performed on a Cary Eclipse 50 UV-Vis Spectrophotometer (wavelength range 250-1000 nm) in conventional configuration. SEM analysis has been made with SEM microscope JEOL JSM-840. AFM data were recorded in a Veeco Nanoscope V in contact mode over an area of 2 µm × 2 µm, at normal pressure and air atmosphere. Diffractograms were recorded with a Philips X'Pert PRO-MPD with a Cu $K_\alpha 1$ (1.5406 A°) source. X-ray photon spectroscopy (XPS) was performed on a Physical Electronics PHI model 5700 equipped with Mg K_α radiation (1253.6 eV) in combination with 4 keV Ar^+ gun sputtering.

3. RESULTS AND DISCUSSION

3.1. Structural, morphological and chemical analysis

Films are homogeneous, transparent and colourless, except 1064 nm Gaussian beam films. **Figure 2** shows SEM photography of films as function of laser wavelength and beam shape. Gaussian films at 532 nm show clusters and small micro-crystals on surface. Surface morphology of IR Gaussian films became irregular, clusters density and micro-crystals increase, with slightly white colour observed; other authors described it grey and high clusters density on film surface while films elaborated with UV lasers are colorless with lower grain size and smooth surface (Craciun, V, *et al.*, 1995; Ianno, N J, *et al.*, 1992). The differences can be explained the by laser-target interactions, the photon energy, the absorption optical coefficient as function of laser wavelength and the size or interaction zone.

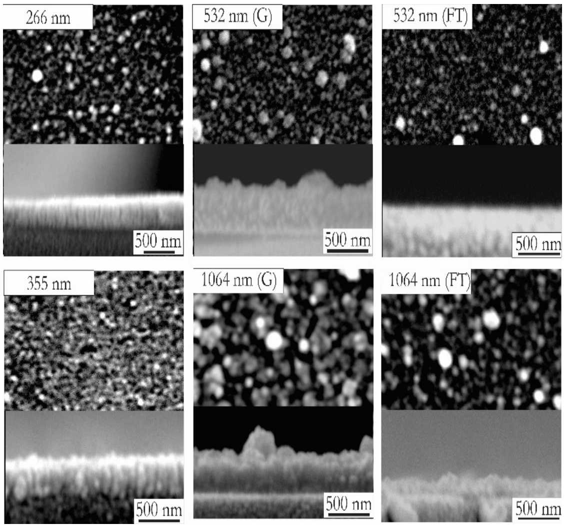
Figure 1. PLD system



If the ablation process uses photons with highest energy that the material band gap, the interactions target-laser are mainly electronic interactions, causing photo-ablation of the material [Ianno, N J, *et al.*, 1992; Hahn, D.W.; Omenetto, N., 2012; Tognoni, E.; *et al.* 2002]. If the ablation photons have lower energy than the band gap material, the laser induces fasts melting of the material that causes hidro-dynamical sputtering, with the increase of the droplets and clusters ejected of the target (Ianno, N J, *et al.*, 1992); films show clusters and irregular morphology. IR and Visible laser are below of the ZnO band gap (3.26 eV) (Craciun, V, *et al.*, 1995) in agree with experimental results.

The influence of beam shape is studied; fluence were similar in all cases; beam shape and energy distribution on target surface causes differences in ablation process (Hahn, D.W.; Omenetto, N., 2012; Tognoni, E.; *et al.* 2002; Mateo, M.P., *et al.*, 2003). Flat top beam shows small damage zone on target causing lowest ablation rates, films show reduction of clusters density and droplets on surface, lowest thickness and roughness, at regardless of laser wavelength. Gaussian beam films show highest thickness and roughness, highest rate deposition and irregular growth zones with respect to flat top films.

Figure 2. SEM pictures of ZnO films as function of laser wavelength. (G) Gaussian beam (FT) Flat-top beam



XPS analysis confirmed ZnO deposition, without traces of metallic Zn, by using Zn $2p_{3/2,}$ Zn 2 $p_{1/2}$ and O 1s peaks. The Zn $2p_{3/2}$ and O 1s peak are displaced (**Figure 3**) Results and calculated film stoichiometry are resumed in **Table 1**. It has been reported that the chemical shift in peak position is related to the charge transfer between the zinc and oxygen ions associate at stoichiometry variations by increment of defect states in the crystalline structure (O interstitial); this variation causes a change in the partial atomic charge and the subsequence chemical shift (Min, C.H., *et al.*,2010). All films were oxygen deficient (Ayouchi, R; *et al.*, 2009), except 1064 nm Gaussian films, it showed oxygen excess. The displacement of O 1s peak at lowest values is associated at oxygen lost in the film; the peak shift of Zn peak at highest values indicates the Zn rich state in the film (Li, H., *et al.*, 2004). In our experiments, the chemical shift and the increment of interstitial O can be associated at differences in interaction mechanisms between laser wavelength-target (Hahn, D.W.; Omenetto, N., 2012; Tognoni, E.; *et al.* 2002; Mateo, M.P., *et al.*, 2003).

ZnO films are polycrystalline, at least (100), (002) and (101) peaks are detected (Fig 4). The preferential growth axis changed as function of the laser wavelength and beam profile. Using Gaussian beam, 532 nm films, the (002) peak intensity is reduced and it can see a weak (101) peak; the 1064 nm films are polycrystalline without preferential direction growth. By using flat top beam, films prepared with 532 nm only exhibit (101) and a low (100) peak; the 1064 nm films show low (101) peak. With the flat top wavelength, a regardless of laser wavelength, all the films exhibit a low intensity in the (101) peak. It has been reported that the films elaborated with UV laser showed preferential growth in (002) plane, and films prepared with visible laser films growth in (101) plane (Craciun, V, *et al.*, 1995; Ianno, N J, *et al.*, 1992). The crystalline quality films are related to kinetics of particles ablated and the clusters density; clusters are stacked and coalesced on substrate surface yield microcrystalline structures (Lemlikchi, S.; *et al.*; 2010). With 532 and 1064 nm Gaussian laser wavelengths, it can observe the increment of polycrystalline of films, in agree with the SEM pictures.

Results show shifts in XRD peak position. The diffraction peak 002 for ZnO films prepared with Gaussian beam 532 and 1064 nm appear at 34.28 and 34.36°,and flat top films with weak intensity at 36.31° and 34.48°, respectively.

Figure 3. Detail of XPS ZnO films as function of laser wavelength (Gaussian beam) (A) Zn $2p_{3/2}$ peak (B) O 1s peak.

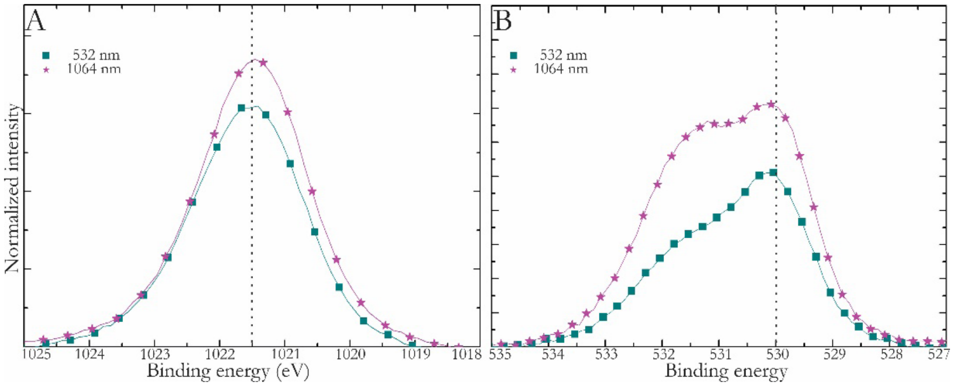


TABLE 1. XPS values as function of wavelength laser ablation.

λ_{LASER} (nm)	Zn $2p_{3/2}$ (eV)	O 1s (eV)	Ratio O/Zn	Optical Band gap (eV)
$532_{GAUSSIAN}$	1021.48	530.10	0.89	3.33
$532_{FLAT\ TOP}$	1021.38	530.02	0.91	3.28
$1064_{GAUSSIAN}$	1021.40	530.15	1.02	3.28
$1064_{FLAT\ TOP}$	1021.39	530.02	0.86	3.33

The shift in angular peak position is associated with a stress in the plane of the film (Zhu, B.L.; *et al.*, 2008; Chen, T.; *et al.*, 2010). In films elaborated at room temperature, film stress is originated by structural defects. The vacancies oxygen shorted the lattice constant (c-axis) and they induce a compressive stress (Zhao, J.; *et al.*, 2007); the interstitials O increase the distance and induce a tension stress along the c-axis (Zhu, B.L.; *et al.*, 2008; Chen, T.; *et al.*, 2010). The experimental data indicate with the increase of the laser wavelength, the reduction of interplanar spacing causing compression stress along the c-axis and polycrystalline of the film (Zhu, B.L.; *et al.*, 2008; Zhao, J.; *et al.*, 2007); it is according to film stoichiometry calculated.

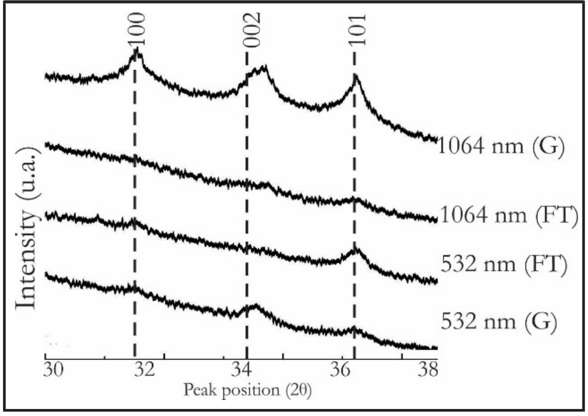
3.2. Optical properties

Films present a good transmittance, with the shift of fall in the UV region typical of ZnO films (spectra do not showed here). We observed the reduction of transmittance films in the visible range (400-800 nm) with the increment in laser wavelength; these effects could be cause by increment of thickness, the compactation of film or the increment in the structural defects. The optical band gap is estimated with the transmittance spectra by meaning of Tauc plot (**Table 1**) (Lemlikchi, S.; *et al.*; 2010; Chen, T.; *et al.*, 2010). The values gap calculated is between 3.28-3.33 eV (Suchea, M, *et al.*, 2009; Zhu, B.L.; *et al.*, 2008). Oxygen vacancies may cause a slightly increase in the optical band gap; although, it can be affected for the variation in the grain size and stress state in the film (Zhu, B.L.; *et al.*, 2008). In this case, the band gap shift is associated at stoichiometry and structural changes as function of laser wavelength.

Photoluminiscence (Pl) emission spectrum from ZnO consists of UV and visible bands (Zhang Y.; *et al.*; 2004; Sun, L.; *et al.*; 2006; Zhao, J.; *et al.*, 2007). In spite of previous papers that have been reported that the films prepared at room temperature do not show PL signal (Zhu, B.L.; *et al.*, 2008), the spectra as function of laser wavelength is shown in **Figure 5**. The UV peak is shifted at blue regions while visible band exhibited a red-shift with the increase in the laser wavelength.

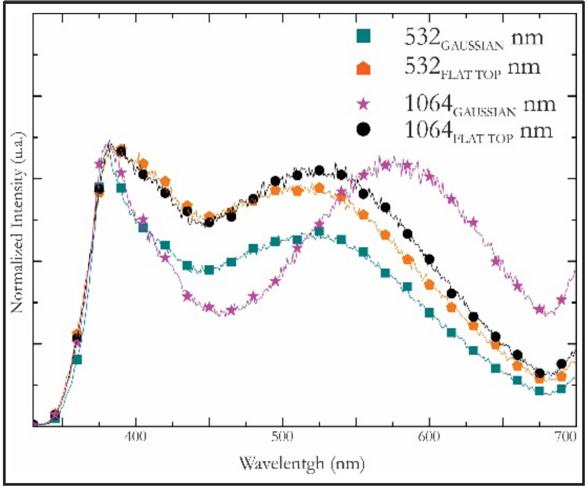
All the films exhibits an oxygen deficiency, highest ratio O/Zn and highest peak position shift is observed with 1064 nm Gaussian beam. In agree with the literature, in ZnO films is more probably the formation of structural defects like oxygen vacancies and interstitial zinc that interstitial oxygen or zinc vacancies (Zhu, B.L.; *et al.*, 2008). Some papers indicate that the PL peak position can indicate the type of defect in the films (Zhao, J.; *et al.*, 2007). There is an agreement in considering to UV emission is due to the transition from the conduction band to the valence band (Li, H., *et al.*, 2004), and can be affect for crystalline quality or stoichiometry (Zhu, B.L.; *et al.*, 2008). The origin of the visible components is not clear. The green band is associated at oxygen vacancies (Wang, Z,; *et al.*, 2015) and the orange-red band is associated at interstitial oxygen (Zhang Y.; *et al.*; 2004;Fan, X.M.; *et al.*, 2009). Taking in account analysis, UV shift is attributed at interstitial oxygen (Zhao, J.; *et al.*, 2007) and the visible band red-shift at interstitial oxygen (Zhao, J.; *et al.*, 2007), Wu, X. L; *et al.*, 2007; Ozerov, I; *et al.*, 2003].

Figure 4. Detail XRD pattern of the ZnO films as function of laser wavelength and beam profile.



The influence in optical properties of beam laser shape is not clear, signal intensity is lowest in flat top films, this can be explained by the lowest thickness film. **Figure 5** show Pl spectra as function of laser beam shape and laser wavelength. In films elaborated with 532 nm, the relative intensity UV/visible peak is highest in Gaussian beam; peak position shifted is not observed. In the 1064 films relative intensity is modified and the peak position shift detected in Gaussian beam is more evident. The results are in agree with the stoichiometry (**Table 1**) and structural variations mentioned previously.

Figure 5. PL room temperature spectra of ZnO films as function of laser wavelength and beam profile.



4. CONCLUSIONS

The films elaborated with 532 Gaussian beam showed an increment in the roughness surface with reduction in the deposition rate. The PL spectra exhibits s visible band shifted at green region in agree with the XPS results. The 1064 Gaussian films are polycrystalline with irregular surface formed for micro-crystals. We determined an oxygen excess in this samples associate at shifted visible band in the PL spectra. The flat top beam, a regardless of the wavelength laser, can obtain films with lowest deposition rate and smooth surfaces, with preferential growth in (101) plane. The films are non-stoichiometric but the density of the defects is the lowest. We can affirm that the laser wavelength and the beam shape is an important parameter in the PLD films, because the interaction between target-laser determined strongly the film characteristics.

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