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Pulsed laser deposition of PbTe under monopulse and multipulse regime

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ABSTRACT

The aim of this paper is to compare PLD technique with monopulse and multipulse nanosecond laser excitation. We show the feasibility of depositing nanometric layers of PbTe employing the regimes already mentioned. Each of the grown layers were characterized by XRD, EDXS, SEM, Spectroscopic Elipsometry, AFM and the thickness was measured by mechanic profilometry. We have conducted comparative experiments to show the advantages and drawbacks of making PLD with mono and multipulse nanosecond laser.

1. Introduction

Pulsed Laser Deposition (PLD) is a technique, which uses pulses of laser energy to remove material from the surface of a target. The vaporized material, containing neutrals, ions and electrons is known as a laser-produced plasma plume and expands rapidly away from the target surface (velocities typically ~10^6 cm s^{-1} in vacuum) [1,2]. Film growth occurs on a substrate upon which some of the plume material condenses. In practice however, the situation is not so simple, since a large number of variables affects the properties of the film, such as laser fluence, background gas pressure and substrate temperature. These variables allow the film properties to be manipulated somewhat, to suit individual applications. Usually, nanosecond single pulse emitting lasers are used. One drawback of using nanosecond pulsed laser is the so called selective ablation, that could conduct to a lack of stoichiometry in the ablation process. This issue could be compensated by a background gas or manipulating the target to sample distance [3,4]. This fact obviously conducts to lose the possibility of depositing layers with a controlled composition. This is worst in the cases where the target is composed by several elements, such lead telluride. To overcome the problem of selective ablation, recently appeared in the literature some studies regarding PLD realized with femtosecond lasers [5–7]. The majority of studies to date that have reported films fabricated using PLD have used lasers in the single-pulse emission regime [8]. By other way, studies have shown that the splashing effect can be significantly reduced or eliminated using a second laser parallel to the target that re-excites the plasma in the respective assisted plasma configuration [9]. Galbács et al. also observed this plasma re-excitation phenomenon using a multi-pulse regime emission laser, where each laser emission was composed of multiple pulses with an interpulse separation between 10 and 100 μs. In this case, the laser pulses were collinear, and re-excitation may have resulted from the laser-plasma interaction of the subsequent pulses or a plasma-plasma interaction, in which the domain of one or the other phenomenon depended on the ablated target and on the interpulse separation. In a previous study, the interactions reported by Galbacs et al. were confirmed using the multi-pulse regime to deposit titanium dioxide (TiO_2) thin films, demonstrating a lower rate of growth than that obtained using the single-pulse regime for the same total emission energy [10].

Recently, Nd:YAG lasers with Q-Switch device based on saturable absorbers have been used for the purpose of exciting samples for elementary analysis by Laser Induced Breakdown Spectroscopy (LIBS). In this regime, the Q-Switch consists of a Chrome-doped YAG crystal (Cr^{4+}:YAG), which is clarified during the pumping process, emitting one or several pulses in the course of a lamp excitation pulse [11]. Jedlinszki and Galbács show that in principle it is possible to reach higher levels of plasma ionization when the laser emits multi-pulse regime [12]. This can be a very interesting point in the PLD technique, as higher plasma ionization levels, with the presence of very energy-rich species, may contribute to the growth of the film by transferring energy to the process.

Lead Telluride is a thermoelectric (TE) material with potential application in electrical energy generation, cooling, and thermal

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sensing. Recently, research has shown that lead chalcogenides with narrow band gaps and face-centered-cubic structure are very promising materials for TE applications [13]. PbTe is a IV–VI compound semiconductor with the rock salt structure and a narrow band gap (E_g=0.32 eV), as many semiconductor it has important thermoelectric properties such as good figure of merit, good chemical stability, low vapour pressure, and high melting point [14–16].

As far as we now, there not not in the wide PLD literature studies done employing nanosecond lasers emitting in a multipulse regime. So the aim of this paper is to show the feasibility of depositing nanometric layers of PbTe employing a multipulse nanosecond laser. We have conducted comparative experiments to show the advantages and drawbacks of making PLD with mono and multipulse nanosecond laser.

2. Experimental setup

Nanometric layers were deposited on soda-lime glass substrates using a commercial PbTe target, whose concentration is 50% of Pb and 50% of Te. The substrates were previously cleaned with alcohol and acetone on an ultrasonic bath. In the ablation process, a Nd:YAG laser with a passive Cr4+:YAG Q-Switch was employed; the laser emits at 1064 nm and was operated at a frequency of 10 Hz. The deposition was performed at 5×10^{-2} Torr, with a target-substrate distance of 5 cm and a substrate temperature of 130 °C. The laser radiation was introduced at 45° with respect to the PbTe surface (Fig. 1).

The laser system was configured to emit in the multi-pulse and mono-pulse regimes. In the initial configuration, the laser module emitted in the multi-pulse regime, where each emission comprised three individual pulses separated by 45 μs (see Fig. 2a) and 60 ns of duration for each pulse. For the second configuration (mono-pulse), a second Cr^{4+}:YAG Q-Switch was added to the optical cavity of the laser, reducing the emission to a single 40-ns pulse (see Fig. 2b). The beam shape shown in Fig. 2 was measured by a Thorlabs instrument model: BC106N-VIS/M beam profiler. The total energy for each emission was adjusted to 104 mJ in both regimes, which fixed the working fluence at 2 J cm^{-2}. For the purpose of this paper, we call laser shot to each pulse (monopulse regime) or bunch of pulses emitted (multipulse regime) by the laser. By other way, we call pulse to each individual peak observed in Fig. 2a.

The obtained nanolayers were studied by AFM in ambient conditions using a Multimode-Nanoscope V (Veeco, Santa Barbara, CA) operating in Tapping Mode with an etched silicon probe model Arrow NCR-50 Nano World (cantilever resonance frequency: 258 kHz, force constant 42 N/m; tip radius 5–10 nm). Typical scan rates were 1–1.5 Hz. Elipsometry experiments were done with a multispectral ellipsometer in the microspot configuration – SOPRA GES5- and the data was analyzed by the use of the Winelli II software. SEM and EDX analysis were achieved with a Carl Zeiss instrument model: Gemini. Crystalline structure was determined by X-ray diffraction (XRD) using the grazing incidence configuration with the incident beam at 1.5° (X Pert PRO MRD, PANalytical). Thickness of the samples were measured with a surface profilometer, Veeco model Dektak 150.

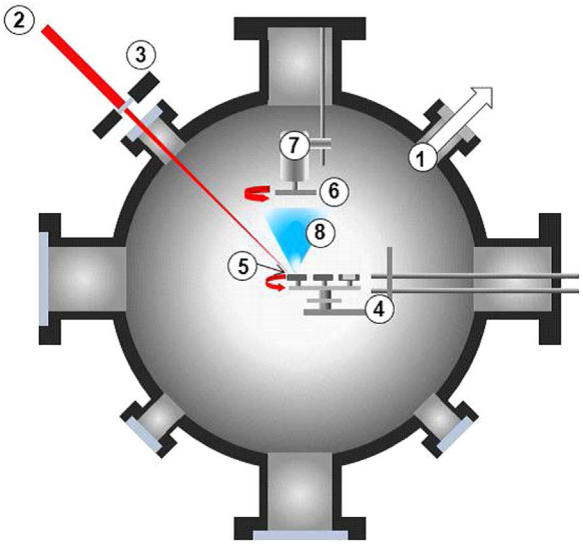

Fig. 1. Schematic of the PLD system employed. (1) Vacuum system, (2) incoming Nd:YAG shot, (3) laser focusing lens, (4) rotary target system, (5) PbTe target, (6) rectangular pieces of glass as substrates, (7) rotary substrate system, (8) laser generated plasma.

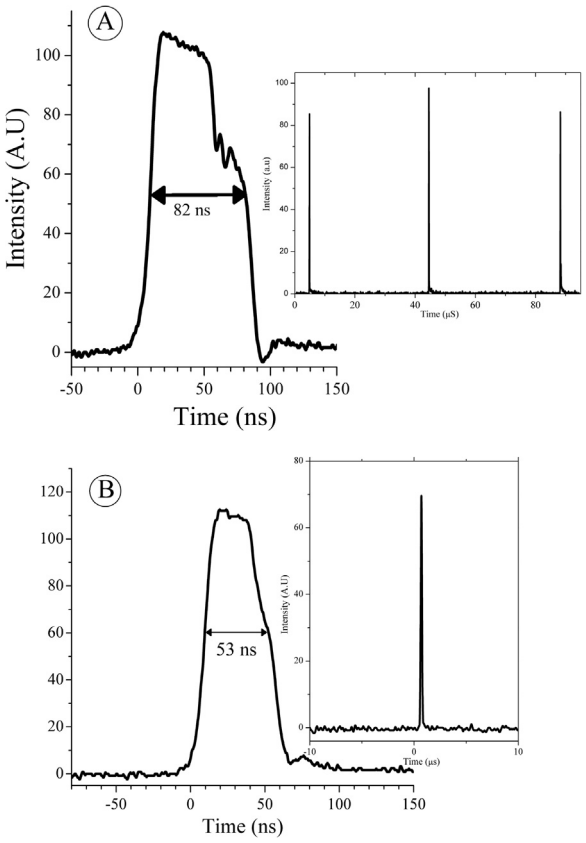

Fig. 2. Temporal profile and pulse width in the investigated ablation regimes: (a) multi-pulse and (b) single-pulse.

3. Results

3.1. Deposition rate

Deposition rate (DR) is an important parameter when thin layers are grown by PLD. This parameter measures the quantity of material deposited by each laser pulse. By definition, it is based on the laser mater interaction, so this parameter shows no linearity between the quantity of deposited material and laser pulses. However, for certain limited range this relation might be linear. To measure the deposition rate it is necessary to know the thickness of the layer and the quantity of impinging laser pulses. In the case of this paper the thickness of each sample was measured by a mechanic profilometer, which was described in the experimental section. Fig. 3 shows the thickness as a function of the number of shots employed to make the thin films. Whith this data, the deposition rates were calculated as (0.28 ± 0.025) Å/pulse and (0.16 ± 0.022) Å/pulse, for the mono-pulse and multi-pulse regimes respectively. Those data were determined by making a least square calculation with the data shown in Fig. 3. The details of all the

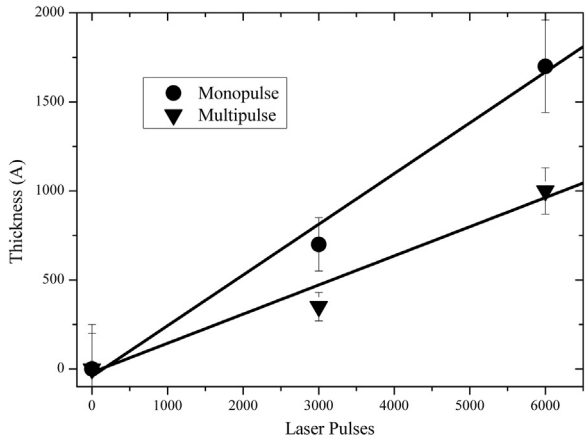

Fig. 3. Linear deposition rate curve for multipulse (triangle) and monopulse (circle) excitation regimes.

Table 1
Parameters of the linear adjustment of the data shown in Fig. 3. In brackets are indicated the standard error of the determination for each intercept and slope.

	Monopulse	Multipulse
Intercept (A)	−42.24 [104]	−19.45 [74]
Slope (A/pulse)	0.28 [0.025]	0.16 [0.022]
R	0.98	0.97

parameters of the regression are shown in Table 1.

3.2. Roughness

The roughness was measured by Atomic Force Microscopy (AFM). This parameter was measured in 6 different parts of each sample and each measure was made in an area of 5 μm^2. The measurement shown in Table 2 represents an average value of the distance between a crest and a valley. Table 2 shows that the roughness measured to the samples produced with multipulse are greater than the monopulse. At the same time, the samples produced with 6000 laser pulses show less roughness than the samples produced with 3000 laser pulses regardless the excitation regime used to produce the nanolayers.

3.3. Quantitative composition of the nanolayers

Table 3 represents the EDS results of the analysis of PbTe nanocrystals where is shown the presence of Pb and Te and their concentrations. The EDS information was acquired with excitation values less than 1.00 keV. The data shows that multipulse generated layers are nearly stoichiometric while the situation is different in monopulse regime. Monopulse excitation regime shows that the ablation does not follow stoichiometry of the target (which is 50% of Pb and 50% of Te).

3.4. Optical properties

The objective is to determine the complex dielectric function, $\varepsilon(E)$, of the PbTe deposited in both regimes, monopulse and multipulse. This

Table 2
AFM Roughness measurements of the samples under monopulse and multipulse excitation.

	Monopulse (nm)	Multipulse (nm)
3000 pulses	21.57 ± 2.57	54.03 ± 6.16
6000 pulses	8.99 ± 1.50	30.50 ± 8.88

Table 3
Concentration measured by EDS of the Pb and Te on the nanolayers obtained with multipulse and monopulse excitation.

	MONOPULSE		MULTIPULSE	
PULSES	**Pb (%)**	**Te (%)**	**Pb (%)**	**Te (%)**
3000	50–62	40–48	44–56	44–56
6000	50–62	40–48	48–56	44–52

optical characterization was done using spectroscopic ellipsometry (SE) at room temperature (angle of incidence θ_i=70^o and polarizer azimuth angle set at θ_p=45^o, for all cases studied).

According to the energy-band structure of the PbTe [17], the dielectric function in the photon-energy range studied (1.5–5 eV) can be described through 3 critical points (each one corresponding to the different interband transitions located at optical frequencies) E_1, $E_2$and E_3 which play an important role in the optical spectra of the thin films under study. The complete function $\varepsilon(E)$ is described taking into consideration more critical points, as E_0(known as the fundamental band gap ~0.3 eV) or E_4, $E_5$and E_6 (~6 eV) which were not included in the model for being out of the range of study. These critical points are included in the Model Dielectric Function (MDF) which uses for representing these three transitions a Damped Harmonic Oscillator −DHO- model:

$$\varepsilon(E) = \varepsilon_\infty + \sum_{j=1}^{3} \frac{C_j}{(1-\chi_j^2)-i\chi_j\gamma_j}, \text{ with } j = 1, 2, 3 \quad (1)$$

where χ_j=E/E_j and $E_j$represents the central energy of the critical point, while C_j and γ_j are the nondimensional strength and broadening parameters, respectively.

For all samples, the SE measurements have achieved an excellent accordance (R^2≅0.999) in comparison with the DHO-MDF model both in monopulse and multipulse regimes. Table 4 shows the evolution of the three critical points $E_j$as the number of pulses increments in both monopulse and multipulse regimes.

Under the described model, Fig. 4 shows the obtained $\varepsilon(E)$=$\varepsilon_r(E)$+$i\varepsilon_i(E)$ behavior for the four cases under study. It is interesting to note that while the imaginary part (Fig. 4B) remains almost invariant within the four samples; the real part (Fig. 4A) presents variations, showing a more negative character (which reminds the typical behavior of the dielectric function in metals) for the multipulse cases than for the monopulse ones. It is interesting to notice that the metal behavior of the PbTe samples is more erratic for the 3000 pulses range while for the 6000 pulses samples the curves seem to converge to the same value.

Also, in the $\varepsilon(E)$ complex function shown in Fig. 4A and B, it is possible to observe the contribution of $E_2$and E_3, while it is worth mentioning that although the E_1 critical point is out of the modeling range, its inclusion is fundamental in the model to achieve the excellent accordance obtained. Moreover, the found values for E_1 are similar to those described in literature.

3.5. XRD analysis

A typical PbTe nanolayer XRD pattern is shown in Fig. 5A (multipulse) and 5B (monopulse) for both multipulse and monopulse excitation regime. XRD analysis was applied to all the samples under study in this paper, and there were no significant differences between 3000 or 6000 impinging pulses. The spectra reveal that in both cases the crystalline structure corresponds mostly with a cubic system. In the XRD pattern, the prominent peak at 27.68° corresponds to the (200) plane of the PbTe cubic phase, indicating that the crystals are predominantly oriented in this direction. The peaks 2Θ=39.51°, 48.91°, 57.13°, 64.66° and 71.54° correspond to (220), (222), (400), (420) and (422) planes of cubic phase of PbTe respec-

Table 4
Evolution of the critical point positions as a function of the number of pulses.

PULSES	E_1 [eV]		E_2 [eV]		E_3 [eV]	
	Mono Pulse	Multipulse	Mono Pulse	Multipulse	Mono Pulse	Multipulse
3000	1.28	1.37	2.12	2.02	3.73	4.14
6000	1.2	1.21	1.92	1.93	3.56	4.13

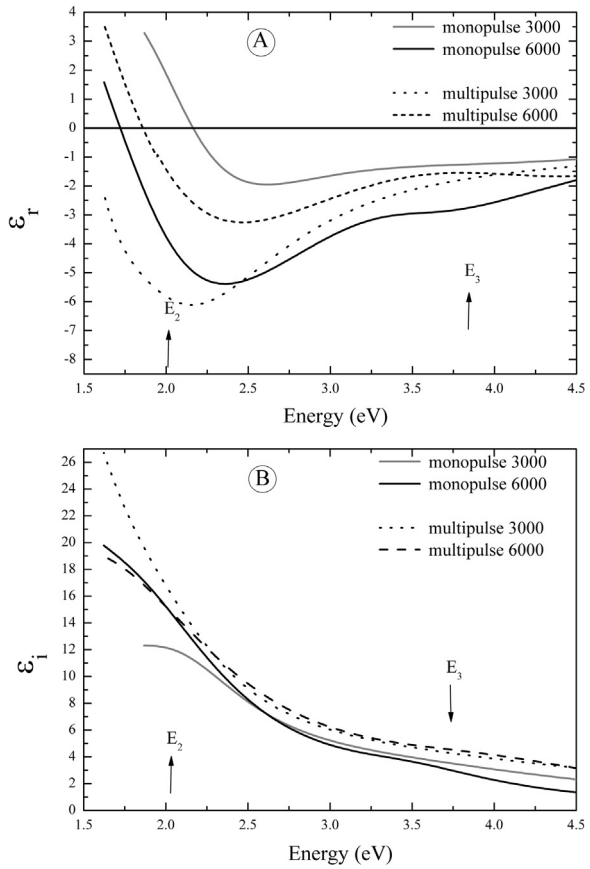

Fig. 4. Dielectric function of the PbTe samples. 3000 pulses (A) and 6000 pulses (B).

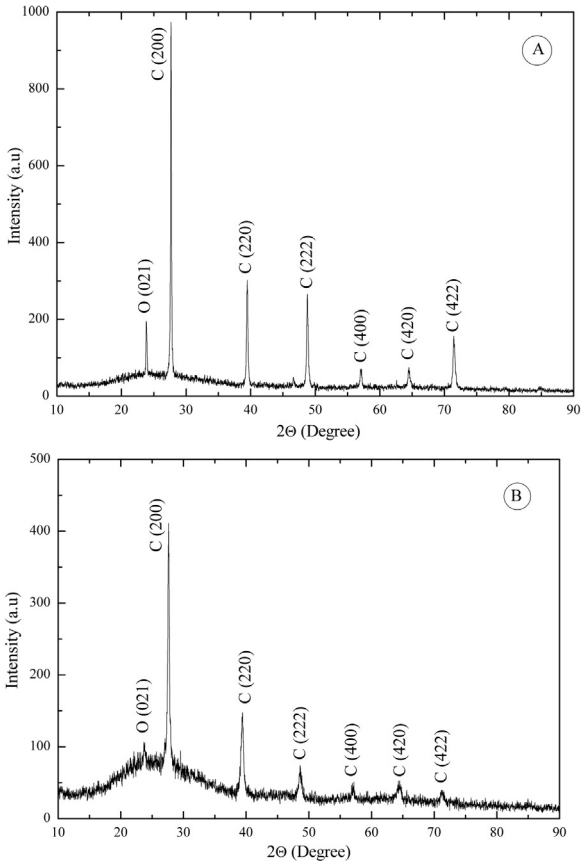

Fig. 5. XRD spectra. (A) Multipulse excitation and (B) monopulse excitation.

tively. The calculated value of the lattice constant is $a = 6.64$Å, which is in good agreement with values already published for PbTe [18]. In both spectra there is only one diffraction peak consistent with the orthorhombic system, which corresponds to the peak at 2Θ=24.77° associated to (021) plane. Finally, the spectra of mono and multipulse generated samples look very similar in their angular position, which means that the crystallography phases in both kind of samples are similar.

We also measured the FWHM of the peaks and by applying the Scherrer equation the size of the crystallites was determined. We have used the original Scherrer equation as published in 1918 (see Ref. [19]). Our findings indicate an average size of (35.5 ± 0.3) nm for multipulse and (31.9 ± 0.4) nm for monopulse nanolayers.

3.6. Morphological analysis

In literature there are 3 accepted growing layer models for thin films. Fig. 6 schematizes these models known as Frank-van der Merwe mode (the layer is formed uniformly over the substrate), Volmer-Weber mode (the layer growths as isolated island) and the Stranski-Krastanov mode (which, more less is a combination of the last two). Fig. 7 shows images obtained by scanning electron microscopy (SEM) of the films grown with 3000 and 6,000 shots, with a mono-pulse and multi-pulse regime, respectively. According to these SEM pictures, lead telluride layers grow in different ways, whether the excitation is a monopulse or a multipulse laser shot. When 3000 monopulse shot are impinging over the target, the growing mode is similar to the Stranski-Krastanov mode (see Fig. 7C). However, the observed islands are not present for the same number of shots in multipulse regime (see Fig. 7D). For the case of 6000 laser shot, the layer is thicker than in the case before, so that could be the reason not to observe the isolated islands. In the case of 6000 laser shot and multipulse excitation some droplets are observed on the PbTe layer (see Fig. 7B), while in the case monopulse excitation the amount of droplets are considerably lesser (see Fig. 7A). The formation of these droplets in these thin films is known as splashing. In particular, it can be noted the peculiar “elongated drop” shape of the bigger particles in the thin film grown with mono-pulses, whose origins are still being determined. The thin films made by multi- pulse ablation have a higher density of particles than the mono pulse ablation regime, the particles size are between 30–280 nm, with 50 nm of average size. While in mono pulse thin films the particle size is between 30 and 600 nm, with average size of 100 nm. A quantitative size distribution is unviable, due to the irregular form of the particles in thin films made with single-pulse ablation.

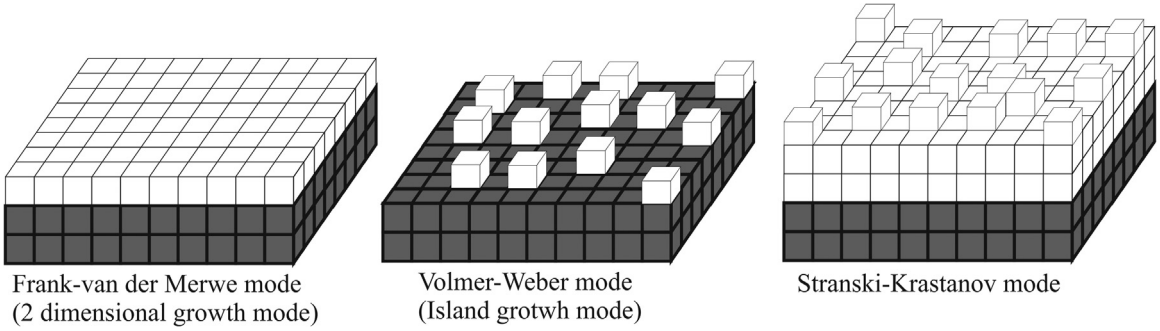

Fig. 6. Schema of the accepted mode of growing of the layers over differents substrates.

4. Discussion

The aim of this paper has been to establish a comparison between monopulse and multipulse excitation regimes for nanolayer growing in pulsed laser deposition method. One of the main parameter to take into account in this comparison is the so called deposition rate (DR). This parameter is experimentally determined and gives a thought about how much of the material is being deposited over the substrate for each incident laser pulse. The DR is a parameter that depends on the incident laser wavelength, the applied laser fluence, the duration of the laser pulse (nanosecond, femtosecond, picosecond) and as we shown in this paper it depends also on mono and multi pulse excitation regime. A greater DR does not mean that one excitation regime is better than other. In other words, if the experiment requires a growing of a layer in as short time as possible with a poor control over the thickness so the DR should be as greater as possible. In other way, if the experiment require a carefully control over thickness of the nanolayer a lesser value of the DR should be perfectly acceptable. In our experiments we found that the DR for monopulse excitation regime is 0.28 A/pulse and in multipulse regime this value is 0.16 A/pulse. At the best of our knowledge in literature there is only 1 paper dealing with multipulse PLD, the measured DR in that paper is in agreement with our results [10]. A possible explanation for this result could be given in terms of the time separation between pulses in multipulse ablation regime. The separation between the pulses in a multi-pulse shot is similar to the average life-time of the plasma [20]. The ablation process starts when the first pulse of the burst imping on the target. The second pulse in the burst arrives 45 μs later while the plasma is still in the extinction process. So a laser plasma interaction occurs. During this interaction, a fraction of the energy of the pulse can be absorbed by the species still present in the plasma. Consequently, the energy impacting on the target is less and, therefore, the amount of extracted material is also less. This pulse-plasma interaction occurs in single pulse regime but in a much lower intensity. The interaction in this last case is between the last nanoseconds of the laser pulse and the first moment of the recently formed plasma.

Another characterization that could be expected to show some differences when the nanolayers are grown under mono or multipulse excitation is the crystalline structure. To clarify this situation we have conducted XRD experiments of the samples, and as it is shown in

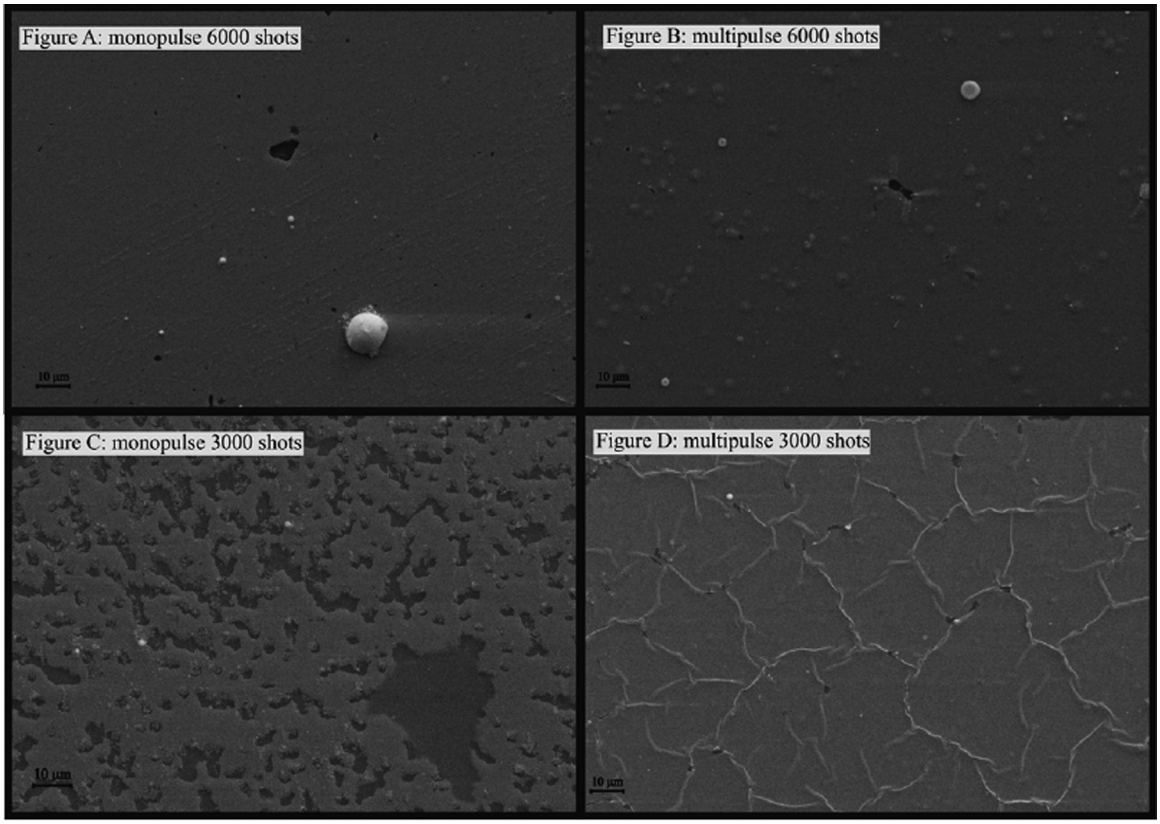

Fig. 7. SEM images of the PbTe deposited layer over glass in a monopulse and multipulse regime.

Fig. 5A and B the diffraction peaks of the samples show no differences. Except for the peak observed at 24° 77' which correspond to an orthorhombic crystalline structure. The rest of the peaks shown correspond to a cubic crystalline structure. This means that the excitation regime has no effect on the crystalline structure whether it be mono or multipulse.

The samples show a roughness which is greater in multipulse regime than in monopulse. A possible explanation for this fact is the presence of particulates or splashing, which as Fig. 7 shows, is bigger in the films grown with multi-pulses. The presence of these micrometric particles in the initial stages of growth of the nanolayers may bring about an accumulative effect of defects on the final surface of the film, thus causing an accelerated increase in the roughness. In addition, it was suggested that the film roughness is influenced by strain induced in the film by a lattice mismatch with the substrate, this affect the roughness in both ablation regimes [21].

Several papers deal with the stoichiometric capabilities of PLD reaching to controversial results. In our results we have found a lack of stoichiometry when the nanolayers are grown with monopulse excitation, while the opposite result was found when the nanolayers are grown under multipulse excitation, which means that the transfer of material from target to substrate is stoichiometric. This result is shown in Table 3. The lack of stoichiometry during the ablation process under a monopulse regime is a matter of discussion in the most up to date literature on laser ablation scientific community. One of the most accepted explanation includes the concept of selective ablation. This means that for a binary alloy, during the ablation process under nanosecond monopulse laser excitation one of the species is selectively extracted by the laser pulse. This selectivity is fully related with the wavelength of the excitation pulse, the duration of the pulse (selective ablation was not shown under femtosecond or picosecond laser excitation) and material properties. It is an important fact of this work, that nanolayers stoichiometry in films grown by multipulse nanosecond laser pulses, has been preserved from the target to substrate. As far as we know this is the first time that a similar result is reported, so we will try to give a possible explanation of this fact. Let´s suppose the four pulses that compose the burst interact completely with surface, this means that we are neglecting the interaction between the second, third and fourth pulse with the plasma generated by the first pulse. In this scenario the first pulse will ablate the surface and will extract 56% of Pb and 44% of Te, leaving the surface of the target enriched in Te (the new composition could be 44% of Pb and 56 of Te). The second pulse will interact with this Te enriched surface, and will produce a selective ablation of the same order that the first pulse. But, since the new surface is already enriched in Te and impoverish in Pb, the quantity of extracted Pb will be less than in the first pulse. By reproducing the same effect with the third and fourth pulse, the overall result would be a balanced extraction of material which conducts to a stoichiometric composition in the grown nanolayer. Of course this explanation needs to be tested experimentally and those experiments will be done in the near future in our labs.

Another important parameter registered in our lab was the determination of the optical properties of the nanolayers grown under different regimes of excitation. In this regard, it is interesting to notice that in both regimes, as the number of pulses increases from 3000 to 6000 pulses, all three critical points decrease their value, approaching to the bibliographic reported values. These results indicate that the morphology of the thin films is dependent of the number of cycles, the optical results suggest that for 3000 pulses, the system is not completely homogeneous probably due to the formation of islands what indicates a Volmer-Weber or Stranski-Krastanov mode formation. However, the decrease of the critical point values should indicate, as already shown in the SEM images of Fig. 7c a more homogeneous formation of the thin film more like a Frank-van der Merwe mode.

5. Conclusions

In this paper, we have compared the monopulse and multipulse pulsed laser deposition technique. We have made several characterizations on the PbTe nanolayer grown under both excitation regimes. We have applied perfilometer analysis (determination of the deposition rate), atomic force microscopy (to determine the roughness), EDS (to measure the quantitative composition), Elipsometery (to determine some optical properties), XRD (to determine the crystalline composition) and SEM (to determine some morphological aspects of the samples).

The samples grown under monopulse excitation have higher deposition rate and have lower roughness than the multipulse samples. So it seems that monopulse excitation is more adequate than multipulse, when roughness is important. However when quantitative composition of the samples are analyzed, it seems that monopulse samples do not follow the stoichiometry of the target, while the multipulse do. Clearly this result cannot be extrapolated to any kind of sample; however it can be indicative of the behavior of the multipulse excitation.

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