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SYNTHETIC PLUMBONACRITE THIN FILMS GROWN BY CHEMICAL BATH DEPOSITION TECHNIQUE

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This work it has the goal to present a way to grow synthetic plumbonacrite thin films. Our characterization started with XRD, from where we get a hexagonal structure of the Plumbonacrite corresponding compound. The following characterization considered was the absorption spectra in the visible region and from the energy band gap was calculated Eg=1.8 eV. Our reaction conditions lead to a thickness around of 375nm of the films, measured by ellipsometry and the resistivity of these thin films was measured giving around 110 MΩ. Also we did X-ray Photoelectrons Spectroscopy which probed that the thin films are mainly composed by lead and oxygen. Finally we are reporting the surface morphology through AFM were it can be observed the roughness at large scale.

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1. Introduction

There are several techniques to prepare the lead oxide thin films for instance Autocatalytic Oxidation [1], reactive DC magnetron sputtering [2], spray pyrolysis [3], and chemical bath deposition [4, 6]. The last technique is preferred when is important to get inexpensive and simply thin films. All of this determines new materials routes with different characteristics. This work shows an alternative way to get lead hydroxide thin films that have similar characteristics than reported in [4].

The XPS studies reveal that these thin films are formed mainly of Lead and Oxygen, the hydrogen element is no showed due to the inability to detect it. However the white colour is of the lead hydroxide thin film like is reported in [4]. The diffraction X-ray studies confirmed that our thin films correspond to an hexagonal face of the Plumbonacrite compound. The thickness of the film is about 375nm, the band gap is direct of 1.8 eV like is reported in [3] and the thin films are highly resistive around 110 MΩ, due to the band gap value these thin films have applications like an optical window. In the next section we can observe the experimental process of the synthesis of the film, after that, we going to talk about the main results.

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2. Experimental

The deposition of Plumbonacrite films was done on glass slide substrates, immersed in a reactive solution prepared into a 100 ml beaker by the subsequent addition of 5 ml of 0.5M lead acetate, 5 ml of 2M sodium hydroxide, 2ml of 1M triethanolamine, the solution was diluted adding 82 ml of deionized water, after it is added 6 ml of a solution of Rongalite-NaOH, finally 5 drops of a buffer (NH_4OH/ NH_4Cl) were added. We use of 1 to 20 hours and 54 ^oC as condition to grown the films. In the second step the appearance of the solution is milky white, but after to add another reactive the solutions start to change to transparent and after makes turbid.

Optical transmission spectra of the bilayer were recorded by an Ocean Optics USB4000-UV-VIS spectrometer in the 280- 850 nm wavelength range, and a XPS (Perkin-Elmer Phi-5100) model used to the chemical composition of the thin films. The X-ray diffraction measurements were performed using a Rigaku Ultima III diffractometer.

3. Results

The Plumbonacrite thin films were grown by the chemical bath deposition at 54^oC this films were white after the finished process of 20 hours like is reported in [5].

The thickness of the Plumbonacrite thin films is more uniform for the thinnest films. The thin films of 20 hours of growth have more variation in the thickness than the thin films of 6 hours, we can therefore affirm that the films have a polycrystalline growing.

The thickness for the 6 hours thin film is about 375nm while for the 20 hours thin film is around of 750 nm.

a) XRD analysis

In the figure 1 the spectrum of X-Rays Diffraction is shown, the indices on some diffraction peaks of our thin film fit with the hexagonal crystalline phase of the Plumbonacrite, reported as the database C 96-900-9519 of the MATCH! version1.10, this film was highly oriented in the plane (220).The XRD pattern was measured in the range of 15 to 50°and fitted with the referred pattern, the result analysis shown the peaks with intensity highest than 200, for instance the predominant peak is located in 26.55, the next in 34.09, the third in 20.8 and the last in 36.05 respectively, therefore this material correspond to polycrystalline film like is reported in [6].

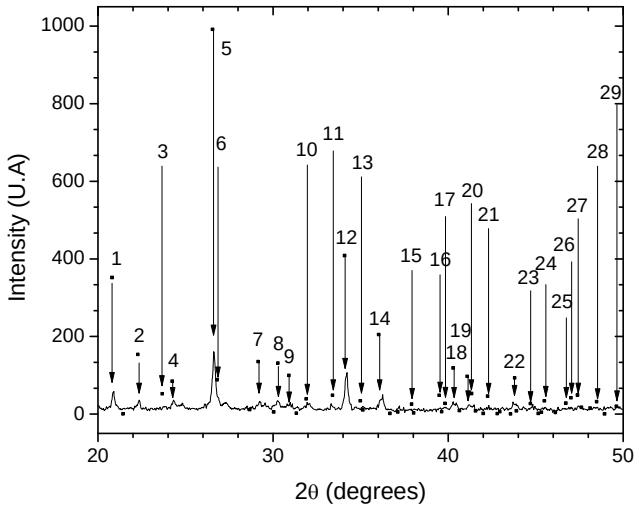

Fig. 1. Here it is displayed the spectra of XRD for the Plumbonacrite thin films developed in this work.

Table1. Associations of intensity peaks for Plumbonacrite thin films.

peak number	2θ	Intensity
1	20.8	351.9
2	22.28	153.8
3	23.66	52.2
4	24.24	84.5
5	26.55	991.7
6	26.8	88.3
7	29.14	135
8	30.27	131.7
9	30.9	99.4
10	31.89	39.2
11	33.4	48.6
12	34.09	408.2
13	34.98	34.1
14	36.03	204.6
15	37.91	25.4
16	39.48	48.7
17	39.82	27.2
18	40.29	118.9
19	41.08	97
20	41.34	52.6
21	42.26	45.9
22	43.8	92.7
23	44.69	27.1
24	45.5	34
25	46.72	28.3
26	47.02	41.7
27	47.39	49.4
28	48.48	31.8
29	49.62	19.6

The table 1 shows the intensity and the 2θ angle of each one of the 29 identified peaks.

b) Optical Response

In the figure 2 the spectra of the absorbance, reflectance and transmittance are shown, in the case of the absorbance this is approximately constant between 400 to 500 nm, after that, the absorbance is decreased with the wavelength.

From the transmittance spectrum, which is increasing almost constantly with the wavelength after 500 nm, this is a good approach to consider an edge for compute the energy band gap of the thin film.

The figure 3 was used to compute the energy band gap, considering direct transitions of the synthetic lead oxide thin film, using the intersection of the straight line with the y axis divided by the slope of a linear fit, the fit parameters are showed in the inset of the same figure 3, the obtained value was Eg=1.8 eV.

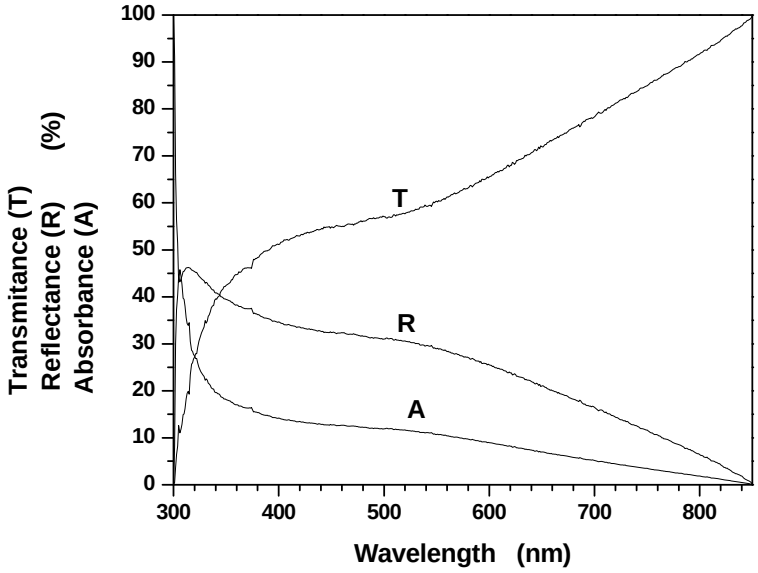

Fig. 2 Are displayed the spectra of the absorbance, reflectance and transmittance for the Plumbonacrite thin films developed in this work.

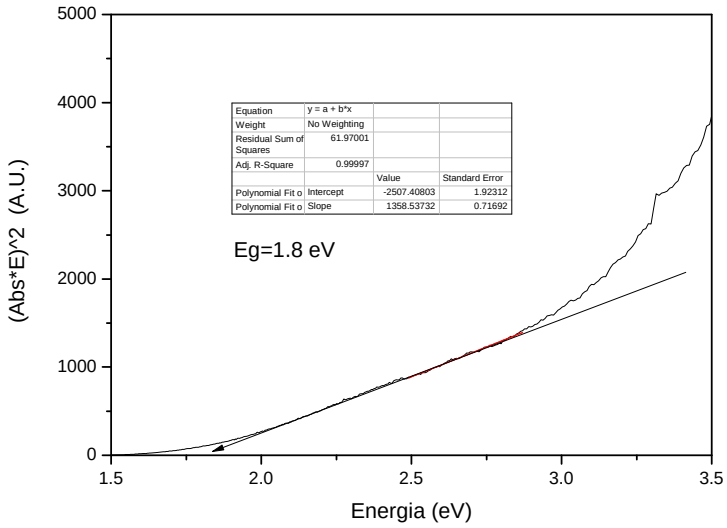

Fig. 3 The square of absorption times energy versus the energy, in order to calculate the energy band gap.

c) XPS Chemical analysis

Figure 4 shows the XPS spectrum of the synthetic plumbonacrite thin film, this spectrum has been done to determine the chemical composition. The oxygen and carbon are two common components due to ambient; nevertheless the oxygen is expected in the chemical composition of the synthetic plumbonacrite thin films. We found the main energies corresponding to lead and oxygen.

In figure 4 the 4, 5, and 7 peaks correspond to oxygen and the 6, 8, 9, 13, 14, and 15 correspond to lead that is expressed in the reduced form in the table 2, respectively.

The leveled 1, 11 and 12 peaks corresponding to Na, Cl and S, respectively, that are precursors of the initial formulation.

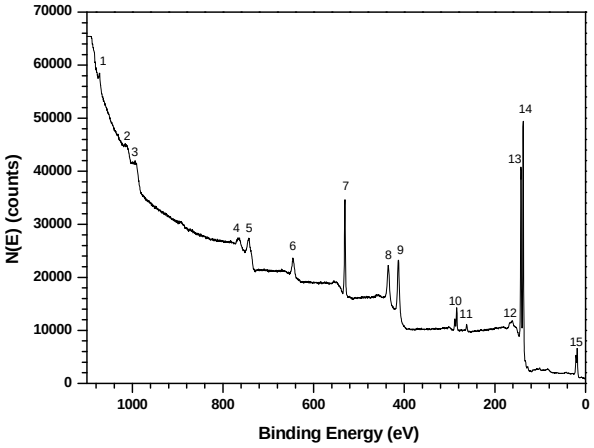

Fig. 4. The spectra of XPS for the synthetic Plumbonacrite thin films developed in this work.

Table1. Associations of level binding energies for Plumbonacrite thin films.

Label	Plumbonacrite	Binding Energy (eV)
1	Na 1s	1073
2	C KVV (Auger)	1013
3	C KVV (Auger)	994
4	O KLL (Auger)	764
5	O KLL (Auger)	743
6	Pb 4p3/2	646
7	O 1s	531
8	Pb 4d3/2	435
9	Pb 4d5/2	413
10	C 1s	284
11	Cl 2s	262
12	S 2p	163
13	Pb 4f5/2	143
14	Pb 4f7/2	138
15	Pb 5d5/2	19

d) AFM morphology of the Plumbonacrite Thin film

In figure 5 we can observe some 2D and 3D surface morphological details of the synthetic plumbonacrite thin film by mean of AFM micrographic characterization, in the part a) the micrograph was taken in an area of 100 μm^2 and the part b) it is the complement in 3D, that exhibits the roughness nature with highest peak around of 1.3 μm, the images of the figure 5 was processed using an image processor [7].

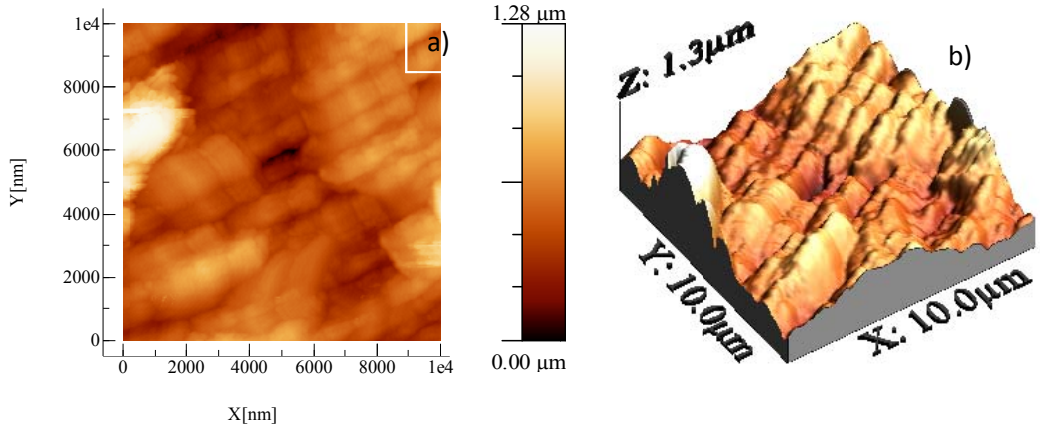

Fig. 5. Shows the 2D and 3D surface micrographs of AFM for the synthetic Plumbonacrite thin film.

The RMS roughness is estimated around 0.18 µm with maximum value of 1.28 □m with less number of events like is observed in the figure 6.

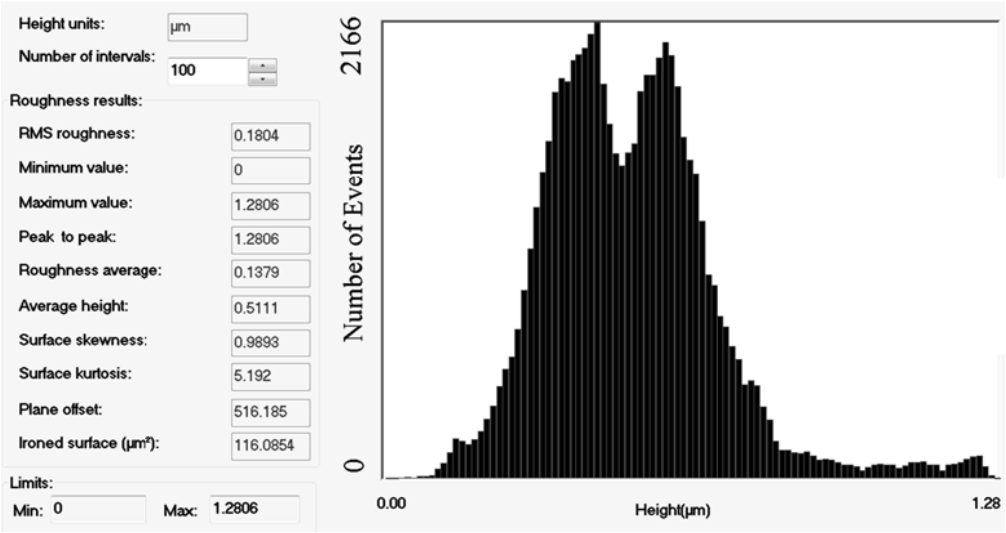

Fig. 6. It is shown the RMS roughness of the Plumbonacrite thin film corresponding to the figure 5.

e) Resistivity measurement

For the resistivity measurement were located two parallel electrodes of silver paint of 0.8 cm of length and 1 cm of separation, it can be seen in figure 7. For this measurement we needed thickness and this was measured by ellipsometry being of 375 nm.

So, the calculated value for the resistivity was 110 MΩ.

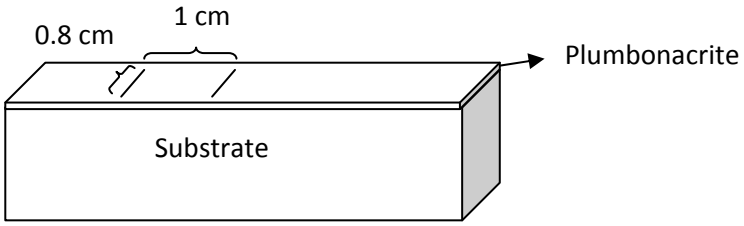

Fig. 7 Arrangement for measuring the resistivity of the synthetic lead oxide.

4. Conclusions

We obtained Plumbonacrite thin films by the chemical bath deposition, the synthesis is relatively new compared with the one reported in [4], the thin film has a direct band gap of 1.8 eV, a resistivity of the order of 110 MΩ, a thickness of 375nm, and the studies of surface chemical analysis (XPS) confirm that the main components of this thin film are Pb and O, while the XRD characterization fit with Plumbonacrite polycrystalline phase compound, the appearance of the final film is white, in the case of the XRD the powder is burned during the measurements. This film could be used like an optical window for low energies of the visible region. AFM measurements indicate that the film material is quite rough, 180nm.

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