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Effects of Relatively High Temperature on Thin Films of PbS Fabricated by Chemical Bath Deposition Method

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Abstract: Chemical bath deposition (CBD) method was used to deposit thin films of PbS. In the experiments, PbS was obtained at four temperatures which are 55, 65, 75 and 85 °C. The effects of relatively high temperature were investigated in detail. The structural properties of the PbS films were examined by using X-ray diffractometer (XRD). According to the XRD, it was found that when temperature was increased, the preferred orientation shifted from (111) plane to the (002) plane at a deposition temperature higher than 75 °C. Besides, crystallite sizes (cs), lattice constants (a), average stresses (S), micro strains (ε) and dislocation densities (δ) were obtained from the XRD results. Surface properties of the PbS thin films were researched by using scanning electron microscope (SEM). It was seen from the SEM images that there was crack on the surface of the films when the film was obtained at 55 °C. On the other hand, no cracks were observed in the SEM images of PbS films of 30000 magnifications produced at temperatures higher than 55 °C.

Göreli Yüksek Sıcaklığın Kimyasal Biriktirme Yöntemi ile Üretilen Pbs İnce Filmleri Üzerindeki Etkileri

Anahtar Kelimeler
Kurşun sülfür,
İnce film,
Kimyasal banyoda büyütme,
Sıcaklık

Özet: Kimyasal biriktirme yöntemi (KBY), ince film PbS'yi depolamak için kullanılmıştır. Deneylerde PbS, 55, 65, 75 ve 85 °C'lik dört farklı sıcaklıkta elde edilmiştir. Nispeten yüksek sıcaklığın etkileri detaylı olarak incelenmiştir. PbS filmlerin yapısal özellikleri X-ışını difraktometresi (XRD) kullanılarak araştırılmıştır. XRD'ye göre, sıcaklık arttığında, 75 °C'den yüksek depozisyon sıcaklığında tercihli yönelimin (111) düzlemden (002) düzleme kaydığı tespit edilmiştir. Ayrıca XRD sonuçlarından, kristalit büyüklükleri (cs), örgü sabitleri (a), ortalama gerilmeler (S), mikro gerilimler (ε) ve dislokasyon yoğunlukları (δ) elde edilmiştir. Üretilen PbS ince filmlerin yüzey özellikleri taramalı elektron mikroskobu (SEM) kullanılarak araştırılmıştır. SEM görüntülerinden, PbS filmler 55 °C'de elde edildiği zaman yüzeylerinde çatlak olduğu anlaşılmaktadır. Diğer yandan, 55 °C'den yüksek sıcaklıklarda üretilen PbS filmlerin 30000 büyütme oranındaki SEM görüntülerinde çatlak olmadığı görülmüştür.

1. Introduction

Semiconductor thin films have been widely investigated because of their unique electronic and optical properties compared to their bulk material. The optical properties of thin films depend on surface characteristics, shape, crystallite size, and other variables such as doping [1].

Lead sulfide (PbS) includes a group of IV–VI semiconductors with relatively narrow band gap, such as 0.41 eV at room temperature and is fabricated for mid-IR detectors. Besides, recently, PbS has been used in solar cells, thin film transistors, and nonlinear optical devices [2].

In the literature, there are many researches on the preparation methods of PbS, such as successive ionic layer, pulse electrodeposition, adsorption and reaction, spray pyrolysis, thermal evaporation, galvanic method, solid-vapor deposition, atomic layer deposition, and chemical bath deposition (CBD).

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Among these methods, CBD is a low-cost method and the simplest experiment to produce thin films of PbS [3].

The mechanisms of reactions for PbS precipitation that take place during the production of PbS by chemical bath deposition are as follows [4]:

$$Pb(NO_3)_2 + 2NaOH \rightarrow Pb(OH)_2 + 2NaNO_3 \tag{1}$$

$$Pb(OH)_2 + 2NaOH \rightarrow Na_2[Pb(OH)_4] \tag{2}$$

$$[Pb(OH)_4]^{2-} \rightarrow Pb^{2+} + 4OH^- \tag{3}$$

$$CS(NH_2)_2 + OH^- \rightarrow CH_2N_2 + H_2O + HS^- \tag{4}$$

$$HS^- + OH^- \rightarrow H_2O + S^{2-} \tag{5}$$

$$Pb^{2+} + S^{2-} \rightarrow PbS \tag{6}$$

There are several studies in the literature on PbS films grown onto glass and other substrates at different temperatures by CBD.

Spectrophotometric studies with UV-vis-NIR have shown that the direct band gap is lying in the range of 2.4-2.81 eV for the films deposited at 1 hour of deposition time at 30-10 °C. It has been reported that the stoichiometry improved by the decrease of deposition temperature [5].

In one study, chemical bath deposition was performed using lead nitrate [$Pb(NO_3)_2$], sodium hydroxide (NaOH), and thiourea [$SC(NH_2)_2$] composition in deionized water at room temperature, at proportion and concentration of 1: 3: 1: 12 and 1/2: 1:1 (mol/l), respectively. However, for this chemical composition, the lead ions needed for the formation of PbS were found to be available only within the first 30 min after the preparation of the solution. After this period of time, PbS film thickness remained unchanged. A preferential orientation in the plane of the PBS thin films produced (2 0 0) was also observed [6].

In another study, four PbS films of five different molarities used for lead acetate and thiourea were prepared at different pH (10.5 and 11) and precipitation temperatures (318 and 333 K). It was found in all XRD spectra that the intensities of (111), (220) and (311) peaks were low compared to the intensities of (200). This is related to the orientation of the grain growth, preferably along the (200) direction. In the same study, it was observed that dislocation density and micro-strain decreased with increasing molarity, pH, and deposition temperature. In addition, by the increase of molarity, pH, and deposition temperature, the average crystallite size was found to increase, but the micro strain and dislocation density decreased [7].

In a different research, PbS films were produced at different deposition temperatures of 40, 60 and 80 °C. According to the XRD analysis of the produced films, the intensities of all peaks tended to increase significantly along with the temperature, since the crystallinity of the films were boosted very sensibly with a small change in temperature. From surface SEM images of PbS thin films deposited at different temperatures, the temperature dependence of grain size was clearly visible. All PbS films demonstrated dense packed microstructures without voids or cracks. Smaller discrete grains were observed dispersed on the surface of the films deposited at 40 °C, while faceted larger grains could be detected in films produced at higher temperatures up to 80 °C [8].

Another research looked into the structural and optical properties of chemically deposited PbS thin films at various temperatures from 60 °C to 80 °C within 4 hours. It was observed that the crystalline of films were improved when the temperature was increased from 60 °C to 80 °C. It was reported that RMS value increased slightly by increasing the temperature. Further, it was found that the grain size of the PbS films increased by the rise in deposition temperature. The SEM micrograph made it clear that the film was uniform and compact and was well adhered to the substrate. Besides, the substrate surface was well covered with spherical grains, mechanism as revealed by AFM. [9].

According to some earlier studies, the initially intrinsic surface energy of the (111) facet was higher than the (200) facet for PbS nanocrystals [10-12]. Furthermore, as revealed by some studies, relatively primary growth occurs along the (111) direction at low temperature (300 K). According to these studies, this was because the interaction strength with the (111) facet was weak at low temperature, and interaction strength was increased as reaction time extended, and this could efficiently decrease surface energies of the (111) facet. Therefore, the lower the growth along the (111) facet, facilitate the growth along the (200) facet [13-15].

It has been reported by Gadave et al. that the orientation of the PbS films with the CBD (200) orientation is the preferred orientation [16]. According to the study of PbS films produced at 80 °C for 3 hours, it was found that the direction of (200) perpendicular to the surface of film in bottom substrate was the preferred orientation [17].

Another study reported that the surface aspects of the SEM images of the PbS films were composed of uniform nanorods structures for the synthesis temperature of 55 to 70 °C; nevertheless, the nanorods size increased slightly with an increase in the synthesis temperature. With a further increase in the synthesis temperature to 80 °C, the nanorods structure broke up into mixed structures with the emergence of spherical nanoparticle and plate-like shapes [18].

In a literature review, the effect of deposition parameters on the kinematics of deposited films was investigated resulting in four factors of deposition rate. These factors were defined as deposition time, temperature of deposition bath, pH of the deposition bath, and the concentration of constituents. The higher the deposition temperature, the higher the reaction rate was according to the results of this study. It was also reported that the longer the deposition time, the higher the deposition rate was. In addition, the higher the pH of the deposition bath, the greater the reaction rate was. The growth rate was achieved by controlling the amount of S^{-2} ions in the reaction mixture so that the growth rate could be controlled "fast" and "slowly" [19].

A research by Altıokka, it was found that the precipitation rate of PbS was an important parameter for obtaining thin films at good quality. In addition, a kinetic model for the rate of precipitation of PbS was developed. It was resulted that PbS has an autocatalytic effect since it acted as a nuclei. This study showed that crystallization and surface morphology were highly dependent on reaction rate for producing PbS films by the CBD method. For this reason, the use of an inhibitor was suggested to slow down the precipitation rate [12].

This research presented the effects of deposition temperature on the structural and morphologic properties of PbS thin films. It was discovered that deposition temperature had great effects on preferred orientation and morphology. According to the XRD analysis of the produced films, the intensities of all peaks tended to increase significantly along with the temperature; and this was because the crystallinity of the films was boosted very sensibly by a small change in temperature. It was determined that the preferential orientation at the deposition temperature higher than 75 °C changed from (111) plane to (002) plane. It was also reported that cracks were not observed in the SEM images of 30000× magnifications of PbS films produced at temperatures higher than 55 °C.

2. Material and Method

100mL aqueous solutions of 0.0089M lead nitrate ($Pb(NO_3)_2$), 0.15M sodium hydroxide (NaOH) and 0.051M thiourea ($CS(NH_2)_2$) were used for the deposition of PbS thin films. All solution was prepared at 11.5 pH. The glass substrates and bath container were cleaned with 5% (w/w) hydrochloric acid and were washed with deionized water afterwards. The depositions duration was 45 min. The depositions were carried out at four different deposition temperatures, which were 55±1 °C, 65±1 °C, 75±1 °C and 85±1 °C.

Gravimetric method was used to calculate film thicknesses, while a PANalytical Empyrean XRD was used to analyze structural properties of the PbS thin films. A Zeiss SUPRA 40VP SEM was used to investigate the surface morphology of the films.

3. Results

3.1. Structural studies of PbS thin films

Gravimetric method was used for calculate of film thicknesses. The thicknesses of the films were calculated as 750 nm, which showed that deposition temperature did not affect film thickness. This was an expected result as the temperature did not affect the number of ions, and therefore, film thicknesses. In a different research in the literature, it was observed in the experiment in which no inhibitor was used in the deposition solution that the lead concentration at room temperature was almost complete in 50 minutes [12]. Lead concentration increases earlier as the deposition temperature increases the reaction rate. Consequently, the lead concentration in all experiments will be almost complete. For this reason, the film thicknesses in all experiments will be almost the same. As there is no lead in the deposition solution, all experiments will be completed within 45 minutes. It is reported in the literature that lead concentration is over even at 30 °C [6,12].

Figure 1 shows the XRD pattern of PbS thin films. XRD patterns show that all films formed in galena type cubic form. Texture coefficients (TC) given in Equation 7 were employed to determine preferred orientation. The TC values are given in Table 1. According to the TC values, preferred orientation varied from (111) plane to the (002) plane by means of the rise in temperature.

$$TC = \frac{I_{(hkl)}/I_{0(hkl)}}{\frac{1}{N}\sum_N(\frac{I_{(hkl)}}{I_{0(hkl)}})} \tag{7}$$

where $I_{(hkl)}$ is the observed relative intensity of a plane (hkl), $I_{0(hkl)}$ is the standard intensity of the plane (hkl) given in ASTM card [20]. It is found in the XRD spectra of the film produced at 85°C that the intensities of (111), (220) and (311) peaks were low compared to the intensities of (002). This is related to the orientation of the grain growth, preferably along the (002) direction. According to some studies in the literature, the initially intrinsic surface energy of (002) facet for PbS nanocrystals is smaller than (111) facet [10-12]. Moreover, some studies [13-15] documented the occurrence of relatively primary growth along the (111) direction at low temperature (300 K). For this reason the interaction strength with (111) facet was poor at low deposition temperature, and when deposition temperature was increased, the reaction rate increased subsequently, and therefore, interaction strength increased too. This could decrease surface energies of (111) facet [13,14,15]. Because of this, the growth of the PBS films (111) along the facet is reduced, facilitating the growth of the (002) facet.

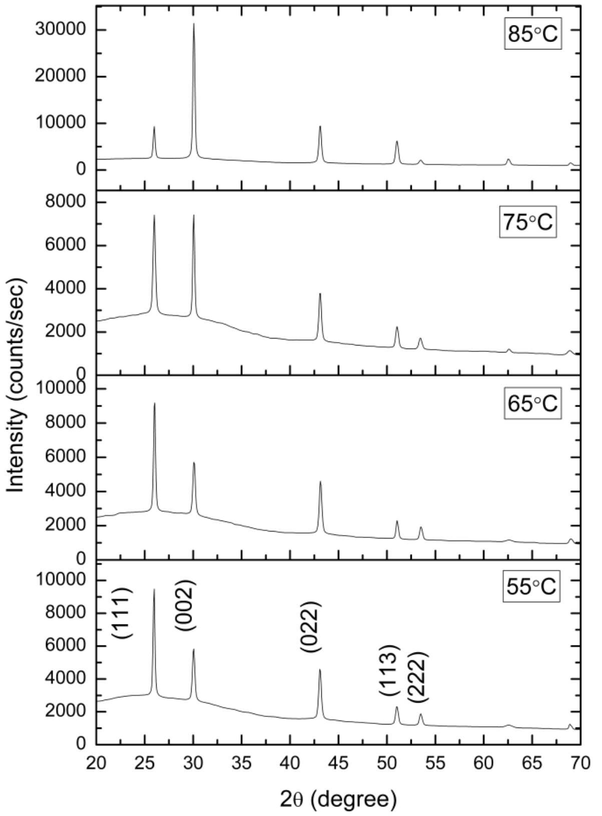

Figure 1. X-ray diffractograms of PbS thin films according to bath temperature.

Table 1. Texture Coefficients according to different planes

Experiments	55°C	65°C	75°C	85°C
T.C.(111)	1.126	1.082	1.013	0.758
T.C.(002)	0.847	0.854	1.021	1.265
T.C.(022)	0.785	0.722	0.687	0.642

Debye Scherrer equation was used to calculate crystallite size and they are demonstrated in Table 2. Debye Scherrer equation is given in Equation 8.

$$\mathrm{cs} = \frac{0.089\ \ 180\ \ \lambda}{314\ \ \beta\ \ \cos\theta_C}\ \mathrm{nm} \tag{8}$$

where β is the full width half maximum, $2\theta_C$ is the peak centre, λ is the wavelength of X-ray radiation (1.54056 Å), β and $2\theta_C$ were obtained by fitting the XRD peak profile [21]. When Table 2 is examined, it can be seen that crystallite size varies from 31 nm to 42 nm.

Table 2. Calculated crystallite sizes

Experiment	Size of crystallite (nm) (111)	Size of crystallite (nm) (002)	Size of crystallite (nm) (022)
55 °C	42	31	32
65 °C	31	42	33
75 °C	31	42	33
85 °C	42	42	33

The lattice constant for the cubic rock salt structure is calculated by using the Equation 9.

$$a = d\sqrt{(h^2 + k^2 + l^2)} \tag{9}$$

where h, k, and l are Miller indices, and d is the distance between planes [17].

Furthermore, mean stress and micro strain for all planes were calculated using Equation 10 and Equation 11, respectively, and are presented in Table 3.

$$S = \varepsilon Y/(2\sigma) \tag{10}$$

$$\varepsilon = (a_0 - a)/a_0 \tag{11}$$

where a_0 is the lattice parameter of the bulk sample (a_0 = 5.936 Å) a is the corrected value of the lattice parameter of a thin film sample, σ is the Poisson ratio of bulk crystal and Y is Young's modulus. The value of Y for PbS is 70,2 GPa and σ value is taken as 0,28. The corrected values were calculated using Nelson-Riley graphs and the results are exhibited in Figure 2.

The calculated lattice parameters of the produced films are plotted versus F (θ) and given in Equation (12).

$$F(\theta) = (cos^2\theta/2) * (\frac{1}{sin^2\theta} + \frac{1}{\theta}) \tag{12}$$

$$(cos^2\theta/2) * (\frac{1}{sin^2\theta} + \frac{1}{\theta}) = 0 \tag{13}$$

Equation 13, which is the linear line cutoff point, provides the presence of the corrected lattice constant and is presented in Table 3 [22]. The deviation of the calculated lattice parameter (a) from the strain face bulk sample (a_0 = 5.936 nm) indicates that the obtained films were under strain [17].

The dislocation density of the produced films can be derived from the crystallite size as given in Equation 14 and presented in Table 3 [7].

$$\delta = \frac{1}{(cs)^2} \tag{14}$$

3.2. Surfaces of PbS thin films

Typical 30000 times magnified SEM photos are presented in Fig. 3. Fig. 3a is a photo of the film obtained at 55±1 °C. When this image is examined, a crack can be seen on the surface. This is undesirable because the presence of the crack may cause short circuit for p-n junction.

Table 3. The dislocation densities confirmed weave parameters, micro strain values, and average stress values of the PbS thin films produced at 55 °C, 65 °C, 75 °C and 85 °C temperature

EXPERIMENT	2θ	LATICE PARAMETER a (VERIFIED) (Å)	MICRO STRAIN $*10^{-4}$	DISLOCATION DENSITY $(lines/m^2)*10^{14}$	AVERAGE STRESS $(10^7 N/m^2)$
55 ⁰C	25,935	5,94038	24,5	5,84	30,8
	30,021	5,94538	29,2	10,2	36,6
	43,060	5,94261	9,56	9,47	12,0
	50,992	5,93954	6,92	8,92	8,68
	53,455	5,94055	3,46	8,73	4,35
	62,535	5,94336	8,96	71,9	11,2
	68,878	5,94035	10,3	4,19	13,0
65 ⁰C	25,988	5,938596	4,37	10,4	**5,48**
	30,068	5,94422	13,85	5,74	**17,4**
	43,113	5,934804	-20,15	9,47	**-2,53**
	51,012	5,93709	18,36	8,91	**2,30**
	53,481	5,935288	-12,0	13,6	**-1,50**
	62,568	5,93852	42,45	7,99	**5,32**
	68,95	5,93669	11,62	29,8	**1,46**
75 ⁰C	25,962	5,944485	14,29	10,39	**17,9**
	30,030	5,95146	26,04	5,740	**32,6**
	43,072	5,940065	6,847	9,470	**8,58**
	51,008	5,93835	3,959	8,916	**4,96**
	53,417	5,941939	10,00	13,64	**12,5**
	62,566	5,93864	4,447	7,99	**5,58**
	68,827	5,945974	16,80	29,80	**21,1**
85 ⁰C	25,994	5,945888	16,6	5,84	**20,9**
	30,075	5,94744	19,2	5,74	**24,2**
	43,067	5,939556	5,99	9,47	**7,51**
	50,973	5,939312	5,58	8,92	**6,99**
	53,432	5,938683	4,51	13,6	**5,67**
	62,510	5,94348	12,6	4,97	15,8
	68,900	5,93886	4,84	1,15	6,06

The crack disappeared when the temperature was increased, as can be seen from Figure 3. Besides, the surface of the films obtained at 65±1 °C and 75±1 °C are composed of cubic grain structure. But, there are clusters on these surfaces. When the temperature was increased to 85±1 °C, the clusters on the surface disappeared. According to these results, compact and crack free films can be produced at 85±1 °C. For the PbS films to be deposited on glass, the nucleation rate is well-considered to be larger than the growth rate due to the great number of nucleation centers that endured on the surface of the substrate. These results explain why PbS films deposited on glass are compact and with small size of particles. [17].

In this study, parallel to the previous studies [5, 7, 12, 15, 17, 23, and 24], it is shown that no cracks were observed on the films obtained at the temperatures of 55 °C and above. This is likely to be due to the increase in the rate of crystal formation as a consequence of the rise in the deposition temperature.

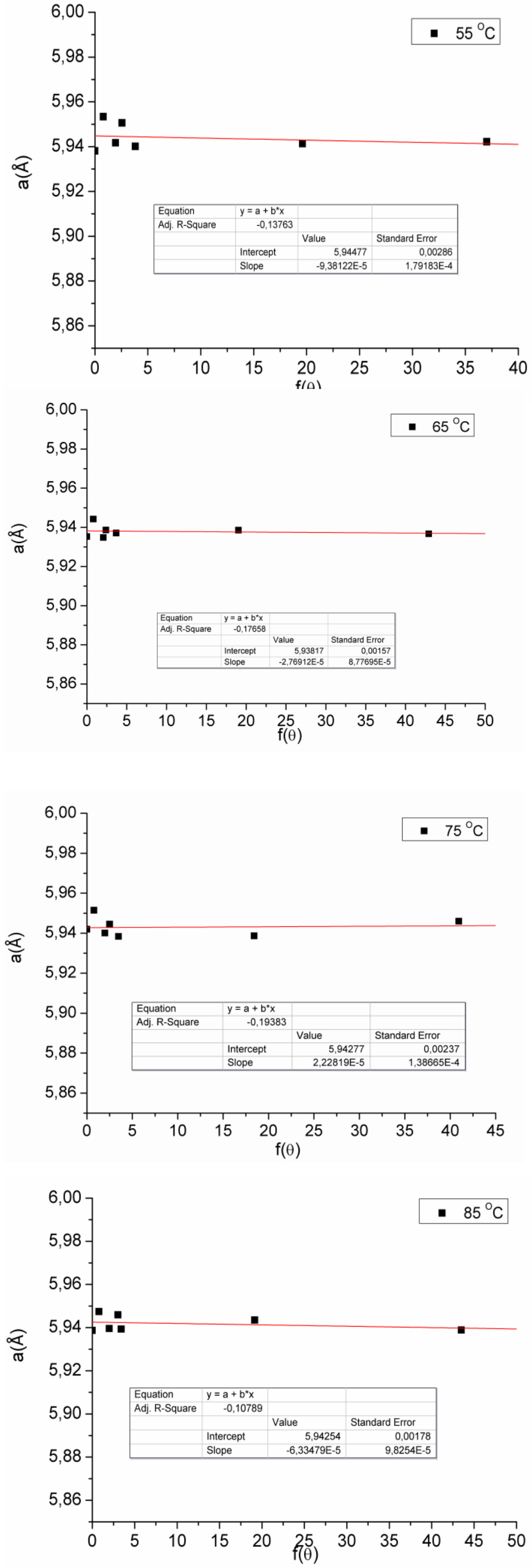

Figure 2. Nelson–Riley plots of PbS thin films obtained at a) 55°C, b) 65°C, c) 75°C, d) 85°C temperature.

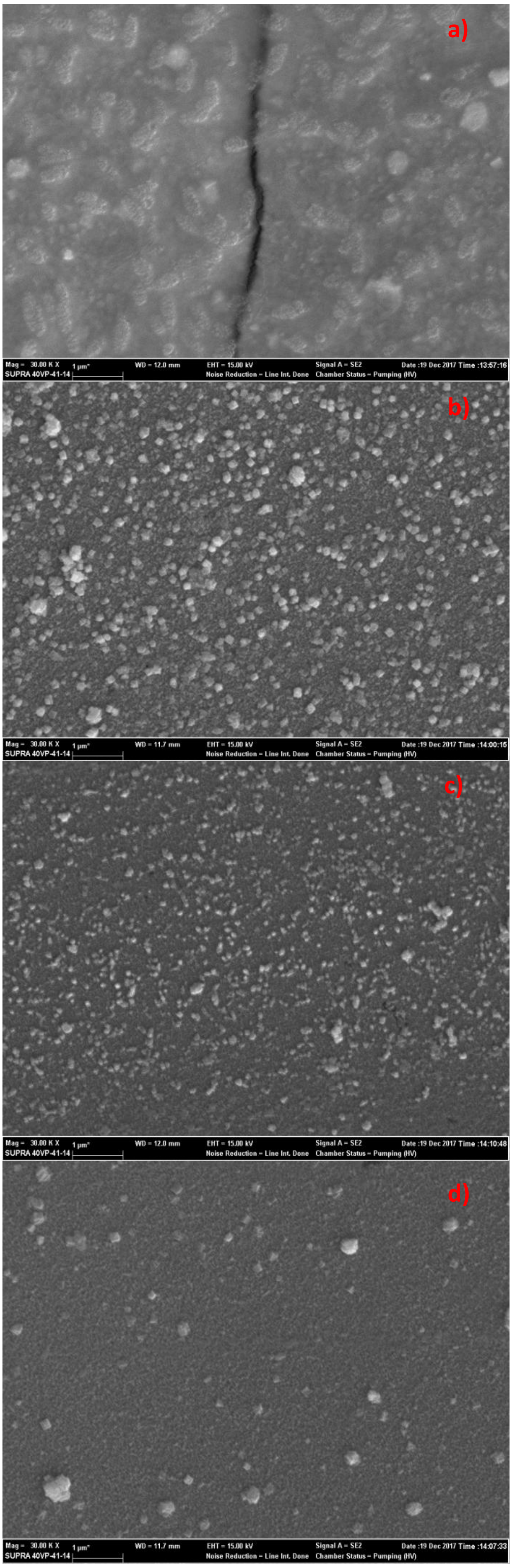

Figure 3. SEM images of the PbS films at a)55 °C b)65 °C c)75 °C d)85 °C temperature, magnified 30000 times.

4. Conclusion

This study presented the obtainment of PbS thin films by chemical bath deposition. Four different temperatures were applied in the experiments. XRD data showed that all films formed in cubic structure and preferred orientation depended on temperature. Besides, the peak intensity of the film obtained at 85±1 °C was nearly 3 times higher than that of the other films. This result shows that good crystallization was obtained at 85±1 °C. The surface morphologies were investigated by using a SEM and the SEM images revealed that when temperature was reduced below 65±1 °C, a crack appeared on the film surface. The study showed that compact and smooth surface can be obtained at 85±1 °C.

Dislocation densities, micro strain, and mean stress values of all films were calculated. The mean stress values of the film obtained at 55 °C temperature was greater than that of the other films. However, among all films, the film obtained at 65 °C temperature had negative average stress and micro strain values. It is clearly understood that negative strain indicates compressive strain present in the synthesized PbS thin films [25].

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