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Fabrication of Pb_xS_{1-x} Detector and Studying the Effect of x Content on its some Physical Properties

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Abstract

A polycrystalline Pb_xS_{1-x} alloys with various Pb content (0.54 and 0.55) has been prepared successfully. The structure and composition of alloys are determined by X-ray diffraction (XRD), atomic absorption spectroscopy (AAS) and X-ray fluorescence (XRF) respectively. The X-ray diffraction results shows that the structure is polycrystalline with cubic structure, and there are strong peaks at the direction (200) and (111), the grain size varies between 20 and 82 nm. From AAS and XRF result, the concentrations of Pb content for these alloys were determined. The results show high accuracy and very close to the theoretical values. A photoconductive detector as a bulk has been fabricated by taking pieces of prepared alloys and polished chemically, etching, and annealing at special condition. photoconductive properties of these detectors were measured.

Key words
Detectors, PbS, photoconductivity

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تصنيع كاشف كبريتات الرصاص ودراسة تأثير محتوى الرصاص على بعض خصائصه الفيزياوية

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الخلاصة

حضرت بنجاح سبائك كبريتات الرصاص المتعددة البلورات عند محتوى الرصاص(٠,٥٥، ٠,٥٧). حدد تركيب ومحتوى السبائك بواسطة حيود الاشعة السينية ومطياف الامتصاص الذري وفلورة الاشعة السينية. بينت نتائج حيود الاشعة السينية بأن التركيب من النوع المكعب المتعدد البلورات، وهناك قمم واضحة بالاتجاهين (١١١) و (٢٠٠). وكانت قيمة الحجم الحبيبي تتراوح بين ٢٠-٨٢ نانو متر. وتم تحديد تركيز محتوى الرصاص من خلال نتائج مطياف الامتصاص الذري وفلورة الاشعة السينية وكانت النتائج تقريبا مقاربة للقيم النظرية. صنع كاشف التوصيل الضوئي من خلال تقطيع السبائك المحضرة وعمل صقل واضهار كيميائي لها، بعد ذلك تم تلدينها تحت ضروف خاصة. تم قياس خصائص التوصيل الضوئي لتلك الكواشف.

1- Introduction

Detector is a device, which senses and converts incident radiation in to electrical signals. Infrared detectors are accomplished with devices, which are sensitive to temperature (thermal detectors), or devices, which are sensitive to photons (photo detectors). The temperature change by the incident photons will usually cause the detector to produce a proportional voltage (thermovoltaic) or a proportional change in conductivity (photoconductivity). In general, photodetectors exhibit significant dependence on wavelength, while thermal detectors are nearly independent of wavelength [1]. A wide range of binary compound and ternary alloy have been used to fabricate IR detectors. Lead sulphide (PbS) is an important semiconductor with a narrow band gap that can be used as photodetector for IR radiation[2,3]. the films are obtained by CBD or vacuum evaporation, these films have photosensitivity as prepared. The photosensitivity that depends on various factors, such as: obtaining method, thickness, composition, structure and the morphology of the film can be improved by introducing oxidants in the reaction environment, reducing agents, or by the application of thermal treatments in oxygen-containing environment[4-9].

During treatments p type PbS films with crystal defects are formed. The resultant junctions are responsible for the photosensitivity.The aim of this research is to fabricate Pb_xS_{1-x} alloys with various Pb content as a detectors

2-Experimental

2-1 Alloys preparation:

The Pb_xS_{1-x} alloys were prepared from pure lead and sulfur powders whose purity is about (99.9999%) with two x content x (0.54,0.55), the weighted lead and sulfur were loaded into a clean and baked quartz tube.

The evacuation of the tube was(10^{-6}) torr, the ampoule was sealed off, the loaded ampoule was first placed inside electric furnace of type (Heraeui). The temperature was slowly raised to 473K, and the ampoule kept at this temperature for 4h(qusi equilibrium phase). Then temperature was then raised by step 200K and kept at 673K for 4h. This procedure was continued to 1073K, and the ampoule was kept at that temperature for 12h. These sequence of 200K increase every 4h was continuous until the furnace temperature was raised to the melting point of alloy 1400K which is above the melting point of lead (600K), Sulfur (388K) and lead sulfide alloy (1391K) .The ampoule was shaken thoroughly, after which it was kept at that temperature for 24h. The ampoule was finally quenched rapidly in cold water to reduce segregation and to obtain more homogenous alloys. The prepared resultant of Pb_xS_{1-x} was taken out to prepare a photoconductive as a bulk after cutting, polishing and .etching it.

2-2 Fabrication of Pb_xS_{1-x} bulk photoconductive detector:

Pieces of $Pb_{0.54}S_{0.46}$ and $Pb_{0.55}S_{0.45}$ alloys have been cut, and chemically polished by mixture of (30HCl, 10 HNO_3, 1 acetic acid). This solution in oven at 323K for a few minute, after that the samples are immersed in this mixture for 3 minute, then immersed in 10% acetic acid. Also chemical etching has been made for this samples by mixing [1HCl, 3solution of thiourea (100)g/l] and putting in oven at 333K,then the samples immersed in resultant solution for (1-10) minute, The color of samples are varying from gray to blue[18]. Annealing was made by putting the sample with a little sulfur into a clean and baked quartz ampoule. After the evacuation (10^{-6}) torr, the ampoule was seal off and placed inside electric furnace of type (Heraeui). Many attempts have been done for this annealing and the only succeed attempt when the temperature was raised to 283K/hr until 873K, and the ampoule kept at these temperatures for 48h and after that the temperature slow down in the same rate then the sample was ready for testing it.

2-3Composition analysis:

The composition of Pb_xS_{1-x} alloys where determined by Shimadzu 601 instrument atomic absorption spectroscopy (AAS).This analysis was used for quantitative measurement of atomic percentage of the material (Pb and S) in the alloys, and by using some acids as solvent for this material. Also, XRF has been used to determine the composition analysis. The structure of the Pb_xS_{1-x} alloys as bulks has been examined by X-ray diffraction with CuKα wavelength (λ) (1.5405)A. The scanning angle has varied in the range

(20^o-60^o) with speed 2cm/min, and current 20mA and voltage 40 kV.

2-4 I-V Measurement

The dark and photo current of Pb_xS_{1-x} photoconductive bulk at different value of x content was measured as a function of applied voltage. The photocurrent have been done under illumination intensity (75,100,120)mW/cm^2 by using Halogen lamp type philips 120W as shown in Fig.(1).

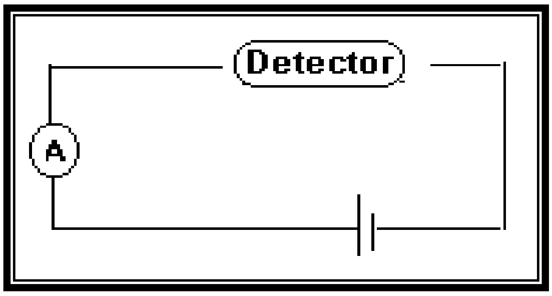


Fig.(1) Circuit diagram for measuring dark and photocurrent for photoconductive

2-5 Detector Characteristic Measurement:

The photocurrent measurement have been done using system consists of IR source (SiC)in the region (1-20μm) monochrometer in the range 0.4-2.5 μm type (infrared spectrometer model 746 from optronic Laboratories, Keithly Digital Electrometer for current measurments. The sample of Pb_xS_{1-x} bulk with different value of x content and T_a were connected to the electrical circuit as shown in Fig.(2). By increasing the incident light wavelength, .The incident power on the detector was obtained by using a calibrated standard detector.

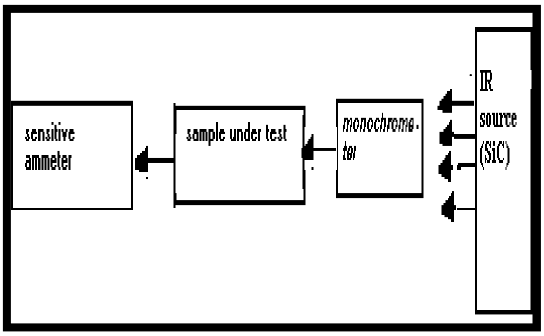


Fig.(2)Photocurrent measurement

The spectral responsivity, quantum efficiency,NEP detectivity (D*) were calculated by using the following relations respectively.

$R_\lambda = I_{ph}/P_{in}$ (Amp/Watt)---------------(1)

Where R_λ , I_{ph} and P_{in} represents responsivity, photo current and incident power respectively.

$\eta_{(\lambda)} = R_\lambda hc/\lambda q = 1.24 R_\lambda/\lambda$----------------(2)

Where $\eta_{(\lambda)}$, h, c, λ, q represents quantum efficiency, blank constant, speed of light, wavelength, charge of electron respectively.

$NEP = I_n / R_\lambda$----------------(3)

Where NEP and I_n represents noise equivalent power, noise current respectively.

$D = 1/NEP = R_\lambda / I_n$ -------------------(4)

$D^* = R_\lambda (a^{`} \Delta F)^{1/2} / I_n$ -------------(5)

Where D, D^*,$a^{`}$ and ΔF represents Detectivity, normalized detectivity detectors area and band width

3-Results and Discussion

Atomic Absorption Spectroscopy (AAS) has been used to examine the concentration of the elements (Pb and S) in the alloys as bulks, by depending on the atomic absorption of the standard elements of these components.

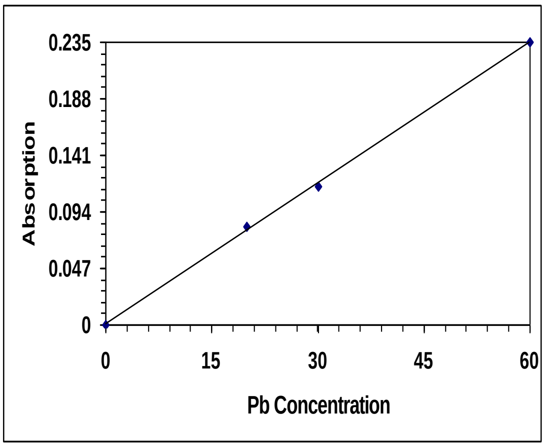


Fig.(3) Working curve for stander Pb concentration.

Also We determined the concentration of element Pb for the prepared alloys as bulks by XRF technique. We observed that the two values are very close which indicate that this method is adequate method.

Table (1) The composition of alloys (bulks) determined by AAS and XRF

Alloy	Theoretical concentration of element%		Experimental concentration of element% byAAS		Experimental oncentration of element% byXRF		Total%
	Pb	S	Pb	S	Pb	S	
$Pb_{0.54}S_{0.46}$	0.54	0.46	0.55	0.45	0.547	0.453	100
$Pb_{0.55}S_{0.45}$	0.55	0.45	0.555	0.445	-	-	100

Fig.(4) shows the X-ray diffraction for Pb_xS_{1-x} alloys as bulks with x content (0.54, 0.55), it appears that these alloys as bulks and films are polycrystalline of FCC cubic structure according to the comprised between our d-values with the ASTM cards data for FCC of Pb_xS_{1-x} , one can observe that the following reflection surfaces [(111), (200), (220), (311), (222)]have been appeared which is similar to the ASTM cards data. Our results are the same as [19-23].

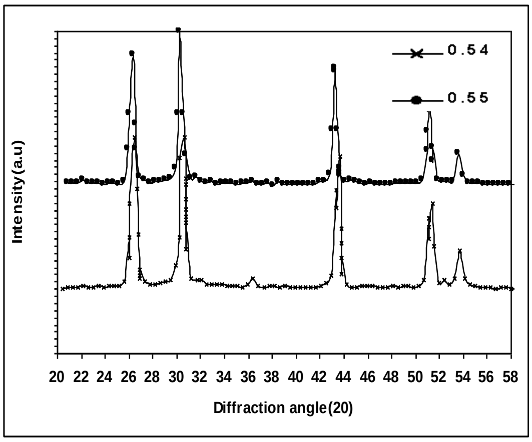


Fig.(4) show the X-ray diffraction for Pb_xS_1alloys as bulks with x content (0.54, 0.55)

The photoconductivity of the Pb_xS_{1-x} bulks has been determined for two x content and power intensities. The current voltage characteristics were measured under darkness and illumination at various power intensity (75, 100, 120)mW/cm^2. The dark and photocurrent against the applied voltage are shown in Fig.(5). We can observe that the current variation under illumination is relatively small, which mean poor photoconductive at room temperatures. The incident radiation decreases the resistivity of polycrystalline lead chalcogenide photoconductive by creating electronic transition in the band gap. The increase in photoconductivity is partially due to increase in the number of the free carriers (electrons and holes), and partially to decrease in the potential barrier at the grain boundaries, which increases the mobility of the carrier at the grain boundaries, and increases the photoconductivity of the carriers [10].

The relation between dark current and the applied voltage for Pb_xS_{1-x} bulk at different Pb content (0.54, 0.55) are shown in Fig.(5) at voltage bias (0-40)V. This figure shows that the dark current increases with increasing voltage bias, at low voltage (0-5)V the increasing of dark current was small because the probability of capturing the free charge carrier by recombination and capture centers which resulting by adding Pb content are increased, this leading to increase the transit time (t_r)between the electrode and reduce the mobility and drift velocity. Whereas at high voltage (V>5V), when the applied electric field on the detector is increased, the drift velocity is increased. For this reason, the defects are become un-affective which leading to reduce the transit time and the behavior of dark current was become linear as a function of high voltage bias, and this is agreement with [24].

The dark currents are increases with increasing x content, and decrease for high value of Pb content. This means that when adding the Pb content, the sensitizing center and free charge carrier will increased, then the dark current increased Whereas at high value of Pb content, all the effective and recombination center will filled and all the free charge carrier will transfer and extinguish, therefore the dark current decreased. From Fig.(5), the dark current were decreased with increasing Pb content from 3.52 to 1.922 μA at 5V for 0.54 and 0.55 respectively due to the same reason which mention previously. The relation between photocurrent and the applied voltage for Pb_xS_{1-x} bulk at different power intensity and at Pb (0.54, 0.55) are shown Fig.(6) at voltage bias (0-40)V. The photocurrent increases with increasing the power intensity as above. For 0.5 content and R.T and 40V,the photocurrent increases about two times when the light intensity increases from 75 to 120mW/cm^2 and this is in agreement with [25,26], and attributed to increasing the arrival charge carrier to the detector electrodes. At low intensity the free carrier will be captured at the capture centers which found at the energy gap and when the power intensity increased, these centers will be filled and has no effect on moving the free charge carriers, this leads to increase the conductivity and decrease the resistivity.

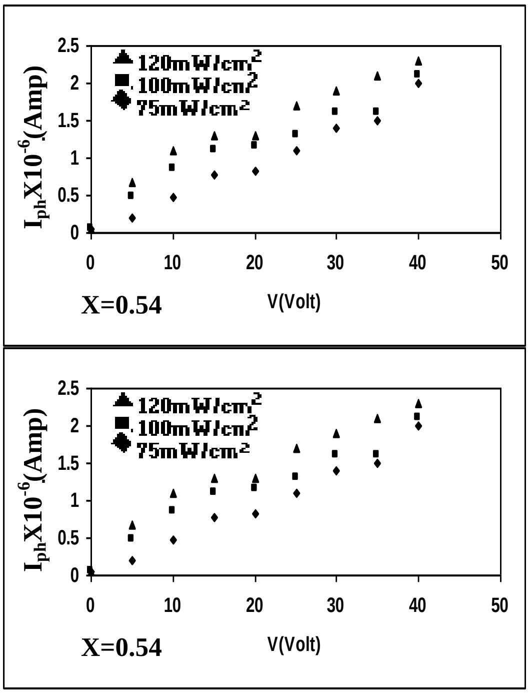


Fig.(5).The relation between dark current and the applied voltage for Pb_xS_{1-x} bulk at x for Pb 0.54 and 0.55.

The photocurrent increases with increasing Pb content, and this attributed to improvement the crystal structure by heating. Adding of Pb content will create a sensitizing centers inside energy gap which responsible on generation and recombination processes, then the photocurrent increased and this is in agreement with [16,27]. The photocurrent for bulks increases with increasing Pb content from 2.3 μA to 3 μA due to the same reason which mention previously for films.

From the measurements of the spectral responsivity(R_λ) for Pb_xS_{1-x} bulks at different x content (0.54,0.55), the spectral responsivity has been studied as shown in Fig.(7) which include the spectral responsivity as a function of wavelength for bulks . It is found that the peaks were divided into three regions. The first region represents the small wavelength region (1-2)μm in which the R_λ have little increases with increasing wavelength and this attributed to the high absorption coefficient at wavelength (1-2)μm and this is in agreement with the result of optical studies for [12,28]. The second region represents the wavelength from 2 to 2.8μm. The maximum value of R_λ was occurs at this region relative to the incident wavelength, because the absorption of light leading to increase R_λ with increasing the wavelength incident radiation. This meaning a high efficiency to separate the electron –hole pairs which generated from the external applied field, and also to decrease the recombination processes comparing with the first region [29].The third region represents long wavelength region 2.8 to 3.5μm in which the R_λ reduced sharply due to approach from the wavelength cut-off (λ_c). So that the absorption decreases and the transmission increases This means that the wavelength absorbed within the materials and this called the recombination within the materials(bulk recombination), so it has small probability for incorporate the carrier, which effect on the value of spectral response consequently the quantum efficiency decreases according to equation(2). Figure (7) illustrates the variation of R_λ with Pb content for Pb_xS_{1-x} bulks. It is obvious that the increasing in Pb content caused increase in R_λ , which due to increase α, so that the quantum efficiency increased and consequently the R_λ increases. The responsivity and rel.responsivity of the bulk has been shown in the Fig.(7) Table (2).The value of R_λ for bulk is varies between (0.283-0.41)A/W, and this value are in agreement with [3,16,27,30].

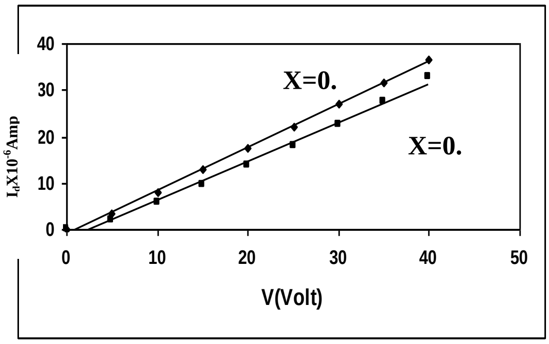


Fig.(6).The photocurrent as a function of applied voltage for Pb_xS_{1-x} bulk for different power intensity and at 0.54,0.55 content.

Table (2)The parameters of Pb_xS_{1-x} photoconductor detectors

Alloy	λ_{peak} (μm)	I_{ph} (μA)	R_λ (A/W)	Quantum efficiency %	NEP(W) $x10^{-12}$	$D^* x10^{12}$ cm.$Hz^{1/2}$/W
0.54	2.6	0.85	0.283	13.5	3.17	0.314
0.55	2.6	1.23	0.410	19.5	1.88	0.520

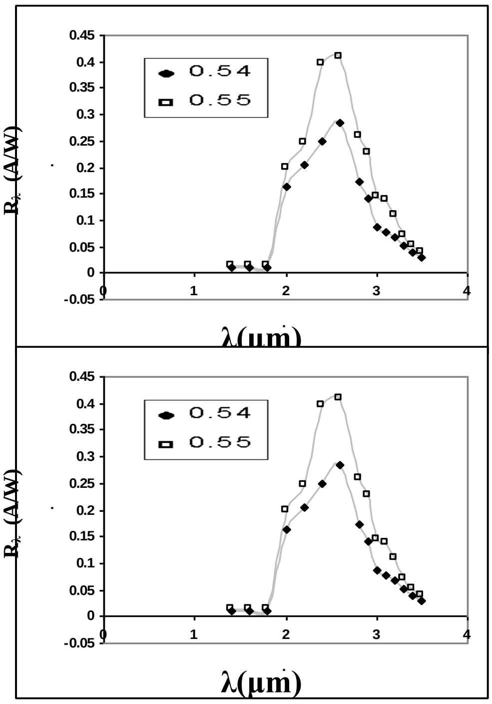


Fig.(7) The variation of R_λ and Rel.R_λ as a function of wavelength Pb_xS_{1-x} bulks

The quantum efficiency (η) values are related to spectral response results and it was calculated by using equations (2). Fig.(8) shows the variation of η versus wavelength of Pb_xS_{1-x} bulks at different x content (0.54,0.55). It can be seen from the Table (2) that the η increases nearly from 13.5% to 19.5% i.e. about 1.5 times, and this is attributed to the increase in α with increasing Pb content. Also increasing states in gap which shifts the peaks to higher wavelength due to decrease E_g ,therefore the η increasing according to the eq(2) The values of η are nearly agreement with [16].

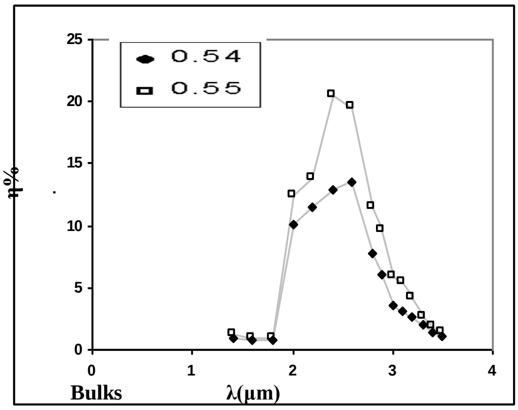


Fig.(8)The variation of η% as a function of wavelength at different Pb content for Pb_xS_{1-x} bulks.

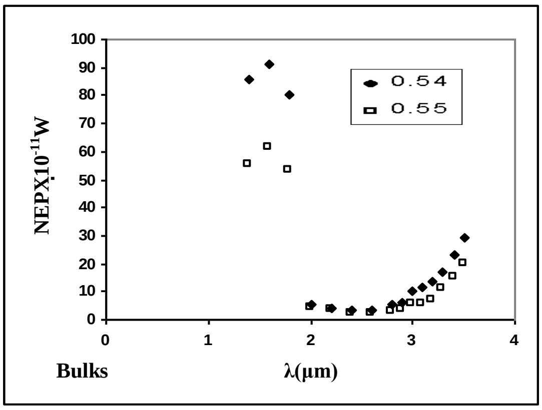


Fig.(9)The variation of NEP as a function of wavelength at different Pb content for Pb_xS_{1-x} bulks.

By using eq(3), we can calculate the NEP for Pb_xS_{1-x} bulks at x content (0.54,0.55), as shown in Fig.(9). The value of NEP for Pb_xS_{1-x} bulk decreases with increasing Pb content.

By using eq(5), we can calculate the D* for Pb_xS_{1-x} bulks at different x content (0.54,0.55), as shown in Fig.(10).

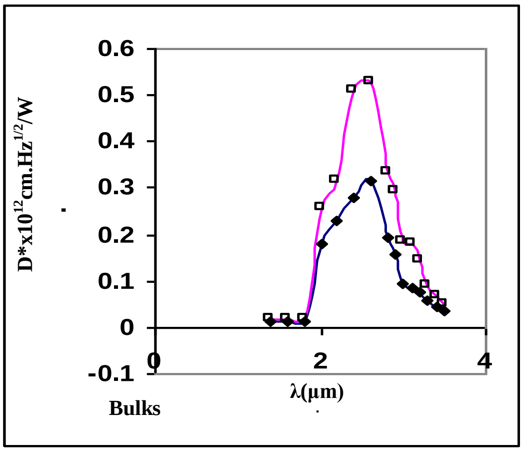
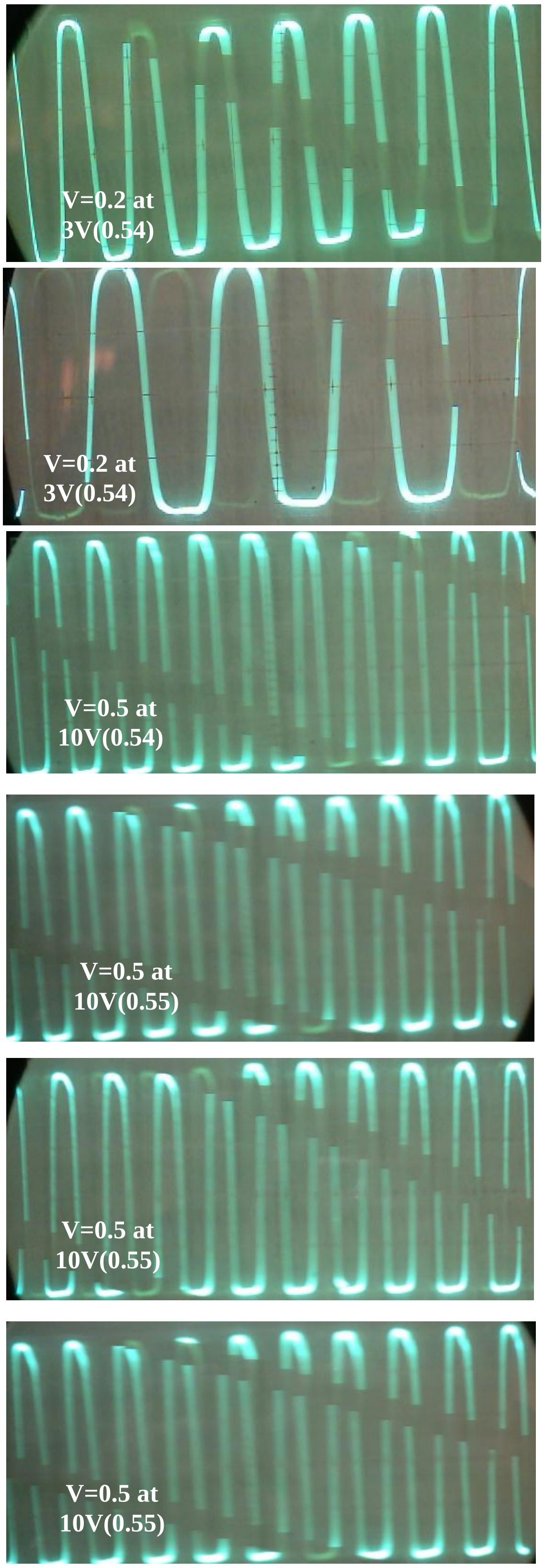


Fig.(10) The variation of D^* as a function of wavelength at different Pb content for Pb_xS_{1-x} bulks.

It is seen from these figures that the higher magnitude of D^* proportional with the higher magnitude of the R_λ because the D^* is a function of the spectral response. Also the D^* increases about two times for bulks with increasing Pb content due to increase R_λ as shown in Fig.(10) . This results are in agreement with the other referense[6]. Heini [15] has prepared PbS films by elecro-deposition technique and found that the value of D^* for PbS films was $1.6X10^6cmHz^{1/2}.W^{-1}$, and Al- Miyali [27] found that D^* for PbS which prepared by chemical deposition was equal to $0.46X10^9cmHz^{1/2}.W^{-1}$. Chopra and Pandya [13], found that the detectivity of a PbS photoconductive device is markedly dependent on the deposition technique, and the detectivity increases at low temperature and its peak shifts to longer wavelength (1-5)μm. And the value of D^*_λ as a high as $10^{12}cmHz^{1/2}W^{-1}$ at 77K and wavelength peak at 2.1μm in 295K and D^*_λ in R.T are $4.5x10^8$ and this is nearly in agreement with our value. Figs.(11)represents the output voltage signal from the Pb_xS_{1-x} PC (bulk)



Figs.(11)represents the output voltage signal from the Pb_xS_{1-x} PC (bulk)

which represents the response value to the IR source(SiC) at different Pb content and T_a, we can see from these figures that the output voltage signal increase with increases Pb content and T_a,

The increasing of output voltage with increasing Pb content is due to improve in crystal structure for these detectors which effect on the carrier life time and mobility lead to increase the response. Also we can see from these figures that the output voltage signal are contain three regions, the first region represents additional generation pairs, the average of total generation was higher than the average of recombination, while in the second region, the materials will be in stable state in which the average of total generation (summation of thermal and photo generation) was equal to the average of recombination , the third region represents the region in which the effect of light was finished and the carrier concentrations are decreases

4- Conclusions

From our results, one can conclude that

A. The Pb_xS_{1-x} alloys for (x = 0.54, 0.55) have successfully prepared, which was used for fabricate photoconductive bulks detectors

B. The XRD tests of alloys at different Pb content showed that the structure is polycrystalline with cubic structure (FCC), and with preferred orientation (200) and (111) orientation.

C. from AAS and XRF studies, the concentration were determined for Pb content and these alloys and the result are of high accuracy and very close to the theoretical values.

D. For the first time a Pb_xS_{1-x} photoconductive detectors as a bulk of alloy from a polycrystalline material was fabricated as a detector.

E The photocurrent for bulks increases with increasing light intensity and Pb content.

F. From the spectral responsivity measurement, It is found that the curve has one peak at 2.6µm

G. It is found that the increase in Pb content caused increase R_λ.

H. The η increases with increasing Pb content from 0.54 to 0.55

I. The value of NEP for Pb_xS_{1-x} bulk decreases with increasing Pb content.

J. The D^* increases about two times for bulks with increasing Pb content.

K.From the output signals, the output voltage signal for PC increases with increasing Pb content.

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