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- الدلالة في كتب الأخطاء الشائعة "العربية الصحيحة لأحمد مختار عمر" أنموذجا).
- اضطرابات النطق لدى عينة من تلاميذ الحلقة الأولى لمرحلة التعليم الأساسي بمدينة مصراته.
- دور الإرشاد النفسي المنبثق عن الشريعة الإسلامية في علاج بعض مشكلات الشباب الليبي المعاصر.
- العناصر التيبوغرافية ودورها في الإخراج الصحفي.
- تقييم بعض مدخلات مؤسسات رياض الأطفال بمدينة مصراتة في ضوء معايير الجودة.
- دراسة الأخطار الجيومورفولجية بمنطقة حوض وادي غاوغاو باستخدام نظم المعلومات الجغرافية والاستشعار عن بعد.
- مفهوم صورة الجسد وعلاقتها بالاستعداد للعصابية لدى طلبة المرحلة الثانوية.
- الصور البيانية في الأمثال النبوية "نماذج مختارة".
- تأثير التلوث الناتج عن صناعة الإسمنت على الأس الهيدروجيني للتربة ومدى تأثيره على نمو النبات "الفول" Vica Faba L.
- المتاشبه اللغوي عند القراء والمفسرين "تأليفا وتطبيقا" .
- رسالة في مباحث البسملة لأحمد بن زين دحلان "ت1304هـ".
- نظرية العبقرية عند كانط.
- ماهية النص الأدبي خطاب إلى متذوقي الأدب.
- كفايات التعليم الإلكتروني ومدى توفرها لدى هيئة التدريس بكلية التربية جامعة المرقب
- استعمال كاف التشبيه حرفا واسما.
- المؤرخ نقولا زيادة وليبيا "دراسة في المعاصرة التاريخية حياة وتأليفا".
- فاعلية المرأة الطوارقية في الرواية الليبية " إبراهيم الكوني أنموذجا".

- ضوابط بيع التقسيط في الشريعة الإسلامية
- أثر دراسة الفقه المقارن في تضييق شقة الخلاف بين المذاهب الفقهية .
- Morphology and composition of $CuInSe_2$ that film deposited by Stacked Elemental Layers for solar cells application
- A novel Piggyback Scheme to Improve the Performance Of MAC Layer Based on IEEE802.11n
- Problems of English prepositions in EFL learners' translation
- L’argent peut-il effacer les valeurs morales ? Le Père Goriot de .Balzac est un type

الافتتاحية

من سمات المجتمعات المتحضرة سعة ثقافة أبنائها وكثرة قرائها، والكتاب لديهم هو أفضل صديق، يرافقهم أينما كانوا وحيثما ما حلوا، فكما أن الطعام غذاء أبدانهم فإن القراءة غذاء أرواحهم، ولا عجب أن للقراءة أهمية عظيمة في الإسلام فهو يدعو إلى التدبر والتفكير والقراءة والتعلم، يكفي أن أول آية نزلت على حبيبنا محمد صلى الله عليه وسلم هي قوله تعالى ﴿ اقْرَأْ بِاسْمِ رَبِّكَ﴾ فكان الأمر بالقراءة فاتحة عقد الاتصال بين السماء والأرض، وللقلم في تثبيت ركائز العلم مكان لذلك خصه المولى عز وجل بالذكر مصاحبا للأمر بالقراءة فـقال ﴿اقْرَأْ وَرَبُّكَ الأَكْرَمُ الَّذِي عَلَّمَ بِالْقَلَمِ* عَلَّمَ الإِنسَانَ مَا لَمْ يَعْلَمْ ﴾ .

ولكن العجب في أمة القرآن، أمة اقرأ أن تكون أمة عازفة عن الكتاب والقلم، تتصدر مجتمعاتها آخر الصفوف، وتبقى القراءة في ذيل اهتمامات أبنائها، فلقد تدنى المستوى الثقافي والمعرفي لديهم إلى أدنى درجة، فالأمم لا تقاس بكثرة المال والأبناء وإنما تقاس بمدى ثقافة أبنائها، ومستواهم المعرفي، وأولى سمات ذلك حبهم للقراءة، والملاحظ والدارس لحال أمتنا في هذا العصر يرى وبكل بوضوح ودون أي مجهود قلة نسبة من يعشقون الكتاب، ومن يقتنونه، وارتفاع نسبة العزوف عن قراءته بل يتجاهلونه.

لقد تسرب إلى قلوب أبنائنا حب المال، ويا ليته من أوجهه السليمة الصحيحة فالثقافة وحب القراءة لا يتعارضان مع النشاط الاقتصادي، بل هما داعمان له ورافدان من روافده، فما علت الأمم الغربية في عصرنا الحاضر وازدهر نموها إلا بالعلم والثقافة، ونحن أمة القرآن أمة الثقافة تأخرنا حتى وصفنا بالتخلف مع أن أسلافنا أخذوا بناصية العلم فسادوا الدنيا بدينهم ولغتهم وثقافهم والشواهد في أواسط آسيا وأدغال أفريقيا باقية إلى الآن خير دليل، فهل لهذه الأمة من صحوة ثقافية ونهضة حضارية تبني بها حاضرها، وتعيد بها مجدها التليد.

هيئة التحرير

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Abstract

The aim of this study is to investigate the morphology and composition of the *CuInSe*$_2$ thin film deposited by Stacked Elemental Layer technique SEL. The films were annealed in different ways to increase the chalcopyrite phase which may improve its' electrical and optical properties. The chalcopyrite phase dominates when samples heated or step heated at 400^0 C for 40 – 50 minutes in argon, and for 15 minutes in vacuum. Deposited films were characterized using Atomic percentage composition and x-ray diffraction techniques. This paper describes the production of well-formed polycrystalline *CuInSe*$_2$ and well defined XRD peaks. Atomic percentage composition has been studied in order to investigate the composition of the films. The thickness of layers were monitored by a quartz crystal monitor.

Keywords: CuInSe$_2$ *thin film, Photovoltaic (PV) energy, the chalcopyrite CuInSe*$_2$

1. Introduction

Photovoltaic (PV) energy promises to become a leading source of electrical power, rivaling the largest existing source of electricity. The first developed photovoltaic systems were 100 times as expensive as conventional electricity source. Today they are only few times expensive. The best examples of success came in the area of "thin films" solar cells. Thin films were in their first stages at 1970s. They now have a substantial of PV market and are growing rapidly. More important, thin films are making rapid advances toward cost competitiveness with conventional electricity sources and are believed to have the potential to achieve cost parity.

The ternary compound (ABX) semiconductors are of special interest because they have a wide range of optical band gaps and carriers' mobility. The deviations of their properties from the binary analogs make them very useful especially in solar cells. They are able to form various solid solutions and to accommodate different dopes. These properties led them to emerge as technologically significant device materials, including their application in photovoltaic cells, light emitting diodes, and various nonlinear optical devices[i, ii].

In the last decades, intensive studies have been carried out to examine the photovoltaic properties of many materials and compounds. These includes, crystalline and amorphous Si Cu_2S, InP, GaAs, CdTe, *CuInSe*$_2$, $CuInGaSe_2$ and others. *CuInSe*$_2$ is a ternary direct gap semiconductor with bandgap of 1.04 eV. *CuInSe*2 based solar cells are very promising material because it is an excellent stable material [iii,iv].

Among these compound, the chalcopyrite *CuInSe*$_2$, either in the form of single crystal or as polycrystalline thin films, has emerged as a promising candidate for solar energy conversion, because it possesses the following useful properties:

- It has a desirable direct band gap of about 1 eV at room temperature, which is in the energy range for optimum solar conversion [v, vi].
- It has very high absorption coefficient, about $10^4 – 10^5$ cm^{-1} near the band gap for polycrystalline thin films, which is one of the highest value reported in the literature for any semiconductor [7, 8].
- It can be made either n- or p- type, which permits the formation of homojunctions, like diffusing Cd or *In* into p-type *CuInSe*$_2$ [vii, viii] or by diffusing *Cu* into n-type *CuInSe*$_2$ [9] and several types of heterojunctions [7].
- p-*CuInSe*2 material with low resistivity $\rho \sim 0.5\Omega$ cm and high mobility of minority carriers $\mu > 500$ cm^{2}V^{-1}S^{-1} , as required for efficient heterojunctions, can be prepared by introducing a slight excess of Selenium in to material [7]. However, difficulties arise in the material's reproducibility with desired electrical parameters, because of the intrinsic doping effects [ix]. Very high short-circuit current densities ~ 39 mA/cm^2 [x].

- Excellent stability.

In spite of the possibility of producing $CuInSe_2$ homojunction devices, the most efficient solar cells use heterojunction with n-type CdS films as a wide band gap window which admit light to the underlying heterojunction with p –type $CuInSe_2$ [xi]. Such devices using 2-4 µm pure $CuInSe_2$ thin film have attained efficiencies as high as 19% [xii] For this reason the heterojunction n-CdS/p-$CuInSe_2$ is currently one of the most promising technologies. The reasons for this are threefold.

- First, it is likely that only polycrystalline or amorphous thin films solar cells could be fabricated in sufficiently low costs to be competitive with conventional bulk power sources.
- Second, for the currently tested polycrystalline or amorphous materials only $Cu(In,Ga)Se_2$ based heterojunction solar cells have been demonstrated to achieve grater than 19% [xiii, xiv,xv] efficiency with excellent stability at relatively low costs. Not much higher efficiencies are possible since the band gap of $CuInSe_2$ is significantly less than 1.45 eV, the approximately optimum band gap required, to match to the solar spectrum [xvi]. In order to increase the band gap to the optimum value, the pure $CuInSe_2$ is alloyed by S or Ga to form $CuIn(S,Se)_2$ and $Cu(In,Ga)Se_2$ [xvii].
- Third, the electro-optical, structural, and morphological properties of $CuInSe_2$, and the corresponding devices performance, are influenced greatly by films composition, intrinsic defect, and growth parameters [8].

The most important factor which characterizes the $CuInSe_2$ thin films is the *Cu*/*In* ratio, and of practical concern is the ease to obtain controllable and reproducible *Cu*/*In* ratio [xviii]. This is technique Simple, low cost and secure techniques to produce the $CuInSe_2$ thin films are needed. These techniques will make $CuInSe_2$ based devices reliable and practical.

2. Experimental Aspect

2.1 Stacked Elemental Layer technique

Stacked Elemental Layer (SEL) method can be considered as a spin off the selenization concepts. In this process only one elemental source is used at a time in depositing elemental layers. The thickness of each layer can be controlled easy. This process differs from selenization method in that it does not *covert* a *Cu-In* metallic films, called "metallic precursor" to a chalcopyrite structure by reaction with *Se*. The precursors itself contains elemental Se layers. *Se* containing precursor has shown some promising results as an alternative to the pure metal precursor [xix].

Some advantages are expected from this method. The technological simplicities in producing *CuInSe*$_2$ thin films and easy composition controlling, stand as a key for developing a wide scale production. SEL method seems to make these conditions reliable.

No principle need to the selenization process in pure *Se* vapour or with H_2Se which are technologically more complicated and very toxic. Additional selenization sometimes is used to improve films quality.

2.2 Apparatus setup

Stacked Elemental Layer structure as shown in Figure I, consists of alternate elemental layers thermally deposited on a microscopic slide glass at room temperature in standard evacuated chamber at $< 10^{-4}$ torr.

The evacuation chamber, which has been used in this work, is a two-step system. First evacuation step accomplished by using a rotation pump. The second step uses a diffusion pump to obtain the final evacuation, $< 10^{-4}$ torr.

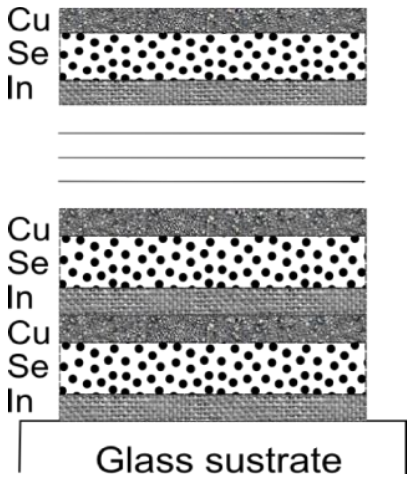


Fig.1: Stacked Elemental Layer Structure

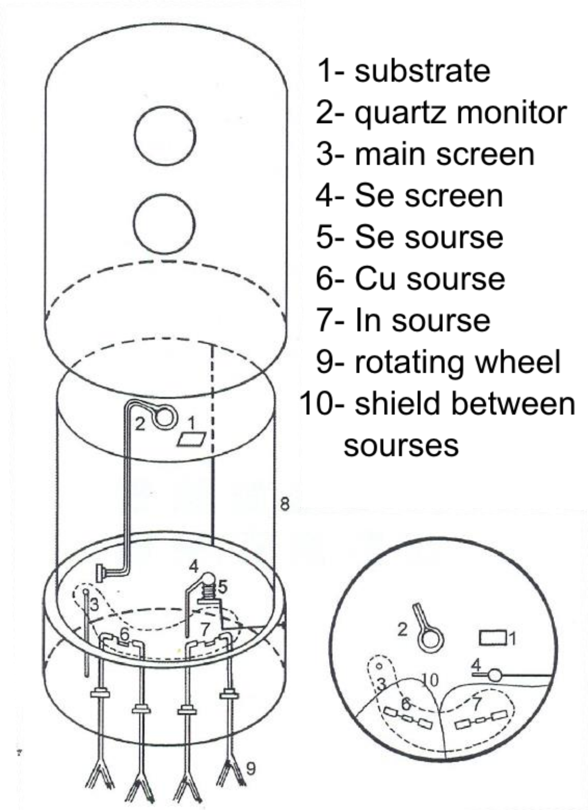


Fig. 2: Scheme of the evacuation chamber of SEL technique.

A microscopic slide glass with dimension 25 × 75 mm, was used as a substrate. It has been mounted inside a holder fixed to the upper side of a rotating wheel, installed inside the evacuation chamber. The substrate was mechanically placed above the elemental sources at about 17 cm, from each of them successively by rotating the wheel. Figure 2 Shows the chamber of SEL technique.

A sheet of copper with the same dimensions as those of the glass substrate was placed on the substrate in order to prevent the temperature increasing of the substrate which may cause unintentional reaction between elemental components during the deposition of the elemental layers. The temperature of the substrate was monitored by control nickel - nickel chrome thermocouple connected to the substrate to observe any increasing in the substrate temperature.

Figure 3 shows the substrate covered by certain thin molybdenum mask to form two rows separated by 3 mm. Each row consists of six squares with 7 mm edge separated by 4 mm.

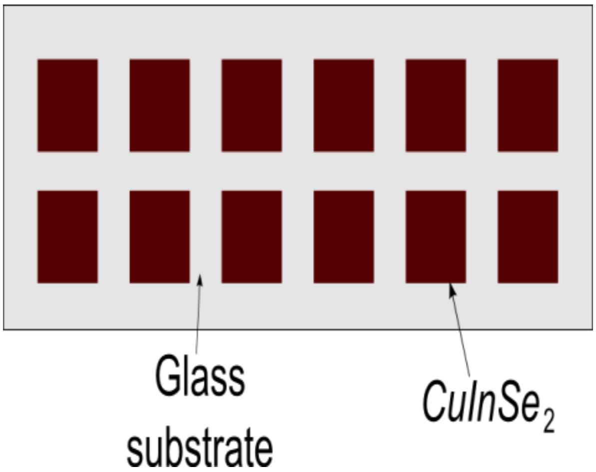


Fig. 3: Schematic diagram of the substrate

Two molybdenum boats with dimensions 3 × 6 × 3 mm were used as *Cu* and *In* sources. A cylindrical ceramic boat with 16 mm. diameter and 8 mm height was used as Se source. This boat was covered by circular ceramic plate with a slit of 2 mm at the centre. The temperature of the *Se* source was monitored by nickel-nickel chromel thermocouple.

Each of these sources was connected to a suitable power supply. Because of the very high temperature needed to evaporate *Cu* and *In* as shown in Table 1 a cooling system has been used to cool down the holders of these sources and to keep a reasonable temperature inside the evacuation chamber.

Deposition rate of the elemental materials and the thicknesses of deposited layers have been monitored by a quartz crystal monitor which was mounted near the upper side of the wheel, at the level of the substrate, and in the middle of the evacuated system. To prevent quarts monitor heating, it was connected to the cooling system also.

Two mechanical controlled shutters have been used. First shutter was used to screen/unscreen both *Cu* and *In* sources simultaneously and the *Se* source the other time, while the second one used as an additional shutter to screen the *Se* source, to ensure shielding of the *Se* source until it cools down the *Se* evaporation temperature, which takes time up to five minutes. Each source has been shielded from other sources by fixed shields installed between the sources.

2.3 Experimental procedure

The deposition of the elemental layers had been carried out as follow:

After achieving a suitable evacuation (< 10^{-4} torr) the substrate was placed above the screened *In* source by rotating the wheel. The *In* source was heated till melting, then the current was increased to boil and evaporate it. Unscreening the *In* source and depositing the it onto the substrate. During *In* evaporation, the current was regulated to keep a reasonable and stable flux of *In* vapour, which was observed as well as the thickness of the deposited layer, on the quartz monitor. When the desired thickness was obtained, the screen was returned again over the *In* source and the power supply was switched off. Then, the system was left for a minute to let the *In* source cool down.

Next, the substrate was placed over the *Se* source which was screened by its own screen, and then screened by the main screen. Then *Se* source has been heated until it was boiled and started evaporating. Unscreened the source and regulating the current to keep stable, steady and reasonable flux of *Se* vapour. The evaporation speed and the thickness of *Se* layer controlled and observed in the same manner as with *In*. When the desired *Se* thickness was deposited, the *Se* source screened again by its own screen and by the main screen also (to insure that *Se* flux do not reach the substrate) and switched off the *Se* power supply.

Before *Cu* was deposed, an enough time must be given to *Se* source to be cooled to ensure the stopping of *Se* evaporation.

To deposit the *Cu*, at first the copper source was screened, and then the substrate was placed over the *Cu* source by rotating the wheel, then the copper source was heated till evaporation, then depositing *Cu* on the substrate by un screening the source, evaporation speed and the deposited layer's thickness controlled and monitored in the same manner as with other sources mentioned above, when the desired thickness was reached, the *Cu* source was screened and the power supply was switched off.

In order to minimize the probability of evaporation of any impurity which may get to elemental sources unintentionally, at temperatures just before the evaporation of each of the elemental materials the temperature was keep for relatively long time (~30 S), and at evaporation temperatures, the substrate stayed at screen position for few seconds to prevent the deposition of the initial vapour which may contain impurities. Each three successive elemental layers named "sandwich". This sandwich can be repeated as many times as wanted, in this work it was repeated up to nine sandwiches, i. e., 27 elemental layers.

2.4 Annealing process

First annealing of the stacked elemental layers, the principal annealing, have been done in different ways. An additional annealing in air at low temperatures (not more than 150ºC) seems to be important to improve the electrical and optical properties of *CuInSe*2 thin films. The principal annealing has been carried out as follow:

Four samples from each run have been annealed as follows:

- First one was continuously annealed in argon ambient for 40 minutes at 400°C.
- Second one was annealed in argon ambient in three different temperature steps, 150°C, 300 °C, and 400°C, 15 minutes each step.
- Third one was continuously annealed in vacuum for 15 minutes at 400°C.
- Fourth one was annealed in vacuum in three different temperature steps, 150°C, 300°C, and 400°C, 5 minutes each step.

3. Characterization of $CuInSe_2$ material

3.1 Stoichiometry determination

The first few samples were consisted from one sandwich (three elemental layers). They have been used to determine the thickness (in kHz) of each of the elemental layer that needed to produced after annealing (with the other elemental layers) a chalcopyrite structure thin film with near stoichiometry composition. The thickness of the films were about 2 µm. The absorption spectrum between 1000 -1400 nm was used as a test for the formation of the chalcopyrite structure of the *CuInSe*2 compound which has absorption edge around 1 eV which close to result at the literature [xx] . Samples which showed absorption edge around 12000 nm tested by electron microprobe to know its atomic composition.

Table 1: Melting points and evaporation temperatures of the elements *Cu*,

In and *Se* [xxi].

Substance	Melting point ºC	Evaporation temperature ºC
Cu	1083	2336
In	156.4	2000 ∓ 10
Se	220	688

3.2 Composition and morphology

The morphology of $CuInSe_2$ thin films is strongly affected by precursor's composition and the following heat processing.

The *Se* content in the initial sandwich did not conserve after annealing, in spite of the *Se* excess in the initial precursors, to insure the P- type conductivity of the following *CuInSe*2, most of the films exhibit *Se* at. % **<** 50, Table 2, that is because of the *Se* re-evaporation due to its volatile nature and due to the exothermic reactions between elements that take place during the first steps of annealing [xxii].

Table 2: Atomic percentage composition in the range *Cu*- poor to *Cu*-rich for $CuInSe_2$.

Cu at. %	*In at. %*	*Se at. %*
22.23	29.25	48.62
23.00	27.48	49.52
23.06	28.16	48.78
25.44	24.97	49.59
26.12	24.72	49.15

The film has *In* as a first layer has the best adhesion to the glass substrate. The adhesion then depends on the *Cu* content, the *Cu*- poor film shows better adhesion. Samples with *Cu*- rich show comparatively large grain which attributed to binary phase formation, like $Cu_{2-x}Se$ [23], and exhibited less adhesion to the substrate with pinholes and cracks.

Films with *Cu* layer first on the glass showed weak adhesion and exhibited cracks. Thin films with *Se* layer first showed very weak adhesion to the glass substrate and very simple to peel up and it is friable.

Sample annealed or step annealed in argon were homogenous and show better electrical and optical parameters than samples with the same composition annealed in vacuum. It is expected that annealing in argon enable oxygen to substitute *Se* vacancies and the samples are less compensated.

Films color is strongly dependant on their composition and the method of annealing used. For a constant content of *Se*, samples with *In* excess in general were more light color, while *In* poor samples were dark. *Cu*-poor samples annealed or step annealed in vacuum have light brown color. Samples with the same composition annealed or step annealed in argon have dark brown color. *Se* lost in sample annealed in vacuum is more. The dark color of samples annealed in argon may be attributed to the impurities in argon and the oxygen traces which may react with the elements of ternary compound.

The annealing interval and temperature has a considerable effect on the quality of the material. The reaction between elemental layers takes place at the first moments of the annealing and the color of the precursor simultaneously change. Samples were annealed at temperatures less than 350ºC for less than 25 minutes seems to be non-chalcopyrite, and dominated by the binary phases. The chalcopyrite phase dominates when samples heated or step heated at 400ºC for 40 -45 minutes in argon, and for 15 minutes in vacuum.

3.3 X- ray diffraction

Sample with initial composition near stoichiometry were annealed under the same environmental condition. Samples show tetragonal phase. X-ray diffraction analysis confirms a single phase chalcopyrite structure for $CuInSe_2$ as shown in Figure 4. The reflections from planes (101) (103) (211) and (213) (known as super lattice reflection) can be seen clearly in the diffraction spectra of the $CuInSe_2$. The presence of these reflections confirms the formation of a good chalcopyrite film [xxiii]. The intensity of the (112) peak is abnormally high for all samples. The (112) preferred orientation in $CuInSe_2$ is required for good lattice matching with CdS in solar cells. The quality of the chalcopyrite phase is demonstrated by the resolved tetragonal splitting of 204/220 peaks, Figure 5. This results are close to the standard data in literature [xxiv].

Fig. 4: X- ray diffraction pattern of $CuInSe_2$ thin films

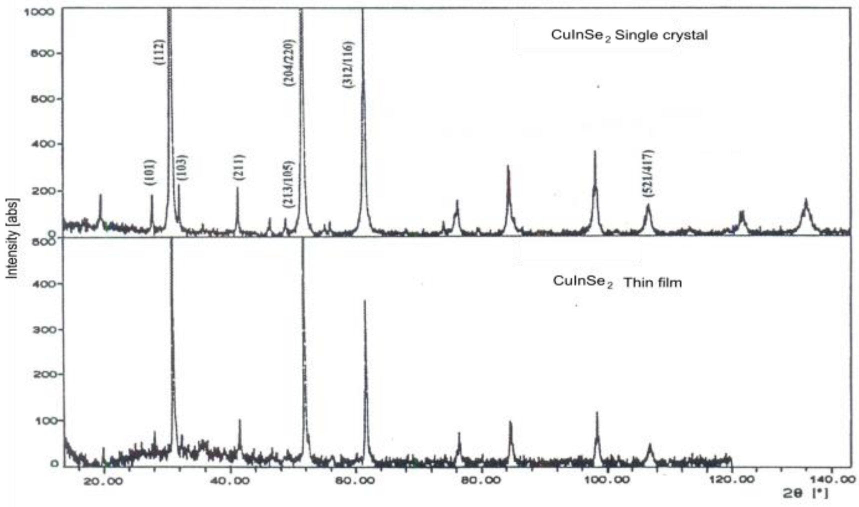
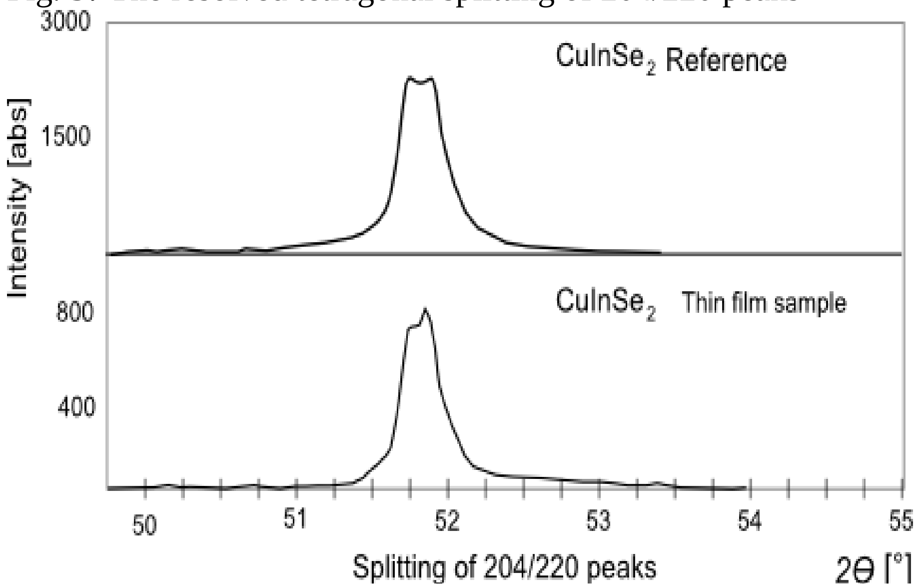


Fig. 5: The resolved tetragonal splitting of 204/220 peaks



4. Conclusion

In this work, $CuInSe_2$ have been successfully produced by sequential thermal deposition of elemental materials (*Cu*, *In* and *Se*) in vacuum chamber onto microscopic glass substrate at room temperature, each three sequential elemental layers are named "sandwiches". It was found that the sequence *In-Se-Cu* gave the best adhesion to the substrate. Films started with *Cu* on the substrate exhibited pinholes and cracks. Films started by *Se* as a first layer on the glass peeled off. The *Se* content in the initial sandwich did not conserve after annealing. Sample annealed or step annealed in argon were homogenous. X-ray diffraction analysis confirms a single phase chalcopyrite structure for $CuInSe_2$.

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الهدف من هذه الورقة البحثية هو دراسة مورفولوجيا وتكوين اغشية $CuInSe_2$ الرقيقة المصنعة بطريقة الرص المتتالي لطبقات العناصر الأساسية SEL. تم تسخين الأغشية بطرق مختلفة لزيادة طور Chalcopyrite الذي قد يحسن الخصائص الكهربائية والبصرية لتلك الأغشية. يسود طور Chalcopyrite عندما تسخن العينات الى درجة حرارة 400^0C مباشرة او بالتدرج لمدة 50 دقيقة بوجود غاز الأرجون، وتسخينه في الفراغ لمدة 15 دقيقة. تم تشخيص مكونات هذه الأغشية الرقيقة بايجاد النسب المئوية للمكونات الذرية وكذلك باستخدام تقنية حيود الأشعة السينية. وتصف هذه الورقة إنتاج اغشية $CuInSe_2$ متعدد البلورات (polycrystalline) ذو قمم حيود واضحة المعالم. وقد دُرست النسب المئوية الذرية للمركب لغرض التحقق من تكوُّن الأغشية المطلوبة. كما استخدمت شاشة الكوارتز البلورية لمراقبة سمك الطبقات المختلفة.

الفهرس

يشترط في البحوث العلمية المقدمة للنشر أن يراعى فيها ما يأتي :

- أصول البحث العلمي وقواعده .
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- يرفق بالبحث تزكية لغوية وفق أنموذج معد .
- تعدل البحوث المقبولة وتصحح وفق ما يراه المحكمون .
- التزام الباحث بالضوابط التي وضعتها المجلة من عدد الصفحات ، ونوع الخط ورقمه ، والفترات الزمنية الممنوحة للعديل ، وما يستجد من ضوابط تضعها المجلة مستقبلا .

تنبيهات :

- للمجلة الحق في تعديل البحث أو طلب تعديله أو رفضه .
- يخضع البحث في النشر لأوليات المجلة وسياستها .
- البحوث المنشورة تعبر عن وجهة نظر أصحابها ، ولا تعبر عن وجهة نظر المجلة .

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