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Effect of Heat and $CdCl_2$ Treatment on the Structure and Photoluminescence of CdTe/CdS thin Films

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ABSTRACT. This is to report on the structural and Photoluminescence (PL) studies of thin films of CdTe on glass or CdS (0.25 μm) / glass substrates which were prepared by the physical evaporation method. The CdS / glass substrate temperature was the variable parameter in this experiment. The effect of chemical and heat processing and substrate temperature on the structural and the electro-optical properties were analyzed. X-ray diffraction results showed a preferential growth in the (111) direction of treated and untreated films.

The Scanning Electron Microscopy results showed considerable grain growth with chemical treatment, and little effect on grain size with heat treatment only. The cycling of the chemical and heat treatment resulted in more growth in grain size. Photoluminescence (PL) spectra, in the region 1.6-1.2 eV, were taken for untreated and treated samples at 12K. All untreated samples showed undetectable PL spectra. The heat treated samples showed very weak PL relative to the chemically treated samples. Cycling the chemical and heat processing resulted in more intense PL spectra and resolved longitudunal-optical phonon replicas indicative of an improvement in film crystallinity. The PL spectra of samples with increasing substrate temperature showed a substantial decrease in intensity with a shift of the maximum emission towards lower energy. This was attributed to diffusion of sulfur in CdTe. The PL spectra, taken for chemically treated samples from the front excited surface and the junction excited surface are of similar intensity and of approximately similar structure. This was evidence of the penetration of the chemical/heat treatment deep in the whole film.

CdTe is considered a good semiconducting material for many device applications (Chu *et al.* 1992, Seto *et al.* 1988, Kim *et al.* 1992, Mandel *et al.* 1986, Meyers 1988) and a promising thin film material for photovoltaic solar cells due to its optimal direct band gap of about 1.5 eV at room temperature (Tribouletand 1973) which coincides with the peak of solar radiation. It also has long term stability, and good resistance to changes in weather conditions (Kazmerski 1993). Heterojunction solar cells constructed from CdTe as an absorber and CdS as a window have been investigated by many authors working in both academic institutions and industry. The recently reported (Chu *et al.* 1992, Kazmerski 1993) cell efficiencies in the vicinity of 15%, 1 cm^2 cell, stands as the highest reported so far. Still a number of problems are listed which deal with film quality, controlled doping, contacting, and junction quality.

PL is considered a good tool to investigate radiative defect states and intentional doping in the material (Chu *et al.* 1991, Amirtharaj 1990). PL spectra taken for films prepared by different techniques have showed different structure. Investigators have listed a number of transitions near the band gap, and have associated them with different sources and mechanisms. Conflicting assignments for some of the transitions exist in the literature. Therefore more work is needed to probe these transitions.

It was found that the commonly used treatment (Brain *et al.* 1991, Ringal *et al.* 1991) of CdTe films by saturated solution of $CdCl_2$ in methanol followed by heat treatment in the temperature range 400-500°C has resulted in the improvement of the efficiency of the CdTe/CdS solar cells (Ringal *et al.* 1991, Rohatgi *et al.* 1991). Apparently this is accompanied by an increase in grain size (Ringal *et al.* 1991, Vaccaro *et al.* 1991) and densification of CdTe (Chu 1991), formation of interfacial CdS_{1-x} Te_x (McCandless 1991, Lee *et al.* 1987) and a relatively stronger PL spectrum. The treatment also produced a gradual decrease in the concentration gradient of oxygen and chlorine with depth from the surface (Moutinho *et al.* 1992).

In this paper, we report the results of X-ray diffraction, Scanning Electron Microscopy "SEM" and the PL spectra of untreated and treated films of CdTe. The PL spectra were taken from the front and the back of the films. The CdTe films were grown on top of CdS films which were kept at different substrate temperatures. This allows one to explore the role of different treatments on the quality of the film, and to correlate it to the diffusion of cadmium, sulfur and chlorine in the CdTe film.

Experimental Details

A commercially available polycrystalline CdTe of 99.9999% purity was used as the source material. The CdTe and CdS films were deposited on a glass substrate by physical vapor deposition. The CdTe films were either deposited directly over the glass substrate which was kept at room temperature, or on CdS (0.25 μm) / glass substrate which was kept at temperature "T" = 18, 50, 150, 250 °C. These structures, in addition to the substrate temperature change, were chosen to study the diffusion of sulfur by observing the changes which occur in the PL spectra.

The prepared films were divided into several smaller parts for different treatment procedures. Saturated solution of $CdCl_2$ in methanol was prepared for the chemical treatment. CdTe / glass films were treated according to the following experimental processing procedures: (1) samples were annealed at 400 °C for 30 mins; (2) same as (1) then dipped in the $CdCl_2$ solution, followed by annealing at 400°C for 30 mins; (3) same as (2) then dipped in the $CdCl_2$ solution, followed by annealing at 400°C for 30 mins; (4) same as (3) then dipped in the $CdCl_2$ solution, followed by annealing at 400°C for 30 mins. All samples were annealed in air at atmospheric pressure. All the chemically treated samples were annealed first, before any further treatments. This annealing before the chemical treatment was found to be an important step for the films to adhere to the substrate and to produce a uniform surface distribution of $CdCl_2$ on drying. The heat treatment before the chemical treatment diffuses cadmium and tellurium into glass (Ahmad-Bitar *et al.* 1995) and probably diffuse sodium, oxygen, ... into the film. The cycling of the chemical treatment (treatment 3 and 4) was done to check on the effect of this treatment on the grain size and PL spectra. It was found by others (Baron *et al.* 1990, Rohatgi *et al.* 1992, Moutinho *et al.* 1992) that films of CdTe on glass which are directly dipped in $CdCl_2$ followed by heat processing had a nonuniform surface distribution of $CdCl_2$ on drying and became separated from the glass substrate following the chemical and heat treatment.

Treated and untreated samples were studied by X-ray diffraction, Scanning Electron Microscopy "SEM", and photoluminescence. For the PL measurements the samples were housed in a closed-cycle He refrigerator and the excitation source was a 19 mW diode laser with 690 nm wavelength. The luminescence was collected with a frensel lens and detected with a photomultiplier tube and the output was electronically processed by an electrometer, a data acquisition unit, and an HP-computer. The luminescence spectra were monitored using a 0.25-m monochrometer calibrated with the laser line and the Hg-lines.

Results and Discussion

1. *Structural Investigation*

The orientation of the treated and the untreated films were found by the X-ray diffraction. The structural orientation for these films confirmed the cubic structure for the CdTe with a predominant strong reflection from the (111) plane and the hexagonal structure for the CdS films with predominant strong reflection from the (002) plane, and a weak reflection from (110) plane. The $CdCl_2$ treatment decreases the intensity of the reflection from the (111) plane and causes the appearance of extra weak lines (non-CdTe or underlying film) similar to what was reported earlier (Baron *et al.* 1990, Moutinho *et al.* 1992).

The topography and morphology of the films were studied by the SEM. Fig. 1 shows the SEM micrographs of untreated and treated samples. The processing of CdTe by chemical ($CdCl_2$) followed by heat treatment provides for the diffusion of Cd, O_2, and Cl inside the film (Ahmad-Bitar *et al.* 1995, Moutinho *et al.* 1992). These elements or some of them mainly chlorine may act as an active element to join either micro-single crystals or small grains together leading to the observed increase in grain size. This suggests that $CdCl_2$ followed by heat treatment acts as a recrystallizing agent. In addition, the cycling of the chemical ($CdCl_2$) / heat treatment leads to a further increase in grain size of the film as can be seen in Fig. 1.

2. *Photoluminescence Investigation*

All as grown CdTe films (untreated) gave undetectable PL spectra. This is interpreted in terms of the high concentration of deep states linked to the vacancies of Cd or Te (Kuhn *et al.* 1992, Krustok *et al.* 1990, Sobiesierski *et al.* 1990, Sobiesierski *et al.* 1988) or the high concentration of trapping centers or both. Also CdTe does not exist in the vapor phase (Chu *et al.* 1991), and it is formed on the substrate after deposition. Small concentration of atomic Cd, Te and amorphous CdTe can't be ignored as a cause of the weak PL.

The CdTe films, annealed at 400 °C, show a weak PL spectra as can be seen in Fig. 2-a and the inset. The treated films however gave a broad band centered around 1.46 ev. Most investigations in the literature reported on the presence of this band for crystals (Sobiesierski *et al.* 1988, Millokhin *et al.* 1991, Cotal *et al.* 1990, Seto *et al.* 1988) and films (Sopanon 1990, Feng *et al.* 1988, Mendoza-Alverez *et al.* 1988, Figueroa *et al.* 1986, Cardenas *et al.* 1984, Norris 1980, Barnes 1975) irrespective of the different preparation methods. The origin of this band is widely discussed, but most investigators have associated it with the recombination of donor-accepter pairs "DAP". This DAP emission is referenced to copper (Seto *et al.* 1988) or silver (Feng

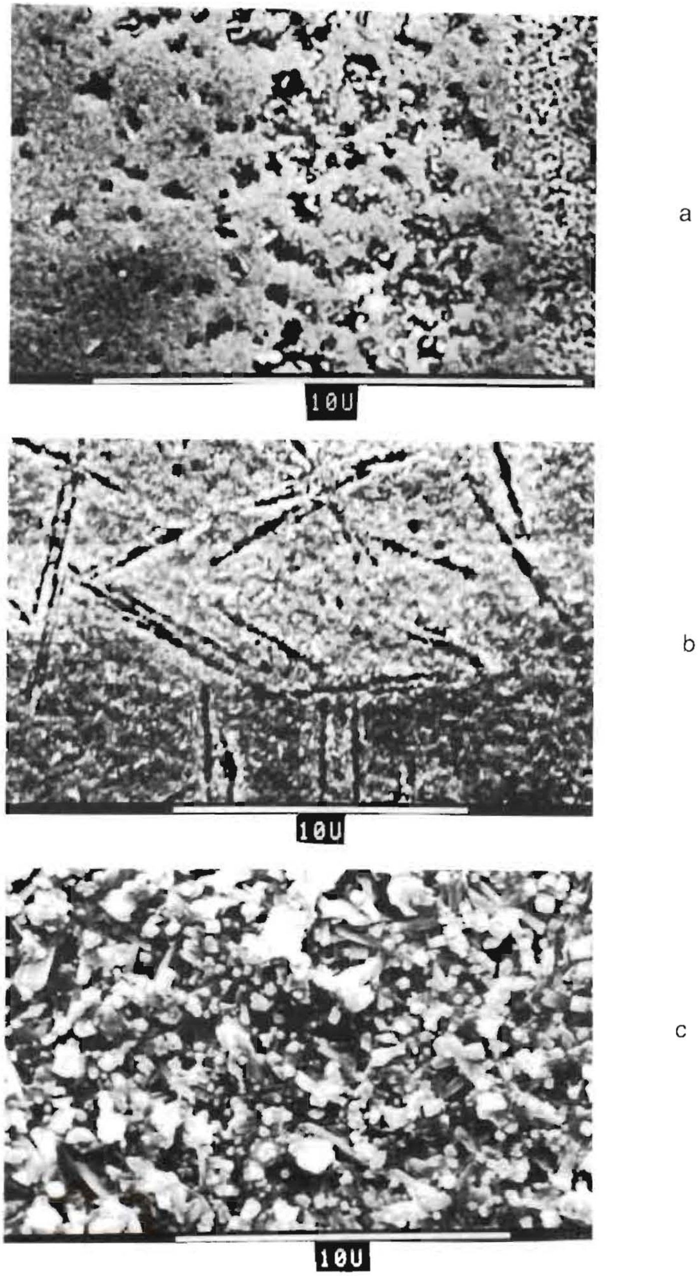

Fig. 1. SEM photomicrographs of a) annealed at 400 °C for 30 mins; b) annealed at 400 °C for 30 mins then dipped in the $CdCl_2$ solution, followed by annealing at 400°; c) same as (b) then dipped in the $CdCl_2$ solution, followed by annealing at 400 °C for 30 mins.

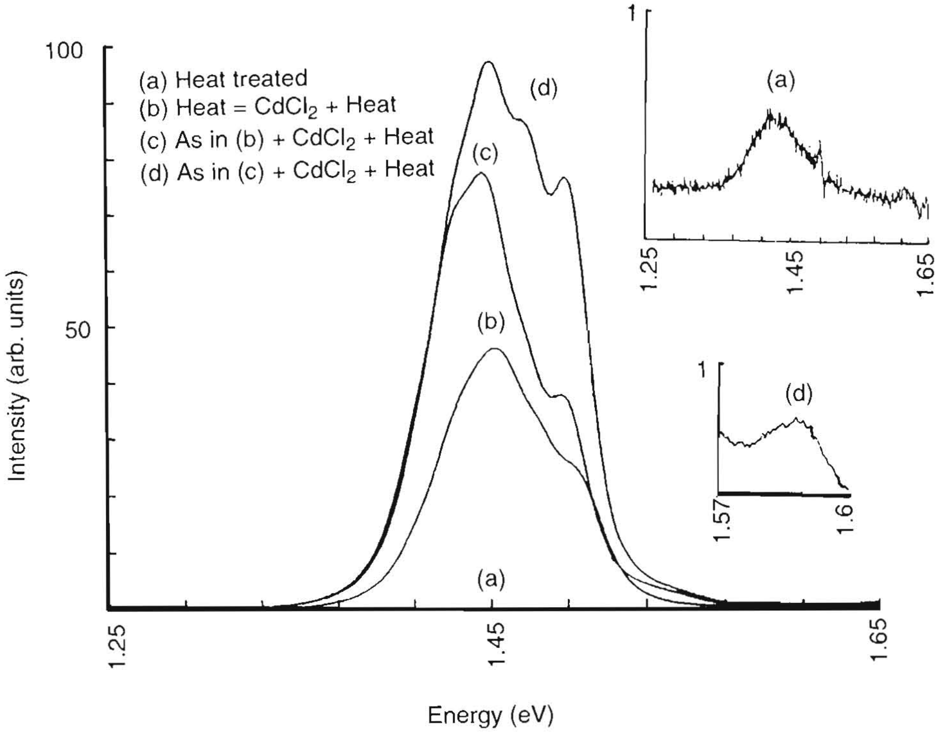

Fig. 2. Photoluminescence spectra for CdTe/glass with different treatment: a) annealed at 400 °C; b) annealed at 400 °C/chemical dipping in CdCl2/annealed at 400 °C; c) as in "b" then followed by chemical dipping in CdCl2/annealed at 400 °C; d) as in "c" then followed by chemical dipping in CdCl2/annealed at 400 °C.

et al. 1988) on cadmium sites which act as a shallow acceptor (Seto *et al.* 1988) or to surface defects (Cardenas *et al.* 1984). Copper is mobile in CdTe and often was observed as a dominant residual impurity (Seto *et al.* 1988, Kazmerrski 1993). Myers *et al.* (1983) have suggested that this band is related, at least in part, to the presence of srface states.

a) Effect of CdCl$_2$ / Heat Processing

The effect of different chemical and heat processing methods were examined by PL to investigate its effect on shallow levels, deep levels and the surface states. Fig. 2 shows the effect of treatment on the PL of CdTe / glass films: (b) is annealed first at 400 °C for 30 mins, then dipped in $CdCl_2$ solution followed by annealing at 400 °C for 30 mins; (c) treated as (b) then dipped in $CdCl_2$ solution followed by annealing at 400 °C for 30 mins; (d) treated as (c) then dipped in $CdCl_2$ solution followed by annealing at 400 °C for 30 mins. The role of the chemical treatment leads to a clear increase in the radiative shallow defect transitions. Cycling the chemical treatment has resulted in an increase in the intensity of the PL spectrum which confirms this role. The appearance of the PL spectrum in this energy range may be due to a decrease in deep defect states or that either chlorine or oxygen have electrically neutralized (*e.g.*, V_{Cd} or V_{Cd} - complexes) them. The increase of the grain size also suggests (see Fig. 1) that the number of traps and defects at the grain surface decreases and chlorine might electrically passivate these regions leading to the observed increase from the shallow levels. The width of this band did not change with the cycling of the treatment implying undetectable change in the distribution of defects producing this band. Cycling the treatment increased the PL intensity and resulted in resolving this broad band into small peaks at 1.43, 1.45, 1.47, and 1.49 eV separated approximately by 0.02 eV suggesting the participation of longitudunal-optical (LO) phonons in producting them. The appearance of these phonon replicas is an indication of the improvement in the crystallinity of the lattice (Moutinho *et al.* 1992, Giles-Taylor *et al.* 1985, Hofman *et al.* 1992). The crystalline quality of the films can be determined by comparing the intensity of the 1.42 band to the intensity of the band originating from the bound excitons at 1.59 eV. The stronger the 1.59 eV band, the better is the crystalline quality of the film. The sample treated as in (d) showed the strongest exciton band at 1.59 eV relative to the other samples. This band is shown in the inset 2 in Fig. 2. The samples treated as in c and b showed an undetectable signal for the 1.59 eV band.

Fig. 3a shows the PL spectrum of the film taken from the excited front surface side, while Fig. 3b shows the PL spectrum taken from the excited CdTe/CdS junction side. The $CdCl_2$/heat processing was done to this film. The figure shows that the PL spectra taken from the front and from the junction surfaces are relatively of similar intensity and of approximately similar structure.

This is a conclusive evidence that this treatment had penetrated deep in the whole film. This result also confirms the active role of $CdCl_2$/heat processing at the junction which implyes the diffusion of oxygen and chlorine deep inside the film and reaching the CdTe/CdS interface. The compositional analysis made by the electron microprobe and the X-ray photoelectron spectroscopy (Moutinho *et al.* 1992) confirmed an oxygen gradient to 5000 A° deep, higher Cd levels, and 7 at. % of chlorine near the surface due to $CdCl_2$/heat processing.

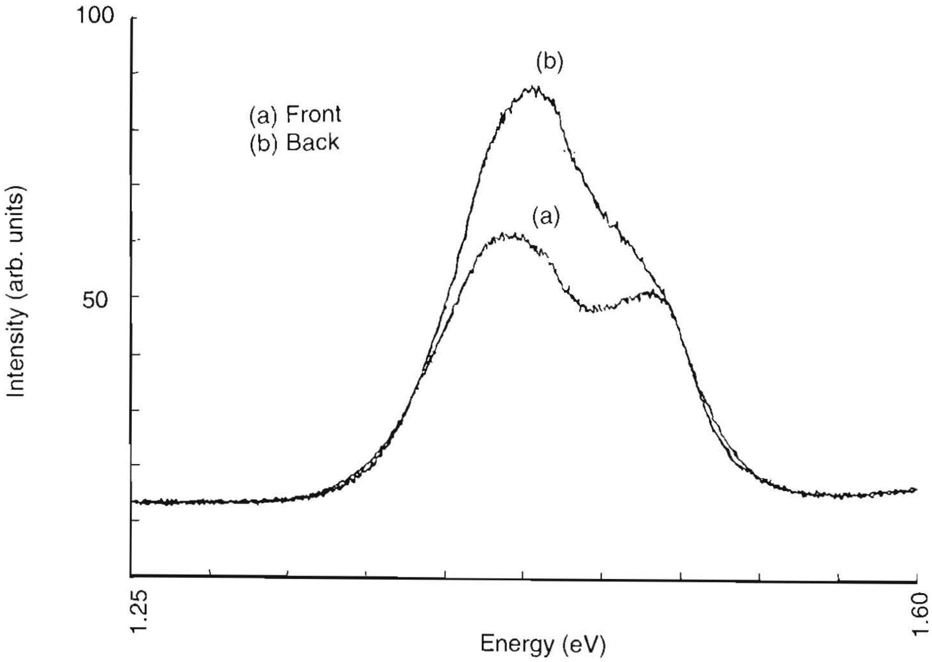

Fig. 3. Photoluminescence spectra for CdTe / CdS / glass taken from the excited: a) front surface side; b) CdTe / CdS junction side.

b) Effect of Substrate Temperature

Fig. 4 shows five PL-spectra of C dTe films on CdS/glass substrates at temperatures T = 18, 50, 100, 150, and 250°C respectively. These films were first annealed at 400 °C for 30 mins then dipped in $CdCl_2$ followed by 400 °C heat annealing. The spectra have shown two main features. Firstly, the maximum PL intensity shifts from about 1.47 eV for the film with 18°C substrate temperature to 1.42 eV for the other films. Secondly, films with substrate temperatures above 50°C showed that the PL intensity decreases very fast with an increase of substrate temperature. The PL spectrum of the 50 °C substrate temperature showed the highest intensity and a relatively strong band at 1.55 eV indicative of a better crystalline quality for this film.

The PL spectrum for the film with 18 °C substrate temperature had the LO-Phonon replicas. The PL spectrum of this film compares relatively well with the PL-spectrum of Fig. 2d and 2c. This shows the importance of the lattice match between CdTe and CdS films. The maximum emission intensity occurs at approximately the same value as the PL spectra for the CdTe/Glass films shown in Fig. 2 and of approximately the same width.

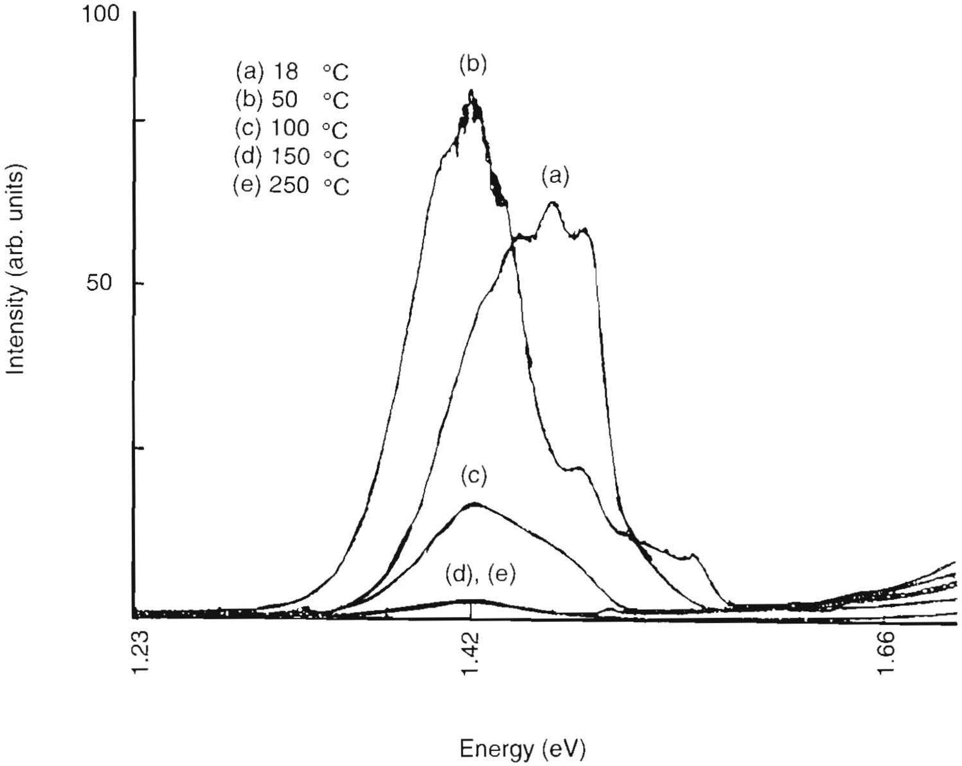

Fig. 4. Photoluminescence spectra for CdTe / CdS / glass with substrate temperatures: a) 18 °C; b) 50 °C; c) 100 °C; d) 150 °C; e) 250°C.

The PL spectra for the 50, 100, 150, and 250 °C substrate temperatures do not show the LO-Phonon replicas and the maximum intensity occurs at 1.42 eV. Also increasing the substrate temperature resulted in a decrease in PL intensity. These resutls can be attributed to the diffusion of sulfur. Sulfur diffusion into CdTe films, which was investigated by secondary ion mass spectrometry (Keyes 1991), was found to be a rapid process even at low temperature (375 K). The increase of sulfur diffusion by increasing substrate temperature might have an effect on the electrically active states. The shift in the position of the maximum emission peak suggests that this band in fact consists of more than one band originating from various impurity acceptor atoms as Cu, Ag, Na,....

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تأثير المعالجة الحرارية وكلوريد الكاديوم $CdCl_2$ على التركيبية و السطوع الضوئي لرقائق CdTe/CdS

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تم دراسة التركيبية والسطوع الضوئي (PL) لرقائق CdTe تم تحضيرها بطريقة التبخير الفيزيائي على طبقة تحتية من الزجاج أو CdS (0.25 µ m)/ زجاج . تم إعتبار درجة حرارة الطبقة التحتية CdS (0.25 µ m)/ زجاج معاملاً متغيراً في هذه التجربة ، كما تم تحليل تأثير المعالجة الحرارية والكيميائية ودرجة حرارة الطبقة التحتية على الخواص التركيبية والكهرو-بصرية .

إن نتائج حيود الأشعة السينية أظهرت، نمواً إنتقائياً للإتجاه (III) للرقائق المعالجة والغير معالجة . إن نتائج المجهر الألكتروني الماسح أظهرت نمواً وافراً للحبيبات نتيجة المعالجة الكيميائية وتأثيرات قليلة على حجم الحبيبات نتيجة المعالجة الحرارية فقط . إن تكرار المعالجات الكيميائية والحرارية أدت إلى زيادة أخرى في حجم الحبيبات . أخذت أطياف PL في المنطقة (1.2-1.6 eV) لعينات معالجة وغير معالجة عند درجة حرارة K12 . لم تعط جميع العينات الغير معالجة أية أطياف PL يمكن إلتقاطها . العينات المعالجة بالحرارة أعطت أطياف PL ضعيفة بالنسبة للعينات المعالجة كيميائياً . إن تكرار المعالجات الكيميائية

والحرارية أنتجت أطياف PL أكثر شدة وصوراً متماثلة ومتحللة للفونون البصري - الطولي مما يدل على تحسن تبلور الرقيقة .

أظهرت أطياف PL للعينات ذات درجات الحرارة المتزايدة للطبقة التحتية إضمحلالاً وافراً في الشدة مع إنزياح لقمة الانبعاث الاعظم بإتجاه الطاقة الصغرى . وقد عزي هذا السلوك إلى إنتشار الكبريت في CdTe .

أطياف PL التي أخذت من السطوح الأمامية المستثارة لعينات معالجة ومن سطح الوصلة المستثارة كانت متشابهة في الشدة ومتقاربة من حيث التركيب . كان هذا دليل على تغلغل المعالجات الكيميائية / الحرارية في عمق كل الرقيقة .