

## INFLUENCE OF 1-GROUP IMPURITIES ON THE PROPERTIES OF CdS FILMS

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The influence of 1-group impurities (lithium, sodium, copper and silver) on electrical, photoelectrical and luminescence properties of CdS films, prepared by the chemical spray pyrolysis method, are investigated. It is shown that, the appearance of photoconductivity band with maximum at  $\lambda = 550$  nm and green emission band at  $\lambda = 510$ -540 nm in CdS are connected with the same center-negatively charged cadmium vacancies. The increase of the intensities of these bands under diffusion doping by lithium and sodium ions and decrease of them under introduction of copper and silver ions, are due to donor-acceptor interaction of diffused 1-group impurities with charged cadmium vacancies.

### 1. Introduction

As it is known, cadmium sulphide thin films are one of the most interesting materials used in optoelectronic techniques. On these basis, the electroluminescent devices of visible spectrum and thin films  $Cu_2S$ -CdS,  $CdTe$ -CdS,  $CuInSe_2$ -CdS heterojunctions are presently made which are quite useful for the creation of effective and unexpensive solar cells [1-7]. Therefore, study of the nature of photosensitivity, radiation recombination centers and effect of different factors (doping, thermal treatment, illumination, etc.) on these centers attract a large interest. In this work, the results on the investigations of the influence of 1 group impurities, introduced by both diffusion and during deposition process and also different thermal treatments on the electrical, photoelectrical and luminescent properties of CdS thin films, prepared by chemical spray pyrolysis method, are presented.

The 1-group impurities in CdS diffuse according to dissociative mechanism, which is connected with their rapid migration along the interstitials as positive ions being subsequently situated in vacancies, where they exhibit an acceptor properties [8]. Since the vacancies in CdS can also be electrically charged (cadmium vacancies are acceptors and sulphur vacancies-donors [9]), donor-acceptor interaction between diffused 1-group impurities and vacancies can mutually influence on the diffusion and concentration of impurities and vacancies [10], and subsequently on the properties of the cadmium sulphide [9,11-15].

It should be noted that, besides the donor-acceptor interaction, the pressure of sulphur vapour can also influence (under diffusion annealing process) on the diffusion and solubility of 1-group impurities in CdS (which contains easy volatile component-sulphur) and permit the control of the vacancies concentration in each sublattice of CdS [10]. Therefore, the investigation of interaction character of 1-group impurities, introduced by diffusion into CdS at different sulphur vapour pressure with vacancies and the influence of such interaction (impurity-vacancy type) on the photoelectrical and luminescent properties of CdS thin films excites some interest.

### 2. Experimental methods

The cadmium sulphide thin films were prepared by spray pyrolysis method which basically involves the

spraying of an aqueous mixture of  $CdCl_2$  (0.025 M) and thiourea  $(NH_2)_2CS$  (0.025 M) into heated ( $\sim 400^\circ C$ ) substrates [11]. As a substrate, a chemically cleaned glass with and without a conductive layer of  $SnO_2$ , was used. The spray rate was about  $5 \text{ ml}\cdot\text{min}^{-1}$ , which provided a deposition rate about  $50 \text{ nm}\cdot\text{min}^{-1}$ . The CdS samples used in this investigation were  $0.5 \text{ cm}^2$  in areas and had thicknesses  $\sim 10 \mu\text{m}$ .

The investigated impurities were lithium, sodium, copper and silver. Diffusion of lithium and sodium was carried out from vapour phase, while diffusion of copper and silver from diffusant layers deposited by vacuum evaporation onto the surface of samples in evacuated quartz ampoules. For the creation of equilibrium pressure of sulphur vapour, certain of sulphur was placed in the amount ampoul. In this case, the pressure of sulphur vapour in ampoul at annealing temperature ( $P_2=0.5$ -1.5 at.) established by sulphur weight and the ampoul volume. The introduction of impurities from a permanent source was realized at the temperature range of  $500$ - $600^\circ C$  and time sufficient for the uniform saturation of samples by studying impurities, the concentration of impurities exceeds the concentration of initial donor impurities.

To check the possible influence of annealing on the properties of CdS, a control sample was subjected to heat treatment under the analogous conditions (but without any impurity). After diffusion process, the surface of samples was etched in aqueous solution of hydrochloric acid and washed in distilled water.

To compare the results obtained on the samples prepared by diffusion doping, a number of CdS films doped by sodium and copper during the spray deposition process were prepared. In this case, the impurities were introduced by the addition of a small amounts of NaCl and CuCl salts into the sprayed solution.

The Ohmic contacts to the samples for the electrical and photoelectrical measurements were made by vacuum evaporation of indium.

The photoconductivity spectra of the samples were measured at room temperature by standard methods using a SPM-2 monochromator.

The photoluminescence spectra of the samples excited by a  $N_2$ -laser ( $\lambda=337 \text{ nm}$ ) were taken by a DFS-24 spectrometer and registered by a synchronized detecting system at 77 K.

## 3. Experimental results and discussion

At first, we consider the effect of I-group impurities introduced by diffusion method on the properties of CdS films.

Diffusion doping of n-type CdS by the impurities (Li, Na, Cu, Ag), results in an essential increase of resistivities of the samples (table 1). After diffusion, conductivity of the samples remain n-type.

Fig. 1 shows the conductivity spectra of CdS samples, doped by lithium(1), sodium(2), copper(3), silver(4) ( $T=600^\circ\text{C}$ ,  $t=3$  hr, at fixed pressure of sulphur vapour  $P_S=1.0$  at.) and as-grown sample(5). It should be noted that, after diffusion annealing, the control sample, unlike the samples doped by I-group impurities (under the same surface treatment) become nonphotosensitive. From comparison of spectra of the samples doped by I-group impurities, one can distinguish the main peculiarity: in the samples doped by lithium and sodium, a wide line of photoconductivity with a maximum at  $\lambda=550$  nm is observed, while in the samples doped by copper and silver, the photoconductivity at this range of the spectra is minimal. The diffusion doping by lithium and sodium extinguishes an intrinsic photoconductivity at  $\lambda<520$  nm.

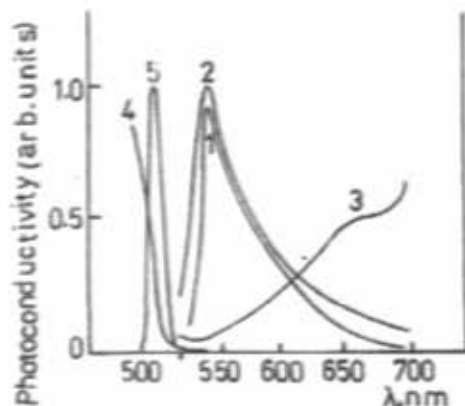


Fig. 1. The photoconductivity spectra of chemically sprayed CdS films, diffusion doped ( $T=600^\circ\text{C}$ ,  $t=3$  hr,  $P_S=1$  at.) by lithium(1), sodium(2), copper(3), silver(4) and as-grown (5) samples ( $T=300$  K).

In Fig. 2 the photoconductivity spectra of control samples annealed at  $T=500^\circ\text{C}$ ,  $t=2$  hr at different sulphur vapour pressures  $P_S=0.5$  at.(1),  $1.0$  at.(2) and  $1.5$  at.(3) are given. It is seen that, the intensity of  $\lambda_{\text{max}}=550$  nm line is raised by increasing the sulphur vapour pressure. The intensity dependence of this line on sulphur vapour pressure (Fig. 2 inset) is described by following relation

$$I_{ph} = B P_S^{1/2} \quad (1)$$

where,  $B$  is a constant.

In Fig. 3 the photoluminescence spectra of the samples doped by sodium(1), copper(2), control sample(3) annealed in sulphur vapour and reannealed sample 3 in cadmium vapour (4) at  $T=500^\circ\text{C}$ ,  $t=3$  hr are given. As it is clear from Fig. 3, the diffusion doping by sodium(1) and annealing in sulphur vapour (3) leads to appearance of an intensive green band of luminescence (so called "edge emission" [17]) in the range  $\lambda=510-540$  nm [17,18], while in the samples doped by copper (2), the green emission is

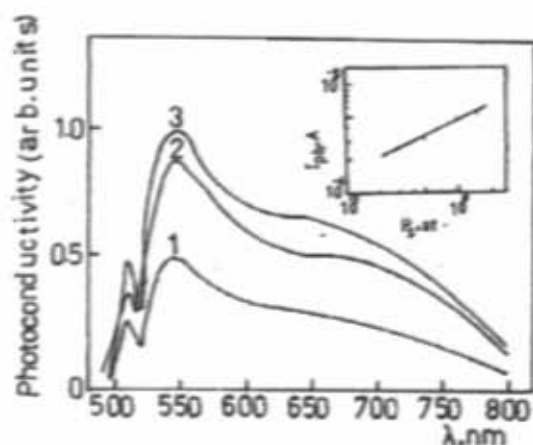


Fig. 2. The photoconductivity spectra of chemically sprayed CdS films, annealed ( $T=500^\circ\text{C}$ ,  $t=2$  hr) at different pressure of sulphur vapour  $P_S=0.5$  at.(1),  $1.0$  at.(2),  $1.5$  at.(3) ( $T=300$  K). Inset shows the photoconductivity peak (at  $\lambda_{\text{max}}=550$  nm) intensity versus the sulphur vapour pressure.

absent. Reannealing the sample (3) in cadmium vapour results in disappearance of green emission (curve 4).

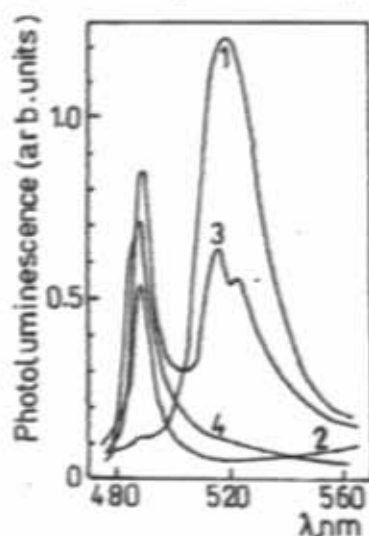


Fig. 3. The photoluminescence spectra of chemically sprayed CdS films, diffusion doped ( $T=500^\circ\text{C}$ ,  $t=3$  hr,  $P_S=1$  at.) by sodium(1), copper(2), annealed in sulphur vapour (3) and the spectrum of the film 3, reannealed in cadmium vapour(4) ( $T=77$  K).

Now we consider the effect of I-group impurities introduced during the deposition process on the properties of CdS films.

In contrast to the diffusion doping, the I-group impurities introduced during the deposition process leads to considerable decreasing of resistivities of CdS films (see, table 1).

Fig. 4 and 5 show respectively the photoconductivity and cathodoluminescence spectra of CdS films, as grown (1) and doped by sodium impurities by addition of 100 mg/l (2) and 200 mg/l (3) NaCl to the solution (the photoconductivity and cathodoluminescence spectra of samples doped by copper impurities show the same appearance, therefore, are not given). "The photoconductivity spectrum of as-grown CdS films (1), besides the funda-

mental absorption line (at  $\lambda < 520$  nm) contains a slight band in the region  $\lambda = 560-800$  nm which is increased by increasing the sodium (and copper) concentration. Such a band is observed and increased by increasing  $Na$  (and  $Cu$ ) concentration at cathodeluminescence spectra, which is the superposition of orange and red bands of luminescence in  $CdS$  [19, 20]. As it is seen from Fig. 4 and 5, in contrast to the diffusion doping, the sodium impurity introduced during the deposition process does not stimulate the appearance of photoconductivity maximum at  $\lambda = 550$  nm and also green luminescence in  $CdS$  films.

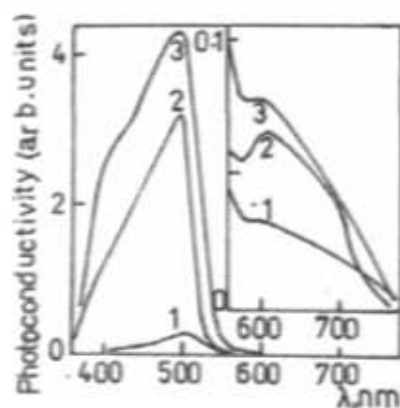


Fig. 4. The photoconductivity spectra of chemically sprayed  $CdS$  films, as-grown (1) and sodium doped during the deposition process by the addition of 100 mg/l  $NaCl$  (2) and 200 mg/l  $NaCl$  (3) to the solution ( $T = 300$  K).

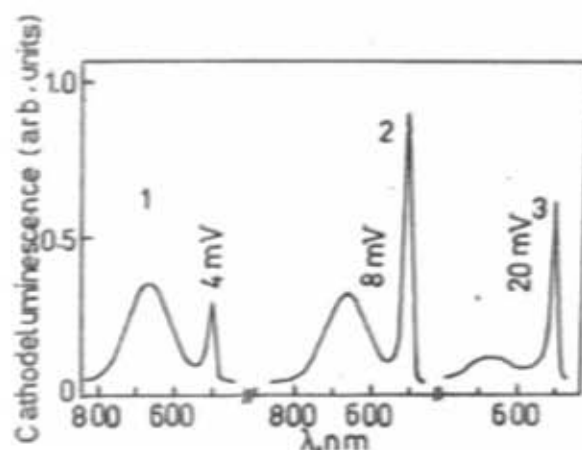


Fig. 5. The cathodeluminescence spectra of chemically sprayed  $CdS$  films, as-grown (1) and sodium doped during the deposition process by the addition of 100 mg/l  $NaCl$  (2) and 200 mg/l  $NaCl$  (3) to the solution ( $T = 77$  K).

The thermal treatment in sulphur vapour ( $P_s = 1$  at,  $T = 500^\circ C$ ,  $t = 2$  hr) of  $CdS$  films doped by sodium and copper during the deposition process (200 mg/l  $NaCl$  and 200 mg/l  $CuCl$ ), essentially changes their photoconductivity and luminescence spectra. In the photoconductivity and luminescence spectra of the films doped by sodium impurity the maximum at  $\lambda = 550$  nm and green emission are observed respectively, while in the films doped by copper the maximum at  $\lambda = 550$  nm and green emission are not observed (as shown in Fig. 1 and 3).

So, from the above given experimental data on joint study of photoconductivity and luminescence spectra of  $CdS$  films, doped by 1-group impurities and annealed in sulphur and cadmium vapour, are followed:

- the photoconductivity line with maximum at  $\lambda = 550$  nm and green emission at  $\lambda = 510-540$  nm are observed in  $CdS$  films, diffusion doped by lithium and sodium impurities and thermally treated in sulphur vapour samples. In the latter case, intensity of the photoconductivity at  $\lambda = 550$  nm is increased by increasing the sulphur vapour pressure.

- in  $CdS$  films, diffusion doped by copper and silver impurities, the photoconductivity line at  $\lambda = 550$  nm and green emission are not observed.

- reannealing the  $CdS$  films (which were previously annealed in sulphur vapour) in cadmium vapour, results in disappearance of photoconductivity at  $\lambda_{max} = 550$  nm and green emission.

- doping of  $CdS$  films by sodium impurity during deposition process (by the addition of  $NaCl$  to the solution) does not lead to appearance of photoconductivity maximum at  $\lambda = 550$  nm and green emission but increases the intensity of existing bands of photoconductivity and luminescence. The same character holds for photoconductivity and luminescence spectra of samples doped by copper impurity.

- thermal treatment in sulphur vapour of the samples, doped by sodium during deposition process gives rise to appearance of photoconductivity maximum at  $\lambda = 550$  nm and green emission, but they are not occurred in the samples, doped by copper impurity.

The given experimental data show that, the appearance of photoconductivity at  $\lambda = 550$  nm and green emission in  $CdS$  films, diffusion doped by lithium, sodium and annealed in sulphur vapour is due to the one and the same center in  $CdS$ .

The obtained results can be explained, assuming that, the photoconductivity at  $\lambda = 550$  nm and green emission are connected with negatively charged vacancies of cadmium. Proceeding from this assumption let us first explain the results on increasing of intensity of photoconduction peak at  $\lambda = 550$  nm by sulphur vapour pressure, given in Fig. 2, and appearance of green emission.

The concentration of charged cadmium vacancies  $N(V_{Cd}^-)$  and sulphur vacancies  $N(V_S)$  in both of  $CdS$  sublattices are connected with each other by mass action law [10].

$$N(V_{Cd}^-) \cdot N(V_S) = K \quad (2)$$

where,  $K$  is a constant of vacancies formation reaction.

By increasing the sulphur vapour pressure  $P_s$ , the sulphur vacancies concentration is decreased, and according to the relation (2), the cadmium vacancies concentration must be increased, which results in an increase of photoconductivity intensity at  $\lambda = 550$  nm and green emission, due to these vacancies. The dependence of cadmium vacancies concentration on partial pressure of sulphur vapour is described by relation

$$N(V_{Cd}^-) = KP_s^{-1/m} \quad (3)$$

where,  $m$  determines the number of atoms of sulphur molecules in gaseous state [10].

Dark ( $\rho_d$ ) and light ( $\rho_l$ ) (under illumination power density  $W=300 \text{ mW/cm}^2$ ) resistivities of as-grown, control, and lithium, sodium, copper, silver doped CdS films, prepared by chemical spray pyrolysis method.

No	Samples	Annealing Conditions	$\rho_d$ Ohm.cm	$\rho_l$ Ohm.cm	$\rho_d / \rho_l$
1	CdS as-grown	-	$7 \cdot 10^3$	$3 \cdot 10^3$	2.3
2	CdS: Li	$600^\circ\text{C}$ , 3hr, $P_3=1$ at	$3 \cdot 10^8$	$6 \cdot 10^3$	$5 \cdot 10^4$
3	CdS: Na	" - "	$2 \cdot 10^9$	$5 \cdot 10^3$	$4 \cdot 10^5$
4	CdS: Cu	" - "	$2 \cdot 10^7$	$10^4$	$2 \cdot 10^3$
5	CdS: Ag	" - "	$5 \cdot 10^8$	$3 \cdot 10^3$	$2 \cdot 10^4$
6	CdS - Control	" - "	$9 \cdot 10^8$	nonphotosensitive	-
7	CdS - Control	$500^\circ\text{C}$ , 2hr, $P_3=1.5$ at	$4 \cdot 10^7$	$5 \cdot 10^3$	$8 \cdot 10^3$
8	CdS - Control	$500^\circ\text{C}$ , 2hr, $P_3=1.0$ at	$3 \cdot 10^7$	$8 \cdot 10^3$	$3 \cdot 10^3$
9	CdS - Control	$500^\circ\text{C}$ , 2hr, $P_3=0.5$ at	$2 \cdot 10^7$	$2 \cdot 10^4$	$10^3$
10	CdS: 100 mg/l NaCl addition to the solution	-	$1.5 \cdot 10^3$	$10^2$	$1.5 \cdot 10^1$
11	CdS: 200 mg/l NaCl	-	$2 \cdot 10^2$	$10^1$	$2 \cdot 10^1$
12	CdS: 200 mg/l CuCl	-	$3 \cdot 10^2$	$2 \cdot 10^2$	$1.5 \cdot 10^1$

The experimentally measured dependence of photoconductivity maximum at  $\lambda=550 \text{ nm}$  on sulphur vapour pressure in exactly described by relation (3), namely  $I_{ph} = BP_s^{1/2}$ , which shows that, in our experiments the sulphur molecules during the diffusion have two atoms preferentially in the gaseous state ( $m=2$ ).

Now let us discuss the results on the effect of I-group impurities diffusion on the photoconductivity and luminescence spectra of CdS films, namely the appearance of photoconductivity at  $\lambda=550 \text{ nm}$  and green emission band under diffusion doping by lithium, sodium, and the absence of these bands under the diffusion doping by copper and silver. At first we consider the influence of electron-hole equilibrium on concentration of charged vacancies of cadmium and sulphur in CdS. It is known that because of interaction between the charged impurities and structural defects, the presence of donors must increase the solubility of acceptors and vice versa [10]. The cadmium and sulphur vacancies in CdS can be ionized by quasi-chemical reactions



where,  $e$  is electron.

Applying the mass action law to the reactions (4) and (5), one can show

$$N(V_{cd}^-) = K_1 N(V_{cd}) n \quad (6)$$

$$N(V_s^+) = K_2 N(V_s) p / n_i^2 \quad (7)$$

where,  $K_1$  and  $K_2$  are the constants of reactions (4) and (5),  $n$  and  $p$  are the electron and hole concentrations,  $n_i$  is the intrinsic charge carriers concentrations. From the relations (6) and (7) it follows that, the concentration of negatively charged cadmium vacancies  $N(V_{cd}^-)$  linearly

increases by concentration of electrons, and the concentration of positively charged sulphur vacancies  $N(V_s^+)$  also linearly increases by increasing the holes concentration. In other words, the doping by donor impurities must increase the concentration of negatively charged cadmium vacancies, but doping by acceptor impurities must increase the concentration of positively charged sulphur vacancies, leading consequently, (according to (2)), to decrease of  $N(V_{cd}^-)$ .

Assuming that lithium and sodium impurities are situated preferentially in interstitials as ionized donors, and the copper and silver impurities in cadmium sublattice as ionized acceptors, then the observed experimental results are well explained on the basis of the considered model above. The diffusion doping of CdS films by the donors (lithium, sodium) increases the concentration of cadmium vacancies  $N(V_{cd}^-)$ , resulting in an increase of photoconductivity at  $\lambda=550 \text{ nm}$  and green emission. The diffusion of acceptor impurities (copper and silver) decreases the cadmium vacancies concentration  $N(V_{cd}^-)$  and therefore, leads to the disappearance of the photoconductivity at  $\lambda=550 \text{ nm}$  and green emission.

The absence of photoconductivity at  $\lambda=550 \text{ nm}$  and green emission in CdS films doped by sodium during the deposition process (by addition of NaCl to the solution) may be due to the precipitation of sodium impurity (as well as copper impurity) in intergrain spaces of CdS films. The presence of sodium and copper impurities in intergrain spaces must increase the recombination barrier (because of zone distortions near the grains surface), which makes difficult for electron capture by s-centers (rapid recombination centres), localized near the boundaries of crystallites [21, 22]. Such a blockading of recombination channels through the s-centers must increase the intensity of present bands of photoconductivity and luminescence of the samples, which are observed in our experiments (Fig. 4, 5). The considerable decreasing of resistivities of CdS films doped during the deposition process (see table 1) can also be a confirmation for the

assumption of precipitation of Na and Cu impurities in intergrain spaces.

The appearance of photoconductivity line  $\lambda=550$  nm and green emission bands in films doped by sodium and the absence of these lines in copper doped films (during deposition process) after annealing in sulphur vapour are due to diffusion of these impurities from intergrain spaces into the crystallites. In this case, the process is analogous to diffusion doping of CdS films by sodium and copper impurities, which was discussed above.

#### 4. Conclusions

The photoconductivity line with maximum at  $\lambda=550$  nm and green band of luminescence in CdS films are connected with negatively charged vacancies of cadmium sublattice. The intensities of these bands can be controlled

by changing the partial pressure of sulphur vapour during thermal annealing.

The observed increasing of intensities of photoconductivity line at  $\lambda=550$  nm and green emission band at  $\lambda=510-550$  nm under lithium, sodium diffusion, and decreasing of these bands under copper, silver diffusion are due to donor-acceptor interaction of introduced I-group impurities with charged cadmium vacancies.

The absence of photoconductivity at  $\lambda=550$  nm and green emission in CdS films, doped by sodium during deposition process (by addition of NaCl to the solution) is attributed to the precipitation of sodium impurities (as well as copper impurities) in intergrain spaces, which blockade the recombination channels through the s-centers, localized near the grain boundaries, leading to an increase of the intensity of present photoconductivity and luminescence bands in CdS films.

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### 1-GRUP AŞQARLARININ CdS TƏBƏQƏLİRİNİN XASSƏLƏRİNƏ TƏSİRİ

1-grup elementlərinin (litium, natrium, mis və gümüş) kimyəvi pulverizasiya üsulu ilə alınmış CdS nazik təbəqələrinin elektrik, fotoelektrik və luminesent xassələrinə təsiri tədqiq olunmuşdur. Litium və natrium aşqarlarının diffuziyası zamanı ( $\lambda=550$  nm) fotoelektrik və yaşı emissiya ( $\lambda=510-540$  nm) zolaqlarının intensivliyinin artması, həm də onların mis və gümüş diffuziyası zamanı azalması 1-grup elementlərinin məufi yüksəlmiş kadmiyum vakansiyalarının qarşılıq təsiri ilə izah olunur.

A.И. Байрамов

### ВЛИЯНИЕ ПРИМЕСЕЙ 1-ГРУППЫ НА СВОЙСТВА ПЛЕНОК CdS

Исследовано влияние примесей 1-группы (литий, натрий, медь и серебро) на электрические, фотоэлектрические и люминесцентные свойства пленок CdS, полученных методом химической pulverизации. Увеличение интенсивности полос фотопроводности при  $\lambda=510-540$  nm при диффузионном легировании CdS литием и натрием, уменьшение их при легировании медью и серебром объясняется донорно-акцепторным взаимодействием примесей 1-группы с отрицательно заряженными вакансиями кадмия.

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