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PREPARATION OF SOME II-VI SEMICONDUCTOR FILMS BY THERMAL OR ELECTRON BEAM EVAPORATION AND BY HOT WALL EPITAXY AND THEIR OPTICAL CHARACTERIZATION

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Thin films of CdS and related materials were grown by thermal evaporation, by electron beam evaporation and by hot wall epitaxy onto various substrates. In the case of evaporation it was necessary to anneal the films after growth, in contrast to the epitaxy. Simultaneous evaporation of two semiconductor materials leads to ternary films with an absorption edge depending on the composition. Alternating evaporation of two materials leads to multilayer heterostructures demonstrating new effects. Absorption and luminescence measurements have been used for optical characterization. Some non-linear optical measurements are also presented concerning mainly photothermally induced optical bistability.

1. INTRODUCTION

In the last five decades the properties of bulk semiconductor materials have been investigated and understood in great detail. The interest has now turned to thin films. In the case of III-V semiconductors with reduced dimensionality such as multiple quantum wells (MQWs) and superlattices, many exciting results and new effects have been found. For a recent review see for example ref. 1 and references therein.

With some delay a similar development started in the field of II-VI₂ semiconductors. New results are presented in refs. 2 and 3. Here we give a review mainly of our results concerning CdS films and related materials.

Thin films of semiconductors can be produced very easily by thermal or electron beam evaporation. Their disadvantage compared with single crystals or epitaxial layers is their poor crystallinity. The best result for this technique is usually a columnar structure of the film oriented perpendicularly to the substrate⁴.

In this paper we present our experience with some CdS-related evaporated II-VI films and compare it with the first results of hot wall epitaxy (HWE). Optimal substrate temperatures and post-deposition annealing lead to an improved crystalline structure and steepness of the absorption edge.

Using two separately controllable sources it is possible to produce films of mixed crystals. In the case for example of $\text{Cd}_{1-x}\text{Zn}_x\text{S}$ the absorption edge can be shifted to the desired wavelength in the range from pure CdS to pure ZnS.

Another use of this set-up is to evaporate alternately materials with different band edges leading to heterostructures. In the case of sufficient quality interesting effects are expected as we know for example from MWQs grown by molecular beam epitaxy (MBE).

Non-linear effects due to thermally induced optical bistability are also presented for different materials and exciting wavelengths.

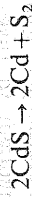
2. PREPARATION

2.1. Thermal and electron beam evaporation

As substrates we used microscope glasses. These were cut to the desired size of $15 \text{ mm} \times 15 \text{ mm}$. After being cleaned in acetone in an ultrasonic bath for 5 min they were rinsed in distilled water and cleaned again in ethanol. Then the substrates were rinsed again in water and dried in a stream of pure nitrogen. The cleaned substrates were fixed onto the holder in the vacuum chamber and heated to 670 K for 1 h to desorb surface impurities. After cooling down to the desired temperature the growth started. As source material we used CdS and ZnS tablets from Balzers with a purity of 99.999% and 99.99% respectively. Deposition rate and substrate temperature were controlled by a quartz oscillator and a thermocouple respectively. In the case of thermal evaporation from a molybdenum crucible it was possible to choose continuously the evaporation rate from zero to the desired value by changing the source current. Electron beam evaporation did not have this advantage. It was not possible to regulate the beam current to achieve a stable evaporation rate. One reason might be the low conductivity of the semiconductor material that leads to charging effects. Small charged particles of the target flow away. Another disadvantage is that only one thin film of about $1 \mu\text{m}$ thickness can be prepared without breaking the vacuum because the position of the electron beam on the target tablet could not be changed. In contrast, thermal evaporation enabled the preparation of about six samples by simply changing a shutter position.

The vacuum chamber was pumped by an oil diffusion pump fitted with a liquid N_2 cooled foreline trap to avoid oil backstreaming into the chamber. Typical pressures during evaporation were in the range 10^{-5} – 10^{-6} mbar.

From earlier examinations⁵ it is well known that CdS dissociates during evaporation according to



As a result cadmium and S_2 have to recombine at the substrate in a certain ratio to create stoichiometric CdS. In reality this does not take place completely and evaporated films have sulphur vacancies^{6,7}.

2.2. Epitaxial layers

The epitaxial layers were grown by HWE. An ultrahigh vacuum chamber with a background pressure of 5×10^{-9} mbar was fitted with hot wall unit. The

temperatures of the source pump was used to minimize surface contaminations performed in the loading, was heated to 1000 K. The for the wall and 500–750 was fixed onto the orifice $\text{BaF}_2(111)$ and $\text{SrF}_2(111)$ cleaned previously with water. The polished surface CdS source materials was contamination of less than

3. RESULTS OF GROWTH

3.1. Thermal evaporation 3.1.1. CdS films

Films evaporated or brown to black for them a poor crystallinity and absorption. At low temperature that of sulphur, resulting increasing substrate temperature finally to yellow for T_{sub} impractically low value constituents.

As a consequence vacuum chamber which improves crystallinity and structures changed to yellow. Avoided in vacuum. For temperature of 450–500 obtained films with optical of the equivalent bulk material.

In an X-ray diffractometer peaks can be observed. The of the hexagonal CdS lattice structure factor of the $\{h$ CdS is always zero⁸. In the than ten peaks from different a result the growth axis is substrate. This fact is in a different substrate temperature scanning electron microscope columnar structure with di

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 possible to produce films of the absorption edge can be CdS to pure ZnS. Specially materials with different sufficient quality interesting is grown by molecular beam optical bistability are also

ere cut to the desired size of trasonic bath for 5 min they thanol. Then the substrates pure nitrogen. The cleaned chamber and heated to 670 K to the desired temperature and ZnS tablets from Balzers position rate and substrate a thermocouple respectively. n-crucible it was possible to he desired value by changing have this advantage. It was stable evaporation rate. One uctor material that leads to arget flow away. Another i thickness can be prepared e electron beam on the target l evaporation enabled the shutter position.

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high vacuum chamber with d with hot wall unit. The

temperatures of the source, wall and substrate could be separately chosen up to 1300 K with an accuracy of 0.1 K. A loading module evacuated by a turbomolecular pump was used to minimize contamination of the growth chamber. Desorption of surface contaminations from the substrates by heating them up to 1200 K was also performed in the loading module. Before the growth was started the hot wall unit was heated to 1000 K. Typical growth parameters were 850 K for the source, 900 K for the wall and 500–750 K for the substrate. During growth the substrate holder was fixed onto the orifice of the cell, which leads to a closed system. Freshly cleaved $\text{BaF}_2(111)$ and $\text{SrF}_2(111)$ and sapphire were used as substrates. The latter had been cleaned previously with degreasing detergent, acetone, methanol and deionized water. The polished surface of the sapphire was perpendicular to its optical axis. The CdS source materials was very pure powder (Puratronic) with a guaranteed metallic contamination of less than 20 ppm.

3. RESULTS OF GROWTH AND CHARACTERIZATION BY ABSORPTION SPECTROSCOPY

3.1. Thermal evaporation

3.1.1. CdS films

Films evaporated on a substrate below 300 K without annealing were dark brown to black for thermal and electron beam techniques. This can be explained by a poor crystallinity and a non-stoichiometry of the film which lead to a high absorption. At low temperatures the sticking coefficient of cadmium is higher than that of sulphur, resulting in a high density of sulphur vacancies in the film. With increasing substrate temperature the colour changes from black to brown and finally to yellow for $T_{\text{sub}} \geq 450$ K. Above 500 K the deposition rate decreases to impractically low values owing to decreasing sticking coefficients of both constituents.

As a consequence we used post-deposition annealing at 730 K for 1 h in the vacuum chamber which increases the steepness of the absorption edge and thus improves crystallinity and stoichiometry. Even brown films grown at low temperatures changed to yellow. Annealing in air is also possible but surface reactions are avoided in vacuum. For this reason we evaporated the films at a substrate temperature of 450–500 K and annealed them after deposition. In this case we obtained films with optical and crystallographic properties almost similar to those of the equivalent bulk material.

In an X-ray diffraction pattern of a thermally evaporated CdS film only two peaks can be observed. They can be identified with the (002) and the (004) reflections of the hexagonal CdS lattice. A (001) or (003) reflection is not observed because the structure factor of the $\{h(2l+1)\}$ crystallographic orientation in the hexagonal CdS is always zero⁸. In the case of unoriented CdS layers one would expect more than ten peaks from different Miller indices owing to their statistical orientation. As a result the growth axis is identical with the optical c axis and is perpendicular to the substrate. This fact is in agreement with other publications and was observed for different substrate temperatures, evaporation rates and thicknesses of the films⁹. In a scanning electron microscopy picture we observe a very flat surface structure and a columnar structure with diameters less than 0.1 μm in the cleaved edge. The width of

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3.1.3. CdS–ZnS multilayers

By evaporating alternating films of CdS and ZnS we produced a heterostructure of the two semiconductor materials with different band gaps.

The equipment was the following: two separately controllable sources filled with CdS and ZnS were heated simultaneously. By turning a shutter from one source to the other it was possible to produce alternating films of CdS and ZnS. The deposition rate was measured by a quartz oscillator. After a programmed thickness the shutter was automatically changed by an electromagnet.

Because of the periodical band gap modulation over small distances (about 100 Å single film thickness) it should be possible to observe quantum effects for the energy of electrons and holes in the quasi-two-dimensional system. Ideally we would observe a blue shift of the fundamental edge and a stepwise absorption structure owing to the higher subbands and excitons. Experiments with MBE-grown III–V superlattices and MQWs have already demonstrated this behaviour¹². First results for HWE-grown ZnSe and ZnTe superlattices are given in ref. 13.

Figure 2 shows the absorption spectra at 4.2 K of a $30 \times (100 \text{ Å CdS} - 150 \text{ Å ZnS})$ multilayer, compared with that of a CdS single film with an equivalent thickness of 0.3 μm. We observe modulations in transmittance due to Fabry–Pérot interferences of the multilayer surface. This fact makes it impossible to distinguish clearly modes and steps of absorption. A rough estimation of the expected blue shift of the band edge of the multiheterostructure as compared with CdS taken at a transmission $T \approx 0.3$ leads to a value of about 15 meV or 3.1 nm. This value is obtained from a simple potential well model on assuming that the band gap discontinuity of CdS and ZnS is completely in the conduction band. In the evaporated sample we observe a shift of more than 40 nm which is ten times the expected value. This finding can only be explained by a mixing of cadmium and zinc in the interface. This results in a blue-shifted absorption according to $\text{Cd}_{1-x}\text{Zn}_x\text{S}$. A homogeneous zinc content of about 24% in the CdS layers explains this shift. This result cannot be a result of diffusion during or after growth, because the substrate temperature was only 370 K and no annealing was used for the heterostructures. To verify this statement, we prepared a two-layer system 0.5 μm ZnS/0.5 μm CdS. Figure 3 shows the absorption edge of this sample for different annealing temperatures during a period of 1 h. It does not change up to 690 K, which indicates

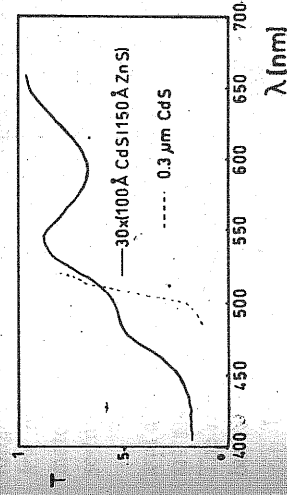


Fig. 2. Transmission spectra of a CdS/ZnS multilayer compared with that of a CdS single film with the equivalent thickness.

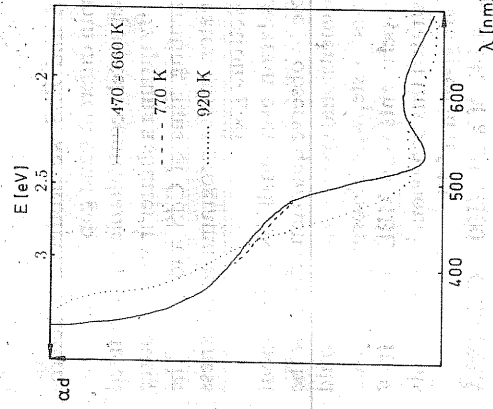


Fig. 3. Absorption of a two-layer system CdS/ZnS after different annealing temperatures. Full scale, about 3.

that no significant diffusion takes place. Only at 950 K do we observe a blue shift of absorption which can be explained by interdiffusion and formation of $\text{Cd}_{1-x}\text{Zn}_x\text{S}$. Therefore, the mixing in the heterostructures must be a consequence of imperfect shutter operation. A sputter depth profile was recorded by Auger electron spectroscopy. 4 keV argon ions with a current density of $5 \mu\text{A cm}^{-2}$ were used for the sputtering process. 3 keV electrons were used for excitation. The chemical composition was calculated from the peak heights of cadmium, zinc and sulphur in the differentiated Auger electron energy spectrum. Figure 4 shows the chemical composition of the film as a function of sputter time. For a constant sputter rate as in

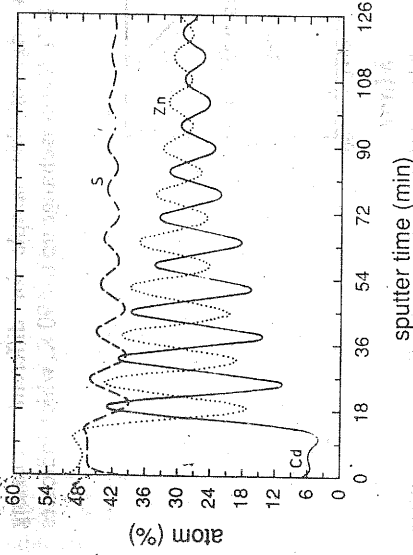


Fig. 4. Depth profile of a CdS/ZnS multilayer.

our case this time concentration with already in the upper modulation degree and to an inhomogeneity can be explained by

3.2. Electron beam evaporation. Films grown by thermally evaporated CdS. They changed to brown at 450 K.

3.3. Hot wall epitaxy. Layers prepared by thermal evaporation carried out. The growth rates might be between 150 nm on sputter and 100 nm on growth rates might mismatches of the substrate. The value of the film, about 12% in the (000) surface of a bulk material. The surfaces of grains or hexagonal plates.

4. OPTICAL CHARACTERISTICS

The luminescence of the CdS/ZnS multilayer was measured with a mercury lamp. The emission spectrum was recorded with a cryostat. Figures 5 and 6 show the emission spectra prepared by thermal evaporation and hot wall epitaxy. They show a broad band in the blue region with a small intensity at the excitation wavelength of the excitons I_1 (2.525 eV); a few millielectronvolts longitudinal optical phonon energy is added to the material, which is usually observed in CdS (e.g. sodium etc.)^{10,14}

In transmission measurements, the absorption coefficient is about 2.63 eV when the wavelength is about 450 nm. The reason for this shift between film and substrate is the interdiffusion. The splittings Δ_{AB} between the bands are in agreement with the values of the evaporated films.

our case this time can be related to the depth. We see a clear modulation of concentration with the depth. However, it can also be seen that there is a mixing already in the uppermost layers that leads to the shifted edge. The decrease in the modulation degree with depth is due to a mixing of the film by the sputtering ions and to an inhomogeneous erosion. The modulation of the sulphur concentration can be explained by slightly different sensitivity factors of sulphur in CdS and ZnS.

3.2. Electron beam evaporation

Films grown by electron beam evaporation exhibited the same behaviour as the thermally evaporated films. At low substrate temperatures the films were black. They changed to brown and finally to yellow for substrate temperatures above 450 K.

3.3. Hot wall epitaxy

Layers prepared by HWE were homogeneously yellow. No annealing was carried out. The growth duration was 1 h and the thickness of the films varied between 150 nm on sapphire, 0.6 μm on BaF₂ and 0.8 μm on SrF₂. The different growth rates might be due to different sticking coefficients and to the lattice mismatches of the substrates. In the case of SrF₂, where we obtained the thickest film, the value of the mismatch is the lowest, almost 1%. Al₂O₃ has a mismatch of about 12% in the (0001) plane and BaF₂ of about 6%.

The surfaces of all samples were shiny as they appear for a good, as-grown surface of a bulk material. A light microscope revealed a very smooth surface. No grains or hexagonal pyramids could be observed.

4. OPTICAL CHARACTERIZATION OF THE FILMS

The luminescence measurements were performed using the UV lines of a mercury lamp. The samples were kept at liquid helium temperature in a bath cryostat. Figures 5-7 show the luminescence and transmission of CdS films prepared by thermal and electron beam evaporation and by HWE. Figures 5 and 6 show a broad band in the range of the donor-acceptor pair luminescence and only a small intensity at the bound exciton region. The epitaxial layer exhibits bound excitons I₁ (2.525 eV) and donor-acceptor pair luminescence at 2.40 eV with their longitudinal optical phonon replicas separated by 35 meV. The I₁ energy is shifted a few millielectronvolts towards lower energy compared with the I₁ line in bulk material, which is usually attributed to excitons bound to neutral acceptors (lithium, sodium etc.)^{10,14}

In transmission we can see small dips in absorption at about 2.549 eV, 2.565 eV and about 2.63 eV which we attribute to the free excitons A Γ_5 , B Γ_5 and C Γ_5 . They are also shifted to lower energies by about 5 meV compared with bulk material¹⁰. The reason for this shift is the strain in the CdS film introduced by the interface between film and substrate.

The splittings Δ_{AB} and Δ_{BC} are approximately 16 meV and 65 meV respectively. These values are in agreement with the splittings in bulk material¹⁰.

The evaporated films do not exhibit such a structure. The modulations in the-

annealing temperatures. Full scale,

to we observe a blue shift of formation of Cd_{1-x}Zn_xS. a consequence of imperfect sorded by Auger electron of 5 $\mu\text{A cm}^{-2}$ were used for r excitation. The chemical dmium, zinc and sulphur in gure 4 shows the chemical a constant sputter rate as in

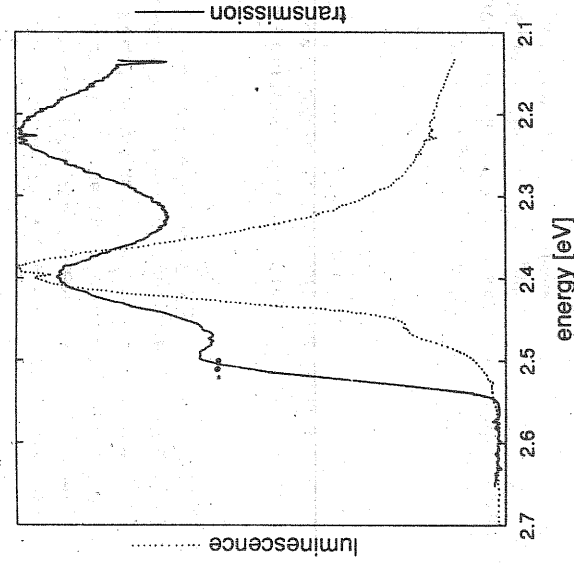


Fig. 5. Transmission and luminescence spectra of a CdS film prepared by thermal evaporation.

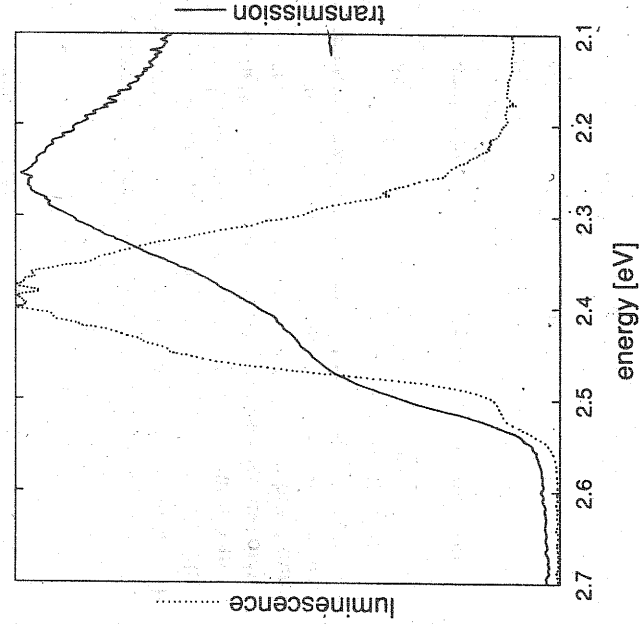


Fig. 6. Transmission and luminescence spectra of a CdS film prepared by electron beam evaporation.

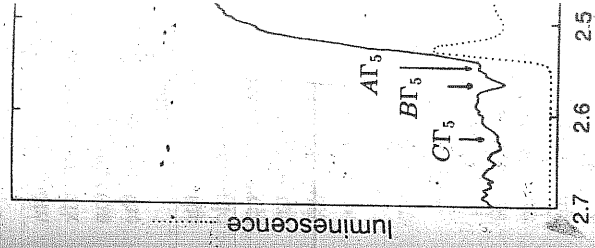


Fig. 7. Transmission and luminescence spectra of a CdS film prepared by thermal evaporation.

transparent region are transparent. The transparent layer does not display the typical structure compared with other II-VI semiconductors.

As a result we can see that the luminescence is well resolved and originates from the three uppermost layers.

5. NON-LINEAR OPTICS

5.1. Photothermally induced non-linear optics

It is well known that II-VI semiconductors can be used as non-linear optical materials.

This means that the refractive index increases exponentially with the intensity of the light. This is strongly excited with light and heats the material. This leads to an increasing absorption coefficient. This feedback is strong enough to cause a self-absorption. This means that the absorption for the same wavelength is the same as in the previous history. If the film is excited without any hysteresis is observed.

y thermal evaporation.

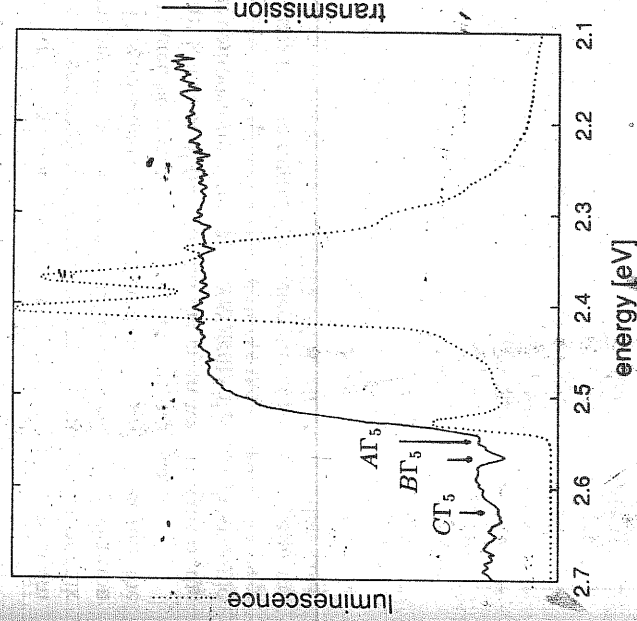


Fig. 7. Transmission and luminescence spectra of a CdS epitaxial layer prepared by HWE on BaF₂.

transparent region are due to Fabry-Pérot interferences in the layer. The epitaxial layer does not display this effect because it is too thin.

As a result we can conclude that the epitaxial layer has an improved crystalline structure compared with the evaporated films. The I₁ and the donor-acceptor pair luminescence is well resolved and in transmission we can see the three Γ₅ excitons from the three uppermost valence bands.

5. NON-LINEAR OPTICS

5.1. Photothermally induced optical bistability

It is well known from earlier publications that the absorption edge in semiconductors can be described by the Urbach-Martienssen rule¹⁵⁻¹⁹

This means that the absorption coefficient below the lowest free exciton increases exponentially when the temperature increases. If such a semiconductor is strongly excited with light in the tail of the absorption band part of the light is absorbed and heats the sample by non-radiative recombination. This leads in turn to an increasing absorption and again in a higher temperature. If this non-linear feedback is strong enough, thermally induced optical bistability (OB) can be observed. This means that a stationary hysteresis exists with two states of different absorption for the same input light intensity. The present state depends on the previous history. If the feedback is not strong enough only a strong non-linearity without any hysteresis is observed. The latter is the case for pure CdS films and bulk

y electron beam evaporation.

material for the 514.5 nm Ar⁺ laser line and the polarization $E \perp c$ at room temperature because the initial absorption is already too high²⁰. In the case of bulk material it is possible to change the polarization to $E \parallel c$. This reduces absorption and OB can be observed. This possibility does not exist for thin films because the crystallographic c axis is oriented perpendicular to the substrate surface.

By adapting the absorption edge of the films to the given wavelengths of the Ar⁺ laser by an appropriate composition of the ternary system Cd_{1-x}Zn_xS we could observe OB for two different wavelengths²¹. Figure 8 shows the hysteresis loops of two different Cd_{1-x}Zn_xS films for two different wavelengths at room temperature. One other possibility is to cool the film ($T \leq 250$ K)²² or to use another laser wavelength²³.

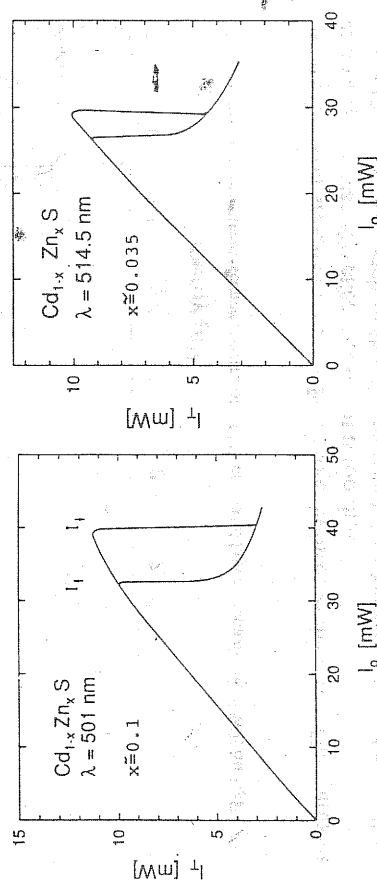


Fig. 8. Hysteresis loop of two different Cd_{1-x}Zn_xS films for two different wavelengths at room temperature.

5.2. Opto-electronic properties

The temperature in the laser spot can also be influenced via Joule heat production through dark current and photocurrent by an electric field. This was possible by evaporation of two metal contacts with a distance of about 100 μ m. This device connects the optical with the electrical properties of the sample and is called a photothermal self-electro-optic effect device (SEED).

If we apply a constant light input intensity and an a.c. voltage with variable frequency we observe a modulated transmitted light intensity. At low frequencies the modulation is nearly constant and decreases with a $1/f$ law for higher frequencies. The -3 dB frequency is about 100 Hz owing to the long thermal time constants. At 10^3 Hz the modulation reaches a minimum and increases again. In this region the probe temperature can no longer follow the modulation and the varying input intensity is simply measured in the transmitted signal. Consequently the phase delay is π at low frequencies and decreases continuously to zero at higher frequencies. More detailed information about OB in evaporated films and SEEDs can be found in refs. 24 and 25.

II-VI SEMICONDUCT

6. SUMMARY

We have shown behaviour to bulk in occurrence of OB. A broad luminescence of the epitaxial layer luminescence of bo acceptor pair transi

7. ACKNOWLEDGME

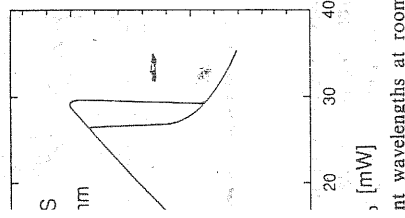
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6. SUMMARY

We have shown that it is possible to grow II-VI films with very similar behaviour to bulk material. In the case of evaporated films this fact can be seen in the occurrence of OB. A disadvantage is their lack of crystallinity. As a result we see only a broad luminescence peak due to donor-acceptor pair recombinations. In the case of the epitaxial layer we achieved an improved crystallinity which leads to the luminescence of bound excitons and well-resolved phonon replicas of the donor-acceptor pair transitions.

7. ACKNOWLEDGMENTS

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SILICIDE FOR AND Mo-Co F

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The solid sta
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1. INTRODUCTION

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* Presented at the 17th
Conference on Thin Film
† Present address: India

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