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The growth of cubic CdS on InP(110) studied *in situ* by Raman spectroscopy

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CdS was deposited onto clean cleaved InP(110) by molecular beam epitaxy (MBE) using a growth rate of 0.2 monolayers/min and a substrate temperature of 440 K (510 K). Raman spectra were taken *in situ* of the clean InP surface and after each evaporation step using an Ar^+ ion laser as a light source. Due to this resonant excitation scattering signals originating from the CdS deposition are observed at coverages as low as 2 monolayers (ML). The number of phonon peaks observed and their selection rules reveal that the cubic modification is present. The spectra are dominated at all coverages by the longitudinal optical (LO) and 2LO phonon scattering intensities and the variation of the 2LO/LO intensity ratio with CdS deposition indicates changes in the electronic structure of the growing CdS. Another spectral feature in the Raman spectra is attributed to a chemically reacted layer at the interface most likely consisting of an In–S compound. The intensity of this feature is found to depend critically on the growth parameters, in particular the substrate temperature, but also on the operating time of the MBE cell. The amount of reaction at the interface also influences the critical CdS film thickness and the development of the 2LO/LO ratio. The results are discussed taking complementary photoluminescence, x-ray diffraction, and photoemission data into account.

I. INTRODUCTION

Raman spectroscopy is often applied as a standard post-growth characterization tool for heteroepitaxial layers. Scattering by the phonon vibrational modes is commonly used to determine the crystal quality and/or the strain in the epitaxial layer. Examples of such application on II–VI/III–V heterostructures, in particular ZnSe/GaAs, are given in Refs. 1–4. We have also applied such post-growth analysis with a view to studying the formation of interfacial reactions and their identification, e.g., in the CdTe/InSb and the ZnSe/GaAs systems.[5,6] Very often the scattering intensity of interfacial compounds is quite weak because of the small scattering volume. Therefore, it is extremely difficult to investigate interfacial properties after the growth of typically micron thick epitaxial layers which screen any interfacial effects. Consequently it would be most advantageous to monitor the growth *in situ* from the initial stages, beginning with the clean substrate surface. Such approach has been successfully applied to study the growth of the semimetals Sb and Bi on the cleavage faces of the III–V semiconductors InP and GaAs.[7,8] It has also been used to study semiconductor heterostructure growth in the case of Ge on GaAs.[9] In this article we describe the first *in situ* Raman results for the epitaxial growth of a II–VI compound on a III–V substrate, namely CdS on InP(110). It will be shown that Raman scattering is a highly sensitive probe to study this closely lattice matched heterostructure system.

II. EXPERIMENTAL

The *in situ* Raman experiment was performed using an ultrahigh vacuum (UHV) vessel integrated in the optical set up of the Raman experiment. The InP crystals supplied by MCP Ltd. were mounted on a sample holder equipped with heating and cooling facilities. The sample temperature was monitored by a thermocouple adjacent to the sample to be 440 K (510 K) and 100 K during the evaporation and the measurements, respectively. Atomically clean InP(110) surfaces were prepared by cleaving the crystals in UHV at a base pressure of $p < 2\times10^{-10}$ mbar using a double notch-double wedge technique. CdS was deposited by molecular beam epitaxy (MBE) using a liquid nitrogen cooled Knudsen cell containing polycrystalline CdS powder supplied by Johnson–Matthey (5N purity). The cell was temperature controlled and kept at a constant temperature of 820 K which leads to a nominal deposition rate of 0.2 ML/min at the sample position. The rate was calibrated using the inten-

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sity attenuation of Auger and photoemission signals. Before deposition the cell was thoroughly outgassed. CdS was deposited in steps and Raman spectra were taken after each evaporation in monolayer intervals at low coverages and larger intervals up to about 150 ML.

Several emission lines of an Ar^+ ion laser at 514.5 nm (2.41 eV), 501.7 nm (2.47 eV), and 457.9 nm (2.71 eV) were used for the *in situ* Raman experiment. The laser beam with maximum power of 20 mW was focused onto the sample to about 100 μm in diameter fulfilling a quasibackscattering geometry.

Two different scattering configurations were chosen for the experiment. First the incident and scattered light were both polarised along the ⟨001⟩ directions of the InP substrate, and secondly the polarization of the incident light was rotated by 90° so that the polarization vector of the incident light was parallel with the ⟨1–10⟩ direction of the sample. By leaving the polarization of the scattered light constant the sensitivity of the monochromator is constant for both configurations which permits straightforward intensity comparisons. In the former configuration, called from now on "parallel," selection rules based on the crystal symmetry of zincblende materials does not allow scattering from either the transverse (TO) nor the longitudinal (LO) optical phonons. However, when the Raman transition susceptibility is expanded in terms of a finite phonon wave vector **q** or a static electric field **E** LO phonon scattering becomes allowed in this configuration. In CdS this contribution to the LO scattering is dominant at excitation near resonance due to the strong Fröhlich electron–phonon interaction. In the latter "crossed" configuration TO phonon scattering is allowed by the symmetry selection rules, while LO phonon scattering is symmetry as well as Fröhlich forbidden.

The scattered light was focused onto the entrance slit of a Cary double monochromator. The slit widths (400/600/400 μm) were adjusted for maximum signal intensity at ultralow coverages leading to a resolution of 4 cm^{-1} at a laser wavelength of 457.9 nm. The scattered light was detected by a GaAs photomultiplier.

III. RESULTS AND DISCUSSION

A. The growth of the cubic CdS modification on InP(110)

In this Raman experiment light scattering by the vibrational modes of CdS are used to characterise the structural properties of the epitaxial layers. The crystal symmetry and the scattering configuration used impose selection rules on the number of modes contributing to the scattering intensities. For instance the InP substrate has the cubic symmetry T_d. This zinc-blende structure offers a longitudinal optical (LO) and a doubly degenerate transverse optical (TO) phonon at wavevector $\mathbf{k} \approx 0$ for the Raman characterization. From the InP(110) face scattering is only symmetry allowed by the TO phonon in the crossed configuration.

The II–VI compound semiconductors very often crystallise in the hexagonal wurtzite as well as the cubic zinc-blende structure. For CdS the wurtzite one is preferred in nature and the cubic is metastable. In a simple picture taking into account that the real space periodicity in the ⟨111⟩ direction of the hexagonal modification is approximately twice that of the cubic modification, the wurtzite phonon dispersion can be obtained from the zinc-blende one by folding. This increases the number of phonon vibrational modes at the Brillioun zone center observable in a Raman experiment. The frequencies of the cubic vibrational modes nearly conincide with hexagonal ones and cannot be used for the purpose of distinguishing the two modifications. However, the number of modes observed should give clear indications on the matter. In the case of wurtzite CdS there are six observable Raman active modes, i.e., E_2 at 43 cm^{-1}, A_1(TO) at 234 cm^{-1}, E_2 at 256 cm^{-1}, E_1(TO) at 243 cm^{-1}, A_1(LO) at 305 cm^{-1}, and E_1(LO) at 307 cm^{-1}.[10] The TO and LO modes of the cubic modification concur with the E_1(TO) and the E_1(LO) of the hexagonal modification.

Two sets of Raman spectra taken in the crossed and the parallel configurations at a laser wavelength of 457.9 nm are shown in Fig. 1. The spectral regions shown cover the range of the CdS vibrational modes (except the E_2 at 40 cm^{-1}) and the first harmonic of the E_1(LO). These spectra are typical for a nominal deposition of 20 ML CdS onto InP(110) at $T_{sub} = 440$ K. The dominant peaks in the spectra are clearly at the LO and 2LO frequency position at 305 and 610 cm^{-1}, respectively. In addition there are a few noticeable differences between the two configurations. First, the LO and 2LO scattering intensities are found to be about three times stronger in parallel configuration. This is in agreement with the Fröhlich selection rules. Furthermore,

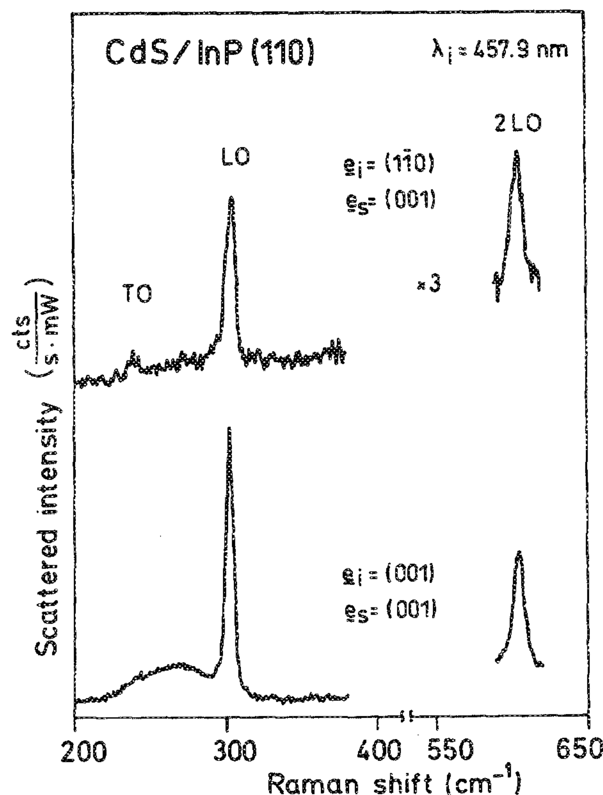

FIG. 1. Raman spectra of a CdS/InP(110) sample after a nominal deposition of 20 ML at $T_{sub} = 440$ K.

the peak at the LO(CdS) phonon position in the crossed configuration is somewhat asymmetric because the symmetry allowed TO(InP) phonon gives rise to scattering at almost the same position. Also in the same spectrum a tiny peak is observed at the TO(CdS) phonon position of 246 cm^{-1}. It obeys the selection rules for a zincblende type CdS TO phonon. Further peaks which could be expected in the case of hexagonal CdS were not detected. A comparison of the TO(CdS) and LO(CdS) scattering intensities indicates that deformation potential scattering for the TO phonon is much weaker than the Fröhlich scattering for the LO. This effect is well known since the Fröhlich interaction in CdS becomes very strong due to the excitonic coupling.[11] Finally a broad feature on the low frequency side of the LO phonon peaking around 260 cm^{-1} is only observed in the parallel configuration. Its origin will be discussed in Sec. III C.

The number of phonon modes observed and their selection rules clearly indicate that CdS grows in the cubic modification. Further evidence is given by the double crystal x-ray rocking curves (DCXRC) in Fig. 2 of a 200 ML thick CdS sample on InP(110). In the lower spectrum a reflection originating from the CdS layer is clearly observable at 24.05° near the strong (022) reflection of the InP substrate. It may, however, be assigned to either the (022) reflection of zincblende CdS or the (011) reflection of the wurtzite CdS. In the latter case one would also expect to observe the (022) reflection at an angle of 29.2°. Since this is not the case as can be seen from the upper spectrum in Fig. 2, the latter case can be excluded. Further evidence is also given by the previous low-energy electron diffraction (LEED) experiment[12] and it thus can be concluded that CdS grows in the cubic modification on InP(110).

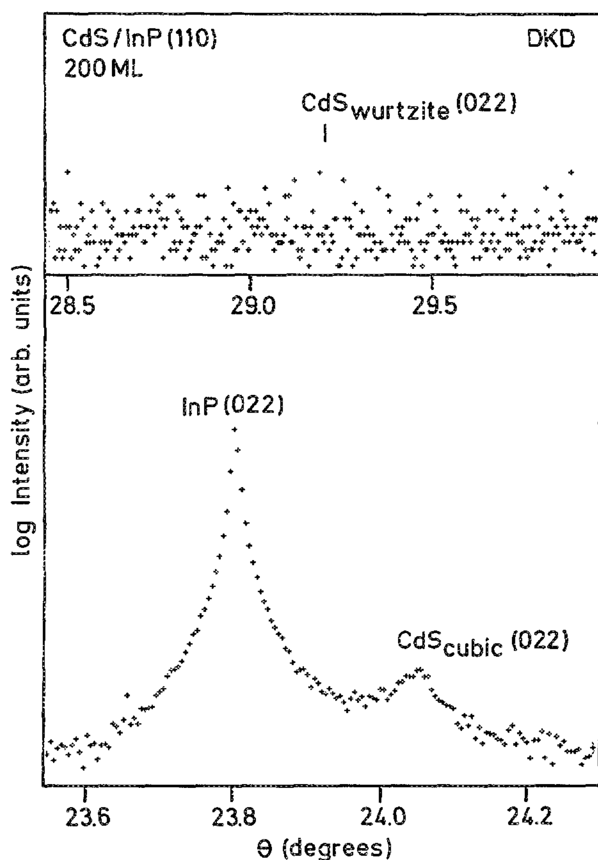

FIG. 2. DCXRC spectra of a 150 ML thick CdS overlayer on InP(110) at two different angular positions.

B. Resonant Raman scattering

The Ar^+ ion laser lines used overlap the band gap of bulk CdS. It is thus expected that these lines are suitable to lead to an resonant enhancement of the scattering signals from the CdS overlayer. Preliminary resonance experiments were carried out and the Raman spectra taken in the parallel configuration at three different wavelengths are shown in Fig. 3. The upmost spectrum is the same as that of Fig. 1. The middle spectrum reveals that at 501.7 nm the strongest signals are obtained. However, it can also be seen that a background increasing towards higher wavenumbers exists. This is caused by the photoluminescence in the CdS layer. Both the strong Raman signal and the luminescence background demonstrate that this laser line is closest to resonance. For the Raman experiment, however, it is desirable to avoid the luminescence background in order to detect weak features in the Raman spectra. The background can be circumvented by using a excitation below the energy gap as shown in the lower spectrum. In this case, on the other hand, also the scattering intensity centered around 260 cm^{-1} is much weaker compared to that observed for the excitation with 457.9 nm. Since the identification of this feature was one of the prime goals of the work, the 457.9 nm line was therefore used for the following growth studies.

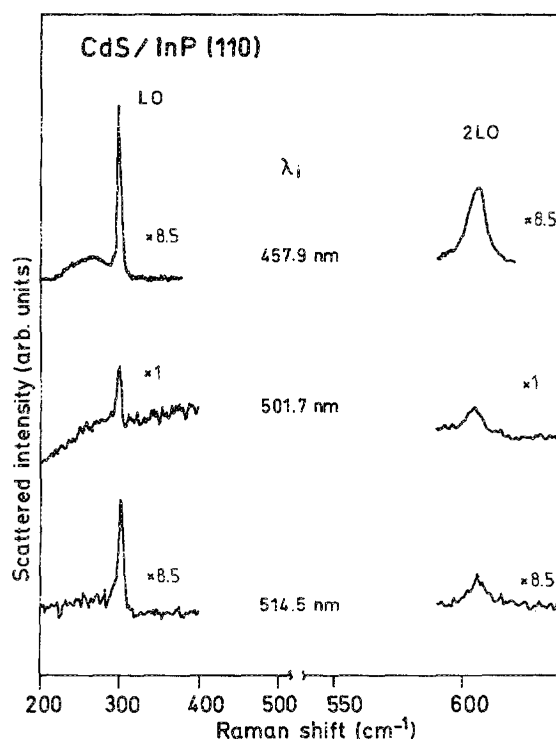

FIG. 3. Three sets of Raman spectra of the same sample as in Fig. 1 taken at different exciting laser wavelengths.

C. Growth and interface reaction

The spectra in Sec. III A proved the scattering intensities of the CdS features at a nominal deposition of 20 ML to be quite strong, ie approximately 12 counts/(mW s) at the maximum of the LO phonon peak. For that reason the possibility of achieving monolayer sensitivity in the Raman experiment was feasible. Figures 4 and 5 present Raman spectra taken in the crossed and parallel configurations for a series of coverages. Considering the crossed configuration in Fig. 4 the deposition of 1 ML of CdS does not induce any apparent change in the Raman spectrum compared to that of a clean cleaved InP substrate. The scattering intensity observed at 304 cm^{-1} is caused by symmetry allowed TO phonon scattering of the InP. At a higher coverage of 4 ML, however, there now is clear evidence of the CdS overlayer. The CdS LO phonon peak almost covers the substrate's TO peak a remainder of which leads to the asymmetric lineshape of the peak. Also in the 2LO region a clear peak has evolved. At the highest coverage shown this 2 LO feature is even stronger than the LO peak itself. In addition the CdS TO phonon shows up as a tiny peak.

The development of the CdS features becomes even more explicit in the Raman spectra of Fig. 5 recorded using the parallel configuration where the LO phonon scattering is Fröhlich allowed. In this case there is no significant contribution to the scattering intensity from the clean surface since the TO phonon scattering from the InP substrate is symmetry forbidden. At a CdS coverage as low as 2 ML the CdS signals are very clearly observable. Considering the LO and 2LO scattering both are found to increase steadily in intensity with coverage up to 80 ML. At the same time the 2LO/LO intensity ratio varies from 0.2 at 2 ML to approximately 1.5 at 80 ML. It is very likely that the variation in the 2LO/LO

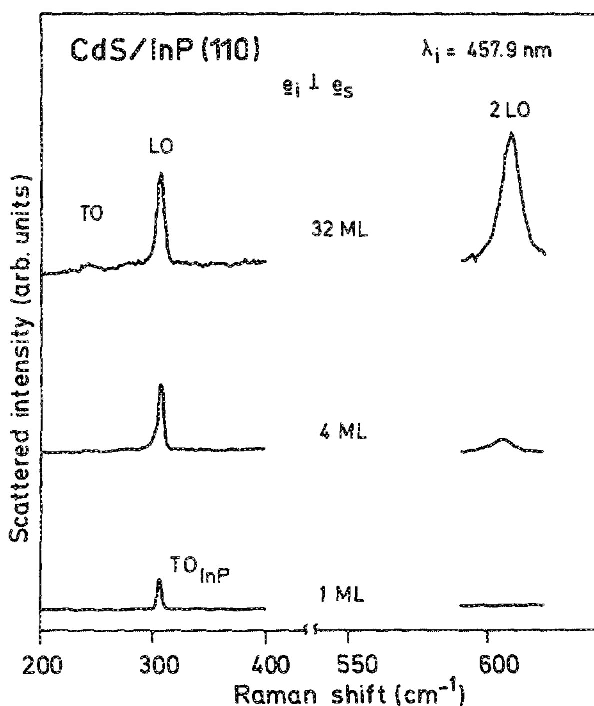

FIG. 4. Series of Raman spectra taken in the crossed configuration for increasing CdS coverages.

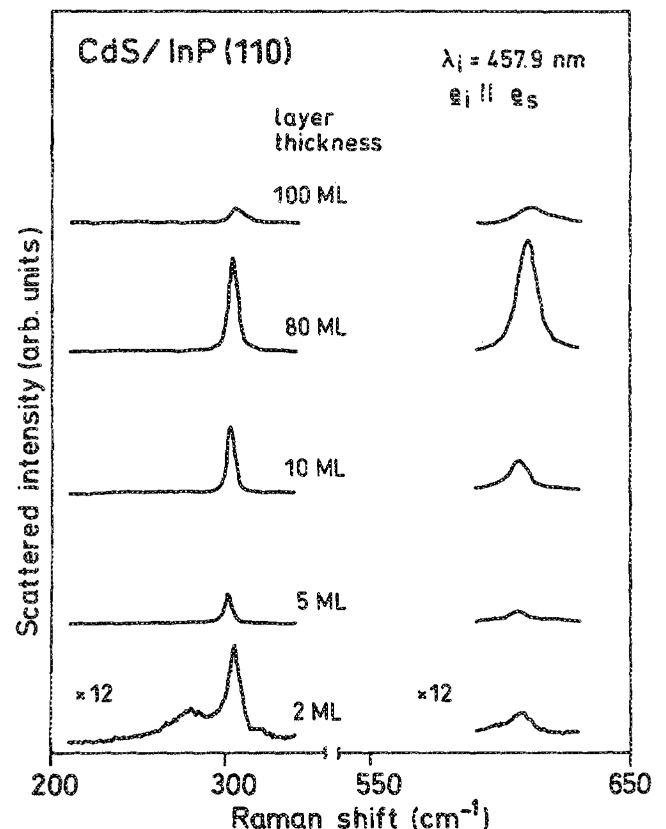

FIG. 5. Series of Raman spectra taken in the parallel configuration for increasing CdS coverages.

ratio reflects the development of the CdS electronic structure since the LO scattering is strongly coupled to the electronic (excitonic) states. The interface electronic structure should also affect the electronic properties of the CdS layer. It is thus not surprising that the saturation value of the 2LO/LO ratio at the higher coverage depends on the amount of reaction at the interface as described below. The evolution of the 2LO/LO ratio is further correlated with a shift observed in the *in situ* photoluminescence spectra.[13]

In the coverage range from 2 to 90 ML the full width at half-maximums (FWHMs) of the LO and 2LO peaks are only significantly broadened by about 2 cm^{-1} at the lowest coverage of 2 ML while the FWHM is almost constant at (6 ± 0.3) cm^{-1} for the LO and (8 ± 0.5) cm^{-1} for the 2LO at the higher coverages. While the ratio of the 2LO and LO FWHMs is very close to that measured for the FWHMs of polished CdS samples,[14] their absolute values sample are much larger (LO: 8.2 cm^{-1} and 2LO: 13.5 cm^{-1}). This may indicate that the epitaxial layers studied here are of extremely good quality. One may also expect the LO (2LO) feature of the cubic modification to be somewhat sharper than of the hexagonal because of the above mentioned folding of the dispersion which leads to the overlap of the A_1 (LO) and E_1 (LO) at almost but not exactly the same frequency position. Due to artifacts of the measurement an analysis of the frequency shifts is not given in this article.

At a coverage of 100 ML CdS a dramatic change can be seen in the spectra. There is a strong decrease in intensity together with a broadening of the LO (FWHM: 9 cm^{-1})

and 2LO (FWHM: 13 cm^{-1}) peaks. This sudden change suggests that the crystal quality has extremely worsened. A possible explanation is the formation of dislocations in the CdS overlayer which occurs when the film thickness reaches its critical value due to the lattice mismatch of the two materials InP and CdS. This assumption is supported by additional *in situ* photoluminescence measurements[13] which reveal not only a strong decrease in intensity too but also drastic changes in the lineshape at the same CdS deposition of 100 ML. The critical film thickness was calculated using the ansatz of Matthews and Blakeslee[15] to be 25 nm which corresponds to 121 ML of CdS. The experimental value lies between 90 and 100 ML which is in fairly good agreement with the calculation. However, it should be already pointed out that the critical film thickness as judged from sudden changes in the Raman as well as in the luminescence spectra strongly depends on the amount of chemical reaction at the interface.

Besides the LO and 2LO peaks there is like in Fig. 1 a broad band on the low frequency side of the LO phonon position which cannot be assigned to any InP or CdS feature. With increasing coverage this broad band disappears. Such a behavior implies that this scattering intensity stems from a chemical phase other than InP or CdS which is formed during the deposition process at the interface. When compared to Fig. 1 it is apparent that this broad feature in Fig. 1 survived to higher coverages whereas the evolution of the LO and 2LO peaks was quite similar. This happened although the samples were prepared under "identical" growth conditions, i.e., the same substrate and cell temperatures. Within the course of this study eight samples were grown under these identical conditions, for all of them the intensity of this broad band was found to differ more or less. Nevertheless there is a certain trend in the development of that feature. The Raman spectrum of the first sample grown after refilling the MBE cell with new CdS powder always exhibited a strong contribution of the broad band. The intensity of this broad band was then getting weaker for the samples grown subsequently. Indeed the Raman spectra in Fig. 5 were taken of a sample which was prepared after the cell was already used for deposition onto three other samples. As judged from the intensity of the broad band it may be called the most "ideal" sample. The trend observed suggested that the stoichiometry of the CdS powder changes with the operation time of the cell. In an x-ray photoelectron spectroscopy (XPS) measurement the original CdS powder and a remainder in the cell after breaking the vacuum were compared. It was in fact found that the former is rich in sulfur compared to the latter. It is therefore essential to consider the operation time of the cell as an additional growth parameter, in particular when, like in this study, a single source is used for deposition. This finding also substantiates the necessity of a better control of the molecular fluxes, for instance by using separate cells.

From the previous experience gained from the CdTe/InSb experiments[5] it is well known that the amount of reaction at the interface can be manipulated by the substrate temperature with a higher temperature usually leading to more reaction. In order to confirm that the broad feature is really caused by the interfacial reaction we have therefore deposited CdS at a higher substrate temperature of $T_{sub} = 510$ K. The upper spectrum in Fig. 6 represents the light scattering from a sample with a nominal deposition of 40 ML of CdS at 510 K. Obviously the broad band is now much stronger relative to the LO intensity which is also less than 1/3 as intensive as the LO phonon of the "ideal" sample at the same coverage. As a result the formation of the reacted layer is favoured at this higher substrate temperature. Another important aim is the identification of the interfacial compound. In analogy to the reactions occuring at the CdTe/InSb[5] and ZnSe/GaAs[6] interfaces In_2S_3 seems to be a likely candidate as a reaction product. This compound is also thermodynamically more stable than InP and CdS. The trend observed for the growth of subsequent samples together with the XPS results at least supports the assumption that the reacted layer has to consist of a sulfur rich compound. Since compounds of the form III_2VI_3 are known to be formed by the heterovalent exchange of the group V and VI elements when III–V substrates are heated in an atmosphere rich in group VI component,[16] we have used this approach to grow an In_2S_3/InP standard. This was done by annealing freshly cleaved InP at 770 K in an H_2S atmosphere. As a result of this procedure one obtains a several micron thick

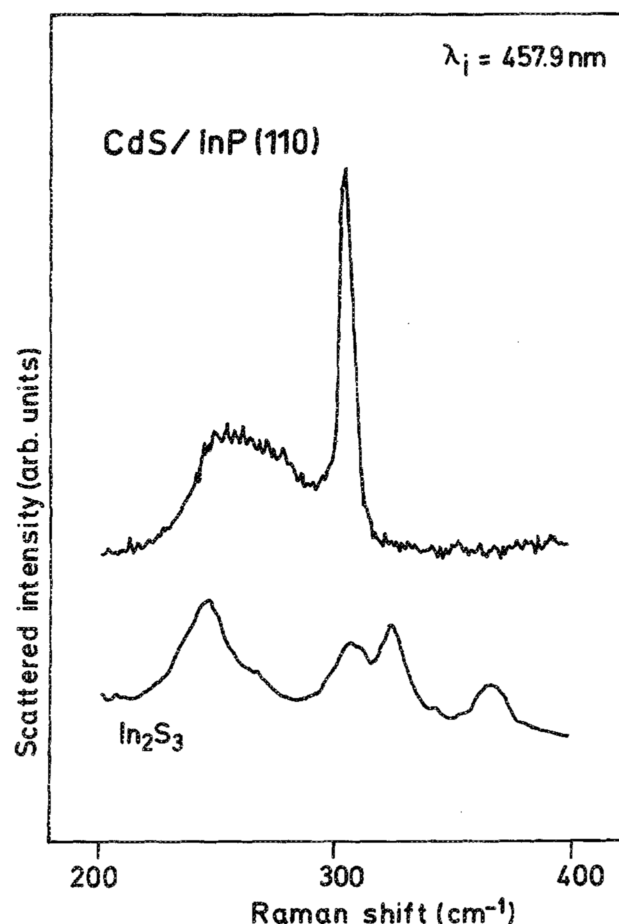

FIG. 6. Upper Raman spectrum represents a nominal CdS deposition of 40 ML onto InP(110) at 510 K. Lower spectrum is obtained from a several micron thick In_2S_3 layer on InP(110).

layer which was identified to consist of well ordered In_2S_3 by x-ray diffraction. A typical Raman spectrum of such a layer is included in Fig. 6. It was taken in the same parallel configuration used for the CdS growth experiment. There is hardly any scattering intensity detectable in the crossed configuration confirming that the In_2S_3 layer is well ordered rather than polycrystalline. Thus the selection rules of the In_2S_3/InP standard and the broad band at the CdS/InP interface are the same. A comparison of the two spectra in Fig. 6 reveals that the strongest In_2S_3 feature almost coincides with the broad band in the upper spectrum. Therefore it seems likely that the broad band represents scattering from an In_2S_3 rich layer at the interface. The fact that the reacted layer is thin and strained may explain the broadening and the shift towards higher wave numbers. However, the possibility that other reaction products such as In_5S_6 or even Cd_xP_y compounds cannot be ruled out entirely from the comparison of the Raman spectra. Nevertheless the In_2S_3 growth experiment demonstrates that In_2S_3 is the favorite compound to be formed when InP is annealed in a sulfur rich atmosphere. The situation during the initial CdS deposition onto InP could be quite similar and the agreement in energy position and selection rules strengthens the assumption that the reacted layer at the CdS/InP interface consists mainly of In_2S_3.

The amount of interfacial reaction affects the growth of the CdS overlayer in two ways. More reacted layer reduces the critical film thickness which can be explained by the larger lattice mismatch between In_2S_3 and CdS of approximately 8%. Second the saturation value of the 2LO/LO ratio decreases to be typically slightly less than 1. This indication of a lower electronic quality is supported by the luminescence spectra which reveal the presence of additional impurities in the CdS overlayer.[13]

IV. SUMMARY

In this article we have described the application of *in situ* Raman spectroscopy to study the epitaxial growth of CdS on InP(110). Raman signals originating from the CdS overlayer were obtained from coverages as low as 2 ML. The spectra supply information on the cubic modification, electronic structure, critical film thickness, and chemical reactions at the interface. The CdS phonon modes observed and their selection rules supported by the x-ray diffraction data confirm that CdS grows in the cubic modification. The possibility of using the 2LO/LO ratio in order to explore the electronic structure is currently being further investigated. Drastic changes of the CdS phonon spectra occurring around 100 ML for an ideal sample are assigned to the critical thickness effects. An additional spectral feature is explained by the formation of a reacted interfacial layer which probably consists of mainly In_2S_3. Further experiments will be performed in order to gain a better understanding of the influence of the interfacial reaction on the overlayer properties and also control over the interface reaction.

ACKNOWLEDGMENTS

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