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Elastic and plastic strain relaxation in ultrathin CdS/ZnS quantum-well structures grown by molecular-beam epitaxy

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We investigate the growth of highly strained ultrathin CdS/ZnS quantum well structures by molecular-beam epitaxy with an emphasis on structural aspects such as surface morphology of the growing layer and strain relaxation. As shown by quantitative reflection high-energy electron diffraction (RHEED) measurements, no true three-dimensional nucleation of CdS takes place despite the high mismatch relative to the ZnS buffer. Nevertheless, the CdS surface reveals a high density of very small typically monolayer islands, leading to a strong elastic relaxation at the surface. A new RHEED technique is applied to distinguish this elastic deformation from plastic relaxation through misfit dislocations. The critical thickness for the onset of the latter is found to be three monolayers. This result is confirmed by transmission electron microscopy and photoluminescence measurements carried out for further characterization, proving the presented RHEED technique to be reliable. [S0163-1829(97)03644-8]

I. INTRODUCTION

Recently, increasing research activity has been devoted to the reduction of dimensionality in semiconductor heterostructures from two-dimensional (2D) quantum wells towards systems showing a quasi-zero-dimensional electronic behavior. The most prominent approach in this field is the self-organized island formation observed, for example, in InAs/GaAs strained layer epitaxy.[1,2] In a different approach, advantage is taken from potential fluctuations in bulk material or quantum wells to achieve three-dimensional carrier confinement. The latter are either induced by alloy disorder[3,4] or by thickness fluctuations in ultrathin or even fractional monolayer quantum wells.[5] In this context we investigate highly strained (-7%) ultrathin CdS quantum wells embedded in a ZnS matrix grown by molecular-beam epitaxy (MBE). Especially, we focus on structural aspects such as initial nucleation, surface morphology, and strain relaxation. The last issue is studied in great detail using quantitative *in situ* reflection high-energy electron diffraction (RHEED) measurements. A technique that allows to distinguish between changes in lattice constant due to elastic and plastic strain relaxation is presented. It is applied to the determination of the critical thickness for plastic strain relaxation by misfit dislocations. The obtained results are verified by transmission electron microscopy (TEM) and photoluminescence (PL) measurements.

II. EXPERIMENTAL DETAILS

A. Growth

All investigated layers were grown in a home-built MBE system with a base pressure in the upper 10^{-10} mbar region. As substrates we used Si-doped GaAs (100) with no nominal miscut. The substrates were degreased and etched in a solution of 4:1:1 H_2SO_4:H_2O_2:H_2O for one minute, rinsed thoroughly in deionized water and methanol and finally dried in a filtered nitrogen jet before mounting on the substrate holder. After loading and pumping down to the 10^{-6} mbar region the substrates are reactively deoxidized by thermal annealing at 490 °C in a flowing hydrogen ambient ($p \approx 5\times10^{-4}$ mbar) before being transferred to the growth chamber. This procedure not only prevents an As depletion of GaAs due to the low annealing temperature but also minimizes an eventual etching by residual sulfur in the vacuum chamber during the deoxidation process. As source materials for epitaxy, polycrystalline CdS and ZnS of 5N5 purity each were evaporated from standard effusion cells equipped with hot-lip PBN crucibles. The use of compound sources is a simpler and therefore better reproducible approach than the more common application of the elements. It also circumvents problems due to the very high vapor pressure of elemental sulfur and the necessity to apply a cracker cell, because both materials, CdS and ZnS, are known to sublime stoichiometrically in such a way that solely S_2 molecules and metal atoms are found in the gas phase.[6] The main disadvantages of the compound source approach are the usually lower material purity available and the II/VI beam flux ratio being fixed to the value of one. Despite these facts, a layer quality being able to compete with films grown from the elements can be achieved, as recently demonstrated for ZnS by our group.[7] For reasons discussed in more detail below, all samples were grown at very low substrate temperatures (typically around 140 °C). An obvious advantage of this choice is that any thermally induced diffusion in the grown quantum wells can be excluded. Further on, sticking coefficients get close to one. Thus, the influence of possible temperature inhomogeneities over the substrate or slight temperature variations from sample to sample on the growth rate are minimized, leading to an improved reproducibility.

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B. Growth rates and determination of quantum-well thicknesses

To obtain well defined thicknesses in the growth of ultrathin quantum-well structures, careful calibration and parameter adjustment are essential, especially when—as in the CdS/ZnS system—no RHEED intensity oscillations are observed. Substrate and effusion cells were therefore kept at the desired temperatures for at least one hour to achieve thermal equilibrium before the actual growth process was started. The beam equivalent pressures, as measured by a nude ion gauge at the growth position, were chosen to be 5.0×10^{-7} mbar for CdS and ZnS, respectively, resulting in a growth rate of 0.3 ML per second for each of the materials. The beam pressures were checked to be stable within 1% for at least 10 min in any case. To calibrate the growth rates, some CdS and ZnS layers of several 100 nm thickness were grown and their thicknesses measured by the analysis of Fabry-Perot modes in the reflection spectra. However, for the determination of the CdS quantum-well thicknesses, the value obtained from the growth time and rate was additionally corrected for the following effects: The effusion rate measured a short time after the cell shutter is opened generally differs from the stationary value and also shows a temporal transient due to the cell being out of equilibrium. This effect was quantitatively taken into account by numerically integrating over the time dependent flux profile measured by the beam flux monitor and assuming the sticking coefficient to be constant. A further correction arises from the large lattice mismatch of -7% between CdS and ZnS. As long as the critical thickness is not exceeded, the CdS quantum well is forced to grow with the smaller lattice constant of the underlying ZnS buffer layer (which can be assumed to be essentially relaxed to its bulk lattice constant). Therefore, an increased number of lattice sites have to be occupied to fill a monolayer in comparison to the unstrained case. Thus, the growth rate in ML/s has to be corrected by a factor $a^2_{\mathrm{ZnS}}/a^2_{\mathrm{CdS}}=0.865$, where $a_{\mathrm{ZnS}}=5.410$ Å and $a_{\mathrm{CdS}}=5.818$ Å are the bulk lattice constants of cubic ZnS and CdS, respectively.

To independently check the described calibration procedure, some short-period CdS/ZnS superlattices were grown. From the analysis of satellite peaks observed in x-ray diffraction, the superlattice period length could be determined and compared to the value calculated from the growth parameters. In all cases excellent agreement was found. First high-resolution TEM measurements also seem to confirm these results, although a quantitative analysis for ultrathin quantum wells is difficult and still under way.

C. RHEED setup

All *in situ* RHEED measurements described below were carried out using a Staib EK-35 RHEED system operated at 13 kV. For quantitative analysis, the diffraction pattern was recorded from the fluorescence screen by a high sensitivity CCD camera connected to a personal computer (PC) equipped with a frame grabber card. Streak positions and distances were measured (usually as a function of time) by scanning the intensity profile along a predefined line parallel to the shadow edge. To determine the streak positions as exactly as possible, the obtained peaks were fitted numerically to Gaussians. This enables us to measure lattice constants to an accuracy of about 0.2%. Streak intensities were measured by integrating above an area around the streak maximum. The whole image processing system is connected to the PC controlling the shutter operations, which enables an exact correlation between growth and RHEED data aquisition.

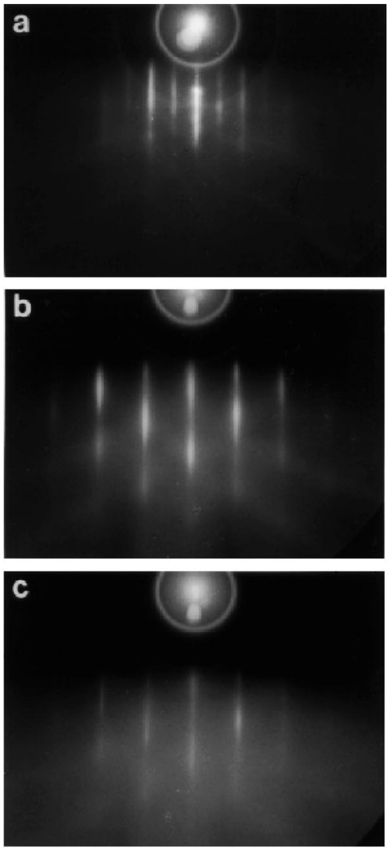

FIG. 1. RHEED patterns obtained along [011] (a) after deoxidation of the GaAs (100) substrate, (b) after growth of 630 nm ZnS buffer, (c) after deposition of a 1.2 ML CdS quantum well.

III. RESULTS AND DISCUSSION

Before starting the growth process, RHEED was applied to investigate the GaAs substrate surface. The typical result obtained for the [011] azimuth is shown in Fig. 1(a). The appearance of a streaky pattern with Kikuchi bands confirmed that deoxidation had been successful. Half order streaks due to a (2×1) reconstructed surface were frequently observed, probably caused by an adsorption of sulfur. After initiating ZnS buffer layer growth, RHEED showed an intermediate cubic spot pattern, indicating a three-dimensional nucleation. However, when growth proceeded, the diffraction spots quickly elongated more and more. Finally, a streak pattern with only slight bulklike modulation was obtained. Figure 1(b) shows as an example the RHEED pattern after deposition of 630 nm ZnS. The strong streaking suggests the surface to be very smooth. On the other hand, the bulklike modulation indicates that there is still some surface roughness enabling more than one monolayer to contribute to the diffraction signal. In general, no or only a weak $c(2\times2)$ reconstruction was observed, suggesting a stoichiometric or only slightly Zn-rich surface.

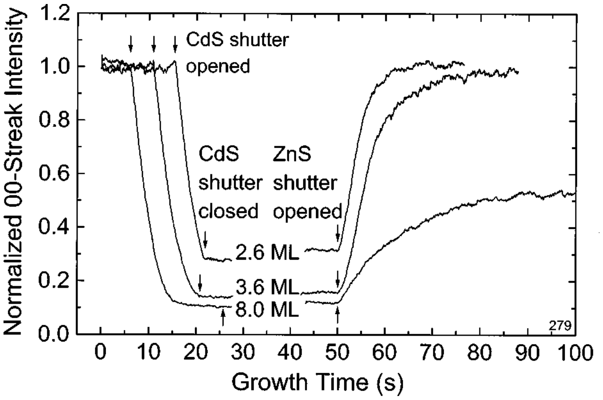

FIG. 2. Temporal evolution of the RHEED 00-streak intensity ([001] azimuth) during deposition of CdS wells with different thicknesses and subsequent ZnS overgrowth. Intensities are normalized to the ZnS buffer starting value. The time scale origin was chosen arbitrarily and has no physical meaning.

It is worth noting that a substantial increase in substrate temperature, connected to an even more Zn-rich surface due to the high volatility of sulfur, led to an increased tendency for three-dimensional growth and eventually to a faceted surface. Further details are found in Ref. 7.

After ZnS buffer layer deposition, growth was interrupted for 2 min to possibly smooth the surface, although no significant effect was observed by RHEED. When opening the CdS shutter to start the quantum-well growth, the diffraction pattern changed drastically, as shown in Fig. 1(c) for deposition of 1.2 ML CdS. On one hand, the pattern remained streaky (also for longer deposition times), indicating that *no* true three-dimensional islands were formed. On the other hand, the reflections broadened and strongly decreased in intensity due to an increased diffuse scattering. [The fact that the streaks may appear even sharper in Fig. 1(c) is only a reproduction artefact caused by the in general higher brightness of Fig. 1(b).] This led to a relatively faint diffraction pattern, which, however, recovered quickly to its former appearance, when the well was capped by only a few monolayers of ZnS, provided that not too much CdS had been deposited before.

Quantitative measurements of the temporal behavior of the 00-streak intensity along the [001] azimuth are given in Fig. 2 for different CdS well thicknesses (2.6 ML, 3.6 ML, and 8.0 ML). As can be seen, even for the growth of only 2.6 ML CdS the measured intensity decreased to less than 30% of its initial value. Although there may be influences due to material contrast by the altered surface chemistry or stoichiometry, changes in beam refraction due to the different inner potentials of CdS and ZnS or other dynamical effects, the observed decrease in intensity is so strong that it hardly can be explained by the mentioned effects alone. This becomes especially clear when comparing the streak intensities after deposition of only a few (e.g., 8.0) monolayers and after deposition of a thick layer (≈ 1 μm) CdS, where the latter shows a significantly higher streak intensity.

For the described reasons, we have to attribute at least a part of the drop in streak intensity to a different origin, namely to a *structural* change in surface morphology. The latter cannot be associated with the formation of misfit dislocations, because 2.6 ML are still less than the critical thickness for CdS/ZnS, as shown below. We attribute it to a strong increase in nucleation density in comparison to ZnS, i.e., a high number of very small islands with typically one or two monolayer height was formed. This is a reasonable assumption, because a high nucleation density is not only supported by the low substrate temperature used, which reduces adatom mobility, but especially by the large lattice mismatch of -7% between CdS and ZnS. The latter leads to a high strain energy in the CdS well, which can be significantly reduced by elastic deformation when growth takes place by the formation of many small nuclei. The RHEED observations described above can be easily explained in this picture: When an increased number of very small islands are formed on the surface, this automatically means an increase in surface disorder due to the increased step density but even more due to the fact that the growing islands can relax their strain to a certain extent by elastic deformation (see below), so that RHEED detects a distribution of lattice parameters on the surface. Both effects mean disorder that will broaden the reflections and decrease their intensity by enhanced diffuse scattering.

Growth of a few monolayers ZnS smooths out the surface until the equilibrium step density for ZnS and a uniform lattice parameter are reestablished. However, as shown in Fig. 2, RHEED intensity only recovered quickly to its original value with ZnS deposition, when less than three monolayers CdS had been deposited before. Already for 3.6 ML CdS, recovery seemed slightly delayed. On first sight, this could be simply explained by the fact that smoothing was easier in the 2.6 ML case, because the step density was smaller than in the 3.6 ML case, because the CdS equilibrium value was not yet fully reached (intensity still dropped significantly with further CdS deposition). However, this explanation can definitely be excluded for the 8 ML curve in Fig. 2. Although the starting conditions should be very similar to the 3.6 ML case (the streak intensity was nearly identical), recovery of the streak intensity was very slow and the initial value was not reached at all, even for longer deposition times. From this observation we can deduce that an irreversible structural change ocurred during deposition of the 8 ML CdS well and probably already for the 3.6 ML well. It is not far-fetched to assign this irreversible change to *plastic* strain relaxation, i.e., the formation of misfit dislocations.

To check this assumption and to further verify the nucleation model described above, the development of the in-plane lattice parameter during growth was investigated as a function of time by measuring the integer streak spacing at the position of the CdS {111} reflections in the RHEED pattern, being proportional to the reciprocal lattice constant perpendicular to the incident electron beam. Care was taken to avoid the disturbing influence of Kikuchi lines by taking the RHEED patterns a few degrees off the actual azimuth. Interpreting the measurements, one has to take care about a potential influence of refraction effects on the RHEED pattern: As already mentioned above, refraction is different in CdS and ZnS which could cause an artificial shift of the RHEED reflexes (a shift which is not connected to a change in the lattice parameter). However, for a relatively smooth layer, refraction should essentially shift the reflexes slightly along the streak direction, which does actually not influence our measurements.

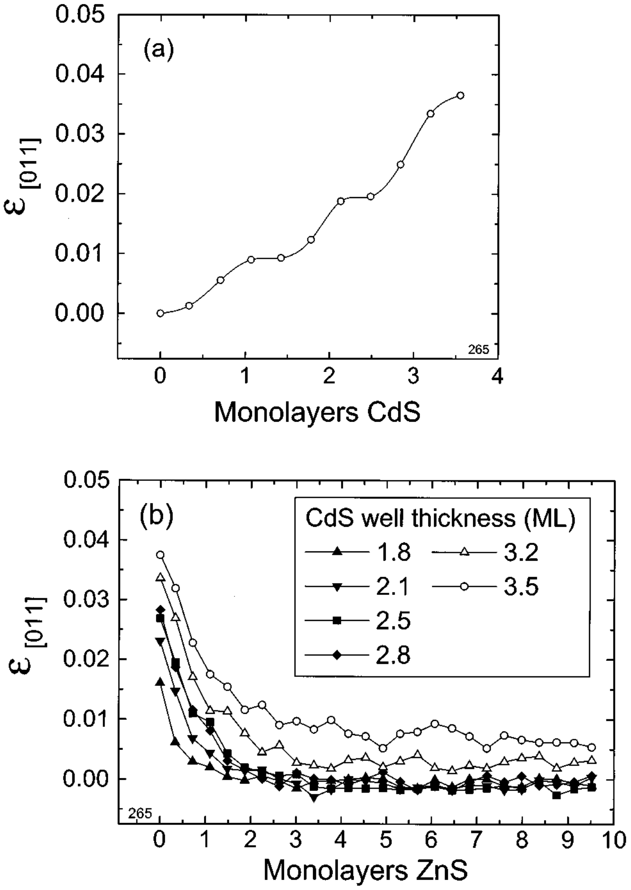

FIG. 3. Relative change in [011] lattice parameter as measured by RHEED ($[01\bar{1}]$ azimuth) during deposition of (a) CdS, (b) ZnS on CdS quantum wells of different thicknesses.

Figure 3 shows the obtained results for the relative change $\varepsilon=(a_{\mathrm{layer}}-a_{\mathrm{ZnS}})/a_{\mathrm{ZnS}}$ of the [011] lattice parameter, where a_{ZnS} and a_{layer} are the lattice parameters before starting CdS deposition and that of the growing layer, respectively. When CdS growth was initiated on the ZnS buffer [Fig. 3(a)] the measured lattice constant quickly increased significantly towards the CdS value, even if only one or two monolayers were deposited. This result, which was obtained in all azimuths observed ($[01\bar{1}]$, [011], [010]), deviates from that reported previously for CdS/ZnS using different growth conditions.[8] Indeed, it is quite unusual, because a constant ε could be expected for coherent growth, until the critical thickness for strain relaxation by misfit dislocations is reached. The increase in lattice constant is, however, a direct confirmation for the model of small nuclei showing the strong *elastic* relaxation presented above. Notably, ε not simply increased but reproducibly showed a certain oscillatory behavior with a period slightly higher than one monolayer, which was to our knowledge not observed in the CdS/ZnS system before. Simular results were, however, obtained in ZnTe/CdTe strained layer epitaxy.[9] The interpretation of our measurements is as follows: When CdS nucleates on the smooth ZnS surface the average lattice constant first increases, because more and more of the surface is covered by CdS islands which expand elastically to reduce their strain energy. However, when the first monolayer is nearly completed and the different islands are growing together, their elastic expansion is prevented and they are forced to adopt the smaller in-plane lattice constant of the underlying ZnS. The same repeats with deposition of the next monolayer, so that an oscillatory behavior is obtained, where one oscillation period corresponds to the deposition of one monolayer. However, this simplified model would only be strictly valid for an ideal layer-by-layer growth mode. In practice, the next monolayer already starts growing before the previous one is fully completed. First, this slightly slows down the oscillations, because part of the beam flux is used to form the new monolayer. Further on, the oscillations will be damped. Especially, the average ε measured by RHEED will not return to zero when a monolayer is completed, because islands of the next monolayer do already exist and show elastic relaxation. When growth proceeds, the increasing average surface roughness will enable elastic relaxation more and more and thus the average in-plane lattice parameter will increase likewise. Finally, the step density will approach its equilibrium value for CdS.

This model explains well the behavior of the in-plane lattice parameter shown in Fig. 3(a), but it was not clarified yet, to what extent *plastic* relaxation possibly contributes to the measured changes in lattice constant. We therefore have to ask for the critical thickness for the formation of misfit dislocations. As already mentioned, the latter can often be determined directly by RHEED, monitoring the lattice parameter of the growing layer: Critical thickness is exceeded, when the lattice parameter starts to deviate from the initial value for coherent growth. Obviously, this method fails, when—as in our case—strong elastic strain relaxation occurs additionally. We therefore applied a special technique that was to our best knowledge not used before: On a 950 nm ZnS buffer we grew several quantum wells with increasing thickness, separated by 90 nm ZnS spacers, and measured the temporal behavior of the in-plane lattice constant by RHEED after initiation of *ZnS spacer* deposition. [It is worth emphasizing, that these measurements are not disturbed by the different refraction in CdS and ZnS or other dynamical effects, because only RHEED investigations of one material (ZnS) are involved and the absolute value of the lattice parameter changes will not be essential for the discussion below.]

The results obtained for the [011] lattice parameter are shown in Fig. 3(b). ε is defined as above. For quantum-well thicknesses smaller than 3 ML, ε dropped to zero very quickly with ZnS growth. The ZnS lattice parameter was essentially reestablished after deposition of only 2 ML. A different behavior was observed for quantum wells thicker than 3 ML. Although ε quickly decreased in this case, too, the ZnS bulk lattice constant was not reached. Instead, ε converged to a finite value, indicating the ZnS spacer to reveal a *residual strain*, the latter being higher for thicker quantum wells. These results can be understood as follows: For quantum-well thicknesses below 3 ML, growth is coherent. Therefore, any increase in the measured in-plane lattice constant is solely due to elastic deformation of the CdS nuclei formed on the surface. When the well is capped by ZnS, mainly the asperities caused by the CdS nuclei are quickly smoothed out. Thus, elastic relaxation is prevented and the surface adopts the underlying ZnS lattice constant again. For quantum-well thicknesses above 3 ML, plastic strain relax-

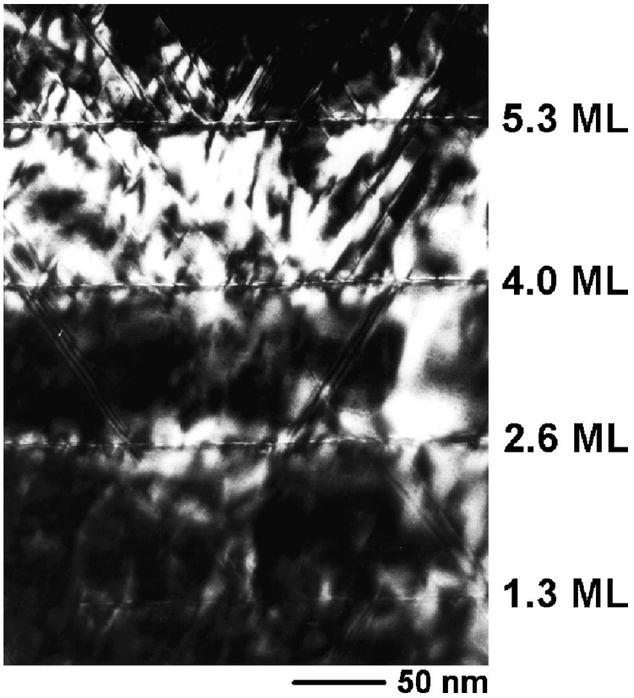

FIG. 4. Conventional two-beam [400] dark-field TEM image of a CdS/ZnS multiple quantum-well sample with different well thicknesses.

ation by the formation of misfit dislocations sets in. Thus, the in-plane lattice constant of the CdS well increases irreversibly. When ZnS is deposited onto the well and elastic relaxation is suppressed, the underlying lattice constant of the quantum well is again adopted by the growing surface, but it now deviates from the buffer value, because the quantum well is no longer coherent to the ZnS buffer. *The residual strain of the ZnS overgrowth can therefore be regarded as a measure for the degree of plastic relaxation in the quantum well*. In a quantitative analysis one should, however, take into account, that the ZnS cap reveals a finite—albeit comparably small—surface roughness, which enables partial elastic relaxation of residual strain. Possibly, this fact could also be partly responsible for the fluctuations in lattice constant that appear in the back part of the 3.2 ML and 3.5 ML curve in Fig. 3(b). They could be interpreted as a ‘‘statistical version’’ of the lattice constant oscillations discussed above, that occur, when growth is initiated on a more rough surface. Further investigations will be necessary to clarify this point. To determine the critical thickness for the formation of misfit dislocations one has to take into account that plastic relaxation could be anisotropic. For this reason, the described RHEED technique was also applied to investigate the behavior of the $[01\bar{1}]$ and [010] lattice parameters. However, the obtained results for elastic and plastic relaxation were quite simular. No significant anisotropy occured. The upper limit for coherent growth can therefore be concluded to be 3 ML for the applied growth conditions.

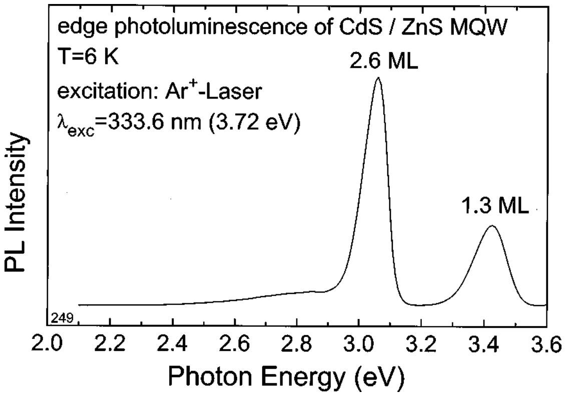

FIG. 5. Edge photoluminescence of the multiple quantum-well (MQW) sample in Fig. 4.

A comparison to the literature values of about 4 ML (Refs. 8 and 10) seems to support this result. Nevertheless, we carried out TEM and photoluminescence investigations to independently confirm the reliability of the presented RHEED technique. Figure 4 shows a conventional two-beam ($\mathbf{g}=[400]$) dark-field TEM image of a CdS/ZnS multiple quantum-well sample. The individual well thicknesses are 1.3, 2.6, 4.0, and 5.6 ML. In agreement with RHEED results, no evidence for the formation of true 3D islands was found (even in high-resolution TEM), in spite of the high lattice mismatch between CdS and ZnS. Stacking faults run through the whole structure, the origin of which, however, can be traced back to the ZnS/GaAs interface. Only for quantum-well thicknesses above 3 ML (4 and 5.3 ML) additional stacking faults were created by the formation of imperfect dislocations in the CdS well. (It should be noted that due to a limited reproduction quality two stacking faults could be assumed to start from the 2.6 ML quantum well in the shown picture. Indeed this is not the case. The latter only ‘‘vanish’’ in the light region directly below the 2.6 ML well and reappear near the 1.3 ML well which possibly cannot be seen very well in the reproduction.) This confirms the critical thickness determined by RHEED. However, TEM is obviously not very sensitive to the onset of plastic relaxation, because only few defects are created when the critical thickness is only slightly exceeded. Therefore, photoluminescence measurements were carried out for a series of single and multiple quantum wells. The obtained spectrum for the TEM sample already discussed is shown in Fig. 5. It was measured at 6 K using the 333.6 nm line of an Ar$^+$ laser for excitation below the ZnS barrier band gap energy. PL was detected from the cleaved edge of the sample to avoid the influence of Fabry-Perot modes. Two strong luminescence bands were observed that could be assigned to localized excitonic emission from the 1.3 and 2.6 ML well by comparison with single quantum-well spectra. In contrast to that, the luminescence efficiency dropped strongly for the wider quantum wells (4.0 and 5.3 ML) due to nonradiative recombination caused by misfit dislocations, so that hardly any PL could be detected. Indeed, the inspection of further samples showed that the thickness limit is very sharp and 2.8 ML was the maximum nominal well thickness resulting in a reasonable luminescence. Thus, the results of the RHEED technique presented above are confirmed impressively.

Nevertheless one should note some limitations of the described technique: On one hand it is an *in situ* method, which should allow in principle the direct determination of critical thickness with the growth of only one sample and no further *ex situ* characterization. On the other hand, the detected changes in the lattice parameter are rather small (parts of a percent) and therefore require careful measurement and a high quality RHEED equipment. Even then the applicability of this technique is restricted to cases of really high lattice mismatch (at least several %) to yield significant results, because for short times after the onset of plastic relaxation the deviation of the lattice parameter from the value for coherent growth would otherwise be too small to be detected. In any case this relatively involved RHEED technique will only pay out when detailed structural information about surface roughness, anisotropic strain relaxation, etc. is required. Otherwise, PL measurements are a much easier and very sensitive approach to detect critical thickness, as also demonstrated in this paper.

IV. CONCLUSIONS

We investigated the MBE growth of highly lattice-mismatched ultrathin CdS/ZnS quantum-well structures with an emphasis on surface morphology and strain relaxation. At least for the growth conditions used, the nucleation of CdS on the smooth ZnS buffer under the influence of strain was shown to take place by the formation of a high density of small islands with 1–2 ML height, leading to the occurence of strong elastic relaxation at the surface. No true 3D islanding was observed. A RHEED technique was presented that enabled us to distinguish between elastic and plastic strain relaxation. The latter was applied to determine the critical thickness for the formation of misfit dislocations in the CdS quantum wells. We obtained a value of 3 ML for the used growth conditions, with no evidence for strong anisotropy of plastic strain relaxation. The determined critical thickness could be verified by TEM and PL measurements, thus proving the reliability of the presented RHEED technique.

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