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Optical characterizations of $CuInSe_2$ epitaxial layers grown by molecular beam epitaxy

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$CuInSe_2$ (CIS) films with Cu/In ratios of $\gamma = 0.82-1.79$ have been grown on a GaAs (001) substrate by molecular beam epitaxy. Piezoelectric photoacoustic (PPA) measurements were carried out from liquid helium to room temperature to investigate nonradiative carrier recombination processes in comparison with photoluminescence (PL) measurements which directly detected radiative carrier recombination processes. Three PPA signal peaks which corresponded to band gap energies of the CIS (*AB* and *C* bands) and the GaAs substrate, were clearly obtained between liquid helium and room temperatures. A free-exciton emission line was observed up to 200 K in the PL spectra. Two additional peaks on intrinsic defects which are Cu vacancy (V_{Cu}) and interstitial In (In_i) were observed in the In-rich CIS samples. The PPA measurements were useful in investigating the defect levels and the band gap energy in the CIS/GaAs thin films. [S0021-8979(99)02720-6]

I. INTRODUCTION

I–III–VI_2 chalcopyrite semiconductors, especially $CuIn_XGa_{1-X}Se_2$ (CIGS), have attracted much attention for solar cell applications due to its large absorption coefficient of more than $10^5\,cm^{-1}$ near the band gap and its band gap energy of between 1.0 and 1.7 eV at room temperature. Despite the lack of accurate material information, conversion efficiencies of the polycrystalline CIGS based solar cells have been significantly improved to $\eta = 17.7\%$.[1] This indicates that CIGS will be a promising material for high efficiency devices. However, the chalcopyrite semiconductor has many intrinsic defects in the crystal which are not well understood. Furthermore, growth of stoichiometric high quality CIGS crystals is difficult in comparison with $CuInSe_2$ (CIS) and $CuGaSe_2$ (CGS) crystals. Therefore, it is important to grow and characterize high quality CIS and CGS crystals for making high efficiency solar cells.

Photoacoustic (PA) measurements have been used in investigating physical properties of the semiconductors.[2–5] One of the great advantages of the PA measurement is a direct monitor of the nonradiative recombination processes. Therefore, the PA technique may complement the photoluminescence (PL) technique which can directly detect the radiative recombination processes. The other advantage is that the PA measurement is sensitive to a small change of optical absorption coefficient in a transparent sample. Zegadi *et al.*[5] reported the PA spectra of CIS thin films grown by an evaporation method using a gas-microphone technique. However, no thorough studies have been accumulated yet in the chalcopyrite semiconductors.

In the I–III–VI_2 chalcopyrite semiconductors, the material properties are known to be strongly dependent on the defects in the crystal. However, detailed material information such as the difference in physical characterization between Cu- and In-rich CIS films is still lacking. Furthermore, the correlation between the defects and solar cell characteristics has not been well understood.

In our previous articles,[6,7] we reported that CIS epitaxial layers on GaAs (001) were grown by molecular beam-epitaxy (MBE). The Cu-rich CIS were concluded to be high quality films since streaky reflection high-energy electron diffraction (RHEED) patterns and sharp free-exciton emission were observed in the PL spectra at a low temperature.

In this article, the CIS epitaxial layers on a GaAs substrate (001) grown by MBE were examined by piezoelectric PA (PPA) spectroscopy between liquid helium and room temperatures for the dependence of the optical characterization in the CIS samples on the Cu/In ratios (γ). Furthermore, the usefulness of the PPA measurements to study the nonradiative carrier recombination processes in the CIS thin films is also discussed.

II. EXPERIMENTAL PRODUCERS

The CIS epitaxial layers were grown on a semi-insulating (SI) (001)-oriented GaAs substrate by MBE at a

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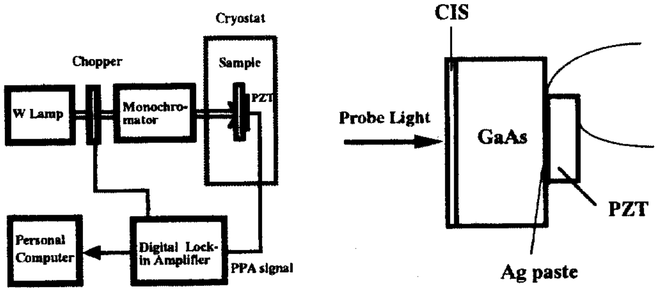

FIG. 1. Measurement system for the PPA spectroscopy and schematic cross-sectional view of the CuInSe$_2$ film on a (001) oriented GaAs substrate.

substrate temperature of $T_S = 450\,°\mathrm{C}$ with three separate effusion cells of Cu (7N), In (7N), and Se (6N). The Cu/In ratio was controlled by changing the In beam flux and excess Se was supplied during growth. Thickness of the obtained samples was about 1.0 μm: The Cu/In ratios were determined by means of an electron probe micro analysis (EPMA) to be from 0.82 to 1.79. The CIS samples exhibited a p-type condition and the net carrier concentrations of $N_A - N_D$ of 2×10^{16} to $2\times 10^{20}\,\mathrm{cm}^{-3}$ were obtained by Hall measurements at room temperature using the Van der Pauw technique.[6]

The PPA spectra were measured between liquid helium and room temperature under the modulation frequency of 100–800 Hz. The signals were detected by a disk-shaped piezoelectric transducer (PZT) attached directly to the sample rear surface (GaAs substrate side) with respect to the incident beam. Conducting silver paste was used to obtain good thermal and mechanical contacts between the sample and the PZT transducer. The observed signals were amplified through a lock-in amplifier and processed by a personal computer. The experimental setup for the PPA measurements is shown schematically in Fig. 1.

III. RESULTS AND DISCUSSION

A. Photoquenching effect

The PPA measurements of the CIS/GaAs epitaxial layers were carried out to observe the nonradiative carrier recombination processes. The typical PPA spectra of the Cu-rich CIS thin films (Cu/In= 1.79) at 80 K is shown in Fig. 2(a). Some distinct peaks were clearly observed in the PPA spectrum. The peak positions at 1.04 and 1.50 eV are in good agreement with the E_g of CIS (AB band)[8] and GaAs,[9] respectively. Furthermore, another peak at 1.26 eV is well corresponded to be the C band in the CIS crystals.[10]

It is well known that deep defect donor level EL2 is a dominant donor to accomplish a SI–GaAs for large scale integration applications. Since EL2 transforms to its metastable state by the monochromatic-light illumination about 1.1 μm (1.1 eV) at the low temperature, so called photoquenching effect. One of the commonly accepted properties about this metastable state (EL2*) is that the initial state before photoquenching (EL2^0) can be revived after annealing the sample above 130 K. In addition, the existence of an optical recovery from EL2* to EL2^0 was observed in a SI–GaAs sample.[3] Therefore, the secondary light ($\lambda = 1100\,\mathrm{nm}$) illuminated the CIS/GaAs thin film until the photoquenching state was reached. The PPA spectrum of the CIS/GaAs after quenching at 80 K are shown in Fig. 2(b). After quenching the PPA signal intensity becomes small and almost disappears below the E_g of GaAs. It is also well known that the photoquenched state of SI–GaAs remains up to 150 K.[11] Therefore, the temperature of the sample decreases again at 80 K after that of the sample increases at 150 K in the cryostat. The observed PPA spectrum of the CIS thin film is corresponded with that of the before quenching. This indicates that EL2 levels are thermally recovered at 150 K. The PPA signals of CIS are overlapped with those of EL2 in the GaAs substrate. Therefore, it is considered that the PPA signals due to the defect levels of CIS or GaAs in the CIS/GaAs films are difficult to obtain less than 150 K. To obtain the PPA signals due to lattice defects of the CIS/GaAs samples at less than 150 K, the secondary light illumination is useful in the photon energy region higher than 1.1 eV.

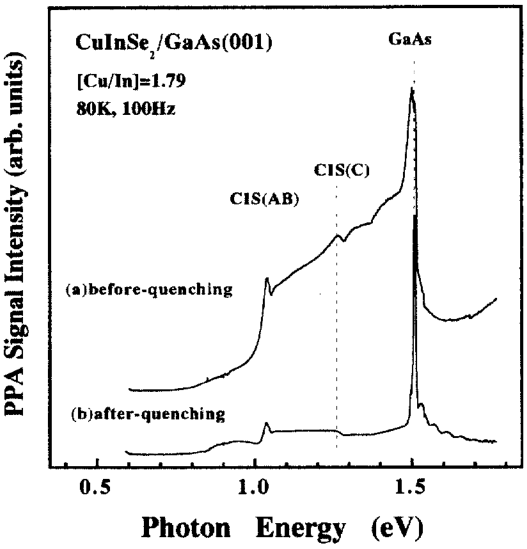

FIG. 2. PPA spectra of a CuInSe$_2$/GaAs thin film grown by MBE before (a) and after (b) quenching with a photon energy of 1.1 eV at 80 K with a modulation frequency of 100 Hz.

B. Temperature dependence

The typical temperature dependent PPA spectra of the Cu-rich ($\gamma = 1.79$) CIS/GaAs thin films between 4.2 and 300 K are shown in Fig. 3. In the PPA spectra at 4.2 K, two peaks and one broad band are observed at 1.04, 1.51, and 1.28 eV which are in good agreement with band gaps of CIS (AB band),[7] GaAs,[11] and CIS (C band),[12] respectively.

As the temperature increased, the intensities of all peaks decreased and, on the other hand, a value of full width at half maximum (FWHM) increased. However, the peaks due to the band gap of GaAs and CIS are clearly observed at room temperature (RT) at 1.410 and 1.005 eV, respectively. These values are in good agreement with those reported by Ikari *et al.*[13] and Zegadi *et al.*,[14] respectively. On the other hand, PL emission is not observed at RT. Earlier indications are that the PPA measurements are sensitive and effective to obtain the nonradiative characterization in the CIS thin film.

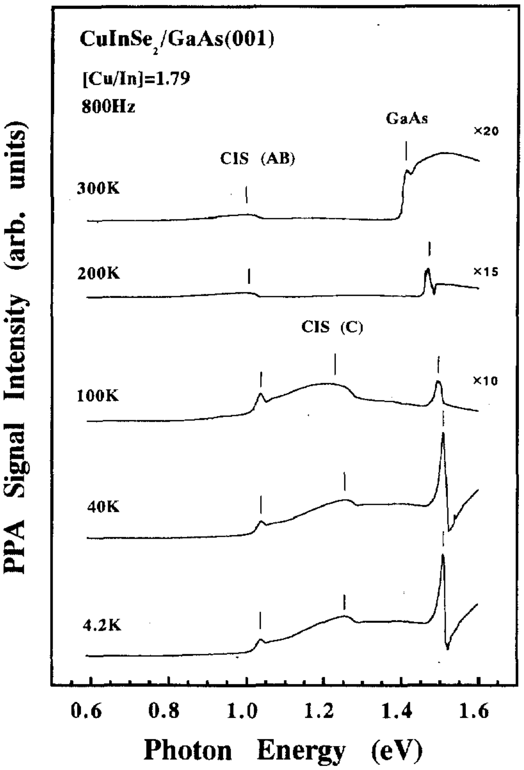

FIG. 3. Temperature dependence of PPA spectra of a Cu-rich (Cu/In $=1.79$) CuInSe$_2$/GaAs thin film grown by MBE between 4.2 and 300 K with a modulation frequency of 800 Hz.

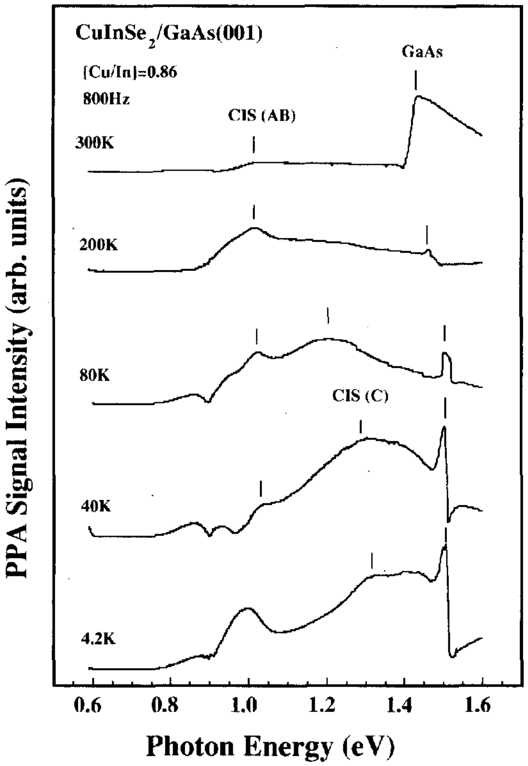

FIG. 5. Temperature dependence of PPA spectra of an In-rich (Cu/In $=0.82$) CuInSe$_2$/GaAs thin film grown by MBE between 4.2 and 300 K with a modulation frequency of 800 Hz.

Temperature variation of the A band which is attributed to the band gap of CIS is plotted in Fig. 4. E_g is almost temperature independent from 4.2 to 60 K and decreases above 60 to 300 K. This decrease can be described by the secondary curvilinear function. The temperature variation of E_g has been shown to follow Varshni's empirical equation[15]

$$E_g = E_g(0) - aT^2/(b+T), \tag{1}$$

where $E_g(0)$ is E_g at 0 K, a and b are the fitting parameter and the Debye temperature, respectively. In the case of the peak A for the band gap of CIS, the best fitting curve using Eq. (1) is also shown in Fig. 4. The obtained value of a and b are 2.1×10^{-4} (eV/K) and 230 (K), respectively. The obtained Debye temperature of 230 is in good agreement with 220 reported by Bachmann *et al.*[16] The energy difference of E_g of CIS between liquid helium and room temperatures is about 33 meV and this value is very small in comparison with 92 meV of GaAs. Furthermore, the C band is also fitted by the Eq. (1). The temperature dependence of the C band is in good agreement with the Varshni's empirical equation by using the same earlier values $[E_g = 1.26, a = 2.1\times 10^{-4}\,(\text{eV/K}), b = 230\,(\text{K})]$. This indicates that the temperature dependence of the C band is similar behavior to that of the AB band of the CIS from 4.2 to 60 K.

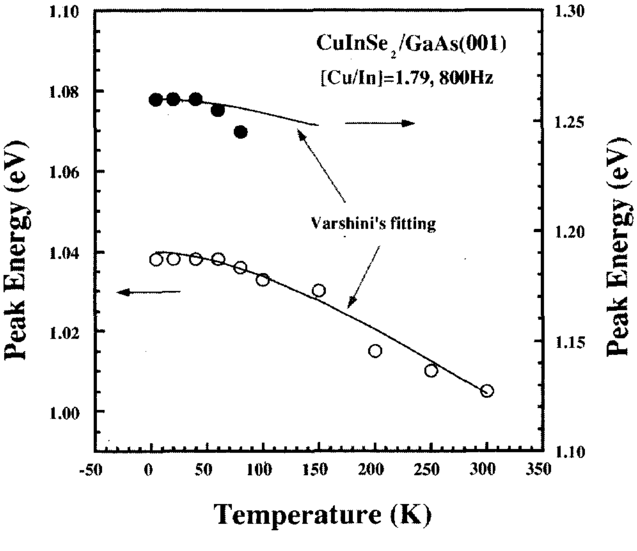

FIG. 4. Temperature variations of the band gap energies (AB and C bands) of a Cu-rich (Cu/In= 1.79) CuInSe$_2$ grown by MBE between 4.2 and 300 K. Solid line indicates Varshini's fitting.

In a previous article,[7] we reported that a free-exciton peak shifts to a higher energy side with increasing the temperature from 7 to 70 K in the PL spectra. The same phenomenon is reported by Yu[8] and Tanda *et al.*[17] in PL spectra at a low temperature. If the band gap energies monotonically shifted to a higher energy side as that of free-exciton peak, the observed temperature dependence of the PPA spectra could not be understood. It is assumed that one of the reasons is that exciton binding energy decrease slightly from 4.2 to 60 K. The other reason is that a hybridization of the copper or silver d-levels with the chalcogen p-levels are not strongly dependent from 4.2 to 60 K.[18]

Figure 5 shows the typical temperature dependent PPA spectra of an In-rich ($\gamma = 0.86$) CIS/GaAs thin film between

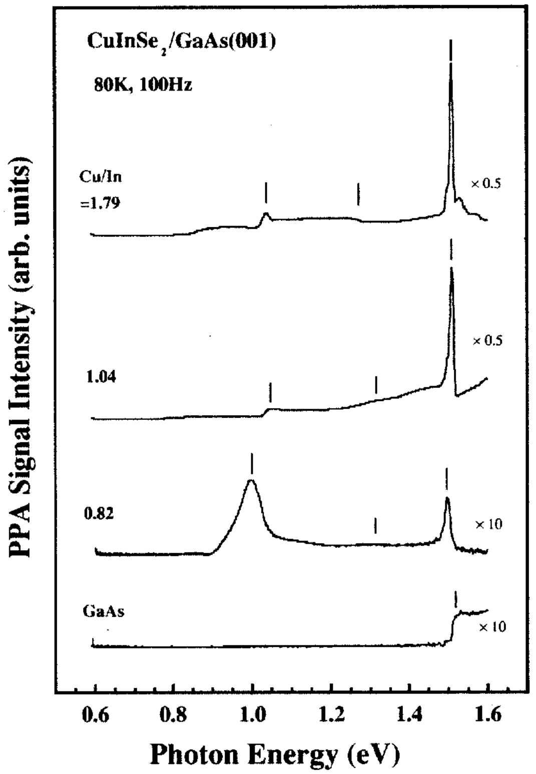

FIG. 6. PPA spectra of a CuInSe$_2$/GaAs thin film grown by MBE (Cu/In $=0.82$, 1.04, and 1.79) and GaAs substrate at 80 K with a modulation frequency of 100 Hz.

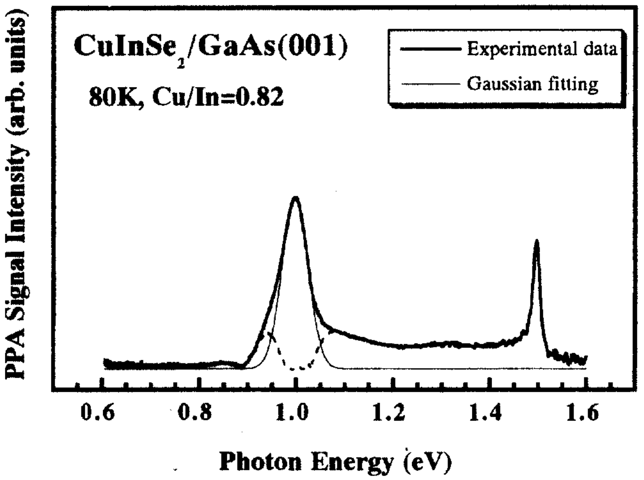

FIG. 7. PPA spectra of an In-rich CuInSe$_2$/GaAs thin film (Cu/In$=0.82$) grown by MBE at 80 K with a modulation frequency of 100 Hz. Bold and thin lines indicate experimental and Gaussian fitting data, respectively. Dotted line obtained by subtracting the Gaussian fitting data from experimental data.

4.2 and 300 K with a modulation frequency of 800 Hz. In the PPA spectrum at 4.2 K one peak and three broad bands at 1.51, 0.86, 0.99, and 1.30 eV are observed. The values of 1.51 and 1.30 eV are in good agreement with band gaps of GaAs and CIS (*C* band) in the Cu-rich sample, respectively. However, the other peak values (0.86 and 0.99 eV) are smaller than the band gap (*AB* band) of the Cu-rich sample. As the temperature increased, the PPA signals are drastically changed around 1.0 eV in comparison with that below 40 K. One broad band of 0.99 eV at 4.2 K began the separating of two bands (1.03 and 0.93 eV) at 40 K. Their values of 1.03 and 0.93 eV are due to the band gap (*AB* band) of the CIS and an intrinsic defect, respectively. However, the value of the *AB* band of the In-rich CIS is smaller than that of the Cu-rich sample. This will be discussed in the next section in detail. The activation energies of the intrinsic defects are estimated to be about 100 and 170 meV from the signals at 0.93 and 0.86 eV, respectively. It is assumed that this sample has Cu vacancy (V_{Cu}), interstitial In (In_i) and substitutional In at the Cu site ($\mathrm{In}_{\mathrm{Cu}}$) defects since this sample is Cu-poor, In-rich, and Se-rich from the EPMA results. Therefore, it is deduced that these peaks are due to V_{Cu}[19] and In_i,[20] respectively.

C. DEPENDENCE OF Cu/In RATIO

The typical PPA spectra at 80 K under a modulation frequency of 100 Hz of In-rich, nearly stoichiometry and Cu-rich CIS epitaxial layers (Cu/In$=0.82$, 1.04, and 1.79) are shown in Fig. 6. All spectra were obtained with secondary light ($\lambda = 1100\,\mathrm{nm}$) illumination. The PPA spectrum of a GaAs substrate is also plotted in comparison with the CIS/GaAs samples. The dominant peak is due to the band gap of GaAs in the PPA spectrum of the GaAs substrate. In the PPA spectrum of Cu/In$=1.04$, two distinct peaks which are due to band gap energy of CIS (*AB* band), and GaAs at 1.04 and 1.51 eV are observed, respectively. A weak band due to the *C* band of CIS is observed at 1.28 eV. The peaks in the spectrum of the other samples can also be ascribed to CIS (*AB* band), CIS (*C* band), and GaAs, while this has already been explained in Secs. III A and III B.

In the spectrum of the In-rich sample (Cu/In$=0.82$), a broad band is clearly observed at 1.0 eV. This peak energy is smaller than the band gap energy of the stoichiometric and Cu-rich sample. The shape of the spectra of the In-rich sample is also different from that of the stoichiometric and Cu-rich sample. Therefore, it is assumed that the broad band at 1.0 eV is due to an intrinsic defect. Therefore, the Gaussian fitting line is described in Fig. 7. By subtracting the Gaussian fitting data from experimental data, two new peaks at 0.94 and 1.06 eV which are due to an intrinsic defect and the *AB* band, respectively. In addition with these peaks, the weak peak is observed at about 0.85 eV. The estimated activation energy of about 120 and 200 meV are well corresponded with those in the sample of Cu/In$=0.86$.

Relationships between the band gap energies of CIS (*AB* band), CIS (*C* band), and GaAs estimated as earlier and Cu/In ratios are plotted in Fig. 8. The E_g of the GaAs does not change in the entire region of the Cu/In ratio from 0.82 to 1.79. The E_g of CIS (*AB* and *C* bands) almost does not change in the region of the Cu/In ratio from 0.82 to 1.04. This result is in good agreement with PL results.[6] On the other hand, the E_g of CIS (*AB* and *C* bands) almost does not change, slightly increased in the Cu/In$=0.82-1.04$. However, Chichibu *et al.*[21] reported that the E_g of CIS (*AB* and *C* bands) become small in an In-rich region with decreasing

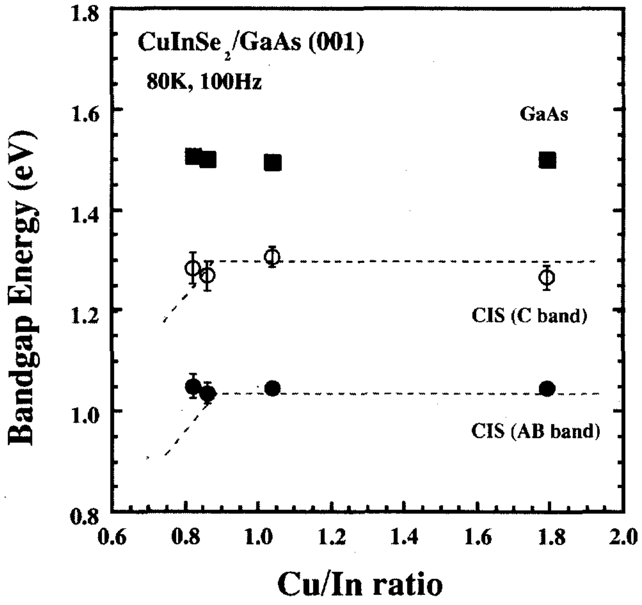

FIG. 8. Relationships between band gap energies (*AB* and *C* bands) of CuInSe$_2$ and Cu/In ratio at 80 K. Dotted lines indicate the band gaps obtained from the absorption spectroscopy in the CuInSe$_2$/glass thin films at room temperature. The band gap energies of GaAs in the CuInSe$_2$/GaAs thin films also plotted in comparison with the CuInSe$_2$.

the Cu/In ratio obtained by absorption spectra at RT in CIS/7059 glass thin films grown by molecular-beam deposition,[21] indicated in Fig. 8 (dotted lines). One of the reasons for this conflict is the difference of the crystalline quality between epitaxial and amorphous layers. The In-rich epitaxial layers on the GaAs substrate may be a growth ordered vacancy compound in the sample. However, the PPA signal of the band gap is overlapped by that of an intrinsic defect since In-rich samples have an intrinsic defect which has shallow activation energy. We need to measure another composition samples (high In-rich) or analyze, for example fitting a theory, of the PPA spectra in detail for further discussing of band gap in the In-rich region.

The peak intensity of the band gap of CIS (I_{CIS}) clearly increases with decreasing the Cu/In ratio as shown in Fig. 6. On the other hand, the intensity of the peak due to the band gap of GaAs (I_{GaAs}) increases with increasing the Cu/In ratio. In other words, the intensity of $I_{\mathrm{CIS}}/I_{\mathrm{GaAs}}$ decreases with increasing the Cu/In ratio. Since now the excitation light is illuminated into the CIS side, if the absorption coefficient (α) of CIS is very large for the thickness (l), ($\alpha\times l>1$), the excitation light is not reached to the GaAs substrate and the signals of GaAs are not obtained. Therefore, one of the reasons seems to be that the absorption coefficient of CIS becomes large with decreasing the Cu/In ratio. Furthermore, the PPA signals are almost enhanced by a thermal diffusion length (L) which is obtained by an equation of $L=(2\beta/\omega)^{1/2}\times 10^3$ (μm), where ω is the modulated frequency (rad/s) and β is thermal diffusivity (cm^2/s). Therefore, the other reasons that if the absorption coefficients are not changed, the thermal diffusion length (that is to say, thermal diffusivity) of the CIS samples become large with decreasing the Cu/In ratio from 0.82 to 1.04. Furthermore, the FWHM of the E_g peak of the CIS become small with increasing the Cu/In ratio in Fig. 3. This indicates that the quality of the CIS epitaxial layers become worth with decreasing the Cu/In ratio from 1.04 to 0.82. It is deduced that stoichiometric CIS epitaxial layers are higher in quality than the In-rich CIS ones.

The CIS epitaxial layers grown by MBE were measured and precise lattice parameters and integrated intensities of chalcopyrite reflections peaks were determined.[22] The corrected lattice parameters of the Cu-rich CIS were nearly consistent with the Joint Committee on Powder Diffraction Standards diffraction data (standard values). However, in the In-rich CIS, the c/a ratio was about 2.028 and the cell volume was smaller than the standard value, which was estimated to be 98.4%. It would appear that the structure of the In-rich composition films include many vacancy-type defects. Furthermore, the specimen with $\gamma=0.98$ had the minimum FWHM and the maximum peak intensity, implying that this is the most perfect crystal. On both sides of $\gamma=0.98$, the value of the FWHM are larger and the peak intensity smaller.

The structural properties of the CIS films were investigated by *in situ* RHEED patterns.[6] In the Cu-rich films, for the $[-110]$ direction, a streaky $(\times 1)$ pattern can be observed, indicating high quality films. On the other hand, in the In-rich films, the RHEED pattern became rather spotty, and nearly identical for the $[-1-10]$ and $[-110]$ directions.

The structural properties of the films were also investigated by cross-sectional transmission electron microscopy.[6] Misfit dislocations are created at the substrate/CIS interface in both of the Cu- and In-rich films, however, the number of dislocations falls off along the growth direction. Within our detection limit, no second phase was found in the near stoichiometric films. However, nucleation of Cu–Se binary compounds tentatively identified as Cu$_2$Se was observed in Cu-rich samples, in good agreement with the results of the x-ray diffraction.[22] On the other hand, no clear indication of a second phase was observed for the In-rich film. However, a large number of twins on {112} planes appeared. We also found a significant change in the lattice constant from standard values when the films become In-rich. The earlier results suggest that the growth mode and the stress relaxation mechanism are quite different between Cu- and In-rich films.

All the films showed p-type conduction, and their hole concentration with respect to the Cu/In ratio are shown in the previous article.[6] The Cu-rich films were strongly p type, and the carrier concentration drops drastically at $\gamma\sim 1$. In-rich films with $\gamma<0.9$ were highly resistive, and the quality of metal–CIS contact was insufficient for measurement.

PL spectra obtained from the samples with different Cu/In ratios are also shown in the previous article.[6] Sharp and distinct emission lines were observed in the Cu-rich samples. The excitation power and temperature dependence of such emission lines made possible the identification of phonon replicas from substantive emissions. On the otherhand, a broad band strong emission appears in the In-rich films. Excitation power dependence of the broad band emission showed that the peak significantly blueshifts with increasing the excitation power, indicating a donor-acceptor pair emission. It also indicates that In-rich CIS films are heavily compensated, in good agreement with the electrical characterization.

IV. CONCLUSIONS

The $CuInSe_2$ epitaxial layers (Cu/In$=0.82-1.79$) were grown on SI (001)-oriented GaAs substrate by MBE at a substrate temperature of $T_S=450\,^\circ C$, and the thickness of these samples was about 1.0 μm. The CIS samples were all *p* types and the net carrier concentrations of N_A-N_D of about from 2×10^{16} to $2\times10^{20}\,cm^{-3}$. PPA measurements of the CIS samples were carried out at from liquid helium and room temperatures in order to know the nonradiative carrier recombination processes. The PPA signals due to the band gap energies of the CIS (*AB* and *C* bands) and GaAs are clearly observed in the Cu-rich CIS samples although the free-exciton emission is observed up to 200 K in the PL spectra. Their peak energies in the PPA spectra are not changed with changing the Cu/In ratio. Temperature variation of the band gap of the CIS is in good agreement with curve fitting by the Varshini's empirical equation. On the other hand, the peak signal of the band gap energy is overlapped with an intrinsic defects in the In-rich sample. The In-rich samples have many intrinsic defects such as the Se vacancy and interstitial In. The PPA measurements are useful in investigating the defect levels or the band gap energy in the CIS/GaAs thin films.

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