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Liquid Phase Epitaxial Growth of Fe-Doped Semi- Insulating InP

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Semi-insulating InP epitaxial layers with a resistivity as high as 8×10^7 ohm-cm have been grown by liquid phase epitaxy using Fe as a dopant and high growth temperatures. Behavior of Fe doping has been well explained by the solubility of Fe in the growth solution and temperature dependence of the distribution coefficient of Fe. Fe concentration in semi-insulating layers has been estimated to be in the high 10^{15} cm^{-3} range by SIMS analysis.

1. Introduction

Semi-insulating (SI) InP epitaxial layers are eminently suitable for current confinement layers in InGaAsP/InP buried heterostructure (BH) lasers. This is because both the leakage current and the parasitic capacitance can be significantly reduced by their use.

The most common method for obtaining SI-InP crystals is doping with transition metals. In particular, Fe and Ti introduce mid-gap states within the bandgap of InP and compensate for residual impurities. SI-InP bulk crystals have been grown by doping with Fe[1] or Ti[2] by the liquid encapsulated Czochralski (LEC) technique. Recently, Fe-doped SI-InP epitaxial layers have also been grown by metalorganic chemical vapor deposition (MOCVD)[3].

However, liquid phase epitaxial (LPE) growth of SI-InP layers doped with Cr[4], Fe[4], and Co[5] has been unsuccessful so far. The cause of the difficulty in LPE growth of SI-InP has been assumed to be that the solubility of transition metals in the growth solution and/or the distribution coefficient are extremely small at the growth temperatures usually employed (below 800 °C).

In this work, we present the first report on LPE growth of semi-insulating InP layers. This has been accomplished by employing Fe as a dopant and high growth temperatures (above 850 °C). We also describe the results of SIMS analysis and electrical measurements on Fe-doped SI layers. Behavior of Fe doping and several characteristics of Fe-doped SI layers are discussed.

2. Experimental procedures

Undoped or Fe-doped epitaxial layers were grown on (100) oriented Sn-doped n^+- or Fe-doped SI-InP substrates. Growth temperatures, T_g, were 800 °C, 850°C, and 900°C. Materials used were 7-nines In, single crystalline InP source with a carrier concentration of about $4 \times 10^{15} cm^{-3}$, and 4-nines Fe powder. The residual carrier concentration of undoped layers was $(2 \pm 1) \times 10^{15} cm^{-3}$. The weight percent of Fe, W_{Fe}, added to the growth solution was between 0.01 wt.% and 2.0 wt.% of the growth solution.

The most serious problem in high temperature growth is thermal damage to InP substrates and InP epitaxial layers. To eliminate this problem, an InP wafer was faced to the InP substrate until growth started, then another InP wafer was faced to the as-grown epitaxial layer immediately after growth.

Resistivities of epitaxial layers were measured using Van der Pauw samples when they were below 1×10^5 ohm-cm. When resistivities exceeded 1×10^5ohm-cm, they were measured using n^+(cap layer)-SI(Fe-doped layer)-n^+(substrate) structure mesa diodes. These diodes were also used to examine electrical characteristics of SI layers.

SIMS analysis was performed on undoped and Fe-doped layers and an LEC grown Fe-doped SI-InP substrate to estimate the concentration of Fe.

3. Results

3-1. Surface morphology

Figure 1 shows Nomarsky microphotographs of as-grown surfaces of the undoped (a) and of the 0.4wt.% Fe-doped (b) layer grown at 900 °C. Both as-grown surfaces were mirror-like and significant degradation by high temperature growth and Fe doping was not observed on the surface morphology.

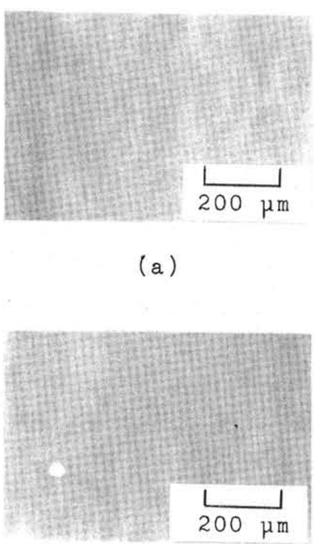

Fig. 1. Nomarsky microphotographs of as-grown surfaces of the undoped (a) and the 0.4 wt.% Fe-doped (b) InP layer grown at 900°C.

3-2. Electrical characteristics

Figure 2 shows the resistivity at room temperature as a function of the weight percent of Fe added to the growth solution. At all growth temperatures, the resistivity increased as the weight percent of Fe increased. In particular, the resistivity increased rapidly above a critical value of weight percent of Fe, and then saturated at about 5×10^6ohm-cm and 5×10^7ohm-cm at the growth temperature of 850 °C and 900 °C, respectively. The highest resistivity achieved was 8×10^7ohm-cm. This is the highest value obtained by LPE and is comparable to the resistivity of Fe-doped SI-InP layers grown by MOCVD[3]. At the growth temperature of 800 °C, SI layers were not obtained.

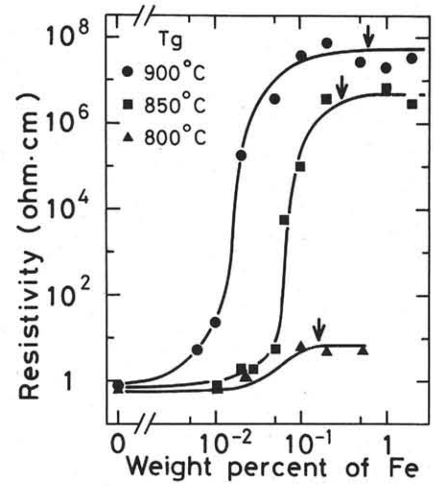

Fig. 2. Resistivity as a function of the weight percent of Fe added to the growth solution.

Typical current density(J)-voltage(V) characteristics of an n^+-SI-n^+ diode are shown in Figure 3. Resistivity and thickness of the SI-InP layer were 8×10^7ohm-cm and 3.3 μm, respectively. At low applied voltage, J-V curves obeyed Ohm's law. Above the Ohmic region, the current was in proportion to the square of the applied voltage, and then at a critical voltage the current increased rapidly.

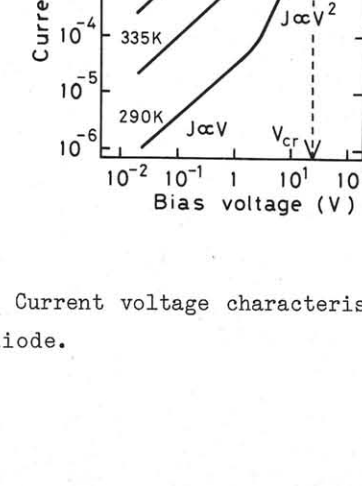

Fig. 3. Current voltage characteristics of an n^+-SI-n^+ diode.

Temperature dependence of the resistivity of the SI layer is shown in Figure 4. The activation energy of the resistivity was 0.62 eV. This value indicates that the Fermi level lies almost at the middle of the bandgap of InP.

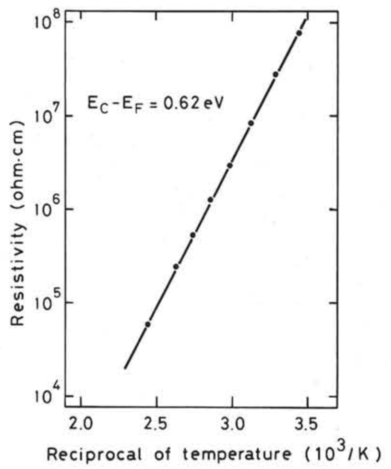

Fig. 4. Temperature dependence of the resistivity.

3-3. SIMS analysis

Table 1 shows the result of SIMS analysis. The resistivity of each layer is also shown. The experimental error was within a factor of two and the detection limit of Fe was about $5\times10^{14}cm^{-3}$. Whereas the concentration of Fe in all undoped layers was below the detection limit, Fe was detected in all Fe-doped layers. The concentration of Fe increased with increase in the weight percent of Fe or the growth temperature. About 8×10^{15} cm^{-3} of Fe was detected in a SI layer with resistivity of 5×10^7 ohm-cm. This was several times as high as the residual carrier concentration of undoped layers. In an LEC grown SI substrate, about $2\times10^{16}cm^{-3}$ of Fe was detected.

Table 1. Result of SIMS analysis.

No.	Tg(°C)	W_{Fe}(wt.%)	$X^s_{Fe}(cm^{-3})$	Resistivity(ohm-cm)
1	800	0.1	1.5×10^{15}	6
2	850	0.1	4×10^{15}	3×10^5
3	900	0.01	2×10^{15}	20
4	900	0.1	7×10^{15}	4×10^7
5	900	1	8×10^{15}	5×10^7

4. Discussion

4-1. Interpretation of behavior of Fe doping

There are two important points in behavior of Fe doping as shown in Figure 2.

The first point is that the resistivity saturates above a critical value of weight percent of Fe. This can be explained by the solubility of Fe in the growth solution. The solubility of Fe in the indium solution at each temperature[4)] is shown with an arrow in Figure 2. Each value agrees well with the weight percent of Fe where the resistivity saturates.

The second point is that the resistivity increases with increase in the growth temperature at the same value of the weight percent of Fe. This can be explained by temperature dependence of the distribution coefficient of Fe. Figure 5 shows the distribution coefficient estimated from the result of SIMS analysis as a function of the reciprocal of growth temperature. It was shown that the distribution coefficient decreases in proportion to the reciprocal of growth temperature. The distribution coefficient was about 2×10^{-5}, 5×10^{-5}, and 1×10^{-4} at 800°C, 850°C, and 900°C, respectively. The distribution coefficient at 1070°C predicted by extrapolating a linear line in Figure 5 agrees well with that reported in reference 7 for LEC grown Fe-doped InP crystals.

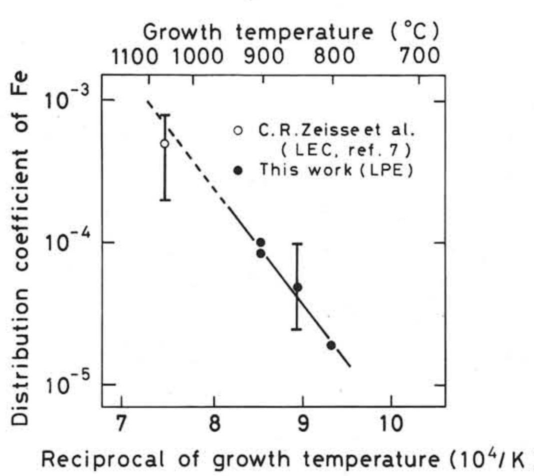

Fig. 5. The distribution coefficient of Fe as a function of the reciprocal of growth temperature.

Therefore, we can conclude that the highest resistivity of LPE grown Fe-doped InP layers at each growth temperature is limited by the solubility of Fe in the growth solution, the distribution coefficient of Fe, and the residual carrier concentration.

4-2. Analysis of current-voltage characteristics

The concentration of electron traps in a SI layer can be estimated by the critical voltage in current-voltage characteristics shown in Figure 3.

Figure 6 shows the critical voltage as a function of the thickness of the SI layer with a resistivity of about 5×10^7 ohm-cm. It was found that the critical voltage, V_{cr}, is proportional to the square of the layer thickness, L. The experimental relation was as follows, where V_{cr} is in volts and L in µm;

$$V_{cr}=1.37\times L^2 \qquad (1)$$

From Lampert's theory of the electron injection current controlled by electron traps[6], the critical voltage is given by

$$V_{cr}=(eP_t/2\varepsilon)\times L^2 \qquad (2)$$

where e is the electron charge, P_t is the concentration of unoccupied electron traps in thermal equilibrium, ε is the dielectric constant

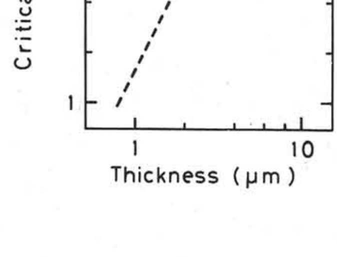

Fig. 6. Critical voltage as a function of layer thickness.

and L is the thickness of the insulator. By using eq. (1) and (2), P_t was estimated to be about $2\times10^{15} cm^{-3}$. This agrees well with the value predicted from the Fe concentration obtained by SIMS analysis and the residual carrier concentration of undoped layers.

InGaAsP/InP BH lasers usually operate at an applied voltage of 1 to 2 V. In order to obtain effective current confinement layers, SI layers of V_{cr}>2 V are necessary. The experimental relation of eq. (1) shows that the SI layer thicker than 1.2 µm is sufficient to satisfy the condition of V_{cr}>2 V. Therefore, LPE grown Fe-doped SI-InP layers can be applied to BH lasers because current confinement layers thicker than 1.2 µm can be easily grown by LPE.

5. Conclusion

Semi-insulating InP epitaxial layers with a resistivity as high as 8×10^7 ohm-cm have been grown by liquid phase epitaxy using Fe as a dopant and high growth temperatures. Behavior of Fe doping has been well explained by the solubility of Fe in the growth solution and the temperature dependence of the distribution coefficient of Fe. Fe concentration in SI-InP layers has been estimated to be in the high 10^{15} cm^{-3} range by SIMS analysis.

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