Studies on InP:Fe growth in a close−spaced showerhead MOVPE reactor

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Introduction

Semi−insulating (SI) InP:Fe has since long time been used in the fabrication of optoelectronic devices operating at high frequency, such as lasers and modulators, due to its inherent low capacitance and high resistivity. Despite a few drawbacks with Fe as a deep acceptor for realising SI InP, such as interdiffusion between Fe and p−dopants and poor current blocking for double injection, it is today the only realistic alternative. Several other deep impurities have been attempted in literature, like the more thermally stable Ti [1] and Ru [2], however, despite demonstrating excellent results, the maturity and commercial availability has hindered the adoption of those dopants in production. In order to improve yield and reduce cost, large−scale reactors have been developed for the epitaxial growth of III−V materials. Although a wide range of results on the growth of InP:Fe by MOVPE have been reported in literature, so far, very few studies have been carried out on the growth of InP:Fe utilising a production type close−spaced showerhead reactor. The showerhead, which consist of a mesh of uniformly distributed injection holes, is placed very close (1−2 cm) to the wafer surface. This is supposed to result in a stagnation point flow, where the boundary layer thickness is unchanged along the susceptor [3]. Therefore, such a reactor has been shown to be less sensitive to the growth conditions like reactor pressure, temperature and rotation [4−5].

We have in this work undertaken the study of InP:Fe both on planar substrate and selectively regrown around mesa structures in a showerhead reactor. The InP:Fe layers have been characterised by current−voltage (I−V) measurements, C−V profiler, and the regrowth behaviour studied for various growth conditions.

Experimental

All experiments have been carried out in a Thomas Swan 6x2” closed−spaced showerhead MOVPE reactor. The growth of InP:Fe has been performed at a pressure of 100 Torr, a temperature of 620 C and using hydrogen as carrier gas. As source gases PH_3 and TMIn have been utilised, and as Fe source ferrocene has been used. A typical growth rate of 1.35 µm/h is employed for an input V/III ratio of 130. The above conditions are chosen having a subsequent regrowth in mind, i.e. at a growth pressure of 100 Torr the selectivity should be high and at a growth temperature of 620C the Zn−Fe interdiffusion kept relatively low. Besides, operating at low pressure is an advantage regarding reactor maintenance, where even after more than 500 growth runs no cleaning of the showerhead has been necessary.

For resistivity evaluation n−InP / InP:Fe / n−InP structures have been grown and processed to circular diodes. Temperature dependent I−V measurements were undertaken between 20 and 80 C. The Fe incorporation was assessed by SIMS measurements and C−V profiler measurements on codoped InP:Sn,Fe structures. The Fe concentration was varied by changing the ferrocene input partial pressure while keeping all other growth parameters constant.

For regrowth experiments, ~2.5 µm high mesa stripes were prepared by RIE and a short wet−chemical etch, in order to create a mask overhang. The regrowth experiments have been carried out at various V/III ratios and growth rates with respect to the standard conditions. In addition, a change in wafer rotation from 50 to 125 rpm was examined.

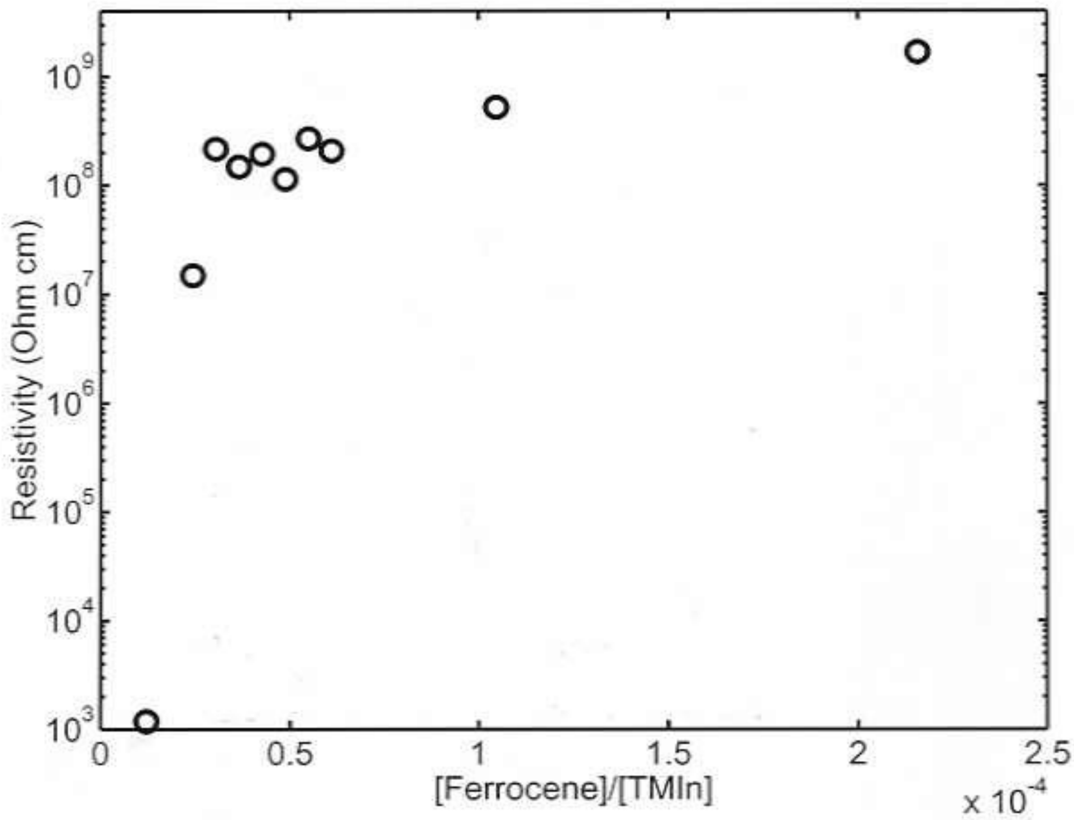

Fig. 1. The resistivity of InP:Fe layers as a function of ferrocene / TMIn mole fraction at a growth temperature of 620 C.

Results and discussion

In a series of experiments n−InP/InP:Fe/n−InP structures were grown for various ferrocene input partial pressures in order to determine the resistivity and the Fe concentration. In Fig. 1 the resistivity derived from I−V measurements is depicted for different [ferrocene]/[TMIn] ratios. As can be expected there is initially an abrupt increase in resistivity corresponding to a total Fe concentration change from < $1x10^{16}$ cm^{-3} to around $1x10^{16}$ cm^{-3} as measured by SIMS. The ease in achieving highly resistive layers can be explained by the very low background doping concentration typically obtained in InP, on the order of $5-9x10^{14}$ cm^{-3}. The maximum resistivity obtained was found to lie around $1x10^{9}$ Ohmcm, which is sufficient for use in devices requiring SI blocking layers. In addition, from the temperature dependence of the I−V measurements an activation energy of ~ 0.67 eV is derived indicating the appearance of the deep Fe acceptor.

In order to assure that all Fe incorporated is activated a study of codoped InP:Sn,Fe structures has been carried out. By performing polaron carrier profiles on InP:Sn layers with an intermediate Sn−Fe codoped layer the activation of Fe can be derived from the difference Δ n in carrier concentration with and without Fe doping. A typical C−V profile is shown in Fig. 2, where a Fe activation of $4.5x10^{16}$ cm^{-3} has been obtained, which also corresponds with the value measured by SIMS. At the given growth temperature of 620C the electrical activation seems to saturate for higher Fe concentrations, which is in agreement to the maximum electrical activation (~ $5-6x10^{16}$ cm^{-3}) for MOVPE grown InP:Fe (obtained by C−V profiling) as reported by Knight et al. [6] and Robein et al. [7].

A few experiments on regrowth of InP:Fe around 2.5 µm high mesa structures have been accomplished. In Fig. 3 the SEM picture of the regrowth of 2 periods of InP:Fe/InP:Sn around a [110] oriented laser mesa is shown. The growth rate was varied between 0.7 and 1.35 µm/h and the input V/III ratio changed from 260 to 130 without showing any difference in regrowth behaviour.

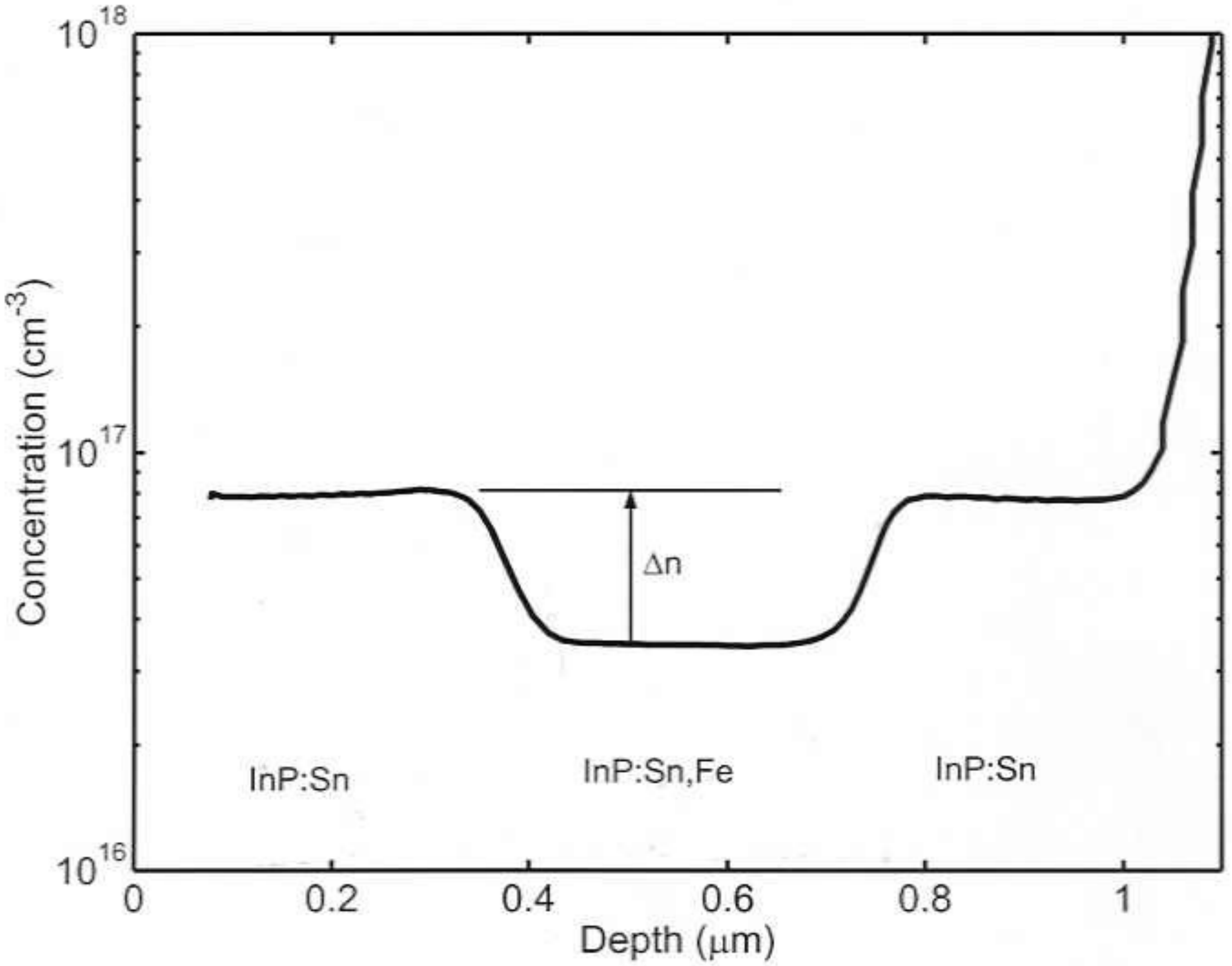

Fig. 2. C−V carrier concentration profile (Polaron) of a InP:Sn / InP:Sn,Fe / InP:Sn structure. In this case the Fe activation is ~ 4.5×10^{16} cm^{-3}, where SIMS gives a value of 4.9×10^{16} cm^{-3}.

In addition, the wafer rotation speed was varied between 50 to 125 rpm without observing any differences. At all conditions no problem of selectivity or mask overgrowth is apparent and the planarisation is about 2µm on each side of the mask. This show on the stability on the growth parameters in such a reactor, provided that mesas are prepared in a proper manner.

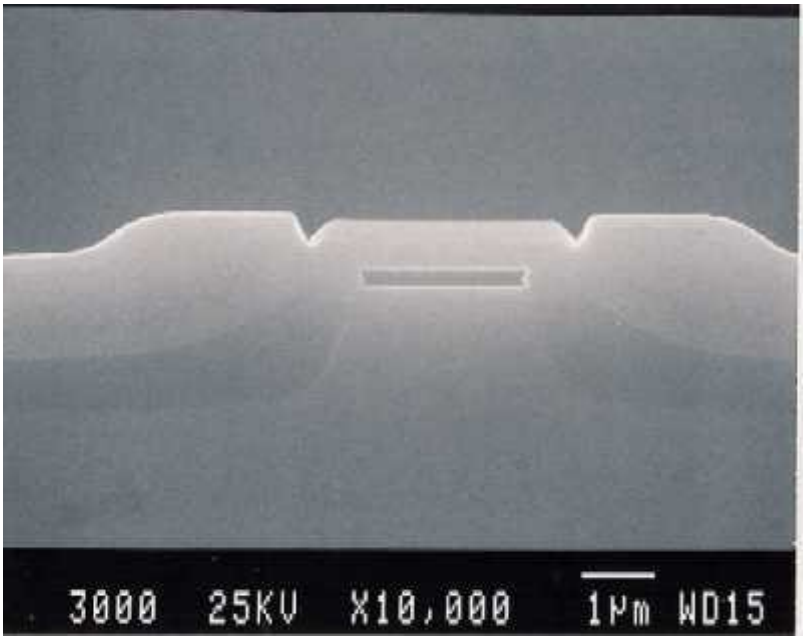

Fig. 3. SEM micrograph after regrowth of 2 periods of InP:Fe / InP:Sn around a 2.5 µm tall laser mesa.

Summary and conclusion

We have demonstrated on the capability of growth of highly resistive semi−insulating InP:Fe and making regrowth around relatively high mesa structures in a production type close−spaced showerhead MOVPE reactor. The stability of the process has been pointed out by changing the growth conditions without observing altering in growth behaviour.

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