Effect of interfacial dopant layer on transport properties of high purity InP

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Temperature dependent Hall measurements were used to investigate the role of unintentional interfacial dopant layers on the electrical properties of high purity InP. Secondary ion mass spectroscopy was used to measure the level of Si contamination present in several samples at the substrate-epilayer interface. We show that the presence of interfacial dopant gives rise to two layer conduction whose temperature dependence mimics the freezeout behavior expected for a deep donor. However, the magnetic field dependence of the Hall data at low temperatures shows the expected behavior for the two layer model, including an order of magnitude variation in the apparent sheet concentration and Hall mobility over the range from 0.1 to 0.6 T at 77 K. We show that the true bulk mobility and carrier concentration can be accurately determined in samples with high interfacial contamination by performing Hall measurements at several magnetic fields at 77 K. © *1996 American Institute of Physics.* [S0003-6951(96)03214-4]

Control of residual impurities is a key issue in the fabrication of compound semiconductor devices based on GaAs and InP. In recent years the technique of metalorganic chemical vapor deposition (MOCVD) has achieved great success in reducing impurity incorporation to extremely low levels. Hall methods are often a primary characterization tool giving valuable information regarding the cleanliness of the reactor and the source chemicals. In this letter, we indicate an important pitfall of performing such Hall measurements at one value of the magnetic field. We show that the presence of an interfacial dopant layer at the epilayer-substrate boundary can give rise to anomalous Hall data, which can lead to several wrong conclusions regarding the electrical properties of the bulk material. Interfacial dopant layers have serious detrimental implications for devices, especially if regrowth is required. Devices which depend upon a high purity buffer layer, such as high electron mobility transistors (HEMTs), field effect transistors (FETs), and metal-semiconductor-metal (MSM) photodetectors are especially sensitive to impurity spikes at the epilayer-substrate interface. For example, impurity spikes in HEMTs or FETs can cause difficulties in obtaining good pinch-off characteristics resulting in inferior noise properties. In this letter, we show that a proper analysis of Hall measurements can be used to assess the degree of interfacial contamination of epitaxial layers as well as providing a technique for measuring the true bulk electrical properties.

The role of two carrier or two layer conduction in interpreting Hall measurements has been reviewed by Stradling.[1] Intrinsic two layer conduction is commonly observed in narrow gap semiconductors such as InAs which suffer from an accumulation layer at the surface due to the lineup of the surface levels with respect to the conduction band minimum. A semiclassical model for the general case of two parallel conducting sheets is presented in Ref. 1. This model assumes a Hall factor of unity. In the case of two conducting layers 1 and 2 the apparent sheet concentration and Hall mobilities are given by

$$n_t=\frac{(n_1\mu_1+n_2\mu_2)^2+(\mu_1\mu_2)^2(n_1+n_2)^2B^2}{n_1\mu_1^2+n_2\mu_2^2+(\mu_1\mu_2)^2(n_1+n_2)B^2}, \tag{1}$$

$$\mu_t=\frac{n_1\mu_1+n_2\mu_2}{n_t}, \tag{2}$$

where μ_i and n_i are the electron mobility and sheet concentrations for each of the two layers. An important prediction of these expressions is a very strong variation in n_t and μ_t with magnetic field if there is a large difference between μ_1 and μ_2. These expressions have been applied with success to the case of the narrow gap material InAs.[2,3] In the present work we examine a qualitatively similar situation of high technological importance involving the presence of a well-characterized Si impurity layer at the substrate-epilayer interface during growth of high purity epitaxial InP on semi-insulating InP. In our case one of the layers is the very high mobility bulk InP epilayer, the other is the unintentional δ-doped layer containing Si which is often observed by SIMS measurements at the epilayer-substrate interface.[4,5] In the present work we look at the magnetic field dependence of the Hall data for such a system, in conjunction with SIMS measurements, and show how quantitative information regarding the properties of both the bulk and interface layers can be obtained.

Growth of InP was conducted using low pressure MOCVD at a pressure of 75 Torr and a temperature of 630 °C. Palladium purified hydrogen was used as the ambient gas, with phosphine and trimethylindium precursors at a V:III ratio of 230:1. All samples were grown under identical growth conditions using whole 50 mm diam wafers of (100) oriented Fe-doped InP, where 4.0 μm of undoped InP was grown at a rate of 1.2 μm/h. Substrate preparation was identical in all cases and included immersion in 5:1:1 H_2SO_4:H_2O_2:H_2O.

Measurements of the sheet concentrations of Si and O were determined by secondary ion mass spectroscopy (SIMS), using a Cameca IMS 4*f* microanalyzer with a Cs^+ primary beam source. The detected ions were ^{28}Si and ^{16}O

TABLE I. Hall data for three different 4 μm samples taken at a field of 5000 G together with SIMS concentrations of Si and O at the substrate epilayer interface.

Sample no.	n(293 K) (cm $^{-2}$)	μ(293 K) (10^3 cm^2/V s)	n(77 K) (cm^{-2})	μ(77 K) (10^5 cm^2/V s)	SIMS[SI] (10^{12} cm $^{-2}$)	SIMS[O] (10^{12} cm $^{-2}$)
1	5.7×10^{10}	4.9	4.8×10^{10}	1.6	0.40	~0
2	9.1×10^{11}	2.7	3.0×10^{11}	0.37	5.3	8.6
3	1.2×10^{12}	2.4	3.4×10^{11}	0.27	7.1	6.0

negative secondary ions, calibrated using a Si implanted standard. The oxygen sheet concentration is estimated. Hall measurements were performed using the van der Pauw method. A Hall factor of unity was assumed in all calculations.

Table I shows a brief summary of transport and SIMS data for three samples reported in this study. All samples were grown under similar conditions. Hall measurements of sample 1 show a significantly lower apparent sheet concentration than samples 2 and 3. However, low-temperature photoluminescence measurements show no appreciable difference between the samples, with very sharp donor bound exciton transitions,[6] and in addition there is no appreciable difference between the bulk SIMS data for the three epilayers. The 77 K mobility of samples 2 and 3 are significantly lower than for sample 1. The lower mobilities and higher apparent bulk concentrations of samples 2 and 3 correlate well with a high level of interfacial Si and O contamination as measured by SIMS. An interesting feature is the large drop in the apparent bulk concentration as the measurement temperature is decreased from 293 to 77 K, suggesting at first sight some form of deep donor freezeout.

Figures 1(a) and 1(b) compare the measured sheet concentrations of a sample without interfacial contamination (sample 1) and with interfacial contamination (sample 3). Sample 1 shows the typical freezeout behavior observed for high purity bulk InP. The observed activation energy of ~6.8 meV obtained for the 100 G data agrees reasonably well with the predicted effective mass donor binding energy of 7.31 meV. The temperature dependent sheet concentration data for sample 3 are strikingly different, and are shown in Fig. 1(b). The data show a strong drop starting from 293 K, which is close to exponential in $1/T$ down to 100 K. This would appear to indicate some sort of freezeout behavior with an activation energy of around 31 meV. However, at temperatures below 10 K, the sheet concentration increases again, back up to its previous room-temperature value. In addition, a very strong dependence of the sheet concentration on magnetic field is observed. The lack of freezeout at low temperatures is consistent with the high level of interfacial Si contamination measured by SIMS. The present data can be easily explained in a two layer picture in which the interfacial Si contributes a high sheet density, low mobility shunt layer. The low mobility layer suppresses the measured mobility and increases the observed sheet concentration compared with samples without an interfacial layer. The mobility of the interfacial layer will be essentially temperature independent. In contrast, bulk high purity InP has a strong variation in mobility with a maximum near 50 K. Thus, in the vicinity of this temperature, the conductivity of the bulk layer becomes high, resulting in a drop in apparent sheet concentration due to the fact that a large fraction of the electron current now flows through the high mobility layer.

The temperature dependent Hall mobility data for sample 1 are shown in Fig. 2(a). The behavior is very similar to previous reports of high purity bulk InP. A very small dependence on magnetic field is observed for this sample as expected for a single carrier, single layer material. In contrast, the Hall mobility data for sample 3 show a very strong dependence on magnetic field. The discrepancy between the two fields is largest at temperatures between 10 and 100 K where the bulk mobility is largest. The mobility data at the lower magnetic field are also in close agreement with the mobility data for the layer without interfacial contamination. As will be shown below, this indicates that the bulk properties of the two layers are very similar.

The 77 K magnetic field dependence of the carrier concentration for samples 1 and 3 is shown in Fig. 3. The high purity sample without the interfacial layer (open symbols)

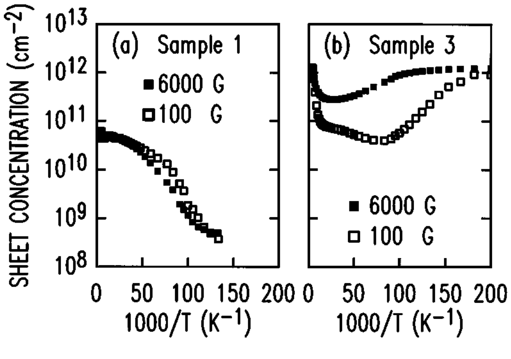

FIG. 1. Sheet concentrations vs temperature at two different magnetic fields for a sample with (a) low levels of interfacial Si and O and (b) high levels of interfacial Si and O.

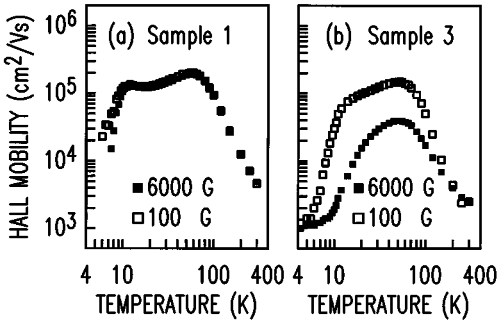

FIG. 2. Hall mobility vs temperature for samples without interfacial contamination (a) and with interfacial Si, O contamination (b).

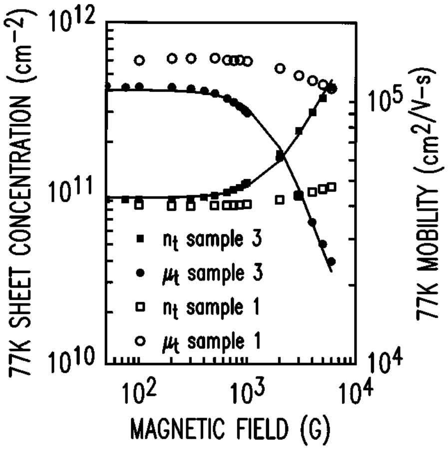

FIG. 3. 77 K Hall mobility and sheet concentration as a function of magnetic field for samples 3 (solid symbols) and 1 (open symbols). The fits to sample 3 which has interface contamination, are based on the two layer conduction model described in the text.

shows a small variation over the field range 100–6000 G which is likely explained by a small field dependence of the Hall factor, r_H at this temperature. The low field limit of the Hall mobility is 1.6×10^5 cm^2/V s confirming the excellent quality of this material. In contrast, sample 3 shows a very strong field dependence in both the mobility and sheet concentration of nearly an order of magnitude (closed symbols). This variation is much too large to be accounted for by Hall factor variations. The solid curve in Fig. 3(b) represents a least-squared fit to the data based on expression (1). The bulk sheet concentration, bulk mobility, and surface mobility were used as adjustable parameters, while the sheet electron density from the Si interface layer was estimated from the low temperature limit of Fig. 1(b) to be 1.0×10^{12} cm^{-2}. Using this value, we obtained values for the bulk sheet concentration and mobility of 5.0×10^{10} cm^2 and 1.5×10^5 cm^2/V s, respectively. The fitted surface sheet mobility of 2.9×10^3 cm^2/V s is somewhat higher than the low-temperature value of $\sim1\times10^3$ cm^2/V s obtained from Fig. 2(b), and may indicate a small temperature dependence of this quantity. The bulk transport values for this sample agree very well with those obtained for sample 1, which has negligible interface contamination. This is proof that the bulk purity of the two layers is essentially identical, and that the strikingly different Hall properties are the result of the interfacial layer in sample 3. This is also consistent with the lack of a difference between the PL spectra of the two samples.

The analysis presented here indicate that meaningful estimates of the bulk mobility and carrier concentration can be obtained even in samples with high interfacial donor contamination, provided the magnetic field is below 500 G. Most routine Hall measurements are performed at a single field of around 2000–5000 G, in which case serious errors can arise regarding the bulk properties of epitaxial films.

The value for the Si donor density determined from the Hall analysis is almost an order of magnitude lower than that obtained by SIMS. Similar discrepancies are commonly observed for activation of bulk donors. In addition, the presence of high levels of oxygen at the interface may have the effect of compensating some of the Si donors. Finally, the semi-insulating substrate certainly should deplete some of the electron density from the interfacial layer.

We have identified two conditions under which interfacial Si and O contamination of the type reported here occurs. The two contaminated samples 2 and 3 were grown in the presence of a very small leak in the inlet manifold to the reactor. Correction of this problem immediately resulted in the elimination of the interfacial layer in subsequent samples. We have also observed similar contamination effects if the wafers were exposed to ambient air for long periods of time prior to growth.

In conclusion, we have shown that Hall measurements are capable of providing detailed information regarding the presence and sheet density of unintentional interfacial electrical impurities in epitaxial growth. With simple field dependent Hall measurements it is possible to extract both the bulk and interface carrier densities and mobilities in epitaxial semiconductors. Without a careful analysis of the field dependence of Hall data, it is possible to arrive at erroneous conclusions regarding bulk purity levels, including the observation of fictitious deep donors.

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