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Enhancement of p-GaN Conductivity Using PECVD SiO_x

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A technique to enhance the hole concentration in activated Mg-doped p-type GaN epitaxial layers is described. The method consists of depositing a porous plasma-enhanced chemical vapor deposited SiO_x layer on top of p-GaN after which the sample is heated to 950°C in nitrogen ambient for 1 min followed by the removal of the SiO_x layer in a buffered HF solution. A significant improvement of the conductivity of the p-GaN layer has been obtained.

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Magnesium-doped p-type GaN layers are used as contact layers in blue, green, and ultraviolet light emitting diodes (LEDs) as well as in blue and UV laser diodes. A low dopant efficiency can be expected since Mg is a deep acceptor in GaN at ~300 meV. Conductivity might be further reduced in metallorganic vapor phase epitaxy (MOVPE) grown p-GaN as Mg atoms are bonded to H atoms incorporated during the growth process leaving the acceptors electrically inactive.[1] An activation treatment to break the Mg-H bond and dispel the hydrogen from the lattice is hence required. Multiple heating steps could improve the hole concentration by 50%.[2] This consists of combining a heat step at 350 to 700°C for a few minutes plus a short pulse to a high temperature. We have tried this method on our p-GaN by comparing the hole concentrations of two samples, one annealed at 900° for 30 s while the second sample was heated at 600°C for 10 min in which three short pulses at 900°C for 10 s were incorporated. The hole concentrations remain quite similar in both cases. Varying the pulse time also did not improve the result. On the other hand, it is well known that plasma etching of GaN leads to nonstoichiometric surfaces because of the faster desorption of N atoms as compared to Ga atoms. The nitrogen vacancies act as donors and hence influence the doping level of GaN layers.[3,4] By contrast, one would expect that Ga vacancies would act as acceptors and hence improve the conductivity of p-GaN epilayers. In this paper we present an original method for improving the p-type conductivity of Mg-doped GaN which involves capping p-GaN epilayers with plasma-enhanced chemical vapor deposition (PECVD) SiO_x layers and the subsequent anneal treatment at high temperature to generate a large number of Ga vacancies that act as acceptors.[5] The role of the SiO_x layer was confirmed using two identical samples, one as-grown and the second covered with a 300 nm PECVD SiO_x layer. Both samples were submitted to a rapid thermal anneal (RTA) step at 950°C for 30 s in N_2 ambient. Hall measurements show typical hole concentrations of $8\text{-}10 \cdot 10^{17}\ \text{cm}^{-3}$ and a mobility of $3.6\ \text{cm}^2/\text{V s}$ for the sample covered with SiO_x against $3\text{-}4 \cdot 10^{17}\ \text{cm}^{-3}$ and $11\ \text{cm}^2/\text{V s}$ for the sample without the oxide layer.

Experimental

A detailed investigation of the SiO_x role was carried out using GaN epilayers grown on the *c* plane sapphire substrates in a 6 × 2 in. MOVPE reactor based on the close-coupled showerhead principle. The GaN samples consisted of a 2 μm thick undoped GaN layer with high resistivity ($>10^8\ \Omega$ cm) capped with a 1 μm thick Mg-doped layer. The Mg doping concentration was varied between 8×10^{18} and $4 \times 10^{19}\ \text{cm}^{-3}$ according to secondary ion mass spectroscopy (SIMS) measurements by altering the flow rate of the Mg precursor, Bis-cyclopentadienyl magnesium (Cp2Mg) relative to that of the Ga precursor, trimethyl gallium (TMGa). The carrier gas during growth was hydrogen, although the Mg-doped capping layer in some samples were grown in a 1:1 mixture of nitrogen and hydrogen gas. Following the growth, the samples were subjected to an anneal treatment in the MOVPE reactor at 800°C in nitrogen for 20 min, and these are designated hereafter as activated samples. Subsequently, the activated samples were submitted to an additional anneal technique; they are capped with a 300 nm thick layer of silicon oxide deposited by PECVD at 300°C and subsequently annealed in a RTA machine at 950°C for 1 min in a nitrogen ambient after having capped the sample with a sacrificial sapphire substrate to avoid nitrogen out-diffusion. These samples are referred hereafter as RTA-annealed samples. After the RTA anneal treatment the SiO_x layer is removed in a buffered hydrofluoric acid (BHF). Hall measurements were carried out on both sets of samples to determine the holes concentration as well as the hole mobilities. The samples exhibit a larger hole concentration after the RTA anneal with the largest improvement at the lower end of the Mg doping range, i.e., with $[\text{Mg}] < 10^{19}\ \text{cm}^{-3}$, up to a carrier concentration of $4 \times 10^{17}\ \text{cm}^{-3}$. Moreover the hole mobility did not suffer from the treatment and remains constant at about 10-12 $\text{cm}^2/\text{V s}$. The results of the Hall measurements are shown in Fig. 1 and 2. It is seen that the quantitative improvement after the SiO_x/RTA treatment is dependent on the material source and can vary significantly. However our method worked successfully on p-GaN from four different sources. Only in one material source the improvement of holes concentration was accompanied with a mobility decrease.

Discussion

A significant enhancement of the conductivity of *in situ* annealed p-type GaN layers is observed after the combined SiO_x PECVD/RTA treatment. This superior conductivity is the result of an improvement in the hole carrier concentration while the hole mobility remains constant suggesting that the crystalline quality of the GaN layer is not degraded by the RTA treatment. It has also been shown that the SiO_x layer plays an important role in achieving this.

We believe that the p-conductivity enhancement is due to the formation of Ga vacancies in the upper GaN layer. The PECVD SiO_x and the subsequent RTA treatment introduce Ga vacancies in the top GaN layer. These Ga vacancies behave as acceptors and hence are responsible for the established improvement in holes concentration. The PECVD SiO_x combined with an RTA step is a well-known method in the literature[6-8] commonly used to create intermixing of GaAs quantum well (QW) structures. The intermixing occurs as annealed SiO_x adsorbs Ga from the semiconductor interface and the annealing drives the Ga vacancies down in the structure resulting in the migration of Al atoms from the AlGaAs barrier layer into the QW causing a blue shift of the characteristic wavelength of the QW. Moreover, following Deenapanray *et al.*[9,10] the quality of the SiO_x layers is essential to allow the adsorption of Ga atoms and hence the

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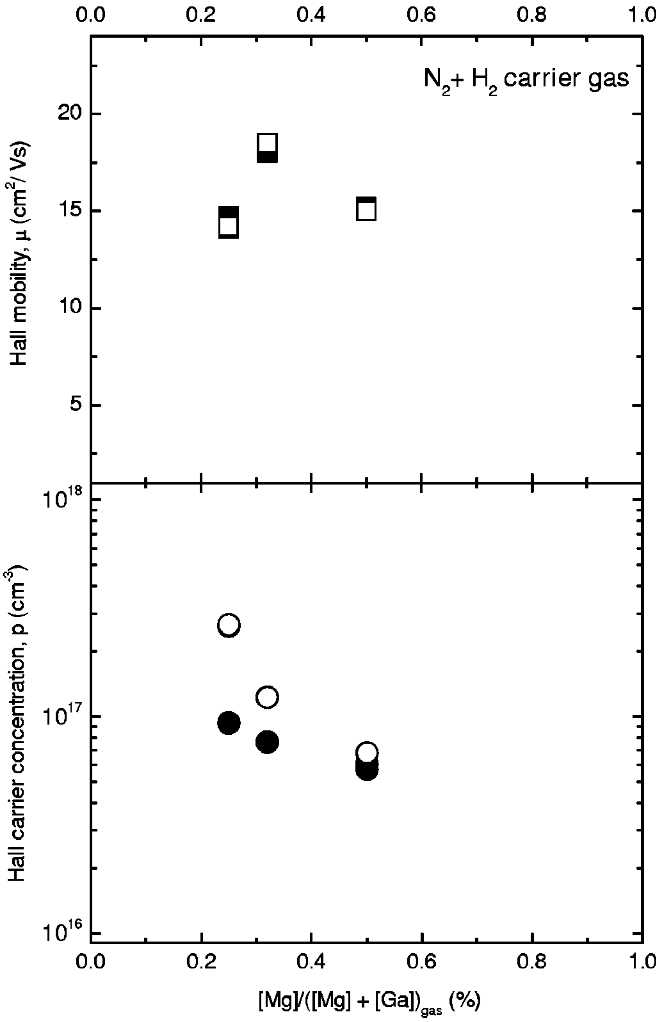

Figure 1. Mobility (top) and hole concentration (bottom) of RTA-treated samples (open symbols) and activated samples (closed symbols) using N_2 + H_2 carrier gas.

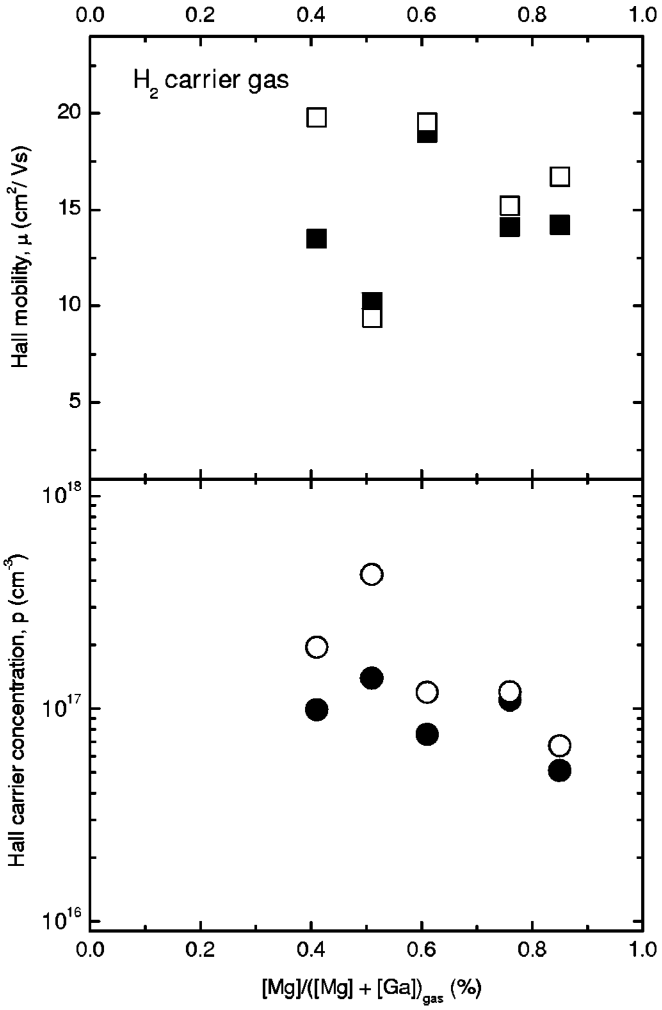

Figure 2. Mobility (top) and hole concentration (bottom) of RTA-treated samples (open symbols) and activated samples (closed symbols) using H_2 carrier gas.

intermixing: the more porous the SiO_x layer, the higher the intermixing effect. The quality of the PECVD SiO_x used has been tested, and it is found that a BHF solution removes the SiO_x layer in a few seconds. This reveals that the SiO_x layer used in our special treatment is rather porous and hence is capable of adsorbing the Ga atoms from the top p-GaN layer resulting in the improvement of the conductivity.

From the literature we trace one patent describing the use of a similar technique on p-GaN in order to avoid nitrogen out-diffusion.[11] However the samples with and without a SiO_x layer lead to the same hole concentrations of 2×10^{17} cm^{-3} and hence the SiO_x layer did not improve the conductivity of the p-GaN layer. This would suggest that the SiO_x layer used in that work[11] was not sufficiently porous to introduce Ga vacancies.

Conclusions

We have demonstrated a significant improvement of the conductivity of MOVPE-grown p-GaN epilayers by applying a SiO_x layer followed by an RTA step at 950°C for 1 min in nitrogen ambient. This postgrowth technique can be easily performed to p-GaN layers for improving the conductivity of p-GaN layers, and hence it is very important for the advancement of GaN-based LEDs and lasers.

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