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P-type Doping of Broad-band Nitride Semiconductors

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Abstract: *GaN-based optoelectronic devices have been an important development direction for semiconductors and have been applied in several fields. P-type GaN thin film implementation is the core process for optoelectronic devices, and a lot of breakthrough results have been achieved in p-GaN research. For example, Si and Mg are used as the main doping elements. However, more effective doping of GaN materials is needed in order to make GaN materials play a greater electrical and optical advantage. In this paper, we take p-GaN as the main object of study and outline the conditions that need to be satisfied for effective doping of GaN materials. Emphasizing several factors that make the quality of p-type materials of GaN a bottleneck for application development, the results achieved in p-GaN research in recent years are presented.*

Keywords: *Semiconductors, Gan Material, P-type Doping*

1. Introduction

In the past 10 years, GaN-based optoelectronic devices have made important progress and are widely used in lighting, projection, display, and medical fields.[1-2] For GaN-based devices to give full play to their excellent electrical and optical properties in the application process, in addition to the good quality of the GaN material itself, it is necessary to effectively dope the GaN material, especially in lasers, LEDs, photodetectors and in light-emitting devices such as solar cells.[3] Theoretically, to achieve effective doping of GaN materials, the following three conditions must be met: (a) The radius of the doped impurity atoms is close to Ga atoms or N atoms to produce substitutional doping. This can prevent the dopant atoms from becoming interstitial atoms to a certain extent, distorting the crystal lattice and affecting the crystal quality of the GaN material. (b) The chemical bonds between the impurity atoms and the surrounding atoms must be sufficiently stable. Because the growth temperature of GaN-based semiconductor materials is usually relatively high, weak impurity bonds will make it difficult for impurity elements to be stably doped into GaN materials. (c) Within the allowable range, the doped impurities should have the lowest ionization energy to ensure that they can be effectively ionized at room temperature. At present, in terms of n-type and p-type doping of GaN materials, Si and Mg are relatively mature doping elements with higher doping efficiency.[4-10]

GaN-based devices require n-type GaN to provide sufficient n-type carriers (such as electrons). It is found that doping Si, Ge, Se and other elements in GaN can get n-type GaN. the activation energy of Si is relatively low about 20 meV, so people usually choose Si as n-type doping element, the dopant to provide Si doping element in practical application is SiH_4.[11-12] The carrier concentration in n-type GaN is proportional to the flow of SiH_4, and the current research n-type carrier concentration can reach (1-2) $\times 10^{19}$ cm^{-3}, and the surface of n-type GaN with high Si doping concentration is still smooth and mirror-like.[8, 13] At the same time, the study shows that GaN materials doped with appropriate Si impurities for the suppression of deep energy level luminescence and improve the intensity of band edge luminescence is also very beneficial.[8]

Unlike n-type doping, the only effective dopant for p-type GaN materials is Mg, and because the ionization energy of Mg in GaN is as high as 170 meV, its ionization probability at room temperature is only about 2%, for a long time, the p-type doping of GaN materials has been one of the main bottlenecks that hinder its application and development.[14] Research and improve the performance of p-type GaN to reduce the operating voltage of related devices, improve its luminous power, reduce the threshold current, etc. have very important significance.

Although the current stage of P-GaN research has achieved many breakthrough results, the quality of the current p-type material of GaN is still a major factor that restricts the performance of related devices, the following problems mainly exist.

2. Method

2.1. Formation of Mg-H complex by Mg-acceptor bonding[15-16]

As the growth of P-GaN by MOCVD equipment is an H-containing process, H atoms are easy to combine with Mg acceptors to form Mg-H complexes and passivate them. To obtain high-performance p-type materials, the Mg acceptors must be annealed and activated, and the most common way is to use rapid thermal annealing. However, in some devices, such as lasers with high In components in the active region, high growth temperature and annealing temperature will make the phase separation of In components in the active region, so low annealing temperature must be used to deal with p-type GaN, but the low annealing temperature will make the efficiency of Mg acceptor activation reduced.

2.2. High ionization energy of Mg in GaN[17-21]

As the ionization energy of Mg is as high as 170 meV, theoretically the Mg subject to ionization at room temperature to produce holes is only about 2%, so in order to obtain more than 1×10^{18} cm^{-3} hole concentration of P-GaN must be re-doped. However, when the Mg doping concentration is higher than a certain value, it will form self-compensation and lead to a lower hole concentration instead, so the Mg doping concentration cannot be too high, which makes it difficult to obtain a high hole concentration.

2.3. The compensation effect of impurities[22-25]

When using MOCVD for GaN material growth, due to its growth process there are C, H, O and other elements, so will be unintentionally introduced with the growth process of these impurity elements, where H will passivate the Mg acceptor, and C impurities in some literature reports indicate that will also compensate for the Mg acceptor. In particular, in some devices, the high growth temperature of p-type layer will affect the quality of the active region, and a relatively low growth temperature must be used to grow p-type GaN, but the low growth temperature will aggravate these unintentionally doped impurities, thus making its compensation effect intensify, and in the low temperature grown P-GaN, C impurities are likely to be the main source of compensation. At the same time, the N vacancies generated by the annealing process will also compensate for the Mg acceptor.

P-type GaN material has a low carrier concentration due to the passivation of Mg acceptor H, strong Mg self-compensation effect and other impurity compensation effects, which severely restricts the application and development of GaN material-related devices.[15] The development of P-type GaN materials is slower than other materials. It was not until 1989 that Amano et al. used low-energy electron beam irradiation (LEEBI) to irradiate Mg-doped GaN, and it was the first time that they obtained good electrical conductivity p-type GaN material.[6] However, the p-type conductivity obtained by this method is not uniform, and only a very thin layer on the GaN surface exhibits relatively good p-type characteristics, about 500 nm, so this annealing method is not in actual devices. Subsequently, in 1992, Nakamura et al. used rapid thermal annealing to anneal Mg-doped GaN, and for the first time obtained a p-type GaN material with good uniformity, and proposed the reason for the low hole concentration in the P-GaN material.[5, 22] It is due to the passivation effect of H on the Mg acceptor, and H can be released from the GaN material by thermal annealing to reduce this passivation effect, so that the Mg acceptor can ionize to generate holes, thereby making the p-type Improved performance. Since then, thermal annealing has become an indispensable process for MOCVD growth of p-type GaN materials. Since then, high-brightness blue and green light-emitting diodes, ultraviolet detectors, photodetectors, etc. have been rapidly developed, began to enter the commercial market.[26-27] In general, the achievements in P-GaN research in recent years can be roughly divided into the following categories:

2.4. Annealing activation in different atmospheres[28-41]

Hull and Chung et al. studied the role of O_2 in the annealing atmosphere during P-GaN annealing. Among them, the latter compared the effect of annealing in N_2 atmosphere with annealing temperature of 850 , annealing time of 20 min, and different oxygen content in the study. It is found that when the content of O_2 in the annealing atmosphere is 1%, the P-GaN film exhibits the best performance after annealing, and it is proposed that when the oxygen content is higher, the donor-type ON will be formed in the GaN film, which is more effective for Mg Compensation by the acceptor will degrade the performance of the P-GaN film instead. Their research pointed out that the presence of O_2 is beneficial to the desorption of H from the GaN epitaxial layer, because the presence of O_2 may form H_xO_y molecules (such as H_2O) with H atoms. Similar conclusions are further confirmed in the research results of D.B.Li and L.L.Wu et al.

Nakagawa et al. also studied the annealing of P-GaN films under N_2 and O_2 atmospheres, and replaced the H in the NH3 atmosphere of the epitaxial layer cooling process with deuterium (D). From the SIMS test results, it is found that H and D in the P-GaN film exhibit two different properties, and therefore it is speculated that there are two different mechanisms for hydrogen in the activation process of the Mg acceptor. The first type requires relatively large energy (0.8-1.5 eV), and the second type requires relatively small energy (0.2-0.5 eV). The former is more closely related to the passivation of the Mg acceptor, while the latter is related to the acceptor's passivation. The electrical compensation effect is more relevant.

Lin et al. studied the activation mechanism of P-GaN films in the air. It is found in the experiment that the increase of the hole concentration in the GaN film is mainly due to the decomposition of the (Mg_{Ga}-H) complex, the formation of (V_{Ga}-H2), and the movement of interstitial Mg atoms to Ga vacancies (V_{Ga}) during the annealing process. And from its PL spectrum, it is found that (V_{Ga}-H_2) complexes will be formed in the P-GaN film when annealing in air. (V_{Ga}-H_2) can promote the decomposition of (Mg_{Ga}-H) complexes and increase the H content in GaN. The desorption rate is therefore conducive to the increase of the hole concentration in GaN.

2.5. Cover metal annealing activation[42-45]

In 2001, Waki et al. proposed for the first time that the annealing of a metallic Ni overlay can effectively activate P-GaN films at low temperatures, and explained the related activation mechanism. They vapor-deposited a 1.5 nm thick Ni layer on the surface of the P-GaN film, and then annealed the sample at a temperature of 200 ℃ to 800 ℃. In the experiment, it is found that the activation effect can be achieved after annealing at a low temperature of 200 ℃ after using the Ni cover layer. Then, the H concentration in the GaN film after the Ni layer is removed is tested with SIMS, and it is found that the H concentration is different from the untreated one. Compared with the sample, there is a significant decrease. The explanation they put forward is that the Ni cladding layer can strengthen the desorption of H atoms in the GaN film and weaken the passivation effect of H on the Mg acceptor. At the same time, they found that when the annealing temperature is increased to 800 ℃, the performance will be worse. The possible reason is that the Ni coating layer at high temperature will aggravate the decomposition of the P-GaN film surface, resulting in a large amount of V_N on the surface of the P-GaN film. V_N will compensate the Mg acceptor, making its performance worse. Subsequently, some other research groups successively used different H storage metals such as Pd, Co, Pt, Mo, etc. as the covering layer for research, and found the same phenomenon. Although their research methods are different, they have caused changes in their performance. The mechanism is roughly the same.

2.6. Other activation methods[46-52]

Eaton's group and Miyachi's group studied the effect of Minority-Carrier injection on the activation of Mg-doped GaN. The former study found that when there is a minority carrier implantation, thin, lightly doped GaN can be annealed at 175 ℃ to achieve the effect of acceptor activation, while thick, heavily doped GaN needs to be at a high temperature above 700 ℃ to activate the acceptor for effective activation, it is proposed that the thin, lightly doped P-GaN activation process satisfies a quadratic dynamic process. The SIMS test of the heavily doped sample found that the minority carrier injection activation only destroyed the (Mg-H) complex, but the H did not leave the P-GaN film. When the sample activated by the minority carrier injection was thermally annealed again, the Mg acceptor will be passivated again.

Kim et al. studied the influence of 248 nm krypton fluoride (KrF) pulsed excimer laser irradiation on the activation of Mg acceptors. They found that when the P-GaN film grown by the MOCVD method was irradiated at an energy density of 590 mJ/cm^2 in an N_2 atmosphere, only a hole concentration of $4.42\times10^{17}/cm^3$ could be obtained. The laser irradiation method is applied to the sample after rapid thermal annealing at 950 ℃ for 1 min, and it is found that the hole concentration of the sample can be increased to 9.42×10^{17}/cm^3 at an energy density of 420 mJ/cm^2. However, the use of this subsequent laser irradiation method requires a layer of SiO_2 film to be grown on the P-GaN surface to prevent laser irradiation from damaging the GaN surface. Although the laser irradiation method can also achieve selective area activation, its process is complicated and the effect is not very satisfactory. There are also the Y.C.C group, Lin group, Wang group and so on that use similar research methods.

S. J. Chang et al. studied the effect of treatment on the activation of Mg-doped GaN. They annealed the Mg-doped GaN samples under 2.45 GHz and 560 W conditions and did different treatment time experiments. At the same time, they made a set of conventional thermal annealing samples with an annealing temperature of 730 ℃ and an annealing time of 20 min as a control. The test results of PL and Hall show that microwave processing is a more effective way to activate P-GaN. Moreover, good results can be obtained after microwave treatment for 5s, but longer treatment cannot significantly improve the conductivity of P-GaN. The principle of Mg acceptor activation is that the H atoms of P-GaN absorb microwave energy and promote the decomposition of the Mg-H complex.

H. Lee and M. Oh et al. studied the activation of P-GaN by the electrochemical constant pressure method. Their experimental procedure was as follows: the P-GaN sample was first activated by conventional thermal annealing in N2 atmosphere at a temperature of 650 ℃, and then P-GaN with metal in dots soldered on the surface of the sample as the anode and metal Pt as the cathode was placed in a 0.5 mol/L KOH solution, kept constant for 4 min, and only the voltage (0-10 V) added between the two electrodes was changed. The results showed that the maximum hole concentration in P-GaN was increased from 1.9×10^{17} /cm^3 at 0 V to 4.5×10^{17} /cm^3 when the voltage between the two electrodes was 3 V. The SIMS test results showed that the H concentration in P-GaN did not decrease significantly at 3 V, while at 7 V, the H concentration decreased more than two times. The reason for this may be that the 3 V voltage only decomposes the Mg-H complex while increasing the voltage promotes the migration of H in P-GaN. The activation mechanism of this method is to make the H in P-GaN leave GaN and combine with -OH in KOH solution to generate H_2O through electrolysis, thus achieving the effect of Mg subjected to the main activation and increasing the hole concentration in P-GaN.

2.7. Presence of compensation mode[25, 53-59]

Obloh et al. found by annealing experiments on samples with different Mg doping concentrations that when the Mg doping concentration reaches a certain level, the concentration of its vacancies generated by annealing activation decreases with increasing Mg concentration, and it was found by PL spectroscopy that there is a strong DAP luminescence peak, i.e., a strong compensatory effect, in the high Mg-doped samples. The analysis shows that in high Mg samples Mg will occupy the N vacancy and become a compensating donor to the Mg acceptor, which is the so-called Mg self-compensation effect. Their study shows that the strong self-compensation effect is significantly enhanced with increasing Mg concentration, and the general compensation effect starts at a doping concentration of 3×10^{19} /cm^3.

Yang et al. found by SIMS and Hall test that residual carbon impurities in GaN materials also compensate for the Mg acceptor, thus reducing the performance of p-type GaN. Their study points out that the C impurity concentration rises with decreasing growth temperature and can be a major factor limiting the performance improvement of p-type GaN in P-GaN grown at low temperatures, while their study points out that this compensating effect associated with C impurities works through the formation of C_N-O_N complexes.

At the same time, some studies have pointed out that when the annealing temperature exceeds 850 ℃ during the annealing process, it will make the P-GaN film surface decomposition and make the V_N increase, from their research shows that the formation of V_N also has a certain compensation for the Mg subject, so that the annealing temperature continues to increase but will reduce the performance of p-type materials.

3. Conclusion

At this stage, although the research of p-type GaN has achieved certain results, but because the effective element type available for doping is only Mg, it still faces many problems. For example, it is difficult to achieve the efficiency of the Mg element subject to primary activation and at the same time organize the Mg element not to combine with H atoms to form complexes, the doping concentration of Mg element affects the change of GaN hole concentration, the compensation effect of C, H, O and other elements, etc. At the same time, the results of the stages studied so far can be divided into annealing activation studies in different atmospheres, such as O_2, covering metal annealing activation studies, such as metal Ni, and other activation and compensation methods. But all in all, scientists still have a long way to go for the doping research of P-type GaN materials.

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