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Submitted to *RSC Advances*, Liu *et al.***Co-doping of Magnesium with Indium in Nitrides: First Principle Calculation and Experiment**Zhiqiang Liu<sup>1,2</sup>, Binglei Fu<sup>1,2</sup>, Xiaoyan Yi<sup>1,2</sup>, Guodong Yuan<sup>1,2</sup>, Junxi Wang<sup>1,2</sup>, Jinmin Li<sup>1,2</sup>, Luna<sup>3</sup>, Ian Ferguson<sup>4</sup><sup>1</sup>*Research and Development Center for Solid State Lighting, Institute of Semiconductors, Chinese Academy of Science, Beijing, 100086, China*<sup>2</sup>*State Key Laboratory of Solid State Lighting, Beijing, 100086, China*<sup>3</sup>*Lyles School of Civil Engineering, Purdue University, West Lafayette, IN 47907, U.S.A*<sup>4</sup>*College of Engineering and Computing, Missouri University of Science and Technology, 305 McNutt Hall, 1400 N. Bishop, Rolla, MO 65409, U.S.A*\*Corresponding authors: [lzq@semi.ac.cn](mailto:lzq@semi.ac.cn); [spring@semi.ac.cn](mailto:spring@semi.ac.cn);

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**Abstract**

In this work, an effective strategy for achieving efficient p-type doping in wide bandgap nitride semiconductors was proposed to overcome the fundamental issue of high activation energy. We demonstrated that a hole concentration as high as  $1.4 \times 10^{18} \text{ cm}^{-3}$  could be achieved through In-Mg co-doping. The electronic structure of the system and the formation energy of impurity were analyzed via first principle calculation to clarify the underlying physics and the ambiguity in understanding of the origin of the high hole concentration. Our results indicated that the original valence band maximum of the host materials could be modified, thus improving the p-type dopability. We showed that the calculated ionization energy  $\varepsilon(-/0)$  of acceptor is only about 135meV, which is much smaller than that of the isolated Mg acceptor.

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## 1. Introduction

As in most wide-band-gap nitride and oxide materials, GaN can be easily doped n type but is difficult to dope p type. It is called “unipolar” or “asymmetric” doping problem. The origins of the difficulty include low dopant solubility, hydrogen passivation, compensation effect.<sup>1-3</sup> Considerable efforts have been made to address the p-type doping issue in nitrides.<sup>4-8</sup> Recent advances in crystal growth technology have shown that the issues of low solubility and hydrogen passivation can, at least to some extent, be overcome by using non-equilibrium growth techniques and high-temperature annealing.<sup>9-10</sup> However, alleviating the more fundamental problem of high activation energies has, to date, not yet been satisfactorily achieved. Actually, the underlying physical mechanism of high acceptor activation energies is coming from the electronic structure of nitrides material. That is: nitrogen is strongly electronegative and has a deep  $2p$  atomic orbital. Thus, the valence band maximum (VBM) of nitrides, which contain mostly N  $2p$  orbitals, is at relatively low energies. This leads to a deep acceptor energy level which makes it very inefficient for thermal activation. To date, the activation energy of Mg, which is the most promising acceptor for GaN, is still in the range of 160 and 200 meV, consequently only a small fraction of Mg can be activated at room temperature.<sup>9-11</sup> It is, therefore, reasonable to believe that modulating the energy position of acceptor states related to the VBM of host materials should be a promising solution for high effective p type doping.

Based on this consideration, various approaches have been developed. One strategy is demonstrated by Peter and Schubert.<sup>12-13</sup> They found that by polarization induced modulation of the valence band edge in a superlattice, the low doping efficiency could be partially overcome. B. Gunning *et al.* proposed another strategy to lower the acceptor impurity states by extremely high doping.<sup>14</sup> They argue: as the electrically active acceptor concentration increases, the isolated deep acceptor levels begin to interact and split into an impurity band, which is closer to the valence band thus lowering the effective activation energy. Simon and Jena<sup>15</sup> also suggested that a 3D hole gas could be produced using the built-in electronic polarization in nitrides. Elevating the VBM of the host material by co-doping was regarded as another important strategy to address this issue,<sup>16-17</sup> for example by Si-Mg co-doping and other mutually passivated defect pair co-doping. However, later theoretical analyses have shown that this type of energy level coupling is too small to significantly reduce the acceptor ionization energy due to different wave-function characteristics.<sup>18</sup> Therefore, although partial successes have been achieved, the mechanisms of those methods are still controversial and poorly understood. Better approaches or alternative strategies to create more stable and shallower acceptors in nitrides are highly desired.

Isoelectronic doping with In has long been used to eliminate defects and also shown its potential in enhancing nitrides hole concentration.<sup>19-22</sup> However most of these works are based on experimental observation and from the perspective of suppressing compensation effect. There still no consensus on the underlying physics of the acceptor ionization process. In this work, an effective method for achieving efficient p-type doping in high bandgap nitride semiconductors by In-Mg co-doping has been proposed. First principle calculation was performed to clarify the ambiguity in the understanding of the underlying physics in these co-doping systems. Furthermore the effective p-type doping is demonstrated experimentally.

## 2. The band-structure and the total energy calculation

The band-structure and the total energy calculation of In-Mg co-doped GaN are performed using the projector-augmented-wave method with generalized gradient approximations, as implemented in the Vienna ab initio simulation package.<sup>23</sup> Here, the generalized gradient approximations (GGA) of Perdew-Burke-Ernzerhof (PBE) functionals are used for the exchange correlation potential.<sup>24</sup> All the atoms are fully relaxed until the Hellmann-Feynman force is less than 0.03 eV/ang. The cutoff energy is chosen to be 800 eV. A reciprocal space k-point mesh of 5 x 5 x 5 is employed.

Wurtzite GaN structures with In-Mg co-doping were examined using 2 x 2 x 2 supercell models. Two Ga atoms are substitute by In and Mg randomly. Actually, we have tried several different configurations. The calculation results (transition energy and densities of states) did not change too much. For simplicity, in this work only the results of the most energetically favorable configuration, in which In and Mg atom aligned parallel to the *c* axis is presented.

## 3. Experimental

Samples in this study were grown on 2 inch (0001) sapphire substrates by a Veeco 3x2 inch metal organic chemical vapor deposition (MOCVD). Trimethylindium(TMIn), trimethylgallium(TMGa), bis(cyclopentadienyl) magnesium (Cp2Mg) and ammonia (NH3) were used as precursors for In, Ga, Mg and N, respectively. The TMIn/Cp2Mg molar flow ratio is remaining at about 5. For the whole growth process, the carrier gas was hydrogen. Firstly, 30 nm GaN was deposited at the surface of sapphire substrate as the buffer layer. Then, a 1  $\mu\text{m}$  undoped GaN layers were deposited followed by 0.5 $\mu\text{m}$  In-Mg codoping GaN layer at 1050  $^{\circ}\text{C}$ . To suppress the formation of compensation center (nitrogen vacancies), a high V/III molar flow ratio of 10000 was used. The TMIn/Cp2Mg molar flow ratio is remaining at about 5. For comparison, conventional Mg-doped GaN was also prepared using the same grow parameters, which acts as the control sample. After the

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growth, the samples were annealed at 650 °C for 30 min in nitrogen to activate the Mg dopant. Secondary ion mass spectrometry measurements were performed with a Physical Electronics with a 6 kV 100 nA Cs<sup>+</sup> beam, at a detection angle of 450 to verify the incorporation and distribution of Mg and In atoms. Temperature-dependent Hall-effect measurements performed at a magnetic field of 0.5 T using the van der Pauw method. NiAu acts as the ohmic contact layer. High resolution x-ray diffraction (HRXRD) was performed to characterize the crystalline quality of the samples using a Philips X'Pert system.

#### 4. Results and discussion

To determine the defect transition energy levels, the total energy for a supercell containing In-Mg complex in charge state  $q$  ( $E(\alpha, q)$ ) and the same supercell of pure GaN ( $E(\text{host})$ ) were calculated. The Brillouin zone integration using special k-points<sup>25</sup> is consistent and can give good results on the total energy and converged charge density. However, this method can only give a poor description on the symmetry, energy levels of the defect state and VBM of system. Furthermore, the obtained results are sensitive to the selection of the k-points sampling. In this work, the hybrid scheme is used to combine the advantages of both the special k-point and gamma point-only approaches.<sup>25-26</sup> The transition energy level of Mg acceptor with respect to the VBM is obtained by:

$$\varepsilon(q/0) = [\varepsilon_D^\Gamma(0) - \varepsilon_{VBM}^\Gamma(\text{host})] + [E(\alpha, q) - (E(\alpha, 0) - \varepsilon_D^k(0))] / (-q) \quad (1)$$

where  $\varepsilon_D^\Gamma(0)$ ,  $\varepsilon_D^k(0)$  are the defect levels at the gamma point and at special k point (averaged);  $E(\alpha, q)$ ,  $E(\alpha, 0)$  are total energies for supercells with defect  $\alpha$  in the  $q$  charge state and in the neutral charge state, respectively;  $\varepsilon_{VBM}^\Gamma(\text{host})$  means the VBM energy of the supercell at the gamma point. The  $\varepsilon_D^\Gamma(0)$  and  $\varepsilon_{VBM}^\Gamma(\text{host})$  are eigenvalues in two different systems. Therefore, to get accurate descriptions of the VBM and defect transition energy levels it is very important to align the potential of defect cell with pure host, especially for shallow levels. Previous calculations have exhibited that the transition energy levels have been severely underestimated without the potential alignment in wide band gap material systems.<sup>27-28</sup>

Based on equation (1), we calculated the ionization energy of the isolated Mg acceptor in bulk wurtzite GaN, which is about 208 meV. It agrees well with previous both calculations and experimental observations.<sup>29-30</sup> For comparison, the ionization energy  $\varepsilon(-/0)$  of In-Mg acceptor is only 135 meV, which is about 70 meV shallower.

To understand the underlying physical mechanism of In-Mg doping, projected densities of states (DOSs) were analyzed to exam the orbital hybridization between doping atoms and the host matrix. As shown in Figure 1, several peaks of the In  $t_{2d}$  states overlap with that of N  $t_{2p}$ , which indicated the strong coupling between them.<sup>31-32</sup> It is because: in the environment of the GaN matrix, In  $4d$  states will split into two-fold  $e_g$  states and three-fold  $t_{2d}$  states. As known, the In  $t_{2d}$  and N  $t_{2p}$  states share the same  $t_2$  symmetry, thus can couple with each other. Many previous reports also mention similar peak of DOSs overlapping and use them to prove the coupling between impurity and some state of the host.<sup>31-32</sup> Based on the band coupling model developed by Dalpian and Wei,<sup>33</sup> such kind of states ( $t_{2p}$ ,  $t_{2d}$ ) coupling will lead to a level repulsion, which pushes up the VBM (mostly composed by N  $2p$  orbitals) and pushes down the In  $t_{2d}$  states. It also could be seen that several peaks of Mg  $t_{2p}$  state overlap with that of In  $t_{2d}$ . However, we argue that such kind of overlapping is coming the fact that Mg  $t_{2p}$  and In  $t_{2d}$  share the same coupling with N  $t_{2p}$  states. Considering the limited atom concentration and large average distance, the direct coupling between In  $t_{2d}$  and Mg  $t_{2p}$  state should be rather weak.

Figure 2 shows the calculated total density of states for pure GaN and In doped GaN. It is clearly visible that several peaks of In doped GaN (blue line) move to higher energies due to the coupling and level repulsion between In  $t_{2d}$  and N  $t_{2p}$ . As a result, a “new” higher VBM, which is composed of the hybrid  $p-d$  orbitals, is formed. As discussed above, the upward shift of the VBM will facilitate acceptor activation and thus better  $p$ -type dopability in GaN. The underlying physical mechanism of the above observation can also be understood in another way: In Figure 3 we plotted calculated charge density of the VBM at the gamma point in the planes containing In-N bond (a) and Ga-N bond (b). We can see from the plots that the VBM states are more localized on the In-N site and shows strong  $p-d$  hybridization orbital character. As know, due to larger In-N bond length and strong coupling between N  $2p$  orbital and In  $4d$  orbitals, the VBM of InN is 1.11 and 2.39 eV higher than that of GaN.<sup>34</sup> In this new scenario, by In doping the VBM of our system turn to be more InN-like, which is higher than pure-GaN. Therefore, the  $Mg_{Ga}$  defect level is closer to the VBM. As a result, the acceptor ionization energy is smaller. It is important to point out that due to the limited concentration (about  $10^{17}/cm^{-3}$  confirmed by secondary ion mass spectrometry) In atom still acts as dopant in our system. That means the band-gap does not change too much. Another question needed to consider is the effect of In incorporation on the crystal quality of nitrides materials. Based on formation energy calculation, Li *et al*<sup>35</sup> argued that due to different bond length it is easier to break the In-N bond than the Ga-N bond to form N vacancy, which act as donors in nitrides and lead to compensation effect. From this point of view, it is critical to suppress the formation of N vacancy to achieve high efficient In-Mg co-doping. Here, high V/III ratios (10000:1) and high

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growth temperatures (1050°C) are used to yield sufficient amounts of active nitrogen species for gallium nitride growth. It is believed that high growth temperature can not only allow for ammonias cracking, but also facilitated transport of atomic N to proper lattice sites.<sup>36-37</sup> The strain analyses by XRD published by our group previously have confirmed the elimination of nitrogen vacancies in Indium doped nitrides by using non-equilibrium growth techniques,<sup>38</sup> which is consist with the results of Chung.<sup>39</sup>

We would like to point out that the concentration of In and Mg have substantial effect on the carriers concentration. With increasing In concentration, the coupling of In  $4d$  and N  $2p$  become stronger, which further push up the VBM and facilitated p-type dupability. While, with the increasing of Mg concentration, the initially isolated acceptor level will interact and result in the formation of acceptor band, which is closer toward the valence band and lowering the effective activation energy.<sup>14</sup> However, all the above assumptions are strongly relied on the suppressing of nitrogen vacancy formation, which lead to the compensation effect. Experimental and theoretical analysis on this topic is highly desired, which will be performed in our next work.

Symmetric (002) and asymmetric (102)  $\omega$  scans for control sample and In-Mg codoping sample were shown in Figure 4. The full widths at half maximum (FWHM) of asymmetric (102) rocking curves of these two sample are almost identical. While, The FWHM of symmetric (002) rocking curve increase from 237.5 to 250.18 arcsec, which indicates that the incorporation of In atoms slightly deteriorate the crystalline quality of GaN material. As shown in Figure 5, secondary ion mass spectrometry measurements were performed to verify the incorporation and distribution of Mg and In atoms. Furthermore, the hole concentration confirmed by Hall measurement is as high as  $1.4 \times 10^{18} \text{ cm}^{-3}$  at room temperature, which is nearly one order of magnitude higher than the typical value by direct doping GaN in our lab. The acceptors activation can be determined by fitting the concentration data. It decreased to about 160 meV as shown in Figure 6 and can be act as a direct evidence to support our calculation results. The hole mobility of In-Mg codoping sample is about  $9.6 \text{ cm}^2 / V.s$  at room temperature, which is slightly decrease compared with the control sample (about  $10.2 \text{ cm}^2 / V.s$  ). We argue that the decrease of hole mobility should be mainly ascribed to the increase of impurity scattering and/or dislocation scattering.

## 5. Conclusion

In summary, an effective strategy for efficient p-type doping was proposed to overcome the fundamental problem of high activation energy in high bandgap III-nitrides by In-Mg codoping. Our results indicated that due to sharing the same  $t_2$  symmetry, In  $t_{2d}$  and N  $t_{2p}$  states can strongly couple with each other. Such kind of



states ( $t_{2p}$ ,  $t_{2d}$ ) coupling will lead to a level repulsion, which pushes up the VBM (mostly composed by N  $2p$  orbitals). Another result of the coupling is: now the VBM state of our new system is more localized on the In-N site and shows strong  $p$ - $d$  hybridization orbital character. That means the VBM of our system is more InN-like, which is higher than pure-GaN. As a result, better  $p$ -type dopability of GaN could be achieved. Furthermore, the hole concentration confirmed by Hall measurement is as high as  $1.4 \times 10^{18} \text{ cm}^{-3}$ .

### Acknowledgements

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**Figure captions**

Fig. 1. Calculated projected density of state of In  $4d$ , N  $2p$  and Mg  $2p$  orbitals.

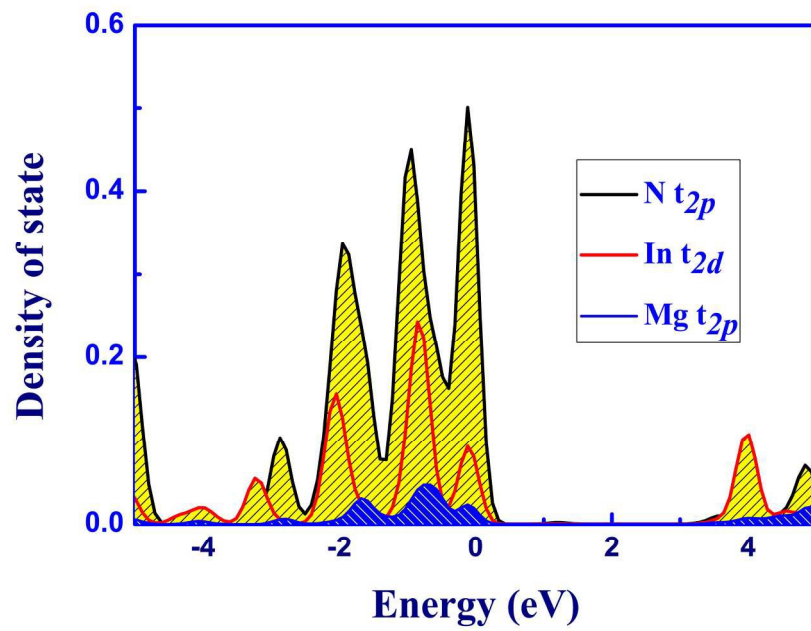
Fig. 2. Calculated total DOS for supercells of pure GaN, and In-Mg co-doping GaN.

Fig. 3. Isosurface charge density plots of VBM of In-Mg co-doping GaN at  $\Gamma$  point in the planes of (a) N-In-N bonds and (b) N-Ga-N bonds.

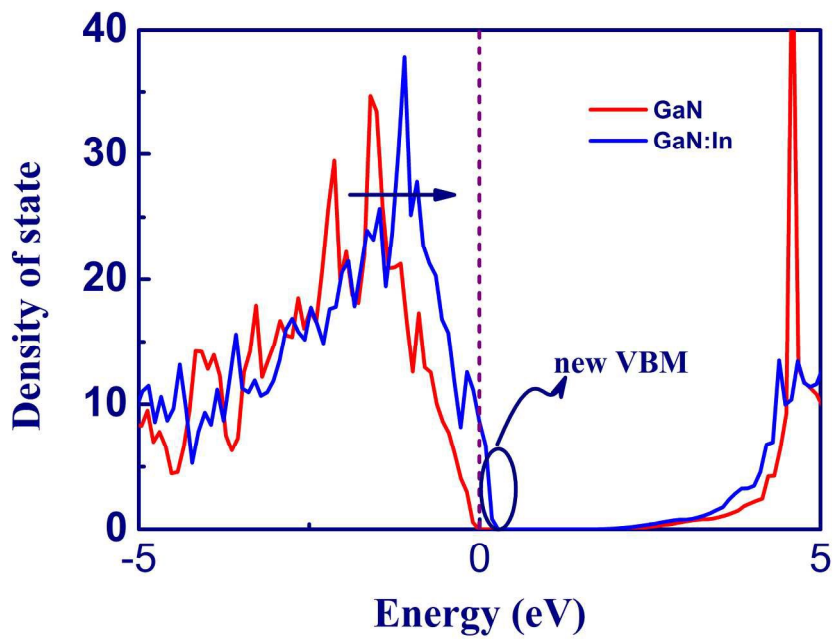
Fig. 4. Symmetric (002) and asymmetric (102)  $\omega$  scans for control sample and In-Mg codoping

Fig. 5. Secondary ion mass spectrometry measurements were performed to verify the incorporation and distribution of Mg and In atoms

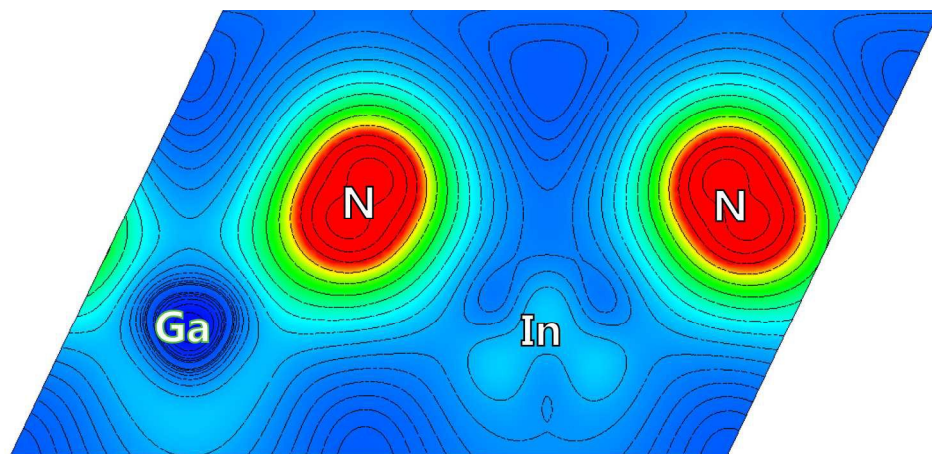
Fig. 6. Hole concentration and mobility as a function of temperature for In-Mg co-doped GaN samples. The fitting curve is shown with solid line.



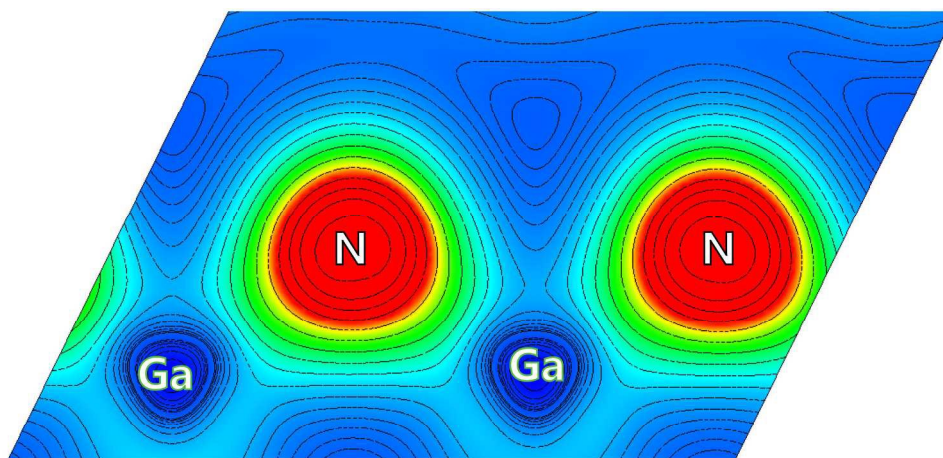
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199x139mm (300 x 300 DPI)

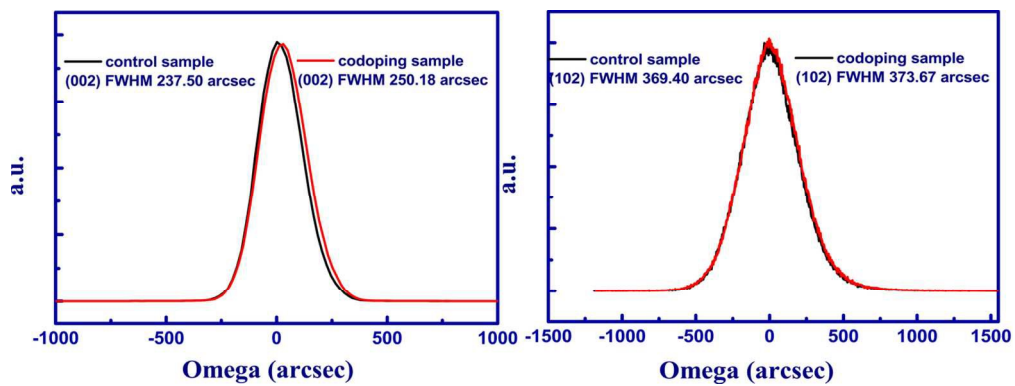


(a)

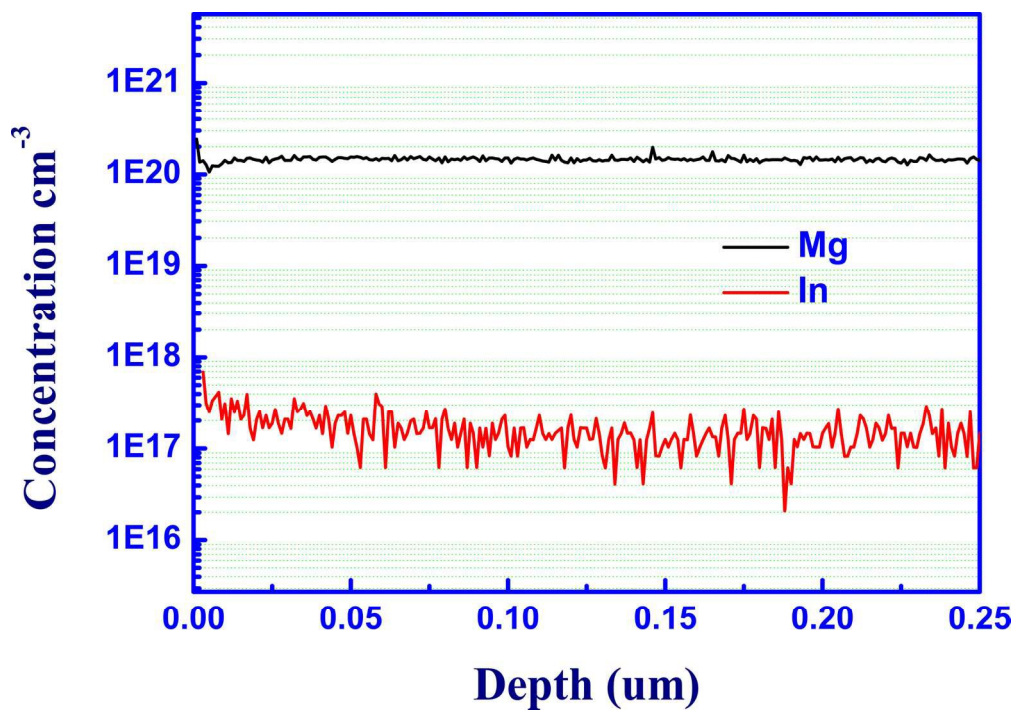


(b)

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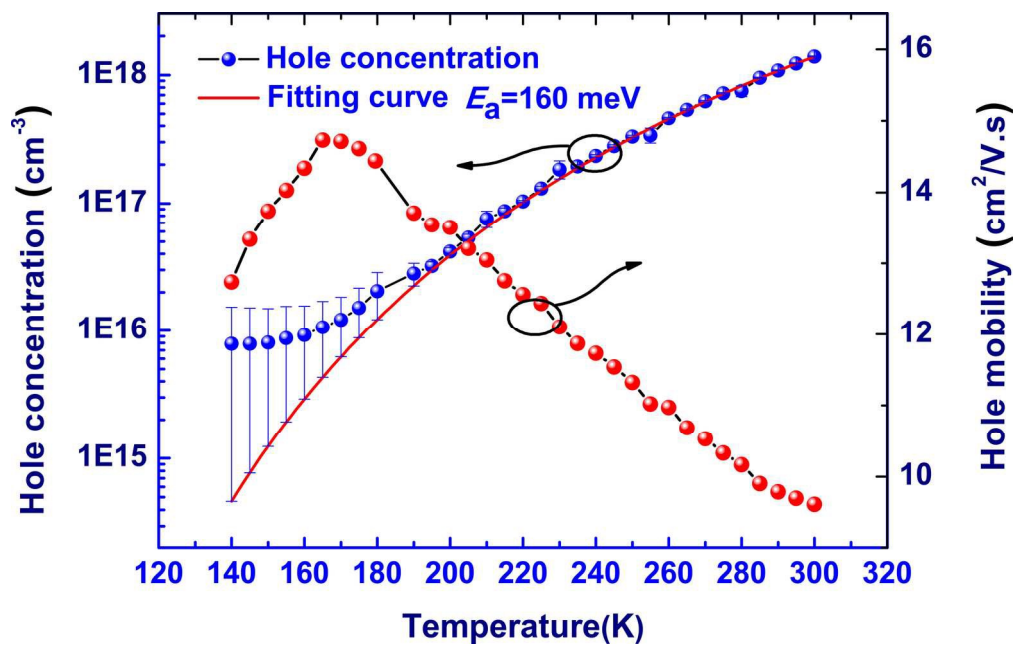


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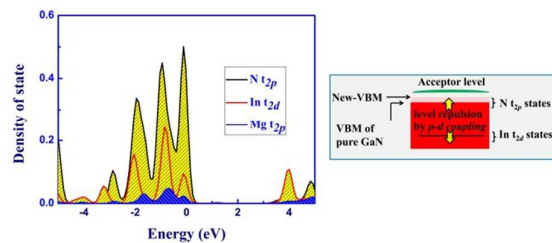


173x120mm (300 x 300 DPI)





169x106mm (300 x 300 DPI)



The valence band maximum could be modified by specific states coupling, thus improving the p-type dopability in In-Mg co-doping GaN.