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Photoinduced doping in hexagonal boron nitride

A. Perepeliuc,^{1,2} R. Gujrati,^{3,4} A. Srivastava,^{1,4} P. Vuong,² V. Ottapilakkal,²
P.L. Voss,¹ S. Sundaram,^{1,4} J.P. Salvestrini,^{1,4,a)} and A. Ougazzaden^{1a)}

¹Georgia Institute of Technology, School of Electrical and Computer Engineering, Atlanta, GA 30332-0250, USA

²CNRS, Georgia Tech – CNRS International Research Lab, 2958, 2 rue Marconi, 57070 Metz, France

³Georgia Institute of Technology, George W. Woodruff School of Mechanical Engineering, Atlanta, GA 30332-0250, USA

⁴Georgia Tech Europe, 2 rue Marconi, 57070 Metz, France

^{a)}Author to whom correspondence should be addressed. E-mail addresses: jean-paul.salvestrini@georgiatech-metz.fr ;
aougazza@georgiatech-metz.fr

Abstract

Hexagonal boron nitride is shown to exhibit very significant persistent photoconductivity after UV illumination. This behavior can be initiated by sub bandgap or close to bandgap illumination. Neither temperature nor pressure affect the build up of photoinduced carriers. The effect persists at least for months at room temperature and is maintained significantly after heating up to 300°C. Up to 6 orders of magnitude increased conductivity has been durably established in the devices and effect is reproducible. Double exponential fitting gives time constants up to 4600 days. Irradiation after having saturated the devices is shown to drastically reduce the decay rate. The bulk origin of such effect has been demonstrated. p-hBN/n-AlGaN diodes based on magnesium doped h-BN have been used to determine the type of conductivity through studies of junction capacitance variations under UV irradiation. Depending on illumination wavelength, both n and p-type durable photoinduced carriers can be produced. These results are of interest for UVC LEDs in which the usual conductive AlGaN layers are still a hurdle toward efficient UV emitters.

Persistent photoconductivity (PPC) refers to light-driven increased conductivity which persists after turning off the light source. At room temperature, semiconductors such as AlGaAs or GaN can exhibit this property [1,2] but most of the effect is lost after few hours at maximum. Lowering the temperature is a way to increase the duration of such effect but it makes it inefficient for practical usage in semiconductor devices. Interestingly, in some complex semiconductor oxides including STO, PPC is maintained for days at room temperature and has been used to write erasable information in such material [3]. Strikingly, PPC was observed in Ga₂O₃ to be effectively permanent at room temperature by exposing the oxide repeatedly to light [4]. Furthermore, photoinduced conductivity was unchanged even after heating the sample at 400 °C. A large lattice relaxation model (LLR) is suggested as an explanation. First proposed by Lang and Logan to explain PPC in Te-doped AlGaAs [5],

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excitation of electrons from donor related defects leads to a stabilizing crystal distortion inducing barrier for electron recapture. From the LLR model, it can be inferred that the larger the distortion the higher the barrier. Accordingly, Rana's ab initio simulations showed crystal distortion up to 0.8 angstrom when electrons are pumped out from oxygen vacancies [4]. Providing more insight on type of conductivity in PPC, first principle simulations were used by Lany and Zunger to describe PPC in ZnO and Chalcopyrites [6], showing that in some materials sub band gap illumination could lead to n-type PPC whereas near bandgap illumination could induce p-type PPC. Interestingly, compared to AlGaAs in which PPC is linked to DX-centers [5], PPC observed in ZnO, Ga₂O₃ or STO are currently related to native oxygen vacancies. PPC in MoS₂ has been explained using a different model [7]. Random spatial fluctuations caused by defects modify the carriers' potential energy, concentrating them in potential wells, leading ultimately to reduced recombination rate. It is to be noted that above room temperature, white light induced PPC in MoS₂ displays LLR model behavior [8]. In each case, PPC is strongly related to the presence of defects [5,9,10,11].

Hexagonal boron nitride (h-BN), an ultrawide band gap 2D material (5.955 eV) [12], is shown in this paper to exhibit such room temperature and extremely long PPC under different UVC light exposures. Photoinduced conductivity increase by 6 orders of magnitude has been maintained for at least 8 months at room temperature upon 213 nm illumination. Such an effect has also been observed under 266 nm irradiation but with a weaker response. No sample pretreatments were required to obtain PPC compared to STO in which pre-annealing under hydrogen rich atmosphere is required [13,14]. These results are of interest for UVC LEDs in which the usual conductive AlGaN layers are still a hurdle toward efficient UV emitters. Due to the increased aluminum content needed for sub 270 nm emitting devices, dopants' ionization energies also increase, lowering the number of available free carriers [15,16]. This issue is especially true for p-layers [17,18]. Having a very large band gap, h-BN is a potential replacement for p-AlGaN layers and can also serve as an electron blocking layer [19]. Indeed, magnesium (Mg) p-doping has been demonstrated but the reported values are still quite low (10^{16} up to 10^{18} free carriers per cm³) [19,20]. Moreover, n-type doping has also been achieved but is still quite inefficient for devices applications [21]. Being able to induce permanent changes in conductivity using UV illumination could allow more efficient conductivity tuning in h-BN and potentially pave the way toward new DUV structures devices. In the present work, conditions under which very long long PPC effects can be triggered in unintentionally doped h-BN are studied. The type of carriers obtained by 213 nm and 266 nm illumination are also investigated using a p-hBN/n-AlGaN junction structure.

Thin 3, 6, 10, 20 nm-thick layers of h-BN were grown on 2" (0001) sapphire substrate using metal organic chemical vapor deposition. Triethylboron (TEB) and ammonia (NH₃) were respectively used as precursors for bore and nitrogen. Layers were grown at 1280 °C in hydrogen ambient at 85 mbar. SEM, XRD and TEM images are provided as Supplementary Material (see

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Figures S1, S2 and S3). More details on growth process can be found in [22]. Non-annealed 500 μm square Ni(30nm)/Au(100nm) contacts were deposited using electron beam deposition. The distance between each pad is also 500 μm in both directions. Schematic, and top view picture of the structure are shown in Figure 1. In addition, p-hBN/n-AlGaIn diode structure were also grown on 2" (0001) sapphire substrate. More details about the structure can be found elsewhere [20]. Samples were illuminated in between pads using pulsed lasers emitting at 213 nm (average power of 3 mW, beam size of 269 μm , oval shape) and 266 nm (average power of 4 mW, beam size of 591 μm , rounded shape) with a repetition rate of 1 kHz and 1 ns pulse-width (FWHM). It is to be noticed that the use of a shorter pulse-width Ti:Sapphire femtosecond laser did not lead to PPC. Interestingly, Kanaev et al observed that 248 nm nanosecond pulses led to persistent modifications of h-BN fluorescence spectra compared to femtosecond pulses [23]. This could be due to the fact that, in the latter case, electronic photo-excitation periods are much shorter than nucleus' dynamics and thus not allowing for enough time for defects to relax to a metastable configuration and ultimately to build up electrical charges. Illumination was performed under three different conditions: 298 K and atmospheric pressure, 298 K and vacuum, 100 K and vacuum. In the latter two cases, pressure was 5×10^{-6} Pa. Current-voltage (I-V), 100 kHz capacitance-voltage (C-V) and 20 V biased current-transient were recorded for months to investigate charges' build up dynamics and carrier type generation for both 213 and 266 nm exposures.

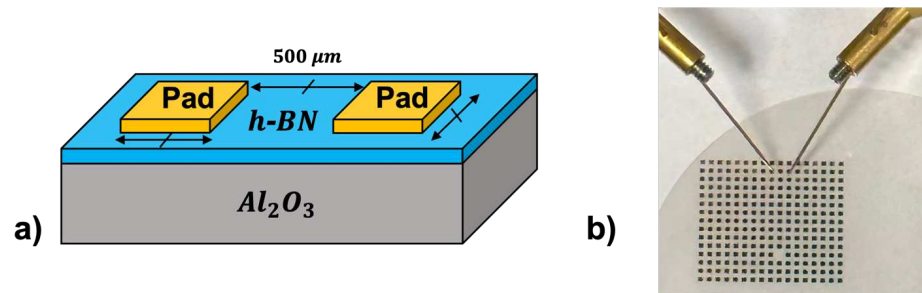


FIG. 1. (a) Schematic of the device. (b) Top view of multiple devices.

I-V measurements up to 200 V were first performed on devices before illumination. Initially, contacts present an ohmic behavior. After illumination, ohmic response is conserved. Such results are given in Supplementary materials (see Figure S4). Due to ohmic behavior of contacts before and after illumination, the devices' electrical resistance can be measured in both cases, allowing us then to calculate conductivity normalized to conductivity before light exposure σ_d . Figure 2(a) shows an example of normalized conductivity transients recorded in 20 nm thick h-BN devices, at 298 K under ambient pressure, during 213 and 266 nm illumination. For each one of them, the laser spot was kept continuously in between the pads. Therefore, the

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dose was essentially concentrated on a single region. Doses of 27 and 180 kJ/cm² were needed to start reaching saturation under 213 and 266 nm irradiation, corresponding respectively to 20 and 90 hours of continuous illumination.

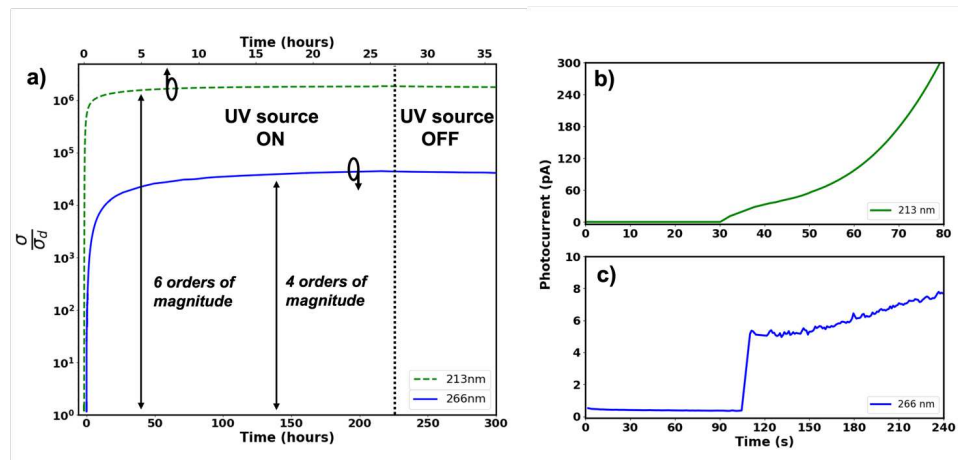


FIG. 2. (a) Build up of photoinduced conductivity in 20 nm h-BN normalized to conductivity before illumination. Dashed and continuous lines correspond respectively to devices exposed to 213nm and 266nm light. Top x-axis corresponds to dashed curve. (b), (c) Photocurrent transients measured at 20 V bias, respectively during 213 and 266 nm exposure.

First, 213 nm illumination leads to 6 orders of magnitude increase whereas 266 nm response is of 4 orders of magnitude. Secondly, saturation has been reached much more quickly for 213 nm exposure than 266 nm. More powerful UV light sources should accelerate the free carriers' build up. In any case, the h-BN material responds more effectively to 213 nm light. Note that these results are very reproducible across different wafers. Also, illumination of devices at 298 K under vacuum, and 100 K under vacuum still led to 6 orders of magnitude conductivity increase. Therefore, neither the temperature nor the pressure seems to have an influence on triggering PPC in h-BN. However, illumination at low pressure led to a slight surface darkening, which could be due to oxidation of elemental boron surface layer created by UVC photons breaking B-N bonds with nitrogen released under gas form [23]. Short-time build up current transients are also presented in Figures 2(b),(c). Once illumination is turned on, a first fast increase in current is observed as expected when pumping electrons from defects in the bandgap. If light source is turned off immediately after, current goes back to the original value very fast. On the other hand, if illumination is prolonged, then after a short plateau, current starts to rise again and charge to build up durably. If light is then turned off, a current increase corresponding to classical photoconductivity is lost but the built up value remains. It should be noted that because the 213 nm response is much faster than 266 nm, turning off the light source right after opening it still leads to small

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PPC and original conductivity cannot be recovered. An illustration of such behavior is given in Supplementary materials (see Figure S5).

Both curves on figure 2(a) also show the conductivity several hours after turning off light source. It can clearly be seen that for these timescales, at least, photoinduced carriers are permanent. A much longer follow up of illuminated devices at room temperature and ambient pressure is shown on Figure 3. Three different devices' conductivity decay were investigated. Two of them were irradiated with 213 nm laser at different doses (devices A and B, respectively blue circles and green triangles) meanwhile the third one has been exposed to 266 nm (device C, red squares). All three curves were normalized to built up conductivity obtained after turning off light source σ_0 .

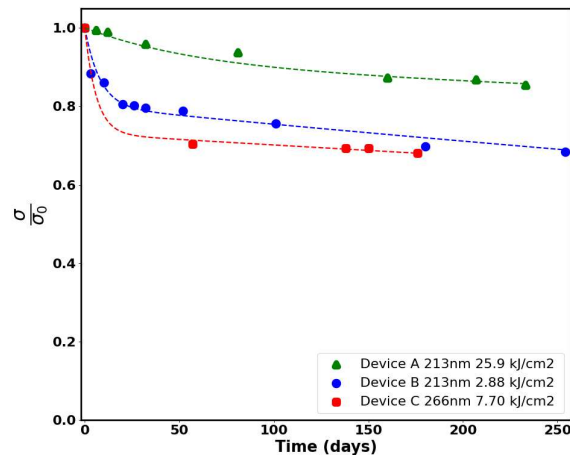


FIG. 3. Decay of photoinduced conductivity normalized to conductivity just after turning off light source, for three devices for both wavelength and different doses. Blue circles, green triangles and red squares correspond respectively to devices A, B and C. Dashed lines correspond to double exponential fitting.

Astonishingly, PPC was maintained above 90% of induced value after 8 months for sample A. For samples B and C, photoinduced conductivity is still above 70% of its initial value after 8 months. Conductivity decay is composed of two phases, a fast one lasting roughly few weeks and subsequently an extremely slow decay. Most of the PPC loss occurred during the first phase. Since there is still debate in the literature on how the decay should be modeled, either by using stretched exponential or double exponential, we have chosen to use the latter such as in [7] because it gave a better agreement with the measured values than stretched exponential. The double exponential model is not intended to provide quantitative explanation of the PPC

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mechanism, but rather to estimate how long PPC will last significantly. From this modelling, two time constants can be extracted. First time constant τ_1 corresponding to first decay goes up to 60 days whereas second time constant τ_2 is two orders of magnitude greater and ranges from 1700 to 4600 days, depending on the dose provided to sample. Time constants used for devices A,B and C are given in table I. Note that first time constant for device C can hardly be fitted accurately due to lack of measurements points. It is clear that the PPC induced in h-BN should last significantly for years. Note that stretched exponentials have also been proposed to model such behavior [24,25] and tried in this study, but double exponential fitting gave us better results.

TABLE I. Time constants used in double exponential fitting

Device	A	B	C
τ_1 (days)	60	8	6
τ_2 (days)	4600	1700	2500

It should be noted that device A decay is much slower than device B. Thus, dose has an impact not only on the actual value of PPC, but also on its decay rate. The higher the dose, the larger the time constants, so that the higher dose probably induces more metastable states and thus more durable crystal distortions. Continuing light exposure after conductivity saturation could thus be a solution to increase the lifetime of the PPC effect. Device C PPC initial decrease is more important but it still reaches a very slow decay rate after few weeks. This implies that 266 nm light triggers h-BN less efficiently as observed in Figure 2. Looking for more optimal wavelengths around 266 nm could be a solution to improve the PPC effect.

Samples highly exposed at 213 nm ($> 10 \text{ kJ/cm}^2$) were heated at 300 °C for one hour and a half. Samples lost between one and two orders of magnitude of doping. Therefore, samples still keep up to 5 orders of magnitude conductivity increase even after annealing at high temperature for long time. Thus, if such h-BN layers were to be used in electronic devices, the increase in conductivity could be maintained under usual operation conditions. The persistent photoconductivity phenomenon is currently related to defects present in the crystal such as vacancies or substitutional impurities which should make it a bulk effect. To confirm such assumptions, we exposed samples with different h-BN thicknesses to 213 nm light and measured by how many orders of magnitude conductivity increased. Results are given in table II.

TABLE II. Conductivity increase under 213nm exposure for different film thicknesses.

	3 nm	6 nm	10 nm	20 nm
OoM ^a increase	1	3	5	6

^aOoM Orders of magnitude

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It can clearly be seen that the thinner the h-BN layer, the less important is the PPC effect. Therefore, we have strong evidence that PPC observed in h-BN is due to bulk effects which could then be potential defects. Furthermore, as described above, being able to induce PPC under atmospheric pressure or vacuum puts away the hypothesis of surface effects due to air molecules triggered by UV light absorption. Conductivity enhancement starts to saturate as thickness increases, which can be explained by the geometry of the electric pads which induced lines of current not spreading in all the layer but essentially close to the surface.

All these results encourage us to consider PPC in h-BN as a potential alternative to conventional doping. However the sign of the photoinduced carriers' charge still needs to be addressed in order to propose PPC for practical devices. Consequently, pn junctions made of p-hBN and n-AlGaN have been used to perform such study. Presence of an internal electrical field has been demonstrated by Mballo [20], indicating the presence of a depletion region in the structure. PN junction capacitances are influenced by the number of activated dopants in each side of the junction, thus increasing or reducing such number should induce a shift in capacitance. As UVC irradiation is shown to increase conductivity in h-BN, pn junction capacitances should increase or decrease permanently depending on what types of carriers have been generated. Figure 4 shows capacitance variations of two such h-BN/AlGaN pn junctions after exposure to 213 and 266 nm light.

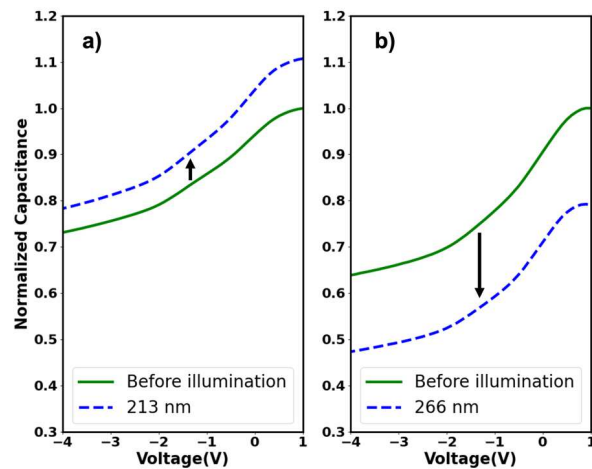


FIG. 4. C/V measurements of two p-hBN/n-AlGaN junctions before and after 213/266 nm irradiation. Both curve are normalized to highest reference capacitance value. Measured capacitances range between 50 and 110 pF. Devices a) and b) received respectively doses of 61 and 350 kJ/cm²

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Both devices experienced a shift in capacitance after UVC illumination. An increase of capacitance has been observed upon 213 nm exposure whereas the second device's capacitance decreased after seeing 266 nm light. Junction capacitance being inversely proportional to depletion region depth, and the latter extending more in less doped regions, 213 nm and 266 nm light are supposed to induce p-type and n-type doping respectively. These two opposite behaviors encourage us to believe that 266 nm induced PPC is not just a weaker version of 213 nm exposure, but a distinct effect caused by triggering a different defect. Capacitance variations are higher for 266 nm illumination given that dose was 5 times higher than with 213 nm laser. Also, h-BN layers used in these pn junctions were previously p-doped with magnesium. If 213 nm is rightly assumed to be inducing p-type PPC, then adding more holes in a region already filled with positive carriers should induce a reduced effect. Moreover, PPC being strongly related to defects, we suggest that introduction of magnesium inside the crystal could potentially kill some of the centers related to PPC. To ensure that these changes in capacitance are only due to h-BN layer, square contacts Ti/Al/Ni/Au (20/200/25/25 nm) were deposited on 500 nm thick n-AlGaIn, grown in the same conditions as the one used in the junctions, and 213/266 nm exposures were performed. We observed the short usual PPC in (Al)GaIn [2], but no such long-term effects as observed in h-BN thin layers. Thus, measured changes in capacitances are related to h-BN layers only. To support the idea that 213 nm is causing p-type PPC, Hall measurements using a 0.5 T DC magnetic field were performed on a 5x5 mm² 20 nm thick h-BN layer that had been evenly illuminated. Such large area light exposure has been performed using a laser scanning procedure described in [26]. Contacts are the same as the one used in the previous devices. Results confirm p-type and give a carrier density of $p=2 \times 10^{16} \text{ cm}^{-3}$ and mobility of $\mu=0.5 \text{ cm}^2 \text{ V}^{-1} \text{ s}^{-1}$. Compared with values reported by Mballo [20], p-photoinduced doping is one order of magnitude more efficient than magnesium doping. This is consistent with I/V measurements performed on both kind of p-doping methods. Such Hall measurements were not performed on 5x5 mm² samples illuminated with 266 nm light, because the induced current was too low we were prevented from achieving accurate measurements.

In summary, different thicknesses of h-BN layers were exposed to 213 nm and 266 nm nanoseconds pulses. As a result, persistent photoconductivity has been observed. Conductivity has been raised by six orders of magnitude under 213 nm illumination whilst 266 nm induced four orders of magnitude. 213 nm exposure triggered a much faster and more efficient PPC compared to 266 nm irradiation. Bulk effect linked to defects has been suggested by showing that conductivity increase is thickness dependent. Post-exposure, devices lost, after 8 months, at most 30% and only 10% in the best case of their induced conductivity with a highly nonlinear behavior. Most of the loss happened during the first weeks and then decay rate became very slow. At room temperature, photoinduced conductivity persists consequently after at least 8 months. Temperature and pressure have been shown to have no influence on building up photoinduced carriers. C/V measurements performed on p-

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hBN/n-AlGaIn junctions exposed to 213/266 nm led to increase of capacitance in first case, suggesting p-type PPC under 213 nm illumination, whereas in second case, 266 nm led to diminished capacitance indicating n-type PPC. Both wavelengths induced effects are very reproducible across different growth runs. The huge persistence of PPC in h-BN is a promising alternative to conventional doping in h-BN or eventually as a booster for already doped h-BN layers. More understanding on the actual defects causing PPC in h-BN is needed. It is to be noticed that the features (dark spots) which are small size pinholes (around 50 nm in diameter) in the thin hBN layers related to the mechanism of the lateral growth layer-by-layer and coalescence in the van der Waals growth, as well as wrinkles that can be seen in Fig. S1 are unlikely responsible for the PPC. The best lead we currently have could be the incorporation, during the crystal growth, of carbon atoms coming from the TEB organo-metallic precursor, into the BN crystal lattice. Consequently, growth conditions could potentially be oriented to induce such defects to improve the strength and lifetime of this effect. Also, more experiments and proof are needed to confirm that 213 nm and 266 nm induce distinct effects as well as different types of photoinduced carriers. This could be a path towards fabrication of h-BN homojunctions. Finally, light needs to be shed on why PPC was not induced under femtoseconds pulses. We suggest that continuous light sources could potentially trigger a much stronger effect.

SUPPLEMENTARY MATERIAL

The Supplementary Material is available free of charge at <https://...>
It shows structural and morphological characterization (HR-XRD, SEM, and HAADF-STEM), as well as electrical characterization of the devices versus voltage, time, and illumination.

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CONFLICT OF INTEREST

The authors declare that they have no conflict of interest.

CONTRIBUTION

J.P.S. and A.O. contributed to design the study and supervised the whole project. A.P., R.G. and A.S. performed device fabrication and electrical measurements, A.P. and J.P.S. wrote the manuscript. S.S., P.V. and V.O. performed the epitaxial

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growth. P.V. and V.O. performed SEM and HRXRD measurements of the epilayers. The manuscript was written through contributions of all authors. All authors have given approval to the final version of the manuscript.

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