Room Temperature GaN-based Spin Polarized Emitters

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ABSTRACT

Wide band gap dilute magnetic semiconductors have recently been of interest due to theoretical predictions of room temperature ferromagnetism in these materials. In this work $Ga_{1-x}Gd_xN$ thin films were grown by Metalorganic Chemical Vapor Deposition. These films were found to be ferromagnetic at room temperature and electrically conducting. However, only GaN:Gd layers and devices grown with a $TMHD_3Gd$ precursor that contained oxygen showed strong ferromagnetism, while materials grown with an oxygen-free Cp_3Gd precursor did not show ferromagnetic behavior. This experimental observation was consistent with first-principles calculations based on density functional theory calculations that we completed that showed the ferromagnetism was mediated by interstitial oxygen. The results confirmed the first successful realization of $Ga_{1-x}Gd_xN$-based spin-polarized LED with 14.6% degree of polarization at 5000 Gauss is obtained.

Keywords: Spin Polarized LED, MOCVD, Nitride DMS

1. INTRODUCTION

Spintronic devices are of great interest since they can be designed to provide higher information storage densities, higher processing and transfer speeds, as well as lower power consumption than current electronic and optoeletronic devices. The introduction of magnetic ions into III-V semiconductors, such as GaAs and GaN, has proved to be an effective approach to realizing spintronic devices [1]. Novel functionalities such as reconfigurable logic, nonvolatile chip-based memory, and a solid state platform for quantum computing may also be possible [2]. Currently, metal semiconductor heterostructures are used for spin injections from ferromagnetic metals into semiconductors due to the absence of magnetic semiconductor material. However, the difference in conductivities between the metal and the semiconductor results in interfacial scattering and low spin injection efficiencies. Therefore, since the 1990s, extensive efforts have focused on making III-V semiconductors ferromagnetic by doping them with magnetic ions however this behavior was typically observed well below room temperature [3].

Ferromagnetism and semiconducting properties can coexist in magnetic semiconductors such as europiums, chalcogenides and semiconducting spinels. These materials have not resulted in any commercial devices, as it is difficult to grow device quality thin films, pn junctions and heterostructures. One class of materials that show promise in this regard is dilute magnetic semiconductors (DMS). These are typically compound semiconductors doped with transition metals (TM) or rare earth (RE) elements to provide magnetic functionality due to their incompletely filled d-shells (TM's) and f-shells (RE's) [4]. They exhibit a net magnetic moment that is exploited in DMS-based materials and devices.

The most widely studied III-V DMS compound is $Ga_{1-x}Mn_xAs$, which is a p-type material based on the relatively shallow nature of the Mn acceptor. It exhibits a strong correlation between carrier concentration and Curie temperature (T_C). The experimental results were first successfully modeled by Dietl *et. al.* [5] employing Zener mean field theory. A strong Ruderman-Kittel-Kasuya-Yosida (RKKY) coupling was found between the localized spins of the Mn center and the delocalized spins of free holes. Thus the long-range ferromagnetic interaction between isolated Mn centers is mediated by holes in $Ga_{1-x}Mn_xAs$. This material has been shown to be effective at providing a spin injection layer in optically based devices [6]. However, the T_C of these systems is limited to less than 170 K which precludes their

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practical uses [7]. Dietl's calculations [5], predicted room temperature ferromagnetism for $Ga_{1-x}Mn_xN$ and $Zn_{1-x}Mn_xO$ with 5 % Mn incorporation and 3.5×10^{20} holes/cm^3. In addition, recent investigation demonstrated that the Mn acceptor in these materials is rather deep ($E_B \approx 1.5$eV) [8], in contradiction to Dietl's assumptions, and the required hole concentration has not been achieved in either material to date. Nevertheless, tremendous progress in obtaining ferromagnetic GaTMN (TM = Mn, Fe and Cr) was made over the last decade by this group and others. However, it is still debated whether the observed ferromagnetism is due to substitutional TM ions in the semiconductor lattice, unwanted precipitates, or a combination of both. TM doping of GaN generally results in highly resistive material, since TMs form deep acceptors, sometimes with compensating point defects. Thus, the observed ferromagnetism in GaTMN can be better explained by a double exchange mechanism involving a spin split impurity band within the band gap. This model was confirmed by the annihilation of ferromagnetism upon n-type and p-type co-doping shifting the Fermi level out of the impurity band.

Gadolinium-doped GaN (GaN:Gd) is one of the most intriguing dilute magnetic semiconductors. Although ferromagnetism above room temperature in Gd-doped GaN was first reported by Asahi *et. al.* [9, 10] it did not achieve further prominence until Dhar *et. al.* [11,12] reported "colossal magnetic moment," apparently up to 4000 μ_B per Gd atom, in these films. This colossal magnetic moment was found to be even stronger in Gd implanted samples [13,14]. There were reports of traces of secondary phases in GaN:Gd [15,16], none of which can explain the physical origin of ferromagnetism above room temperature. Paramagnetic/ferromagnetic resonance studies also found no conclusive signals that could account for the high temperature magnetism, although some evidence for either Gd or GdN clusters were reported [17].

Initially the origin of this colossal magnetic moment was suggested to be the polarization of the surrounding host medium [11,18]. However, later studies point toward defects in the crystal lattice, since implantation led to even higher magnetization but annealing somewhat reduced it [13,14]. X-ray magnetic circular dichroism (XMCD) studies completed on the Gd *L*3 edge show that the main origin of the magnetism does not appear from Gd itself [19,20]. Several theoretical models have been proposed to explain these observations, but none of these models completely address the question of the cause of such large magnetic moment. The microscopic mechanisms in play in GaN:Gd are still subject to extensive debate, but many first-principles calculations based on density functional theory (including this group) predict that crystal defects are important contributors to the observed magnetic moments [21,22]. In particular, interstitial oxygen (O_i) has been predicted to have a negative formation energy in GaN:Gd and to add to the net magnetic moment of the material [21].

This research group has produced the only reports of GaN:Gd thin films grown by Metalorganic Chemical Vapor Deposition (MOCVD) to date [23], which is the standard commercial growth technique for producing device-quality compound semiconductor thin films. These films were found to be ferromagnetic at RT and electrically conducting although the mechanism for the ferromagnetism was not well understood [24]. In this work, we have completed a more systematic investigation of interrelationship of chemical, magnetic, and electrical properties of GaN:Gd. First-principle calculations based on density functional theory have shown that the ferromagnetism was likely mediated by interstitial oxygen. This was confirmed, in part, because ferromagnetism was only observed in GaN:Gd thin films grown using $TMHD_3Gd$, an oxygen containing metalorganic Gd precursor. It was found that the GaN:Gd was residually n-type (mid 10^{17}cm^3) rather than the highly resistive material grown using TMs and could be effectively n-type (Si) or p-type (Mg) doped without losing their ferromagnetic properties. These Si and Mg Co-doped GaN:Gd films could then be used for spin-injection into spin light emitting diodes (LEDs) and other spintronic devices. Preliminary spin-polarized GaN:Gd-based LEDs where produced which had a maximum spin polarization of 14.6% at 5000 Gauss and exhibited magnetic hysteresis at room temperature.

2. EXPERIMENTAL

GaN:Gd films, typically 500 nm thick, were grown on 2 μm thick GaN templates (GaN/sapphire) by MOCVD using a commercial rotating disk reactor with a short vertical jar configuration. The reactor has a specially modified flow flange injection system with dual injector blocks to minimize pre-reactions of the nitrogen and TM/RE precursors in the transport phase. The gadolinium sources were Tris(2,2,6,6-tetramethyl-3,5-heptanedionato)gadolinium ($(TMHD)_3Gd$) and Tris(cyclopentadienyl)gadolinium (Cp_3Gd). The key difference between these sources is that the organic ligand in $TMHD_3Gd$ contains oxygen, while the ligand in Cp_3Gd does not. In addition, films with n-type (Si) and p-type (Mg) co-doping were also grown and standard p-GaN activation steps were applied to p-$Ga_{1-x}Gd_xN$. An energy dispersive spectrometer (EDS) attached to a scanning electron microscope (SEM) was used to estimate film compositions. Room

temperature (RT) magnetization curves were obtained by vibrating sample magnetometry (VSM). Hall measurements were performed to study the electrical properties of undoped and doped GaN:Gd layers. The LED structures were grown using n- and p- type layers that were fabricated into device structures for testing.

3. RESULTS AND DISCUSSION

3.1. The Theory of Ferromagnetism of Gd in GaN

The Gd ion in GaN introduces its own electronic states to the GaN band structure. Before taking into account coupling and hybridization effects between Gd and GaN states it is feasible to first only consider the electronic states of the isolated Gd ion in the GaN matrix. The energy schemes of Gd ions are derived from classical atom theory [25]. They differ considerably between different oxidation states. For example, the electronic structure of Gd^{2+} is strongly influenced by spin-orbit coupling, while Gd^{3+} with its exactly half filled f-shell has an orbital momentum equal to zero and, thus, is unaffected by spin-orpit coupling. In addition, the electronic structure of Gd ions is influenced by the crystal field of GaN. This effect is about three orders of magnitude smaller than for TM ions because the partially filled 4f shell is shielded by 5s and 5p electrons. The splitting caused by the crystal field can still be resolved in luminescence, transmission or magneto resonance spectra providing valuable information on the environment of the Gd ion. Carrier mediated spin coupling is of particular significance are charge transfer transitions between Gd and the bands of the host crystal, i.e., Gd may act as a donor or acceptor. Accordingly the energy needed to accomplish such a charge transfer process can be correlated with a position in the GaN energy band structure. Establishing this position is central for the evaluation of any DMS.

We have previously studied the effects of interstitial oxygen on the ferromagnetism in Gd-doped GaN system via first principles calculations, including both total energy and band structure calculation [22]. In particular, the formation energies of different interstitial O atomic configurations in GaN:Gd, the resulting ferromagnetism and its stability in n-type GaN:Gd have been studied, Figure 1. It was found that splitting interstitial sites O was a likely candidate for defect induced magnetism in N-type GaN:Gd system. Through p-d hybridization, Gd spin polarization could magnetize p state of interstitial O and in turn this hybridization renders a ferromagnetic coupling state among all Gd dopant, and a TC above room temperature. It is suggested that both the 5d and 4f orbitals of Gd take part in the coupling to O 2p orbitals. Furthermore, based on the results of formation energy of different interstitial O atomic configurations, it was found that the split-interstitial O, which could support ferromagnetism, was not the most stable site energetically. It is thought that this could be the reason why many previous experimental observations of magnetic behaviors have been contradictory.

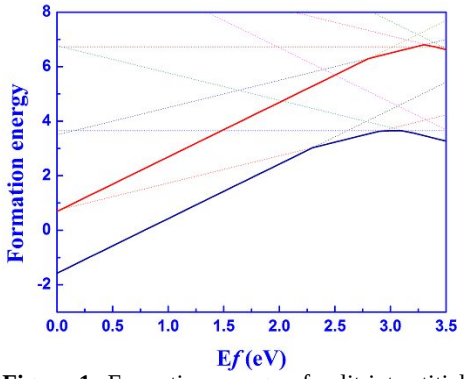


Figure 1: Formation energy of split-interstitial sites and channel center site oxygen. It was found that the channel center site, which cannot support magnetism in GaN:Gd, was always the energetically favorable configuration.

3.2. Study of effects of Gd precursor

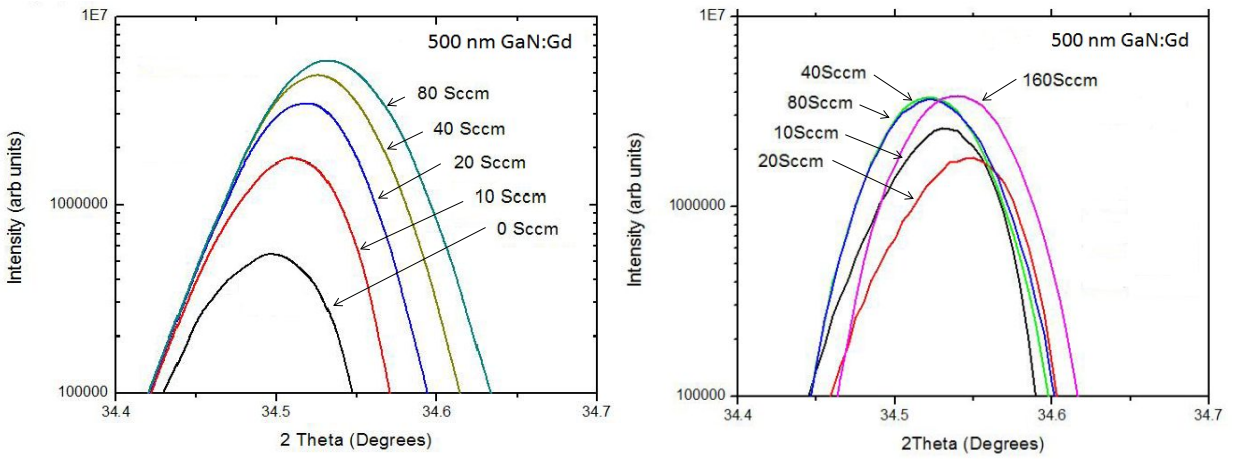
A series of GaN:Gd films were grown by MOCVD using two different Gd sources to study the role of oxygen in the observation of ferromagnetism in these materials. In this instance, Tris(2,2,6,6-tetramethyl-3,5-heptanedionato)-gadolinium ($TMHD_3Gd$) and Tris(cyclopentadienyl)-gadolinium (Cp_3Gd) were used as the Gd sources. The key difference between these sources is that the organic ligand in $TMHD_3Gd$ contains oxygen, while the ligand in Cp_3Gd does not. This means that one series of samples grown using $TMHD_3Gd$ should have potentially have oxygen incorporated in them, depending on the growth conditions, and the other series of samples grown with Cp_3Gd should not. Table 1 shows the atomic composition of GaN:Gd films produced with varying flow rates of the two precursors. Flow rates are given here because of the difficultly of measuring the actual Gd composition. The samples produced with the $TMHD_3Gd$ precursor all showed the presence of both gadolinium and oxygen in the films, however varying the flow rate

of this precursor did not appear to systematically change the observed Gd density measured by EDS. The films produced using the Cp_3Gd source all showed much higher Gd content than the $TMHD_3Gd$ films, but contained no oxygen.

Table 1: EDS data for MOCVD-grown GaN:Gd samples.

Precursor, Flow	Ga %	N %	Gd %	O %
$TMHD_3Gd$, 10 sccm	44.46	44.41	0.06	1.18
$TMHD_3Gd$, 40 sccm	49.07	48.46	0.01	0.88
$TMHD_3Gd$, 80 sccm	47.74	48.61	0.01	1.55
Cp_3Gd, 10 sccm	46.51	47.03	0.35	0.00
Cp_3Gd, 80 sccm	46.33	46.37	0.34	0.00
Cp_3Gd, 160 sccm	47.48	46.34	0.53	0.00

XRD measurements of the films showed no discreet evidence of macroscopic secondary phases of Gd_2O_3, GdN, or Gd metal; only a peak around GaN 002 was observed. 2Theta-Omega scans for the series of $TMHD_3Gd$ and Cp_3Gd films are shown in Figure 2 (a) and (b), respectively, enlarged to show detail in the GaN 002 peak. The $TMHD_3Gd$ samples showed a systematic shift of the GaN 002 peak to higher diffraction angles with increasing precursor flow. This is likely due to the incorporation of oxygen, as oxygen typically substitutes for nitrogen in GaN, and oxygen has a smaller covalent radius than nitrogen, leading to a slight decrease in lattice size. The Cp_3Gd films did not show this trend, which supports the theory that the shift is likely due to oxygen incorporation.



(a) (b)

Figure 2: 2Theta-Omega scans of the GaN 002 peaks from (a) $TMHD_3Gd$ films and (b) Cp_3Gd films Gd precursor flows are indicated for each curve.

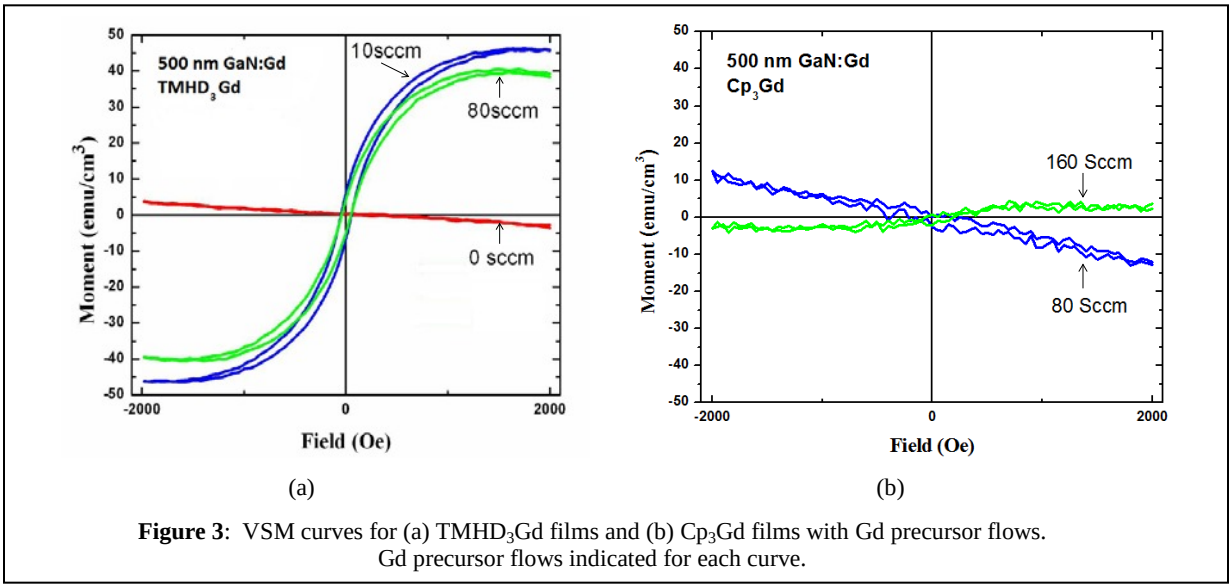
The magnetic properties of films produced with both precursors were studied using VSM. Figure 3 shows magnetization curves for (a) $TMHD_3Gd$ films and (b) Cp_3Gd films. The GaN:Gd films produced with $TMHD_3Gd$ exhibited smooth, well-defined "S" curves, with saturation magnetic moments of approximately 40 emu/cm^3. The films produced with Cp_3Gd showed much lower magnetic moments. The film produced with 80 sccm appeared to be diamagnetic, similar to the undoped GaN reference, while the film with 160 sccm precursor flow exhibited very weak ferromagnetism with a saturation magnetization of ~5 emu/cm^3. These results supported the theory that oxygen incorporation in GaN:Gd enhances the ferromagnetic behavior of the material although the actual incorporation of the oxygen and the type of interstitial, if it exists, has not yet been investigated.

3.3. A study of co-doped GaN:Gd

Ferromagnetic GaTMN (TM = Mn, Fe and Cr) has been produced by this research group and others over the last decade [8]. However, TM doping of GaN generally results in highly resistive material since TMs form deep level acceptor-like impurity bands, sometimes with compensating point defects. It is likely that observed ferromagnetism in GaTMN is explained by a double exchange mechanism involving a spin split impurity band within the band gap. This model was confirmed, in part, by the annihilation of ferromagnetism upon n-type and p-type co-doping shifting the Fermi level out

of the impurity band. It is still debated whether the observed ferromagnetism is due to substitutional TM ions in the semiconductor lattice, unwanted precipitates, or a combination of both. However, both the native formation of highly resistive material for the undoped material GaTMN and the destruction of the ferromagnetism with n-type and p-type co-doping means that these materials do not operate like a traditional Dilute Magnetic Semiconductors (DMS). The GaN:Gd films grown by MOCVD showed residual n-type conductivity, typically mid $10^{17}cm^3$, which opens the possibly to control the conductivity of these films with co-doping without destroying the ferromagnetism.

A series of n-type (Si) and p-type (Mg) GaN:Gd films were grown with expected Gd concentrations between 0.5 % and 4 % in the gas phase were grown. Typical p-GaN activation steps were applied to p-GaN:Gd. GaN:Gd films with an apparent Gd incorporation from 10^{12} cm^{-3} to 10% appeared possible in this growth system, at least in the gas phase. However, it should be understood that there are always tremendous difficulties in accurately calibrating new material compositions. X-ray diffraction (XRD) and Raman spectroscopy revealed good structural quality with no significant deterioration or strain induced through the Gd incorporation even though Gd is a large atom. XRD measurements of the samples did not detect any secondary phases or precipitates, as depicted in Figure 2. In addition, atomic force microscopy (AFM) studies revealed smooth sample surfaces with no apparent surface precipitations. Room temperature (RT) magnetization data for GaN:Gd have been obtained by vibrating sample magnetometry (VSM), Figure 4. It was observed that as the Gd flow rate is increased, a transition from diamagnetism to ferromagnetism occurs; the magnetization strength observed was 20 emu/cm^3 for 4 % Gd flow for undoped material. Additionally, the GaN:Gd could be systematically co-doped with Si or Mg and the saturation magnetization was found to be enhanced by this n-type or p-type co-doping (Figure 5). A maximum magnetization of ~500 emu/ cm^3 was obtained for p-type $Ga_{1-x}Gd_xN$ with (x = 4 %) following annealing to activate the dopant. This is the first report of observed RT ferromagnetism in p-type GaN:Gd or successful co-doping of GaN:Gd with Mg. At this time, the underlying mechanism for the observed magnetism remains unknown but this will be further investigated by Anomalous Hall Effect and Circular Magnetic Dichroism measurements since, unlike TM doped GaN, these Gd doped GaN layers were found to be conducting.



(a) (b)

Figure 3: VSM curves for (a) $TMHD_3Gd$ films and (b) Cp_3Gd films with Gd precursor flows. Gd precursor flows indicated for each curve.

Room temperature (RT) and low-temperature (LT) PL measurements performed on the epitaxial $Ga_{1-x}Gd_xN$ (x = 4 %) layers show the presence of peaks in the range of 3.1–3.3 eV in addition to the GaN emission. In the literature these peaks has been attributed to the internal transition associated with Gd^{3+} and other deep levels [26] and this could be used in optimizing the material properties.

3.4. Preliminary GaN:Gd spin-LED Results

Even though the precise mechanism for the ferromagnetism in the co-doped GaN:Gd is not well understood this does not stop the development of devices using this material as a potential spin injector. In addition, if these devices work they provide additional insight into the physical mechanism for the ferromagnetism. A series of consecutive light emitting

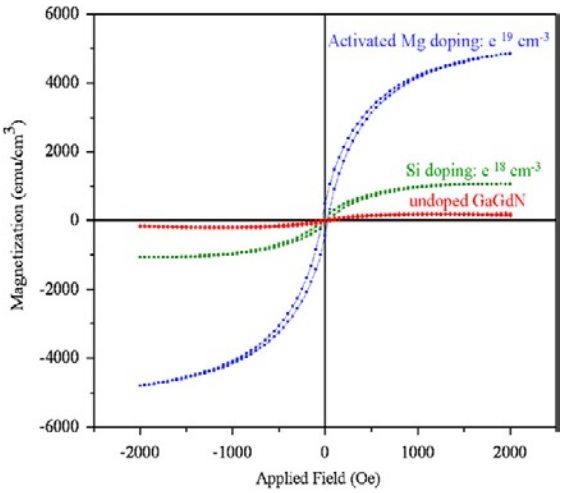


Figure 4: Room temperature magnetization curves for undoped (as grown) GaN:Gd and co-doped GaN:Gd with Si and Mg. The co-doped GaN:Gd show higher levels of saturation magnetization.

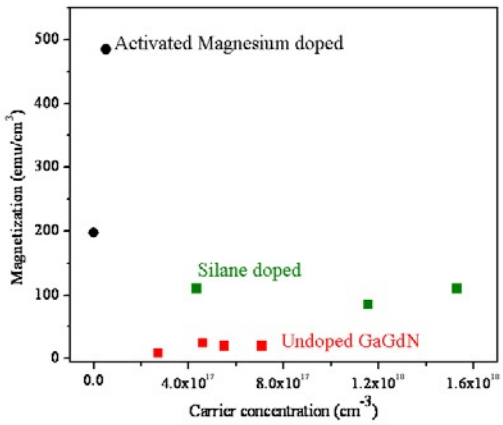


Figure 5: The effect of carrier concentration on the room temperature ferromagnetization of undoped, and Si and Mg co-doped GaN:Gd. This was the first reported observation of p-typed ferromagnetism in GaN suggesting that the Gd center strongly couples into the valence band.

diode structures were grown with and without the GaN:Gd spin injector layer. Both n-type and p-type spin injection layers were used in these devices. Simplified schematics of the GaN-based reference LED structure and the GaN:Gd-based spin-polarized LED are shown in Figure 7. These device structures consisted of a 500 nm n-type layer, followed by GaN/InGaN multiple quantum well (MQW) active region, and finally a p-type region. The n- and p- type regions in the spin LED were GaN:Gd layers based on the layers discussed in Section 3.3 and have the same physical properties. The I-V curves for the two LEDs are shown in Figure 2(c) and it can be seen that the GaN:Gd LED has a larger series resistance and a slightly higher turn on voltage. The larger series is most likely due to the slightly higher resistance of the p-type GaN:Gd compared to the p-type GaN grown under the same conditions.

The GaN:Gd LED and the reference GaN LEDs were both mounted on a non-magnetic DIP package for functional testing under magnetic field. This testing was performed in a Faraday configuration, in which the LED is placed inside the poles of an electromagnet capable of generating up to 5000 Gauss magnetic field. Current is passed through the devices, and the resulting electroluminescence (EL) is collected and focused through a quarter wave (QW) plate. As expected, no response for was seen for the reference GaN LED with magnetic field for either Right Circularly Polarized (RCP) or Left Circularly Polarized (LCP) EL from these devices, Figure 8. This was not the case for the GaN:Gd LED which showed a systematic variation of the RCP and LCP emission in with magnetic field, Figure 9. The primary figure of merit for a spin LED is the degree of polarization, P_{spin}, which is defined as the difference between the left and the right circularly polarized light intensities divided by their sum. A maximum EL polarization of 14.6% was observed at an applied field of 5000 Gauss at room temperature shown evidence for spin injection at room temperature. This is comparable to the 22.1% polarization at 10,000 Gauss and 10 Kelvin reported for a $Ga_{1-x}Mn_xAs$ LED [27].

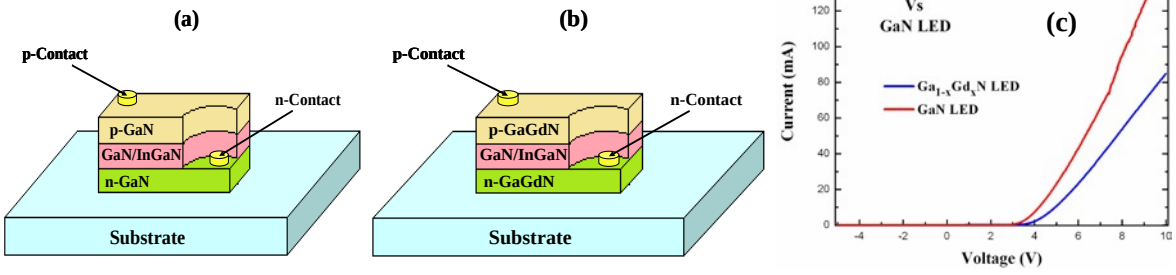


Figure 7: Schematic of LED device structures of (a) reference LED, (b) $Ga_{1-x}Gd_xN$ LED, and (c) I-V curves for both structures.

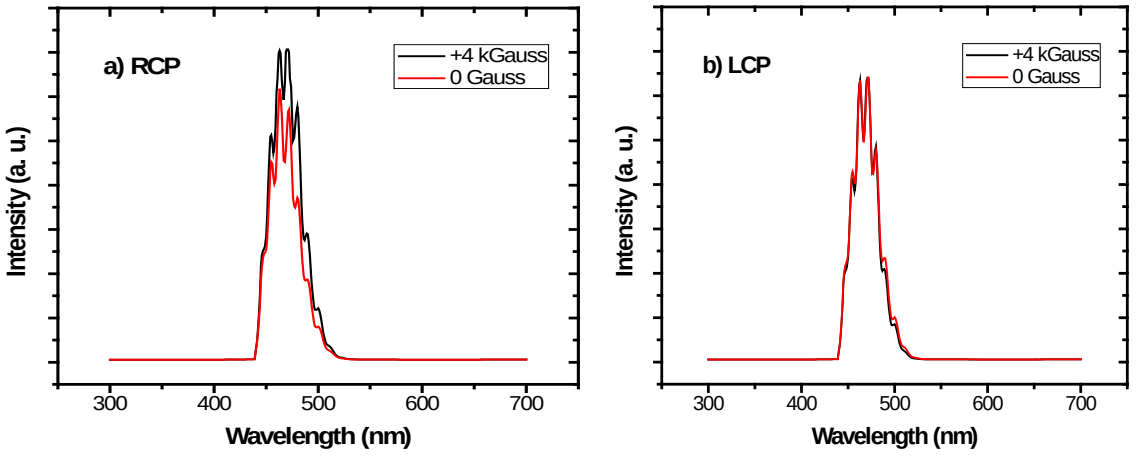


Figure 8: (a) Right and (b) Left circularly polarized EL from reference GaN-based LED.
Both right and left circularly polarized emission are minimally affected by a 4 kGauss applied field.

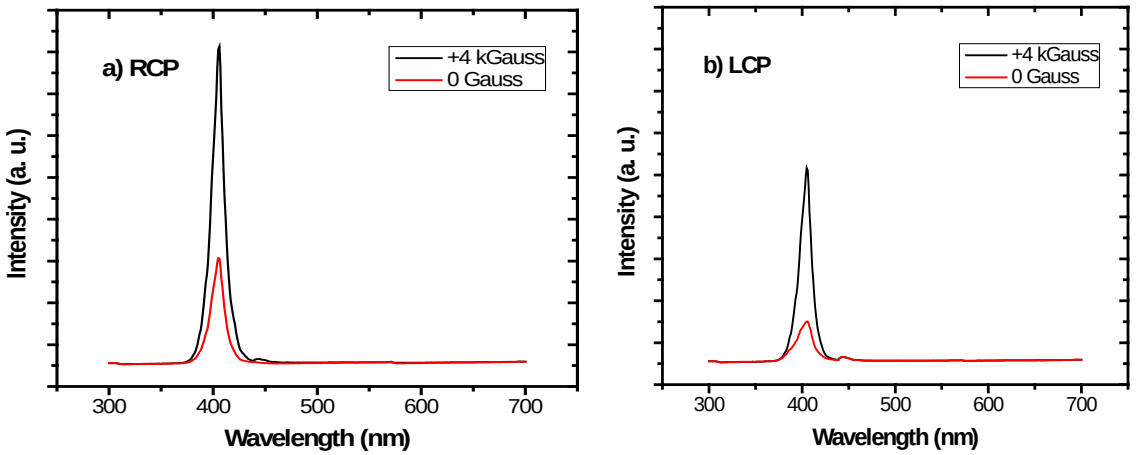


Figure 9: (a) Right and (b) left circularly polarized EL from GaN:Gd-based spin LED (plotted on same scale).
Both right and left circularly polarized light are significantly increased by a 4 kGauss applied field.

Figure 10 shows the normalized values of P_{spin} for the GaN:Gd LED at sequentially applied magnetic fields. These values form one quadrant of a hysteresis curve starting at 0 Gauss, sweeping up to 5000 Gauss, then back to 0 Gauss. The EL polarization of 14.6% was highest observed in this study at an applied field of 5000 Gauss however the EL polarization did not appear to have saturated at this value of field. The final measurement shows a persistent EL polarization of 9.3% after removal of the applied magnetic field, i.e. at 0 Gauss. It was found that the sign of the applied magnetic field did not change the sign of the EL spin polarization for this device. Additional studies are needed to fully understand this behavior and to investigate the performance of alternate GaN:Gd-based device structures.

4. CONCLUSIONS

Gadolinium-doped GaN (GaN:Gd) is one of the most intriguing dilute magnetic semiconductors. However, the microscopic mechanisms for the incorporation of Gd in GaN and its electrical, optical and magnetic properties are still subject to extensive scientific research and debate. Many first-principles calculations based on density functional theory (including those by this research group) predict that crystal defects are important contributors to the observed magnetic moments. In particular, interstitial oxygen has been predicted to add to the magnetic moment of substitutional Gd in GaN, and early experimental results from the PI have supported this hypothesis. The interplay of the electrical, optical and magnetic properties of GaN:Gd as spin-injection layers is not well understood and require further investigations. The apparent role of oxygen in coupling the magnetic moments of dilute concentrations of Gd in GaN:Gd suggests that ZnO:Gd could also be a viable spin-injection material due to the large concentration of oxygen in the crystal lattice. The chemical and physical effects of this interaction require future systematic studies in order to develop room temperature III-Nitride spintronic devices.

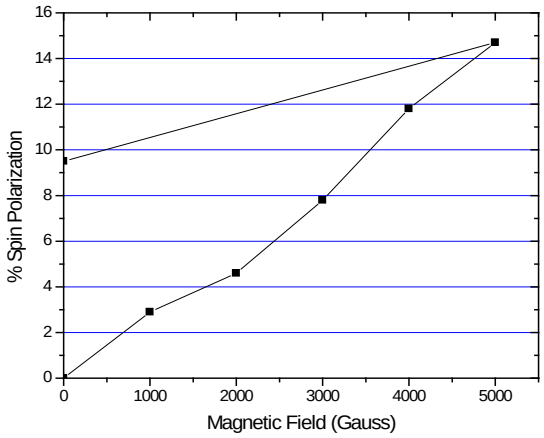


Figure 10: Degree of polarization of EL from $Ga_{1-x}Gd_xN$-based LED at sequentially applied magnetic fields (room temperature).

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