Process dependence of H passivation and doping in H-implanted ZnO

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We used depth-resolved cathodoluminescence spectroscopy (DRCLS), photoluminescence (PL) spectroscopy and temperature-dependent Hall-effect (TDHE) measurements to describe the strong dependence of H passivation and doping in H-implanted ZnO on thermal treatment. Increasing H implantation dose increases passivation of Zn and oxygen vacancy-related defects, while reducing deep level emissions. Over annealing temperatures of 100–400 °C at different times, 1 h annealing at 200 °C yielded the lowest DRCLS deep level emissions, highest TDHE carrier mobility, and highest near band-edge PL emission. These results describe the systematics of dopant implantation and thermal activation on H incorporation in ZnO and their effects on its electrical properties.
Process dependence of H passivation and doping in H-implanted ZnO

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Abstract
We used depth-resolved cathodoluminescence spectroscopy (DRCLS), photoluminescence (PL) spectroscopy and temperature-dependent Hall-effect (TDHE) measurements to describe the strong dependence of H passivation and doping in H-implanted ZnO on thermal treatment. Increasing H implantation dose increases passivation of Zn and oxygen vacancy-related defects, while reducing deep level emissions. Over annealing temperatures of 100–400 °C at different times, 1 h annealing at 200 °C yielded the lowest DRCLS deep level emissions, highest TDHE carrier mobility, and highest near band-edge PL emission. These results describe the systematics of dopant implantation and thermal activation on H incorporation in ZnO and their effects on its electrical properties.

(Some figures may appear in colour only in the online journal)

1. Introduction
The wide band gap semiconductor ZnO \( (E_g \approx 3.4 \text{ eV}) \) is a leading candidate for next generation opto- and micro-electronics due to its high exciton binding energy, thermo-chemical stability, environmental compatibility and potential applications for light-emitting devices and photovoltaics [1, 2]. As-grown un-doped ZnO is naturally n-type, usually attributed to native defects [3, 4] and/or various donor impurities, such as Al, In, H [5–7]. However, the origin of the n-type conductivity is still controversial, and the ability to understand and control the electrical behaviour of intrinsic and impurity related defects in ZnO remains a fundamental, unresolved challenge. Hydrogen is one of the most common but important impurities during various synthesis processes of ZnO [8, 9]. It could contribute to n-type ZnO conductivity by passivating ionized compensating acceptors [10–15] or acting as shallow donors [7, 16–24]. Previous theoretical studies predicted that H is likely to passivate zinc vacancy \( (V_{Zn}) \) and substitutional lithium on zinc site \( (Li_{Zn}) \) defects by forming neutral \( X_{Zn}–H \) complexes [13, 14], and the H passivation of acceptor defect states have been confirmed by photoluminescence (PL) spectroscopy [10, 11], infrared (IR) absorption spectroscopy [12] and positron annihilation spectroscopy (PAS) [15]. Different local vibrational modes in the IR spectra were related to different hydrogen-related defects in ZnO [12, 25–27]. Hydrogen is also a shallow donor in ZnO according to first-principles calculations [7, 17], confirmed experimentally [9, 16, 18–24]. Various experiments identify two types of H donors: (1) a bound exciton emission peak described as \( I_4 \) in PL spectroscopy [19, 20], which was later identified as hydrogen bound with an oxygen vacancy [24], \( H_{O_4} \), consistent with Janotti and Van de Walle’s theory [7]; (2) hydrogen at the bond-centred interstitial site [24], \( H_{BC} \) from IR absorption spectroscopy [12]. Moreover, H has also been suggested for possible p-type fabrication by improving acceptor solubility through forming H-acceptor complexes [13]. Here we present results aimed at understanding the influence of dopant implantation and thermal activation on H incorporation in ZnO in order to control its electrical properties.
Based on combined depth-resolved cathodoluminescence spectroscopy (DRCLS), PAS and other surface science techniques, our previous work correlated the commonly observed 1.9–2.1 eV ‘red’ and 2.3–2.5 eV ‘green’ luminescence with zinc vacancies and vacancy clusters (denoted (V\textsubscript{Zn})\textsubscript{n})-related and oxygen vacancy (V\textsubscript{O})-related defects, respectively [28]. PAS and secondary ion mass spectrometry (SIMS) profiles demonstrate that, compared with V\textsubscript{Zn}, the Li\textsubscript{Zn} acceptor is the dominant trap for H in hydrothermally grown (HT-) ZnO [29]. H implantation could form an n\textsuperscript{+} layer in ZnO due to H donor and implantation-induced defects [22]. It has also been shown that the H is trapped in ZnO for temperatures below 250°C [30]. Moreover, temperature-dependent Hall-effect (TDHE) measurements show that 200°C annealing in Ar after H implantation results in the highest carrier mobility [31, 32]. However, the implantation and thermal dependence of H incorporation and its electrical behaviour in ZnO is still under debate.

In this work, we report on the passivation and donor effect of ion-implanted H with various concentrations up to 3 × 10\textsuperscript{18} cm\textsuperscript{-3} in single-crystal bulk hydrothermal (HT) ZnO. We correlated deep band DRCLS emission intensities with implanted H concentration, the thermal process dependence of defect-H passivation, and the resultant electrical properties of H-implanted ZnO. From combined experiments, we summarize optimal annealing conditions for H doping and defect passivation.

2. Experimental

Six n-type HT-ZnO wafers with a size of 10 × 10 × 0.5 mm\textsuperscript{3} were used in the study. They are labelled HR, H1, H2, H3, H4 and HH4. HR represents as-grown high resistivity HT-ZnO, while H1–H4 specimens were HT-ZnO implanted on the O face at room temperature (RT) with multiple H\textsuperscript{+} beam energies to produce approximately box-like profiles of different H concentrations, shown by the simulated profiles in figure 1 using the SRIM-code [33]. The as-grown HT-ZnO has unintentionally doped high Li background concentration (1–5) × 10\textsuperscript{17} Li cm\textsuperscript{-3}) and high resistivity (1–10 kΩ cm) due to self-compensation and the low energy barrier for Li to switch between interstitial and substitutional sites [14, 34]. One quarter of each wafer (HR-3, H1-3, H2-3, H3-3 and H4-3) was heat treated from annealing temperature (T\textsubscript{a}) 100°C to 400°C in 100°C steps, with DRCL spectra acquired after each annealing step. In the annealing process, the temperature rose from RT to set temperature (100°C, 200°C, 300°C or 400°C) in 30 min, and kept at set temperature for 1 h. Annealing at different times was carried out on another H-implanted ZnO HH4, which has same H implantation dose as H4. HH4 was annealed at 200°C, 300°C and 400°C for 30 min, 1 h, 2 h and 4 h, respectively. Two quarters of HR, H2, and H4 wafers received annealing treatments in different sequences: they were first annealed at 100°C (HR-1, H2-1 and H4-1) and 200°C (HR-2, H2-2 and H4-2), and then annealed at 300°C (HR-1, H2-1 and H4-1) and 400°C (HR-2, H2-2 and H4-2). TDHE and PL features were measured for these specimens after each annealing.

![Figure 1. Simulated profile of H concentration in ZnO after H implantation using the SRIM-2006 code. The H concentrations are box profiles extending to depths of ~3.5 μm.](Image)

DRCLS measurements were obtained on ZnO (0001) faces at 80 K in ultrahigh vacuum. See [35, 36] for DRCLS experiment details. Monte Carlo simulation [37] provides depth distributions of the electron–hole pairs generated by the incident electron beam versus E\textsubscript{B}. For incident energy E\textsubscript{B} = 1, 2, 3, 4 and 5 keV, electron–hole pair excitation is maximum at U\textsubscript{B} = 7, 18, 32 and 72 nm, respectively. The energy power of the incident beam was P\textsubscript{B} = 2 mW with a spot diameter of 0.5 mm. References [38, 39] provide descriptions of the TDHE and PL measurements.

3. Results

3.1. Deep band defect passivation with H implantation

Figure 1 shows the simulated profile of H concentration as a function of depth for samples H1, H2, H3 and H4. The multiple H-implantations in each sample produce box areas of roughly constant H concentration: 3 × 10\textsuperscript{17} cm\textsuperscript{-3}(H1), 6 × 10\textsuperscript{17} cm\textsuperscript{-3}(H2), 1 × 10\textsuperscript{18} cm\textsuperscript{-3}(H3) and 3 × 10\textsuperscript{18} cm\textsuperscript{-3} (H4) that extend to depths of ~3.5 μm. E\textsubscript{B} = 5 keV CL spectra in figure 2 display the evolution of defect emissions with increasing H implantation dose. Each CL spectrum was normalized relative to its near band-edge (NBE) emission intensity. Figure 2 shows that the deep band emissions at ~2.06 eV and ~2.50 eV both decrease with increasing H implantation dose. A minor exception to this trend is H2, which also deviated in other trends as well. Overall, the highest H dose corresponds to the lowest deep band emission, which suggests that the implanted H passivates not only the Zn vacancy, but also the oxygen vacancy. Figure 2 illustrates that the passivation effect increases with increasing H implantation dose. The inset in figure 2 displays the evolution of deconvolved broad deep level emission peak intensity I\textsubscript{D} normalized to NBE intensity I(NBE), denoted as I\textsubscript{D}/I(NBE) for H(1) and H(2). Both ratios
Figure 2. Dose-dependent 80 K \( E_{\text{in}} = 5 \) keV CL spectra of H-implanted ZnO. H implantation reduces the intensity of \( V_{\text{Zn}} \) (∼2.06 eV) and \( V_{\text{O}} \) (∼2.50 eV). Inset shows the deep band emission decrease with increasing H implantation dose.

Figure 3. 80 K CL spectra for H-implanted H4-2 after annealing at different temperatures. The inset shows the changes in H4 deep band emission versus post-implant anneal temperature. The 200 °C temperature has the lowest deep band emission.

3.2. Electrical properties of H-implanted and annealed ZnO

Tables 1–3 display the results of electrical measurements on H-implanted and annealed ZnO samples H1–H4. They show that annealing temperature has a strong effect on the electrical properties of H-implanted ZnO. HR, H2 and H4 carrier mobilities all reach their maximum values after a 200 °C anneal, then decreased steadily with increasing temperature above 200 °C. This mobility decrease is attributed to increased removal of H from otherwise passivated ionized scattering centres such as \( \text{Li}_{\text{Zn}}, V_{\text{Zn}} \) and other defects reported previously [10–12, 29]. For H2 and H4, H passivation can be attributed to the implanted H, while the rather limited passivation of HR could come from ‘hidden hydrogen,’ identified previously as a hydrogen molecule [40, 41].

Carrier concentrations in table 2 show ∼10⁴ higher values for H2 versus as-grown HR and 10²–10³ higher values for high-dose implanted H4 versus HR. Correspondingly, H2’s \( \rho \) is much lower than that of HR and H4. This is consistent with the deviation of H2 from the defect emission intensity versus dose trend in figure 2. Note: ∼5% of the HT-wafers (Goodwill SPC) have higher than expected \( n \) at RT. Tables 2 and 3 show that \( n \) for H-implanted H2 and H4 versus anneal temperature exhibits maxima for 300 °C anneal, i.e. 100 °C higher than the temperature for the highest carrier mobility. The TDHE measurements in figure 5 illustrate these differences in \( n \) and \( \mu \) behaviour with annealing temperature. After the 200 °C post-implant anneal, figure 5(a) shows that H4 has the highest carrier mobility, while figure 5(b) shows that \( n \) reaches its maximum value after a 300 °C anneal. Section 4 discusses this in detail.
3.3. NBE emission of H-implanted ZnO

The PL spectra in figure 6 show that H4 annealed at 200 °C has the highest NBE emission of all anneal temperatures. The donor bound-exciton transitions at 3.3610 and 3.3617 eV first increase after 200 °C annealing, especially for the peak around 3.3617 eV. With higher annealing temperatures, the peak around 3.3610 eV disappears and the peak around 3.3617 eV decreases more than twice. Previous theory and experiment [42–44] have identified the bound-exciton luminescence at 3.3610 eV as O–H bonds at Zn vacancies, in the form of 4(O–H)–VZn, which gives rise to shallow donors. The peak around 3.3617 eV could be related to 3(O–H)–VZn, which gives rise to shallow donors. The peak around 3.3617 eV as O–H bonds at Zn vacancies, in the form of 4(O–H)–VZn, which gives rise to shallow donors. The peak around 3.3617 eV as O–H bonds at Zn vacancies, in the form of 4(O–H)–VZn, which gives rise to shallow donors.

According to our previous calibration of the DRCLS defect density [28], the (VZn)NBE concentration of (1–2) × 1018 cm−3 in HT-ZnO corresponds to \( I_D/(\sim 2.06 \text{ eV})/\text{NBE} \sim 1 \) in CL spectra. Figure 2 shows \( I_D/(\sim 2.06 \text{ eV})/\text{NBE} \sim 0.88 \) for as-grown HT-ZnO sample, corresponding to a (VZn)NBE concentration of (0.9–1.7) × 1018 cm−3. Likewise, the concentration of (VZn)NBE in the highest H dose implanted H4 corresponds to \( \sim 1.1 \times 10^{17} \text{ cm}^{-3} \) after H passivation of Zn vacancy defects.

Figure 3 shows that H reduction of deep level emission and defect passivation are optimal at 200 °C. Moreover, figure 4 indicates that 1 h anneal is the optimal annealing time for H to passivate the defects, corresponding to the lowest CL defect emission. \( I_D/(\sim 2.06 \text{ eV})/\text{NBE} \sim 0.01 \) of H4 after the 1 h 200 °C anneal corresponds to a minimum (VZn)NBE concentration of \( \sim 2 \times 10^{16} \text{ cm}^{-3} \). At higher temperatures, H passivated defects begin to dissociate. This is consistent with the trap limited diffusion model for implanted H in ZnO, in which implanted H trapped in ZnO will diffuse out starting from 250 °C [31]. The TDHE measurement of highest \( \mu \) for H4 after a 200 °C anneal in figure 5 is consistent with the implanted H passivating the LiZn, VZn and other defects, decreasing the scattering by charged centres and thus increasing the carrier mobility. This effect was also observed on previously annealed hydrogen-implanted HT-ZnO [32].

Lizn is also an acceptor in HT-ZnO, which shows a CL peak around 3.0 eV [45]. Because of the relatively low Li concentration in these HT-ZnO samples, the Lizn acceptor could not be detected by our system. However, PAS results of H-implanted ZnO clearly demonstrate the passivation of Lizn by H; furthermore, H passivates Lizn before VZn acceptors [29]. H passivates VZn by forming H5+ donors through the following

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**Table 1.** RT electrical properties of as-grown HT-ZnO sample HR after annealing in air at the indicated temperatures. Annealing at 200 °C produced the highest \( \mu \) and \( n \) plus the lowest \( \rho \).

<table>
<thead>
<tr>
<th>Annealing T (°C)</th>
<th>100</th>
<th>200</th>
<th>300</th>
<th>400</th>
</tr>
</thead>
<tbody>
<tr>
<td>( \mu ) (cm² V⁻¹ S⁻¹)</td>
<td>109</td>
<td>112</td>
<td>108</td>
<td>106</td>
</tr>
<tr>
<td>( n ) (cm⁻³)</td>
<td>7.75 × 10¹⁴</td>
<td>7.83 × 10¹⁴</td>
<td>7.71 × 10¹⁴</td>
<td>7.61 × 10¹⁴</td>
</tr>
<tr>
<td>( \rho ) (Ω cm)</td>
<td>74.0</td>
<td>71.4</td>
<td>75.3</td>
<td>77.48</td>
</tr>
</tbody>
</table>

**Table 2.** RT electrical properties of H-implanted HT-ZnO sample H2 with H box concentration of 6 × 10¹⁷ cm⁻³ after annealing in air at different temperatures. Annealing at 200 °C produced the highest \( \mu \) nearly the highest \( n \), and the lowest \( \rho \).

<table>
<thead>
<tr>
<th>Annealing T (°C)</th>
<th>100</th>
<th>200</th>
<th>300</th>
<th>400</th>
</tr>
</thead>
<tbody>
<tr>
<td>( \mu ) (cm² V⁻¹ S⁻¹)</td>
<td>146</td>
<td>152</td>
<td>143</td>
<td>127</td>
</tr>
<tr>
<td>( n ) (cm⁻³)</td>
<td>9.04 × 10¹⁸</td>
<td>9.0 × 10¹⁸</td>
<td>9.34 × 10¹⁸</td>
<td>7.19 × 10¹⁸</td>
</tr>
<tr>
<td>( \rho ) (Ω cm)</td>
<td>0.00472</td>
<td>0.00457</td>
<td>0.004663</td>
<td>0.006809</td>
</tr>
</tbody>
</table>

**Table 3.** RT electrical properties of H-implanted HT-ZnO sample H4 with H box concentration of 3 × 10¹⁸ cm⁻³ after annealing in air at different temperatures. Annealing at 200 °C produced the highest \( \mu \), but annealing at 300 °C produced the highest \( n \), and the lowest \( \rho \).

<table>
<thead>
<tr>
<th>Annealing T (°C)</th>
<th>100</th>
<th>200</th>
<th>300</th>
<th>400</th>
</tr>
</thead>
<tbody>
<tr>
<td>( \mu ) (cm² V⁻¹ S⁻¹)</td>
<td>112</td>
<td>170</td>
<td>149</td>
<td>142</td>
</tr>
<tr>
<td>( n ) (cm⁻³)</td>
<td>9.34 × 10¹⁵</td>
<td>4.45 × 10¹⁷</td>
<td>7.30 × 10¹⁷</td>
<td>1.33 × 10¹⁷</td>
</tr>
<tr>
<td>( \rho ) (Ω cm)</td>
<td>5.98</td>
<td>0.0823</td>
<td>0.05743</td>
<td>0.3311</td>
</tr>
</tbody>
</table>

---
reaction [7]:
\[
\text{VO} + \text{H}^+ \rightarrow \text{H}_2\text{O}.
\]

(1)

This is consistent with both (i) figure 2 showing that higher H implantation leads to lower 2.5 eV VO-related defects and (ii) tables 1–3 showing that \(n\) increases with the H implantation. The H\(2\)\(_0\) has been identified as donor bound exciton I\(_4\) at 3.3628 eV by PL [24]. Figure 6 indicates this 3.3628 eV peak at a 200°C anneal. Its relatively low intensity may be due to H passivating the acceptor-like defects before forming donors can account for the low I\(_4\) intensity in H-implanted H\(4\).

The H\(+\)O has been identified as donor bound exciton I\(_4\) at 3.3628 eV by PL [24]. Figure 6 indicates this 3.3628 eV peak at a 200°C anneal. Its relatively low intensity may be due to H passivating the acceptor-like defects before forming donors can account for the low I\(_4\) intensity in H-implanted H\(4\).

TDHE measurements support this interpretation. Figure 5(a) shows that H\(4\) after a 200°C anneal reached the highest \(\mu\) due to H passivating most of the acceptors and other charged scattering centres. Likewise, 5(b) shows that H\(4\)'s \(n\) increases to its highest value after the 300°C anneal due to implanted H passivating VO and forming shallow H\(2\)\(_0\) donor without dissociating the passivated acceptor defects. However, with more donors, ion scattering increases, thereby decreasing \(\mu\). This trend accounts for the \(n\) value maximum at a higher anneal temperature than that of the \(\mu\) value maximum. At higher temperatures, these H passivated complexes begin to dissociate, leaving the initial ionized scattering centre. Other H-implanted ZnO studies have also shown that 200°C anneal in Ar leads to highest carrier mobility [32, 33], the same as our annealing results in air, demonstrating the consistence of 200°C as the optimal annealing temperature. It also indicates that at low annealing temperature, below 400°C as in this study, annealing environment does not affect the annealing effect substantially. Here the 200°C annealing increases the RT carrier mobility of H\(4\) by \(\sim 1.5 \times\) relative to HR. These results show clearly that H passivation of ion scattering centres increases carrier mobility.

4.2. H passivation effect versus donor effect

TDHE measurements of H plasma-treated ZnO film demonstrated that H not only passivates defects and acceptors but also forms shallow donors [46]. Previous research also showed that H in low-defect ZnO samples grown by vapour phase transport will form H\(2\)\(_0\) donors that are detected by PL and labelled I\(_4\) [7,18–21,24]. For HT ZnO, however, Zn vacancy, O vacancy and Li\(_{Zn}\) acceptor defect intensities are relatively high so that implanted H passivation of these defects before forming donors can account for the low I\(_4\) intensity in H-implanted H\(4\).

Figure 6. Photoluminescence spectroscopy of H-implanted H\(4\) at low temperatures (4 K). Post-implanted H\(4\) annealed at 200°C has the highest NBE emission intensity.

Figure 5. TDHE measurement of H-implanted H\(4\) shows \(\mu\) (a) and \(n\) (b) versus anneal temperature. The H\(4\) \(\mu\) reaches a maximum after 200°C annealing, while \(n\) reaches its maximum after 300°C annealing.
passivation of acceptors and formation of donors, while the lower $\mu$ values is due to a net increase in scattering centres.

4.3. TDHE measurement of donor and acceptor states

The TDHE measurements fitted with $E_{LO} = 69$ meV (details of the fitting procedure can be found elsewhere [47]) in figure 5 show that the donor concentration of approximately $1 \times 10^{18}$ cm$^{-3}$ for H4-2 200 °C is relatively high compared with the background concentration of other donor impurities in as-grown HT-ZnO such as Al, which is on the order of $10^{17}$ cm$^{-3}$ [48]. Furthermore, figure 1 shows that the implanted H is about $3 \times 10^{18}$ cm$^{-3}$. Therefore, implanted H accounts for the high donor concentration with approximately 30% implanted H activated as donors and the remaining implanted H passivating acceptors and defects.

Figure 5 shows that the donor activation energy is 28 meV [33], close to the activation energy of hydrogen bound in an oxygen vacancy $V_{Zn}$, reported previously [9, 23]. IR spectroscopy would be useful to identify the H configuration in these samples; however, its sensitivity is not high enough to study concentrations of about $3 \times 10^{18}$ H cm$^{-3}$ in layers with a thickness of only 3.5 $\mu$m (see, e.g. [24]). The background bulk concentration of H is a few times $10^{17}$ H cm$^{-3}$ in these samples (500 $\mu$m thick) and this will dominate the FTIR spectra. In addition, the acceptor concentration in figure 5 is $5.5 \times 10^{16}$ cm$^{-3}$—close to the DRCLS estimate of $\sim 2 \times 10^{16}$ cm$^{-3}$($V_{Zn}$)$_{H}$ density in H4 and consistent with ($V_{Zn}$)$_{H}$ as the main acceptor after H passivation of LiZn [29].

As-grown sample HR and H-implanted H2 also showed similar CL, TDH and PL trends as H4 with different annealing temperatures. All of these results indicate that 1 h 200 °C is the optimal annealing condition for H to passivate defects in ZnO and increasing H concentration increases the defect passivation. The implanted H also increases the HT-ZnO carrier mobility and concentration and decreases resistivity by passivating ionized scattering centres, releasing compensated carrier mobility and concentration and decreases resistivity by passivating ionized scattering centres, releasing compensated.

5. Conclusions

This study provides several main conclusions. (1) Implanted H in HT-ZnO decreases deep band $V_{Zn}$ and $V_{O}$-related defect emissions with increasing H dose as H passivates Zn and O vacancies. (2) 1 h annealing at 200 °C is the most effective post-implant anneal condition to passivate deep band defects and increase carrier mobility. (1) The 300 °C anneal activates more shallow donors while preserving acceptor passivation. (4) Annealing at higher temperatures or longer time weakens the passivation effect as implanted H dissociates, increasing deep band emission, lowering carrier mobility, as well as reducing NBE PL emission.

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