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## Research Article

### STRUCTURAL, OPTICAL AND SQUID STUDIES OF SILICON AND MANGANESE ION CO-IMPLANTED GALLIUM NITRIDE

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#### ABSTRACT

In the present work, 5 MeV Si<sup>++</sup> ions for the ion fluence of  $1 \times 10^{16} \text{ cm}^{-2}$  and 325 keV Mn<sup>+</sup> ions for the fluence of  $2 \times 10^{16} \text{ cm}^{-2}$  co-implanted into un-doped GaN film of thickness 1.90  $\mu\text{m}$ , grown on sapphire substrate. Structural, optical and magnetic properties of non-implanted, Mn-implanted and co-implanted GaN samples were studied using X-ray diffraction, Raman scattering and superconducting quantum interference device techniques. XRD studies of non-implanted, Mn-implanted and co-implanted GaN samples showed the c-plane lattice constant was found to be 0.5181 nm, 0.5158 nm and 0.5181 nm respectively. The result revealed the expansion of GaN lattice due to the incorporation of Mn<sup>+</sup> ions in the samples. Raman spectra showed the peaks at  $300 \text{ cm}^{-1}$  and  $670 \text{ cm}^{-1}$  assigned to vibrational modes of gallium and nitrogen vacancy related defects respectively. SQUID studies showed the enhancement in magnetic properties after co-implantation with silicon ion. The Curie temperature estimated from zero field and field cooled curves for Mn-implanted and co-implanted with silicon ion into GaN samples was found to 301 and 326 K.

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#### INTRODUCTION

The gallium nitride and gallium antimonide based dilute magnetic semiconductors have recently attracted an interest due to their applications in magnetic semiconductor devices. These devices have wide applications such as magnetic field sensors, switches and high power devices [1, 2]. Many methods have been developed to prepare the (Ga, Mn)N dilute magnetic semiconductors but obstacle occurs to maintain the prominent percentage of manganese element into gallium nitride because of the solid solubility limit [3, 4]. Ion implantation has obvious advantages to overcome this problem by introducing impurities in an excess of the equilibrium of solid solubility [5]. However, it causes damage in the crystal lattice as the ions loose energy during travel in the crystal. Some efforts have been made in the past to synthesize the (Ga,Mn)N dilute magnetic semiconductor by ion implantation technique and their re-crystallization [6-9]. SQUID and TEM studies of 350 keV manganese ion implanted gallium nitride for the fluence of  $5 \times 10^{16} \text{ ions cm}^{-2}$  showed the ferromagnetic behavior up to 250 K and platelet structures of Ga<sub>x</sub>Mn<sub>1-x</sub>N respectively [6]. SQUID study of 200 keV manganese ion implanted GaN for the fluence of  $5 \times 10^{16} \text{ ions cm}^{-2}$  showed the ferromagnetic behavior up to the temperature 270 K after rapid thermal annealed at 850 °C for 20 s. Whereas, photoluminescence measurement showed the optical transitions related to manganese at 2.5 and 3.0 eV corresponds to donor-

Mn acceptor and conduction band-Mn acceptor transitions respectively [7]. SQUID and XRD studies of 1 keV manganese ion implanted GaN/Al<sub>2</sub>O<sub>3</sub> for the fluence of  $2.5 \times 10^{14} \text{ ions cm}^{-2}$  showed the hysteresis loop at 293 K and the (Ga,Mn)N structure respectively [8]. SQUID Study of 75 keV manganese ion implanted GaN for the fluence of  $8 \times 10^{17} \text{ ions cm}^{-2}$  revealed the ferromagnetic behavior at 100 K after annealing at 850 °C for 30 min [9].

In this work, gallium nitride thin film grown on sapphire substrate was implanted with 325 keV manganese ions for the fluence of  $2 \times 10^{16} \text{ ions cm}^{-2}$  at room temperature. The implanted samples were again implanted with 5 MeV Si<sup>++</sup> ions for ion fluence of  $1 \times 10^{16} \text{ ions cm}^{-2}$  at 350 °C substrate temperature. The structural, optical and ferromagnetic properties of non-implanted, Mn-implanted and co-implanted samples were investigated using XRD, Raman scattering and SQUID techniques.

##### Experimental details

Un-doped GaN film of thickness 1.90  $\mu\text{m}$ , grown on sapphire substrate by metal organic chemical vapor deposition (MOCVD) were uniformly implanted with 325 keV Mn ions at temperature using 1.7 MV Tandemron accelerator at IGCAR, Kalpakkam. The projected range (Rp) and standard deviation ( $\Delta\text{Rp}$ ) of 325 keV manganese ions in gallium nitride calculated

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from SRIM code were found to be 261.3 nm and 102.8 nm respectively [10]. The beam current density was about 50 nA cm<sup>2</sup> during implantation. The scanned beam was further collimated through a collimator of diameter 12.5 mm for uniform implantation over the entire area of the sample. During implantation, the vacuum in the target chamber was maintained at 10<sup>-7</sup> mbar. The gallium nitride samples were implanted with manganese ions for fluence of 2.0 × 10<sup>16</sup> ions cm<sup>-2</sup>. The samples implanted with Mn<sup>2+</sup> ion for the fluence of 2 × 10<sup>16</sup> ions cm<sup>-2</sup> were again implanted with 5 MeV Si<sup>++</sup> ions for the fluence of 1 × 10<sup>16</sup> ions cm<sup>-2</sup> at 350 °C substrate temperature. The 5 MeV Si<sup>++</sup> ion implantation was performed for re-crystallization of the GaN film and to remove the Mn<sup>+</sup> implantation damage. The energy of silicon beam was chosen such that it pass through the GaN film and stops in the sapphire. X-ray diffraction of non-implanted implanted and after implanted with 5 MeV Si<sup>++</sup> ion samples were recorded on X-ray diffractometer (JEOL model JDX- 8030) equipped with Cu Kα (λ~ 0.1540 nm) radiation source. The primary excitation current for the experiment was 30 mA at 40 kV. The detection period and step size were kept 1.0 degree per minute and 0.02 degree respectively. Raman spectra of the samples were recorded on a LABRAM-I Micro-Raman spectrometer (JobinYvonSpex make) at Bhabha Atomic Research Centre (BARC), Mumbai. These measurements were carried out at room temperature using 488 nm line of the Ar<sup>+</sup> laser for excitation and the scattered light was detected in a backscattering geometry using multi-channel CCD detector. The ferromagnetic properties of Mn<sup>+</sup> implanted gallium nitride samples were studied using superconducting quantum interference device (SQUID) magnetometer (MPMSXL-Quantum design Co. Ltd.) For this measurement, implanted samples were mounted plane parallel to the applied field. Magnetization was measured as function of applied magnetic field and temperature to determine the coercive field and other magnetic properties. Zero-field-cooled (ZFC) and field-cooled (FC) magnetization curves as function of temperature were recorded. In the ZFC mode, the samples were cooled in zero field from 350 K to 1.8 K. After stabilization of the temperature, the constant field 500 (Oe) and 1000 (Oe) were applied. The data were recorded while heating the samples. In the FC mode, the samples were cooled from 350 K to 1.8 K in the presence of a constant magnetic field of 500 (Oe) and 1000 (Oe) and then the measurements were carried out while heating the samples in the same field.

## RESULTS AND DISCUSSION

### X-ray Diffraction (XRD) Studies

Figure 1(a), 1(b) and 1(c) show the XRD patterns of non-implanted, Mn-implanted and co-implanted with 5 MeV silicon ion samples in the 2θ range 30°- 40° respectively. The non-implanted sample (Fig. 1(a)) shows the two peaks; one peak at 34.75° and other at 35.05° corresponds to GaN (002) and sapphire substrate (006) reflections respectively. After implantation with manganese ion, the XRD peak of as-implanted sample (Fig. 1(b)) from (002) reflection show the reduction in intensity and shifted toward the lower 2θ value with respect to that of the non-implanted sample. These indicate the disorder and defects in the implanted layers. However, co-implantation with silicon ion (Fig. 1(c)) XRD

pattern regained the crystalline structure. The lattice constant of tetragonal gallium nitride structure calculated using following equation;

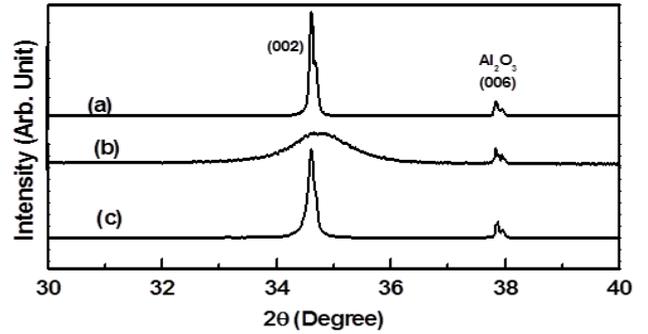


Figure 1 XRD spectra of GaN samples; (a) non-implanted, (b) Mn-implanted and (c) after co-Implanted with 5 MeV silicon ions.

$$\frac{1}{d^2} = \frac{4}{3} \left( \frac{h^2 + hk + k^2}{a^2} \right) + \frac{l^2}{c^2} \quad (1)$$

where, d is inter planar distance, h, k and l are the planes, a and c are the lattice constants. The c plane lattice constant for Mn-implanted and co-implanted samples was found to be 0.5158 nm and 0.5181 nm respectively. However, the corresponding estimate of lattice constant for non-implanted GaN was 0.5181 nm. The full widths at half maxima (FWHM) for the samples Mn-implanted and co-implanted were found to 1.2520 and 0.1493 degrees respectively. Whereas, estimated FWHM for non-implanted GaN sample was found to be 0.1226 degree. The increase in the lattice constant and FWHM indicated that the Mn atoms were incorporated in the wurtzite GaN structure as substitutional on the Ga sub-lattice [11].

### Raman Scattering Studies

Raman spectra of non-implanted, Mn-implanted and co-implanted with Si<sup>++</sup> ion samples shown in Figure 2(a), 2(b) and 2(c) respectively. Raman spectra of non-implanted GaN sample showed a single intense peak at ~567 cm<sup>-1</sup> attributed to Raman active phonon mode (E<sub>2</sub><sup>H</sup>) of gallium nitride [Fig.2 (a)]. However, the peaks of LO modes and low-frequency E<sub>2</sub> (E<sub>2</sub><sup>L</sup>) mode were found to be inherently very weak, the weak peaks may be due to experimental limitations. After implantation with Mn ions, the peaks appeared at ~140 cm<sup>-1</sup>, 295 cm<sup>-1</sup>, 300 cm<sup>-1</sup>, 420 cm<sup>-1</sup>, 529 cm<sup>-1</sup>, 549 cm<sup>-1</sup>, 670 cm<sup>-1</sup>, 674 cm<sup>-1</sup> and 720 cm<sup>-1</sup> were assigned to as disorder activated phonon modes<sup>14</sup>. The 420 cm<sup>-1</sup> peak attributed due to phonon mode, while the peaks at 140 cm<sup>-1</sup>, 529 cm<sup>-1</sup>, 549 cm<sup>-1</sup> and 720 cm<sup>-1</sup> respectively assigned to the low-frequency nonpolar phonon (E<sub>2</sub><sup>L</sup>), E<sub>1</sub>(TO), A<sub>1</sub>(TO) and LO modes. Whereas, the peaks at 300 cm<sup>-1</sup> and 670 cm<sup>-1</sup> are believed to have originated respectively from the vibrational modes of gallium and nitrogen vacancy related defects [Fig.2(b)]. The intensity of Raman active phonon mode (E<sub>2</sub><sup>H</sup>) of gallium nitride for the sample co-implanted with silicon ions were found to decrease than that of Mn-implanted sample. However, the other modes found to be increased [Fig.2(c)].

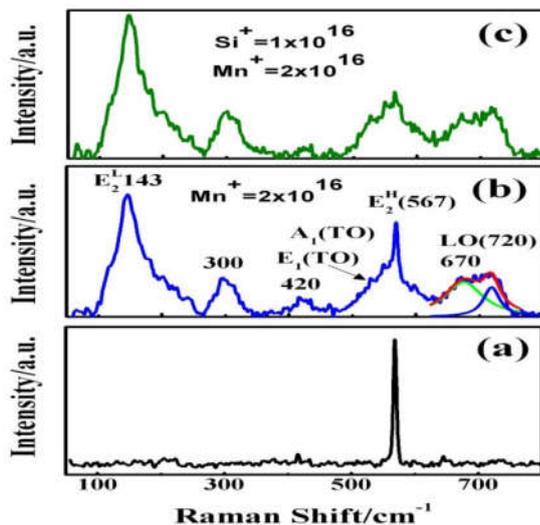


Figure 2 Raman spectra of GaN samples; (a) non-implanted, (b) Mn-implanted and (c) after co-implantation with 5 MeV silicon ions.

### Magnetic Studies

Figure 3(a) and 3(b) show the magnetization (M-H) curves of Mn-implanted and co-implanted with 5 MeV silicon ions samples respectively. It is seen from the Figure that the co-implanted gallium nitride sample showed the anomalous increase in the ferromagnetic properties. The magnetic properties such as coercive field, remanent magnetization and saturation magnetization of Mn-implanted and co-implanted GaN samples estimated from Figure 3 are given in Table-I. It is evident from the Table-I that the coercive field, remnant magnetization and saturation magnetization were increased after co-implantation with 5 MeV silicon ions. These results indicate that almost all Mn<sup>+</sup> ions contribute to ferromagnetic behavior in the sample after silicon ion implantation.

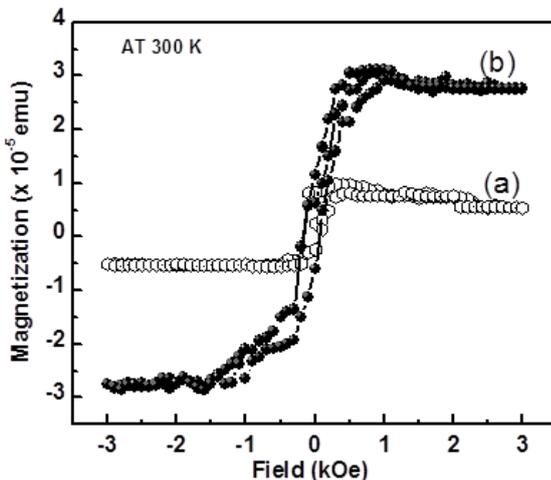


Figure 3 Magnetization (M-H) curves of GaN sample implanted with 325 keV Mn ions for the fluence of  $2.0 \times 10^{16}$  ions  $\text{cm}^{-2}$  (a) Mn-implanted and (b) after co-implantation with 5 MeV silicon ions.

**Table I** Values of coercive field, remanent magnetization and saturation magnetization of Mn- implanted and after co-implanted with silicon ion into GaN samples.

| Sample   | Mn-implanted | co -implanted |
|--|--------------|---------------|
| Coercive field (H <sub>c</sub> ) (Oe)                              | 163          | 180           |
| Remanent magnetization (M <sub>r</sub> ) ( $\times 10^{-6}$ emu)   | 0.61         | 11.66         |
| Saturation magnetization (M <sub>s</sub> ) ( $\times 10^{-6}$ emu) | 3.38         | 29.6          |
| Curie temperature (T <sub>C</sub> ) K                              | 310          | 326           |

### CONCLUSIONS

We have investigated the structural, optical and magnetic properties of 325 keV Mn<sup>2+</sup> ion implanted gallium nitride samples before and after implantation with silicon ion using X-ray diffraction, photoluminescence and superconducting quantum interference device techniques. XRD study revealed the re-crystallization of the implanted samples after implantation with silicon ion. Photoluminescence study showed bands at 2.28 eV and 2.45 eV due to YL and donor-manganese (D, Mn) transitions respectively. SQUID studies showed the significant increase in coercive field, remnant magnetization, saturation magnetization and Curie temperature (~ 326 K) after implantation with silicon ion.

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