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CEMS study of defect annealing in Fe implanted AlN

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Abstract An AlN thin film grown on sapphire substrate was implanted with 45 keV ^{57}Fe and ^{56}Fe ions at several energies to achieve a homogeneous concentration profile of approximately 2.6 at.%. in the AlN film. Conversion electron Mössbauer Spectroscopy data were collected after annealing the sample up to 900 °C. The spectra were fitted with three components, a single line attributed to small Fe clusters, and two quadrupole split doublets attributed to Fe substituting Al in the wurtzite AlN lattice and to Fe located in implantation induced lattice damage. The damage component shows significant decrease on annealing up to 900 °C, accompanied by corresponding increases in the singlet component and the substitutional Fe.

Keywords AlN · ^{56}Fe/^{57}Fe implantation · Defect annealing

1 Introduction

Group III-nitrides that have properties such as band gaps covering the full visible spectrum and high melting temperatures are attractive materials for the fabrication of a range of opto-electronic devices such as blue–green LEDs and lasers, as well as electronic devices for

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high power and high temperature applications. In order to fabricate such devices and integrate them into circuits, selective-area doping is required. Ion implantation is an attractive means of achieving this as it offers accurate control of dopant ions, their concentration and their implantation profile. However, ion implantation creates lattice damage which must be removed by annealing before the implanted dopants are rendered electrically active. In the low fluence implantation regime, annealing has been observed to result in significantly reduced lattice damage [1, 2], and, in some cases, even to complete lattice recovery [3]. However, the dopant levels achieved are insufficient to produce effective electrical or optical parameters for most device applications. Emission channeling (EC) measurements on Li and Na ions implanted in AlN and EC and γ-γ perturbed angular correlation (PAC) studies on AlN (and GaN) implanted with $^{111}In^{+}$ and $^{89}Sr^{+}$ have been conducted by Ronning et al. [4]. The lighter ions were observed to occupy interstitial sites on implantation, but relocated to substitutional sites after annealing. The In and Sr ions, after room temperature implantation, were found to occupy substitutional sites in heavily perturbed surroundings of point defects. Annealing at 1473 °C resulted in partial removal of the point defects, but produced no lattice site change. High fluence implantations, on the other hand, lead to heavily damaged regions in the host, that contain secondary defects such as stacking faults, dislocation loops and amorphous regions [5–7]. These defects have been found difficult to remove by thermal annealing, and to contribute to compensation of the implanted dopants, thus rendering them electrically and optically inactive [8–10]. A technique to obviate the build-up of implantation induced defects was followed by Osov et al. [11] with ion implantation in several implantation steps with annealing between each step. This method resulted in better lattice recovery compared to single energy implantation and annealing at high temperatures.

In the present contribution we focus on defect recovery in wurtzite AlN which has a direct band gap of 6.28 eV and a melting temperature of 3000 °C, and hence has been the subject of several studies on the incorporation of n- and p-type dopants. For example, Tanisayu et al. [12] achieved n- and p-type conductivity by incorporation of Si and Mg in AlN during growth, but the resistivity of the material increased significantly when the Mg doping level increased beyond 2 at.%.

We have sought to achieve increased dopant incorporation in AlN via ion implantation, but controlling the local concentration by utilisation of implantation at several energies and fluences so that a box shaped implantation profile is achieved. We have studied the nature of implantation sites taken up by implanted ^{57}Fe ions and the annealing behaviour of implantation induced lattice damage in an AlN sample, utilizing conversion electron Mössbauer Spectroscopy (CEMS). Unlike Emission Channeling (EC) measurements, Mössbauer spectroscopy does not allow the direct determination of the lattice sites of implanted ions. Instead, information on the nature of the sites of the probe nuclei are inferred from the hyperfine parameters (isomer shift (δ) and electric quadrupole splitting (ΔE_Q)) that characterise the fit components in the Mössbauer spectra due to Fe nuclei in different environments in the host lattice.

2 Experimental

A commercially acquired AlN sample, in the form of a 1000 nm thick epilayer grown on a sapphire substrate (MTI Corporation (USA)) was implanted with ^{56}Fe and ^{57}Fe ions. The implantation energies and fluences (listed in Table 1) were chosen, following TRIM

Table 1 Energy

Ion	Energy	Fluence ($\times 10^{16}/cm^2$)
^{56}Fe	300 keV	3.10
	170 keV	1.15
	90 keV	0.81
^{57}Fe	45 keV	0.45

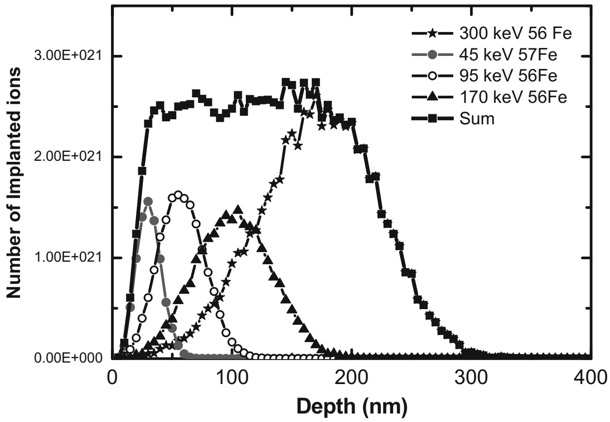

Fig. 1 Implantation profiles of ^{57}Fe and ^{56}Fe ions implanted in AlN with the energies indicated

[13] simulations, to achieve a 'box' shaped implantation profile (see Fig. 1) which allowed 5.5×10^{16} Fe ions/cm^2 to be incorporated in the sample while the concentration was kept at 2.6 at.%. The aim was to keep lattice damage below the level at which secondary defects are formed [5–7].

CEMS data were collected on the as-implanted sample and after annealing the sample for 30 minutes in flowing nitrogen at temperatures up to 900 °C utilising a parallel plate detector filled with acetone gas at 30 mbar pressure

3 Results and discussion

The CEM spectra, presented in Fig. 2, resemble those observed by Borowski et al. [14] for AlN implanted with Fe to a concentration of 12 at.%. Consequently, a similar fit model was adopted, with the spectra being analysed with a singlet and two quadrupole split doublet components. The spectral components were assumed to have Voigt line shapes with a Lorentzian width $\Gamma = 0.30$ mm/s and Gaussian broadening σ. The fit parameters (isomer shift δ, quadrupole splitting ΔE_Q) and areal fractions (f) of the as-implanted sample and after annealing at 900 °C are listed in Table 2, and the area fractions are plotted in Fig. 3 as a function of annealing temperature.

The singlet component has an isomer shift close to the value observed by Borowski et al. [14] and has been attributed to small Fe clusters in the AlN matrix. We now consider doublet D1 which has an isomer shift of 0.53 mm/s and a quadrupole splitting of 0.64 mm/s after

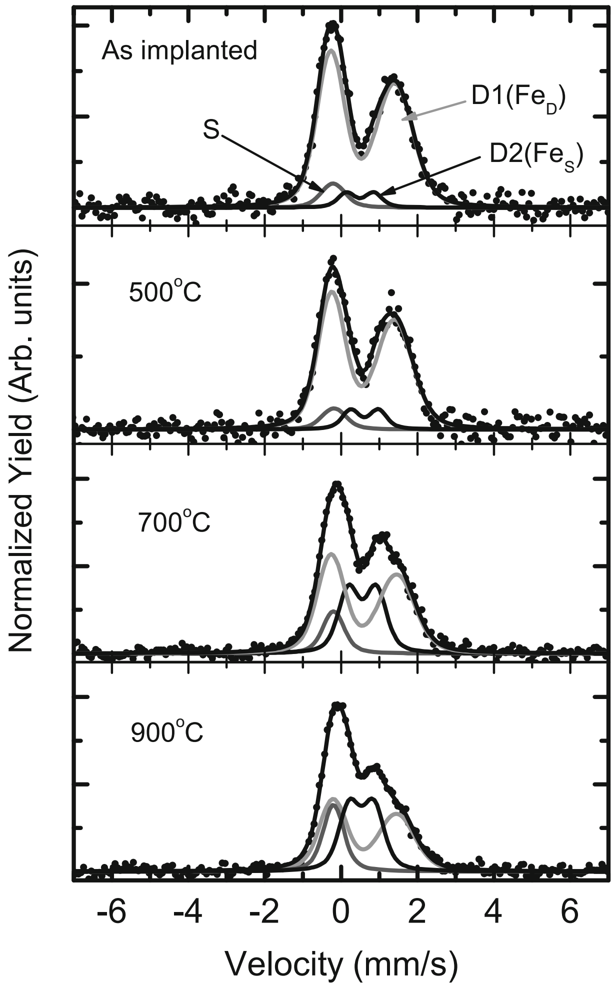

Fig. 2 CEM spectra of AlN implanted with the profilerm ^{57}Fe and ^{56}Fe shown in Fig. 1, as a function of annealing temperature

the annealing at 900 °C. A similar component has been observed by Borowski et al. [14], and assigned to Fe surrounded by 4 N atoms. In the wurtzite AlN lattice, this corresponds to Fe substituting an Al atom (Fe_S).

Doublet D2 has both a large broadening as well as a large mean quadrupole splitting. It dominates the spectra at the lower annealing temperatures, and is attributed to Fe ions located in implantation induced lattice damage, Fe_D.

Figure 3 presents the dependence of area fractions of the spectral components on the annealing temperature. It is evident that on annealing above 700 °C the lattice damage decreases appreciably, with Fe_D falling from >80 % in the as implanted sample to below 50 % after annealing at 900 °C. The single line component and doublet D1 (Fe_S) show corresponding increases, from 6 % in the as-implanted sample to 18 and 34 %, respectively, after the 900 °C annealing. There is no evidence of site change of the implanted probes.

Table 2 Hyperfine parameters extracted from fits to the CEM spectra shown in Fig. 2

Annealing temperature	Spectral component	δ (mm/s)	ΔE_Q (mm/s)	σ (mm/s)	f (%)
As implanted	S	−0.21(3)	–	0.50	6(2)
	D1 (Fe_S)	0.49(3)	0.71(4)	0.30	6(2)
	D2 (Fe_D)	0.57(3)	1.67(7)	0.70	88(6)
900 °C	S	−0.20(2)	–	0.40	18(2)
	D1 (Fe_S)	0.53(2)	0.64(2)	0.40	34(3)
	D2 (Fe_D)	0.62(3)	1.66(7)	0.70	48(4)

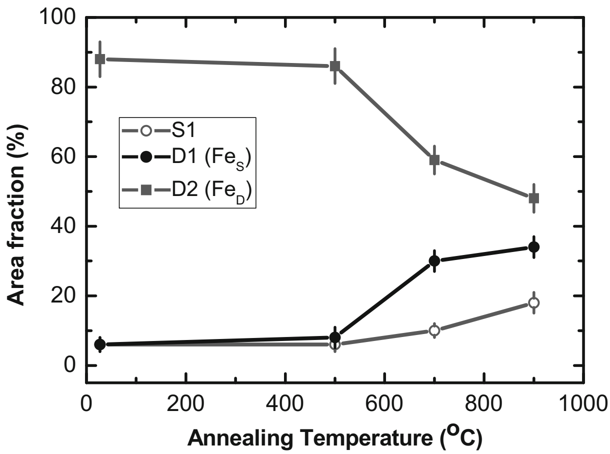

Fig. 3 Areal fractions of the spectral components observed as a function of annealing temperature

4 Conclusions

We have implanted ^{57}Fe and ^{56}Fe ions in AlN at several energies to achieve a homogeneous concentration profile of 2.6 at.% in the AlN epilayer. CEM spectra collected after annealing the sample up to 900 °C were fitted with a single line (attributed to small Fe clusters), and two quadrupole split doublets attributed to Fe substituting Al in the wurtzite AlN lattice (Fe_S) and to Fe located in implantation induced lattice damage (Fe_D).

The damage component shows strong decrease on annealing, accompanied by corresponding increases in the singlet component (S) and the substitutional Fe (Fe_S). The increase in S shows that the Fe ions located in defects are partially accumulated into clusters on annealing. Our results also show that the point defects in the perturbed surroundings of the substitutional Fe ions are also partly removed on annealing, in agreement with the EC and PAC studies of Ronning et al. on ^{111}In and ^{89}Sr implanted AlN [4]. The present study illustrates that implantation at several energies and fluences to achieve a homogeneous profile in which the dopant concentration is kept within reasonable limits can lead to the incorporation of appreciable fractions of dopants on regular lattice sites.

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