

Annealing behaviour of GaAs implanted with 70 MeV ^{120}Sn ions

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Single crystal GaAs substrates implanted with 70 MeV ^{120}Sn have been investigated by X-ray diffraction (XRD) and electrical resistance measurements after annealing in the temperature range 373-823 K. XRD measurements for the samples annealed up to 723 K show two peaks, one due to the substrate and another due to the implant damaged layer. The strain parameter from the measured separation between the substrate and layer peaks of several symmetric and asymmetric reflections after each annealing step has been calculated. Strain recovery occurs in two predominant annealing stages, one at about 500 K and the other at about 700 K. Temperature dependence (100-300 K) of resistance of these samples indicates that electrical conduction in the samples annealed up to 723 K, is dominated by variable range hopping. Localized states density at the Fermi level $N(E_F)$, estimated from temperature dependence of sample resistance after each annealing step, shows two annealing stages similar to those observed from XRD measurements. Isothermal annealing carried out at two different annealing temperatures (523 and 573 K) indicates the activation energy $E_a = 0.16$ eV for the first annealing stage.

Keywords: MeV implantation, Radiation defects, GaAs

1 Introduction

Ion implantation of GaAs with ions of energy up to few hundred keV is a well established technology¹. The process is routinely used for applications such as metal-semiconductor field effect transistors and monolithic microwave integrated circuits^{2,3}. With the availability of high energy accelerators providing ions of energy extending to 100 MeV and more, the modifications of the materials subjected to ions of this high energy range has been explored. The higher energy ions provide deeper implants. The potential applications of such implants for semiconductor devices requiring deep buried conducting or isolating regions have also been studied^{4,5}. The nature of damage produced by such high energy ions is different from that caused by the same ions at lower keV energies. Similarly, the annealing properties may also be different. In this paper, the effects of implanting GaAs with ^{120}Sn ions at energy of 70 MeV on properties (i) structure probed by X-ray diffraction (XRD) and (ii) electrical resistance as well as the results of annealing the samples in the temperature range 373-823 K have been studied.

2 Experimental Details

The samples used in this experiment were mirror polished <100> semi-insulating and lightly doped n -GaAs wafers with background doping concentration of $2 \times 10^{16}/\text{cm}^3$ and thickness of 400 microns. The samples were carefully cleaned in organic solvents, trichloroethylene, acetone and methanol rinsed in de-ionized water and then immediately loaded into the target chamber of the NEC 16 MV Tandem Van de Graff type electrostatic accelerator⁶ (Pelletron) at Nuclear Science Centre, New Delhi. The implantations have been carried out at room temperature on the polished side of the sample with 70 MeV ^{120}Sn ions to a fluence of 1×10^{18} ions/ m^2 in a non-channeling direction. Implantation details have been described elsewhere⁷. The implanted samples have been isochronally annealed for 10 min at different temperatures up to 823 K in high purity hydrogen ambient. To prevent the out diffusion of As from GaAs surface during the high temperature heat treatment, the implanted sample were capped with clean piece of virgin GaAs. Unimplanted samples were used as reference during the annealing

experiments to monitor any change in the sample characteristics caused by surface degradation. The X-ray diffraction patterns were recorded on a Phillips X'pert system having a channel cut, four crystal Ge (022) (Batlels type) monochromator for CuK_α X-ray beam of divergence 12 arc seconds in the scattering plane and a 2° open detector. Profiles of several reflections, symmetric and asymmetric, were recorded for the as-implanted sample and samples annealed at different temperatures in $\omega/2\theta$ scans after optimizing the tilt and azimuth angle. The electrical characteristics of the as-implanted *n*-GaAs sample and samples annealed at different temperatures were also investigated. The as-implanted samples were divided into several pieces. Each piece was used for annealing treatment at a selected temperature. Ohmic contacts were fabricated on the back side of these samples by evaporating a uniform coating of Au-Ge-Ni alloy. The top ohmic contacts on the implanted side were made by evaporating Au-Ge-Ni square dots of area 0.0045 cm² through a metal mask. The contacts were then alloyed at 723 K for one minute in pure hydrogen ambient. Ohmic contacts were made before the implantation for those samples which were annealed to temperatures less than 723 K. Current voltage (*I-V*) measurements were carried out over a temperature range 100-300 K by using a programmable voltage source, a Keithley digital electrometer and a variable temperature cryostat. The *I-V* measurements were also carried out for two samples isothermally annealed at two different temperatures 523 and 573 K for various time durations.

3 Results and Discussion

XRD profiles of all the reflections of the implanted samples up to the highest scattering angle of $2\theta_B$ of about 152°, show two peaks similar to those for the (004) reflections seen in the 300K annealed plot in Fig. 1. One peak is due to the substrate (marked S) and the other is attributed to the implant damaged layer (marked L) which extends from the sample surface up to a depth equal to the range of the implanted ions (~10.8 μm). The XRD profile of the unimplanted GaAs substrate (not shown here) has only a single sharp peak located in the vicinity of S. The relative intensities of the layer and the substrate peaks as well as their separation depend upon the glancing angle of incidence and the scattering angle. However, these separations at the four different

azimuths, separated by 90°, are about the same indicating a negligible tilt of the layer with respect to the substrate. The appearance of the damage related peak at an angle smaller than the substrate Bragg peak shows expansion of the lattice with damage, which is a well known effect⁸. The damaged layer in the tetragonal distortion model is characterized by the perpendicular (ϵ_1) and parallel (ϵ_2) strain parameters, which are defined with respect to the substrate in the XRD study as⁹:

$$\epsilon_1 = (d_{\perp} - d_{\text{sub}}) / d_{\text{sub}} \quad \dots(1)$$

$$\epsilon_2 = (d_{\parallel} - d_{\text{sub}}) / d_{\text{sub}} \quad \dots(2)$$

where d_{sub} is the substrate spacing, d_{\perp} and d_{\parallel} are the out-of-plane and in-plane lattice parameters for the strained damaged layer, respectively. To obtain ϵ_1 and ϵ_2 of the damaged layer, the measurements of several symmetric and asymmetric reflections have been carried out as presented in Table 1. The average separation $\Delta\omega_{L,S}$ between the substrate peak S and the damaged layer peak L for all the measured reflections is presented in Table 1. For the present work, we have

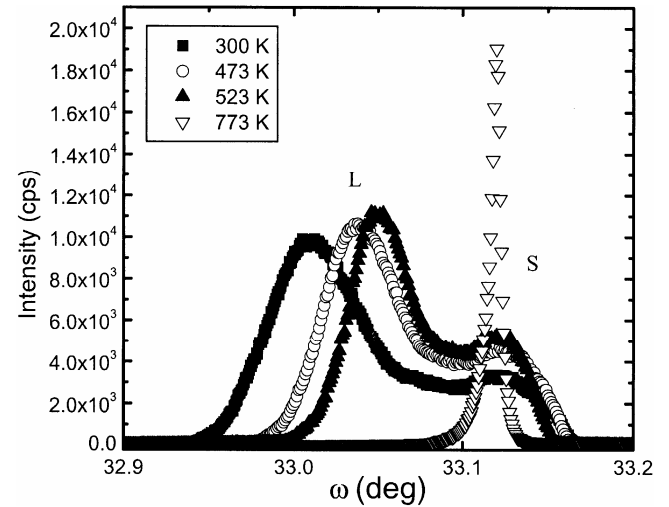


Fig. 1—XRD (004) reflection profiles for as-implanted samples and samples annealed at four different temperatures

Table 1—Layer to substrate peak separations for different reflections*

(hkl)	004	115L	115H	026L	026H	006	117L
$\Delta\omega_{L,S}$ (deg.)	0.121	0.221	0.123	0.317	0.209	0.255	0.886

*L and H are low and high glancing incidence.

Least Square estimates: $\epsilon_1 = (3.302 \pm 0.065) \times 10^{-3}$; $\epsilon_2 = (-0.358 \pm 0.545) \times 10^{-3}$

Parameter correlation (ϵ_1, ϵ_2) = -0.07

adopted a simplified analysis of these measurements assuming that the separation $\Delta\omega_{L,S}$ can be expressed in terms of linear combination of parallel and perpendicular strain parameters described by¹⁰:

$$\Delta\omega_{L,S} = k_1\varepsilon_1 + k_2\varepsilon_2 \quad \dots(3)$$

$$k_1 = \cos^2 \varphi \tan \theta_B + 1/2 \sin 2\varphi \quad \dots(4)$$

$$k_2 = \sin^2 \varphi \tan \theta_B - 1/2 \sin 2\varphi \quad \dots(5)$$

where θ_B is the Bragg angle for the particular reflection and φ is the angle between the reflecting plane and the sample surface. The asymmetric planes have two reflection modes (i) low incidence in which the angle of incidence of the X-ray beam is $(\theta_B - \varphi)$ and (ii) high incidence for which the angle of incidence $(\theta_B + \varphi)$. Eqs (4 and 5) are for the low incidence mode. For the high incidence mode, the angle φ reverses sign. It is clear from Eqs (4 and 5) that only the asymmetric reflections with $\varphi \neq 0$ can yield information about the iplane strain ε_2 . Using the $\Delta\omega_{L,S}$ values from Table 1 and the values of k_1 and k_2 for various reflections calculated from Eqs (4) and (5), the values of ε_1 and ε_2 and their standard deviations were obtained by least square fitting procedure as applied to Eq. (3). The results shown in Table 1 clearly indicate a negligible in-plane strain ε_2 and significant out-of-plane strain ε_1 together with low correlation between the two strain parameters. Negligible in-plane strain has been reported earlier in several implantation studies^{11,12}. In order to investigate the annealing of the structural defects by X-ray measurements, XRD (004) reflection profiles were recorded for the as-implanted sample and after isochronal annealing of the same sample at different temperatures varying from 373 to 773 K in steps of 50 K. However, for the sake of clarity, we have shown only four XRD profiles in Fig. 1. The substrate feature S gradually becomes narrower and its strength increases after each successive annealing. The damaged layer feature L also becomes narrower with annealing. However, change in its strength is small up to annealing temperature of 525 K. At annealing temperatures greater than 525 K, the strength of the layer feature starts decreasing and then practically vanishes at 775 K. The other significant change is the

shifting of the layer peak, closer to the substrate peak, with each annealing step. The separation between the S and L peaks is a measure of the maximum strain in the layer. The reduction of strain with each annealing step thus represents recovery of the implant damage. The XRD measurements of several symmetric and asymmetric reflections after each annealing step have been carried out. From the measured peak separations $\Delta\omega_{L,S}$, the values of the average perpendicular strain ε_1 were obtained and plotted in Fig. 2. The damage recovery seems to occur in two predominant stages, one at about 500 K the other at about 700 K.

The electrical characteristics of the *n*-GaAs implanted substrates have been investigated by current-voltage (*I-V*) measurements for as-implanted sample and samples annealed at different temperatures. We find that the *I-V* curves for the as-implanted samples and samples annealed up to 723 K are linear and the resistance values can be directly obtained from them. The sample annealed to 823 K shows a weak non-linearity in the *I-V* characteristics and as such we have estimated the resistance of this sample from the high current region in the *I-V* curve where the series resistance is dominant. The room temperature values of the sample resistance after different annealing temperatures, are presented in Table 2. The resistance values increase

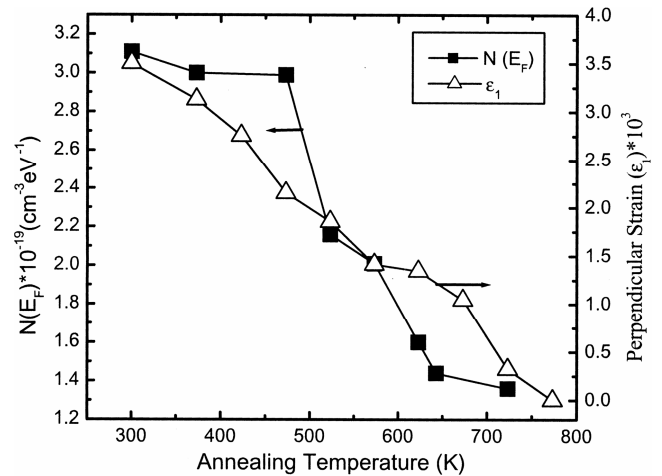


Fig. 2—Perpendicular strain ε_1 obtained from XRD measurements and localized density of states at Fermi level $N(E_F)$ obtained from hopping conduction measurements as a function of annealing temperature. The lines are drawn by simply joining the experimental points to guide the eye

Table 2—Room temperature resistance after different annealing temperature

Annealing temperature (K)	300	373	473	523	573	623	643	723	823
Resistance (Ω)	245	265	330	452	500	890	6.5×10^3	3.5×10^4	1.5×10^7

with each annealing step indicating recovery of the damage. In order to estimate the density of defects, the temperature dependence of resistance of these samples has been carried out. We observe that the resistance of the as-implanted sample and samples annealed up to 723 K satisfies the relation $R \propto T^{-1/4}$ in the temperature range 100-300 K as shown in Fig. 3. These plots suggest that the conduction mechanism in this temperature range is dominated by variable range hopping between defect energy levels in the forbidden gap as described by¹³:

$$\rho(T) = \rho_0 \exp(T_0 / T)^{1/4} \quad \dots(6)$$

The values of T_0 are obtained from the slopes of $\log R$ versus $T^{-1/4}$ plots. The localized states density $N(E_F)$ at the Fermi level, for these samples can be estimated by the expression¹⁴ $N(E_F) = (c^4 \alpha^3 / T_0 k)$, where $c^4 \cong 20$, $\alpha(\text{cm}^{-1}) \cong (2m^* / h^2)^{1/2} (E_g / 2)^{1/2}$ is the attenuation distance of the wave function for the localized states and m^* is the effective mass of the electron. The estimated values of $N(E_F)$ after annealing at different temperatures are shown in Fig. 2. We see a clear annealing stage at about 500 K, which is very analogous to the one with that observed from XRD measurements. XRD measurements indicate another annealing stage at about 700 K. Considerable annealing of the defects is also observed around the temperature range 600-700 K from the electrical measurements. These annealing stages were

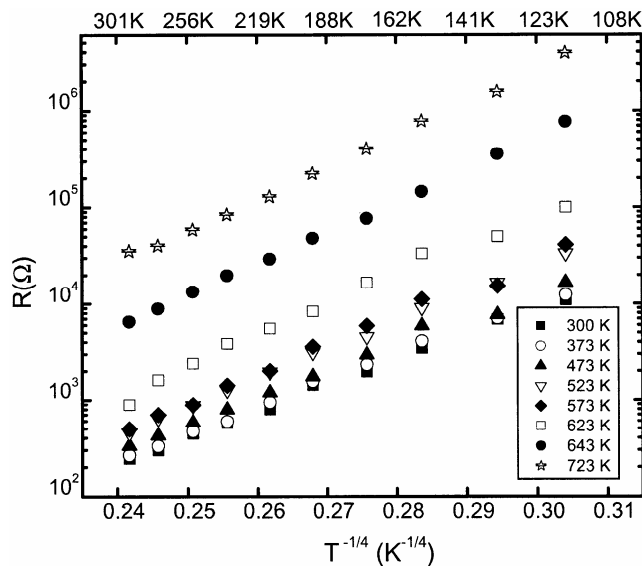


Fig. 3—Logarithm of resistance versus $T^{-1/4}$ for *n*-GaAs substrates implanted with 70 MeV ¹²⁰Sn ions to a dose of 10^{14} ions/cm² sample and annealed at different temperatures up to 725 K

also obtained from earlier reported experiments on GaAs irradiated by fast neutrons¹⁵. Our previous optical measurements¹⁶ have also shown these two annealing stages. Additionally, another dominant annealing stage seems to occur at around 825 K, as seen from the large increase in the sample resistance value presented in Table 2. At this annealing temperature, there is a change over of the conduction mechanism from variable range hopping transport between the defects states to the hopping conduction between the neighbouring defect sites⁷.

In addition to the isochronal annealing experiments, we have carried out isothermal annealing at two different annealing temperatures (523 K and 573 K) and measured the electrical characteristics from these measurements, $N(E_F)$ was obtained after each annealing time as shown in Fig. 4. This data has been used as below to obtain a characteristic energy of annealing of the implant damage in the region of the first stage of annealing.

If N is the total number of disordered states per unit volume, and assuming that the annealing process to be a first order reaction, then the change of N is described by¹⁷:

$$\frac{dN}{dt} = -K_0 \exp(-E_a / kT) N \quad \dots(7)$$

where the frequency factor K_0 is constant, T the annealing temperature and E_a is the activation energy of annealing. If we consider that the localized density of states at Fermi level $N(E_F)$ is proportional to the defect density N . Then

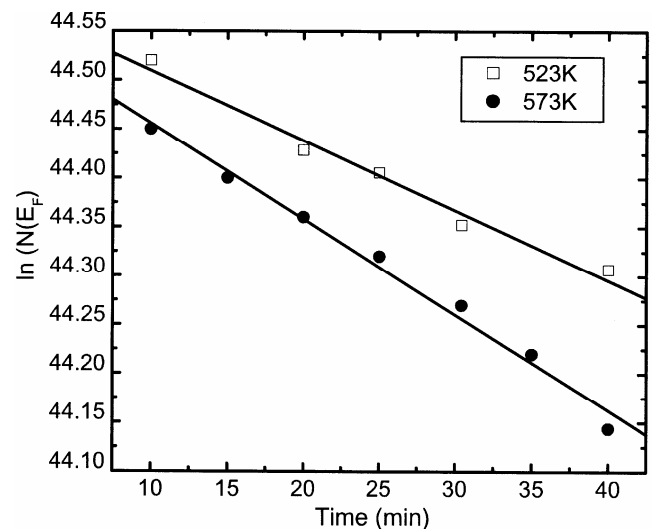


Fig. 4— $\ln(N(E_F))$ versus time of annealing for two different annealing temperatures

$$\frac{dN(E_F)}{dt} = -K_0 \exp(-E_a / kT) N(E_F) \quad \dots(8)$$

and hence

$$\ln N(E_F) = \ln N_0(E_F) - K_0 \exp(-E_a / kT) t \quad \dots(9)$$

where $N_0(E_F)$ is the localized states density at $t = 0$.

The activation energy E_a for the first annealing stage is obtained from the ratio of the slopes of the two plots in Fig. 4 and it is found to be 0.16 eV. This activation energy is close to the value found from our optical measurements¹⁶. This value is quite small compared with the activation energy of annealing of point defects for GaAs, which are in the range 1.0-1.5 eV obtained from experiments on GaAs irradiated by electrons and fast neutrons¹⁵ at an annealing stage which also occurs at about 500 K. The first annealing stage seems to occur at roughly similar temperatures in these different studies, it is definite that the actual damage and its annealing are very different. The damage due to electron irradiation and gamma irradiation will largely consist of isolated point defects, whereas that due to MeV energy ion implantation will consist of regions of large defect clusters due to nuclear energy loss (nuclear damage) as well as relatively small defect regions due to electronic energy loss (electronic damage). It is not possible at this stage to separate the annealing of these regions precisely. The estimated activation energy is possibly related to the annealing of high concentration of defect clusters.

4 Conclusion

We have implanted ¹²⁰Sn ions in single crystal GaAs substrates at a fluence of 1×10^{14} ions/cm² and energy 70 MeV. The resulting damage in the implanted samples and after annealing them over a temperature range 373-823 K, was studied by XRD and electrical resistance measurements. XRD measurements for the implanted sample show two peaks one is due to the substrate and another is attributed to the implant damaged layer. We measured the separation between the substrate and layer peaks of several symmetric and asymmetric reflections after each annealing step and from the measured peak separations $\Delta\omega_{L,S}$, the values of average perpendicular strain ϵ_1 are obtained. Strain recovery seems to occur in two predominant stages, one at about 500 K and the

other at about 700 K. The electrical characteristics of the implanted substrates have been investigated by current-voltage (*I-V*) measurements for the as-implanted sample and samples annealed at different temperatures. The resistance of the implanted samples is found to increase with annealing temperature. The conduction below room temperature for the samples annealed up to 723 K is dominated by variable range hopping between defect energy levels in the forbidden gap. We have estimated the localized states density at Fermi level $N(E_F)$, after each annealing step. The electrical measurements also show two annealing stages analogous to those observed from XRD measurements. Another dominant annealing stage occurs at around 825 K, as seen from the large increase in the resistance values. The isothermal annealing carried out at two different annealing temperatures (523 K and 573 K) indicates the activation energy $E_a = 0.16$ eV for the first annealing stage which appeared at around 500 K.

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