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EFFECTS OF 3.0 MeV ELECTRON IRRADIATION ON 4H-SiC WAFER PROPERTIES

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ABSTRACT

Effects of 3 MeV electron (10 mA) irradiation at room temperature on the phase, microstructure, electrical and life time properties of 4H-SiC wafer were investigated by scanning electron microscopy (SEM), X-ray diffraction (XRD), four point probe current-voltage measurements and positron annihilation spectroscopy. It was found that irradiation damage in SiC wafer is significantly increased with the increase of radiation dose as observed in SEM. Irradiation also resulted in modification of crystallite size as identified by XRD. The resistance of a sample before irradiation was found to be 0.8 MΩ, whereas for a sample irradiated at 200 kGy, the resistance as measured by four point probe was 5.2 MΩ. It seems that the increase of resistance hence, reduction in conductivities could be due to defects induced by the radiation dose received then created leakage currents at both reverse and low-forward biases and creation of traps in the SiC. Meanwhile positron annihilation spectroscopy (PAS) was used to analyse the life time of irradiated samples which nonetheless shows that all irradiated sample have similar life time of 151 ps. It was observed that that no degradation process of materials experienced by SiC wafer irradiated at 500 kGy.

Keywords: crystallite, electron irradiation, positron annihilation spectroscopy, resistivity, silicon carbide.

INTRODUCTION

There have been numerous research done on SiC, due to its unique properties such as being a wide band gap semiconductor and high resistant to radiation damage. Many of these research focus on the electrical applications of SiC in diodes. Such a scope is of special interest to nuclear industry as radiation detectors due to its potential to be used in harsh conditions and environments as mentioned in References (Lu et al., 2003; Cinar et al., 2009; Cinar et al., 2010). For example, SiC MOSFETs can be applied at very high voltages and temperatures as compared with normal silicon based devices (Neudeck, 2006). In the presence works, more focus were given on effects of radiation to SiC, (Ferrero et al. 2002), its optical characteristics (Le Donne et al., 2005), differences between different forms of SiC such as produced from hot pressed, reaction bonded and chemical vapour deposition (Hodgson et al., 2011) and also on the differences between its polymorphs (Ching et al., 2006; Cinar et al., 2009; Cinar et al., 2010). These investigations were carried out to correlate with SiC electrical properties. Polytypes SiC that are mostly studied are 4H-SiC and 6H-SiC. Structural investigations on the differences between these two polytypes have been reported (Ching et al., 2006; Cinar et al., 2009; Cinar et al., 2010; Hodgson et al., 2011) with the main

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differences are the band gap and therefore the barrier heights in SiC diodes, series resistances, electronic structure, bonding and optical properties for example those discussed by Ching et al. (2006) and Cinar et al. (2010).

The work reported in this paper is a part of the investigation to analyse the characteristics of SiC before and after irradiation using SEM, XRD and TEM techniques and the study of defect formation due to irradiation by electron beam. The tests chosen for this investigation include SEM and XRD to determine phase properties, crystallites sizes and morphology changes and electrical characteristics (I-V) and four point probe of the sample during forward and reverse bias. Initial investigation using PAS was also carried out.

EXPERIMENT WORKS

The commercialised wafer samples from MTI.XTI (USA) of orientation (0001), one surface polished, crystal n-type and dimensions 7 x 5 x 0.3 mm (length x width x height) were used in this investigation. The as-received wafers were already polished on one side. To clean the samples of any dust, acetone and deionised water was used. XRD measurements were carried out with reflection from the polished surface (Cu-K_α, 1.514 Å, PanAnalytical Instrument) on samples that are unirradiated, irradiated at 100 kGy and 200kGy. Peak broadening and position were determined using X'pert software whereas *d*-spacing, crystalline sizes and lattice strains were calculated using HighScorePlus software's. For the SEM test, the samples (unirradiated, irradiated at 100 kGy, at 200 kGy and at 300kGy) were cleaned with acetone and deionised water prior analysed. The I-V characteristics of the sample (unirradiated and irradiated at 200 kGy samples) were determined and the resistivity was measured by four points probe (Keithley 2401). To prepare the samples for the I-V characterization, silver paste was applied as the contacts and annealed at 100°C in a furnace for approximately 30 minutes to form a better contact-sample interface. For irradiation measurements, samples were exposed to electron beam of 3 MeV and 10 mA with a total dose of 100 kGy, 200 kGy up to 500 kGy (depending on the test) at a rate of 50 kGy per pass. The electron irradiation process was done by using electron beam facility at the Malaysian Nuclear Agency (EPS 3000, Electron Processing System). The irradiated samples were then examined again by the same characterization methods done on the as-received samples. Positron annihilation spectroscopy (PAS) analysis was carried out at Helsinki University Finland facility. Samples for lifetime test were irradiated at 100, 200, 300 and 500 kGy using electron beam. All samples were measured using the same source and analysis done with the same corrections.

RESULTS AND DISCUSSION

The XRD patterns for irradiated (at 100KGy and 200KGy) and unirradiated samples are shown in Figure 1. The highest peak was observed at (111) at a very small peak was also observed at (222). The (111) peak is sharp for all samples and this indicates strong crystalline structure. The diffraction peaks in Figure 1 correspond to the database XRD patterns of the β-phase SiC. The XRD pattern and intensity for unirradiated and irradiated samples are similar, indicating that SiC phases and crystalline are not susceptible to modifications due to electron radiation exposure. Table 1 and 2 shows the peak broadening and position and the calculated lattice strains, crystallite sizes and *d*-spacing for first and second peaks of the XRD patterns respectively. The second peak (222) is considered as insignificant as its peak intensity is very low. As shown in Figure 1 and Table 1 the position of the first diffraction peak shifted to lower diffraction angle and slightly become sharper with increased radiation dose. The peaks were found to have shifted after exposure to electron

irradiation with no significant shape changes. This suggests that irradiation does not significantly affect the strain of material. Measurement by XRD indicated that the strain reduced only slightly from 0.258% for unirradiated sample to 0.255% and 0.208% for samples irradiated at 100 and 200 kGy respectively.

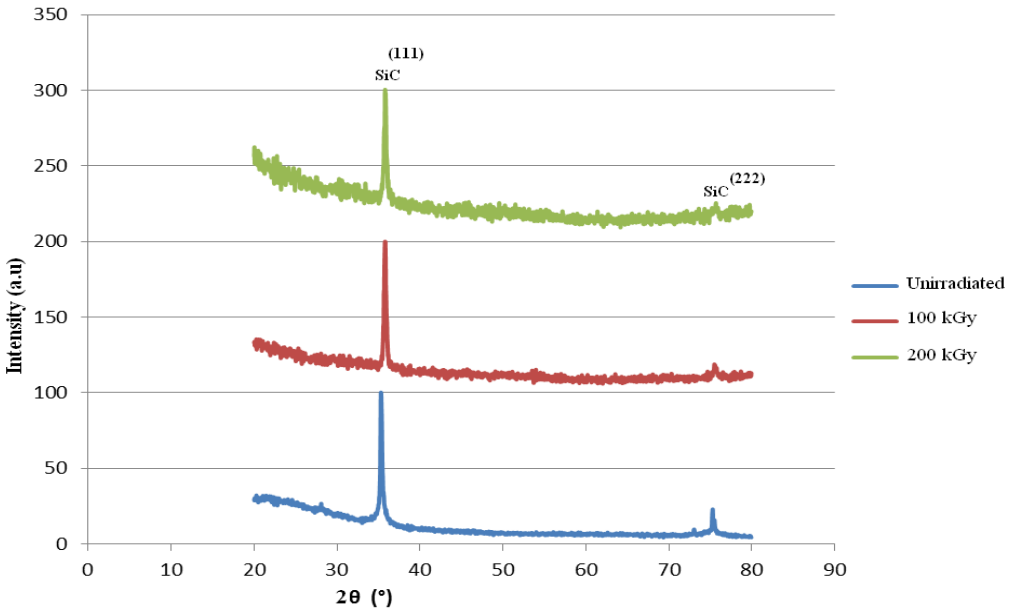

Fig. 1: A comparison of the three samples; An XRD graph showing peaks for the SiC before, 100 kGy and 200 kGy irradiation

The crystallite size for the first peak of the unirradiated sample was found to be 575Å. This is almost the same to the crystallite size of sample obtained from 100 kGy irradiation which is 576Å. However, for 200 kGy irradiation, the crystallite size increased quite significantly that is, 746Å. This is possibly caused by thermal heating of the sample by electron radiation thus sintering effect occurred.

Table 1: Shows the lattice strains and crystallite sizes for the first peak (111).

Peak 1					
Sample	FWHM [°2Theta]	Peak position [°2Theta]	Lattice strain [%]	Crystallite size [Å]	d-spacing [Å]
Unirradiated	0.195	35.320	0.258	575	2.541
100 kGy	0.195	35.802	0.255	576	2.508
200 kGy	0.162	35.853	0.208	746	2.505

Table 2: Shows the lattice strains and crystallite sizes for the second peak (222).

Peak 2					
Sample	FWHM [°2Theta]	Peak position [°2Theta]	Lattice strain [%]	Crystallite size [Å]	d-spacing [Å]
Unirradiated	0.119	75.277	0.061	1454	1.261
100 kGy	0.475	75.493	0.266	236	1.258
200 kGy	0.950	75.552	0.534	112	1.257

Fig. 2(a) - (c) shows SEM micrographs for unirradiated and irradiated (100, 200 and 300 kGy) samples. The images show the radiation damage due to ionizing effects (possibly surface vaporization). Radiation damage was observed on the surface of the 100 kGy irradiated sample, and further increase in damage is observed on the surface morphology of the 200 and 300 kGy irradiated samples. The microstructures observed determine such surface damages extended into bulk medium and occur at much smaller scale.

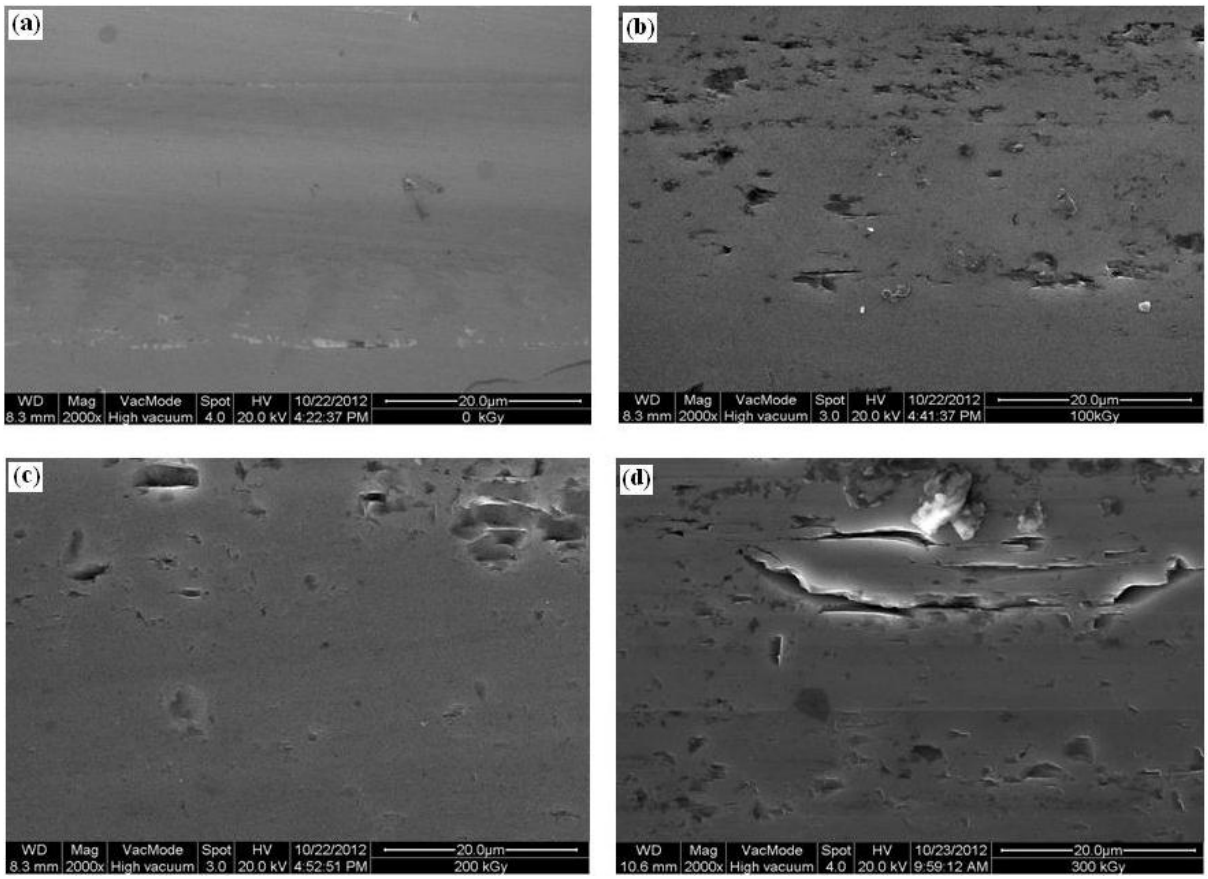

Fig. 2: SEM Micrographs of 4H-SiC wafers surface for sample **a)** unirradiated **b)** Irradiated 100 kGy **c)** Irradiated 200 kGy and **d)** Irradiated 300 kGy

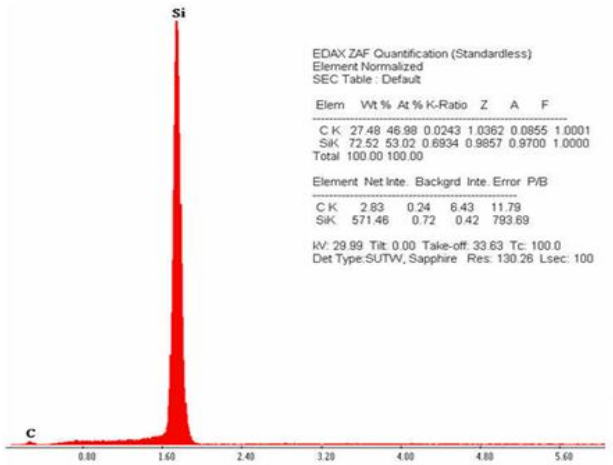

Fig. 3: An EDX graph confirming the elements present in SiC.

Table 3: Four point probe resistance readings for the unirradiated and 200 kGy samples

Sample	Resistance (MΩ)	Resistivity (Ωm)	Conductivity (Sm^{-1})
Unirradiated	0.8	1087.8	$9.2x10^{-4}$
200 kGy	5.2	7070.5	$1.4x10^{-4}$

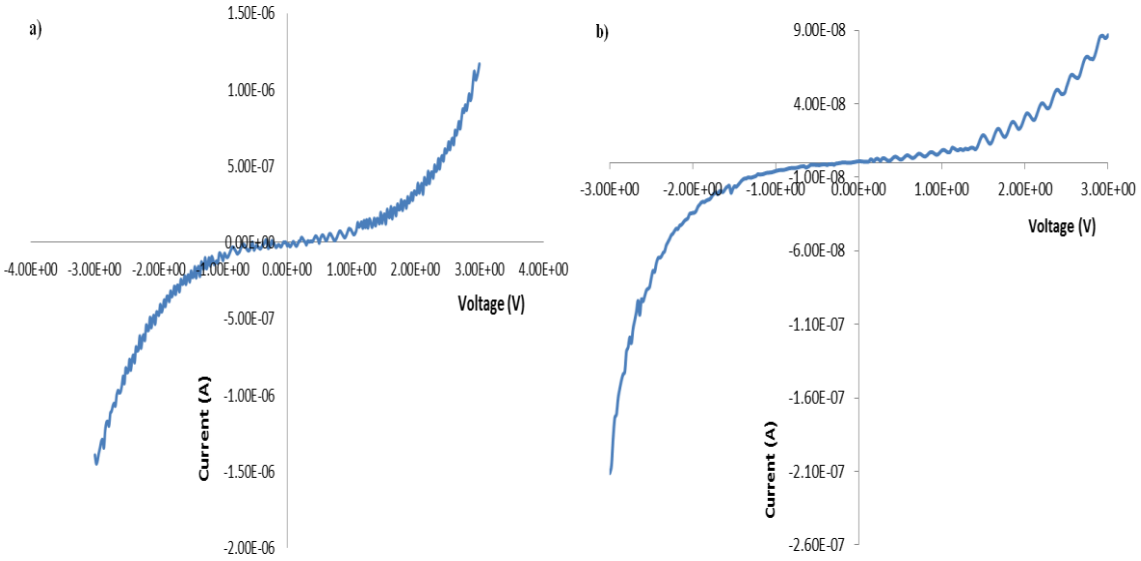

Fig. 4: Current versus voltage graph for 4H-SiC **a)** Unirradiated and **b)** 200 kGy irradiation.

The current-voltage (I-V) test results were shown in Figure 4. The result for unirradiated sample is shown in Figure 4(a) with the resulting graph showing non-linear relationship and perhaps varies exponentially. The graph indicates that the application of silver paste by annealing does not affect its electrical properties. Assuming that the silver paste induced a built in voltage, because both sides of the wafer were coated and annealed with the same process, the flow of current in the forward and reverse bias should follow the same characteristics with similar magnitudes. Using the four point probe, the value of resistance of the unirradiated sample is 0.8 MΩ, whereas the value for sample irradiated at 200 kGy, this 5.2 MΩ as shown in Table 3. To get the resistivity, the well-known formula below (1) was applied.

$$\rho = \frac{\pi d}{ln2} R \qquad (1)$$

Where ρ is the resistivity, d is the thickness of the sample and R is the resistance. Using this formula the resistivity value obtained for the unirradiated sample and 200 kGy irradiated is 1087.8Ωm and 7070.5Ωm respectively with the conductivity just being the inverse of it. The I-V graph for 200 kGy irradiated sample as shown in Figure 4 displays a similar trend with unirradiated sample. However 200 kGy irradiated sample does not show similar characteristics with similar magnitudes for the forward and reverse bias. Again, the resistance of the sample varies depending on the position of the slope taken into consideration. Furthermore, taking the reverse bias section from -2V to -3V gives a resistance of 5MΩ which is similar in value to the four point probe reading.

Although it was not possible to obtain the I-V trend for unirradiated sample, the same approach gave a reading of 0.5MΩ. The different areas of the graphs were chosen because the resistance value was found to correspond to the four point probe value. The changes in resistances hence the conductivities could possibly be due to microstructure changes induced by the radiation but the details are yet known as Positron annihilation spectroscopy (PAS) analysis shows defects propagation occurring. The PAS spectrum is shown in Figure 5 which typical for all samples irradiated by 100, 200, 300 and 500 kGy as well as unirradiated sample. The results show that the samples have one lifetime component of about 151 ps. Time resolution was in the range 262-264ps. This possibly means that the degradation through defect propagation may not possible even when sample is irradiated up to 500 kGy.

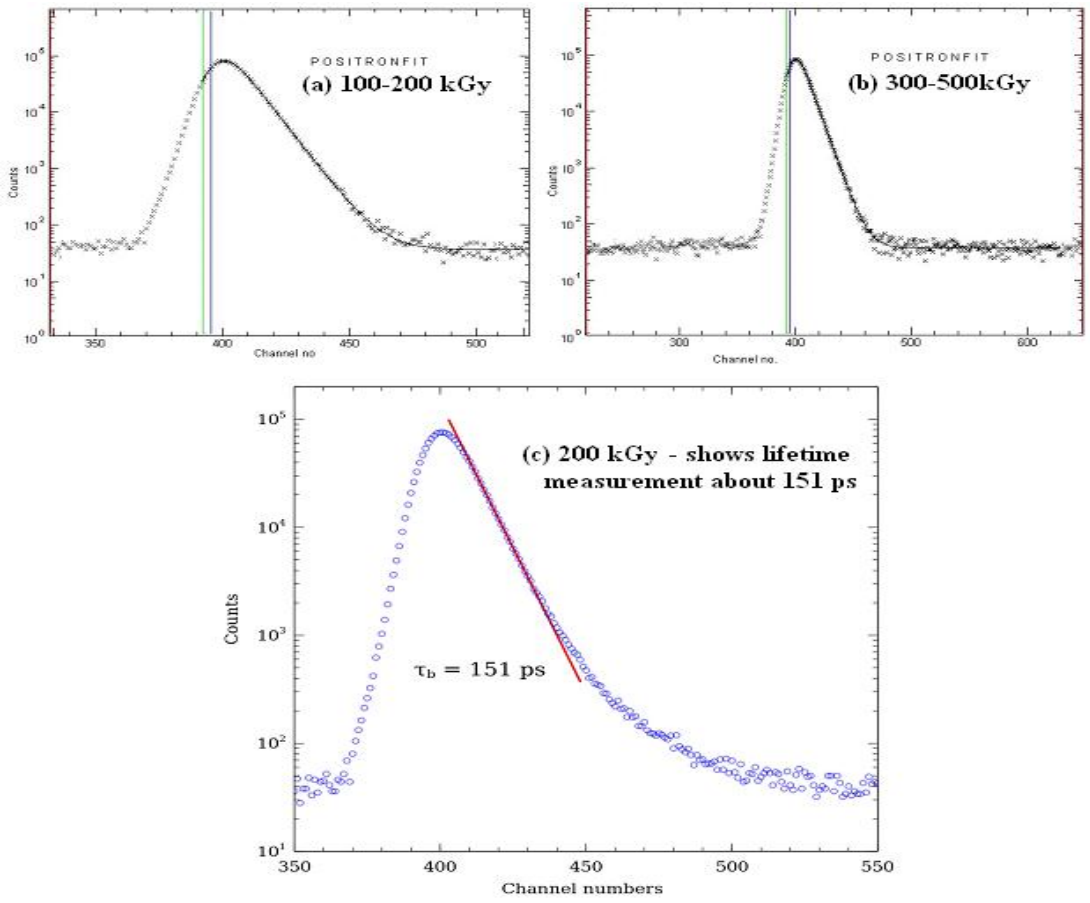

Fig. 5: Positron measurement of electron irradiated samples.

CONCLUSION

The lattice strain values calculated using the (111) peak was found to decrease with increasing electron irradiation dose. The second peak (222) does not give reliable result as peak intensity is very low. The peaks were found to have shifted after exposure to electron radiation with no significant shape changes suggesting a uniform strain and thermal effect may cause sintering of sample that resulted in increased crystalline sizes. The radiation damage on the surface may be due to ionizing effects as observed on the SEM micrographs. Resistivity of the sample was found to be significantly increased after irradiation. PAS analysis nonetheless shows the electron irradiated samples all have same life time and this suggest no propagation of defects on the samples.

ACKNOWLEDGEMENT

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