CHAPTER 8 SUMMARY OF RESULTS

The following topics: in-diffusion, radiation enhanced diffusion of silver, retained radiation damage and annealing of radiation damage in single crystalline 6H-SiC were investigated by RBS, RBS-C and SEM. For in-diffusion investigation two methods were used. In the first, thin silver films (100 nm) were deposited onto the samples followed by annealing while in the second, the samples with silver thin films deposited on their surface were encapsulated followed by annealing. For investigations into radiation enhanced diffusion of silver, retained damage and annealing of radiation damage, silver was implanted into samples at room temperature, 350 ^{o}C and 600 ^{o}C followed by subsequent annealing. This chapter summarises the results of this study.

8.1 SILVER IN-DIFFUSION RESULTS

In-diffusion of silver into 6H-SiC was first investigated at a temperature below the melting point of silver (960 ^{o}C) using un-encapsulated 6H-SiC samples with 100 nm deposited silver. No silver in-diffusion was observed at 800 ^{o}C but disappearance of silver occurred at this temperature. For the encapsulated samples, no in-diffusion of silver was observed at 800 ^{o}C, 900 ^{o}C and 1000 ^{o}C but silver disappeared from the samples' surfaces and was found on the walls of the quartz glass ampoule. The disappearance of the silver layer from the SiC surface was investigated at temperatures from 200 ^{o}C to 700 ^{o}C in steps of 100 ^{o}C. This phenomenon was found to be due to the wetting problem between silver and SiC.

8.2 ROOM TEMPERATURE IMPLANTATION

Radiation enhanced diffusion in 6H-SiC was investigated after silver implantation at room temperature by studies of annealing at temperatures from below the melting point (960 ^{o}C) of silver up to 1600 ^{o}C. This was performed by both isochronal annealing and isothermal annealing. The radiation enhanced diffusion was observed by comparing the FWHM of silver implanted depth profiles at different temperatures with those of the post annealing samples.

The room temperature implantation resulted in a completely amorphous surface layer of approximately 270 nm thick. Epitaxial re-growth from the bulk was already taking place during annealing at 700 ^{o}C and the crystalline structure seemed to be fully recovered at 1600 ^{o}C, as regards samples that were sequentially isochronally annealed from 700 ^{o}C in steps of 100 ^{o}C up to 1600 ^{o}C. However, no silver signal was detected at this temperature which left some doubts as to the crystalline structure of the samples at this temperature. It was speculated to be due to thermal etching of the top original amorphous layer while the damage consisted of epitaxial re-growth from the bulk. The decomposition of SiC, giving rise to carbon peaks in the RBS spectra due to the evaporation of Si, was clearly observed on the same samples at 1600 ^{o}C. Isothermal annealing at 1300 ^{o}C for 10 h cycles up to 80 h caused epixatial re-growth from the bulk during the first annealing cycle (10 h). No further epitaxial re-growth from the bulk was observed up to 80 h. This was believed to be due to the amorphous layer re-crystallising into crystals that were randomly oriented to the 6H-SiC. These crystals could be other crystalline forms of SiC such as 3C, 4H, etc. These results were confirmed by Raman spectroscopy but transmission electron microscopy is necessary to further confirm them.

No diffusion of silver was observed at temperatures below 1300 ^{o}C but silver seemed to form precipitates at these temperatures. Diffusion of silver towards the surface accompanied by silver loss from the surface began at 1300 ^{o}C and was very high at 1400 ^{o}C, with silver profiles becoming asymmetric and closer to the surface. The loss of silver was already taking place at 1100 ^{o}C. By means of SEM and RBS analyses, silver loss was found to be due to the following:

- Diffusion of silver towards the surface.
- The mass flow of silver via holes that were observed to be growing larger with higher annealing temperatures on SiC surfaces.
- Thermal etching of SiC.

Isothermal annealing at 1300 ^{o}C for 10 h up to 80 h caused diffusion of silver during the first annealing cycle whereas no further diffusion was observed for any further annealing at the same temperature up to 80 h. The diffusion coefficient was not calculated due to the lack of information on the structural evolution of SiC during the first annealing cycle. Isothermal annealing at 1300 ^{o}C and 1350 ^{o}C for 30 minute

cycles up to 120 minutes caused high diffusion during the first cycle and reduced diffusion during the second cycle, while no diffusion was observed for any further annealing longer than the second cycle. The higher diffusion during the first 30 minutes was due to ion induced amorphazation. The diffusion of silver in the amorphised SiC was measured at different temperatures in the range 1300 ºC to 1385 ^{o}C and yielded to $D_o \sim 1.4 \times 10^{-12}$ m^{2}s^{-1} and $E_a \sim 3.3 \times 10^{-19}$ J. These values were found to be approximately the same as the values of silver diffusion in polycrystalline CVD-grown SiC found by our group, stemming from grain boundary diffusion: $D_o \sim 4\times10^{-12}$ m^2 s^{-1} and $E_a \sim 4\times10^{-19}$ J.

To clearly understand silver diffusion in 6H-SiC especially in the amorphised 6H-SiC, the following still need to be further investigated.

- The diffusion of silver in the amorphised 6H-SiC allows further in-diffusion studies of silver into amorphised 6H-SiC. This would be carried out by performing encapsulated in-diffusion experiments in the amorphised 6H-SiC that is available. If these experiments succeed, the same experiments will be performed on the 6H-SiC amorphised by Si implantation to reduce the complication of measuring the diffusing silver. Since there will be no implanted silver in the amorphous 6H-SiC, it will be easier to measure the silver that is present in-diffusion to amorphised 6H-SiC.
- The annealing of amorphised 6H-SiC after the first cycle needs to be further investigated by Transmission Electron Microscopy (TEM) to verify our suggestion that amorphous SiC re-crystallizes into other SiC polytypes such as 3C-SiC.

8.3 HIGH TEMPERATURE IMPLANTATIONS

Implantation of silver at 600 ^{o}C retained crystallinity, although distortions occurred in the implanted region, while implantation at 350 ^{o}C also retained crystallinity, but a larger number of distortions occurred as compared to silver implanted at 600 ^{o}C. This was caused by the fact that, at 600 ^{o}C, the displaced atoms were more mobile because of their higher thermal energy than at 350 ^{o}C. The greater energy increased the probability that the displaced atoms would combine with their original lattice sites. Annealing of these samples at 1300 ^{o}C, 1350 ^{o}C and 1500 ^{o}C caused some annihilation of defects but a number of defects were retained.

No diffusion of silver was observed during annealing of the samples (implanted at 350 ^{o}C and at 600 ^{o}C) at 1300 ^{o}C, 1350 ^{o}C and 1500 ^{o}C but silver moved towards the surface at 1500 ^{o}C. The upper limit of diffusion coefficient of $D<10^{-21}\ m^2s^{-1}$ was obtained at 1300 ^{o}C which was of the same order of magnitude as results found in literature at 1500 ^{o}C: $D< 5\times10^{-21}\ m^2s^{-1}$, even though our temperature was 200 ^{o}C less. This indicates that our RBS has a better resolution than XPS. The movement of silver towards the surface was found to be due to thermal etching at 1500 ^{o}C. Neutron irradiation of these samples caused no silver diffusion but silver ^{110m}Ag due to ^{109}Ag capturing a neutron during neutron irradiation was detected in the samples. No annealing was performed on neutron irradiated samples, to reduce the chances of contaminating the oven with radio-active isotopes of silver with a long half-life and the danger of high doses of gamma-rays for subsequent users.