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FORMATION OF GaN NANOCRYSTALS IN SiO_2/Si

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Ga and N ions were implanted into a silicon dioxide layer on crystalline silicon. Ion energies were adjusted for obtaining an overlap of the depth profiles at a depth close to the surface. The chosen ion fluences result in maximum concentrations of about 9at.%. Rapid thermal annealing with halogen lamps was performed in nitrogen/ammonia ambient. Optimum annealing conditions for retaining the implanted species within the samples were found to be 1000°C and times between 30 and 120 s. Formation of Ga-N bonds was demonstrated by extended X-ray absorption fine structure measurements. Best results were obtained after annealing at 1000°C for 90 s. In this case pictures of cross section transmission electron microscopy show dark areas which are related to the higher mass of Ga atoms. High-resolution pictures reveal the existence of nanocrystals.

Introduction

Silicon (Si) is still the most widely used semiconductor for device production. Accordingly there is a continuous interest in research for broadening the application of this well developed technology to new types of devices or applications. For obtaining new functionalities nanostructures are often useful. Nanoprecipitates of nanocrystals can be generated by ion implantation and subsequent thermal annealing in Si or in SiO_2 on Si wafers (SiO_2/Si) (see e.g. [1,2] and references therein). The advantage of these processes is their compatibility to the classical Si technology. Furthermore, there is also a more general interest in understanding the physical processes of formation of nanocrystals by ion implantation and subsequent annealing. In this contribution the production of GaN nanocrystals in SiO_2/Si is investigated.

Experimental Conditions

Thermally oxidised Si wafers were subsequently implanted first with 180 keV Ga and second with 50 keV N ions. The ion energies were chosen in order to obtain a good overlap between the depth distributions of the two ion species. The applied ion fluences of 6×10^{16} Ga/cm^2 and 7.5×10^{16} N/cm^2 result in a peak Ga and N concentration of about 8at.% and 9at.%, respectively. An excess N concentration is chosen to account for the higher probability of N loss during annealing [3]. Implantation was performed at nominal room temperature with fluxes below 6×10^{12} cm^{-2}s^{-1}. The implanted samples were annealed using rapid thermal annealing (RTA) with flash lamps. The temperature varied between 1000 and 1300°C for times between 30 and 600s. To avoid N loss during annealing, the atmosphere during RTA was a mixture of 50% N_2 and 50% ammonia [3]. Own experiments as well as results in Ref. [3] showed strong N loss during annealing without ammonia.

Rutherford backscattering spectrometry (RBS) with He ions was used to measure the amount of Ga and N after implantation and annealing. For enhancing the visibility of N, the enhanced non-Rutherford scattering cross section of He on N around 3.6 MeV He ion energy was used [4]. The formation of GaN bonds was studied with X-ray absorption at the Synchrotron Radiation Storage Ring: BESSY (Berlin) and by Raman spectroscopy. Furthermore, transmission electron microscopy (TEM) was employed for demonstrating the formation of nanoclusters or nanocrystals.

Results and Discussion

Figure 1 shows a typical set of RBS spectra for 3.6 MeV He ions backscattered on implanted SiO_2/Si after annealing at 1000°C for different times. Backscattering on the SiO_2 layer occurs at channel number 235-295 (Si part) and 125-190 (O part). The signal of the implanted Ga atoms is seen at channel numbers 400-420 and that of N atoms around 153. The signal from the Si substrate starts at channel 235. The different height in this part is due to channelling effects in the crystalline Si substrate.

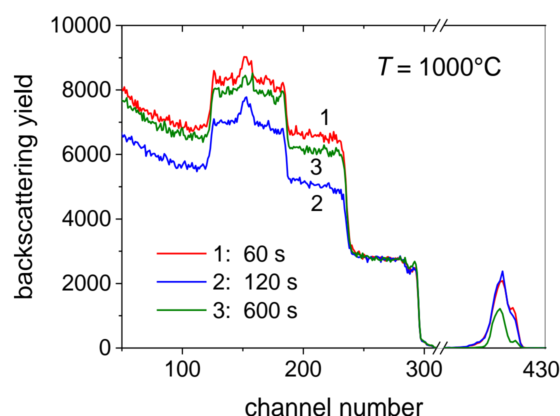

Fig. 1. RBS spectra of 3.6 MeV He ions for Ga and N ions implanted SiO_2/Si after annealing at 1000°C for different times. The thickness of the oxide layer was about 1µm.

In order to investigate the amount of Ga and N atoms remaining in the samples after annealing, the corresponding peaks in the RBS spectra were integrated. For Ga, the spectra measured with 1.4 MeV He ions were used. For N, the peak in the 3.6 MeV spectra was subtracted from the background before

integration. In Fig. 2 the integrated yields are shown normalised to the value after implantation. Part (a) demonstrates the effect of temperature for a fixed time of 30 s. Part (b) shows the results for a fixed temperature of 1000°C as a function of annealing time.

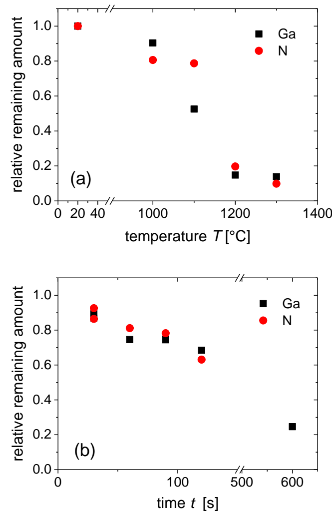

Fig. 2. Relative amount of Ga and N atoms remaining after annealing (a) as a function of temperature for a fixed time of 30 s and (b) as a function of time for a fixed temperature of 1000°C.

From the results shown in Fig. 2 optimum annealing conditions were found to be 1000°C and times between 30 and 120 s. For the samples annealed under these conditions EXAFS (extended X ray absorption fine structure) measurements were performed to search for Ga-N bond. The corresponding Fourier transform amplitudes are shown in Fig. 3. From the comparison of the curves with that measured for crystalline bulk GaN, the occurrence of Ga-N bonds is clearly evident. A detailed analysis of the data reveals closest agreement with bulk GaN for the sample annealed for 90 s. In this case the number of nearest and second nearest neighbours was 4.2 and 10.2, respectively, compared to 4 and 12 for bulk GaN. The occurrence of GaN was further confirmed by Raman spectroscopy. The spectra measured after annealing show a weak band of nanocrystalline GaN around a Raman shift of $735 cm^{-1}$ (not shown).

Fig. 4 shows a first cross section TEM picture of sample annealed under optimum conditions. The occurrence of nanocrystals is clearly evident.

Conclusion

GaN nanocrystals were formed in SiO_2/Si by ion implantation of Ga and N atoms and subsequent RTA. Optimum conditions were found to be 1000°C and 90 s in nitrogen/ammonia ambient. The occurrence of Ga-N bonds was detected by EXAFS. For these annealing conditions, cross section TEM reveals clusters containing heavy elements such as Ga. A preliminary high-resolution picture shows a

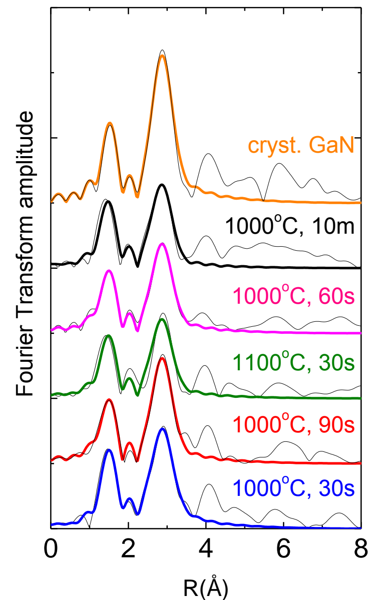

Fig. 3. Fourier transform amplitudes of EXAFS spectra *(color thick lines: fitting, black thin lines: experiment).* For comparison a curve for crystalline GaN is shown (see upper panel of the figure).

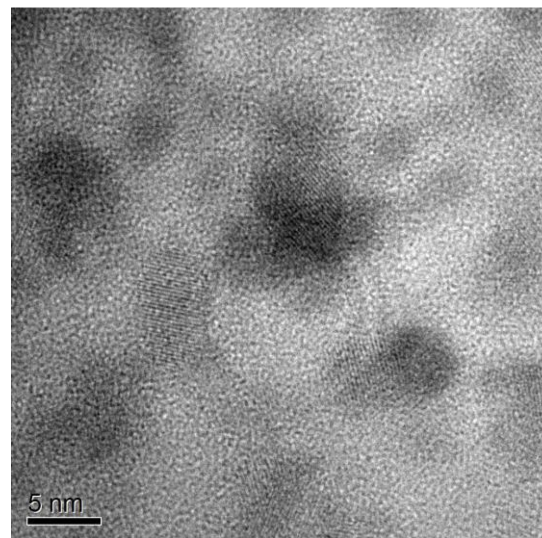

Fig. 4. Cross section TEM picture of Ga and N implanted SiO_2/Si after annealing at 1000°C for 90 s.

crystalline structure within these clusters which most probably can be related to GaN.

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