An EPR Study of Substitutional Phosphorus in Neutron Transmutation Doped Silicon

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The irradiation of silicon with thermal neutrons to obtain uniformly phosphorus doped silicon is gaining technological importance. The donor production is based on the nuclear reactions $^{30}Si+n \rightarrow {}^{31}Si+\gamma$ and $^{31}Si \rightarrow {}^{31}P+e^-$. Unavoidably, these reactions also create substantial radiation damage. Therefore it requires high temperature anneal to restore the host lattice and to force the phosphorus atoms to occupy the regular donor sites.

This process was investigated in an isochronal annealing procedure, with anneal times of 1 hour at successively higher temperatures up to 800 °C, by means of electron paramagnetic resonance (EPR). Measurements were performed in a K-band superheterodyne spectrometer (frequency 23 GHz) tuned to observe the dispersion part of the susceptibility, with the sample at a temperature of about 8 K. The phosphorus atoms on substitutional lattice sites are detected unequivocally by the associated EPR spectrum with a g-value g = 1.9985 and the characteristic hyperfine splitting of 42 G. The donor concentrations were determined accurately by comparing the resonance amplitudes with those of a conventionally doped reference sample of known resistivity. Experiments were performed on silicon irradiated with 1.3×10^{18} thermal neutrons per cm^2. For this fluence one expects a production of 2.2×10^{14} phosphorus per cm^3 corresponding to a room temperature resistivity of 22 Ohm.cm.

From this experiment the four main conclusions can be summarized as follows. Already before any intentional anneal treatment has been given the concentration of phosphorus on substitutional sites amounts to 6×10^{13} atoms per cm^3. This means that immediately after the primary production event and the subsequent spontaneous relaxation as much as about 25% of the phosphorus atoms has found a regular lattice position in a largely undistorted environment. Upon anneal the substitutional phosphorus content increases, quite gradually in the broad temperature range from 250 to 700°C. In this temperature range apparently the phosphorus atoms are released by anneal of a variety of radiation defects of which they were constituents. Observation of the EPR signal to its full intensity requires illumination of the sample with white light, or with silicon-filtered near-bandgap light. Only after anneal above 550°C the phosphorus EPR spectrum is also visible when the sample is kept in the dark. Between 550°C and 700°C the electron traps disappear and the Fermi level recovers to the normal n-type position. Both processes, i.e. occupation of regular sites by

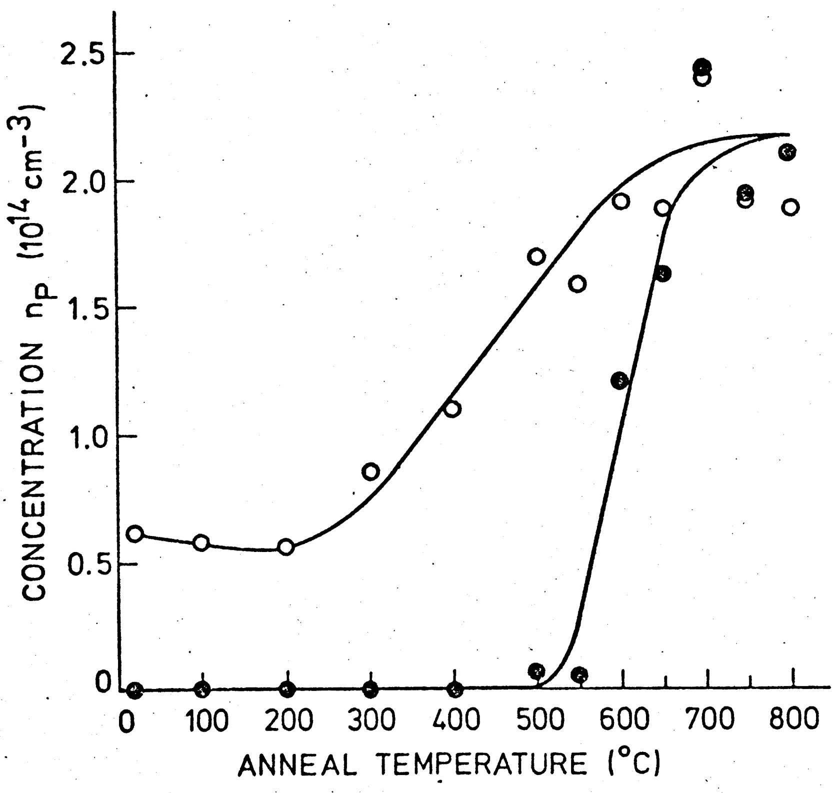

Figure 1. Concentration n_P of substitutional phosphorus in neutron transmutation doped silicon after isochronal anneal of 1 hour at temperature T_{anneal}. Measurements made in the dark ●, and under illumination 0.

phosphorus atoms and the anneal of electron traps, reach completion at the same anneal temperature of 700°C. This suggests that the non-substitutional phosphorus participates in complexes capable of electron trapping. The data on which these conclusions are based are presented in figure 1.

After the anneal at 700°C the EPR intensity corresponds to a phosphorus concentration of 2.4×10^{14} per cm^3. The corresponding room temperature resistivity of 20 Ohm.cm was confirmed by a four-point-probe measurement.