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Electron trap annealing in neutron transmutation doped silicon

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Silicon doped by neutron transmutation to 1.2×10^{14} phosphorus atoms/cm^3 was investigated with deep level transient spectroscopy using evaporated Au/n-Si diodes. Seven bulk electron traps were identified which appear after 30 min N_2 anneal at temperatures between 425 and 725°C. Five of these annealed in the manner characteristic of intrinsic defects studied by EPR and ir spectroscopy. Two may be related to residual oxygen and carbon complexes.

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Within the last few years neutron transmutation doping[1] has been proven a viable technique for producing silicon with extremely high purity and homogeneity.[2,3] Substantial damage, however, is induced during the irradiation process. Recently Schulz and Lefèvre[4] concluded that deep level transient spectroscopy[5] (DLTS) constitutes a particularly useful method for detecting the residual defects in annealed material, but made no attempts to characterize the observed traps. In this work the annealing of electron traps is studied in more details by means of DLTS.

n-type dislocation-free float-zone silicon with starting resistivity >1000 Ω cm and carbon and oxygen concentrations $<2\times10^{16}$ cm^{-3} was used in the present study. Doping to a level of 1.2×10^{14} phosphorus atoms/cm^3 (40 Ω cm) was carried out in a thermal nuclear reactor with a thermal–to–fast-neutron flux ratio of 50 : 1, and at a temperature of approximately 135 °C. Wafers cut from the irradiated crystal were etched and subsequently polished on one side. After 30 min anneal in N_2, 100 μm was removed from either side of the wafer by chemical polishing in order to suppress surface effects. DLTS measurements were performed on evaporated Au/n-Si diodes 2 mm in diameter utilizing a standard Boonton 72B capacitance meter. The double sampling circuitry[6] used as the required rate discriminator had a trap detection limit of 0.1% of background doping. Appropriate corrections were made to account for the time constant of the bridge.[6]

Figure 1 shows the position of the more prominent spectral lines due to majority-carrier traps after anneals at 450 °C or above. Spectra obtained up to 725 °C all include one or more of these lines with varying amplitudes. Activation energies were determined in the usual manner[5] of plotting the emission rate e' times $(300/T)^2$ versus $1/kT_{peak}$. A combined plot for the above defects is presented in Fig. 2. Energy levels and cross sections identified after anneals in the range 425–800 °C are tabulated in Table I. A few traps, however, which were found in concentrations less than 4×10^{11} cm^{-3} are not listed. As for (h) it appears to be a surface-generated acceptor-type defect with a diffusion constant of 10^{-8} cm^2/sec at 500 °C. As usual the activation energies incorporate temperature dependencies in the cross sections and possible Poole-Frenkel effects, therefore the cross sections should only be accepted as a guide line.

Trap concentrations as a function of annealing temperature are illustrated in Fig. 3. Below 425 °C the defect concentration was so large that free-carrier freeze-out prevented any capacitive measurements. In the temperature range up to 600 °C the annealing

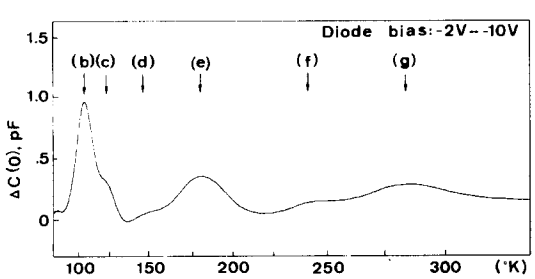

FIG. 1. Electron trap spectrum produced in transmutation doped FZ silicon (40 Ω cm) after 30 min N_2 anneal at 500 °C. Emission rate, 180 sec^{-1}. Defect labels refer to Table I.

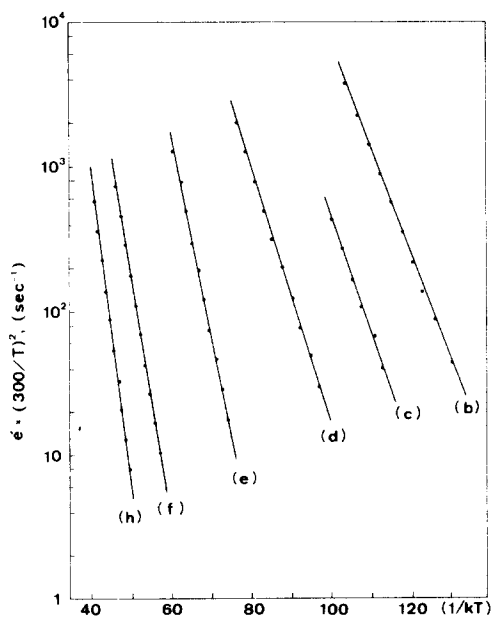

FIG. 2. Arrhenius plot of corrected e' values versus $1/kT$ for defects shown in Fig. 1.

TABLE I. Electron traps present in neutron-irradiated FZ silicon after 30 min N_2 anneal.

	$E_C - E_T$ (eV)	Cross section (cm^2)	Temperature range (°C) (min, max)
(a)	0.15	2×10^{-16}	• • •, 450
(b)	0.17	4×10^{-16}	475, 550
(c)	0.19	2×10^{-16}	450, 625
(d)	0.20	4×10^{-17}	450, 725
(e)	0.32	1×10^{-15}	• • •, 550
(f)	0.40	3×10^{-16}	450, 525
(g)	0.46	3×10^{-15}	• • •, 425
(h)[a]	0.50	2×10^{-15}	450, 525

[a]Appears to be a surface effect.

behavior bears strong resemblance to the results obtained by EPR,[7] i.e., the disappearance of the Si-P-1 spectrum between 450 and 500 °C, and also to those of ir absorption spectroscopy, i.e., the generation and disappearance of electronic higher-order bands (HOB)[8] and the complete anneal of band-edge absorption at 550 °C.[9,10] Above 750 °C no defects were detected in agreement with Herzers findings[3] for reactors with a cadmium ratio larger than 10 : 1.

On this basis it is tempting to associate the (g) level with the pentavacancy responsible for the Si-P-1 spectrum,[11] (a) and (b) with the two HOB defects, and (e) and (f) with the (vacancy) defects causing the residual band-edge absorption. Unfortunately, structural details which might substantiate these assignments are only available for the pentavacancy and for the HOB defect responsible for the 1102-, 1048-, and 1124-cm^{-1} resonance lines.

For the pentavacancy the symmetry of the g tensor below room temperature indicates a strong relationship to defects which have primarily one dangling bond in the ⟨111⟩ direction (donor-vacancy complexes) or two parallel ⟨111⟩ bonds (single negative charge of the divacancy).[11] DLTS data published on the E centers[12] show levels at 0.44 eV (phosphorus), 0.42 eV (arsenic), and 0.39 eV (antimony), while the energy level 0.41 eV has been identified for the divacancy.[13] Hence an activation energy of 0.46 eV for the (g) level and a cross section of 2×10^{-15} cm^2, although large, appear not to be unreasonable.

As for the HOB defect it is believed to be a vacancy complex and the resonance lines to be associated with electron transitions between the ground state and a number of excited states.[8] In this context an activation energy for electron emission of 0.17 eV is quite acceptable for the (b) level.

For the remaining traps (c) and (d) a specific defect assignment is less obvious. Evidence of an oxygen-vacancy complex which anneals out at 620 °C has been given,[14] but for oxygen-rich material. Also the return of interstitial carbon to substitutional sites at temperatures above 600 °C is well documented.[10,14] In analogy to electron-irradiated silicon this may generate substitutional-interstitial carbon complexes within a certain temperature range. It should be emphasized though that defect interactions at this stage of anneal are poorly

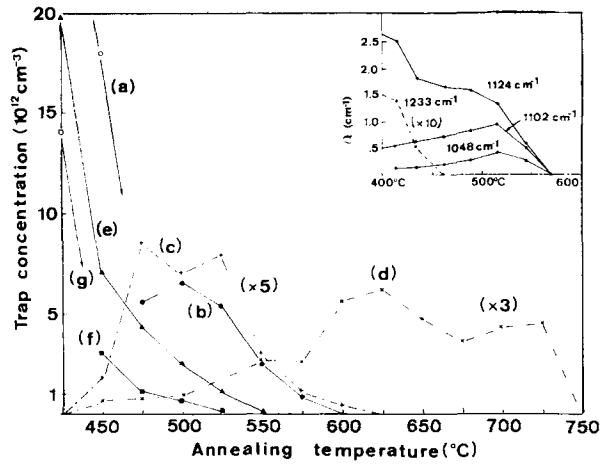

FIG. 3. Annealing of bulk defects in neutron transmutation doped FZ silicon (40 Ω cm). Inset shows annealing of higher-order infrared absorption bands (Ref. 8).

understood. In particular the factors governing the generation and annihilation of higher-order bands are not sufficiently explored. Moreover Si *di*-interstitials and interstitial carbon complexes have been identified in p-type material,[15,16] but the role of Si and carbon interstitials in n-type material needs further elucidation.

In summary evidence has been presented that electron traps observable with DLTS follow the defect annealing behavior established by other investigations. Furthermore, in those cases where detailed descriptions of defect structures are available, the activation energies derived may be successfully correlated with other defect properties.

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