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High resolution deep level transient spectroscopy applied to extended defects in silicon

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Abstract

Deep level transient spectroscopy (DLTS) and high resolution Laplace DLTS (LDLTS) have been applied to p-type Czochralski silicon that contains dislocations that have and that have not been locked by oxygen. The stress-induced dislocations have been immobilized by oxygen during heat treatment, which prohibits glide under certain applied shear stresses. The DLTS spectra show typical broad features between 100 and 320 K, characteristic of those seen in other dislocated silicon reported in the literature, and several components are present in the LDLTS spectra. In addition, DLTS spectra show a sharp narrow peak at 40 K at a rate window of 200 s^{-1} in the case of the locked dislocations, but not in the case of the sample where there is no oxygen locking. LDLTS shows that this deep level consists of more than one component and it is proposed that this peak is likely to be due to electrical activity associated with oxygen at the dislocation core. For hole emission at temperatures above 100 K, in the sample with unlocked dislocations, LDLTS detects a change of the emission rate of the carriers from some, but not all, of the components of the broad peak when the LDLTS fill pulse length is changed. This change is ascribed to band edge modification as the electronic states associated with the dislocation charge up during the fill pulse, and causes local electric field-driven emission of trapped charge during the reverse bias phase of the measurement. The LDLTS features which remain constant with fill pulse are proposed to be due to point defects in the material, which are not physically near dislocations.

1. Introduction

Dislocations are undesirable in semiconductors for various reasons. Dislocations in silicon, in particular, have associated deep electronic states in the band gap whose properties may vary depending upon the level of decoration of the dislocation by the impurity [1]. The mechanical strength of semiconductors is also governed by the density of *mobile* dislocations, and therefore

their presence adversely affects device performance for many reasons. Czochralski-grown (Cz) silicon is favoured as the starting material for Si device manufacture due to its superior mechanical strength over float-zone Si. This is attributed to the effect of the oxygen present in the Cz material on the dynamic activity of dislocations under stress [2]. It has since been shown experimentally that oxygen can inhibit the dislocation motion very effectively [3]. Furthermore, this work was able to provide quantitative analysis of oxygen transport to the dislocations. The proposal in [3] was that the higher the unlocking stress, the more oxygen there is at the dislocation core.

However, if any element is to be used to lock dislocations in place, the associated electrical activity needs to be understood. Dislocations decorated with known concentrations of oxygen atoms provide an interesting model system for investigating the electronic effects of impurities in silicon, and their interaction with the strain and electric fields associated with extended defects. Deep level transient spectroscopy (DLTS) has been used since the 1980s to try to understand and analyse the properties of dislocation-related electronic states [4–8]. However, all attempts at detailed trap analysis have been limited by the inherent very broad nature of the peaks due to dislocations. DLTS signals which can be related to electrical activity associated with the presence of dislocations are generally found in the temperature range 100–300 K.

Generally, conventional DLTS measurements alone are not sufficient to identify whether a deep state is associated with a point-like defect or an extended defect. Other experiments need to be performed, such as characterizing the activation energy as a function of DLTS fill pulse, or carrying out capture-cross-section measurements. The activation energy, E_a, of a well behaved isolated point defect is independent of fill pulse duration, but an extended defect charges up and the change in local band bending results in variations in both E_a and the capture cross section, σ [9]. There is a well established theory that says that if the electron capture is into extended defects, causing a Coulombic barrier to evolve with time, the DLTS signal exhibits a dependence on the logarithm of the fill pulse time, t_F [10]. This relationship is given by

$$n_T(t_F) = \sigma v_{th} n \tau N_T \ln(t_F/\tau) \quad (1)$$

where $n_T(t_F)$ is the time-dependent trap occupancy, σ, n, and v_{th} are the capture cross section, the carrier concentration and the thermal velocity respectively, t_F is the fill pulse duration and τ is a measure of the time required for the trapped charge to begin to affect the Coulomb barrier. Plotting the change in capacitance as a function of the logarithm of the fill pulse duration yields a straight line in the special case of emission from dislocation-related traps.

High resolution Laplace DLTS (LDLTS) [11] has recently been applied to dislocated silicon [12] and showed a complex series of emission rates which were difficult to interpret. However, it was clear that the emission rate varied as the fill pulse length in the LDLTS experiment was varied. This is consistent with previous findings of variations in apparent activation energy of the deep level, measured by DLTS, as a function of fill pulse length [13], but the use of LDLTS facilitates a direct measurement of the same effect.

In this paper we exploit the LDLTS technique to characterize new peaks observed in the DLTS spectra of Cz silicon samples containing deliberately introduced dislocations. Some of the samples have been heat-treated to lock the dislocations with some of the oxygen present in the starting material. We demonstrate the strength of LDLTS in identifying the difference between point- and extended-defect-related DLTS peaks in these samples.

2. Sample preparation

The starting material was dislocation-free p-type Cz silicon with an interstitial oxygen concentration of 1.05×10^{18} cm^{-3} (DIN 50438/I). Bars measuring approximately

Table 1. The dislocated and annealed samples used in the study. The higher the unlocking stress required, the greater the amount of oxygen at the dislocation core.

Sample name	Annealing time at 500 °C (h)	Required dislocation unlocking stress (MPa)	Approximate concentration of oxygen atoms per unit length of the dislocations (cm^{-1})
A	35	150	2×10^7
B	15	75	1×10^7
C	0	<10	$\sim 1 \times 10^6$

3.5 mm × 20 mm × 0.69 mm were then cleaved from this material and the cleaved edges were mechanically polished. The bars were indented with a Vickers diamond tip with a 10 g load and a 5 s dwell time. Indents were made in three regions of the bars, at 40 μm intervals. The punched-out dislocation half-loops were grown by a four-point bend at 550 °C. The specimens were then annealed at 500 °C for different times and the unlocking stress and approximate concentration of oxygen atoms per unit length of dislocation were estimated for each sample using data from previous experiments [14, 15]. Table 1 describes the samples after the bending and dislocation locking. The location of the dislocations was determined by differential interference contrast (Normarski) microscopy and this information was used when deciding where to locate the Schottky diodes for the electrical experiments. About 50 μm of material was removed from the surface of the specimens by mechanical and chemomechanical polishing to remove the indents and the effects of oxygen out-diffusion. The samples were not etched, in order to avoid any unintentional introduction of hydrogen.

1 mm diameter Schottky diodes were formed by sputtering Ti and Al on the top surface, through a mask, and the ohmic contact was formed on the rear by Al evaporation. Each contact was thought to overlay roughly the same number of dislocations in each specimen but accurate estimation of their number was not possible. A high stability cryostat was employed for the LDLTS measurements. This technique records the capacitance transient due to carrier emission at a fixed temperature and applies mathematical algorithms to extract the emission rates present in the transient. The choice of temperature is dictated by the original DLTS measurement, i.e. a temperature is chosen at which there is a peak in the DLTS spectrum. Several thousand capacitance transients were averaged, which ensured that the signal-to-noise ratio was $\geqslant$1000:1, which is necessary to separate transients with closely spaced emission rates. The transient was analysed and a plot of peak intensity as a function of emission rate produced. If the transient is truly exponential, only one peak is visible in the LDLTS spectrum, and the area under each sharp peak is proportional to the concentration of the deep level with that emission rate.

3. Results and discussion

Figure 1 shows the DLTS of the three samples A, B and C. The reverse bias applied during DLTS and LDLTS was chosen using I–V and C–V measurements and were −4, −1.5 and −2 V for samples A, B and C respectively. At these voltages, the respective values for the quiescent capacitance, C_o at 300 K were 75, 50 and 28 pF. The fill pulse voltage was 0.1 V for all measurements. There are two distinct regions of interest and we shall discuss these separately. The first is in the temperature region <100 K and the second is above 100 K. Taking the lower temperature region first, it is clear that there is no sharp peak below 100 K in sample C, which is the sample that has not been annealed. The lack of annealing means that

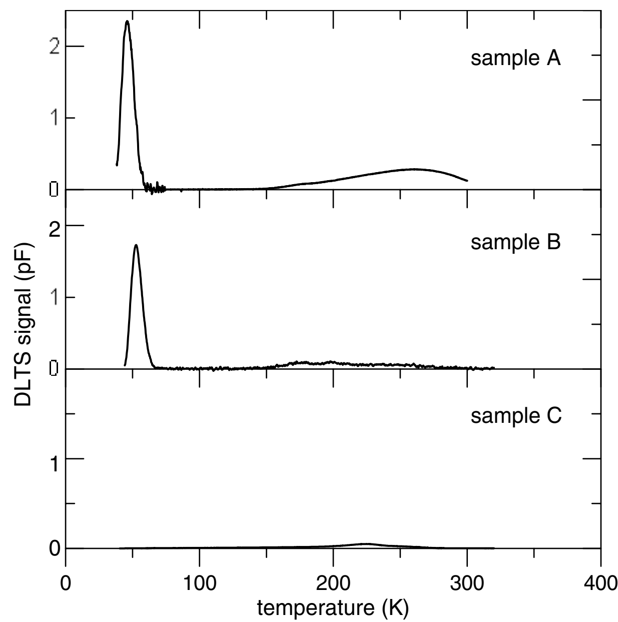

Figure 1. DLTS of samples A, B and C at a rate window of 200 s^{-1} and a fill pulse of 1 ms. The plots are to scale.

Table 2. Activation energies of the low temperature peak, calculated from Arrhenius plots at two fill pulse lengths for samples A and B.

Sample	Fill pulse duration (ms)	Activation energy (meV)
A	1	250
	10	220
B	1	280
	10	260

the dislocations are not intentionally immobilized by oxygen. The required unlocking stress is therefore very low, less than 10 MPa, which arises unintentionally whilst cooling down from 550 °C after the dislocation growth stage. Hereafter we refer to these dislocations as not being locked by oxygen. We return to the higher temperature features later in the paper.

Despite the fact that the peak at about 40–50 K in samples A and B is sharp and narrow, and looks like point-defect-related emission, there is a strong suggestion from these results that it may be related to the presence of oxygen at the core of the locked dislocations. The peak is larger in specimen A than B and apparently absent in specimen C, its height correlating reasonably well with the expected oxygen concentrations at the dislocation cores inferred from their unlocking stresses. If the peak is indeed due to oxygen at the dislocation core it should exhibit properties more likely to be observed from deep levels in the vicinity of extended defects, such as an activation energy that changes with fill pulse length [5, 13], and maybe more than one closely spaced electronic level contributing to the overall carrier emission [12]. The former can be tested by plotting Arrhenius plots from the DLTS data with different fill pulse lengths, and the latter by carrying out LDLTS.

Table 2 shows the results from Arrhenius plots at two fill pulses of the low temperature peak observed in samples A and B. The activation energies are related to the distances from

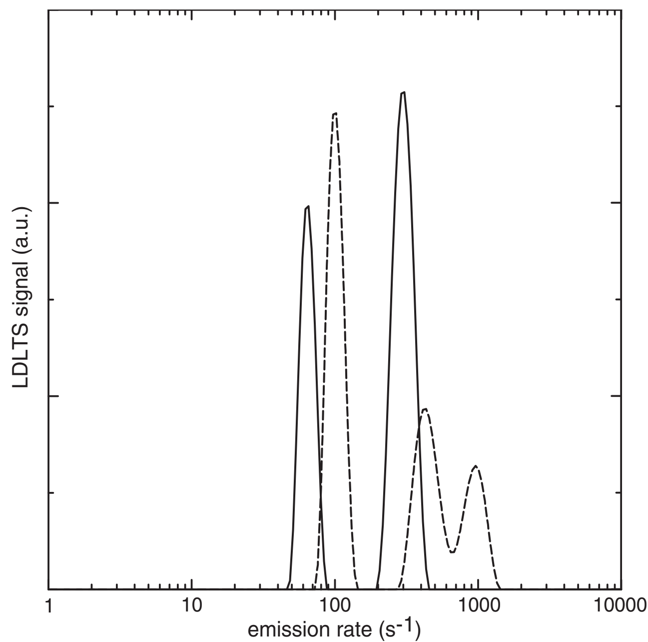

Figure 2. LDLTS of sample B, which contains oxygen-locked dislocations, measured at 50 K with two fill pulses, 1 ms (——) and 50 ms (- - - -).

the valence band, as the polarity of the capacitance transient clearly showed that hole (majority carrier) emission was being observed. If an isolated point defect exhibited a peak at such a low temperature, the emission energy would be expected to be low, or even related to a shallow dopant. In the present cases the emission energies are much higher than those expected for dopants, and also exhibit a dependence on the fill pulse duration, in the same sense as previously observed for decorated stacking faults in silicon [13]. These preliminary results suggest that this feature in the DLTS is not simple point-defect-like, and add to the hypothesis that it is related to the oxygen at the dislocation core. We attribute the unusually large activation energies at this DLTS range to the fact that the emitting centre(s) is (are) in the strain field of the dislocation. Both capture and emission at the traps will be complicated processes in which the charging of the dislocation has a significant effect which evolves with time.

It is worth mentioning that the thermal double donor (TDD) could exist in silicon annealed at 450 °C. Although it exhibits a peak at these temperatures in DLTS [16], the peak is due to electron emission, and the spectrum in figure 1 results from hole emission. It is postulated that the temperature of the anneal in this work is too high at 500 °C and the times too short for formation of the TDD. Typically a hundred or more hours are required. There is no evidence of type inversion in the behaviour of the Schottky diodes or changes in the C–V profile with annealing time.

In order to test whether there is more than one energy level emitting in the region of 50 K, LDLTS was carried out at this temperature and figure 2 shows a typical LDLTS result from sample B measured at fill pulse lengths of 1 and 50 ms. Both samples A and B showed similar behaviour at this temperature. This confirms that the new peak at this low temperature is not due to a simple point defect, as it has more than one energy level contributing to the emission seen in the conventional DLTS spectrum. Furthermore, as the fill pulse duration is increased an extra higher emission feature appears in the spectrum. This is attributed to a longer fill pulse being

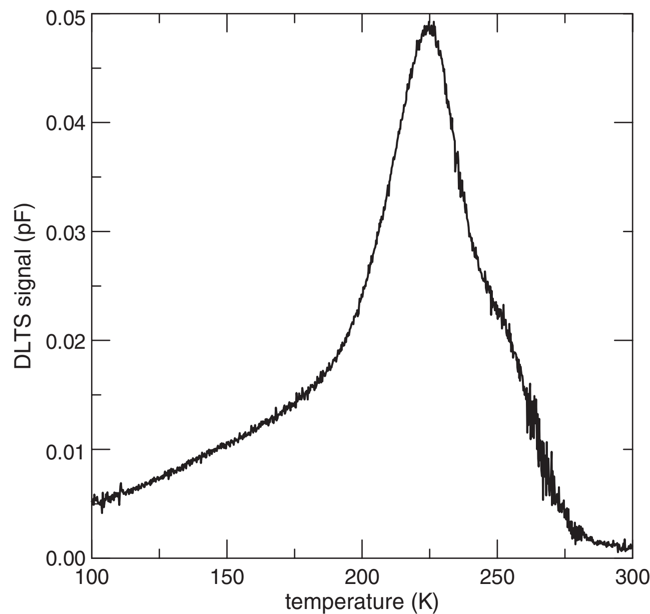

Figure 3. DLTS of sample C, which contains unlocked dislocations, at a rate window of 200 s^{-1} and a fill pulse of 1 ms.

required to enable another state to fill, and emit, because of the repulsive effect of dislocation charging on carrier capture during the LDLTS experiment. We return to this point below.

LDLTS was then applied to the higher temperature peaks in the DLTS spectra shown in figure 1. Samples A and B (shown later) revealed a complicated emission spectrum with many lines in the LDLTS spectra. However, the unannealed sample C shows an interesting feature in the conventional DLTS at higher temperatures, and this was investigated further by LDLTS. Figure 3 shows the DLTS of sample C at a rate window of 200 s^{-1} between 100 and 300 K. There is a symmetrical peak centred at 225 K plus higher and lower temperature shoulders indicating that the sharp peak is superimposed on a broad background peak. The symmetrical peak is quite narrow and is a candidate for hole emission from isolated point defects. Figure 4 shows two LDLTS spectra from this sample measured at 225 K, the temperature at which the narrow peak is centred in the DLTS, at two different fill pulse lengths, 1 and 10 ms. If the emission is from defects in the vicinity of a dislocation, changing the fill pulse duration should change the emission rate because of local band bending as the dislocation charges up during the experiment. This is directly observable in figure 4 for some, but not all, of the peaks. Additionally, an extra line appears in the spectrum after a 10 ms fill pulse. One simple explanation could be that the extra peak could be considered as a hole state possessing a low capture cross section that only fills after a very long fill pulse. This is related to the concept of deep states near a dislocation becoming harder to fill as the dislocation charges up because of repulsive carrier band bending effects [9, 10]. The effect was also observed at 50 K in sample B, as mentioned above. However, there are possible alternative explanations related to multiple states at the dislocation core filling at different rates depending upon the occupancy of the other states [12].

The peaks with slower emission rates can be considered to have a constant emission rate with fill pulse. Figure 4 has a logarithmic x axis in order to show all the data: figure 5 shows

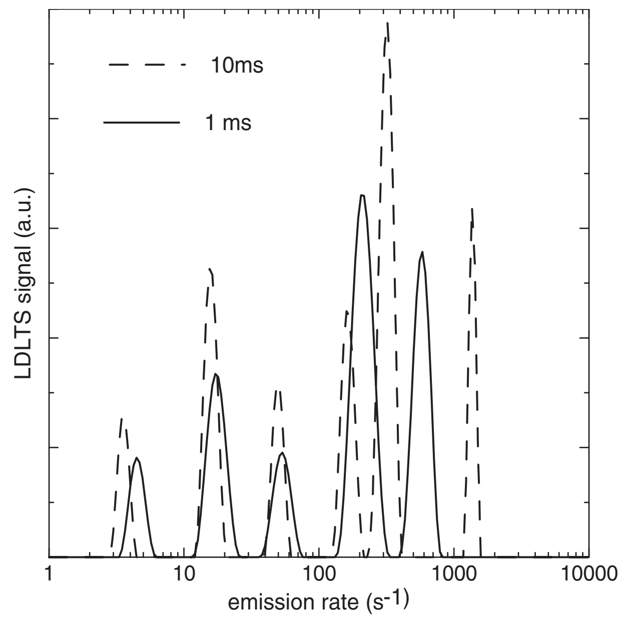

Figure 4. LDLTS of sample C, which contains unlocked dislocations, measured at 225 K, at two different fill pulse lengths.

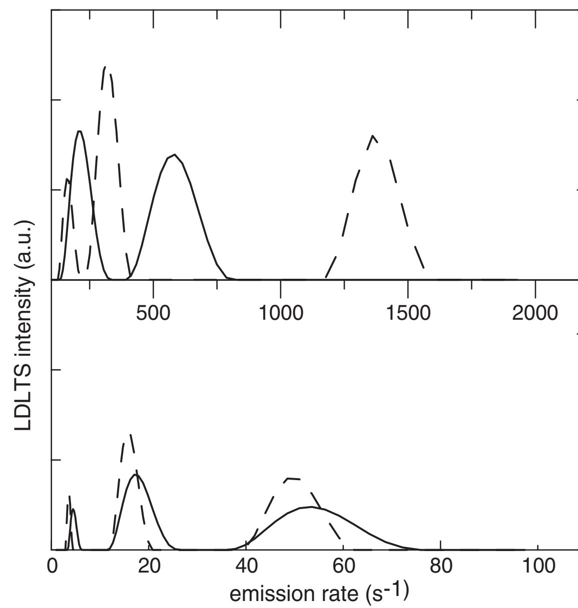

Figure 5. Linear plots of LDLTS obtained from sample C, which contains unlocked dislocations, measured at 225 K, showing the change in emission rate of the deep states which emit at a rate $>100\ s^{-1}$.

the low and high emission rates separately with a linear axis to confirm that some of the peaks are invariant with fill pulse duration. The very small variations observed for the low emission rate peaks are well within LDLTS error bars [11]. This confirms that the high temperature

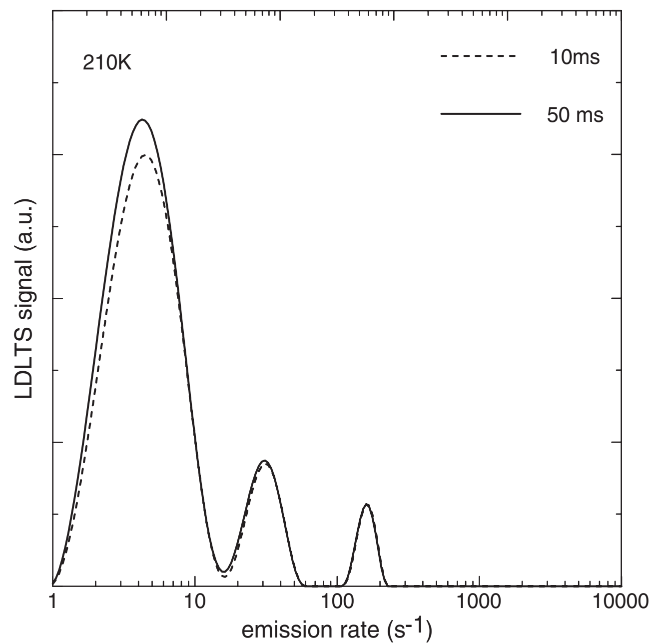

Figure 6. LDLTS of sample B, which contains oxygen-locked dislocations, measured at 210 K, with fill pulse lengths 10 and 50 ms.

feature in sample C, with dislocations not locked by oxygen, consists of emission from both isolated point defects and extended defects. Arrhenius plots of the peak centred at 225 K in figure 3 in DLTS revealed a constant value of E_a with fill pulse duration, i.e. the emission is from isolated point defects. The emission seen as the broad background peak in sample C is therefore deduced to be from defects in the vicinity of the dislocations, as evidenced by the change in emission rate in the LDLTS of some of the peaks. Samples A and B do not exhibit this effect. Figure 6 shows LDLTS from sample B at two fill pulse durations, 10 and 50 ms, measured at 210 K. Three invariant peaks are observed, at very similar emission rates to the invariant peaks in figure 5, from sample C. Sample A (not shown) exhibits similar behaviour to sample B.

It is concluded that the oxygen locking may significantly change the dislocation-related deep state population in these samples. In the locked samples the extended-defect-related states are observed between 40 and 50 K in DLTS but have anomalously large activation energies. In sample C, which contains dislocations not locked by oxygen, the states related to extended defects appear in DLTS above 200 K. The use of LDLTS has enabled this identification.

4. Conclusions

High resolution Laplace DLTS has been applied to the study of p-type dislocated silicon and has identified deep states which are modified when the dislocation motion is inhibited by oxygen at the dislocation core. Before locking, hole emission is detected at >200 K in the DLTS spectrum, but after locking, a new peak appears at 40–50 K. In both cases the activation energy varies with fill pulse duration, and LDLTS shows that there is more than one energy level contributing to this emission. From these results it is deduced that this deep level may be associated with oxygen at the dislocation core.

All the dislocated samples, whether the dislocations are locked or not, exhibit a typical broad emission in the DLTS between 100 and 300 K. High resolution LDLTS has successfully been used to establish that this feature only has a components due to emission from deep states in the vicinity of dislocations, when those dislocations are not oxygen locked.

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