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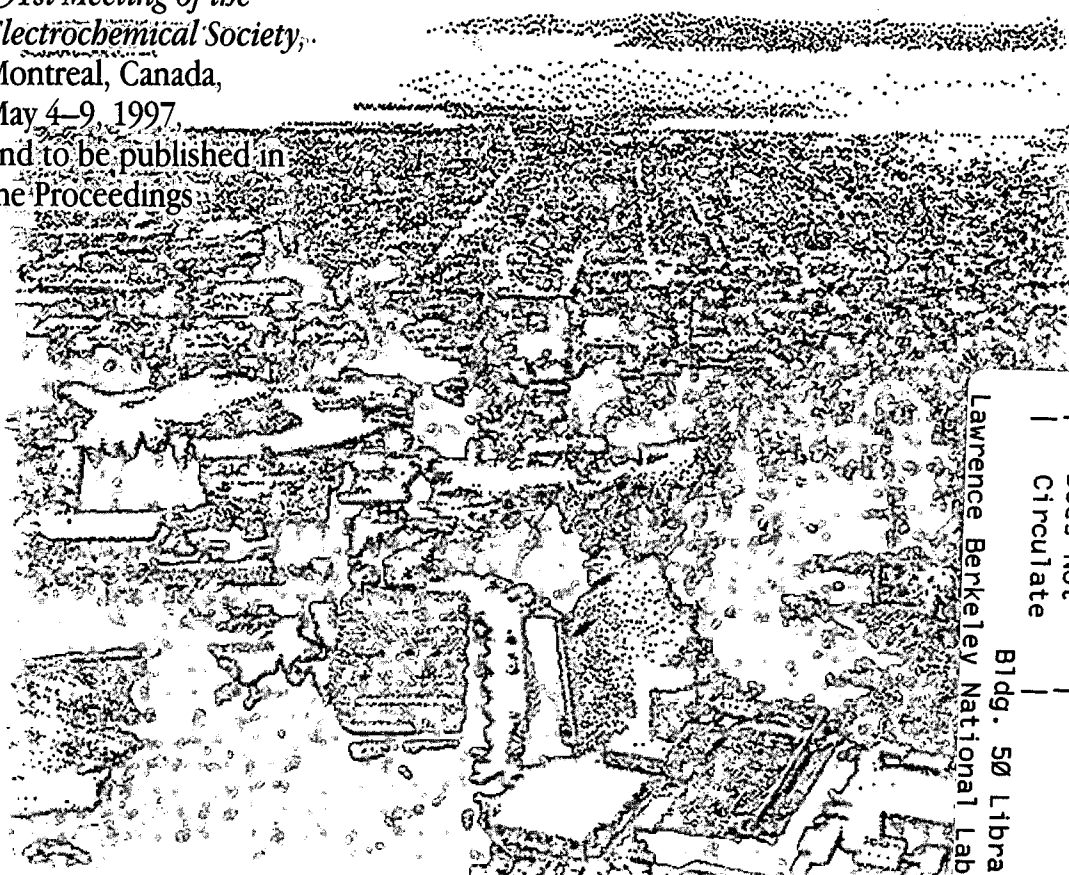


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Lithium-Ion Drifting: Application to the Study of Point Defects in Floating-Zone Silicon

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ABSTRACT

The use of lithium-ion (Li^+) drifting to study the properties of point defects in *p*-type Floating-Zone (FZ) silicon crystals is reported. The Li^+ drift technique is used to detect the presence of vacancy-related defects (D defects) in certain *p*-type FZ silicon crystals. SUPREM-IV modeling suggests that the silicon point defect diffusivities are considerably higher than those commonly accepted, but are in reasonable agreement with values recently proposed. These results demonstrate the utility of Li^+ drifting in the study of silicon point defect properties in *p*-type FZ crystals. Finally, a straightforward measurement of the Li^+ compensation depth is shown to yield estimates of the vacancy-related defect concentration in *p*-type FZ crystals

INTRODUCTION

We report on the use of lithium-ion (Li^+) drifting in an electric field to study the properties of point defects in *p*-type Floating-Zone (FZ) silicon crystals (1). The properties of Li^+ in FZ silicon are well known from the many studies on the Li^+ drift process itself and from the interaction of Li^+ with point defects and impurities (2,3). In the following we first briefly describe the Li^+ drifting process and then show some Li^+ compensation results that we have obtained on a few *p*-type crystals. We next discuss how we have used the Li^+ drift technique to detect the presence of vacancy-related defects in certain *p*-type FZ silicon crystals and to estimate the silicon self-interstitial and vacancy diffusivities at 950 °C (4). We conclude by showing that the Li^+ compensation depth can be used to estimate, in *p*-type FZ silicon crystals, vacancy-related defect concentrations. We propose that measurement of the Li^+ compensation depth be employed to complement the Gate Oxide Integrity (GOI) test in the evaluation of FZ crystal quality (5).

Li⁺-ION COMPENSATION

The properties of interstitial Li⁺ in crystalline germanium and silicon were initially explored over forty years ago (6). These early studies indicated that the coupling between Li⁺ and various defects in the semiconductor was a sensitive probe for the nature of these defects and for their distribution in a crystal. With the subsequent realization that thick semiconductor regions could be made nearly intrinsic by Li⁺ compensating acceptors, fabrication of Li⁺ compensated silicon (Si(Li)) detectors was initiated at various laboratories (7). This is now the principal application for this Li⁺ compensation technique in silicon.

While we have reviewed our fabrication of Si(Li) detectors elsewhere, for the following we need to briefly highlight a few aspects of this process (8). Figure 1 illustrates the salient points. The fabrication of a Si(Li) detector begins with the diffusion of lithium (375 °C, 15 minutes) into a *p*-type silicon wafer (~ 3 mm thick, ~ 1,000 Ohm-cm) to form a *n*⁺-*p* junction. The resulting lithium diffusion profile is approximated by the complementary error function shown by the dotted line in the figure. We then heat the wafer to ~110 °C and apply a ~500 V reverse bias across the *n*⁺-*p* junction to drive (with the electric field) Li⁺ from the *n*⁺ lithium diffused region into the *p*-type bulk region. After some time, the Li⁺ profile assumes the shape represented by the solid line in Fig. 1. The

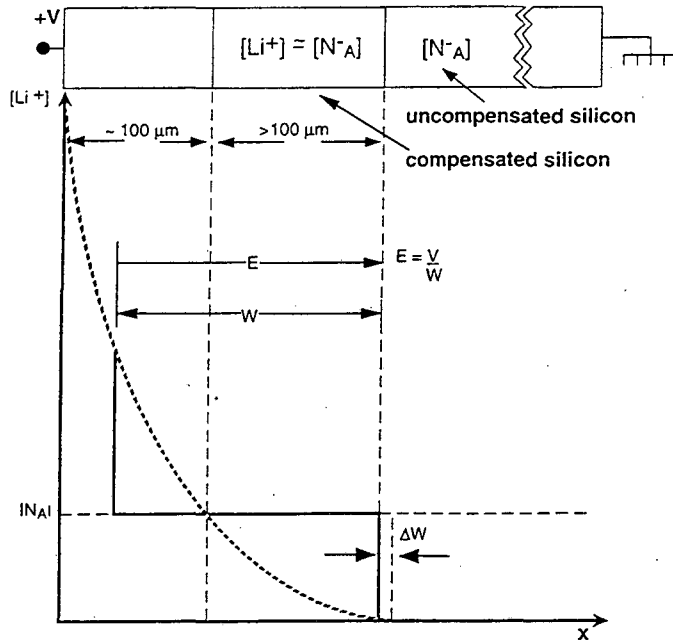


Figure 1. The approximate lithium ion distribution after the initial diffusion (dotted line) and after some of the lithium ions have been drifted into the *p*-type bulk silicon (solid line). The Li⁺ concentration in the bulk is approximately equal to the acceptor concentration, [N_A].

following differential equation then describes the growth rate of the Li^+ compensated region, W (9):

$$\begin{aligned} N_A dW/dt &= \mu_{Li} N_A E - N_L W R_L, \text{ or} \\ &= \mu_{Li} N_A V/W - N_L W R_L, \\ \text{or, } dW/dt &= \mu_{Li} V/W - N_L W R_L / N_A \end{aligned} \quad [1]$$

where μ_{Li} is the Li^+ mobility, N_A is the net-acceptor concentration in the p -type wafer, V is the voltage applied across the compensated region, N_L is the concentration of lithium-ion precipitation sites and R_L is the average rate of Li^+ loss due to these sites. Assuming that V , μ_{Li} , and R_L remain constant, the compensated region width, $W(t)$, after a drift time, t , is:

$$\begin{aligned} W(t) &= W_{max} (1 - e^{-2t/\tau})^{1/2} \\ \text{and } W_{max} &= (\mu_{Li} V \tau)^{1/2} \end{aligned} \quad [2]$$

where $t > 0$ and $\tau = N_A / (N_L R_L)$ is the effective Li^+ lifetime. If the Li^+ loss is significant, then $\tau \ll t$ and the maximum compensated depth can only reach W_{max} , independent of the drift time.

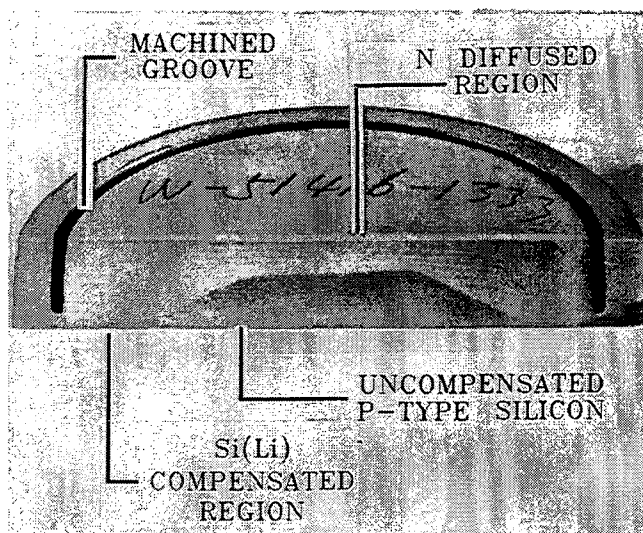


Figure 2. A photograph of a 50 mm diameter, 5 mm thick Si(Li) detector (fabricated on a 50 mm diameter FZ p -type FZ crystal) that we sectioned and copper stained to reveal the Li^+ compensation profile.

Figure 2, which is a photograph of a sectioned Si(Li) detector, shows the effect of a short Li^+ lifetime on the compensation depth realized. Here we subjected the wafer to a drifting time that should have resulted in Li^+ compensation throughout the full wafer thickness. Yet, as can be seen in the figure, the center region of the wafer is not compensated. The results in Fig. 2 actually display an often observed characteristic of certain p -type FZ crystals. Guislain and DeLaet, much earlier, observed a similar profile on p -type FZ crystals that "were quenched very fast", and suggested that the defect responsible had a "relatively high mobility at even moderate temperatures"(10). In our p -type

crystals we have attributed this profile to the presence of a high concentration of vacancy-related point defects that act as Li^+ precipitation sites at the center of these crystals (11).

NATIVE POINT DEFECTS IN SILICON

In the following we will only outline the present understanding of some extended native point defects in silicon. Prior to 1975 the two principal observed defects in FZ single crystals (the so called A and B swirl defects) were assumed to be vacancy clusters. Since 1975 there has evolved a general agreement that these A and B defects in silicon crystals are agglomerations of silicon interstitials (12). Since we will refer to pre-1975 publications, we have reinterpreted, when necessary, the earlier results on Li^+ behavior (drift and precipitation) in view of the present understanding of the A and B defects. In addition to the A and B defects, D defects have been described and identified as vacancy-related structures. A discussion of these D defects effect on Li^+ compensation is the main focus of this paper.

For FZ crystals it has long been recognized that the concentrations of the A, B and D defects are dependent on the silicon crystal growth rate. Roksnoer and Van den Boom, and, more recently, Abe and Kimura have reported that, in FZ silicon, there are specific radial regions of the crystal where one defect type dominates over the others (13,14). Abe and Kimura's observation that the central region of FZ crystals can have high concentrations of D defects correlates well with our experimental observations that certain *p*-type FZ crystals have central regions with apparently high Li^+ precipitation rates.

In the past decade the removal of detrimental imperfections in silicon crystals (gettering) has been the subject of extensive theoretical and experimental research (15). And, in silicon, the consensus view is that interstitials play a primary role in a variety of gettering techniques. In particular, Abe and Kirmura have shown that, with a gettering schedule of 20 minutes at 1000 °C in wet oxygen, they could reduce the D-defect concentration in their FZ crystals. The oxidation produced silicon interstitials that apparently combined with the D defects. However, a high concentration of phosphorus at the silicon surface is reported to be a more efficient source of silicon interstitials than an oxide layer alone (16). We have now employed a phosphorous-glass ($POCl_3$) gettering process, with different temperatures and times, on a series of *p*-type crystals, using Li^+ compensation to assess the effectiveness of the gettering procedure. A comparison of the Li^+ compensated regions between an ungettered and gettered wafer is shown in Figure 3.

At this point we have not determined the exact nature of the point defect related mechanism that is responsible for results such as those shown in Figure 3. Ferman has suggested that Li^+ precipitation in silicon can take place via a reaction with oxygen-vacancy (O-V) centers (17). In modeling our $POCl_3$ gettering experiments, we have assumed that the injected interstitials are combining with or annihilating the vacancies present in the central regions of our crystals. We have not examined the effect that the

gettering process might have on the oxygen-vacancy pairs (A-center) present. Therefore we cannot confirm the applicability of Ferman's model to our crystals even though our range of values for the vacancy-related point defect concentrations, $\sim 10^8$ to $10^{12}/\text{cm}^3$ is similar to Ferman's estimates for the [O-V] precipitation centers in his crystals.

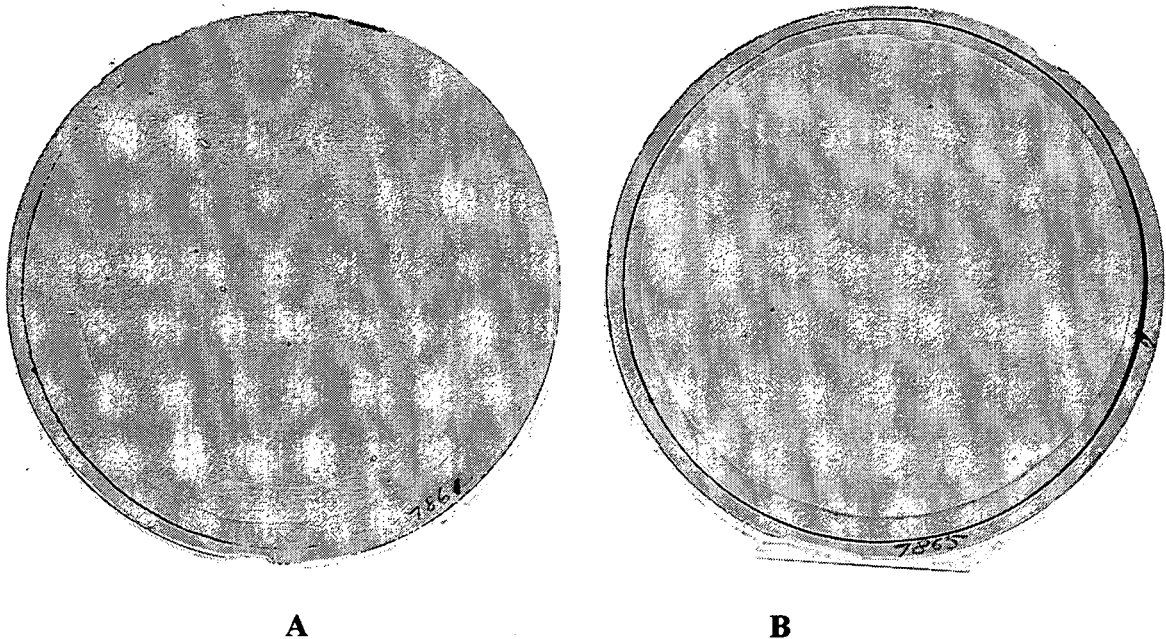


Figure 3. A photograph of an ungettered (A) and a gettered (B) 75 mm diameter, 3 mm thick *p*-type FZ wafers from the same crystal (75 mm diameter), which we Li^+ compensated and then copper stained. The lightly shaded region on each is the L^+ compensated region. The wafer in (A) was actually drifted for twice as long as that in (B) without the Li^+ drifting through the wafer. This result in (A) is similar to that shown in Figure 2.

GETTERING RESULTS

In the following we describe our use of Li^+ compensation to examine the native point defect diffusivities and concentrations, and to detect the presence of native point defects in nitrogen-doped FZ silicon. In both instances we Li^+ drifted the nominally 3 mm thick samples for a fixed time-voltage duration. We then sectioned the samples and decorated the compensated region with a copper staining solution, which is essentially a room temperature copper-silicon displacement reaction (18). The profile shown in Figure 2 was obtained with this technique.

Point defect diffusivities and concentrations

The diffusivity and concentration of the native point defects in silicon crystals have been debated for many years before and since the work by Tan and Gösele to reconcile the differing models (19). It has been proposed that the interaction between these native point defects can be described by the following coupled differential equations (20):

$$\partial C_I / \partial t = \nabla \cdot (D_I \nabla C_I) - K_r (C_I C_V - C_I^* C_V^*) \quad [4]$$

$$\partial C_V / \partial t = \nabla \cdot (D_V \nabla C_V) - K_r (C_I C_V - C_I^* C_V^*) \quad [5]$$

where K_r is the bulk recombination constant between vacancies and interstitials, D is the diffusivity, C is the concentration, and the superscript * denotes equilibrium values while the subscripts I and V refer to, Si interstitials, and vacancies, respectively.

The process modeling program SUPREM-IV contains a flexible solution routine to Eqns. 6-7 and we have used this program to model our POCl_3 gettering process on 3 mm thick wafers (21). To simulate the reduction of vacancies over a depth of ~ 1.5 mm, as shown in Fig. 5, we have had to adjust the SUPREM-IV program default coefficients for D_I , D_V , C_I^* and C_V^* . Our latest values for these parameters are:

$$D_I = 1 \times 10^3 \exp(-2.44 \text{ eV/kT}) \text{ cm}^2/\text{sec} \quad [6]$$

$$C_I^* = 2.5 \times 10^{19} \exp(-2.36 \text{ eV/kT}) \text{ cm}^{-3} \quad [7]$$

$$D_V = 1 \times 10^2 \exp(-2.92 \text{ eV/kT}) \text{ cm}^2/\text{sec} \quad [8]$$

$$C_V^* = 5 \times 10^{13} \exp(-1.08 \text{ eV/kT}) \text{ cm}^{-3} \quad [9]$$

We expect to adjust the coefficients in Eqs. 6-9 further upon completion of a series of planned experiments and subsequent analyses (22). Equations 6-9 are plotted in Fig. 4 along with the limiting point defect diffusivity and concentrations values recently proposed by Gösele, Plöbl, and Tan (23). Our present values for the native point defect parameters are in reasonable agreement with the proposed values. This agreement lends support to our assumption that the POCl_3 gettering process is removing vacancy-related defects from the p -type FZ crystals.

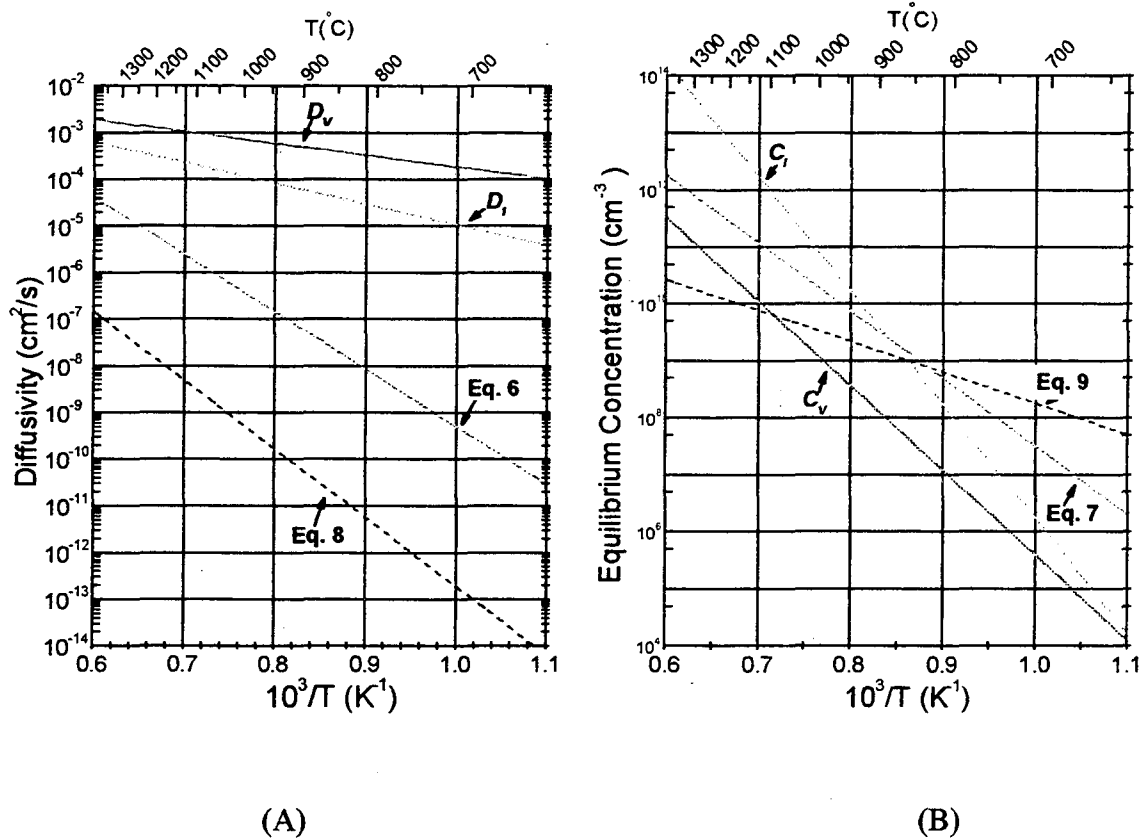


Fig. 4. (A) Theoretical diffusivities of silicon interstitials and vacancies as proposed by Gösele, Plöbl and Tan (labeled D_I and D_V) and those derived by modeling of our POCl_3 gettering process (labeled Eqs. 6 and 8). (B) Theoretical concentrations of silicon interstitials and vacancies as proposed by Gösele, Plöbl and Tan (labeled C_I and C_V) and those derived by modeling of our POCl_3 gettering process (labeled Eqs. 7 and 9).

Nitrogen-doped FZ silicon

By employing nitrogen doping to increase the nitrogen concentration in FZ crystals improved GOI test results have been confirmed by v. Ammon et. al. who proposed that complete suppression of D defect formation may be possible if the N_2 concentration is sufficiently high (25). It was therefore of interest to verify this proposal with Li^+ compensation. We obtained some p -type wafers from a nitrogen doped (N-doped) FZ crystal that shown improved GOI results over that of a normally doped crystal. The N-doped crystal characteristics are listed in the Table I along with those of a normally doped crystal.

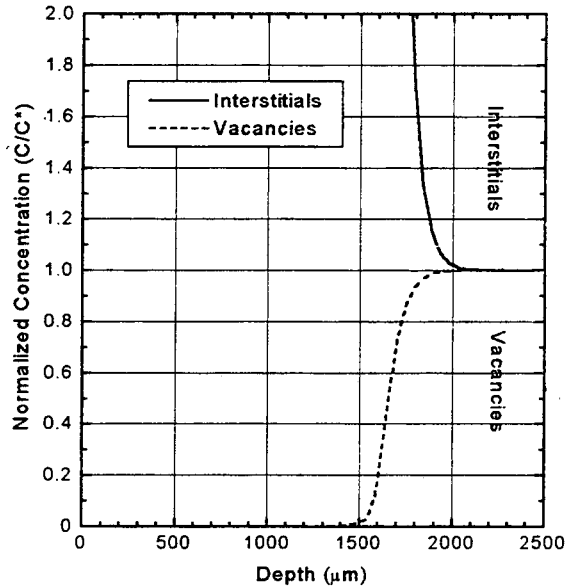


Fig. 5. The SUPREM-IV computer program simulation of the normalized vacancy and silicon interstitial concentrations for a 100 minute, 950 °C POCl_3 gettinger process that produces a ~ 1.5 mm wide region in which the vacancy concentration is significantly reduced. The coefficients used here are given in Eqs. 6-9.

Table I. *p*-Type, 111 Orientation, FZ Crystal Characteristics

Crystal	ρ (Ohm-cm)	τ (μ -sec)	Diam (mm)	Growth rate (mm/min)	[N] /cc	[C]/cc	[O]/cc
N-doped	1,250	900	103	2.5	3×10^{14}	5×10^{15}	2×10^{15}
Normal	$\sim 1,000$	$\sim 1,000$	100	2.5	1×10^{14}	3×10^{14}	2×10^{15}

For processing convenience, we cut 11 mm² square samples from across the center of 3 mm thick N-doped wafer. Using the fabrication procedure described earlier, each sample was Li^+ drifted to produce a *p-i-n* diode structure. We then sectioned these diodes and Cu stained them to determine the Li^+ drift depth profile. A plot of the Li^+ drift depth as a function of radial distance across the wafer is shown in figure 6. The plot (data points shown for only half of the wafer diameter) shows that the Li^+ drift depth is shallow in the central region of the wafer (~ 50 mm), at ~ 75 mm the Li^+ drift depth increases significantly and at ~ 80 mm the total 3 mm thickness of the wafer is Li^+ compensated. A similar plot is shown in Figure 7 for the Li^+ drift depth in a normally doped FZ crystal.

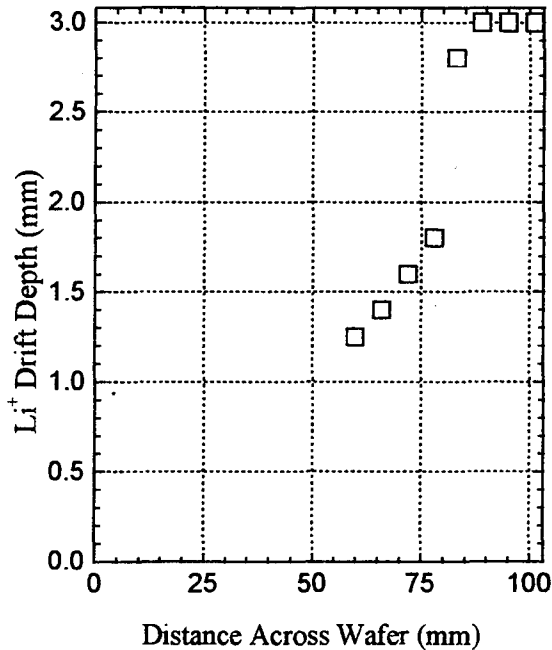


Figure 6. The Li^+ profile in a wafer from the N-doped crystal in Table 1. At the center the maximum drift depth, W_{max} , is ~ 1.2 mm.

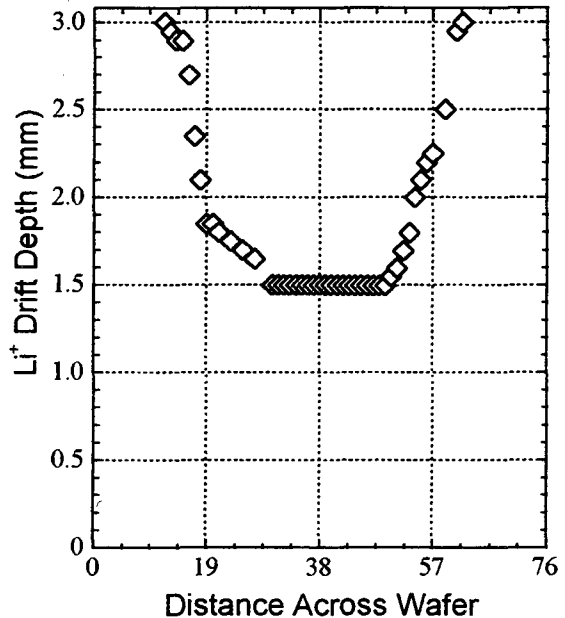


Figure 7. The Li^+ profile in a wafer from the normal crystal in Table 1. At the center the maximum drift depth, W_{max} , is ~ 1.5 mm.

The undrifted regions of both the N-doped and normal FZ Si crystals are similar (Figures 6 and 7) indicating that the N doping has not completely removed the D defects. Our results suggest that, although an improvement in the GOI test results has been observed, the D defect regions are still present (25). In other words, the concentration of D defects may have been reduced below the detection limits the GOI test, but not below that of the Li^+ compensation method.

VACANCY-RELATED DEFECT CONCENTRATION

The use of lithium in silicon crystal defect studies is normally associated with either the precipitation of lithium from a supersaturated concentration in the crystal or in the decoration of point defects for x-ray topography (26). Both of these applications require long (many hours), relatively high temperature (~ 450 °C) diffusions to disperse the lithium throughout the sample. Several groups have used the data from lithium precipitation along with Ham's theory of precipitation at point defects to arrive at an estimate of the point defect concentration (27). Both lithium precipitation studies and x-ray topography suffer from limitations in the measurements and in the interpretation of the data. Specifically, with precipitation studies the lithium precipitation rates are not constant and with the

topography analysis the lithium only appears to decorate A defects (28). Li^+ drift overcomes these difficulties.

In discussing Eqs. 2 and 3 we noted that if the Li^+ lifetime was short, there was a maximum depth to which the Li^+ could be drifted into the silicon wafer, i.e. W_{max} . Following the work of Guislain et.al., we can rewrite Eq. 3 as (29):

$$1/\tau = \mu_{Li}V/W_{max}^2, \quad [10]$$

which, following Ferman, we can then equate to Ham's estimate of the precipitation relaxation time (17):

$$1/\tau = 3D(\nu C_o)^{1/3}(4\pi N_p/3)^{2/3} F(e), \quad [11]$$

where, for our case, D is the Li^+ diffusivity, C_o is the Li^+ concentration, ν is the precipitate volume/solute atom, N_p is the vacancy-related defects/cm³, and $F(e)$ is the precipitates shape factor, which is ~ 1 for Li^+ precipitates. Combining Eqns. 12 and 13 and using the Einstein relationship, $D = (kT/q)\mu$, we arrive at an estimate of the vacancy-related defect concentration as:

$$N_p = (3/4\pi)(Vq/3kT(\nu C_o)^{1/3})^{3/2} (1/W_{max})^3, \text{ or} \quad [12]$$

$$N_p = (4.3 \times 10^9)(1/W_{max})^3/\text{cm}^3, \quad [13]$$

using Ferman's value for $\nu = 4 \times 10^{-23} \text{cm}^3$, and assuming a drifting voltage $V = 500$ Volts, $C_o = [Li^+] = [N_A] = 1 \times 10^{13}/\text{cm}^3$ ($\sim 1,400$ Ohm-cm, p -type), and the drifting temperature of 110°C . The values for N_p obtained with Eq. 13 are plotted in Figure 8.

In our examination of various p -type FZ Si crystals we have observed $0.1 \text{ cm} < W_{max} < \sim 2.0 \text{ cm}$ corresponding to $\sim 4.2 \times 10^{12}/\text{cm}^3 > N_p > 5.4 \times 10^8/\text{cm}^3$. This range of values is in reasonable agreement with those of Ferman as well as those of de Kock and Boonen who used a value of $\nu = 3.2 \times 10^{-22} \text{cm}^3$ in their calculations (26). Further, the Li^+ profiles in Fig. 6 (N-doped crystal) and Fig. 7 (normal crystal) have W_{max} values at the wafer center of 1.5 mm and 1.2 mm, respectively. These yield values for N_p at the wafer center in the "normal" crystal of $\sim 1.3 \times 10^{12}/\text{cm}^3$ and in the N-doped crystal of $\sim 2.5 \times 10^{12}/\text{cm}^3$. At the wafer periphery $N_p < 1.6 \times 10^{11}/\text{cm}^3$ for both crystals.

CONCLUSIONS

We have demonstrated the ability of Li^+ compensation to detect vacancy-related defects in p -type FZ silicon. In particular we have shown that Li^+ compensation indicates the presence of these defects even when GOI results would suggest otherwise.

Given the relatively high diffusivities of the silicon native point defects, any FZ crystal evaluation procedure that involves substantial thermal cycling, such as growing an oxide for the GOI test, may substantially alter the point defect distribution in the crystal.

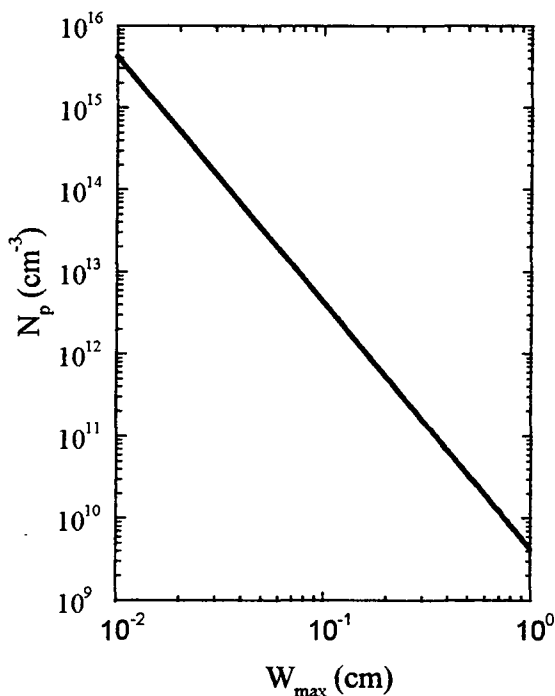


Figure 8. The estimated vacancy-related point defect density (N_p) as a function of the maximum drift depth (W_{max})

Li^+ drifting appears to offer a higher sensitivity to the presence of vacancy-related FZ crystal defects than the GOI test and does not require high-temperature processing. Further the straightforward measurement of the Li^+ compensation depth can yield an estimate of the vacancy-related defect concentration in *p*-type FZ silicon. Therefore, in the ongoing effort to model and control defects in FZ silicon, a renewed application of Li^+ compensation to FZ crystal evaluation appears to be now opportune.

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