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Dopant concentration imaging in crystalline silicon wafers by band-to-band photoluminescence

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In this work, we present two techniques for spatially resolved determination of the dopant density in silicon wafers. The first technique is based on measuring the formation rate of iron-acceptor pairs, which is monitored by band-to-band photoluminescence in low injection. This method provides absolute boron concentration images on p-type wafers, even if compensating dopants such as phosphorus are present, without reference to other techniques. The second technique is based on photoluminescence images of unpassivated wafers, where the excess carrier concentration is pinned by a high surface recombination rate. This rapid technique is applicable to either p- or n-type wafers, when the bulk carrier lifetime is much longer than the transit time to the surface. The relative sensitivities and advantages of the two techniques are discussed. © 2011 American Institute of Physics. [doi:10.1063/1.3664859]

I. INTRODUCTION

Accurate knowledge of the dopant concentration of silicon wafers is of considerable interest with the advent of solar-grade silicon materials for solar cell fabrication, which often contain multiple dopant species.¹ Conventionally, dark conductance measurements via four-point probe analysis or eddy current mapping are used for the determination of net dopant concentrations. The main drawback of these methods lies in their limited spatial resolution, as well as the slow nature of their point-by-point mapping. Several alternative techniques have been developed in recent years. Zierer *et al.*² have introduced a technique for the determination of the net doping concentration and mobility in compensated silicon using free carrier absorption (FCA), and Geilker *et al.*¹ have added the analysis of Fourier transform infrared spectroscopy (FTIR) data. This technique generates an average value of dopant density of the material. Mitchell and Trupke introduced an imaging method to determine bulk minority carrier lifetimes and dopant densities of silicon bricks using photoluminescence (PL) imaging technique,³ based on a two-filter method.⁴ PL imaging has found numerous useful applications in spatially resolved solar cell and wafer characterization and for inline quality control in silicon solar cell manufacturing^{5–9} over the last few years. In this paper, we investigate two new approaches for the characterization of variations of the dopant density in both p-type and n-type silicon material also based on photoluminescence imaging technique. The first method allows imaging of the total acceptor concentration in boron-doped p-type wafers by monitoring the iron-boron re-pairing rate using band-to-band photoluminescence. The second is based on measuring the PL image of silicon wafers that are strongly dominated by

surface recombination, in which case a nearly linear relationship between photoluminescence intensity and net dopant density exists. We will demonstrate that both can have a high relative sensitivity and generate high spatial resolution images.

II. EXPERIMENTAL METHODS

PL measurements in this work were made with a BT imaging LIS-R1 instrument where images of the sample are taken with 160 μm resolution. Excess carriers are generated by illumination with a steady-state 808 nm laser during the PL measurement. The band-to-band photoluminescence emitted as a result of radiative recombination from the sample is captured by a one megapixel silicon charge-coupled device camera through a filter that eliminates laser illumination reflected from the sample. The intensity of the laser can be adjusted from below 1 sun equivalent (approximately 0.1 W/cm^2) to an upper limit of 10 suns.

The samples used in this work were 0.88 $\Omega\text{ cm}$ B-doped float-zone grown silicon wafers. For the surface limited technique, the samples were imaged in their as-cut state, which creates a very high recombination rate at the surfaces. Measurements using the FeB method were made on well-passivated samples that contain sufficient traces of iron which either exists in natural form or was introduced through deliberate implantation. Fig. 1 shows the N_A image of a 10 cm diameter passivated 15 $\Omega\text{ cm}$ float zone (FZ) wafer derived using FeB method based on natural iron content.

A special sample was used to investigate a possible correlation between the interstitial iron concentration $[\text{Fe}_i]$ and the dopant density. This 232 μm thick sample was implanted with five doses of 70 keV Fe ions, with each implantation covering an area of $30 \times 7\text{ mm}^2$, and doses of $1.16 \times 10^{10}\text{ cm}^{-2}$, $2.32 \times 10^{10}\text{ cm}^{-2}$, $4.64 \times 10^{10}\text{ cm}^{-2}$, $1.39 \times 10^{11}\text{ cm}^{-2}$, and $4.64 \times 10^{11}\text{ cm}^{-2}$, yielding target volume concentrations of

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FIG. 1. Image of a whole 100 mm diameter 15 Ω cm FZ wafer in 160 μ m resolution measured using FeB pairing technique with 3.7×10^{15} photons $\text{cm}^{-2} \text{s}^{-1}$ laser flux intensity and a measurement time of 10 s for each data point. The natural iron concentration was $7 \times 10^{11} \text{cm}^{-3}$. PL intensity measured in the FeB dissociated state and that measured in the associated state differed by a factor of two.

$5 \times 10^{11} \text{cm}^{-3}$, $1 \times 10^{12} \text{cm}^{-3}$, $2 \times 10^{12} \text{cm}^{-3}$, $6 \times 10^{12} \text{cm}^{-3}$, and $2 \times 10^{13} \text{cm}^{-3}$, respectively. After further surface cleaning, the sample was annealed at 900 $^{\circ}\text{C}$ for 60 min in nitrogen to distribute the Fe evenly throughout the thickness of the sample. SiN films were then deposited by plasma-enhanced chemical-vapor deposition (PECVD) on both surfaces, to passivate them and allow accurate measurement of the bulk lifetime. The sample was measured under 1.4×10^{16} photons $\text{cm}^{-2} \text{s}^{-1}$ for a total exposure time of 10 s for dopant concentration determination and under a higher laser power of 4.9×10^{16} photons $\text{cm}^{-2} \text{s}^{-1}$ for a period of 10 s for an investigation on measurement of dopant concentration in a region exceeding true low injection conditions.

A second 206 μ m thick sample was processed for the application of the dopant measurement technique based on FeB pairing. After surface cleaning and etching, it was subjected to implantation of 70 keV Fe ions with a dose corresponding to a target volume concentration of $2.96 \times 10^{12} \text{cm}^{-3}$ in the central area of size $30 \times 29 \text{mm}^2$, and annealed and passivated as above. FeB dissociation was achieved by illuminating the sample with multiple flashes of white light of high intensity with a maximum peak of 400 suns. The sample was then measured using an incident laser photon flux of 1.03×10^{16} photons $\text{cm}^{-2} \text{s}^{-1}$ for an integration duration of 10 s per data point over the FeB re-pairing process. Given the upper limit of the sample wafer lifetime which is 4 μ s, measured using a quasi-steady-state photoconductance lifetime measurement (QSSPC), such an incident flux generated a maximum steady state excess carrier concentration Δn value of around $2 \times 10^{12} \text{cm}^{-3}$ which is well below the crossover point of the Fe_i and FeB characteristic lifetime signatures,^{10,11} and meets the low injection measurement requirement, as described below.

III. ACCEPTOR CONCENTRATION DETERMINATION VIA IRON-BORON PAIRING

A. Technique fundamentals

This technique is based on measuring the formation rate of iron boron pairs after they are dissociated by external

factors, such as optical or thermal perturbation.^{12–14} The time constant for the re-pairing reaction is dependent on the acceptor concentration N_A and is given by¹⁵

$$\tau_{\text{assoc}} = \frac{BT}{N_A} \exp\left(\frac{0.66}{kT}\right), \quad (1)$$

where the constant $B = 5.7 \times 10^5 \text{s/Kcm}^3$.¹⁶ The temperature T is in Kelvin, N_A is the acceptor concentration (cm^{-3}), Boltzmann's constant $k = 8.617 \times 10^{-5} \text{eV/K}$ and τ_{assoc} is in units of seconds.

As a change in the proportion of interstitial Fe present as FeB pairs has a pronounced effect on the carrier lifetime of a p-type silicon sample, the re-pairing rate of FeB can be directly measured by monitoring the carrier lifetime. As shown previously,¹⁷ low injection measurements of the band-to-band PL intensity provide a convenient way to monitor the re-pairing reaction, and can be used to determine the acceptor concentration via the equation above.

The τ_{assoc} value derived from low-injection PL data during FeB re-association can be processed either as a single area-averaged value, as done previously,¹⁷ which gives a high signal-to-noise ratio, or at a pixel-by-pixel level, resulting in a spatially resolved N_A image. This involves processing a series of PL images measured at intervals after the breaking of FeB pairs. N_A is then computed for each pixel via a linear regression approach, resulting in an image with resolution limited by the detector, which is 160 μ m in this work. Processing of a larger number of images can reduce uncertainty in the computation of the regression line of τ_{assoc} , hence leading to clearer contours and images. Also, the PL intensity in the FeB associated state should be more than three times higher than that in the dissociated state for accurate derivation of the dopant concentration. Finally, this technique based on FeB pairing is applicable only to p-type material, and allows the total acceptor concentration to be obtained, as distinct from the net doping.

Uncertainty in the average dopant concentration is contributed to by factors such as temperature variation, and fluctuations with time in the laser intensity. Measurements of the laser intensity taken every 2 min over a 40 min period revealed intensity variations of less than 7%. Correspondingly, the PL signal difference in each consecutive measurement data should be larger than the margin of flux intensity variations for accurate measurement. The total absolute uncertainty in the measured N_A values is estimated to be less than 10% for the images presented in this work. As the measurement is based on the rate of FeB pairing computed pixel by pixel, it is in principle immune to excitation flux inhomogeneity across the sample, sample surface variations, and bulk lifetime variations.

B. Measurement optimization

In principle, the FeB re-pairing rate, and hence, the resulting acceptor concentration, should be independent of the concentration of interstitial Fe. This was confirmed by applying the N_A imaging technique on the sample with graded Fe concentrations. Fig. 2 shows the PL image of a $16 \times 32 \text{mm}^2$ section of this sample measured with an

FIG. 2. (Color online) Image of Fe-implanted FZ sample showing five regions of different implant concentration.

incident flux of 1.4×10^{16} photons $\text{cm}^{-2}\text{s}^{-1}$ and a total acquisition time of 10 s, revealing the regions of different Fe concentration. Fig. 3 shows that the PL count rate decreases monotonically with the iron concentration. Fig. 3 also shows that the standard deviation in the PL count rate increases as a function of the iron concentration, reflecting the lower lifetimes and lower signal to noise ratio in the high iron concentration regions.

The Fe implanted sample was then measured using the FeB pairing technique for the determination of the dopant density. Measurement was performed using an incident photon flux of 1.4×10^{16} photons $\text{cm}^{-2}\text{s}^{-1}$ and total exposure time of 10 s per data point at 33 °C. The time resolved PL count rate obtained in the $16 \times 32 \text{ mm}^2$ section of a sample yielded a τ_{assoc} of 712 s from a mono-exponential fit, which corresponds to the average N_A density of $1.82 \times 10^{16} \text{ cm}^{-3}$, showing good agreement with dark conductance measurements, which is $1.7 \times 10^{16} \text{ cm}^{-3}$. An image generated by pixel-by-pixel calculation of the dopant density of the section of $16 \times 32 \text{ mm}^2$ of the graded Fe-implanted sample is shown in Fig. 4.

FIG. 3. (Color online) PL count rate and standard deviation as a function of the iron concentration.

FIG. 4. N_A density image of a $16 \times 32 \text{ mm}^2$ section of a graded Fe-implanted silicon sample showing a consistent mean value of $1.8 \times 10^{16} \text{ cm}^{-3}$ across the sample with higher noise level in regions of higher Fe_i concentration (left side).

A plot profile of N_A density along the y direction of the sample is shown in Fig. 5(a). Consistent dopant values of around $1.8 \times 10^{16} \text{ cm}^{-3}$ are observed across the sample irrespective of the large variation in iron concentration. This confirms the expected independence of the formation time constant, and thereby, the resulting acceptor concentration on the Fe concentration. A higher noise level in the N_A density value was observed in the regions implanted with the highest Fe doses, reflecting the lower signal-to-noise ratio, as caused by the much lower lifetimes in those regions. The signal-to-noise ratio can be improved by increasing the illumination intensity, or by applying binning in the image acquisition, which will result in a reduction of the image resolution. However, care should be taken in setting the illumination intensity, to ensure that the measurement will not exceed the true low injection conditions required by the technique.¹⁷ This is demonstrated in Fig. 5(b), which shows a cross section of the generated N_A density profile measured with a higher incident photon flux of 4.9×10^{16} photons $\text{cm}^{-2}\text{s}^{-1}$ for a duration of 10 s. Deviations in the N_A density

FIG. 5. (Color online) Cross sections of N_A densities viewed from y direction: (a) *Top*. Consistency in the derived N_A concentration values across five $[\text{Fe}]$ regions (with higher $[\text{Fe}]$ on the left side) confirms independence of measured N_A on $[\text{Fe}]$ concentration. Correlation between noise level and $[\text{Fe}]$ is also evident. (b) *Below*. Deviation in N_A concentration across the sample was observed when measurement was performed in regions exceeding true low injection conditions.

in regions of lower Fe concentration due to measurement regions lying nearer the crossover point^{5,17} can be seen. Regions of higher Fe concentration in Fig. 5(b), on the other hand, actually show improved signal-to-noise ratios, around a consistent average N_A density value, as a result of the increased illumination intensity. Due to its wide range of excess carrier densities resulting from the deliberately large range of implanted Fe concentrations, the optimization process in this graded Fe sample is rather strongly constrained. In practice, most samples will exhibit a much smaller range of Fe concentrations, allowing optimal measurement conditions to be established more easily and with greater tolerance. Our experiments have also shown that accurate doping imaging using iron-acceptor pairing is more difficult with gallium or indium doped silicon, due to the lower cross-over points compared to boron-doped silicon,¹⁸ resulting in measurements being performed in the region with lower signal to noise ratios.

C. Demonstration of the FeB pairing method

Fig. 6 shows the N_A image of a $30 \times 29 \text{ mm}^2$ section of the Fe-implanted $0.88 \Omega \text{ cm}$ boron-doped FZ sample generated via the FeB pairing technique (the PL intensity in the FeB associated state was 4.5 times higher than that in the dissociated state). Multiple ring-like structures of the dopant concentration are evident in this quarter wafer. Position (180, 0) is located near the centre of the ingot; hence, the ringlike structures are radial variations extending from the ingot centre. A mean dopant density of $1.77 \times 10^{16} \text{ cm}^{-3}$ with a standard deviation of 6.8% is obtained from this measurement. This measurement technique generates an absolute N_A density, which requires no further calibration.

IV. DOPANT CONCENTRATION DETERMINATION VIA SURFACE LIMITED PL MEASUREMENTS

A. Technique fundamentals

Band to band recombination is proportional to the product of the concentrations of electrons and holes in the material. In low injection, the PL intensity is proportional to both the net doping, equal to the acceptor concentration N_A in

FIG. 6. (Color online) N_A concentration image of a $30 \times 29 \text{ mm}^2$ of a $0.88 \Omega \text{ cm}$ FZ wafer measured using FeB pairing technique shown in $160 \mu\text{m}$ resolution revealing partial circular ringlike structures.

non-compensated p-type silicon, and to the average excess carrier density Δn . Generally, the latter can vary widely from wafer to wafer and even within a given wafer, due to variations in effective minority carrier lifetime. However, for samples with sufficiently high bulk lifetimes (i.e., bulk lifetime larger than the transit time by at least two orders of magnitude will allow N_A variations of less than 5% to be distinguished), and non-passivated surfaces that cause instant recombination for any carriers that reach them, the carrier density profile within the wafer can be easily determined to a good degree of accuracy. In other words, the effective lifetime τ_{eff} of such wafers is approximately equal to the transit time τ_{transit} , defined as the average time required for generated carriers to diffuse to a surface. The average carrier density Δn_{av} is then equal to $G\tau_{\text{transit}}$, where G is the generation rate. For uniformly absorbed infrared light under steady-state conditions, the transit time is given by¹⁹

$$\tau_{\text{transit}} = \frac{W^2}{12D_n}, \quad (2)$$

where W is the sample thickness, assumed to be much lower than the carrier diffusion length and D_n the minority carrier (electron) diffusivity. The PL intensity will then be proportional to $N_A GW^2/12D_n$. Since the electron diffusivity D_n is only weakly dependent on the doping concentration, the PL intensity will be approximately linear in N_A . This provides a method to image variations in the net doping in surface-limited silicon wafers. Expressed alternatively, by pinning the value of Δn , courtesy of the lifetime being surface limited, any variations detected in the PL intensity are almost entirely attributable to variations in N_A concentration. A prerequisite for this method to work is thus a sufficiently high bulk lifetime across the wafer.

In practice, PL images are not performed with perfectly uniform photogeneration. The imaging system used in this work uses 808 nm illumination. Using the simulation package QSSModel (Ref. 20) and applying Klaassen's mobility model to determine the electron diffusivity,²¹ the transit time can be modeled for this illumination wavelength and for a range of doping levels and wafers thicknesses. Fig. 7 shows that this transit time is only weakly dependent on the dopant concentration, due to the dependence of the carrier

FIG. 7. (Color online) Relationship between transit time and dopant concentration from QSSModel in low injection, showing weak dependency.

FIG. 8. (Color online) Relationship between detected PL intensity and dopant concentration, showing an almost linear relationship. The lines represent fits of the equation $PL = A \cdot N_A^{1.1}$.

diffusivity on the doping. It is possible to model the intensity of the detected PL emission from the carrier profile established under the 808 nm illumination in surface-limited conditions.²⁰ Fig. 8 gives the results as a function of doping, for three different wafer thicknesses. The graphs show that the relationship between PL intensity and dopant concentration is almost linear. The slightly super-linear dependence (the line fits in Fig. 8 are actually exponential functions with an exponent of 1.1) arises again from the mild dependence of the transit time, via the diffusivity, on the doping. A rigorous approach can account for this slight non-linearity,²² but for the purposes, here, it is sufficient to assume that changes in the PL intensity are caused only by variations in N_A concentration, an assumption which will cause errors only in the range of 10% or less.

The carrier lifetime measured using the QSSPC technique²³ on the test wafer for this work in the as-cut state was less than 1 μ s, but the bulk lifetime was confirmed to be more than 1 ms, as verified by a lifetime measurement performed on a sister wafer after surface passivation with PECVD SiN films. The vast difference between the bulk life-

FIG. 9. (Color online) N_A concentration image of a $30 \times 29 \text{ mm}^2$ section of a $0.88 \text{ } \Omega \text{ cm}$ FZ sister wafer measured using surface limited method shown in $160 \text{ } \mu\text{m}$ resolution revealing radial circular structures extending from center of ingot around position $x = 180, y = 0$.

FIG. 10. (Color online) Dopant density profiles along scan line indicated in Figs. 6 and 9 obtained with the two different techniques.

time and effective lifetime measured on the as-cut sample confirms that the latter is completely surface limited.

B. Demonstration of the surface limited method

Fig. 9 shows the N_A density image of a $30 \times 29 \text{ mm}^2$ section from the same position as the test sample of an as-cut $0.88 \text{ } \Omega \text{ cm}$ boron-doped FZ sister wafer measured with an illumination intensity of $1.3 \times 10^{17} \text{ photons cm}^{-2} \text{ s}^{-1}$ for an acquisition time of 10 s. The N_A values shown were calibrated using an average N_A density of $1.75 \times 10^{16} \text{ cm}^{-3}$, obtained from four point probe measurements. Higher quality images with improved signal-to-noise ratios are yielded in these images due to higher illumination intensity used in the measurement. Circular doping structures extending from the origin can be clearly seen. A direct comparison of dopant density profiles along the x-axis scan line of Fig. 6 (FeB technique) and Fig. 9 (surface limited technique) is shown in Fig. 10, reflecting qualitative similarity, with some differences in the details.

The ringlike variation of doping concentrations in float zone determined in this work has in fact been observed before, for example, by Janus, who reported macroscale and microscale fluctuations in n-type float zone silicon samples, where they were measured by a $100 \text{ } \mu\text{m}$ step-length spreading resistance technique,²⁴ and more recently, by Veirman *et al.* who observed ringlike inhomogeneities in compensated Czochralski silicon.²⁵

As float zone silicon was used in this experiment, the ringlike structures observed are unlikely to result from lifetime variations related to oxygen induced defects.²⁶ The fact that these variations are not bulk lifetime-related is confirmed as follows. First, the acquisition of the dopant density based on the FeB repairing method is solely derived from the rate of the FeB re-association and hence is independent of the sample lifetime. Second, the doping ringlike structures were not present in images of these samples after they were surface passivated in which only a nearly uniform lifetime distribution was observed, despite a much greater effective lifetime and PL count rate. The lack of correlation in these images measured before and after passivation rules out the

TABLE I. Technique measurement requirements comparison.

	FeB pairing technique	Surface limited technique
Sample preparation	Requires surface passivation, and Fe implantation if insufficient natural Fe	No pre-processing, as-cut surfaces ideal
Image acquisition	Multiple images required over many minutes	Single image acquisition
Calibration	Does not require separate calibration	Requires separate calibration
Carrier injection, laser intensity	True low injection, low intensity	Low injection, high intensity
Accuracy	Unaffected by surface condition, bulk lifetime or illumination intensity homogeneity	Sensitive to illumination intensity homogeneity and very low bulk lifetimes
Relative sensitivity	Low	High
Application	Absolute acceptor density, B-doped p-type only (also compensated)	Net doping density, any p- or n-type dopants

possibility that the PL variations acquired in the surface limited condition are due to bulk lifetime variations.

V. CONCLUSIONS

In this paper, we have demonstrated and compared two methods for imaging dopant densities in silicon wafers by band-to-band photoluminescence. Table I summarizes their respective advantages and disadvantages. The first technique determines the doping concentration in B-doped p-type wafers by monitoring the rate of FeB pairing, with no requirement for a separate calibration. Since the analysis relies on relative changes in the PL signal as a function of time, there is no need for absolute PL measurements. It has the capacity to measure the absolute acceptor concentration, with possible application to compensated materials. It may offer the opportunity to determine images of both n- and p-type dopants in compensated materials, when used in combination with the surface limited technique. It may also be used for absolute calibration of relative values for the doping concentration that are obtained from the surface limited PL method in cases where the Fe concentration is sufficiently high to allow a reliable measurement of the FeB formation time constant but sufficiently low to yield surface limited lifetime. Another advantage of the FeB technique is that it is insensitive to artifacts introduced by variations in sample thickness, bulk lifetime or optical conditions of the surfaces, or inhomogeneity of the incident illumination.

The second method determines the net doping concentration by measuring photoluminescence from a sample in surface limited conditions. It is a convenient and fast technique, applicable to both p- and n-type silicon and only requires the acquisition of a single image. It is not subject to true low injection limitations as required by the FeB pairing technique for N_A derivation and so can obtain much higher signal to noise ratios, which results in high resolution images. The key drawback of this method, however, is that it is a relative measurement which requires an independent calibration in order to make it quantitative. Another limitation is also found for highly doped materials due to the effect of Auger limited lifetime. For resistivities $< \sim 0.05 \Omega \text{ cm}$, the reduction in effective lifetime starts dominating the PL response. Compared to the results above this causes the opposite variation of PL signal with increasing doping density, i.e., a reducing PL count with increasing doping density for

resistivities $< 0.01 \Omega \text{ cm}$.²⁷ While applicable to silicon wafers in microelectronic applications, this complication is largely irrelevant for PV applications, where higher resistivities are generally used.

In this work, both FeB pairing and surface limited techniques show the existence of a circular doping structure within float zone silicon wafers, confirming previous studies. The high resolution and precision of the images obtained demonstrates the potential of the two techniques in characterizing wafer substrate quality and enhancing understanding of silicon crystal growth. The surface limited method is in general the preferred technique for qualitative dopant imaging, due to its fast and simple implementation. However, it should also be noted that this technique strongly relies on uniform surface properties and thickness. Variations in these quantities can be difficult to separate, which constitutes the accuracy limits of this technique when applied in practice.

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