A. Haarahiltunen, H. Väinölä, M. Yli-Koski, E. Saarnilehto, and J. Sinkkonen, Detection of iron contamination in internally gettered p-type silicon wafers by lifetime measurements, The Electrochemical Society Proceedings Vol. 05, High purity silicon VIII, Editors: C. L. Claeys, M. Watanabe, R. Falster and P. Stallhofer, p 135-145 (2004).

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DETECTION OF IRON CONTAMINATION IN INTERNALLY GETTERED P-TYPE SILICON WAFERS BY LIFETIME MEASUREMENTS

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We have studied the applicability of standard lifetime methods (SPV, μ–PCD) to detect iron in internally gettered p-type silicon wafers. Conventional high - low - high anneals were performed to produce a series of wafers with varying denuded zone (DZ) width and oxygen precipitate density. The wafers were intentionally iron contaminated to a level of about 1-2E13 cm^{-3}. Different kinds of gettering treatments were applied to obtain a wide range of dissolved iron concentrations. The results indicate that the iron concentration down to 1E11 cm^3 can be quantitatively measured in internally gettered wafers by lifetime methods. In internally gettered wafers μ-PCD has lower detection limit for iron than SPV but in both cases the detection limit depends on the internal gettering process. The reference measurements were done by DLTS.

INTRODUCTION

Iron is one of the most common transition metal contaminant in silicon wafer. It diffuses relatively fast and it is a strong recombination center, thus, it has deleterious effects on the device performance even when present in small concentrations. In internal gettering (IG) iron is captured by oxide precipitates and related defects which are formed conventionally in the bulk of wafer by a three-step high - low - high anneal (1).

There are several well-known detection methods for iron, such as standard lifetime measurements techniques, Surface Photo Voltage (SPV) and microwave detected PhotoConductive Decay (μ-PCD) (2,3) and deep level transient spectroscopy DLTS (4). SPV and μ-PCD are indirect measurements: the interstitial iron (Fe_i) concentration is determined from the change in the diffusion length or the lifetime after dissociating iron-boron (FeB) pairs. DLTS can be considered as a direct measurement as it measures the trap densities in the band gap, which can be identified to be either interstitial iron or iron-boron pairs.

DLTS is often used when characterizing the internal gettering efficiency of iron (5-7). DLTS has high sensitivity for iron and oxide precipitates have no effects on the measurements. However, DLTS is hardly suitable for large area detection i.e. wafer mapping, as an additional contact preparation is needed.

SPV and μ-PCD measurements do not need any additional sample preparation and wafers can be mapped routinely. This makes it possible to detect non-homogeneities in the gettering efficiency. Previously, SPV and μ-PCD have been used to study the internal gettering efficiency (8-10), however, in theses studies SPV and μ-PCD have not been considered as quantitative measurements. In Ref. (9) the IG processes include 40 min

nucleation anneal in 800 ^{o}C and 1-2.5 h growth anneal in 1000 ^{o}C. This means that in Ref. (9) the DZ is wide and bulk defect density is low, thus the SPV measurement can be expected to give a correct iron concentration. In Ref. (8) the nucleation and growth times of IG processes were long and the DZ width varied between 30 to 70 μm so the validity of homogeneous approximation is questionable. Therefore, the measured iron concentrations were considered as effective values.

In this work we have studied the effect of the high-low-high process on iron detection by SPV and μ-PCD. After the high-low-high annealing wafers were iron contaminated to the level of 1-2E13 cm^{-3}. Different kinds of gettering treatments were applied to obtain a wide range of dissolved iron concentrations. The reference measurements were done by DLTS.

EXPERIMENTAL

High - Low- High anneals

Table I High - low - high processes and oxygen losses during process. In all processes the growth annealing was 16 h at 1100 ^{o}C.

Sample	1. High	Low	Oxygen loss
	^{o}C ; h	^{o}C ; h	Ppma
B1	---	750 ; 2	8.5
B2	---	750 ; 2	8.7
A1	---	750 ; 2	5.2
A2	---	750 ; 2	5.5
B3	1150 ; 4	650 ; 6	8.2
B4	1150 ; 4	650 ; 6	7.7
B5	1150 ; 4	650 ; 6	8.0
A3	1150 ; 4	650 ; 6	4.3
A4	1150 ; 4	650 ; 6	4.9
A5	1150 ; 4	650 ; 6	4.8
B6	1150 ; 4	550 ; 6	9.1
B7	1150 ; 4	550 ; 6	9.4
B8	1150 ; 4	550 ; 6	9.5
B9	1150 ; 4	550 ; 6	9.1
A6	1150 ; 4	550 ; 6	7.2
A7	1150 ; 4	550 ; 6	6.7
A8	1150 ; 4	550 ; 6	5.9
A 9	1150 ; 4	550 ; 6	8.1
B10	1150 ; 16.6	550 ; 6	9.0
B11	---	650 ; 6	---
A10	1150 ; 16.6	550 ; 6	6.5

The silicon wafers used in experiments were boron doped and their resistivities were 20-50 Ωcm. The oxygen content (11) in A-series and B-series were 14 ppma and 16 ppma, respectively. Thickness of wafers was 525 μm in A-samples and 625 μm in B-samples. The different high - low - high and low-high processes (Table I) were applied in order to study the effect of the denuded zone (DZ) width and the precipitation site density on the detection limit and detection accuracy of iron by lifetime methods. In all processes the growth annealing was 16 h at 1100 ^{o}C. As-grown samples (C-samples) were included in the gettering processes for reference use.

Based on the results in Ref. (12) the DZ width of the wafers can be estimated to be less than 30 μm in low-high processes, and about 30-40 μm in processes where the first high anneal is 4 hours and 60-80 μm in processes where the first high anneal is 16 hours 40 minutes. Oxygen loss due to the out-diffusion and precipitation was measured after each process using FTIR. From the oxygen losses (Table I) it can be seen that oxygen precipitates have been successfully formed.

Iron contamination and gettering

The wafers were contaminated in a SC1 (NH_4OH:H_2O_2:H_2O 1:1:5) solution with added iron (30 ppb) impurities. Iron was in-diffused for 50 min at 850 °C where the iron contamination level is determined by the solubility. The surface contamination was removed by etching wafers in a H_2O:HF:H_2O_2 (24:1:1) solution and by cleaning in a sequence of SC1, SC2 (HCl:H_2O_2:H_2O 1:1:5) and HF-dip (H_2O:HF 25:1). After surface cleaning the wafers were dry-oxidized for 20 min to grow 10 nm thick oxide and annealed for 15 min in nitrogen ambient at 900 °C. 35 min anneal at 900 °C allows the iron contamination to spread uniformly through the wafer so the initial condition before cooling is known. In one set of test samples from C-series 950 ^{o}C oxidation temperature was used instead of 900 ^{o}C. The final contamination level was same, which indicates that the surface iron contamination was efficiently removed by cleaning. The background contamination level was measured to be in the order of 7E10 cm^{-3}.

The gettering was done by slow cooling from 900 ^{o}C or by isothermal annealing. The cooling after anneal at 900 °C was performed in five different ways. In each case the wafers were cooled from 900 °C to 850 °C at a rate of 4°C /min. From 850 °C the wafers were cooled at rate of 2 °C /min to 800,750, 700, 650 or 600 °C. The wafers were then pulled out of the furnace at a velocity of 30 cm/min. The contamination level (1-2E13 cm^{-3}) in samples pulled out at 800 ^{o}C is the initial contamination level as our previous results (8) indicate that no gettering occurs at high temperatures.

The enhanced gettering technique (10) is used to study detection of a low iron concentration, as the gettering during cooling is not very efficient (8). The enhanced gettering was done by annealing wafers at 700 ^{o}C or at 600 ^{o}C.

Iron detection

The remaining interstitial iron concentration was measured by SPV and μ-PCD from the front side of the wafer. By using homogeneous wafer approximation the iron concentrations can be calculated with aid of (2,3)

$$[\mathrm{Fe}]_{SPV} = 7.8\mathrm{E}15 \cdot \left(\frac{1}{L_2^2} - \frac{1}{L_1^2} \right) \quad \mathrm{cm}^{-3} \qquad (1)$$

$$[\mathrm{Fe}]_{\mathrm{PCD}} = 3.4\mathrm{E}13 \cdot \left(\frac{1}{\tau_1} - \frac{1}{\tau_2} \right) \quad \mathrm{cm}^{-3} \qquad (2)$$

where L (μm) is a diffusion length and τ (μs) is a lifetime. Index one indicates lifetime (diffusion length) when iron is paired with boron and index two indicates lifetime (diffusion length) when iron is in the interstitial form. The dissociation of FeB-pairs was done by using a high-intensity flashlight (2). In the measurements the back sides of the wafers were passivated by negative corona charge (-1.27 $\mu C/cm^2$) and the front sides of the wafers were charged positively (0.64 $\mu C/cm^2$). The front side of wafer was recharged (0.25 $\mu C/cm^2$) after the wafer was exposed by flashlight. Equations 1 and 2 are not generally valid in IG-wafers which have a denuded zone, *i.e.*, the inverse of measured effective lifetime is not a sum of inverses of lifetimes of different regions (13).

In the case of SPV the measurement signal is mostly coming from DZ, thus Equation 1 gives the correct results if the iron concentration is so high that both diffusion lengths are much smaller than the DZ-width. We can define the background diffusion length to be the measurement result without iron contamination. The background diffusion length is determined by penetration depths of lasers, DZ-width and bulk diffusion length (14). In the case of low iron contamination, in our case [Fe]<1-2E13 cm^{-3}, the measured diffusion length before FeB-pairs dissociation is nearly equal to the background diffusion length. The iron concentration can also be calculated by using the diffusion length when only Fe_i reduces the charge carrier lifetime. This enables us to estimate the detection limit of SPV

$$[\mathrm{Fe}]_{\mathrm{SPVlimit}} \approx 7.1\mathrm{E}15 \cdot \left(\frac{1}{L_b^2} \right) \quad \mathrm{cm}^{-3} \qquad (3)$$

where L_b (μm) is the background diffusion length. The prefactor is calculated using an electron diffusion constant of 35 cm^2s^{-1}, electron capture coefficient of 5.5E-7 cm^3s^{-1} (15) and the FeB-pairs dissociation efficiency of 90 % (2). Figure 1 shows the estimated SPV detection limit with different background diffusion lengths. The DZ-width can be estimated from the background diffusion length. Typically in our case DZ-width is about 1-3 times the background diffusion length. The iron concentration calculated from SPV results is lower than it is in reality as the fixed value of the background diffusion.

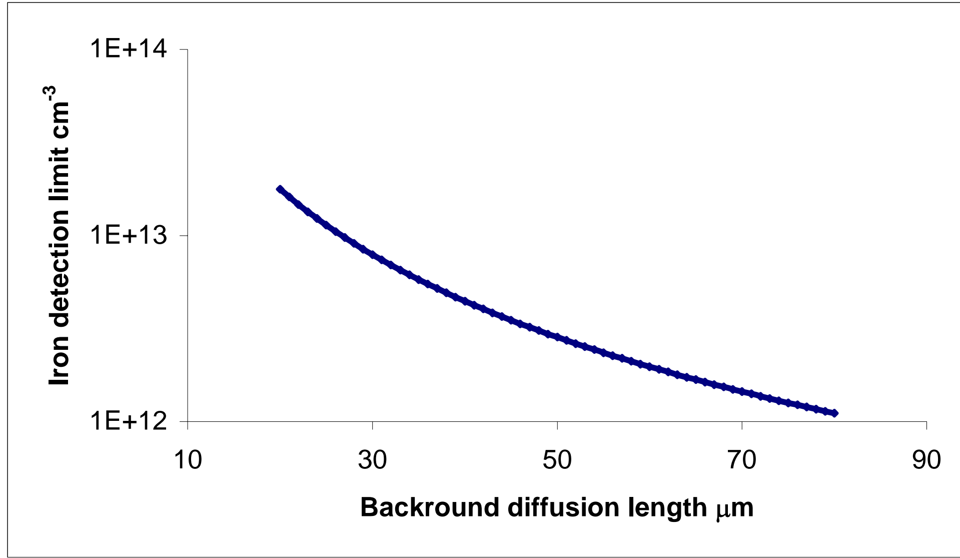

Figure 1. The detection limit of SPV.

In the case of μ-PCD measurement at low injection level the inverse of the effective measured lifetime is (16)

$$\frac{1}{\tau_{eff}} = \frac{1}{\tau_{DZ}} + \frac{1}{\frac{d^2}{\pi^2 D} + \frac{d}{s - B}} \tag{4}$$

where τ_{DZ} is the lifetime in DZ, d is the DZ-width, D is the charge carrier diffusion constant, s is the frontsurface recombination velocity and B is the effective interface recombination velocity. Equation 2 gives the correct result if it is assumed that the second term on the right in Equation 4 is constant. This assumption means that FeB-pairs dissociation does not affect B. As a consequence the second term can be considered as the background lifetime which sets the detection limit: the change in measured lifetimes caused by FeB-pairs dissociation must be more significant than the random errors in the measurement. Figure 2 shows the detection limit of μ-PCD measurement with different relative measurement errors.

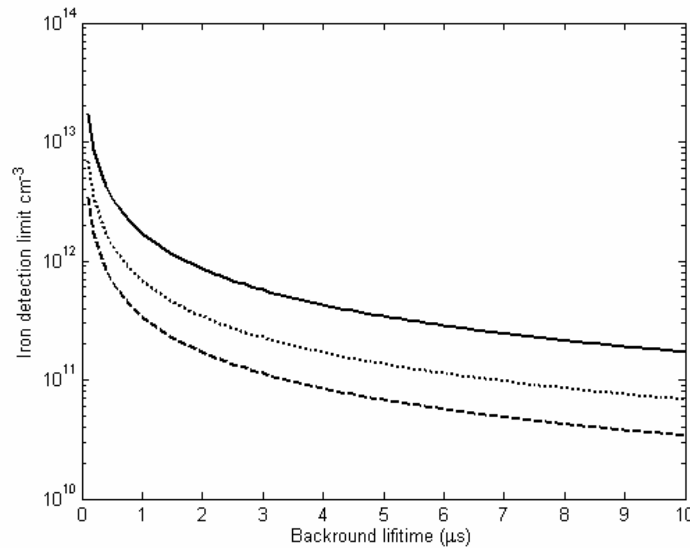

Figure 2. Iron detection limit with different background lifetimes, when five percent (solid line), 2% (dotted line) and 1 % (dashed line) measurement error is assumed. In calculation we have assumed that before dissociation of FeB-pairs the lifetime in DZ is limited by FeB-pairs and after dissociation of FeB-pairs the lifetime in DZ is limited by Fe_i.

The lifetime measured by μ-PCD is injection dependent, thus it must be somehow confirmed that the lifetime values before and after FeB-pairs dissociation is measured at the same injection level. In our case the approximately same injection level is attained as the beginning of the transient, time from generation pulse to about two times the lifetime, is not used to determine the lifetime. This enables the excess carrier decay to reach nearly the same level before each measurement.

The reference iron concentration near the wafer surface was measured by DLTS (4). Before DLTS-measurements pieces about a size of 1×1 cm were cleaved from the wafers. Dry-oxide was removed from the pieces by a diluted 1:10 HF solution and aluminium Schottky-contacts of about 100 nm thick were evaporated onto the top of the pieces. An indium/gallium alloy was used to make ohmic contacts on the back sides of the pieces.

RESULTS AND DISCUSSION

Samples gettered by slow cooling

The C-samples were used to check the correctness of Equations 1 and 2 in case of homogeneous wafers. It was observed that Equation 2 gives the same iron concentration as DLTS. In the case of SPV measurements the iron concentrations calculated from Equation 1 were about 2.2 times higher than those measured by DLTS, thus the conversion factor in Equation 1 must be divided by 2.2. It was also noticed in Ref. (2) that iron concentration determined by SPV is higher than the one measured by DLTS.

Table II. The average measurement results of SPV and μ-PCD after slowly cooling samples to pull out temperature. Sample C3 was dry-oxidized at 950 °C. The star marks the samples that were also measured by DLTS.

Sample	L_1 μm	L_2 μm	Fe cm^{-3}	τ_1 μs	τ_2 μs	Fe cm^{-3}	Pull out °C
*B1	24.83	12.44	1.72E+13	1.056	2.411	1.81E+13	700
***B2**	**23.31**	**14.48**	1.04E+13	**1.507**	**2.851**	1.06E+13	**600**
*A1	41.33	16.63	1.07E+13	1.835	6.011	1.29E+13	700
***A2**	**38.58**	**16.44**	1.07E+13	**1.804**	**5.617**	1.28E+13	**600**
*B3	34.18	13.76	1.57E+13	1.035	3.149	2.21E+13	800
*B4	34.22	14.95	1.28E+13	1.055	3.451	2.24E+13	700
***B5**	**36.87**	**18.95**	7.26E+12	**1.698**	**4.545**	1.25E+13	**600**
*A3	42.62	15.29	1.32E+13	1.39	4.273	1.65E+13	800
*A4	42.88	14.06	1.60E+13	1.301	4.728	1.89E+13	700
***A5**	**45.36**	**18.01**	9.21E+12	**1.888**	**6.422**	1.27E+13	**600**
B6	32.05	13.2	1.71E+13	0.811	1.538	1.98E+13	800
B7	16.76	11.7	1.33E+13	0.665	1.293	2.48E+13	750
B8	17.4	12.4	1.13E+13	0.67	1.305	2.47E+13	700
B9	18.85	12.2	1.38E+13	0.685	1.353	2.45E+13	650
A6	22.29	13.8	1.14E+13	0.918	2.013	2.01E+13	800
A7	26.84	14.5	1.19E+13	1.064	3.036	2.08E+13	750
A8	27.42	13.7	1.42E+13	1.103	3.46	2.10E+13	700
A9	21.67	13.8	1.11E+13	0.791	1.71	2.31E+13	650
*C1	58.85	14.47	1.59E+13	1.381	3.908	1.59E+13	800
*C2	56.4	13.77	1.76E+13	1.38	4.697	1.74E+13	600
*C3	56.83	14.13	1.67E+13	1.365	4.946	1.80E+13	800
*C4	59.25	15.4	1.40E+13	1.518	7.924	1.81E+13	800
*C5	53.11	15.3	1.39E+13	1.4	7.22	1.96E+13	650

In the Table II SPV and μ-PCD results are shown. The results confirm our previous observation (8) that during cooling gettering occurs at low temperature. The result indicates that irrespective of the IG-process the gettering occurs only after cooling to 600 °C and in every case the gettering efficiency is between 30 to 50 %. The inefficient gettering can be explained by the fact that before cooling all the iron is dissolved and there are not any iron nuclei, which could grow during cooling. Formation of iron nuclei, even in case of heterogeneous nucleation, seems to need a high degree of supersaturation. This phenomena is also observed in Ref. (10). Only in sample A2 gettering did not occur, which can be explained by a short nucleation time of oxygen and by lower initial oxygen concentration than in B2.

It can be observed from Table II that in SPV-measurement the approximation of the constant L_1 does not hold exactly in similarly processed samples, however the change is quite small (e.g. B3-5). The detection limit of all samples estimated from Equation (3) is lower than 8E12, when a background diffusion length of 20 μm is used (values in Figure 1 are divided by 2.2). In practice this limit is also affected by the minimum significant change in the diffusion length which in our case is about 2 μm. The diffusion length after FeB-pairs breaking, L_2, is 14±2 μm, except in wafers where gettering occurred. This only

states the fact that the diffusion length in DZ (determined by Fe_i) is so low that bulk recombination has only a minor effect on the measurement result.

The results from the μ-PCD indicate that the lifetimes are also very sensitive to oxygen loss and there is scattering in lifetime values even in the same process, e.g. the samples A6-9. μ-PCD detection limit can be estimated from Figure 2 (with the aid of the 2 % line) to be lower than 6E11 cm^{-3} when a 1.3 μs background lifetime is assumed. This is about a decade lower than detection limit of SPV. In practice the background lifetime value can be estimated to be the measured lifetime after dissociation of FeB-pairs.

Samples gettered by isothermal annealing

To obtain lower iron concentrations samples A/B6, A/B9, C4 and C5 were annealed 1 hour at 600 °C. The samples, which were cooled to 800 and 650 °C, were chosen so that any possible effect of the previous cooling could be observed. Samples A10 and B10 were included in the 600 °C anneal as they have longer DZ, which should decrease the detection limit. Additionally samples B7 and B11 were annealed at 700 °C for 1 or 2 hours, respectively. The measurement results are collected in Table III.

Table III. The average measurement results by SPV and μ-PCD after isothermal annealing, temperature and time is indicated in the right hand side column. The star marks the samples that were also measured by DLTS

Sample	L_1 μm	L_2 μm	Fe cm^{-3}	τ_1 μs	τ_2 μs	Fe cm^{-3}	°C ; h
B6	17.7	17.41	-	1.772	1.779	-	600 ; 1
B9	17.27	17.1	-	1.616	1.626	-	600 ; 1
A6	21.03	20.27	-	2.307	2.351	-	600 ; 1
A9	19.94	19.45	-	1.912	1.941	-	600 ; 1
C5	54.06	19.57	1.23E+13	2.424	10.550	1.48E+13	600 ; 1
C4	55.99	16.27	8.04E+12	1.850	9.398	1.08E+13	600 ; 1
*A10	37.89	37.23	-	4.286	4.405	1.54E+11	600 ; 1
*B10	29.54	29.41	-	3.020	3.062	2.14E+11	600 ; 1
*B11	-	-	-	1.741	1.705	-	700 ; 2
*B7	-	-	-	2.206	2.390	1.19E+12	700 ; 1

The results reveal that the iron concentration drops so low in IG-samples that it cannot be detected by SPV, *i.e.*, the diffusion length change is smaller than 2 μm. It also seems that iron gettering in IG-samples does not depend on previous cooling. However, in sample C5 (slowly cooled to 650 °C) the iron concentration is lower than in C4 (slowly cooled to 800 °C). From results it can be also concluded that this homogeneous nucleation or out-diffusion is not so strong as heterogeneous nucleation in IG wafers.

The change of the lifetime in sample B11 is 0.036 μs (2 %) after dissociation of FeB-pairs, so the iron cannot be detected. DLTS gives the iron concentration 2.23E11 cm^{-3} in

sample B11 indicating that it is well below the theoretical detection limit, which is about 6E11 cm^{-3}. The absolute and relative change of lifetime in B11 can be used as decision values, *i.e.*, the iron has been detected if both the absolute and the relative change are greater than 0.036 μs and 2 %, respectively. Using this selection rule iron is detected in samples A10 and B7 in which the both decision values hold true. Of course iron is also detected in the C-samples. In A/B6 and A/B9 neither of decision values are fulfilled. In the sample B10 the absolute change is high enough (0.042). The relative change is too small (1.4 %) but the calculated iron concentration is still used in Figure 3 as it seems to be correct.

Iron can be detected from samples A/B10 as they have a wider DZ than A/B6 and A/B9. The wider DZ increases the background lifetime and decreases the detection limit. The iron concentration in A10, B10 and B7 measured by DLTS was 2.7E11, 2.0E11 and 9.1E11 cm^{-3}, respectively. The iron concentration of 2E11 cm^{-3} is clearly below the detection limit when the background lifetime is between 1.7 to 2.3 μs as in samples A/B6 and A/B9. The iron concentration of 2E11 cm^{-3} is just within the detection limit when the background lifetime is between 3 to 4 μs as in samples A/B10. It is also easy to see that the iron concentration is clearly above the detection limit 3.5E11 cm^{-3} in sample B7.

DLTS results

The slow cooling should produce a rather homogeneously distributed dissolved iron concentration through the whole wafer, as the effective iron diffusion coefficient (17) remains so high at temperatures above 600 °C. The dissolved iron concentration after long annealing is also homogeneously distributed. Thus the iron concentration determined by the lifetime methods should yield the same results as DLTS measurements from near the wafer surface. This is confirmed in Figure 3. In Figure 3 measurement results obtained by different methods are compared. It is important to note that even lifetime values are sensitive to IG-process (e.g. oxygen loss) the calculated iron concentrations are in good agreement with DLTS results.

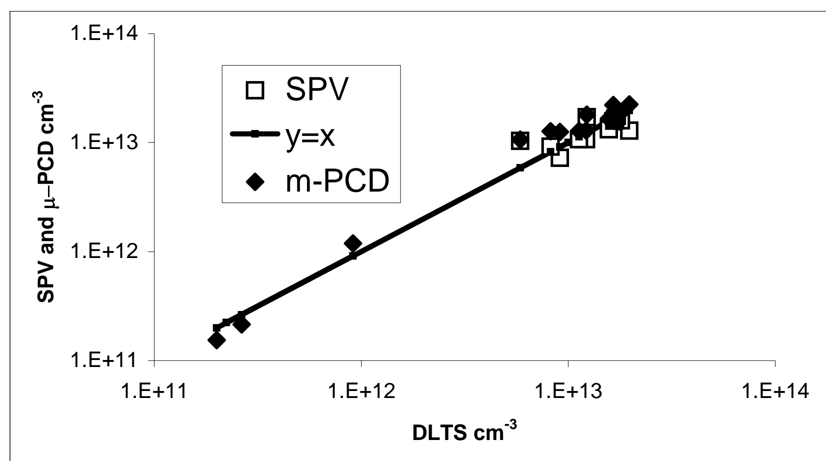

Figure 3. The comparison between DLTS, SPV and μ-PCD results.

CONCLUSIONS

During slow cooling the iron needs to have high supersaturation before any precipitation can happen. This study reveals that the critical temperature is between 600 ^{o}C and 650 ^{o}C when the iron contamination level is 1-2E13 cm^{-3}.

The iron can be quantitatively measured from IG wafers by lifetime methods if the iron concentration is nearly homogeneous. The detection limit of SPV is set by the IG-process, which determines the DZ-width and oxygen precipitation and related defect concentrations (bulk diffusion length). In conventional IG the bulk diffusion length is short and therefore the detection of iron is mainly limited by the DZ-width. The μ-PCD's detection limit for iron is also set by the IG-process but in our case it is a about decade lower than the detection limit of SPV.

ACKNOWLEDGEMENTS

The authors acknowledge the financial support from the Finnish National Technology Agency, Academy of Finland, Okmetic Oyj, Micro Analog Systems Oy and VTI Technologies Oy.

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