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ADVANCED METHODS OF INCREASING AND MONITORING THE LIFETIME OF NONEQUILIBRIUM MINORITY CHARGE CARRIERS IN MASTER DIES FOR HIGH-PERFORMANCE SILICON SOLAR CELLS *

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The distribution of nonequilibrium minority charge carrier lifetime $\tau_{n,p}$ in the depth of single-crystal silicon wafers was investigated by the improved method of stationary photoconduction decrease and by the standard method of photoconduction decay. The wafers of p- and n-type conduction used in hardware products for the electronic engineering were tested. To increase $\tau_{n,p}$ in the near-surface regions the wafers were subjected to the gettering annealing and deep chemical etching. Basing on the comparative analysis of resulting $\tau_{n,p}$ values it is proposed to use silicon wafers treated by chemical etching as master dies for domestic manufacture of alternative high-performance multijunction photovoltaic converters with vertical diode cells.

KEY WORDS: *master silicon dies, lifetime, nonequilibrium minority charge carriers, photoconduction*

1. INTRODUCTION

The operating efficiency of photovoltaic converters (PVC) is estimated by measurements and analysis of their photocurrent values, output data and diode parameters [1]. A design-engineering solution (DES) of such devices has important effect on their photocurrent and diode parameters, determining the output data and, consequently, on the coefficient of PVC efficiency [2]. Moreover, the DES influence on the photocurrent, output data and diode parameters occurs via the dependence of above-mentioned parameters on the lifetime $\tau_{n,p}$ and diffusion length $L_{n,p}$ of nonequilibrium minority charge carriers (MCC) in the base semiconductor material of PVC.

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In the process of manufacturing monocrystalline silicon PVC their master die undergoes different chemical and thermal actions influencing on the above-mentioned MCC parameters. Therefore, a quantitative monitoring of $\tau_{n,p}$ and $L_{n,p}$ is essential at the stage of updating the DES of photovoltaic converters.

We have realized [3] and further improved [4] the method of MCC lifetime determination in the master dies of Si-PVC by measuring the voltage drop during the idle running after the cutoff of light flux being incident onto the PVC photoreceiving surface. Approbation of this method has shown [4] that when manufacturing PVC on the base of high-quality silicon with an initial $\tau_{n,p} \geq 100$, under complex action of different processing factors, the lifetime of MCC in the master dies of such PVC decreases to less than 60 µs.

At present the use of cheap silicon, having considerably less values of $\tau_{n,p}$, instead of high-quality solar silicon, is proved to be promising. Therefore, of current importance is to develop methods allowing one to increase and to control $\tau_{n,p}$, at least, in the near-surface region of the master die where the PVC p-n junction takes place. (Recombination in the zone of rectifying junction depletion exerts the most negative influence on the instrumental structure parameters on the whole).

It is possible to increase $\tau_{n,p}$ and $L_{n,p}$ in the near-surface region of the master silicon die by realizing the methods known in the literature [5,6]. For example, one uses the annealing in air to clear the die from the background and other uncontrolled impurities penetrating in silicon during mechanical and chemical treatment after wafer cutting off from the initial monocrystalline ingot [5]. During annealing we observed the gettering of impurities from the crystal bulk into the SiO_2 oxide growing on the surface. The deep etching of silicon in the alkali solution [6] removes the near-surface layer containing microcracks and other defects caused by mechanical treatment of the initial silicon wafer surface. A further positive result of the deep chemical etching is a chance to decrease the silicon wafer thickness from 400 to 150-200 µm that corresponds to the world tendencies in updating the DES of high-performance silicon PVC [7]. Thus, the diffusion length of MCC exceeds the master die thickness thereby decreasing the recombination loss in its volume. Among the known methods of $\tau_{n,p}$ determination [8], with the purpose of monitoring the parameter under consideration in the master dies for Si-PVC, the most effective can be the method of stationary photoconduction and the method of photoconduction decay. Namely these methods, in our opinion, permit to measure the $\tau_{n,p}$ distribution in the thickness of the tested silicon dies being illuminated by the monochromatic light of a different wavelength. However, unlike the method of voltage drop during PVC no-load running [3] these methods do not require the presence of a diode structure in the sample under investigation.

In view of the aforesaid, the purpose of the present investigation is to approve the advanced methods of increasing the MCC lifetime in the master crystals of high-performance PVC. One more purpose is to improve the method of stationary photoconduction for monitoring the $\tau_{n,p}$ value distribution from the surface into the master die bulk.

2. TEST OBJECTS AND EXPERIMENTAL METHODS

The test samples were based on the monocrystalline silicon of p- and n-type conduction, of grades KEF-4.5 and KDB-7.5, respectively, having the planar dimensions of 17x12 mm and the thickness of (380±20) µm.

The first set of check samples, cut of the initial silicon wafers, was not subjected to any special exposure, excepting the forming of ohmic contacts necessary for determination of $\tau_{n,p}$ by the improved method of stationary photoconduction and the method of photoconduction decay [8]. The latter was used to confirm the reliability of $\tau_{n,p}$ values obtained by the first method.

To increase $\tau_{n,p}$ and $L_{n,p}$ the second set of test samples was annealed in air for the purpose of gettering the background impurity from the crystal volume into the SiO_2 oxide, growing on the surface. The annealing temperature, the same as in [5,9], was 900°C and the annealing time of 3 h, for good cleaning of the near-surface crystal layer from the impurity, was taken according to the known data on the mobility of different impurity atoms in silicon [9,10].

To approve one more method of MCC performance increasing, the samples of the third set were treated by deep etching in the 20% solution of NaOH in order to remove a near-surface fractured layer. The measured sample thickness after etching during 47 min was (170±5) µm. Then the samples were annealed in air at a temperature of 900°C during 3 h. This annealing is necessary to clean the crystals being treated from the penetrating high-mobile alkali ions of the etching solution which form many recombination centers significantly decreasing $\tau_{n,p}$ [11]. After annealing the samples of the second and third sets were subjected to the chemical etching in the concentrated HF during 5 min to remove the grown oxide containing the impurities, appearing after gettering, from the crystal volume.

For determination of $\tau_{n,p}$ on the surface of samples of all three sets by means of thermodiffusion enrichment with *n*-Si bismuth [12] and *p*-Si aluminum [12] there formed were ohmic contacts (10x5 mm) with 5 mm slot between their large sides. Aluminum electrodes of 0.5 µm thick were applied, by vacuum condensation, directly on these contact areas.

The offered advanced method of stationary photoconduction intended for determination of $\tau_{n,p}$ in the test samples was realized with the help of the measuring equipment being conceptually analogous to that in [8]. The procedure was the following. The sample surface, illuminated by the light from the slot between the aluminum electrodes, was iris-loaded so that the light quanta should not reach the near-electrode slot regions to exclude the influence of metal-semiconductor contacts on the sample photoconduction. The light source was a universal light-emitting diode lighter [13] with a wavelength varying in the range of $370 \le \lambda \le 960$ nm. The rectangular pulses, generated by this device, were of 150 µm duration with a pulse ratio of 50 ms that provided realization of stationary photoconduction effect in the sample regions being irradiated during the majority of time of the lighting act. To neutralize the level of shallow traps, the influence of which distorted true values of $\tau_{n,p}$, in the process of measurements according to [8], the stationary lighting of samples was performed by the polychromatic radiation from the other source.

The stationary photoconduction ΔG at different values of λ from the above mentioned wavelength range was calculated using the well-known equation

$$\Delta G = \frac{1}{R_C} - \frac{1}{R_T}, \tag{1}$$

where R_T is the dark resistance of the sample determined by means of the universal multimeter R4833 in the dc bridge condition; R_C is the dark resistance of the illuminated sample determined by the voltage waveform on the sample with the help of a digital storage oscilloscope S9-8.

The $\tau_{n,p}$ values in the test samples were calculated using on the $\Delta G(\lambda)$ values obtained by the model of inhomogeneous distribution of recombination centers in the silicon die volume [14] in the following way.

Using the expressions of [14] for the stationary photoconduction of a sample having inhomogeneous thickness and the concentrations of light-generated MCC in such a sample, it is possible to write for the stationary photoconduction in the case of absorption coefficient values α, at which $\alpha L \geq 1$, the following equation

$$\Delta G = \frac{E_0(\lambda)\left[1 - R_{ref}\right]\lambda\alpha(\lambda)\tau_{n,p}q\mu_{n,p}\left(1+b\right)}{hc}\left[d - \frac{s\tau\left(\frac{kT\mu_{n,p}\tau_{n,p}}{q}\right)^{1/2}}{\left(\frac{kT\mu_{n,p}\tau_{n,p}}{q}\right)^{1/2} + s\tau}\right], \tag{2}$$

where d is the sample dimension in the direction of light flux; $E_0(\lambda)$ is the incident radiation power on the sample surface unit; $R_{ref}(\lambda)$ is the coefficient of reflection from the sample surface; q is the absolute electron charge value equal to $1.6 \cdot 10^{-19}$ coulombs; $b = \mu_n / \mu_p$; μ_n, μ_p are the mobility values for electrons and holes, 0.12 m^2/(B·c) and 0.045 m^2/(B·c) [15] respectively, for silicon dies of both types; s is the surface recombination rate; k is the Boltzmann constant equal to $1.38 \cdot 10^{-23}$ J/K; T is the sample temperature; h is the Planck constant equal to $6.63 \cdot 10^{-34\,4}$ J·s; c is the velocity of light in vacuum equal to $3 \cdot 10^8$ m/s.

Using equation (2) and illuminating the sample by the irradiation with a wavelength of 940 nm, when the preferential light absorption takes place at a distance from the crystal surface, one can calculate $\tau_{n,p}$ in the crystal volume. The $\tau_{n,p}$ values obtained on the test samples of n- and p-types are given in the table for $x = 30$ μm, that corresponds to the distance from the photoreceiving surface of the silicon die, on which about 63% of light quanta with $\lambda = 940$ nm are absorbed [16,17].

If the sample is exposed to the shorter wave radiation, that corresponds to the case $\alpha L >> 1$ the expression for the stationary photoconduction takes the following form [14]:

$$\Delta G = \frac{E_0(\lambda)\left[1 - R_{ref}\right]\lambda \tau_{n,p} q \mu_{n,p}\left(1+b\right)}{hc}\left[1 - \frac{\alpha(\lambda) s \tau_{n,p}\left(\frac{kT\mu_{n,p}\tau_{n,p}}{q}\right)^{1/2}}{\left(\frac{kT\mu_{n,p}\tau_{n,p}}{q}\right)^{1/2} + s\tau_{n,p}}\right]. \quad (3)$$

Substituting in (3) the $\tau_{n,p}$ value, obtained in the solution of equation (2), the quantity s can be calculated. The MCC lifetime τ_s in the layer, corresponding to the depth of absorption of the radiation with a wavelength λ, is related with the recombination rate by the equation

$$\tau_s = \frac{\tau_{n,p}\left(kT\mu_{n,p}\right)^{1/2} q^{-1/2}}{\tau_{n,p}\left(kT\mu_{n,p}\right)^{1/2} q^{-1/2} + s\tau_{n,p}^{1/2}}. \quad (4)$$

By measuring and determining the above-mentioned quantities in samples exposed to the monochromatic irradiation in the wavelength range from 370 to 960 nm we can determine the distribution of τ_s in the depth of the test crystal.

The MCC lifetime was determined in the same crystals by the decrease of their photoconduction, similarly to [8], under conditions of monochromatic irradiation such as in the case of $\tau_{n,p}$ determination in the corresponding crystals by the above-described method of stationary photoconduction. The radiation wavelength, varying as in the case of the improved method of stationary photoconduction, permits to determine the $\tau_{n,p}$ distribution in the depth of test crystals.

3. RESULTS AND DISCUSSION

The MCC lifetime data obtained in different depths of silicon wafers by the method of stationary photoconduction and the method of photoconduction decay are given in the table. The absorption depths for each wavelength of the radiation used were determined from the spectral dependence of the coefficient of light absorption in silicon [18] by the formula $x = 1/\alpha$. Comparison of the data shows that the τ_s values, determined for the test samples by the method of stationary photoconduction are in good accordance with the τ_s values calculated by the method of photoconduction decay. Hence, the observed pattern of τ_s distribution in the crystal depth is conditioned by the characteristics of test samples not by the measurement peculiarities. Analysis of the data obtained shows that for initial silicon wafers, i.e., samples from the control group, a pronounced irregularity of τ_s distribution in the crystal depth is inherent. As a result, the τ_s value in the near-surface regions of crystals having ~ 1 μm thickness does not exceed 5% of the $\tau_{n,p}$ values in the volume of test samples. As the optimum depth of the p-n-junction position in the high-performance PVC is from 0.2 to 0.6 μm [16], the silicon of a similar quality level can not be applied for the manufacture of such devices.

Due to this fact it is necessary to carry out search and approbation of methods of τ_s increase in the near-surface regions of silicon wafers. One of such methods, as mentioned above, is a gettering annealing. However, the pattern of τ_s distribution in the annealed samples demonstrates that the gettering annealing during 3 h does not lead to the appreciable increase of τ_s in the near-surface regions of silicon wafers investigated. There is no positive tendency in the pattern of τ_s distribution in the sample depth and, therefore, the increase of the gettering annealing duration will not give effect. Basing on the results obtained we suppose that the τ_s decrease, observed in the near-surface regions of silicon wafers, can not be caused by the detrimental impurity contamination. Probably it is related with the presence of many recombination centers, the nature of which is determined by the technology specifity in particular, cutting and subsequent grinding with diamond suspension [3]. This procedure leads to the fracture of the near-surface layer and microcracking penetration in the crystal to a depth of 100-150 μm. Taking into account that initial wafers, cut from monocrystalline silicon bars, have a thickness of ~ 500-600 μm [19] and the silicon wafers become, after grinding and polishing, of (380±20) μm thick, the residual effects can exerts the above-mentioned negative influence on the τ_s value in the near-surface region. Therefore, one more, known in the literature, method of τ_s increase was tried with the use of deep chemical etching of silicon wafers in the alkali solution that makes it possible to remove completely the near-surface layer and the remainder of possible deep microcracks [6]. The obtained τ_s distributions in the depth of silicon wafers evidence that the supposition on the presence in them of recombination centers in the near-surface layers is not without foundation. According to the Table 1, in the case of chemically etched silicon wafers with *n*- and *p*- type conduction, the lifetime τ_s retains about 70% of its value in the depth corresponding to the p-n junction position in the high-performance PVC, which generally equals to 0.2-0.6 μm. Besides the fractured layer removal, this the elimination of the crystal surface influence on τ_s can take place that should lead to the significant decrease of the surface recombination rate.

TABLE 1: MCC lifetime values in microseconds determined for the test samples by the method of stationary photoconduction (SPC) and the method of photoconduction decay (PCD) as a function of the radiation absorption depth *x* in the monocrystalline silicon

***x*, μm**	***n*-type conduction**						***p*-type conduction**					
	Initial		**Annealing**		**Etching + annealing**		**Initial**		**Annealing**		**Etching + annealing**	
	SPC	**PCD**	**SPC**	**PCD**	**SPC**	**PCD**	**SPC**	**PCD**	**SPC**	**PCD**	**SPC**	**PCD**
0.1	0.6	0.7	0.9	0.7	8.7	8.5	3.0	2.0	4.0	2.0	52.0	62.0
0.7	1	1.5	1.7	1.5	8.9	8.6	7.4	12.0	12.4	12.0	52.4	64.0
1.5	3.8	3.8	4	3.8	9.4	8.8	14.8	25.0	24.3	25.0	52.9	67.0
2.5	9.4	6.8	7.3	6.8	9.8	9.1	40.3	71.0	69.5	71.0	53.2	71.0
3.5	12.4	9.0	9.6	9.0	10.2	9.6	44.1	74.0	72.0	74.0	56.4	72.0
30	19	13.1	15.3	13.1	12.4	12.0	70.8	82.0	87.0	82.0	79.2	76.0

To check the supposition that the fractured near-surface layer influences on τ_s we have carried out the mathematical simulation of the τ_s distribution in the depth of silicon dies. The physical model of [14] was used considering a semi-infinite semiconductor the recombination on the surface of which influences on τ_s in the near-surface region of the semiconductor. The relation, obtained for the given conditions, was used in the calculation of $\tau_s(x)$ distribution in the depth of the semiconductor exposed to the monochromatic radiation [14]

$$\tau_s(x) = \frac{\tau_{n,p}}{\alpha^2 L_{n,p}^2 - 1}\left[\frac{\alpha L_{n,p}^2 + s\tau_{n,p}}{L_{n,p} + s\tau_{n,p}} e^{-x/L_{n,p}} - e^{-\alpha x}\right]. \quad (5)$$

Using this model and Eq. (5) for the wavelength of 400 and 470 μm, the mathematical simulation of $\tau_s(x)$ distribution in the silicon die with *p*-type conduction treated by gettering annealing was carried out (see Figure). In the Fig. 1 for this sample also shown is the range of experimental dependences $\tau_s(x)$, the presence of which is due to the fact that the light diode radiation peak half-wide of about 20 nm does not permit to produce a pure monochromatic radiation.

As is seen from the Figure, the behavior of the experimental dependence $\tau_s(x)$ is qualitatively similar to the theoretical curves that evidences on the presence of a fractured near-surface layer, exerting negative influence on the MCC lifetime. This assertion can be valid also for the initial samples of silicon of *p*-type conduction, as well as, for the samples of silicon of *n*-type conduction (initial and treated by gettering annealing). As is seen from the table these samples have a qualitatively similar pattern of τ_s distribution in the near-surface crystal region.

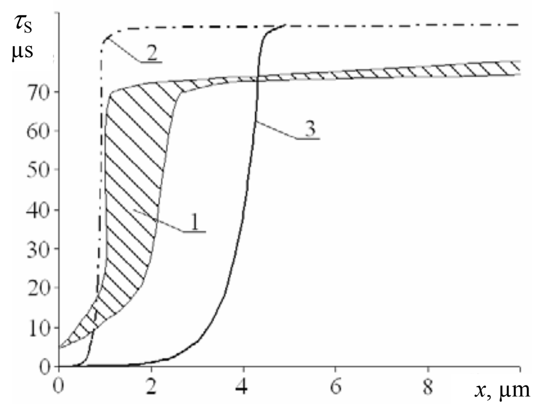

FIG. 1: The $\tau_s(x)$ distribution for the wafer of silicon with of *p*-type conduction: 1 – range of experimental dependences of $\tau_s(x)$; 2 – theoretical dependence $\tau_s(x)$ for the wavelength of 400 μm; 3 - theoretical dependence $\tau_s(x)$ for the wavelength of 470 μm

At the same time, there is no fractured near-surface layer in the samples treated by deep chemical etching. Therefore, the pattern of τ_s distribution in the near-surface crystal region is qualitatively different and does not allow one to describe correctly this distribution with the help of the model proposed in [14].

Another positive result of the monocrystalline wafer treatment by chemical etching is its mass decrease. This effect leads to the significant increase of the power produced by PVC relative to the device mass that agrees with present-day tendencies in the development of high-efficient monocrystalline Si-PVC [7].

At the same time in [3] is shown that decrease of the thickness of master dies in domestic Si-PVC without simultaneous creation of an effective system for capture and keeping of an active light component leads to the decrease of their photocurrent and, consequently, their efficiency too. This is related with the decrease of the number of electron-hole pairs in the diode structure of such Si-PVC as a result of its photoactive volume decreasing.

However, for domestic manufacture of single-junction monocrystalline Si-PVC presently it is economically inexpedient to realize an effective light-capture system including an updated frontal surface texture of fine inverted pyramids [20] and a high-efficient back-surface reflector [21].

At the same time, to reach the record high manufacturing characteristics of monocrystalline Si-PVC it seems promising to use master silicon dies with a significantly decreased thickness for implementation of a modified design-engineering solution in a multijunction (MJ) Si-PVC with vertical diode cells (VDC). It is conditioned by the following. The rectifying junctions of MJ Si-PVC are located perpendicularly to their photoreceiving surface, and the VDC size in the direction of light propagation (0.7-0.8 mm) is quite enough for almost full absorption of its photoelectrically active (for Si-VDC) component. Thus, the decreased thickness of master dies will allow one to increase the coefficient of MCC collection by the rectifying junctions, and to increase significantly the number of electron-hole pairs in the VDC volume. As a result, the photocurrent and the efficiency of such MJ Si-PVC will be increased. The use of high-efficient reflectors [20] based on the indium-tin oxide along the vertical boundaries of VDC allows one to develop the domestic manufacture of alternative high-efficient multi-junction Si-PVC of various destination, the production price of which will be lowered.

4. CONCLUSION

Investigations on the MCC lifetime distribution in the depth of wafers, containing monocrystals of silicon with *p*- and *n*-type conduction, carried out using the improved method of stationary photoconduction and the method of photoconduction decay, have demonstrated the following.

The values of τ_s in the near-surface layers of initial crystals and crystals treated by the gettering annealing in air are no more than 5% of the values of a corresponding parameter in the crystal volume. This fact can be due to the presence of microcracks and large number of point defects in the near-surface layer.

The results of mathematical simulation of the τ_s distribution in the depth of crystals, subjected to the gettering annealing, also support the supposition on the presence of a fractured near-surface layer exerting a negative influence on the MCC lifetime.

Decreasing the thickness of master silicon dies by deep chemical etching with subsequent annealing in air it is possible to remove the fractured near-surface layer and to increase τ_s in the near-surface regions to no less than 70% as compared to its value in the crystal volume.

The results obtained show that relatively cheap silicon wafers, treated by the deep chemical etching, can be used as master dies for domestic manufacture of alternative high-performance multijunction PVC with vertical diode cells.

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