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Revue Phys. Appl. **22** (1987) 637-643 JUILLET **1987,** PAGE 637

Classification
Physics Abstracts
72.20J — 72.40 — 66.30J

Influence and passivation of extended crystallographic defects in polycrystalline silicon

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(*Reçu le 20 octobre 1986, révisé le 5 février 1987, accepté le 27 mars 1987*)

Résumé. — L'influence et la passivation de défauts cristallographiques étendus sont étudiées dans du silicium polycristallin à gros grains aussi bien dans des plaquettes que dans des photopiles solaires à jonctions N^+ P. Quand la taille moyenne des grains dépasse 1 mm, l'influence des joints de grains sur les propriétés électriques devient négligeable, tandis que celle des défauts intragrains prédomine. Les défauts n'ont pas, par eux-mêmes, une activité recombinante importante, et c'est la ségrégation d'impuretés (oxygène), qui semble les activer. Deux techniques de passivation par l'hydrogène ont été utilisées : l'implantation d'ions et le recuit à basse température dans un flux de gaz. Les améliorations de longueur de diffusion des porteurs minoritaires, ainsi que les augmentations de photocourant obtenues avec des spots lumineux focalisés ($\lambda = 940$ nm), montrent que les effets des hydrogénations sont essentiellement des effets volumiques. Toutefois, la profondeur de passivation ne dépasse pas 30 μm après implantation d'ions et semble plus faible encore après recuit dans un flux de gaz. Comme la désorption de l'hydrogène se produit à des températures inférieures à 500 °C, la passivation par l'hydrogène paraît plus résulter de réactions chimiques avec des impuretés ségrégées aux défauts cristallographiques étendus que de la saturation de liaisons pendantes.

Abstract. — Influence and passivation of extended crystallographic defects are investigated in large grained polycrystalline silicon wafers and N^+ P solar cells. When the mean grain size exceeds 1 mm, the influence of intragrain defects becomes predominant. It was found that the defects have not by themselves a noticeable recombination activity and that the segregation of impurities (oxygen...), could be the main source of trap centres. Two techniques of passivation by hydrogenation are used : ion implantation and anneal in gas flow. The improvements of minority carrier diffusion lengths, and the increase of light beam induced currents at $\lambda = 940$ nm, indicate that hydrogen effects are essentially bulk effects. Nevertheless, the material is improved up to a depth of about 30 μm only after ion implantation or annealing in gas flow. As desorption of hydrogen occurs at temperatures lower than 500 °C, it is assumed that the passivation is more related to chemical effects with impurities segregated at extended crystallographic defects than the result of the saturation of dangling bonds.

1. Introduction.

The various polycrystalline silicon materials (cast ingots, ribbons) used to realize solar cells present many defects. The most obvious are extended crystallographic defects such as grain boundaries (G.B.'s) or intra-grain defects (subgrain boundaries, twins, dislocations, stacking faults...). In addition come point defects, dissolved and precipitated impurities. The existence of these defects, whose distribution and density vary from one type of sample to another, is a consequence of the conditions of preparation (rapid growth...) imposed by the search for a low cost material. Hence, it would be of great interest to find methods which may be able to passivate the defects and impurities especially when less refined silicon feedstocks are used.

What kind of defects and impurities should be passivated ? Grain boundaries have been the object of a great deal of attention mainly because they are easily accessible and can be studied individually. However, their influence is greatly reduced when the average grain size is greater than one mm, and it becomes clear that intragrain defects as dislocations, subgrain boundaries or decorated twins, affect the minority carrier lifetime. Interaction between impurities (especially oxygen) and defects must also be considered.

It was first admitted that the recombination centres arised from the existence of dangling bonds at G.B.'s and dislocations. However, the experimental results have not supported this assumption and it was necessary to emphasize that segregated impurities, precipitates or clusters could be the origin of these centres. Consequently, passivation techniques may utilize the neutralization of the recombination centres by diffusion to the defects of appropriate impurities (hydrogen ; lithium...).

The present paper summarizes results obtained by the group of the University of Marseille, in the field of electrical characterization of defects and passivation by hydrogen. Details have been given in previously published papers.

2. Experimental.

2.1 CHARACTERIZATION TECHNIQUES. — The investigated materials were large grained P-type wafers cut out of cast ingots prepared by Wacker Heliotronic (Silso) or by Laboratoires de Marcoussis C.G.E. (Polyx) (grain size > 0.3 mm ; boron concentration $\simeq 10^{16}$ cm^{-3} ; interstitial oxygen concentration $[O_i] \simeq 10^{17}$-10^{18} cm^{-3} ; substitutionnal carbon concentration : $[C_s] \leq 5 \times 10^{17}$ cm^{-3}).

Solar cells were made by Photowatt S.A. by means of conventional technology (N^+ P junctions).

Samples of 2 × 1 cm were cut from the cells. On each sample an array of 12 mesa diodes (1.5 × 1.5 mm^2) was produced, which allow a statistical measurement of photovoltaic and junction properties of each sample. Light beam induced current (L.B.I.C.) scan maps were obtained by scanning the diodes with focused monochromatic light from a monochromator ($\lambda = 0.94$ μm ; spot size ≅ 10 μm) [1]. E.B.I.C. contrast and scans have been made using a scanning electron microscope at 20 kV. Effective diffusion lengths L_n were measured by means of the S.P.V. method, in the wavelength range $0.8 < \lambda < 1$ μm, by illuminating the full area of the diodes. Interfacial recombination velocity S of G.B.'s was evaluated by Zook's method [2].

Samples were also cut from unprocessed wafers, and then chemically polished. Ohmic contacts were made on one face by diffusion of aluminium at 500 °C during 30 min. The deposition of 200 Å thick chromium film was used in order to obtain semitransparent Cr-Si diodes (2 × 2 mm) on the other face. The Cr layers were removed to passivate the defects, and then deposited again at the same place.

Samples of 20 × 2 mm^2 with ohmic contacts at the extremities were used to draw photoconductance scan maps [3]. Bicrystals of different orientations were also investigated.

Finally, the diodes were etched with Sirtl etch to evaluate the mean density of dislocation etch pits N_{dis} and the length of G.B.'s L_j per unit area.

2.2 PASSIVATION TECHNIQUES. — Hydrogen ion implantations were made by means of a Kaufman source, the voltage and the ion current of which were 1.4 kV and 1.4 mA.cm^{-2} respectively. The temperature of the samples was not regulated and can reach 400 °C after 4 min. The implanted surface of the samples were lightly etched in order to remove an eventual dammaged region.

Hydrogenations in gas flow consisted of anneals of the samples during 4 to 10 h in a silica tube heated at temperatures in the range between 200 and 400 °C, when 99.99 % pure hydrogen gas circulates (≃ 0.15 l/min). Some anneals were made in argon flows in the same conditions as blanks.

Desorptions were made in vacuum ($p \simeq 10^{-5}$ torr) by heating the hydrogenated samples at temperatures higher than 300 °C during a few hours.

3. Results. Influence of G.B.'s and dislocations.

The local measurements of L_n, of N_{dis} and L_j allow to correlate the values of L_n and the density of defects. Figure 1 represents the variation of L_n

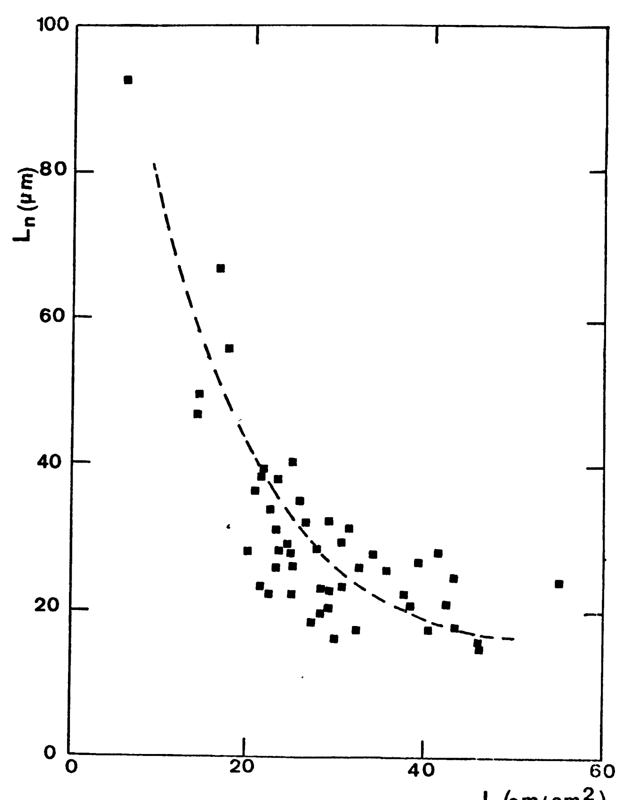


Fig. 1. — Dependence of effective electron diffusion lengths on the length of G.B.'s per unit area for diodes where N_{dis} is found below 5×10^4 cm^{-2}.

versus L_j for diodes where $N_{dis} \leqslant 5 \times 10^4\ cm^{-2}$. Figure 2 illustrates the variation of L_n towards N_{dis}, for diodes where $20 \leqslant L_j \leqslant 30\ cm.cm^{-2}$. The dispersion of the results is mainly due to the unaccurate evaluation of the defects densities, to the random distribution of intragrain defects and to a variable recombination activity of the defects.

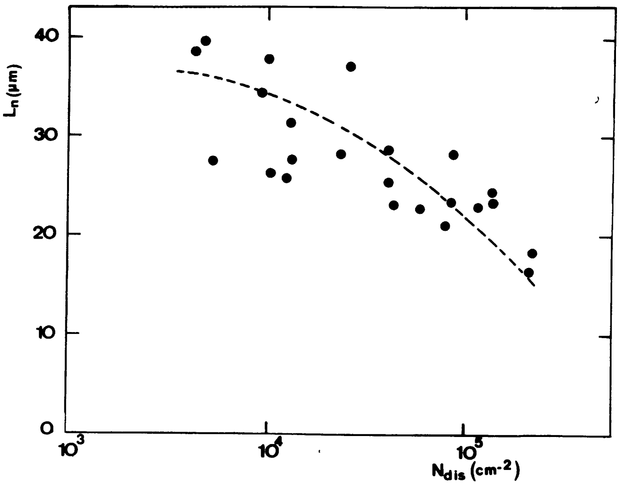


Fig. 2. — Variation of effective electron diffusion lengths *versus* mean etch pit densities N_{dis} for diodes where $20 \leqslant L_j \leqslant 30\ cm.cm^{-2}$.

It was found that L_n obeys the empirical laws :

$$L_n^{-1} = 12.5\ L_j - 1.33\ ,$$
$$L_n^{-2} = 1.5 \times 10^4 (N_{dis})^{1/4} - 6.6 \times 10^4\ ,$$

when L_n is in cm, L_j in $cm.cm^{-2}$ and N_{dis} in cm^{-2}.

The variations of the photocurrents J_{sc} *versus* L_j and N_{dis} are similar to those of L_n, although J_{sc} and L_n are not measured with the same light level, indicating that sunlight is insufficient to saturate the traps.

The different activity of G.B.'s on a given sample, and also along a given G.B. is well illustrated by the photoconductance scan map in figure 3, where the current peak intensity is depending on the local G.B. barrier height. A similar behaviour was observed with dislocations, comparing E.B.I.C. contrast and microphotograph of diodes.

It was not found a dependence of the G.B. recombination activity on the type of boundary. This was confirmed by the study of C.Z. bicrystals, the G.B.'s of which are inactive unless they have been annealed at temperatures above 500 °C.

The irregular activity of the defects and the enhancement of this activity by anneals suggest that its origin is certainly related to the segregation of

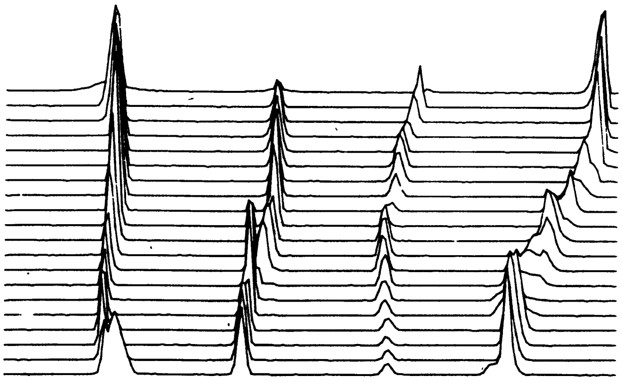


Fig. 3. — Photoconductance scan map of a sample containing several G.B.'s showing the local variation of the recombination activity.

impurities or to the presence of precipitates. Notice that the segregation of impurities can purify the grains within a recombining G.B. as illustrated by the microphotograph of figures 4a and 4b. Segregated impurities could be fast diffuser atoms, or more probably oxygen [4].

Dangling bonds which are theoretically distributed along the defects uniformly, seems to be less involved in the trapping of minority carriers, probably because a reconstruction of the lattice occurs at G.B.'s and dislocations. Several recent papers confirm the predominant influence of impurity defect interaction [5-7].

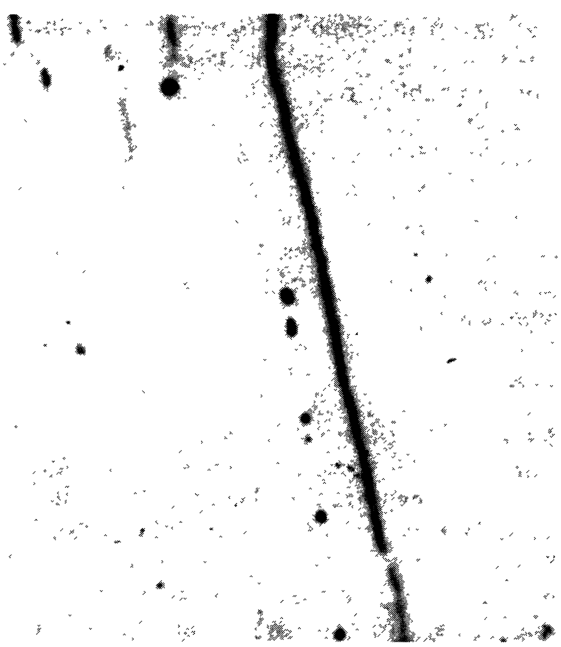
4. Results.

4.1 HYDROGEN ION IMPLANTATION. — At the beginning of this works, Seager *et al.* [8] have established that hydrogen implantation passivates G.B.'s and increases the photocurrent of solar cells, while Aucouturier *et al.* [9] and Jastrebski *et al.* [10] have reported that passivation is also extended to intragrain defects, particularly, to dislocations. The present results confirm that the improvements are due to bulk effects, as electron diffusion lengths are increased.

The best improvements are obtained after an exposure time of 4 min. Longer times dammage the surface in agreement with the observations of Panitz *et al.* [11]. The relative increase of L_n is essentially depending on the initial value L_{n0}, i.e. of the defects density, and $\Delta L_n / L_{n0}$ is the higher that L_{n0} is smaller, provided L_{n0} is in the 20 to 90 µm range, as shown in figure 5.

The E.B.I.C. contrast in figure 4c and the L.B.I.C. scan maps in figure 6 show that the response of the grains is increased 4 times, while the G.B. interfacial recombination velocities S which were initially in the 10^4-$10^5\ cm.s^{-1}$ range are decreased by one or two orders of magnitude.

a) b)



c)

Fig. 4. — (a) Microphotograph of an etched diodes : three different grains appears (twinned ; heavily dislocated ; homogeneous) separated by two G.B.'s A and B. (b) E.B.I.C. contrast of the same diode : notice that G.B. A is recombining, G.B. B not, and that contrast is reduced within G.B. A. (c) E.B.I.C. contrast after hydrogen ion implantation (1.4 keV ; 1.4 mA.cm^{-2} ; 4 min).

Drawn with different wavelengths, the L.B.I.C. scan lines indicate that passivation depth of G.B.'s is limited to about 30 μm, but can be more extended in heavily dislocated intragrain regions, as reported by Dube *et al.* [12]. However, the relative increases of L_n depend weakly on N_{dis} and L_j.

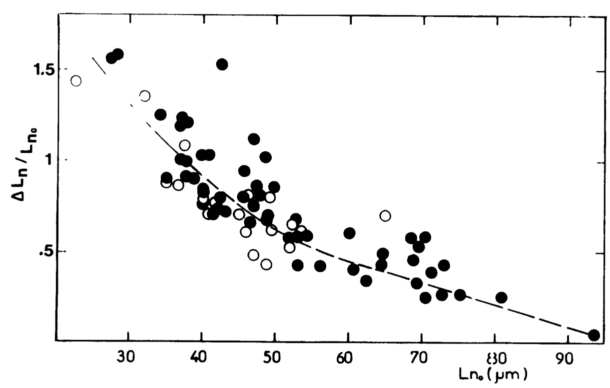


Fig. 5. — Dependence of the relative increase of effective diffusion length on the initial value L_{n0} : ● Cr-Si diodes ; ○ N$^+$ P mesa diodes.

Photoconductance scan maps confirm the improvement of L_n in the grains, and the reduction of S and barrier height of G.B.'s. They indicate also that anneals at temperatures above 450 °C in vacuum are required to suppress in few hours the hydrogen effect [13], in agreement with measurements of L_n by the S.P.V. method.

The passivation is not homogeneous : the G.B.'s of a given wafer do not all behave in the same way, and there is little or no correlation with the type of G.B.

Applied to N$^+$ P mesa diodes, hydrogen ion implantation improves the values of J_{sc}, of V_{oc} and of L_n [14]. E.B.I.C. and L.B.I.C. contrast and scans indicate that the improvements occur also in regions of the polycrystal where no G.B.'s or dislocation

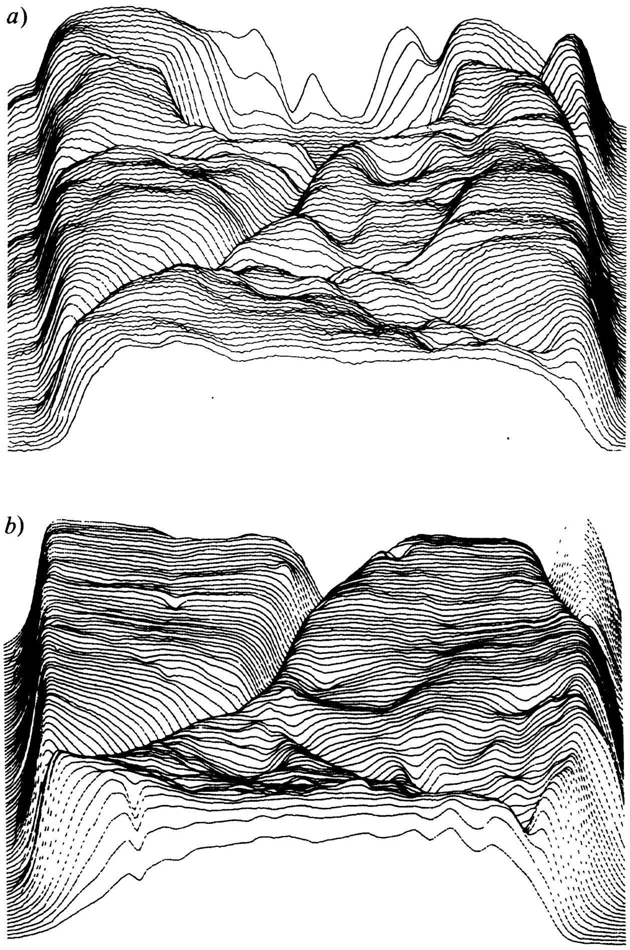


Fig. 6. — L.B.I.C. scan maps of a diode (λ = 940 nm) before (a) and after (b) hydrogen ion implantation (full scale sensitivities : 25 and 100 μV respectively).

etch pits have been revealed. The increase of V_{oc}, J_{sc} and L_n are the higher that the initial values are smaller (or the defect densities are higher). Consequently, in increases of J_{sc} cannot be explained by a change of surface reflectivity only.

In addition to the passivation of defects, when the substrate of the samples is heated at 300 °C, the implantation produces a compensation of the P-type material few μm below the surface [15]. A semi-insulating region appears within the surface, which can be moved when the sample is electrically biased, suggesting that hydrogen diffuses in silicon as H^+ ions [16].

4.2 ANNEALS IN HYDROGEN GAS FLOW. — Although anneals in hydrogen gas have been successfully used to passivate interfacial states in MOS devices and to increase the electrical conductivity of microcrystalline silicon [17, 18], their influence on the properties of large grained silicon wafers and solar cells was not well admitted.

The following results and few recent publications [19, 20, 21] indicate that such anneals are able to passivate extended crystallographic defects and to improve polycrystalline silicon wafers and cells.

Anneals in argon flow at temperatures in the range between 200 and 350 °C have not any influence on the properties of blank samples, but in hydrogen gas flow, electron diffusion lengths L_n are increased provided the initial values L_{n0} are below 70 μm as shown in figure 7.

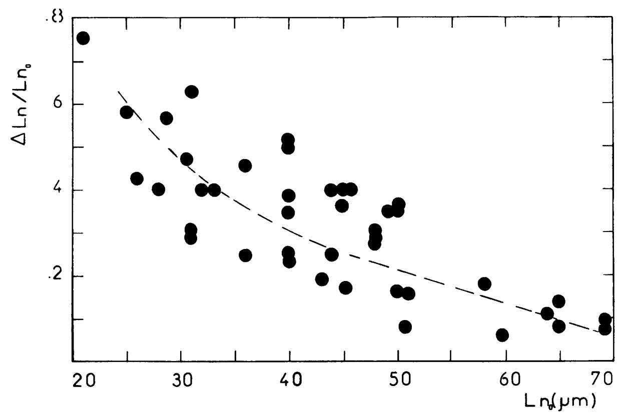


Fig. 7. — Dependence of the relative increase of effective electron diffusion lengths on initial value L_{n0} (280 °C – 4 h).

The relative improvements of L_n are smaller than those measured after ion implantation, but the variation with the initial value L_{n0} is very similar.

The higher increases are obtained for annealing temperatures within 280 °C, as shown in figure 8,

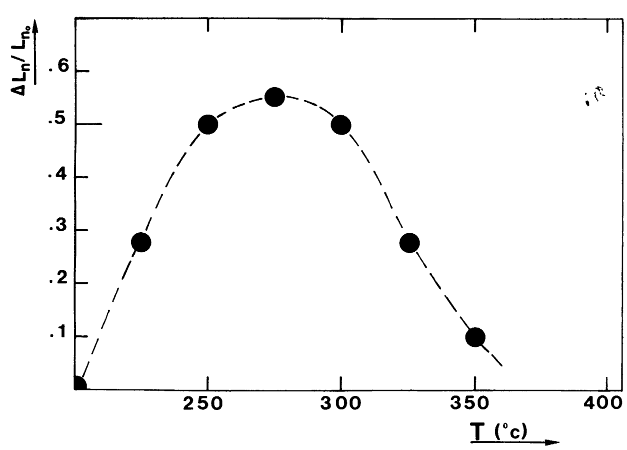


Fig. 8. — Variation of the mean value of L_n/L_{n0} *versus* annealing temperature (each point correspond to 28 diodes where $30 \leqslant L_{n0} \leqslant 40$ μm). Duration : 6 h.

and $\Delta L_n/L_{n0}$ tends to saturate after four hours. Below 200 °C and above 350 °C, no improvements are observed. The variations of $\Delta L_n/L_{n0}$ are well correlated with those of N_{dis} and L_j suggesting that in-diffusion of hydrogen occurs *via* the defects emerging at the surface [19].

L.B.I.C. and photoconductance scan lines drawn at different wavelengths show that passivation is limited at a depth about 30 μm below the hydrogenated surface. The scan lines within G.B.'s indicate that S decreases from the initial means value 5×10^4 cm.s^{-1} below 10^4 cm.s^{-1}.

The observed improvements are reversible : they disappear after annealing in vacuum during two hours at 350 °C, and they are restored by a new treatment.

Applied to N^+ P mesa diodes, front-side hydrogenations increase the photovoltage V_{oc} by about few tens of mV, but J_{sc} and L_n are practically not modified. *I-V* curves indicate that the junctions are improved by reduction of leakage and recombination currents and these reductions are well correlated with the increases of V_{oc}.

When front-side and back-side hydrogenations were done after removing the ohmic contact, L_n and J_{sc} are improved : L_n values in the 60 to 80 μm range are measured and J_{sc} values up to 23 mA.cm^{-2} without antireflection coating are achieved [22]. The relative increases of J_{sc} are all the higher that the initial values are smaller, as in implanted cells. The improvements of L_n, J_{sc} and V_{oc} are not affected when a back ohmic contact is made by aluminium diffusion at 600 °C during 10 min in argon flow.

4.3 TENTATIVE COMPARISON AND DISCUSSION. — The two hydrogenation methods can passivate polycrystalline silicon wafers and cells, and the passivations are the consequence of a bulk defect neutralization.

Ion implantation is the most efficient method. A high dose of hydrogen is introduced directly in the material through the entire surface [23], and then can diffuse in the bulk *via* the crystallographic defects, and also in homogeneous regions of the grains where microdefects and perhaps impurities could be passivated.

During the annealings, adsorption of hydrogen between 200 and 350 °C, is the most likely mechanism which could explain the observed results. Although molecular hydrogen cannot be absorbed on clean surfaces of silicon single crystals [24], Lemke and Haneman [25] have observed that the presence of microcracks at the surface can favour the reaction between hydrogen molecules and silicon. It may be that in the investigated material, the emergences of G.B.'s and dislocations behave as microcracks and constitute adsorption sites. Like hydrogen can diffuse in the bulk preferentially *via* G.B.'s and dislocations, the adsorption by the emergences of these defects could be enhanced. Consequently the defects may be passivated, and the variation of diffusion lengths (and photocurrent) are well correlated with the densities of extended defects L_j and N_{dis}.

The two methods lead to a limited passivation depth, which do not exceed 30 μm at G.B.'s, in spite of a high diffusion coefficient of hydrogen in silicon at low temperatures [26]. This could be partly explained by the model of Capizzi [27] based on a diffusion limited by trapping and detrapping near impurities, precipitates or dangling bonds.

The limited depth of passivation involves a variation of diffusion length with depth into the samples, and the S.P.V. method yield an average of the L_n values, as reported by Micheels *et al.* [28], which is smaller in hydrogen annealed than in ion implanted samples.

The low desorption temperatures corresponding to the disappearance of the improvements in the implanted and annealed samples are not compatible with the formation of Si-H bonds. Desorption at 340 °C was observed by Kazmerski [29] and attributed to the destruction of SiH_2 bonds. It may be that in dislocations and G.B.'s such bonds predominate, and this could explained why in the hydrogen annealed samples, the values of L_n and J_{sc} return progressively to their initial values when the temperature overpasses 350 °C. For implanted samples, the critical temperature is higher, about 450 °C, and the reason of this difference is not actually clear. Perhaps, exchanges of hydrogen with microstructural modifications of the surface, which result from the bombardment of even low energy hydrogen ions [29, 30], could bring an explanation.

An another difference between the two hydrogenation method appears when hydrogen is introduced by the front surface of solar cells. While passivation of bulk defects occurs in implanted samples, hydrogen annealed diodes are passivated at a depth about few microns below the surface, only.

This could be explained by a reduction of the adsorption because in highly doped N^+ regions the formation of impurity precipitates or agglomerates seems to be impeded, possibly due to enhanced solubility or complexing of impurities with dopant atoms.

It sems that the two hydrogenation techniques produce a similar passivation of the extended defects. The high dose of hydrogen introduced in the samples, the deeper penetration and the possible passivation of microdefects and impurities in the homogeneous intragrain regions could explain the higher efficiency of implantation.

5. Conclusion.

The present results indicate that in large grained polycrystalline silicon, intragrain defects constitute the main source of recombination centres, and that the segregation of impurities, probably oxygen, could explain the variations of this activity.

The defects can be passivated by mean of methods which neutralize segregated impurities. Implantation of hydrogen ions appears to be the most efficient technique as it can passivate in few minutes wafers or solar cells. However, the implanted surfaces are generally dammaged. Adsorption of hydrogen is less efficient, but presents the advantage to be a simple method. The two hydrogenation techniques yield bulk improvements in wafers, which can be verified by the increases of effective electron diffusion lengths and infrared L.B.I.C. scans.

The desorption of hydrogen is obtained in vacuum at temperatures below 500 °C, suggesting that hydrogen forms Si-H_2 bonds.

The applications of the passivation techniques described in the present paper are obviously limited to improve ordinary cells exhibiting photovoltaic conversion efficiency below 11 %. They can also homogenize the properties of all the wafers cut from a given ingot.

Newertheless, the actual lack of knowledge about the exact mechanism of the passivation by hydrogen requires still more investigations, especially with high resolution microanalysis methods.

Acknowledgments.

This work was supported by CNRS-PIRSEM (contract n° 5003) and Agence Française pour la Maîtrise de l'Energie (contract n° 505 1026).

The author would like to thank Dr. J. Fally and Dr. C. Belouet of Les Laboratoires de Marcoussis-GGE for helpfull discussions, wafers supplying, and hydrogen ion implantations.

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