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A HIGH-SPEED CHARACTERIZATION TECHNIQUE FOR SOLAR SILICON

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ABSTRACT

High-speed crystal growth techniques demand high-speed characterization techniques to allow a timely feed-back of information to the crystal growers. The unique properties of the Sielectrolyte-contact (SEC) provide for an extremely fast and simple measurement of the light-induced photo-current for any piece of Si without lengthy preparation of the specimen. Electropolishing at high anodic current densities allows for insitu generation of fresh surfaces whereas preferential etching of defects in various modes is possible at low current densities. In n-type Si a simple estimation of the minority-carrier diffusion length is possible in many cases. Laser-scanning enables local probing of the photocurrent and provides data about the homogeneity of a sample. The experimental realization of the method is described in detail and examples are given and discussed.

INTRODUCTION

Material analysts in the Siemens laboratories have been confronted lately with an unprecedented variety of "solar" Si specimens. Chunks of metallurgical Si from two carbo-thermic reduction furnaces /1/, poly- or single crystals grown from this material, "supported Web" (S-Web) /2/ specimens, Sisheets made by sintering Si powder, Si-ribbons obtained by roller-quenching, and reference material from outside vendors were to be characterized as comprehensively as possible and, needless to say, as quickly as possible.

"Characterizing" a piece of solar Si usually calls for statements about basic morphological and structural properties (e.g. flatness, presence of microcracks, grain size, texture, dislocation density, inclusions and precipitates); electronic properties (conduction type, resistivity, carrier mobility, minority-carrier diffusion length L and life-time τ , density and type of deep levels), and ultimately solar-cell properties

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(efficiency, open-circuit voltage U, short-circuit current I and fill-factor). For the potential user of solar Si, the short-circuit current I is the most interesting material parameter because it is dominated by the bulk properties of the Si (the exact magnitude of U and of the fill-factor are rather junction- and contact properties). I is basically determined by the diffusion length L which in turn depends mostly on the concentration, the type, and the spatial distribution of recombination centers ("deep-levels"). Deep levels, finally, are created by impurities, by lattice defects (grain-boundaries, dislocations, precipitates) or by combinations of both. A grain boundary or dislocation is called "electrically active" if it is associated with deep levels and therefore acts as recombination center for minority carriers.

A quick, simple and reliable method that could provide I $_{sc}$ -data and information about defects certainly would be most welcome. The Silicon-Electrolyte-Contact (SEC) can do just this; its mechanism and its application is the subject of this paper. Emphasize will be placed on the implementation of the technique. In order to provide easy reading for "silicon people", the necessary electrochemistry will be presented in semiconductor language rather than in electrochemical nomenclature. For more elaborate treatments of the electrochemistry of semiconductors the reader is referred to refs. /3-9/.

THE SI-ELECTROLYTE-CONTACT: BASIC PROPERTIES

ALC: NOT APPROXIMATELY

In a first approximation, the SEC may be thought of as a Schottky-contact with an electrolyte substituting the metal. With a counter-electrode of arbitrary material (but preferably chemically inert) that "contacts" the electrolyte, a voltage may be applied and a current passed through the Si-electrolyte junction. The important differences to a proper Schottky-contact at this point are: i) There is a measurable cell- or batteryvoltage U that depends on the properties of the Si, the counter electrode and the electrolyte, and ii) current flow is inherently tied to a chemical reaction. With HF-based electrolytes, the chemical reactions at the Si-electrode are the reduction and liberation of hydrogen-ions for the cathodic reaction (i.e. electron transfer from the Si to the electrolyte) and the dissolution of the Si for the anodic reaction (i.e. hole transfer from the Si to the electrolyte).

Fig. 1a shows typical U-I-characteristics for a mono-crystalline p-type sample with $\approx 0.5~\Omega$ cm resistivity in 2.5 % HF and a particular set of experimental conditions that will be discussed later. The main features are:

 The basic diode-characteristic is evident. For large reverse voltages, junction break-down occurs; for forward bias large currents flow (only limited by the resistivity of the electrolyte).

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- Illumination produces a photo-current, carried by the light-generated minority carriers (i.e. electrons); the magnitude of which is proportional to the light intensity.
- iii) Zero-current conditions are obtained when the applied voltage exactly compensates the built-in cell-voltage. The light-induced current is not superimposed on the darkcurrent for small voltages and thus does not induce a shift in the cell-voltage that would correspond to U of a solar-cell.
- iv) The forward-characteristic shows a peculiar structure with two current peaks, P, and P₂, and strong current oscillations for $U > U(P_2)$.
- v) For current densities $J < J(P_1)$ a film exhibiting interference colors grows on the Si surface which is called the "porous Si layer" (PSL) /10-12/; if the current density is raised beyond $J(P_1)$ this layer peels off instantaneously.

What can be done with this? Firstly, the junction breakdown voltage at reverse-bias condition carries information about the resistivity of the sample. Secondly, the magnitude of the dark current provides a good measure of the surface quality of the sample. Third, illuminating the sample with a calibrated light-source produces a photo-current I_{ph} which equals closely the I_{sc} -value of a solar-cell that were to be made from the sample. To give an example: Good single crystals illuminated under "air mass 1" (AM1) conditions (925 W/cm², spectral distribution corresponding to sun-light) produce about 30 mA/cm²; "Silso" material yields ≈ 25 mA/cm² and metallurgical Si may be as low as 1 mA/cm². Fourth, if the specimen surface is poor, it may be electro-polished by raising the forward-voltage beyond U(P₂). Finally, defects may be etched preferentially at forward current densities ≤ 1 mA/cm².

Before going into details, n-type Si needs to be discussed because it behaves very differently from p-type Si. Fig. 1b shows typical I-U-characteristics for n-type Si. Important points to note are:

- With reference to p-type Si, the reverse- and forward-current regions are interchanged, but the chemical reactions are not. H₂ is still developed for the cathodic reaction (now forward-current region of the SEC) and the specimen dissolves in the anodic region.
- ii) With light impinging on the Si, a photo-current flows in the anodic region. At very high light intensities the I-Ucurve is similar to p-Si for anodic currents because of current limitation by the electrolyte. The photo-current, however, is not proportional to the light intensity but may be twice as high as the light-induced minority-carrier current.
- iii) Electropolishing can only be achieved if the light intensity is high enough to allow for the current oscillations.
 Otherwise defect etching occurs as will be discussed later.

EXPERIMENTAL SET-UP

The experimental set-up comprises the electrolytic cell with the specimen holder, a pumping circuit for the electrolyte, light-source, potentiostat, and x-y-recorder. A description together with experimental details will be given in the appendix.

EXAMPLES OF MEASUREMENTS

p-type Si: Cathodic reactions

Fig. 2 compares I-U-characteristics of various Si specimens as they are obtained with Schottky-contacts (3 nm Cr, 5 nm Cu, 1 nm Cr, Al-grid, 18 mm² area) and, after removal of the metal layers in-situ by a short electropolishing treatment, with the SEC. The area in the latter case was 100 mm²; the dark currents nevertheless were comparable to, or even smaller than the Schottky-contact case. The result from these and many other measurements is that the photo-induced current $I_{\rm ph}$ can be measured quantitatively with the SEC-method. The possibility of repeated measurements with fresh surfaces produced by electropolishing is an additional advantage not available with other methods.

As already discussed, the SEC-method does not provide data about the open-circuit voltage U . But this is no serious disadvantage because U is mainly determined by the nature of the junction and thus by the technology used for its formation.

p-type Si: Anodic reaction

Electropolishing (i.e. etching of the sample to a mirror-finish) has already been discussed; it should be performed in the current-oscillation regime of the I-U-characteristics ($\approx 300 \text{ mA/cm}^2$ for [HF] = 2.5 %; cf. Fig. 1a). In contrast, very clear etching of defects is achieved for small current-densities around ≈ 500 _uA/cm². In this current regime the lgI-U-characteristics often display classical diode-behaviour (i.e. lgI \propto U with proportionality factors typical for diffusion- or recombination- currents, cf. /13/). The etching of defects at low current densities has been described before by one of the authors /14/, but was then not well understood. It is still not well understood, but with the following items in mind, it is rather simple to produce etching patterns at least as good or superior to those obtained by the traditional chemical etches:

i) "Electrically active" defects are always etched below a certain critical current density (roughly 1 mA/cm²); i.e. etch-pits or grooves are formed (cf. /14/). For good defect delineation a current-density x etching-time product of $\approx (3-5)$ mA.min.cm² is recommended. At very low current densities (≤ 100 /uA/cm²) the etching behaviour may be complex.

- ii) Whether or not electrically non-active defects are preferentially etched at somewhat higher current-densities as was stated in /14/ is an open question at present. Great care has to be exercised in judging if a grain-boundary was truly etched (i.e. a groove was formed) or if only a step was developed.
- iii) For all current-densities, including rather large ones, the etching rate depends initially on the surface orientation which manifests itself by steps at grain-boundaries. The maximum step heights observed depend somewhat on the current-density but seem to remain constant after a certain time of etching.
- iv) The colored layer always present at low current-densities partially obscures the etching pattern, partially enhances it (by showing different colors in different grains). It is easily removed by either raising the current-density J for a few seconds beyond J(P₁) or by immersing the specimen for a few seconds in KOH.

The preferential etching effects must be closely related to the mechanisms of carrier-transport across the Si-electrolyte interface. In a first approximation, it can be envisioned that for low current-densities recombination currents at defects are larger than the diffusion-currents flowing in more perfect areas of the sample. This view is supported by the observation that etch-pits disappear rather suddenly if the current-density exceeds a certain value. In any case, preferential etching of defects using the SEC-method is tied to current transport mechanisms in defected junctions and thus should allow a deeper understanding of the correlation between etching behaviour and defect properties.

Notwithstanding the difficulties of a detailed interpretation of the preferential etching phenomena in p-type Si, it is a simple and straight-forward procedure to obtain high-quality etching-patterns that allow to determine grain-sizes, dislocation densities, presence of precipitates etc. Fig. 3 serves to illustrate the aforesaid. A major advantage in comparison to many purely chemical etching procedures lies in the fact that defects are always etched, irrespective of the surface orientation, and that qualitative judgements about the electrical activities of defects are possible.

n-type Si: Cathodic and anodic reactions

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The cathodic current regime is rather uninteresting. The SEC is forward-biased; large currents flow; hydrogen is developed and not much can be learned about the Si specimen.

The anodic current regime corresponds to reverse bias of the SEC; the currents flowing therefore are interpreted as leakage currents (in the dark) or photo-currents with illumination. Since anodic current always go hand in hand with dissolution of the Si-electrode, the amount of Si locally dissolved is a direct measure of the time-integrated current flowing through that area.

As in the case of p-type Si, illumination induces a photocurrent. In contrast to p-Si, this photo-current is not identical with the light-induced minority-carrier current flowing to the Si surface, but is generally larger by a factor lying between 1 and 2. This phenomena is caused by an electron-injection process at the Si-surface which is triggered by the arrival of a hole and the concomitant breaking of a Si-Si bond. Much simplified, this process may be envisioned as follows: Surface atoms at the Si-electrolyte interface are tied with two bonds to the Si-crystal; the other two bonds are saturated with F ions. A hole arriving at the surface breaks one bond. The remaining one is either broken by another hole that happens to come along or, if that does not come to pass within a certain time, is broken by the injection of an electron into the Si. The free atom then enters the solution as SiF₂, an unstable species that immediately disproportionates according to

 $2 \operatorname{SiF}_2 \longrightarrow \operatorname{Si} + \operatorname{SiF}_4$

or oxidises as follows

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 $SiF_2 + 2 HF \longrightarrow SiF_1 + H_2$.

This process may also occur during the anodic dissolution of p-type Si at small current densities and probably plays a crucial role in the formation of the colored layer (PSL) formed for current densities smaller than $J(P_1)$.

Unfortunately, this electron-injection process makes direct measurements of the interesting quantity, the light-induced hole-current, very difficult. The injected electron-current has to be separated from the measured current and therefore has to be known. It is possible to "calibrate" a given experimental set-up (Fig. 4) but detailed measurements have not been attempted in the present work.

Preferential etching of defects now may be achieved either with the dark- or photo-current. Fig. 5 gives a rather spectacular example of a poly-Si sample that was grown from metallurgical Si and thus contains a high concentration of metallic impurities. Although nominally of 0.05 Ω cm n-type, it also contained a significant concentration of boron. The etching pattern obtained with the dark-current shows irregular features only loosely associated with the grain-structure of the specimen. Most likely, it mirrors the distribution of some metal that acts as generation center for minority carriers. The peculiar appearance of the etching pattern suggests that the main process governing the incorporation of the metal was constitutional supercooling of the Si melt. Fig. 5b shows the etching pattern on an area adjacent to that shown in Fig. 5a, but now the current was light-induced. Since the photo-current in "bad" samples is sensitive to the width of the space-charge region which varies with the dopant concentration, the appearance of striations is understandable.

Of particular interest is the etching-profile across a grain-boundary. Since a portion of the minority carriers generated in the vicinity of a grain-boundary will recombine at the grain-boundary, the photo-current density around the grainboundary is smaller than in the interior of the grain. The current increases with increasing distance from the boundary; the bulk-value will be reached at a distance roughly given by the minority-carrier diffusion length. Since the etching-profile mirrors the current-density profile, grain-boundaries, if etched with the photo-current, should appear as ridges with a basewidth of roughly twice the diffusion length. Fig. 6 proofs that this is indeed the case. The base-width of the grain-boundary ridges and the minority-carrier diffusion length (as obtained by the surface photo-voltage method) are in basic agreement.

Laser-scanning

As already mentioned, laser-scanning by simply moving the focussing lens is easily possible. The resolution is limited to a few 10 um because of light-scattering in the plastic window and the electrolyte. A particular advantage is the possibility to perform a defect-etch in-situ after the laser-scan which often allows a direct correlation between the photo-current profile obtained and structural properties of the sample; Fig. 7 gives an example. The specimen was a single crystal of ptype, but partially compensated by phosphorus. The photo-current was small, due to the presence of metallic contaminants, and therefore sensitive to the width of the space-charge region. It is seen that photo-current maxima correspond perfectly to the striations revealed by a short etching at ≈ 500 u/A/cm² and which therefore are interpreted as the maxima of the phosphorus distribution.

CONCLUDING REMARKS

The examples given demonstrate that the SEC does in fact provide a method for a "high-speed" characterization of solar Si. Some peculiarities of the method certainly do exist and some experience is needed for its proper application. But whoever has mastered chemical etching techniques and I-U-measurements can also handle the SEC. An initial effort is rewarded with a facility that can provide substantial data about a "raw" sample (e.g. broken off from a ribbon still being grown) within 15 min after it has been received.

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There is also ample opportunity for more involved measurements utilizing the SEC. Suffice it to mention the possibility of measuring the dependence of $I_{\rm ph}$ on the wavelength which may be used for diffusion-length determinations /15/, of deep-level spectroscopy /16/ and, with defined electropolishing steps between measurements, of carrier concentration profiling /17,18/. There is also much leeway for basic research: The etching behaviour of defects in both p- and n-type Si exhibits many particular features that wait for proper explanations. Moreover, the chemistry of the dissolution process, though much investigated, is far from being clear. The nature and the formation of the so-called porous Si-layer (PSL) still is not fully explained despite of its possible use in integrated-circuit technology (cf. /19/).

APPENDIX

EXPERIMENTAL DETAILS

The basic experimental set-up used for this investigation is shown in Fig. 8. Its main parts are the specimen holder and the electrolyte cell, the pumping circuit for the electrolyte, the light-source, the potentiostat and the x-y-recorder.

The specimen holder employed is depicted in Fig. 8b. It should allow an easy electrical contact to the back-side of the specimen, accommodate specimens of various sizes and shapes, define the sample area exposed to the electrolyte as precisely as possible and it should accommodate the reference electrode. It should not, above all, leak electrolyte to the sample backside and it should not obstruct too much the flow of the electrolyte. The specimen holder shown in Fig. 8b is a working compromise, better constructions are certainly possible. An interesting alternative, e.g., is described in ref. /20/; an other desirable option would be to rotate the specimen.

The electrolyte cell is a suitable container with fixtures that allow the insertion of the specimen holder, the counter electrode, and, if so desired, a thermometer and a pH-probe. It contains an inlet and outlet for the circulation of the electrolyte and a window to allow the illumination of the specimen. All parts of specimen holder, cell and pumping circuit in contact with the electrolyte should be HF-resistent; PVC or TEFLON are recommended.

Circulation of the electrolyte is essential for troublefree measurements. A jet of electrolyte streaming against the specimen (via a suitable nozzle connected to the electrolyte inlet) insures that currents are not limited by diffusion of molecules to or from the electrode. More important, it removes hydrogen bubbles which otherwise could stick to the speci-

men and then obscure measurements of I_{ph} . Fig. 9 shows the effect of pumping on the I-U characteristics of p-type Si. The jitter of the photo-current for the unpumped case is due to H_2 -bubbles. In the anodic region marked differences occur because of current limitations for the unpumped case. Rotating the specimen or the use of an ultrasonic generator, however, may serve the same purpose as pumping.

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The pumping circuit contains a TEFLON filter (10 ,um) to remove small particles and dust from the electrolyte. This is advantageous if a focussed laser-beam is used as a light source. An intermediary container allows quick and easy adjustment of the electrolyte level irrespective of pressure differences in pump and filter by moving it up or down. As a pump, any HF-resistant pump strong enough to overcome the flow resistance of the circuit will do.

As electrolyte, HF in a concentration of ≈ 2.5 % by weight was chosen. Electrolytes containing no fluorides do not give satisfactory results; fluorine salt solutions, e.g. NH_HF in H₂O, are too current limiting for electropolishing. HF in much higher concentrations leads to inconveniently high currents (the height of the peaks P₁ and P₂ is proportional to the HF-concentration) in the anodic regime and concentrations below ≈ 2 % are too current-limiting. Very diluted HF, however, does not wet clean Si-surfaces very well. The addition of a wetting agent therefore is essential. In our experiments a few drops of a commercially available wetting agent for acidic environments ("Mirasol"; producer: Tetenal) proved sufficient for the purpose.

The light source used was a 150 W halogen lamp adjusted to give a homogeneous intensity within an area \approx 5 cm in diameter. The intensity was adjusted to approximate AM1 conditions by placing a calibrated solar cell in the location of the specimen. As it was found that this intensity produced a photo-current of (30 + 2) mA/cm² in any good single crystal of p-type Si investigated (resistivities ranging from 0.1 Ω cm - 100 Ω cm), a 1 Ω cm sample was permanently mounted in a specimen holder and used as reference standard. Alternatively to homogeneous illuminations, a He-Ne laser-beam focussed to a spot size of \approx 30 µm could be used. An inexpensive camera-lens (f = 100 mm) was used for focussing; "laser scanning" was achieved by simply moving the lens with a motor-driven table.

A few words about the potentiostat and the reference electrode: The former is essentially a device that keeps the voltage across the semiconductor-electrolyte junction at a constant, pre-determined value and supplies the current necessary to maintain this potential-difference. A built-in ramp generator allows to scan the junction voltage between pre-determined values and thus to record I-U-characteristics. As in any precision measurements of I-U-characteristics, the electrochemical equivalent of a "4-point-probe" configuration is used for the

measurements. Two current leads contact the sample backside and the counter-electrode and two potential probes measure the voltage. One is simply connected to the sample backside, the other one to a "reference electrode" that is located next to the specimen surface. This reference electrode not only eliminates the voltage drop across most of the electrolyte (which is normally nonlinear with the current) but serves as the reference point for the voltage scale. Since any electrode immersed in an electrolyte develops an electrochemical potential that varies with the electrode material, electrolyte composition, temperature, etc., an "ohmic" contact to the electrolyte is impossible. The difference between the electrochemical potentials of the Si-electrode and the reference electrode therefore is superimposed on the applied voltage and measurements are meaningful only if the potential of the reference electrode is constant and known. For very precise measurements therefore standard reference electrodes such as a saturated calomel electrode should be used, for the objective of this work, however, a simple Pt-wire will be adequate. The counter-electrode could be used as reference electrode, too. (Simplifying the set-up to a "2-point-probe"), but then the measured voltage will include the voltage drop in the electrolyte (quite significant at higher current densities) and unaccounted changes of the potential since the counter-electrode has to pass current and therefore cannot maintain a constant potential.

The potentiostat should be able to deliver about 1 A/cm^2 but should also allow measurements in the μA -region. Its voltage range should be large (e.g. -10 V - +10 V) and, considering the diode-character of the SEC, it should be fool-proof.

The samples should fit into the specimen holder, otherwise no restrictions concerning size or shape are necessary. Very irregular samples, or samples with holes (e.g. metallurgical grade Si or porous sintered Si) that would leak electrolyte to the backside contact, can also be simply contacted with an insulated wire (insulate the contact point with wax) and immersed into the electrolyte. The illuminated area then has to be defined by an aperture and high dark-currents may be encountered. The quality of the sample surface is of no importance since an electrically good surface (i.e. passing only small reverse currents) can always be obtained by electropolishing; this is demonstrated in Fig. 10. However, under-etching at the circumference of the rubber-seal may occur which often is accompanied by noticeable dark-currents. It is therefore good practice to etch unpolished samples before insertion in the specimen holder for a few minutes either in KOH (20 % at 80 °C) or in CP4.

The samples need to have an ohmic back-side contact which can be produced by the usual methods (e.g. evaporating and alloying Al in p-type Si). For "high-speed" measurements, it has been found sufficient to paste Ga and liquid Ga-In-eutecticum on the sample backside in conjunction with some scratching. It is good practice to make two contact spots in this way and to measure the I-U-characteristics between them. This is easily done with the potentiostat; if an ohmic characteristic is obtained the contacts are good enough for measurements.

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 $\begin{array}{c} \underline{\text{Fig. 1}} \\ \text{Typical I-U-characteristics of $\approx 0.5 Ω cm p-Si (Fig. 1a)} \\ \text{and for n-type Si (Fig. 1b) in a 2.5 $\%$ HF-electrolyte} \\ \text{for various light intensities.} \end{array}$



Fig. 2 Comparison between I-U-characteristics obtained with the SEC and with Schottky (MIS) contacts. The current densities in the MIS case are smaller because of lightloss in the metal layers.

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Fig. 3 Examples of etching-patterns obtained by anodic etching. The right-hand micrograph demonstrates pronounced differences in the etching behaviour of twin-related boundaries most likely related to differences in the electronic activity of the defects.







Fig. 5 Etching pattern of n-type poly-Si obtained a) in the dark; b) with illumination.