New Modes of Fast Fourier Impedance Spectroscopy Applied to Solar Materials Characterization and Semiconductor Pore Etching

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Impedance spectroscopy (IS) essentially measures the current or voltage response of a complex system to small perturbations of the applied voltage or current, respectively. Multi-mode FFT IS as introduced here includes responses to (possibly local) perturbations of light coupled to photo-electrical semiconductor devices such as solar cells or (photo) electrochemical cells, and allows quick measurements by employing a FFT technique (≈ 1 s per spectrum) and thus in-situ data acquisition. In conjunction with device models many parameters can be assessed simultaneously, quickly, and with high spatial and/or temporal resolution. Examples given include electrochemical pore etching in Si where so far inaccessible parameters like the process valence or the pore depth are recorded in-situ, and simultaneous mapping of more than 8 solar cell parameters such as life time, doping, surface recombination velocity, series resistance, or reflectivity.

1. Introduction

Solar Cells and certain photo-electrochemical Si "devices" employed for electrochemically etching so-called macropores (cf. (1, 2) for reviews concerning porous Si) have several features in common. Of particular interest with respect to the topic of this paper are:

- i) Minority carriers are essential for the device and generated well above equilibrium levels by illumination.
- ii) The photo-generated minority carriers can recombine in many ways but only the percentage flowing across some interface drives the device.
- iii) The illumination may be coupled to the frontside or the backside of the device.
- iv) There is typically an ohmic contact and a blocking contact (i.e. a contact that only passes minority carriers) as integral part of the device.
- v) Device operation employs external voltages V or currents I linked by some global IV characteristics.
- vi) The local parameters on some pixel on the device surface (with device dimension in 100 cm^2 range and pixel dimensions in the 1.000 μ m² range) may be quite different from the (averaged) global parameters.
- vii) The hardware (and to some extent the software) for measuring electrical properties of solar cells or solid-liquid junctions in electrochemical cells is rather the same. Always needed is a potentiostat / galvanostat that can impress a constant voltage V or (large) current I on the global system relative to some reference while detecting very small dI or dV responses, respectively. In addition, an integrated illumination source capable of delivering high light intensities P with very good spatial and temporal uniformity is needed that also must offer the possibility to modulate the intensity in a large range of frequencies.

Characterizing these devices globally as well as locally in shrot times is a demanding task. A "simple" solar cell, for example, calls for two (related) sets of parameters that need to be determined:

- i) Electrical and optical parameters: Mainly short circuit current I_{SC} , open circuit voltage U_{OC} , fill factor *FF*, series and shunt resistors R_{SE} and R_{SH} , respectively, the coefficient of reflection *R*, and resulting from all of that the efficiency η .
- ii) Semiconductor parameters: Diffusion length L or lifetime τ , surface recombination velocities S, junction properties (e.g. pre-exponential factors or ideality factors), doping and related bulk resistances and contact properties.

Of course, the technically interesting electrical parameters follow "somehow" from the semiconductor parameters. Be that as it may, measuring this set of parameters *globally* is not easy, and mapping these parameters *locally* is a formidable task. Measurement times restricted to a few seconds (necessary for in-line controls of solar cells) do not make this task any easier. "Parallel" techniques (essentially producing pictures) like lockin thermography (3 - 5) or the emerging photo- or electroluminescence techniques (6, 7)are fast but do not deliver all the information wanted and classical scanning techniques like, e.g. LBIC (light beam induced current) (8, 9), always have resolution – measurement time trade-offs and typically can only access one or two of the parameters listed above.

For the electrochemical pore etching device the situation is even more complicated because in contrast to a solar cell, its "mode of operations" is not yet fully understood and it changes all the time because Si is dissolved. Characterizing this device thus includes understanding what exactly is going on while the device is "on", i.e. current flows and dissolves silicon, and to follow it in time as things keep changing. Besides the technique reported here there is no other local in-situ technique capable to assess what is going locally at the tip of a growing macropore with a diameter typically in the 1 μ m region at a depth of some μ m to > 300 μ m. Measurement time is of essence once more because the system may change quite a bit within a few seconds since the interface is etched, i.e. moves into the bulk Si all the time.

It will be shown that "impedance spectroscopy" (IS) in the sense of analyzing the linear response of the devices to small perturbations of either voltage, current or light intensity at various frequencies allows to extract a large amount of meaningful parameters from matching experimental spectra to calculated ones. This necessitates new modes, i.e. photo impedance (PI) where dI/dP or dj/dP (I = current, j = current density, P light intensity) are measured (PI is not a classical impedance since its unit is not " Ω ") for either frontside or backside illumination, an FFT impedance technique to meet measurement time restrictions, dedicated light sources fully integrated into a computer controlled system (including a focused scanned Laser beam for local measurements), many optimization routines to overcome inherent signal to noise problems and, most important, extensive modeling of the devices under investigation. Taken together, these demands translate into custom-build systems, developed over many years, which are now marketed via ET&TE GmbH (10).

2. Macropore Etching in Silicon

2.1 Basics and Modeling Efforts



Figure 1. Schematic illustration of macropore etching using backside illumination. P(bsi) denotes the backside illumination intensity, the various d for depth the geometry of wafer and pores as indicated, j_b and j_P are the current density right at the backside or at the tip of the pores, respectively, and S_P is the interface recombination velocity at the plane define by the tip of the pores varying by ΔS_P between the tips (where it should be very large) and the region between tips.

Fig. 1 schematically shows the paradigmatical set-up for the first basic situation: etching so called macropores in n-type Si under backside illumination and with lithographically defined points of pore nucleation (cf. (1, 2) for reviews of this standard technique). For what follows we look at an advanced model of this pore-etching situation (cf. (11 - 14) for details). An ohmic (but light transparent) contact defines the backside of the (pore etching) device; the contact to the frontside is achieved via an electrolyte (dilute HF in this case) and shows essentially Schottky behavior. In some (pseudo) steady state as shown in Fig. 1 the holes produced at the backside diffuse a distance $d_{\rm b}$ to the virtual plane defined by the pore tips and either recombine in the bulk (characterized by the bulk diffusion length $L = (D\tau)^{1/2}$ (D = diffusion coefficient of holes (= minority carriers), $\tau =$ minority carrier lifetime) or at the virtual plane with an interface recombination velocity that differs by some $\Delta S_{\rm P}$ between pore tips (symbolized by a star) and the area between pores (symbolized by a cross)). The hole current flowing through the pore tips dissolves Si by some chemical reaction and thus produces pore growth into the bulk of the silicon. "Operation" of the device, i.e. growing pores with constant diameters in the $(0.5 - 5) \mu m$ region to depth of $> 500 \,\mu\text{m}$ (cf. (2) for details and possible applications of macroporous Si) is usually done under conditions where the current is kept constant (or follows some pre-determined profile with time) at a potential that is also kept constant or at some profile with time; the control parameter then is the light intensity P that is adjusted via a feedback loop. The interface of interest is by definition inhomogeneous – pore growth is

synonymous to a high current density at the pore tips and a low current density at the pore walls and the original surface. What do we want to characterize in-situ, i.e. while the device is running, i.e. pores are etched? The first obvious parameter is the depth of the pores d_{Pore} at any moment of the formation process. It is clear that d_{Pore} can not grow with a constant speed since it becomes progressively more difficult to transport reactants and reaction products down or up a pore as it gets longer and longer. For optimal pore growth this translates into keeping the valence n of the dissolution constant at about n = 2.7 (11, 15). The valence n of the pore etching process simply determines how many electrons need to flow through the external circuit in order to bring one Si atom into solution; it is a non-integer because at the interface several processes with different valences occur simultaneously. We also need the diffusion length L of the Si (which can be taken as a constant) and in particular the difference of the interface recombination velocity $\Delta S_{\rm P}$ at the plane of the pore tips. Omitting a relatively trivial consideration of the hole production at the backside, and assuming that all holes recombining at the pore tips are converted into a Si-etching current, the photo impedance $Z_{Photo}(\omega, d_B, L, \Delta S_P) = dI/dP$ (ω is the circle frequency of the disturbance signal) can be calculated analytically by solving the corresponding (time-dependent) diffusion equation of the problem as sketched in Fig. 1. One obtains

The second term takes into account (in linear order) the non-constant interface recombination velocity at the pore tips and between pores (cf. **Fig. 1**); it is necessary (against earlier expectations (15)) to account for the experimental impedance data. While Eq. [1] already provides for a rather good fit to the measured spectra, it is not yet perfect. Perfection can be achieved by adding a semi-empirical diffusion term that takes into account some of the as yet not fully understood chemistry of the process in the form:

$$Z_{Complete}(\omega, d_B, L, \Delta S_b) = A_1 \left(Z_{Sem}(\omega, d_B, L) + \frac{A_0}{\frac{1}{Z_{Sem}(\omega, d_B, L)} + A_2 \sqrt{i\omega}} \right) \left(\frac{\frac{1}{\sqrt{\frac{1}{L^2} + \frac{i\omega}{D}}}}{\frac{1}{\sqrt{\frac{1}{L^2} + \frac{i\omega}{D}} + \Delta \frac{D}{S_b}}} \right)$$
[2]

The new term essentially describes a diffusion process and the parameters A_0 and A_2 contain information about the "chemistry" in some lumped form; A_1 is simply a trivial proportionality constant.

If we now consider the standard U/I impedance, here in the form Z = dI/dU, a firstprinciple theoretical modeling is not possible since this would require a precise knowledge of the interface reactions, which are to some extent the unknown processes we are after. What can be done, however, is to describe the sold-liquid junction by a suitable mixture of relaxation and diffusion processes that have to meet the following conditions i) it must reproduce the measure U/I impedance spectra; ii) it must be compatible to the existing knowledge about charge transfer processes at the solid – liquid interface.

One of the standard equations often used for IS spectroscopy meets these conditions, it is

$$Z(\omega) = R_s + \frac{1}{\left(\frac{i\omega\tau}{(R_p + \Delta R_p)(1 + i\omega\tau)} + \frac{1}{R_p(1 + i\omega\tau)}\right) + i\omega C_p}$$
[3]

 R_s embodies a simple series resistance and describes ohmic losses, C_p describes the capacitance of the interface, and R_p the chemical transfer resistance of the chemical dissolution process. The chemical dissolution splits up into two known processes with different reaction rates characterized by the relaxation time τ of the slow process (= oxidation of Si) and the time constant R_pC_p of the fast process (= direct dissolution of Si). The difference ΔR_p describes the increase in the chemical transfer resistance at higher frequencies.

It is helpful to realize that impedance spectroscopy in any mode measures the system responses at different frequencies and thus primarily time constants of processes described by relaxation, resonance or just diffusion (via $L = (D\tau)^{1/2}$). Keeping in mind that Si dissolution, whatever the details, contains a slow and a fast process as outlined above, allows to extract the valence *n* of the process (physically given by the competition between the slow process (oxidation) with a valence n = 4 and the fast process (direct dissolution) with $n \approx 2$ to

$$n = \frac{4}{2 - \frac{\Delta R_p}{R_p + \Delta R_p}}$$
[4]

All variables contained in Eqs. [2], [3], and [4] can be extracted in-situ by matching recorded spectra to the proper equations. In-situ data are thus obtained; and they are *localized* measurements up to a point because they only describe (in summary) what happens at the pore tips.

2.2 Some results

The apparatus developed for this purpose is capable of recording a complete I/U and I/P spectrum in one second each, and a pore etching experiment running for e.g. 6 hrs produces > 7.000 spectra or around 6 GB of data that are fitted in real time, with the ex-



tracted parameters displayed as a function of etching time or pore depth, respectively. Figs. 2 and 3 show some measured impedance spectra and Fig. 4 the time development of the parameters extracted.

obtained during etching. a) Nyquist plot; b),c) the corresponding Bode the imaginary (b) and the real plots for the imaginary (b) and the real (c) part of the impedance. Measured data (squares) the fit employing Eq. [2] are and the fit employing Eq. [3] shown. are shown.

Figure 2. Typical UI spectra Figure 3. Typical bsi-PI specmacropore tra. a) Nyquist plot; b),c) the corresponding Bode plots for (c) part of the impedance. Measured data (squares) and

The example shown in **Fig. 4a** is of particular interest; it was taken with a new and optimized electrolyte that allows etching pores far faster and deeper than before (16). Shown are most parameters contained in the impedance equations from above plus the valence n and a curve displaying the ratio of the global etching current I and the illumination intensity (expressed as current I_{illu}). It is clearly visible that the system is in some "chaotic" state (the word "chaos" here is not used in its strong form as an expression of deterministic chaos, although that is probably the reason for this behavior) but that this is not expressed in the pore morphology obtained ex-situ by scanning electron microscopy (SEM). Note also that the valence n, while approaching the "magic" value of 2.7 on average, shows strong fluctuations.



Figure 4. Etching conditions where pore growth repeatedly almost stops for short time periods. An aqueous electrolyte with an HF concentration of 5 wt.% with 0.85g/l of carboxymethylcellulose salt (CMC) was used. a) Left side: results of *VI* impedance as described by Eqs. [3] and [4]. Right side: results of PI impedance as described by Eq. [2] plus the logarithm of the ratio between DC etching current and DC illumination intensity. b) A cross-sectional SEM picture of the pores obtained.

Space does not permit a detailed discussion of the information contained in the data shown above. Suffice it to state that they allow monitoring the etching process closely and reliably and provide for substantial progress towards a fuller understanding of the pore etching process. They also prove beyond reasonable doubt that the electrochemistry of Si contains a stochastic component that expresses itself in fluctuations as shown in the spectra but also in some feedback processes that ensure, for example, uniform pore morphology despite parameter fluctuations.

3. Characterization Of Solar Cells

3.1 The CELLO Techniques

Before we introduce and discuss the (local) application of photo impedance $Z_{Photo} = dI/dP$ to standard Si solar cells, it is helpful to briefly introduce the standard CELLO technique which analyzes the response to a perturbation with just one (small) frequency and serves as the basis for the more involved impedance measurement. CELLO is short for Solar CEL LOcal Characterization and works by:

i) Fixing the global illumination intensity P and one of the two remaining global values (I or U) of the external variables (for temperature T = const.). This must be done with high precision. It is not sufficient, for example, to insure "open circuit" i.e. I = 0 A or short circuit (i.e. U = 0 V) by simply opening or short-circuiting the two "wires" of a solar cell since this would neglect the serial resistances inherent in the solar cell and its leads and thus the voltage drop therein. A four probe configuration is required,

with (current less) potential probes on the solar cell and an active feedback potentiostat / galvanostat that ensures proper conditions with an accuracy of about 10^{-5} .

- ii) Disturbing the system "solar cell" locally at coordinates (x,y) by a sine modulated Laser beam will induce a response of the global solar cell either by some dI(x,y) for potentiostatic conditions (i.e. a constant impressed voltage U) or a dU(x,y) for galvanostatic conditions. The sine modulation here is only needed to allow lock-in data acquisition techniques.
- iii) Recording these dI or dU responses at some suitable working points of the global characteristics (e.g. at the point of maximum power generations, short circuit, open circuit, or for reverse bias) produces all the information needed for extracting mostly electrical parameters as defined in the introduction and allows to produce maps of e.g. the local series resistance R_{SE} .

Parameter extraction from the raw data is not easy and involves a substantial theoretical effort towards modeling the CELLO system, not to mention overcoming signal to noise problems by dedicated soft- and hardware. For details the reader is referred to (17 -19); **Fig. 5** shows two typical CELLO results. **Fig. 5a** shows a direct dI(x,y) map taken at short-circuit conditions (i.e. U = 0 V); this map is relatively easy to obtain and comparable to typical LBIC results. **Fig. 5b** shows a series resistance (R_{SE}) map. This map is computed from separate dI and dU measurements on the base of extensive modeling (19). There is no other technique offering this capability without destroying the solar cell. Note that both maps come with full quantitative color-coding as shown in the histograms. The inscribed red square in the upper left hand corner marks the area shown in **Fig. 6**



Figure 5. Examples of CELLO results. a) (Differential) short-circuit current map; b) Map of local series resistances R_{SE} .

3.2 The CELLOplus Techniques

CELLOplus is the abbreviation for CELLO *plus* photo-impedance spectroscopy. In principle, a CELLO system is used with the added feature that the Laser beam intensity is now modulated with several frequencies (simultaneously) in the range of typically 4 kHz to 50 kHz and the response is measured with regard to amplitude and phase for all frequencies (by FFT). This is not as easy as it appears since very high precision is needed. For example, signals of $dI \approx 100 \mu A$ must be extracted from a background of several A with a precision in the phase of a fraction of a degree over a large range of frequencies. This necessitates, for example, that the frequency response of the external circuit must also be determined as a (parasitic) system variable and "subtracted" from the data obtained.

CELLOplus has a certain advantage in comparison with CELLO or other characterization techniques for solar cells: the theoretical photo impedance for short-circuit conditions can be calculated from pretty much "first principles" and thus provides a solid base for parameter extraction from measurements. For a standard solar cell one obtains for the frontside illumination (fsi) photo impedance Z_{fsi}

$$Z_{fsi} = Z_{Si} \cdot Z_{RC} \cdot Z_{Res}, \qquad [5]$$

with

$$Z_{Si}(\tau, D, S_B, R, \alpha, d_W, \omega) = qF(1-R)$$

$$\frac{1}{1 - \left(\frac{G}{\alpha}\right)^2} \frac{\frac{G}{\alpha} \left(1 - \frac{S_B}{D\alpha}\right) - \tanh(d_W G) \left(\left(\frac{G}{\alpha}\right)^2 - \frac{S_B}{D\alpha}\right)}{\frac{G}{\alpha} + \tanh(d_W G) \frac{S_B}{D\alpha}}$$
and $G \coloneqq \sqrt{\frac{1}{L^2} + \frac{i\omega}{D}}$, [7]

with $Z_{Si} = PI$ of the "ideal" Si substrate, $Z_{RC} = PI$ of the series resistance / capacitance circuit, and $Z_{Res} = PI$ of the external feed back circuit. The variables are: $\tau =$ lifetime of minority carriers; L = diffusion length of minority carriers; qF = intensity of illumination (expressed as photo generated charge); R = reflectivity; $\alpha =$ penetration depth of the light; $S_B =$ surface recombination velocity at the back side of wafer or solar cell; D = diffusion coefficient of the minorities; $d_W =$ wafer thickness.

The $Z_{\rm RC}$ term in Eq. [5] is given by

$$Z_{RC} = \frac{1}{1 + i\omega R_{ser}C},$$
[8]

with R_{ser} = serial resistance from the illuminated spot/area to the reference electrode; C = capacitance of illuminated spot/area. This term simply recognizes that the system has

some resistances and capacitances that is not contained in Eq. [6], stemming mostly from the series resistance of the Si and the capacitance of the space charge layer.

The Z_{Res} term due to the resonant loop of the complete control circuit can be described with sufficient accuracy by a simple and strongly damped resonant process via

$$Z_{\text{Res}} = \frac{1}{1 - \left(\frac{\omega}{\omega_0}\right)^2 + iK_F\omega},$$
[9]

with ω_0 = resonance frequency; K_F = damping constant.

The total impedance for the case of a standard Si solar cell thus is a function of 11 variables, $Z_{fsi} = Z_{fsi}(\tau, D, S_B, R, \alpha, d_W, \omega, R_{ser}, C, \omega_0, K_F)$, one of which is trivial (thickness d_W), and two of which are not of much interest (resonance frequency ω_0 and damping constant K_F).

All one has to do now is to extract the 8 parameters of interest by matching measured fsi-PI spectra to Eq. [5]. While this may appear hopeless, it is actually not too difficult as will be shown in the next section.

3.3 Some results of the CELLOplus technique

Fig. 6 shows just four maps taken from the same solar cell shown in Fig. 5. Only the quadrant indicated in Fig. 5 with the red square is shown in order to demonstrate the good spatial resolution.

Shown are maps of parameters that are either directly contained as variables in the impedance equation [5] or that can be computed from these variables in a straightforward manner. The series resistance map in **Fig. 6a**), while in arbitrary units, nevertheless shows essentially the same structures as the independently obtained CELLO map in **Fig. 5a**) and thus demonstrates the reliability of the impedance technique. A comparison with R_{SE} maps obtained with a slow and destructive needle probing method (so-called corescan technique (20)) also demonstrates the correctness of the results obtained by CELLO and CELLOplus (5).

The maps in **Figs. 6b**) – **d**) were selected because they show properties that to the best of our knowledge cannot be obtained by any other technique. It is thus not possible to demonstrate the correctness of the quantitative data by comparison with other measurements; the numbers displayed are, however, in the expected range. This is of some importance for solar cell R&D because the degree of surface and interface passivation as measured by recombination velocities is gaining much interest since it is one of the decisive parameter that need to be controlled for future high-efficiency Si solar cells.



Figure 6. Examples of CELLOplus results. a) Map of the series resistance (in arbitrary units) directly comparable to the independently obtained CELLO map in **Fig. 5a**). b) Map of the diffusion coefficient of the minority carriers. c) Map of the surface recombination velocity of the back side of the solar cell. d) Map of the doping concentration.

4. Conclusions

It has been shown that multi-mode FFT impedance spectroscopy is a powerful tool for exploring junction and bulk properties of semiconductor devices such as solar cells or current-carrying liquid-solid junctions. In particular, multi-mode FFT impedance spectroscopy allows to obtain in-situ data in situations where no other methods can be used. Properties and parameters not easily obtained by other methods thus become accessible. This is also true for devices not mentioned in this paper (e.g. electrochemical devices with semiconductors other than Si or non-Si solar cells). The method needs to be based on optimized hard- and software, a FFT technique, several measurement modes, and extensive theoretical modeling in order to convert raw impedance data to device properties. After extensive efforts to provide for these ingredients in the past, the method is now ready for large-scale applications.

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