Analysis and minimisation of plasma process instabilities during thin silicon film deposition

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ABSTRACT

With the thin-film silicon industry facing the problems of high-quality material deposition at high rates and narrowing deposition process windows, the "no-drift regime" is an important part of this development. In the case of plasma-enhanced chemical vapor deposition (PECVD) of thin silicon films, the inconstancy of the concentration of silicon-containing particles (SCP) in the plasma leads to changes in deposition conditions, causing a deterioration of film properties, therefore decreasing the performance of the solar cells. During the last few decades, evidence about the process instabilities has been accumulated in different laboratories. In this study, Fourier transform infrared absorption spectroscopy (FTIR), optical emission spectroscopy (OES), self-bias voltage and plasma impedance controls were applied as in-situ process diagnostics during the deposition of amorphous and microcrystalline silicon thin-films. Results of the study were then discussed.

Introduction

The in-situ process diagnostics clearly indicate inconstancy of plasma properties and therefore of deposition conditions. These instabilities could take several minutes and are comparable with the film deposition time. Two scales for process drifts could be identified: a strong short-term drift or initial transient state phenomena (ITS) for the first several minutes of deposition, and a long-term drift (LTD) for the whole deposition process time.

For example, a variation of the bias voltage of ±10% and of the SiH* emission intensity (6% decrease) during the first few minutes was observed. The FTIR measurement of reactant concentration in the process chamber evidence that the strong SCP concentration drop (about 50%) in a plasma is the cause of the short-term drift of OES signals (SiH* emission), plasma impedance and self-bias voltage signals. On the other hand, a weak plasma impedance change is one of the reasons for the long-term drift of OES signals and self-bias voltage signals as the silicon particle concentration in the exhaust remains constant. In this article, the influences of the deposition chamber geometry and technological parameters on process drifts are considered. It is shown that the decrease of the gas residence time in the reactor leads to a decrease of ITS, while options for active and passive process control during plasma deposition are discussed. The improvement of solar cell performance based on thin silicon films is demonstrated when drifts are reduced.

An important step in the production of Si-based thin-film solar cells is the preparation of amorphous or microcrystalline thin silicon layers by plasma-enhanced chemical vapor deposition (PECVD). The inconstancy of the concentration of silicon-containing particles (SCP) in the plasma leads to changes in deposition conditions, thus possibly decreasing solar cell performance [1,2]. In the eighties, several researchers from different laboratories showed that the PECVD process has an initial transient state phenomenon (ITS) in semi-batch reactors [3,4,5]. This is an important phenomenon, because the quality of the material is closely related to the plasma parameters and the improvement of the film interface properties is crucial for raising the performance of thin-film devices. It is clear that the interface properties depend on the history of a PECVD chamber (cross-contamination [6], chemical memory [7], remaining impurities, silicon powder etc.) and the initial transient state of the plasma.

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Both Y. Nakayama et al. [5] from the University of Osaka and Y. Ashida et al. [3] from Mitsui Toatsu Chemicals Inc. have observed the instability of emission intensities I_{SiH*} and I_H measured during the deposition process. Similar observations were made by M. N. van den Donker [1]: after starting the glow discharge, the intensity of the SiH radicals (I_{SiH*}) decreased and then leveled off. Following an increase, the atomic hydrogen intensity stabilised. Similar observations were made in a modified KAI-S large area industrial reactor ($37 \times 47 cm^2$) from Oerlikon Solar AG even when gas residence time was very small (about 1s) [8]. In each of these studies, using different reactors, the ITS of the plasma took place over times ranging from 2 to 60 seconds and, more importantly, a strong influence on the devices' response was observed. For example, Y. Nakayama et al. [9] showed that the deposition rate, hydrogen content, hydrogen bonding states and photoluminescence spectra of the a-Si: H interface layer correlated strongly with the initial transient state of the plasma and differ from those of the bulk deposited in the steady state of the plasma, especially at a high consumption of SiH_4.

Today, the production of industrial PECVD systems is growing. The backmixed reactor type is the most popular concept, because it offers good control of reaction speed, lower labor and handling costs, has a low shutdown time to empty, clean out, and refill, and an acceptable quality control of the product [10]. The backmixed reactor (semi-batch, stirred tank reactor or constant flow stirred tank reactor), a common ideal reactor type where gas is uniformly mixed, has the same composition both within the reactor and at the chamber exit. However, the inherent property of such reactors is ITS (start time).

This paper addresses the latter aspect and focuses on the origin of ITS and its influence on device fabrication.

Fab & Facilities

Materials

Cell Processing

Thin Film

PV Modules

Power Generation

Market Watch

The aims are increasing layer homogeneity in the growing direction and an increase of deposition preciseness.

Experimental details

The deposition reactor used in this work has a $30 \times 30 cm^2$ deposition area (see [11,12] for more details). The vacuum chamber volume is $4 \times 10^5 cm^3$. The system has a radio frequency (rf = 13.56 MHz) showerhead electrode jointly developed by Forschungs- und Applikationslabor Plasmatechnik (FAP GmbH, Dresden, Germany) and IEF-5 Photovoltaik at Forschungszentrum Jülich. The electrode distance was 1cm and, therefore, the plasma volume between the electrodes was about $1000 cm^3$. The rf-power feeding, process gas flow and pumping unit were optimised to provide homogeneous plasma conditions in a deposition regime.

The deposition system was also equipped with Fourier transform infrared absorption spectroscopy (FTIR) installed in the deposition chamber exhaust line with a path length of 1m between two ZnSe windows, which was used to measure the partial pressure of silane. Previously, this method had been successfully used in Centre de Recherches en Physique des Plasmas (Ecole Polytechnique Federale de Lausanne, Switzerland), for the optimisation of microcrystalline silicon deposition efficiency (gas utilisation efficiency) [13,14]. The Fourier transform of the detector signal yields a spectrum in the $600-4000 cm^{-1}$ range with an instrumental spectral resolution of $0.5 cm^{-1}$. To obtain a reasonable signal-to-noise ratio, the measurements were performed by averaging over 32 spectral scans, for static measurements (reference measurement before process start) and one scan for dynamic measurements every three seconds. To determine the SiH_4 density, a calibration curve for the integral of the Q branch versus the SiH_4 pressure was obtained from SiH_4 gas spectra at different silane partial pressures and a reactor heater temperature of 200°C. The integral limits were chosen to be from 2163 to 2199 cm^{-1}. The SiH_4 density for the different plasma conditions was then obtained from the Q branch area using the calibration curve.

Parallel to real-time control of the SiH_4 concentration in the exhaust line, optical emission spectroscopy (OES), rf – electrode self-bias voltage (V_{bias}) monitoring and plasma (together with part of the reactor) impedance control were applied as in-situ process diagnostics during deposition of amorphous and microcrystalline silicon thin films. The plasma-emitted light was guided by the optical fiber to a monochromator (type Ocean Optics HR2000). The I_{SiH^*} line at 414nm was monitored.

The rf voltage (V_{rf}) and plasma impedance (phase shift - φ) were measured with the rf voltage–current probe (Z'SCAN, Advanced Energy). The detector
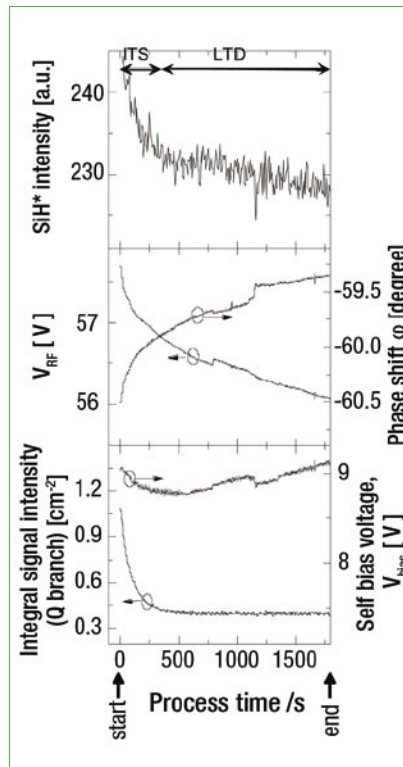

Figure 1. Time dependence of OES signal (I_{SiH^*}), of the rf probe signals (V_{RF}, φ, V_{bias}) and integral intensity of Q branch after turning on the rf discharge for regime A.

and control point of the rf bias voltage were situated as close as possible to the rf electrode. The accuracy of the V_{rf} (peak-to-peak value) measurement is greater than 0.2V or 0.2% of the measured value, while the precision is lower (about 0.4V) due to rf signal deviation in the generator output, vibration of a matching box etc. The inductance of the rf antenna between the Z'Scan probe and the powered electrode is about 0.1μH. Rf power is supplied by an rf generator (Dressler V1000) via an L-type matchbox.

The deposition parameters are described in the following section. 350nm thick p-i-n thin amorphous silicon film solar cells were deposited. Details of the preparation and measurement of the typical amorphous (a-Si:H) thin silicon film solar cells can be found in [15,16].

Results and discussion

Origin of the initial transient state

Figures 1 and 2 show variations of OES signals, of the rf probe signals and integral intensity of the Q branch after turning on the rf discharge for the different deposition regimes. The technological parameters (hydrogen, silane gas flows, rf power, heater temperature (T_h) and gas pressure in the chamber) of the regimes are presented in Table 1.

After starting the glow discharge, the I_{SiH^*} strongly decreases (-6.5%) (ITS), becomes more stable, but still shows a long-term drift (LTD). The initial transient time is defined as a time when the FTIR integral
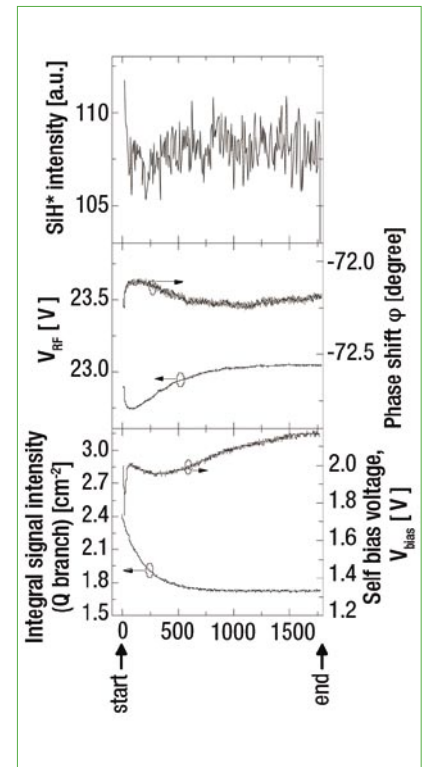

Figure 2. Time dependence of OES signal (I_{SiH^*}), of the rf probe signals (V_{RF}, φ, V_{bias}) and integral intensity of Q branch after turning on the rf discharge for regime B.

intensity signal becomes 102.5% of its stable value. It is important to note that a linear relation between SiH* emission and SiH_4 density is usually assumed, since excited SiH radicals (denoted as SiH*) originate from the electron-impact dissociative excitation of SiH_4 [17].

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ITS and LTD effects are also observed in other in-situ signals. Inconstancies of the rf voltage, plasma impedance and self bias voltage on the powered electrode are observed. It is interesting to note that in both deposition regimes, rf voltage and phase shift signals show opposite trends.

The ITS in the in-situ signals is longer for regime B compared to regime A (about 530 s vs. 370 s). Although the relative change of V_{rf}, φ and V_{bias} signals is not so strong as for OES, these changes cannot be ignored because even a small change of V_{rf} on an electrode has a very strong, nonlinear influence on ionisation degree and electron energy in the plasma [18].
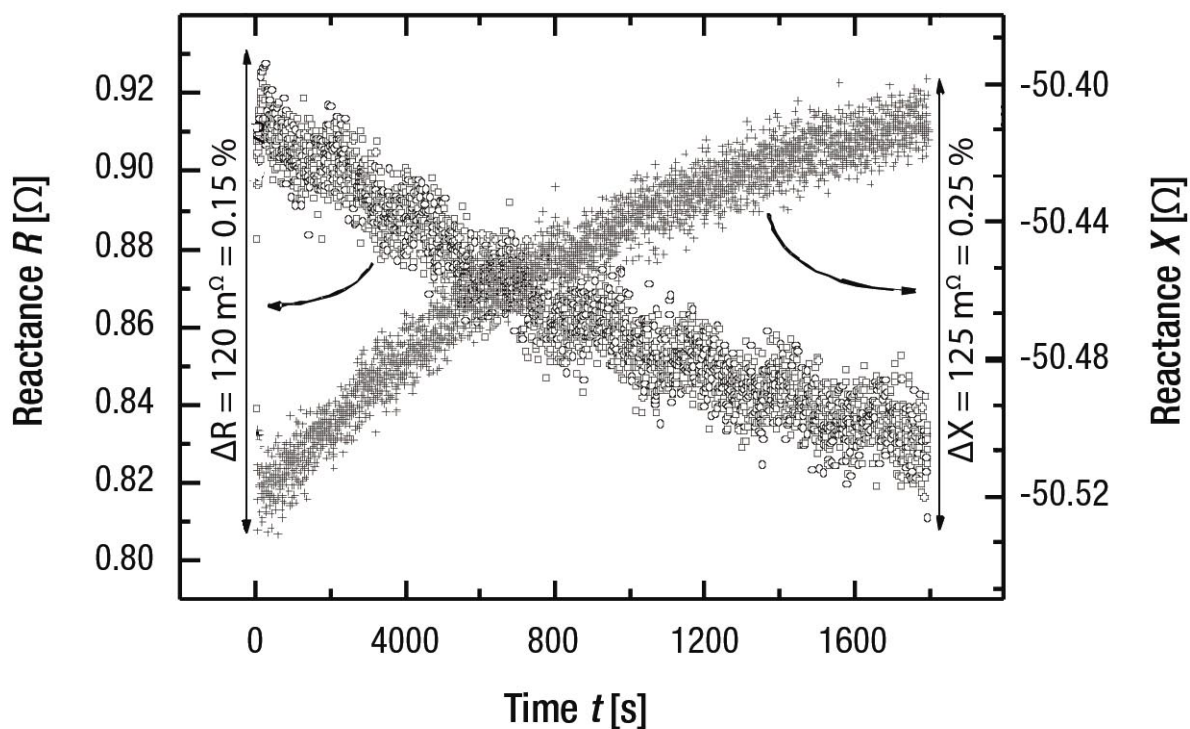
Figure 3. Resistance and reactance during 30 minutes' measurement in vacuum. The characteristic is reproducible.

For example, a good correlation is found between the character of drifts of the OES signal and V_{rf} in Figures 1 and 2, because the SiH* intensity depends not only on the SCP concentration in the plasma, but also on the electron energy and density [19], therefore depending on the rf voltage applied. In turn, inconstancy of a V_{bias} signal is an additional fact showing the inconstancy of deposition conditions.

The FTIR signals in Figures 1 and 2 clearly demonstrate that the SCP concentration is stable after ITS (after about 400 s under regime A and after about 600 s under regime B). This confirms observations made earlier [3,4,5,9] that the initial stabilisation time depends on the variation of silicon-containing radicals and polymers in $SiH_4 + H_2$ plasma rather than on the other parameters. Therefore the ITS is a drift of plasma parameters due to the drop in silane concentration in the plasma.

Nevertheless, the LTDs were observed when the SCP concentration in the plasma was stable. It is very likely that the gradual reactor heating could be a reason for LTD, because increasing wall and gas temperatures have a strong influence on plasma properties [18.] and therefore on plasma + reactor impedance (see inconstancy of impedance signals in Figures 1 and 2). Another reason for LTD could be the phenomena of silicon powder formation and growth in the plasma bulk, topics that have been extensively investigated recently [20,21,22,23].

The next experiment demonstrates the influence of reactor heating on reactor impedance. Figure 3 shows the instability of reactor impedance (without plasma ignition). A decrease in the resistance

of about 15% and a rather small increase of the absolute reactance of 0.25% are observed for the 30-minute experiment in the small-area rector (about 10L reactor volume). The relatively small change of the reactance appears negligible, but the decrease of the resistance seems to be considerable. Since the plasma is not

ignited, the delivered electrical power of 20W is only dissipated in the stray elements of the reactor. It is likely that parts of the reactor are heated up and the contact resistances between different elements (screws, for example) determine the measured resistance. Since the components of the reactor expand during
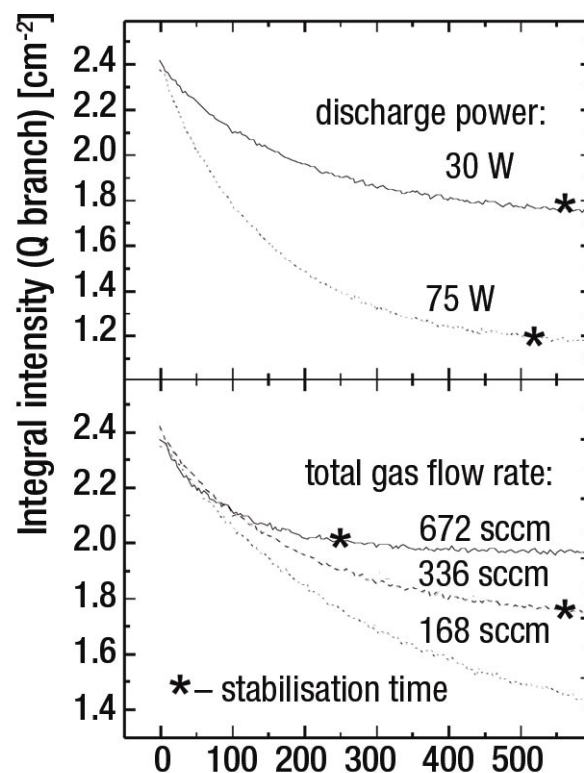

Figure 4. FTIR signal after turning on various rf powers (a) and for different total gas flow rates (b).

heating, the contact could improve and the measured resistance could decrease. It is important to note that during deposition this effect of rf power heating plays a smaller role because the resistance of the plasma is much higher and the major part of the power dissipates in the plasma. Nevertheless, the experiment shows the influence of reactor heating on rf matching.

Influence of the technological parameters on the initial transient state

Figure 4 shows the ITS for different power and total gas flow rate regimes. The inconstancy is shorter when the rf power and the total gas flow rate are higher. Figure 4(a) demonstrates that the variations of SCP concentration in the reactor depend on the degree of SiH_4 consumption since this value strongly depends upon the discharge power.

Figure 5 shows the ITS time plotted as a function of the gas residence time in different reactors and under different deposition conditions (substrate temperature, process gas pressure). The gas residence time is the time required to process one reactor volume of feed measured under specified conditions [10]:

$$t = \frac{V}{v_0} = \frac{(reactor\ volume)}{(volumetric\ feed\ rate)} \quad (1)$$

Figures 4 and 5 clearly demonstrate that the increase of the total gas flow rate (decrease of residence time) is effective in order to decrease the transient time; interestingly, in all cases the ITS time is about four times longer than the residence time in the deposition chambers. This means that equilibrium or steady-state conditions result when reactor volumes are refreshed about four times by the gas feed.

It is important to note that since the gas volumetric feed rate depends on the temperature of the gas and the pressure in accordance with the ideal gas law, the decrease of process temperature and increase of total gas pressure in the reactor should lead to an increase of ITS magnitude.

The decrease and leveling off of silane density in the exhaust line (see FTIR signal, Figures 1 and 2), decrease of ITS time with increasing silane consumption in the reactor (Figure 4(a)) and dependence upon gas residence time (see Figure 4(b)), all imply that a variation of SCP concentration at the plasma region is the main origin of the initial transient state period. These findings correlate with the result for pure SiH_4 [5] and CH_4 [24], which found that the consumption of reagent plays an important role in the ITS phenomenon. Unfortunately, the shortening of ITS time by the decrease of the gas residence time (increasing total gas flow rate) conflicts with the problem of increasing silane consumption rate in industry.

Influence of ITS on amorphous thin-film silicon solar cell fabrication

The ITS is harmful to the deposition of films with high requirements for uniformity of composition. Thus the transient depletion of the initially present SiH_4 source gas induces the formation of an amorphous incubation layer that prevents crystallite nucleation in the i-layer, leads to poor microcrystalline solar cell performance [2,25,26] and induces low-quality material deposition at the sensitive p-i interface of amorphous silicon solar cells [3]. As shown above, an effective way of reducing ITS is to shorten the gas residence time. This parameter could be reduced by: (i) increasing the total gas flow rate, (ii) using a PECVD system with a small reactor volume, or (iii) decreasing the process pressure. Unfortunately, reducing the gas residence time is restricted within a rather narrow process window. Using a PECVD system with a small reactor volume and/or using a shutter needs the reconstruction or even a completely new design of the PECVD system.

A possible solution is a silane profiling method (also known as continuous plasma method (CP) [27]). Under this condition, the process was started by using H_2 plasma, after which SiH_4 was fed in, keeping the same H_2 flow used to ignite the plasma. The p-i-n structure of thin solar cells has a sensitive p-i interface located at the interface layer, which is usually deposited in the ITS regime. Therefore, it is quite likely that the performance of this type of solar cell is sensitive to the ITS of the plasma. The deposition conditions of amorphous silicon intrinsic layer (a-Si:H) with a thickness of 350nm was regime B (see Table 1). The usual solar cell characteristics and the solar cell characteristics when the continuous plasma method was used are shown in Table 2. As compared to cells
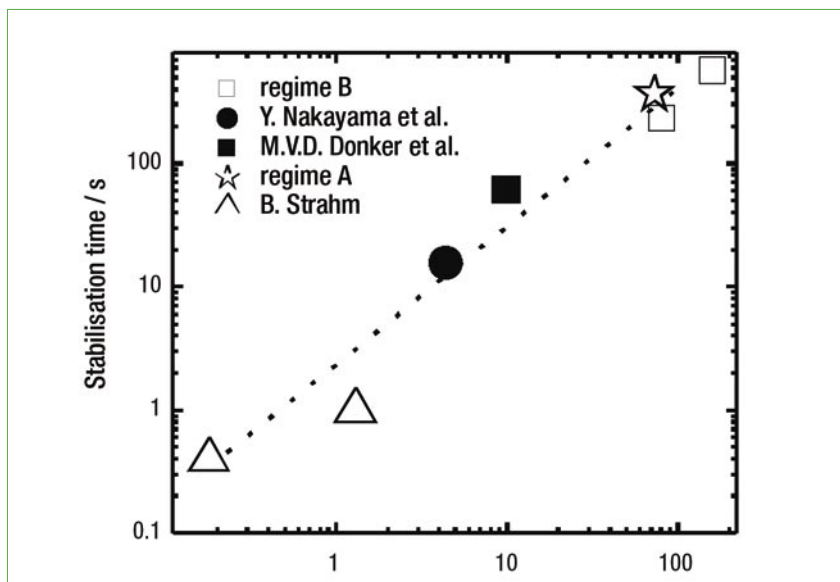

Figure 5. Stabilisation time (ITS) of I_{SiH^*} plotted as a function of gas residence time in different deposition chambers. Gas residence times were calculated for specified conditions described in [1, 5, 8].

Deposition regime:	H_2 (sccm)	SiH_4 (sccm)	rf power (W)	T_h (°C)	Pressure (Pa)
A	2000	20	400	150	1065
B	300	36	30	180	400

Table 1. Deposition conditions of regimes A and B. H_2, SiH_4, and T_h are flows of hydrogen, silane and substrate temperature, respectively.

Deposition regime:	η (%)	FF (%)	V_{oc} (V)	J_{sc} (mA/cm^2)
No CP method	9.45	68.9	0.91	15
CP method	10	72	0.93	15.1

Table 2. Performance of a-Si:H solar cells with and without the continuous plasma deposition method. Solar cell parameters are: cell efficiency (η), fill factor (FF), short-circuit current (J_{sc}) and open-circuit voltage (V_{OC}) (the maximum current and voltage, respectively, from a solar cell). The FF is defined as the ratio of the maximum power from the solar cell to the product of V_{oc} and I_{sc}.

produced without CP, the efficiency of cells produced by the silane control method was higher due to the higher fill factor (FF).

These results indicate that the initial plasma instability period leads to the deposition of a low-quality (void- and defect-rich) interface layer. Experiments show that the CP method used to minimise the ITS in the plasma deposition of this silicon layer plays an important role in the uniform composition of material, and consequently in the performance of devices.

> "The main reason for plasma instability is a drop in the silane concentration of the plasma."

Conclusions

The origin of the initial transient state in the silane plasma is investigated using optical emission spectroscopy, plasma impedance monitoring and FTIR of exhaust gases. The main reason for plasma instability is a drop in the silane concentration of the plasma. The instabilities may take several minutes and are comparable with the film deposition time. Two scales for process drifts could be distinguished: a strong short-term drift (ITS) for the first several minutes of deposition and a long-term drift (LTD) for the whole deposition process time. By using the silane profiling method, the influence of the ITS on the fabrication of thin silicon film amorphous solar cells is shown. An improvement in solar cell performance of 5% is demonstrated when drifts are reduced.
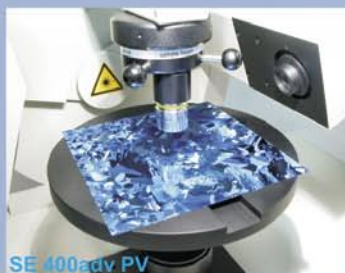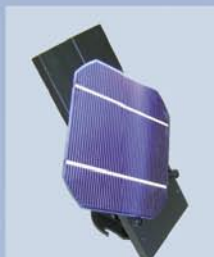
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Dr. Dzmitry Hrunski received his diploma degree in design and technology of radio frequency devices from the Belorussian State University of Informatics and Radioelectronics in Minsk in 1992. He obtained his Ph.D. from the same university on the topic of "Deposition of a-Si:H films in two frequency discharge" in 2001. He has been working in field plasma technology and thin silicon film deposition since he began his diploma, and in 2002 he joined Dr. B. Schroeder's research group in Kaserslautern as a post doc to learn the Hot-Wire CVD method. He has been working on in-situ diagnostics and optimization of PECVD processes at the Research center of Jülich IEF-5 Photovoltaik since 2005.

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Uwe Stickelmann carried out his diploma work at Forschungszentrum Jülich GmbH, where he worked on in-situ impedance analysis in PECVD systems. His main focus was on plasma process diagnostics during the fabrication of thin silicon films. After completing his graduate studies in electrical engineering in 2008, he joined the development department of SMA Solar Technology.

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Wolfgang Appenzeller obtained his engineering degree in 1975 from Fachhochschule Aachen, Germany. From 1975 until 1991 he worked as an engineer in the University of Düsseldorf, Institute of Applied Physics, Germany. Since 1991 he has been working at Forschungszentrum Jülich GmbH, IEF-5 Photovoltaik. He is responsible for maintenance and developing PECVD and sputter systems and since February 2002 has been working on $30 \times 30 \text{cm}^2$ substrate size solar module technology.

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