Monitoring and controlling possibilities in wet chemical etching processes for c-Si solar cell production

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ABSTRACT

Quality assurance and process control are becoming increasingly important in the industrial production chain to the manufacturing of silicon solar cells. There are a number of relevant wet chemical processes for the fabrication of standard screen-printed industrial solar cells, mainly for texturization and cleaning purposes. While one-component systems like pure HF for oxide-removal are easy to monitor, i.e., by conductivity measurements, typical texturization processes are much more complex due to the number of constituents.

For acidic texturization of multicrystalline silicon wafers, typical mixtures involve amounts of hydrofluoric acid (HF), nitric acid (HNO₃) and water. It has also been documented that mixtures can be found where additional additives like phosphoric acid (H₃PO₄), acetic acid (HOAc) and sulphuric acid (H₂SO₄) have been used [1, 2]. In alkaline random pyramid texturization for monocrystalline wafers, a base like potassium hydroxide (KOH) or sodium hydroxide (NaOH) and organic additives like 2-propanol (IPA) are used [3]. In addition to these processes, recently developed high-efficiency cell concepts require several additional wet chemical process steps like advanced cleaning processes, chemical edge isolation or single side oxide removal processes [4].

In order to obtain continuously stable and reproducible process results and to overcome process operations based on operator experience, a reliable monitoring of the bath concentrations is essential. Such quality control has the potential for significant cost reductions due to optimized durations between replacements of bath mixtures or shortening of processing times. In this context, the application of on-line analytical methods, either by means of chemical, optical or electrical measurement techniques, is of particular interest.

Introduction

Wet chemical etching processes represent standard procedures in modern batch or inline-based production lines for crystalline silicon solar cells. However, continuous and accurate process monitoring is still not widely used, while physically measurable parameters like temperatures, gas flows or process pressures are continuously measured.

Similar to other relevant production steps, significant cost reduction might be achieved by continuous process monitoring resulting in:

- an increase in process quality and stability which results in an increased overall production yield; and
- optimized durations between replacements of bath mixtures or shortening of processing times.

Process monitoring in the case of wet chemical processes necessitates the use of inline (or even online) bath analysis methods, which would overcome modern typical operation based on operator experience. As an example of the importance of an elaborate process control, chemical consumption data for a typical acidic texturing process can be taken; assuming an average industrial bath operating time of around 80h, the amount of dosed HF and HNO₃ during that time period accumulates to a factor of 10 to 15 higher than the original amount of HF and HNO₃ used for the fresh bath make-up. These figures demonstrate that basically the overall process performance is mainly driven by an accurate dosing of the consumed chemicals and therefore their exact determination is of great importance.

Additionally, from a more scientific point of view, a better understanding of the underlying mechanisms especially for the texturing processes could be achieved, which will ultimately support further process optimization.

"Continuous and accurate process monitoring is still not widely used."

Only a few applications exist for process monitoring, already exist, mainly for the alkaline or acidic texturing processes, an overview is given in Table 1. Titration is a very robust and widely used method for the determination of acids and bases, but also allows the concentrations of oxidizing agents like hydrogen peroxide are measurable [5, 6]. Maintenance and calibration costs are quite low, since most of the used stock solutions are commercially available. The measurement cycle is in the range of 10 to 15 minutes. A constraint for the usage of the titration is the limited number of components that can be detected simultaneously. For example, the acidic texturization involves the inclusion of additional titration steps for the analysis of all components. The determination of organic components like 2-propanol is not possible.

A more fast and flexible method is chromatography, where inorganic acids and bases and organic additives are measurable, with measurement cycles are commonly shorter than 10 minutes [7]. Commercially available high-end equipment has a high automation level, which reduces the running costs to a minimum.

A real inline system is shown via NIR spectroscopy [8]. Since NIR spectroscopy is a non-selective method, interactions between the measured components as well as the influence of varying temperatures have to be included into the calibration dataset. This leads to a high workload for the calibration, but calibration models for most purposes in photovoltaic industries are commercially available. After setting up a calibration model, there is very little need for further maintenance. Modern FT-NIR (Fourier Transform) instruments reach a measurement cycle of 30 seconds so that NIR-spectroscopy enables a real-time process control.

For surface active components like 2-propanol, determination of the air-to-

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	Detectable species in acidic etching mixtures	Detectable species in alkaline etching mixtures
Titration	HF, HNO ₃ , H ₂ SiF ₆ [Henßge 2007] / [Weinreich 2007]	KOH, K ₂ Si(OH) ₂ O ₂ , K ₂ CO ₃ [Grosvenor 1982]
HPLC/IC	HF, HNO ₃ , H ₂ SiF ₆ , HNO ₂	2-Propanol
Surface tension		2-Propanol
NIR-Spectroscopy	HF, HNO ₃ , H ₂ SiF ₆ , HNO ₂ * *addition of UV/Vis channel	KOH, K ₂ Si(OH) ₂ O ₂ , 2-Propanol

Table 1. Overview of existing inline/online analysis techniques for wet chemical process baths.



chemically textured silicon surfaces. The different techniques can be carried out either by means of inline or offline measurement tools.

liquid surface tension represents a viable measurement method [9].

In order to establish a closed-loop process control methodology, all relevant interdependencies between chemical bath compositions and effects on the final solar cell performance, have to be determined. Fig. 1 represents the step-by-step methodology for final optimization of the solar cell's output performance, which is based on an exact inline/online control of the most important process parameters. Such a procedure is principally transferable to any kind of technology step; in our study we applied this approach to wet chemical texturing processes. In order to reach an optimized texturing process, first of all the most suitable surface morphology of the designated surface texture has to be determined. This includes structural information about the resulting surfaces (reflectance, feature homogeneity and sizes, roughness) as well as information on the resulting electrical quality of the surfaces. All relevant process parameters (e.g. concentrations, process time, temperature, etc.) influencing the final





surface morphology need to be identified and control strategies for these parameters need to be defined. For continuous process control in modern production lines, especially online measurement, techniques are of special interest. When measuring the chemical bath components, inline chromatography or spectroscopic methods are usually applied. Optical control of the textured surfaces is performed by means of surface reflectance measurements or simple optical scanning of the wafer surface. Electrical analysis involves either lifetime measurements of the textured surfaces (typically only local information) or camera-based analysis techniques like PL (photoluminescence) imaging [10].

In the following sections we will focus on inline/online control possibilities of the main bath components for alkaline and acidic texturing processes, and will discuss how concentration variations influence the final texturing result.

Alkaline texturing process

Alkaline etching with sodium hydroxide (NaOH) or potassium hydroxide (KOH) has different etch rates for different crystallographic orientations. Hence this anisotropy results in small pyramids with a square base randomly distributed over the wafer surface for monocrystalline silicon wafers with <100> orientation. To improve the lateral uniformity and the anisotropy of the etching process, isopropyl alcohol (IPA) is added to the etching solution. After texturing the wafers are typically cleaned in hydrochloric (HCl) and hydrofluoric (HF) acid with intermediate rinsing in DI water. Alkaline texturing is typically performed within batch processes, where wafers are held in carriers that allow chemicals to wet the entire surface. For standard process control, the carriers are weighed before and after etching to determine an average etching depth. Typical process temperatures range between 70 and 80°C, which is close to the boiling point of the IPA (82°C). Constant evaporation of IPA occurs during the etching process, which represents a major process uncertainty and results in the need for regular redosing. In order to simplify the redosing of the additive and to get a higher reproducibility of the initial IPA concentration, it should be measured and controlled.

The concentration of organic molecules like alcohols in aqueous solutions can be analyzed by high-performance liquid chromatography (HPLC). The IPA concentration of an alkaline wet chemical bath used during a solar cell process can be measured directly without dilution by HPLC. The alkaline sample is separated while IPA is retarded and the inorganic components are accelerated compared to water. The IPA peak area is used to relate to the IPA concentration. IPA is a surface active compound which lowers the surface tension of air in the liquid. When surface tension and IPA concentration of the bath solution are measured during a texturing process, the surface tension follows the IPA concentration reciprocally proportionally. The surface tension of an alkaline texturing bath solution increases with decreasing IPA concentration. The decreases in IPA concentration can be seen in Fig. 2 as the surface tension increases during a texturization process [9].

"The evaporation rate depends not only on the concentration but also on the etch rate."

In order to reach the initial IPA concentration, the IPA has to be redosed. Different dosing amounts were tested. The IPA redosing is lower for run four than for run six in Fig. 2, resulting in a lower IPA concentration. As the incorrect amount of IPA is redosed, the initial IPA concentration is different for different runs. The reflection of KOH/IPA textured wafers is usually high as the surface tension of the etching solution and the etch rate are also high. However if the correlation between IPA concentration and surface tension are known, the exact amount of IPA can be dosed while



Figure 3. IPA concentration analyzed by HPLC and IPA concentration calculated from surface tension in alkaline texturing solution with correct IPA dosing.

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knowing the actual surface tension. The initial IPA concentration can be reached for several runs as IPA is redosed in the correct amount (Fig. 3, run two to six). The target concentration was the initial IPA concentration of run two. The IPA bath condition is well reproducible for several runs (Fig. 3).

Rather than redosing per run, redosing per time is also possible. As the time in between redosing gets shorter, the IPA concentration stays constant or increases because the evaporated IPA is replaced; however it is difficult to dose IPA accurately without a real online control parameter. The evaporation rate depends not only on the concentration but also on the etch rate. This means that a wrong redosing leads to high concentration variations. Therefore an IPA redosing was developed that was dependent on the surface tension (Fig. 4) [9]. The IPA dosing is controlled by a proportional-

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Figure 4. Surface tension measurement set-up. Right: HPLC as reference measurement.



integral-derivative (PID) controller, reading a constant IPA level during a texturing process as shown by the HPLC data in Fig 4. When the surface tension target value was changed to a higher value, the dosing stops until the actual surface tension value corresponds to the target surface tension.

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The following texturization results were obtained using the IPA controller

for a constant IPA concentration in the bath solution. Different KOH and IPA levels during texturization reveal different textured surfaces (Fig. 5). A low IPA concentration level leads to small pyramids with a few big pyramids in between. A low KOH concentration in the bath solution coupled with a high constant IPA concentration results in some flat pyramids





and a high weighted reflection of the wafer surface is high (ca. 25%, Fig. 5, right).

The flat parts between the pyramids lead to this high reflection. For a high KOH concentration, the standard deviation of the height data taken from confocal microscope pictures is higher than for low KOH concentration (2.0µm vs. 1.6µm). A high KOH and IPA concentration results in more homogeneous pyramids sizes. The pyramids on the textured wafer surface are relatively small (< 5µm wide and < 3µm high, Fig. 5, right).

In order to control all components of the alkaline etching bath with one measurement method, an NIR spectroscopy model has been developed for this process [11]. In contrast to most inorganic components like KOH, IPA has a separate –CH-group with separate vibration modes, which enables a very precise calibration for this component [16]. The model was created in a temperature range from 60°C up to 80°C under laboratory conditions. The transfer to the process tool required an offset for IPA and silicate as well as a slope and bias adjustment for the KOH concentration.

The IPA concentration measured with NIR spectroscopy shows the expected good correlation with the IPA concentration from the online HPLC-measurements. The concentration increases in the beginning of the process due to the initial IPA dosing and decreases during the process to a level of 70% of the initial concentration (Fig. 6).

Acidic texturing process

Acidic texturization, as an isotropic process, does not depend on the crystallographic orientation and thus is most suitable for multicrystalline silicon. Typically, a solution of HF, HNO₃ and water is used on an industrial scale either in batch or inline type production equipment. For the more common inline application, the wafers move horizontally on rolls through different tanks. A typical sequence involves the texturing itself, lowly concentrated KOH for porous silicon removal, HCl and HF clean and air drying, between chemical treatments. Spray rinses are used to minimize cross-contamination and to stop chemical reactions.

The concentrations of HF, HNO_3 and H_2SiF_6 are accessible by titration in two steps. The first step is an acid-base titration, which delivers the total acid amount as well as the concentration of dissolved silicon. This is followed by a precipitation titration. The concentrations of free HF and HNO_3 are calculated from the total fluoride concentration, the total acid concentration and the dissolved silicon. The detection of dissolved N(III)-species is not possible.

A more flexible measurement technique for the analysis of components in acidic texturization baths is ion chromatography. After the separation of the ionic species in the chromatographical column, fluoride, nitrate and nitride ions are measured in a conductivity channel; the detection of dissolved silicon is not possible by conductivity measurement. Therefore a post-column derivatization with sodium molybdate was used in order to form a yellow reaction product that can be measured in a UV/Vis-detector.

An advantage of ion chromatography in contrast to titration is the ability to monitor the concentration of N(III)-species. A detection of these species is also possible with spectroscopic methods, when the instrument allows an extension to the UV/ Vis range, where the nitrogen oxides occur in the range of 340nm–400nm.

Near-infrared (NIR) spectroscopy is a suitable method for the concentration monitoring of acidic texturization baths. The method is based on the observation of overtone and combination vibration bands of water. The position as well as the shape of these bands is influenced by the temperature and by the presence and concentration of ionic species like HF, HNO₃ and H₂SiF₆.

A partial least squares (PLS) model was built for the determination of the HF-, HNO₃ and H_2SiF_6 concentration. The measurement accuracy of the model was estimated from a cross validation. The standard error of validation was 1.8g/l for



HF, 3.5g/l for HNO₃ and 1.8g/l for H₂SiF₆. This model was used to determine the bath composition of the acidic texturization bath twice a minute.

The control algorithm was written in LabVIEW and the communication to the FT-NIR spectrometer was achieved via OPC protocol. The dosing events were transferred to the PLC of the process tool via an optocoupler interface card. A rising edge on the PLC triggers the dosing of a variable volume of the selected chemistry. The PLC responds a signal indicating the business of the dosing unit.

The used NIR model was validated with reference measurements achieved by inline ion chromatography. Over a time period of one week, a slight change in the offset was observed, which necessitated a further optimization of the NIR hardware. The developed dosing algorithm was used to control the concentrations of HF and HNO₃ in dependence of the etched silicon amount over one process day (Fig. 7). The algorithm was able to stabilize concentrations the resulting in a deviation of the etching rate of only 2% relative.

The main influencing parameters on the texturization quality are the educt concentrations as well as the concentration of the hexafluorosilicic acid (H_2SiF_6) that is generated during the etching process. H_2SiF_6 is an acid that enlarges the oxidation potential of HNO₃ by moving the chemical equilibrium between N_2O_3 and NO⁺ towards the nitrosyl cation. H_2SiF_6 also etches silicon dioxide, leading to the conclusion that the increasing



Figure 8. Correlation between total acid concentration, resulting etch rates and surface morphologies. High acid concentrations result in flat, more polished surface structures, whereas lower acid concentrations result in a surface roughening with preferential etching of crystal defects.

concentration of H_2SiF_6 has to be compensated by a decrease of the HF- and HNO₃ concentration (Fig. 7).

Too high an acid concentration leads to a smooth surface with a high reflectance, while too low concentrations result in very rough surfaces that are difficult to passivate. A reason for the low efficiencies at low reflection might be the presence of etch attacks at grain boundaries (GBs), which tend to occur strongly, under the etching conditions that are needed for a low reflecting texture. Due to these etch attacks, v-shaped trenches are formed at GBs. These trench structures are barely deeper than ordinary etch cavities, but they are very steep, while still being narrow (ca. 3μ m). Because of the steepness of their facets, incident light is reflected to the opposite side leading to another chance for absorption (double-bounce effect). Therefore, trench structures appear as dark lines on the wafer surface; on a macroscopic scale, these lines appear in ball-of-wool-like clusters.

Trench structures are more preferably formed at a low etch rate than at a high etch rate. Therefore one possibility for reducing the fraction of the surface area covered by trench structures is to implement an accurate control of the etch activity, e.g. etch rate, of the texturing bath.

The appearance of trench structures on the wafer surface has been mentioned in earlier publications and is often recognized as harmful to solar cell performance. Trench structures could lead to shunts after firing as the passivation-layer inside them is thinner. Furthermore, the solar cell surface area is enhanced by trench structures leading to more surface recombination. Wafer breakage is also increased by the appearance of trench structures [12]. On the other hand, trench structures offer an impressive reduction of surface reflection which might compensate some of their negative effects.

A method for in-line measurement of preferred grain boundary etching caused by acidic texturization (trench structures) uses a line camera to scan wafers moving on a conveyor belt [13]. The system provides the required diffuse illumination and still measures sharp and contrast-rich images [14].

The number of trench structures on these images is quantified by a newly developed automatic algorithm (Fig. 9, left). The algorithm uses a threshold value to distinguish trench structures from the surrounding texture. All pixels that have a grey value below the threshold value are counted as trench structures, the other pixels as texture. As the threshold value is linearly dependant on the wafer reflection/ brightness, it had to be determined via an appropriate calibration with a set of texturized wafers.



Figure 9. Left: Marked rift structures determined by a colour vision system on a textured multicyrstalline silicon wafer. Right: Relation between material quality and texture: the fraction of rift structure area f_R on the wafer surface as well as the fraction of crystal defect area determined by as-cut photoluminescence images has been quantified using a newly developed algorithm.

By regarding the measurement method in detail, it could be shown that the true area fraction of trench structures (fT) only lies between 0.3 and 1.8%. This is only 15% of the value expected from looking at the wafer or from camera images. The size of trench structure areas seems to broaden because the camera system has a much lower resolution (a pixel has a length of 130µm) than the width of trench structures (<5µm). The measurement method, as well as the algorithm, were tested by measuring fT for 150 multicrystalline wafers with a broad variety in the number of trench structures and comparing the results to manually obtained results. The variety in the number of trench structures was obtained by choosing wafers representing a typical cross-section of industrially available mc-Si materials and texturizing them with two different textures: an acidic texture with strong etch attacks and an acidic texture with weak etch attacks. The number of trench structures strongly depends on the number of crystal defects in as-cut wafers and on the texture strength (Fig. 9, right). The fact that the expected correlation could be reproduced well indicates the viability of the measurement method.

The strong dependence of fT on material quality and texture strength indicates that the number of trench structures can be reduced by choosing an appropriate texture, e.g. an adjustment of the chemical concentrations. If trench structures are as harmful as reported, their prevention could result in an increase in solar cell efficiency.

Conclusion

An online process control of acidic and alkaline wet chemical texturization processes was successfully demonstrated with different analysis methods. In the case of alkaline texturing processes, the online bath control was directly utilized to control the dosing of 2-propanol during the process. By stabilizing the 2-propanol concentration, more homogeneous pyramidal surface structures as well as a higher process-to-process reproducibility was achieved.

In the case of acidic texturing processes, either inline ion-chromatography or nearinfrared spectroscopy might be used for online bath control. Beside the accurate control of the chemical concentrations, optical as well as electrical analysis of the resulting surface morphologies is necessary in order to obtain an optimized texturing process. Depending on the original material quality (especially the appearance of crystal defects), the etching solution has to be adjusted to avoid the formation of rift structures. A closed-loop process control therefore necessitates the material analysis by means of photoluminescence imaging prior to the texturization process and the control of the chemical components during, as well as an optical analysis of the resulting surfaces after, the process itself.

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