

A cheaper, faster and greener solar panel manufacturing process

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ABSTRACT

The cleaning performance of three different fluorine-containing precursors – sulphur hexafluoride (SF_6), nitrogen trifluoride (NF_3) and molecular fluorine (F_2) – is compared from theoretical, experimental and commercial points of view. Experiments were performed using an Oerlikon Solar KAI Gen 5 (1300mm × 1100mm) R&D platform. For the experiments with F_2 , an ‘on-site/on-demand’ generator from The Linde Group was installed at the Oerlikon Solar facility in Trübbach, Switzerland. The SF_6 -based cleaning process was found to be up to 75% less efficient than the corresponding NF_3 or F_2 process. A comparison between NF_3 and F_2 indicates that a significantly larger process window is available for reactor cleaning when F_2 is used in place of NF_3 . This leads to both time and gas mass savings, improving productivity and bringing down the cost of ownership of the reactor cleaning process. As a direct consequence, Oerlikon Solar has decided to transfer the process to their production KAI MT plasma-enhanced chemical vapour deposition (PECVD) platforms.

Introduction

The removal of deposited silicon in a plasma-enhanced chemical vapour deposition (PECVD) chamber is an essential step for thin-film Si PV production. In situ cleaning using gases containing fluorine (F) is a widely adopted process that offers many advantages over mechanical cleaning: the latter requires scheduled downtime of the equipment, resulting in higher operational costs and lower throughput. The established cleaning gases of choice for PECVD chamber cleaning are fluorine-containing gases, such as nitrogen trifluoride (NF_3) and sulphur hexafluoride (SF_6). From these gases, fluorine radicals are created; these then react with the unwanted silicon deposits, creating volatile SiF_4 , which is subsequently pumped out.

“The removal of deposited silicon in a PECVD chamber is an essential step for thin-film Si PV production.”

As the chamber cleaning process represents a significant part of the gas cost

for the PECVD process, manufacturers have been looking for more cost-effective and environmentally friendly alternatives, as both of these greenhouse gases have high global-warming potential. This paper presents the progress made in the implementation of molecular fluorine (F_2) as the cleaning gas on the Oerlikon Solar KAI platform.

Theoretical comparison of processes that use SF_6 , NF_3 and F_2

Fluorine radicals are created either by in situ RF activation or by using a remote plasma source (RPS). The latter technique has been implemented during the development of NF_3 as a cleaning gas, in order to achieve full usage of the molecule at high flows of the precursor gas. As will be seen later, this is a requirement for an efficient NF_3 -based cleaning process, but not essential when molecular fluorine is used.

The cleaning reactions will first be reviewed from a theoretical point of view, independently of activation technology or recipe development. Understanding the possible reaction pathways will allow the precursor to be identified for which cleaning is expected to be more efficient. Recipe development and a clean performance

comparison on a KAI R&D platform will be discussed in the last part of this paper.

1. The active radicals

The plasma created during the cleaning process of a PECVD reactor is a very complex medium. It contains neutral atoms and molecules, and different types of ions, electrons, photons, neutral molecular fragments, etc. It is believed that the PECVD cleaning processes used by Oerlikon Solar are mainly driven by neutrals (atoms, molecules and molecular fragments), and not by ions, for the following reasons:

- The KAI PECVD reactors are almost symmetrical, leading to very low DC bias values, so that the positive ions receive little acceleration in the plasma sheath.
- The cleaning processes occur at pressures above 0.3mbar, and, under some conditions, pressures higher than 1.0mbar are used. In such cases the energy of positive ions reaching the surface is very much reduced because of collisions with other molecules in the plasma sheath.
- Measurements show that the ion density in such plasmas can lie below $1\text{E}+10\text{cm}^{-3}$, but in order to achieve the observed etching rates of several tens of angstroms per second, a density of active radicals greater than $1\text{E}+14\text{cm}^{-3}$ is required. This is the same order of magnitude as the densities of neutral species and not of positive ions.

Since the number of negative ions is lower than that of positive ions, it can be concluded that the cleaning processes in the Oerlikon Solar chamber are driven by neutral species and not by ions.

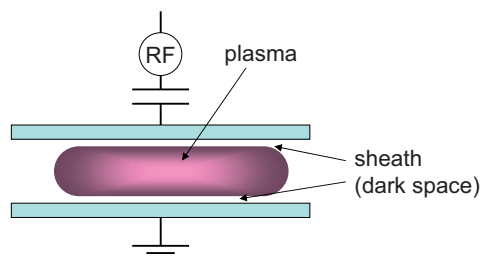


Figure 1. Plasma sheath.

The most important cleaning radical is atomic fluorine (F) [1]. The fluorine atoms reaching the surface react with the deposited silicon, forming SiF₄, which is volatile and is in turn pumped away:



Although less important than atomic fluorine F, another neutral – molecular fluorine F₂ – also plays a role in the cleaning process [2], via reactions such as:



The reaction pathway for the creation of those neutrals from the different fluorine-containing gases that are used as the precursor will be discussed next.

2. Creation/destruction of active radicals

The main reaction pathway for the creation of atomic fluorine, in plasma, is via electron dissociative attachment [3,4]:



The activation energy for these reactions is very low (below 1eV), so the likelihood of them happening is very high. The corresponding neutral dissociation has a higher energy threshold (several eV) and is therefore of less importance.

The negative ions are further neutralized through ion–ion recombination:



where 'I' might be SF₅, NF₂ or F₂ (or one of the smaller radicals).

Another neutralization mechanism goes through detachment, for example:



where 'J' might be SF₅, NF₂ or F₂ (or one of the smaller radicals).

It can be concluded that in all cases it is fairly easy to dissociate the first fluorine atom from the precursor molecule; this is the main reason why all of these precursors can be (and are) used.

In contrast with the creation of the first fluorine atom, the mechanisms for destruction of the cleaning radicals are very different for the said three precursors, and this fact leads to significant differences in cleaning performance.

SF₆ plasma

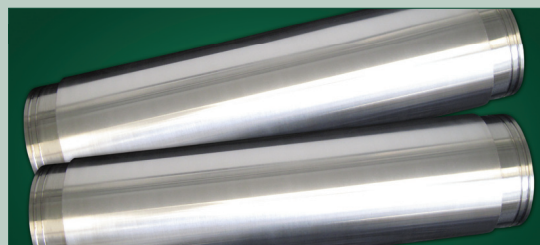
In the case of SF₆ there is a very strong recombination reaction channel of the form:



where x = 0, 1, ... 5. This reaction is so rapid that the resulting density of atomic fluorine in SF₆ plasmas is quite low, despite the ease with which atomic fluorine is created. One way of reducing the impact of recombination on cleaning performance is the introduction of oxygen (O₂) into the discharge. The molecular oxygen is dissociated in the discharge, and then reacts with SF_x components, producing components such as SF_xO_y, which are fairly stable and do not recombine further with atomic fluorine. This method is used but has some disadvantages:

- The atomic oxygen will react not only with the SF_x radicals but also with the deposited Si, creating SiO₂, which is more difficult to clean than Si.

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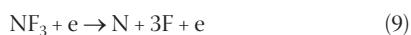
- The dissociation of oxygen requires energy, since one channel lies around 5.12eV, leading to the decrease of electron density and consequently slowing down all of the other reactions in plasmas.

The limitation induced by the recombination channel (Equation 8) is inherent in SF₆ plasmas. It can be reduced but not overcome by process measures, such as increasing the applied power or adding some more reactants.

NF₃ plasma

There are two cases that can be distinguished.

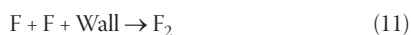
Case 1: If the applied power is high enough to completely dissociate the NF₃ molecule via a reaction of the type



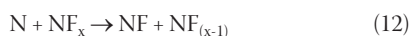
(the reaction must be seen as the integral results of the reactions in Equations 4, 6 and 7) then a very fast recombination channel between N atoms appears:



This channel prohibits the F recombination back to NF_x radicals, leaving only the following possibility for recombination of the F atoms:



Case 2: If the NF₃ molecule is not completely dissociated, then the following scenarios might occur:



The reactions given in Equations 12 and 13 can be summarized as $\text{N} + \text{F}_2 \rightarrow \text{NF} + \text{F}$ with NF_x as the catalyzer, which is a destruction channel for atomic nitrogen as well as directly for cleaning radicals (out of a molecule of F₂ only one F atom remains as a cleaning radical – the cleaning activity of NF has not been demonstrated). In these circumstances, the reaction



becomes a relevant destruction channel for cleaning radicals.

A threshold applied power is therefore expected, above which should yield full conversion of NF₃ to cleaning radicals, with N₂ as a by-product. This observation led to the introduction of RPSs for NF₃ plasma, allowing high power to be used for dissociation outside the PECVD chamber. The high level of power coupling in an RPS allows a large dissociation rate of high flows of NF₃ so that recombination does

not have a negative impact on the cleaning rate [5]. For low NF₃ flows, the in situ power coupling is sufficient to allow a large dissociation rate of NF₃ precursor.

F₂ plasma

Considering now the case of F₂ plasma, it is observed that all of the previously discussed plasmas are a combination of a F₂ plasma and something else:



in the case of SF₆ plasmas, and



in the case of NF₃. The first advantage of using F₂ becomes apparent, namely that the energy usage is the most efficient in a pure F₂ plasma: all of the other molecules lead to energy loss, slowing down the cleaning reactions.

“The biggest advantage of F₂ plasma is the absence of loss channels.”

The biggest advantage of F₂ plasma is the absence of loss channels. The only recombination possible, shown in Equation 11, leads to F₂ formation, which still has some cleaning properties, even if lower than those of atomic fluorine (F) (see Leu et al. [2] for more details). In conclusion, for a given set of parameters (pressure, flow, power coupled to the plasma), the F₂ cleaning is expected to be more efficient than cleaning using any other precursor.

3. The impact of negative ions

All cleaning plasmas are electronegative, i.e. there are a significant number of negative ions in plasmas (see reactions in Equations 3, 4 and 5). Although a very interesting feature from a scientific viewpoint, it is however of less importance from a technological one. The main reason for this, as mentioned previously, is the fact that our processes are driven by neutrals and not by ions.

The presence of negative ions can nonetheless indirectly affect the cleaning processes in two ways, both of which are easy to overcome by technological means:

- The impedance of an electronegative plasma (as in the cleaning process) is very different from the impedance of an electropositive plasma (as in the deposition process). This means that the requirements for the RF matching box are different for deposition and cleaning. The challenge is then to use a suitable RF matching box that is able to match both deposition and cleaning plasmas. This challenge was met by designing a

matching box that covers the whole required parameter window.

- The difference in plasma impedance might lead to some inhomogeneities in the case of cleaning plasmas in large area reactors if the reactor was primarily designed for deposition use. This challenge was met by careful design of the PECVD reactor.

If necessary, for very special cleaning processes, some electropositive gases (such as Ar or N₂) could be added to the cleaning recipes: these significantly reduce the electronegativity of the plasma and consequently improve the process homogeneity.

4. Comparison from cost and environmental points of view

The F₂ molecule consists (in terms of mass) of 100% fluorine, while the NF₃ molecule has only 80% fluorine, and SF₆ only 78%. By switching from NF₃ to F₂ and neglecting all of the previously mentioned process advantages of F₂, there is a reduction in mass consumption (in terms of mass/run) of 20%. Because gas is purchased on a per kilogram basis, this means that even if NF₃ and F₂ were purchased at the same price, it will be 20% cheaper to use F₂ while performing the same cleaning. As will be seen below, the unique performance of F₂ as a cleaning agent leads to an increase in mass saving that is well above this minimum theoretical value of 20%; when combined with a lower price per kilogram of F₂ than of NF₃, the effect of the total cost saving from on-site fluorine on annual gas consumption is considerable.

Molecular fluorine also has zero global-warming potential, whereas NF₃ (with a global-warming potential of 17,200) and SF₆ (22,600) are well-known greenhouse gases. Conversion to fluorine will therefore help dramatically reduce the CO₂-equivalent footprint of the solar panel manufacturing process.

On-site/on-demand generation of F₂: successful Linde concept

Given the chemical properties of molecular fluorine, the adoption of fluorine by the PV industry has raised some genuine concerns. The conservativeness of the PV industry, as well as its reluctance to introduce new materials in general, convinced both groups – Oerlikon and Linde – to develop a systematic approach in order to overcome all safety concerns and technology hurdles, and offer end users a proven solution for chamber cleaning. Working together with Linde – who have more than thirty similar systems installed worldwide, along with an excellent safety and reliability record – Oerlikon have addressed these concerns and installed an on-site fluorine generator at their solar facility in Trübbach. The generator, a Generation F 800 SC, is able

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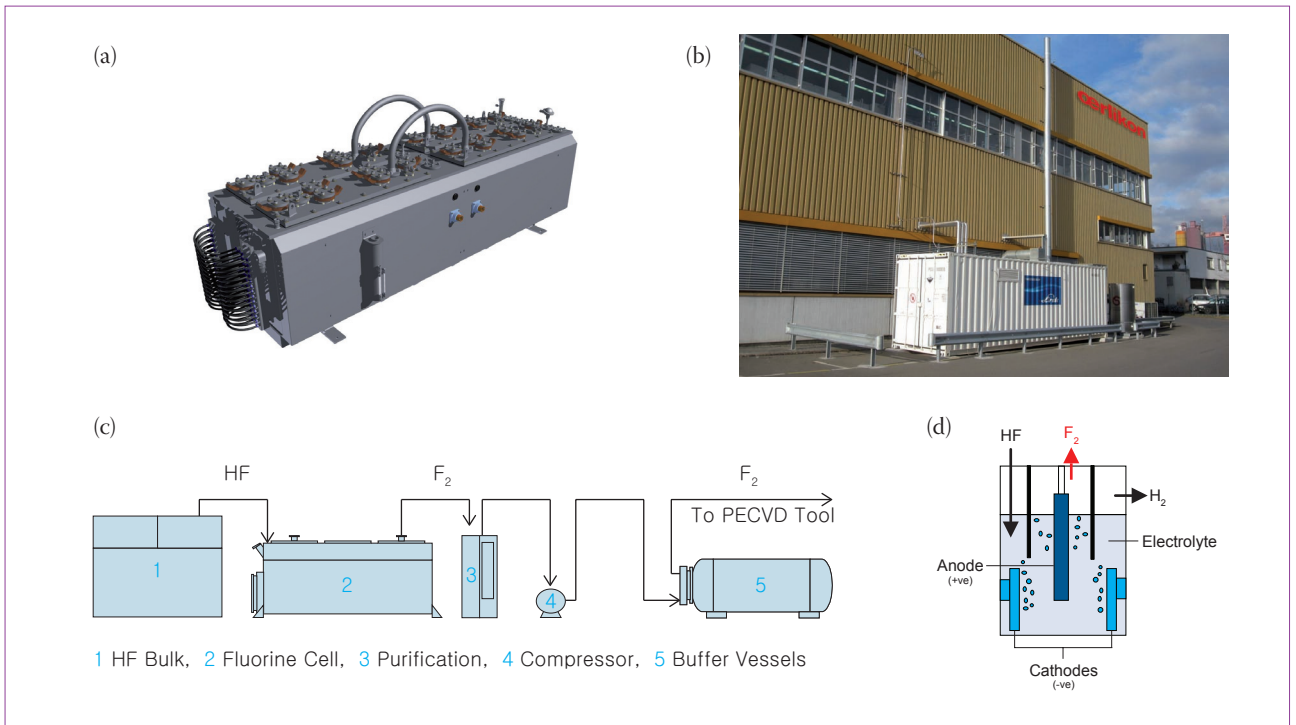


Figure 2. (a) Electrolysis cell; (b) containerized F₂ generator; (c) process flow chart; (d) principle of fluorine generation.

to produce 800 standard litres per hour (corresponding to a consumption of 32kg/day).

“The only viable supply of molecular fluorine for production purposes is on-site generation.”

Safety regulations severely limit the amount of fluorine that can be stored or transported in a high-pressure cylinder. Because of the large amount of cleaning gas to be considered for a PV production line, the only viable supply of molecular fluorine for production purposes is on-site generation. This concept has been successfully developed and implemented by Linde in the past few years, with the installation of hundreds of tons of on-site fluorine production capacity worldwide for semiconductor, flat-panel display and photovoltaic manufacturing.

In contrast with SF₆ or NF₃, fluorine is a highly reactive material, even in its molecular form, and therefore material compatibility of all wetted materials from the generator to the KAI process chamber must be ensured. Before using F₂ in the PECVD chamber, the piping from the buffer tank to the process chamber must be passivated. However, there are no particular concerns regarding the material used in the chamber and downwards to the abatement system, as the configuration has been proved for dissociated NF₃ or SF₆.

On-site and on-demand generation of fluorine offers many benefits, including

a simplified supply chain (no fluorine transport is required), a low site inventory (as fluorine is produced on demand), and a safer delivery path (there is no F₂ cylinder exchange operation) compared to a cylinder supply because of the much lower pressures involved.

On-site fluorine generation is based on the electrolytic decomposition of anhydrous hydrofluoric acid (HF), first used by Moissan in his isolation of elemental fluorine in 1886. Anhydrous HF can be supplied in either gas or liquid phases to the working fluid, KF₂HF (which is a liquid only above 70°C), contained in a fluorine-resistant alloy vessel that also serves as the cathode. Current applied through the proprietary anodes determines the rate of fluorine production, and the evolved fluorine and hydrogen are physically

separated to prevent recombination. The H₂ is diluted for direct disposal, or can be easily and completely abated in situ with proprietary passive catalytic oxidation.

Fluorine is filtered and purified to reduce residual vapour-phase HF to levels below 20ppm; it can then be used at its nominally atmospheric production pressure, or compressed and buffered to a safe working pressure of up to around 20psig. To supply processes that have been developed using diluted sources of fluorine, a near-zero pressure drop blender can supply on-demand blends of fluorine and inert gases, such as argon and nitrogen, in a dilution range of 0–100%. On-site fluorine generators are sized according to the total volume required, and all rely on the same simple low-pressure, low-temperature and low-inventory process design.

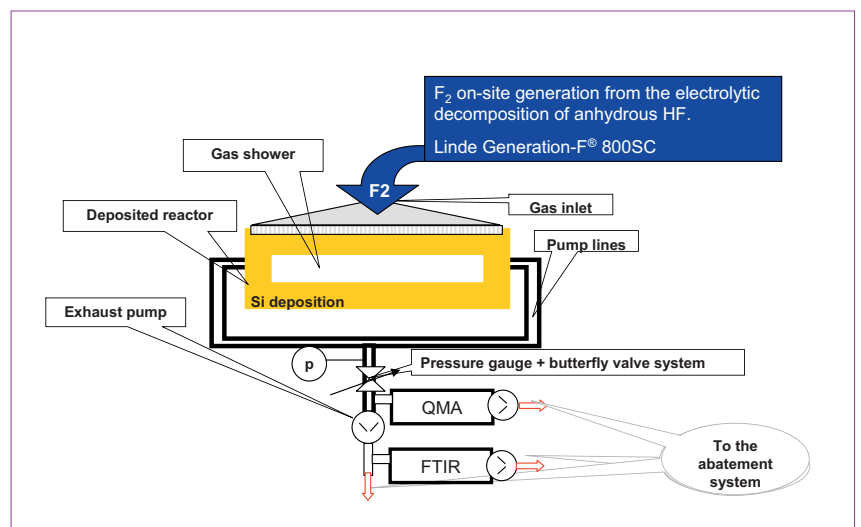


Figure 3. Experimental set-up.

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KAI reactor and experimental set-up

Molecular fluorine was used to carry out a series of cleaning tests on a KAI R&D platform, a process chamber with a single Gen 5 (1300mm × 1100mm) reactor; the results were compared to those previously obtained using either NF_3 or SF_6 . To monitor the cleaning performance of the different gases, different analytical tools were installed, including mass spectrometry (QMA) and infrared absorption spectroscopy (FTIR) (see Fig. 3).

A typical overview mass spectrum (QMA) for NF_3 cleaning is shown in Fig. 4, while a typical infrared spectrum (FTIR) is shown in Fig. 5. A detailed analysis of these two methods or of the taken spectra is beyond the scope of the present paper. In principle, in both cases one obtains spectra, from which two pieces of information can be inferred: the composition of the exhaust gas and the relative amount of every component. Ideally we want quantitative data from the chamber only – QMA gives us good information but it is difficult to interpret. FTIR, however, is easier to understand but the data is compromised by the measurement point being downstream of the pumps, so it is difficult to know if the data is the result of reactions in the process chamber or in the pumps downstream of the chamber.

By monitoring some of the QMA lines over a complete cleaning cycle, the curve shown in Fig. 6 can be generated. To compare different cleaning procedures, an arbitrary measure is used, specifically the ‘cleaning rate 10%’ (CR10%), defined as the thickness of the deposited layer divided by the time until the main cleaning signature (amu85 – SiF_3) decreases to 10% of its maximum. This is not the actual cleaning time but offers a good way of comparing different cleaning processes for a given deposition (see Leu et al. [5] for more details about this type of measurement applied to another type of cleaning).

The etching rate on a deposited glass was also measured by means of ellipsometry: the deposition thickness was measured before and after a short cleaning treatment performed with the glass inside the reactor. The results are expressed in terms of atomic fluorine flow so that a direct comparison of the efficiency of the cleaning can be made on the basis of the number of atoms of F supplied. For example, each molecule of NF_3 contains three F atoms, while a F_2 molecule contains two; thus, to supply the same number of F atoms, you need to supply 50% more F_2 molecules (flow) than NF_3 molecules.

F_2 etching uniformity

The first series of tests was the measurement of the a-Si etch rate on a deposited glass. The film thickness

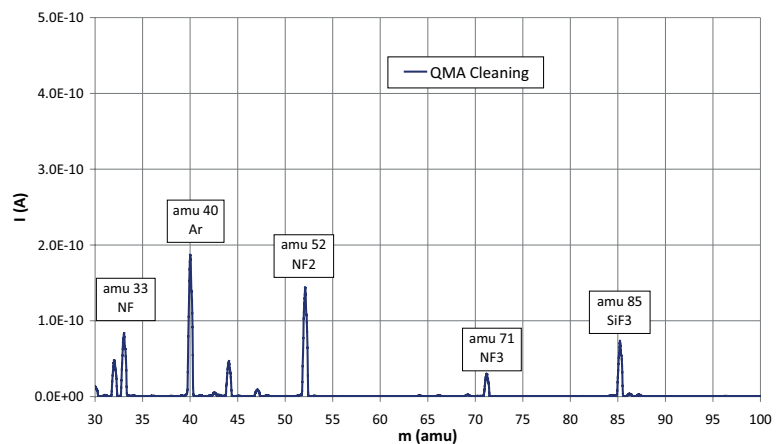


Figure 4. Typical overview of the QMA spectrum.

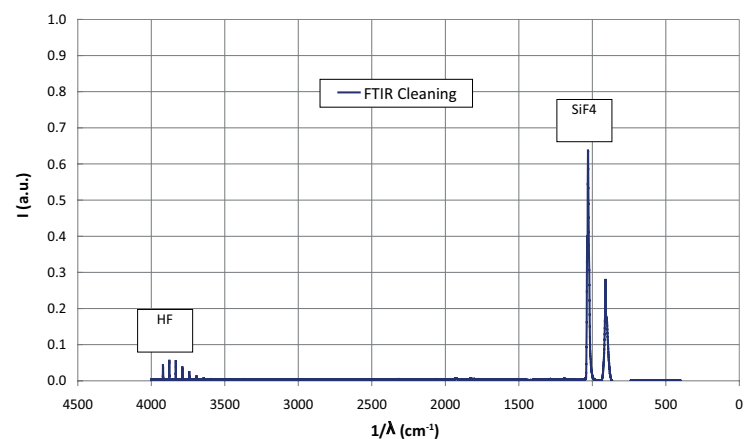


Figure 5. Typical overview of the FTIR spectrum.

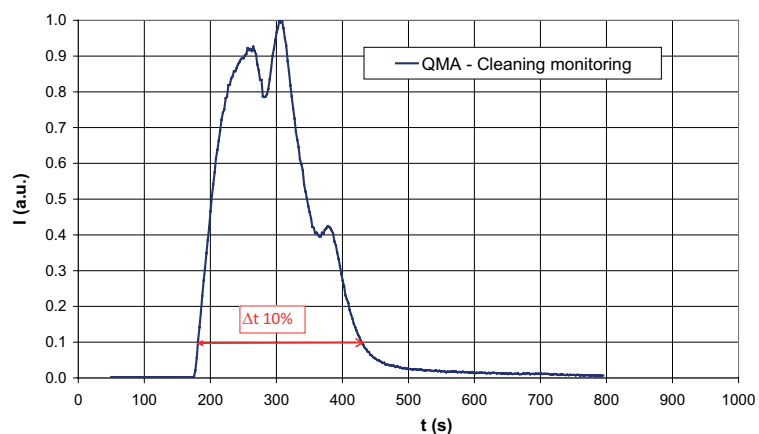


Figure 6. QMA cleaning monitoring.

was measured immediately after the deposition process. The reactor was then cleaned, and the glass reintroduced into the clean reactor; the cleaning process was performed for a few tens of seconds, and the film thickness on the glass measured again. The deposition thickness before and after the short F_2 etching is shown in Fig. 7. The etching uniformity is remarkably good

across the whole measured parameter window, confirming the results already obtained from another KAI R&D chamber, albeit with a different configuration [6].

Comparative evaluation of cleaning gases

A comparison was made between the best known method recipes for SF_6 and NF_3 , and a series of NF_3 and F_2 cleaning

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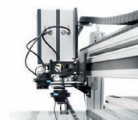
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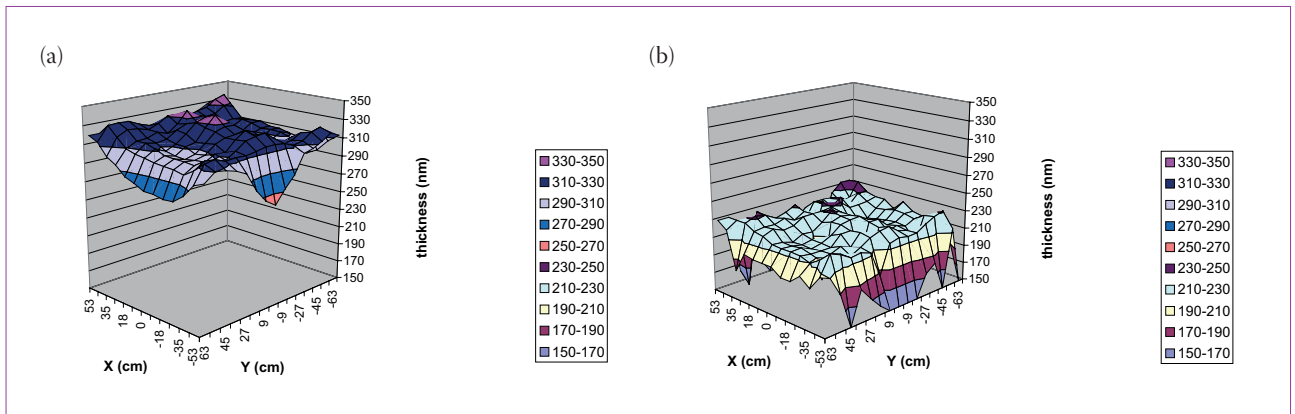


Figure 7. F₂ etching experiments:(a) before F₂ cleaning, and (b) after short F₂ cleaning.

development recipes (see Fig. 8). No direct comparison between SF₆ and NF₃ cleaning was possible, because SF₆ requires the addition of oxygen (see the discussion about the theoretical comparison between different cleaning precursors), and the presence of oxygen changes the power balance in the plasmas.

The SF₆ cleaning was found to be up to 75% less efficient than NF₃ and F₂. The comparison between the best known NF₃ method recipe and the corresponding (in

terms of flow of atomic fluorine) F₂ recipe shows that the F₂ cleaning is about 10% faster. By increasing the F₂ flow at constant power, a linear increase in cleaning rate was measured. In comparison, an increase of NF₃ flow at constant power was shown to lead to insignificant increases in etching rate. An increase in RF-coupled power is necessary to obtain increased etching rates with increasing NF₃ flow; even using higher RF powers, the etch rate in the case of NF₃ tends to saturate. This behaviour

confirms the hypothesis made above that SF₆ cannot reach the performance of NF₃, because of the rapidity of the volume recombination channel: SF_{x-1} + F → SF_x. For low gas flows the NF₃ is dissociated into N and F atoms, the recombination channel N + N + Wall → N₂ prohibits the recombination of F to NF_x radicals, and consequently the performances of NF₃ and those of F₂ are comparable. With increasing NF₃ flows, higher and higher power levels are required in order to achieve the dissociation of NF₃ molecules into atoms, making the NF₃ process less efficient than the F₂ one.

A second series of tests was carried out using a quadrupole mass analyzer (QMA) to monitor the signal of mass 85 (corresponding to SiF₃ and indicating the presence of SiF₄) until it reached 10% of its maximum value (see Fig. 9). The results also confirm the hypothesis made from the theoretical comparison between different cleaning precursors.

For low flows at a given RF power, the gas usage is almost 100% for both NF₃ and F₂, and the cleaning rate is much the same for similar flows of atomic fluorine. If the flow is increased at constant power, the cleaning rate will increase; however, a point will eventually be reached at which an increase in flow has no further effect on the cleaning rate, as the power available can only activate so many molecules, and non-activated molecules will just be pumped through the system without making any contribution to the cleaning. Thus, there is a maximum flow for optimum cleaning for a given coupled power.

This main advantage of F₂ over NF₃ is that the maximum flow for optimum cleaning is higher for F₂ than for NF₃: higher flows of F₂ can be used for the same coupled power while still having almost the full usage of precursors. As a consequence, depending on the target to be achieved – a higher cleaning rate or a lower gas consumption (see Fig. 10 for a comparison of the NF₃ and F₂ processes in terms of gas consumption) – there are two development strategies for F₂ cleaning:

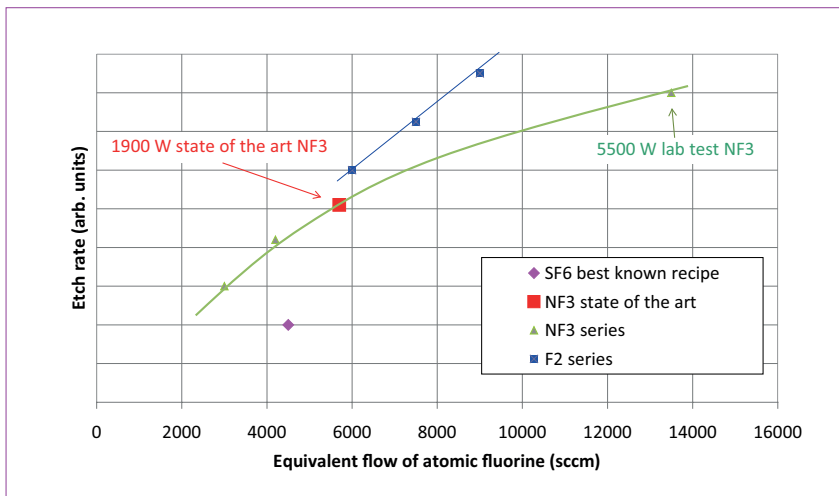


Figure 8. Etching experiments: comparison of SF₆, NF₃ and F₂.

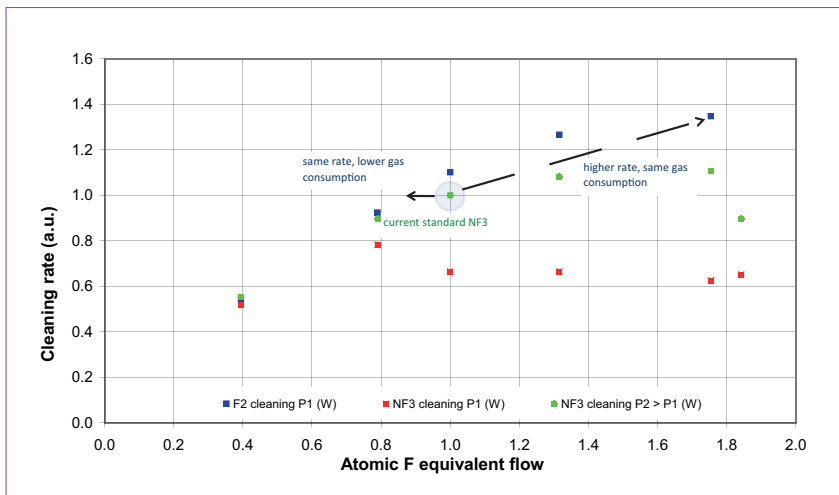


Figure 9. CR10%: comparison of NF₃ and F₂.

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- Using a higher F_2 flow can lead to a 35% decrease in cleaning time compared to NF_3 while keeping the cleaning gas mass consumption per module at the same level.
- Using a lower F_2 flow leads to the same effective cleaning time, whereas the F_2 -mass consumption is reduced by up to 30% compared to NF_3 .

“A significantly larger process window is available for reactor cleaning when F_2 is used in place of NF_3 or SF_6 .”

Conclusions

The results obtained show that a significantly larger process window is available for reactor cleaning when F_2 is used in place of NF_3 or SF_6 . This leads to both time and gas mass saving, improving productivity and bringing down the cost of ownership of the reactor cleaning process.

The successful experiments on the KAI R&D platform with Gen5 glass size has allowed Oerlikon Solar to confidently transfer the process for use on KAI MT platforms, the PECVD production platforms commercialized by Oerlikon Solar.

The joint development work being carried out between two key companies in the thin-film solar industry is helping to reduce the cost of chamber cleaning, and establish molecular fluorine as the mainstream cleaning gas for thin-film silicon solar cell technology, to the benefit of both the customer and the environment.

Acknowledgement

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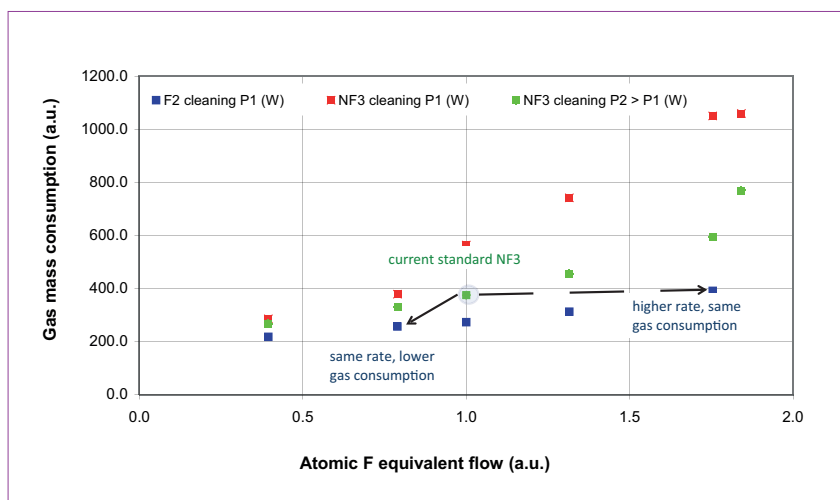


Figure 10. Gas mass consumption: comparison of NF_3 and F_2 .

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