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Increasing the Deposition Rate of Microcrystalline and Amorphous Silicon Thin Films for Photovoltaic Applications - Phase IV: 1997 - 1999

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ABSTRACT

The project aim was to test the feasibility and usefulness of Very High Frequency (VHF) plasma operation in large-area reactors suitable for volume production of solar cell panels, particularly with respect to microcrystalline silicon deposition for the new generation of IMT Neuchâtel ‘micromorph’ solar cells. Faster deposition is necessary because the current rate of about 1 Å/s is prohibitively slow for industrial production. The goal of the project was to achieve 10 Å/s whilst maintaining a high degree of crystallinity, good adhesion (low stress), and suitable material properties such as low defect density.

Two approaches for increasing the deposition rate of microcrystalline silicon were studied: an alternative plasma chemistry by addition of silicon tetrafluoride (SiF_4) to standard silane / hydrogen mixtures, and an alternative plasma source using a high current DC arc.

It was shown by experiment and analytical modelling that SiF_4 contributes little to the deposition rate because the fluorine generated from the dissociation of SiF_4 etches away the film as fast as it is deposited. The deposition rate of microcrystalline silicon is therefore determined by the silane flowrate, provided that the silane is almost completely dissociated by the plasma and the plasma composition is essentially hydrogen. The VHF technique is therefore well-suited for microcrystalline silicon deposition because of the high dissociation rates which can be obtained, for a given power, relative to the conventional 13.56 MHz frequency.

Extremely fast deposition rates, up to 100 Å/s, were obtained in the high current DC arc reactor. The film was highly microcrystalline but with a defect density and uniformity insufficient for photovoltaic applications in these preliminary experiments. The rapid rate is due to the high power density in the arc compared with radio-frequency (RF) plasmas, and proves that there is no physical limitation to microcrystalline silicon deposition well above the required 10 Å/s.

In conclusion, to increase the deposition rate of low defect density, uniform, microcrystalline silicon thin films, it is clear that the power density in large area RF reactors must be raised to several kW/m^2. In this case, the high RF voltages which result in film damage from energetic ion bombardment can only be avoided by using higher VHF frequencies (above 60 MHz) which, unfortunately, cause non-uniform plasma power distribution in large area reactors. The optimum RF contact configuration for large area reactors was calculated for the design of future VHF high deposition rate reactors with 1 m^2 electrodes.

CONTENTS

1) PROJECT AIMS

The project aim is to test the feasibility and usefulness of Very High Frequency (VHF) plasma operation in large-area reactors suitable for volume production of solar cell panels, particularly with respect to micro-crystalline silicon deposition for the new generation of IMT Neuchâtel 'micromorph' solar cells. Faster deposition is necessary because the current rate of about 1 Å/s is prohibitively slow for industrial production. The goal of the project is to achieve 10 Å/s whilst maintaining a high degree of crystallinity, good adhesion (low stress), and suitable material properties such as low defect density.

2) INTRODUCTION

Production of microcrystalline silicon requires the deposition of hydrogenated silicon and, at the same time, a mechanism for converting, or 'conditioning', the film into a micro-crystalline phase. There are several 'conditioning' mechanisms, but essentially this entails the removal of energetically-unfavourable Si:H groups by an etchant species. In plasmas of silane/hydrogen mixtures, this etchant species is atomic hydrogen. Generally, the silane is almost completely depleted in the plasma, and the overall deposition process is rate-limited by the generation of atomic hydrogen. Rapid deposition of microcrystalline silicon has been optimised for high power, Very High Frequency (VHF) plasmas in hydrogen-diluted silane (P. Torres, "Hydrogenated Microcrystalline Silicon by VHF-GD for Thin Film Solar Cells", IMT thesis Neuchâtel 1998). The deposition rate could be further improved by using a higher silane flowrate provided that the supply and/or etching efficiency of the etchant species can be simultaneously increased. This could therefore be achieved in two ways :

a) Use an alternative plasma chemistry to the standard silane/hydrogen mixture by adding gases containing a more effective etchant species. Atomic fluorine is known to be a strong etchant for silicon, but fluorine gas is extremely corrosive and special precautions must be used. Silicon tetrafluoride (SiF_4) was chosen as a suitable fluorine-containing additive to silane/hydrogen for this work.

b) Use an alternative plasma source with a more efficient production of atomic hydrogen, while keeping the standard silane/hydrogen mixture. The high current DC arc at CRPP is known to be a prolific source of atomic hydrogen, and this reactor was converted for silicon deposition experiments.

An account of both approaches is given after description of the experiment and its diagnostics.

3) DEVELOPMENTS IN REACTOR TECHNOLOGY AND DIAGNOSTICS

Technical considerations for Large Area VHF Deposition of Microcrystalline Silicon

Experiments were carried out in the Balzers KAI-1 Plasma-Box industrial reactor, modified as described in preceding reports. Our previous studies show that VHF advantages, such as enhanced dissociation rate, ionisation degree and low-energy ion flux, continue to increase as the frequency rises. However, the voltage uniformity degrades as the electrode dimensions approach a quarter of the free-space wavelength at the radio-frequency used. Since fixed frequency generators are much less costly than wideband amplifiers, the question arises: What is the optimum frequency for micro-crystalline silicon deposition? A compromise upper limit frequency is determined by the degree of non-uniformity which can be tolerated, and the technical complexity of multi-point RF contacts employable. For our 47 cm x 57 cm electrode geometry, with a single back-face-centred RF connection, and a uniform gas showerhead with single side pumping, a film thickness uniformity of better than 5 % was obtained for 70 MHz VHF operation. Consequently, a 2 kW Dressler generator at 67.8 MHz (5th harmonic of the conventional 13.56 MHz) was purchased, financed by the CRPP, for high power VHF studies in industrial size reactors.

High power operation also necessitated upgrading of the matching box, vacuum current feedthrough, and *in vacuo* RF connection to the reactor box. Typical plasma parameters used during this study were 70 MHz excitation frequency, 400 W power, 0.05 to 4 mbar total pressure, 250 ºC electrode temperature with 24 mm electrode gap, and gas mixtures of silane, hydrogen, silicon tetrafluoride and argon.

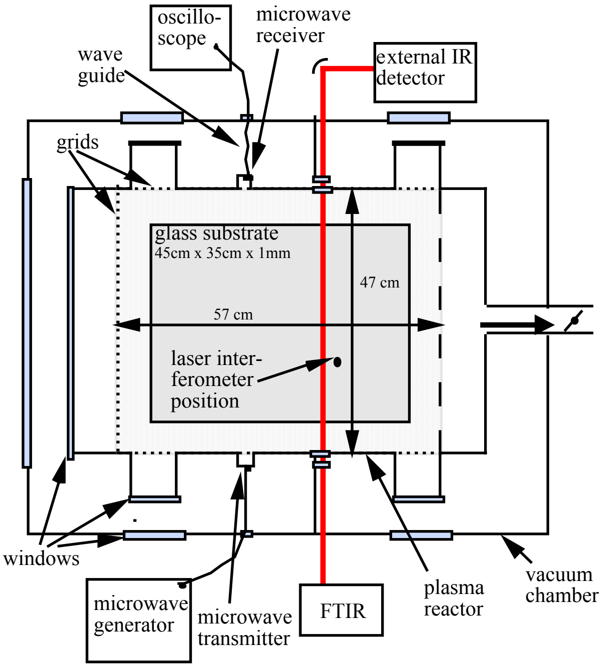

Figure 1 *Schematic of the plasma reactor showing the infrared and microwave diagnostics.*

Diagnostics: Development and Implementation

The panoply of available diagnostics was developed throughout the various OFEN projects as described in previous reports. Only the new developments are mentioned below.

i) *Fourier Transform Infrared Absorption Spectroscopy* is applied to measure the silane fractional depletion in the plasma during the deposition process. The single-pass line-of-sight arrangement is compatible with industrial design requirements; it uses an external infrared detector to measure the absorption spectrum using a commercial Fourier Transform spectrometer. Sapphire windows used in previous work have sufficient transmission for measuring the depletion of silane, but sapphire has a cut-off at the infrared wavelengths corresponding to silicon tetrafluoride. Consequently, thin silicon windows (200 micron) were chosen since silicon allows infrared transmission at 1000 cm^{-1}, corresponding to silicon tetrafluoride, and has sufficient resistance to plasma etching.

ii) *Emission spectroscopy* of silicon tetrafluoride plasmas has the advantage that several of its radicals (SiF, SiF_2, and SiF_3) can be observed by emission spectroscopy, in contrast to silane where only the SiH radical emits visible light.

iii) *Cavity Ringdown Absorption Spectroscopy* has been employed on the KAI reactor. This relatively recent technique, applied for the first time in an industrial-scale reactor, consists of a cavity formed by two high-reflectivity mirrors (better than 99.99 %) in vacuum. A pulsed dye laser (4 mJ, 4 ns pulse at 566 nm, 10 Hz repetition frequency) is coupled into the back face of the entrance mirror and the light energy is reflected back-and-forth several thousand times, giving an effective optical path length of a few kilometres in the plasma. The exponential decay time, measured by a photomultiplier at the exit mirror, is simply related to the absorption coefficient at the laser wavelength of any plasma species, such as negative ions, radicals and powder particles. The diagnostic was shown to be highly-sensitive to nanometric particles formed in the plasma.

iv) *Mass spectroscopy* was installed to monitor the relative proportions of fluorinated exhaust products during deposition and etch cleaning. It became clear that standard mass spectrometry is inappropriate for fluorine-containing gases since the hot tungsten filament, which serves as the electron beam source, is rapidly corroded. Improved cross-beam ion sources will be investigated in consultation with Balzers Instruments AG.

v) *Voltage probes* for RF electrode voltage and voltage uniformity.

vi) *Microwave Diagnostics for Plasma Density Measurements*. Microwave interferometery in the range 7 to 12 GHz (Band X) is a reliable, sensitive and rapid absolute measurement of the free electron density. The KAI-1 represents a strongly over-moded cuboid cavity in which the high wavenumber density of modes would seem to preclude identification and tracking of a single eigenmode. Fortunately, imperfections in the reactor cavity such as windows, pump ports etc. mean that the higher modes are strongly damped and in practice, individual modes can be identified. The measurement consists in comparing the shift in mode spectrum obtained by frequency-sweeping the microwave source. This diagnostic has been used to measure the electron density dependence on excitation frequency.

vii) *Thin film interferometry* for measuring the deposition or etch rate during the plasma was a principal diagnostic. Its reliability and ease of alignment was greatly improved by a new construction using high-temperature, vacuum compatible optical fibres.

viii) *X-ray diffraction, electron microscopy and infrared absorption spectroscopy* were all employed as standard *ex situ* diagnostics of films deposited on silicon wafers.

ix) *Ellipsometry* consists in measuring the alteration in polarisation degree on reflection from a surface, from which the dielectric constant can be deduced along with various other properties (roughness, crystallinity...) according to the model applied. In fact, the interpretation has proven more difficult than anticipated: the measured dielectric function is extraordinarily difficult to fit to library values for various forms of silicon (micro-crystalline, large and small grained poly-crystalline, amorphous, porous, crystalline, or any mixture of these). It appears to us (in collaboration with U. Kroll of IMT Neuchâtel) that the roughness of the micro-crystalline silicon overlayer is strongly affecting measurements of the film bulk.

4) RESULTS

A. UNIFORMITY IN LARGE AREA REACTORS FOR VHF OPERATION

We have addressed three of the most critical parameters influencing uniformity, namely, voltage, gas supply, and edge plasma uniformity. The aim is to optimise the homogeneity of each parameter (voltage, gas pressure, radical composition, plasma density) individually, rather than look for a combination of compensating effects, since the latter approach would only yield uniform films for a narrow process window.

Voltage Uniformity in Large Area Reactors for VHF operation

The standard RF connection position at the electrode edge gives a strong variation in RF voltage amplitude over the electrode surface at VHF frequencies. This results in a non-uniform plasma power dissipation and deposition thickness. We developed an analytic model which accurately reproduces the measurements by means of an expansion in Green functions. The principal non-uniformity is due to a singularity in the vicinity of the RF connection (and similarly, at the ground connection). Due to the skin effect, the solid RF electrode plate is effectively a double-skinned electrode in which continuity of the RF current across the top and bottom surfaces is via the edges of the RF electrode. Therefore, the RF contact singularity can be separated from the plasma zone by displacing the RF connection to the back face centre of the RF electrode. A RF passage and central back-face connector has been designed and installed at the CRPP. It is now routinely used to produce films at 70 MHz with a homogeneity well within the tolerance limits required for integral 35 cm x 45 cm solar cell substrates. Our model and voltage probe technique were further confirmed by measurements on the new IMT large area reactor. Multiple RF connections have been investigated and offer the possibility of further improvements in voltage uniformity, limited only by the degree of technical complexity which can be tolerated.

Gas Supply Uniformity

Although a uniform interelectrode RF voltage is *necessary*, it is not in itself a *sufficient* condition for uniform deposition: the plasma *composition*, as well as its RF voltage, must also be homogeneous. This is often overlooked in the reactor design of gas source and pumping flows. A one-dimensional analytical approach based on continuity equations with and without plasma, demonstrates that an input gas flow which is uniformly distributed over the whole electrode surface theoretically gives a homogeneous composition of the plasma. In other words, for a uniform gas showerhead, the individual densities of the silane gas and each of the plasma-produced radicals are the same everywhere in the reactor. This is not an intuitive result, because even if the showerhead gas *source* is uniform, the gas *flow*, which increases towards the pumping port, is not uniform over the electrode area.

Nevertheless, there are ranges of plasma parameters for which 'dome' or 'hollow' film-thickness profiles persist: the question is whether the 1-D model neglects 3-D density gradients and diffusive flows. An analytic solution of the Navier-Stokes fluid equations demonstrates that density gradients near to the reactor sidewalls lead to a non-uniform plasma composition. This applies to a length scale roughly equal to the electrode gap which explains why the useful substrate size is always somewhat smaller than the actual plasma dimensions. A complete 3D numerical model is near completion.

A uniform showerhead gas source is therefore necessary. However, the design of such a showerhead is not trivial, since any pressure gradients behind the showerhead will cause non-uniform gas flow into the plasma. An analytical solution for the pressure gradients and flow yields a scaling factor to determine the expected non-uniformity in terms of the showerhead geometry. This will be useful for scaling up the reactor to square-metre areas for industrial production of solar cells.

Plasma Uniformity

The plasma itself must also be homogeneous. For example, the region near the electrode edges, which is the junction between the RF electrode and ground, can be a region of strong electric fringing fields and, consequently, a zone of intense plasma with different radical concentrations compared to the central plasma. Lateral density gradients would then arise, perturbing the otherwise uniform composition of the plasma, leading to film inhomogeneity extending over areas beyond the edge region itself. This may be the cause of the unexpectedly strong non-uniformities described in U. Kroll *et al*, "Fast Deposition of a-Si :H Layers and Solar Cells in a Large-Area (40x40 cm) VHF-GD Reactor" (MRS spring meeting 1999). 2D numerical modelling is being used to design special profiles for the electrode edges.

Finally, if a glass substrate is not completely flat on the electrode, the enhanced sheath capacitance due to the electrode-glass 'vacuum gap', in series with the plasma sheath, will cause a locally-reduced RF potential, resulting in zones of reduced film thickness. This is more serious for thin (< 1 mm) glass substrates, where even small (~ 0.1 mm) surface irregularities degrade deposition uniformity, than for thick glass (> 3mm) substrates used for square metre-sized solar cells.

B. MICROCRYSTALLINE SILICON DEPOSITION

Micro-crystalline silicon has been shown to be a useful material for solar energy in the novel ‘micromorph’ cells developed at IMT Neuchâtel. A broad experimental investigation of plasma parameters clearly showed that a necessary condition for micro-crystalline silicon deposition is the almost complete depletion of silane gas, resulting effectively in a nearly pure hydrogen gas plasma. Given our observation that VHF plasmas are more efficient in dissociating silane, this explains why it is that *VHF operation is naturally more effective for micro-crystalline deposition* than conventional frequencies, as borne out by empirical observation by IMT Neuchâtel and subsequently in other laboratories.

Further work confirmed that a copious source of atomic hydrogen is also necessary, probably for silicon atom ‘rearrangement’ mechanisms, for example by selective etching of disordered material. These observations lead to a simple description in terms of gas continuity equations (as discussed in section C below) which graphically illustrate that the key factor is the *gas composition within the plasma*, which is generally not the same as the relative composition of the input gas flowrates, because of plasma dissociation and radical deposition. A corollary of this ‘obvious’ but often overlooked result is that a universal recipe for micro-crystalline silicon deposition requires not only the values of RF power, pressure and flowrates, but also factors which vary from one reactor to another, such as different pumping speed. The analytical model presented after the experimental observations draws all of these variables into a single coherent explanation.

Experimental observations

i) The relative etch rates of amorphous and micro-crystalline silicon were measured by thin film interferometry: first, a film of amorphous or micro-crystalline silicon was deposited; secondly, the film was immediately exposed to a hydrogen plasma with given parameters (pressure, flow rate and power). The interference fringe evolution reversed, indicating an etching by the hydrogen plasma. For the same hydrogen plasma conditions, the micro-crystalline silicon was etched five times more slowly than the amorphous phase, confirming the selective etch mechanism during deposition in hydrogen-diluted silane plasmas.

ii) *Layer-by-Layer Deposition of Micro-crystalline Silicon.* This technique was extended to micro-crystalline silicon deposition by the layer-by-layer technique, where brief periods (12 seconds) of amorphous silicon deposition were alternated with longer periods (160 seconds) of exposure to a pure hydrogen plasma. The idea is to 'transform' amorphous layers into micro-crystalline silicon by the hydrogen plasma. The interferometer measurements clearly show the alternate periods of deposition and etching; the remarkable result is that the etching for the early layers is at least twice as rapid as the later stages. We interpret this as follows: initially, only amorphous silicon is deposited which can be rapidly etched and eventually becomes converted to a micro-crystalline-containing film. This micro-crystalline base acts as a substrate for the next deposition period which subsequently facilitates a higher degree of micro-crystalline growth by a sort of 'epitaxial' effect. Since micro-crystalline silicon is slower to etch than amorphous silicon, the decrease in etch rate with time is explained.

iii) *Acceleration of the Micro-crystalline Silicon Growth Rate.* This above-mentioned result suggests that the growth rate of micro-crystalline silicon could be *accelerated* as the deposition proceeds: presently, each micro-crystalline silicon deposition experiment is carried out with fixed plasma parameters from start to finish in order to guarantee that the layer properties are more-or-less homogeneous, as required for analysis by *ex situ* measurements. However, the *initial* slow deposition is obligatory only to ensure the development of a 'seed' micro-crystalline layer; once deposited, the plasma parameters could be adjusted (more power, pressure and/or silane fraction) to accelerate growth since the 'epitaxial' growth of micro-crystalline silicon on a micro-crystalline silicon substrate is faster. We have already performed accelerating growth experiments, but it was soon realised that reliable *in situ* diagnostics for micro-crystallinity are absolutely necessary to check that there is no transition to the amorphous phase by exaggerating the acceleration. These considerations, along with the limitation on the number of depositions per day, have driven the current search for *in situ* measurement of micro-crystallinity.

Alternative Plasma Chemistry for Microcrystalline Silicon Deposition

The potential of SiF_4 for accelerating microcrystalline silicon deposition has been investigated in our industrial reactor at the VHF frequency of 60 MHz as a function of pressure, power, flowrate and concentration in silane/hydrogen mixtures. The system of plasma chemistry and surface reactions has too many unknown parameters to justify predictive modelling, and so a phenomenological approach was adopted whereby the individual gases, binary mixtures and final triple mixture were systematically investigated. The conclusions are briefly presented :

Silicon tetrafluoride plasma was found to *deposit* very slowly (0.1 Å/s) on amorphous hydrogenated silicon (a-Si:H), but rapidly *etch* (3 Å/s) fluorinated silicon (a-Si:F:H). There is therefore a strong substrate dependence for SiF_4 plasma, which further complicates the issue by increasing the number of unknown parameters. The deposition rate of silane, for the same plasma conditions, is 60 times faster than for SiF_4. Therefore we have an indication, which will be supported and explained below, that SiF_4 contributes only inefficiently to deposition.

Silicon tetrafluoride/silane plasma does deposit rapidly (6 Å/s), but only amorphous material. Strong powder formation also occurred, probably because both are electronegative gases which give rise to trapped negative ions.

Silicon tetrafluoride/hydrogen plasma gave little or no deposition. There is negligible conversion of the SiF_4 to SiH_x -type precursors. We deduce that deposition originates from silane only, and that if SiF_4 dissociation products do deposit, they are efficiently removed by other SiF_x radicals. These observations can be understood by considering the order of bond dissociation energies shown in figure 2.

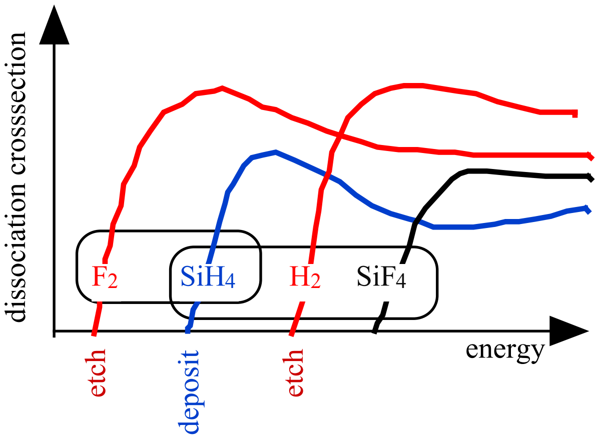

Figure 2 *The ordering of bond dissociation energies for the gases considered*

This shows that silane is preferentially dissociated in silane / silicon tetrafluoride mixtures ; that hydrogen cannot spontaneously replace fluorine in SiF_x radicals, and that SiF_4 and HF are stable etch products.

Silicon tetrafluoride / silane / hydrogen plasma was investigated to find the most rapid deposition of microcrystalline silicon given the constraints on RF power density. Different pressure, concentration and flowrate scans confirmed that the deposition rate was fixed by the silane flowrate, independently of the other gases, provided that ~100 % silane depletion prevailed. Therefore, the goal was to increase the silane flowrate until microcrystalline silicon could no longer be obtained. Microcrystalline silicon films were obtained using plasmas in silane / hydrogen / SiF_4 mixtures with flowrate ratio approximately 1:10:10. The deposition rate limit was reached for 3 Å/s, which was almost twice as fast as silane / hydrogen mixtures without silicon tetrafluoride. For certain plasma conditions, amorphous silicon deposition obtained using a silane / hydrogen mixture could be transformed to microcrystalline deposition by the admixture of SiF_4. However, the net deposition rate was not greatly improved at these moderate powers (< 1 kW).

C. MODELLING OF MICROCRYSTALLINE SILICON PLASMA DEPOSITION

A simple analytical plasma chemistry model is now developed in an attempt to account for the experimental observations and to describe the various empirical approaches in a single framework. One explanation for the deposition of microcrystalline silicon is that simultaneous etching of poorly coordinated bonds and amorphous tissue occurs during deposition. Since the silicon etch probability by atomic hydrogen is small, a large flux of atomic hydrogen is required. Conventionally, this is arranged by strongly diluting the silane in hydrogen, which unfortunately results in slow deposition. Experiments designed to increase the deposition rate use higher RF power, a higher proportion of silane (restrictive dilution), and Very High Frequency (VHF) for the excitation frequency.

First, we write down the simplest set of equations which can describe plasma dissociation, deposition and etching. No attempt is made to account for secondary reactions between radicals, nor to model the hydrogen content of the deposited film. A more complete treatment would have to include surface and solid state phenomena. The aim here is to set up illustrative analytical solutions, where clarity takes priority over rigour. The equations are steady-state and zero-dimensional (independent of position in the reactor) which is justified for a uniform showerhead.

For a silane/hydrogen mixture, we reduce the complex plasma chemistry to the simplest reaction set which preserves the overall stoichiometry in the plasma:

$$\text{Plasma dissociation}: \ \mathrm{SiH_4} \xrightarrow{k_{\mathrm{SiH_4}}} \mathrm{SiH_2} + \mathrm{H_2}$$

$$\text{Deposition}: \ \mathrm{SiH_2} \xrightarrow{S} \mathrm{Si_{film}} + \mathrm{H_2}$$

$$\text{Plasma dissociation}: \ \mathrm{H_2} \xrightarrow{k_{\mathrm{H_2}}} 2\mathrm{H}$$

$$\text{Etching}: \ 4\mathrm{H} + \mathrm{Si_{film}} \xrightarrow{G} \mathrm{SiH_4}$$

where $k_{\mathrm{SiH_4}} \approx \sigma_{\mathrm{SiH_4}} \sqrt{8 k_B T_e / \pi m_e}\, \exp\left(-e E_{\mathrm{SiH_4}} / k_B T_e\right)$ is the rate constant for dissociation of silane ($\sigma_{\mathrm{SiH_4}}$ is the electron impact cross-section, T_e [K] the electron temperature, and $E_{\mathrm{SiH_4}} \approx$ 6 eV is an effective threshold energy for dissociation). For hydrogen, $k_{\mathrm{H_2}}$ is similarly defined, with $E_{\mathrm{H_2}} = 8.8$ eV. The rate of deposition is written as $S n_{\mathrm{SiH_2}}$ where S includes the diffusion of SiH_2 radicals to the substrate and the sticking probability. The etch rate by atomic hydrogen, $G n_{\mathrm{H}}$, is similarly defined. The corresponding conservation equations for each species in the plasma, in units of particles·$m^{-3}s^{-1}$, are:

$$\begin{array}{rl} \mathrm{SiH_4}: & \Phi_{\mathrm{SiH_4}} - (n_e k_{\mathrm{SiH_4}} + a) n_{\mathrm{SiH_4}} + G n_{\mathrm{H}} = 0 \\ \mathrm{SiH_2}: & n_e k_{\mathrm{SiH_4}} n_{\mathrm{SiH_4}} - (S + a) n_{\mathrm{SiH_2}} = 0 \\ \mathrm{H_2}: & \Phi_{\mathrm{H_2}} + n_e k_{\mathrm{SiH_4}} n_{\mathrm{SiH_4}} + S n_{\mathrm{SiH_2}} - (n_e k_{\mathrm{H_2}} + a) n_{\mathrm{H_2}} = 0 \\ \mathrm{H}: & 2 n_e k_{\mathrm{H_2}} n_{\mathrm{H_2}} - (4G + a) n_{\mathrm{H}} = 0 \end{array}$$

where $\Phi_{\mathrm{SiH_4}}$ and $\Phi_{\mathrm{H_2}}$ are the silane and hydrogen molecular flow rates per unit volume into the reactor ($\Phi[\mathrm{molecules\,m^{-3}s^{-1}}] = \phi[\mathrm{sccm}] \big/ \left(600 V k_{\mathrm{B}} T_{\mathrm{gas}}\right)$ and V [m^3] is the reactor volume), n_e is the electron density, and a is the inverse of the effective residence time of the gases, i.e. an effective pumping speed. The latter is determined by the usual experimental condition that the total number density $n_{\mathrm{total}} = p/k_{\mathrm{B}}T_{\mathrm{gas}}$ is held constant, for example, by feedback to a throttle valve in the pumping line. Generally, for a large area parallel plate reactor with electrode gap much smaller than the substrate dimensions, the rate of diffusion to the electrodes is much greater than the pumping loss rate. Therefore, S and $G \gg a$ unless the sticking coefficient or etching probability is very low. It follows that no reactive radicals leave the reactor, and only molecular hydrogen and silane are pumped. Without plasma, $a_0 n_{\mathrm{total}} = \Phi_{\mathrm{SiH_4}} + \Phi_{\mathrm{H_2}}$; in the limit where all the silane is dissociated in the plasma, and S, $G \gg a$, the pumping speed can double and so a is bounded by $a_0 \le a < 2a_0$. The system of four conservation equations and the condition of constant total pressure is a closed set of equations readily solved to give a and the number densities $n_{\mathrm{SiH_4}}$, $n_{\mathrm{H_2}}$, $n_{\mathrm{SiH_2}}$ and n_{H} in terms of the plasma electron density n_e and the values $\Phi_{\mathrm{SiH_4}}$, $\Phi_{\mathrm{H_2}}$, S, G, $k_{\mathrm{SiH_4}}$ and $k_{\mathrm{H_2}}$.

The radicals H and SiH_2 have much smaller number densities than H_2 and SiH_4 because they diffuse rapidly to the electrode surfaces where they can react. Although the radical densities are small, their flux to the substrate surface, however, is significant and determines the ratio Q of etch and deposition rates, $G n_{\mathrm{H}}$ and $S n_{\mathrm{SiH_2}}$, respectively:

$$Q = \frac{G n_{\mathrm{H}}}{S n_{\mathrm{SiH_2}}} = \frac{\left[2 + \left(1 + a/n_e k_{\mathrm{SiH_4}}\right)\left(\Phi_{\mathrm{H_2}}/\Phi_{\mathrm{SiH_4}}\right)\right]}{\left[2\left(1 + a/n_e k_{\mathrm{H_2}}\right) + \left(\Phi_{\mathrm{H_2}}/\Phi_{\mathrm{SiH_4}}\right)\right]}.$$

Q is an indicator for the degree of microcrystallinity in the deposited film, since etching of poorly coordinated silicon is a requirement for a high degree of microcrystallinity. The expression does not depend on the precise values taken for S and G provided that S, $G \gg a$. Deposition dominates for $Q < 1$, and etching dominates for $Q > 1$. From the expression for Q it can be seen that the microcrystallinity of the deposition is determined by three dimensionless numbers:

i) the dilution ratio $\Phi_{\mathrm{H_2}}/\Phi_{\mathrm{SiH_4}}$;

ii) the ratio between the silane dissociation rate and the effective pumping speed, $n_e k_{\mathrm{SiH_4}}/a_0$;

iii) the plasma dissociation rate constant ratio. This ratio is fixed by the cross-sections and threshold energies of the gases and varies only with electron temperature. Note that $k_{\mathrm{SiH_4}} \gg k_{\mathrm{H_2}}$ principally because the threshold energy for silane dissociation is below that for hydrogen.

The boundary between deposition and etching is at $Q = 1$, for which the dilution ratio $\Phi_{\mathrm{H_2}}/\Phi_{\mathrm{SiH_4}} = 2k_{\mathrm{SiH_4}}/k_{\mathrm{H_2}} \approx 50$ for all values of pumping speed and electron density. This corresponds approximately to experimental observation where it is also found that the net rate of deposition falls close to zero for about 2% of silane in hydrogen, and smaller fractions of silane eventually lead to etching of the film. For low power, low pressure plasmas, $n_e k_{SiH_4}/a_0 < 1$, and $Q = k_{H_2}\Phi_{H_2}/\left(2k_{SiH_4}\Phi_{SiH_4}\right)$. Experimentally, it is then found that microcrystalline silicon can only be obtained for less than about 4% silane in hydrogen for these conditions, corresponding to $Q > 0.5$. We can therefore estimate an approximate range $0.5 < Q < 1$ for microcrystalline silicon growth. To summarise:

$$\begin{array}{lcl} Q < 0.5 & \Rightarrow & \text{a-Si:H deposition} \\ 0.5 < Q < 1 & \Rightarrow & \mu\text{c-Si:H depositior} \\ Q > 1 & \Rightarrow & \text{etching.} \end{array}$$

Using the general expression for Q, we can now draw figure 3 showing the ranges of silane dilution and dissociation rate to pumping rate ratio for which microcrystalline deposition occurs. The advantage of describing the plasma parameters for microcrystalline silicon deposition in terms of the dimensionless number $n_e k_{SiH_4}/a_o$ is that factors which vary from reactor to reactor such as different reactor volumes, pumping speeds and electron density, are automatically accounted for.

For a given flow rate of silane, the maximum possible deposition rate occurs when all the silane is converted into silicon in the film. We therefore define the deposition yield by the ratio of the net deposition rate (deposition minus etching) to the silane flow rate:

$\eta = \dfrac{Sn_{SiH_2} - Gn_H}{\Phi_{SiH_4}} = \left[1 - \left(\dfrac{k_{H_2}\Phi_{H_2}}{2k_{SiH_4}\Phi_{SiH_4}}\right)\right] \Big/ \left[1 + \dfrac{a}{n_e k_{SiH_4}}\right]$ which again depends on the three dimensionless numbers defined above. From this we see that hydrogen dilution progressively reduces the deposition yield, as does a small ratio of dissociation to pumping rate, resulting in more and more of the silane being carried out by the hydrogen flow and then lost to the pump.

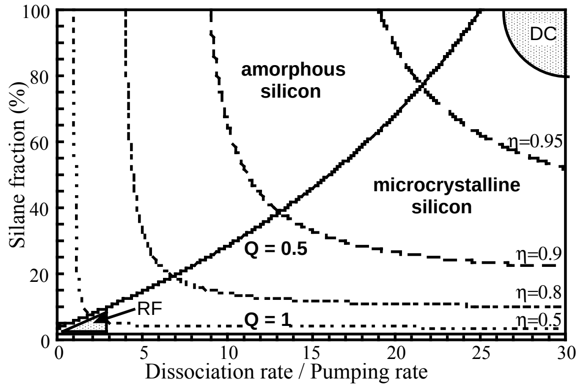

Figure 3 *The range of silane fraction (as a percentage of the total flow) vs the ratio of dissociation rate to pumping rate. The region for which microcrystalline silicon deposition is predicted lies between the boundaries Q = 0.5 and Q = 1 (bold lines). The dashed lines indicate different values for the deposition yield. The labels 'RF' and 'DC' mark the estimated parameter space for conventional high-dilution RF experiments, and for the high current DC plasma, respectively.*

The curves for deposition yields of 0.95, 0.9, 0.8 and 0.5 are represented in Fig. 3. For optimising the deposition rate, yield and microcrystalline quality, it is necessary to choose plasma parameters that give values as high as possible for the electron density and gas pressure (see ii) above), and then to use the highest possible silane fraction commensurate with $Q > 0.5$. Since Φ_{SiH_4} is in units of molecules·m^{-3}s^{-1}, the deposition rate is given by:

$$R\,[\text{Å/s}] = \eta R_{\max} \approx 10^{10}\,\eta \Phi_{\mathrm{SiH}_4} m_{\mathrm{SiH}_4} d \big/ (2\rho_{\mathrm{Si}})$$

where m_{SiH_4} [kg] is the mass of a silane molecule, ρ_{Si} [kgm^{-3}] is the density of the silicon film, and d [m] is the electrode gap (the factor of two is because the film is deposited on both electrode surfaces).

The gas composition of the plasma is also determined by Q, and hence by the same three dimensionless numbers. The ratio of partial pressures of molecular hydrogen and silane is:

$$\frac{n_{\mathrm{H}_2}}{n_{\mathrm{SiH}_4}} = Q\frac{2k_{\mathrm{SiH}_4}}{k_{\mathrm{H}_2}},$$

and since $0.5 < Q < 1$ for microcrystalline silicon deposition, this shows that the hydrogen gas density is always at least 25 times more than the density of silane. Therefore, we have the key result that *the gas composition of all silane / hydrogen plasmas suitable for microcrystalline deposition is almost entirely composed of hydrogen, independently of the flow rates used.* This key result is confirmed by infrared absorption measurements in the discharge of our large area reactor, where the absorption peak of the silane is very small for plasma conditions which produce microcrystalline silicon films.

Microcrystalline silicon can also be deposited using high power and pure silane, because even in this case, the plasma consists of almost pure hydrogen, the by-product of the dissociation and deposition reactions. The use of high power and pure silane is simply the extreme case of high RF power and restrictive dilution mentioned above. The VHF technique is well-suited to microcrystalline silicon deposition because of the high dissociation rates which can be achieved compared with the conventional 13.56 MHz frequency.

The important point is that the gas composition within the plasma is generally not the same as the relative composition of the input gas flowrates, because of plasma dissociation and radical deposition. Conditions for microcrystalline silicon thin film deposition therefore cannot be defined only in terms of the dilution used, but also require knowledge of the pressure, reactor volume, and plasma power density used. In practice, the high power, high pressure conditions may be limited by powder formation, a consequence of secondary reactions which are neglected in this model.

Silicon Tetrafluoride and Fluorine

A study of plasma deposition using gas mixtures of fluorine (F_2) and silicon tetrafluoride (SiF_4) gives an opportunity for comparison with the hydrogen (H_2) and silane (SiH_4) mixtures, with H replaced everywhere by F. Atomic fluorine is a strong etchant for silicon compared with hydrogen, and so it might be expected to facilitate the deposition of microcrystalline silicon. The comparison is especially interesting because the relative order of bond strengths between the pairs of molecules is reversed : The bond dissociation enthalpy of H-H (4.48 eV) is greater than that for Si-H (3.06 eV), whereas the bond dissociation enthalpy for F-F (1.6 eV) is much *less* than that for Si-F (5.57 eV). Therefore, F_2 / SiF_4 mixtures allow an investigation of a system with $k_{\mathrm{SiF}_4}/k_{\mathrm{F}_2} \ll 1$ instead of $k_{\mathrm{SiH}_4}/k_{\mathrm{H}_2} \gg 1$, which changes the third dimensionless factor mentioned above. However, inspection of the expression for Q now shows that $Q = 1$ for $\Phi_{\mathrm{F}_2}/\Phi_{\mathrm{SiF}_4} = 2k_{\mathrm{SiF}_4}/k_{\mathrm{F}_2} \ll 1$, and the etch rate is predicted to dominate over deposition except for a negligibly-small addition of F_2 to SiF_4. Experimentally, it was indeed found that no, or very slow, deposition occurs even with pure SiF_4 gas. This is not because fluorine passivates the growing surface, but because the liberated fluorine from SiF_4 dissociation removes the film as fast as it is deposited.

In conclusion, for these binary gas mixtures, it is necessary for the dissociation rate constant of the silicon-depositing gas to be less than that of the etchant gas. This is most easily satisfied when its dissociation threshold energy is below the threshold for dissociating the etchant gas, as is the case for silane / hydrogen. For this reason, silicon tetrafluoride / fluorine mixtures are unsuitable for microcrystalline silicon deposition. Mixtures of three or four of these gases is beyond the scope of the present simple model.

D. AN ALTERNATIVE PLASMA SOURCE

A High Current DC arc has been used to deposit microcrystalline silicon at extremely high rates, up to 100 Å/s, for substrate temperatures below the annealing temperature. The reactor configuration resembles a 'hot wire' device, except that the tungsten filament is replaced by a 50 cm-long plasma column (Fig. 4). Microcrystalline silicon is produced for all dilutions, even for no hydrogen dilution, with a mean crystallite size of 12 ± 3 nm. Infrared absorption spectra shown in Fig. 5 exhibit very narrow absorption peaks at 615, 2080 and 2100 cm^{-1}, whereas amorphous material exhibits broader absorption peaks, and a broad peak around 2000 cm^{-1}. Together with Raman scattering measurements, this indicates a high crystallinity in these films.

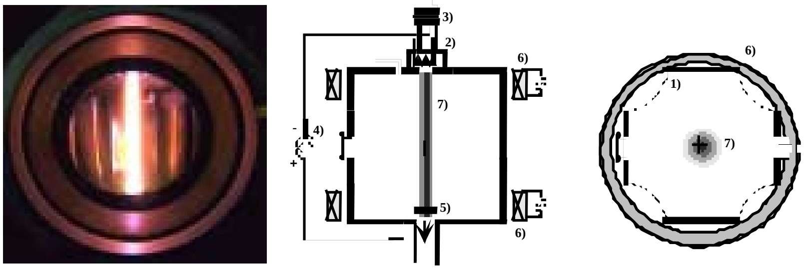

Figure 4 *Schematic and side view of the DC arc reactor*

This DC arc has $n_e \approx 10^{17-18}$ m^{-3} and p = 150 Pa for a flowrate : volume ratio comparable to the RF-PECVD example. The ratio of plasma dissociation rate to pumping rate, $n_e k_{SiH_4} / a_0$, is several hundred times larger than for the RF reactor, principally due to the elevated electron density. Referring to Fig. 3, this explains why the DC arc can deposit microcrystalline silicon ($Q > 0.5$) even for no hydrogen dilution, and consequently with a high degree of silane utilisation. One reason for the high electron density is that the DC plasma power, several kW, is much higher than for typical RF plasmas of comparable volume. Such a high power density in an RF plasma would cause film damage and stress from energetic ion bombardment across the capacitive sheaths, whereas the substrates in the DC arc are in the low ion energy diffusion zone outside the plasma column. The film adhesion is, in fact, excellent, even for a thickness of several microns, with none of the peeling often found for RF deposition using substrates which were not carefully cleaned beforehand. The material quality, tested at IMT Neuchâtel, suffers from rather high defect density, but it must be stressed that these were preliminary tests with no special attention paid to contamination or impurities. Also, post-hydrogenisation could potentially passivate defects. Furthermore, this type of reactor is not well adapted in its current form for uniform coverage of large areas. This technique has now been patented (number CH/794/99), and this interesting approach could be followed up in a future project, most probably in collaboration with Balzers AG.

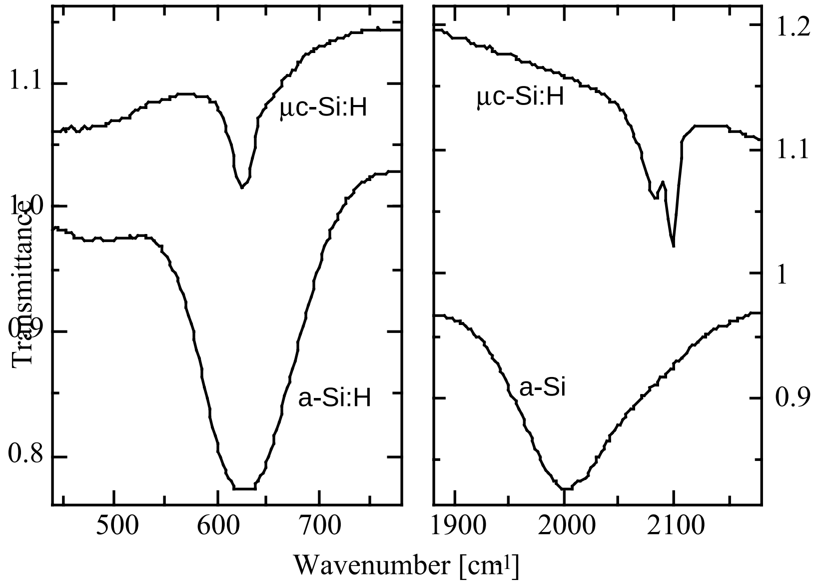

Figure 5 *Infrared absorption spectra for amorphous and microcrystalline silicon.*

5) COLLABORATIONS, CONTACTS WITH INDUSTRY

Our group continues to benefit from excellent relations with Balzers SA in Liechtenstein and Palaiseau, France (Drs J. Schmitt, J. Perrin, E. Turlot, J. Dutta and F. Leblanc). The topic of rapid growth of microcrystalline silicon by addition of silicon tetrafluoride was proposed to CRPP by our co-workers Drs. U. Kroll, H. Meier, and Professor Shah in the lnstitut de Microtechnique, Neuchâtel, within the framework of our long-standing collaboration. The CRPP also contributed the techniques of voltage uniformity measurement and modelling, and film homogeneity by whole surface interferometry, which were then presented as part of a joint paper "Fast Deposition of a-Si:H Layers and Solar Cells in a Large Area (40 x 40 cm) VHF-GD Reactor", U. Kroll, D. Fischer, J. Meier, L. Sansonnens, A. Howling, and A. Shah, at the MRS Spring Meeting, San Francisco 1999.

Student education at the CRPP during 1999 included the diploma work of Luc Chevalley and a Rapport de Stage by P. Brianceau in the framework of a collaboration with ESPEO Orleans. CRPP expertise in Cavity Ringdown Spectroscopy was transferred by F. Grangeon to the group of Dr. P. Roca in l'Ecole Polytechnique de Palaiseau, France, for investigation of nano-crystallite formation in plasmas and its possible contribution to film growth of polymorph solar cell material. CRPP data was sollicited by Professors S. Wagner (Princeton, USA) and A. Gallagher (Arizona, USA) for information on plasma powder production and negative ion formation respectively.

During the project, the CRPP also benefited from the help and expertise of several external collaborators. These include Dr. V. Nosenko from the Physics Institute in Kiev, Ukraine via a Schweizerische Akademie der Technischen Wissenschaften (SATW) exchange, C. Monard via a ‘stage’ from the GREMI laboratory in Orleans, Hiden Analytical, and Suzanne van Egmond from the Technical University of Eindhoven (funded by the group of

Professor Schram). International interest is witnessed by continued invitations to present the work in the USA, Germany and Japan, and by visits to the laboratory by companies such as Sanyo Electric.

The CRPP organised and hosted an international workshop: "Low Temperature Plasma Diagnostics" with the theme "Diagnostics of Industrial Plasmas" in Saillon, 15-19 February 1999. The scientific committee, speakers and participants represented a mix from university and industrial backgrounds to discuss the specific question of university/industry exchange and transfer. The workshop proceedings of 67 papers are available as Lausanne Research Paper LRP629/99 and on the conference website http://crppwww.epfl.ch/LTPD99. In addition, the First Swiss Plasma Meeting, organised and hosted by the CRPP, was held on 1 December 1999. The meeting brought together 23 industrialists and 22 university and technical school members to consider the possibility of a co-ordination of plasma research and applications in Switzerland. It is planned to have follow-up meetings alternately in industry and in academic institutions.

6) CONCLUSIONS AND PERSPECTIVES

The deposition of micro-crystalline silicon occurs in conditions of almost complete depletion of silane, where the plasma composition is essentially hydrogen. It is shown that a principal barrier to rapid microcrystalline silicon deposition in RF plasmas is the limited plasma power density for gas dissociation which can be applied without incurring destructively-high ion bombardment energies. The VHF method has a strong advantage over conventional frequencies in this respect. The novel High Current DC Arc technique provides extremely high deposition rates well beyond the range required by industry, provided that work on improving the material quality is carried out.

An analytical model has been developed to investigate the plasma chemistry of microcrystalline silicon deposition for silane diluted in hydrogen, and for silicon tetrafluoride diluted in fluorine. This description combines the various empirical approaches of RF and High Current DC arc in a single framework. It is shown why microcrystalline silicon deposition rates are much higher in a high power DC arc than in a conventional, hydrogen-diluted RF plasma.

Finally, thanks to the work carried out in this and previous OFEN projects, the CRPP and its collaborators in IMT Neuchâtel have been granted a CTI project: "Large Area and High-Throughput Coating System (PECVD) for Silicon Thin-Film Solar Cells" in collaboration with Europe's leading thin film deposition industry, Balzers Process Systems. The project aims to design and build a 1 m^2 plasma reactor suitable for industrial deposition of photovoltaic solar cells. This provides excellent continuity with the OFEN projects, and will be the next step in a realistic strategy towards creating an embryo industry for photovoltaic power generation in Switzerland.

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