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Quarterly Progress Report

DIAGNOSTICS OF A GLOW DISCHARGE USED TO PRODUCE

HYDROGENATED AMORPHOUS-SILICON FILMS

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During this quarter we have concentrated on developing and using film-deposition monitors to rapidly and accurately measure a-Si:H deposition at various surfaces of the discharge. We have succeeded in measuring deposition rates in-situ within a few minutes, so that many different discharge conditions can be probed in a short time. The deposition measurements have been carried out on a 250 C substrate as well as on room temperature surfaces. We have used the frequency shift of quartz crystals as the thickness monitors. This is a well established technique for film deposition in vacuum, but adapting it to deposition from discharges and at elevated substrate temperatures requires special procedures. As such in-situ measurements may also be of general use to other contractors, we will describe our method here. This is followed by some of the results.

Quartz crystal wafers, typically ~ 0.3 mm thick and ~ 2 cm diameter are used in most stable oscillators, and they are readily available and inexpensive. The crystal undergoes a coupled mechanical and electrical oscillation at a frequency $\nu = (1.67/D)$ MHz, where D is the crystal thickness in mm. It is coupled to the electrical circuit by a thin metal layer (generally vacuum deposited Au or Ag) on each of the two surfaces. The metal layers generally cover only a portion of each surface, and essentially only the section of the crystal with metal on both sides undergoes the mechanical thickness oscillation. If a film is deposited on

either side of this oscillating portion of the crystal, the mechanical frequency decreases by an amount $1.32 d \text{ Hz}/\text{\AA} \times (f/5\text{MHz})^2$, where d is the film thickness of a film of the density of crystal Si. The accuracy limitation is set by the temperature coefficient of the crystal frequency, which is $\sim 8 \text{ Hz}/^\circ\text{C}$. Thus deposition of a few $\text{\AA}$ thickness can be readily measured if the crystal temperature is stable.

In order to measure film deposited from a discharge, it is necessary to protect the crystal circuit from the discharge current and to correct for temperature changes induced by the discharge and by the deposition chemistry. This can be done by switching the discharges on for a minute or so, then off and waiting for the frequency to stabilize at the new value (generally a few minutes for our crystal mounts), or by measuring dv/dt during steady-state deposition once the initial thermal changes have stabilized (also a few minutes). We observe net crystal heating of typically $\sim 5^\circ\text{C}$ by the discharge.

We have mounted nominally 5 MHz crystals with the rf-ground surface exposed to the discharge, and in a light mount to allow rapid thermal equilibration. The transistor oscillator circuit is outside the vacuum system, and is connected to the back surface of the crystal by a single, unshielded lead (to minimize capacitance to ground). Lead lengths up to 50 cm have been used successfully, although there is a tendency for self oscillation to terminate sooner as a function of film thickness for longer lead lengths.

The present crystal mount design is shown in Fig. 1; this is a simple design chosen for ease of construction and assembly, while satisfying various criteria. First, it is desirable for the entire oscillating portion of the crystal obtain a uniform deposit, since the Q of the crystal will be decreased by thickness variations. For this purpose we have generally etched away portions of the two electrodes until overlap occurs only in the center, under the opening in the mount. (Crystals with electrodes of this form can also be purchased directly.) We have used the original Ag or Au electrodes since an a-Si:H film quickly covers the exposed electrode and deposition independent of electrode material is obtained. Next, the crystal is held in place with a weak spring force that preferably contacts the mechanically inactive area of the crystal. Either or both of the rf-ground and rf carrying electrodes can be coupled to the oscillator circuit by capacitors, so that the crystal may be floated in the discharge circuit or the crystal rf lead can be simply disconnected during discharge operation.

We have measured deposition rates at the screen cathode and at the substrate of a dc-proximity discharge in pure SiH_4 . We have then estimated the total deposition areas in order to calculate the total rate of silicon deposition. This has been related to the rate of SiH_4 depletion, so that the efficiency for producing coated Si is obtained, i.e. much of the depleted SiH_4 is pumped away as Si_2H_6 . This ratio r of deposited Si atoms to depleted SiH_4 molecules is represented by filled circles in Fig.

2, where it is plotted as a function of W/F , the discharge power (W) to flow (F) ratio. The fractional depletion f of SiH_4 by the discharge is determined by W/F ; f is plotted as a line in Fig. 2. This quantity f is an excellent basis of comparison for discharges of different powers, flow rates, geometries, and types (rf vs. dc), since it represents the amount of gas altered in the discharge. The measured ratio R of Si_2H_6 density to depleted SiH_4 density is also shown in Fig. 2. Since most of the depleted SiH_4 goes into a film or Si_2H_6 , $r+R \cong 1$ should be observed. This is indeed approximately seen at the higher values of f , but the measured $r+R$ exceeds one at the smaller f . We suspect that gas temperature and differential pumping speed effects cause this inconsistency in measuring f for small f values, but this has not yet been verified. Aside from this uncertainty, the low f data in Fig. 2 indicates that a significant portion ($\sim 30\%$) of the initially depleted SiH_4 deposits as an a-Si:H film, while the remaining $\sim 70\%$ forms Si_2H_6 and a small amount of the heavier silanes. (The neutral radicals leading to these products are always a small fraction of the total molecular density.) At the higher f values the Si_2H_6 is becoming a major fraction of the gas. Here the measured R is decreasing, indicating that much of the Si_2H_6 is being dissociated in the discharge before being pumped away. The D data then indicates that some of the radicals from this Si_2H_6 dissociation contribute to the deposition at the higher f values. The present $r+R \neq 1$ inconsistency precludes more detailed interpretation at this time.

Fig. 1. The holder is made from a piece of sheet stainless steel 1/2" by 1-1/2" by .004". A 1/4" diameter hole is punched in it, and a thin wire loop is spot welded around the hole. This will be the ground contact for the front of the metalized crystal. The holder is bent into the above shape with the loop on the inside and with enough spring tension to hold the crystal in place. The mica, rf lead wire, and the crystal are placed in the holder as shown in the side view diagram (which is not to scale).

Fig. 2. The line is the fractional depletion of SiH_4 . The lower points, $\times$ and Δ , are I/qFf , the fraction of depleted Si atoms that are carried to the cathode as ions. The filled circles, $\bullet$, are the fraction of depleted Si atoms that go into wall coatings. The upper points, $+$ and $\circ$, are the fraction of depleted Si atoms pumped away as Si_2H_6 . W is the discharge power and F the gas flow.