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BLACK a-Si:H SPUTTERED FILMS FOR PHOTOVOLTAIC SOLAR CELLS

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INTRODUCTION

From an examination of the reported literature of the preparation and characterization of a-Si:H films by rf-sputtering⁽¹⁻¹⁶⁾, it is clear that only a limited range of preparation variables have been studied even though the resulting physical properties cover a very wide range. As in the glow-discharge method, the plasma in the rf-sputtering process is complex and highly dependent on all the preparation parameters including system geometry and even the coatings on the wall of the system. Thus, optimum conditions established in one system will not necessarily be the same as that in any other system. In order to fully understand a sputtered thin film material, then, it is necessary to establish the complex preparation-structure-composition-property relations. Such is the basis of our general research approach.

During the first quarter, we established three important results:

1. The a-Si:H films could be anisotropically etched in a manner similar to that previously found for a-Ge^(17,18) and a-Si⁽¹⁹⁾. The resulting film microstructure is a dense array of columns with a spacing on the order of 1000-3000Å. The total surface reflectance is decreased from ~40% (for a smooth, as-deposited film) to ~3% for the etched film.
2. It has been shown that both a-Si and a-Si:H films can be classified according to the structure zone model⁽¹⁹⁾ and thus provides a means of comparing various samples made in a single study or samples from various laboratories. Since it is well established that the microstructure of a-Si:H films has an important effect on resulting film properties^(20,21), this classification scheme should be important. Further, the anisotropic etching behavior is useful in that it more clearly

reveals the microstructure such as in films which would otherwise display conchoidal fracturing. Also, the etching behavior is expected to be related to post-deposition oxidation and the porous nature of this material.

3. We have shown, for the first time apparently, that rf-sputtered a-Si:H film have a columnar microstructure similar to that reported for glow-discharge samples⁽²⁰⁾. The columns are aligned in the direction of the impinging vapor stream.

During the second quarter, we have extended our sputtering preparation experiments to include variations in the partial pressures of H₂ and Ar. A significant result is that we have demonstrated the wide control of Si-H bonding by variations in the sputtering gas pressure. In particular, we have prepared sputtered a-Si:H films which contain a predominance of monohydride units ($2000\text{cm}^{-1}/2090\text{cm}^{-1}$ IR peak ratio of $\sim 3-5$) and have no noticeable post-deposition oxidation as detected by IR measurements. The IR spectra for our samples prepared at 5mTorr total pressure (Ar + 0.22% H₂ and Ar + 1.7% H₂) are very similar to that reported by Knights et al.⁽²¹⁾ for their low power anode samples prepared by glow discharge, which were reported to have the lowest defect density, highest resistance to post-deposition degradation, and good physical property characteristics for device applications. In all previous reports (except the recent paper by Jeffrey et al.⁽¹⁶⁾; see text for details) on IR spectra of sputtered a-Si:H the highest 2000/2090 ratio has been ~ 1 ⁽²⁾. Since this represents a significant advance in the sputtering preparation technique, further evaluation on these samples is underway. The details of these and other results during the second quarter are given below.

EXPERIMENTAL RESULTS AND DISCUSSION

Film Preparation

A series of films were prepared by rf-sputtering in which both the partial pressure of H_2 (0-7 mTorr) and total gas pressure of $Ar + H_2$ (5-70 mTorr) were varied. Both the rf-power level ($0.8W/cm^2$; 100W on 12.5cm dia. Si target) and substrate-target distance (32mm) were held constant. In each of the tests, various substrates were placed for use in the different structural, chemical, and physical investigations.

Characterization

1. Etching Behavior. A number of chemical etchants have been evaluated experimentally with regard to capability of texturing film surface in such a manner as to reduce optical reflectivity in the active photon region and to reveal the microstructure of these a-Si:H films. These experiments have been performed for films prepared over the whole range of P_H for $P_T = 20$ mTorr (see First Quarterly Report for details of preparation). This total pressure is approximately that at which a-Si:H films with optimal photovoltaic properties have been prepared previously⁽¹⁾, and the range of P_H spans all practically possible conditions. Thus, the evaluation of etchants for this series of films can generally be expected to demonstrate the feasibility of chemical texturing, even though the number of films tested is limited in number and variation of preparation conditions. A summary of the results for each etchant type is given below. In all cases, etching was performed on films deposited on 1" x 1" glass slips. Film thickness was on the order of $10\mu m$. For each film tested, the time dependence of texturing effects was investigated by immersing overlapping regions, for example to give a set of immersion times of 20, 40 and 60 seconds for a single film in a single

etchant.

a. HF (reagent grade, 49%)

Etching response to this reagent is quite varied over the range of films studied. Although results are somewhat nonsystematic, the following generalizations appear valid. For low P_H , films are unstable (peeling and flaking off substrate), and no desirable etching effects are observed. Whether or not this instability is due to substrate effects (e.g., penetration of HF to the glass substrate with subsequent dissolution of the glass and solvent migration parallel to the glass surface) was tested by performing the same experiment on films prepared at the same time, but on stainless steel substrates. Results in this case were very much the same, however. For moderate P_H , films underwent surface texturing/erosion. Desirable effects were observed, with a dull purple-black finish in one case. For high P_H films appeared much less reactive, exhibiting slight film dissolution (without obvious texturing) even after 5-10 minutes immersion.

b. HNO_3 (conc):HF(49%): CH_3COOH (conc): H_2O

Etching response in this chemical system was studied in terms of a) $[\text{HNO}_3]/[\text{HF}]$, and b) $([\text{H}_2\text{O}] + [\text{CH}_3\text{COOH}])/([\text{HNO}_3] + [\text{HF}])$. That is, the oxidizing and solubility effects of HF and HNO_3 as well as the solubility and moderating effects of H_2O and CH_3COOH are being varied. Moderation is taken here to mean control of immersion period

required for a given reaction. Moderating is found to be quite important, e.g. combination of the above reagents can be found to completely dissolve 10 μ m-thick films in less time than the most rapid manual immersion - H₂O quench possible (i.e. approx. 1 sec.).

For HNO₃:HF:H₂O, (20:4:1 vol) to 20.4:9), for a given etchant, decreasing film reactivity with increasing P_H is seen. For intermediate P_H, significant darkening is observed (very sensitive to immersion period), while for higher P_H one can etch, in steps, completely through the film to the substrate without great difference in surface texture. Darkened surfaces are in some cases obtained in 1-2 second immersions. Increasing [H₂O] over the range specified above, increased the length of time required (a few-fold) for an equivalent amount of etching, but appears to give less blackening. Also, the effect of increasing [H₂O] appears to "saturate" rather quickly, that is, the moderating effect of H₂O appears over a relatively small region, and further increases in [H₂O] tend to give essentially "no reaction."

For HNO₃:HF:CH₃COOH:H₂O, the most significant result is the ability of CH₃COOH to moderate without significantly changing the nature of the texturing reaction. For example, for moderate P_H films, for HNO₃:HF:CH₃COOH:H₂O (1:1:X:0), varying x from 0 to 8, one can change reaction time from a few seconds to a few minutes, and yet give approximately the same "blackness."

It is concluded that 1) any of the films may be textured to yield a (qualitative) significantly reduced reflectivity, the exact reactivity being fairly well controlled by varying $[HF]/[HNO_3]$, and 2) $[CH_3COOH]$ can be employed to moderate texturing without significantly altering the actual texture.

c. 20% NaOH (90°C)

Texturing effects are observed here. Total time of immersion required is generally 5-10 minutes, increasing with P_H . The best decrease in reflectivity is observed as a grayish, matte appearance. From the general appearance of some films with prolonged immersion the question of actual bulk chemical change is raised.

d. 30% KOH (90°C)

Somewhat surprisingly, the reactivity of 30% KOH is apparently less than that of 20% NaOH (both 90°C), although final appearance of etched surface is pretty much as that produced by NaOH.

e. HCl (conc)

No reaction apparent.

f. Na_2O_2

No reaction apparent.

Etching experiments on the samples prepared during this quarter, as described above, are still in progress and will be described in the next report.

2. IR Spectroscopy. Infrared absorption spectral measurements have been used extensively for both chemical and microstructural analysis of a-Si:H thin films. Although better techniques exist for obtaining the chemical information ^(22,23), they have been used primarily to "calibrate" the IR data. Because of its wide use, IR spectroscopy provides a convenient tool for comparing not only our set of samples but also for comparison with films prepared by other investigators.

The IR transmission spectra for the films prepared at 5, 20, 40, and 70 mTorr total gas pressure (Ar + H₂) are given in Figs. 1, 2, 3, and 4, respectively. The films were deposited onto polished silicon crystal wafers in which one side was slightly roughened in order to minimize interference effects. In each figure there are spectra presented for the samples prepared at various partial pressures of hydrogen (P_H) and is represented as a percentage of the total gas pressure (P_T).

$$(P_H\% = \frac{P_H}{P_T} \times 100).$$

The film thickness, as measured by a Talysurf 10 profilometer, is also given. Since these transmission spectra are neither normalized to equal thickness nor converted to spectral absorption coefficients, (both of these conversions are in progress) we will now discuss only the general comparative features.

The IR transmission spectra were taken between 1-2 months after the samples were prepared. Although the samples were stored in a vacuum dessicator, the total exposure to air between preparation and measurement exceeds several days in all cases. Anderson et al.⁽¹⁾ have demonstrated the air-exposure time-dependence on IR spectra and indicate that the samples stabilize after several days. This

stabilization process was attributed to post-deposition oxidation. Most of their reported data is on "stabilized" samples and thus our data should be comparable.

IR absorption and Raman studies on a-Si:H films^(5,14,24,25) have led to an understanding of Si, O, and H bonding in these materials. In the following, we will discuss the assignments of each of the observed absorption modes, relate these to our set of data (Fig. 1-4) and discuss trends and their significance.

- An absorption peak at 480cm^{-1} , apparently due to a fundamental Si-Si stretching vibration⁽⁵⁾ is present in all our spectra and has no obvious relation to the Si-H bonding, as discussed below. This is similar to previous reports.
- The broad absorption band at $630\text{-}630\text{cm}^{-1}$ is also seen in all spectra. It has been attributed to both an SiH group wagging mode and an SiH₂ group rocking mode, the former being associated with an SiH group bending mode at 2000cm^{-1} . The sharper peak centered at 630cm^{-1} and seen in a number of the spectra is apparently due to an absorption in the crystalline Si substrate and can be disregarded. This $630\text{-}650\text{cm}^{-1}$ band is qualitatively similar for all the samples. At the high P_T high P_H conditions this peak is considerably reduced, likely due to low H-incorporation.
- In the $800\text{-}1050\text{cm}^{-1}$ range there is a broad-band mode due to bulk oxidation⁽¹⁾. Also, in this region there are several Si-H modes between 800 and 950cm^{-1} which

stabilization process was attributed to post-deposition oxidation.

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will be described below. The Si-O mode is due to oxidation occurring during-deposition. Another broad peak at $1000\text{-}1200\text{cm}^{-1}$ has been assigned to surface oxidation, presumably due to oxidation down the void networks and occurring post-deposition⁽¹⁾. This peak is due to the stretch modes of the SiO_2 groups in which the antisymmetric and symmetric stretch modes are at 1100 and 1200cm^{-1} , respectively. This mode separation can be seen in our spectra. The 5mTorr samples are highly resistant to post-deposition oxidation and show only bulk oxidation ($800\text{-}1050\text{cm}^{-1}$ mode). The higher P_T samples, however, show considerable post-deposition oxidation in addition to bulk oxidation. There is a direct relation between the post-deposition oxidation and the etching behavior, which will be discussed in the next quarterly report. Shimizu et al.⁽²⁶⁾ have reported similar results for rf-sputtered a-Si films (without H) although they offered no explanation. Their films prepared at higher gas pressures gave an IR absorption band at $1000\text{-}1200\text{cm}^{-1}$ while films at low pressures, but with oxygen intentionally added during deposition had a broad absorption only at $800\text{-}1050\text{cm}^{-1}$.

- There are four absorption peaks in the $800\text{-}950\text{cm}^{-1}$ regime which consist of two overlapping doublets⁽²⁴⁾. The 845 and 890cm^{-1} doublet is associated with the 2090cm^{-1} mode (which can shift $\pm 10\text{cm}^{-1}$ depending on

conditions) and is due to $(\text{SiH}_2)_n$ chain like groups. The higher frequency doublet (862 and 907cm^{-1}) is related to deformation modes of the SiH_3 group (degenerate and symmetric deformation, respectively). Due to the closeness of the two doublets they generally appear to be a single doublet. Our current IR data isn't sufficiently resolved to separate these doublets and thus we cannot distinguish between $(\text{SiH}_2)_n$ and SiH_3 bonding at this point. More detailed work is in progress.

- The three modes clustered at 2000 , 2090 , and 2140cm^{-1} , as discussed above, have been assigned to SiH , $(\text{SiH}_2)_n$, and SiH_3 group bonding-modes, respectively. For the 5mTorr ($0.22\%\text{H}$) sample, we see no distinguishable $845\text{-}890\text{cm}^{-1}$ modes, which corresponds with the virtual absence of any multiply H-bonded modes at either 2090 or 2140cm^{-1} . The dominant SiH bending mode at 2000cm^{-1} is clearly associated with the $630\text{-}650\text{cm}^{-1}$ SiH wagging mode. With increasing P_{H} (1.7%) at 5mTorr the 2090cm^{-1} mode becomes distinguishable and is accompanied by an emerging peak at 890cm^{-1} . With increasing P_{T} the 2090cm^{-1} peak increases along with an increase in the $800\text{-}950\text{cm}^{-1}$ range doublets. The more detailed interrelationships between these modes and the preparation conditions are being studied.
- Modes at $\sim 2250\text{cm}^{-1}$ and $\sim 2900\text{cm}^{-1}$ have been reported but not assigned. Anderson et al.⁽¹⁾ and Freeman and Paul⁽⁵⁾ both show that the $\sim 2250\text{cm}^{-1}$ peak increases with post-

deposition film degradation while the latter further shows the simultaneous occurrence of three peaks at $\sim 2900\text{cm}^{-1}$.

We also see both modes only in samples which show extensive post-deposition degradation (40 and 70mTorr samples).

However, from our spectra, there appears to be no direct relation between these two peaks.

From the above IR transmission spectral results, we have clearly demonstrated the wide range and control of the Si-H bonding in a-Si:H sputtered films by varying P_H and P_T . Furthermore, we have now shown that a-Si:H sputtered films can be prepared with a predominance of monohydride units. Similarly, Jeffrey et al.⁽¹⁶⁾ have shown that by increasing the rf-power level sputtered films can be attained with high monohydride/dihydride ratios. Until recently, it was believed that sputtered films might be intrinsically inferior to glow discharge material due to an impossibility to completely breakdown the polysilane units in the rf-sputtering deposition process⁽²⁷⁾.

Not only do the present results dispell this pessimism, but, along with Jeffrey et al.⁽¹⁶⁾ results, indicate that there may well be a wide range in preparation-parameter-space where monohydride can be predominantly attained and controlled. Jeffery et al. suggested that high energy ions and neutral atoms incident at the growing film either prevent or destroy dihydride bonding configuration and thus is a beneficial effect. Anderson et al.⁽¹⁾ and Pawlewicz⁽¹¹⁾, on the other hand, argue that bombardment effects are detrimental and should be minimized. Although we have no direct evidence at present, our previous experience⁽²⁸⁾ indicates that high self-bias voltages are attained at low total gas pressures (eg. 5mTorr), which can lead to significant bombardment effects. Such measurements are currently in progress. Also, tests are being designed

Figure 1 IR Spectra of rf-sputtered a-Si:H films prepared at a fixed $P_T = 5\text{mTorr}$ and at various P_H (as expressed as a percentage of P_T). The film thickness is also given.

Figure 2 IR Spectra at $P_T = 20\text{mTorr}$

Figure 3 IR Spectra at $P_T = 20\text{mTorr}$.

Figure 3 IR Spectra at $P_T = 40\text{mTorr}$

Figure 4 IR Spectra at $P_T = 70\text{mTorr}$

to better understand bombardment effects and are discussed in our renewal proposal.

Several remaining points will now be discussed. First, the 0% samples (sputtered in pure Ar) with increasing P_T have an increasing (and significant) H-content and show an increasing post-deposition degradation. It is not yet determined whether the hydrogen is due to post-deposition hydration or incorporation during deposition due to either pump oil backstreaming or water vapor desorption from the system walls. These results seem to have some bearing on Pawlewicz's⁽¹¹⁾ somewhat surprising conductivity and optical absorption results. Second, the 70mTorr samples had a lower deposition rate in general. The 70mTorr (10%H) test, in particular, had an extremely low deposition rate. [See film thicknesses; all tests were run approximately the same times.]. The absence of any Si-H modes could be due in part to the low thickness, although low H-content is also likely.⁽¹⁾

3. Hydrogen Analysis by SIPS/SIMS. In the course of using sputter-induced photon spectrometry (SIPS) to characterize hydrogen in the a-Si:H films, we have encountered the problem of a high background which makes quantification of hydrogen difficult. This background signal is due to residual hydrogen in our present vacuum system, 1×10^{-7} torr, and reflection of the photon emission by the sample surface. To overcome this background problem in hydrogen detection, we have undertaken to design and build a new surface analytical instrument featuring the combination of two ion-beam sputtering techniques, SIMS and SIPS. A schematic diagram is shown in Fig. 5. The ion beam system consists of a Colutron ion source, mass filter and two electrostatic lenses and differentially pumped with a turbomolecular pump.

The target chamber can be evacuated to $\sim 10^{-10}$ torr with a turbo-molecular pump and titanium sublimation pump. The secondary ions are detected with a mass spectrometer which is an Extranuclear quadrupole

type with a specially designed hemispherical energy filter. The photons emitted from the excited sputtered atoms are detected with a Princeton Applied Research optical multichannel analyzer. Both the operation of SIMS and SIPS are computer-controlled. Other novel features include a secondary ion imaging system which generates a picture of elemental distribution on the sample surface, and an optical microscope which directly views the sample with transmitted or reflected light.

The uniqueness of this instrument lies in the fact that the collection of both secondary ions and emitted photons is completely maximized without sacrificing the sensitivity of either technique, and that SIMS and SIPS data can be collected simultaneously on the same ion-bombarded region. The primary ion beam can be focussed to a diameter $\sim 5\mu\text{m}$. In the depth-profiling mode of operation, the beam is rastered across a square pattern and electronically gated so as to avoid contributions from the walls of the bombarded crater. All elements in the periodic table from hydrogen to uranium can be detected with isotopic information.

The new SIMS-SIPS instrument will be completed shortly and should see operation some time in April 1980. Quantitative analysis of hydrogen in a-Si:H films should not be a problem with this new system.

WORK IN PROGRESS

During the next quarter, our preparation experiments will be on doping of our a-Si:H sputtered films. Most of our effort, however, will be directed toward the characterization of the series of films prepared during this quarter. This detailed characterization will include the following:

1. IR Spectroscopy. Transform transmission data to absorption coefficients and do detailed comparative analysis of the data. Measure long range time dependence of spectra.
2. Etching behavior. The relation of anisotropic etching to film microstructure, reflection reduction, preparation parameters, and properties will be studied.
3. Chemical analysis. Si, Ar, and O will be measured by electron microprobe analysis. Both nuclear reaction analysis (at Oak Ridge National Laboratory) and interpretation of IR absorption data will be used for determining H-content.
4. Optical absorption. Optical bandgap measurements will be made and compared.
5. Our preliminary atom probe field ion microscope studies of a-Si:H films will be reported.
6. Deposition rate and macroscopic film stress.

Figure 5 Block diagram of the SIMS-SIPS system

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