THIN FILM POLYCRYSTALLINE SILICON SOLAR CELLS
QUARTERLY REPORT NO. 1

1 OCTOBER 1979 - 31 DECEMBER 1979

PREPARED FOR

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UNITED STATES
DEPARTMENT OF ENERGY
DIVISION OF SOLAR TECHNOLOGY
AND
SOLAR ENERGY RESEARCH INSTITUTE

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MOTOROLA PROJECT NO. 2377

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PREFACE

This Quarterly Report No. 1, covering the period October 1, 1979 to December 31, 1979, was prepared by the Solar Energy R&D Department, I. A. Lesk, Manager, Motorola Inc., Phoenix, Arizona 85008. This report describes the work performed under SERI Contract No. XS-9-8277-1. K. R. Sarma is the principal investigator. Others who participated in this program are R. W. Gurtler, M. J. Rice, R. Legge, B. Sopori, R. J. Ellis and W. Salina.

ABSTRACT

During this first quarter, the MoSi_2 separation layer growth rate has been studied as a function of time and temperature. The presence of small amounts of O_2 in the silicon deposition ambient were found to inhibit the growth rate of the MoSi_2 layer and also to affect the reliability of shear separation. Void formation in silicon at the Si- MoSi_2 interface, due predominantly to diffusion of silicon through the MoSi_2 layer was observed. This is believed to be responsible for shear separation occurring in the silicon film. Gas chromatographic procedures were developed for characterizing the silicon deposition process. Coherent twin bundles in the grain-enhanced silicon films were not found to adversely influence solar cell efficiency. Several 1 cm x 2 cm solar cells were fabricated. Performance characteristics of these cells are discussed; the best device had a conversion efficiency of 10.7% (under simulated AM1 illumination) with $V_{\text{OC}} = 0.545$ V, $J_{\text{SC}} = 28.65 \text{ mA/cm}^2$ and $\text{FF} = 68.3\%$.

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1.0 INTRODUCTION

The objectives of this research program are: a) investigation of a semi-continuous plasma deposition system as a means for efficient, high rate deposition of polycrystalline silicon films, b) development of low-cost reusable substrates for deposition and shear separation of the silicon films, c) optimization of subsequent grain enhancement of the films through laser RTR recrystallization, and d) demonstration of at least 12% efficient, large area (greater than 10 cm^2) solar cells fabricated on grain-enhanced silicon films.

During this first quarter, growth rates of MoSi_2 separation layers as functions of time and temperature were determined experimentally. Effects of small amounts of oxygen in the deposition ambient, on the MoSi_2 layer growth rate and shear separation were also determined. Sources of O_2 in a continuous deposition system can be from a lack of complete effectiveness of the gas seals, and from the N_2 used in the gas seals. Further structural characterization of interfaces in the Mo TESS substrate was necessary to determine the shear separation mechanism.

Several experiments were performed in the semicontinuous plasma deposition system, to verify key concepts of continuous silicon film deposition processes. Gas chromatographic procedures were developed for analysis of exhaust gases from this silicon film deposition reactor.

Effects of twin bundles (with (111) twin planes) in the grain enhanced silicon films on solar cell conversion efficiency were investigated. Several $1 \text{ cm} \times 2 \text{ cm}$ area solar cells have been fabricated. The performance characteristics of these solar cells are discussed.

2.0 SUBSTRATE STUDIES

During this quarter, a study of the interaction of Mo substrates with CVD silicon was continued to better determine the mechanism of shear separation of the silicon from the substrate.

2.1 RATE OF MoSi_2 LAYER GROWTH

It was previously shown (1) that formation of MoSi_2 was required for reliable shearing of CVD silicon films from temporary Mo substrates. The rate of MoSi_2 formation was determined by measuring the thickness of the silicide layers on sectioned samples with a microscope. Samples were prepared by depositing silicon at temperatures of $1000^\circ - 1200^\circ\text{C}$ for reaction times of 10 to 90 minutes. For reaction times of greater than 90 minutes samples were subjected to successive 90 minute depositions. Samples were mounted and polished by standard metallographic techniques. It was found that staining the sections with an FeCl_3 solution (10 g. FeCl_3 , 2 ml. HCl , 95 ml. H_2O) for a few seconds aided in identifying phase boundaries.

Data for the growth of MoSi_2 are presented in Figure 1. The plot of thickness squared vs. time in Figure 1 indicates that the kinetics follow a parabolic law, except for the initial portion of the reaction since the extrapolated curve does not pass through the origin. Experimental points for short times are high since it was not possible to quench the samples in the apparatus used, so that the small amount of growth which continued during cool down was a significant amount of the total growth.

Figure 2 shows a least square fit for the MoSi_2 growth rate at 1150°C . Also shown are the data of Gage and Bartlett (2) for comparison. They used a pack cementation method of silicide formation and obtained a higher reaction rate. Their growth rate was also proportional to $\text{time}^{\frac{1}{2}}$, indicating a diffusion limited

FIGURE 1

(MoSi₂ Thickness)² vs. TIME

FIGURE 2: MoSi₂ thickness as a function of time at 1150°
G&D - Data of Gage and Bartlett.

reaction. The difference may be ascribed to the fact that in the CVD process the initial stages of the reaction are controlled by the nucleation of silicon, island formation, and surface coverage before the diffusion of silicon becomes the rate-determining factor.

2.2 EFFECTS OF OXYGEN ON SILICIDE FORMATION

Several experiments were performed in which small amounts of oxygen were added to the process gases during deposition. Figure 3 shows the silicide thickness obtained from these experiments compared with the thickness obtained under normal conditions. Two effects are observed from the addition of oxygen; the rate of formation of MoSi_2 is reduced, and the silicon does not shear when oxygen is added throughout the deposition. The effect of oxygen is therefore postulated as impeding silicon diffusion through the MoSi_2 layer.

2.3 MECHANISM OF SHEAR SEPARATION

Successful shearing of silicon ribbons from Mo substrates was found to have a time-temperature dependence suggestive of a diffusion process. When depositions were carried out at $1000 - 1050^\circ$, shearing did not take place reliably. However, a 1050° deposition followed by 15 - 30 minutes of anneal at 1200° resulted in shearing. One Mo substrate with a strongly adherent Si ribbon was annealed at 800°C . When cooled after 80 hours, 3/4 of the ribbon sheared. After 40 additional hours at 800°C , the remaining ribbon sheared.

Van Gorp (3) made the generalization that, for transition metals, silicon is the primary diffuser in silicon-rich silicides and the metal is the primary diffuser in metal-rich silicides. With MoSi_2 , silicon would then be expected to diffuse faster than Mo. This would cause vacancies in the silicon adjacent to the MoSi_2 surface; these vacancies would then coalesce into Kirkendall voids. Figure 4 is an SEM

FIGURE 3: EFFECT OF OXYGEN IN PROCESS GASES ON MoSi₂ Growth

FIGURE 4: VOID IN SILICON ADJACENT TO
MoSi₂-Si INTERFACE

photograph of a void in the silicon, and Figure 5 shows voids formed in the Mo adjacent to the Mo_5Si_3 layer. Similar void formation was observed by Borders and Sweet (4) in Si-Ge alloys with tungsten films, in which Si was the primary diffuser. The voids, caused by the diffusion of Si act as stress raisers when the substrate is cooled. The stress can be as much as three times greater (5) at the edges of the voids, depending on their size and shape, than in the bulk. It is postulated that the large stress due to the different expansion coefficients of Mo and Si, further concentrated by the voids, causes fracture to occur in the silicon immediately adjacent to the MoSi_2 layer.

2.4 SILICON NUCLEATION BEHAVIOR

It had been observed during many depositions that a used Mo substrate produced more uniform silicon ribbon with more reliable shearing. A similar effect was observed with substrates coated with a Si_3N_4 film. In contrast, when investigating barrier layers of SiO_2 , it was found that uniformity was poor and it was ascribed to poor nucleation. Figures 6 - 8 show ribbons grown on different surfaces. The samples with poor nucleation are extreme cases, but illustrate these difference very well.

Figure 9 shows 100X SEM photographs of 5 minute depositions on various surfaces. Nuclei were counted and the following results obtained

<u>SURFACE</u>	<u>N</u> (No./cm ² sec)
SiO_2	19
Mo (virgin)	34
Si_3N_4	111
Mo (N_2 added to gas)	283
MoSi_2	general coverage.

FIGURE 5: VOIDS IN Mo AT Mo_5Si_3 INTERFACE.

Extreme case of
Poor Nucleation on
Virgin Mo. 2X

Deposited on
 MoSi_2 coated Mo
2 X.

Figure 6

Extreme case of
Poor Nucleation on
 SiO_2 Coated Si.
2X.

Figure 7

Deposited on
 Si_3N_4 Coated Mo
2X.

Deposited on
 MoSi_2 coated
Mo
2X.

Figure 8

Mo

SiO₂

Si₃N₄

MoSi₂

FIGURE 9: NUCLEATION OF Si ON VARIOUS SURFACES

The MoSi_2 surface was best for nucleation, followed by Mo in which a small amount of nitrogen was added to the reactant gases. The Si_3N_4 surface was significantly better than virgin Mo or SiO_2 surfaces, confirming the previous observations.

3.0 PLASMA DEPOSITION

During this quarter, several experiments were performed using the semi-continuous plasma deposition system developed during the previous quarter (1). This system was designed around a square cross-section quartz reaction chamber. Silicon films approximately 5 cm wide, with lengths up to 60 cm, can be produced from the Mo substrates translated continuously on one wall of the deposition chamber. The other three walls of the quartz chamber are covered with stationary Mo substrates to prevent their devitrification due to silicon deposition. While our ultimate goal is to utilize all the silicon deposited in the system by continuous translation of substrates on all walls of the deposition chamber, operation of the present semicontinuous system serves to aid in the verification of key concepts of continuous silicon film deposition processes.

The concept of gas barriers (alternating series of N_2 and vacuum curtains) for sealing the deposition system from the outside ambient was successfully verified. The concept of continuous shear separation was also successfully demonstrated. In a batch type deposition system, the substrate is maintained at a uniform temperature during silicon deposition, and it is cooled uniformly to separate the silicon film from the substrate, all at one time. However, in a continuous system, as the substrate comes out of the deposition system, silicon film separation should initiate at the leading edge of the substrate and propagate slowly to the other end of the substrate. A successful progressive (continuous) shear separation has been inferred by our ability to produce 60 cm long silicon films without breakage, using the present deposition system. When the leading edge of the 60 cm long substrate (with the deposited silicon film separated) comes out of the deposition system, silicon deposition is still taking place on the other end of the substrate.

To investigate whether silicon film etching reactions were occurring at the down-stream end of the deposition system, the direction of substrate travel was reversed in some experiments. The substrates were introduced from the gas exhaust side and withdrawn along with the separated silicon films from the plasma introduction side. Some problems were encountered in these experiments. By the time the Mo substrates (with deposited silicon films) reached the plasma introduction end, silicon film separation had occurred. The plasma arced to the thin separated silicon film, causing it to melt and form spheres of silicon. The separated silicon ribbons (with the spheres attached to them) could not come out of the clearance provided in the gas curtains without breakage. Thickness measurements made on the broken silicon films did not indicate any significant effect due to direction of substrate travel. Hence, instead of pursuing this further, we have gone back to the initial substrate travel direction (along the direction of the plasma).

One of the limitations of the present semicontinuous system was found to be the length of time it could be run continuously. Progressive silicon build-up on the three passive walls of the reactor impedes smooth substrate travel along the active wall, thus forcing the run to be terminated after about 4 hours. However, this problem will not be present when all four walls are made active to produce four silicon sheets simultaneously.

Gas chromatography (G.C.) is a valuable analytical technique for characterization of a conventional CVD or a plasma CVD process. Application of GC for characterization of silicon CVD processes has been described in the literature (6). Since the sample gas to be analyzed is cooled to the GC column temperature ($\approx 100^{\circ}\text{C}$), this technique is not suitable for studying chemical equilibria in the high temperature silicon CVD (or plasma CVD) environment. However, GC analysis of the cold exhaust gas can be used to obtain reactant utilization as shown below.

The gas sampling arrangement for GC analysis is shown schematically in Figure 10. Exhaust from the plasma deposition reactor is split into two streams as shown in Figure 10. The large gas stream is sent to the burn-off box. The small gas stream is sent to the six-port gas sampling valve in a 5840 Hewlett Packard Reporting Gas Chromatograph with a thermal conductivity bridge detector. The GC column was about 10' long, and 1/8" O.D. and made of 316 stainless steel. The column was packed with 80 - 100 mesh chromosorb-W solid support; the solid support was loaded by about 20% with SF-96 liquid. When the gas sample (containing a mixture of components) to be analyzed is injected into the helium carrier gas, passing through this column, the components in the sample separate according to their distribution coefficient with respect to the He carrier gas and the SF-96 absorbent liquid phase. The separated individual gas components in the sample come out of the end of the column in a specific order. The thermal conductivity detector (TCD) detects the individual components as they emerge from the column. It is necessary to calibrate the instrument with known components for their retention times in the column and the detector bridge signal response factors. The detailed theory and practice of gas chromatography can be found in many of the standard books on the subject such as reference (7).

FIGURE 10: SCHEMATIC SHOWING THE GAS SAMPLING ARRANGEMENT FOR GC ANALYSIS.

The thermal conductivity detector (TCD) temperature was maintained at 85°C during analysis. The column oven temperature is time programmed as shown in Figure 11, to effectively separate the chlorosilanes and yet complete the analysis in a short time.

Figure 12 shows the chromatogram of the exhaust gases from the deposition reactor when the input gases were H₂ (30 LPM) and SiHCl₃ (9 grams per min.) and the furnace temperature was 1000°C, in the absence of the plasma (RF power = 0). The big negative peak is thought to be due a combination of the pressure peak and hydrogen peak. The retention times of HCl, SiH₂Cl₂, SiHCl₃ and SiCl₄ are 1.82, 3.14, 4.02 and 5.14 min. as indicated in the figure. From the calibrated response factors of these gases for the TCD signal, the composition of the exhaust gas stream was found to be: HCl = 45.8%, SiH₂Cl₂ = 2.5%, SiHCl₃ = 35.7% and SiCl₄ = 16.0% (by volume or mole basis). Thus this analysis indicates relative percentages of various components in the exhaust gas stream. Similarly Figure 13 shows the chromatogram with the plasma beam (RF power = 2.5 KW), under otherwise identical conditions. From this chromatogram, the composition of the exhaust stream is found to be: HCl = 51.3%, SiH₂Cl₂ = 1.2%, SiHCl₃ = 28.3% and SiCl₄ = 19.2%.

Knowing the Si/Cl ratio in the input gas stream, $(\frac{Si}{Cl})_{in}$, and the exhaust gas stream, $(\frac{Si}{Cl})_{out}$, the efficiency of silicon deposition, η , can be calculated from the equation:

$$\eta = \frac{Si/Cl_{(in)} - (Si/Cl)_{out}}{(Si/Cl)_{in}} \times 100 (\%)$$

When using SiHCl₃ as the input silicon bearing gas $(\frac{Si}{Cl})_{in}$ will be equal to

$\frac{1}{3} \cdot (\frac{Si}{Cl})_{out}$ can be calculated as follows:

FIGURE 11: TEMPERATURE PROGRAM EMPLOYED FOR COLUMN OVEN.

FIGURE 12: CHROMATOGRAM OF THE EXHAUST GAS WITHOUT PLASMA (RF = 0 KW) WHEN H_2 = 30 LPM, $SiHCl_3$ = 9 GPM and FURNACE TEMPERATURE = 1000°C.

FIGURE 13: CHROMATOGRAM OF THE EXHAUST GAS WITH THE PLASMA BEAM (RF = 2.5 kW)
WHEN H_2 = 30 LPM, SiHCl_3 = 9 GPM AND FURNACE TEMPERATURE = 1000°C .

$$\left(\frac{\text{Si}}{\text{Cl}}\right)_{\text{out}} = \frac{100 - m_{\text{HCl}}}{m_{\text{Cl}}}$$

Where $m_{\text{Cl}} = m_{\text{HCl}} + 2 m_{\text{SiH}_2\text{Cl}_2} + 3 m_{\text{SiHCl}_3} + 4 m_{\text{SiCl}_4}$, and m_{HCl} , $m_{\text{SiH}_2\text{Cl}_2}$, m_{SiHCl_3} and m_{SiCl_4} are mole percentages of HCl, SiH_2Cl_2 , SiHCl_3 and SiCl_4 in the exhaust gas stream as determined by GC analysis.

$$\therefore n = \frac{\frac{1}{3} - \frac{100 - m_{\text{HCl}}}{m_{\text{Cl}}}}{1/3} \times 100$$

$$\text{i.e. } n = \left[1 - \frac{1}{3 m_{\text{Cl}}} (100 - m_{\text{HCl}})\right] \times 100\%.$$

Using the above equation, the efficiency of silicon deposition was calculated to be 32.4% and 26.7% with and without the plasma beam respectively under the set conditions described above. These values appear to be in agreement with the efficiencies calculated from weight gain measurements in the horizontal plasma deposition system under similar conditions (1). We plan to continue this work for a detailed characterization of the deposition process as a function of composition and flow rate of the input reactant gases, furnace temperature, and RF power of the beam.

5.0 GRAIN ENHANCEMENT AND CHARACTERIZATION

A detailed microstructural analysis of grain enhanced silicon films was initiated with a view to identifying specific features that can have a significant influence on the solar cell conversion efficiency. Because of the very large grain size (1) in grain enhanced silicon films, the performance of a solar cell is not expected to be significantly affected by the grain boundaries. Twin boundaries, however, were found to be quite numerous in some regions of the grain enhanced films. Figure 14 shows an SEM photograph of a region containing closely spaced twin boundaries (twin bundles).

To determine the effect of these twin bundles on solar cell efficiency parameters, solar cells were fabricated on grain enhanced ribbons containing regions of twin bundles as shown in Figure 15. By x-ray back reflection Laue measurements, these twin boundaries were found to be (111) planes.

The dark and illuminated (simulated AM1) I-V characteristics of such a cell (V143-1128E) are shown in Figure 16. This cell was sectioned into three regions as shown in Figure 18 and the individual pieces labelled as A, B, and C; thus, only region B contains a high density of twin bundles. The numbers indicated in Figure 18 are minority carrier diffusion lengths (measured by OCPV) in the regions indicated. Table 1 shows the results of the analysis of regions A, B, and C.

Under AM1 illumination, the performance of region B appears to be superior to that of regions A and C. Columns J_{SC} (Blue) and J_{SC} (Red) in Table 1 are the relative blue ($\lambda = 4400\text{\AA}$) and red ($\lambda = 9000\text{\AA}$) responses from regions A, B, and C. The blue response results from carriers collected from the n+ diffused region and the depletion region only, since the penetration depth ($1/\alpha$) is only $0.4\text{ }\mu\text{m}$. However, penetration for $9000\text{\AA}$ is $31\text{ }\mu\text{m}$ and hence the short circuit current for this wavelength is directly related to the minority carrier diffusion length in the bulk. The twin bundle region appears to have

FIGURE 14: SEM PHOTOGRAPH OF A GRAIN
ENHANCED SILICON FILM SHOWING
TWIN BUNDLES.

FIGURE 15: EXAMPLE OF A SOLAR CELL CONTAINING A TWIN BUNDLE REGION. TWIN BUNDLE REGION, OCCUPIES APPROXIMATELY $1/3$ OF THIS 2 cm^2 AREA CELL. (V143-1128E).

FIGURE 16: ILLUMINATED (SIMULATED AM1) AND, DARK I-V CHARACTERISTICS OF THE CELL SHOWN IN FIGURE 15.

higher red response and lower blue response compared to regions A and C. To determine whether this effect is due to possible doping differences in heavily twin bundled region (B) and lightly twin bundled regions (A and C), the carrier concentrations were determined in regions A and B by C-V measurements. As indicated in Table 1, no such inhomogeneity was observed. (The reason that resistivity determined by C-V measurements is lower than that given by the 4-point probe measurements may be due to highly resistive grain boundaries). The poor blue response from the twin bundle region may be a result of the large surface area of this region due to ridges (see Figure 14), which result in higher surface recombination. The higher red response from region B, compared to A and C, is in agreement with diffusion length measurements indicated in Figure 17.

These results indicate that twin bundles are not necessarily bad. However, it should be emphasized that these are coherent twin boundaries (twinning on {111} planes). It is possible that incoherent twins, such as twinning on {112} or {211} planes, can be detrimental to solar cell efficiency due to large atomic disorder at these boundaries.

TABLE 1: SUMMARY OF THE ANALYSIS OF THE CELL (V143-1128F)
SHOWN IN FIGURE 15.

REGION	V_{OC} (volts)	AM1 J_{SC} (mA/cm ²)	F.F. (%)	η (%)	J_{SC} BLUE (mA/cm ²)	J_{SC} RED (mA/cm ²)	N_A (cm ⁻³)	ρ (ohm-cm)
ENTIRE CELL	0.498	25.3	67.1	8.46				2.6
A	0.502	24.86	67.8	8.46	0.0577	0.122	5.69×10^{15}	1.15
B	0.524	26.34	67.3	9.29	0.048	0.144	5.83×10^{15}	1.13
C	0.483	24.36	65.0	7.65	0.0947	0.137

FIGURE 17: SECTIONING SCHEME OF THE CELL SHOWN IN FIGURE 15. THE NUMBERS ARE THE DIFFUSION LENGTH VALUES MEASURED AT REGIONS INDICATED.

Several more 1 cm x 2 cm solar cells have been fabricated on the silicon films obtained from the batch plasma system after grain enhancement. The best device showed a conversion efficiency of 10.7%, the highest to date, with $V_{OC} = 0.545V$, $J_{SC} = 28.65 \text{ mA/cm}^2$ and $FF = 68.3\%$. The I-V characteristics of this cell are shown in Figure 18. Even though the ribbons appeared to be reasonably uniform, in terms of grain size, variations in solar cell performance were observed from ribbon to ribbon, and along the same ribbon. Table II shows an example of the variation in performance of solar cells fabricated on one ribbon. Cells 2 and 3 are high performers with efficiencies of 10.1% and 10.3% respectively. While J_{SC} is respectable in cells 1 and 4, their fill factors particularly, are inferior. It is possible that this is due to problems in metallization of the cells, conceivably caused by the Pd/Ag plated metallization depositing metal on regions of the p-n junction that were exposed as a result of the process sequence interacting in a deleterious fashion with structural defects in the ribbon. Major effort will be devoted to investigating this possibility.

Short circuit currents in our present solar cells are high (e.g. 31.1 mA/cm^2). This indicates a long minority carrier diffusion length, which in turn indicates that the amounts of harmful impurities (e.g., Mo) in the ribbons are low.

FIGURE 18: ILLUMINATED (AM1) AND DARK I-V CHARACTERISTICS OF THE HIGHEST EFFICIENCY SOLAR CELL FABRICATED TO DATE.

$J_{SC} = 28.65 \text{ mA/cm}^2$
 $V_{OC} = 545 \text{ V}$
 $FF = 68.3\%$
 $\eta = 10.66\%$

TABLE 2

VARIATION IN PERFORMANCE OF SOLAR CELLS FABRICATED
ON RIBBON V150-1136C, UNDER AM1 ILLUMINATION.

CELL NO.	J_{SC} (mA/cm ²)	V_{OC} (V)	FF (%)	η (%)
1	27.3	0.494	56.1	7.6
2	31.05	0.511	63.4	10.1
3	31.1	0.515	64.5	10.3
4	28.75	0.480	40.0	5.5

7.0 PLANS FOR NEXT QUARTER

During the next quarter we plan to:

- . Continue TESS substrate system experiments.
- . Continue optimization of the semicontinuous plasma deposition system parameters.
- . Conduct a detailed characterization of variations in the deposition process as a function of deposition parameters, by gas chromatography, and
- . Fabricate and characterize solar cells on silicon films obtained from the semicontinuous plasma deposition system (after grain enhancement).

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