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FLOATING SUBSTRATE PROCESS

Large Area Silicon Sheet Task Low-Cost
Silicon Solar Array Project

Third Quarterly Progress Report, June 21, 1976—September 24, 1976

M. Garfinkel
R. N. Hall

September 27, 1976

Work Performed Under Contract No. NAS-7-100-954350

General Electric Company
Schenectady, New York

ENERGY RESEARCH AND DEVELOPMENT ADMINISTRATION
Division of Solar Energy

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Large Area Silicon Sheet Task
Low-Cost Silicon Solar Array Project

Third Quarterly Progress Report
Subcontract No. 954350

by

M. Garfinkel, Program Manager
R. N. Hall, Principal Investigator

Period Covered: June 21, 1976 to September 24, 1976

September 27, 1976

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ABSTRACT

The Seeded Growth Furnace was completed and operated extensively during the third quarter. Optimum thermal geometry, gas flows and withdrawal rates are being determined. Surface growth was obtained having growth velocity as high as 5-6 mm/min. The surface growth has generally taken the form of interlocking crystals and is thin enough to follow the liquid surface. Its thickness has been determined in one case to be 20 microns.

A molybdenum susceptor has been made for the seeded growth furnace. It is anticipated that this new design will eliminate the problems caused by films on the melt and seed crystal and lead to improved crystal structure in the silicon growth.

A series of experiments were performed in order to determine whether the reactor gas streams were sources of contamination. Nitrogen reacts with silicon at 1100°C to coat its surface with a film 30Å-150Å thick. Silicon heated in hydrogen at temperatures between 1000°C and 1200°C maintains a very clean, film-free surface.

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Section 1

INTRODUCTION

This research program began January 6, 1976. Its objective is to demonstrate the feasibility of the floating substrate sheet growth process for the growth of silicon sheet. This process is an approach to the formation of single-crystal silicon by direct epitaxial conversion from a gaseous silane. Incoming feedstock gas impinges upon a silicon substrate which is floating upon a thin pool of liquid tin. Single-crystal silicon grows to the desired thickness by means of vapor phase epitaxy. Nucleation of fresh substrate silicon takes place by rapid growth from the edge of the growing sheet into a region at the surface of the liquid tin which is supersaturated with silicon. The process lends itself to continuous operation with the growing sheet being continuously withdrawn from the growth zone.

Since the epitaxial growth of silicon is well known and presents no problems, the current effort will focus upon the nucleation and rapid growth of substrate silicon from supercooled melts. We will perform studies of (1) supercooling; (2) crystal growth from a supercooled Sn-Si alloy; (3) silicon uptake into tin from silanes; and (4) surface growth. The goals of this contract are the growth via this process of single-crystal silicon having 0.5 cm^2 area and the determination of the velocity of propagation of single-crystal silicon along a hot tin surface which has been supersaturated with silicon.

During the first quarter, laboratory apparatus was designed, built, and used to study the supercooling of Sn-Si melts and the uptake of silicon from silane. Values of supercooling of Sn-Si melts as high as 78°C at 1100°C and 39°C at 1200°C were observed. These results were limited by nucleation caused by foreign material floating on the surface of the melt. Spontaneously nucleated planar platelet growth was observed at the Sn-Si supercooled surface having growth rates of approximately 0.5 cm/min .

Homogeneous nucleation of silane decomposition was studied at 1015°C and 1135°C . The flow rates of HCl required to suppress gas phase decomposition were determined for silane flow rates up to $4.5 \times 10^{-3} \text{ mole/min}$. Initial silicon uptake experiments showed that at least 20 percent of the incoming silicon contained in a flowing silane gas stream can be incorporated into liquid tin at 1040°C .

In the second quarter, the studies of supercooling and of the interaction of silane with tin melts were continued and extended.

A Horizontal Boat Furnace which permits the surface scum to be blown out of the crystal nucleation zone was built and operated. Supercooling results obtained in this apparatus are similar to those obtained earlier and appear

to be limited by nucleation at the melt-crucible surface. An electromagnetic pump was successfully incorporated into this apparatus. First examples of seeded surface growth were observed using web-dendrite material as the nucleating seed.

An experimental configuration was developed permitting accurate measurements of the uptake of silicon from silane. Single pass efficiencies greater than 30 percent, corresponding to the deposition of solid silicon at 1.8 microns/min, were measured. It was determined that silicon is incorporated into liquid tin at the same rate as it is deposited upon silicon substrates. The rate of incorporation is independent of the degree of melt saturation.

Tin melts have been driven into saturation by means of silicon input from silane and dichlorosilane. Silicon melt concentrations achieved by gas stream input were limited to the saturation value because of crystal growth nucleated by silicon deposited upon the tray walls.

The Seeded Growth Furnace was operated extensively in the third quarter.

Section 2

TECHNICAL DISCUSSION

SEEDED GROWTH FURNACE

MODEL 1 -- GRAPHITE SUSCEPTOR

The original seeded growth furnace, shown in Figure 1 of the Second Quarterly Progress Report, was operated during August and September. Several minor design changes were made in order to improve the performance and ease of operation, but the basic design was as indicated above.

After some initial break-in experiments it was found that rapid surface growth could be achieved by directing the cooling jet so that it impinged on the melt surface just ahead of the growing edge of the solid silicon. As reported in Monthly Report No. 8, growth velocities of 5 to 6 mm/min were achieved. Similar results were obtained during subsequent experiments, SGF-10 through 12. This surface growth was thin enough that it could easily be seen to bend as the seed crystal was moved during the growing operation, but it was composed of many small crystals which had little relation to the orientation of the seed crystal that was used to initiate growth. This material will be characterized in more detail in a later section of this report.

Figure 1 shows an example of the results achieved using this furnace. In this experiment, SGF-11D, a layer of silicon 35 mm long was grown, but it separated from the seed as it was being drawn off. The furnace was then shut down so the results could be photographed. In addition to the polycrystalline mass of silicon that was being grown on the end of the seed crystal, there was another "peninsula" of silicon approximately 6 x 12 mm in size, which can be seen growing out from the corner of the boron nitride tray. Elsewhere over the surface of the tin were small silicon crystals which formed as the system was cooling down.

This furnace had several shortcomings which needed to be corrected. The most serious difficulty was that a layer of foreign material, or scum, always formed on the surface of the Sn-Si alloy and interfered with the growth process. Most of it could be blown aside by the gas jet, but there always appeared to be some attached to the end of the seed crystal. It seems probable that this scum formation was the principal reason for our failure to achieve single-crystal growth; the origin of the scum is not clear, but there appear to be several potential sources. The graphite susceptor may absorb oxygen or other gases while the furnace is open and result in the evolution of CO during operation and thus lead to the formation of SiC on the melt surface. Also, reaction with the boron nitride tray may produce a film of BN on the melt surface. In addition, there are several places

Figure 1. Crystal growth produced during SGF-11D. The furnace was shut down after the growth separated from the seed crystal. An area of unwanted growth extends toward the viewer from the corner of the boron nitride tray.

where hot quartz is in contact with the graphite susceptor. Since these materials have been observed to react with each other, their products may contribute to the scum.

Another problem was that, while the gas jet provided an effective means for cooling the melt surface, it also caused the surface of the melt to flow in an uncontrolled manner with the result that it was impossible to isolate the effect of melt circulation on crystal growth. It appeared that a cold finger placed close to the surface of the melt would provide a better means for achieving local cooling ahead of the growth edge.

MODEL 2 -- MOLYBDENUM SUSCEPTOR

A new susceptor, which is expected to eliminate these difficulties, has been constructed for the seeded growth furnace. Figure 2 shows a cross-section view of this furnace, which was assembled and which is now undergoing initial testing. The susceptor consists of a block of molybdenum, which is slotted so that trays of various different materials can be evaluated as to their suitability for melt containment. The tray is made in the form of a shallow box with sides which fit together snugly around a bottom plate and are held together by the slots of the susceptor. Trays made of quartz,

Figure 2. Seeded growth furnace with molybdenum susceptor.

alumina, sapphire, beryllia, or tungsten can be readily obtained in this way. Electrodes for electromagnetic stirring have also been provided. Local cooling is produced by a water-cooled copper cold finger having a sapphire plate, which can be adjusted to be in close proximity to the melt surface, attached to its lower surface. A boron nitride trough was also provided in order to facilitate the withdrawal of the silicon through the exit port. A thermocouple and chart recorder were added for more convenient operation and data-taking.

CRYSTAL GROWTH USING THE SEEDED GROWTH FURNACE

The floating substrate process involves the rapid growth of a thin single-crystal sheet over the surface of a supersaturated Sn-Si melt. The thickness of this sheet would be increased as required by vapor-phase epitaxial deposition as the sheet travels down through the deposition zone. The purpose of the seeded growth furnace was to determine the conditions that would be required to produce such surface growth and to learn what growth velocities could actually be achieved.

In our initial experiments we directed the cooling jet onto the seed crystal close to the end at which growth was to be initiated. Some growth was produced, but it was rather slow and much of the growth took place downward toward the bottom of the tray making it difficult to draw the silicon

out through the exit port. We also tended to use excessive electromagnetic stirring in the belief that this would accelerate the growth by bringing fresh solution to the growing edge. It now appears that vigorous stirring serves mainly to homogenize the temperature and composition of the melt so that growth becomes nearly isotropic. We now believe that surface growth is favored by using little or no forced circulation in the melt.

In experiments SGF-9 through 12 the cooling jet was directed upon the liquid just ahead of the crystallized silicon. It was found that the growth rate could be increased to over 5 mm/min and that it propagated as a very thin layer over the surface of the liquid. So far its structure has been very poor. Most of the growth consists of a network of tiny crystals and platelets which are interconnected to form a thin layer, often with openings within which tin has been trapped. The silicon often shows areas several millimeters in extent; these appear to be continuous single-crystal regions, although they exhibit a distinct surface texture. Figures 3 and 4 are microphotographs of the top and bottom surfaces of a fragment broken from one of these regions, the field of view being $2.2 \times 1.7 \text{ mm}^2$. The top view, Figure 3, shows a smooth "spine" attached to a region with a pronounced horizontal texture reminiscent of the unseeded planar growth reported in our First Quarterly Progress Report. The same fragment seen from below in Figure 4 appears to be formed from many co-planar platelets all having the same crystallographic orientation with their shiny $\langle 111 \rangle$ faces lying in the plane of the original growth surface. From the appearance of these platelets, it appears that they must have grown by liquid phase epitaxy upon the original surface after it had first formed. The same crystal fragment was examined on edge in an optical microscope and measured to have a thickness of approximately 20 microns. Edge views of this crystal taken in a scanning electron microscope are shown in Figures 5 and 6.

These observations demonstrate that thin single-crystal sheets of silicon can be induced to grow over the melt surface under the proper circumstances. The rates of growth that have been observed are within a factor of four of that which had been originally hoped for. The poor crystal structure that has been achieved so far is discouraging but may be caused by the presence of scum on the surface of the melt. The new seeded growth furnace with the molybdenum susceptor is expected to eliminate the scum problem and should result in substantially improved crystal perfection.

GAS STUDIES

The investigation of unwanted film formation begun in Month 8 has been continued. The procedure followed was to use a clean specular silicon surface as the detector. Films formed during the course of an experiment, either by reaction of silicon with some component of the gas stream or in some other manner, were detected and measured ellipsometrically. Since ellipsometer sensitivity is very high, it allows film thickness measurements with a reproducibility better than $0.5 \text{ \AA}$ to be made quite easily.

Figure 3. Top view of a fragment of crystal from SFG-9D.
The field of view is $2.2 \times 1.7 \text{ mm}^2$.

Figure 4. Same crystal shown in Figure 3 viewed from below.

The wafers used were chem-mechanically polished with $\langle 111 \rangle$ orientation. The native oxide on the surface was removed by dipping the wafers in 10 percent HF solution, and residual fluoride was removed by rinsing in deionized water. The wafers were loaded into the horizontal reactor within one minute of being etched and washed. They were placed directly upon the susceptor and run at approximately 1100°C under various gas flow conditions for about 45 min. Specific details of the experimental conditions and brief summaries of results are given in Table 1.

Figure 5. Edge view of crystal from SGF-9D. Some platelets that grew down into the Sn-Si alloy and which adhered to the Si can be seen on its lower surface.

Figure 6. Edge view at higher magnification of the right end of the crystal shown in Figure 5.

Table 1
GAS FLOW STUDIES

Rup No.	H ₂ ℓ/min	N ₂ ℓ/min	T °C	t min	Thickness Å	Surface	Comments
G11	2	0	1100	45	6.5	slight etching	Si coated graphite susceptor
G12	0	2	1095	45	26	smooth	Si coated graphite susceptor
G13	2	0	1085	40	6	thermal etch	Graphite susceptor
G14	0	2	1085	50	28	thermal etch	
G15	2	0	1075	43	4	thermal etch	New endplate
G16	2	0	1075	45	4	thermal etch	Heavy etching under jet
G17	0	2	1075	55	30-130	smooth	
G18	2	2	1075	45	30-150	smooth	
V2-1	10	0	1075	40	-	severe thermal etching	Experiment performed in epitaxial reactor
V2-2	0	3	1075	45	35	smooth	Experiment performed in epitaxial reactor
V2-3	0	3	1200	32	53	smooth	Experiment performed in epitaxial reactor
V2-4	0	3	1075	123	49	smooth	Experiment performed in epitaxial reactor

Considerable variability was seen in the early experiments G1-G8 described in Monthly Report No. 8. The results obtained for runs which were carried out with nominally identical operating conditions differed from each other. There appeared to be a pattern in this variability--films obtained from later runs seemed generally to be somewhat thinner than those obtained in earlier experiments.

All of these experiments were performed using a graphite susceptor and interleaved quartz paper/molybdenum foil reflective insulation. Both of these are possible sources of variability and contamination, since they can absorb considerable quantities of air and water vapor upon exposure to the atmosphere, releasing these gases with subsequent heating.

This hypothesis was tested in runs G11 and G12. The quartz paper was removed for these experiments, leaving only molybdenum foil reflective insulation. The surface of the graphite susceptor was also sealed with a silicon coating. In a further attempt to carry out these experiments in as clean a configuration as possible, the old endplate with its single static seal was used. The film obtained from G11 which was run in hydrogen was 6.5Å. The surface also exhibited mild thermal etching. These results are essentially the same as those obtained earlier with hydrogen: the surface was very clean. The 6.5Å film is about as small a value as any that can be measured under common laboratory conditions. It is easily accounted for by either the the growth or adsorption of two monolayers of water or of an oxide. Run G12 which

was operated in nitrogen generated a smooth film 26 Å thick. While similar to the films seen earlier for nitrogen gas flow, its thickness is somewhat thinner than the 34 Å and 40 Å obtained for G1 and G2, respectively.

Runs G13 and G14 are repeats of G11 and G12 using an uncoated graphite susceptor. This susceptor was made from Union Carbide high-density graphite and was fired in vacuum at 1700°C prior to being used. The results obtained were identical with those from the sealed susceptor. We thus conclude that: (a) the quartz paper was indeed the source of our variability, and (b) hydrogen-graphite reactions to form hydrocarbons with attendant contamination of the gas stream is not a problem.

The high-density graphite susceptor was used in subsequent experiments G15-G18. The new furnace endpiece with its auxiliary gas jet and additional sliding O-ring seals was used in order to determine the effect of the gas jet directed at the sample surface and to check whether or not these seals leaked.

G15 and G16 were performed with a pure hydrogen jet directed at a portion of the sample surface. The results were the same as those obtained earlier. Away from the jet, where the gas flow was relatively slow, the sample surface was smooth, allowing ellipsometer measurements. The surfaces came out of the reactor very clean, exhibiting 4Å-7Å films when measured within a minute or less of exposure to air. The regions of the samples near the jet where the hydrogen flowed along the surface at high velocity developed a pronounced thermal etch pattern as a result of the fact that the silicon evaporated from the clean surface is carried off by the gas stream. This pattern, which is centered about the jet flow, may be seen in Figure 7, an overall view of sample G18. Figure 8 is a microphotograph of the "hazy" region of G16. The thermally etched pattern with <111> crystal symmetry is clearly visible.

The results for G17 and G18, run with 100 percent and 50 percent nitrogen, respectively, are quite similar. The surfaces are very smooth. The "bulls-eye" film pattern seen earlier for cases of mixed hydrogen-nitrogen jet flows described in Monthly Report No. 8 is absent. Film thicknesses ranging from 30Å to approximately 150Å are seen, with the thicker films developing near the jet flow.

These experiments confirm our prior conclusion that some surface film forms upon the surface of silicon wafers and thus presumably upon any silicon seed which is heated in the presence of nitrogen. We have also identified quartz paper as a serious source of contamination. More satisfactorily, these experiments have shown that it is possible to maintain silicon surfaces in a very clean state in pure hydrogen.

Several additional experiments were carried out in a vertical epitaxial reactor (AMT model #800). This experimentation was done in order to

Figure 7. Overall view of sample G16 which was heated in a hydrogen atmosphere. The bright area is heavily etched thermally and is centered at the location where the hydrogen jet impinged upon the wafer.

Figure 8. A highly magnified view of the thermally etched region of G16.

determine whether the results of the gas studies were peculiar to our horizontal reactor or were more general in nature. These are included in Table 1 and are indicated as runs V2-1 through V2-4. The results for this series were gratifyingly similar to those obtained in the horizontal reactor. Sample V2-1, run in hydrogen, exhibited severe thermal etching resulting from the high gas flow across the wafer in this reactor. While this result made it impossible to measure film thickness, it is a reasonable conclusion that the rate of any film formation is well below that of silicon surface evaporation, which is quite low at 1080°C (the equilibrium vapor pressure is 2×10^{-7} Torr.). Runs V2-3 through V2-4, which were run in nitrogen, developed smooth films 30Å-50Å thick. Film thickness increased with both time and temperature, as is shown in Table 1.

The atmosphere in the seeded growth furnace was also tested using the procedure described above. After run SGF-13, the graphite susceptor with its boron nitride tray and associated quartz, molybdenum, and tungsten parts was assembled empty and fired to 1300°C in N₂ to clean it out. A silicon test wafer was then placed in the tray and heated to 1070°C in H₂ for 45 min using a gas flow of 10 liters/min. Upon examination, the wafer was found to have a non-specular film on its surface. The ellipsometer measurement of this surface indicated an apparent thickness of 463 Å and an index of 3.13. Since this result showed that a considerable deposit had been formed on the silicon, we removed all the furnace parts except the graphite susceptor and ran another test in H₂. This time the test wafer came out with a clear specular surface and showed a film that was 385 Å thick with an index of 1.50. It was easily removed with HF and was presumed to be SiO₂ which grew as the result of back-diffusion of air through the exit port at the end of the furnace. This port has a 7 x 16 mm² opening in an endplate which is only 12 mm thick. We therefore extended the length of this opening to 36 mm to reduce back-diffusion and conducted another test, this time obtaining a reading of 7.5 Å, indicative of a clean H₂ atmosphere. This last result had been obtained with only a brief exposure of the graphite susceptor to air when the furnace was opened to introduce the silicon wafer. We next repeated this experiment except that the susceptor was left exposed to air overnight in order to find out whether or not it would become contaminated. This time the ellipsometer measurement indicated a 6 Å film, substantially the same as that obtained after the brief air exposure, again indicating clean operating conditions.

These experiments showed that the graphite susceptor itself was not a source of contamination, at least as evaluated by the silicon wafer test procedure. Back-diffusion of air through the exit port was observed but was eliminated by extending the length of this opening. Serious contamination of the test wafer was observed when the other parts of the seeded growth furnace were included with the susceptor. A distinct deposit extending from places where they had been in contact with graphite was seen on quartz surfaces, making it evident that a reaction between quartz and graphite had been taking

place. The new furnace with its molybdenum susceptor is expected to be free of materials which could react in the way.

PROBLEMS

While thin, rapid surface growth has been achieved, it is not yet properly seeded and is quite polycrystalline. The presence of a film of foreign material on the surface of Sn-Si melts has been noted in previous reports. This film interferes with crystal growth by providing additional crystallization nuclei. Surface films are formed upon silicon seeds when they are heated in nitrogen. We have used nitrogen ambient in most of our work, and it appears likely that the resultant surface film on the seed crystal interfered with the seeding. The nitrogen ambient may also have contributed to the film on the melt surfaces.

Another likely source of foreign contamination is the tray material which is used to contain the melt. Both quartz and boron nitride react to some extent with the Sn-Si alloy so that these reaction products may be responsible for this film. The new design for the seeded growth furnace should eliminate both of these sources of contamination.

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Section 3

TENTATIVE CONCLUSIONS AND RECOMMENDATIONS

The use of a gas jet to provide local cooling ahead of the growth region in the seeded growth furnace results in uncontrolled circulation within the Sn-Si alloy, making it difficult to evaluate the effects caused by electromagnetic melt circulation. The gas jet will be replaced by a water-cooled cold finger to provide the local supercooling needed for crystal growth.

The gas studies performed during this quarter have demonstrated that nitrogen gas is a source of contaminating film growth upon silicon maintained at the temperatures required for crystal growth. Hydrogen, on the other hand, permits the maintenance of clean silicon surfaces and presumably clean melt surfaces. We will use pure hydrogen ambient in future work.

Section 4

PROJECTED ACTIVITIES

The major focus of our efforts for the next period will be upon surface growth.

We plan to continue working with the seeded growth factor in order to determine the growth conditions that are most favorable for rapid surface growth. The new molybdenum susceptor version of this furnace is completed and will be operated with various tray materials under what are expected to be much cleaner growth conditions. We will also test the effectiveness of gas conduction cooling to a nearby cold finger as a means of maintaining strong thermal gradients in the neighborhood of the crystal growth region.

Section 5

NEW TECHNOLOGY

There are no reportable items of new technology.

Appendix I

UPDATED PROGRAM PLAN

PROGRAM PLAN FLOATING SUBSTRATE PROCESS

Appendix II

MAN HOURS AND COSTS

Previous man hours were 1086 hours of scientist/engineer effort and 1232 hours of technical specialist and shop time. Total charges for the first two quarters, not including fee, were \$104,555. Third quarter charges to Sept. 24, 1976 are 520 hours of scientist/engineer time and 765 hours of technical specialist, and shop time. Charges for this quarter, not including fee, are \$59,685. Cumulative totals for the first three quarters are 3603 man hours and \$164,240 dollar costs, not including fee.

Planned/Incurred Costs
Direct Labor Hours
Contract 954350
Begun January 6, 1976

Floating Substrate Process