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ERDA/DPL/954373-75/1

HEAT EXCHANGER-INGOT CASTING/SLICING PROCESS

Silicon Sheet Growth Development for the
Large Area Silicon Sheet Task of the Low Cost
Silicon Solar Array Project

First Quarterly Progress Report
by
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Covering Period from November 20, 1975, to December 31, 1975
December 31, 1975

JPL Contract No. 954373

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ABSTRACT

Designs for changes to the existing crystal casting furnace and multi-wafer slicer were completed prior to the contract. Equipment supplies necessary for the modifications and silicon material were ordered during the first one and one-quarter month period covered by this quarterly report. Assembly preparation and check-out of all systems is on schedule.

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SUMMARY

The object of this program is to demonstrate that large single-crystal ingots can be economically cast by the Heat Exchanger Method (HEM) and economically sliced into thin sheets with a multi-wafer slicer. Crystal Systems has developed the Heat Exchanger Method (HEM) for casting the largest sapphire available and has interest in extending the process to other materials especially silicon because of its potential for photovoltaic generation of electricity.

The goals for producing silicon sheet under this contract are:

Ingot Growth Project

Physical Dimensions: 6" diameter by 4" high

Growth Rate: Solidification time 8 hours

Dislocation Density: $< 10^4 \text{ cm}^{-2}$

Crystal Structure: Single crystal

Slicing Project

Physical Dimensions: 8 mils $\pm$ 1mil thickness for sheet
cross sections of 4" diameter, 4"x 4" and 6" x 3"

Slicing Rate: 10 mils/minute (per wire/blade)

Surface Steps: 0.2 mils

Wire/Blade Thickness: 3 mils wire; 4 mils blade

Parallel Slices: 100

This is an experimental program designed to determine the potential and isolate the limiting parameters for crystal casting and slicing with the Heat Exchanger Method (HEM) and the multi-wafer slicer.

CRYSTAL CASTING

The crystal casting furnace is illustrated in Figure 1; it is a conventional vacuum graphite resistance furnace with the heat exchanger extending into the heat zone.

SCHEMATIC OF CRYSTAL CASTING FURNACE

For silicon casting, an oriented seed is centered and held on the bottom of a quartz crucible. The quartz crucible is then loaded with silicon melt stock and positioned on the

heat exchanger so the seed is centered over the heat exchanger as illustrated in Figure 1. After evacuation, the furnace temperature is increased above the melting point of silicon. Helium is forced through the heat exchanger at low flow rates to prevent melting of the seed. The temperature of the heat exchanger is then increased for nucleation of the liquid on the seed. Growth of the crystal is initiated by slowly increasing the helium flow rate to decrease the solid temperature gradient. The liquid gradient is independently decreased by lowering the furnace temperature.

The distinguishing feature of the Heat Exchanger Method (HEM) is the ability to independently control the liquid-solid temperature gradients without moving the crucible, heat zone or crystal. Crystals grow from the bottom to the top of the crucible suppressing convection that causes temperature and concentration fluctuations at the solid-liquid interface. This prevents constitutional super-cooling even at low temperature gradients and promotes uniform growth resulting in high crystal perfection and chemical homogeneity. The growth interface is protected from external contaminants and vibration by the liquid over it. This eliminates the need to flow expensive high-purity Argon over the silicon melt to sweep away Silicon Monoxide particles that cause spurious nucleation and contamination for CZ growth. Vibration effects on the surface, such as standing waves, etc., are damped out beneath the surface making the Heat Exchanger Method (HEM)

much less sensitive to vibration.

An important feature of the technique for growing silicon is the ability to refine the melt stock. Purification will take place by processing in vacuum by directional solidification. Low purity, irregular-shaped silicon melt stock can be utilized by this method since volatile impurities are removed by a vacuum of 0.1 Torr; other impurities such as quartz particles from the crucible and SiO will float to the surface and not effect the growth interface.

High effective growth rates are achieved since the growth interface expands three dimensionally from the seed to the crucible wall. This effect becomes more significant since the effective growth rate increases exponentially with size. Large size is also helpful in growing higher quality crystals since the greater mass minimizes temperature and concentration fluctuations. Crystals assume the shape of the crucible allowing for casting of square or hexagonal shapes.

The Heat Exchanger Method (HEM) can be completely automated since growth depends on heat flow which is controlled by programming the heat exchanger and furnace temperature. After solidification, the crystal is annealed in-situ just below the melt temperature.

Emphasis will be placed on: (1) Achieving the optimum refining environment (ie., vacuum and temperature), (2) Establishing proper seeding conditions, and (3) Achieving the fastest

growth rate for pure and impure melt stock.

CRYSTAL SLICING

For ingot casting to be economically effective, slicing must be upgraded to slice larger sheets thinner with less kerf loss and at faster rates than is now practiced.

The current state of the art for slicing silicon with a multi-blade wafer machine involves circulating mixtures of oil and SiC or B₄C over hardened steel blades that reciprocate back and forth through the silicon work piece. Very large ingots 8" x 8" x 6" can be cut by this method, but it is slow. For a 3-inch diameter silicon piece, the cutting rate is about 0.1mm per minute. The abrasive slurry is free to roll between the hardened blades and the silicon and, therefore, removes material by a combination of rolling and dragging, an inefficient mechanism for cutting. The viscous oil and abrasive is re-circulated without removing the swarth; this has a detrimental effect on slicing.

Crystal Systems has developed a blade abrasive system and modified a multi-blade wafer machine to cut sapphire. This was accomplished by holding the abrasive particles fixed on the cutting edge of the blade and increasing cutting pressure.

Abrasive particles held in place on the cutting edge greatly improved cutting speed over free abrasive slurries. However, for this condition, cutting rate becomes heavily

dependent on the pressure of the cutting edge against the work. Cutting pressures can be increased by increasing the force of the blades against the work and/or by using thinner blades. Both methods result in blade wander. Cutting pressure was effectively increased by rocking the work with a special mechanism to decrease the blade contact length; this eliminated blade wander.

A total systems approach will be used to update the current multi-wafer slicing technique.

(1) The slicing machine must be made sensitive to cutting pressure.

(2) The sharpest most effective blade/wire abrasive must be used, i.e., diamond held in place on the cutting tool.

(3) Swarth must be removed to prevent agglomeration and loading of the cutting tool.

(4) Silicon must be made to cut faster using chemicals such as NaOH; this will also reduce damage and prepare the surface for junction formation.

QUARTERLY PROGRESS

Crystal Casting

The furnace that will be used for the silicon casting was previously used for sapphire growth. The possibility of aluminum contamination existed if this heat zone was used; therefore, a new heat zone will be installed.

Since the silicon growth temperature is much lower than the sapphire growth temperature, thinner insulation is sufficient to achieve the same thermal control. The insulation was re-designed to a total thickness of 2.5 inches on all sides. This allowed the heater diameter to be expanded from 12 inches to 14 inches I.D. This increase will allow a crystal growth crucible size up to approximately 10 inches in diameter.

The entire furnace interior, including the insulation, will be fitted into a cage in order to maintain geometric stability. The cage and interior can be easily removed from the furnace chamber to allow for cleaning and modifications, if required. In addition, a re-designed heater, base plate, hearth plate, covers and assorted parts were ordered.

It was felt that CS-grade graphite, the type used in sapphire growth was not sufficiently characterized or refined for this phase of the crystal casting task. Standard CS-grade graphite contains approximately 1.2% total ash (12,000 parts per million). Ultra-Carbon Grade U-120 was chosen since it is thermally treated to reduce the contaminant level to approximately 100 parts per million. The impurities will be specified with a certified analysis.

The furnace exterior (the steel water-cooled shell) was subjected to analysis in terms of the silicon growth process. It is felt that an additional sight port is required in order to monitor the furnace temperature properly. This port was

designed, and the work required was ordered.

A helium flow control system was designed so the helium flow rate can be controlled by the heat exchanger temperature or helium flow. The independent and dependent variability will be recorded simultaneously throughout the solidification cycle.

Silicon material was ordered. The poly-silicon obtained has an impurity content equivalent to approximately 200 ohm-cm. "N" type and 2000 ohm-cm. "P" type. The initial seeds ordered are (111) float-zone material, zero (approximately) dislocation count, 30 ohm-cm. "N" type.

The silicon boule will be grown in a high-purity fused quartz crucible approximately six inches in diameter and six inches high. Since this crucible is too small to accommodate all the poly melt stock but large enough for the boule, an extension was designed to allow loading of all the required raw material and consequent re-use of the extension in subsequent growth operations. Should a warping of the extension occur, a graphite lever may be provided to maintain the shape. The crucible will be supported by a graphite support. A graphite retaining plate was designed for catching and retaining any spillage of the melt.

Crystal Slicing

Prior to the contract, the need for increased sensitivity in the crystal-slicing feed system was analytically established by an analysis that related the interaction between the cutting

force, workpiece motion and wire tension. The analytical analysis was verified by slicing tests that were conducted on a multi-wafer slicing machine that had been modified to double the sensitivity. The feed mechanism could not respond to the small variation in cutting forces that occur during slicing with wires. To achieve the necessary sensitivity (an order of magnitude more sensitive), the stock feed mechanism must be replaced with a light-weight low-friction apparatus. Various approaches were studied to achieve this sensitivity. Prior to the contract, a mechanical mechanism was selected and designed. During the contract period the parts were detailed and ordered. The initial slicing tests will be conducted with diamond impregnated wire. Wire 0.003 and 0.008 thick impregnated respectively with 8 and 45 micron natural-type diamond has been ordered for the slicing tests.

All phases of the crystal casting and slicing tasks are on schedule.

CONCLUSIONS AND RECOMMENDATIONS

All phases of the crystal casting and slicing tasks are on schedule. Progress during the pre- and post-contract period is encouraging. No obstacles have been encountered or are now foreseen to cause deviation from the program plan.

APPENDICES

1. Updated Program Plan

The program plan was submitted to JPL for approval. Work is progressing according to the plan.

2. Man-Hours and Costs

Total man-hours and costs for the first quarter are 500 and \$31,921 respectively.

3. Engineering Drawings and Sketches Generated During the Report Period

Engineering drawings are details of parts and, therefore, are not submitted.

4. Summary of Characterization Data Generated During the Report Period

A subcontract for characterization is in the final stages of approval at JPL.

5. Action Items Required by JPL

None.

6. New Technology

Quarterly progress has been reported for the first 1-1/4 month period. There is no new technology to report.

SCHEDULE OF MILESTONES--CRYSTAL CASTING

PAGE 1 OF 2

T _E M	DESCRIPTION	Month No.																	
		1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18
1	A. Modify and calibrate	○	△																
2	furnace/Purchases	●																	
3																			
4	B. Optimize refining		○	△															
5	conditions																		
6																			
7	** Design & Performance		○	△															
8	Review																		
9	C. Establish seeding con-			○					△										
10	dition for 6" Ø x 4"																		
11	single crystal																		
12																			
13	D. Establish single crystal								○									△	
14	casting parameters for																		
15	pure silicon melt stock																		
16																			
17	E. Establish single crystal																	○	
18	casting parameters for																		△
19	solar energy grade silicon																		
20	melt stock																		
21	F. Final Report																		○△
22																			

LEGEND: ○ Estimated Start △ Estimated Complete ● Actual Start ▲ Actual Complete

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SCHEDULE OF MILESTONES--CRYSTAL SLICING

PAGE 2 OF 2

ITEM	DESCRIPTION	Month No.																	
		1	2	3	4	5	6	7	8	9				10	11	12			
1	A. Blade and equipment	○	△																
2	modification and purchases	●																	
3																			
4	B. Slice silicon test piece		○			△													
5																			
6	** Design & Performance		○	△															
7	Review																		
8																			
9	C. Blade hardness varied and					○		△											
10	alternate blade systems																		
11																			
12	D. Particle size varied							○		△									
13																			
14	E. Diamond type varied									○					△				
15																			
16	F. Special effects on cutting														○		△		
17																			
18	G. Final Report																○	△	
19																			
20																			

LEGEND: ○ Estimated Start △ Estimated Complete ● Actual Start ▲ Actual Complete

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