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.
Fifth Quarterly Progress Report, September 25, 1976—December 16, 1976**

**F. Schmid
B. Reynolds**

December 20, 1976

Work Performed Under Contract No. NAS-7-100-954373

**Crystal Systems, Incorporated
Salem, Massachusetts**

**ENERGY RESEARCH AND DEVELOPMENT ADMINISTRATION
Division of Solar Energy**

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Fifth Quarterly Progress Report
by

Frederick Schmid and Blake Reynolds

Covering Period from September 25, 1976, to December 16, 1976

Date of Report: December 20, 1976

JPL Contract No. 954373

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MAN-HOURS AND COST TOTALS

<u>Previous</u>		<u>Current</u>		<u>Cumulative</u>	
M/Hr.	Cost	M/Hr.	Cost	M/Hr.	Cost
3,261	\$162,686	1,254	\$43,773	4,515	\$206,459

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ABSTRACT

Casting experiments during the quarter were directed towards determining the maximum growth rate, characterizing the mode of heat transfer to the gaseous helium in the heat exchanger, developing a sintered crucible coating, and determining the cause of a SiC deposition on the melt stock prior to melting.

The growth rate was heavily dependent on the rate of furnace temperature decrease. For fast growth rates the furnace and heat exchanger temperatures were decreased rapidly. The heat exchanger temperature had to be decreased more rapidly with increase in growth rate to conduct away the heat of fusion to prevent breakdown of the interface. It was determined that most of the heat transfer in the heat exchanger occurred in the injection tube.

Cracking of silicon ingots was prevented in laboratory size crucibles by applying sintered silica coating to the walls of the crucible. The sintered coatings separated freely from the silicon ingot during cool down and prevented cracking.

Silicon carbide deposits that form on silicon melt stock near the crucible wall during heat up could not be traced to carbon transported from ground crucible surfaces

or from graphite support rings in contact with the fused silica crucible.

Slicing tests during this quarter have demonstrated that increasing the concentration of diamond impregnated into the copper-plated wire improved abrasive life.

Reduced cutting rates were experienced when diamond impregnated wire was intermittently used over a period of time. As the length of time out of service increased, the cutting rate decreased, suggesting that corrosion may degrade the copper diamond bond and decrease abrasive life.

To reduce wander, a wire support system was installed. Grooved rollers support and guide the wire on either side of the workpiece. This reduced wander an order of magnitude.

EXPERIMENTAL RESULTS

SILICON INGOT CASTING

Casting experiments during this quarter were directed toward determining the maximum growth rate, characterizing the mode of heat transfer to the gaseous helium in the heat exchanger, developing a sintered silica crucible coating, and determining the cause of a SiC deposit on the melt stock prior to melting. The operating parameters for runs 34 through 50 are presented in Table I.

Determining Maximum Growth Rates

Runs 34, 35, 36, 44, 45, 46, 47, 48, and 50 were conducted to optimize seeding and maximize the growth rate. Runs 34 and 35 were designed to run at superheats less than 10°C with a 90°C and 100°C heat exchanger undercooling. The furnace superheats were 7°C for runs 34 and 35. The boules were observed during cool down with a fibre optic light source. For boule 34 the boule cracked between a temperature of 800°C and 500°C . It was cooled at 40°C/hr in this temperature range. Boule 35 was cooled at 15°C/hr in the 600°C temperature range and it shattered into many small fragments between 575°C and 275°C . Apparently elastic energy was stored during the slow cool down causing the boule to fragment completely below 575°C .

TABLE I.

TABULATION OF HEAT-EXCHANGER AND FURNACE TEMPERATURES

RUN	PURPOSE	SEEDING		GROWTH		REMARKS
		FURN. TEMP. ABOVE M.P.	H.E. TEMP. BELOW M.P.	RATE OF DECREASE H.E. TEMP. FURN. TEMP.		
34	Seed and propagate single crystal	7°C	90°C	200°C/11 hr. 18.2°C/hr.	7°C/11 hr. .636/hr.	Seed melted out. Columnar grains ½" Ø extending to concave growth interface at top of ingot.
35	Seed and propagate single crystal	7°C	100°C	326°C/14 hr. 23.3°C/hr.	7°C/14 hr. .5°C/hr.	Seed melted out. Slow cool crystal causing it to totally shatter.
36	Seed and propagate single crystal, lower furnace temp.	5°C	100°C	290°C/11 hr. 26.4°C/hr.	5°C/11 hr. .455°C/hr.	Seed melt back partially with good seeding and complete crystal to top and crucible wall.
37	Seed with new-type seed	4°C	90°C	-- Power terminated --		Heat exchanger failure
38	Develop crucible coating to prevent cracking	12°C	No H.E.	N/A	12°C 5 hr.	Thick coating prevented crack- ing in one area.
39	Develop crucible coating to prevent cracking	10°C	No H.E.	N/A	10°C 5 hr.	One crucible ingot combination produced without cracking.

TABLE I. (Cont.)

TABULATION OF HEAT-EXCHANGER AND FURNACE TEMPERATURES

RUN	PURPOSE	SEEDING		GROWTH		REMARKS
		FURN. TEMP. ABOVE M.P.	H.E. TEMP. BELOW M.P.	RATE OF DECREASE H.E. TEMP.	FURN. TEMP.	
40	Determine cause of SiC deposit. Use unground crucible.	6°C	No heat exchanger	No heat exchanger		Heavy SiC deposits on melt stock facing crucible walls.
41	Develop crucible coatings to prevent cracking.	6°C	No heat exchanger	No heat exchanger		Crucible and ingot cracked--coating too thin.
42	Determine mode of heat transfer to helium gas in heat exchanger. Determine cause of SiC deposit.	6°C	Varied			Light SiC deposits.
43	Determine cause of SiC deposit. Partial vacuum with helium atmosphere.	6°C	194°C	No growth cycle.		Light deposit distributed uniformly on seed and melt stock.
44	Seed and propagate crystal growth.	10°C	50°C	150°/15.75 hrs. 9.52°C/hr.	10°C .3°C/hr.	Good seeding with single crystal growth to top and crucible wall

TABLE I. (Cont.)
TABULATION OF HEAT-EXCHANGER AND FURNACE TEMPERATURES

RUN	PURPOSE	SEEDING		GROWTH		REMARKS
		FURN. TEMP. ABOVE M.P.	H.E. TEMP. BELOW M.P.	RATE OF DECREASE H.E. TEMP.	FURN. TEMP.	
45	Determine maximum growth rate. Increase rate of heat exchanger temperature decrease.	10°C	70°C	85°C/4.3 hr. 19.8°C/hr.	10°C 2.3°C/hr.	Good seeding with growth spherical interface to within 3/8" of top surface.
46	Determine maximum growth rate. Increase rate of heat exchanger temperature decrease.	9°C	70°C	170°C/3.5 hrs. 48.5°C/hr.	9°C 2.6°C/hr.	Good seeding with growth spherical interface to within 1/2" of top.
47	Determine maximum growth rate. Increase rate of heat exchanger temperature decrease.	9°C	70°C	262°C/5 hr. 52.4°C/hr.	9°C 1.8°C/hr.	Good seeding with flat interface to 1/8" from top. Cracking minimized.
48	Develop crucible coating to prevent cracking.	5°C	70°C	189°C/2.4 hr. 78.75°C/hr.	7°C 2.9°C/hr.	Cracking near top of ingot.

TABLE I. (Cont.)

TABULATION OF HEAT-EXCHANGER AND FURNACE TEMPERATURES

RUN	PURPOSE	SEEDING		GROWTH		REMARKS
		FURN. TEMP. ABOVE M.P.	H.E. TEMP. BELOW M.P.	RATE OF DECREASE H.E. TEMP.	FURN. TEMP.	
49	Develop crucible coating to prevent cracking	1°C	1280°C	-- Run terminated --		Silicon powder did not sinter and silicon permeated graphite crucible to form SiC
50	Determine maximum growth rate. Increase rate of heat exchanger temperature decrease	9°C	70°C	551°C/4 hr. 138°C/hr.	9°C 2.25°C/hr.	Growth to top of boule. Partial seeding single crystal in center section.

For boule 34 the interface was hemispherical and advanced to within one-half inch from the top of the boule. The major portion of the ingot was very large grained material since the seed melted out, but the grain size above the solidification interface and near the edges of the ingot were much smaller. Some directional solidification in the grain orientation was noted and the physical alignment of these grains was in accordance with the normal triangular shaped section noted from growth using (111) seed plates.

The electrical resistivity of the material in the section examined--surface #3--was all "P" type with resistivities in the range of 10-50 ohm-cm. This range of resistivities is similar to that measured on previous boules.

For run 36 the automatic pyrometer was calibrated and realigned so that the melt point temperature could be observed and the superheat temperature could be controlled to $\pm 1^{\circ}\text{C}$. It was superheated to 5°C and the heat exchanger was supercooled 100°C . The heat exchanger temperature was reduced to 1022°C to force the growth interface to grow to the top surface. The interface broke through the surface as a disc about the size of a dime and increased in diameter until the top was completely solidified. When the growth interface was not forced to the surface, dendrites and a skin formed on the surface. The cooling cycle was modified by spiking the temperature of the ingot from 900°C to the melting point in

one hour to degrade the bond between the ingot and the crucible. After reaching the melt point, the power was terminated. Cracking was minimized in the boule especially at the crucible ingot interface. Cracking nevertheless was unacceptable since the boule was broken into two large pieces. X-ray Laue photographs showed that the fracture surface was almost a (110) plane. This is to be contrasted to the tilted (111) fracture surface obtained on (111) seeded growths. The crystallographic relationships are consistent with the expected results to be obtained from a (100) seed.

The outline of the seed showed that very little melt back occurred but good seeding was achieved. Figure 1 illustrates the heavy concentration of a contaminant, probably SiC, outlining the shape of the initial shape of the interface at the beginning of growth. The (100) oriented ingot grew through the hemispherical band of contamination with almost no breakdown of crystallinity. Single crystallinity extended to the crucible wall and to the top of the ingot.

The heat exchanger was starting to deteriorate since it had been used for 26 runs. Before installing a new heat exchanger, it was decided to seat a seed crystal directly on the heat exchanger and have it project through a precision

Figure 1.

Etched Cross Section of Boule 36 Illustrating
Single Crystal Growth and Shape of Growth Interface
Near Beginning of Growth

Superheat 4°C

Heat Exchange Undercooling 100°C

hole machined into the crucible. A .005"-thick tungsten disc was placed between the heat exchanger and the seed and the crucible was loaded as usual and heated to 1416°C . The heat exchanger was held at 1280°C until the polysilicon was completely melted. The heat exchanger temperature was then increased to 1322°C . After holding at 1322°C for 20 minutes, the top of the heat exchanger melted since silicon diffused into the tungsten and reached the eutectic composition causing melting to occur at 1400°C .¹ This shows that the thermocouple indication inside of the heat exchanger is 80° low, i.e., at a temperature of 1322°C , the actual heat exchanger surface temperature must have been 1400°C . When the heat exchanger melted through, helium entered the chamber and tripped the power out causing the temperature to decrease from 1416°C to 1100°C in about 45 minutes. The resulting ingot grew from the seed and the side, walls, and top. An etched cross-section showed that there was seed melt back and single crystal growth from the seed. The single crystal growth from the seed was surprising, considering the rapid decrease in furnace temperature. This seeding method looks interesting for future work, but the seed will have to extend further below the crucible so the heat exchanger will be out of the heat zone and will not approach the eutectic temperature, or a graphite disc must be placed between the seed and heat exchanger.

1. Ref. 1, p. 5.

Run 44 was conducted to seed and propagate single crystal growth. Temperature sensing equipment was instrumented so that the furnace temperatures could be controlled to $\pm 1^{\circ}\text{C}$. For this run the heat exchanger was held at 1362°C and cooled to 1212°C in approximately 15 hours. The melt was superheated 10°C , good seeding and growth was achieved, and single crystallinity was pushed to the top and crucible wall with some breakdown in crystallinity near the crucible wall. The center of the (100) oriented boule broke out in the form of a pyramid. The sides of the pyramid were (111) planes.

Run 45 was the first of a series of runs (46, 47, 48 and 50) to determine the maximum growth rate for a 1" diameter heat exchanger in a 15 cm diameter fused silica crucible. For these runs the rate of heat exchanger temperature decrease was increased from 19.8°C/hr for run 45 to 138°C/hr for run 50, while the furnace temperature was decreased from 9°C superheat at approximately 3°C per hour. Run 45 was superheated 10°C above the M.P. and the heat exchanger was held at 1342°C . The furnace and heat exchanger temperatures were decreased at 2.8°C/hr and 19.8°C/hr respectively for 4.3 hours. Dendritic growth was observed on top of the melt indicating that the melt was supercooled before solidification was complete. Good seeding and single crystal growth was achieved. The total growth time for

solidifying 2.5 kg of silicon was 4.3 hours. Dendrites froze across the top before solidification was complete. A spherical interface was forced to within 3/8" from the top and edge of the boule. Run 46 was conducted similar to run 45, except the heat exchanger temperature was decreased at 48.5°C/hr to 1119°C in 3.5 hours. The spherical interface was forced to within 1/2" from the top and O.D. of the boule before solidification broke down. Dendritic freezing on top of the melt indicated that the melt was supercooled before solidification was complete.

Run 47 was conducted similar to run 46 using 9°C superheat and 70°C heat exchanger undercooling, except the furnace temperature decrease was changed from 2.6 to 1.8°C per hour. This increased the time to solidify from 3.5 to 5 hours. Dendrites again froze across the top before solidification was complete, but the single crystallinity was pushed to 1/8" from the top of the ingot. Figure 2 is an etched cross section of boule 47 illustrating single crystal growth and shape of growth interface near the end of growth. Good seeding and crystal growth was again achieved. Breakdown in crystallinity away from the center section appears related to the SiC contamination. Run 50 was processed similar to the previous runs except that the rate of heat exchanger temperature decrease was increased from 50°C per hr. to 138°C/hr and the furnace temperature was decreased

Etched Cross Section of Boule 47 Illustrating
Single Crystal Growth and Shape of Growth Interface
Near End of Growth

Figure 2.

at 2.25°C/hr. For this run the interface broke through the top of the liquid in the form of a ½" diameter circle in four hours. The size of the circle increased until solidification was complete to the wall of the crucible. The resulting ingot was single crystal in the entire center section where it had seeded. The outer periphery was polycrystalline due to poor seeding.

Development of Crucible Coatings

Runs 38, 39, 41, 48 and 49 were undertaken to develop a sintered silica coating that would prevent cracking of the ingot and crucible. Various proportions of colloidal silica binders were mixed with fused silica powder or crystalline quartz powder to develop a slurry for dip coating. Laboratory size clear crucibles were prepared for dip coating by etching with HF. To achieve a uniform coating on the inside of the crucible, the crucibles were rotated under a heat lamp while the slurry was applied to the inside surface. All attempts to coat the inside of the crucible by this method failed since the coating did not wet the smooth crucible wall. It was determined that the coatings would have to be applied with an air brush. Mixtures consisting of 50-weight percent colloidal silica binder and 50-weight percent 200-mesh fused silica powder or 200-mesh quartz powder mixed by tumbling. The three colloidal silica binders used

consisted of the composition presented in Table II. The composition of the fused silica and crystalline quartz powder is presented in Table III.

Nine crucibles with various coating and binder modifications were sintered at 500°C for one hour. For the crucibles coated only with colloidal silica binders, thin films formed and peeled off. The quartz coatings also separated from the wall of the crucible, probably due to its higher thermal expansion coefficient. The three crucibles on which the quartz powder coatings separated were recoated and dried only under a heat lamp. The three crucibles with sintered fused silica coatings and the three crucibles with the unsintered quartz coatings along with an uncoated crucible were loaded with approximately 50 grams of silicon. The crucibles were heated to 1420°C in 15 hours, held above the melt point for 4 hours, and cooled to room temperature in 40 hours. The plain crucible and ingot were totally fragmented into pebble. All the coated crucibles and resulting ingots were cracked. There were areas, however, where the coating prevented the silicon from contacting the crucible wall and eliminated cracking. There was no adherence of the ingot to the sintered coating and this prevented cracking. It appeared that thicker and more uniform coatings would be required. To make the coating more uniform, a mixture of 26 weight per cent fused silica or quartz powder and

TABLE II
TYPICAL PROPERTIES OF COLLOIDAL SILICA

	1	2	3
Stabilizing counter ion	Sodium	Sodium	Ammonium
Particle Charge	Negative	Negative	Negative
Particle Size, nanometer	14-15	21-24	13-14
Specific surface area, sq m/g	195-215	125-140	210-230
Silica (as SiO ₂), wt %	30	49.5	30
pH (25°C, 77°F)	8.4	8.9	9.6
Titratable alkali (as Na ₂ O), wt %	0.10	0.21	0.09 ⁽¹⁾
SiO ₂ /Na ₂ O (by wt)	300	230	120 ⁽²⁾
Chlorides (as NaCl), wt %	0.003	0.03	0.002
Sulfates (as Na ₂ SO ₄), wt %	0.003	0.10	0.003
Viscosity (25°C, 77°F), cp ⁽³⁾	14	50	20
Weight per gallon (25°C, 77°F), lb	10.1	11.6	10.1
Specific gravity (25°C, 77°F)	1.21	1.39	1.21
Conductance (25°C, 69°F), microohms	1570	-	2630

(1) Sol contains 0.25% NH₃ and 0.09% Na₂O

(2) SiO₂/NH₃ (by weight)

(3) Measured by Ostwald-Fenske pipette No. 100 or 200,
depending on viscosity range

TABLE III

COMPOSITION OF FUSED QUARTZ AND CANDLE QUARTZ

	FUSED QUARTZ	CANDLE QUARTZ
Description	White Powder	White Powder
Form	Amorphous	Water clear hexagonal prisms
Density	-	2.66
Refractive Index	-	1.55
Particle Size	200-mesh 70 μm	200-mesh 70 μm
Analysis	Silica (SiO_2) (99.99%)	Iron oxide (Fe_2O_3) (0.4 ppm)
	Aluminum (Al) (27 ppm)	Titanium dioxide (TiO_2) (0.25)
	Sodium (Na) (10 ppm)	Aluminum oxide (Al_2O_3) (34.2)
	Potassium (K) (6 ppm)	Calcium oxide (CaO) (1.0)
	Iron (Fe) (6 ppm)	Magnesium oxide (MgO) (0.6)
	Calcium (Ca) (6 ppm)	Sodium oxide (Na_2O) (8.7)
		Potassium oxide (K_2O) (4.2)
		Lithium oxide (Li_2O) (5.1)

74 weight per cent liquid binder was used. The crucibles coated with fused silica were heated to 500°C for one hour, recoated and reheated to 500°C. Crucibles with quartz coatings were dried under a heat lamp. The three quartz and three silica coated crucibles along with a plain crucible were loaded with approximately 50 grams of silicon and heated to 1420°C in 20 hours. The crucibles were held above the melt temperature for 4 hours and cooled to room temperature in 30 hours. A crucible coated with fused silica powder and colloidal silica binder #1 was not cracked. The ingot came out of the crucible uncracked also. A crucible with quartz particles and colloidal silica binder #1 had only one crack and the ingot was uncracked. The crucible and ingots that failed appeared to crack due to flaking off of the coatings allowing the liquid silicon to contact the crucible wall. It appears that binder #1 is aiding in holding the coatings to the crucible wall. Fortuitously, this binder has the lowest impurity concentration.

Run 41 was conducted to develop the sintered silica coatings. Coating mixtures with 20, 33.3 and 42.8 weight per cent fused silica powder in the low sodium colloidal silica binder were mixed by tumbling. After thorough mixing, the liquid mixtures were sprayed onto the inside surface of fused quartz crucibles that were rotating under a heat lamp. The crucibles were then heated to 1100°C for approximately

an hour. All three crucibles were cracked due to devitrification. The Na ion in the binder apparently diffused into the fused silica to cause devitrification.

Crucibles with the same coatings were coated a second time and heated at 1000°C for one hour. Coatings with 33.3 and 42.8 weight per cent fused silica powder sintered and stayed attached to the wall, but the coating with 20 per cent silica sintered and shrank away from the wall. There was no sign of devitrification for this sintering temperature. The two crucibles with coatings intact were loaded with 50 gm of silicon heated to 1415°C for $2\frac{1}{2}$ hours and cooled to room temperature in 24 hours. In some areas the coatings pulled free of the crucible allowing the liquid silicon to contact and attach to the crucible. This caused the ingots to crack in the area of attachment. In the area where the coating remained intact, the ingot separated freely from the coating. No sign of devitrification cracking could be observed on any of the crucibles heated to 1415°C in vacuum. It appears that in vacuum the volatile Na ion is pumped out of the system before it causes the fused silica to devitrify.

For run 48, the crucible size was scaled up to approximately 3" diameter. A coating mixture consisting of 26 per cent fused silica and 74 per cent binder #1 was sprayed on the inside of the 3" diameter crucible and two $1\frac{1}{2}$ " diameter crucibles. The crucibles were sintered at 500°C for 12 hours

and loaded with silicon. The large crucible was seated on the heat exchanger and the two smaller ones were placed beside the large crucible. The crucibles were heated to 1420°C in 20 hours and held at this temperature for two hours until the silicon had melted. After stabilization at 1417°C for one hour, the melt was directionally solidified in 2 hours and cooled to room temperature in 12 hours. The directionally solidified ingot was cracked near the top and the ingots from the smaller crucibles were uncracked although the crucibles were broken. The ingot and crucible cracking appears to occur in areas where the crucible coating had flaked off and the liquid silicon contacted the fused silica crucible.

For run 49 the 200 mesh fused silica powder was mixed in a 1:1 ratio with DI water to form a slurry. This slurry was applied to the inside surface of a 2½" diameter graphite crucible. The coating was non-uniform and powdery since there was no binder and it could only be sintered at 350°C. The crucible was loaded with 100 gm of silicon and taken up to the melting temperature in 20 hours. Upon reaching the melting temperature, there was severe outgassing. The power was terminated and the furnace was cooled to room temperature in less than 8 hours. It was apparent that the coating did not sinter or adhere to the inside wall of the crucible since silica powder was present in the bottom of the

crucible. Liquid silicon penetrated the pores of graphite to form SiC, causing the crucible to crack due to expansion on formation of SiC.

Determining the Source of SiC Contamination

Runs 40, 42, and 43 were conducted to determine the cause of SiC contamination. For run 40 silicon melt stock was loaded into a crucible that did not have a ground bottom surface. Melt stock adjacent to the ground surface in run 9 was completely covered with the silicon carbide contamination. It appeared that the carbon contamination was coming from the ground surface, either from SiC particles left from grinding or from organic contaminants that penetrated into pores opened by the grinding operation. The loaded crucible was placed into the furnace, evacuated, and heated to the melt temperature in 20 hours. This was about five hours longer than usual. It was held at 1415°C for 15 minutes before decreasing the temperature. For this run slight melting occurred and heavy deposits of the yellow SiC covered the material that faced the crucible wall. The deposit was not apparent on the silicon melt stock one layer inside of the crucible or on top. The yellow deposit was identified as beta SiC by Debye-Scherrer Powder patterns. This experiment proved that the carbon was not coming from ground surfaces. It appeared that the deposits were located opposite graphite support pieces.

For run 42 a graphite support ring was sectioned to go only half way around the crucible. This experiment was conducted to determine if carbon was coming from the graphite supports, diffusing through the crucible wall and combining with the silicon melt stock to form SiC. The loaded crucible was heated to 1415°C in 28 hours and held at 1415°C for approximately 1.5 hours. Very little SiC deposits were apparent on any of the silicon melt stock. Free carbon did not appear to be coming from the graphite support. The source of free carbon available for reaction with the silicon appears to vary from run to run, possibly due to absorbed moisture from the heat zone or back streaming of vacuum pump oil.

Run 43 was conducted to determine if SiC deposit forms under a cover of helium gas. After the chamber was evacuated to .1 Torr, it was back filled with helium, re-evacuated, and back filled to a 50 Torr of helium. The chamber was isolated from the vacuum system, heated to 1418°C in 27 hours and held at that temperature for one-half hour. The pressure of the stagnant helium cover gas rose to 225 Torr at this temperature. Seventeen kilowatts of power was required to maintain this temperature as compared to 7.6 kilowatt that is consumed at 0.1 Torr. Some melt stock and seed had a thin pale green coating on its surface.

The pale green coating was more uniform and not concentrated only on melt stock near the crucible wall. The impurity was identified as SiC by an 8-hour exposure of Debye-Scherrer film.

SILICON CRYSTAL SLICING

Summary

During the reporting period slicing testing was continued on the modified multi-blade wafering machine, using diamond-impregnated wire blades and coordinated motion between blades and workpiece.²

Testing during this period involved attempts to improve cutting rates, abrasive life, wafer surface condition, and blade wander.

Attempts were made to obtain abrasive life data, to measure effects on slicing performance of blade and coolant silicon-contamination, and to recharge blades in mid-cut.

Methods employed included use of wire blades with higher concentrations of diamond, changes in machine operation, modification of the wire blade pack, and increased care in blade alignment.

Also involved were continued slicing with blades from previous testing, cleaning of blades, use of an alternate coolant, and recharging blades without disturbing blade or workpiece positioning.

Modifications to machine components were begun for the purposes of simplifying feed force control and reducing

2. Ref. 2, pp. 23-33, and ref. 3, pp. 29-56.

blade wander.

During the same period efforts continued toward simplification of the data reduction presently required.

Wire blade samples were characterized with tests of ultimate strength, hardnesses of core and coating materials, and scanning electron microscope photography of wire surfaces and cross sections.

Run 7

Run 7 was intended to measure effects of higher diamond concentration and more-continuous machine operation on cutting rate, abrasive life, and wafer surface damage.

A silicon (111) single crystal workpiece 1.75 in. high and having a 1.5 in. kerf length was cut with 25 wire blades.

The blades were made from 0.006 in. diameter chromium-nickel stainless steel wire, coated with a 0.001 in. thick copper coating and charged two times with 45 μm natural diamonds.

The wire blades were pretensioned to 200,000 psi. The nominal feed force used was 0.15 pounds per blade, and the machine was run at a speed of 160 strokes (or 80 cycles) per minute. Feed force and machine speed were increased gradually during startup until the nominal values were obtained. A constant feed force was maintained during

prolonged cutting.

Preliminary values of cutting rate appeared to vary throughout the run between 1.5 and 2.0 mil/min, the variations being due most largely to shutting down and starting up the machine. Interruption of cutting normally has reduced cutting rate, at least temporarily. Variations in the present startup, shutdown, and re-start procedures are expected to accomplish continuing improvements in cutting rate and the other dependent variables.

Two abnormal events occurred during run 7, which may have caused unanticipated abrasive damage.

On two occasions components in the rocking linkage loosened, temporarily halting the rocking of the workpiece, while cutting continued.

A comparison of cutting rates provided by run 8³ suggests some reduction of cutting rate may have occurred as a result of the malfunctions.

Comparison of preliminary cutting rates from run 7 to those from run 2B⁴ indicates that abrasive life was at least doubled in run 7.

After cutting more than twice the distance, cutting rates were obtained in run 7 at 160 strokes per minute which were at least comparable to those from the end of run 2B at 180 strokes per minute.

A value for abrasive life was not obtained for run 7,

3. P. 26.

4. Ref. 3, p. 36.

because the blades were removed at the end of the cut for later inspection. However, they were still cutting at a rate of from 1.5 to 2.0 mils per minute.

Visual comparison of wafer surfaces from run 7 to those from runs 2 and 2B indicates a large reduction in the quantity and severity of scoring produced.

Wafer surface damage which occurred in run 7 appeared to be caused by two or three different sources, which are expected to lend to continuing improvements. These include: blade alignment, flexing of components in the workpiece support system, and vibration in the form of jerk at the end of each stroke, due to the high unbalanced inertial forces of the present reciprocating bladehead and bladehead drive system.

In each of these areas, component redesign is expected to reduce causes of wafer surface damage, when the order of priorities warrants additional machine development effort.

Run 8

Run 8 was intended to provide a measure of the effectiveness, in reducing blade wander, of blade restraints bonded to the wire blades on either side of the workpiece.

For this purpose lightweight wood and epoxy bridges were positioned and bonded to the wires approximately 10

inches apart. This reduced the unrestrained blade span from 15 inches to 10 inches, and provided 0.25 in. clearance on either side of the workpiece.

In addition to the blade restraints, in run 8 the wires were aligned more precisely than in any tests to date, and the machine was shut down fewer times during the run than in any prior tests.

Other conditions were the same as for run 7, although no mechanical malfunctions occurred in run 8.

Preliminary cutting rates from run 8 varied between 2.0 and 2.5 mils per minute throughout the run, similarly to the cutting rates from run 7, but 20 to 25 per cent higher.

The higher cutting rates in run 8, compared to those in run 7, may have been due in part to the two interruptions of the rocking motion during run 7.

Such interruptions of rocking while the feed force is at or near operating levels could cause blade damage, according to our model for blade/workpiece interaction. This would occur when wire blades contact sharp crystal edges while moving in an unfavorable direction.

The effects of blade restraints of the type employed in run 8, in reducing bow and taper, will best be determined from statistical measurements. Only a small influence, if any, is visible to the eye.

Improvements in blade wander from increased blade tension and wire support are alternatives currently under consideration.⁵

Wafer surface quality from run 8 appears slightly improved over that from run 7, particularly in the larger central portion of the cut which was done non-stop on the second of the three days during which the cut was made.

Startup, shutdown, and restart procedures are expected to improve the types of damage apparent between the three different days' cutting. Other improvements as discussed above in conjunction with run 7 should improve the other surface texture characteristics observed on run 8 wafers.

As in run 7 life values had not been obtained from run 8 data at the time of this writing.

Run 9

Run 9 was intended to measure the abrasive life limit of the wire blades used in run 8.

At the end of run 8 cutting rates were holding in the range of approximately 2 to 2.5 mils per minute, as they had throughout the run.

Run 9 was begun 13 calendar days after the end of run 8. Wire tension was verified to have remained unchanged and other conditions were maintained essentially identical

5. P. 46.

to those of run 8, except for omission of the blade restraints.

Cutting rate during run 9 averaged approximately 0.5 mil/min (20 per cent of the run 8 value) and the run was terminated after cutting only 0.25 inches when cutting rate had decreased to approximately 0.25 mil/min (10 per cent of the run 8 value).

Decreases in cutting rate have been observed after shutdown and restart in the past, as mentioned above.⁶

It has been reported⁷ that for silicon slicing the strength of phosphor bronze ID blades was adversely affected by some coolants which chemically corroded the material. The Cupric Ion Process for chemical-mechanical polishing of silicon is further support that copper and silicon react with each other. On the basis of industrial experience in slicing silicon with nickel-bonded ID blades, nickel may be less susceptible to degradation by corrosion. Nickel coatings also have the advantage that they can be heat treated to control hardness.

Previous interruptions of cutting have typically been for overnight periods, and wafer surface damage has typically been associated with restarting, as discussed above.⁸

Data from run 9, which followed a shutdown of 13 days in length, suggested that decreases in cutting rate and the

6. P. 25.

7. Ref. 4

8. P. 28.

occurrence of wafer surface damage after shutdown and re-starting may result from blade deterioration. This may occur through corrosion.

Since no precautions against such deterioration of the wire blades have been utilized in the past, except for superficial drying, it now appears that attention to this in the future may significantly extend abrasive life for slicing with fixed abrasive wire blades.

Run 10

Run 10 was intended to measure the effects on slicing performance of silicon-contamination of blades and coolant.

Twenty-four wire blades from runs 8 and 9 were cleaned with a degreasing solvent, then with detergent and water. The water and ethylene glycol solution used as the coolant in runs 8 and 9 was replaced with a dilute detergent and water solution.

Under 40X magnification the wires in the cleaned state were much closer in appearance to the bright copper color of new wires, as opposed to the gray appearance of uncleaned wires.

The concentration of diamonds on the cutting surfaces of the wires was sparse compared to that of unused wire, perhaps less than 10 per cent.

Cutting rates in run 10 were approximately the same as in run 9 (approximately 10 per cent of those of run 8).

If effects from silicon removal were present, statistical data reduction would best provide whatever comparison might be made.

Run 10 would seem to confirm most of all the importance of high diamond concentration for high cutting rates.

Run 11

Run 11 was intended to measure the effects of re-charging the wire blades from runs 8, 9, and 10 in mid-cut, with an in-house charging procedure.

The 24 wires from runs 8, 9, and 10 were charged with 45 μ m natural diamonds under a light pressure and for a relatively small number of strokes of the blade carriage. This was a simplified version of the charging procedure used in run 6.⁹

Cutting rate during two and one half hours of cutting at the same feed force and carriage speed as run 10, varied between 0.6 and 0.8 mil/min. This was an increase over the rate in runs 9 and 10 but still well below an acceptable value, such as the 3.5 to 4 mil/min achieved with the charging procedure in run 6.

Additional testing will be undertaken for the purpose of evaluating charging and re-charging methods.

9. Ref. 3, pp. 40 and 53.

Machine Modifications

Changes were made in components of the vertical feed mechanism for the purpose of improving the response of feed force to air pressure. The small adjustment in pressure needed during a typical test was reduced by 50 per cent. Further modifications are expected to completely eliminate the need for such adjustments.

Such changes may improve wafer surface texture while reducing the manpower required for machine operation and data reduction.

A system for guiding and supporting the wire blades on either side of the workpiece has been fabricated and installed. Testing underway at the time of this writing has confirmed that in slicing with wire blades, support guides at these locations reduce blade wander by an order of magnitude.

Wire Blade Characterization

A small number of plain wire and wire blade samples were tested for strength, hardness, and composition, and were photographed with a scanning electron microscope at 300X and 2000X magnification.

Evaluation of these and previous such data is continuing. Preliminary observations include the following.

Impregnation

Differences in appearance between wire blades impregnated with different procedures suggest that damage to wire surfaces and fracturing of diamonds during impregnation, with resulting pullout of diamonds during slicing, can be reduced through further development of impregnation procedures. The experience from run 6¹⁰ would support this conclusion.

In Figure 3 the photographs labelled "Process A" are of one of the wire blades charged with a Crystal Systems process in run 6 and selected randomly before cutting. How representative the sample was is uncertain.

The sample labeled "Process B" in Figure 3 is a randomly selected segment of the double impregnated blade material used in runs 7 and 8, in its as-received condition.

Core Materials

Damage to drawn wires, in the form of nicks or gouges, very likely accounts for significant differences in tensile strengths of different wire samples.

In Figure 4 a random comparison between two drawn stainless steel wires suggests how damage (top photographs) could result from drawing, reducing the tensile strength of the core wire and hence that of the finished metal-

10. P. 31.

Figure 3. Cu-steel wire, 0.008 in D, 45 μ m diamonds, impregnated (300X and 2000X). (Top) Process A. (Bottom) Process B.

Figure 4 . Stainless steel wire, 0.005 in D (300X and 2000X).
 (Top) Hard drawn. (Bottom) Annealed.

Figure 5. Copper coated stainless steel wire, 0.008 in OD (300X and 2000X).

coated impregnated wire blade made from it.

Metal Coatings

Electroplated metal coatings in general vary in properties dependent on plating conditions. Consistently durable metal coated impregnated wire blades presumably depend on consistency in the electroplated metal coating.

Figure 5 presents two photographs of a randomly selected segment of copper coated stainless steel wire from the same lot as was charged with a Crystal Systems process in run 6. Particularly the 2000X closeup suggests the beginning of breakdown of the electroplating from a laminar to a columnar (less ductile) structure.

DISCUSSION OF RESULTS

Determining the Maximum Growth Rate

Crystal growth is controlled by heat transfer to and extraction from the crystal interface. The growth rate is determined by the difference in rate of heat transfer to and away from the interface.

If the melt has a high superheat, a high liquid gradient, $\frac{dT_l}{dr_l}$, a high rate of heat input to the interface results. The solid gradient $\frac{dT_s}{dr_s}$ must also be high, low heat exchanger temperature, to extract the heat rapidly enough to make growth proceed.

The maximum growth rate is achieved by maximizing the rate of heat extraction and minimizing the heat input to the solid liquid interface. This is controlled by the rate of decrease of the heat exchanger and furnace temperature.

In runs 44, 46, 47 and 50, the rate of heat input and extraction were varied by changing the rate of decrease of furnace and heat exchange temperature decrease.

These experiments show that the growth rate is most sensitive to the rate of furnace temperature decrease. For example, the furnace and heat exchanger temperature decrease for boule 45 was 2.3°C/hr and 19.8°C/hr respectively. The

interface advanced to 3/8" from the top of the boule in 4.3 hours. For boule 50 the furnace and heat exchange decrease was 2.25 and 138°C/hr respectively. Even though the heat exchanger temperature decrease was 6.95 times as fast, the growth rates were nearly the same. For these two runs the furnace temperature decrease was nearly the same, 2.3 and 2.25°C respectively, and the growth times were 4.3 and 4 hours respectively.

If the furnace temperature is decreased rapidly to achieve a fast growth rate, the heat exchanger temperature must be decreased rapidly to maintain a planar interface. This was demonstrated by run 45. The rate of heat exchanger temperature decrease was 19.8°C/hr. The interface broke down about 3/8" from the top of the boule because heat was not conducted away from the interface quickly enough. For run 50 the heat exchanger temperature was decreased at 138°C/hr. This was sufficient to force the interface to the top surface.

It is apparent that both the furnace and heat exchanger temperature must be decreased rapidly to achieve fast growth. There may be a limitation on how low the heat exchanger temperature can be reduced without inducing structural defects such as dislocations and twins. For run 50 the heat exchanger temperature was decreased to 791°C. This did not induce cracking of the ingot. Characterization is underway to determine the structural effect of this low heat exchanger temperature.

Determining the Mode of Heat Transfer in the Heat Exchanger

Run 42 was conducted to determine the mode of heat transfer to the helium gas. The furnace temperature was held at 1415°C while the helium flow was varied in increments of 5 liter/minute.

The temperature of the heat exchanger surface was measured with a platinum 10% rhodium thermocouple and the helium inlet and exhaust temperatures were measured with chromel alumel thermocouples. The position of the thermocouples is shown in Figure 6.

Heat exchange temperatures along with the helium inlet and exhaust are plotted as a function of the helium flow rate in Figure 7.

The temperature of the heat exchanger was never more than 50°C different than the inlet and exhaust temperature. It is interesting to note that the inlet temperature is higher than the exhaust temperature for flows higher than 25 liter/minute. Apparently most of the heat exchange occurs in the injection tube since the inlet and exhaust temperatures are nearly the same. For higher flow rates, i.e., above 25 liter/minute, the expansion from the inlet tube into the heat exchanger is sufficient to cool the exhaust gas below the inlet gas temperatures.

Figure 6 .
 Thermocouple Position to Measure Heat Exchanger
 Temperature and Helium Inlet and Exhaust Temperature

Figure 7.
Temperature of the Heat Exchanger and
Helium Inlet and Exhaust Temperature
as a Function of Helium Flow Rate

Determination of Stress Due to Thermal Expansion Mismatch

It has been shown that cracking is caused by localized stress between the silicon and silica.¹¹ Stress between two materials with different expansion coefficients rigidly attached to each other can be expressed as follows:

$$S = \alpha L \Delta T$$

α = linear thermal expansion coefficient

L = length

ΔT = temperature change

S = change in length

strain e (deformation per unit length) =

$$e = \alpha \Delta T$$

for two materials with different expansion coefficients

$$e = (\alpha_1 - \alpha_2) \Delta T$$

assume

$$\alpha_{\text{silicon}} = 4.5 \times 10^{-6} \text{ } ^\circ\text{C}^{-1}$$

$$\alpha_{\text{silica}} = .9 \times 10^{-6} \text{ } ^\circ\text{C}^{-1}$$

$$e = (3.6 \times 10^{-6}) \Delta T$$

$$\sigma = e E$$

where

σ = stress and E is the modulus of elasticity

$$\sigma = (3.6 \times 10^{-6}) E \Delta T$$

$$E = 1.9 \times 10^{12} \text{ dynes/cm}^2 \text{ } \{111\} \text{ direction}$$

$$\sigma = 6.83 \times 10^6 \text{ dynes/cm}^2 \text{ } ^\circ\text{C}^{-1} (\Delta T)$$

11. Ref. 1, p. 11.

Cracks have been observed at about 650°C. The fracture stress of silicon at 650°C is 25×10^8 dynes/cm².

$$\Delta T = \frac{\sigma}{6.83 \times 10^6} = \frac{25 \times 10^8}{6.83 \times 10^6} = 366^\circ\text{C}.$$

The calculation indicates that a 366°C temperature change is required to cause the silicon to fracture. This indicates that below 1000°C the silicon no longer plastically deforms to release the stress due to the thermal expansion mismatch. The solution for linear stress is extended to the two dimensional case below:

$$\begin{aligned} \Sigma F_x &= \sigma AB \cos 45 + \sigma AD \cos 45 = \sigma^1 \frac{AB}{\cos 45} \\ &= 2 \sigma AB \cos 45 = \sigma^1 \frac{AB}{\cos 45} \\ \sigma &= \sigma^1 \end{aligned}$$

This solution shows that the linear stress is the same as the resolved stress on a two-dimensional element.

Development of Crucible Coating to Prevent Ingot Cracking

It has been demonstrated that a sintered silica coating can be used to prevent cracking of the crucible and ingot. Silica coating prevents the silicon from contacting and attaching itself to the fused silica crucible. The liquid silicon does not form a strong bond with the sintered coating and therefore the ingot breaks free of the sintered coating preventing cracking of both the crucible and ingot.

Ingot cracking appears to occur in coated crucibles when the coatings break loose from the crucible wall allowing silicon to contact the crucible. This appears to be due to a weak bonding between the coating and crucible. The coatings were applied to smooth crucible surfaces since it was felt that coating would have to break free from the crucible. This is not the case. It appears in fact that it will be necessary to form a strong bond between the coating and crucible to prevent the coating from detaching from the crucible. A roughened surface will be used in the future to make it easier to apply the coatings and to allow for better bonding between the coating and crucible.

Reducing Wander in Slicing

Wander of the cut is one of the factors that could limit the slicing yield. Thin wafers .015 cm cannot be sliced if the wires wander more than .0025 cm. Up to 0.025 mil .01 cm taper was observed in wafers cut to date. Wander is caused by various factors such as (1) low wire tension, (2) non-uniform distribution of diamond on the cutting edge, (3) poor machine alignment and (4) loading of the cutting edge with swarf.

Wander can be minimized by taking corrective action for these conditions. In addition, a wire support system can be used to prevent the wire from wandering.

On the basis of slicing tests it was felt that a wire support system would greatly reduce blade wander.

In run 8 a lightweight wood and epoxy bridge was positioned and bonded to the wires approximately 10 inches apart. This reduced the unrestrained blade span from 15 inches to 10 inches, and provided 0.25 in. clearance on either side of the workpiece.

In addition to the blade restraints, in run 8 the wires were aligned more precisely than in any tests to date, and the machine was shut down fewer times during the run than in any prior tests.

The blade restraints bonded to the wire on either side of the workpiece did not have a significant effect in

reducing wander.

For typical wire blade slicing operations support pulleys are located on either side of the work. This supports the wire so that high feed forces can be used with no breakage and with greatly reduced wander.

A system using grooved rollers was installed to guide and support the wires.

With rollers supporting the wire on either side of the workpiece, much larger forces could be applied without breaking the wires. This support may be necessary when larger forces are applied for slicing larger cross-sectional area with thinner wires or blades.

Run 13 was conducted using the wire support system. In this run wander was reduced by an order of magnitude compared to previous runs.

Changes had to be made to the wire support system during the run. As a result, additional values for slicing performance will be obtained from further testing. The wire support system appears to be a significant step in reducing wire wander. Refinement of this system, higher tension, more uniform diamond distribution, better machine alignment, and better swarf removal will be employed to attempt to reduce wander to less than one mil (.0025 cm.)...

CONCLUSIONS

1. The amount of single crystal material solidified was increased by increasing the rate of heat extraction; i.e., increasing the rate of heat exchanger temperature decrease.

2. Growth rate was heavily dependent on the rate of furnace temperature decrease. For fast growth rates, fast decrease in furnace temperature, the heat exchanger temperature had to be decreased rapidly to conduct away the heat of fusion to prevent breakdown of the interface.

3. Sintered fused silica coatings prevent cracking of silicon ingots in laboratory size fused silica crucibles. Sintered coating separates freely from the silicon during cool down to prevent cracking.

4. Silicon carbide deposition on the silicon melt stock was not traced to carbon transport from ground crucible surfaces or from graphite support rings in contact with the fused silica crucible.

5. Most of the heat transfer in the heat exchanger occurs in the injection tube.

6. Increasing the concentration of diamond on the copper-plated wire has improved the abrasive life. Only

slight degradation in cutting rate has been observed after slicing through a $1\frac{1}{2}$ " cube of silicon.

7. Reduced cutting rates were experienced when diamond impregnated wire was intermittently used over a period of time. As time out of service increased, cutting rate decreased, suggesting that corrosion may degrade the copper diamond bond and decrease abrasive life.

8. Grooved rollers can be used to support and guide the wire on either side of the workpiece. This reduced wander an order of magnitude.

SCHEDULE OF MILESTONES--CRYSTAL CASTING

PAGE 1 OF 2

ITEM	DESCRIPTION	Month 1976												Dec	Jan	Feb	Mar	Apr	May	June	July	Aug	Sept	Oct	Nov
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

CRYSTAL SYSTEMS, INC.
CONTRACT # 954373

SCHEDULE OF MILESTONES--CRYSTAL SLICING

PAGE 2 OF 2

	DESCRIPTION	Month 1976											
		Dec	Jan	Feb	Mar	Apr	May	June	July	Aug	Sept	Oct	Nov
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

CRYSTAL SYSTEMS, INC.
CONTRACT # 954373

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