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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

Seventh Quarterly Progress Report, March 22, 1977—June 30, 1977

Frederick Schmid
Chandra P. Khattak

July 1, 1977

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

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ENERGY RESEARCH AND DEVELOPMENT ADMINISTRATION

Division of Solar Energy

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MAN-HOURS AND COST TOTALS

Previous		Current		Cumulative	
M/Hr.	Cost	M/Hr.	Cost	M/Hr.	Cost
6,474	$285,172	2,118	$ 98,168	8,592	$383,340

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ABSTRACT

Efforts in crystal casting during the last quarter were directed towards obtaining crack-free, single crystal boules. A crack-free 15cm diameter silicon ingot has been cast using thin-wall, clear silica crucibles. A number of crack-free ingots have been solidified on smaller scales using sintered silica liners and coatings. Single crystal growth has been achieved all the way to the top of the seed as well as laterally to the crucible walls.

Sixty-four wafers per inch (5-7mils, 0.125-0.175mm thick) were sliced using fixed diamond abrasive particles. The diamond impregnated wire fails because of diamond pull-out, thereby having shorter life as compared to the diamond plated wire. Impregnation of diamond can be carried out to high concentrations in house. This wire after plating is expected to yield a suitable blade for silicon slicing. A cost analysis has shown that the cost of expendable materials, diamond and wire, are negligible for slicing silicon with fixed diamond abrasive.

EXPERIMENTAL RESULTS

CRYSTAL CASTING

Efforts during this quarter were directed towards obtaining a crack-free silicon boule with single crystal growth all the way to the top as well as to the walls of the crucible. A number of fused silica liners and casting procedures were developed to prevent cracking of the ingot after solidification. The fused silica alternatives studied include hot-pressed, slip-cast, pressure-cast, glazed and spray-coated crucibles. Some of these have been obtained from trade sources while others have been developed in house.

Ingot Casting

During last quarter,[1] good growth was generally achieved above the seed. However, at the bottom of the crucible around the seed not much lateral growth was obtained. During this quarter the heat flow conditions were altered so that they are conducive to crystal growth and single crystal growth was obtained above the seeds as well as laterally. The experimental conditions are summarized in Table I.

TABLE I.

TABULATION OF HEAT-EXCHANGER AND FURNACE TEMPERATURES

RUN	PURPOSE	SEEDING		GROWTH CYCLE			REMARKS
		FURN. TEMP. ABOVE M.P. °C	H.E. TEMP. BELOW M.P. °C	TEMP. DECREASE H.E. TEMP. °C/HR.	TEMP. DECREASE FURN. TEMP. °C	GROWTH TIME IN HOURS	
70-C	Achieve lateral growth	< 3	23	81	0	6.5	Insufficient melt-back of seed
71-C	Achieve lateral growth	< 3	19	59	0	8.75	Graphite ring at bottom broke
72-C	Achieve lateral growth	< 3	34	92	0	6.8	Good melt-back achieved
73-C	Sintering of crucible liners at approx. 840°C	-	-	-	-	-	No change in appearance of crucibles after the run.
74-C	Achieve lateral growth	< 3	33	85	0	5.75	Good melt-back and lateral growth achieved
75-C	Provide better heat conduction to the heat exchanger	< 3	79	96	0	7.25	A graphite plug was used at the hole in center of crucible. Seed lost.
76-C	To avoid cracking of ingot using SiO_2 liners	~ 10	-	-	-	4.0	Silicon in high purity liner has not cracked.
77-C	Achieve lateral growth	?	57	87	0	4.25	Instrumentation mal-function. Seed melted.

TABLE I. (Cont.)

TABULATION OF HEAT-EXCHANGER AND FURNACE TEMPERATURES

RUN	PURPOSE	SEEDING		GROWTH CYCLE			REMARKS
		FURN.TEMP. ABOVE M.P. °C	H.E.TEMP. BELOW M.P. °C	TEMP. DECREASE H.E. TEMP. °C/HR.	FURN. TEMP. °C	GROWTH TIME IN HOURS	
78-C	To avoid cracking of ingot using SiO_2 liners	< 3	-	-	-	-	Regular grade thin crucible showed no cracking of ingot
79-C	To achieve lateral growth	< 3	44	86	0	6.0	Not enough melt-back of the seed achieved
80-C	To achieve lateral growth	< 3	38	58	0	8.0	Good growth achieved to one side of the seed
81-C	Casting in seeded free standing crucible liner	3	77	46	0	6.25	Seed melted.
82-C	Achieve lateral growth	<3	64	90	0	9.5	Good seed melt-back. Single crystal above the seed as well as good lateral growth.
83-C	Achieve lateral growth	<3	70	88	0	8.5	Growth above the seed as well as in the lateral direction.
84-C	Testing of hot pressed and high purity crucibles. Sintering of pressure cast crucibles.	<3	137	51	2	9.0	The silicon in the hot pressed crucible did not show any cracking.

TABLE I. (Cont.)

TABULATION OF HEAT-EXCHANGER AND FURNACE TEMPERATURES

RUN	PURPOSE	SEEDING		GROWTH CYCLE			REMARKS
		FURN.TEMP. ABOVE M.P. °C	H.E.TEMP. BELOW M.P. °C	TEMP. DECREASE H.E. TEMP. °C/HR.	FURN. TEMP. °C	GROWTH TIME IN HOURS	
85-C	Achieve good growth	<3	75	79	0	6.0	Good seed melt-back and growth above the seed and sides
86-C	Testing of spray coated crucibles	<3			0		Coatings generally "crazed."
87-C	Study effect of new viewport	<3	62	67	3	12.6	Melt-back of seed near sides only.
88-C	Improve heat transfer	<3	59	-	0	-	Run aborted. Heat exchanger damaged.
89-C	Study effect of ultrasonic sensor rods	8	51	139	8	5.5	Seed melted out.
90-C	Casting in spray-coated, pressure-cast and hot-pressed crucibles.	4	137	118	6	10.75	Crackfree silicon in two pressure-cast crucibles. Silicon in the spray-coated crucibles showed fine cracks.
91-C	Prevent cracking using thin, clear crucible.	7	75	155	-	-	Run aborted due to crucible failure.
92-C	Prevent cracking using low-density, slip-cast silica crucible.	16	217	-	-	-	Run aborted due to crucible failure.

TABLE I. (Cont.)

TABULATION OF HEAT-EXCHANGER AND FURNACE TEMPERATURES

RUN	PURPOSE	SEEDING		GROWTH CYCLE			REMARKS
		FURN. TEMP. ABOVE M.P. °C	H.E. TEMP. BELOW M.P. °C	TEMP. DECREASE H.E. TEMP. °C/HR.	TEMP. DECREASE FURN. TEMP. °C	GROWTH TIME IN HOURS	
93-C	Prevent cracking using thin, clear crucible.	4.5	97	177	8.5	7.5	Crack-free ingot cast.
94-C	Casting in glazed slip-cast crucible.	<3	94	164	0	2.5	Crack-free ingot cast.

It was felt that heat was radiating from the bottom of the crucible directly to the cold spot in the setup--the heat exchanger. Therefore, sufficient melt back at the circumference of the seed was not achieved resulting in growth of the material in this area to be random. An evidence of this can be seen in Figure 1 which shows a polished and etched cross-section of boule 67-C. It can be seen that good growth was achieved above the seed all the way to the top surface. In run 68-C melt-back of the seed was only around the top circumference of the seed, hence growth was in about a 45^{o} direction (Figure 2). To avoid cooling the bottom of the melt by direct radiation to the heat exchanger, insulation of graphite felt and tube was put around the exposed heat exchanger tube. This caused the extraction of heat from the sides to be via the seed to the heat exchanger resulting in lateral growth as well as at the top of the seed. Figure 3 shows sections of boules 72-C, 74-C, 82-C and 85-C where lateral growth has been achieved. In run 83-C (Figure 4) essentially all the material solidified as a single crystal. The orientation of the grown material and the seed was checked by back-reflection Laue and found to be the same. As an example of this, areas marked 1, 2 and 3 for run 74-C (Figure 3) were x-rayed. Position #3 is across from the crack but of the same orientation as the seed, emphasizing that cracking of the ingot occurred during

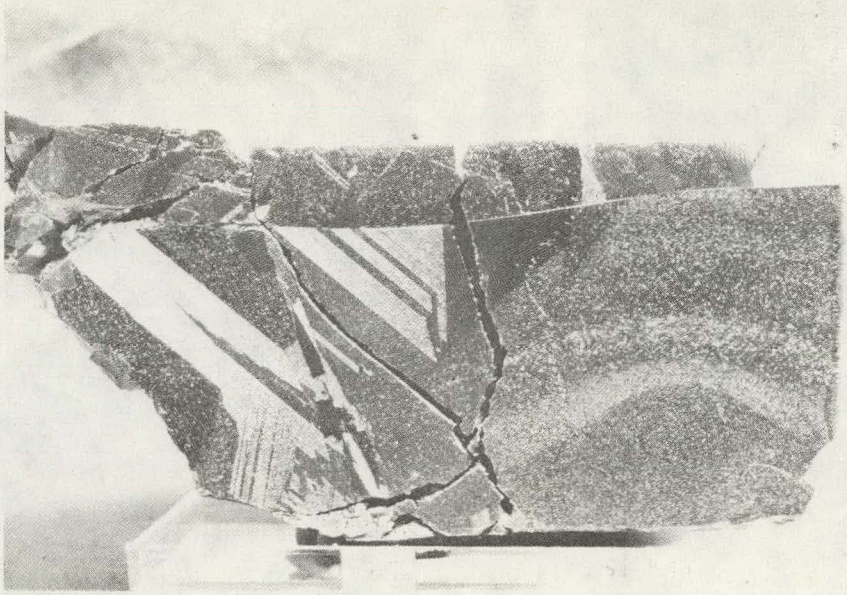

Figure 1. Polished and etched cross-section of boule 67-C.

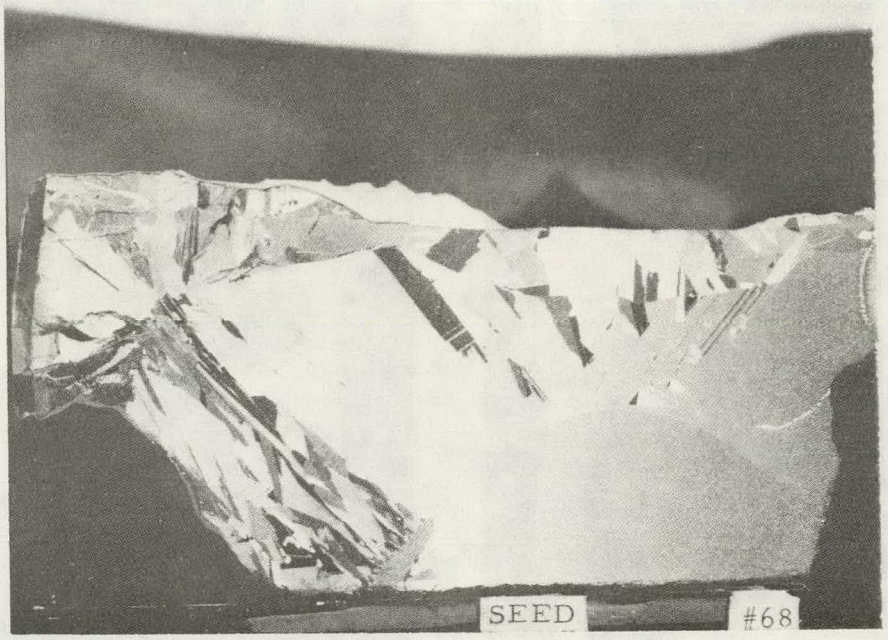

Figure 2. Polished and etched cross-section of boule 68-C.

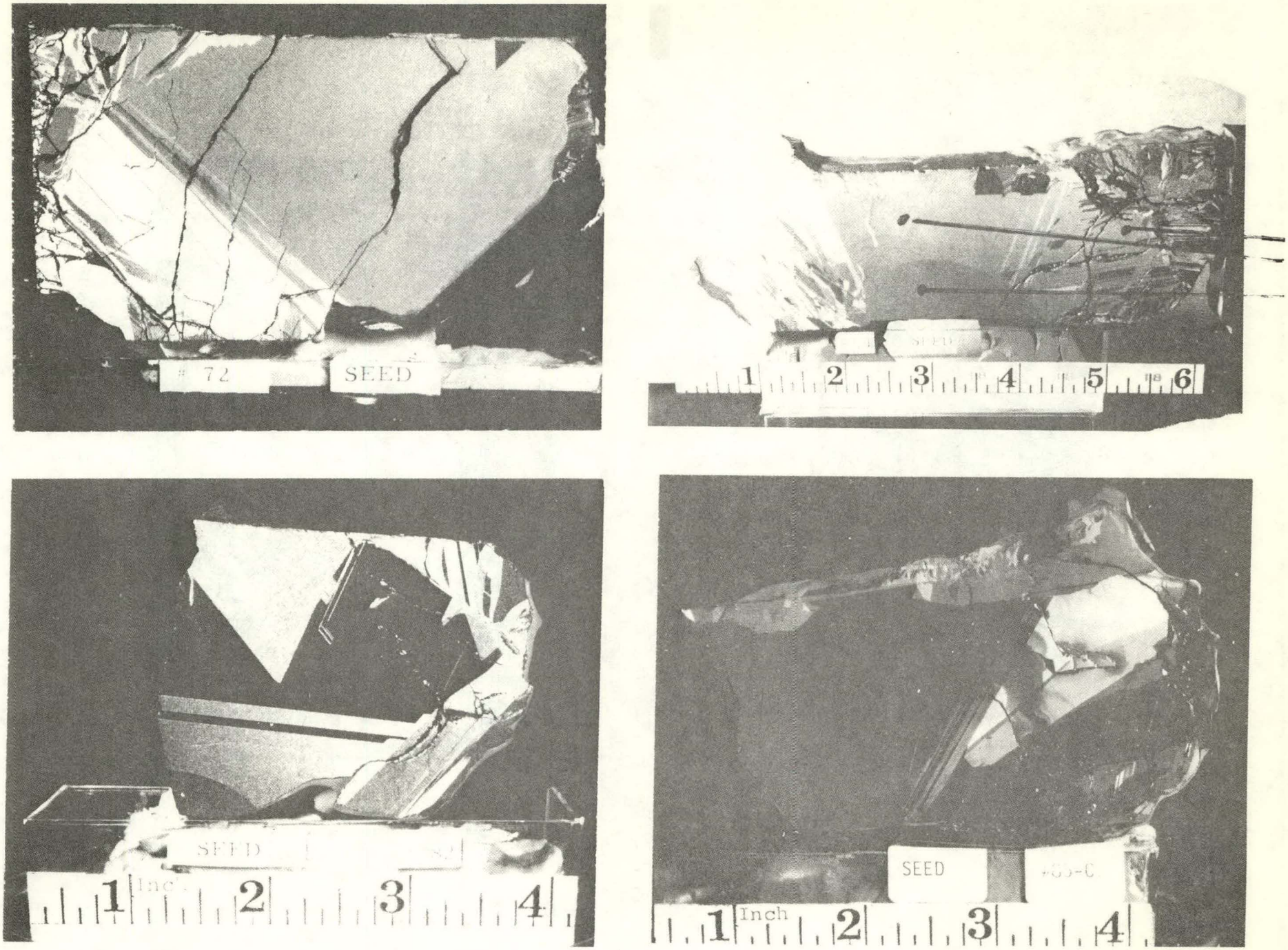

Figure 3. Polished and etched sections showing lateral growth.

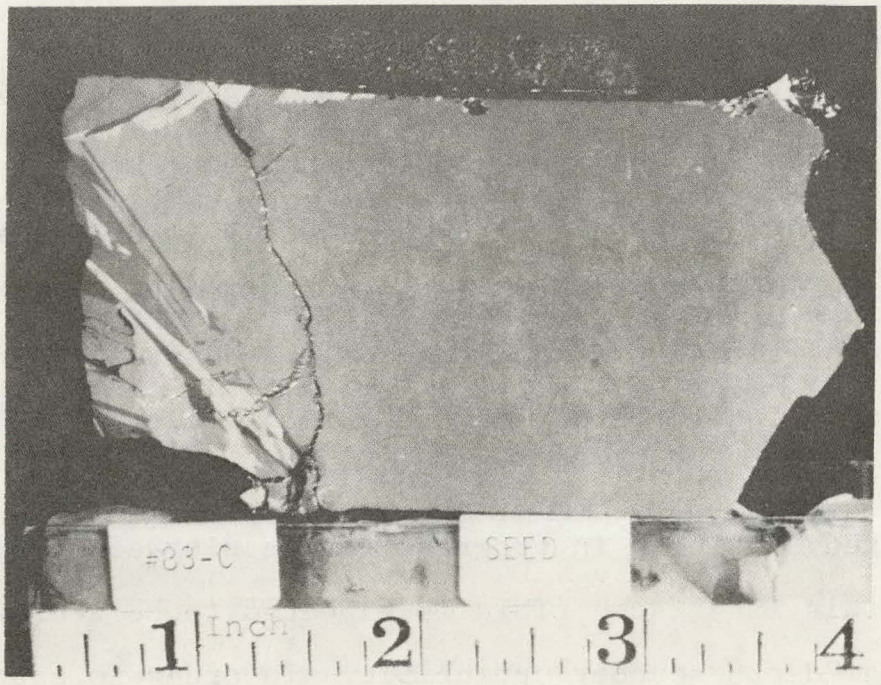

Figure 4. A polished and etched section of run 83-C.

the cool down cycle after solidification.

So far the viewport at the top of the furnace gave a limited view of the solidifying ingot. The completion of the growth cycle was monitored by observing a faster drop of the output of a radiation pyrometer focussed at the lower part of the ingot. This indicated that the contribution of heat of solidification was negligible. At times this has resulted in dropping the power when a small amount of liquid was still present; this formed a polycrystalline layer on the top outside edge of the ingot. It has been felt that a knowledge of the position of the solid/liquid interface during the growth cycle is necessary.

A study has been in progress to use ultrasonics to monitor this interface. This is expected to be achieved using the "depth sounder" principle. In order to adapt the ultrasonic sensors the top port of the furnace chamber had to be modified. Along with this modification a bigger viewport was incorporated to get a better insight during crystal growth. Run 87-C was made with these changes but without the ultrasonics in place in order to study the change in heat flow characteristics due to a bigger sight port. It was found that heat loss through the control port resulted in seed melt-back only on the sides. Single crystal growth was, therefore, observed only in areas of

seed melt-back. Growth of silicon in the Heat Exchanger Method is achieved by keeping the furnace temperature constant and decreasing the heat exchanger temperature. The bigger sight port helped to see that there was still some liquid present when the heat exchanger had reached a minimum temperature based upon its capacity. Under such steady state conditions the furnace temperature had to be lowered to achieve complete solidification.

It has been felt that the limiting factor in obtaining fast growth rates is the extraction of heat from the solidified ingot. This is dependent on the thermal conductivity of silicon and the clear vitreous silica crucible. The thermal conductivity of vitreous silica drops almost exponentially with decrease in temperature[2] around the "seeding" temperatures. Thus the heat extraction is progressively resisted by the crucible during the growth cycle. The efficiency of heat extraction can be increased by either increasing the capacity of the heat exchanger or by changing the crucible medium. In run 75-C a hole was made in the crucible bottom which was plugged by a $1^1/_8$" diameter silicon carbide coated graphite piece. This would improve the heat extraction and the heat flow will be through the seed resulting in better crystal growth characteristics. In run 75-C the furnace temperature was high which melted the seed but there was no

flow of liquid silicon around the plug. A similar experiment was designed for run 88-C. During this melt molten silicon penetrated around the plug due to seed loss just before the start of the growth cycle and the run had to be aborted.

During run 89-C the ultrasonics were set up to measure the velocity of sound in liquid silicon to aid in the determination of the solid-liquid interface. The summary of the principal results of the ultrasonic tests are as follows:

(a) Longitudinal wave sound speed in molten silicon $\approx$ 750 m/s.

(b) Longitudinal wave sound speed in fused silica at 1400°C $\approx$ 5000 m/s.

(c) Heat transfer up the translucent rods of fused silica, of ½" diameter, is not excessive. No cooling is necessary at the transducer location above the furnace.

(d) Attenuation of longitudinal and shear waves at 2 MHz was excessive, but attenuation of longitudinal waves at 1 MHz was relatively small. These statements apply to translucent fused silica rods at thermal equilibrium with one end in or near the melt, with an estimated length of 2 ft at or below 1000°C, and 1 ft between 1000 and 1400°C.

(e) Longitudinal wave sound speed in solid silicon at room temperature = 9070 m/s. Poisson's ratio $\tilde{\sim}$ 0.15.

(f) The calculated energy reflection coefficient at the seed/liquid interface at 1412°C is 62%, for longitudinal waves at normal incidence.

(g) The use of ultrasonic frequencies below 1 MHz, possibly as low as 0.1 MHz, appears desirable with respect to attenuation in translucent fused silica. If this low a frequency could be effectively combined with rods of smaller cross section, such that extensional waves could be guided and propagated without dispersion, the interface monitoring system would be relatively immune to the details of rod end geometry and to the seed interface curvature.

(h) The quartz rods used for the ultrasonic sensors were found to have expanded in diameter near the end which was near/in the hot zone. At the cold end a deposit, presumably SiO, was seen.

Another variable that was incorporated in run 89-C was to increase the superheat of the melt. In this way when the heat exchanger reaches a limiting temperature, growth of single crystal can be achieved by lowering the furnace temperature.

In an effort to obtain crack-free ingots, thin-walled

clear silica crucibles were used. Under such conditions the crucible is expected to be weak enough to shatter before it builds enough stresses to crack the silicon. During run 91-C the crucible failed during the growth cycle resulting in loss of the melt. The solidified portion illustrating the shape of the solid-liquid interface is shown in Figure 5. A similar experiment was carried out during run 93-C when a crack-free silicon boule was obtained (Figure 6).

Solar cells of 2cm x 2cm nominal size were fabricated at Optical Coating Laboratory, Inc., from the material cast in run 26-C, 72-C, and 75-C. The cells from the latter two runs were processed simultaneously. Two control samples were processed; one with the test samples while the other in production. The testing was carried out under Air Mass Zero (AMO) conditions and cell temperatures of 28°C. The results for run 26-C are shown in Figure 7 while those from the other runs in Table II. In column 5 and 6 the readings are shown under a tungsten and xenon light source respectively. In the last two columns the exact length and width of the cells are tabulated. The V_{oc} values for 26-C samples are rather low which is attributed to the high resistivity of about 50 Ω-cm as against the value of 2 Ω-cm for the control. In comparison, the 72-C and 75-C samples showed higher V_{oc} but lower I_{sc}. The drop in I_{sc}

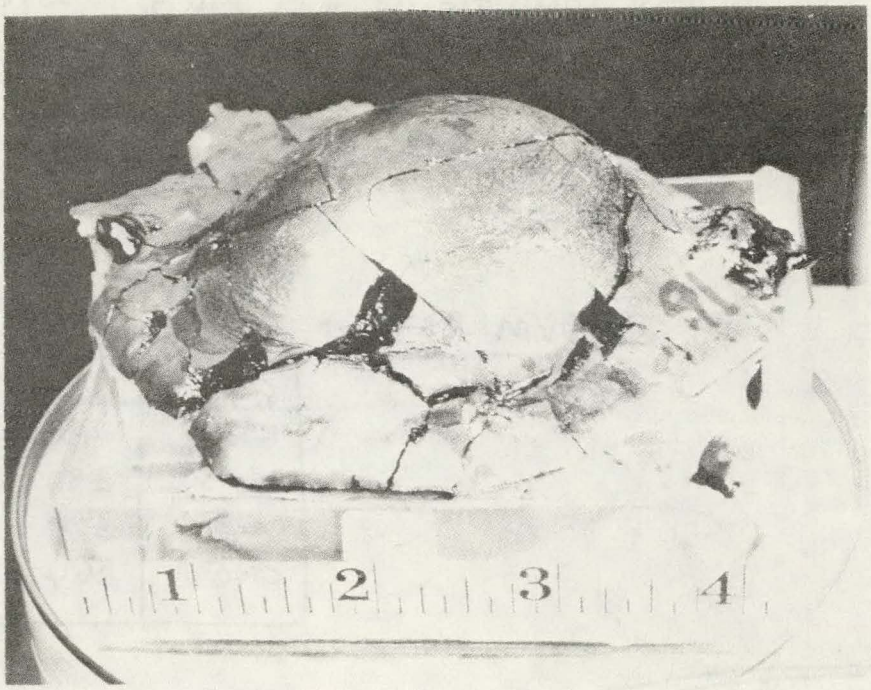

Figure 5. The shape of the solidifying solid/liquid interface.

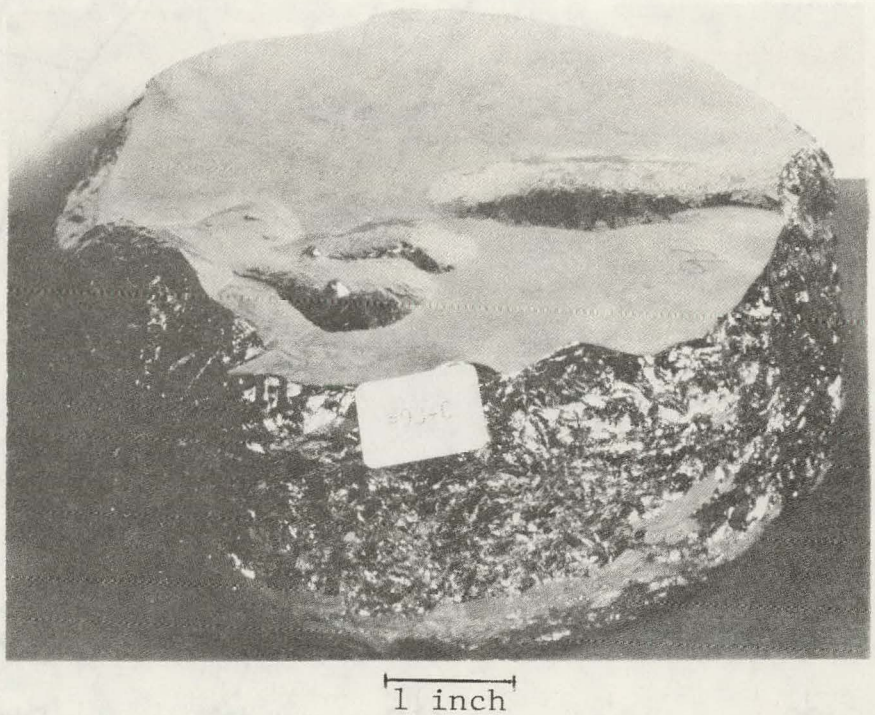

Figure 6. Crack-free silicon boule obtained in run 93-C.

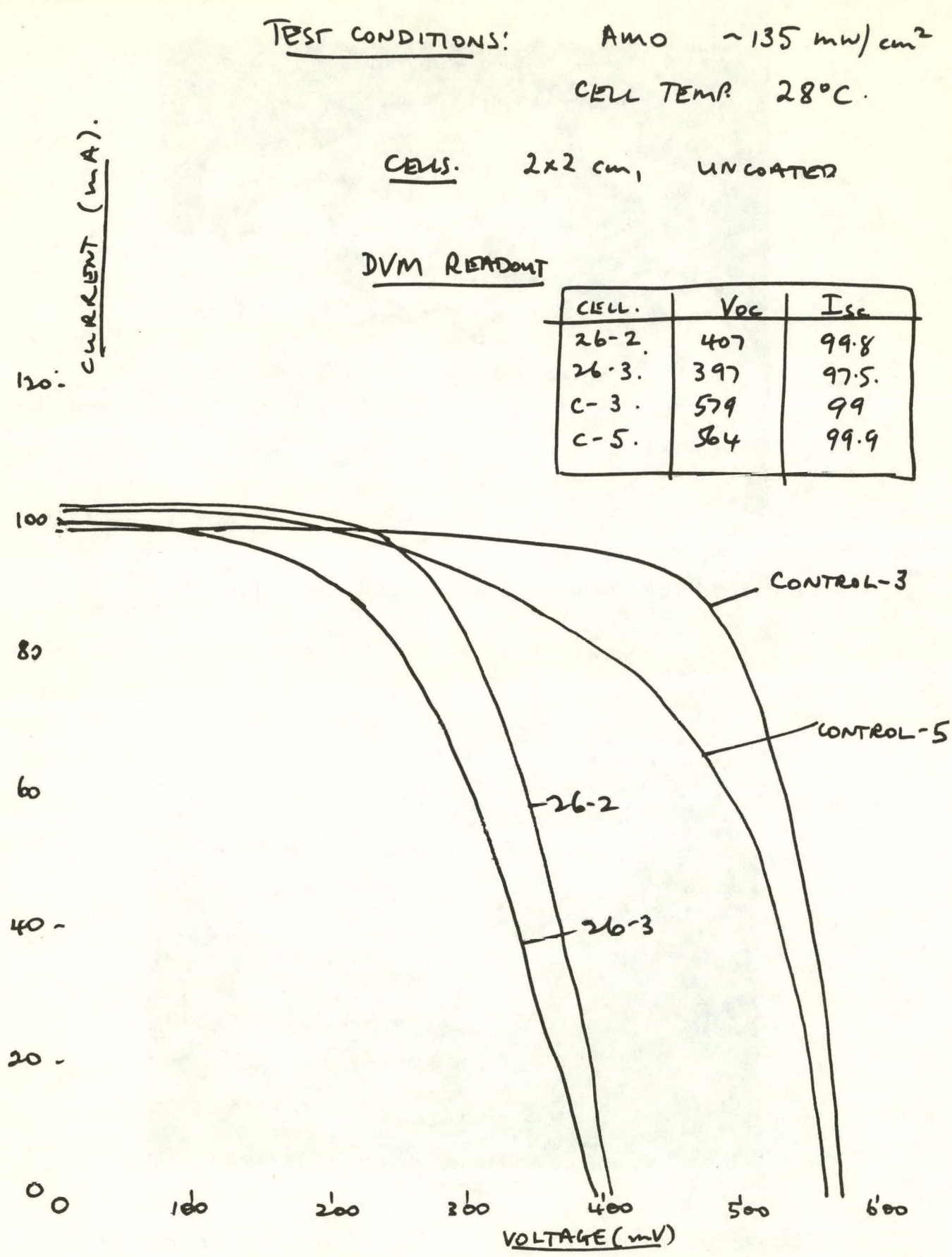

Figure 7. I-V Curves and Digital Readout for V_{oc} and I_{sc} for Solar Cells Made from Boule 26-C and Control Samples

TABLE II.

I-V PARAMETERS (AM0)

(DVM Readings)

INGOT	#	Voc (mV)	Isc (mA)	Tu (mA)	Xe (mA)	I_{400} (mA)	I_{450} (mA)	CFF	L Mils	W Mils
72C	1*	534	73.6	34.1	37.5	66.1	58.4	.67	780	779
	2*	537	73.6	34.1	37.8	67.5	60.8	.69	779	780
	3	525	72.5	34.1	36.5	54.2	44.8	.57	781	783
	4	538	75.3	35.9	37.5	63.4	53.8	.63	782	780
75C	1*	524	70.6	32.1	36.5	56.5	45.2	.61	774	780
	2*	529	68.0	31.2	35.1	61.3	53.1	.68	780	775
	3	536	67.4	31.0	34.3	63.3	53.6	.70	776	783
	4	536	66.7	30.7	34.2	63.1	54.4	.70	774	783
Control	1Ø	579	105.8	63.2	43.1	101.8	98.1	.72	787	787
	2+	579	100.8	61.0	40.0	99.0	96.2	.74	786	786

* KOH etch
Ø Processed with samples
+ Diffused in production

can be associated partly with a build-up of impurities in the crystal growth furnace which are being incorporated in the boule. Estimates of efficiency on equal-area basis and assuming AR coating show that the test cells are about 6.4 - 6.9% (AMO), i.e., over 7% AM1.

Crucible Development

A number of alternatives to the clear silica crucibles using fused silica are being studied to prevent cracking of the silicon ingot after solidification. This has involved the use of commercially obtained crucibles as well as liners and coatings developed at Crystal Systems. This work has been carried out on smaller, experimental ingots and crack-free silicon has been obtained in a number of cases.

A slip-cast Candle Quartz crucible containing a colloidal silica binder was fired at 800°C during run 73-C. This crucible was loaded with 50 gms of silicon and heated to 10°C above the melting point of silicon (run 76-C). It showed evidence of shrinkage due to sintering at the high temperature resulting in cracking. Based upon related experience it is felt that the binder containing compositions undergo large shrinkages during sintering, thereby cracking.

A high purity fused silica liner obtained from a

commercial source was used for casting silicon in run 76-C. On cooling the silicon did not show any evidence of cracking although the liner itself did crack in places, as illustrated in Figure 8. A similar crack-free silicon was cast in a regular grade crucible during run 78-C. During these runs directional solidification was not carried out. In run 84-C a high-purity fused silica crucible was tested to obtain directionally-solidified, seeded growth. It was found that the seed was still apparent and not bonded to the crucible, whereas the grown material was bonded and thus shattered during solidification.

A hot-pressed fused silica crucible was tested for solidification of silicon. This showed no cracking in the silicon although cracks did develop in the crucible (Figure 9). However, on directional solidification in a similar crucible during run 90-C the cast silicon was in pieces. This crucible was not supported in a graphite retainer.

In run 86-C fused silica coatings on clear vitreous silica crucibles were tested. One crucible coated with 3μ particle size fused silica showed no evidence of cracking of the silicon even though it was directionally cooled. Another coated crucible which was not placed on the heat exchanger also produced crack-free silicon.

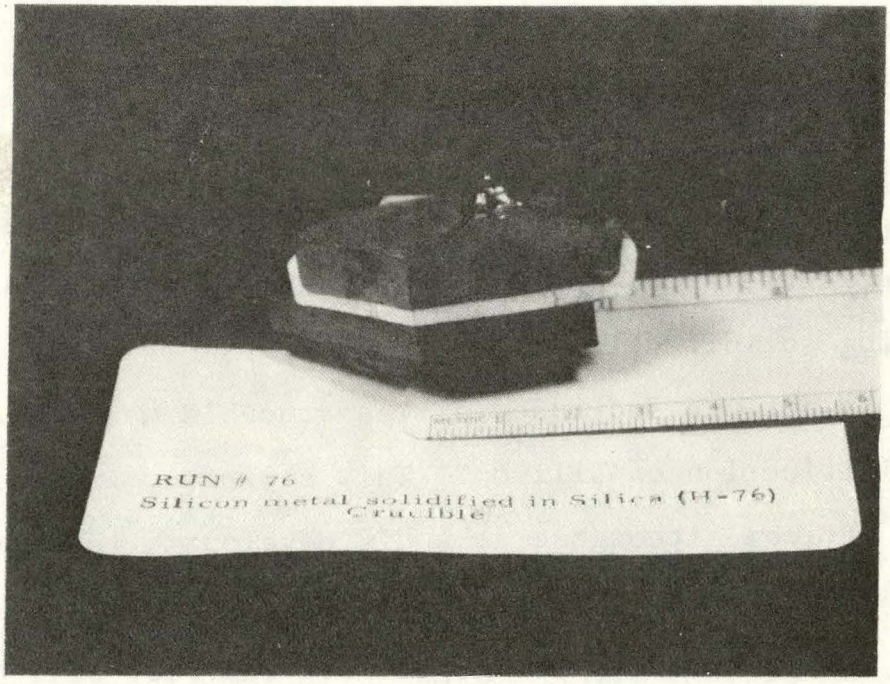

Figure 8. Cross-section through crack-free silicon solidified in high purity silica crucible during run 76-C.

Figure 9 . . A section of the crack-free silicon cast in a hot-pressed curcible.

These two samples are shown in Figure 10. It was generally found that the coatings had "crazed."

During run 90-C two pressure-cast fused silica crucibles developed at Crystal Systems, Inc., were loaded with silicon. These crucibles were made using slips containing 8μ and 25μ particle size silica. The former was free standing while the latter was supported in a graphite retainer. No directional solidification was carried out. Both crucibles produced crack-free silicon as shown in Figure 11.

The main problem in obtaining crack-free silicon ingots is that a tenacious bond is formed at the silicon/silica interface on solidification. On cooling silicon is in tension and silica is in compression, resulting in cracking of the crucible as well as the ingot. This problem can be overcome if the crucible is weak and thereby cracks itself during the cool down cycle before cracking the ingot. However, during the growth cycle the silica is sintered to a high density which makes it less susceptible to failure. The use of low density crucibles has the associated problem of penetration of molten silicon, as experienced in run 92-C. It was felt that if a fused silica crucible can be glazed on the inside it will make it resistant to penetration. The thin layer of the glaze is expected to have low strength and fracture

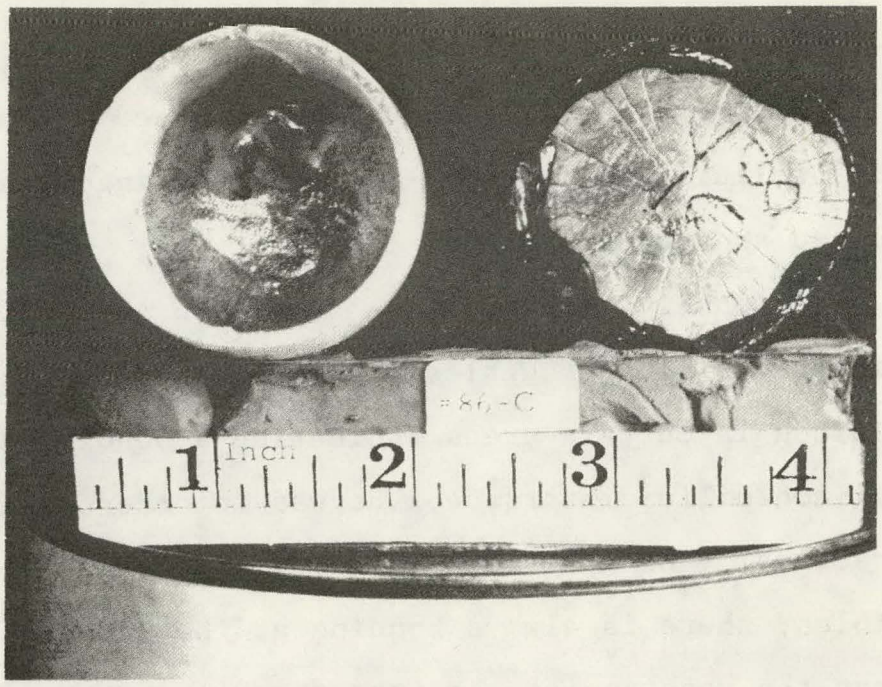

Figure 10. Crack-free silicon cast in coated crucibles. The material on right was directionally solidified.

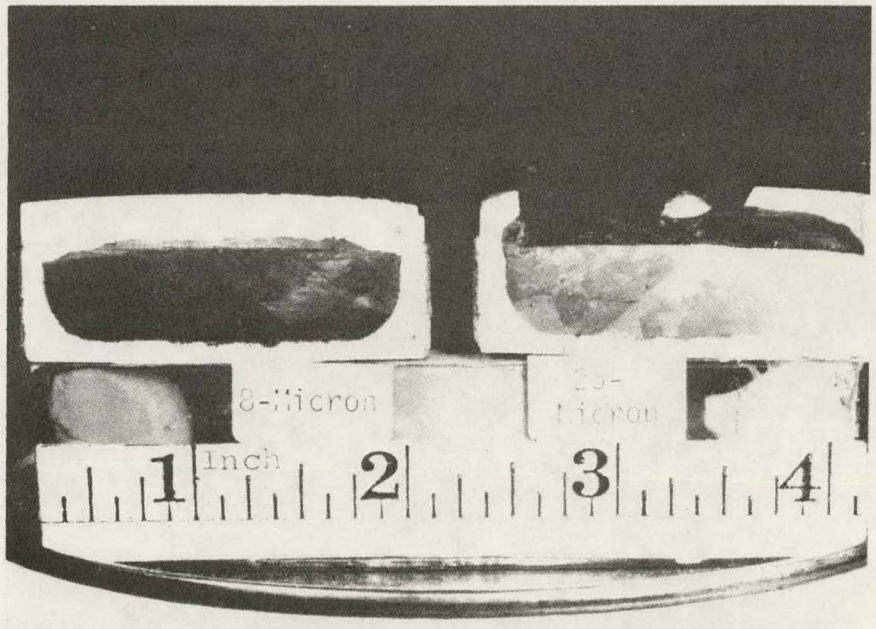

Figure 11.. Crack-free silicon cast in pressure cast crucibles. (Run 90-C)

during the cool-down cycle. During run 94-C a glazed, slip-cast, fused silica crucible was used and directionally solidified. A crack-free silicon ingot was obtained as shown in Figure 12.

An observation which is common to all forms of sintered silica crucibles is that it shows attachment to silicon in certain areas indicating a bonding at the silicon/silica interface, whereas in other areas there is practically no adherence. In cases of clear silica crucibles, there is always bonding at the interface. It seems that the reaction at the interface is quite complex and is a function of the density, thickness, purity, particle size, and surface roughness of the crucible. Work has been initiated in order to understand this reaction.

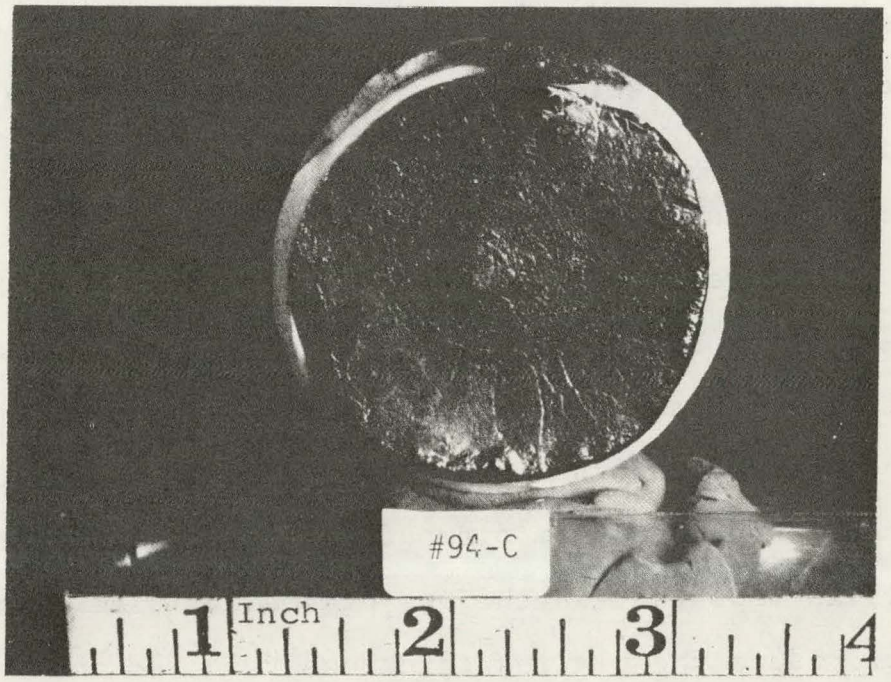

Figure 12. Crack-free silicon cast in a glazed crucible

CRYSTAL SLICING

Silicon slicing efforts were directed towards increasing the number of wafers sliced per unit length. Experiments were performed using different types of plated and impregnated wires. Characterization of the wafer and wire were carried out in conjunction with the experimental slicing. A silicon slicing summary is presented in Table III.

Life tests were carried out during runs 45-S through 47-S on the 5mil tungsten wire plated with 45μm diamonds. This is the same plated wire used in runs 18-S through 36-S. Good quality wafers were sliced. During run 37-S it was established that 20μm diamond will cut satisfactorily. This is important as the use of smaller size diamonds will reduce the plating thickness required and thereby cut down on the kerf losses.

Runs 38-S and 39-S were conducted using optimized slicing conditions used for plated wire. It was found that the diamond impregnated wires failed due to diamond pull out as evidenced in Figure 13 for a used section of wire. This problem was further enhanced for a 5mil, 0.125mm diameter wire used in run 42-S (Figure 14).

In run 41-S an 8mil, 0.2mm wire impregnated with 45μm diamond was spaced at 15mil, 0.375mm intervals to

TABLE III.

SILICON SLICING SUMMARY

RUN	PURPOSE	FEED FORCE/BLADE lb.	gm	AVERAGE CUTTING RATE mil/min	mm/min	WIRE TYPE	REMARKS
37-S	Life test .125 Ø impregnated wire	088	40	N/A	N/A	Single impregnated 20μm diamond in copper plated wire .125 mm diameter	Good cutting rates were achieved at low feed force indicating that 20μm will cut well if concentration can be maintained. No hard data due to excess wire breakage.
38-S	Life test of .2mm Ø diamond impregnated wire	.15	68	2.1	.053	Single impregnated 45μm diamond in copper plated wire .2mm diameter	Good wafer quality.
39-S	Continuation of life test from run 38-C	.15	68			Same wire.	Aborted due to low cutting rate and wire breakage.
40-S	Close .375mm, 15 mil spacing to produce 66 wafers per inch	.1	45			Single impregnated 20μm diamond in copper-plated wire .125mm diameter	Aborted due to loss of wafer and problems in support system.
41-S	Demonstrate close spacing .015 inch, .375mm spacing to produce 66 wafers per inch	.08		.6	.015	Single impregnated 45μm dimaond in copper-plated wire .2mm diameter	Very low forces and low cutting rates used to slice 66 wafers, average wafer thickness 5 mil - .125mm.
42-S	Close spacing same as run 41-S	N/A		N/A		Same wire.	Good data not available due to loss of wires. Cut completed in approx. 30 hrs., approx. 50% good 5 mil thin wafers.

TABLE III. (Cont.)

SILICON SLICING SUMMARY

RUN	PURPOSE	FEED FORCE/BLADE lb	gm	AVERAGE CUTTING RATE mil/min	mm/min	WIRE TYPE	REMARKS
43-S	Test CSI impregnated wire			N/A		0.005 copper coated stainless steel impregnated with #15 diamond (12-22μ)	Run aborted after 0.600" due to poor cutting rates.
44-S	Test CSI impregnated wire			N/A		0.008 copper coated stainless steel 45μm diamond	Run aborted due to poor cutting rates.
45-S	Life test	0.15	67.5	1.13	0.03	0.008 nickel diamond plated wire used in runs 18-36	Good wafer quality.
46-S	Life test	0.15	67.5	1.04	0.025	0.008 nickel diamond plated wire used in runs 18-36	Good wafer quality. Poor support rollers.
47-S	Life test	0.2	90.0	2.5	0.06	0.008 nickel diamond plated wire used in runs 18-36	0.2 lb. required for good cutting rates.
48-S	Test new plated wire	N/A		N/A		5mil, 0.125mm stainless steel core, 45μm diamond nickel plated. 8.5mil total kerf.	Run aborted due to wire wander and wafer breakage.
49-S	Test plated wire	N/A		N/A		Same as 48-S	Failure due to wire breakage because of embrittlement during plating.
50-S	Test CSI impregnated wire	N/A		N/A		8mil, 0.2mm copper coated stainless steel wire.	Run aborted due to poor cutting rates in the middle of blade pack. Wires have uneven tension.

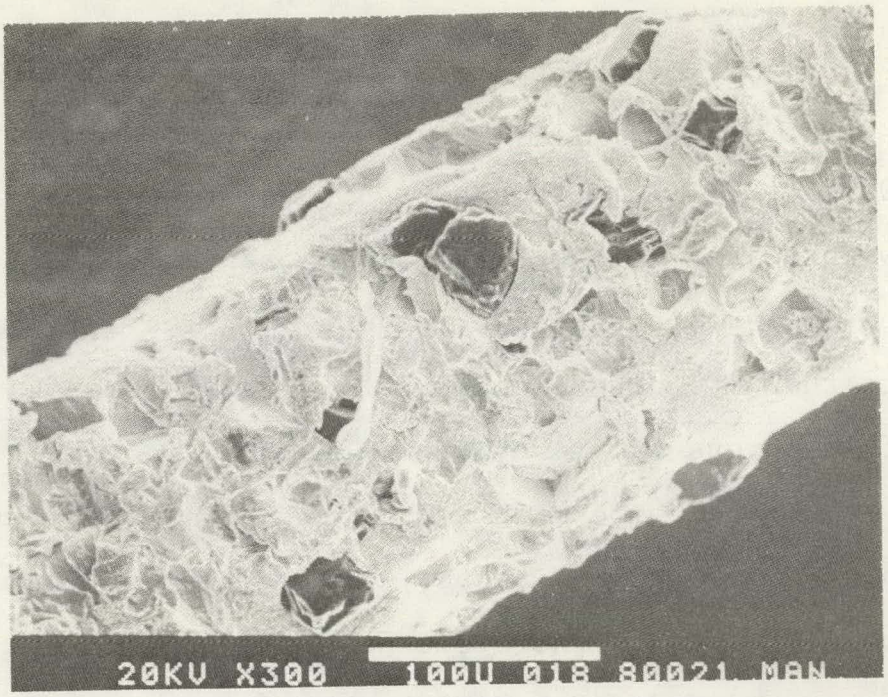

Figure 13. A section of diamond impregnated wire.

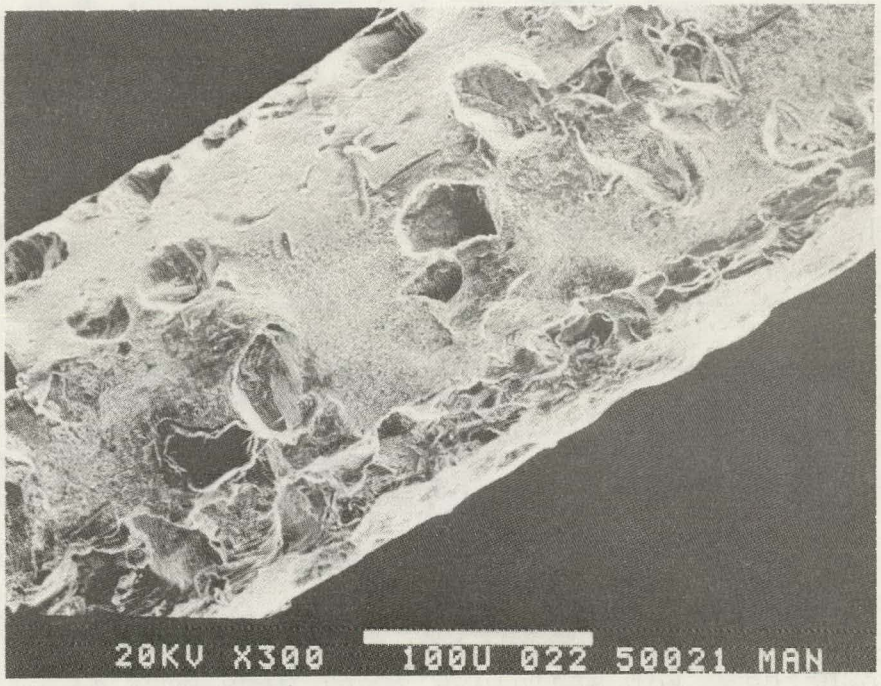

Figure 14. A section of diamond impregnated wire used in run 42-S showing diamond pull-out.

produce 64 wafers per inch. None of the wires came together during the run. The wafers ranged from 5 to 7mils, 0.125 to 0.175mm thickness. Wafer roughness was measured with a Rank Tallysurf Profiliometer in the areas outlined on the tracing shown in Figure 15. The surfaces parallel to the wire direction 3 and 4 were smoother than the profile across from the cut 1, 2 and 5.

Plated wire with a 0.075mm tungsten core was nickel plated with 20μm diamonds. This wire was found to break when mounted in the blade head. An examination of the fractured ends (Figure 16) showed that the wires had been stretched and the center core had necked down and the nickel coating was separated from the core. Further, an examination of the longitudinal section of the wire in Figure 17 revealed that very low concentration of diamond particles were encapsulated in the nickel plating.

Thus the use of 0.075mm diameter core wire to cut the kerf losses produces blade wander and low strengths. There are also problems with achieving uniform coatings and high diamond concentrations. It was, therefore, decided to use 0.125mm core wire and reduce kerf losses by using smaller diamond sizes and thinner platings.

One of the main problems with plated wires has been the variation in diamond concentration obtained from batch to batch. For stainless steel wire embrittlement has been

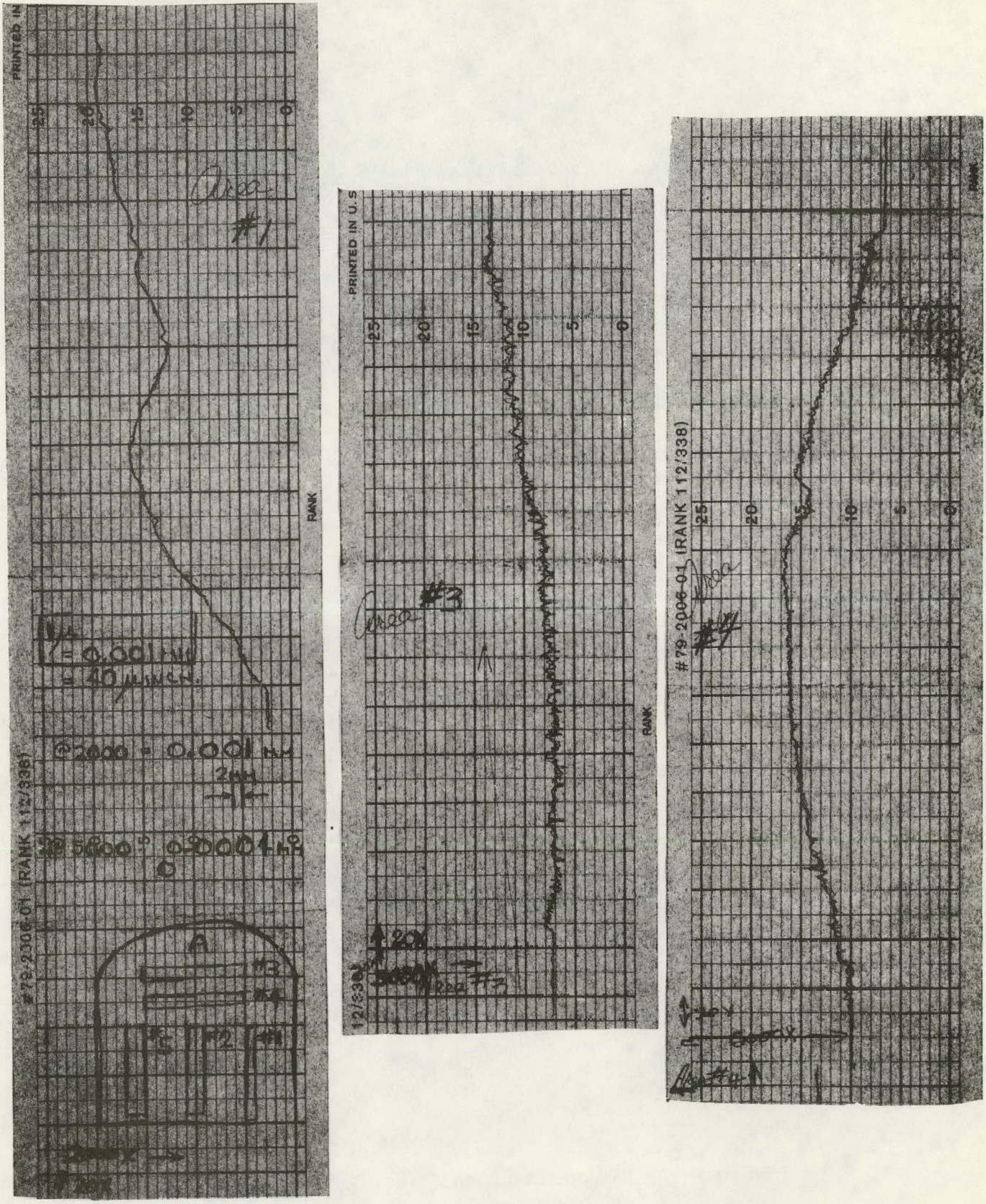

Figure 15. Profiliometer tracing of 0.0055 silicon wafer 41-S

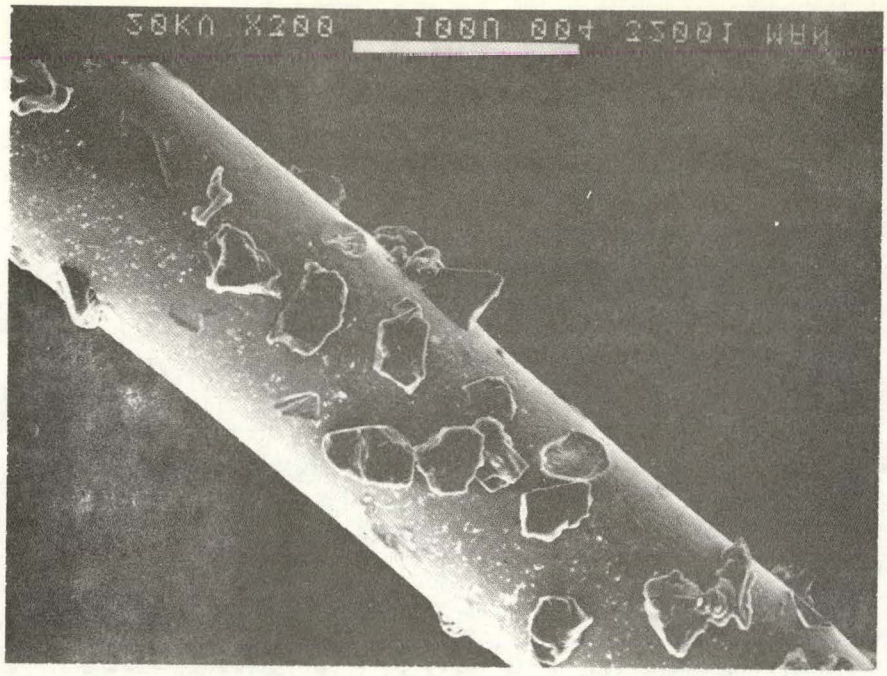

Figure 16. Fractured surface of 0.075mm tungsten core, nickel plated wire.

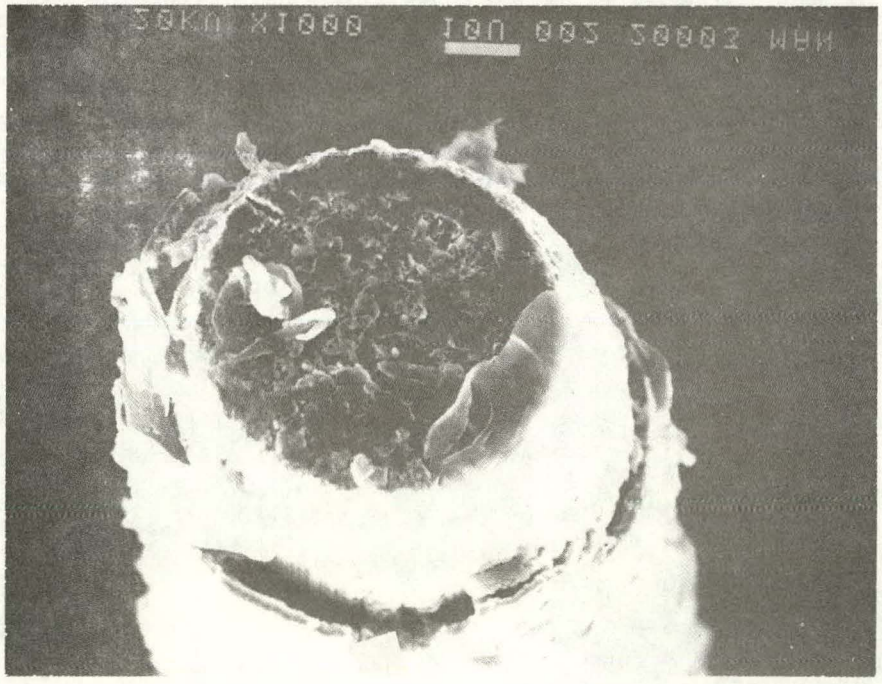

Figure 17. Longitudinal section of wire in Figure 16.

experienced after plating causing run 48-S and 49-S to be aborted. A number of sources are presently being pursued to obtain repeatable diamond plating on a 5mil, 0.125mm core. Meanwhile impregnation of 25μm diamonds into copper plated 0.125mm stainless steel wire has been carried out at Crystal Systems. A SEM examination of this wire (Figure 18) shows good diamond concentration was achieved. A higher magnification picture (Figure 19) indicates that the impregnated diamonds are held well by the coating. The Crystal Systems impregnated wire was tested in runs 43-S and 44-S. As in the case of other impregnated wires, the failure was because of diamond pullout.

It has been established that diamond impregnation can be carried out to high concentrations in house. Impregnation is conducted after the blade pack is set up and the slicing of wafers is by the cutting action of only half the circumference of the wire. The impregnation can be adjusted so that the diamonds are only impregnated on the cutting surface with minimum impregnation on the sides to reduce kerf loss. The wire suffers failure due to diamond pull out. However, this may be avoided if it is plated after impregnation. Efforts are on hand to plate this wire with nickel by electroplating and an electroless process. It is expected this wire will be suitable blade for reliable crystal slicing.

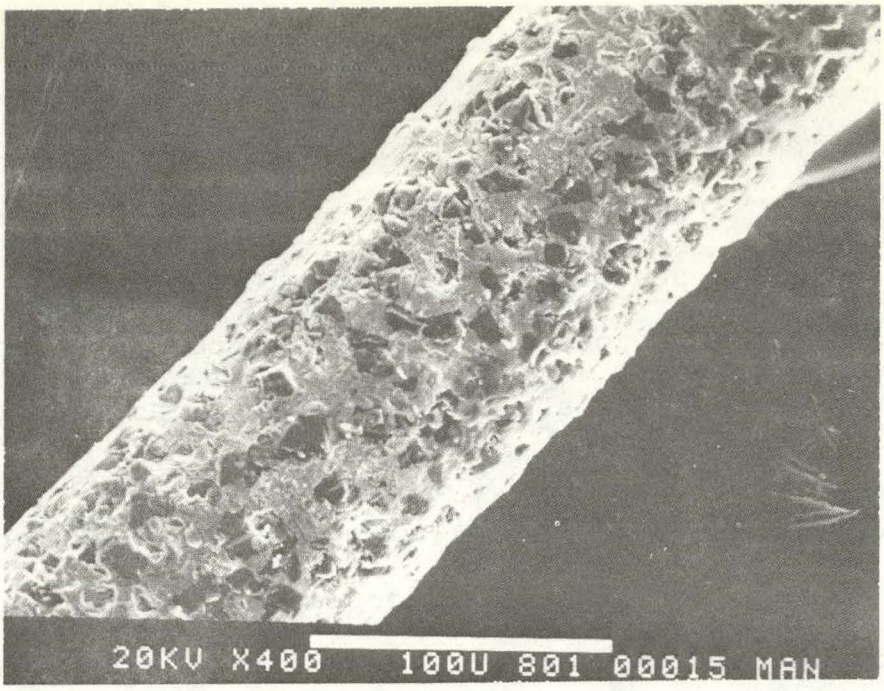

Figure 18. Longitudinal section of a Crystal Systems diamond impregnated wire.

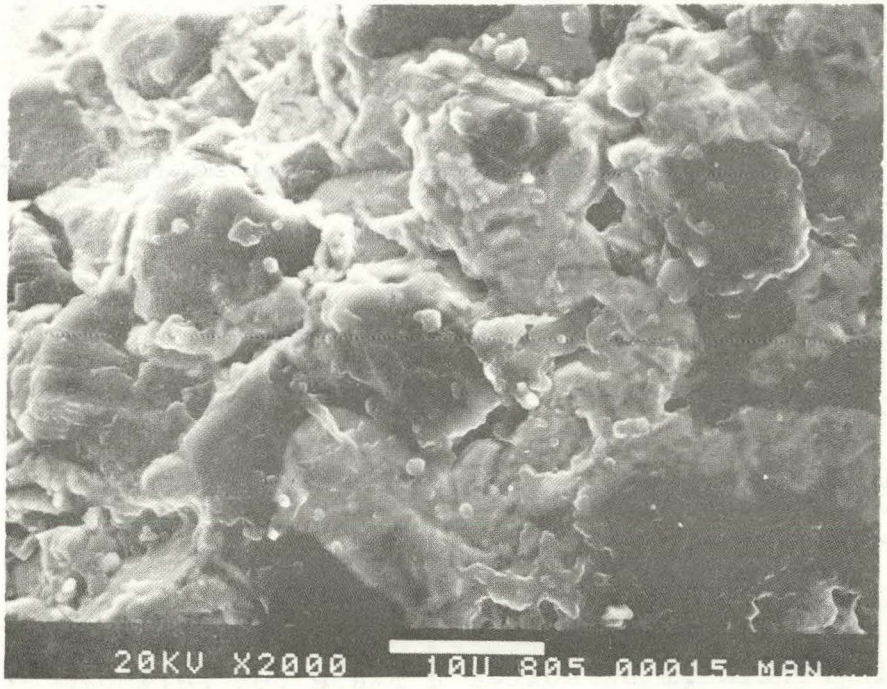

Figure 19. A magnified portion of wire in Figure 18.

Cost of Expendable Material

A cost analysis of diamond and wire for wire slicing was made to determine the cost of expendable materials per square meter of silicon material sliced. Two cases were analyzed--5 and 3 mil core material producing 9 and 5 mil kerf respectively.

Data: 100 Concentration of diamond = 72 carats/in^3 of bond (NBS Standard)

Cost of diamond = \$2.25/carat

Cost of 5 mil wire = \$40/lb or \$0.0035 per wire blade

Cost of 3 mil wire = \$45/lb or \$0.0014 per wire blade

Assume 8" stroke of machine, 9" abrasive length

Case 1: 5 mil, 0.125 mm wire core

1.5 mil, 37.5 μm plating thickness

2 mil, 50 μm diamond

$$\text{Volume of bond per wire} = \frac{\pi}{4}(D_2^2 - D_1^2)\,L$$

$$= \frac{\pi}{4}\{(8 \times 10^{-3})^2 - (5 \times 10^{-3})^2\}9$$

$$= 0.276 \times 10^{-3}\ in^3$$

$$\text{Carats of diamond per wire} = 72 \times 0.276 \times 10^{-3}$$

$$= 19.84 \times 10^{-3}$$

$$\text{Cost of diamond per wire} = 2.25 \times 19.84 \times 10^{-3}$$

Cost of diamond per wire = $ 0.0446

Cost of wire = 0.0035

Total cost of wire blade = $ 0.0481

Assume each wire cuts 4 sq meter of silicon,

Cost of expendable material per square meter of silicon slicing = $0.0120

Case 2: 3 mil, 0.075 mm wire core

0.75 mil, 19 μm plating thickness

1 mil, 25 μm diamond

$$\text{Volume of bond per wire} = \frac{\pi}{4}\{(4.5 \times 10^{-3})^2 - (3 \times 10^{-3})^2\}9$$

$$= 0.079 \times 10^{-3} \text{in}^3$$

$$\text{Carats of diamond per wire} = 72 \times 0.079 \times 10^{-3}$$

$$= 5.72 \times 10^{-3}$$

Cost of diamond per wire = $2.25 \times 5.72 \times 10^{-3}$ = $0.0129

Cost of wire = 0.0014

Total cost of wire blade = $0.0143

Assume each wire cuts 4 sq. meter of silicon,

Cost of expendable material per sq. meter of silicon slicing = $0.0036

It is apparent that the cost of expendable materials for wire slicing is very low, $0.012 and $0.0036 per square meter of silicon sliced for 9 and 5 mil kerf removal respectively. This assumes that four meters of silicon can be cut with each wire. Since the costs for expendable materials are so low, it is not necessary to have a long abrasive life. This can be reduced by an order of magnitude to .4 square meters and the costs will still only be $0.120 and $0.036 per square meter of silicon produced.

CONCLUSIONS

1. A 2.3kg crack-free boule has been cast using thin-wall, clear silica 15cm-diameter crucibles.

2. Crack-free silicon has been cast in fused silica sintered crucible liners and coatings in approximately 5cm-diameter experimental melts. This has been achieved with high purity and technical grade slip cast crucibles obtained from trade sources. Hot-pressed, low density slip cast, and pressure cast crucibles have also cast crack-free silicon. In these crucibles the silicon is not always tenaciously bonded to the silica as in the case of clear vitreous crucibles.

3. Control of thermal flow characteristics has resulted in significant progress towards crystal growth. Under proper conditions single crystal has been achieved above the seed all the way to the top surface and laterally to the crucible wall.

4. It was demonstrated that thin wafers 5 to 7mils, 0.125 to 0.175mm, thick would be sliced with a kerf loss of approximately 8mils, 0.2mm. Sixty-four wafers per inch were sliced.

5. A cost analysis for wire slicing with fixed diamond abrasive particles has shown that the cost of expendable

materials, diamond and wire, are low, $0.0120 and $0.0036 per square meter of silicon sliced for 9 and 5mil kerf removal respectively.

6. Under identical slicing conditions, commercial diamond impregnated wire has a much shorter life compared with diamond plated wire blades. The former suffers failure because of diamond pull-out.

7. Diamond impregnation of copper-coated wire can be carried out to high concentrations at Crystal Systems. This wire, after plating, is expected to be suitable for crystal slicing.

REFERENCES

1. F. Schmid, "Heat Exchanger-Ingot Casting/Slicing Process," ERDA/JPL 954373, Crystal Systems, Inc., Quarterly Technical Progress Report No. 6, March 1977.

2. R. W. Powell, C. Y. Ho and P. E. Liley, Thermal Conductivity of Selected Materials, NBS Reference Data Series, NBS-8, Category 5, November 1966.

SCHEDULE OF MILESTONES: INGOT CASTING

ITEM	DESCRIPTION
1	Modify instrumentation
2	
3	Develop crucible/coating com.
4	
5	Develop max. growth rate
6	
7	Program & Design Review
8	
9	Characterization
10	
11	Determine actual growth rate
12	
13	Growth rate function of h.e.
14	
15	Growth rate function of crucible
16	
17	Monitor interface position
18	
19	Growth rate function of orienta.
20	
21	Growth rate function of diameter
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23	Draft final report
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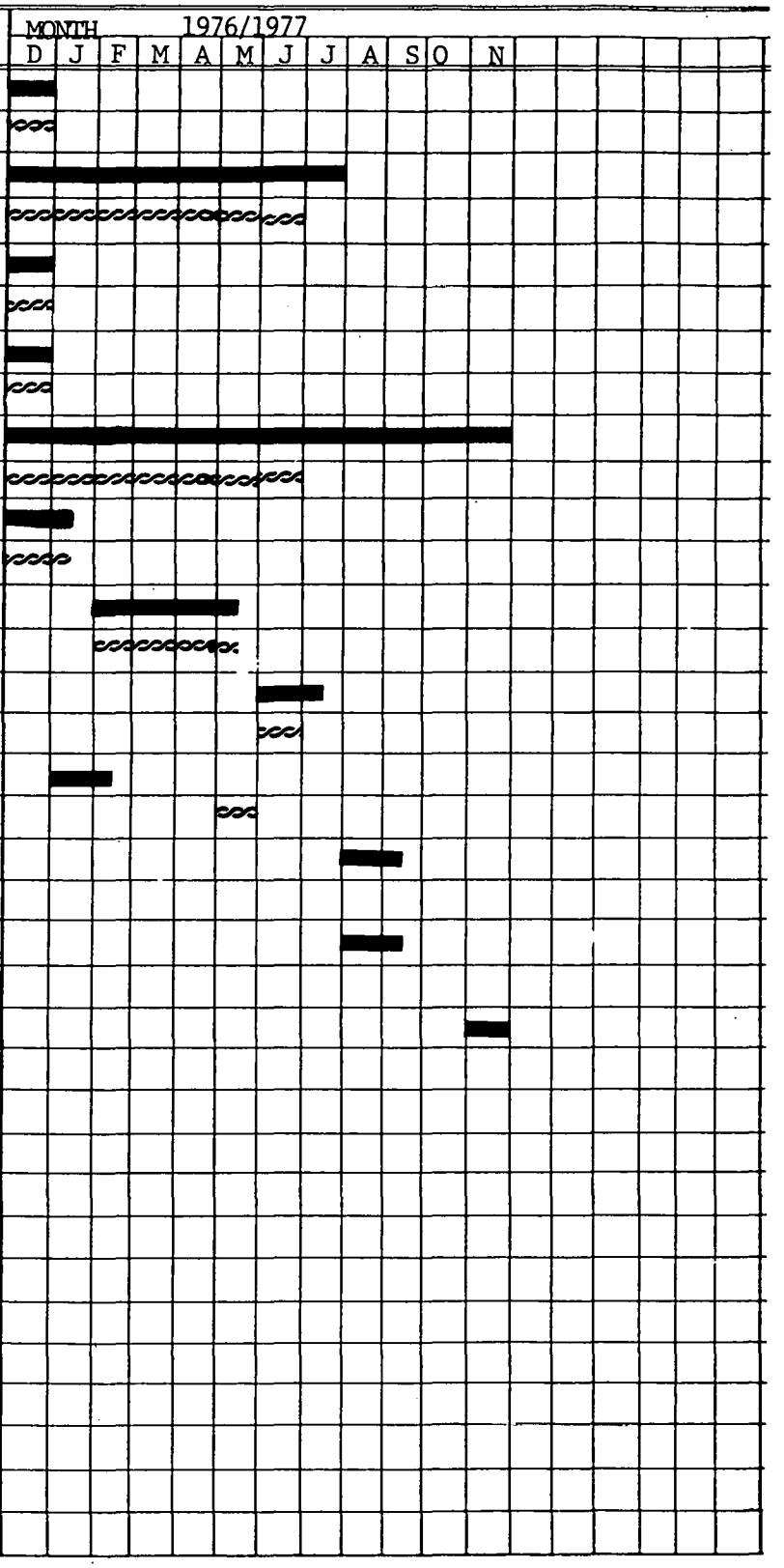

SCHEDULE OF MILESTONES: INGOT SLICING

ITEM	DESCRIPTION
1	Characterization
2	
3	Program/Design Review
4	
5	Modify Varian wire support
6	
7	Modify Varian coolant system
8	
9	Diamond-plated tool .005" Ø
10	
11	Diamond-impregnated tool .005" Ø
12	
13	Slicing test -- 4 cm cube
14	
15	Diamond-impregnated tool .003" Ø
16	
17	Diamond-plated tool .003" Ø
18	
19	Slicing tests - 10 cm kerf length
20	
21	Slicing tests - .015 cm wafers
22	
23	Slicing test - 10 cm x 15 cm
24	
25	Slicing test - .015 wafers
26	
27	Draft final report
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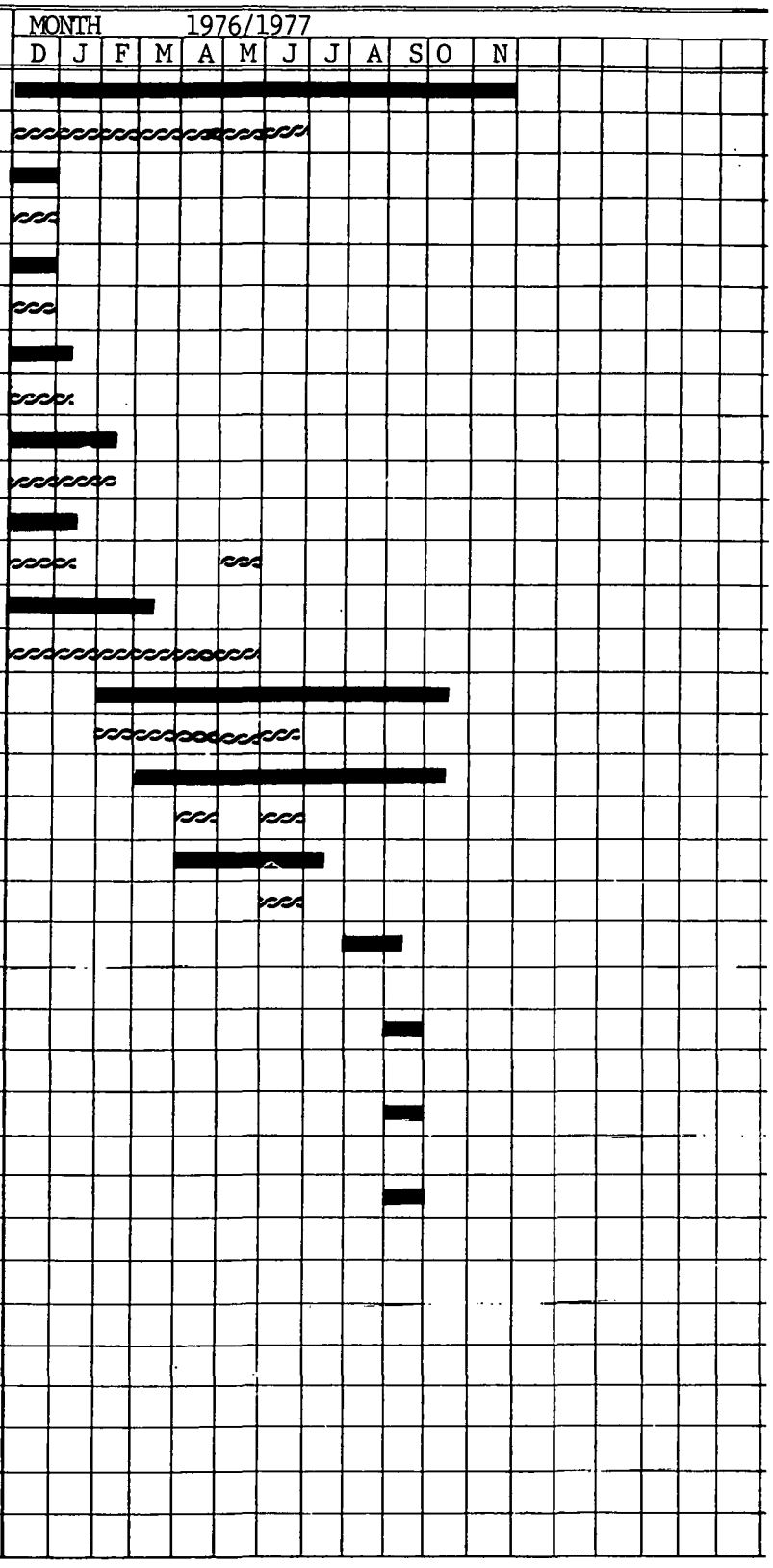

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