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HEAT EXCHANGER METHOD—INGOT CASTING FIXED ABRASIVE
METHOD—MULTI-WIRE SLICING. PHASE II

DRW-16

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

Quarterly Progress Report No. 1, November 21, 1977—December 31, 1977

By
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Chandra P. Khattak

January 6, 1978

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(PHASE II)

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ABSTRACT

A high degree of crystallinity has been achieved in ingots cast in this quarter. Since most of the growth took place near the solidification temperature, the top portion of the ingot was solidified by freezing from the surface. The thickness of this layer was reduced with the control of thermal flow characteristics in the furnace. The crucibles used in this study have a non-uniform bottom which is not conducive to proper heat transfer during solidification.

In an effort to achieve high packing density of solar cells in the module with maximum material utilization, an attempt was made to cast a square cross-section ingot. Even though some minor cracking occurred in the first ingot, it appears feasible to cast square cross-section ingots by the Heat Exchanger Method.

Higher feed forces results in higher cutting rates. However, this is accompanied by wire wander and increased surface damage depth. It has been established that the life of an impregnated blade can be prolonged by plating it after impregnation.

CRYSTAL CASTING

The first phase of this contract has been completed. In this phase, the proof of concept of silicon casting by the Heat Exchanger Method (HEM) has been established. The HEM is a directional solidification process. Such methods have been used to grow crystals of metals and alkali halides.¹ However, they are useful when there is contraction of the material upon solidification or it does not stick to the container walls. In the case of silicon a tenacious bond is formed² between silicon and silica and it expands on freezing. The mismatch of the thermal expansion coefficients^{3,4} of silicon and silica causes the ingot to go into tension and the crucible in compression. Above the ductile-brittle transition temperature,⁵ 650°C, silicon flows plastically to stress relieve itself, but below this temperature stresses are sufficient to crack the ingot. At Crystal Systems graded silica crucibles have been developed which fail during the cool-down cycle, thereby relieving stresses and casting a sound ingot. Boules as large as 3.3 kg have been cast using this approach.

The controlled growth, heat flow and cool-down has yielded silicon with a high degree of single crystallinity. In regions where growth instabilities occurred, very large grains were formed. Results⁶ of dislocation density measurements, minority

carrier diffusion length and lifetime data along with solar cell conversion efficiencies are very encouraging for photovoltaic applications.

In an effort to reduce impurities, silicon carbide formation has been identified with the use of graphite retainers in contact with silica crucibles. Higher growth rates have been achieved with the use of a graphite plug at the bottom of the silica crucible.

During the second phase of crystal casting emphasis will be placed on heat flow and other thermal parameters to increase growth rate and improve the silicon cast for solar cell applications. The present ingot technology approach produces circular cross-section ingots. However, the highest packing efficiency of solar cells in a panel is obtained with square or rectangular solar cells. For effective utilization of material and to reduce costs it is desirable to grow a square cross-section ingot. The possibility of growing square shaped ingots will be studied during the second phase of this contract.

Summary of Progress

During the Phase II portion of the quarter, emphasis was placed on the study of the heat flow on the crystallinity of the cast ingot. The experimental parameters of the various runs are shown in Table I. The etched and polished cross-sections of some of the ingots are shown in Figures 1-6. A study of these cross-sections shows that except for the formation of a grain, most of the central portion grows as a single crystal. On the sides, the single

Figure 1. Polished and Etched Cross-section of Run 2-001-C.

Figure 2. Polished and Etched Cross-section of Run 2-002-C

Figure 3. Polished and Etched Cross-section of Run 2-003-C

Figure 4. Polished and Etched Cross-section of Run 2-004-C

Figure 5. Polished and Etched Cross-section of Run 2-005-C

Figure 6. Polished and Etched Cross-section of Run 2-006-C

TABLE I. TABULATION OF HEAT-EXCHANGER AND FURNACE TEMPERATURES

RUN	PURPOSE	SEEDING		GROWTH CYCLE			REMARKS
		FURN. TEMP. ABOVE M.P.	H.E. TEMP. BELOW M.P.	RATE OF DECREASE H.E. TEMP. °C/HR.	FURN. TEMP. °C	GROWTH TIME IN HOURS	
2-001-C	Study of heat flow	17	175	125	17	10.6	Good melt back of seed and good growth.
2-002-C	Study of heat flow	5	172	167	5	10.3	Good melt back of seed and good growth.
2-003-C	Study of heat flow	11	176	214	11	14.2	Good melt back of seed and good growth.
2-004-C	Study of heat flow	8	206	172	8	14.8	Good melt back of seed and good growth.
2-005-C	Study of heat flow	<3	194	168	~3	9.0	Good melt back of seed and good growth.
2-006-C	Study of heat flow	<3	178	142	~3	9.0	Good melt back of seed and good growth.
2-007-C	Study of heat flow	9	182	230	9	7.0	Good melt back of seed and good growth.
2-008-C	Casting a square cross-section boule	<3	132	170	0	4.5	Attachment of boule to bottom of crucible resulted in minor cracking.
2-009-C	Casting in a duplex crucible	7	112	200	7	8.0	The liner attached to the boule.

crystallinity is not high; however, the size of the grains is large. The bottom of the crucibles used in run 2-002-C through 2-006-C is very thick and uneven, over 1 cm. The seed placed at the bottom is not in good contact with the crucible. A good example of this can be seen in Figure 6 where the molten metal has run under the seed. An idea of the uneven bottom of the crucible can be obtained from the curvature of the cross-sections (Figures 2-6). It has been found that most of the growth takes place near the solidification temperature. Therefore, the nucleation of the material on the sides is from the bottom of the crucible and not off the seed. This is further confirmed by Figure 1. In run 2-001-C the crucible had a smoother bottom, thus lateral growth was achieved all the way to the sides of the crucible.

The central grain formed in some cross-sections is attributed to the breakdown of the interface due to probing. A quartz rod is being used to estimate the position of the solid-liquid interface. This grain progressively decreases in size in sections examined in areas off-center of the boule.

The top portion of cross-sections shows an area where the solidification took place from the surface of the melt, hence it was random. With a control of the heat flow by adding insulation in certain areas, this top freeze-over has been considerably reduced. Once again the thick and uneven bottom of the crucible is not conducive to efficient heat transfer for growth to high crystallinity.

For run 2-008-C a square cross-section crucible with inside dimensions $4\frac{1}{2}$ " x $4\frac{1}{2}$ " x 8" was fabricated. The crystal growth cycle did not show any problems different from other runs. However, the bottom of the crucible was attached to the ingot. As a result some cracking of the ingot was observed. The problem seems to be due to the heat treatment of the crucible. Further runs for casting square boules are planned in the near future.

In an effort to reduce the impurities in silicon from the silica crucible a duplex crucible was used in run 2-009-C. This crucible was similar to the opaque graded crucible except that a high purity silica liner was used. The initial attempt in fabricating this kind of crucible resulted in uneven thickness of the liner, 2-5 mm. The liner enveloped the ingot showing attachment and resultant cracking of the ingot. It appears that to use this kind of crucible the thickness of the liner layer has to be optimized.

CRYSTAL SLICING

In the just completed Phase I of this contract, it has been established that Fixed-Abrasive Multiwire (FAM) slicing is a viable method of producing silicon wafers. The slices obtained were as thin as 4 mil, 0.1 mm thick with excellent surface quality, i.e., surface roughness and 3-5 μm surface damage. The blade development effort has shown that impregnated wires have a shorter life as compared with the plated types. The former fails due to diamond pull-out, thereby losing cutting effectiveness. Wires plated after impregnation hold the most promise, since the diamond size and distribution can be best controlled. In plated wires, 30 μm diamonds are the smallest that can be plated to a reasonable concentration. To reduce kerf smaller diamonds may be necessary, hence impregnated blades may again have the advantage. This route is cheaper, also. Crystal Systems has developed a charging method which imbeds the diamonds only in the cutting edge, thereby reducing the costs further. A lighter and longer blade carriage may be useful for increasing the surface speed and thus the cutting effectiveness. In the Phase II program, further machine modifications will be made and larger workpieces sliced. Even though 64 slices/inch, 25/cm have been sliced, it is intended to produce 100/inch, 39/cm by reducing the kerf further.

Summary of Progress

It has been established that fixed diamond abrasive blade suffers failure due to diamond pull-out. This problem is more severe in case of impregnated blade as compared to the plated blades. In run 2-001-S (details in Table II) the effect of higher feed forces on the cutting effectiveness was studied. The plated blade pack was used. As expected, much higher cutting rates were achieved--5.6 mil/min, 0.14 mm/min; however, wire wander seemed to be a problem. This was exhibited in the bow and taper of the wafers produced. A study of the work damage produced in these wafers was in the 12-20 μm range as compared to 3-5 μm produced in wafers sliced with low feed forces, e.g., run 2-002-S.

It was felt that once the wires have been used with high feed forces, the cutting effectiveness is reduced considerably when the feed forces are reduced. In run 2-002-S the same wires used in the earlier run were used at lower feed forces. As expected, the cutting rates were reduced; however, wire wander was also reduced. The damage depth of the wafers sliced in this run was 3-5 μm .

During testing in run 2-003-S CSI impregnated wires were used. These wires were copper-coated tungsten core impregnated in the cutting edge with 30 μm diamond particles. The wires were electroless nickel coated after impregnation. This run was aborted due to non-uniform cutting and wire breakage. The wires that were not cutting and broke were not adequately impregnated because of misalignment

TABLE II. SILICON SLICING SUMMARY

RUN	PURPOSE	FEED		AVERAGE		WIRE TYPE	REMARKS
		FORCE/BLADE lb.	gm	CUTTING RATE mil/min	mm/min		
2-001-S	Study effects of high feed forces	0.20	90.80	5.60	0.142	45 μ m diamond, nickel plated tungsten wire. 5 mil, 0.125 mm core.	High cutting rates achieved. Increased wire wander observed.
2-002-S	Comparison with low feed forces	0.08	36.32	1.36	0.035	Same wire	Lower cutting rates. Less wire wander and reduced surface damage in wafers.
2-003-S	Test CSI impregnated/plated wire	N/A	N/A	N/A	N/A	0.3 mil, 7.5 μ m electroless nickel plated CSI impregnated wire	Run aborted because of wire breakage. Misalignment during impregnation.
2-004-S	Lifetest of Lastec impregnated-plated wire used in runs 77-S, 78-S and 79-S	N/A	N/A	N/A	N/A	Lastec 8 mil, 0.2 mm super wire, 45 μ m diamonds--0.3 mil, 7.5 μ m electroless nickel plated	Run aborted because of loss in cutting effectiveness.

of the charging mechanism. One of the wires which did not fail was examined with the SEM. Figure 7 shows the micrograph of the cutting edge with good diamond distribution. The top side of the blade, opposite the cutting edge, is shown in Figure 8. It shows that the diamonds are impregnated only in the cutting edge. The side surface of the blade, Figure 9, shows diamond pull-out. One of the wires that failed during the cutting process was also examined. The cutting edge of this wire is shown in Figure 10. There seems to be abrasion of the wire in the center. This is attributed to die-marks due to misalignment during impregnation. The surface opposite the cutting surface, Figure 11, shows no diamonds. The side view of the blade in Figure 12 shows considerable diamond pull-out. In order to optimize the impregnation process, it has to be studied as a function of various variables, viz. (i) die design, (ii) size of the core of the wire, (iii) plating thickness for impregnation, (iv) diamond type and size, (v) type of plating and hardness, and (vi) plating thickness to prevent diamond pull-out. Experiments incorporating these variables are being planned.

In run 2-004-S, the blade pack used was the same as used in three earlier runs,⁶ 77-S, 78-S, and 79-S. The wires used are commercially impregnated wires electroless plated with 0.3 mil, 7.5 μm thickness nickel after impregnation. The run was aborted during the final stages as the wires had lost their cutting effectiveness. Figure 13 shows the cutting edge of the wire and Figure 14 the top surface (opposite the cutting edge). It can be seen that plating has cracked near the corners of the diamond.

Figure 7. Micrograph of Cutting Edge of Impregnated Wire

Figure 8. Micrograph of Opposite Edge of Wire in Figure 7

Figure 9. Side View of the Wire in Figure 7.

Figure 10. Cutting Edge of Poorly Impregnated Wire

Figure 11. Opposite Edge of Wire in Figure 10.

Figure 12. Side View of Wire in Figure 10.

A comparison of Figure 13 and Figure 14 shows that the plating is not worn on top, but it is on the bottom cutting edge. Not much diamond degradation or pull out can be observed in either of these SEM photographs. The cutting edge (Figure 15) of the wire was observed with the SEM at a higher angle and lower magnification to get a better perspective of the cutting edge. Considerable diamond pull-out is apparent in Figure 15. This probably accounts for the loss in cutting effectiveness. Even though run 2-004-S was aborted, the total cumulative depth of cut achieved with plated impregnated wires was 7.2 inches (18 cm) as against 2.7 inches (6.75 cm) for impregnated wires.

Figure 13. Cutting Edge of Wire Used in Run 2-004-S.

Figure 14. Opposite Edge of Wire in Figure 13.

Figure 15. A Tilted View of Figure 13.

CONCLUSIONS

1. The crystallinity of the new grown material was good whenever proper seeding took place. Even with mechanical probing of the solid-liquid interface, breakdown was limited to the local area.
2. Most of the growth in silicon took place near the solidification temperature.
3. A square cross-section can be cast with the Heat Exchanger Method.
4. It has been established that the life of an impregnated blade can be prolonged by plating it.
5. In order to impregnate wires with diamonds on the cutting edge only, the alignment during impregnation is very important.
6. Higher cutting rates can be achieved with higher feed forces. However, under these conditions, wire wander and greater surface damage is obtained.

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