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SILICON INGOT CASTING—HEAT EXCHANGER METHOD MULTI-WIRE SLICING—FIXED ABRASIVE SLICING TECHNIQUE. PHASE III

Quarterly Progress Report No. 4 for July 1—September 30, 1979

By
Frederick Schmid
Chandra P. Khattak

October 1979
Report Issued

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

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SILICON INGOT CASTING: HEAT EXCHANGER METHOD, MULTI-WIRE SLICING: FIXED-ABRASIVE SLICING TECHNIQUE (PHASE III)

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

Quarterly Progress Report No. 4
by

Frederick Schmid and Chandra P. Khattak

Covering Period from July 1 through September 30, 1979

Report Issued: October 1979

JPL Contract No. 954373

CRYSTAL SYSTEMS, INC.
35 Congress Street
Salem, MA 01970

The JPL Low-Cost Solar Array Project is sponsored by the U. S. Department of Energy and forms part of the Solar Photovoltaic Conversion Program to initiate a major effort toward the development of low-cost solar arrays. This work was performed for the Jet Propulsion Laboratory, California Institute of Technology, by agreement between NASA and DOE.

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ACKNOWLEDGEMENT

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ABSTRACT

This contract is for casting silicon ingots by the Heat Exchanger Method (HEM) and slicing by multi-wire Fixed Abrasive Slicing Technique (FAST).

Significant advancements have been made in the area of crystal casting during the period of August through October 1979. It has been demonstrated that nearly single crystal ingots can be cast with a single HEM solidification of upgraded metallurgical grade silicon. The impurities were rejected to the last material to freeze--near the wall of the crucible. The resistivity of the silicon after directional solidification by HEM was 0.1 - 0.2 Ω-cm. Macroscopic impurities, presumably SiC, did not break down the solid-liquid interface and, in some cases, caused only localised twin formation. This material may be used for making solar cells directly. It has the potential for decreasing the cost below the DOE goals and avoiding a silicon short fall.

The HEM process has been scaled up to solidify 22 cm x 22 cm square cross-section ingots weighing up to 10.5 kg. The 22 cm square cross-section ingot is nearly all single crystal material.

For silicon slicing using FAST significant progress has been made in demonstrating high throughput of the slicer and extended life of the wires. Cutting rates exceeded 1986 goals by more than 40%. This has been achieved with the combination of high speeds of the slicer and improvement in the blade. A blade life of two slices per wire has been demonstrated.

Emphasis in the area of blade development has been with impregnation using CSI technology of impregnating diamonds only in the cutting edge. A systematic study of impregnation has shown considerable improvement in diamond concentration, cutting effectiveness and wire life.

SILICON INGOT CASTING -- HEAT EXCHANGER METHOD

Efforts during the present quarter have resulted in significant advancements. It has been demonstrated by one HEM solidification that upgraded metallurgical grade silicon can be solidified into nearly single crystal ingots. Ingot size has been scaled up to 22 cm x 22 cm square cross-section weighing 10.5 kg. Crucible delamination has been improved to eliminate any attachment.

Use of Low-Purity Meltstock

An important feature of the HEM is that the crystal growth takes place from the bottom of the crucible to the top. Therefore, impurities which float on the surface of the melt do not interfere with the growing solid-liquid interface. Usually low-purity meltstocks have high carbon content and are coated with an oxide layer. On melting, the silica and silicon carbide float to the surface of the melt.

Most impurities in silicon have a distribution coefficient significantly less than unity;[1] therefore, during directional solidification these impurities are rejected to the melt and will eventually pile up in the last material to freeze. For HEM growth, this material is along the crucible wall. Cast ingots will have to be sectioned to maintain dimensional accuracy; hence during the sectioning operation the contaminated material will be removed. In a typical casting process the control of the solidification interface, if any, is only marginal and growth takes place from the sides to the center; thereby the last material to freeze is in the central region. Another important feature of HEM is the enlargement of the interfacial area as growth proceeds. Thus the effect of pile-up of impurities at the melt interface is minimized and constitutional supercooling is suppressed.

In view of the above, run 353-C (details in Table I) was carried out using upgraded metallurgical silicon meltstock. Operational parameters were kept similar to runs using high-purity meltstock. Considerable amount of contaminants were seen floating on the surface of the melt throughout the solidification. This HEM ingot did not show any signs of cracking as seen in Figure 1. A polished and etched section of this ingot is shown in Figure 2. It can be seen that a very high degree of single crystallinity has been achieved. Macroscopic examination of the surface shows dark precipitates, presumably SiC, on the surface of the melted-back seed. This did not disturb the interface. With the Czochralski method, where the interface is on the melt surface, single crystallinity could not be achieved during the first pull using this melt stock. This demonstrates the stability of the submerged interface.

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	RATE OF DECREASE H.E. TEMP. °C/HR.	RATE OF DECREASE FURN. TEMP. °C	GROWTH TIME IN HOURS	
339-C	Improve crucible delamination	< 3	96	380	0	5.3	Very good crystallinity.
340-C	Test effect of larger plug	< 3	101	340	0	6.5	Very good crystallinity.
341-C	Improve crucible delamination	< 3	102	356	0	6.75	Attachment of ingot to crucible.
342-C	Modified heat treatment of crucible	< 3	98	338	1	7.25	Non-uniform graded structure of crucible.
343-C	Test larger plug	< 3	110	354	2	6.5	Very good crystallinity.
344-C	Modified heat treatment of crucible	< 3	107	440	1	8.0	Non-uniform graded structure of crucible.
345-C	Cast 8.3 kg ingot	< 3	121	501	0	15.5	Very good crystallinity
346-C	Cast 8.3 kg ingot	3	128	170	3	11.5	No "chipping" of ingot.
347-C	Improve crucible delamination	12	140	478	12	6.3	Partial delamination of crucible. Very good crystallinity.
348-C	Improve crucible delamination	4	132	413	4	6.25	Partial delamination of crucible. Very good crystallinity.
349-C	Cast 6.5 kg ingot	< 3	112	389	1	10.75	Very good crystallinity.

(cont.)

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

RUN	PURPOSE	SEEDING		GROWTH CYCLE			REMARKS
		FURN. TEMP. ABOVE M.P. °C	H.E. TEMP. BELOW M.P. °C	RATE OF DECREASE H.E. TEMP. °C/HR.	RATE OF DECREASE FURN. TEMP. °C	GROWTH TIME IN HOURS	
350-C	Cast 7.5 kg ingot	3	128	370	3	11.3	Good crystallinity.
351-C	Cast 7.5 kg ingot	6	121	393	4	10.5	Good crystallinity. Limited attachment of crucible.
352-C	Improve crucible delamination	-	-	-	-	-	Run terminated due to helium pump malfunction.
353-C	(4) Crystal growth of upgraded metallurgical silicon meltstock	< 3	113	420	0	7.75	No cracking of ingot. Single crystallinity achieved. Impurities segregated during directional solidification.
354-C	Solidify 10.5 kg, 22 cm square ingot	< 3	111	353	0	9.8	No cracking of ingot. Very good crystallinity all the way to the top of the boule.
355-C	Solidify 8.1 kg 16 cm square ingot	4	91	478	4	10.0	No cracking of ingot.
356-C	Solidify 16 cm square ingot	4	93	450	-	-	Control instrumentation malfunction. Power shut down prematurely.
357-C	Optimize post-solidification cycle	11	109	484	11	6.7	Attachment of crucible in some areas.
358-C	Optimize post-solidification cycle	7	112	438	7	7.0	Very limited attachment of crucible.

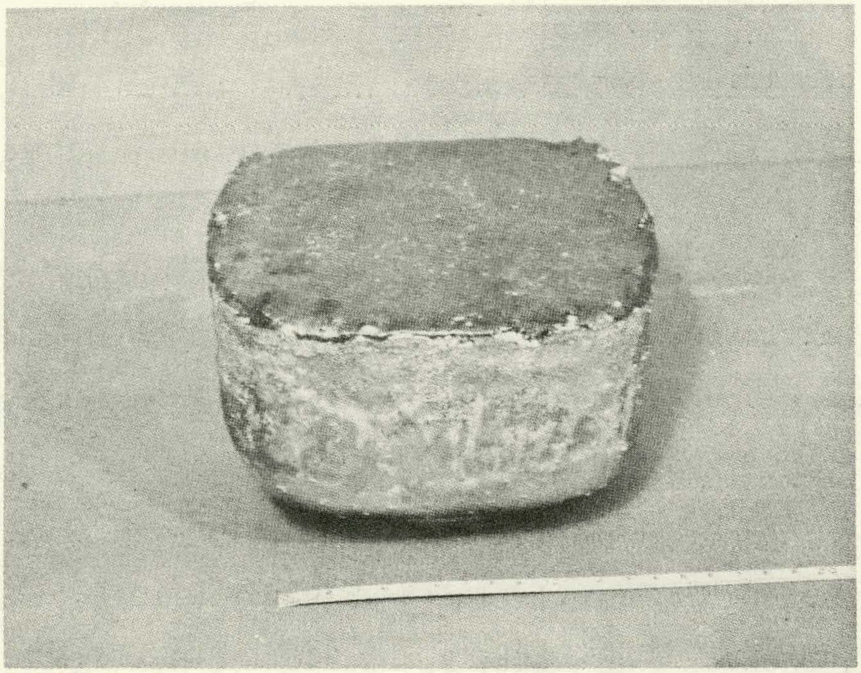


Figure 1. A 5.5 kg, 16 cm x 16 cm cross-section ingot cast in run 353-C using upgraded metallurgical silicon.

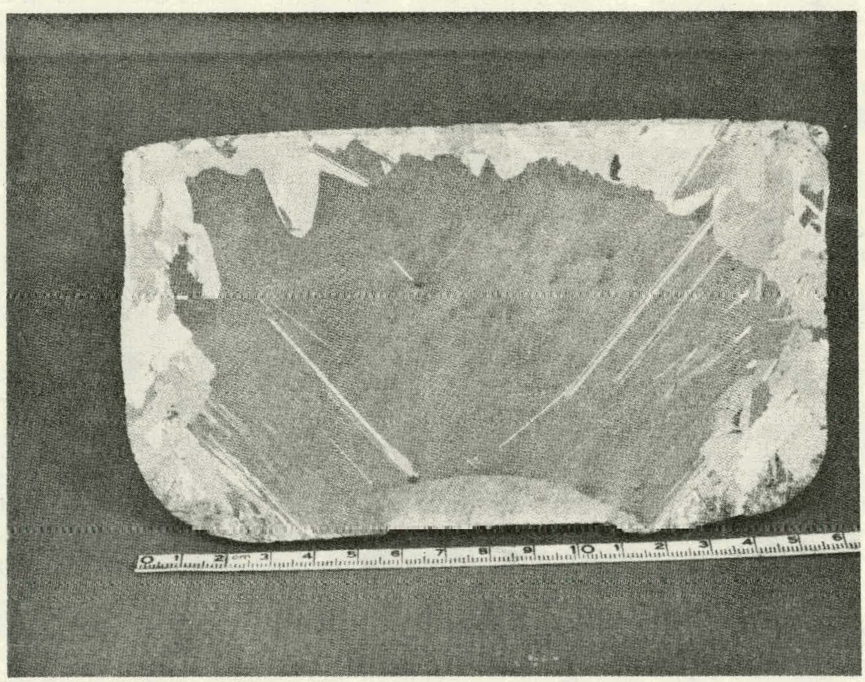


Figure 2. Cross-section of the ingot shown in Figure 1.

The origin of the twins visible in Figure 2 can be traced to dark precipitates, again showing that such large particulates cause only local disturbance of the interface. A closer examination of the cross-section shows that the concentration of these precipitates is more in areas which are last to solidify. Around the periphery where growth was enhanced by lowering the furnace temperature, very large grains were formed.

Four-point probe resistivity measurements of the surface shown in Figure 2 gave a resistivity value of 0.1 - 0.2 Ω-cm. Such material should, therefore, be usable for making solar cells directly.

Scale-up in Size of Ingots

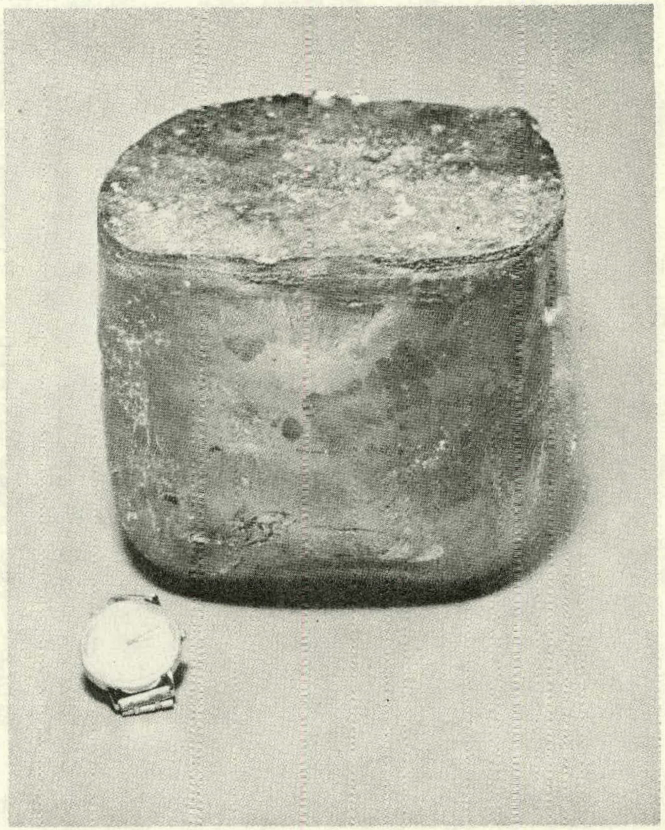
One of the biggest problems of casting silicon in silica crucibles is the attachment of the ingot to crucible at high temperatures which results in cracking of the ingot. In order to prevent cracking a graded silica crucible has been developed which delaminates during the cool-down cycle. The heat treatment of the crucible to develop a graded structure has so far been carried out manually. With the large-size crucibles presently being used, non-uniformity of heat treatment occurs in small areas. This has resulted in attachment in these areas causing "chipping." Initially about 4 kg ingots were cast. Efforts were made to increase the uniformity of the graded structure and, at the same time, scale up the size of the ingots. It was found that high gradients during the heat treatment were necessary to cause delamination of the crucible. Evidence of progress is shown in Figure 3 which shows two of the 16 cm x 16 cm square cross-section crack-free ingots cast by HEM. These ingots weigh over 8 kg.

One of the advantages of the HEM is that the size and shape of the ingot is determined by crucible. Once growth parameters are established scale-up in size is a simple extension of the process. Figure 4 shows the first attempt at 22 cm x 22 cm cross-section, 10.5 kg ingot. This is the biggest and heaviest silicon ingot cast by HEM. An additional advantage of large sizes is the faster growth rates achieved because of enlarged interface size. A comparison of the growth times of run 354-C with 355-C shows that the growth time is slightly shorter for the 22 cm square ingot even through it has about 30% more meltstock.

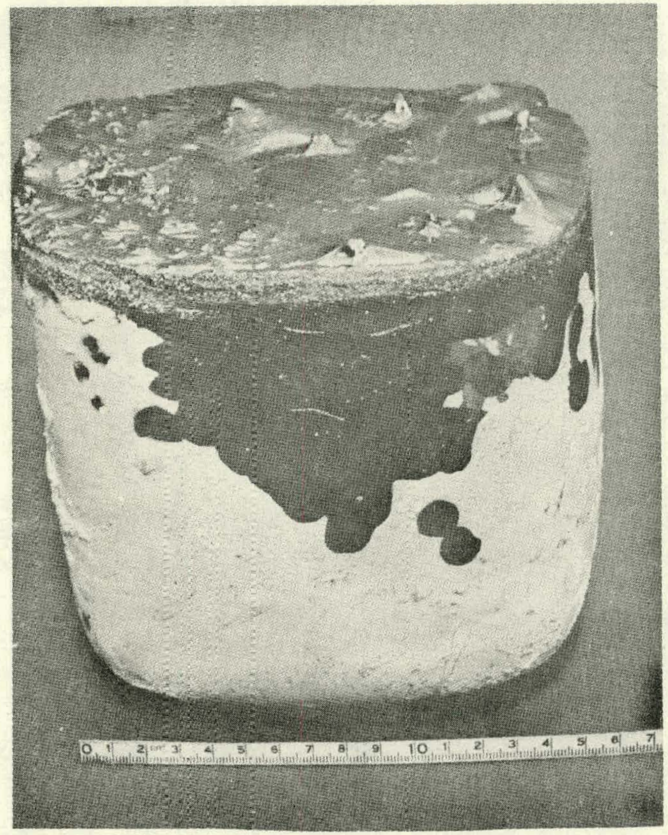
At the present time the scale-up in size of ingots has been carried from 10 cm square to 22 cm square via an intermediate size of 16 cm square cross-section. An idea of the growth in size of ingots can also be had from the three crucibles used. These crucibles are shown in Figure 5 along with their nominal square dimensions.

Characterizations

In addition to crucible development the improvement of crystallinity has also progressed very well. Figure 6 shows an

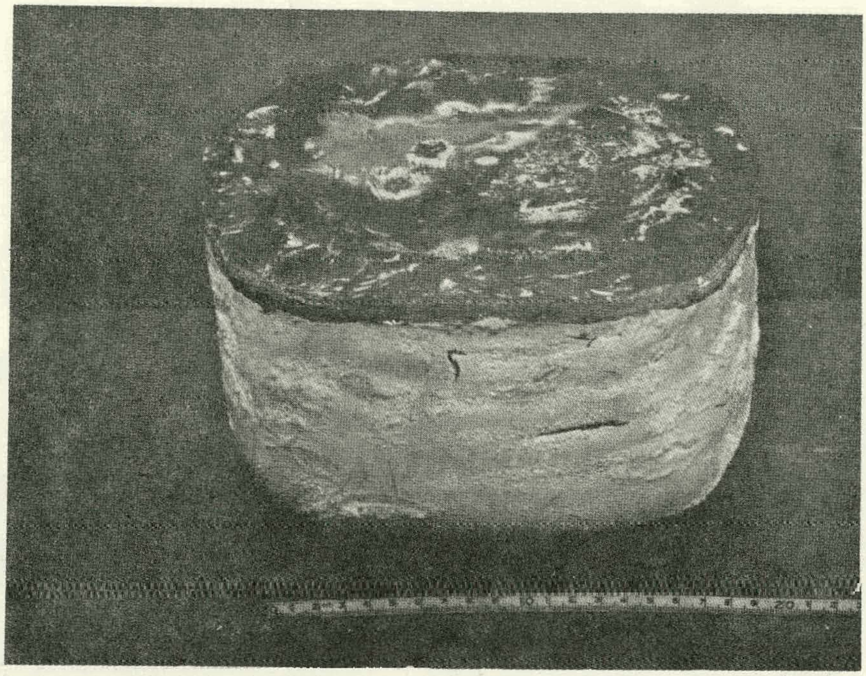


(a)



(b)

Figure 3. Two of the 16 cm square ingots cast by HEM
(a) Run 346-C (8.3 kg) and (b) Run 355-C (8.1 kg)



(a)

(b)

Figure 4. (a) As cast surface of 10.5 kg, 22 cm square ingot; (b) After removal of attached silica

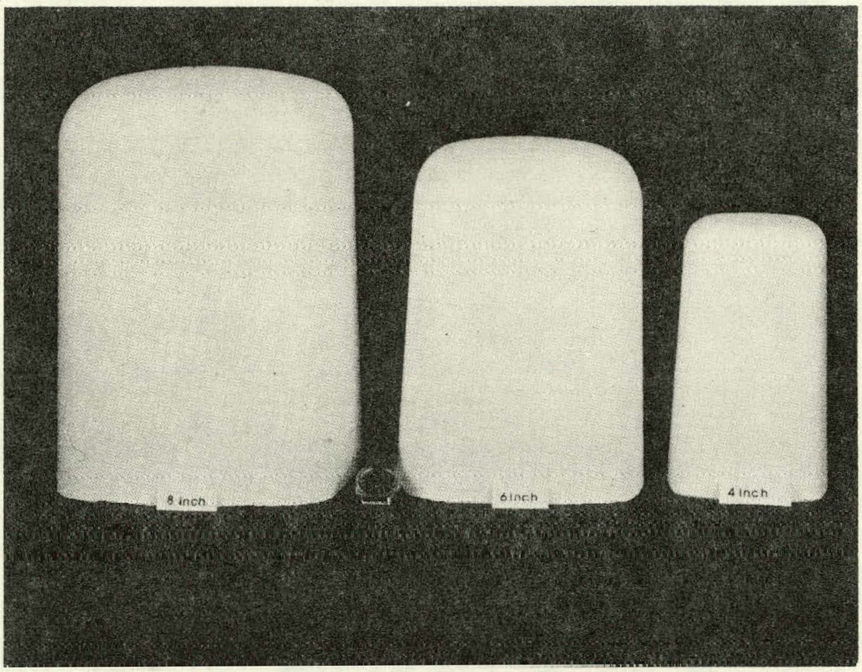


Figure 5. Square crucibles used during scale-up by HEM -- 22 cm x 22 cm, 16 cm x 16 cm, 10 cm x 10 cm cross section

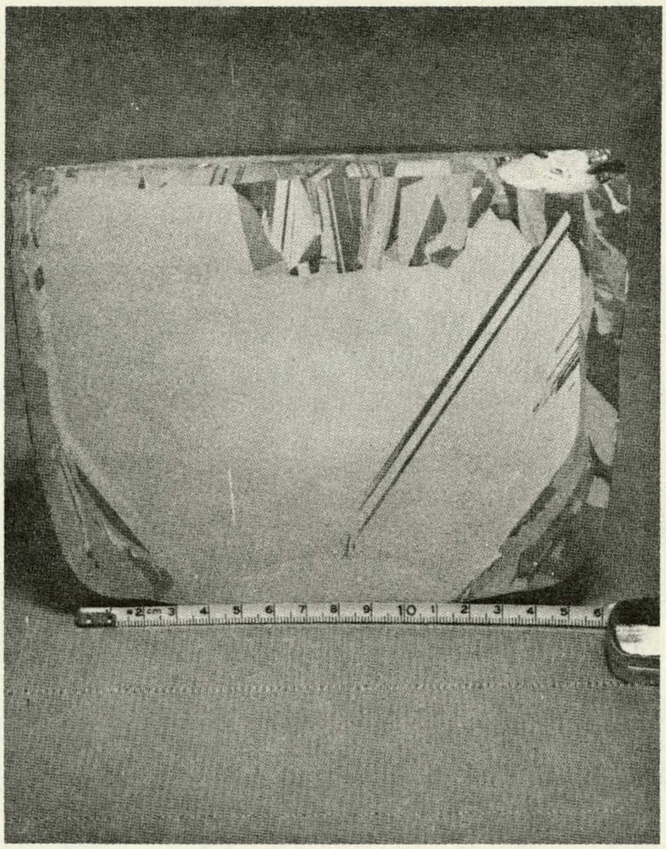


Figure 6. Polished and etched section of ingot cast in run 345-C (8.1 kg)

example of the structure obtained in a 8.3 kg ingot (run 345-C). The breakdown in structure near the top of the ingot was caused by a dendrite floating on the melt that was pushed down onto the growing interface by the interface probe. Figure 7 shows the structure of a 16 cm x 16 cm square ingot cast in run 351-C. It can be seen that except in the periphery of the ingot, the material is single crystal. During this run the furnace power was decreased towards the end of the solidification cycle while there was still some molten silicon. This has resulted in the solidification of large-grain silicon. A slab from run 338-C was sectioned perpendicular to the growth direction. This material showed more than 90% single crystallinity (Figure 8). In run 349-C single crystal growth was achieved all the way to the top surface of the ingot (Figure 9). A similar single-crystal growth was also obtained in the first run at 22 cm x 22 cm square cross-section ingot shown in Figure 10 (run 354-C).

In summary, the process of silicon crystal growth by HEM has been scaled up to 22 cm square cross-section 10.5 kg ingots while maintaining a very high degree of single crystallinity. It has been demonstrated that low-purity, upgraded metallurgical silicon can be processed to single crystal material which can be used for photovoltaic applications.

It may be significant to point out here that Crystal Systems, Inc., was awarded the I-R 100 award for square single-crystal ingot for solar energy applications. This prize is

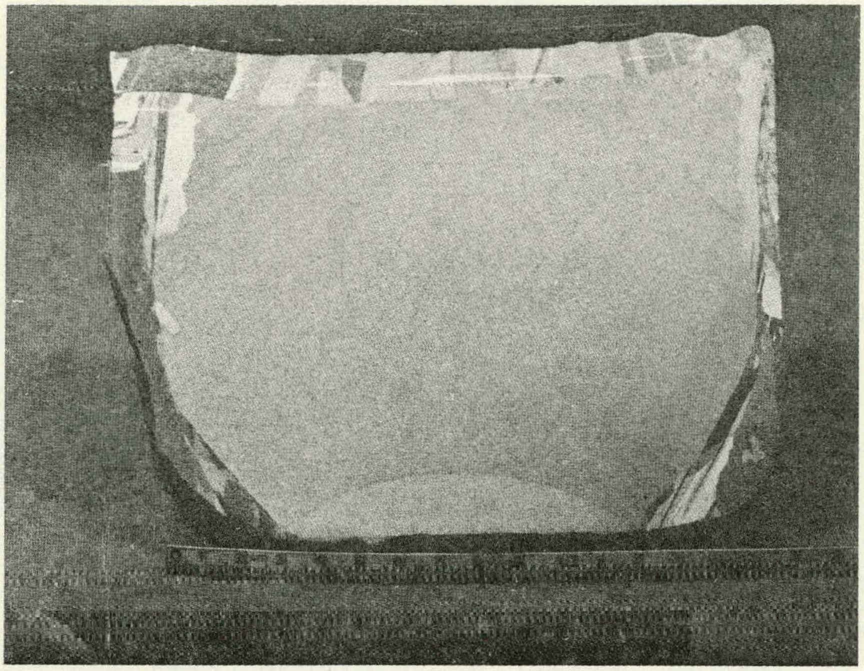


Figure 7. Crystal structure of ingot cast in run 351-C

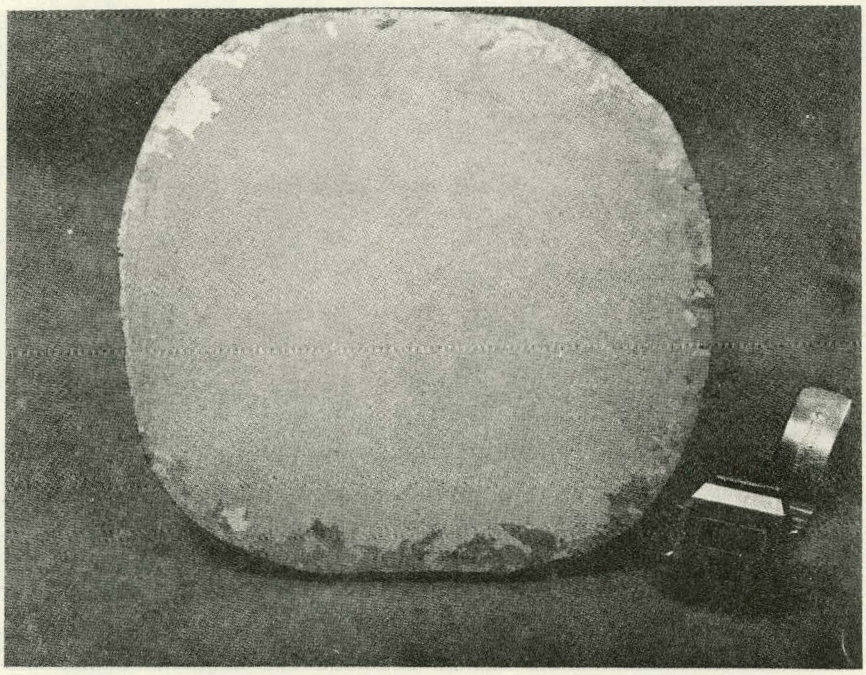


Figure 8. Polished and etched slab sectioned perpendicular to growth direction showing cross-section and crystallinity

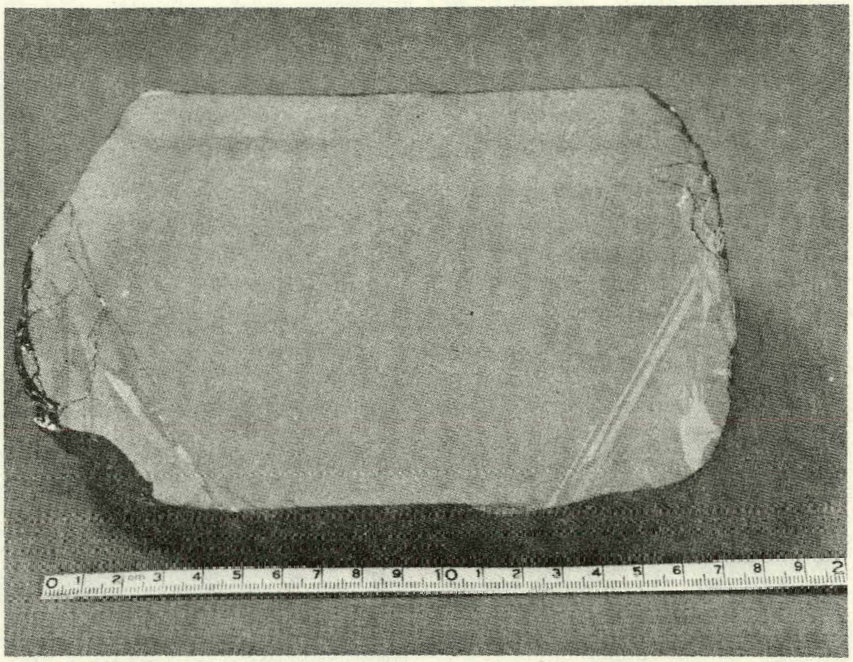


Figure 9. Polished and etched section of ingot cast in run 349-C

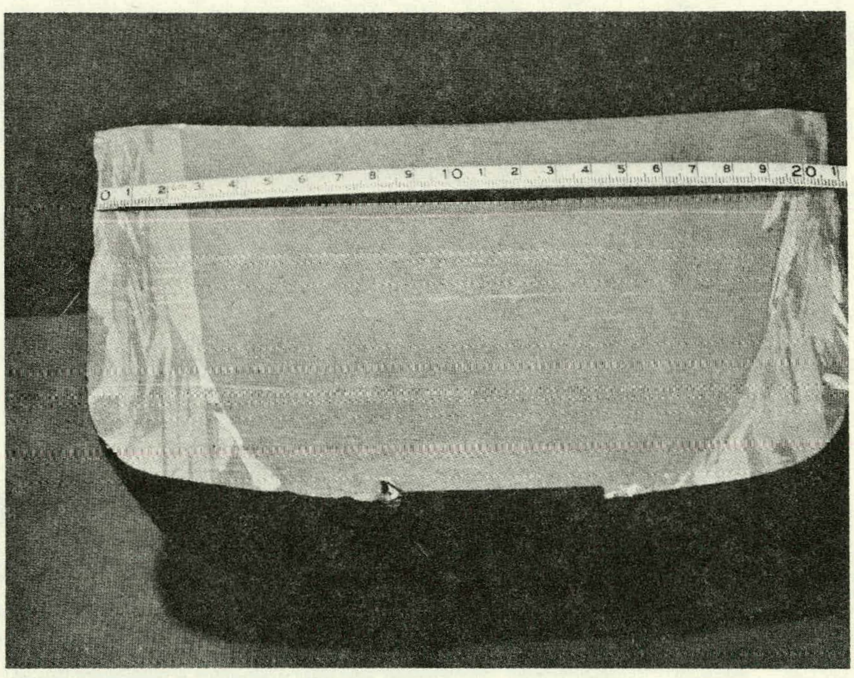


Figure 10. Polished and etched section of 22 cm x 22 cm cross-section ingot cast in run 354-C

given by Industrial Research/Development magazine for developing one of the 100 most significant technical products in 1978.

MULTI-WIRE SLICING--FIXED ABRASIVE SLICING TECHNIQUE

Emphasis in the area of crystal slicing using the Fixed Abrasive Slicing Technique (FAST) during the present quarter was placed on blade development and testing. Significant progress was made in demonstrating high throughput of the slicer and extended life of the wires. Cutting rates of about 40% more than the projected estimates used in the economic analysis were achieved. Considerable progress was made in the area of blade development using impregnated wires in showing cutting effectiveness as well as life.

Testing

During last quarter[2] it was demonstrated that the cutting effectiveness was considerably improved at high speeds using the Fixed Abrasive Slicing Technique (FAST). This was shown using commercially impregnated wires to slice 10 cm diameter silicon ingot. During run 328-SX (details in Table II) this performance was repeated. Good quality wafers were sliced at an average cutting rate of 5.62 mil/min, 0.143 mm/min. These cutting rates are about 40% more than the projected cutting rates used in the economic analysis.[3] Figure 11 shows a plot

TABLE II. SILICON SLICING SUMMARY

RUN	PURPOSE	FEED FORCE/BLADE lb	FEED FORCE/BLADE gm	AVERAGE CUTTING RATE mil/min	AVERAGE CUTTING RATE mm/min	WIRE TYPE	REMARKS
328-SX	Blade life test	0.093	42.4	5.62	0.143	Commercially impregnated wire with 0.3 mil, 7.5 μm electroless nickel	Good quality wafers; 77% yield
329-SX	Life test continuation	0.095	43.0	4.82	0.122	Same as 328-SX	Lateral movement of guide roller gave poor yield (28%)
330-SX	Life test continuation	0.095	43.0	3.44	0.087	Same as 328-SX	Wire breakage due to work hardening
330-S	Test CSI impregnated blades	0.080	36.2	1.8	0.045	5 mil, 0.125 mm W core; 0.7 mil, 15.5 μm Cu sheath; 30 μm natural diamonds impregnated in cutting edge only	Very good quality wafers; 58% yield
331-S	Test electroplated blades	0.070	31.7	1.7	0.042	30 μm synthetic diamonds electroplated in cutting edge only	67% yield. Loss of cutting effectiveness with time.
332-S	Life test	0.075	34.3	0.82	0.020	Same as 331-S	Poor cutting rates
333-S	Test CSI impregnated wires	0.083	37.7	1.6	0.041	5 mil, 0.125 mm W core; 0.7 mil, 15.5 μm Cu sheath; 30 m natural diamonds impregnated in cutting edge only; 0.3 mil, 7.5 μm electroless nickel plating	Very good quality wafers; 86% yield.

(cont.)

TABLE II. SILICON SLICING SUMMARY (cont.)

RUN	PURPOSE	FEED FORCE/BLADE lb	FEED FORCE/BLADE gm	AVERAGE CUTTING RATE mil/min	AVERAGE CUTTING RATE mm/min	WIRE TYPE	REMARKS
334-SX	Study effect of surface speed on slicing performance using rectangular workpiece	0.084	38.3	2.13	0.054	Commercially impregnated wire with synthetic diamonds; 0.4 mil, 10 μm electroless nickel plating	Problem with ways for bladehead
335-S	Test CSI impregnated wires using 45 μm natural diamonds	0.078	35.8	2.31	0.057	5 mil, 0.125 mm stainless steel core; 1 mil, 25 μm Cu sheath; 45 μm natural diamonds impregnated in cutting edge only; 0.3 mil, 7.5 μm electroless nickel plating	100% yield; very good quality wafers
336-S	Life test	0.081	36.7	1.54	0.038	Same as 335-S	Good quality wafers; 74% yield. Second run with bladepack.
337-SX	Test electroplated wires	0.080	36.2	3.90	0.097	5 mil, 0.125 mm music wire electroplated with 30 μm natural diamonds	Trouble with bladehead.
338-S	Life test continuation	0.084	38.3	0.89	0.022	Same as 335-S	Third run with blade pack. 58% yield.
339-S	Test CSI impregnated wires using 30 μm natural diamonds	0.076	34.8	2.62	0.065	5 mil, 0.125 mm stainless steel core; 1 mil, 25 μm Cu sheath; 30 μm natural diamonds impregnated in cutting edge only; 0.3 mil, 7.5 μm electroless nickel plating	100% yield. Very good quality wafers.

TABLE II. SILICON SLICING SUMMARY (cont.)

RUN	PURPOSE	FEED FORCE/BLADE lb	FEED FORCE/BLADE gm	AVERAGE CUTTING RATE mil/min	AVERAGE CUTTING RATE mm/min	WIRE TYPE	REMARKS
340-S	Life test	0.080	36.2	1.54	0.038	Same as 339-S	Good quality wafers. Second run with bladepack. 73% yield.
341-S	Life test continuation	0.078	35.6	0.325	0.008	Same as 339-S	Poor cutting during third run with bladepack.
342-SX	Test realigned bladehead	0.096	43.8	2.59	0.064	Commercially impregnated wires with 45 μm natural diamond; 0.3 mil, 7.5 μm electroless nickel	No machine problems; 50% yield.
343-S	Test commercially impregnated wire batch	0.062	28.9	1.70	0.042	Similar to 342-SX	Wafers lost at the end of run due to poor bond of ingot to plate.

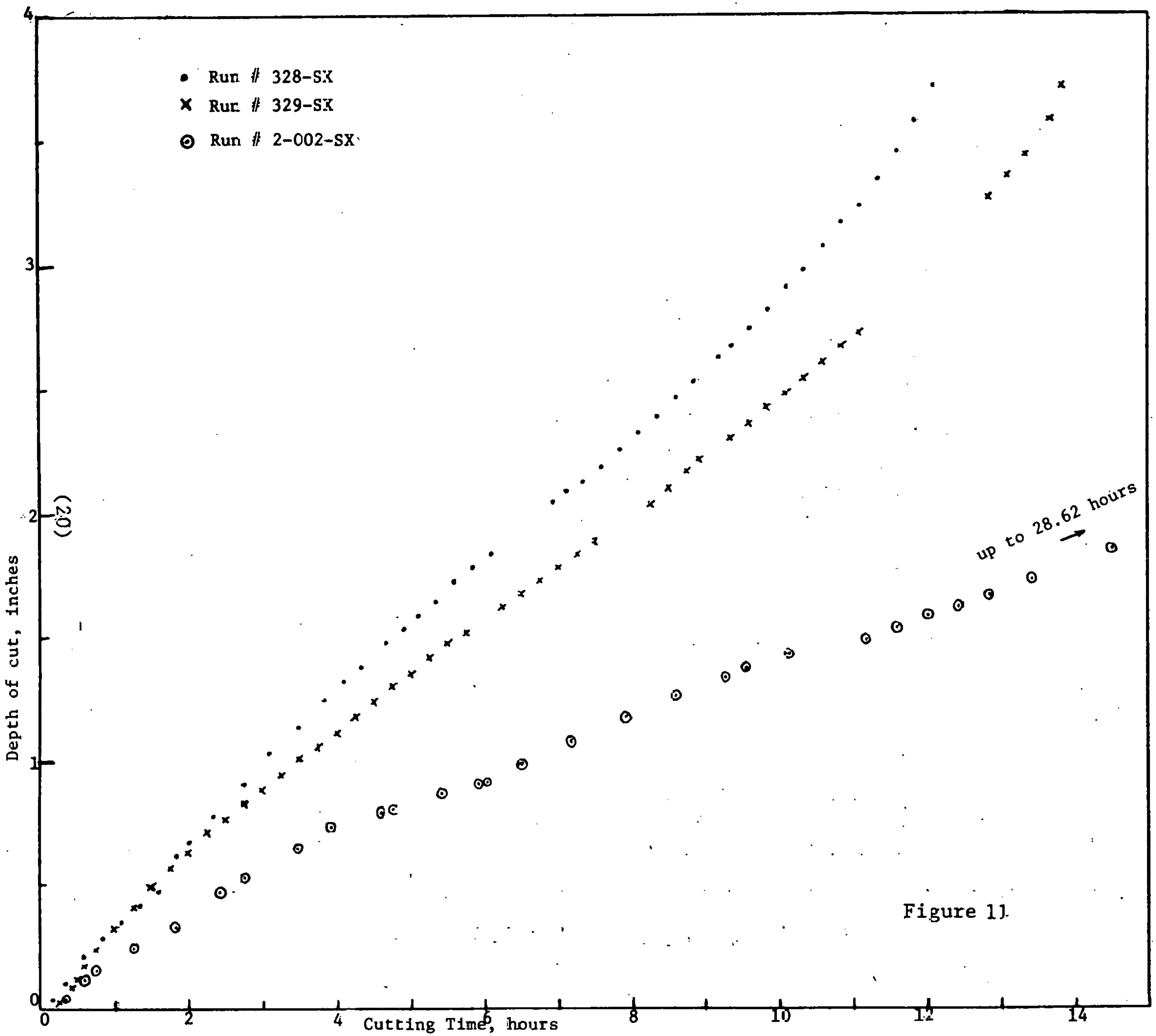

Run # 328-SX
Run # 329-SX
Run # 2-002-SX
up to 28.62 hours
Depth of cut, inches
Cutting Time, hours
0
1
2
3
4
0
2
4
6
8
10
12
14

Figure 11

of the depth of cut with time during runs 328-SX and 329-SX. It shows that except at the start of slicing and towards the end the cutting rate is quite linear. This demonstrates that the varying kerf length during slicing of a 10 cm diameter crystal is minimized by rocking the workpiece during FAST slicing. In regions where non-linear cutting rates were observed, the kerf length is changing rather rapidly and higher cutting rates are observed.

One of the main criteria in reducing costs is the cost of expendable materials. The goal of the present phase is to demonstrate slicing of two 10 cm diameter ingots with the same bladepack. This goal was met at the end of run 329-SX. The same bladepack was used in runs 328-SX and 329-SX. The cutting effectiveness of run 329-SX was good; however, poor yield was achieved because one of the guide rollers was moving laterally causing wafer breakage.

Also shown in Figure 11 is the data for run 2-002-SX when the surface speed of the machine was 200 ft/min. Runs 328-SX and 329-SX were sliced at 400 ft/min using the same set of wires. The latter run shows the deterioration of the blade. Comparison of the data for runs 2-002-SX and 328-SX clearly shows that a significant improvement in cutting performance is achieved when surface speed is increased from 200 ft/min to 400 ft/min. By doubling the speed the average cutting rates increased from

2.33 mils/min, 0.059 mm/min to 5.7 mils/min, 0.145 mm/min, a factor of 2.45.

In run 331-S and 332-S, an electroplated bladepack with 30 μm synthetic diamonds in the cutting edge only was used. It was found that a loss of cutting effectiveness occurred with time. This is probably because diamonds were of synthetic variety.

Emphasis has been placed on CSI impregnated wires as they have the greatest potential for lowering the cost and giving the lowest kerf. A systematic analysis of diamond size, kind, sheath thickness, impregnation parameters, and plating thickness is in progress. The data in Table II for runs 330-S, 333-S, 335-S, 336-S, 338-S, 339-S, 340-S and 341-S show a consistent improvement in performance of cutting effectiveness. It is significant to point out that a set of wires with 45 μm natural diamonds was used in three runs, viz. 335-S, 336-S and 338-S. Life of these wires for cutting three silicon samples is a clear indication of the improvement in the blades. In all the runs good quality wafers were sliced and in run 335-S no wafers were broken during slicing. A similar life of three runs (338-S through 340-S) was also demonstrated for 30 μm diamond impregnated using CSI technology.

Blade Development

Commercially available wire is of 5 mil, 0.125 mm core with a 1.5 mil, 38 μm thick copper sheath into which 45 μm diamonds are impregnated. These wires suffer diamond pull-out during slicing silicon; however, their life can be prolonged by nickel plating. A 0.3 mil, 7.5 μm nickel plating is applied as thicker plating buries the diamonds and anything less does not prevent diamond pull-out. These wires have diamonds impregnated over the entire circumference of the wire. This results in use of more diamonds as only the bottom of the wire is used for slicing. Further, the diamonds on the top surface degrade the guide rollers.

With Crystal Systems technology, diamonds are impregnated only in the cutting edge. Diamonds are impregnated only on the bottom circumference by impregnating the wires after the bladepack is assembled and wires are in tension. Impregnation on the side of the wire is controlled by the dimension of the grooves in the impregnation die. These wires do not degrade the support rollers and seat better in the grooves of the rollers. Further, with CSI technology the kerf thickness is less because the diamonds are not present on the sides of the wire. The improvement in guiding of the wires and lower kerf has resulted in better accuracy of the wafers and high yields in slicing. It is intended to optimize the diamond size, copper sheath and nickel plating thicknesses in addition to the impregnation parameters for efficient slicing.

A bladepack consisting of 5 mil, 0.125 mm core wire with 0.7 mil, 15.5 μm copper sheath was impregnated with 30 μm natural diamonds. It was plated with 0.3 mil, 7.5 μm electroless nickel after plating. The diamond concentration achieved was equivalent to the best commercial wire available. Good quality wafers were sliced with 86% yield when this pack was used in run 333-S. (See Table II). A study of the effect of time of impregnation on diamond concentration showed that initially the concentration increased with time; however, after some time the diamonds were dislodged leaving the copper sheath abraded. Subsequent impregnation into this abraded copper resulted in poor impregnation and diamonds fell off the wire during handling and/or during plating. Using this data two bladepacks have been impregnated with 45 μm and 30 μm diamonds into 1 mil, 0.025 mm copper sheath to a greater concentration than is commercially available.

Diamond impregnation (45 μm) into a 1 mil, 25 μm copper sheath using the above-mentioned process was used in runs 335-S, 336-S and 338-S and very good cutting effectiveness and life was demonstrated. This is the first time CSI impregnated blades have shown cutting life of three boules. A similar bladepack using 30 μm diamond was used in runs 339-S through 341-S. These blades also showed a life of three boules; however, compared with the 45 μm diamond size the cutting rates were poor.

Further correlation of sheath and plating thickness with cutting effectiveness will be pursued with a new impregnation machine for making large blade packs.

CONCLUSIONS

1. It has been demonstrated that upgraded metallurgical grade silicon can be solidified into nearly single crystal ingots with one HEM solidification.
2. The scale-up of ingots has been extended to 22 cm x 22 cm square cross-section weighing 10.5 kg.
3. Single crystallinity has been maintained in nearly all of the 22 cm square ingots.
4. Cutting rates of about 40% more than the projected estimates to meet 1986 goals have been demonstrated.
5. A blade life of two slices per wire has been demonstrated.
6. Impregnation has been improved to give high concentration of diamonds on the cutting edge. This has resulted in better cutting effectiveness and longer blade life.

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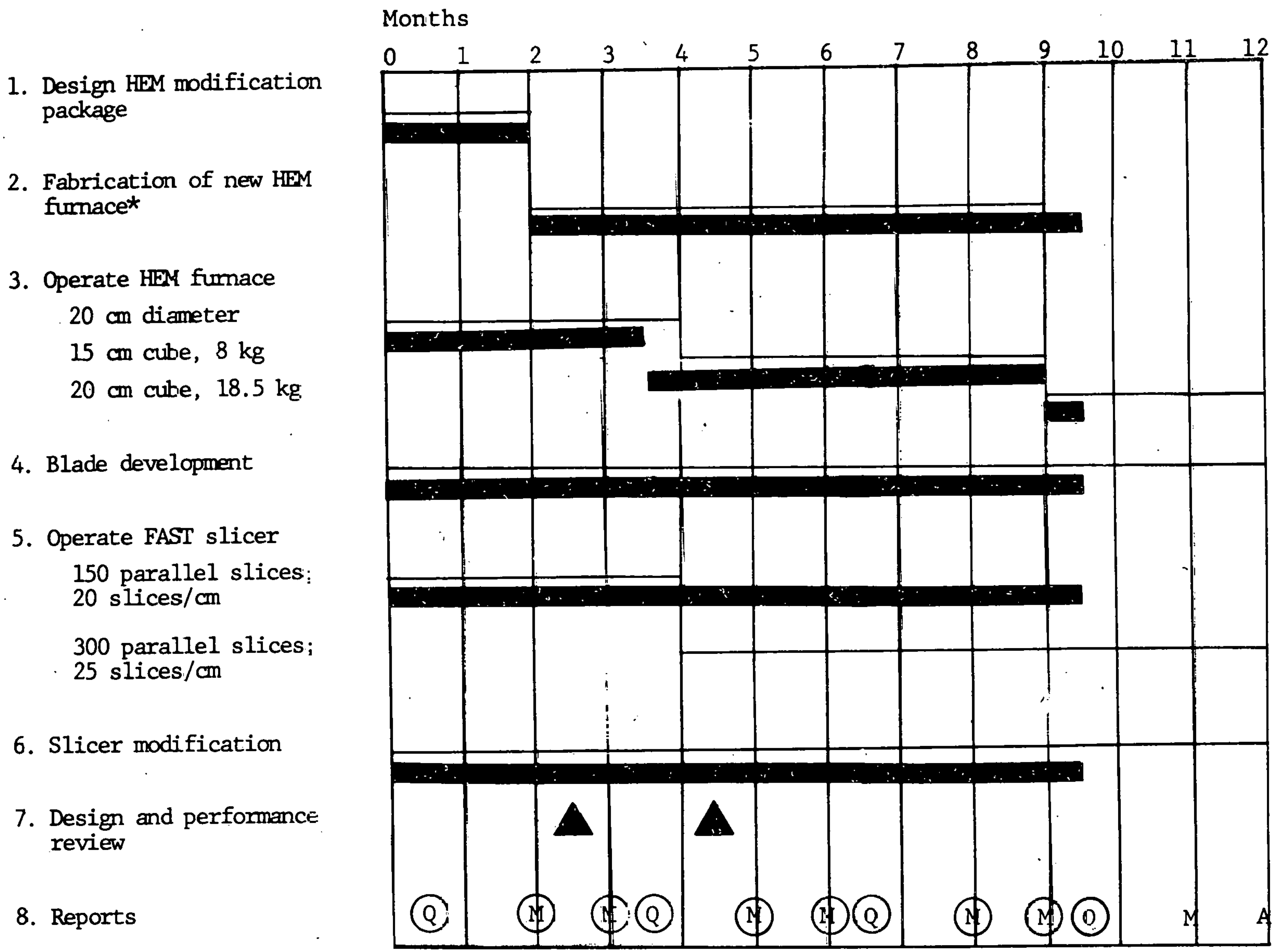
3. F. Schmid and C. P. Khattak, "Heat Exchanger Method--Ingot Casting/Fixed Abrasive Method--Multi-Wire Slicing (Phase II)," Doe/JPL 954373, Crystal Systems, Inc., Quarterly Progress Report No. 3, July 15, 1978.

Contract 954373 (III)

MILESTONE CHART

Crystal Systems, Inc.



(*Modified milestone)

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