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SILICON INGOT CASTING—HEAT EXCHANGER METHOD (HEM) MULTI-WIRE SLICING—FIXED ABRASIVE SLICING TECHNIQUE (FAST)

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

Quarterly Progress Report No. 3, July 1—September 30, 1980

By
F. Schmid
C. P. Khattak
M. Basaran

October 1980
Report Issued

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

Crystal Systems, Inc.
Salem, Massachusetts

U.S. Department of Energy

Solar Energy

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Covering Period from July 1, 1980 through September 30, 1980

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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.

ACKNOWLEDGEMENT

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ABSTRACT

The size of ingots cast by HEM has been increased to 45 kg with 34 cm x 34 cm and 32 cm x 32 cm cross-sections. A new crucible has been developed which has a better shape factor. It has shown considerable improvement in yields of square ingots. A 45 kg ingot was solidified in this crucible.

Optimization of the solidification cycle has shown that as the height of the ingot is increased, the gradients at the bottom, as well as instrumentation control, have to be very precise for efficient solidification.

A new cutting head has been fabricated and assembled with the present drive unit of the FAST slicer. In addition to the salient features of rigidity and accurate alignment, the bladehead is lighter and larger to accommodate a 30 cm wide wirepack. Surface speeds of 500 ft/min were achieved with minimum vibration.

Encouraging results have been achieved with the new cutting head. High cutting rates and yields, 5.1 mils/min and 96%, respectively, have been seen from electroplated bladepacks. Electroplated wires with diamonds in cutting edge only have been used during the present reporting period with good slicing performance. The impregnated wirepacks have also demonstrated cutting effectiveness with the new bladehead.

SILICON INGOT CASTING--HEAT EXCHANGER METHOD (HEM)

The effort on scaling up the size of the ingot as well as on improving the square shape and crystallinity of the ingot was continued during this quarter. Significant progress has been achieved. A new square crucible was developed which has a better shape factor. These crucibles will give yield of the cast ingot similar to that achieved by welding flat plates. An ingot of 32 cm x 32 cm cross-section weighing 45 kg has been cast using such a crucible. Details of experiments are shown in Table I.

Casting of 34 cm x 34 cm Ingots

During the previous quarter[1] some cracking was observed during the heat treatment of 34 cm x 34 cm x 40 cm crucibles prior to use in the HEM furnace. These crucibles are very large as compared with the previously used crucibles. The heat treatment is necessary to produce a graded structure that prevents cracking of the ingot during the cooldown cycle. It was demonstrated that high gradients during the heat treatment aid in the delamination of the crucible from the ingot; however, these conditions cause stresses in the crucible and cause cracking. The cooldown procedure was changed to reduce stresses so that cracking was prevented prior to the use of the crucible in the HEM furnace. A crucible using this procedure was used in run 41-25C. The heat treatment was carried out in such

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	DECREASE OF H.E. TEMP. °C/HR.	DECREASE OF FURN. TEMP. °C	GROWTH TIME IN HOURS	
41-22C	Effect of solidification parameters	12	-	-	12	12.5	Single crystal growth not achieved throughout the height of ingot
41-23C	Effect of solidification parameters	4	-	-	3	32.0	Single crystal growth achieved to the top surface of ingot
41-24C	Use shallow gradients at bottom	25	-	-	27	21.5	Freezing occurred on top surface
41-25C	Cast 34 cm x 34 cm, 45 kg ingot	17	-	-	17	36.0	No solidification problems. Slight cracking near top periphery.
41-26C	Cast 34 cm x 34 cm, 45 kg ingot	-	-	-	-	-	Run terminated during melt-down. Cracking in a corner of the crucible.
41-27	Improve growth cycle	32	-	-	40	36.5	No significant improvement in solidification time.
41-28	Cast 34x34cm^2, 45 kg ingot	39	-	-	40	42	Good solid ingot
41-29	Cast 34x34cm^2, 45 kg ingot. Test 2"Ø heat exchanger.	-	-	-	-	-	Run aborted during meltdown.

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	DECREASE OF H.E. TEMP. °C/HR.	DECREASE OF FURN. TEMP. °C	GROWTH TIME IN HOURS	
41-30C	Test 32 cm x 32 cm crucible with 45 kg charge	-	-	-	-	-	Run aborted during meltdown.
41-31C	Test 32 cm x 32 cm crucible with 35 kg charge	9	-	-	9	28	Good solid ingot, flat sides
41-32C	Test welded crucible	10	-	-	12	5.5	
41-33C	Test 32 cm x 32 cm crucible with 45 kg charge	-	-	-	-	-	Run aborted during meltdown.
41-34C	Test 32 cm x 32 cm crucible with 45 kg charge	16	-	-	20	31	Good solid ingot, flat sides.
41-35C	Test 23 cm x 23 cm crucible with 15 kg charge	14	-	-	18	10	Freezing occurred on top surface
41-36C	Test 23 cm x 23 cm crucible with 15 kg charge	31	-	-	36	12	Food solid ingot, flat sides.

a manner that low gradients occurred near the top while higher gradients were used for the rest of the crucible. A 45 kg charge was solidified in this run. No problems of containment of the melt and solidification were encountered. The 45 kg, 34 cm x 34 cm x 20 cm ingot cast is shown in Figure 1. Crucible delamination was very good except near the top of the ingot where some attachment was observed leading to some cracking. This is the largest silicon ingot solidified.

In run 41-26C, the above experiment was duplicated. Cracking in a corner of the crucible during meltdown necessitated aborting the run. This procedure, however, was repeated in run 41-28C and a 45 kg ingot was solidified. No problems were encountered during the meltdown, growth, and cooldown cycles. Complete delamination of the crucible was achieved. A view of the ingot is shown in Figure 2. The ingot is laid upside down after removal from the crucible to show relationship of the heat exchanger surface to the area of the ingot. A 1-inch diameter heat exchanger was used.

In run 41-29 another ingot of 45 kg and with 34 cm x 34 cm cross-section was attempted. A 2-inch diameter heat exchanger was used in this run to achieve faster growth rates. The experiment was aborted due to sudden change in vacuum during meltdown.

In run 41-30 a new design of the crucible with a squarer shape was used. A 45 kg load was put in the crucible. The experiment was aborted during the meltdown cycle because of cracking of the crucible. This crucible was supported better than before. It is felt that the problem could be high point loading due to the

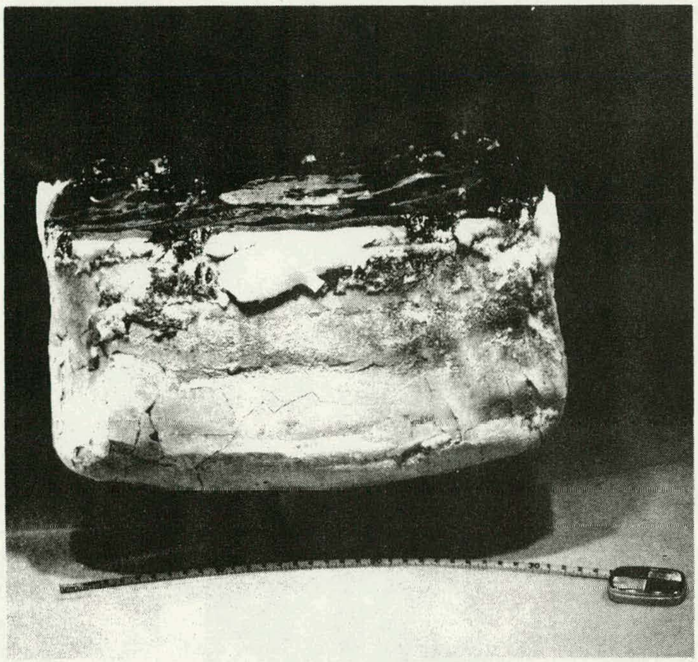
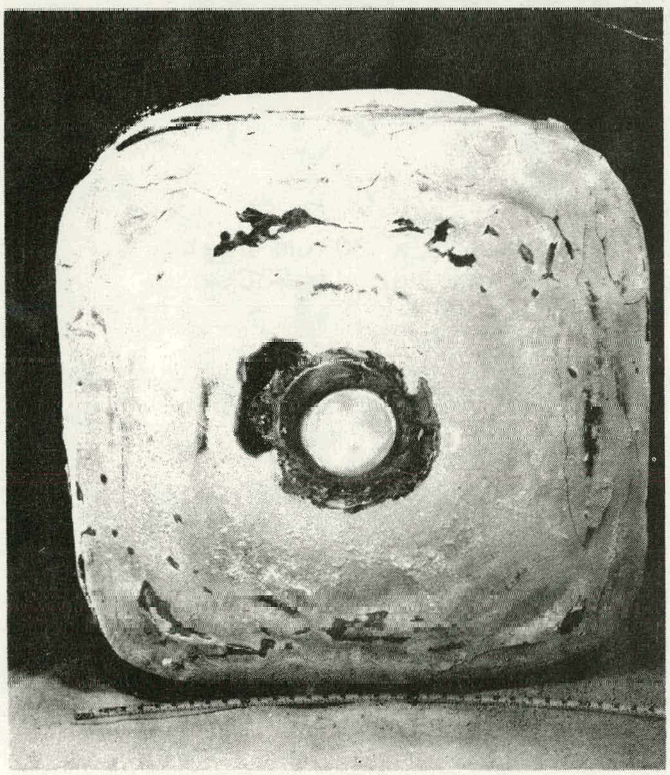
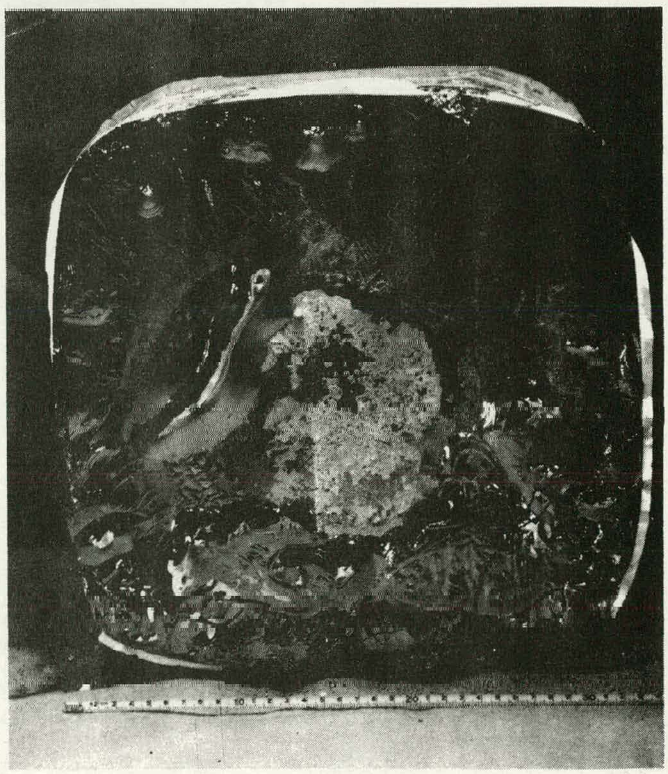


Figure 1. Three views of the 34 cm x 34 cm x 20 cm, 45 kg ingot cast in run 41-25C; (a) side view showing some attachment of crucible near top surface; (b) bottom showing no delamination problems; and (c) top showing minor cracking along one side where crucible attached.

(a)



(b)



(c)

Figure 2. A view of the 34 cm x 34 cm x 20 cm high,
45 kg ingot cast by HEM in run 41-28C.

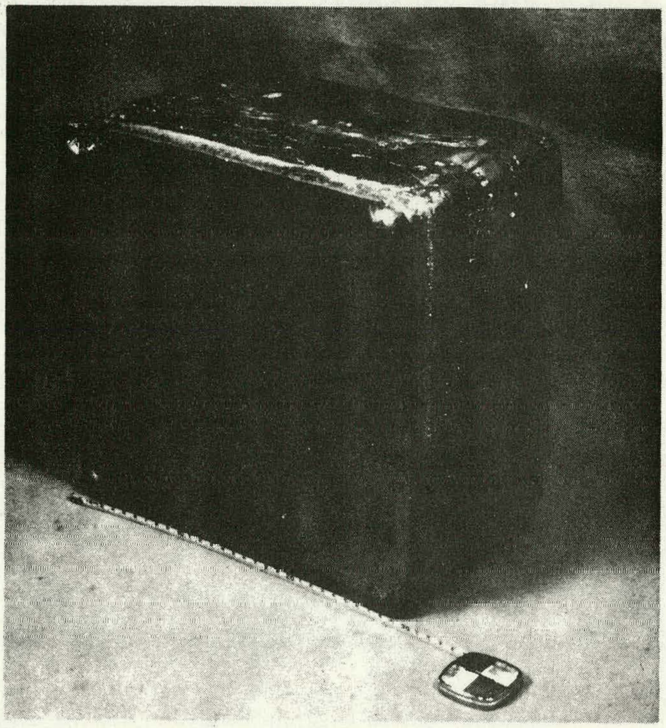
heavy load of pieces and expansion of meltstock due to heating. This may cause stresses in the weak, ceramic crucible.

An important change was made to support the crucible in run 41-31. The crucible was supported by a modified graphite box. This box provides better support to the sides of the crucible. A very good 35 kg ingot with flat sides was cast (Figure 3). A 45 kg ingot was solidified in a similar manner in run 41-34 and once again a good ingot with flat sides was produced (Figure 4). These ingots were cast in crucibles of new design. As is evident from Figures 3 and 4, the ingots have square corners and flat sides. This will provide significantly higher yields of square wafers and thereby reduce costs.

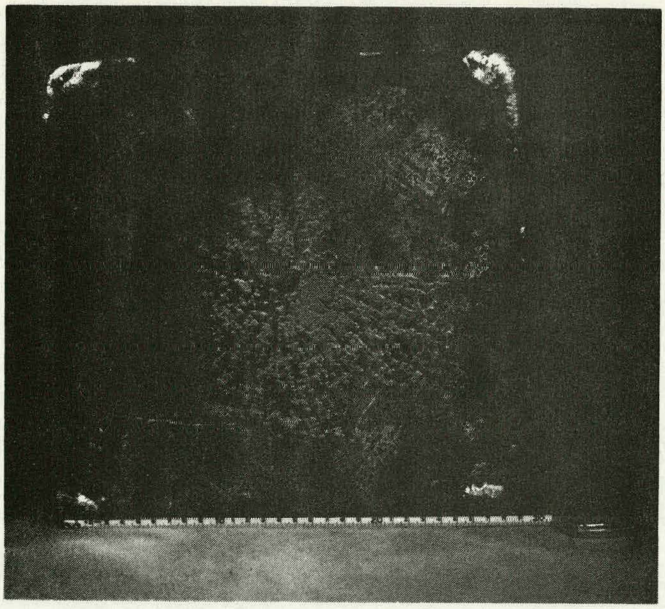
The square crucibles with improved shape factor were also fabricated in 23 cm x 23 cm size. Figure 5 shows an ingot cast in such a crucible. The flat sides and bottom of the crucibles improves yields as well as allows a better support by the graphite retainer box. The crucible can then conform to the shape of the rigid box at high temperatures and improve the square shape. When proper support is not provided by the retainers, the crucible bulges in certain areas thereby deforming the square shape of the ingot.

Crucible Development

The crucibles used in HEM solidification are slip cast and the inside surface is heat treated to develop a graded structure. These crucibles are weak and a 45 kg load appears to provide a



(a)



(b)

Figure 3. Two views of 32 cm x 32 cm cross-section, 35 kg ingot cast in the improved design crucible with better shape factor: (a) an isometric view; (b) the top surface of ingot showing last material to freeze is in the corner.

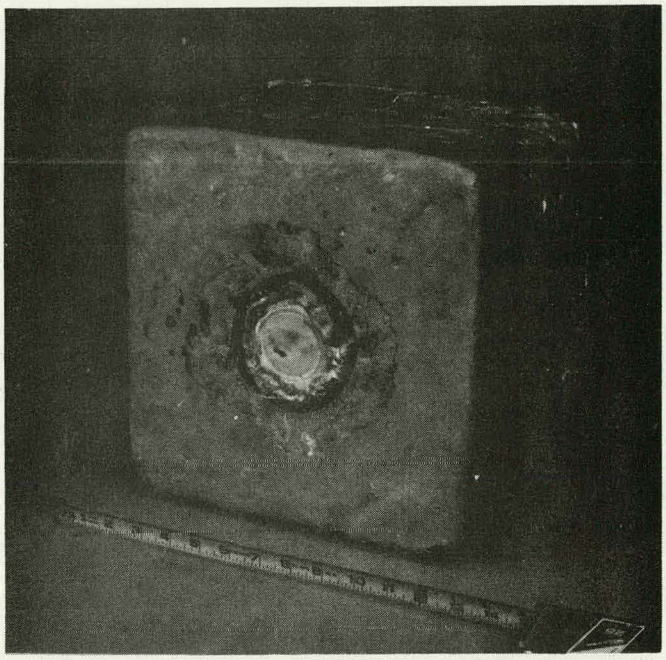


Figure 4. A 45 kg ingot cast in run 41-34C.

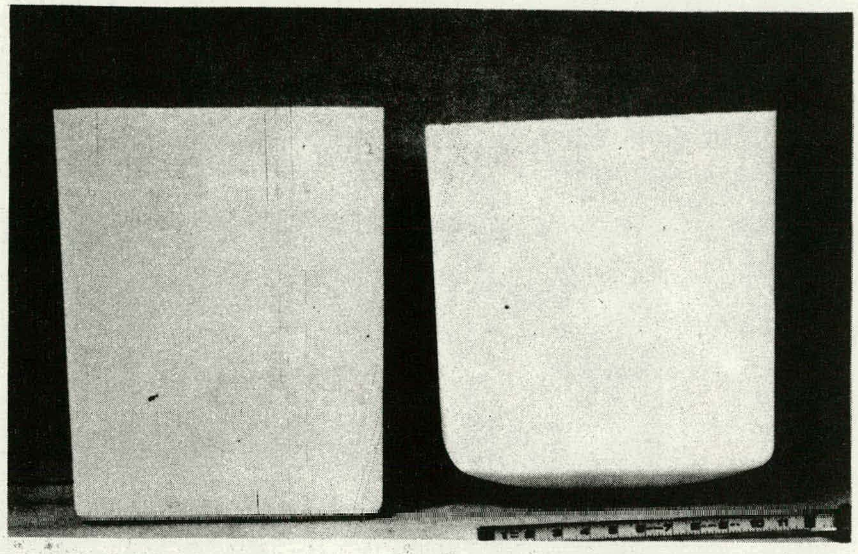
Figure 5. A 23 cm x 23 cm cross-section ingot cast in run 41-36C.

heavy loading. Further, the bottom of the crucible is contoured and the sides have a curvature and taper for ease in slip casting. Under heavy loads the crucible needs to be supported properly. A graphite plate with a contour similar to the bottom of the crucible has shown that a good support is provided in this area. The sides are supported with flat graphite plates forming a box around the crucible. Because of the taper in the crucible this graphite box supports the area near the top of the crucible; in this area the loading is less than the lower part of the crucible. Furthermore, the shape factor of the crucible is such that poor yields of square material are achieved from these squarish crucibles. This has a significant impact on the economics of the process.

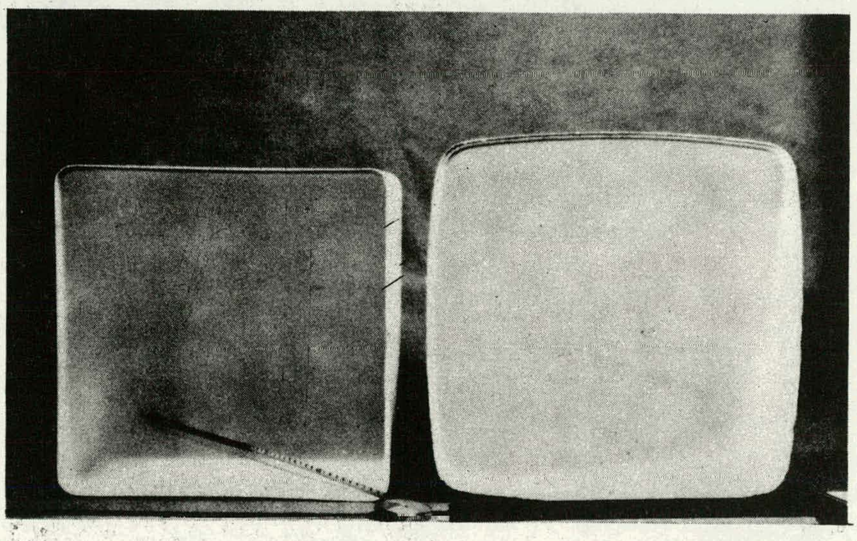
In view of the above a square crucible was developed. This crucible has a flat bottom with no curvature and minimal taper on the sides; the corners have a smaller radius. This crucible (shown in Figure 6 in comparison with its predecessor) is expected to produce squarer ingots and higher yields.

Process Optimization

The effect of solidification parameters was studied on 20 cm cube ingots in runs 41-22C and 41-23C. In the former experiment the melt temperature was reduced rapidly and the solidification time was shortened. Single crystal growth and directional solidification was not achieved to the top surface. In run 41-23C the melt was kept at a low superheat. Single crystal growth was achieved all the way to the top surface (Figure 7); however, the growth time was very long. A slight change in the process parameters prolongs the solidification time considerably. The gradients at the bottom have to be such that they aid extraction of heat by the



(a)



(b)

Figure 6. Views of the two crucibles used to cast 30 cm x 30 cm cross-section ingots. The crucible on the left is the new design (a) view of the side, (b) view of cross-section.

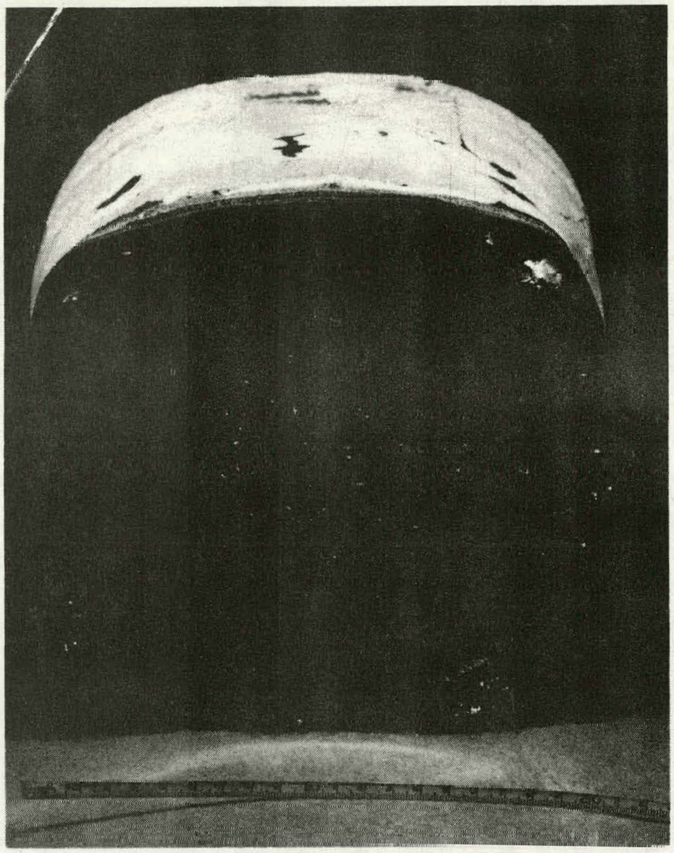


Figure 7. A view of the top surface of the ingot cast in run 41-23C.

heat exchanger. This becomes more important when the height of the crystal is increased.

In run 41-24C very shallow gradients were used at the bottom of the ingot. During this experiment the effect of the heat exchanger was minimized and the melt could only be solidified by dropping the furnace temperature below the melt point of silicon. Thus solidification at the top of the ingot was by freezing over rather than directional solidification by the heat exchanger. Very large grains were formed in this area. This ingot has been sent to JPL for solar cell evaluation. It is intended to use samples from this ingot to study the effect of grain size and grain boundaries on solar cell performance.

These studies have led to changes in heat flow at the bottom of the crucible towards optimization of the solidification.

MULTI-WIRE SLICING--

FIXED ABRASIVE SLICING TECHNIQUE (FAST)

During the third quarter emphasis was placed on machine and blade development for FAST. A new bladehead has been installed. The main features of the modification are higher speed, more rigidity, accurate alignment and larger capacity. Through the experiments carried out using this new bladehead, encouraging results were obtained by way of high cutting rates and improved yields.

Machine Development

The slicer feed mechanism was not rigid; therefore, considerable vibration was transmitted to the workpiece which contributes to wafer breakage during slicing. In addition, misalignment caused wire wander which resulted in poor yields. Vibration was reduced by reciprocating the bladehead at lower speeds. However, this affected adversely the cutting rate and wire life.

A new slicing head was designed and fabricated. The salient-features of this bladehead are a very high degree of rigidity and accurate alignment. A smooth granite faceplate is used as a base on which the wirepack frame is mounted. This granite surface is used to align the reciprocating frame as well as the workpiece carriage, vertical feed and rocking assembly. Misalignments, if any, can also be checked easily from time to time. The heavy granite block also gives the bladehead rigidity. The support of the vertical feed has been designed for rigidity. The reciprocating wirepack frame rides in a ball slide which gives it a very accurate alignment during travel. The rocking mechanism is driven with an anti-backlash lead screw. A synchronous stepping motor powers the unit so that the rocking angle and profile can be changed.

In addition to rigidity and accurate alignment features of the bladehead, the wirepack frame has been made lighter to achieve higher speeds, and it has been enlarged to accommodate 750 wires (25 wires/cm). The rocking angle has also been increased. A view of the new bladehead is shown in Figure 8.

Initial testing with this new bladehead has shown that speeds as high as 500 feet per minute can be achieved and this is limited by the drive unit rather than the bladehead. After debugging the machine, all components appeared to perform satisfactorily.

Blade Development

During the last quarter[1] it was reported that it is possible to electroplate diamonds selectively over the circumference of the wire. A wirepack in which diamonds were electroplated over less than half the circumference of wire was used in run 426-SX. During the slicing run unusually high vibrations were transmitted to the

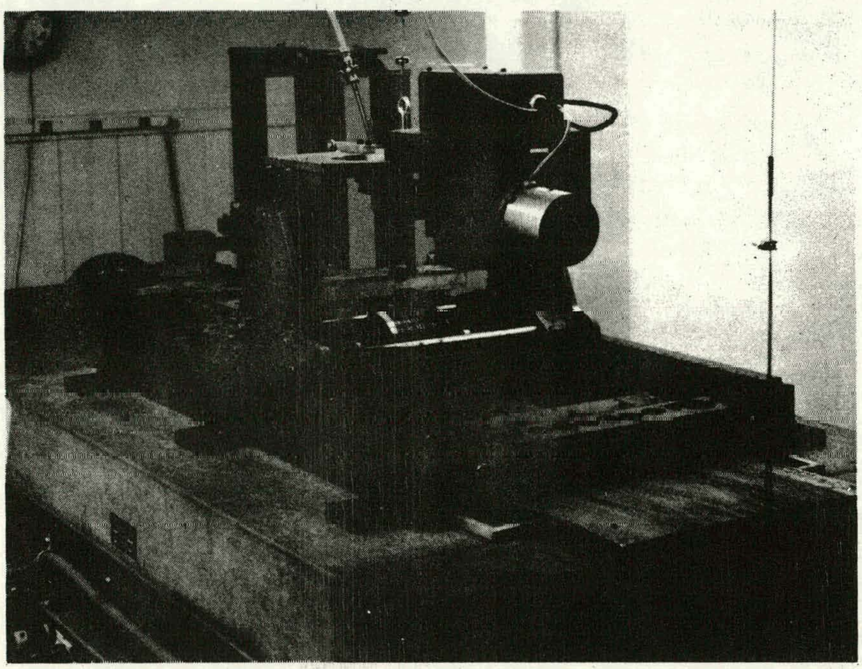
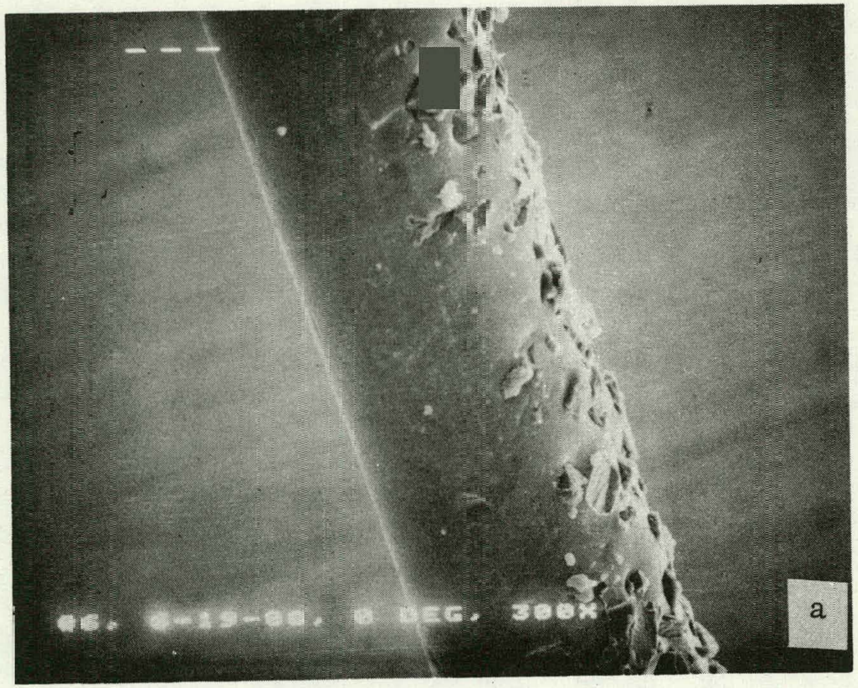


Figure 8. FAST slicer showing new bladehead

workpiece which resulted in only 53% yield. SEM examination of a wire (Figure 9) showed that the diamonds were not even enough to clear the wires through the workpiece. The wires were, therefore, rubbing against the crystal; abrasion marks are visible in Figure 9. This same wirepack was used again in run 429-SX to see if the abrasion during the first slicing test was enough to clear the wires. Vibrations to the workpiece were still being observed; hence, low feed forces were used. As a result, low cutting rates were achieved. Slicing during the third life test of these wires (run 430-SX) improved the cutting rates when the feed forces were restored to the usual value. These runs and the rest of the experiments that were carried out in this quarter are shown in Table II.

The effort of electroplating diamonds on one side was continued for another wirepack. It was found that when selective electroplating is carried out, some diamonds, even though they are in very low concentration, still get plated on the top surface of the wires. During the slicing test these diamonds cause perturbation for the support rollers. The top surface was masked during the plating operation to prevent diamonds from being plated on top. Considerable wire wander was observed when this wirepack was used in run 431-SX. Examination of these wires under a microscope showed that masking during plating formed a flat surface on the top of the wires. During the slicing operation this flat surface was not seating in the grooved rollers and caused severe wire wander.



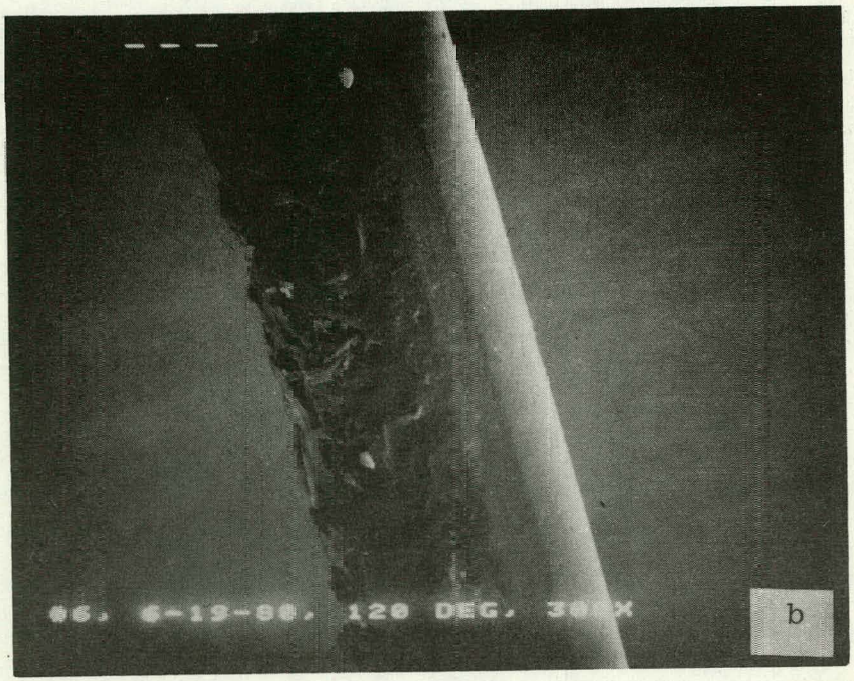


Figure 9. SEM photographs of wire used in run 426-SX (300X)

TABLE II. SILICON SLICING SUMMARY

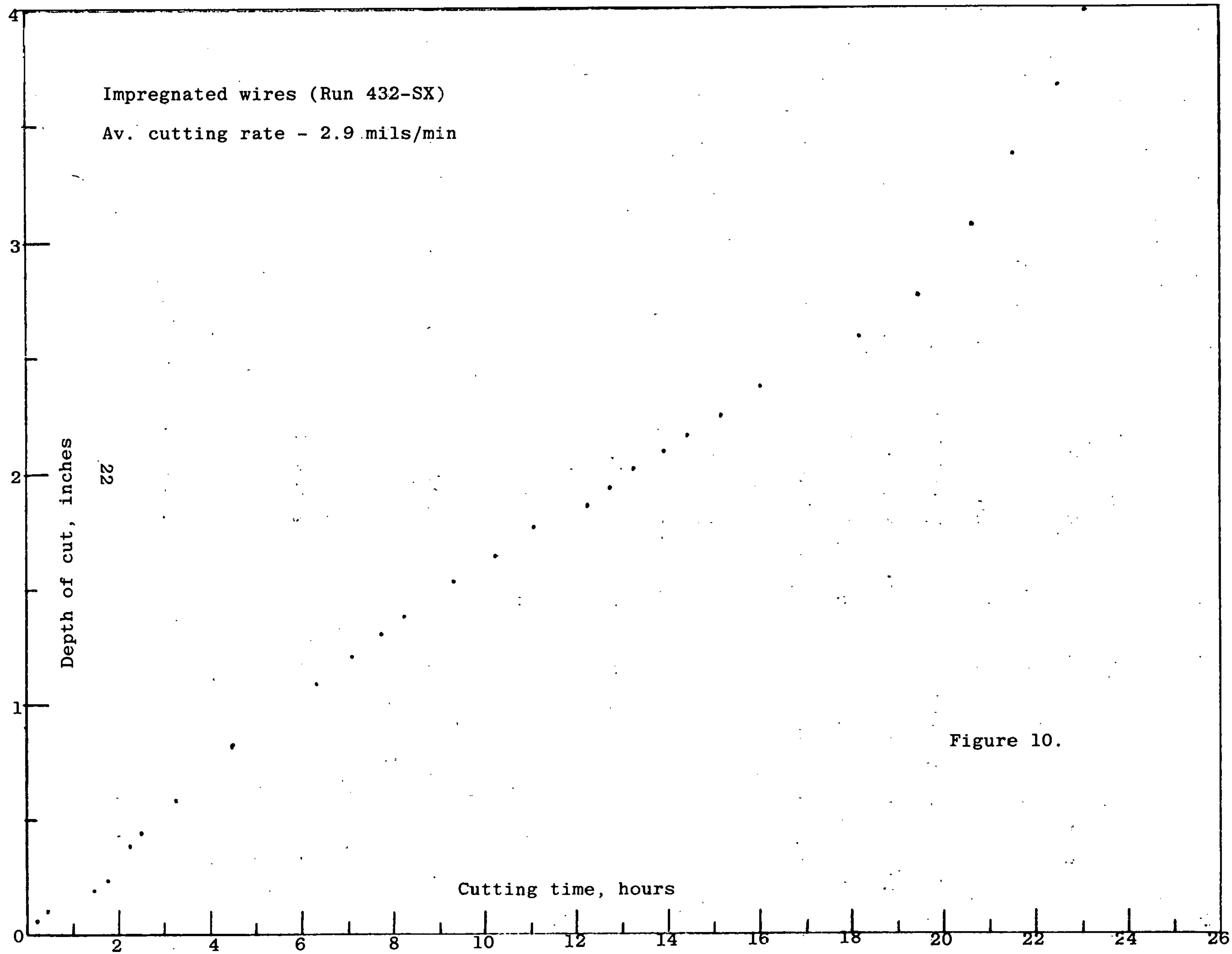
RUN	PURPOSE	FEED FORCE/BLADE lb	FEED FORCE/BLADE gm	AVERAGE CUTTING RATE mil/min	AVERAGE CUTTING RATE mm/min	WIRE TYPE	REMARKS
429-SX	Life test (2nd run) to test codeposited bladepack	0.062	27.28	1.3	0.032	Same as 426-SX (5 mil, 0.125 mm wire codeposited with 45, 30, 15 μm diamonds)	33% yield. Diamonds on wire not sufficient to clear wires. Low feed forces used to minimize vibrations from abrasion.
430-SX	Life test (3rd run)	0.072	32.94	2.3	0.052	Same as 426-SX	27% yield. Higher cutting rates achieved when feed forces were restored.
431-SX	Test new bladehead	0.054	24.56	-	-	5 mil, 0.125 mm W wire codeposited with 45, 30, 15 μm diamonds	Significant improvements because of rigidity and accurate alignment of new bladehead
432-SX	Test CSI impregnated wire	0.071	32.55	2.9	0.072	5 mil, 0.125 mm stainless steel core; 1.5 mil, 0.0375 mm copper sheath, 45 μm diamonds, electroless nickel	75% yield; loss of wafers during last inch of cut.
433-SX	Test codeposited bladepack	0.071	32.55	5.1	0.127	5 mil, 0.125 W wire, codeposited with 45, 30, 15 μm diamonds	83% yield with good surface quality
434-SX	Life test	0.073	33.34	3.3	0.082	Same as 433-SX	53% yield

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
435-SX	Life test (3d run)	0.073	33.68	3.0	0.075	Same as 433-SX	43% yield.
436-SX	Test CSI impregnated wire	0.071	32.16	2.7	0.067	5 mil, 0.125 mm stainless steel core; 1.5 mil, 0.0375 mm copper sheath, 60 µm diamonds, electroless nickel	40% yield.
437-SX	Test electroplated bladepack	0.054	24.85	4.0	0.100	5 mil, 0.125 mm W wire codeposited with 45, 30, 15 µm diamonds	52% yield.
438-SX	Life test (2nd run)	0.060	27.18	5.0	0.125	Same as 437-SX	37% yield.
439-SX	Test electroplated bladepack	0.054	24.70	4.0	0.100	5 mil, 0.125 mm W wire codeposited with 45, 30, 15 µm diamonds	96% yield.
440-SX	Life test (2nd run)	0.060	27.62	3.8	0.095	Same as 439-SX	48% yield.

In run 432-SX an impregnated bladepack was used. The wires were 5 mil (0.125 mm) core with a 1.5 mil (0.037 mm) copper sheath, impregnated with 45 μm diamonds. An electroless nickel thickness of 0.3 mil (7.5 μm) was coated to prevent diamond pullout. In this run high yields (75%) and high cutting rates (2.9 mil/min) were achieved in comparison with the previously used impregnated wires. This was the first slicing test with the new bladehead. During the experiment two problems were encountered, *viz.*, the rocking mechanism stopped for short periods and the feed was held up towards the end of the cut. The rocking mechanism circuitry was corrected for this malfunction. The feed mechanism was held up during the last one inch of the cut by hitting against a bolt-head. Under this situation the wire blades were rubbing rather than cutting and most of the diamonds were pulled out. The bolt-head was removed and the experiment continued. Most of the breakage of wafers occurred during this portion of the experiment. In spite of this problem, high yields and cutting rates were achieved. A plot of depth of cut vs. cutting time is shown in Figure 10. Same type of wire was used in run 436-SX except that 60 μm size diamonds were impregnated.

An electroplated wirepack was used in runs 433-SX, 434-SX and 435-SX. A mixture of diamonds, 45, 30, 15 μm, was electrodeposited on a 5 mil, 0.125 mm core wire. The diamonds were fixed mostly in the cutting edge.[1] Some diamonds were plated on the top surface as well. In the first run a very high yield, 83%, and cutting rate, 5.1 mil/min, were obtained. In the second run the cutting rate was still high, 3.3 mil/min. The yield, however, dropped to 53%. After



Impregnated wires (Run 432-SX)
Av. cutting rate - 2.9 mils/min
Depth of cut, inches
Cutting time, hours
0
1
2
3
4
2
4
6
8
10
12
14
16
18
20
22
24
26

Figure 10.

the first slicing test diamonds in the cutting edge may become dulled as compared with diamonds on the sides, thereby causing wire wander and, therefore, poor yields. Measurements of average wafer thickness and kerf were also made. In the first run these values were 10.6 and 10.2 mils, respectively. In the second run, however, 12.3 mils average wafer thickness and 8.5 mils average kerf were found. This is not conclusive proof that diamonds on the sides of the wire were pulled out because after electroplating there are always some diamonds which are not strongly bonded to the matrix and they fall out during the first slicing test. SEM examination of these wires is in progress. A plot of the data for the three runs is shown in Figure 11. A big improvement in slicing performance has been seen with the new bladehead. A compilation of the data from an earlier electroplated wirepack on the old bladehead is shown in Figure 12. A comparison of these two figures shows that the slicing rates are very much enhanced. This should also improve the life of the wirepacks.

More experiments have been conducted on the new bladehead, runs 437-SX, 438-SX, 439-SX and 440-SX. A very high yield was obtained in run 439-SX. Low yields in run 437-SX are attributed to crystal loosening from the holder during the run.

All these experiments indicate that FAST slicing with the new bladehead is improved. Cutting rates of the order of 4 to 5 mils/min and yield of 80 to 90 per cent have been achieved. The main problems are associated with poor and inconsistent quality of wirepack.

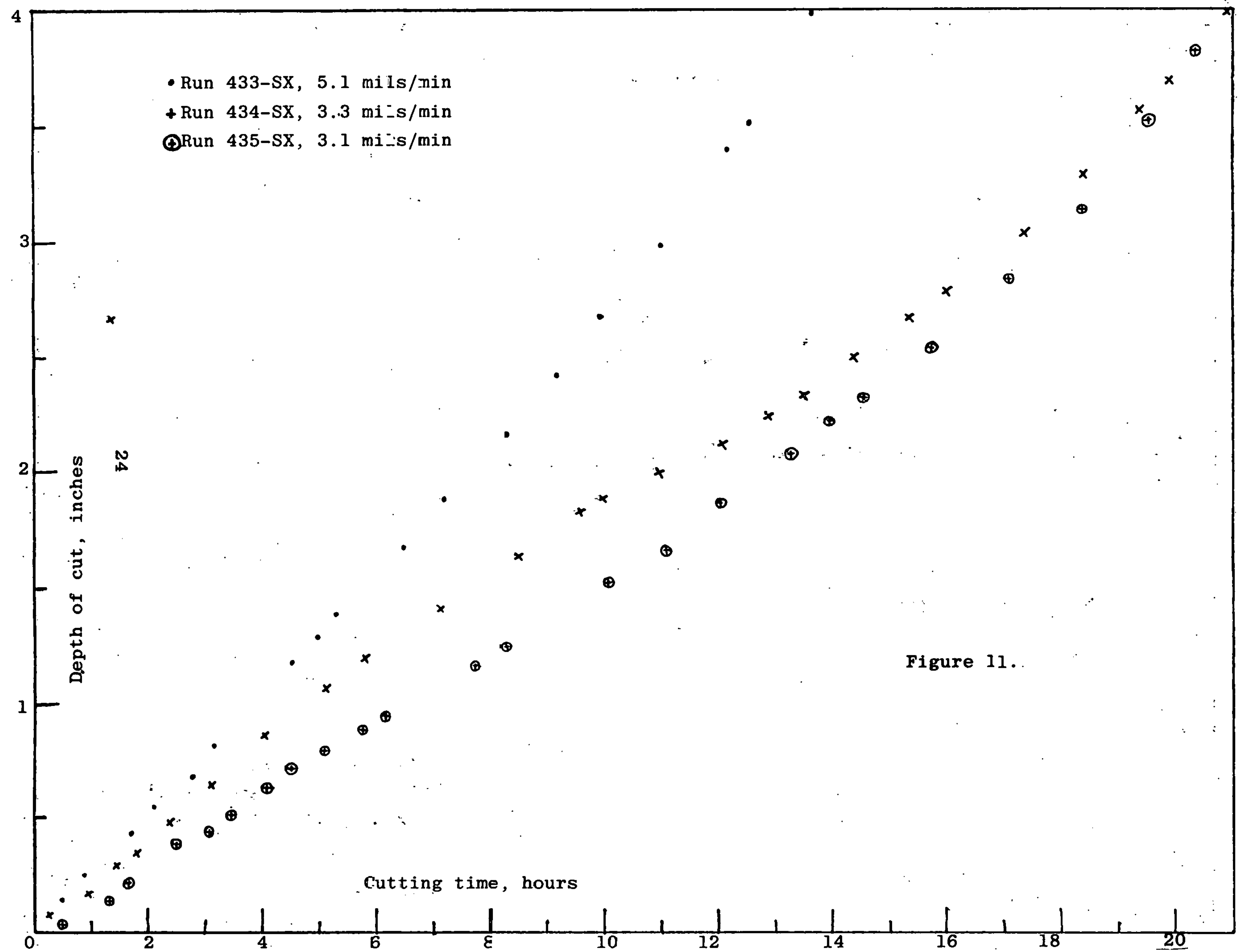

• Run 433-SX, 5.1 mils/min
+ Run 434-SX, 3.3 mils/min
⊕ Run 435-SX, 3.1 mils/min
Depth of cut, inches
Cutting time, hours
0
1
2
3
4
6
8
10
12
14
16
18
20

Figure 11.

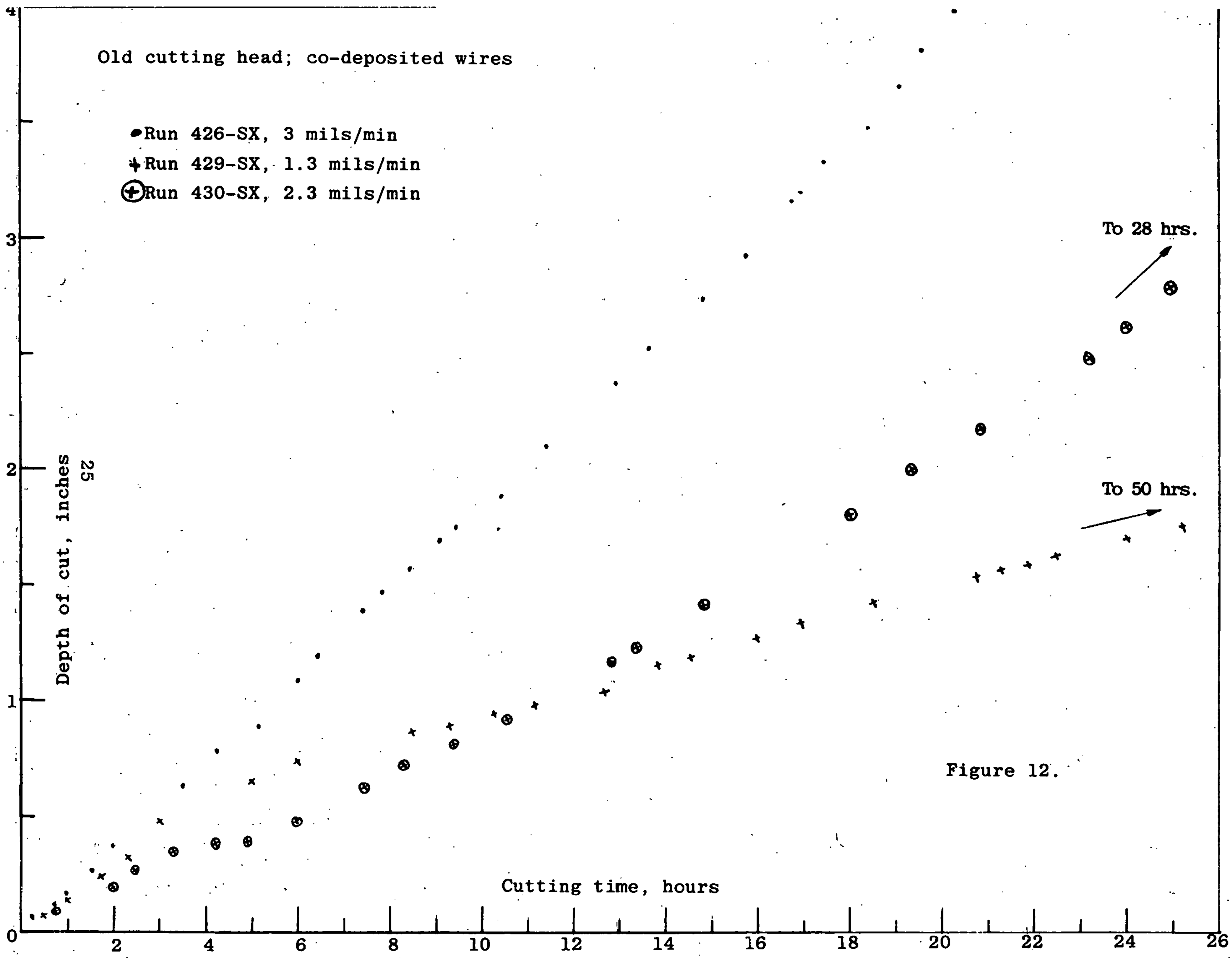

Old cutting head; co-deposited wires
Run 426-SX, 3 mils/min
Run 429-SX, 1.3 mils/min
Run 430-SX, 2.3 mils/min
To 28 hrs.
To 50 hrs.
Depth of cut, inches
Cutting time, hours
0
1
2
3
4
2
4
6
8
10
12
14
16
18
20
22
24
26

Figure 12.

SUMMARY

1. A new crucible of 32 cm x 32 cm cross-section has been developed which has a much better shape factor. This has improved yields of the cast ingot.
2. Forty-five kg crack-free ingots of 34 cm x 34 cm cross-section were solidified.
3. When the height of the ingot is increased, high throughput is more difficult to achieve.
4. A new bladehead has been fabricated and installed on the FAST slicer with more rigidity and accuracy and over 500 surface feet per minute have been achieved.
5. Masking of the top surface to prevent diamond electroplating forms a flat which causes wire wander during slicing.
6. A 75% yield and 2.0 mils/min cutting rate was achieved with CSI impregnated wires.
7. Yield and cutting rate were higher in codeposited bladepacks, *i.e.*, up to 96% and 5.1 mils/min, respectively.
8. In the second run of codeposited bladepack, the cutting rate, 4 mils/min, was higher than the previously conducted second runs.

REFERENCE

1. F. Schmid, C. P. Khattak, and M. Basaran, "Silicon Ingot Casting--Heat Exchanger Method (HEM)/Multi-Wire Slicing--Fixed Abrasive Slicing Technique (FAST), Phase IV," DOE/JPL 954373, Crystal Systems, Inc., Quarterly Progresss Report No. 2, July 1980.

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