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SLICING OF SILICON INTO SHEET MATERIAL

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

TWELFTH QUARTERLY REPORT

By

J. R. FLEMING

DOE/JPL 954374-78/1

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By

J. R. FLEMING

Reporting Period December 30, 1978 to March 30, 1979

JPL Contract No. 954374

Varian Associates Lexington Vacuum Division 121 Hartwell Avenue Lexington, Massachusetts 02173

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Test Summary Program Plan (Updated)

1.0 SUMMARY

Testing of low-cost low-suspension power slurry vehicles is continuing. Results to date indicate that commercial cutting oils are unlikely to work, but a mineral oil with additives should be workable. Further testing to select the proper additives is necessary. Water based vehicle now appears possible, but again there are incidental problems and much testing may be required.

Two different abrasives were tested. A cheaper silicon carbide from Norton gave excellent results except for excessive kerf loss: apparently the particles are too big. An abrasive treated for lubricity showed no lubricity improvement in mineral oil vehicle.

The bounce fixture was tested for the first time under constant cut rate conditions (rather than constant force). Although the cut was not completed before the blades broke, the blade lifetime of thin (100 μ m) blades was 120 times the lifetime without the fixture.

The large prototype saw has completed a very successful run, producing 90% cutting yield (849 wafers) at 20 wafers/cm. Although inexperience with large numbers of wafers caused cleaning breakage to reduce this yield to 74%, the yield was high enough that we regard the concept of the large saw to be proven workable.

For the first time we have obtained recycled abrasive from used slurry and will test it shortly.

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2.0 PROGRESS

2.1 Slurry Tests

Previous testing of mineral oil slurry vehicle has shown that drag forces are a major problem, cutting times may be made reasonable by proper choice of conditions, excellent wafers can be produced, and if drag forces are sufficiently reduced by addition of sufficient lard oil, then the only problem left is low yield due to wafer breakage near the end of the run for unknown reasons.

It seems reasonable that some characteristic of the lard oil may be responsible for the wafer breakage. Thus, we decided to try additives different from lard oil, namely commercial cutting oils and cutting oil additives which do not use lard oil. The price was not a consideration in this series, since we have found that oils with little suspension power are easily recycled by one to two days settling, and if a workable oil proved too expensive, we would at least have a good starting point from which to develop low-cost low-suspension power slurry vehicles.

We consulted the White & Bagley Company of Worcester, MA and picked three test vehicles. W & B cutting oil #1 is a low priced, general purpose cutting oil. W & B cutting oil #2698 is a medium cost, very high sulfur-chlorine-fat content oil for hard to machine materials. Both oils are thin, on the order of 100-200 SUS. W & B HD soluble oil 2213 is an all-purpose extreme pressure additive for oil or water, containing no sulfur or fat but with a high chlorine content.

Test #2-3-29 was run using a vehicle of W & B cutting oil #1. All other conditions were standard. During the first quarter of the cut, fuses blew regularly and the saw could not be run over 60 RPM (60% of standard speed). We terminated the test and will not use W & B cutting oil #1 again as there seems to be no promise of making it work.

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Test #2-3-32 was run using a vehicle made up of 85% by volume 100 SUS mineral oil and 15% by volume W & B #2213. All other conditions were standard.

The initial cutting rate was low, about 70% of the usual rate with PC oil slurry. One fuse blew during the first day of running, and speed was decreased to 80 RPM. On the second day of cutting, about 1/8 of the way through the ingot, it proved impossible to run the saw over 30 RPM without blowing fuses and the run was stopped. Again, since insufficient lubricity was obtained at the highest recommended concentration, and also since it seemed that some component had evaporated or settled out causing higher drag than with 100 SUS mineral oil along, we will not investigate this system further.

Test #2-3-31 was run, again using standard conditions, but using W & B cutting oil #2698. Results were identical to results of Test #2-3-29: blown fuses and inability to run the machine at full speed.

Our conclusions are that we have not yet found the proper mineral oil system, but such a system is workable. Further research is necessary, combining careful consideration of the necessary properties with judicious selection of additives for experimentation. It is unlikely that commercial cutting oils will prove suitable, in view of the results of Tests #2-3-29 and #2-3-31. The workable system will consist of mineral oil and a carefully selected one or two additive package.

A previous test (#2-3-21) tested a silicon carbide produced by the Norton Company which they claimed used a cheaper production process than currently available processes. The abrasive was labelled #500, but Norton claimed that it was the equivalent of the #600 from Micro Abrasives which we have been using. The test showed the new abrasive to perform well, but the kerf loss indicated that the particle size was too high.

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At our request, Norton produced a new sample with smaller particle size. This sample, designated MCA 1632 by Norton, was tested in run #2-3-30. All conditions except the identity of the abrasive were standard.

The results were essentially the same as in Test #2-3-21. Cutting time was 30 hours, yield was 99%, taper was 51 µm (.002 in.), and bow was 44 µm (.0015 in.). All these results are quite good. Unfortunately, the abrasive kerf loss was 98 µm (0.004 in.) rather than the 60 µm (0.0024 in.) expected with #600 abrasive. We will continue to work with Norton on cheaper abrasives, although the major cost reduction is expected to come from recycling.

Test #2-3-36 was run using an unusual abrasive. The Mosher Company, a local manufacturer and distributor of lapping equipment and supplies, provided a sample of Micro Abrasives #600 silicon carbide (our standard abrasive) which they had treated using a proprietary process to provide lubricity when suspended in oil. They claimed we could use this abrasive with straight mineral oil (100 SUS).

Unfortunately, this did not work. Even at 80% of standard reciprocation speed, fuses blew regularly from the beginning. We terminated the run after 1/16 of the cut, and concluded that the treated abrasive offered no improvement over the untreated abrasive in straight mineral oil.

A sample of VCI-309 anodic-cathodic-vapor phase corrosion inhibitor was delivered to Dr. Paul Tung of JPL, along with blade samples, for fatigue testing. Dr. Tung has reported that blades tested in distilled water broke "very quickly" but the spread was large; blades tested in 5 wt.% VCI-309 lasted more than 10^6 cycles (3 tests); and one blade tested in 1 wt.% VCI-309 lasted more than 10^6 cycles. If cycles in Dr. Tung's tests correspond to load cycles in the saw, these lifetimes correspond to 84 hours of cutting, which is much more than required for even two cuts through a 100 mm diameter ingot.

In light of these promising results, we ran Test #2-3-25 using a distilled water slurry vehicle containing 5% (by weight) of Cortec VCI-309. (5% is the maximum recommended concentration.) The results of the tests were promising, but not as good as hoped. The total running time was 21 hours, including three night shutdowns. One blade broke at 5 hours, 40 minutes; one blade broke at 9 hours, 5 minutes; and several blades broke between 13 hours and 21 hours. The vehicle tended to form a stable foam, which caked on the saw. After 21 hours, the cut had only progressed 25 mm (1 inch) into the work, and all the abrasive was trapped in dried foam. In view of the clogging of the machine, we shut down the run.

The fact that all but a few blades lasted at least 21 hours is heartening. Still, an acceptable water based vehicle must allow minimum blade lifetimes longer than this. One problem in testing is that the statistics are extreme rather than mean value statistics (i.e., we are interested in the lower tail of the blade lifetime distribution rather than the average). This makes it difficult to predict saw performance on the basis of relatively few laboratory tests.

Still, the performance of VCI-309 is sufficiently interesting that we will run another test using a defoaming agent.

2.2 Miscellaneous Techniques Tests

The 686 bounce fixture was tested for the first time. This is a low-mass, low-spring-constant support for the workpiece in a 686 which reduces the shock due to vertical workpiece motion at the end of each stroke. It includes an electric motor feed replacing the air cylinder, which tended to stick when isolated from the bounce. The system is designed to operate in closed loop control, at a constant cutting force. Initial tests must be conducted using constant cutting rate, since the cabinets for the electronics (on order for six months) have not yet arrived.

Test #2-5-21 was run to test the bounce fixture. At the request of JPL, we used a 100 μ m (0.004 in.) thick blade and 300 μ m (0.012 in.) thick spacer to cut 25 wafers/cm. The cut rate chosen was 0.64 μ m/sec (0.0015 in/min). All other conditions were standard.

From the beginning, the fixture rocked excessively with the stroke. Adjusting the cut rate (and, therefore, spring compression) made no difference. After 32 hours of cutting, most of the blades broke. They were worn to 38% of their original height. The blade wear was much more than expected. The cut depth was 57 mm, 57% of the full cut.

The fixture was a success in that the blade lifetime was significantly extended over that obtained without the fixture (.25 hour typical). The rocking must be eliminated. It is uncertain now whether the rocking was caused by insufficient pin diameter in the bushings or by an insufficiently tight press fit between the bushings and housing.

2.3 Prototype Tests

As discussed earlier, almost all our difficulties with the large prototype saw have been due to our inexperience with the saw and the techniques of using it. The only problems we have had with the mechanics and electronics are: 1. short bearing lifetime due to insufficient slurry shielding, 2. electronics failures due to the breadboard nature of construction, and 3. lack of and indication of end of stroke "bounce" so the operator had difficulty deciding when to shorten the stroke. The bearing lifetime problem has not yet been solved, although design and fabrication of components to resolve this problem has started. We have also started design and construction of a more reliable, better built electronic system. A bounce readout has been fabricated and installed, and the noise sensitivity has been decreased by careful grounding and shielding.

Test #2-7-06 was run as a test of the bounce readout device. The blade pack was our "baseline" 150 µm (.006 in.) blade and 350 µm (.014 in.) spacer, yielding 20 wafers/cm. 940 blades were easily extended to full elongation. All other conditions were standard.

Some minor mechanical and electrical problems were encountered during the run (e.g., slurry drain blockage), but none were serious enough to cause termination of the run. Cutting time was 39 hours, although this number is somewhat suspect because of the large number of starts and stops to fix minor problems. Very near the end of the run, two groups of wafers broke off near one end of the ingot, totaling 90 broken wafers. Thus, cutting yield was 90%. Solely since we are not experienced with such large numbers of wafers and do not have enough casettes to hold them all, cleaning breakage reduced the yield to a still respectable 74%. Average wafer thickness was 267 μ m (.0105 in.), taper was 124 μ m (0.005 in.) and bow was 155 μ m (0.006 in.)

Although the wafer thickness was somewhat low, the taper was somewhat high, and the bow was very high, we feel this run was very successful. The thickness bow and taper we attribute to the starting and stopping to fix minor problems. This run proves that the large saw is capable of producing high yield runs of 100 mm diameter silicon wafers, using baseline conditions, producing 20 wafers/cm.

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Since this run, we have strengthened the gear train in the stroke adjustment system and replaced some bearings. A run has been started which will cut 22 wafers/cm. This run is stopped right now since the main rods on which the workpiece carriage support bushings ride are worn, and these are difficult to replace even without the constraint of saving a partial run. We hope to be able to repair this system and complete the run in the next reporting period.

2.4 Other Progress

We have been investigating centrifugal separation of abrasive from used slurry. We have obtained a sample of almost pure abrasive separated from used PC oil slurry in a Barrett centrifuge. We will test this abrasive during the next reporting period.

3.0 PROBLEMS

No significant problems occurred during the reporting period.

4.0 PLANS

Plans for the next period include:

- Repair of the prototype and completion of the current test (22 wafers/cm).
- Tests of recycled abrasive.
- Modification and tests of the bounce fixture.
- Further mineral oil and water based slurry tests.

SLICING TEST SUMMARY

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PARAMETER TEST	2-3-29	2-3-30	2-3-31	2-3-32
Material	Si	Si	Si	Si
Size (mm)	100 Dia	100 Dia	100 Dia	100 Dia
Area/Slice (cm ²)	78.5	78.5	78.5	78.5
Blade Thickness (mm)	0.15	0.15	0.15	0.15
Spacer Thickness (mm)	0.36	0.36	0.36	0.36
Blade Height (mm)	6.35	6.35	6.35	6.35
Number of Blades	150	150	150	150
Load (gram/blade)	85	85	85	85
Sliding Speed (cm/sec)	64.2	64.2	64.2	64.2
Alrasive (type/grit size)	SiC/#600	SiC/#600	SiC/#600	SiC/#600
Oil Volume (liters)	6.3*	6.3	6.3*	6.3*
Mix (kg/liter)	0.36	0.36	0.36	0.36
Slice Thickness (mm)		0.260		
Kerf Width (mm)		0.248		
Abrasive Kerf Loss (mm)		0.098		
Cutting Time (hours)		30.33		
Efficiency (full test)		1.178		
(typical)		1.474		
(maximum)		2.317		
Abrasion Rate (fuli test)		0.064		
(cm ³ /hr/bl) (typical)		0.080		
(maximum)		0.126		
Productivity (full test)		2.590		
(cm ² /hr/bl) (typical)		3.220		
(maximum)		5.072		
Yield		99%		
Slice Taper (mm)		0.051		
Slice Bow (mm)		0.044		
Abrasive Utilization (cm ³ /kg)		129.03		
Oil Utilization (cm ³ /liter)		46.45		(
Blade Wear Ratio (cm ³ /cm ³).		0.040		
	*W&B #1	*Norton MCA	*W&B #2698	*100 SUS M. & W&B #221

SLICING TEST SUMMARY

PARAMETER TEST	2-3-35	2-3-36	2-5-21	2-7-06
Material Size (mm) Area/Slice (cm ²)	Si 100 Dia 78.5	Si 100 Dia 78.5	Si 100 Dia 78.5	Si 100 Dia 78.5
Blade Thickness (mm) Spacer Thickness (mm) Blade Height (mm) Number of Slades	0.15 0.36 6.35 150	0.15 0.36 6.35 150	0.10 0.30 4.76 150	0.15 0.36 6.35 940
Load (gram/blade) Sliding Speed (cm/sec)	85 64.2	85 64.2	* 64.2	85 64.2
Abrasive (type/grit size) Oil Volume (liters) Mix (kg/liter)	SiC/#600 6.3* 0.36	SiC/#600 6.3(100 SUS) 0.36	SiC/#600 6.3 0.36	SiC/#600 37.9 0.36
Slice Thickness(mm)Kerf Width(mm)Abrasive Kerf Loss(mm)Cutting Time(hours)				0.267 0.241 0.091 38.83
Efficiency (full test) (typical) (maximum) Abrasion Rate (full test) (cm ³ /hr/bl) (typical) (maximum) Productivity (full test) (cm ² /hr/bl) (typical) (maximum)				 0.049 2.023
Yield Slice Taper (mm) Slice Bow (mm) Abrasive Utilization (cm ³ /kg Oil Utilization (cm ³ /liter) Blade Wear Ratio (cm ³ /cm ³))	0	0	70%/74% * 0.078 0.085 130.56 47.0
	*H ₂ 0 + VCI-30	09 * treated	*cut rate 0.64 µm/sec	* before/aft cleaning

MAN-HOURS AND COSTS (PHASE II)

During the reporting period of December 30, 1978 to March 30, 1979, total man-hours were 599 hours and total costs were \$17,830. Previous expenditures were 13,899.2 hours and \$617,488. As of March 30, 1979, total program man-hours were 14,498.2 hours and total program costs were \$635,318.

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Varian Associates/Lexington Vacuum Division JPL Contract 954374 Starting Date: 1/9/76 (I) 5/19/77 (II)

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(PHASE II) M J J A S O N D J F M A M J J A S O N D J F M A I SLURRY Task I Low Cost Oil Analysis of Suspension Oils Survey Low Cost Oils Test Suspension Qualities Fabricate/Purchase Oils Test Oil Mixtures Test Abrasive Size Mixes Lifetime Enhancement Task 3 Evaluate Degradation SEM Analysis of Abrasive/ Silicon Debris Reclamation of Oil/Abrasive Analyze Lifetime Effects	PROJECT MILESTONES	L			1	977								1	197	8								19	79		
SLURRY Task I Low Cost Oil Analysis of Suspension Oils Survey Low Cost Oils Test Suspension Qualities Fabricate/Purchase Oils Task 2 Cutting Tests - Slurry Test Suspension Oils Test Oil Mixtures Test Abrasive Size Mixes Lifetime Enhancement Task 3 Evaluate Degradation SEM Analysis of Abrasive/ Silicon Debris Reclamation of Oil/Abrasive Analyze Lifetime Effects	(PHASE II)	M	IJ	,1	1	AS	0	N	D	J	F	Μ	Á	М	J	J	A	S	0	N	D	J	F	М	A	Μ	J
Task I Low Cost Oil Analysis of Suspension Oils Survey Low Cost Oils Test Suspension Qualities Fabricate/Purchase Oils Task 2 Cutting Tests - Slurry Test Suspension Oils Test Oil Mixtures Test Abrasive Size Mixes Lifetime Enhancement Task 3 Evaluate Degradation SEM Analysis of Abrasive/ Silicon Debris Reclamation of Oil/Abrasive Analyze Lifetime Effects	LURRY																				-	T					
Analysis of Suspension Oils Survey Low Cost Oils Test Suspension Qualities Fabricate/Purchase Oils Task 2 Cutting Tests - Slurry Test Suspension Oils Test Oil Mixtures Test Abrasive Size Mixes Lifetime Enhancement Task 3 Evaluate Degradation SEM Analysis of Abrasive/ Silicon Debris Reclamation of Oil/Abrasive Analyze Lifetime Effects	Task I Low Cost Oil	9	-	-	-	4	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-			
Survey Low Cost Oils Test Suspension Qualities Fabricate/Purchase Oils Task 2 Cutting Tests - Slurry Test Suspension Oils Test Oil Mixtures Test Abrasive Size Mixes Lifetime Enhancement Task 3 Evaluate Degradation SEM Analysis of Abrasive/ Silicon Debris Reclamation of Oil/Abrasive Analyze Lifetime Effects	Analysis of Suspension Oils	9		+	¥	-	-	-	-	-	-	A															
Test Suspension Qualities Fabricate/Purchase Oils Task 2 Cutting Tests - Slurry Test Suspension Oils Test Oil Mixtures Test Abrasive Size Mixes Lifetime Enhancement Task 3 Evaluate Degradation SEM Analysis of Abrasive/ Silicon Debris Reclamation of Oil/Abrasive Analyze Lifetime Effects	Survey Low Cost Oils		1	£	-	¥.	-	-		-	-	Y															
Fabricate/Purchase Oils Task 2 Cutting Tests - Slurry Test Suspension Oils Test Oil Mixtures Test Abrasive Size Mixes Lifetime Enhancement Task 3 Evaluate Degradation SEM Analysis of Abrasive/ Silicon Debris Reclamation of Oil/Abrasive Analyze Lifetime Effects	Test Suspension Qualities		2	-	-	Y		-	-	100	-	Y															
Task 2 Cutting Tests - Siurry Test Suspension Oils Test Oil Mixtures Test Abrasive Size Mixes Lifetime Enhancement Task 3 Evaluate Degradation SEM Analysis of Abrasive/ Silicon Debris Reclamation of Oil/Abrasive Analyze Lifetime Effects	Fabricate/Purchase Oils					Y_		-		_		-	-	-	_		-	-	-	-	-	+-	-	-			
Test Suspension Oils Test Oil Mixtures Test Abrasive Size Mixes Lifetime Enhancement Task 3 Evaluate Degradation SEM Analysis of Abrasive/ Silicon Debris Reclamation of Oil/Abrasive Analyze Lifetime Effects	Task 2 Cutting Tests - Slurry	y	1	2	-	-	-	-	5	2	-	-	-	-	-	-	-	_	-		-	-	-	-			
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Test Abrasive Size Mixes Lifetime Enhancement Task 3 Evaluate Degradation SEM Analysis of Abrasive/ Silicon Debris Reclamation of Oil/Abrasive Analyze Lifetime Effects	Test Oil Mixtures			1	2		-	5	7_	-	-	-	-	-		-	-	-5	7								
Lifetime Enhancement Task 3 Evaluate Degradation SEM Analysis of Abrasive/ Silicon Debris Reclamation of Oil/Abrasive Analyze Lifetime Effects	Test Abrasive Size Mixes				1	-	-	-	1	-			-	-	-	-	-	_	-	3							
Task 3 Evaluate Degradation SEM Analysis of Abrasive/ Silicon Debris Reclamation of Oil/Abrasive Analyze Lifetime Effects	Lifetime Enhancement	1	1			1	<u>P</u>	-	7	7																	
SEM Analysis of Abrasive/ Silicon Debris Reclamation of Oil/Abrasive Analyze Lifetime Effects	Task 3 Evaluate Degradation		-				2-	-	-	2	2		-	-	_	-	-	_	-	-	-	+	-	-			
Reclamation of Oil/Abrasive	SEM Analysis of Abrasive/ Silicon Debris	ORI	REP			0	2	_	7	2																	
Analyze Lifetime Effects	Reclamation of Oil/Abrasive	E	B	1				2_	-	7	7		•	-	-	-	-	-	-	-	-	+-	÷	\vdash			
	Analyze Lifetime Effects	AI.	E				0	P	-	5	7																
Identify Low Cost System	Identify Low Cost System	PA	图							4	7																
Task 4 Test Low Cost Slurry 35	Task 4 Test Low Cost Slurry	GE									2		_	_		_		2	-	-	-	+-	-	-			
Evaluate Cutting Lifetime	Evaluate Cutting Lifetime	1 7	\$ 0							9	2		-	-				-	-	-	+	+-	+	-			
Evaluate Impact on Accuracy	Evaluate Impact on Accuracy Rate, Wafer Yield, etc.		POOR										2			7	7		_			-	L	L			

SCH 6/15/77 Updated 3/30/79

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SLICING OF SILICON INTO SHEET MATERIAL

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SLICING OF SILICON INTO SHEET MATERIAL

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Updated 3/30/79

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SCH 6/15/77 Updated 3/30/79

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Phase II

SLICING OF SILICON INTO SHEET MATERIAL

Varian Associates/Lexington Vacuum Division JPL Contract 954374 Starting Date: 1/9/76 (I) 5/19/77 (II)

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REPORTS							-		-	-	-		-	-	-	-	-	-	-	-	-	-	-	-	-	-
Financial Package		V	V	V	7	V	V	V	V	V	Ø	V	Y	V	V	V	V	V	V	V	V	-	V			
Monthly Technical Progress		V		V	7		V	V		V	A		V	V	-	V		1	V		1	-	-	ľ	V	ľ
Quarterly Technical Progress			7		-	1		1	1		F	7			7			-		1	-	-		-		
Interim Summary																		I								
Draft Final Report										•															7	-
Final Report																										
TRAVEL																										
Project Integration Meetings			-	7		7	7-5	7	4		V	, 1	7	1	7 1		-		6	V	7	-		A	,	
MAJOR EQUIPMENT																								V		
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SLICING OF SILICON INTO SHEET MATERIAL

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PROGRAM COST SUMMARY

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