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HEAT EXCHANGER METHOD, INGOT CASTING; FIXED ABRASIVE METHOD, MULTI-WIRE SLICING: PHASE II

Silicon Sheet Growth Development for the Large-Area Silicon Sheet Task of the Low-Cost Silicon Solar Array Project. Quarterly Progress Report No. 2, January 1—March 31, 1978

By
Frederick Schmid
Chandra P. Khattak

April 7, 1978

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

Crystal Systems Inc.
Shetland Industrial Park
Salem, Massachusetts

U.S. Department of Energy

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

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Covering Period from January 1, 1978 through March 31, 1978

Date of Report: April 7, 1978

JPL Contract No. 954373

CRYSTAL SYSTEMS, INC.

35 Congress Street
P.O. Box 1057
Salem, MA 01970

The JPL Low-Cost Silicon 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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ABSTRACT

A crack-free silicon ingot has been cast in a graded, semiconductor purity silica crucible. More than 90% single crystallinity has been achieved in 2.5 kg cast ingots. The impurities on the surface of the melt have been reduced with the use of a rapid heat-up cycle and absence of graphite retainers.

Solar cells fabricated out of HEM cast material have shown conversion efficiency up to 14% under AM1 Xenon source illumination.

Considerable progress has been achieved in casting square cross-section ingots. The growth in the corners has been obtained but the problem area is in fabricating a custom-made graded crucible.

Kerf loss was reduced to 6.2 mil, 0.155 mm in slicing 4 cm x 4 cm cross-section with 100% yield. The abrasive life of plated impregnated blades was increased by hardening the electroless nickel layer.

In an effort to prevent diamond pull-out and thereby improve the abrasive life, the plated layer was increased

from 0.3 mil, 7.5 μm to 0.5 mil, 12.5 μm. The extra thickness buried the diamonds. A thinner copper sheath for impregnation and a thicker nickel coating to prevent diamond pull-out is expected to improve the abrasive life.

Higher feed forces increased the cutting rates but resulted in deeper surface damage.

CRYSTAL CASTING

One of the advantages of the Heat Exchanger Method (HEM) is the ability to cast shaped ingots. A major goal in Phase II of this program is to cast square-shaped silicon ingots. Efforts during this quarter have been directed towards improving crystallinity in the cast silicon, improvement of growth rates, and crucible development for shaped ingots.

Crystal Structure

A number of runs, details in Table I, were undertaken to optimize the growth conditions and achieve a high degree of crystallinity in the cast silicon ingots. Polished and etched cross-sections of two boules are shown in Figure 1. It is apparent that single crystallinity is almost complete. This is surprising since the solid-liquid interface is mechanically probed during the growth cycle. Further, the bottom of these crucibles is not flat or uniform in thickness, which is a hindrance to extraction of heat from the melt and promoting growth. Towards the end of the growth cycle, the melt temperature is lowered which results in freezing from the top. This is apparent in Figure 1. In run 2-015-C a crucible with a uniform wall thickness was used. It can be seen from Figure 2 that a

TABLE I. TABULATION OF HEAT-EXCHANGER AND FURNACE TEMPERATURES

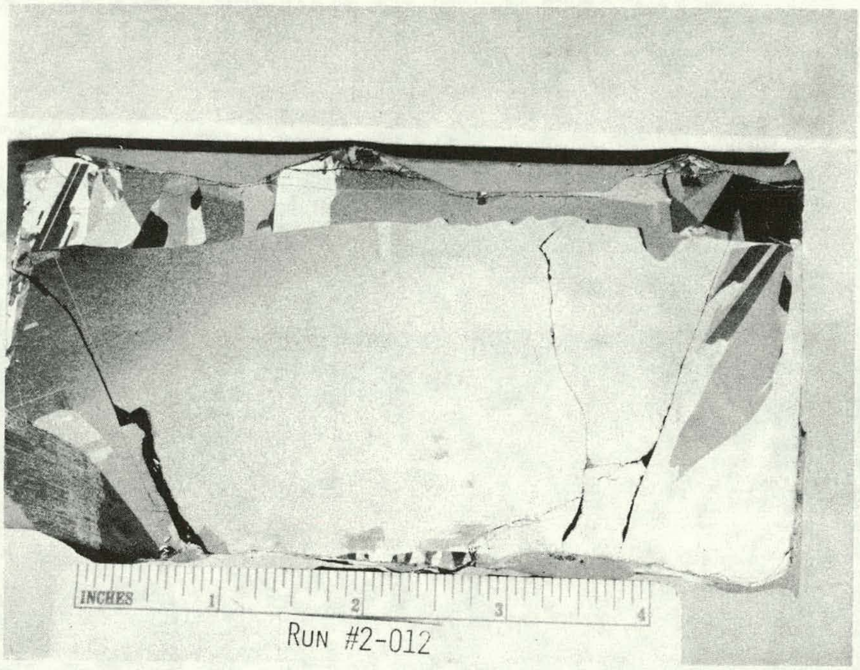
RUN	PURPOSE	SEEDING		GROWTH CYCLE			REMARKS
				RATE OF DECREASE			
		FURN. TEMP. ABOVE M.P.	H.E. TEMP. BELOW M.P.	H.E. TEMP. °C/HR.	FURN. TEMP. °C	GROWTH TIME IN HOURS	
2-010-C	Cast square cross-section boule	< 3	252	182	~ 3	7.0	Boule attached to crucible bottom; excessive seed melt-back
2-011-C	Cast square cross-section boule	3	142	192	~ 3	8.5	Boule attached to sides and bottom of crucible; excessive seed melt-back.
2-012-C	Rapid melt cycle. Improve crucible detachment from silicon.	< 3	142	127	~ 1	15.0	Improvement in crucible detachment from boule, minor cracking bottom periphery only.
2-013-C	Rapid melt cycle. Rapid cool-down cycle.	< 3	150	185	~ 1	8.0	Less attachment of crucible to side and bottom, but increase in cracking on bottom of ingot.
2-014-C	Rapid melt cycle. Prevent crucible attachment.	≈ 5	118	174	5	14.5	Crucible attachment at bottom of silicon.
2-015-C	Rapid melt cycle. Casting with tapered crucible.	≈ 6	104	109	6	14.0	Minimum of cracking; 95% single crystal material.
2-016-C	Rapid melt cycle. Heat-treated crucible (1200°C/1 hr)	≈ 3	132	152	~ 3	13.0	Some attachment of crucible bottom. Material appears cleaner (no SiC).

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

RUN	PURPOSE	SEEDING		GROWTH CYCLE			REMARKS
				RATE OF DECREASE			
		FURN.TEMP. ABOVE M.P.	H.E.TEMP. BELOW M.P.	H.E. TEMP. °C/HR.	FURN. TEMP. °C	GROWTH TIME IN HOURS	
2-017-C	Improve delamination of crucible by heat treatment (1300°C/1 hr)	6	166	238	6	14.0	Crucible shows attachment. Seed lost.
2-018-C	Improve delamination of crucible by heat treatment (1400°C/1 hr)	6	152	220	6	9.0	Crucible shows attachment. Seed lost.
2-019-C	Effect of modified cool-down cycle	<3	149	208	~2	13.5	Crucible shows attachment.
2-020-C	Test heat treated duplex crucible (1200°C/1 hr)	6	147	220	6	11.5	Strong attachment of crucible to silicon.
2-021-C	Study effect of carbon from graphite retainers	5	140	251	5	6.6	Very good growth and crystallinity.
2-022-C	Cast square cross-section boule	10	132	264	10	21.25	Good crystallinity. Minor cracking due to attachment.
2-023-C	Improve solidification rate with graphite plug	3	150	173	0	6.5	Growth has been all the way to the top. Very good crystallinity.

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

RUN	PURPOSE	SEEDING		GROWTH CYCLE			REMARKS
				RATE OF DECREASE			
		FURN. TEMP. ABOVE M.P.	H.E. TEMP. BELOW M.P.	H.E. TEMP. °C/HR.	FURN. TEMP. °C	GROWTH TIME IN HOURS	
2-024-C	Cast square cross-section boule	<3	111	146	~2	13.7	Crucible shows attachment
2-025-C	Cast square cross-section boule using graphite plug	19	94	161	19	9.25	Good crystallinity. Crucible shows attachment.
2-026-C	Cast square cross-section boule using graphite plug	5	94	164	5	6.5	Growth to the top surface. Crucible shows attachment.
2-027-C	Test high purity graded crucible	17	82	170	17	16.0	Seed floated but still good crystallinity achieved
2-028-C	Test high purity partially graded crucible	12	111	172	12	12.25	No attachment in areas where grading of crucible was developed
2-029-C	Reduce growth time with graphite plug	N/A	N/A	N/A	N/A	N/A	Run aborted. Melt stock shiny with no silicon carbide layer
2-030-C	Test high purity graded crucible	19	129	177	19	16.0	No attachment. Crackfree ingot cast.



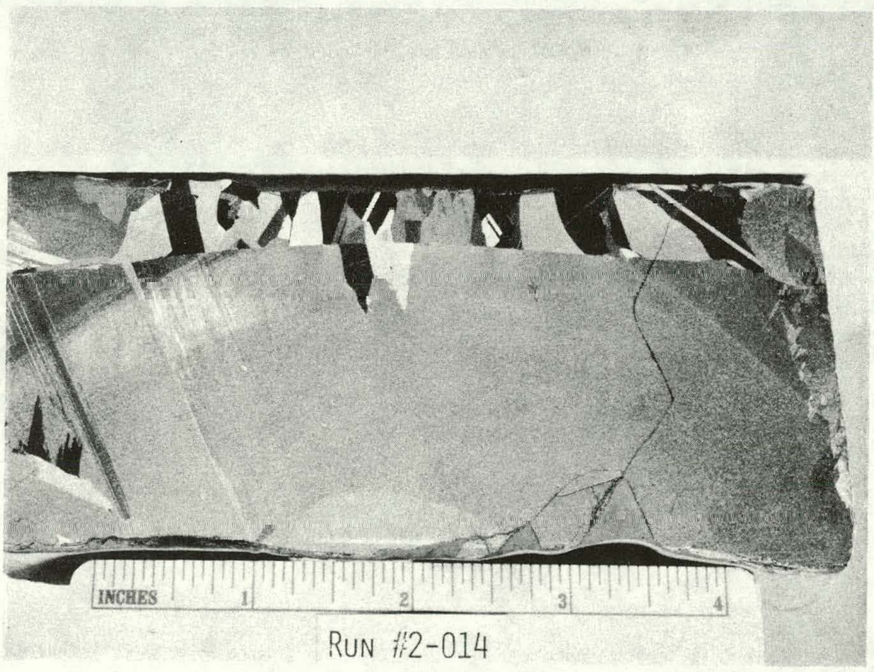


Figure 1. Polished and etched cross-section of two ingots cast in a crucible with non-uniform thickness

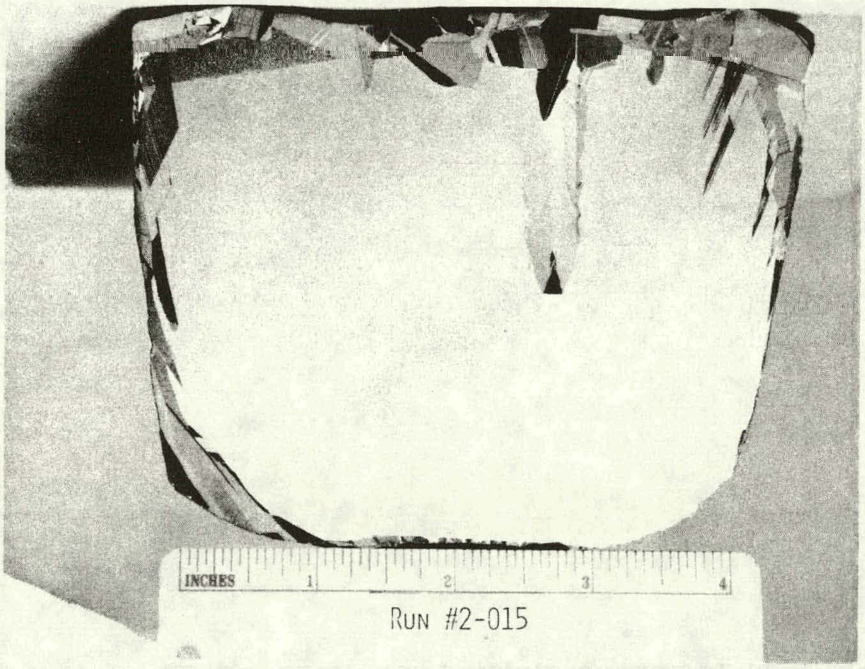


Figure 2. Polished and etched cross-section of ingot 2-015-C cast in a crucible with uniform thickness.

very high degree of crystallinity was achieved with minimal freezing from the top.

As a convenience, up to recently the furnace temperature was raised slowly overnight. In this procedure about 1200°C was achieved in 12-14 hours. From run 2-012-C onward a rapid melt-down cycle was adopted whereby first signs of melting were observed in 3-4 hours. The new method of melt-down should be compatible with the eventual sequence when the process is adopted for production. It has been reported earlier[1] that silicon carbide impurities are formed when silica crucibles are used in contact with graphite retainers. In recent runs, the graphite retainers are not used. It has been observed that with the adoption of the rapid melt-down cycle and the absence of the graphite retainers the surface of the melt has been much cleaner. Generally the impurities float and can be seen as contamination of the surface of the melt. This is also evident in the appearance of the cast ingots. The use of graphite retainers resulted in a layer of SiC formation on the top surface, whereas with the absence of the graphite retainers this surface is clean and shiny. Besides the contamination of the cast ingot the impurities act as nuclei and thereby promote freezing from the top surface. Therefore, the removal of impurities on the surface of the melt has improved the crystallinity of the cast ingots remarkably.

Some experiments were carried out to cast square cross-section boules. It was found that solidification was complete into the corners. This was encouraging since this was considered to be a potential problem. Figure 3 shows a polished and etched section of boule cast in run 2-010-C. In this case the seed was melted back more than usual and the non-uniform thickness of the bottom of the crucible caused growth off the seed; however, very large grains were formed.

In run 2-027-C a high purity silica crucible was used. This crucible has been fabricated according to semiconductor industry specifications for purity. A graded structure was developed in this crucible by heat treatment. The bottom of the crucible had a curvature so that the seed was not seated well. During the melt-down cycle molten silicon flowed under the seed and it floated. Therefore, unseeded growth progressed. Since the thickness of the crucible was uniform a high degree of crystallinity was achieved. This is evident from Figure 4 where the "floating" seed can also be seen.

Crucible Development

One of the major problems in casting silicon in silica crucibles is the attachment of silicon and thereby cracking of the ingot during cooling.[2] The solution to this problem has been achieved with the development of the graded crucible. However, this crucible is not fabricated according to semi-conductor industry purity standards. During this quarter a high-purity silica crucible meeting these requirements was fabricated and a graded structure was developed by heat treatment. In run 2-027-C

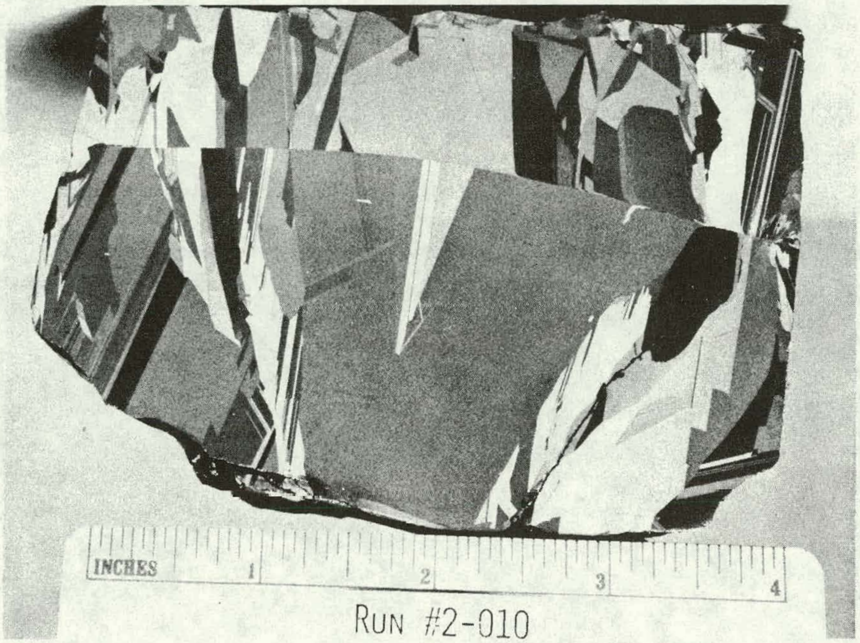


Figure 3. Polished and etched cross-section of a square ingot 2-010-C cast in a crucible with non-uniform thickness and structure

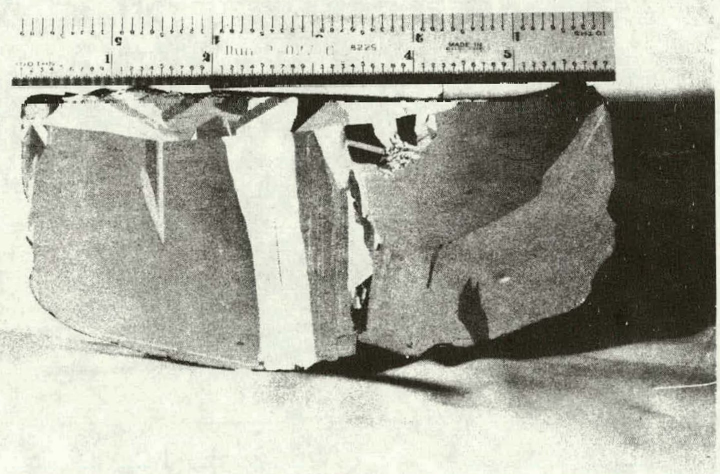


Figure 4. Polished and etched cross-section of boule cast in run 2-027-C cast in high purity graded silica crucible

this crucible showed no attachment to the cast ingot except in one area where the graded structure was not fully developed. A single crack was observed in the ingot which split it along (111) plane, the easiest slip plane for silicon. The river markings on the fractured surface traced the source of the crack to the bonded area of the ingot to the crucible. To establish this further two runs were carried out using the graded high purity crucible. In run 2-028-C the impervious inner layer in the graded structure was not fully developed and it caused considerable cracking of the ingot due to attachment. In run 2-030-C the fully graded structure showed no attachment and thereby no cracking of the ingot.

The unique requirement of square cross-section boules creates the need for custom-made crucibles. Only one vendor has so far supplied such crucibles. As mentioned above, the graded structure is essential for casting crack-free ingots. The square crucibles obtained are not fully graded; however, they are potentially low-cost and fit the overall goals of the contract. Therefore, efforts were made on adapting these crucibles. One of the methods considered for improving delamination of the crucibles was by heat treatment. The crucibles were heated at 1200°C, 1300°C, and 1400°C for an hour and used in runs 2-016-C, 2-017-C and 2-018-C respectively. It was found that the crucible was attached to the ingot. Of these three runs, the best delamination of the crucible took place with the 1200°C heat treatment. A degree of crystallinity was achieved (Figure 5).

In an effort to use a low-cost crucible and still achieve high purity a duplex crucible was fabricated. This crucible had a high purity liner. Such a crucible was used in run 2-009-C.[3] It was found that the liner was too thick and attached to the boule. It was felt that with heat treatment of such a crucible enough microcracking may be set up in the crucible/liner interface to aid delamination. As mentioned above best results were achieved with 1200°C heat treatment. In run 2-020-C a 1200°C heat treated duplex crucible was tested. Delamination was not achieved and the liner enveloped the ingot.

Improvement of Heat Extraction through the Crucible

In the Heat Exchanger Method (HEM) heat is extracted through the bottom of the crucible. Most of the graded crucibles used have a non-uniform thickness. This results in nucleation off the bottom of the crucible rather than off the seed. Moreover, the thermal conductivity of silica is poor near the melting point of silicon and it drops rapidly as the temperature is lowered.[4] The combination of non-uniform, rather thick bottom of the crucible together with the poor conductivity of silica has resulted in the top section of the boule to solidify by freezing from the top. It has been demonstrated[2] that a graphite plug can be used through a hole at the bottom of a silica crucible for more efficient heat transfer.

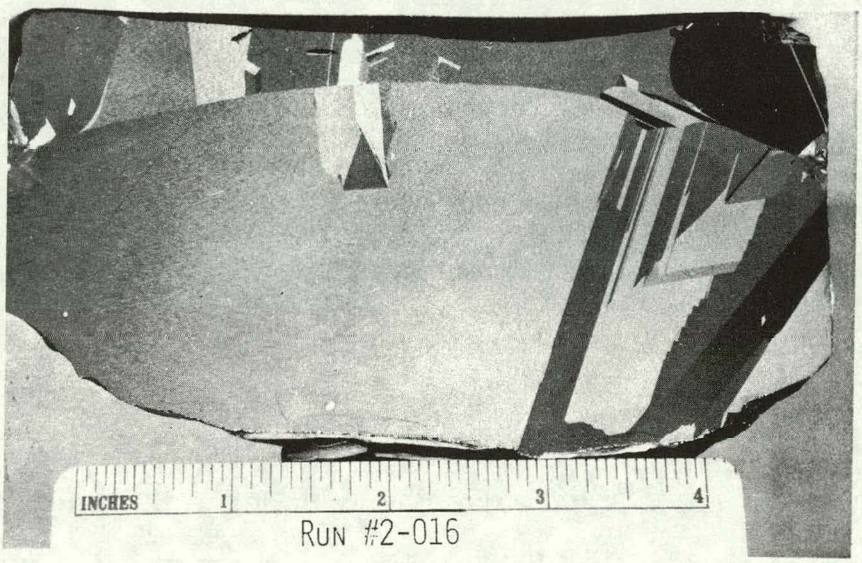


Figure 5. Photograph of polished and etched cross-section of ingot 2-016-C cast in crucible with non-uniform thickness and structure; heat treated at 1200°C for 1 hour

A number of runs (Table I) during this quarter were carried out to study the effect of the graphite plug. It was found that very good crystallinity was achieved in shorter growth periods. Further, growth of the ingot was achieved all the way to the top surface of the ingot without any freezing from the top. A typical polished and etched cross-section of the boule cast in run 2-023-C is shown in Figure 6.

As an economic measure, a crystal growth experiment was conducted to determine if the graphite plug is reusable. One of the plugs used in an earlier run was re-used in run 2-026-C and there was no problem, confirming that the graphite plugs are reusable.

Solar Cell Performance

Material from run 2-021-C was used to make 2 cm x 2 cm solar cells. The measured resistivity on the as cast sample was 4.3 - 4.6 ohm-cm. Data were taken under AM1 conditions using a Xenon source as well as a tungsten source in order to study the effect of impurities in the samples. These data are shown in Table II. Conversion efficiencies of 13.3 - 14% were achieved using Xenon light source. Measurements under AMO conditions have shown a mean conversion efficiency of 11.7% with a high of 11.9% and a low of 11.5%. The three control CZ samples fabricated with these cells showed conversion efficiencies of 11.8, 11.7 and 11.7% under AMO conditions.

Data in Table II shows that the improved efficiency expected under AM1 conditions as compared to the AMO conditions

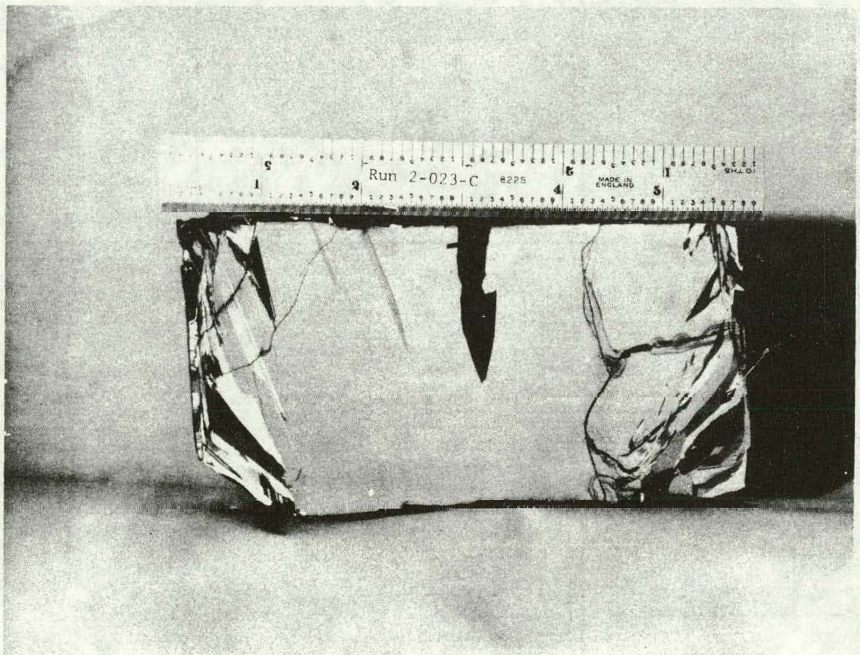


Figure 6. Polished and etched cross-section of boule cast in run 2-023-C using a graphite plug

TABLE II. I-V Parameters under AM1 conditions for AR coated (Ta_2O_5) solar cells fabricated from run 2-021-C using Xenon and Tungsten source illumination.

Sample #	Xenon Source				
	I_{sc} (mA)	V_{oc} (mV)	CFF	P_{max} (mW)	η (%)
1	114.2	609	0.771	53.6	13.3
2	116.8	612	0.764	54.6	13.6
3	114.5	610	0.784	54.8	13.6
4	117.2	615	0.782	56.4	14.0
5	116.2	613	0.784	55.9	13.9
6	113.3	612	0.792	54.9	13.7
Mean	115.4	612	0.780	55.0	13.7

Sample #	Tungsten Source				
	I_{sc} (mA)	V_{oc} (mV)	CFF	P_{max} (mW)	η (%)
1	95.9	598	0.771	44.2	11.0
2	99.0	602	0.770	45.9	11.4
3	95.5	599	0.787	45.0	11.2
4	100.2	605	0.784	47.6	11.8
5	98.0	602	0.789	46.6	11.6
6	97.0	602	0.788	46.0	11.4
Mean	97.6	601	0.782	45.9	11.4

does not show up under tungsten source illumination. This shows that the extra efficiency is obtained in the blue but not in the red region of illumination. Further, the I_{sc} is lower than the control cells. This effect is expected from material that has high impurities.

The V_{oc} measured is rather high for the measured resistivity of the cast sample. In this run, the ingot was cooled rapidly which may have resulted in entrapped oxygen which diffused during cell processing.

CRYSTAL SLICING

Efforts in crystal slicing during this quarter were directed towards blade development to reduce kerf and increase life, slicing tests to measure cutting effectiveness and blade life, along with blade and wafer characterization.

Slicing Tests

Slicing tests were carried out with the impregnated blade as well as plated blades (Table III). It has been established that diamond pull-out in impregnated blades can be prevented by plating after impregnation.[2]

In runs 77-S, 78-S, 79-S, and 2-004-S[3] commercially impregnated wire plated with 0.3 mil, 7.5 μm electroless nickel showed good cutting effectiveness through 6" depth of cut as shown in Figure 7. It was felt that a thicker coating of 0.5 mil, 12.5 μm may hold the diamonds better and thereby prolong the blade life. Such a wire was used in run 2-005-S through run 2-007-S. It was found that the initial cutting rates were lower than usual but it cut through 6" of silicon with high yields. In an effort to hold the diamonds firmly an impregnated blade pack plated with 0.5 mil, 12.5 μm nickel was heat treated at 500°F in order to harden the coating to approximately 680 VH. This blade was tested in runs 2-011-S through 2-014-S where it cut through 8"

TABLE III. 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
2-005-S	Life test	0.08	36.2	2.69	0.068	Commercial 8 mil, 0.2 mm super impregnated 45 μ diamond 0.5 mil, 12.5 μm electroless nickel plated after impregnation	Good wafer quality. Cutting rates lower than usual.
2-006-S	Life test	0.08	36.2	1.75	0.044	Same as 2-005-S.	Good wafer quality. Cutting rates low.
2-007-S	Life test	0.08	36.2	1.31	0.033	Same as 2-005-S.	Cutting rate degradation. Curve same as in past runs but lower.
2-008-S	Test CSI impregnated plated 0.005 wire	0.042	19.4	1.31	0.033	5 mil, 0.125 mm copper coated stainless steel CSI impregnated 0.3 mil, 7.5 μm electroless nickel after impregnation	Initial cutting rates over 4 mils/min. Very good wafer quality.
2-009-S	Life test	N/A	N/A	N/A	N/A	8 mil super impregnated 45 μ diamond plated with 1 mil, 25 μm electroless nickel after impregnation	Run aborted. Low cutting rates due to excess nickel buildup.
2-010-S	Test CSI impregnated wire	N/A	N/A	N/A	N/A	5 mil, 0.125 mm tungsten core, 0.7 mil, 17.5 μm copper sheath 30 μm diamonds	Run aborted due to diamond pullout.
2-011-S	Life test of commercially impregnated, plated wire	0.09	40.41	2.81	0.071	8 mil, 0.2 mm wire, 45 μm diamond, electroless nickel plated 0.5 mil, 12.5 μm	Good wafer quality and good cutting rates. 88% yield.

TABLE III. 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
2-012-S	Life test	0.09	40.41	2.12	0.054	Same as 2-011-S	Very good wafer quality. 97% yield.
2-013-S	Life test	0.09	40.41	1.98	0.050	Same as 2-011-S	Very good wafer quality. 96% yield.
2-014-S	Life test	0.09	40.41	1.41	0.036	Same as 2-011-S	Good wafer quality. 91% yield.
2-015-S	Test CSI impregnated blade	N/A	N/A	N/A	N/A	5 mil, 0.125 mm tungsten core, 0.7 mil, 17.5 μm copper sheath, impregated with 30 μm diamonds, electroless nickel plated 0.5 mil, 12.5 μm	Run aborted due to poor cutting. Diamonds are buried under the plating.
2-016-S	Life test of plated blade	0.083	37.7	3.10	0.078	5 mil, 0.125 mm tungsten core, nickel plated with 22 μm diamonds	Very good wafer quality and good cutting rates. 100% yield. 6.2 mil, 0.155 mm kerf.
2-017-S	Life test continuation	0.083	37.7	1.23	0.031	Same as 2-016-S.	Good wafer quality. 100% yield.
2-018-S	Life test, CSI impregnated blades	0.080	36.2	2.50	0.063	5 mil, 0.125 mm tungsten core, 0.7 mil, 17.5 μm copper sheath; CSI impregnation 45 μ diamonds 0.5 mil, 12.5 μm electroless nickel; heat treatment 500°F for 1 hr.	Very good wafer quality. 99% yield.

TABLE III. 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
2-019-S	Life test continuation	0.080	36.2	1.6	0.040	Same as 2-018-S.	Good wafer quality. 97% yield.

of silicon and produced good quality wafers with high yields.

The cutting effectiveness of impregnated and electroless plated wires is shown in Figure 7. The cutting rates are plotted as a function of cumulative depth of cut. It can be seen that unplated impregnated wires, shown by squares, lose cutting effectiveness very rapidly because of diamond pull-out. The best life was obtained by electroless plating and heat treating at 500°F to increase the hardness of the electroless nickel coating from approximately 580 to 680 VH. The best initial cutting rates were achieved with a 0.3 mil, 7.5 μm plating thickness. Apparently the 0.5 mil, 12.5 μm coating buried some of the diamonds and decreased the initial cutting rates.

Blade Development to Reduce Kerf

Wires were electroplated with diamonds and impregnated using the Crystal Systems impregnation technique to reduce the kerf. A non-uniformity in impregnation of diamonds was observed in wires charged by the Crystal Systems technique. This was attributed to charger misalignment and was corrected. A blade pack was fabricated by impregnating into a 0.0007 thick copper plating on tungsten wire. This wire pack was used in run 2-010-S which gave good initial cutting rates, but the cutting rate decreased as usual due to diamond pull-out. A blade-pack of 100 wires charged by the CSI method was electroless plated with

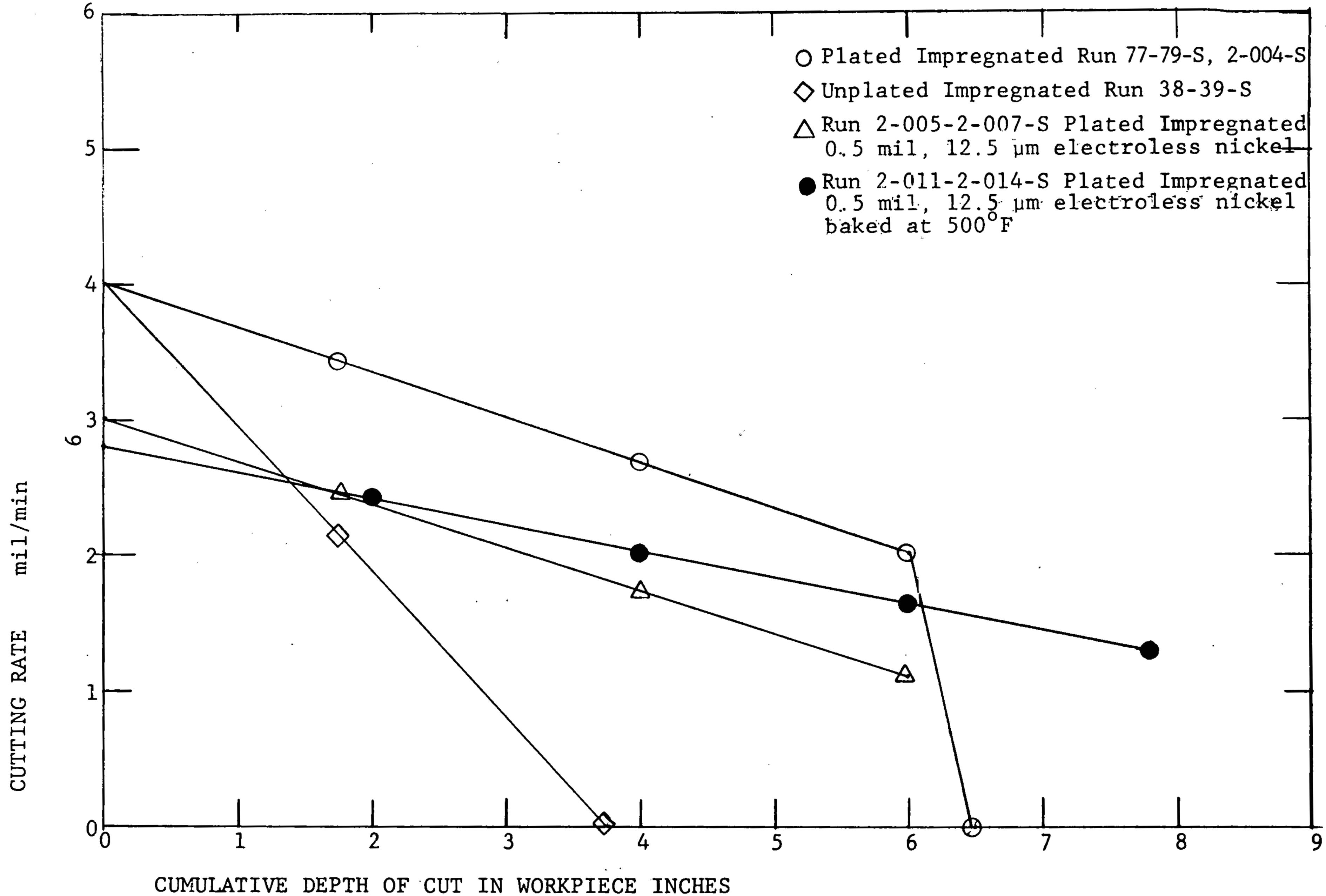


Figure 7. Cutting rate as a function of depth of cut for various wire blades.

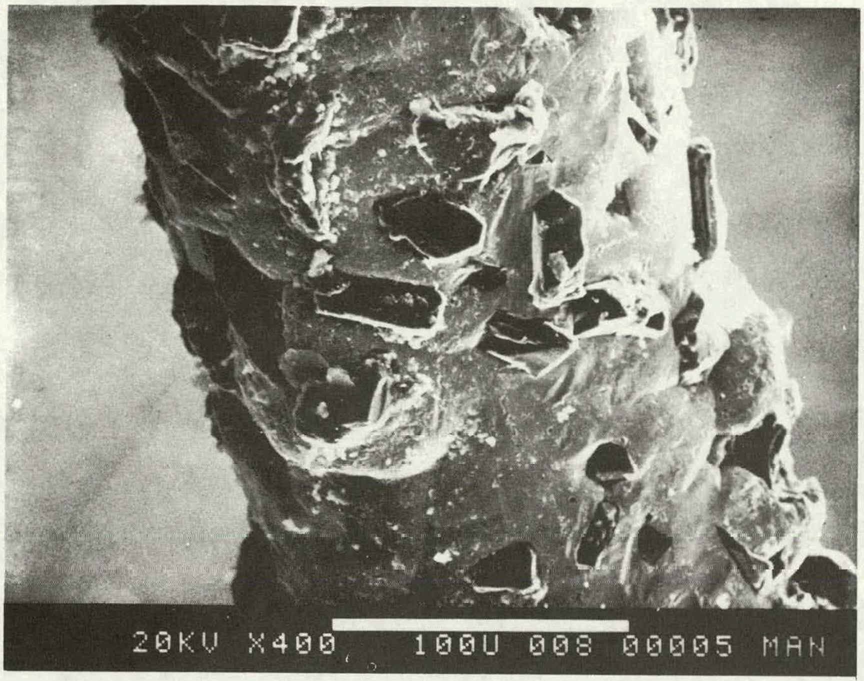
12.5 μm, 0.5 mil nickel and heat treated at 500°F to a Vickers hardness of 580. In runs 2-018-S and 2-019-S the wires cut at 2.5 mil/min, 0.062 mm/min, through the first 4 cm cube and at 1.6 mil/min, 0.04 mm/min through the second 4 cm cube. The kerf loss was reduced to about 8 mil, 0.2 mm and the yield was 99 and 97% respectively.

In an effort to reduce the kerf even more wires with a 3 mil stainless steel core and 1 mil sheath were impregnated with 30 μ diamonds and electroless plated with 7.5 μm of nickel. Initial cutting rates of 4 mil/min, 0.1 mm/min were achieved. The kerf was reduced to 7 mils, 0.178 mm and high quality wafers were produced.

In order to reduce kerf further efforts were made to electroplate 22 μm diamonds directly onto a 5 mil, 0.125 mm tungsten core wire. This blade pack was used in runs 2-016-S and 2-017-S when 100% yield was obtained with kerf reduced to 6.2 mils, 0.155 mm.

Blade Characterization

It has been found[2] that the plated diamond wires do not suffer from diamond pull-out as in the case of impregnated wires. In run 2-001-S cutting was carried out at higher than usual feed forces followed by normal feed forces in run 2-002-S. Figure 8 shows the top surface, unused section, of the blade as well as the cutting edge. It can be seen that even under high feed forces diamond pull-out is not a severe problem.



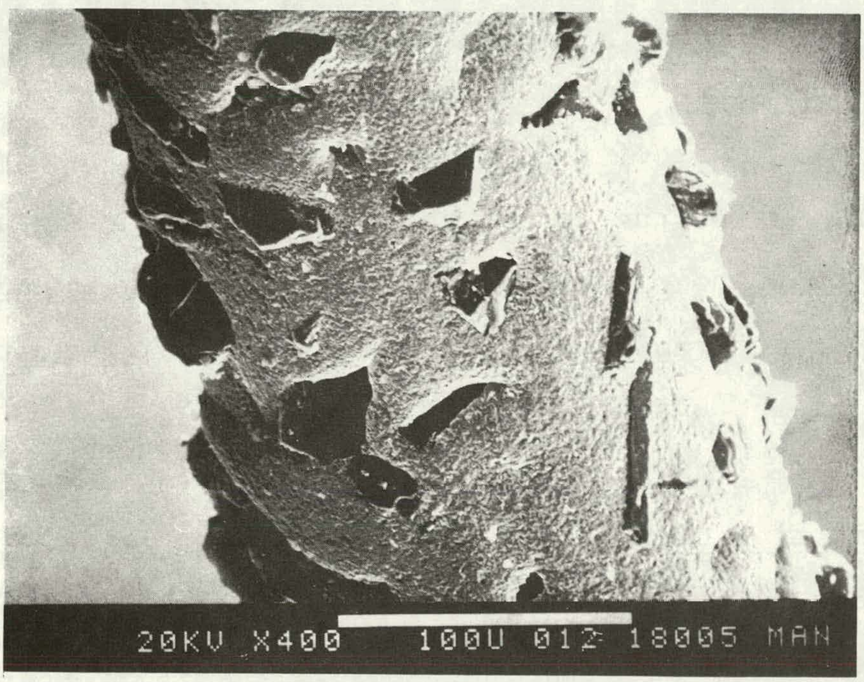


Figure 8. Unused section of plated diamond wire (above) and section used in runs 2-001-S and 2-002-S (below)

In runs 2-005-S through 2-007-S an impregnated blade plated with 0.5 mil, 12.5 μm nickel and hardened at 375°F to 580 VH was used. Better cutting performance was achieved as compared to unhardened blades. Figure 9 shows the unused and used sections of these wires. It can be seen that practically no diamonds can be seen in the cutting edge.

Another blade pack impregnated with diamonds and plated with 0.5 mil, 12.5 μm nickel was hardened at 500°F to 680 VH and used in runs 2-011-S through 2-014-S. These wires exhibited better cutting performance than the 375°F hardened pack. Figure 10 shows the top unused edge as well as the cutting edge of these wires.

It appears that the plated impregnated blade prevents diamond pull-out; however, a thickness of 0.5 mil, 12.5 μm plating buries most of the diamonds. It is intended to reduce the thicknesses of the copper sheath and the plating after impregnation to hold the diamonds more effectively and expose the diamonds for rapid cutting. This will also result in reduction of the overall kerf.

Characterization of Work Damage

It has become apparent the depth of damage can have a major cost impact on sheet production because of wasted silicon, wasted acids, and wasted time. In light of this, a study was undertaken to evaluate the depth of damage caused by multi-wire slicing

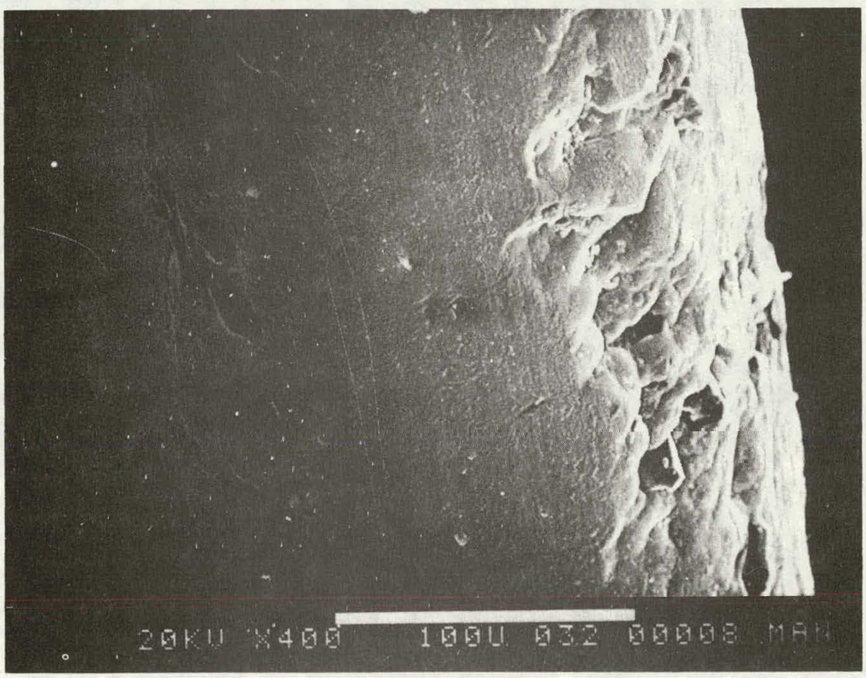


Figure 9. Unused (above) and used (below) section of impregnated blade used in runs 2-005-S through 2-007-S

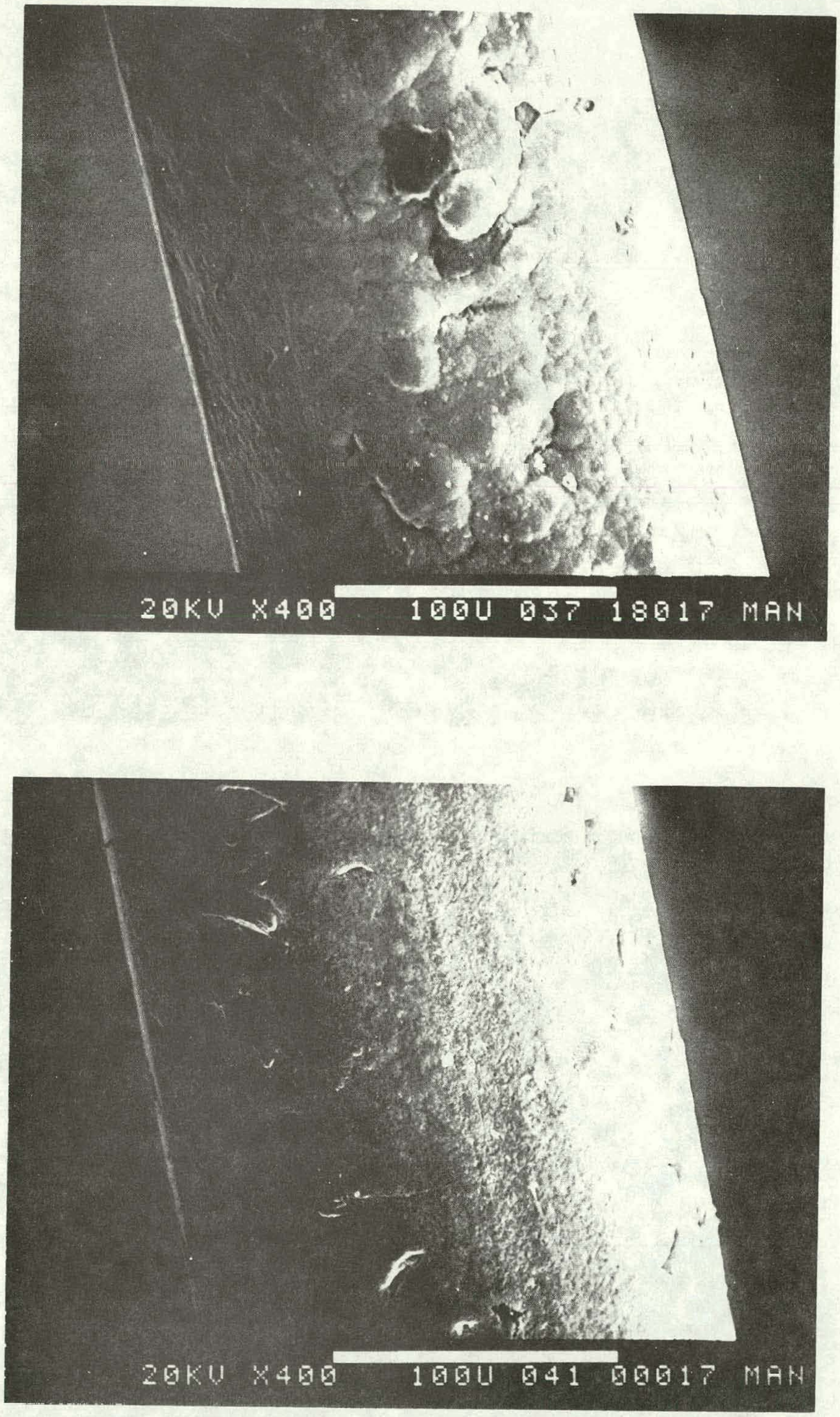


Figure 10. Unused (above) and used (below) section of impregnated blade used in runs 2-011-S through 2-014-S

by the Fixed Abrasive Method (FAM). Data obtained during that initial investigation showed a high concentration of dislocations evidenced by the etch pits near the cut surfaces, but that the concentration decreased rapidly away from the surfaces.

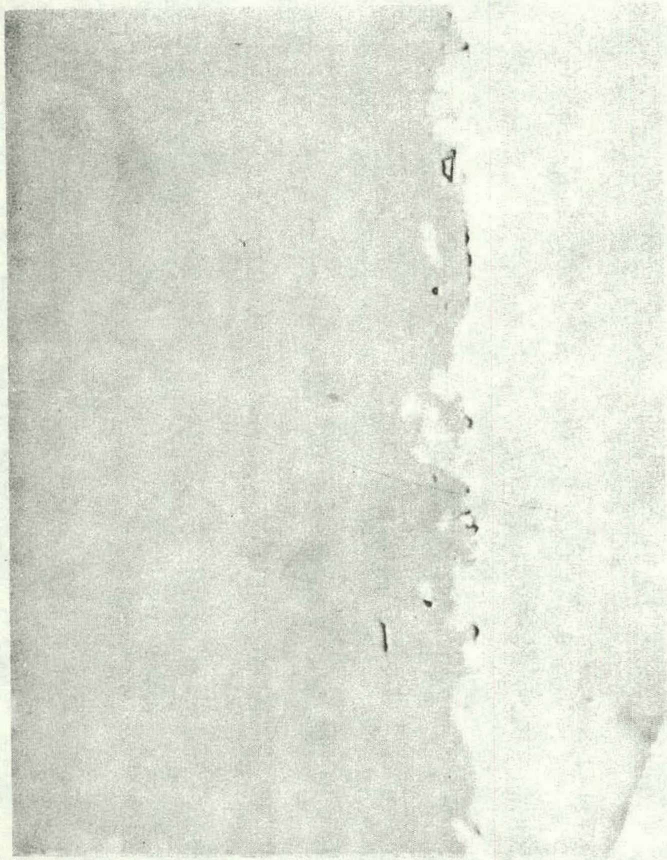
Two types of samples were obtained from each specimen. In one case, the wafer was oriented and then cut so that a (110) surface at 90^{o} to the FAM-sliced surface was exposed. The sample was mounted so that all polishing and etching would be done on that (110) surface. Hence, this type of sample permitted examination of a cross-section of the wafer with one edge being the original cut surface.

As a second check on the depth of the damage, samples of each of the two wafers were mounted and processed so as to produce "tapered sections." These samples were mounted on the (111) surfaces, the original cut surfaces, and then polished on a wedged fixture so that a taper would be developed across the surface, thus producing a mechanical magnification along the dimension that was tapered.

Sample 76-S was tapered 5.8^{o} to give a mechanical magnification of 10X, and sample 67-S was tapered 2.3^{o} to produce a mechanical magnification of 25X.

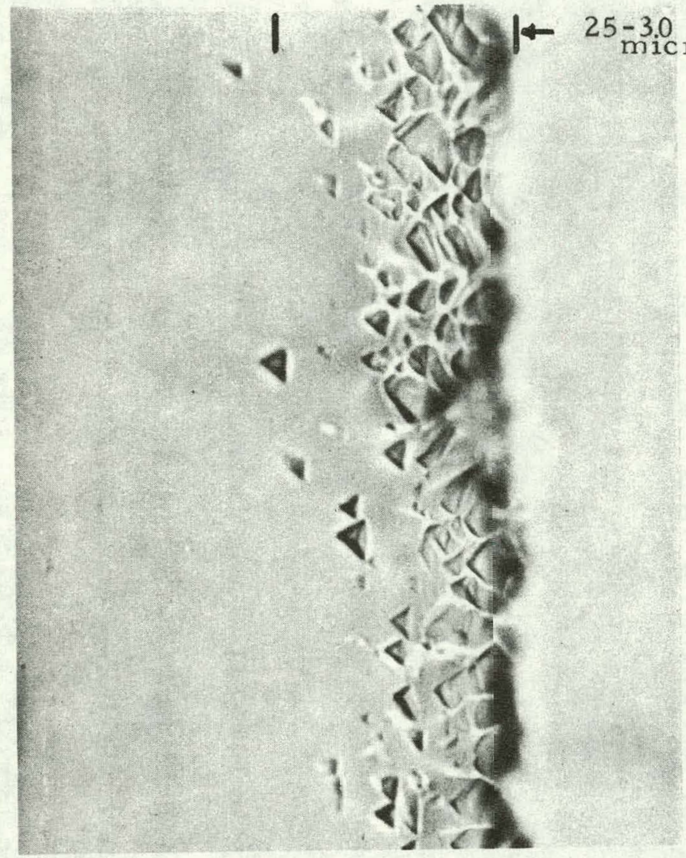
Figures 11 and 12 are 1000X photomicrographs of polished and etched (111) and (110) sections taken from wafer 76-S. Comparison of the two figures shows that the depth of damage is about 3 μm as determined by either the tapered or cross-sectional method.

1000 X   Tapered Section(Dimension 10X)



76S   (111) Silicon   As polished   Ni

1000 X   Tapered Section (Dimension 10X)



76S   (111) Silicon   Etched   Ni

Figure 11. Photomicrographs of polished and etched (111) sections of wafer 76-S (1000X)

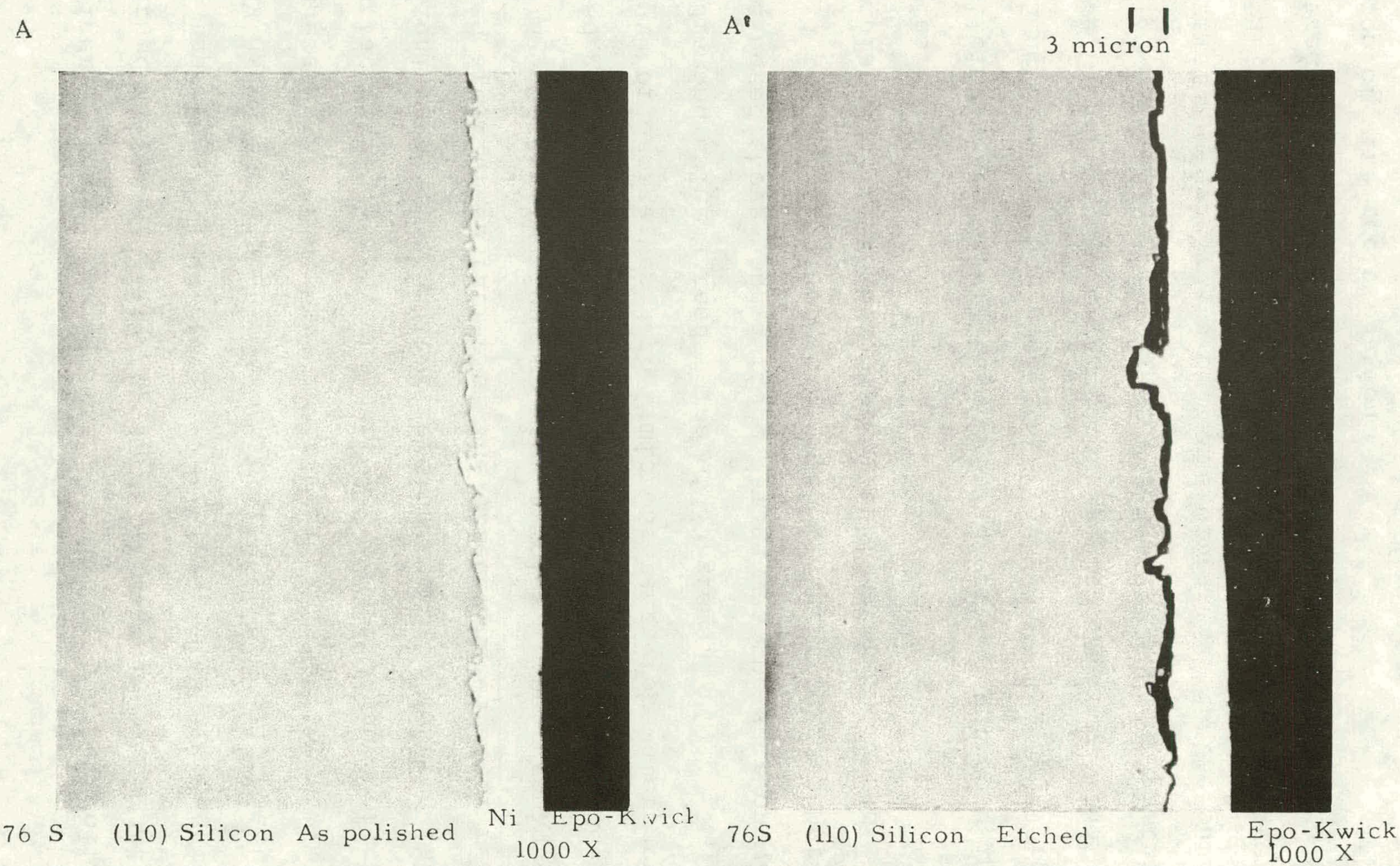


Figure 12. Photomicrographs of polished and etched (110) sections of wafer 76-S (1000X)

Figures 13 and 14 are 1000X photomicrographs of polished and etched (111) and (110) sections of wafer 67-S. Figure 12, a 2.3^{o} tapered section, shows a depth of damage of about 3 microns as compared to 6 microns by the etched (110) section. The tapered section appears to be more reliable in determining the surface irregularity and depth of damage because of the mechanical magnification that results from the taper. The data reported are representative of the effects noted after repeated polishing and observations of many samples. The techniques employed give reproducible results and it can be concluded that the depth of damage was 3 to 4 microns range for wafers cut with 30 μm diamond using a feed force of 35 grams.

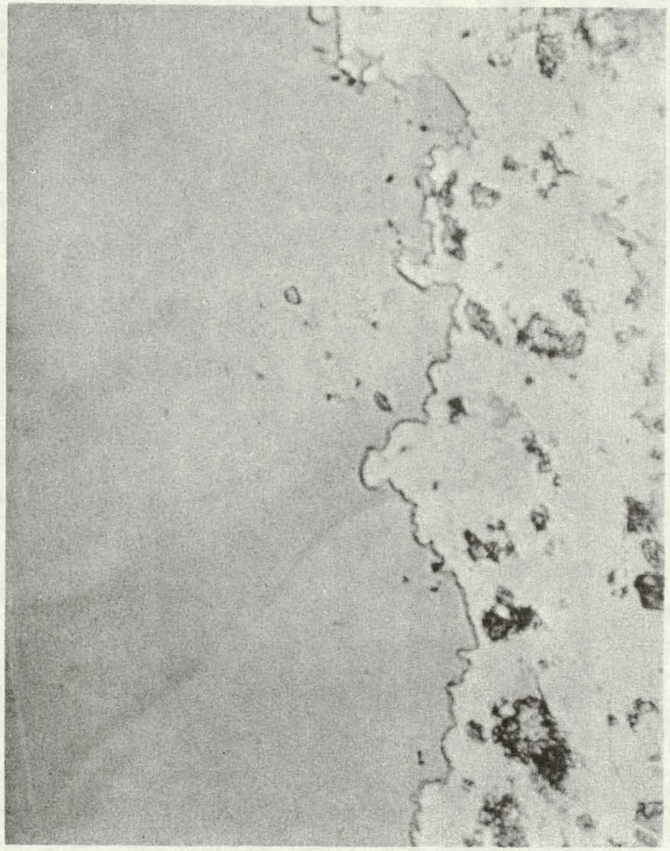
As noted above, the surface damage was the same on wafers cut at a feed force of 35 grams with wire that was either electroplated or impregnated with 30 μm natural diamond particles.

Slicing tests 2-001-S and 2-002-S were conducted using wire with 45 μm diamonds but using feed forces of 36.3 and 90.8 gms per wire. The average cutting rates were 0.035 mm, 1.36 mil/min and 0.142 mm/min, 5.6 mil/min for the 36.3 and 90.8 gm feed forces respectively. There was a dramatic difference in the surface irregularity as illustrated in Figure 15, a Tallysurf profiliometer tracing of the surface topography perpendicular to the direction of wire reciprocation.

Tapered sections of the (111) surfaces of wafers 2-001-S and 2-002-S were made to determine the surface damage as a

1000 X Tapered Section-Dimension 25 X

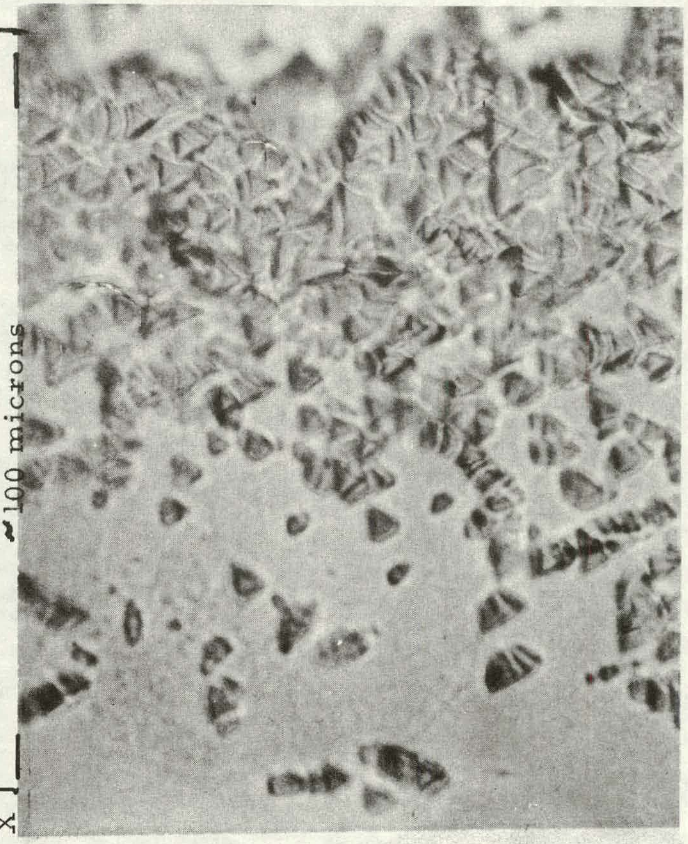
B'



9/19 #4 Silicon (111)
As polished Ni

1000 X Tapered Section - Dimension 25 x

B''



9/19 #4 Silicon (111) Etched Ni

Figure 13. Photomicrographs of polished and etched (111) sections of wafer 67-S (1000X)

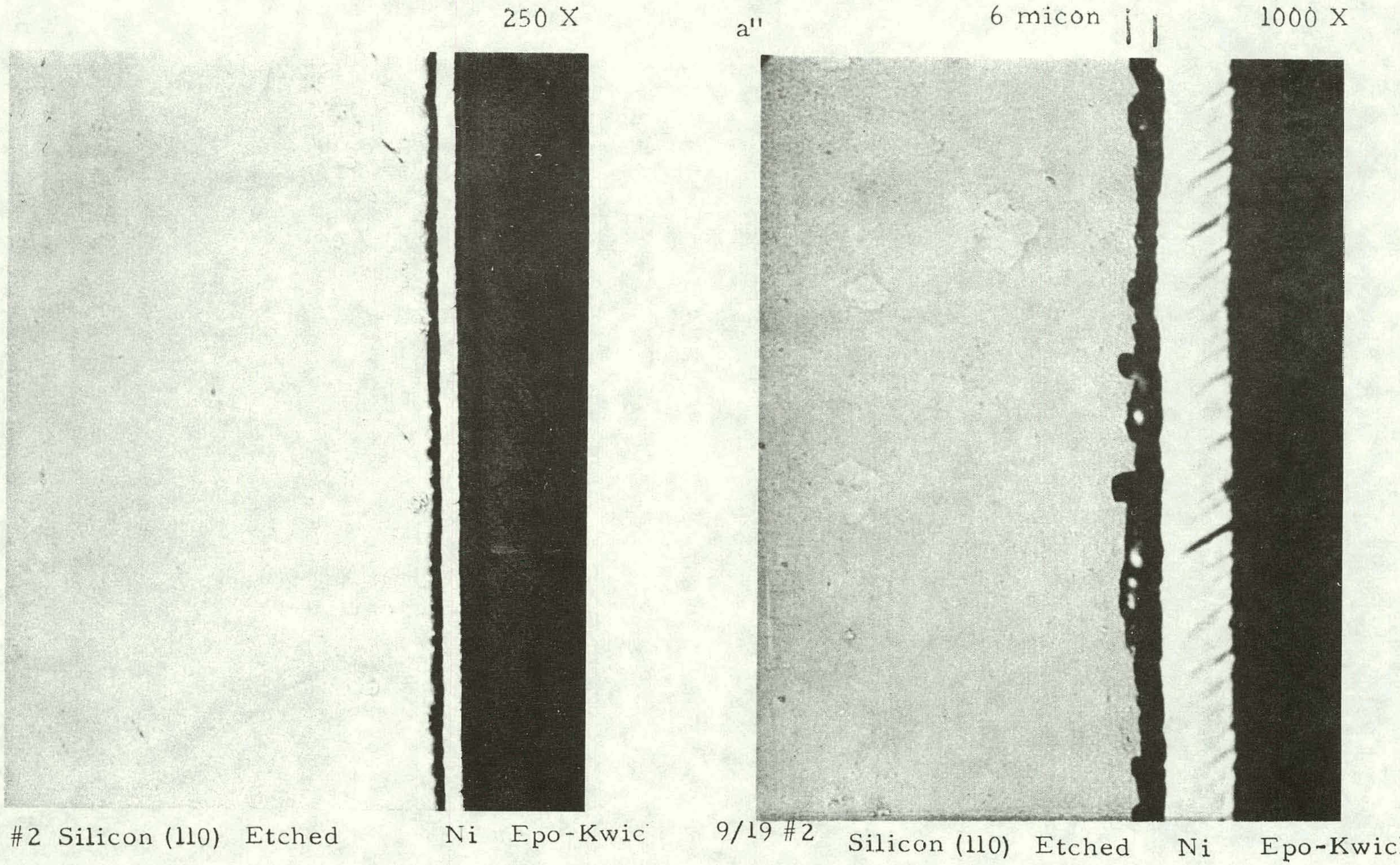


Figure 14. Photomicrographs of polished and etched (110) sections of wafer 67-S (1000X)

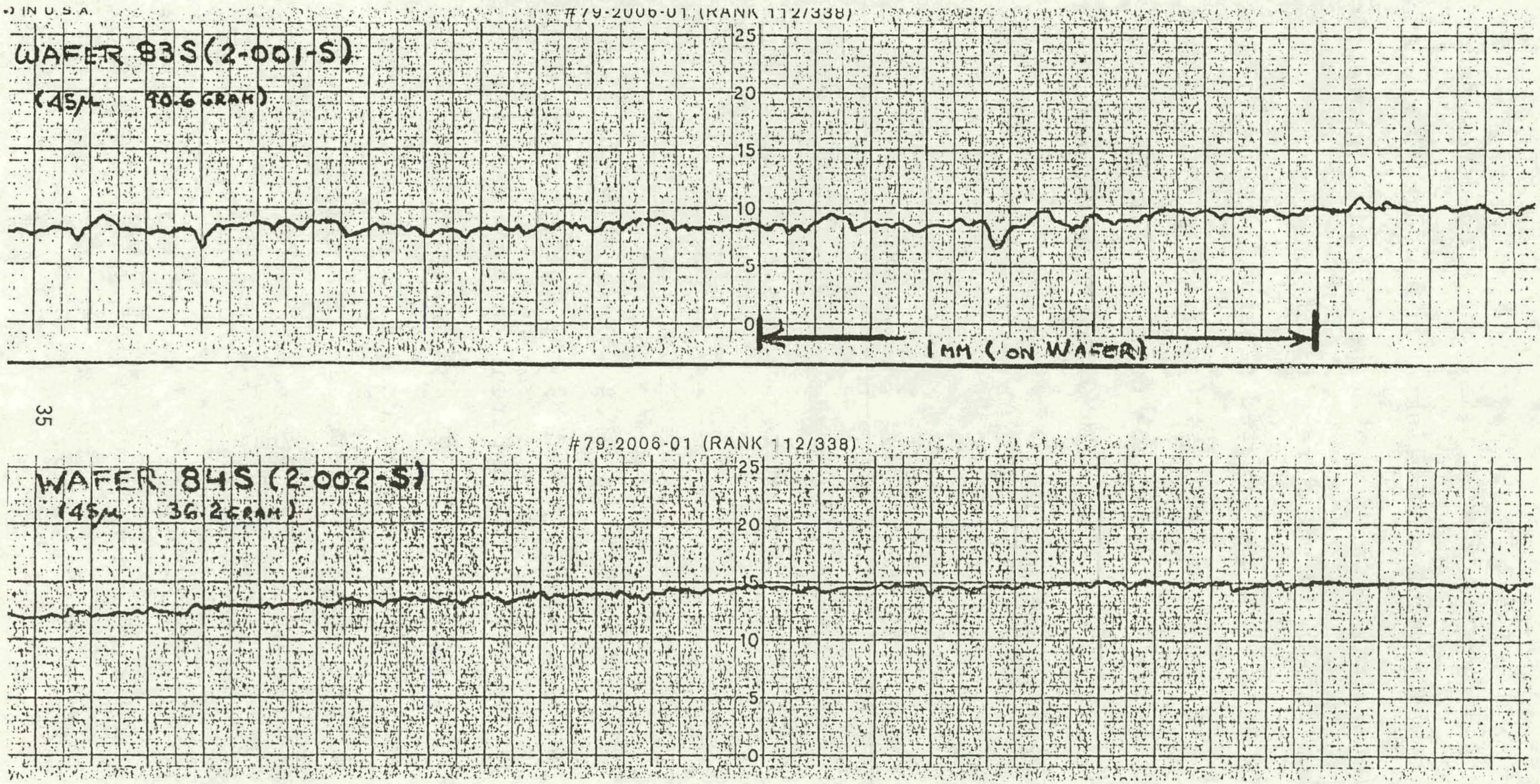


Figure 15. Profiliometer tracing of the surface topography perpendicular to direction of wire reciprocation for run 2-001-S and run 2-002-S

function of feed force.

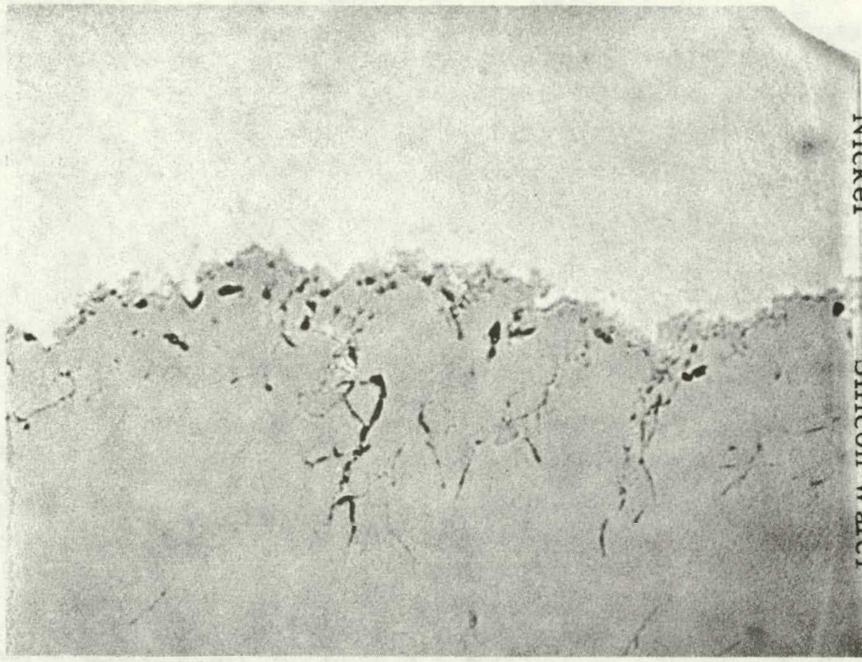
The samples were prepared using the procedures described earlier. It was ground and polished on a wedged fixture such that the final surface was a 5.8^{o} taper giving a geometric magnification of 10X in the direction of the taper.

The features of these tapered sections after the polishing and etching are shown in Figures 16 and 17, photomicrographs at 1000X magnification. The surface contours, distorted due to the tapering, agree qualitatively with the measurements taken by the Talleysurf instrument.

Wafer	Talleysurf Irregularity	Figure 16 and 17 Irregularity
84-S	about 1 micron	about 0.5 micron
83-S	about 4 microns	about 1 micron, but with deep cracks down to about 4 microns

The "as polished" surfaces do, in general, show the same degree of physical undulations as noted by the Talleysurf measurements, but on the photographs it is apparent that some of the damage does extend well into the bulk of the material.

The extent of this sub-surface damage becomes more apparent after the wafers are etched. The wafers were etched in the Dash etch ($5HNO_3$/3 HF/c Acetic) for about 5 seconds. The surfaces, after the etching, do show the typical triangular shaped (111) etch pits concentrated at or very near the cut surface plus a distribution of the etch pits into the bulk of the



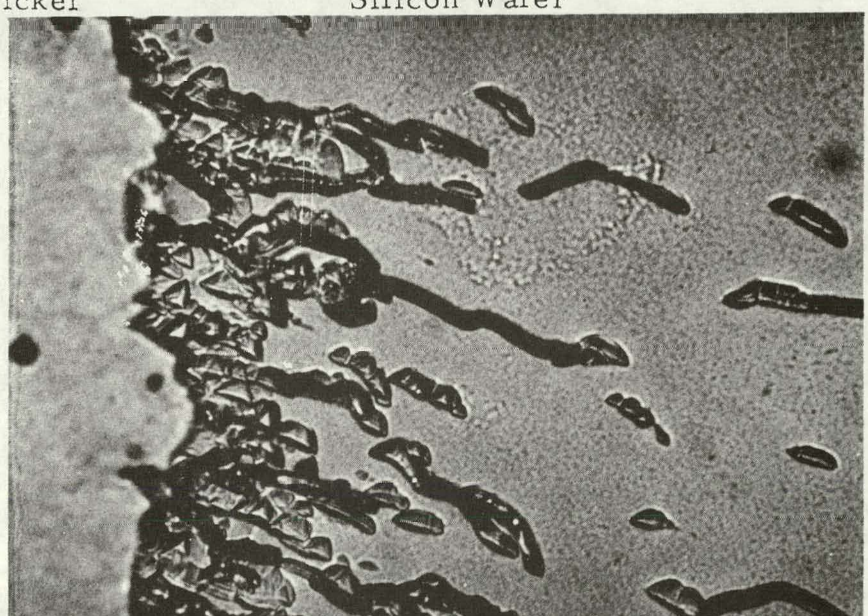
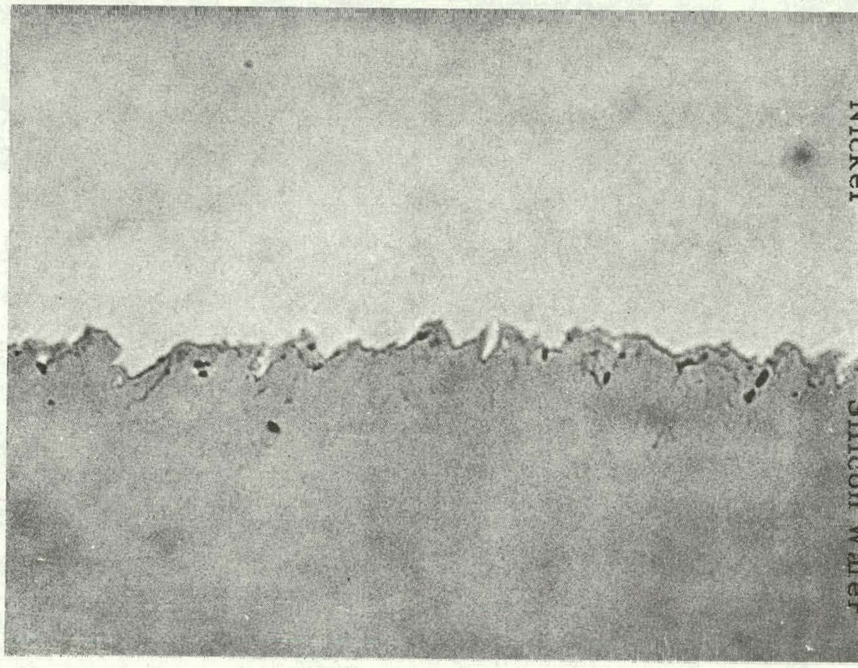


Figure 16. Photomicrograph of polished and etched tapered section of wafer 83-S (1000X)



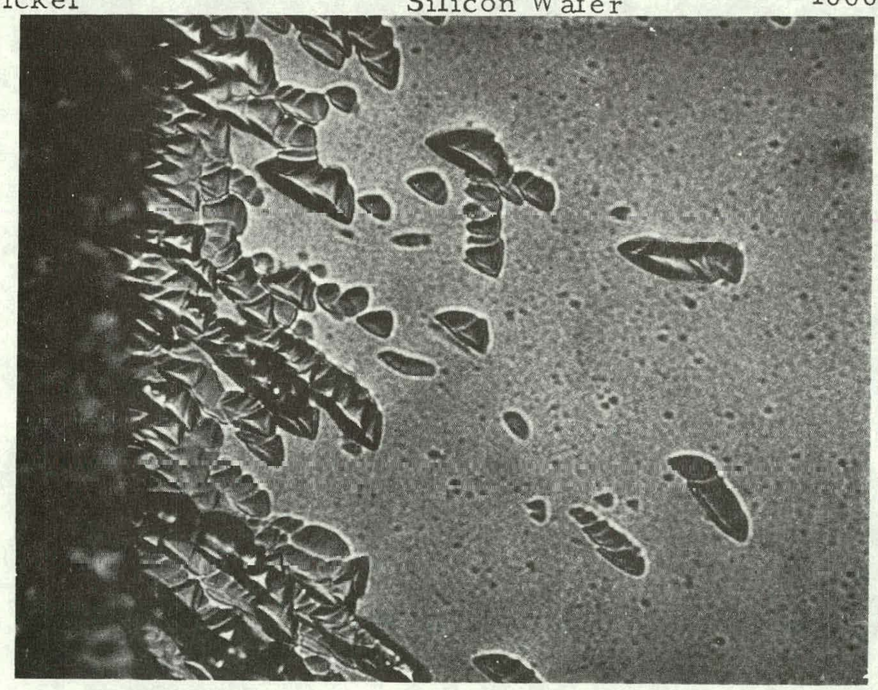


Figure 17. Photomicrograph of polished and etched tapered section of wafer 84-S (1000X)

material. The estimated extent of the damaged layer under the surface is:

Wafer 2-001-S	Heavy concentration of dislocations 1-2 microns deep with evidence of the damage to about 7-8 microns
Wafer 2-002-S	Heavy concentration of dislocations 1-2 microns deep with evidence of the damage to 15-18 microns. The damage in this particular wafer seems to be deep fissures or cracks

The tapered section shows that the depth of work damage is heavily dependent on the feed force. Although larger feed forces increase the cutting rate, it has a detrimental effect on the surface irregularity, depth of work damage, and wafer accuracy, i.e., bow and taper.

The time saved by increasing cutting rates with application of higher feed forces appears to be misleading. Feed forces should not be increased to increase cutting rates, since the loss of silicon, acids, and time will more than offset the time gained in rapid slicing.

Slicing Machine Modification

Due to current budget limitations, a complete system study could not be undertaken to determine the type of machine that would be best suited for increasing the surface speed of multi-wire blades while reducing vibrations. Machines using continuous wire should be compared with machines using wires stretched in a light-weight carriage that reciprocated on linkages or on slides.

For our present program, a machine in which wires are reciprocated was reviewed in terms of either producing a modification of the existing machine or designing a "breadboard" type of unit using the same principal. It became apparent that there were many advantages in building a second machine rather than attempting modification of the present machine. It would not interrupt the work output during construction, and it would permit experimentation with various cutting parameters, once the new unit was completed, without interference with the Program Plan.

In addition, modifying the old machine would seriously limit the areas in which new mechanical techniques could be explored. Since the specific areas in which new data is desired involve increased speeds and strokes, a new head assembly and drive systems will be required. The only useful parts on the present machine would be the base casting and the ways. Both of these restrict the configurations of the slide and drive mechanisms, and would result in more expensive components than those needed by a breadboard type of machine with properly shaped mounting plates attached to a welded stand. Thus it was decided that the construction of a machine such as that shown in Figure 18 would be both less costly and more useful than modifying the existing equipment.

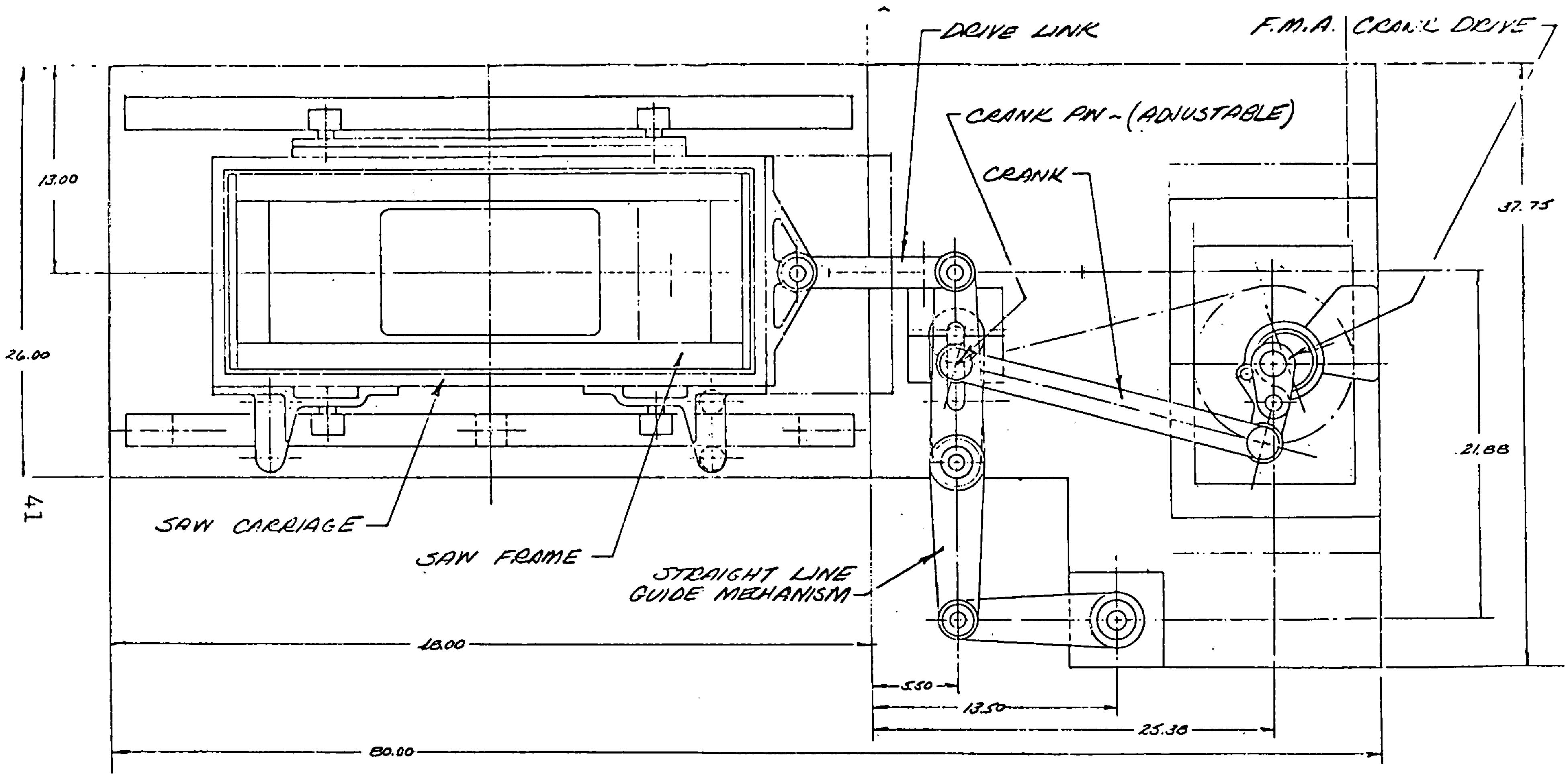


Figure 18. Crystal Slicer - Plan View

The higher speed multi-wire slicing machine is shown in plan view in Figure 18. The saw carriage is an aluminum weldment that is reciprocated on ground steel rails. The saw frame is aligned and bolted in place after stringing. The carriage is moved back and forth by a link guided by a straight line motion mechanism which is crank actuated. By shifting the crank pin location on the guide mechanism, different strokes can be obtained. Beneath the guide mechanism is a second crank which has a mass equal to the carriage weight attached to its end. This crank is driven 180° out of phase with the carriage motion, and thus serves to damp the inertial loads of the system. An FMA developed sinusoidal crank drive mechanism has been adapted to the output cranks to provide equivalent accelerations at each end of the stroke.

This counterbalanced drive arrangement is mounted on its own plate and shock isolated from the machine frame. This arrangement should radically reduce vibration to the saws and the crystal mounting. The crystal mount is attached to an air/oil cylinder-driven rocking assembly which attaches to a roller slide mounted to a bracket on the back of the rail support plate. Feed mechanism is shown in Figure 19. Vertical support of the slide and crystal mount is provided by a Bellofram cylinder attached to the machine frame.

Feed force is controlled by adjusting the air pressure into the Bellofram cylinder. Carriage speed is adjusted by changing

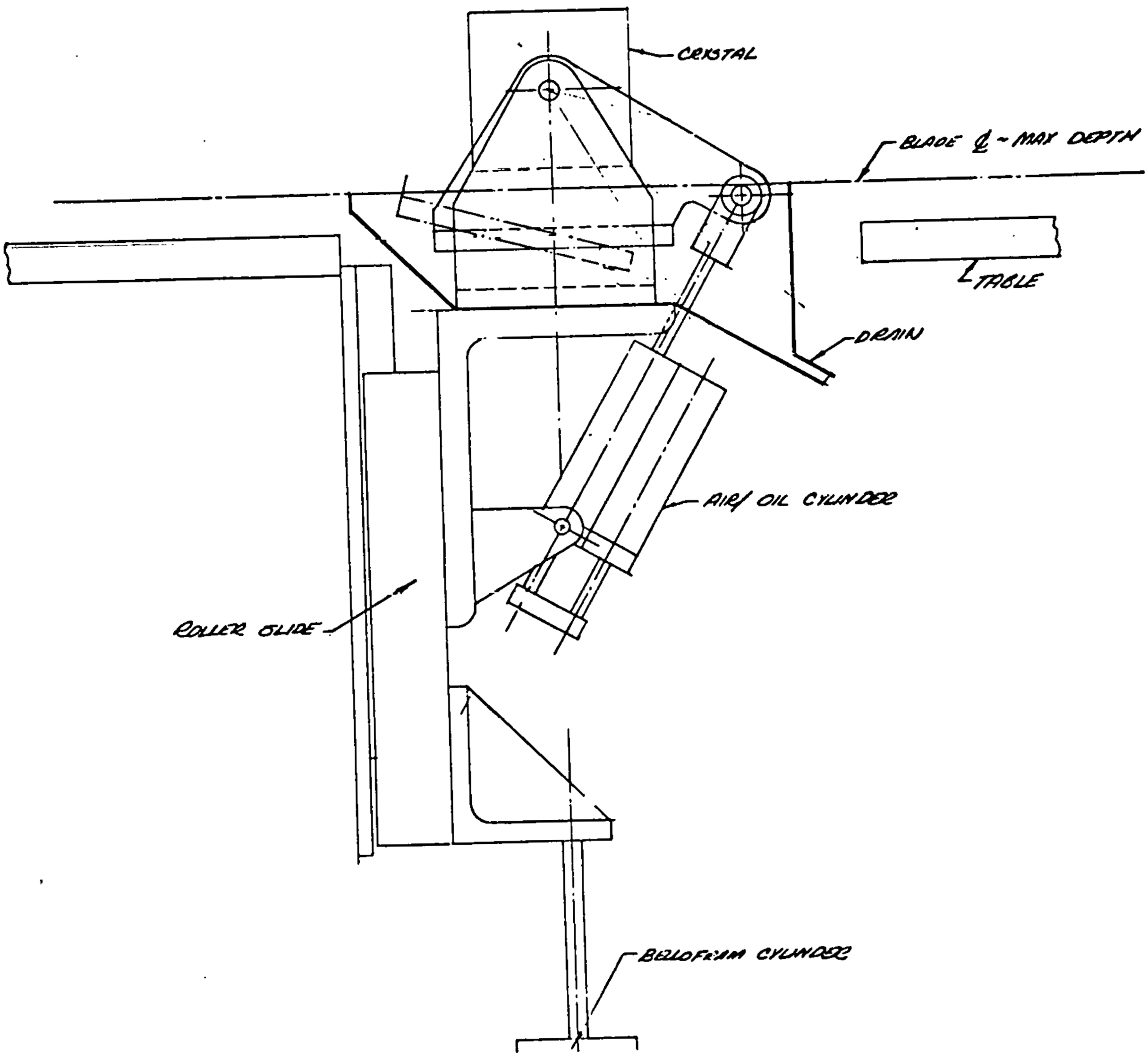


Figure 19. Feed Mechanism

the input speed to the drive gear box by a variable pitch V-belt drive from a 5 HP motor. Stroke length may be changed from 12" to 16" by shifting the location of the crank input pin on the guide mechanism. Crystal rocking action is controlled by adjusting the air/oil cylinder stroke up to angles of $\pm 15^{\circ}$.

The higher speed multi-wire slicer is being detailed and will be ready for fabrication soon.

CONCLUSIONS

1. Solar cells fabricated out of HEM cast material have shown conversion efficiencies of 13.3-14% under AM1 illumination.

2. A crack-free silicon ingot has been cast in a graded high purity semiconductor grade silica crucible.

3. A high degree of crystallinity has been achieved in the cast ingots.

4. The use of graphite plug through the bottom of silica crucibles has improved heat flow and resulted in more rapid growth to the top surface of the ingot.

5. The absence of graphite retainers and a rapid heat-up cycle has resulted in lowering of impurities on the surface of the melt.

6. The low-cost custom made square crucibles do not have a fully graded structure. These crucibles were heat treated to improve delamination and best results were obtained with 1200°C heat treatment.

7. A duplex crucible with a high purity liner was heat treated to induce microcracking and thereby delamination. The liner was too thick causing ingot cracking.

8. Kerf loss was reduced to 0.155 and 0.175 mm respectively by developing electroplated wires with 22 μm diamonds and by

impregnating wires using the Crystal Systems impregnation technique. In both cases high quality wafers were produced with nearly 100% yields.

9. The abrasive life of impregnated wire blades was not improved by increasing electroless nickel plating thickness from 7.5 μm to 12 μm. The extra plating seems to bury the diamonds. A thinner copper sheath and a thicker nickel coating is expected to hold the diamonds without burying them.

10. Abrasive life of plated impregnated blades was increased by hardening the electroless nickel-plated layer.

11. Higher feed forces, i.e., 90.8 vs. 36.3 gms per wire, increased cutting rate from 35 to 142 μm per minute, as well as surface damage from 8 to 18 μm.

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MILESTONES

ITEM	DESCRIPTION	1977 N	D	1978 J	F	M	A	M	J	J	A	S	O	N	D
1	2.5 kg HEM crack-free ingot/square cross-section greater than 10 cm x 10 cm	▲	━	━	━	━	—	—	—	—	—	—	△		
2	Design/fabricate wafering machine		▲	━	━	━	—	—	—	—	—	—	—	—	△
3	Ingot samples/wafer samples			▲	━	━	—	—	—	—	—	—	—	—	△
4	Characterization			▲	━	━	—	—	—	—	—	—	—	—	△
5	Crucible development		▲	━	━	━	—	—	—	—	—	—	—	△	
6	Crucible cost studies		▲	━	━	━	—	—	—	—	—	—	—	—	△
7	Growth rate studies		▲	━	━	━	—	—	—	—	—	—	—	—	△
8	Blade development		▲	━	━	━	—	—	—	—	—	—	—	—	△
9	Fabricate/evaluate solar cells					▲	—	—	—	—	—	—	△		
10	Design analysis study		▲	━	━	━	—	—	—	—	—	—	—	—	△
11	Monthly report			▲	▲		△	△		△	△		△	△	
12	Quarterly report		▲			▲			△			△			
13	Annual report														△

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