

## Large-Area Silicon Sheet Task

A.D. Morrison, Chairman

Presentations were made by seven contractors and by JPL on silicon (Si) sheet efforts and related work.

Westinghouse Electric Corp. reviewed progress on the Si dendritic-web growth contract. A set of new computer models was used successfully to define a growth system configuration that was then built and used to grow web with lower thermally generated stress than has any configuration previously developed by empirical means.

Mobil Tyco Solar Energy Corp., which is conducting research on the edge-defined film-fed growth (EFG) method of making Si ribbon, reported that a significant increase in cell efficiency was demonstrated in large areas ( $50 \text{ cm}^2$ ) of ribbon grown at high speed ( $3.5 \text{ cm/min}$ ). The best cells gave 11.7% efficiency (AM1 and AR coated), just short of this year's goal of 12%.

In the Semix Inc. semicrystalline cast Si program, a technique was developed to determine base resistivity and carrier lifetime in semicrystalline wafers. Also,  $100\text{-cm}^2$  cells of 13.5% efficiency (AM1) were made in limited quantities.

Kayex Corp., which has just completed its effort on advanced Czochralski Si ingot growth, reviewed achievements since contract inception. These included automated growth of 150 kg of 15-cm-dia ingot material per crucible, with after-growth yields of greater than 90% at throughputs of 1.5 kg/h.

Cornell University reported on scanning transmission electron microscopy (STEM) and microprobe investigations of processed EFG ribbon. The following process-induced changes in the defect structure (as compared to unprocessed EFG ribbon) were noted:

- (1) Processing introduces regularly spaced, sub-boundary-like dislocation arrays in the bulk (base section) of the material.
- (2) Some (but not all) of the dislocation nodes in these networks act as nucleation centers for small ( $d < 100 \text{ Angstrom}$ ) precipitates.
- (3) Large precipitates ( $d \approx 1 \mu\text{m}$ ) are formed in the bulk of the material.

The chemical composition of the large precipitates was studied by non-dispersive X-ray analysis in a JEOL 200 CX STEM and by dispersive analysis (for C) in a JEOL 733 Superprobe. Elements identified were Ti, Fe, W, Mo, Cl, Ca and C. None of these elements were found in the matrix. It appears that the precipitates act as gettering centers for impurities. Cl is traceable to the Cl bakeout of the graphite dies.

The structural arrangement and the electrical activity of dislocations at or close to the central twin plane in processed material was studied by electron-beam induced current (EBIC) microscopy on a shallow-bevel specimen.

## LARGE-AREA SILICON SHEET TASK

The majority of the dislocations in the twin plane are regularly spaced and mostly straight arrays of dislocation of like sign, accommodating a tilt component. Dislocation density in the twin plane is high, and the dislocations are effective recombination centers. Inspection of straight sections under higher magnification shows that the electrical activity varies along the dislocation, possibly due to precipitates (TEM will be carried out after completion of EBIC to clarify this point). Temperature-dependent EBIC is being carried out in order to determine the electronic energy levels associated with the various sections.

JPL in-house research program results were presented on the electrical and structural properties of grain boundaries in silicon, particularly those concerning electrical and enhanced diffusion along the grain boundaries.

Temperature-dependence measurements of zero-bias conductance, a photo-conductivity technique, and deep-level transient spectroscopy (DLTS) were developed to investigate potential barrier, carrier recombination velocity, and electronic states, respectively. The studies of potential barrier have revealed that considerable variation in the activation energy along grain boundaries often exists, presumably due to variation of local disorders; the activation energy usually increases with annealing temperature, and the potential barrier decreases with increasing light intensity. The recombination velocity measurements show that the velocity increases with boundary state density and light intensity. The preliminary result from the DLTS experiments indicates a trend: the density of states generally increases with the distance from the edges of the band gap. However, the details vary considerably from sample to sample, a result that can be attributed to local variation of disorders.

A grooving and staining technique, secondary ion mass spectroscopy, and EBIC measurements in scanning electron microscopy were used to study enhanced diffusion of phosphorus at grain boundaries in polycrystalline silicon. The results show that the enhanced diffusion occurs only at high-order grain boundaries having high carrier recombination and the depth of the enhanced diffusion varies drastically from boundary to boundary, making any quantitative measurement difficult unless the boundary can be characterized well.

The University of Illinois at Chicago is studying the fundamental mechanisms of abrasion and wear and the deformation of Si by a diamond in various fluid environments. The abrasion rates and depths of damage of  $\langle 100 \rangle$  and  $\langle 111 \rangle$  p-type Si in three fluid environments (acetone, ethanol, and water) were determined, and the surface deformation mechanism was found to change when the fluid was varied.

Applied Solar Energy Corp. presented results on the efficiency of solar cells made from EFG ribbon and Semix Inc. material. For EFG material, a baseline process was applied to ribbons grown with or without  $\text{CO}_2$  in the ambient. In general, cells made from EFG ribbon grown in  $\text{CO}_2$  performed better. However, the results from both groups were lower than those reported previously.

For the Semix material, work continued on Ingot 5848-13C. High-efficiency processes were applied and the results were presented. A series of more severe gettering schedules was performed on identified portions of the

## LARGE-AREA SILICON SHEET TASK

ingot. It was shown that short-circuit current improved with gettering up to a limiting value (which was still below that of the control cell). Light-biased diffusion length measurements showed that there was a negative light-biased effect (the minority carrier diffusion length decreased) that limited the improvement of short-circuit current in the more severely gettered cells. Also, the baseline process was applied to 10 x 10-cm Semix wafers randomly selected (not from a single ingot).

## ADVANCED DENDRITIC WEB GROWTH DEVELOPMENT

WESTINGHOUSE ELECTRIC CORP.

<b><u>Technology</u></b> Single crystal ribbon growth	<b><u>Report Date</u></b> 04/21/82
<b><u>Approach</u></b> Silicon dendritic web growth <b><u>Contractor</u></b> Westinghouse Electric Corp. Research & Development Center JPL Contract 955843	<b><u>Status</u></b>  <ul style="list-style-type: none"> <li>• First generation computer models developed and verified</li> <li>• Experimental web growth machine completed and operational</li> <li>• Melt-replenished steady-state web growth demonstrated at intermediate growth rate of 7 cm<sup>2</sup>/min</li> <li>• Web growth rate of 27 cm<sup>2</sup>/minute demonstrated under transient conditions</li> <li>• Growth width To 5 cm demonstrated</li> </ul>
<b><u>Goals</u></b> <ul style="list-style-type: none"> <li>• Develop computer models for characterizing and understanding web growth</li> <li>• Develop experimental web growth machine for use with models</li> <li>• Demonstrate melt-replenished steady-state web growth</li> <li>• Demonstrate 25 cm<sup>2</sup>/minute web growth rate</li> <li>• Demonstrate 5 cm web growth width</li> </ul>	

## Program Emphasis

- Deformation Is Major Limitation Of Ribbon Width And Throughput Rate
- Deformation Is Correlated To Thermally Generated Stress
- Computer Models Provide Understanding Of Web Growth And Thermal Requirements For Stress Reduction And Optimized Throughput

## **LARGE-AREA SILICON SHEET TASK**

### **Principal Activity**

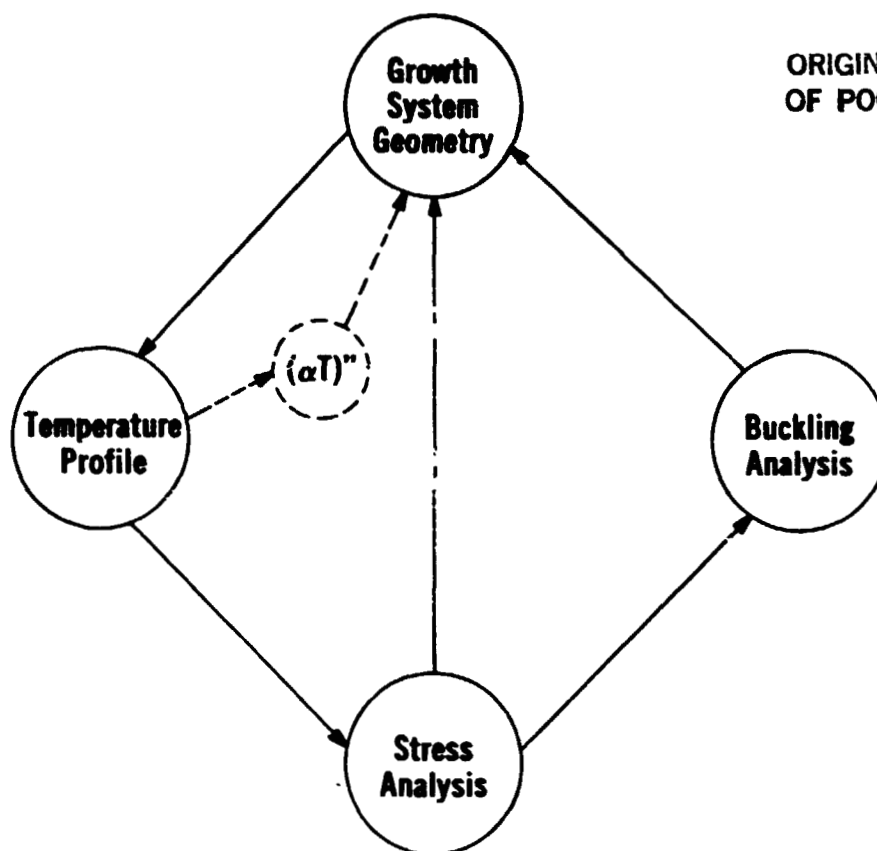
- **Develop Computer Models For Web Growth**
- **Develop Experimental Web Growth Machine Capable Of Automated, Melt-Replenished, Steady-State Growth**
- **Utilize Computer Models And Experimental Growth Machine For Development Of Advanced Web Growth**

### **Computer Models to Characterize Web Growth**

- **Compute Web Temperature Distribution Generated By A Specified Growth Configuration**
- **Compute Thermal Stress Generated By A Specified Web Temperature Distribution**
- **Compute The Critical Buckling Conditions For A Specified Thermal Stress**

## LARGE-AREA SILICON SHEET TASK

### Application of Computer Models

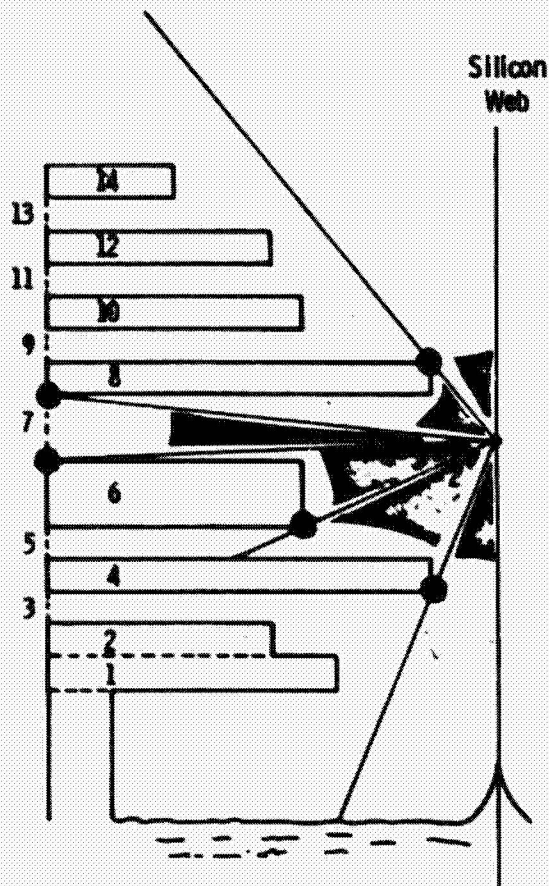


### Status of Computer Models

- Web Temperature Model Has Been Expanded To Be More Definitive And Has Been Verified As Adequate For Next Generation Of Increased Web Throughput
- Thermal Stress Model Is Complete And Verified
- Critical Buckling Model Is Complete And Verified

## LARGE-AREA SILICON SHEET TASK

### Viewing Regions From a Point on the Web



### Steady-State Web Growth Is Necessary

- For Process Analysis, Understanding And Improvement
- For Subsequent Process Standardization

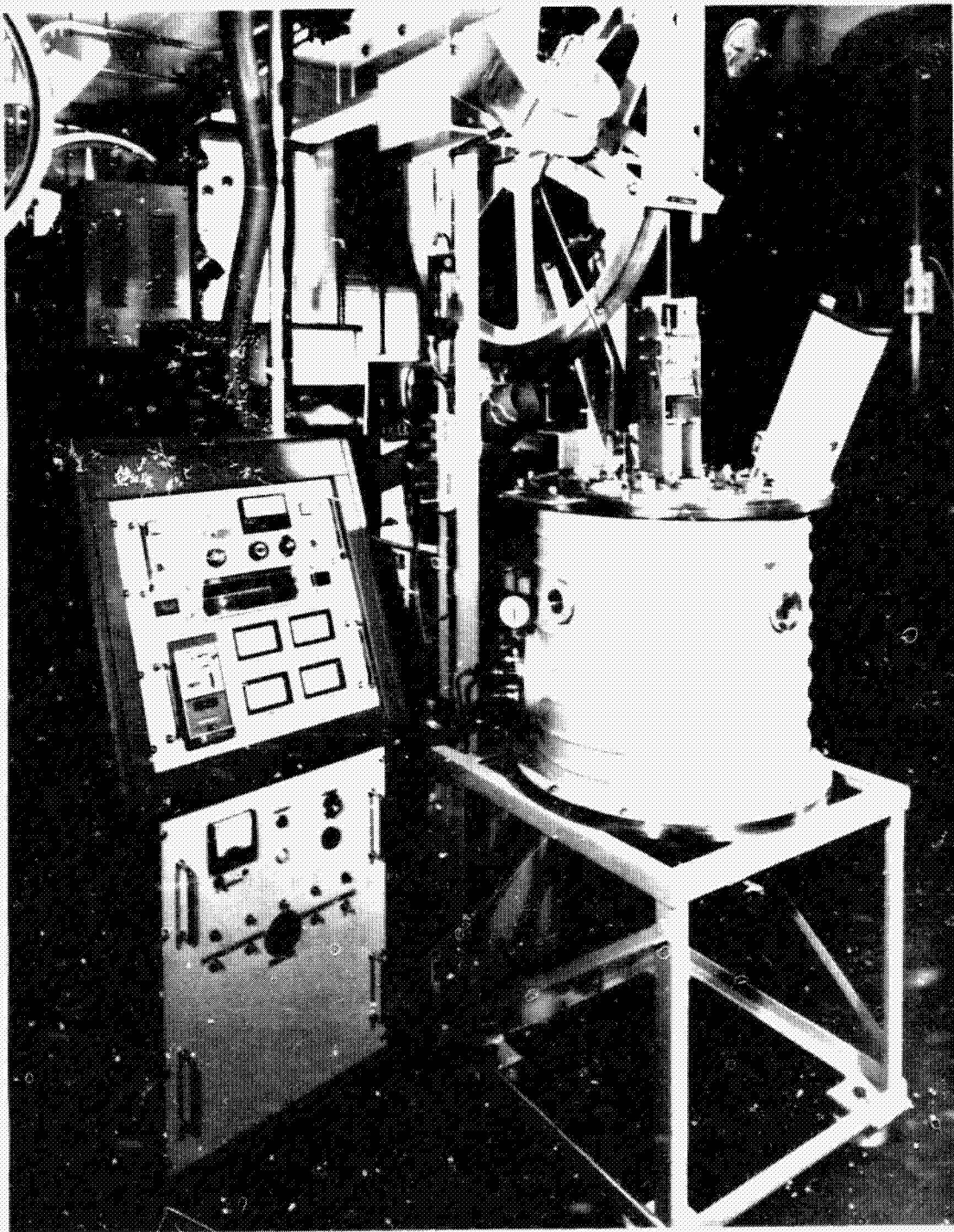
## **LARGE-AREA SILICON SHEET TASK**

**Experimental Web Growth Machine Provides Basic Requirements for Automated Steady-State Growth:**

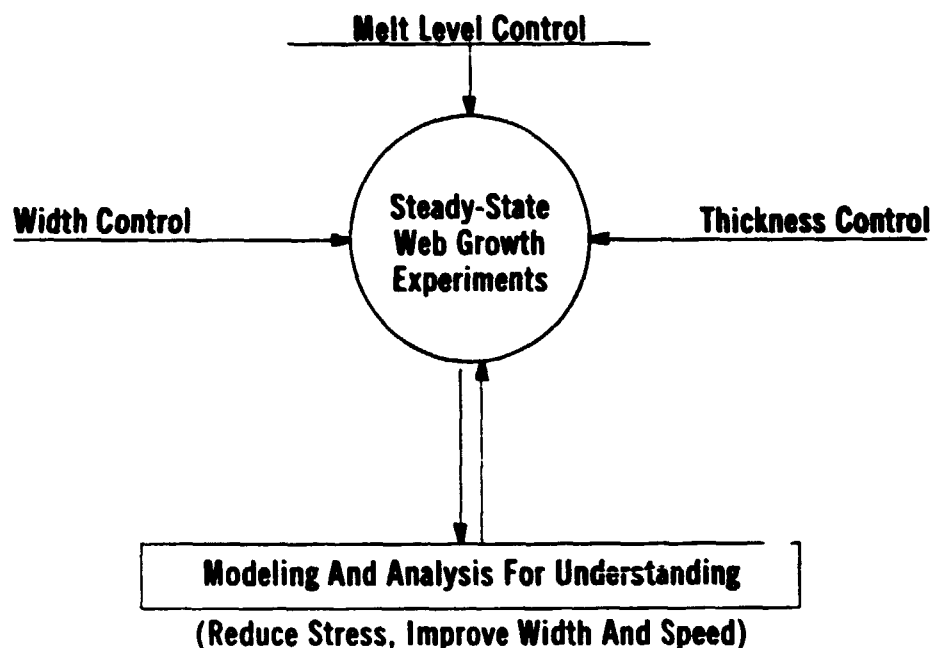
- **Flat Temperature Profile In Growth Region Of Melt**
- **Controlled Constant Temperature**
- **Controlled Constant Melt Level**
- **Controlled Constant Thickness**
- **Controlled Constant Width**



Dendritic Web Experimental Sheet Growth Unit (ESGU)



## Combined Use of Models and Experimental Web Growth



## Results of First Use of Models

- In First Application Models Were Verified By Comparison With A Previously Characterized Growth Configuration. The Model Identified System Limitations And Suggested Modifications Which Resulted In Width Increase Of 25% (To 5 cm)
- Automated Steady-State Web Growth Achieved At Intermediate Rate ( $7 \text{ cm}^2/\text{minute}$ )
- Use Of Models Proven As Route For Understanding And Improvement Of Web Growth

## **LARGE-AREA SILICON SHEET TASK**

### **Problems and Concerns**

- **Present - None. Understanding And Improvement Of Process Proceeding As Planned**
- **Future - Availability Of Low-Cost Polysilicon In Pellet Form**

### **Summary**

- **Computer Models Of Web Growth Completed And Verified**
- **Experimental Web Growth Machine Proven In Automated Steady-State Growth**
- **Web Growth Improved By Application Of Models**

## EDGE-DEFINED FILM-FED GROWTH

MOBIL TYCO SOLAR ENERGY CORP.

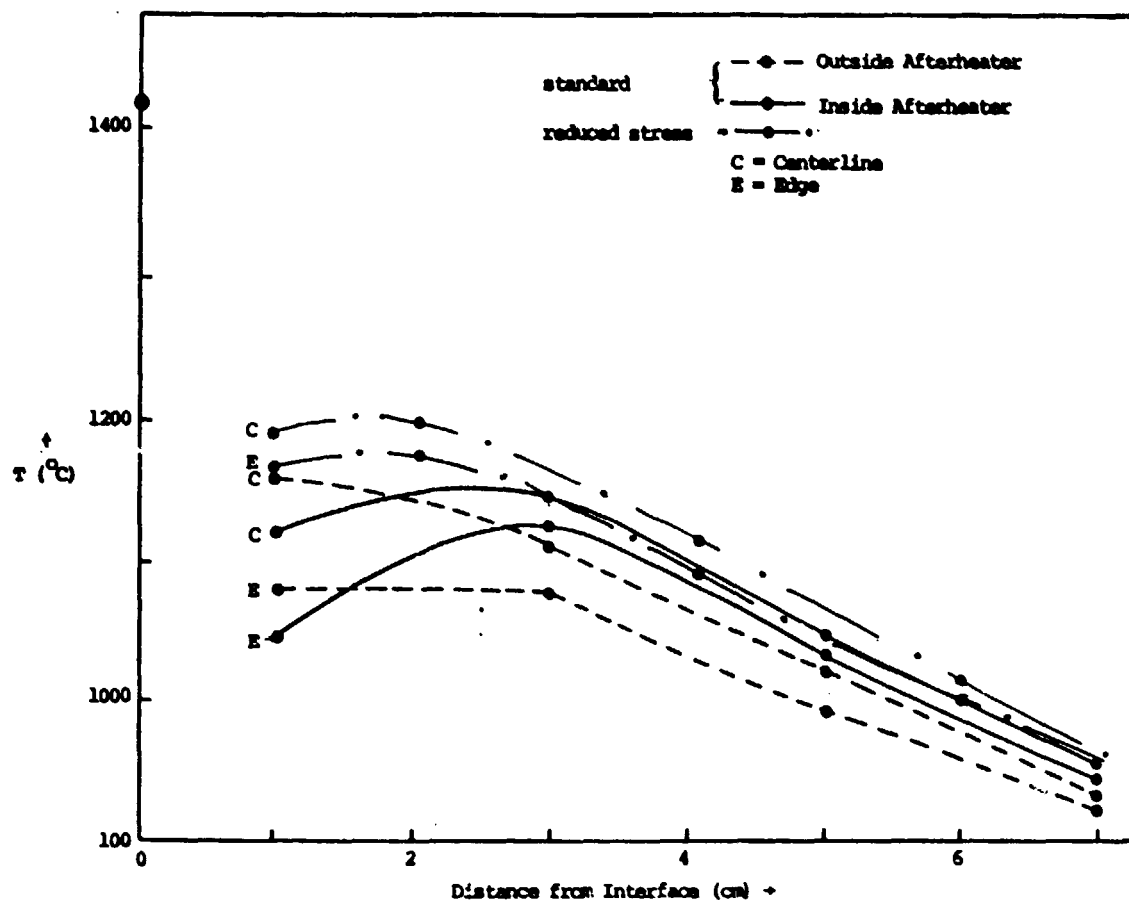
<b>TECHNOLOGY</b> LARGE AREA SILICON SHEET BY EFG	<b>REPORT DATE</b> 4/21/82
<b>APPROACH</b> MULTIPLE GROWTH OF 10 CM WIDE SILICON RIBBON AT 4 CM/MINUTE.  <b>CONTRACTOR</b> MOBIL TYCO SOLAR ENERGY CORPORATION	<b>STATUS</b> <ul style="list-style-type: none"> <li>● CARTRIDGE DESIGN CHANGES HAVE REDUCED RIBBON STRESS AND BUCKLING LEVELS FOR 200 <math>\mu</math>M THICK RIBBON TO LEVEL WHERE FABRICATION OF LARGE AREA CELLS IS POSSIBLE.</li> <li>● LARGE AREA (50 <math>\text{cm}^2</math>) CELLS OF 11.7% (AM1 AND AR COATED) HAVE BEEN ACHIEVED FOR RIBBON GROWN IN HIGH SPEED SYSTEM (AT 3.5 CM/MINUTE).</li> <li>● NEW MULTIPLE RIBBON FURNACE HAS BEEN BUILT AND TESTED (OPERATION HAS BEEN SET ASIDE DUE TO REDUCTION OF PROGRAM).</li> </ul>
<b>GOALS</b> <ul style="list-style-type: none"> <li>● REDUCE STRESS AND IMPROVE FLATNESS OF RIBBON GROWN AT 4 CM/MINUTE AND 200 <math>\mu</math>M THICKNESS.</li> <li>● DEMONSTRATE 12% CELL EFFICIENCY ON LARGE AREAS (50 <math>\text{cm}^2</math>) FOR RIBBON GROWN IN HIGH SPEED SYSTEM.</li> <li>● DESIGN, CONSTRUCT AND OPERATE NEW MULTIPLE RIBBON FURNACE FOR GROWTH OF FOUR 10 CM WIDE RIBBONS.</li> </ul>	

## Progress in Stress Studies

- INFLUENCE OF CARTRIDGE COMPONENT DESIGN ON STRESS AND BUCKLING LEVELS IN 10 CM WIDE RIBBON HAS BEEN IDENTIFIED:
  - TEMPERATURE FIELDS IN LINEAR COOLING PLATES OF SEVERAL DIFFERENT DESIGNS HAVE BEEN MEASURED.
  - IMPROVED FLATNESS WAS ACHIEVED IN 200  $\mu$ M THICK RIBBON GROWN WITH A MODIFIED DESIGN CARTRIDGE.
  - CHANGE IN HORIZONTAL ISOTHERM SHAPE IS PROBABLE CAUSE FOR REDUCED STRESS AND BUCKLING LEVELS.

# LARGE-AREA SILICON SHEET TASK

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Comparison of center and edge temperature profiles in 10 cm cartridge linear cooling plates.

### Stress Studies: Plans

- CAPABILITY IS NEEDED TO PREDICT: (i) MOVING RIBBON TEMPERATURE PROFILES GIVEN SYSTEM COMPONENT TEMPERATURES AND GEOMETRY, AND (ii) STRESS DISTRIBUTIONS AND BUCKLING THRESHOLDS FROM RIBBON TEMPERATURE FIELDS.
- EMPIRICAL APPROACH WILL: (i) GUIDE THEORETICAL STUDIES AND ESTABLISH BOUNDARY CONDITIONS FOR MODELING, AND (ii) ARRIVE AT REDUCED STRESS GROWTH CONFIGURATIONS THAT ARE COMPATIBLE WITH ACCEPTABLE GROWTH CONDITIONS AT 4 CM/MIN.

IMPORTANT TO SYNTHESIZE OUT OF THIS APPROACH A SYSTEM THAT WILL PRODUCE 200  $\mu$ m THICK RIBBON AT 4 CM/MIN WITH SUFFICIENTLY LOW STRESS AND FLATNESS TO MEET DEMANDS IN YIELD AREA.

### Progress in Quality Improvement

- SIGNIFICANT IMPROVEMENT IN CELL EFFICIENCY OF HIGH SPEED GROWN RIBBON TO 11-12% HAS BEEN ACHIEVED.
  - AMBIENT CONTROL HAS PROVEN TO BE VERY IMPORTANT PARAMETER IN OBTAINING CONSISTENT ELECTRONIC QUALITY RIBBON.
  - RIBBON EXIT GAS SEAL, MORE UNIFORM INTERFACE GAS CONTROL SYSTEMS IMPLEMENTED SUCCESSFULLY.

# LARGE-AREA SILICON SHEET TASK

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SPV Diffusion Length Data for 10 cm Wide Ribbon Grown with  
Stretched Cartridge in Machine 17. Bulk Melt Doping was  
4  $\Omega$ -cm for All Runs.

Run No. and Segment	M.Z. Argon Flow Rate ( $\ell$ /minute)	Cartridge Gas Composition	Cartridge Gas Flow Rate ( $\ell$ /minute)	Gas Seal	Speed (cm/minute)	$L_D$ ( $\mu$ m)
17-199-1A	10	argon	1.5	no	3.0 - 3.2	23.0
-1C					3.8	35.0
17-200-1A	6	argon	1.0	no	3.5	33.0
-1D		0.45% $CO_2$ + 45 ppm $O_2$	1.8		3.8 - 3.9	43.0
17-201-1B	6	argon	1.0	yes	3.3	33.6
-1D	3				3.3 - 3.4	30.0
17-202-1A	3			yes	3.5 - 3.6	43.0
-1B	2				3.8	39.0
-1C	2				3.8	52.0
-1D	0				3.8	36.0
17-203-1A	6	argon	1.0	yes	3.3	41.0
-1C		0.17% $CO_2$ + 18 ppm $O_2$	1.2		3.5	45.0
17-204-1A	6	argon	1.0	yes	3.5	49.0
-1C		0.29% $CO_2$ + 29 ppm $O_2$	1.4		3.5	36.0
17-205-1B	6	0.17% $CO_2$ + 17 ppm $O_2$	1.2	yes	3.5	45.4

Solar Cell Data for Phosphine Processed Large Area ( $50 \text{ cm}^2$ )  
Solar Cells Made from 10 cm Wide Ribbons.

$100 \text{ mW/cm}^2$ , Xenon Light,  $28^\circ\text{C}$ , AR Coated.

Run No.	Growth Ambient	Speed (cm/min)	Average Resistivity ( $\Omega\text{-cm}$ )	Diffusion Length ( $\mu\text{m}$ )	Cell Parameters				
					$J_{sc}$ ( $\text{mA/cm}^2$ )	$V_{oc}$ (V)	FF	$\eta$ (%)	Mean $\eta$ (%)
17-143	0.2% $\text{CO}_2$	2.5	1.5	27	26.5	0.523	0.608	8.4	9.6
					26.5	0.531	0.705	9.9	
					27.7	0.534	0.677	10.0	
					26.2	0.530	0.699	9.7	
					28.6	0.538	0.634	9.7	
					26.2	0.529	0.717	9.9	
					26.6	0.533	0.806	9.9	
17-174	Quartz in melt	3.5	1.0	35	25.3	0.527	0.667	8.9	9.4
					26.7	0.534	0.697	9.9	
17-175	0.2% $\text{CO}_2$ + 30 ppm $\text{O}_2$	3.5	1.0	36	26.8	0.539	0.735	10.6	10.3
					27.7	0.545	0.706	10.7	
					26.1	0.537	0.720	10.1	
					27.5	0.547	0.641	9.7	
17-178	1% $\text{CO}_2$ + 100 ppm $\text{O}_2$	3.5	1.0	34	26.4	0.518	0.696	9.5	9.1
					26.2	0.517	0.642	8.7	
17-181	0.23% $\text{CO}_2$ + 23 ppm $\text{O}_2$	3.5	4.0	43	29.0	0.525	0.603	9.2	10.0
					28.8	0.522	0.713	10.7	
17-204	Stretched cartridge with gas seal 0.29% $\text{CO}_2$ + 29 ppm $\text{O}_2$	3.5	4.0	36	29.1	0.517	0.726	11.6	11.1
					28.3	0.530	0.629	9.5	
					29.7	0.541	0.688	11.1	
					29.3	0.546	0.732	11.7	
					29.8	0.537	0.704	11.3	
					30.5	0.542	0.669	11.1	
					29.8	0.546	0.716	11.7	



### EFG Ribbon Quality: Status

- HIGH SPEED GROWN RIBBON EFFICIENCIES AT 11-12% ARE STILL BELOW BEST EFG RIBBON AVAILABLE, WITH PARTICULAR SHORTFALL IN  $V_{oc}$  AND FF.
- ISSUES TO SETTLE:
  - WHAT ARE UNDERLYING MATERIAL QUALITY DEFICIENCIES?
  - CAN THIN RIBBON, PROCESSED WITH IMPROVED GETTERING AND BSF SCHEMES, ACHIEVE GOALS ON PRESENT MATERIAL?

### Annealing Studies

- HIGH TEMPERATURE HEAT TREATMENTS (800-1100°C) IN NITROGEN AND OXYGEN AMBIENTS LEAD TO CONSISTENT DEGRADATION OF (DARK) SPV DIFFUSION LENGTHS INDEPENDENTLY OF GROWTH CONDITIONS, INTERSTITIAL OXYGEN LEVEL OF RIBBON ( $CO_2$  ON OR OFF, OR QUARTZ IN THE MELT).
- $PH_3$  TREATMENT DURING ANNEAL CAN IMPROVE DARK DIFFUSION LENGTH AND APPEARS TO BE NECESSARY TO PRODUCE LIGHT ENHANCEMENT.

## LARGE-AREA SILICON SHEET TASK

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OF POOR QUALITYSchottky Barrier SPV Diffusion Length Measurements Before  
and After Heat Treatment of 10 cm Wide Ribbon in an  
Oxygen Ambient.

Heat Treatment	Growth Run	Growth Ambient	No. of Sample Pairs	Diffusion Length $L_B$ Before Heat Treat.			Diffusion Length $L_A$ After Heat Treat.			Ratio ( $L_A/L_B$ )		
				$\bar{x}$ ( $\mu\text{m}$ )	s ( $\mu\text{m}$ )	s/ $\bar{x}$	$\bar{x}$ ( $\mu\text{m}$ )	s ( $\mu\text{m}$ )	s/ $\bar{x}$	$\bar{x}$ ( $\mu\text{m}$ )	s ( $\mu\text{m}$ )	s/ $\bar{x}$
5 hrs at 800°C in O <sub>2</sub> . Sister sample measurement.	CZ	-	3				83.4	5.42	0.06			
	17-117	argon	8	43.9	21.0	0.48	33.3	7.94	0.24	0.78	0.64	0.65
	17-166	quartz in melt	7	48.7	17.5	0.40	34.3	7.97	0.23	0.78	0.28	0.36
	16-258	0.425 L/min of 1% O <sub>2</sub>	8	48.2	21.3	0.44	35.5	15.8	0.44	0.77	0.30	0.39
50 mins at 900°C in O <sub>2</sub> . Sister sample measurement.	CZ	-	3				73.7	15.3	0.21			
	17-117	argon	8	40.6	16.0	0.39	21.6	8.36	0.39	0.57	0.25	0.43
	17-166	quartz in melt	7	43.7	26.5	0.57	30.1	13.1	0.44	0.82	0.65	0.79
	16-258	0.425 L/min of 1% O <sub>2</sub>	8	51.0	25.4	0.50	29.0	13.8	0.48	0.60	0.24	0.30
7.5 hrs at 900°C in O <sub>2</sub> . Sister sample measurement.	CZ	-	4	111.0	5.29	0.05	32.6	2.79	0.08	0.294	0.025	0.086
	17-148	argon	6	35.7	8.8	0.25	17.0	9.89	0.58	0.57	0.27	0.48
	17-147	quartz in melt	6	37.2	13.9	0.37	15.7	14.3	0.91	0.41	0.36	0.87

 $\bar{x}$  = mean value; s = standard deviation.Schottky Bar. . SPV Diffusion Length Measurements  
Before and After H<sub>2</sub> Diffusion for 10 cm Wide  
Ribbon.

Heat Treatment	Growth Run	Growth Ambient	No. of Sample Pairs	Diffusion Length $L_B$ Before Heat Treat.			Diffusion Length $L_A$ After Heat Treat.			Ratio ( $L_A/L_B$ )		
				$\bar{x}$ ( $\mu\text{m}$ )	s ( $\mu\text{m}$ )	s/ $\bar{x}$	$\bar{x}$ ( $\mu\text{m}$ )	s ( $\mu\text{m}$ )	s/ $\bar{x}$	$\bar{x}$ ( $\mu\text{m}$ )	s ( $\mu\text{m}$ )	s/ $\bar{x}$
900°C diffusion: 10 mins O <sub>2</sub> /N <sub>2</sub> + 30 mins H <sub>2</sub> + furnace cool to 650°C in 100 mins. Same sample measurement.	CZ	-	2	155.0	2.12	0.01	146.0	1.13	0.01	0.94	0.02	0.02
	17-142	argon	7	28.3	12.0	0.42	38.8	11.6	0.30	1.43	0.31	0.22
	17-174	quartz in melt	7	31.3	6.86	0.22	27.6	5.76	0.21	0.88	0.32	0.35
	17-175	0.3% CO <sub>2</sub> + 30 ppm O <sub>2</sub>	5	37.7	7.12	0.19	37.5	23.1	0.62	1.11	0.95	0.95
			4	40.3	4.80	0.12	27.9	9.69	0.35	0.70	0.23	0.33
800°C diffusion: 1 hr in O <sub>2</sub> /N <sub>2</sub> + 1 hr in H <sub>2</sub> + 4 hrs in O <sub>2</sub> /N <sub>2</sub> . Sister sample measurement.	CZ	-	3	157.9	6.77	0.04	116.8	7.74	0.07	0.74	0.97	0.10
	17-117	argon	7	26.5	7.92	0.30	34.9	12.4	0.36	1.40	0.57	0.41
	17-175	0.3% CO <sub>2</sub> + 30 ppm O <sub>2</sub>	6	42.8	11.6	0.27	53.3	10.7	0.20	1.33	0.47	0.36
	17-177	1% CO <sub>2</sub> + 100 ppm O <sub>2</sub>	8	43.2	7.49	0.17	43.2	10.8	0.25	1.01	0.24	0.23
	17-174	quartz in melt	7	46.4	15.4	0.33	44.3	15.6	0.35	0.96	0.19	0.20

## LARGE-AREA SILICON SHEET TASK

### Status of EFG Multiple-Ribbon Program: Needs

- NEW FURNACE FOR FOUR 10 cm WIDE RIBBONS IS OPERATIONAL WITH AUTOMATIC WIDTH CONTROL AND MELT REPLENISHMENT. WORK TO BE DONE CONCERNS:
  - ESTABLISHING RELIABILITY, LIFETIME FOR FURNACE COMPONENTS IN LONG TERM OPERATION.
  - IMPLEMENTING AMBIENT CONTROL AT LEVEL WHICH ENSURES QUALITY CONSISTENCY.
- FUNDAMENTAL QUESTIONS REGARDING VIABILITY AND COST-EFFECTIVENESS OF FURNACE IN PRODUCTION MODE REMAIN:
  - YIELDS FOR THIN RIBBON MUST BE HIGH: LOW STRESS, FLATNESS AT 150-200  $\mu$ m THICKNESS, 4 cm/min ARE ABSOLUTE NECESSITY.
  - SIMULTANEOUS ACHIEVEMENT OF HIGH DUTY RATE, CONSISTENT QUALITY, HIGH YIELDS MUST OCCUR.

### Problems and Concerns

- STRESS AND NON-FLATNESS IN THIN (200  $\mu$ m) RIBBON DO NOT PERMIT FABRICATION OF RIBBON GROWN AT HIGHEST SPEEDS (~4 cm/minute) INTO CELLS WITH ACCEPTABLE YIELDS.
- DEMONSTRATE THAT BEST CELL PERFORMANCE LEVELS OF 11-12% ACHIEVED IN SINGLE CARTRIDGE FURNACES CAN BE OBTAINED CONSISTENTLY IN A MULTIPLE RIBBON FURNACE.
- DEMONSTRATE RELIABILITY OF MULTIPLE RIBBON FURNACE OPERATION OVER THE LONG TERM, WITH ACCEPTABLE DUTY RATES, THROUGHPUT AND MATERIAL QUALITY.

## SEMICRYSTALLINE PROCESS DEVELOPMENT

SEMIX INC.

<b>TECHNOLOGY</b> LARGE AREA SILICON SHEET	<b>REPORT DATE</b> April 22, 1967
<b>APPROACH</b>  SEMICRYSTALLINE CRYSTALLIZATION PROCESS DEVELOPMENT AND VERIFICATION  <b>CONTRACTOR</b> SEMIX INCORPORATED	<b>STATUS</b>  WORK IS CONTINUING TO DEVELOP AN UNDERSTANDING OF THE BASIC MECHANISMS IN THE CRYSTALLIZATION OF SEMICRYSTALLINE MATERIAL.  A TECHNIQUE HAS BEEN DEVELOPED TO DETERMINE BASE RESISTIVITY AND CARRIER LIFETIME IN SEMICRYSTALLINE WAFERS. EXTENSIONS OF THIS TECHNOLOGY ARE BEING PURSUED FOR EVALUATION OF UCP BRICKS.  SEMIX HAS ACHIEVED CELLS OF 13.5% EFFICIENCIES IN LIMITED QUANTITIES.  WE HAVE COMPLETED THE DESIGN PHASE AND ENTERED THE PROCUREMENT AND CONSTRUCTION PHASE OF A PROTOTYPE 3 UCP SYSTEM CAPABLE OF SOLIDIFYING 42 KILOGRAMS OF SILICON. WE ARE INVESTIGATING THE CRITICAL ELEMENTS NECESSARY TO DEMONSTRATE HIGH THROUGHPUT CAPABILITY.
<b>GOALS</b> INVESTIGATE STRUCTURAL PROPERTIES OF UCP MATERIAL.  ASSESS AND DEVELOP TECHNIQUES TO CHARACTERIZE SEMICRYSTALLINE MATERIAL.  DEMONSTRATE 14% AMI EFFICIENCY WITH 100 CM <sup>2</sup> SOLAR CELLS.  EVALUATE AND INVESTIGATE THE CRITICAL ELEMENTS NECESSARY FOR HIGH THROUGHPUT UCP SYSTEMS.	

## Principal Areas of Research

- A. FUNDAMENTAL STUDIES OF SEMICRYSTALLINE MATERIAL
- B. HIGH EFFICIENCY SEMICRYSTALLINE SOLAR CELLS
- C. WAFERING MECHANISMS

## Areas of Fundamental Study

### CRYSTALLIZATION

INVESTIGATION OF THE ROLE OF MICROSTRUCTURE ON THE  
PERFORMANCE OF SEMICRYSTALLINE SILICON, MOST NOTABLY  
THROUGH:

GRAIN ORIENTATION STUDIES, AND  
INVESTIGATION OF STRUCTURAL IN-  
HOMOGENEITIES

### CHARACTERIZATION

DEVELOPMENT OF TECHNIQUES FOR THE CHARACTERIZATION OF  
SEMICRYSTALLINE SILICON FOR BOTH:

RESISTIVITY  
CARRIER LIFETIME

## Crystallization

PURPOSE: TO DETERMINE THE ROLE OF GRAIN VOLUMES AND GRAIN  
BOUNDARIES ON THE QUALITY OF SEMICRYSTALLINE  
MATERIAL.

APPROACH: DETERMINATION OF THE RELATIVE ORIENTATION OF NEIGH-  
BORING CRYSTALLITES.

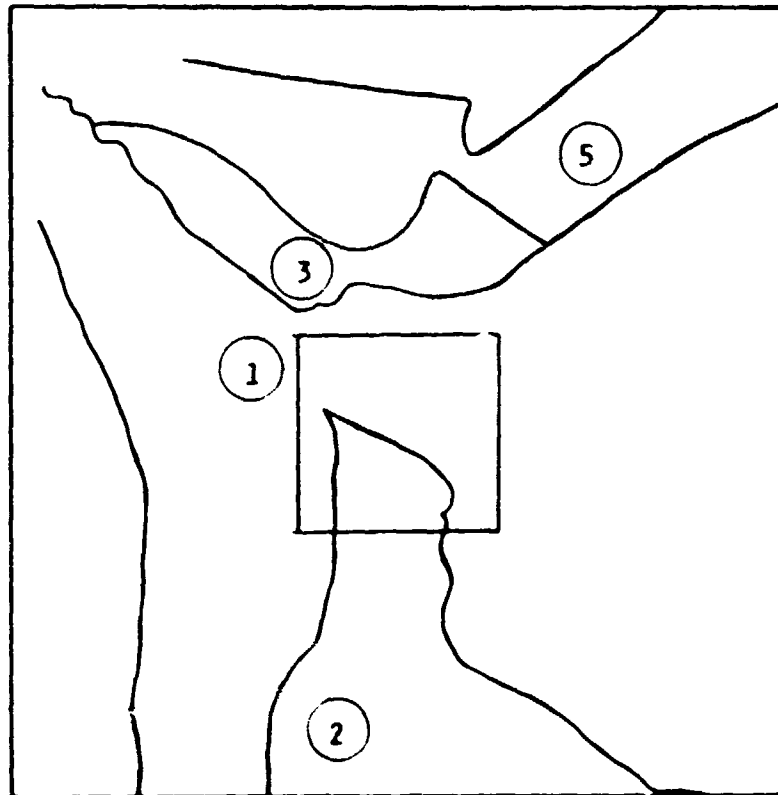
INVESTIGATION OF THE IMPACT OF MICROSTRUCTURAL  
DEFECTS ON CELL PROPERTIES:

INDIVIDUAL DISLOCATIONS

DISLOCATION SUBGRAIN BOUNDARIES

# LARGE-AREA SILICON SHEET TASK

## Relative Positions of Grains in Sample Semicrystalline Material



# LARGE-AREA SILICON SHEET TASK

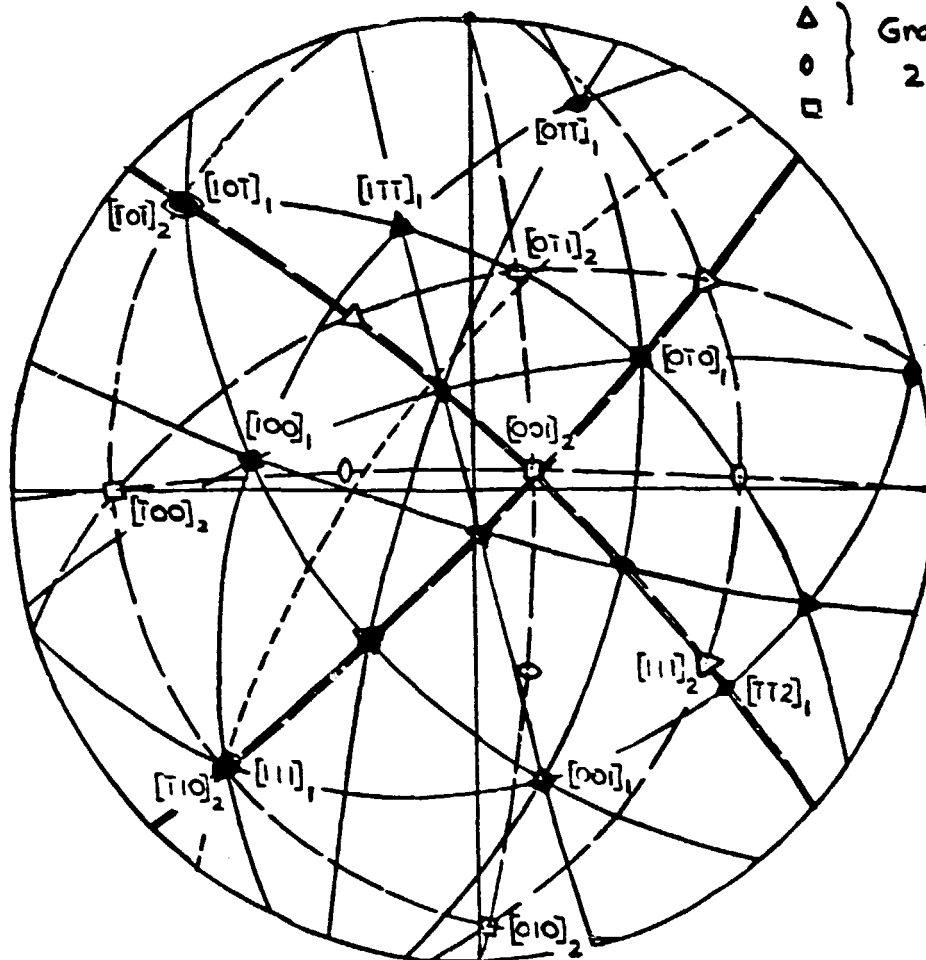
ORIENTAL POLYMER  
OF FOUR QUALITY

$\triangle$   $\langle 111 \rangle$   
 $\circ$   $\langle 110 \rangle$   
 $\square$   $\langle 100 \rangle$

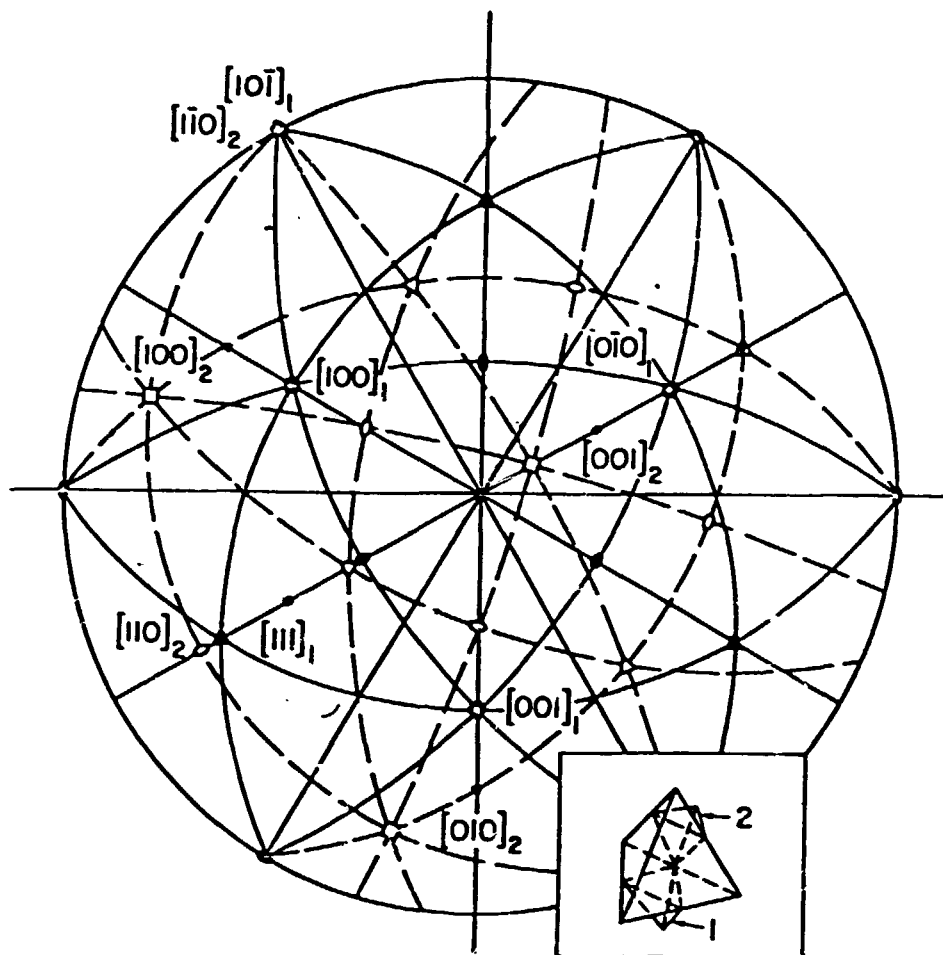
Grain  
1

$\triangle$   
 $\circ$   
 $\square$

Grain  
2

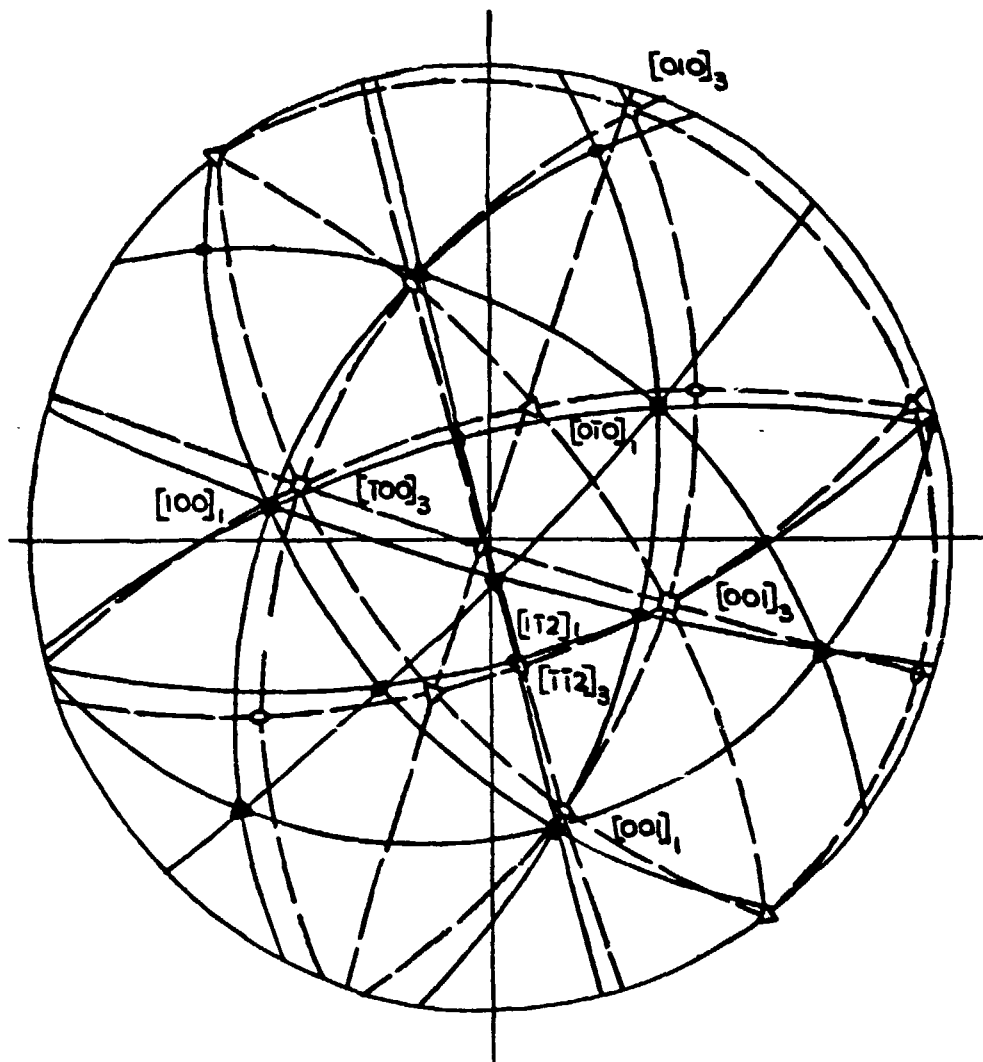


ORIENTATIONS OF GRAINS 1 and 2 IN SAMPLE SEMICRYSTALLINE MATERIAL

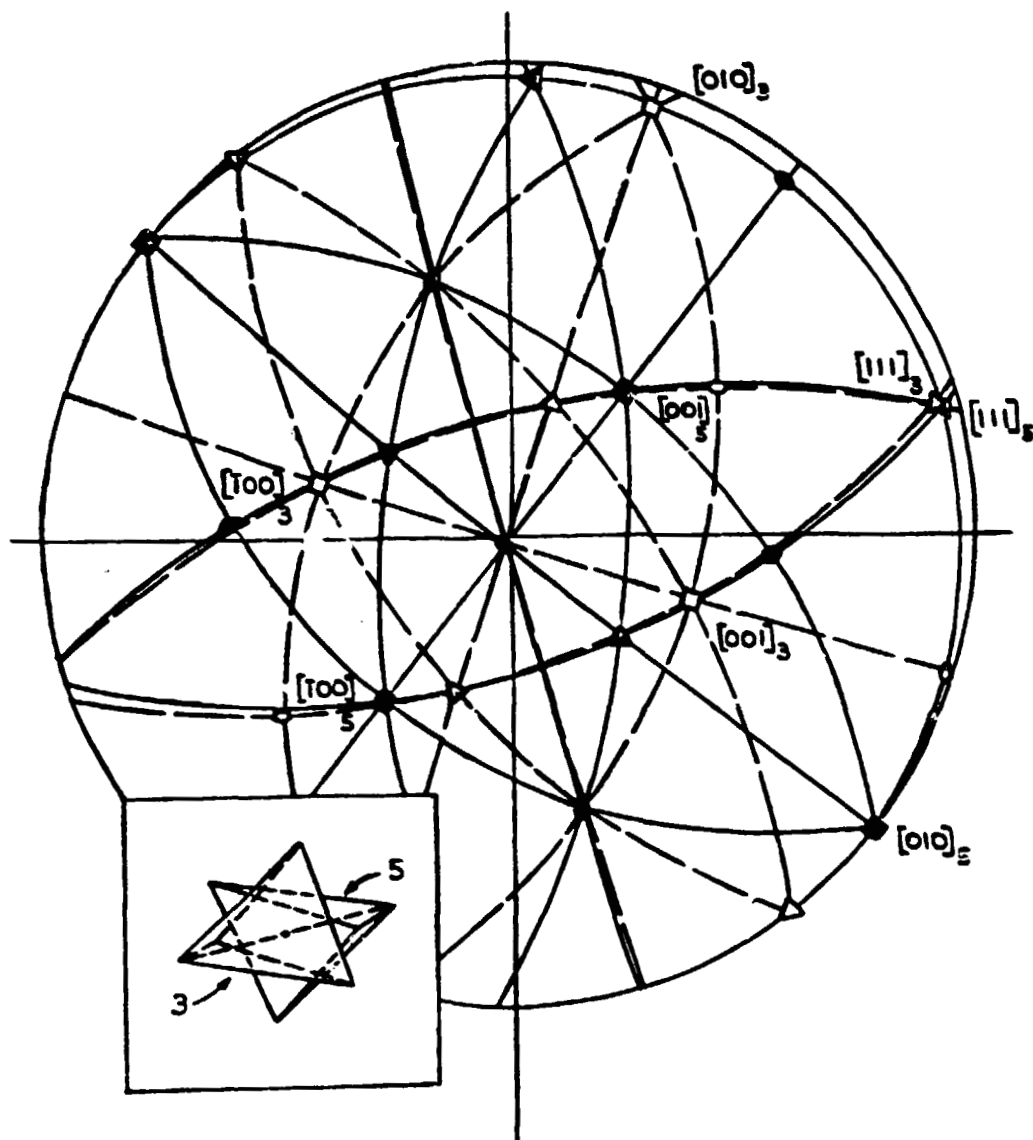


Second Order Orientation Relationship of Grains  
1 and 2 in {111} Twin Geometry Relative to a Third  
Grain





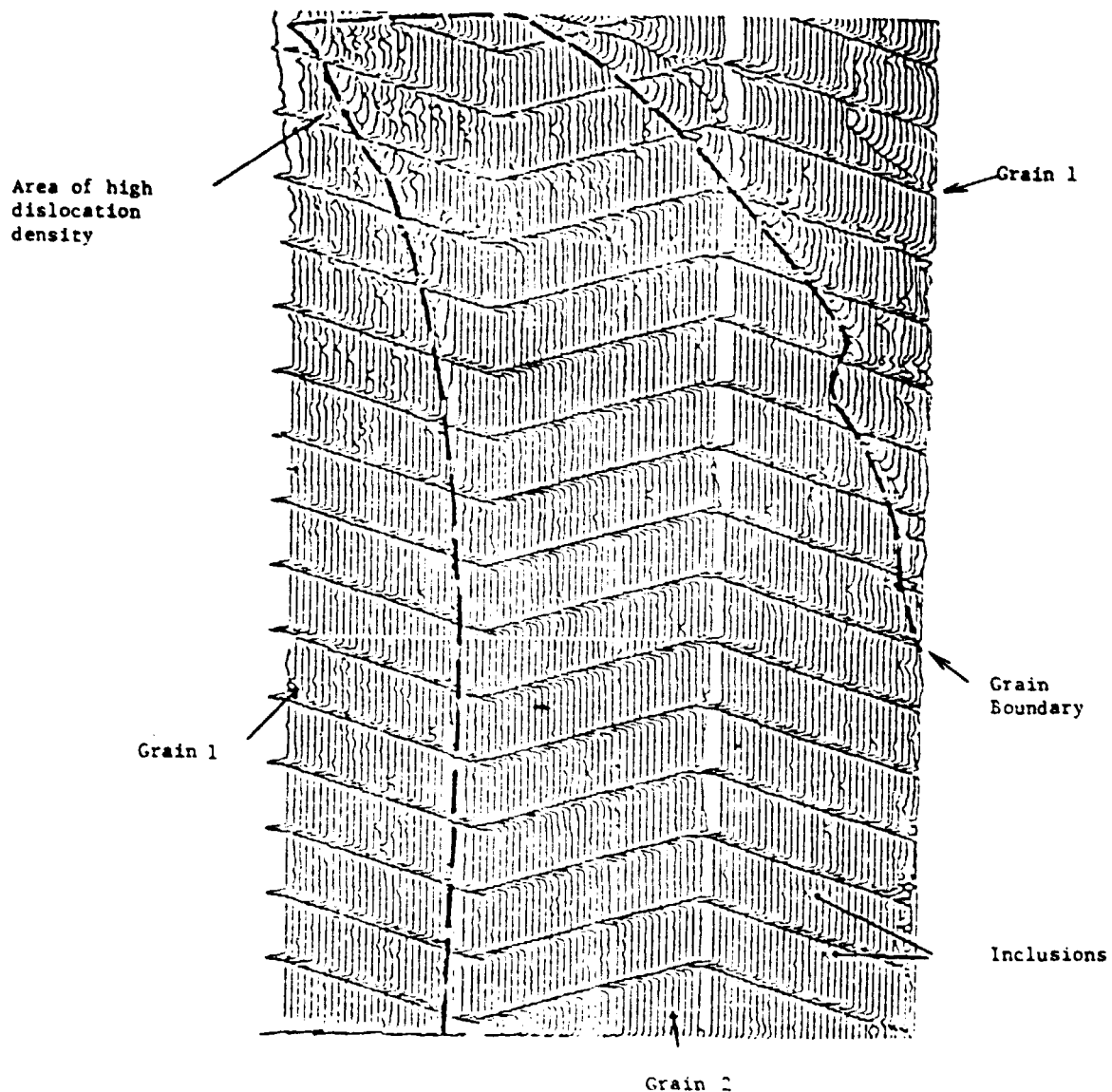
Orientation Relationship for Grains 1 and 3 of 90°  
Rotation About Nearly Common  $\langle 112 \rangle$  Axes



(111) Twin Orientation Relationship of Grains 3 and 5

# LARGE-AREA SILICON SHEET TASK

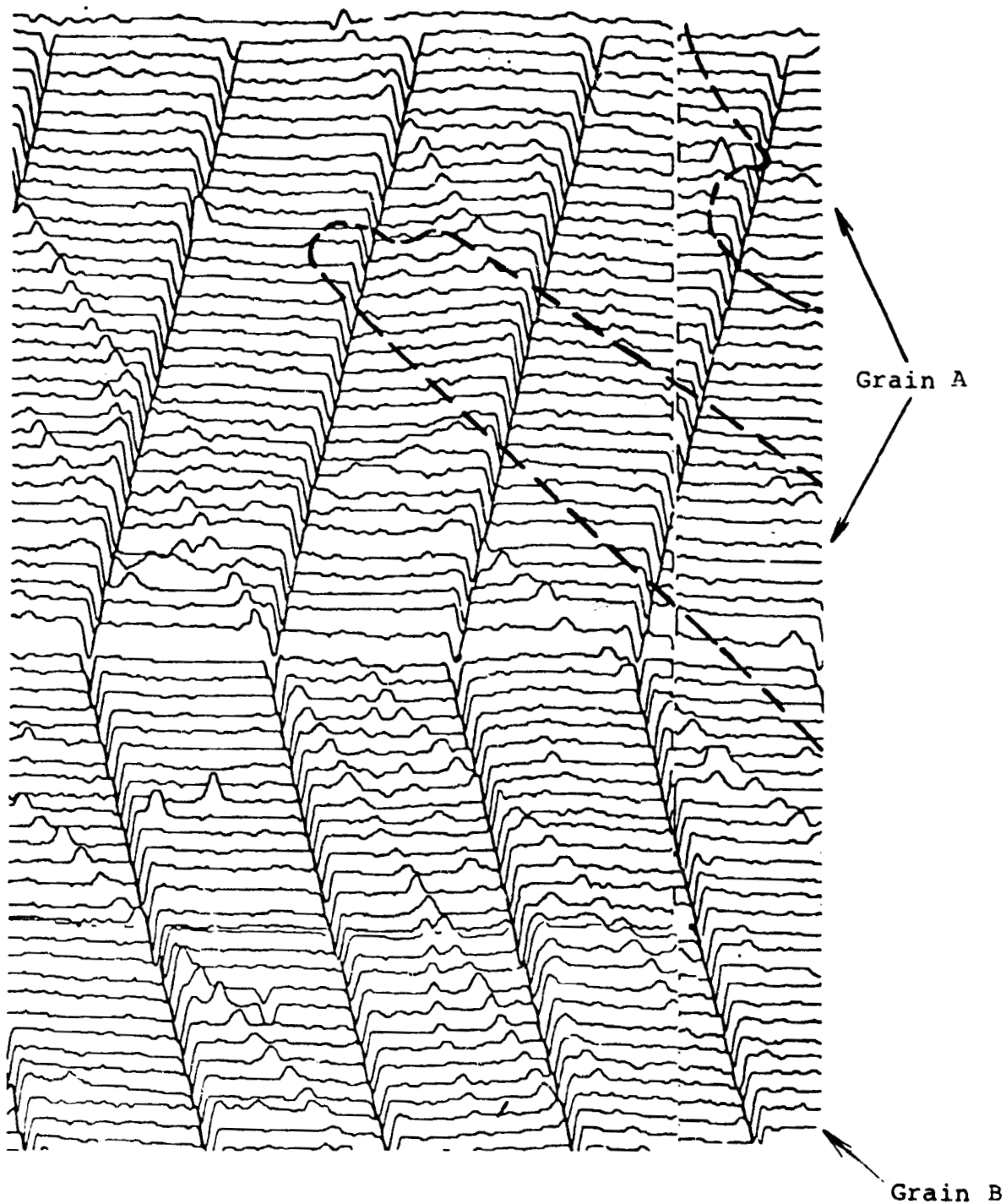
ORIGINAL FACE IS  
OF POOR QUALITY



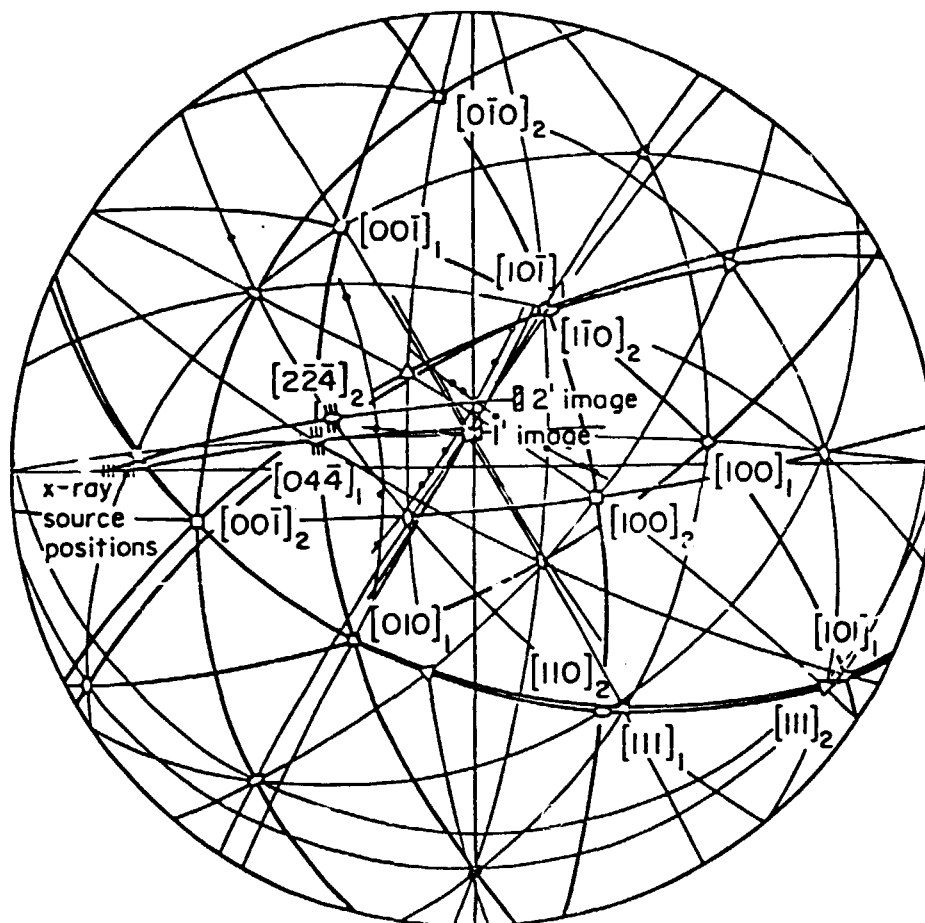
Scanning Photoresponse Pattern of an Area of a 2cm x 2cm  
13% AM1 Efficient Cell Showing the Location of the Grain  
Boundaries and an Area of High Dislocation Density

LARGE-AREA SILICON SHEET TASK

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Scanning Photoreponse Pattern of an Area of a  
2cm. x 2cm. Cell, No. 4726-C3-4, Including the Location  
of the Electrically Inactive Grain Boundaries



Second Order Twin Relationship of Grains 1 and 2  
Oriented to Give  $(044)_1$  and  $(224)_2$  Skew Reflections

## Conclusions

IN UCP SEMICRYSTALLINE SILICON, MOST GRAIN ORIENTATIONS APPEAR TO BE CRYSTALLOGRAPHICALLY RELATED BY A MULTIPLE ORDER TWINNING RELATIONSHIP, AND MOST GRAIN BOUNDARY INTERFACES ARE CRYSTALLOGRAPHICALLY DETERMINED. AS LONG AS THE CRYSTALLOGRAPHICALLY DETERMINED BOUNDARIES CONTAIN NO DISLOCATIONS, THE BOUNDARIES SHOW NO DELETERIOUS ELECTRICAL BEHAVIOR.

BECAUSE OF THE LARGE GRAIN SIZE OF THIS MATERIAL, AND THE LOW DENSITY OF NON-CRYSTALLOGRAPHICALLY RELATED BOUNDARIES, GRAIN BOUNDARIES HAVE A MINIMAL EFFECT ON THE PHOTOVOLTAIC PROPERTIES OF SEMICRYSTALLINE SILICON.

MINIMIZING INTERNAL GRAIN DEFECTS THEN BECOMES THE KEY TO HIGH WAFER QUALITY.

THE INTERNAL GRAIN ORDER IS AFFECTED BY MANY FACTORS INCLUDING:

- INCLUSIONS
- INDIVIDUAL DISLOCATIONS
- IMPURITIES
- DISLOCATION SUBGRAIN BOUNDARIES

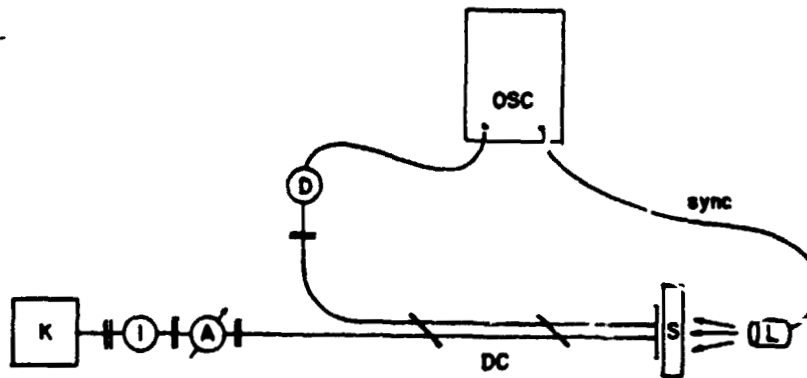
OCCASIONALLY, THE LARGE CRYSTAL WILL DEVELOP A SUBGRAIN DEFECT STRUCTURE. THIS STRUCTURE IS COMPRISED OF NUMEROUS SUBGRAINS THAT HAVE GRAIN DIAMETERS ON THE ORDER OF ONE MILLIMETER OR LESS AND APPEAR TO BE ROTATED BY  $5^{\circ}$  -  $7^{\circ}$  ABOUT SPECIFIC CRYSTALLOGRAPHIC DIRECTIONS SUCH AS A  $\langle 110 \rangle$  AXIS.

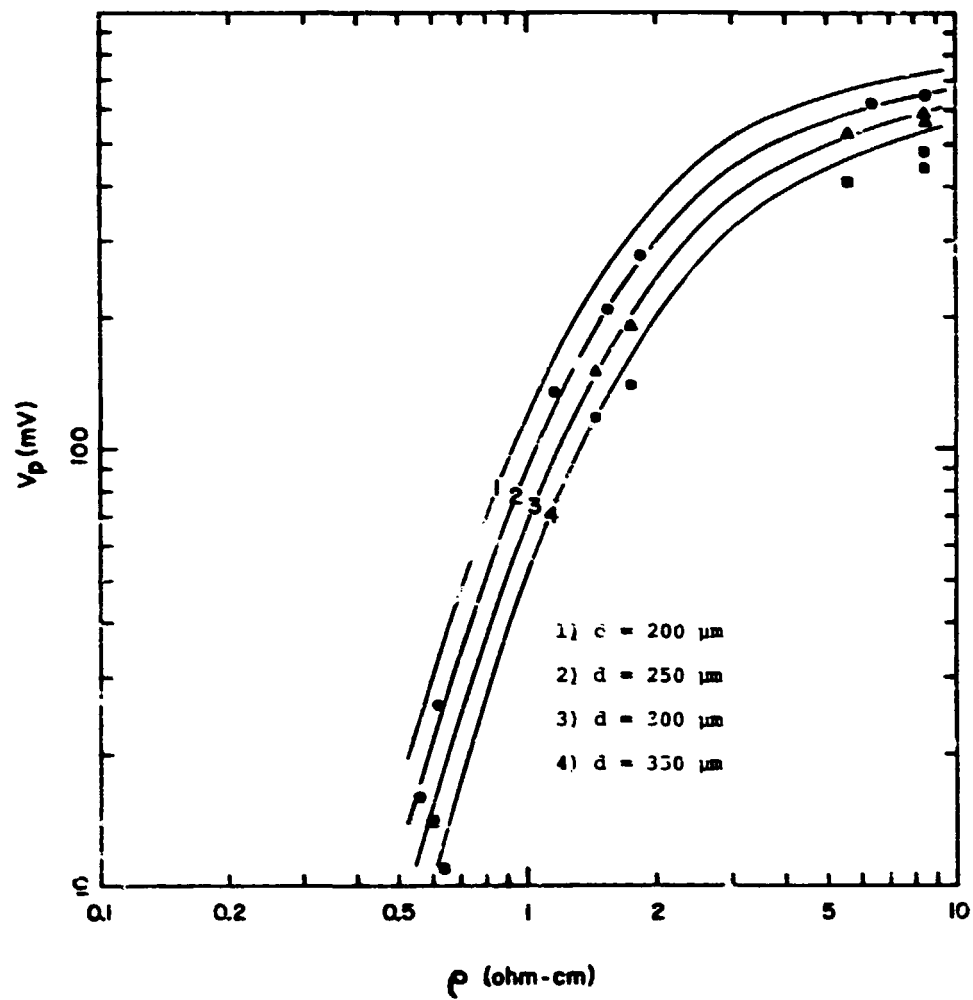
### Determination of Lifetimes in Semicrystalline C

PURPOSE:

DEVELOP A CONTACTLESS TECHNIQUE FOR THE MEASUREMENT  
OF CARRIER LIFETIMES IN SEMICRYSTALLINE MATERIAL.

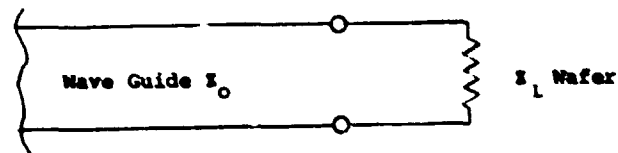
UNIQUENESS OF APPROACH:



Variation in Measured Response vs Base  
Resistivity and Sample Thickness



# Theoretical Model



## ASSUMPTIONS

High Conductivity Limit

$$\sigma \gg \omega \epsilon$$

Exponential Carrier Distribution

No Diffusion of Carriers

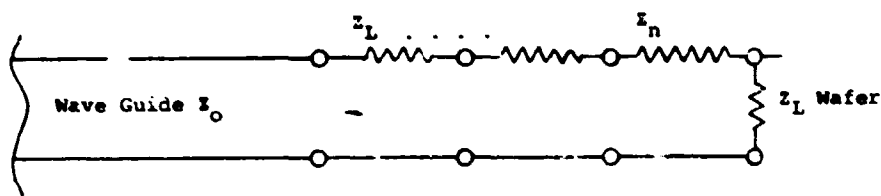
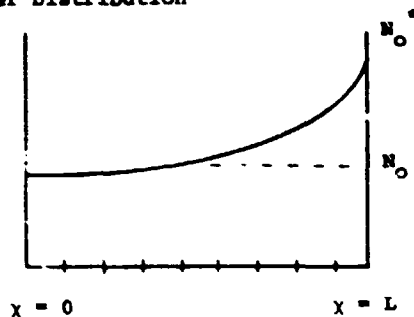
No Surface Recombination Velocity

Carriers Decay Exponentially with Time

$$N(t) = N_0 e^{-t/\tau}$$

## Transmission-Line Model

Carrier Distribution



Reflection Coefficient

$$R = \frac{Z_L - Z_0}{Z_L + Z_0} ; R \ll N_0 + N_0^*$$

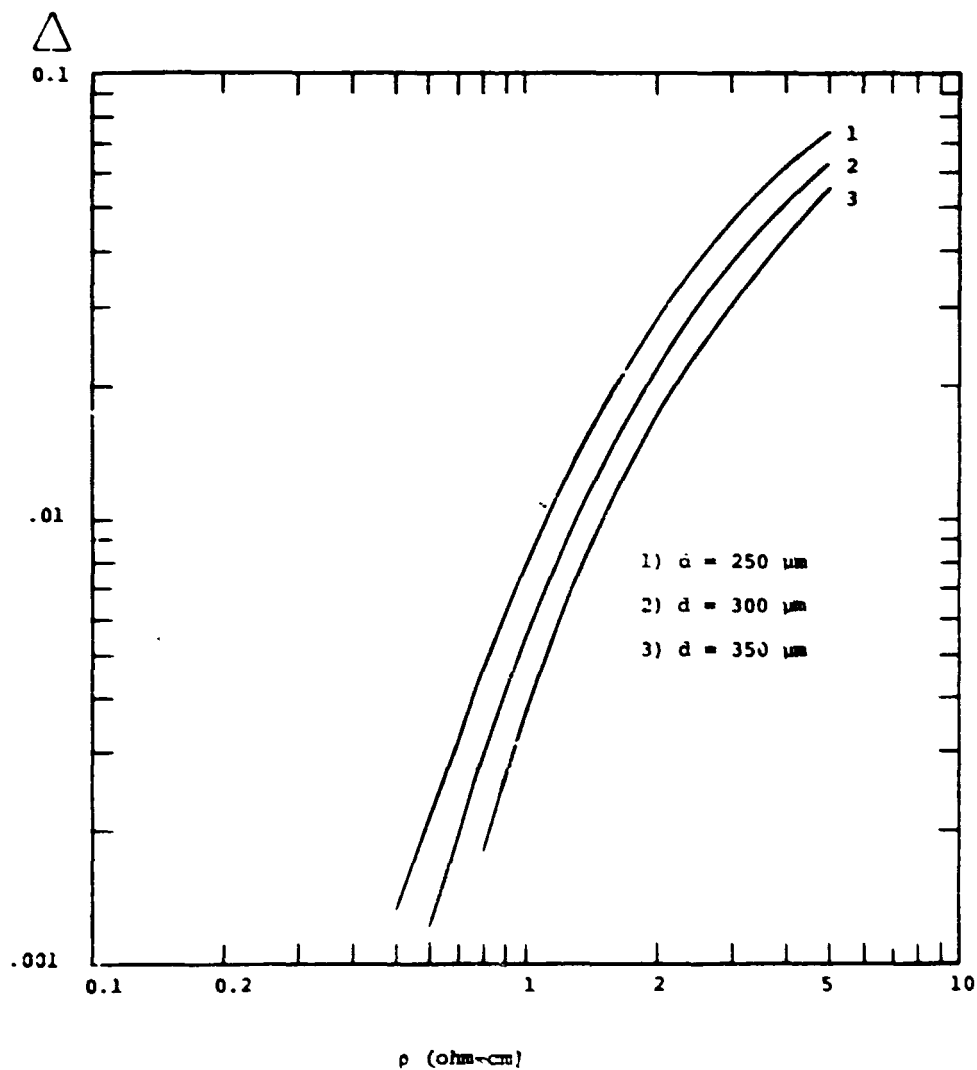
Reflected Power

$$P_M = RR^* \ll V_M$$

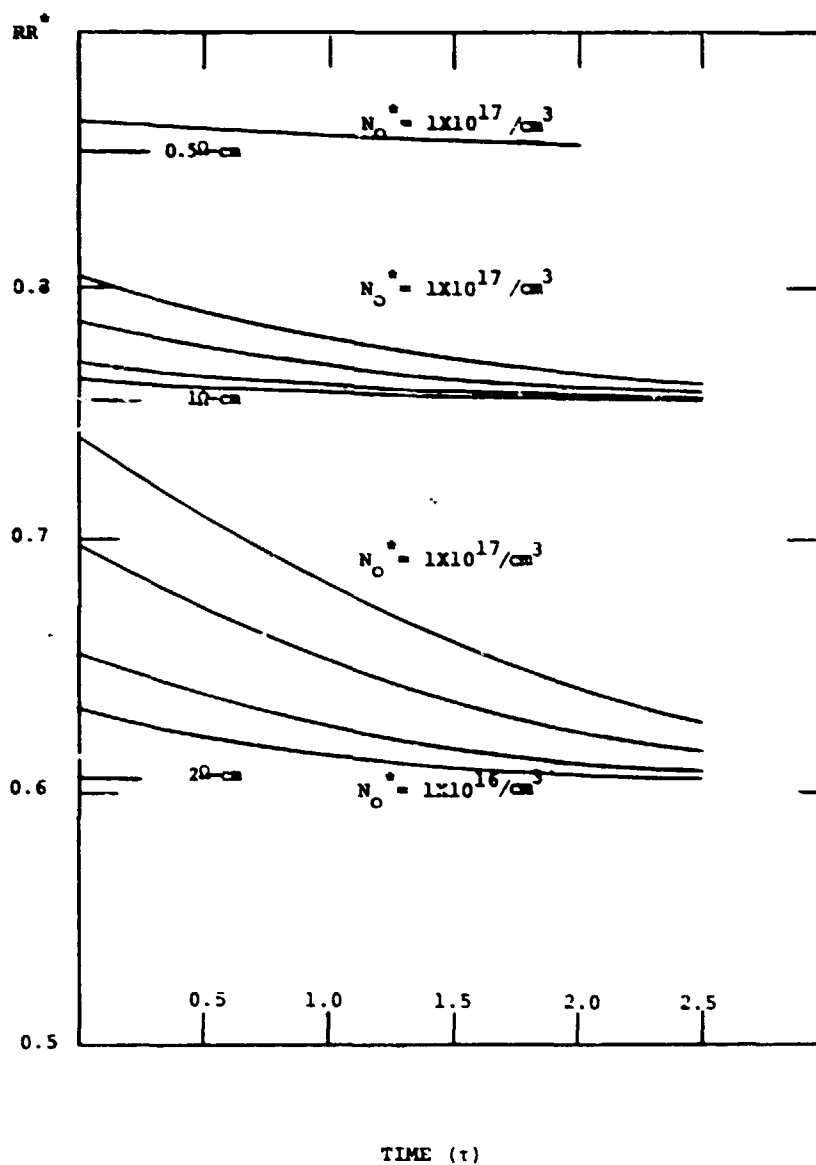
Normalized Modulation

$$\Delta = \frac{V_M^* - V_M}{V_H}$$

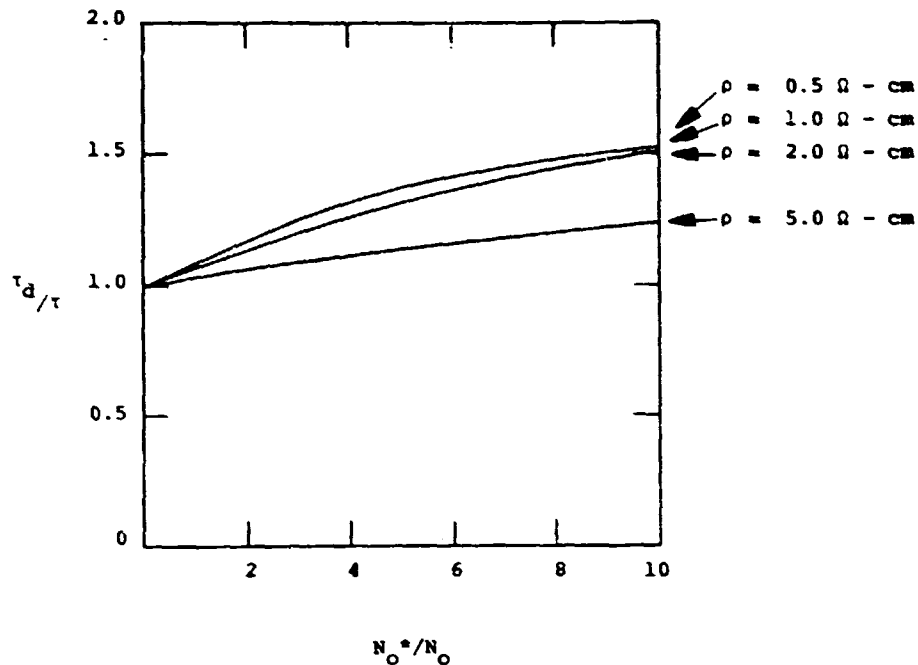
Normalized Modulation Calculated vs Base  
Resistivity and Sample Thickness



## Change in Reflected Power With Time



### Variation in Measured Decay Time vs Base Resistivity and Generated Carrier Concentration



### Conclusions

THIS IS A RELIABLE TECHNIQUE FOR THE CHARACTERIZATION OF SEMI-CRYSTALLINE MATERIAL. THE MODEL ACCURATELY PREDICTS THE OBSERVED BEHAVIOR ASSOCIATED WITH CHANGES IN BASE RESISTIVITY AND SUBSTANTIATES THE EMPIRICALLY OBSERVED BEHAVIOR FOR THE PREDICTION OF CARRIER LIFETIMES.

SURFACE PASSIVATION IS NECESSARY TO OBTAIN CONSISTENT AND MEANINGFUL RESULTS.

THE MEASURED DECAY TIME IS SOME MULTIPLE OF THE CARRIER LIFETIME, DEPENDENT UPON THE:

SAMPLE THICKNESS  
BASE RESISTIVITY  
ILLUMINATION INTENSITY

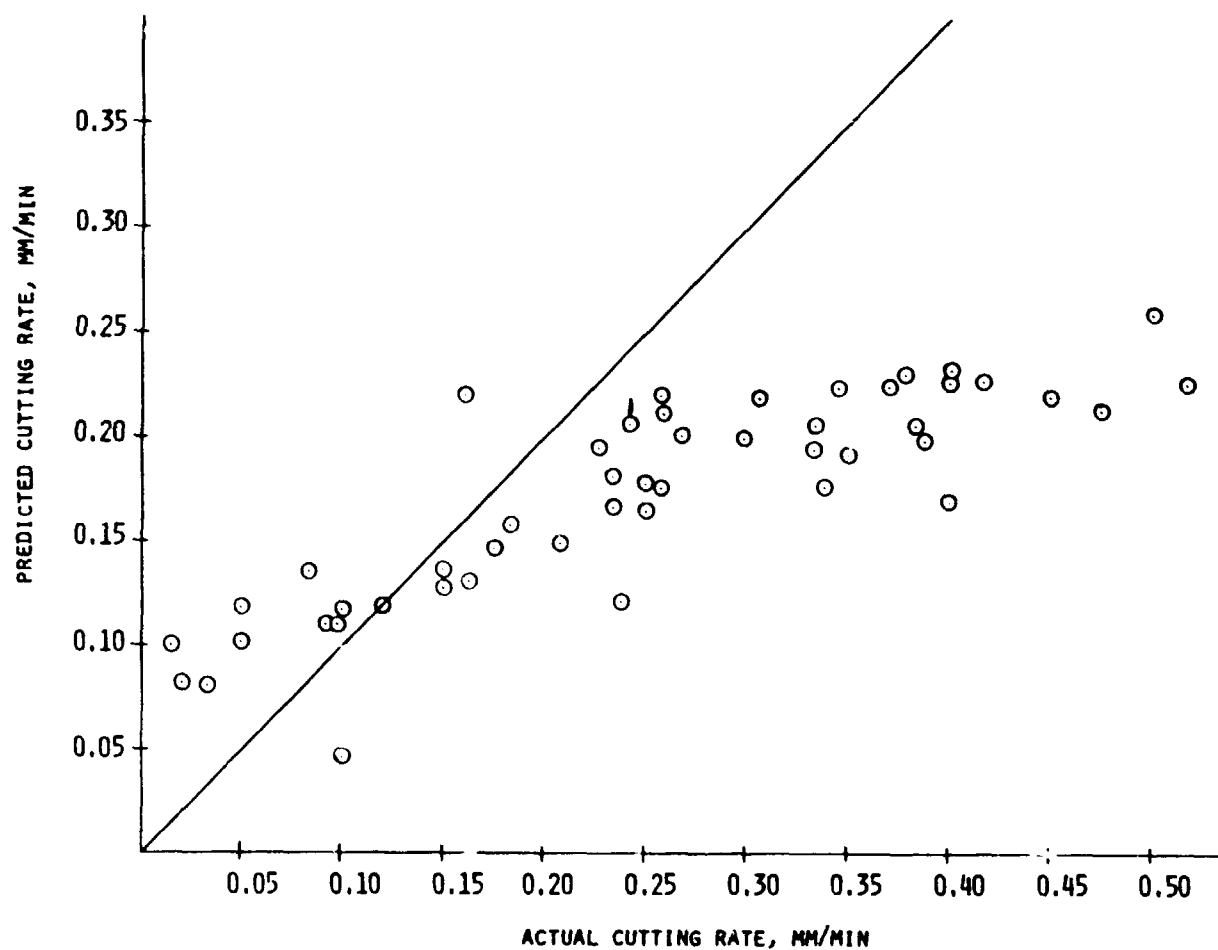
## LARGE-AREA SILICON SHEET TASK

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### Wafering Mechanisms

PURPOSE: TO DETERMINE IF THE HSMBS WAFERING TECHNOLOGY IS A  
VIABLE METHOD FOR THE ECONOMICAL PRODUCTION OF SHEET  
SILICON FOR PHOTOVOLTAICS.

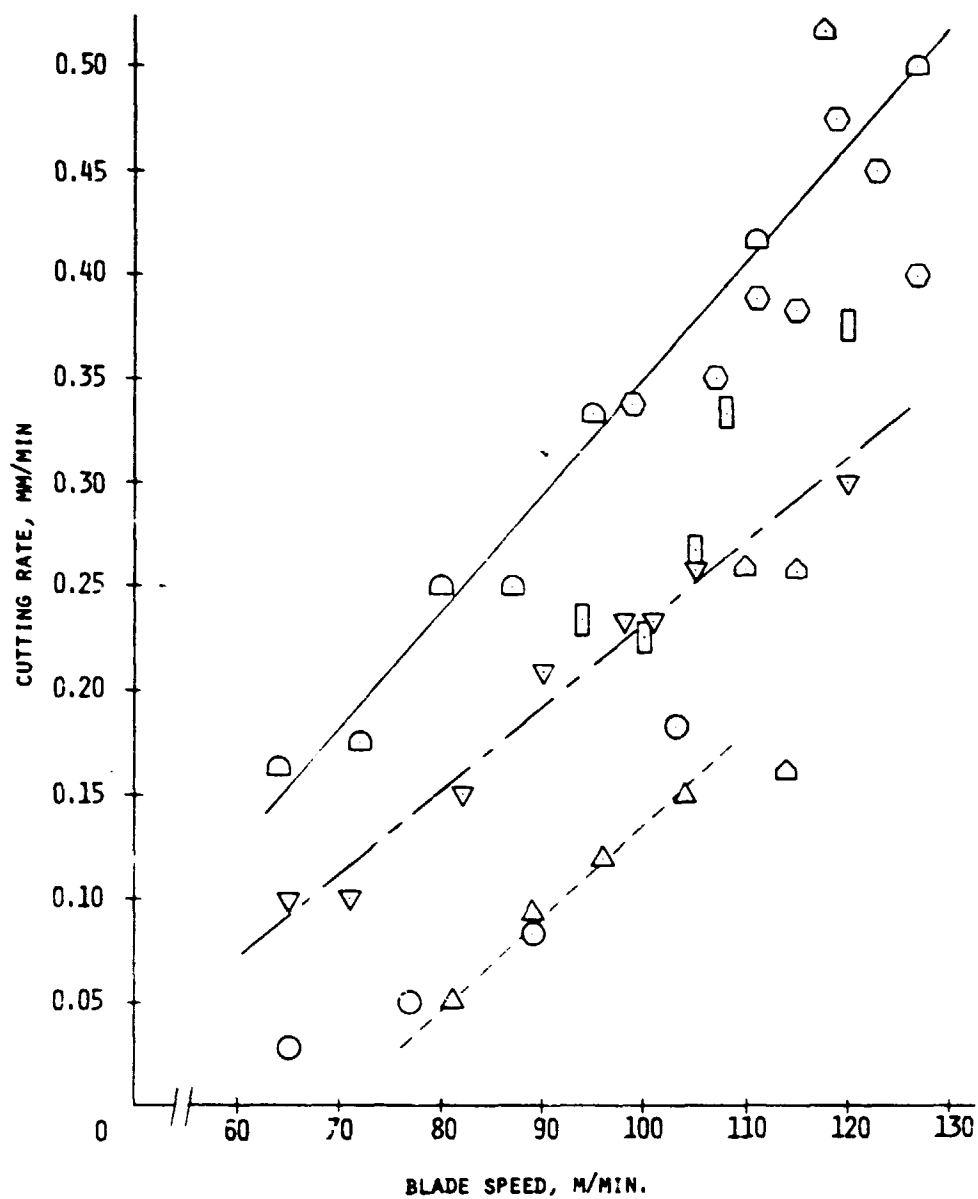
### High-Speed MBS Saw

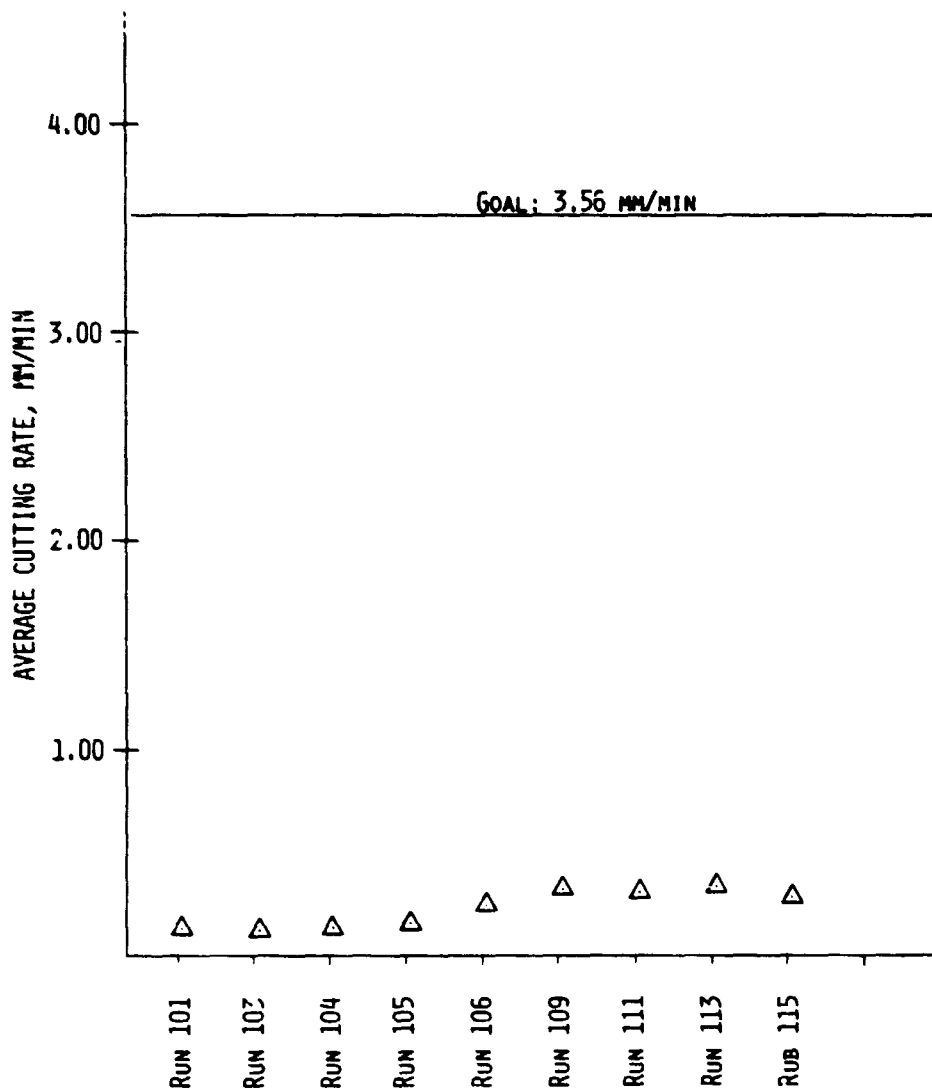


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High-Speed MBS Saw: Cutting Rate vs Blade Speed



High-Speed MBS Saw: Average Cutting Rates  
(Complete Runs Only)

## Conclusions

WITH THE HIGH MATERIALS COST ASSOCIATED WITH THE MBS TECHNOLOGY A CUTTING RATE OF  $1\text{ m}^2/\text{HR.}$  IS NECESSARY.

BASED UPON THE RESEARCH PERFORMED THUS FAR, IT WOULD BE NECESSARY TO SUSTAIN A CUTTING RATE OF  $3.5\text{ MM/MIN.}$

THE CURRENT TECHNOLOGY IS NOT CAPABLE OF MEETING THIS CRITERIA.



## ADVANCED CZOCHRALSKI INGOT GROWTH

KAYEX CORP.

ADVANCED CZOCHRALSKI  
INGOT GROWTH

KAYEX CORPORATION  
APRIL 22, 1982

**GOALS:**

GROWTH OF 150 KG OF INGOTS FROM  
ONE CRUCIBLE USING PERIODIC MELT  
REPLENISHMENT

DIAMETER 15 CM

THROUGHPUT - 2.5 KG/HR

RECHARGE MELTING RATE 25 KG/HR

AFTER-GROWTH YIELD 90%

MICROPROCESSOR CONTROLS PLUS  
IMPROVED SENSORS FOR:

MELT TEMPERATURE

DIAMETER

MELT LEVEL

PROTOTYPE EQUIPMENT FOR HIGH VOLUME  
PRODUCTION, TRANSFERABLE TO INDUSTRY

**STATUS:**

ONE 150 KG RUN PERFORMED DURING  
THIS CONTRACT

ACHIEVED

1.46 KG/HR, 150 KG RUN

14.3 KG/HR

ACHIEVED; 52% MONO, BALANCE POLY

CONSTRUCTED, INTERFACED & DEMONSTRATED

JPL MOD 2000 → CG6000

RL 4/22/82

LARGE-AREA SILICON SHEET TASK

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ADVANCED CZOCHRALSKI  
INGOT GROWTH

KAYEX CORPORATION  
APRIL 22, 1982

APPROACH:

CONSTRUCT AN IMPROVED CRYSTAL  
GROWER HAVING THE PERFORMANCE  
REQUIRED TO ACHIEVE GOALS

CONSTRUCT AN AUTOMATED SYSTEM  
WHICH WILL OFFER RELIABLE  
PERFORMANCE LEADING TO IMPROVED  
YIELDS AND REDUCED LABOR COST

CONDUCT PROCESS DEVELOPMENT ON  
LARGE SIZE CRYSTAL GROWTH, MELT  
REPLENISHMENT AND IMPROVED  
THROUGHPUT AND YIELDS

CONDUCT PARALLEL ANALYTICAL PROGRAM  
TO HELP UNDERSTAND THE PROCESS

STATUS:

INGOT SIZE ACHIEVED, BUT NOT  
THROUGHPUT

SYSTEM OPERATIONAL - INSUFFICIENT  
DATA TO CONFIRM YIELD & LABOR

INGOT SIZE 6" DIA x 37-1/2 KG  
ACHIEVED  
THROUGHPUT BELOW TARGET

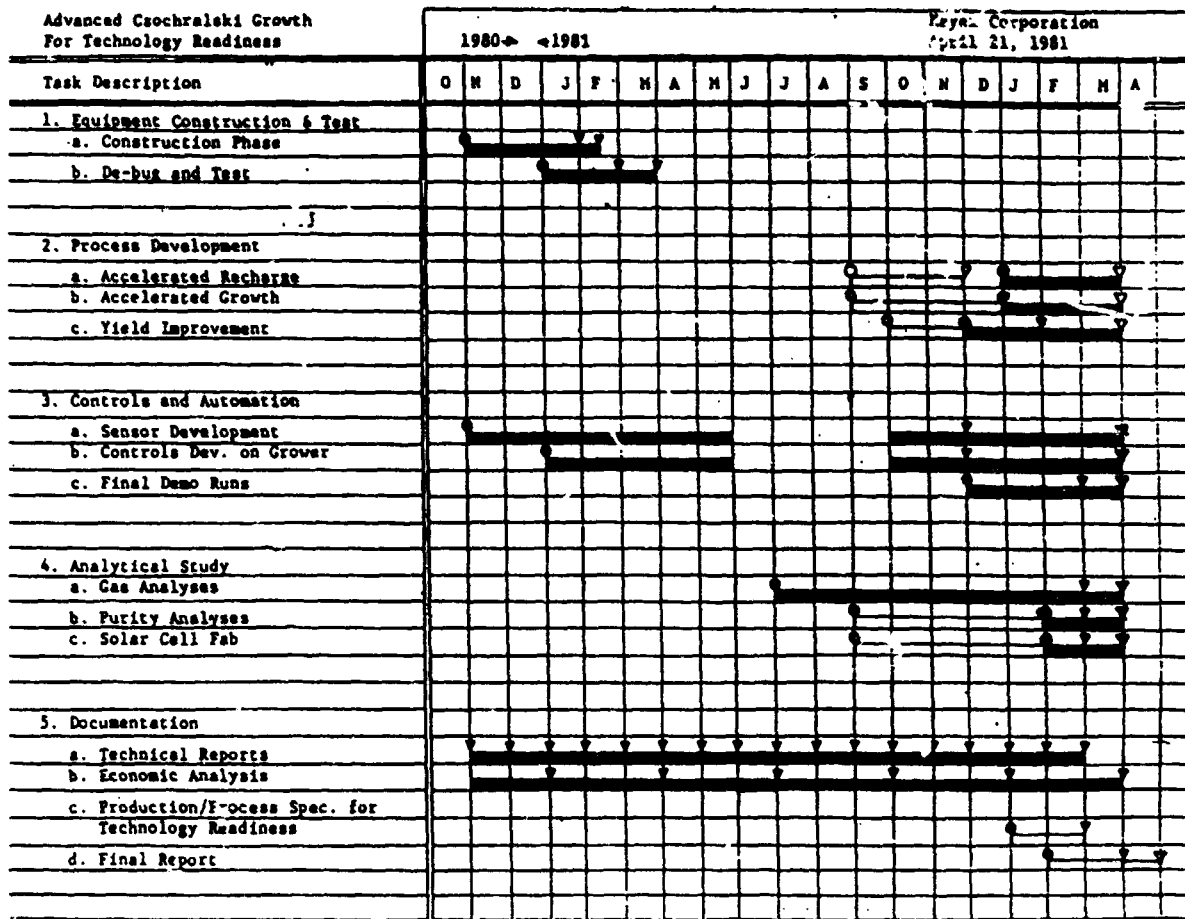
FURNACE GAS ANALYSES  
CRUCIBLE DEVITRIFICATION STUDY  
SOLAR CELL FAB AND TEST

RLL 4/22/82

# LARGE-AREA SILICON SHEET TASK

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## Program Plan, Revision 2



## Program Plan

THE PROGRAM PLAN CONSISTS OF, FIRST, A CONSTRUCTION PHASE, WHICH WAS COMPLETED IN THE FIRST FIVE MONTHS.

A PARALLEL SENSOR DEVELOPMENT PROGRAM WAS CARRIED OUT INITIALLY ON A SECOND COMMERCIAL MACHINE AND, SUBSEQUENTLY, THE SENSORS WERE INTERFACED TO THE JPL FACILITY.

FOR A PERIOD OF TIME IN LATE 1981, VERY LITTLE EFFORT WAS EXPENDED, EXCEPT FOR THE CONTINUED DEVELOPMENT OF THE GAS ANALYSIS SYSTEM.

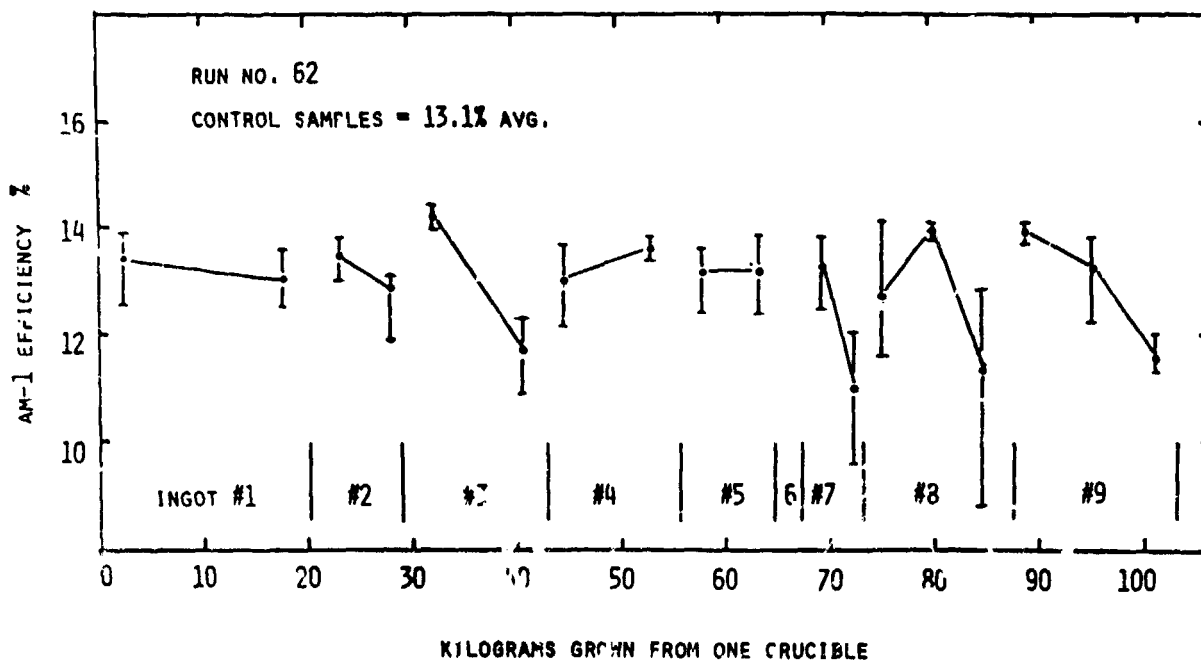
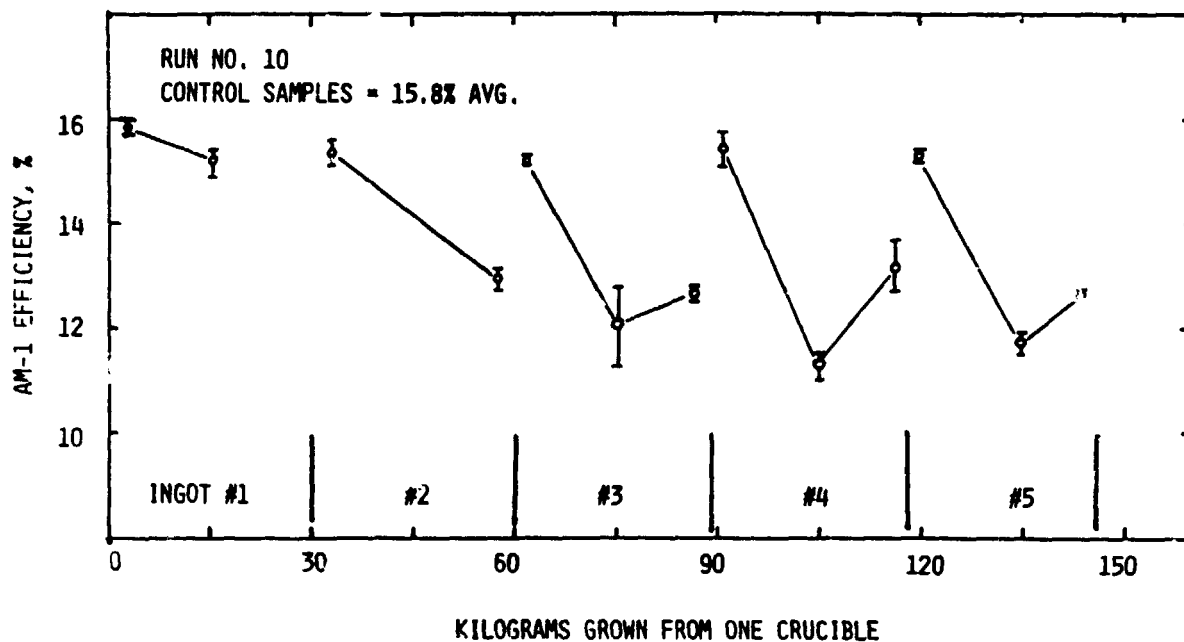
FOR THE LAST SEVERAL MONTHS, THE EFFORT WAS REDIRECTED THROUGH A TDM TO EMPHASIZE PROCESS UNDERSTANDING RATHER THAN EXTENSIVE DEMONSTRATION OF 150 KG RUNS.

THE EXPERIMENTAL WORK IS NOW COMPLETE AND THE FINAL DOCUMENTATION IS IN PROCESS.

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## Solar Efficiency vs Kilograms Grown



# LARGE-AREA SILICON SHEET TASK

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ADVANCED CZOCHRALSKI		KAYEX CORPORATION	
INGOT GROWTH		DOE/JPL 955733	
PROBLEMS - CONCERNS			
<u>PROBLEM AREA</u>		<u>APPROACH</u>	
● YIELD OF MONOCRYSTAL, LOWER EFFICIENCY OF POLY MATERIAL		● STUDY STRUCTURE LOSS MECHANISMS, PRIMARILY MELT CONTAMINATION	
		- CRUCIBLE DISSOLUTION	
		- CRUCIBLE DEVITRIFICATION	
		- GAS AMBIENT PURITY & FLOW	
● THROUGHPUT IS RELATED TO RATE LIMITING FACTORS		● IMPROVE HOT ZONE DESIGN	
- MELTING RATE		- FURTHER WORK WITH RADIATION SHIELDING	
- CORKSCREWING		- TEMPERATURE PROFILING OF MELT	
- STABILIZATION OF MELT TEMPERATURE		- IMPROVED TUNING OF MICROPROCESSOR TO SPEED UP STABILIZATION, SEEDING AND NECKING	

ADVANCED CZOCHRALSKI ENERGY DISPERSIVE X-RAY ANALYSIS OF ROSETTE DEFECTS ON INNER SURFACE OF USED CRUCIBLE				
1. COMPOSITION AT RIM OF DEFECT				
<u>ELEMENT</u>	<u>ATOMIC WEIGHT PERCENT</u>	<u>ATOMIC PERCENT</u>	<u>OXIDE FORMULA</u>	<u>OXIDE PERCENT</u>
SI	87.39	89.92	SiO <sub>2</sub>	91.53
S	1.97	1.78	SO <sub>3</sub>	2.52
CL	6.15	5.01	CL	3.11
K	2.91	2.15	K <sub>2</sub> O	1.75
CA	1.58	1.14	CAO	1.09
2. COMPOSITION OF CENTER OF DEFECT AND BULK SiO <sub>2</sub> GLASS CONTAINED NO DETECTABLE IMPURITIES.				
RLI 4/21/82				

## LARGE-AREA SILICON SHEET TASK

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### CO and H<sub>2</sub> vs Run Time

IN AN ATTEMPT TO UNDERSTAND THE MECHANISM OF STRUCTURE LOSS, WE HAVE CONSTRUCTED A DEVICE WHICH SAMPLES AND ANALYZES THE GROWER EXHAUST GAS FOR CARBON MONOXIDE, HYDROGEN, AND WATER. IF CARBON IS CONTAMINATING THE MELT, IT WILL BE CONCENTRATED IN THE RESIDUAL MELT AND COULD LEAD SUBSEQUENTLY TO SILICON CARBIDE PRECIPITATION, AS MORE AND MORE CRYSTALS ARE GROWN.

CARBON MONOXIDE HAS BEEN FOUND IN SURPRISINGLY HIGH CONCENTRATIONS, AND IS A FUNCTION OF TEMPERATURE. APPROXIMATELY 5000 PPM ARE SEEN DURING MELTDOWN.

# LARGE-AREA SILICON SHEET TASK

## MATERIAL CHARACTERIZATION

CORNELL UNIVERSITY

TECHNOLOGY	REPORT DATE
<p>LARGE AREA SILICON SHEET - ANALYSIS</p>	<p>April 17, 1982</p>
<p><b>APPROACH</b></p> <ul style="list-style-type: none"> <li>• Optical Microscopy and Etching.</li> <li>• Transmission Electron Microscopy</li> <li>• Electron beam Induced Current Microscopy</li> <li>• Chemical Analysis (<math>e^-</math>, ion, neutron, mass spec).</li> </ul> <p><b>CONTRACTOR</b></p> <p>Cornell University/Material Science</p> <p><b>GOALS</b></p> <ul style="list-style-type: none"> <li>• Characterize structure and chemical composition of point, line and planar defects in un-processed LASS material.</li> <li>• Characterize structure and chemical composition of point, line and planar defects in processed LASS material.</li> <li>• Evaluate crystal growth/defect relation</li> <li>• Evaluate processing/defect relation.</li> </ul>	<p><b>STATUS</b></p> <ul style="list-style-type: none"> <li>• EFG TEM and STEM analysis of defects in processed EFG completed. Chemical make up of large precipitates identified.</li> <li>• WEB RT EBIC of processed WEB completed. Temperature dependent EBIC being carried out to determine local energy levels. Rutherford backscattering completed.</li> <li>• HEM Optical Microscopy and etching completed. EBIC completed.</li> <li>• <u>NEW ANALYTICAL TOOLS ADDED SINCE LAST PIR</u> <ol style="list-style-type: none"> <li>1) JEOL 200 CX STEM with EDX</li> <li>2) Temperature dependent EBIC</li> <li>3) General Ionix Accelerator for back-scattering analysis.</li> </ol> </li> </ul>

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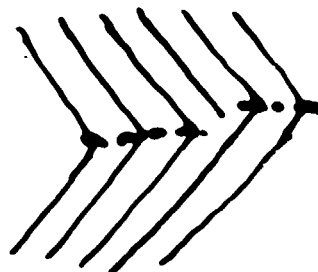
## Coherent Twins

- $60^\circ$  rotation on (111) plane
  - perfect first nearest neighbor fit
- Periodicity of three
  - SIGMA 3 boundary
- Small deviations from ideal twin are accomodated by DSC dislocations
 

$b = (a/6) 112$

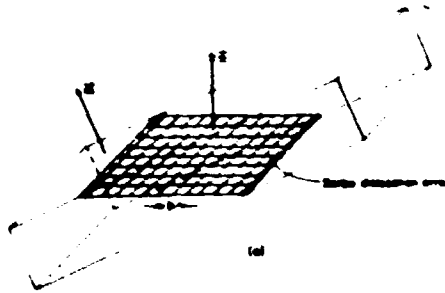
These dislocations are the analogue of complete dislocations in small angle grain boundaries.
- Dislocations are necessarily associated with a step in the boundary
  - presents possibility to study effects of jogs.

C C C C C  
 B B B A A  
 A A A B B  
 C C C C C  
 B B B B B  
 A A A A A

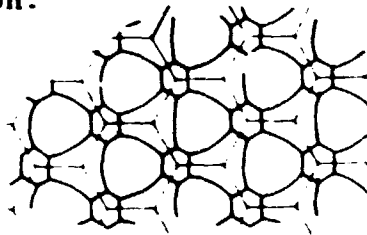


### Twist Boundaries

Network of screw dislocations



On  $(111)$  dislocations react to form  
hexagonal networks - low angle - twin  
relation.



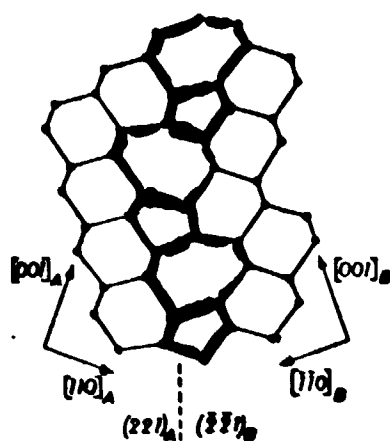
Houstra, J.  
1959Physica 25  
409-472

Fig. 6. Symmetrical tilt boundary with  $\theta_1 = 38^\circ 57'$  and zig-zag arrangement of dislocations.

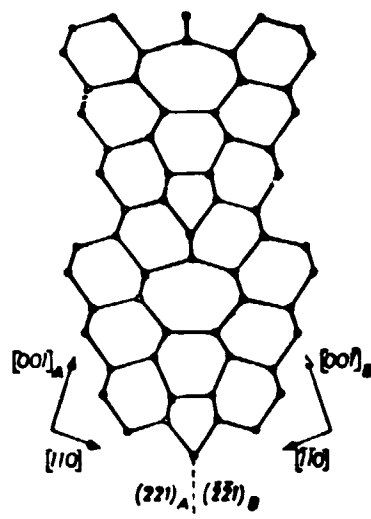
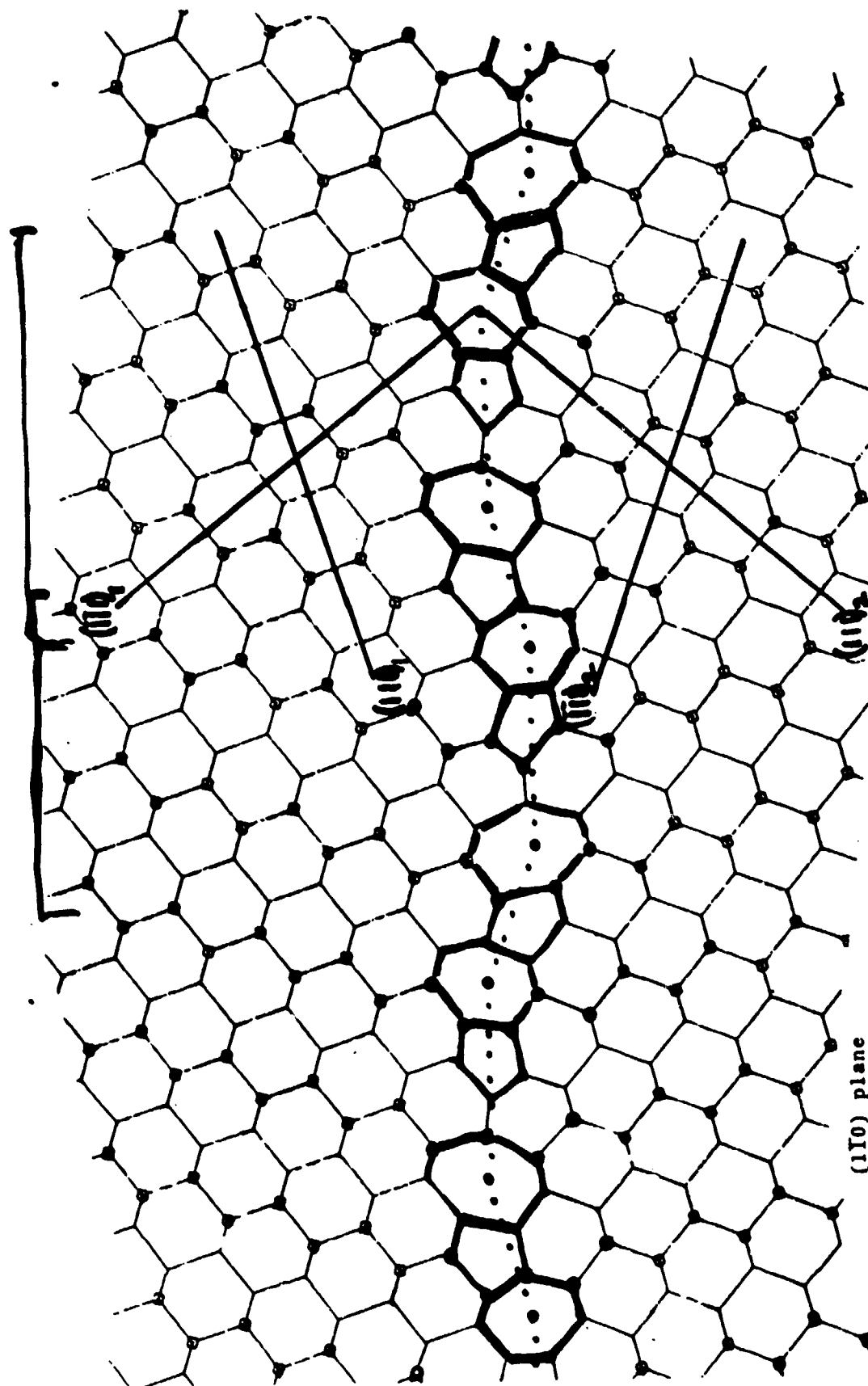


Fig. 7. As Fig. 6 but with overlapping dislocations, thus forming double dislocations.

For ~~the model~~ ( $\theta_1 = 31^\circ 35' = \arccos 23/27$ ) the model ~~is~~  
~~the same as in Fig. 6 but with overlapping dislocations, thus forming double dislocations.~~ Thus,

# LARGE-AREA SILICON SHEET TASK

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Proposed model for a  $\Sigma=5$ ,  $\theta=31.58^\circ$  /  $[1\bar{1}0]_{1/2}$

GBP =  $(SS2)_1$ ;  $(SS2)_2$

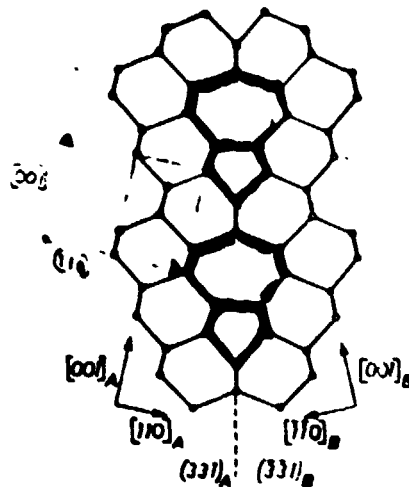
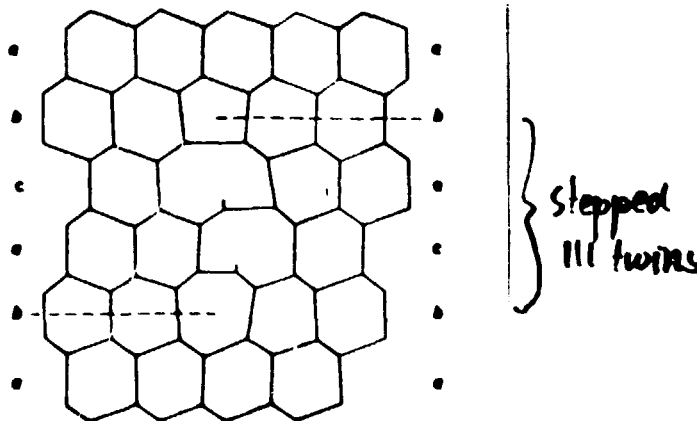
Hornstra, J.  
1959Physica 25  
409-422

Fig. 5. Projection on  $(1\bar{1}0)$  of a symmetrical tilt boundary with tilt axis  $[110]$ , angle of tilt  $\theta_1 = 26^\circ 32'$  and median plane  $(110)$ . The height of the atoms above the plane of projection is expressed in  $z = \frac{1}{2}a\sqrt{2}$ , where  $a$  is the lattice constant.

*J. Phys. Chem. Solids* Pergamon Press 1958, Vol. 5, pp. 129-141.



## 8. DISLOCATIONS AND TWIN BOUNDARIES

As has been shown in Fig. 13(c), a shift of the twin boundary involves a partial dislocation. A shift of one (double) atomic plane involves a partial of type I, a shift of two planes one of type II, a shift of three planes, however, involves a lattice defect of a different kind. The possible structure of it is shown in Fig. 19(a) for the diamond lattice and in Fig. 19(b) for the f.c.c. lattice. It is no dislocation as its Burgers vector is zero.

## Summary

### SIGMA = 3

- BOUNDARY PER SE NOT ELECTRICALLY ACTIVE
- ELECTRICAL ACTIVITY CORRELATES WITH PRESENCE OF PARTIAL DISLOCATIONS.
- PARTIAL DISLOCATIONS SHOW ENHANCED ACTIVITY COMPATIBLE WITH JOG MODEL
- EVIDENCE FOR KINK ACTIVITY FROM CURVED PARTIALS.

### SIGMA = 9

- 111/115 TWIN SHARES HABIT PLANE WITH FIRST ORDER TWIN - FREQUENTLY MIS-IDENTIFIED AS THE LATTER.
- ELECTRICAL ACTIVITY COMPATIBLE WITH BROKEN BOND MODEL.
- ALTERNATING SECTIONS GIVE DOT-LIKE EBIC CONTRAST SIMILAR TO PARTIAL DISLOCATIONS IN FIRST ORDER TWINS.

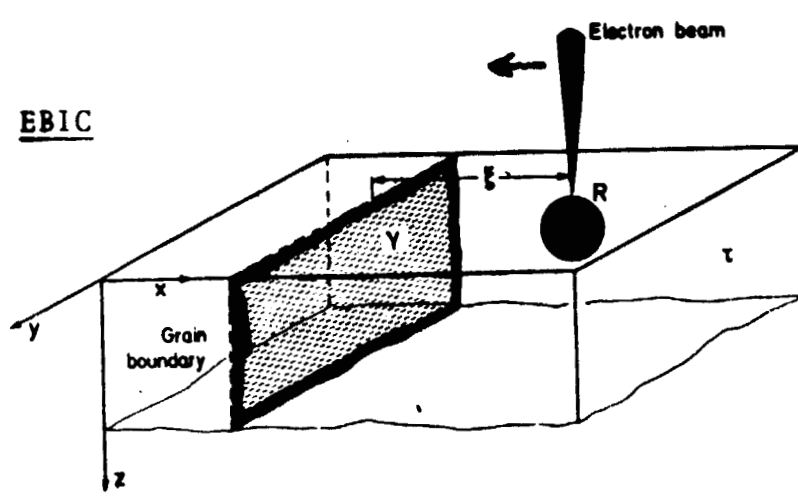
### SIGMA = 27

- CHARACTER OF BOUNDARY DEPENDS ON BOUNDARY PLANE VARIES OVER SHORT DISTANCES ( 0.1  $\mu\text{m}$  ).
- UNSYMMETRIC GBP CORRELATED TO DISSOCIATED BOUNDARIES:  
SIGMA27 = SIGMA9 • SIGMA 3
- MICROFACETTING IN ACCORDANCE WITH HORNSTRA.
- UNDISSOCIATED BOUNDARY ON SYMMETRIC GBP HAS STRUCTURE OF 5 and 7 MEMBERED RINGS SHOULD LEAD TO GAP STATES.

Summary of Sigma = 27

- o Undissociated 255/255 is symmetric.  
and located on second highest density  
CSL (highest 115/115)
- o Dissociated section is un-symmetric.  
Components : Symmetric SIGMA 9 112/112  
(2 nd highest after 111/115)
- o Largest facet is SIGMA 3 111/111 coherent  
twin
- o Other facet: probably 111/115
- o 3 Step periodicity correlated to stacking  
sequence - Hornstra model of atomistic faceting.  
(Brockman).

## Techniques

EBIC

EBIC contrast of grain boundaries can be calculated by solving the 3-D diffusion equation under the following assumptions (J.Marek)

- o  $\infty$  recombination velocity at the boundary plane
- o  $R_p \gg$  depletion layer thickness.

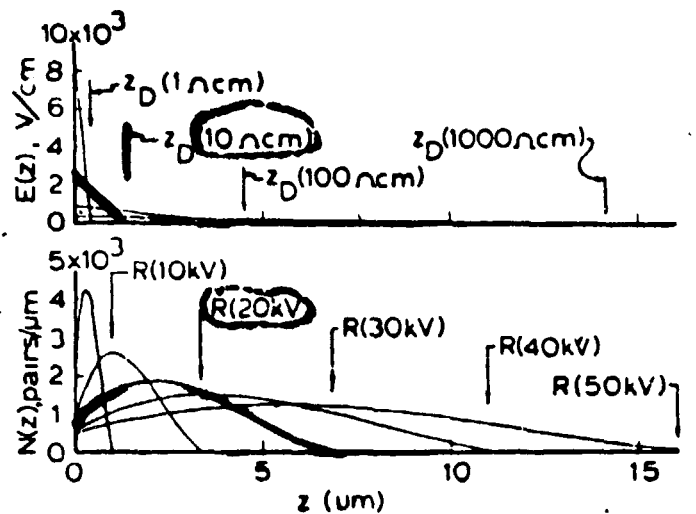
Typical operation conditions for 10 cm Si

$$d \approx 1\mu\text{m}$$

$$R_p \approx 6\mu\text{m} \text{ (30 Kev)}$$

Maximum contrast when  $R_p \approx L$ .





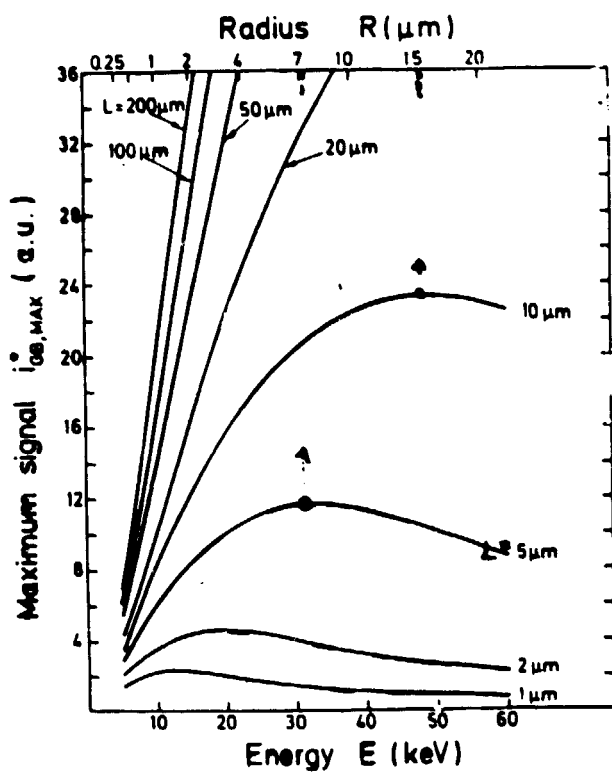
After Leamy et al.

$$R = \alpha E^{1.75}$$

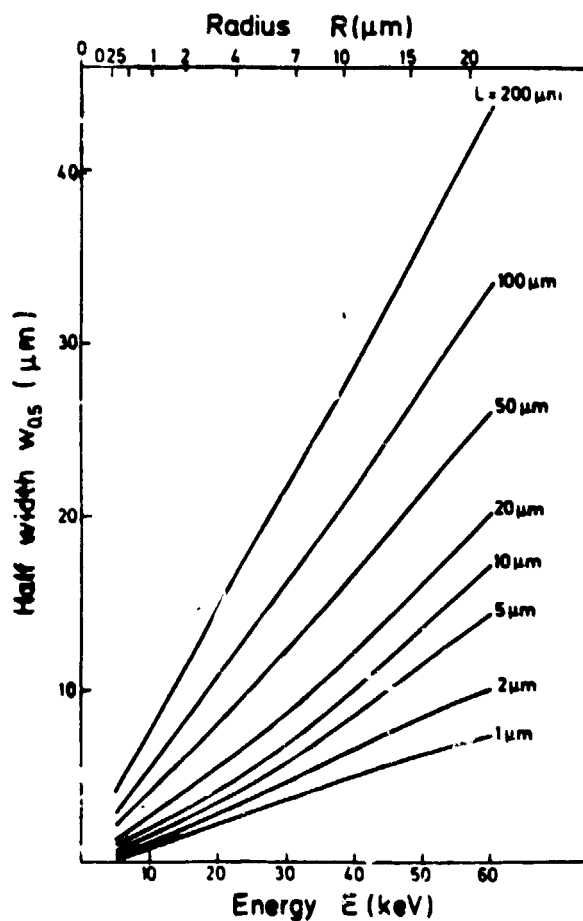
Recent results indicate  $\alpha$  may be twice as high as indicated above.

# LARGE-AREA SILICON SHEET TASK

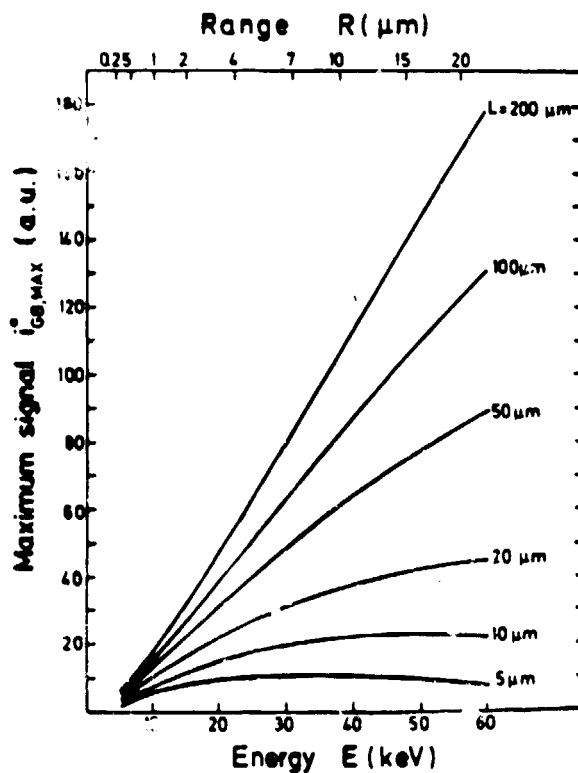
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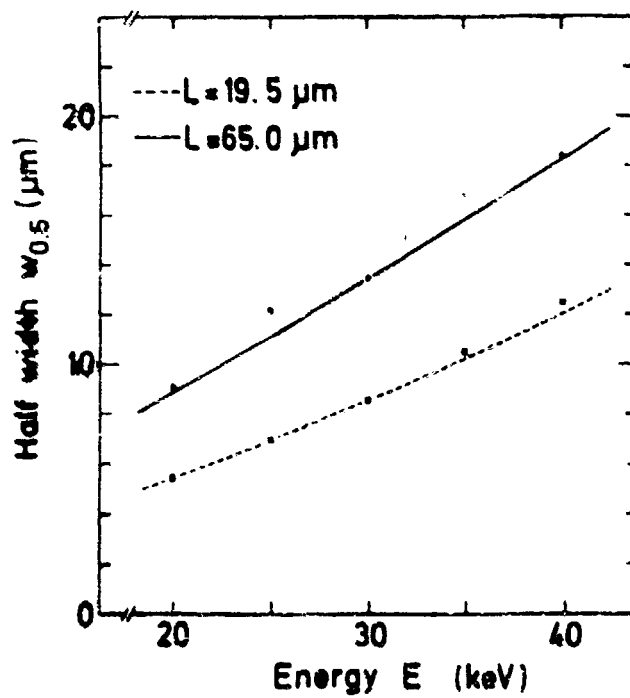
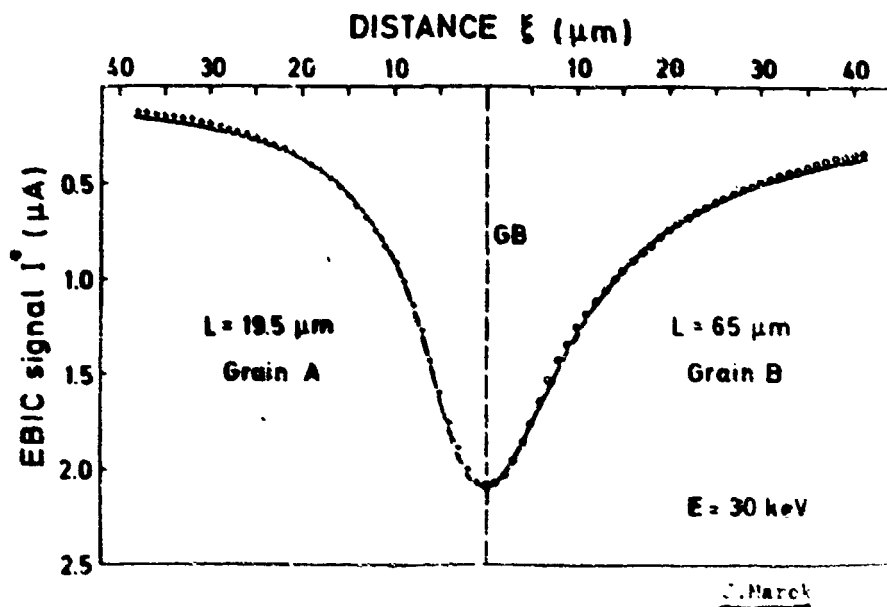
J. Marek



J. Marek



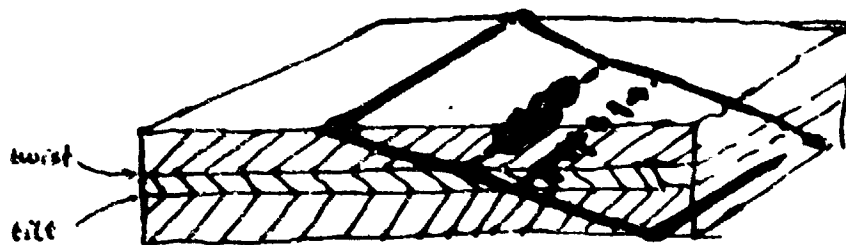
J. Marek



J. Marek

Web

- 0 Contains one or several twin planes in center plane of ribbon



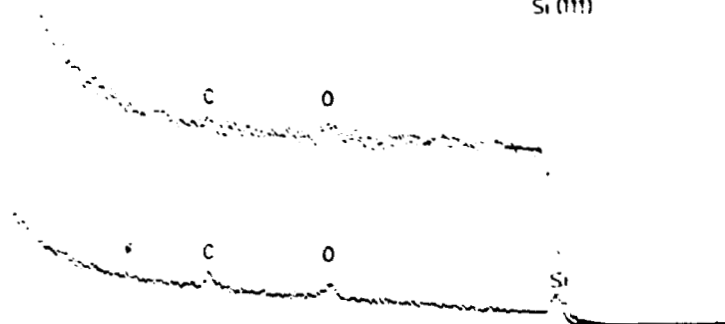
- 0 Shallow bevel allows EBIC imaging of dislocation network on these twins
- 0 Rotational misfit of seconds of arc results in sufficiently large spacing to be resolved by EBIC.

# LARGE-AREA SILICON SHEET TASK

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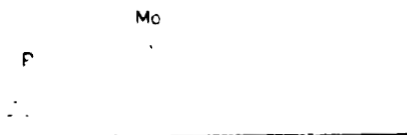
20784 5062  
50 0 LT= 656 CT= 662 4096FS 562 0

Rutherford Backscattering  
2 MeV 15  $\mu$ C  
Processed Web Cell  
Si (111)



19412 5062  
50 0 LT= 5956 CT= 5960 LOG 562 0

Rutherford Backscattering  
1 MeV 50  $\mu$ C  
Processed Web Cell  
Si (111)



## Problems and Concerns

- Statistics of Results .

Time and manpower restrictions confine analysis to a few specimen of a given material. Thus, some caution must be exercised when applying results to the population as a whole, especially for materials in which the crystal growth and processing techniques are continuously refined.

## GRAIN BOUNDARY INVESTIGATION

### JET PROPULSION LABORATORY

L.J. Cheng

#### Participants

LI-JEN CHENG

GERRY CROTTY

TAHER DAUD

KATHY DUMAS

SANDY HYLAND

TOM MacCONNELL

RINDGE SHIMA

CHIN-MIIN SHYU

KATE STIKA

#### Objective

TO DEVELOP BETTER POLYCRYSTALLINE SILICON SOLAR CELLS  
THROUGH BETTER UNDERSTANDING OF THE BEHAVIOR OF GRAIN  
BOUNDARIES IN SILICON

#### Approach

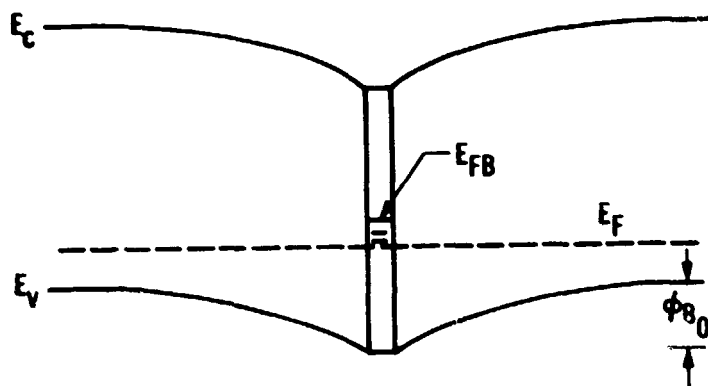
EXPERIMENTAL STUDIES ON SAMPLES OF

- LARGE GRAIN SILICON INGOTS  
(WACKER, SEMIX, AND HEM)
- CZ BICRYSTALS WITH CONTROLLED  
LATTICE MISMATCH

## Subjects Under Study

- BICRYSTAL GROWTH
- ELECTRONIC TRANSPORT PHENOMENA
  - POTENTIAL BARRIER
  - CARRIER RECOMBINATION
  - TRAPPING STATES
- ATOMIC TRANSPORT PHENOMENA
  - ENHANCED DIFFUSION OF IMPURITIES
  - IMPURITY GETTERING
- EFFECTS ON SOLAR CELL PERFORMANCE

## Energy-Band Diagram at Boundary Region for p-Type Si



$$\phi_{B0} = E_A + T \frac{\partial \phi_{B0}}{\partial T}$$

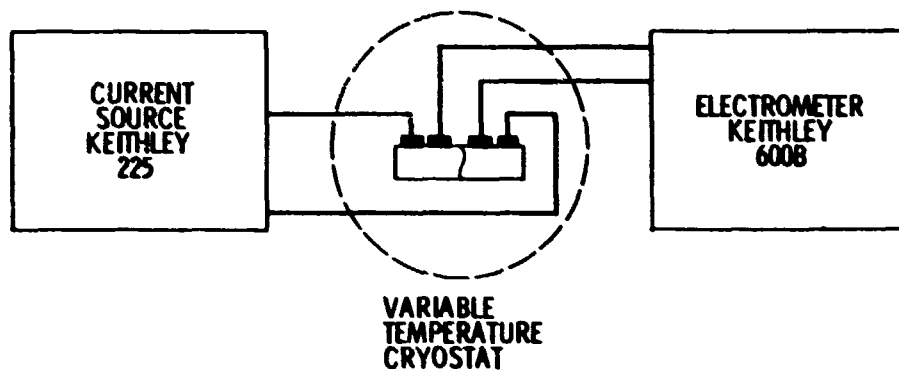
$$\phi_{B0} = \frac{qQ^2}{8\epsilon_0 \epsilon N_A}$$

$$Q = \frac{\epsilon_0 \epsilon N_A}{C_0}$$

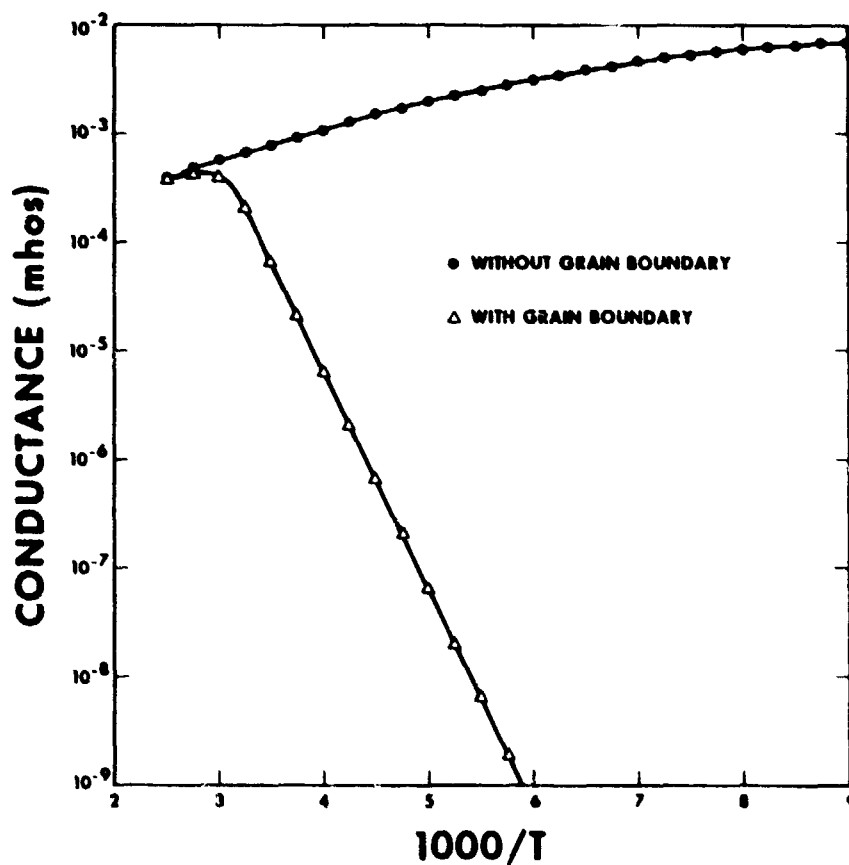
$$Q = \int_{E_{FB}}^{E_F} dE, \sigma dE$$

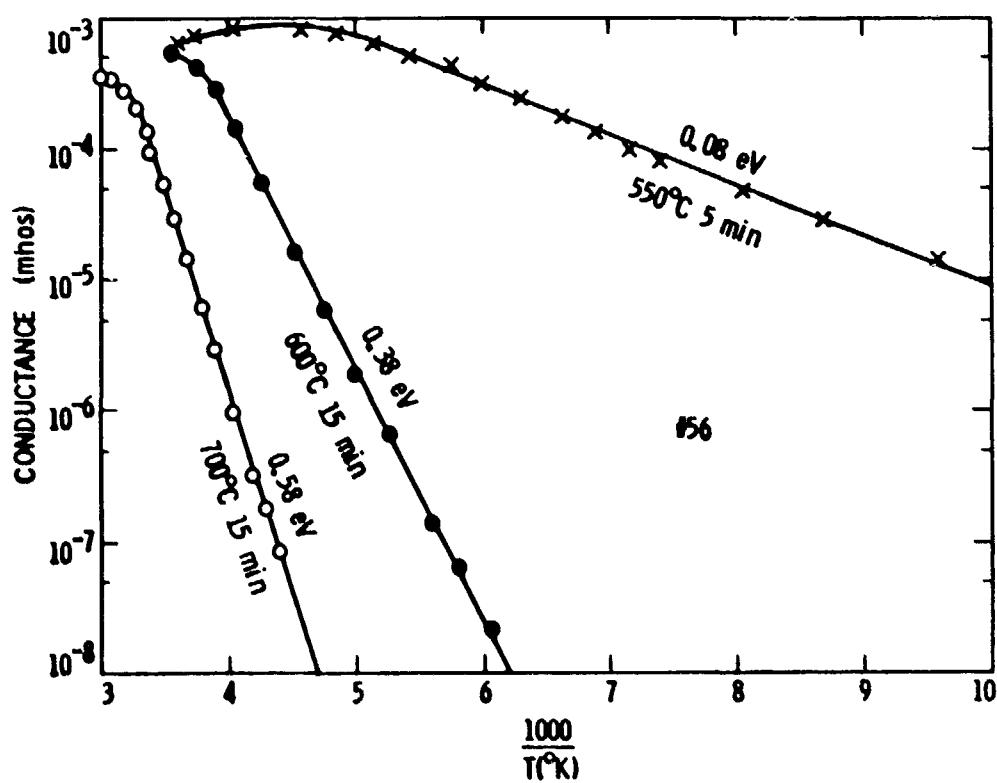
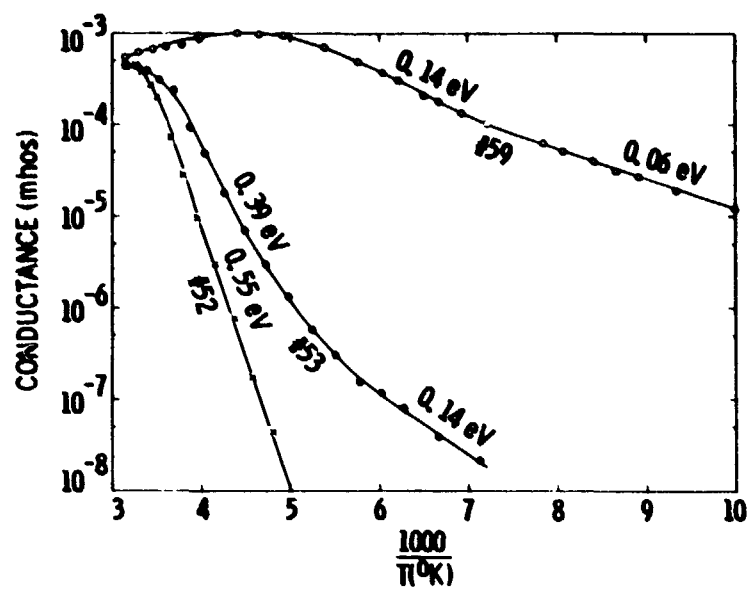


### Experimental Arrangement for Zero-Bias Conductance Measurements



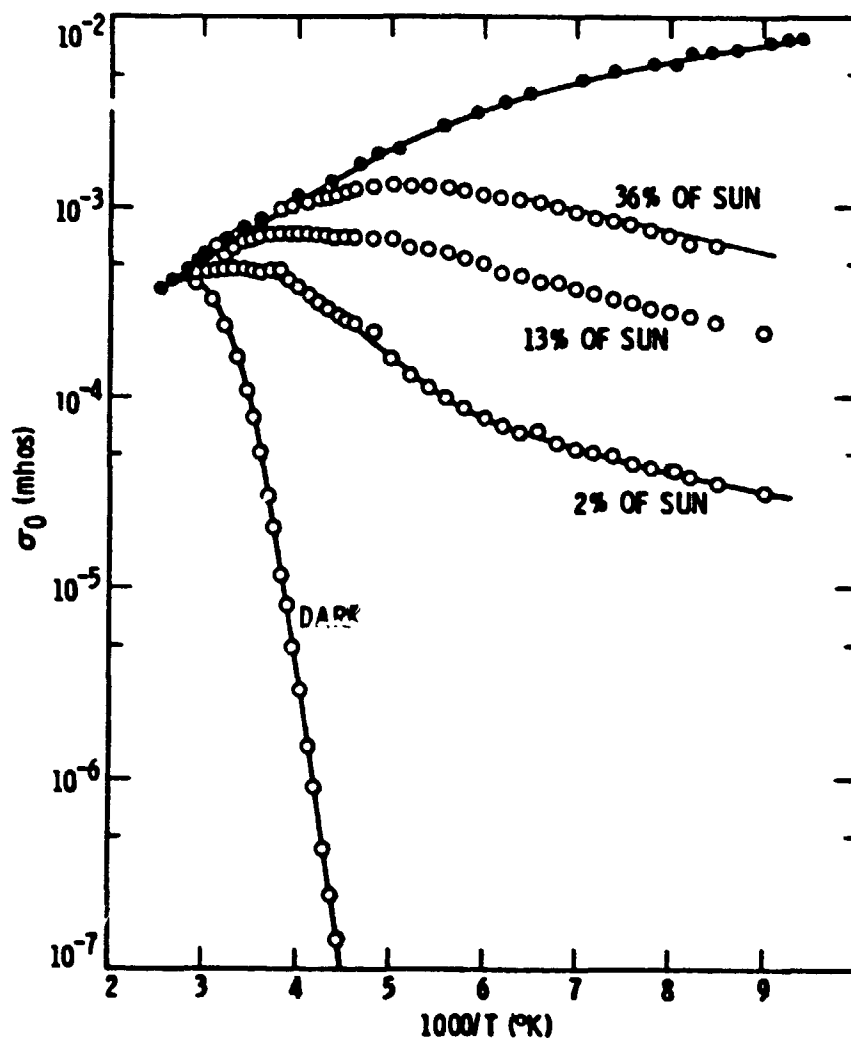
### Temperature Dependence





# LARGE-AREA SILICON SHEET TASK

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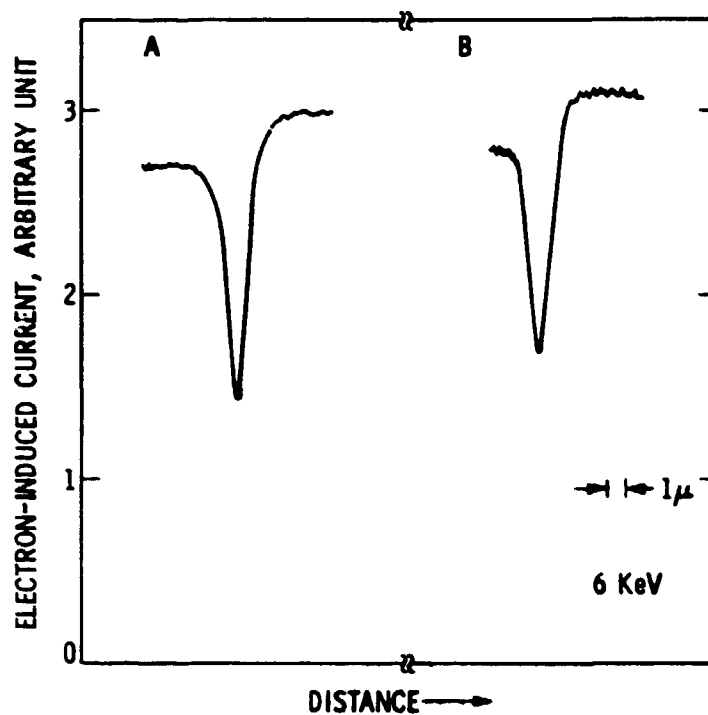
## Potential Barrier

### RESULTS:

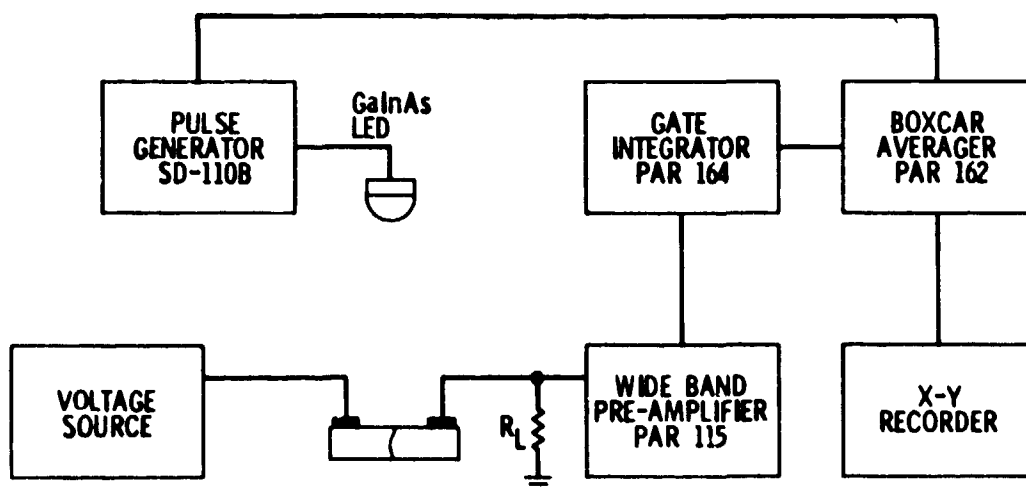
- CONSIDERABLE VARIATION IN  $E_A$  ALONG GRAIN BOUNDARIES, PRESUMABLY DUE TO VARIATION OF LOCAL DISORDERS
- INCREASE OF  $E_A$  WITH ANNEALING TEMPERATURE, LIKELY DUE TO LOCAL DEFECT CHANGES AND IMPURITY GETTERING
- DECREASE OF  $\phi_B$  WITH LIGHT INTENSITY, CAUSED BY MINORITY CARRIER TRAPPING

# LARGE-AREA SILICON SHEET TASK

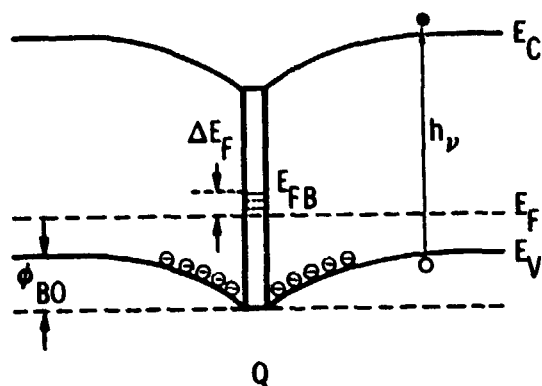
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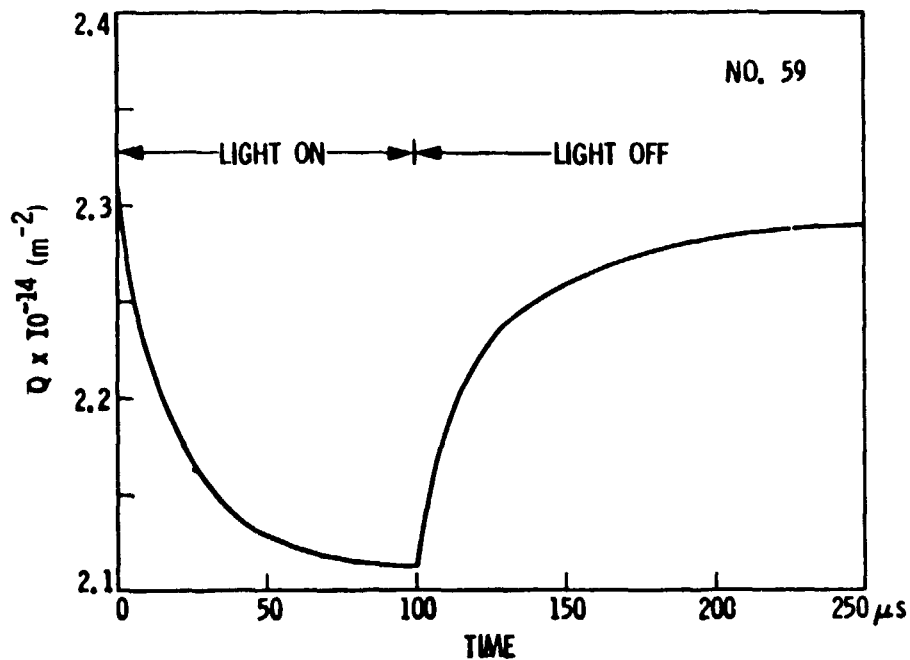
## Experimental Arrangement for Photoconductivity Measurements



## Light Effects on Grain Boundary Properties



- DECREASE OF  $Q$
- INCREASE OF  $G_0$
- CREATION OF CARRIER RECOMBINATION CURRENT



# LARGE-AREA SILICON SHEET TASK

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UNDER ILLUMINATION,

$$\frac{dQ}{dt} = J_{maj} - J_{min}$$

$$(J_{maj} = J_{min} \text{ AT THE EQUILIBRIUM})$$

AND, IN THE DARK AFTER THE LIGHT IS OFF,

$$\frac{dQ}{dt} = J_{maj},$$

WHERE

$$J_{maj} = (2cA - B) \exp(-(E_F + \phi_{BO})/kT)$$

$$J_{min} = \frac{D_e}{e L_e} (n_{\infty} - n_0)$$

---

RECOMBINATION VELOCITY AT THE GRAIN BOUNDARY

$$S = \frac{J_{min}}{e n_0}$$

## Electrical Properties of Grain Boundaries

Sample No.	$E_g$ (eV)	$G_0$ (mho/m <sup>2</sup> )	$\phi_{BO}$ (eV)	$Q$ (/m <sup>2</sup> )	$I^*$	$S$ (m/s)
51	0.55	$5.98 \times 10^2$	0.12	$5.66 \times 10^{14}$	0.66I	2.5
					I	6.4
59	0.10	$2.45 \times 10^3$	0.02	$2.22 \times 10^{14}$	I	0.88
					1.8I	1.09

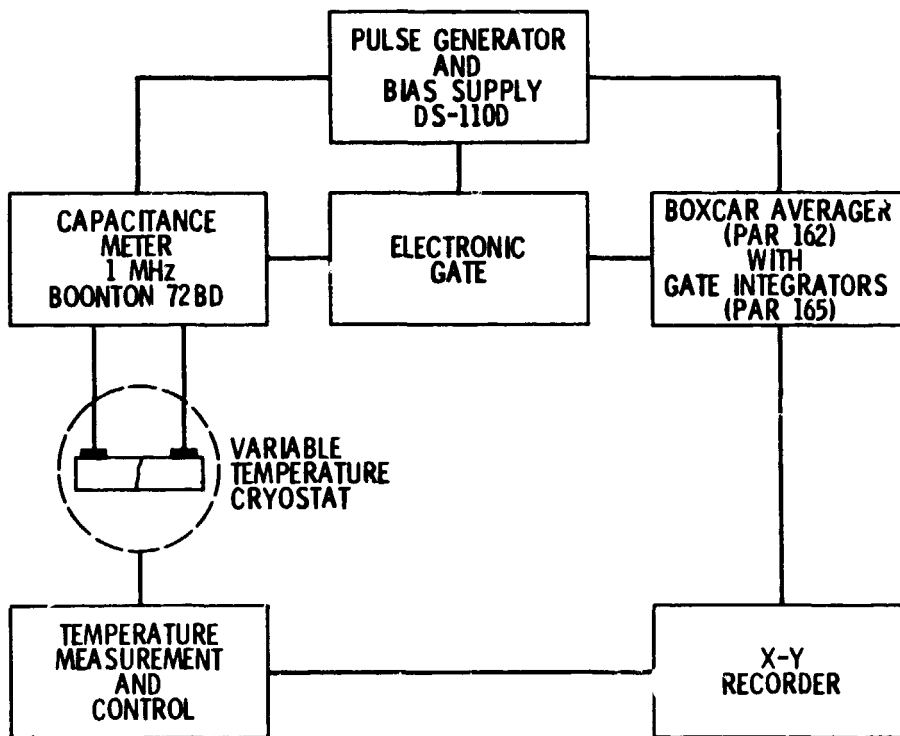
\*I = Light intensity which creates an equilibrium minority carrier density of  $1.08 \times 10^{16}$  electrons/m<sup>3</sup> in the bulk of the sample.

## Recombination Velocity

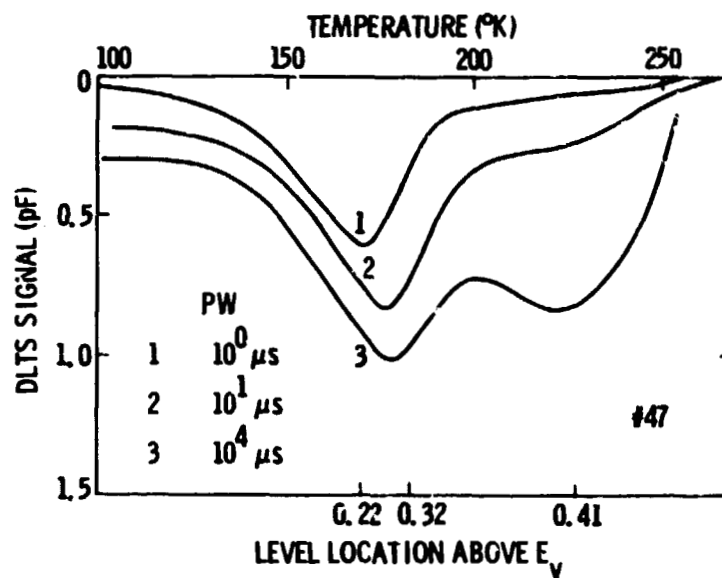
### RESULTS:

- DEVELOPED A TECHNIQUE USING PHOTOCONDUCTIVITY IN CONJUNCTION WITH  $\phi_{BO}$  (D) AND Q(D) MEASUREMENTS FOR THE MEASUREMENT OF MINORITY CARRIER RECOMBINATION VELOCITY AT THE GRAIN BOUNDARY
- OBSERVED INCREASES OF S WITH BOUNDARY STATE DENSITY AND LIGHT INTENSITY

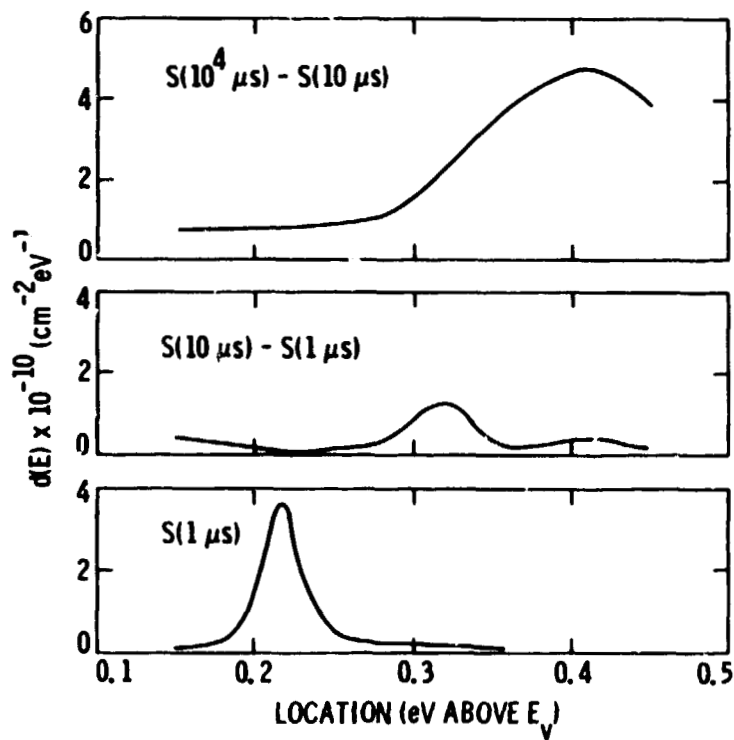
### Experimental Arrangement for DLTS Measurements



## Pulse Width Dependence



## Distribution of the Density of States



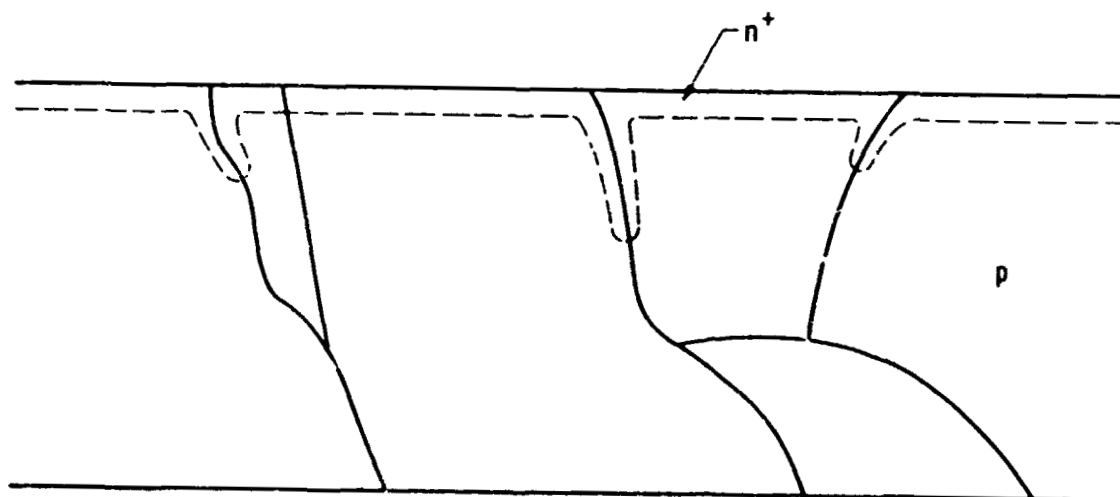


## Electronic States

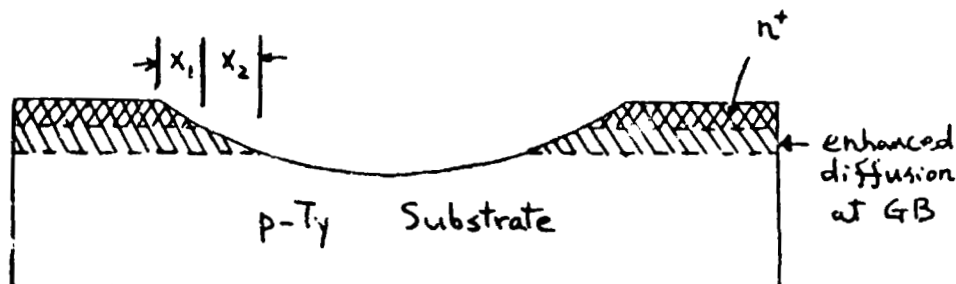
### RESULTS:

- DEMONSTRATED THE APPLICATION OF DEEP LEVEL TRANSIENT SPECTROSCOPY (DLTS) IN THE STUDY OF ELECTRONIC STATES AT GRAIN BOUNDARIES OF SILICON
- OBSERVED A TREND THAT THE DENSITY OF GRAIN BOUNDARY STATES IS GENERALLY INCREASING WITH THE DISTANCE FROM THE EDGES OF THE BAND GAP. HOWEVER, THE DETAILS VARY CONSIDERABLY FROM SAMPLE TO SAMPLE WHICH CAN BE ATTRIBUTED TO LOCAL VARIATION OF DISORDERS

Cross Section of Phosphorus-Diffused Polycrystalline Silicon



Cross Section of Grooved Sample at Grain Boundary



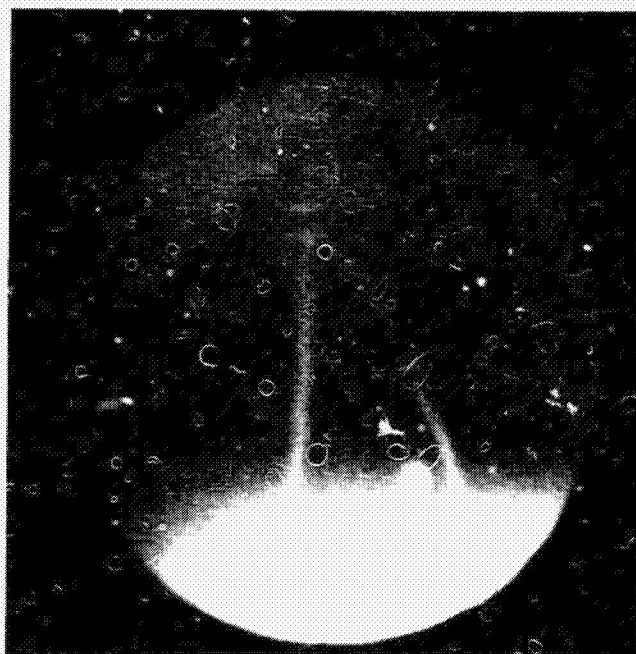
Staining !

F with light

Enhanced Diffusion of Phosphorus



GROOVED AND STAINED

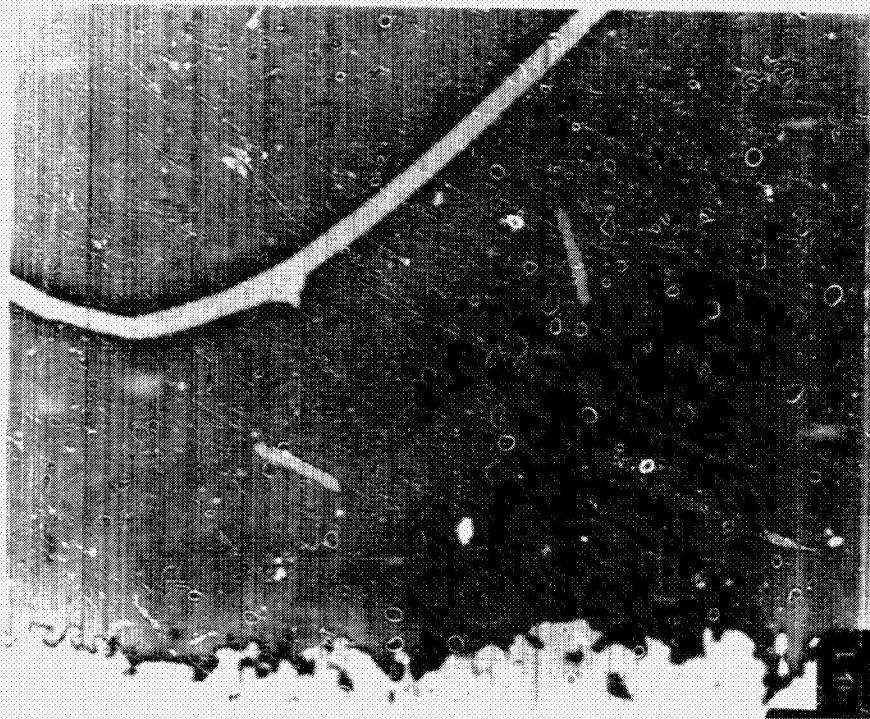


SiP<sup>-</sup> ION IMAGE

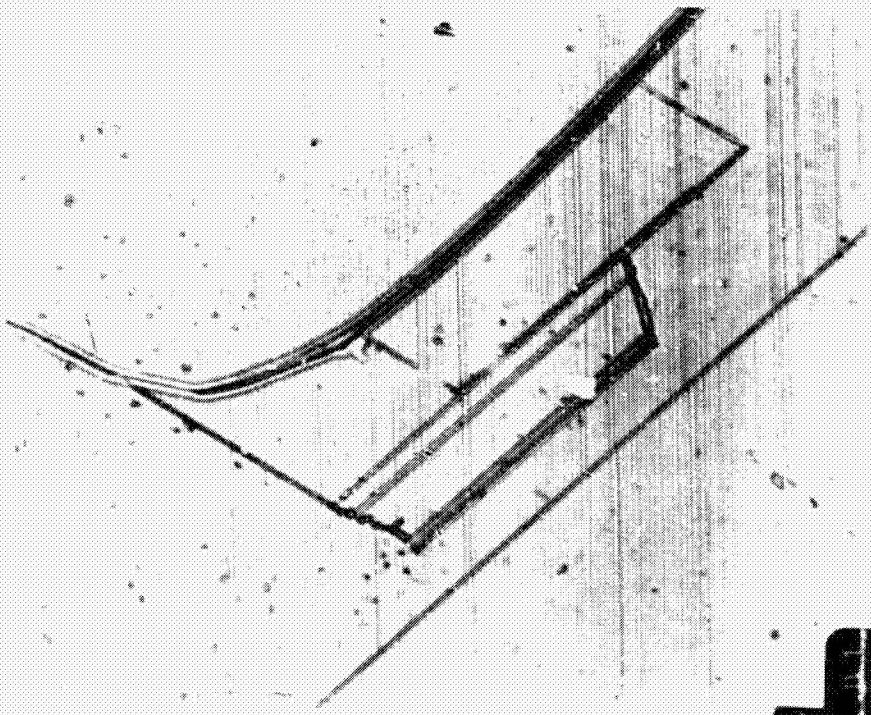
LARGE-AREA SILICON SHEET TASK

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Grooved and Stained



Sirtl-Etched





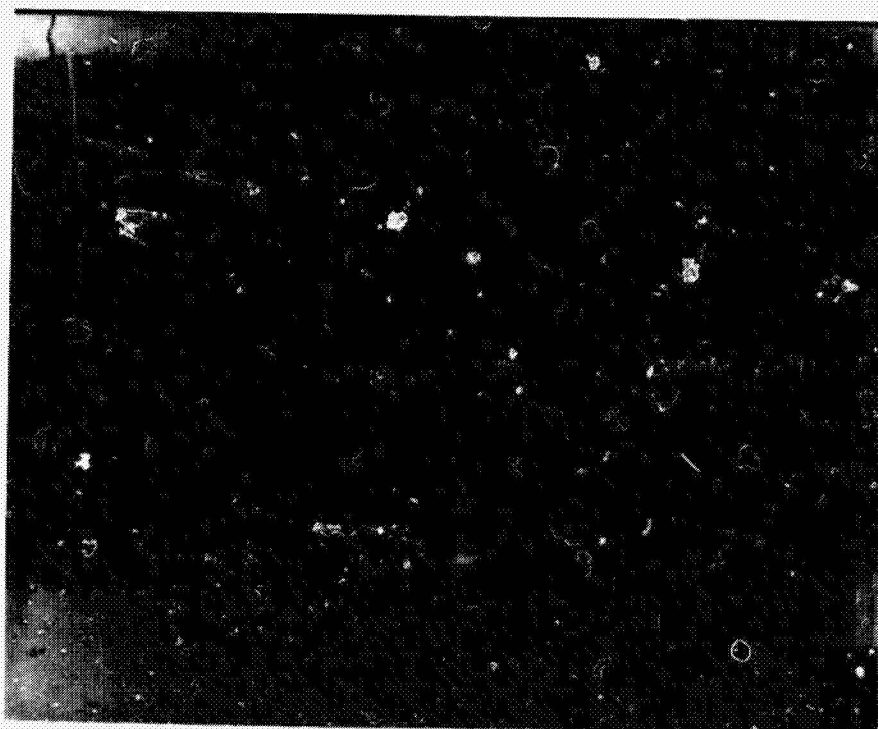
LARGE-AREA SILICON SHEET TASK

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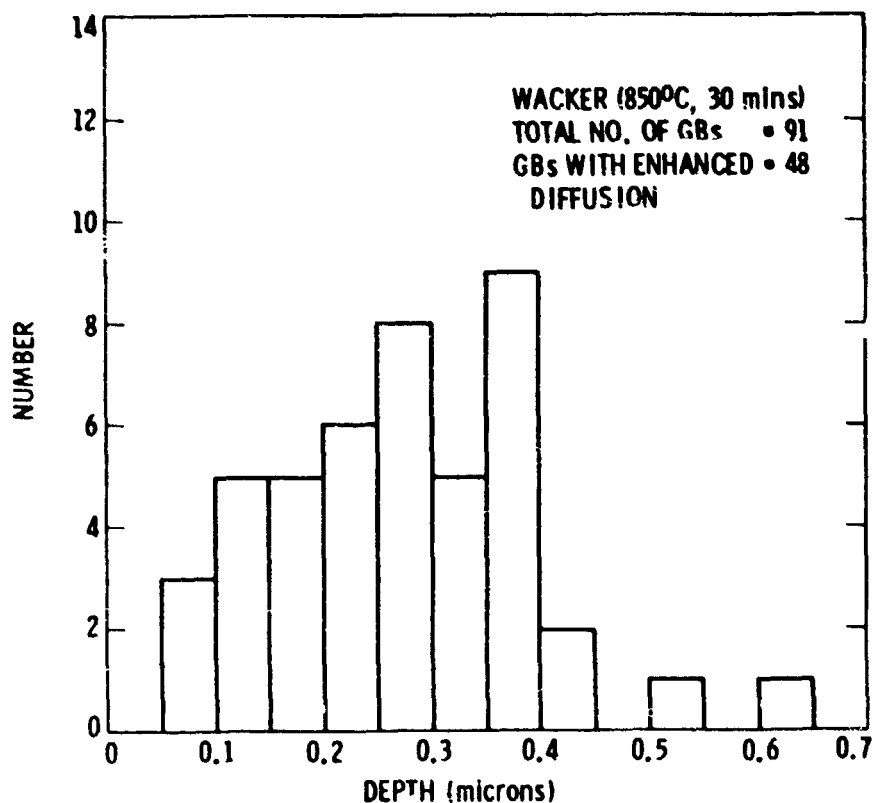
EBIC



Grooved and Stained



## Depth of Diffused Regions at Grain Boundaries



## Enhanced Diffusion of Phosphorus

## RESULTS:

- THE GROOVING AND STAINING TECHNIQUE IS A SUITABLE METHOD FOR THE OBSERVATION OF ENHANCED DIFFUSION OF PHOSPHOROUS AT GRAIN BOUNDARIES IN SILICON
- THE ENHANCED DIFFUSION OCCURS ONLY AT "HIGH-ORDER" GRAIN BOUNDARIES, GENERALLY ASSOCIATED WITH HIGH CARRIER RECOMBINATION
- THE DEPTH OF THE ENHANCED DIFFUSION VARIES DRASTICALLY FROM BOUNDARY TO BOUNDARY, WHICH MAKES THE QUANTITATIVE MEASUREMENT DIFFICULT UNLESS THE GRAIN BOUNDARY IS WELL CHARACTERIZED

## **LARGE-AREA SILICON SHEET TASK**

### **Present Activities and Plans**

- **DENSITY OF STATES, RECOMBINATION VELOCITY, AND BARRIER HEIGHT  
AS FUNCTIONS OF LATTICE MISMATCH AND PROCESS PARAMETERS  
(INCLUDING PASSIVATION)**
- **QUANTITATIVE STUDIES ON ENHANCED DIFFUSION OF PHOSPHOROUS**
- **IMPURITY BEHAVIOR (e.g., CARBON, AND OXYGEN)**
- **EFFECTS ON SOLAR CELL PERFORMANCE**

## STUDY OF ABRASIVE-WEAR RATE OF SILICON

UNIVERSITY OF ILLINOIS AT CHICAGO

J. Clark  
D.S. Lim  
S. Danyluk

<b>TECHNOLOGY</b> Materials Properties Modification	<b>REPORT DATE</b> April 22, 1982
<b>APPROACH</b> An experimental program is carried out to study the fundamental mechanisms of abrasion and wear, and deformation of silicon by a single crystal diamond in various fluid environments.	<b>STATUS</b> The abrasion rate and depth of damage of (100) and (111) p-type silicon in three fluid environments has been determined. The surface deformation mechanism was found to change when the fluid was varied.  The diamond geometry affects the wear rate.  There appears to be a correlation between the wear rate and the dielectric constant of the fluid. The brittle lateral crack model does not appear to describe the wear rates measured.
<b>CONTRACTOR</b> University of Illinois at Chicago	
<b>GOALS</b> Develop a model for surface-mechanical property modification of silicon under the influences of fluid environments	

## Introduction

Optical and scanning electron microscopy are used to determine the wear rate and deformation mechanism of diamond abrading (100) and (111) Cz silicon in water, ethanol and acetone. A multi-scratch experiment is used to determine the effects of normal force on the abrading diamond and fluid on the abrasion rate and depth of damage. These results are compared with a lateral crack model of abrasion of brittle materials.

## LARGE-AREA SILICON SHEET TASK

### Variables

Fluid, Temperature, Voltage, Photo-Irradiation, Normal Force ( $F_N$ ), Orientation, Abrasion Speed.

### Data

Groove depth vs. variables, SEM of groove surface, depth of damage.

### Analysis

Stress analysis, depth of damage, lateral crack model.

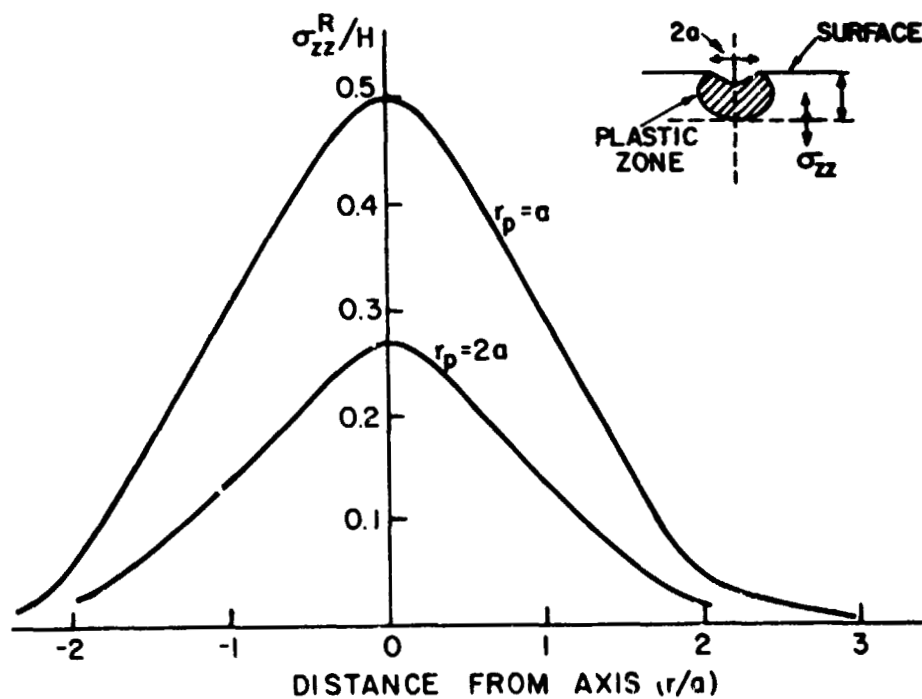
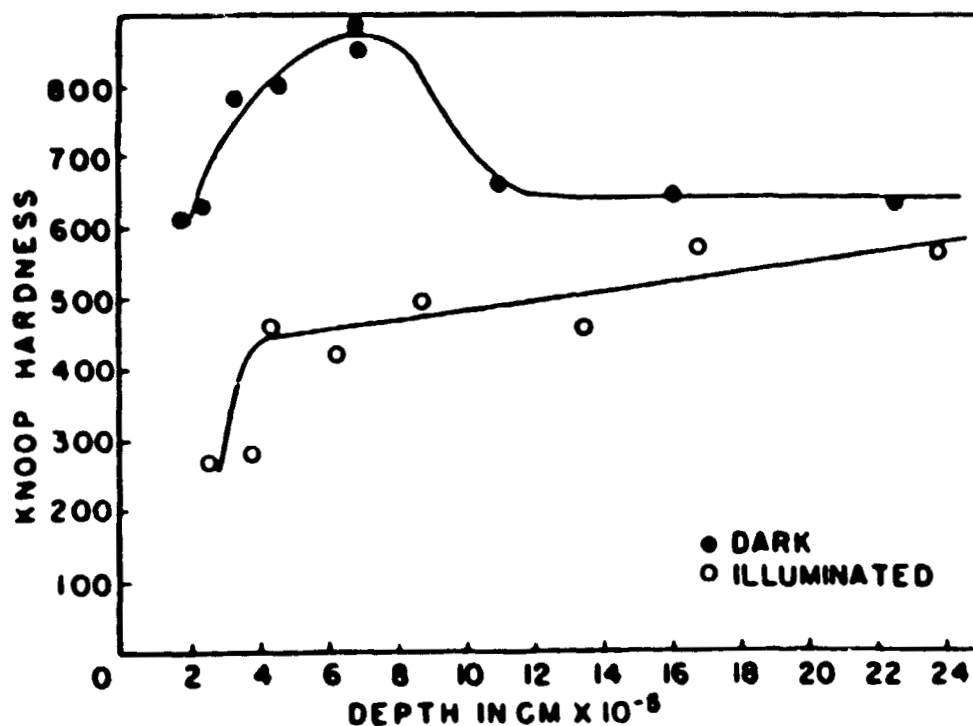
### Summary of Results

1. Wear rate varies by ~100% (acetone; ethanol; water).
2. Depth of damage larger for water than ethanol.
3. (100) and (111) wear rates are different.
4. Dielectric constant of the fluid related to the silicon hardness.
5. Lateral crack model describes wear rate when  $F_N \gtrsim 60$  g.



## Changes in Surface Hardness of Silicon

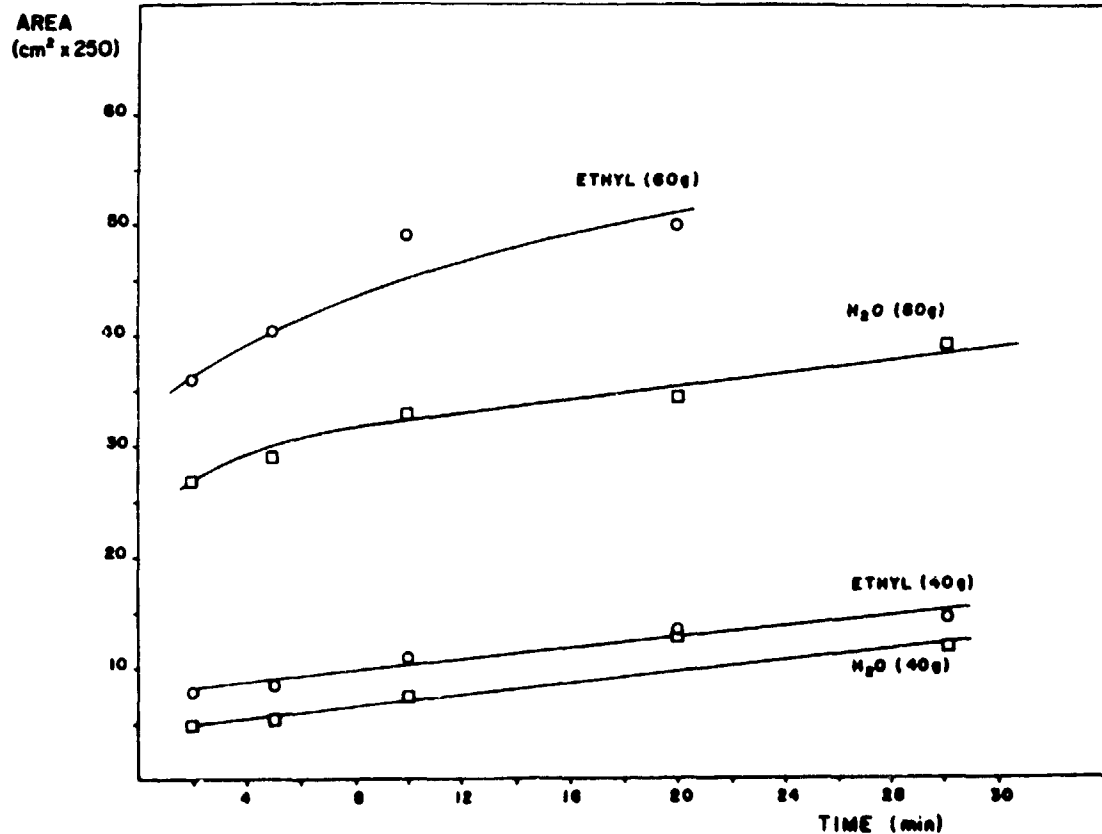
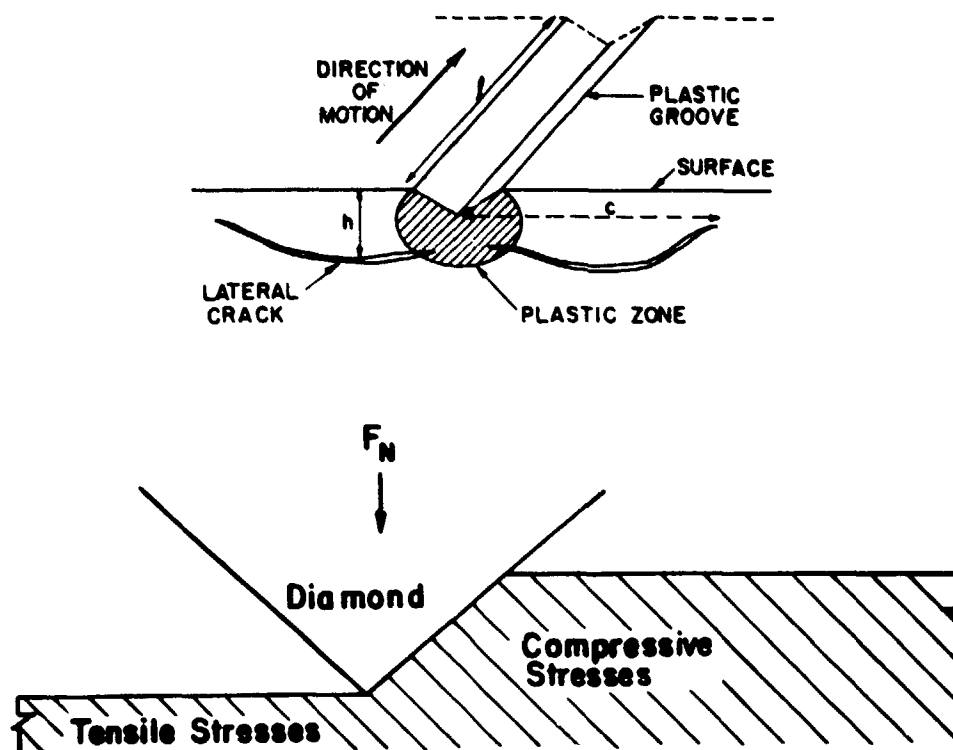
<u>Reference</u>	<u>Effect</u>	<u>Percent Softening</u>	<u>Comments</u>
Kuczynski and Hochman	Photon irradiation	70% softening	Intensity and surface preparation important; microhardness test
Ablova	H <sub>2</sub> O adsorption	Softening	Surface preparation and impurity content important; microhard- ness test
Westbrook and Gilman	Potential between indenter and crystal	60% softening	Disappeared at elevated temperatures; micro- hardness test
Yost and Williams	NaCl and Na <sub>4</sub> P <sub>2</sub> O <sub>7</sub>	50-80% softening depending on concentration	Zeta-potential measurements of crushed silicon
Cuthrell	CCl <sub>4</sub> and H <sub>2</sub> O adsorption	Not determined	Adsorption changed mode of drilling
This work	H <sub>2</sub> O, ethanol, acetone adsorption	Up to 70% soften- ing dependent on type of fluid and F <sub>N</sub>	Pyramid diamond scratch test



The spatial dependence of the residual tensile stress along a plane through the intersection of the plastic zone with the penetration axis: the stresses were estimated from an analytic elastic/plastic solution for a spherical cavity and an elastic solution for a half space.

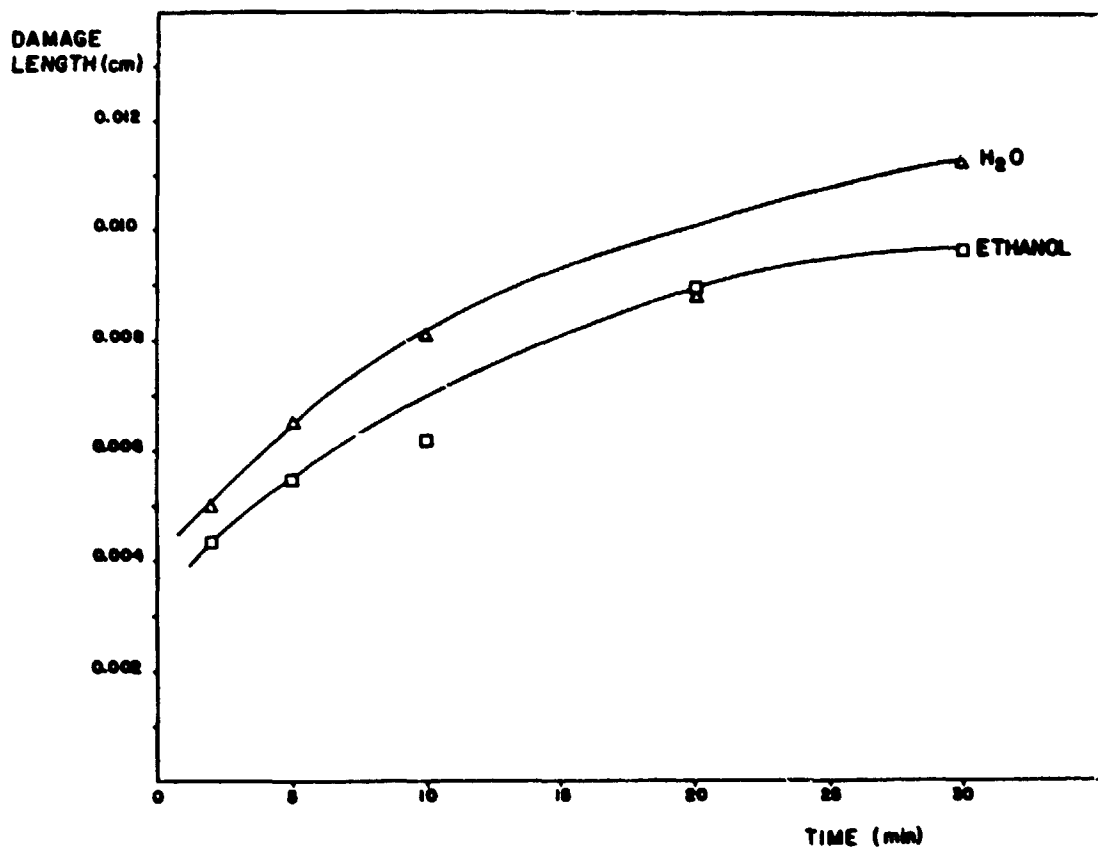
# LARGE-AREA SILICON SHEET TASK

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# LARGE-AREA SILICON SHEET TASK

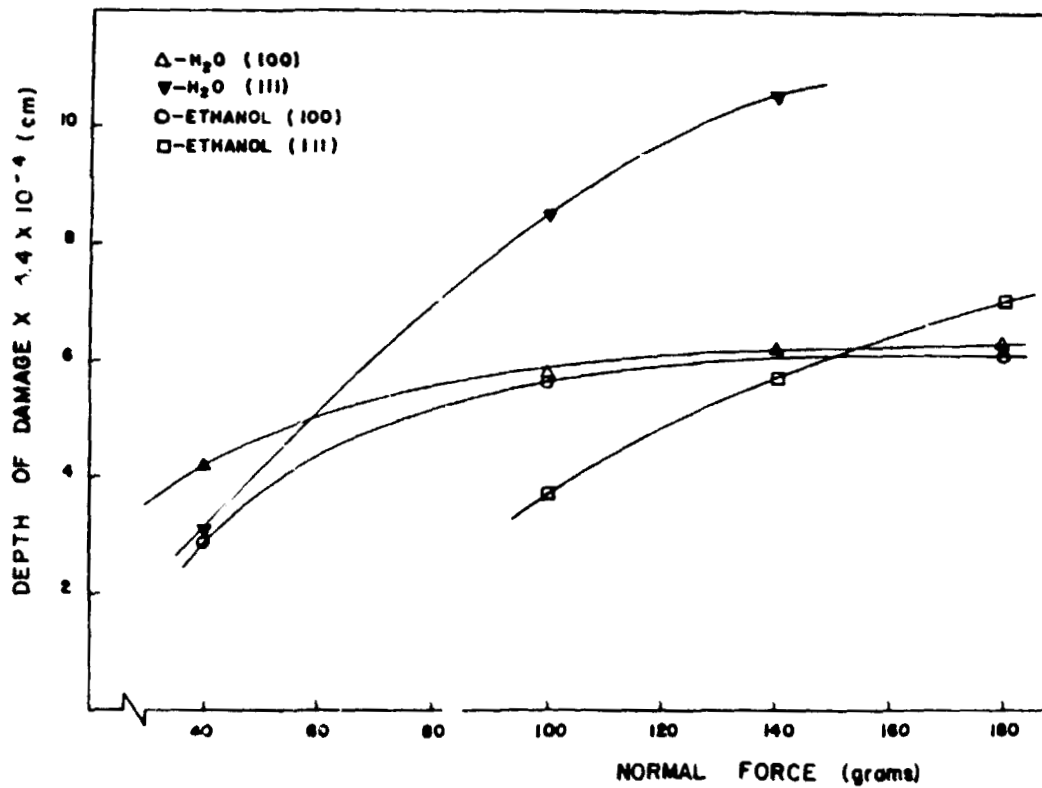
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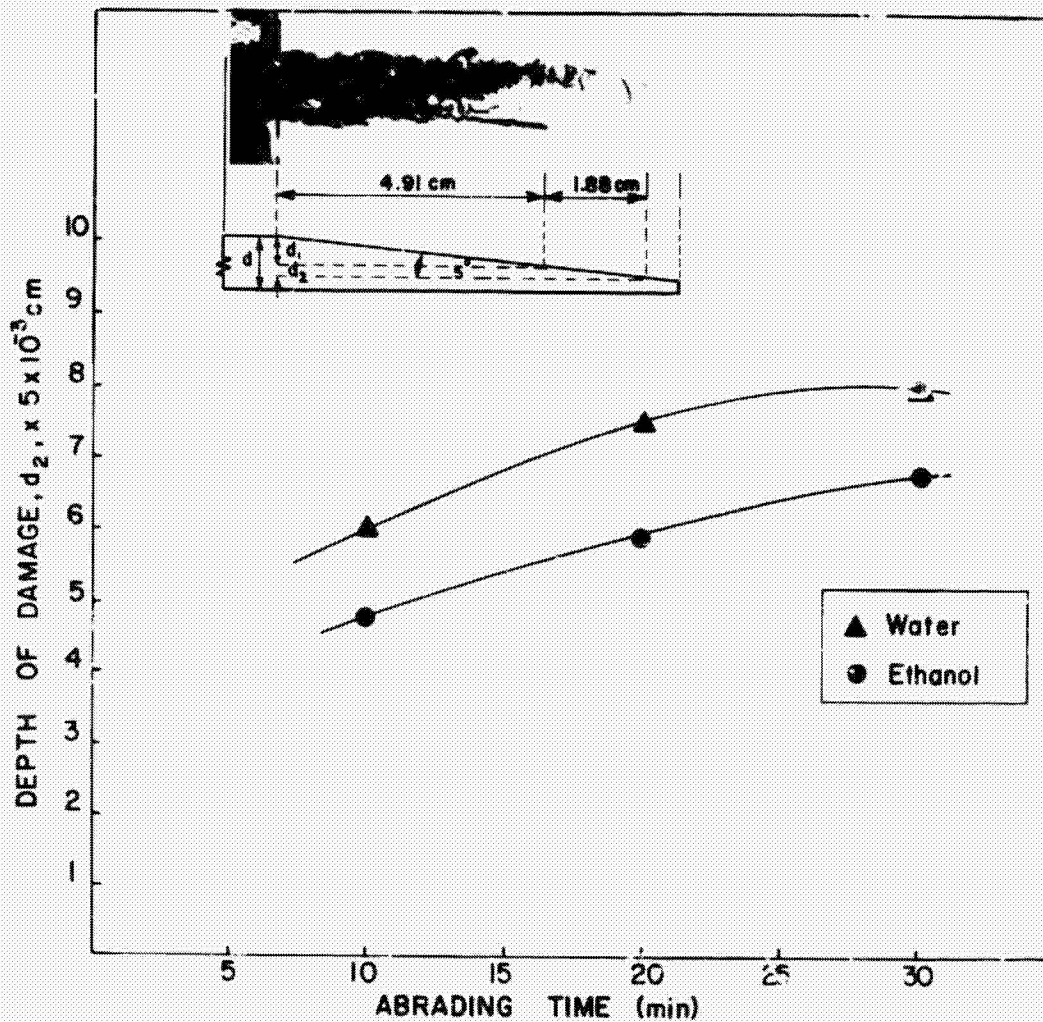


LARGE-AREA SILICON SHEET TASK

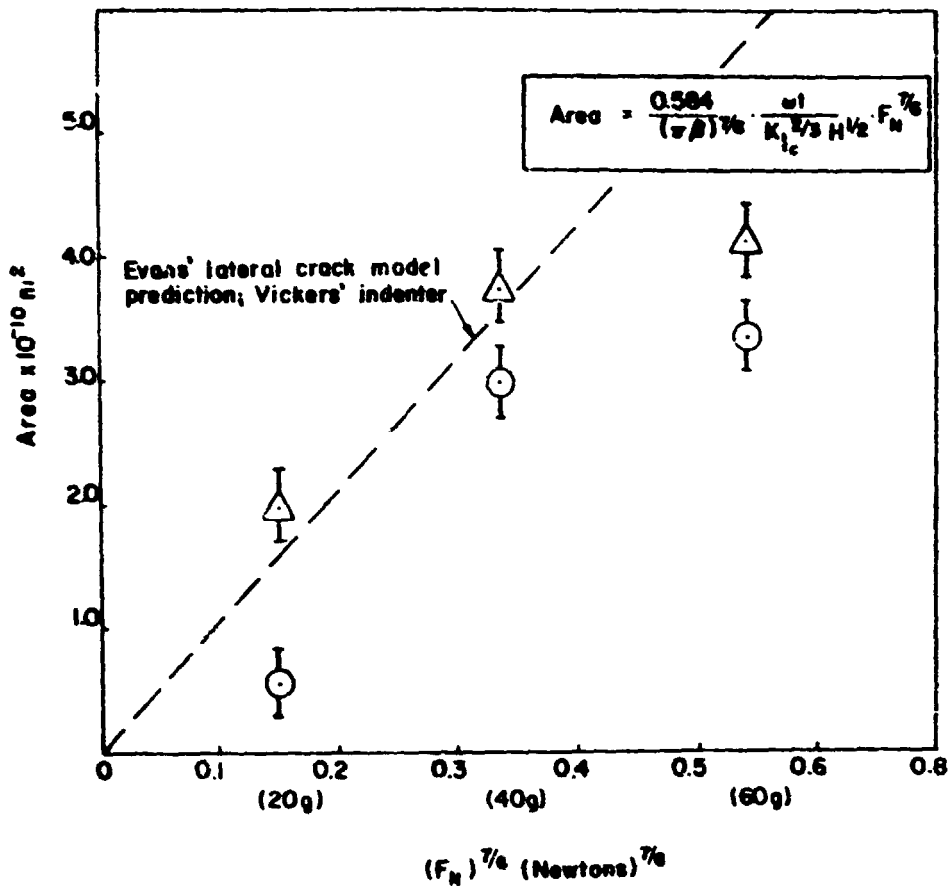
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Depth of Damage From Single Scratch Abrasion on Silicon





Depth of Damage in (100) p-Type Silicon Formed by a Diamond at Room Temperature vs Abrasion Time (min.). The Fluid Environment Was Varied. The Normal Force Was  $F_N = 40 \text{ g}$ .



### Problems and Concerns

The diamond geometry changes during the abrasion experiments due to microcracking at the diamond surface.

The diamond vibration must be stabilized.

The temperature at point of contact between the diamond and the silicon is unknown.

## SOLAR CELL FABRICATION AND ANALYSIS

APPLIED SOLAR ENERGY CORP.

<b>TECHNOLOGY</b> SOLAR CELL FABRICATION & ANALYSIS	<b>REPORT DATE</b> APRIL 22, 1962
<b>APPROACH</b> 1) FABRICATION OF SOLAR CELLS BY BASELINE & ADVANCED PROCESSES POSSIBLY INCLUDING GETTERING AND ANNEALING. 2) ANALYSIS USING DARK AND LIGHT I-V, DIFFUSION LENGTH MEASUREMENTS, SPECTRAL RESPONSE. <b>CONTRACTOR</b> APPLIED SOLAR ENERGY CORPORATION	<b>STATUS</b>
<b>GOALS</b> 1) AN UNDERSTANDING OF THE MECHANISMS THAT LIMIT THE DEFICIENCIES OF SOLAR CELLS MADE FROM VARIOUS SILICON SHEETS. 2) AN UNDERSTANDING OF THE EFFECT ON SOLAR CELL EFFICIENCY OF VARIATIONS IN GROWTH PARAMETERS.	

## 1. EFG (MOBILE TYCO)

10 CM WIDE MATERIAL GROWN WITH OR WITHOUT CO<sub>2</sub>.

## 2. UCP (SEMIX)

HIGH EFFICIENCY PROCESS ON MATERIAL FROM INGOT 5464-13C.  
MORE SEVERE GETTERING ON MATERIAL FROM INGOT 5464-13C.  
10 CM X 10 CM CELLS ON MATERIAL FROM RANDOM SOURCES.

## 3. HEM (CRYSTAL SYSTEM)

MORE SEVERE GETTERING ON MATERIAL FROM INGOTS 4141C AND 4148.



## Summary of Solar Cells Made From EFG 17-200 Series

		Voc (mV)	Jsc (mA/cm <sup>2</sup> )	FF (%)	(%)	REMARKS
17-200-1A (4 CELLS)	AVE. S.D. RANGE	495 ±10 480-504	22.5 ±1.5 20.2-23.6	71 ±4 65-73	7.9 ±.7 7.1-8.6	CO <sub>2</sub> OFF
17-200-113 (2 CELLS)	AVE. S.D. RANGE	515 ±7 510-520	23.0 ±1.2 22.1-23.8	76 ±1 75-77	9.0 ±.5 8.6-9.3	CO <sub>2</sub> ON
17-200-1D (3 CELLS)	AVE. S.D. RANGE	529 ±5 524-534	24.2 ±.2 24.0-24.3	74 ±3 70-76	9.4 ±.4 9.0-9.7	
17-202-1C (4 CELLS)	AVE. S.D. RANGE	505 ±17 486-516	22.6 ±1.6 20.8-24.2	73 ±1 72-74	8.3 ±.9 7.4-9.2	
17-203-1D (2 CELLS)	AVE. S.D. RANGE	499 ±1 498-500	21.0 ±.3 20.8-21.2	73 ±1 72-74	7.7 ±.3 7.5-7.9	
17-203-1E (2 CELLS)	AVE. S.D. RANGE	487 ±7 482-492	19.8 ±1.4 18.8-20.8	71 ±4 68-74	6.9 ±.2 6.7-7.0	
ACCUMULATIVE AVE OF "CO <sub>2</sub> ON" CELLS (13 CELLS)		508	22.4	73	8.4	

IC2 CONTROL (4 CELLS)	AVE. S.D. RANGE	583 ±2 580-584	27.9 ±.4 27.4-28.3	78 ±1 77-79	12.6 ±.2 12.3-12.9
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LARGE-AREA SILICON SHEET TASK

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Summary of Solar Cells Made From 17-175 Series

		VOC (mV)	Jsc (mA/cm)	CFF (%)	(%)	REMARKS
17-175-1A-2 (7 CELLS)	AVE	519	21.2	72	7.9	CO <sub>2</sub> OFF
	S.D.	$\pm 8$	$\pm 1.15$	5	$\pm .8$	
	RANGE	504-530	19.8-22.8	62-75	6.4-9.0	
17-175-1A-6 (5 CELLS)	AVE	493	20.1	61	6.2	CO <sub>2</sub> OFF
	S.D.	$\pm 34$	$\pm 1.2$	$\pm 16$	$\pm 2.1$	
	RANGE	434-518	19.4-21.8	61-74	2.7-8.3	
ACCUMULATIVE AVERAGE OF "CO <sub>2</sub> OFF" CELLS (12 CELLS)		508	20.7	67	7.2	
17-175-1E-52 (8 CELLS)	AVE	539	22.3	73	8.9	CO <sub>2</sub> ON
	S.D.	$\pm 12$	$\pm 2.1$	$\pm 3$	$\pm 1.1$	
	RANGE	516-554	19.3-24.7	68-77	7.0-10.4	
17-175-1E-56 (6 CELLS)	AVE	505	21.3	59	6.4	CO <sub>2</sub> ON
	S.D.	$\pm 40.4$	$\pm 2.4$	$\pm 15$	$\pm 2.2$	
	RANGE	432-546	17.6-22.6	35-70	3.4-9.1	
ACCUMULATIVE AVERAGE OF "CO <sub>2</sub> ON" CELLS (14 CELLS)		524	21.9	67	7.8	

CZ CONTROL (4 CELLS)	AVE	585	28.2	75	12.4
	S.D.	$\pm 2$	$\pm .6$	$\pm 3$	$\pm .4$
	RANGE	582-586	27.5-28.9	71-78	12.0-12.7

# LARGE-AREA SILICON SHEET TASK

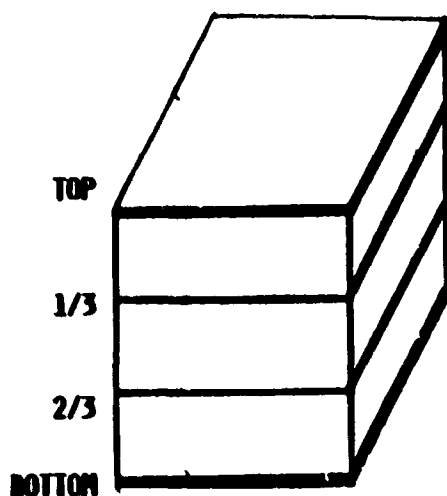
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OF POOR QUALITY

## Distribution of $J_{SC}$ on EFG Ribbon 17-175-1E-52

19.3	24.7	22.4	21.2
20.0	24.7	21.8	24.5

*growth direction*

## UCP Ingot No. 5848-13C

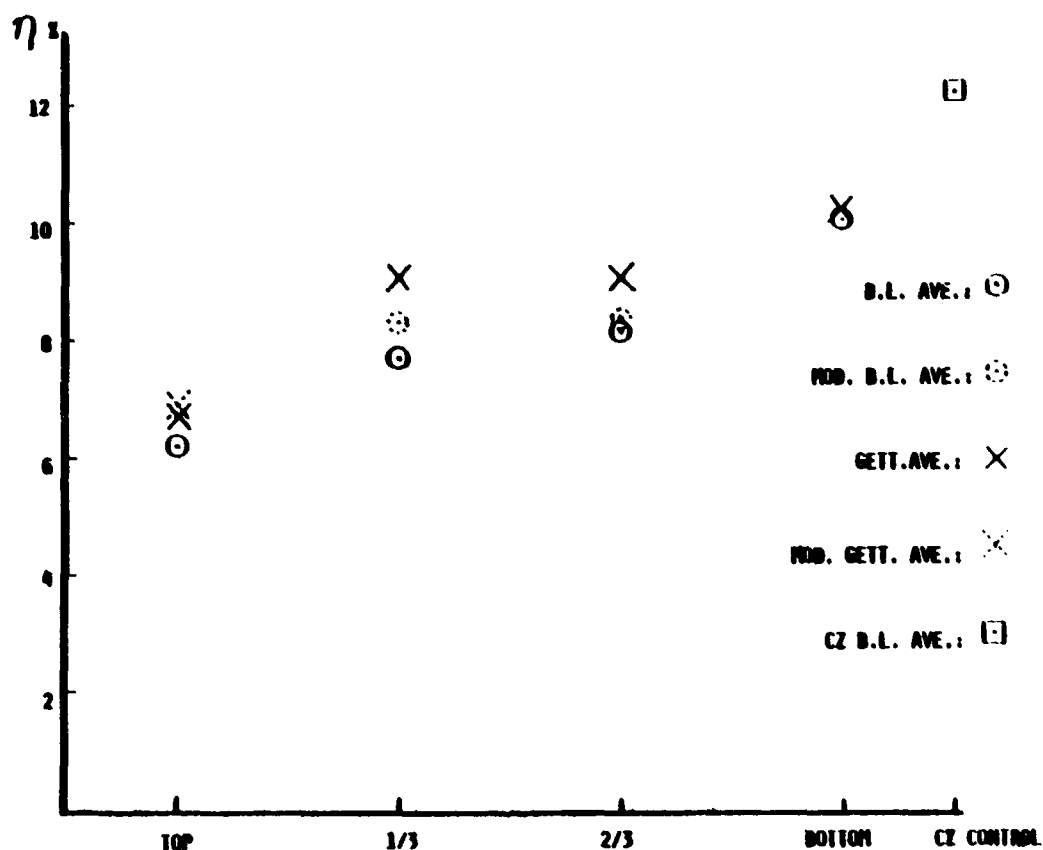


~ 4" x 4" x 4.5"

1	2	3	4
5	6	7	8
9	10	11	12
13	14	15	16

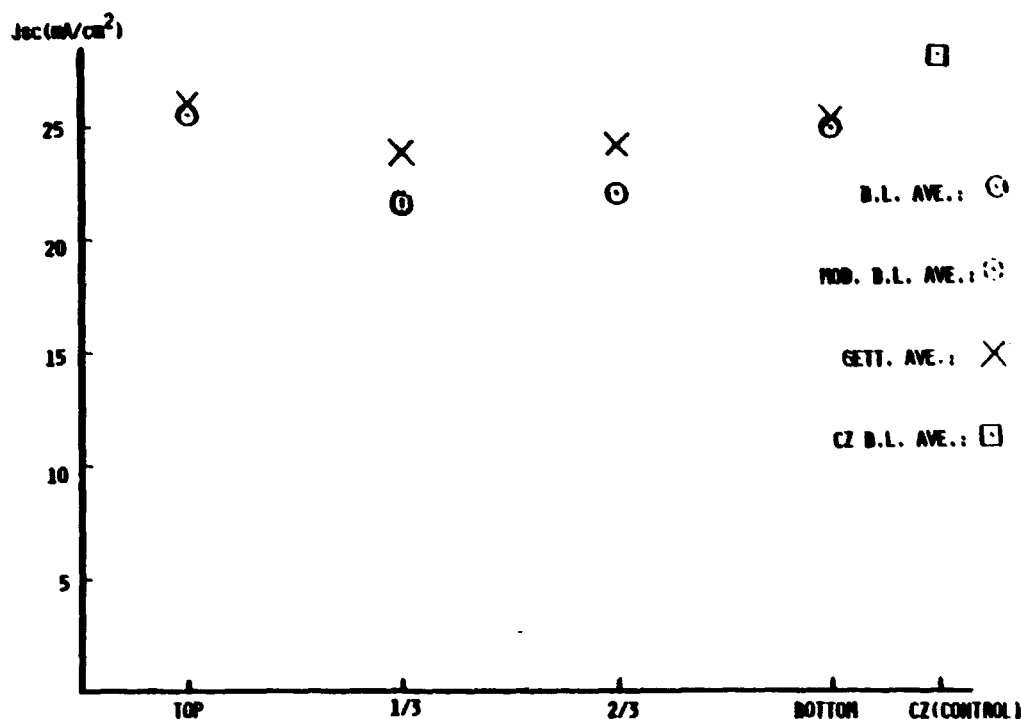
THE CELL'S # AND THEIR RELATIONS TO THE ORIENTATION OF  
THE QUARTER INGOT ARE MARKED

## Average Efficiency of Different Layers of Ingot 5840-13C



## Summary of SJ, BSF and MLAR Cells From UCP Ingot 5848-13C

		Voc (mV)	Jsc (mA/cm <sup>2</sup> )	CFP (%)	$\eta$ (%)
Evaporated Al BSF (UCP - 12 Cells)	A.V.	561	28.7	76	12.2
	S.D.	6	0.6	4	0.8
	RANGE	550-570	27.4-29.4	62-79	9.9-13.1
CZ Control (3 Cells)	A.V.	593	32.6	78	15.1
	S.D.	1	0.2	1	0.2
	RANGE	592-594	32.4-32.8	77-79	14.9-15.3

Average  $J_{sc}$  of Different Layers, Ingot 5848-18CSummary of  $J_{sc}$  From Cells From More Severe  
Gettering Tests (UCP Ingot No. 5848-13C)

Gettering Treatment	Wafer	Ave. $J_{sc}$ (mA/cm <sup>2</sup> )	$J_{sc}$ * of The Cell Covered With SiO <sub>2</sub>
None	1/3 2/3	22.5 23.7	- -
875°C 1/2 Hr.	1/3 2/3	24.6 24.3	24.7 24.8
875°C 1 Hr.	1/3 2/3	25.5 26.3	25.2 26.6
950°C 1 Hr.	1/3 2/3	27.0 26.3	24.8 25.9
CZ Control (No Treatment)		28.2	-
1050°C 1 Hr.	1/3	26.2	25.9
CZ Control (No Treatment)		28.2	-

\*  $J_{sc}$  of the cell covered with CVD SiO<sub>2</sub> during gettering diffusion.

Results of Light-Bias Minority Carrier  
Diffusion Length Study on Getter

PROCESS	CELLS #	$L_{D1}$ ( $\mu\text{m}$ ) D.C. DARK	$L_{D2}$ ( $\mu\text{m}$ ) 0.05 SUN	$L_{D3}$ ( $\mu\text{m}$ ) 1 SUN	$L_{D4}$ ( $\mu\text{m}$ ) DARK AFTER LIGHT TURN OFF
BASELINE	2-1	11	16	17	12
8.75°C 1/2 Hr GETTERING	2-4 2-8*	29 29	44 40	60	30
875°C 1 HR GETTERING	2-13 2-9*	159 100	65 60	72	140
950°C 1 HR GETTERING	2-12 2-15*	182 107	89 49	72	167
1050°C 1 HR GETTERING	2-12 2-9*	212 135	116 62	69 81	244 117
CZ CONTROL	1	150	152	156	137

\* CELLS WERE COVERED WITH SiO<sub>2</sub> DURING GETTERING DIFFUSION.Summary of Results From 10 x 10 UCP Cells  
From Random Sources

	Voc (mV)	Jsc (mA/cm <sup>2</sup> )	CFF (%)	(%)
AVE.	553	26.9	72	10.8
S.D.	6	.9	1	.5
RANGE	546-558	25.2-27.6	72-74	10.0-11.3

AREA = 98 cm<sup>2</sup>

NO. OF CELLS = 6

Comparison of J<sub>sc</sub> From HEM Cells Gettered for 1 h at 1050°C  
With HEM Baseline Cell From Corresponding Area

INGOT #	BASELINE Jsc	GETTER Jsc
41-41C	25.6	26.3
41-48	27.6	28.1