SILICON MATERIALS TASK OF THE LOW
COST SOLAR ARRAY PROJECT (PART 2)

Third Quarterly Report, 1 April 1976—30 June 1976

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1976

Work Performed Under JPL Contract No. 954331

Westinghouse Research Laboratories
Pittsburgh, Pennsylvania

ENERGY RESEARCH AND DEVELOPMENT ADMINISTRATION
Division of Solar Energy

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Westinghouse Research Laboratories

and

J. R. McCormick
Dow Corning Corporation

Contract No. 954331

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Approved:

R. Mazelsky, Manager
Crystal and Vacuum Technology

NEW TECHNOLOGY

No new technology is reportable for the period covered by this report.

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1. SUMMARY

The objective of this program, Part 2 of the Silicon Materials Task, is to develop and define purity requirements for solar grade silicon by exploring the effects of metal impurities on the performance of terrestrial silicon solar cells. The project has been increased in duration to twelve months to accommodate the growth and evaluation of more doped ingots, improvements in sample analysis, and refinements to data collection and evaluation.

During this quarter we completed the growth of all first, second, and nearly all third generation ingots, and initiated the growth of fourth generation ingots. Several boron-doped silicon dendritic web baseline samples have been grown as well as one web doped with chromium. Chemical analysis of the ingots is proceeding on schedule, though, as expected, difficulties in assessing the impurity levels of lightly-doped ingots have developed.

Lifetime measurements have been completed for all 38 ingots grown to date. In addition, lifetime measurements have been used to deduce the presence of gettering and precipitation during various heat treatments and diffusion cycles employed in cell processing. These studies show that even during the 550°C contact sintering lifetime can be affected. At 825°C, the diffusion temperature, both precipitation and gettering occur. The latter result is confirmed by Auger spectroscopic measurements of cell impurity profiles which show that Cr, Ni and Cu each tend to pile up in the n^+ layer during the diffusion cycle.

Recent cell measurements verify our initial conclusions that Ti and V sharply degrade cell performance. Wafers with nominal

10^{13} atom cm^{-3} Ti and V contents exhibit cell efficiencies only 83 and 75% of the baseline cells, respectively. In contrast, when the Mn content is reduced to 10^{14} atoms cm^{-3} cell efficiency rises to 90% of the baseline value.

Cell data for the first set of doubly-doped ingots suggest that Cr, Mn and Cu act independently with respect to their impact on cell performance. For example, wafers containing Cr/Cu and Mn/Cu combinations (each element at 10^{15} atoms cm^{-3}) exhibit cell efficiencies about 70% of baseline -- similar to wafers containing Cr and Mn alone (Cu itself hardly reduces cell output). When Mn and Cr are combined, relative cell efficiency falls to 41% of baseline, illustrating the combined effect of the two harmful impurities.

2. INTRODUCTION

2.1 Program Extension

During the last quarter we received JPL approval to extend the duration of the program to 12 months from the original nine-month period. The increased time facilitates information feedback from the cell performance evaluation to crystal growth so that more complete data analysis can be made prior to ingot doping selection. The added program effort (Fig. 9, Section 5) is focused on increasing the number of doped ingots and webs to be grown and evaluated, improving the analytical results, and refining the data analysis.

Because of the change in program schedule and the rather recent publication of our Second Quarterly Report,² we have confined ourselves here mainly to tabulations of data collected since that last report and some brief descriptive comments. A detailed analysis of the results will follow in later reports.

2.2 Program Objectives

Our objective in Part 2 of the Silicon Materials Task (LCSSAP) is to develop and define purity requirements for solar cell grade silicon material. In a broader sense we are developing a data base which delineates the effects of various impurities and impurity levels on the performance of terrestrial silicon solar cells.

2.3 Program Approach

The basic approach we have taken is to establish, as unambiguously as possible, what concentrations of the impurities commonly

found in silicon starting material (metallurgical grade silicon) can be tolerated in silicon wafers produced by (a) standard Czochralski pulling and (b) rapid sheet growth (dendritic-web) without degrading solar cell performance. Other impurities, e.g., those entering the material via particular purification or sheet growth processes could also be examined within the framework of our methodology as they are identified. Our experimental methods have been described extensively in previous reports.^{1,2}

2.4 Program Personnel

This program is being conducted by a team of scientists from the Westinghouse Electric Corporation and Dow Corning Corporation whose technical responsibilities are identified below.

Westinghouse Research Laboratories

Dr. R.H. Hopkins -- Technical Manager and Dendritic Web Studies

Dr. J.P. McHugh -- Web Growth

Dr. P. Rai-Choudhury -- Device Diffusion

Mr. J.R. Davis -- Solar Cell Testing and Analysis

Mr. P.D. Blais -- Lifetime Studies, Photolithography and Metallization

Dow Corning Corporation

Dr. J.R. McCormick -- Czochralski Ingot Preparation and Evaluation

Dr. R. Mazelsky, Westinghouse, has administrative responsibility for the program.

3. TECHNICAL PROGRESS

3.1 Impurity Matrix

The two-dimensional impurity matrix employed in the present program for evaluating the effects of elements not found in semiconductor grade silicon, but which may occur in a low cost solar grade material is shown in Fig. 1. Also depicted in the figure is the current status of ingots containing those impurities. While we have attempted to obtain first generation Czochralski ingots doped with 5×10^{14} to 5×10^{15} impurity atoms per cm^3 , this has proven exceedingly difficult for the elements magnesium and zinc due to their volatility, and titanium, vanadium, and zirconium due to their extremely small effective segregation coefficients. All ingots except one have been doped with 3.5×10^{15} atoms/ cm^3 ($\pm$ 30 percent) boron as the acceptor impurity.

In addition to the single element impurity matrix shown in Fig. 1, first generation ingots doped with combinations of impurities have been prepared: Mn/Cu, Cr/Cu, Cr/Mn, and Zr/Ti.

3.2 Ingot and Web Preparation

3.2.1 Ingot Preparation

The details of the ingot preparation and characterization have been discussed in previous project reports.^{1,2} The dopant elements, their characteristics and the segregation coefficients used to establish the melt concentrations of the Czochralski ingots are collected in Table 1. The values shown in parentheses in the last column of the table are the best estimate of effective segregation coefficient based on data collected during this program. Two values of segregation coefficient

Impurity Element	Ingot Status				
	Concentration Range				
	10^{14}	5×10^{14} To 1×10^{15}	1×10^{15} To 5×10^{15}	5×10^{15} To 1×10^{16}	$> 10^{16}$
Cr	G, F	G, F, T	G, F, T	G, F, T	G, F, T
Mn	G, F, T	G, F, T	G, F, T		
Cu		G, F, T	G, F, T		
Ni		G, F, T	G, F, T		
Fe			G, F, T		
Ti	G, F, T	G, F, T			
V	G, F, T	G, F, T			
Mg	G, F, T	G, F, T			
Zn	G, F, T				
Zr		G, F, T			
Al					

Fig. 1. Status of the metal-doped ingots which form the impurity matrix for this program.

Table 1. Characteristics of Dopant Impurities

Dopant	Form	Purity %	Melting Point (°C)	Temperature For 1 mm Vapor Pressure (°C)	Segregation Coefficient*
Aluminum ⁺	Wire	99.99	659	1284	1.6×10^{-3} ($2.8 \times 10^{-3}/3 \times 10^{-2}$)
Chromium	Pellets	99.999	1900	1504	10^{-5} ($.95 \times 10^{-5}$)
Copper	Zone Refined Ingot	99.9997	1083	1628	4×10^{-4} (4×10^{-4})
Iron	Sponge/ Ingot	99.999/ 99.999	1535	1783	0.8×10^{-5} (---)
Magnesium	Ingot	99.99	651	605	$\sim 10^{-3}$ (1×10^{-5})
Manganese	Flake	99.99	1244	1251	10^{-5} (1.35×10^{-5})
Nickel	Sponge/ Wire	99.999/ 99.97	1455	~ 1884	0.8×10^{-5} (3.2×10^{-5})
Titanium	Crystal	99.95	1668	~ 2500	10^{-5} ($.36 \times 10^{-5}$)
Vandium	Dendrite	99.9	2190	2550	10^{-5} ($.45 \times 10^{-5}$)
Zinc	Rod	99.999	419	487	10^{-5} (---)
Zirconium	Foil	99.99	2127	2450	10^{-5} ($<.45 \times 10^{-5}$)

* The segregation coefficients shown are best values obtained from a review of the literature on impurities in silicon. The quantity in parentheses represent calculated segregation coefficients based on this work.

⁺ Two segregation coefficients are shown for aluminum. The first value is based on electrically active aluminum determined by resistivity measurements while the second value is based on total aluminum content in the ingot as determined by mass spec.

are provided for aluminum due to its occurrence in both an electrically active and non-electrically active species. The value of 2.8×10^{-3} relates to the electrically active species (that which affects resistivity). The effective segregation coefficient of magnesium is more than two orders-of-magnitude smaller than the value previously reported in the literature while the effective segregation coefficients for titanium, vanadium, and zirconium are significantly smaller than previously published values.

During this abbreviated reporting period, seven ingots were prepared. The status of these and previously grown ingots is summarized in Table 2. In the table the letter designation indicates the ingot has been grown (G), sliced (S), analyzed (A), and processed into cells (P). Thirty-eight ingots have now been prepared, twenty-eight of these doped with one or more impurities. Analysis is up-to-date for all but two of the doped ingots.

We have encountered considerable difficulty in growing some multiply-doped ingots due to the extremely high melt impurity concentrations required. Although single crystal ingots have been pulled from melts containing from 1 to 2 percent impurity concentration, yield has been quite low, as might be expected.

3.2.2 Web Preparation

During the past quarter we put into use a new, larger diameter web furnace which accommodates a melt volume about three times larger than the original furnace we had used. This change, coupled with changes in the furnace heat shielding and coil design, provided the conditions necessary to assure that web material could be produced each run. The initial debugging of the furnace has been completed. Twelve web segments varying in length from 4 cm to 74 cm, the maximum travel distance of our withdrawal mechanism, have been grown. The general characteristics of these first dendritic webs can be visualized with the aid of Fig. 2, which illustrates a representative sample of the crystals grown.

Table 2. Ingot Status

<u>Ingot Identification</u>	<u>Status</u>	<u>Ingot Identification</u>	<u>Status</u>
W-001-00-000	G,S,A,P	W-020-00-000	G,S, P
W-002-00-000	G,S,A,P	W-021-Mg-001	G,S,A,P
W-003-00-000	G,S,A,P	W-022-00-000	G,S, P
W-004-Cr-001	G,S,A,P	W-023-00-000	G,S
W-005-Mn-001	G,S,A,P	W-024-Mg-002	G,S,A,P
W-006-Ni-001	G,S,A,P	W-025-00-000	G,S, P
W-007-Cu-001	G,S,A,P	W-026-Mn-003	G,S,A,P
W-008-Ti-001	G,S,A,P	W-027-Mn/Cu-001	G,S,A,P
W-009-V-001	G,S,A,P	W-028-Al-001	G,S,A,P
W-010-Ni-002	G,S,A,P	W-029-Cr-003	G,S,A
W-011-Zr-001	G,S,A,P	W-030-Cr/Cu-001	G,S,A,P
W-012-Cr-002	G,S,A,P	W-031-Cr/Mn-001	G,S,A,P
W-013-Mn-002	G,S,A,P	W-032-Mg-003	G,S,A
W-014-00-000	G,S,A,P	W-033-Ti-002	G,S,A,P
W-015-Zn-001	G,S,A,P	W-034-00-000	G,S
W-016-Fe-001	G,S,A,P	W-035-V-002	G,S,A,P
W-017-Cu-002	G,S,A,P	W-036-Zr-002	G,S
W-018-Fe-002	G,S	W-037-Zr/Ti-001	G,S
W-019-Cu-003	G,S,A,P	W-038-Al-002	G,S,A

Recall that in the web growth process two coplanar silicon dendrites penetrate into a supercooled liquid pool. Surface tension raises a thin liquid sheet between the dendrites. When the dendrites are translated vertically by an appropriate mechanism, the liquid sheet also moves upward. The liquid temperature falls and it crystallizes to a thin solid ribbon or web, bounded by the supporting dendrites. Under some conditions -- for example, an overly large initial supercooling, a rapid melt temperature excursion or possibly a high melt impurity concentration -- the smooth web/liquid interface breaks down forming an extra dendrite or "third". (If the perturbation is severe, several dendrites may form.) The formation of thirds in some of our first webs, viz. Nos. 12-1, 15-3, and 17-1 is evident in Fig. 2.

Another characteristic apparent from the figure is the tendency of the webs to widen as they grow. Typically, the initial width of the web (not including the dendrites) is 0.4 cm broadening to a little over 1 cm by the end of the run. Since the web width tends to be apparatus limited, and generally less than 1 cm for a significant proportion of these samples, we will probably deviate from our standard 1 cm x 1 cm solar cells for web evaluation. This should pose no problem for rating the relative effects of various impurities on web growth.

The webs grown so far were each boron-doped to provide a target resistivity in the 2-6 Ω cm range used in the Czochralski studies. The measured values have fallen in the 3-4 Ω cm range. So far, web widths have ranged from 0.4 cm to 0.9 cm, while web thickness may vary about $\pm$ 2 mils along the length of a given web. Depending on the growth conditions, the web thickness varied from 8 to 25 mils for the different webs illustrated in Fig. 2.

Our first attempt to test the effect of metal doping on web growth was sample DW 17-1 which contained chromium and boron as intentionally added impurities. Smooth growth was obtained over the

Fig. 2. Typical boron-doped silicon dendritic webs so far grown in our laboratory furnace.

first 28 cm length of the web at which point a temperature fluctuation produced a third dendrite. Unique about this particular event was the branched nature of the extra dendrite. This feature, never observed with boron as the sole dopant, suggests that constitutional supercooling may in fact play a role in the interface breakdown of the rapidly propagating web.

At this point preventing the intrusion of thirds is a major focus for our attention since thirds reduce the yield of usable test material. The width and flatness of the webs appear sufficient to meet the objectives of this program.

3.3 Ingot Evaluation

3.3.1 Resistivity, Defect Density, and Carbon/Oxygen Analysis

The resistivity, etch pit density and carbon/oxygen concentration are collected in Table 3 for all Czochralski ingots. The targeted resistivity for all samples except W023-00-000 was 4 ohm-cm. The normal variation found in preparing doped crystals is $\pm$ 25 percent. The tang end values shown in the table represent the position in the ingot when approximately 50 percent of the melt has been removed (50 percent pulled). Only one singly-doped ingot (W038-AL002) prepared during this reporting period fell significantly outside the targeted resistivity range; it was doped only with aluminum. A low value was chosen for the effective segregation coefficient prior to growth which explains this result.

The etch pit density in slices taken from the seed end of ingots prepared during this reporting period were nominal ranging from 0 to 1500. A significantly higher etch pit density with gross lineage was encountered in tang end slices taken from ingot W037-ZR/TI001.

Carbon and oxygen concentrations found in all ingots were nominal. The carbon concentration typically detected in undoped ingots produced in the crystal pulling furnaces used on this program varies

Table 3. Ingot Analysis (Resistivity, Dislocation Density, and Carbon/Oxygen Concentration)

Ingot Identification	Resistivity Seed/ Resistivity Tang (ohm-cm)	Etch Pit Density of Seed End (Etch Pits/cm ²)	Carbon Conc./ Oxygen Conc. (10 ¹⁶ atoms/cm ³)
W-001-00-000	2.8/3.2	0	7.5/94
W-002-00-000	3.0/4.0	0	8.3/57
W-003-00-000	3.1/3.6	0	12.5/94
W-004-Cr-001	4.6/4.3	500	6/88
W-005-Mn-001	3.6/3.1	600	6/126
W-006-Ni-001	4.1/3.5	300	7.3/96
W-007-Cu-001	5.2/3.7	0	7.5/49
W-008-Ti-001	4.2/3.1	1800	14/150
W-009-V-001	4.1/3.4	0	18/100
W-010-Ni-002	3.9/3.4	200	12.5/140
W-011-Zr-001	4.4/3.6	400	8/87
W-012-Cr-002	4.1/3.9	400	5/170
W-013-Mn-002	4.3/3.2	200	5/160
W-014-00-000	4.3/3.2	1000	5/160
W-015-Zn-001	3.9/3.6	500	9/100
W-016-Fe-001	4.6/5.2	0	5.5/95
W-017-Cu-002	3.8/3.6	2200	5.5/78
W-018-Fe-002	5.9/5.0	500	7/80
W-019-Cu-003	3.8/3.5	200	4.2/96
W-020-00-000	4.0/2.9	0	3.2/160
W-021-Mg-001	3.8/3.4	500	8/110
W-022-00-000	4.1/3.2	500	7/140
W-023-00-000	0.15/0.15	2500	N/A
W-024-Mg-002	3.8/3.6	3750	8.8/46
W-025-00-000	5.1/4.7	2000	9.6/96
W-026-Mn-003	4.5/3.9	1000	9.4/140
W-027-Mn/Cu-001	8.4/6.3	1250	<2.5/110
W-028-Al-001	2.9/2.4	750	17/80
W-029-Cr-003	5.2/4.6	0	2.5/150
W-030-Cr/Cu-001	7.3/6.9	500	3.8/140
W-031-Cr/Mn-001	8.8/4.7	1250	11/110

Table 3. Ingot Analysis (Resistivity, Dislocation Density, and Carbon/Oxygen Concentration) (cont.)

<u>Ingot Identification</u>	<u>Resistivity Seed/ Resistivity Tang (ohm-cm)</u>	<u>Etch Pit Density of Seed, End (Etch Pits/cm²)</u>	<u>Carbon Conc./ Oxygen Conc. (10¹⁶ atoms/cm³)</u>
W-032-Mg-003	4.5/4.1	0	9/90
W-033-Ti-002	4.5/4.2	0	10/100
W-034-00-000	4.4/4.2	0	2.9/130
W-035-V-002	4.4/3.9	1500	5.1/135
W-036-Zr-002	4.4/4.1	500	5.5/137
W-037-Zr/Ti-001	5.1/4.5	500	8.3/190
W-038-Al-002	2.2/1.55	0	63/143

Table 4. Ingot Analysis (Intentionally Added Impurity)

Ingot Identification	Target Conc. (10^{15} atoms/cm ³)	Calc. Conc. (10^{15} atoms/cm ³)	Mass Spec Conc. (10^{15} atoms/cm ³)	NAA Conc. (10^{15} atoms/cm ³)
W-001-00-000	-	-	-	-
W-002-00-000	-	-	-	-
W-003-00-000	-	-	-	-
W-004-Cr-001	1	0.82	0.95	<4 ¹
W-005-Mn-001	1	0.82	1.35	0.6
W-006-Ni-001	1	0.17 ²	<0.5	NA
W-007-Cu-001	1	1.18	1.0	1.65
W-008-Ti-001	1	0.82	0.36	NA
W-009-V-001	1	1.19	0.31	NA
W-010-Ni-002	1	0.56	4.0	NA
W-011-Zr-001	1	2.67 ³	0.45	Incomp
W-012-Cr-002	0.2	0.18	<0.5	NA
W-013-Mn-002	0.2	0.15	<0.5	NA
W-014-00-000	-	-	ND	-
W-015-Zn-001	1	<0.0006 ⁴	<0.5	NA
W-016-Fe-001	1	0.75	<3.0	NA
W-017-Cu-002	10	7	40	25
W-018-Fe-002	2	1.6	<3	NA
W-019-Cu-003	0.2	0.53	<0.5	-
W-020-00-000	-	-	-	-
W-021-Mg-001	1.0	0.59 ⁵	<0.5	NA
W-022-00-000	-	-	-	-
W-023-00-000	-	-	-	-
W-024-Mg-002	10	7.3	<0.5 ⁶	NA
W-025-00-000	-	-	-	-
W-026-Mn-003	0.01	0.0089	<0.5	NA
W-027-Mn/Cu-001	1/1	0.73/0.64	1/1.4	1.1/2.2
W-028-Al-001	1	-	26	NA
W-029-Cr-003	0.01	0.0083	<5 x 10	NA
W-030-Cr/Cu-001	1/1	0.82/0.75	1/1.4	0.5/2.5

Table 4. Ingot Analysis (Intentionally Added Impurity) (cont.)

<u>Ingot Identification</u>	<u>Target Conc. (10^{15} atoms/cm)</u>	<u>Calc. Conc. (10^{15} atoms/cm)</u>	<u>Mass Spec Conc. (10^{15} atoms/cm)</u>	<u>NAA Conc. (10^{15} atoms/cm)</u>
W-031-Cr/Mn-001	1/1	0.85/0.82	1/2.5	-
W-032-Mg-003	1	0.24	0.3	NA
W-033-Ti-002	0.01	0.011	<0.3	NA
W-034-00-000	-	-	-	-
W-035-V-002	0.01	0.013	<0.2	NA
W-036-Zr-002	2	1.74	<0.45	NA
W-037-Zr/Ti-001	1/1	0.95/0.97	<0.45/0.35	NA
W-038-Al-002	3.4	1.84	34	NA

1. NAA indicates impurity concentration of less than 4×10^{15} atoms/cm³ which represented the limit of detection.
2. Nickel powder used as dopant. Dopant partially lost during furnace evacuation. Figure of 0.17×10^{15} atoms/cm² indicative of actual impurity concentration.
3. The high Zr-concentration is obviously in error since it greatly exceeds the amount added to the melt.
4. Zinc loss from the melt by evaporation was essentially complete.
5. Modest loss of magnesium occurred due to evaporation.
6. No Mg detected indicating much smaller segregation coefficient than suspected. Best estimate is a concentration of 1×10^{13} atoms/cm³ for W-021 and 1×10^{14} atoms/cm³ for W-024.

from 2.5×10^{16} atoms/cm³ to 25×10^{16} atoms/cm³ while oxygen concentrations will normally range from 50×10^{16} atoms/cm³ to 150×10^{16} atoms/cm³.

No significant deviations from the above ranges were observed. Neither oxygen nor carbon concentration was sufficiently great to produce crystal growth problems, however, impurity complexing with oxygen may affect device performance.

3.3.2 Impurity Analysis

The primary objective of the ingot analysis effort is to establish what concentration of the intentionally introduced impurities, Fig. 1, is actually introduced into the ingots.

The spark source mass spectrometer has been the primary tool used in ingot analysis. This has been supplemented by neutron activation analysis for those samples which may readily be analyzed by this technique, and by analysis of melt samples at the completion of crystal growth to establish the impurity concentration of the melt. The latter technique was used primarily to ascertain whether significant impurity loss from the melt occurred during growth. Due to the potential non-uniform impurity incorporation within slices taken from a doped ingot, and the inherent microscopic sampling of the mass spectrometer, multiple samples were taken from all first generation ingots to determine the uncertainty involved with the measurements.

The analytical results for the first 38 ingots prepared for solar cell evaluation are compiled in Table 4. In the table the target impurity concentration is derived from the amount of impurity added to the melt and the segregation coefficients shown in Table 1. The calculated impurity concentration is based on the measured melt impurity concentration at the completion of crystal growth (corrected for the volume change of the melt) and the segregation coefficients of Table 1. The next two columns report the result of mass spectrographic analysis and, where applicable, neutron activation analysis. For brevity, only the first three digits of the ingot designator are used in discussing these results.

During the reporting period, a heavily magnesium-doped ingot was evaluated and magnesium detected at 0.3×10^{15} atoms/cm³. The amount determined by mass spectrographic analysis was consistent with the value calculated from melt analysis data. These results indicate the proper effective segregation coefficient for magnesium is approximately 10^{-5} . Lightly-doped (10^{13} atoms/cm³) ingots containing titanium (W033) and vanadium (W035) were also prepared. These impurity concentrations are below the detection limit of mass spec and neutron activation analysis.

A heavily doped ingot using zirconium as the impurity was prepared with a targeted zirconium concentration of 2×10^{15} atoms/cm³. Melt analysis was consistent with the amount of zirconium added to the melt; however, zirconium was not detected by the mass spec below the 0.45×10^{15} atoms/cm³ concentration which we believe to be the lowest reliable limit of detection for zirconium. Based on all evaluation of zirconium doped ingots, the effective segregation coefficient for this element is significantly less than the 10^{-5} value which appears in the literature.

Two aluminum doped ingots have been prepared and analyzed. For both ingots the aluminum concentration determined by mass spectrographic analysis has been approximately an order-of-magnitude higher than the electrically active concentration based on resistivity data. This type of disparity has been observed in the past, and based on preliminary measurements of oxygen in these ingots, is ascribed to the formation of non-electrically active aluminum oxide complexes.

We note that several melt analysis samples were collected in the preparation of ingots W036 and W037 due to the large discrepancy observed in the analysis of the melt from ingot W011-ZR001. The results are quite consistent with predicted melt concentrations and indicate that melt sampling is applicable for all impurities under investigation.

As with the previous quarterly report,² a best estimate is made of the impurity concentration in each ingot, Table 5. These values which are subject to updating as more data are collected should be used in evaluating results. Changes made for the first thirty-one ingots are as follows:

- Zirconium concentration reduced for W011-ZR001 from 0.45×10^{15} to $< 0.25 \times 10^{15}$,
- Iron concentration for W016-FE001 reduced from $< 3 \times 10^{15}$ to $< 1.5 \times 10^{15}$,
- Magnesium concentration for W021-MG001 changed from $< 0.5 \times 10^{15}$ to 0.003×10^{15} ,
- Magnesium concentration for W024-MG002 changed from $< 0.5 \times 10^{15}$ to 0.03×10^{15} and
- Aluminum concentration for ingot W028-AL001 established at 26×10^{15} .

All of the above concentrations are in atoms/cm³. The aluminum concentrations for ingots W028 and W038 are based on mass spectrographic analysis. The concentration of electrically active aluminum is approximately one order-of-magnitude smaller for both ingots.

3.4 Wafer Evaluation

3.4.1 Spreading Resistance

The radial resistivity data for twelve ingots are listed in Table 6. In general, the resistivity variation in the metal-doped ingots is somewhat larger than the baseline or audit ingots. The Cr/Mn doped ingot in particular shows pronounced resistivity striations, possibly associated with non-uniform incorporation of the two dopants. The high resistivity at the seed end of ingot W032-MG is yet unexplained.

3.4.2 Microstructural Evaluation

As our past work has indicated, etch pit density data from individual wafers confirms within the scatter of the method the results obtained for the bulk ingot. EPD generally falls in the "zero" to

Table 5. Best Estimate of Impurity Concentration

<u>Ingot Identification</u>	<u>Best Estimate of Impurity Conc. (10¹⁵ atoms/cm)</u>	<u>Ingot Identification</u>	<u>Best Estimate of Impurity Conc. (10¹⁵ atoms/cm)</u>
W-001-00-000	--	W-020-00-000	10
W-002-00-000	--	W-021-Mg-001	0.003
W-003-00-000	--	W-022-00-000	--
W-004-Cr-001	0.95	W-023-00-000	--
W-005-Mn-001	1.0	W-024-Mg-002	0.03
W-006-Ni-001	0.5	W-025-00-000	--
W-007-Cu-001	1.0	W-026-Mn-003	0.01
W-008-Ti-001	0.36	W-027-Mn/Cu-001	1.0/1.0
W-009-V-001	0.31	W-028-Al-001	26
W-010-Ni-002	4.0	W-029-Cr-003	0.01
W-011-Zr-001	<0.25	W-030-Cr/Cu-001	1.0/1.0
W-012-Cr-002	0.2	W-031-Cr/Mn-001	1.0/1.0
W-013-Mn-002	0.2	W-032-Mg-003	0.3
W-014-00-000	--	W-033-Ti-002	0.0035
W-015-Zn-001	.0001 - .1	W-034-00-000	--
W-016-Fe-001	<1.5	W-035-V-002	0.003
W-017-Cu-002	10	W-036-Zr-002	<0.45
W-018-Fe-002	<3	W-037-Zr/Ti-001	<0.25/0.36
W-019-Cu-003	0.1 - 0.5	W-038-Al-002	34

Table 6. Radial Resistivity Variations for Metal Impurity Doped and Audit Ingots

<u>Ingot No.</u>	<u>Wafer Location</u>	<u>Average Resistivity ohm-cm</u>	<u>Fractional Standard Deviation</u>
W016-FE	seed	4.525	6.081×10^{-2}
	tang	4.599	6.419×10^{-2}
W021-MG	seed	3.673	4.529×10^{-2}
	tang	3.344	2.509×10^{-2}
W025	seed	4.402	5.592×10^{-2}
	tang	4.442	2.674×10^{-2}
W026-MN	seed	3.903	3.359×10^{-2}
	tang	3.714	3.032×10^{-2}
W027-MN/CU	seed	5.713	9.077×10^{-2}
	tang	5.596	4.444×10^{-2}
W029-CR	seed	5.884	1.302×10^{-1}
	tang	5.244	7.252×10^{-2}
W030-CR/CU	seed	6.845	5.163×10^{-2}
	tang	6.753	6.516×10^{-2}
W031-CR/MN	seed	6.921	2.065×10^{-1}
	tang	5.438	7.443×10^{-2}
W032-MG	seed	10.40	7.095×10^{-2}
	tang	6.619	1.119×10^{-1}
W033-TI	seed	3.617	4.973×10^{-2}
	tang	6.725	5.643×10^{-2}
W035-V	seed	3.624	6.101×10^{-2}
	tang	4.093	4.453×10^{-2}

3000 cm⁻². Since the range of etch pit densities is rather narrow, we expect, and so far have found, little effect of the dislocations per se on cell performance or electrical characteristics. The effect of defects like dislocations is probably an indirect one, e.g., acting as precipitate nucleation sites. This conclusion is consistent with the x-ray topographic results described below.

Initially, we utilized x-ray topography as a quality control technique to assure that no gross deviation in crystal perfection had occurred in any of the ingots. We routinely survey at least one wafer from each ingot. The overall features of the topographs, as noted before,² are consistent with the general level of perfection deduced from EPD measurements.

However, when the topographs are examined in greater detail, other features emerge. For example, the variation in fine structure from ingot to ingot is clearly apparent in Fig. 3, which illustrates topographs from ingots W008-Ti, W017-Cu, W010-Ni and baseline ingot W002. Each wafer was in the as-grown condition when the topographs were run. The baseline ingot exhibits many long straight dislocation segments, sharply imaged and with no evidence for decoration. In contrast, the wafers containing the fast diffusing impurities Cu and Ni show considerable decoration of the dislocation lines by precipitates. Precipitation is apparently so heavy in the Cu-doped sample that the normal x-ray extinction conditions are modified to the point that the dislocation lines themselves virtually disappear, only the strain fields of the precipitates being imaged. For the Ti-doped sample the effect of precipitation on dislocations is somewhat less apparent than for Cu and Ni. The dislocations generally show a fringed contrast in the presence of Ti with individual precipitate spots difficult to discern.

On the basis of these results, it seems that a shift in emphasis for the metallurgical evaluation is now warranted. Attention will be focused in the future on evaluating via topography and etching

Baseline

Ti $3.6 \times 10^{14} \text{ cm}^{-3}$

0.05 cm

Cu $1 \times 10^{16} \text{ cm}^{-3}$

Ni $4 \times 10^{15} \text{ cm}^{-3}$

Fig. 3. (111) X-ray transmission topographs of wafers from ingots doped with different metals; strings of dark spots are precipitates decorating the dislocations

the effect of specific impurities on wafer structure at various processing stages. For example, we plan to perform diffusions on some of the samples illustrated in Fig. 3 to explore more carefully the effects of gettering (see Section 3.4.4).

3.4.3 Recombination Lifetimes Measured by Photoconductive Decay (PCD)

A rigorous mathematical analysis required for converting apparent (measured) lifetime values, τ_r' to true bulk lifetimes, τ_r , was developed in the previous quarterly report.² In the period since that report minor refinements in the measurement technique were completed and the measuring equipment was also modified to further increase the accuracy and range of the lifetime measurements. Lifetime measurements were extended to 14 additional ingots and the correlation between τ_r and impurity concentration was briefly explored. Finally, PCD measurements were used to deduce the effects of heat treatment on the electrical activity of metallic impurities.

3.4.3.1 PCD Equipment Modifications

The thin silicon optical filter used to skew the spectral output from the pulsed LED was removed from the system. Measurements performed on thick specimens indicated that the filter did not materially shift the spectrum. The instrumentation for monitoring the test current, I_t , was replaced by an Ayrton-Perry Shunt type metering system; the maximum possible current was increased to 100 ma. This modification is necessary to maintain a reasonable signal-to-noise ratio when measuring low values of τ_r . To elucidate the magnitude of the problem, an analysis of the differential signal observed on the oscilloscope has been made and is discussed below.

The concentration of minority carriers within the bulk silicon for the case of uniform optical generation rate G_L and negligible electric field, E , or surface recombination velocity, v , is described by the differential equation,

$$\frac{d(\delta n)}{dt} = G_L - \frac{\delta n}{\tau_r} \quad (1)$$

where δn is the deviation from thermal equilibrium ($n - n_p$). The solution for the case when $\delta n = 0$ at $t = 0$ is,

$$\delta n(t) = G_L \tau_r \left[1 - e^{-t/\tau_r} \right] \quad (2)$$

where t is the time following the application of the light pulse. The initial differential test signal, δV , observed on the oscilloscope when the LED is turned off is equal to $I_t \delta R$. The change in specimen resistance due to the excess minority carriers is

$$\frac{\partial R}{\partial n} = \frac{\partial R}{\partial \rho} \frac{\partial \rho}{\partial n} \quad (3)$$

Using the relationships that $R = \rho l / 2\omega d$ and $\rho = 1 / e\mu_n n$, Eq. (3) may be explicitly written as,

$$\frac{\partial R}{\partial n} = - \frac{l}{2\omega d e \mu_n^2} \quad (4)$$

The peak signal voltage, δV , is equal to

$$\delta V = - \frac{I_t l}{2\omega d e \mu_n^2} \delta n \quad (5)$$

The value of δn obtained from Eq. (2) when substituted into Eq. (5) yields the desired expression for δV .

$$\delta V = - \frac{I_t G_L \tau_r}{e \mu_n^2} \left(\frac{l}{2\omega d} \right) \left(1 - e^{-t/\tau_r} \right) \quad (6)$$

This equation predicts that the signal voltage, δV , will decrease with low values of τ_r and resistivity, ρ . The range of pulse lengths, t , possible with the LED source is controlled by the HP 212 Pulse Generator which is variable between 0-10 μsec . For small τ_r the value of t can easily be adjusted such that $t \gg \tau_r$. Under these

conditions equilibrium is achieved between generation rate, G_L , and recombination rate, $\delta n/\tau_r$. Equation (6) can be corrected, under these equilibrium conditions, for surface recombination by using τ_r' determined by Eq. (7),³

$$\tau_r' = \tau_r \left\{ 1 - \frac{(sL/Dd) \sinh(d/L)}{[1/L \sinh(d/L) + (s/D) \cosh(d/L)]} \right\} \quad (7)$$

where D is the diffusivity and L the diffusion length for the minority carriers. Equation (7) is plotted in Fig. 4 for the parameters appropriate in this project. Equations (6) and (7) when used for $t \gg \tau_r$ also provide a method for measuring the recombination lifetime. The technique has the disadvantage that G_L must be known, but the advantage that no transient measurements are required.

The signal voltage δV can be increased to compensate for low values of τ_r and ρ by increasing either I_t or G_L . Increasing G_L is difficult to accomplish since increasing the pulse current through the LED increases the operating temperature thus reducing conversion efficiency. Increasing the LED efficiency, e.g., by improving the material lifetime, will increase the infrared radiation decay time. A suitable alternative is to increase I_t (a change which must be performed with due consideration for the maximum electrical field sustainable in the silicon specimen). A pictorial-schematic diagram for the modified PCD measurement apparatus is shown in Fig. 5.

We attempted to obtain correction factors for the decay time of the LED source by measuring the system response time as the effective lifetime, τ_r' , of the specimen was reduced toward zero. The effective lifetime of the specimen was reduced by chemically thinning the sample, thereby allowing the surface recombination velocity to dominate and $\tau' = d^2/D_n \xi_o^2$. For small values of sd/D_n , the value of $\xi_o = \sqrt{sd/D_n}$ and

$$\tau' = d/s \quad (8)$$

Fig. 4. Photoconductive lifetime correction factors for various values of surface recombination velocity on a 0.254 mm thick p-type silicon specimen with a resistivity of 4 ohm-cm.

Fig. 5. Schematic diagram of photoconductive decay lifetime measurement apparatus.

As an example of the method; $\tau' = 0.127 \mu\text{sec}$ for a specimen with a thickness of 0.0254 mm and a typical value of surface recombination velocity, $s = 1 \times 10^4$. Because the sample fractured before it was etched sufficiently thin, the results of this initial experiment were inconclusive. The experiment will be repeated during the next period.

3.4.3.2 Surface Recombination Velocity Measurements

The photoconductivity decay lifetime correction factor is a function of the surface recombination velocity, which is in turn dependent on the surface treatment applied to the silicon test specimen.² The standard surface treatment used so far in our studies is a five-minute etch in a $\text{HNO}_3\text{:HF}$ (20:1) solution. The surface recombination velocity for this standard treatment was determined to be $1 \pm 0.5 \times 10^4 \text{ cm/sec.}^2$ A series of experiments were performed this quarter to determine the reproducibility and accuracy of this value for s_o . A second objective was to find a surface treatment which would lower the recombination velocity, thereby making the measurements less sensitive to variations in s_o . The method outlined in Section 3.4.3.3 of the Second Quarterly Report was used to measure s_o .

The results of these experiments are presented in Table 7, while the composition and etch rate for each etch solution tested appears in Table 8.

The surface recombination velocity for the Standard Etch is now more accurately determined to be $1.34 \pm 0.066 \times 10^4 \text{ cm/sec.}$ The tolerance is the probable error calculated as 0.6745σ . The lowest surface recombination velocity was achieved with CP-4A and is equal to $8.99 \pm 1.40 \times 10^3 \text{ cm/sec.}$ The error in assuming an infinite value for s_o with a sand blasted surface is readily apparent.

A surface treatment of 10 seconds in CP-4A will be used henceforth in all photoconductivity decay lifetime measurements with the appropriate value of s_o . This decision was made in spite of the improved reproducibility obtained with the standard solution, since lower correction factors are considered more desirable.

Table 7. Surface Recombination Velocity Measurements

$$s_o = \sqrt{\frac{D_n}{\tau_r}} \tan \sqrt{\frac{d^2}{\tau_r D_n}}$$

TI Lopex, 15-25 Ω -cm, $D_n = 47.82$, $\tau_r = 576$ μ sec

Surface Treatment	s_o (cm/sec)	$\sigma(n)$
Std. Etch	1.34×10^4	9.82×10^2 (5)
CP-4 Etch	1.539×10^4	-----
CP-4A Etch	8.99×10^3	2.08×10^3 (3)
White Etch	1.080×10^4	2.05×10^3 (2)
CP-6 Etch	1.066×10^4	-----
CP-8 Etch	1.423×10^4	-----
Polish Etch	1.293×10^4	-----
H ₂ Anneal, 1 hr @ 500°C	1.553×10^4	-----
O ₂ Anneal, 1 hr @ 500°C	1.095×10^4	1.98×10^3 (2)
Sandblast	1.741×10^4	-----

Table 8. Etch Solutions

Etch Solution	Etch Composition (ml)				Etch Rate ($\mu\text{m}/\text{min}$)
	HF	HNO ₃	CH ₃ COOH	Addition	
Std. Etch	1	20	--	--	1.60
CP-4	15	25	15	0.3 gr Br ₂	0.15
CP-4A	15	25	15	--	10.0
White Etch	20	80	--	--	1.0
CP-6	2	10	--	--	15.0
CP-8	6	10	--	--	52.0
Polish Etch	16.2	48.7	35.1	0.08 gr I	11.4

3.4.3.3 Lifetime Measurements on Czochralski Wafers

In Table 9 are collected the most recent lifetime data for ingots W001 through W035 (multiply-doped). The data up to W020 were previously reported,² but have since been updated to reflect measurements taken on additional samples. The sample size studied has been added to Table 8 to assist in judging the accuracy of the data. The number of significant figures for each entry has also been reduced to correspond to the standard deviation.

3.4.3.4 Correlation of Lifetime and Impurity Concentration

The recombination rate ($1/\tau$) is directly proportional to the density of recombination centers in the bulk silicon. The relationship can be expressed as

$$\tau = \frac{1}{\sigma v_{th} N} \quad (9)$$

where N is the density of electrically active recombination centers, v_{th} is the thermal velocity of the minority carriers, and σ is the capture cross-section of the centers. The capture cross-section for

Table 9. Bulk Lifetime, τ_r , Corrected PCD Measurements

Lot No.	Impurity Type	Concentration (cm ⁻³) Note (1)	Lifetime (As-Grown)		Lifetime (Diffused)	
			τ (μ sec)	σ Note (2)	τ (μ sec)	σ Note (2)
W001	Baseline	N/A	7.1	1.2(4)	6.9	0.5(3)
W002	Baseline	N/A	6.3	0.6(2)	8.6	1.6(2)
W003	Baseline	N/A	11.6	2.3(2)	8.4	0.7(2)
W004	CR001	0.95×10^{15}	0.35	0.2(3)	1.1	0.2(4)
W005	MN001	1.35×10^{15}	1.8(3)	--	<0.3	0.0(2)
W006	NI001	1.7×10^{14}	11.3	2.7(2)	7.7	0.2(2)
W007	CU001	1.0×10^{15}	6.9	0.9(4)	6.6	3.2(3)
W008	TI001	3.6×10^{14}	2.0	0.5(3)	0.6	0.1(4)
W009	V001	3.1×10^{14}	0.4	0.1(3)	1.9(3)	2.2(3)
W010	NI002	4×10^{15}	6.8	2.6(3)	9.3	0.4(3)
W011	ZR001	4.5×10^{14}	2.6	0.2(2)	1.9	0.2(2)
W012	CR002	$1.8 \times 10^{14*}$	<0.4	0.1(2)	4.9	1.0(5)
W013	MN002	$1.5 \times 10^{14*}$	1.2(3)	1.0(3)	10.5(3)	4.7(5)
W014	Audit	N/A	7.5	0.4(2)	8.3	0.1(2)
W015	ZN001	$1 \times 10^{15*}$	7.2	0.6(2)	5.3	0.4(4)
W016	FE001	$1 \times 10^{15*}$	0.5	0.0(2)	3.8	1.5(4)
W017	CU002	4×10^{16}	8.8	0.1(2)	7.1	0.7(2)
W018	FE002	$2 \times 10^{15*}$	8.1	5.5(2)	0.8	0.1(2)
W019	CU003	$2 \times 10^{14*}$	4.3	3.3(2)	5.7	0.5(2)
W020	Audit	N/A	7.0	1.7(2)	7.3	1.4(2)
W021	MG001	$1 \times 10^{15*}$	8.2	0.8(2)	7.7	0.1(2)
W022	Audit	N/A	7.8	1.0(2)	9.1	0.2(2)
W023	Audit	N/A	Note (4)		Note (4)	
W024	MG002	1×10^{13}	7.5	0.9(2)	10.2	0.9(2)
W025	Audit	N/A	7.6	0.0(2)	12.7	1.0(2)
W026	MN003	$1 \times 10^{13*}$	5.1	0.2(2)	9.3	0.0(2)
W027	MN/CU001	$1/1.4 \times 10^{15}$	22.3(3)	2.3(2)	0.5	0.2(2)
W028	AL001	$1 \times 10^{15*}$	2.9	0.2(2)	1.9	0.0(2)
W029	CR003	8.3×10^{12}	1.1	0.6(2)	6.2	0.4(4)

Table 9 (con't)

Lot No.	Impurity Type	Concentration (cm ⁻³) Note (1)	Lifetime (As-Grown)		Lifetime (Diffused)	
			τ (μ sec)	σ Note (2)	τ (μ sec)	σ Note (2)
W030	CR/CU001	1/1.4 x 10 ¹⁵	<0.3	0.0(2)	0.4	0.1(2)
W031	CR/MN001	1/2.5 x 10 ¹⁵	Note (5)	--	<0.3	0.0
W032	MG003	3 x 10 ¹⁴	7.2	1.1	7.1	1.1
W033	TI002	1 x 10 ¹³ *	3.1	0.0	2.9	0.1
W034	Audit	N/A	--	--	--	--
W035	V002	1 x 10 ¹³ *	1.2	0.0	1.5	0.2

Note 1. Impurity concentration measured by Mass Spec or crucible remains unless marked by * to indicate a target value. For best estimate of impurity concentration, see Table 5.

Note 2. Sample size shown in parenthesis.

Note 3. Lifetime measurements subject to large errors due to extreme shallow trap density.

Note 4. Lifetime measurements not practical due to low resistivity.

Note 5. Lifetime measurements not possible due to very low lifetime.

gold centers⁴ has been measured as $\sim 5 \times 10^{-15} \text{ cm}^2$. Generally, the capture cross-section varies only slightly (± 10 times) for various impurities. The thermal velocity, v_{th} is equal to $\sqrt{3kT/m}$ and is approximately 10^7 cm/sec at room temperature. The density of centers N can be measured directly by thermally stimulated current measurements. Equation (9) assumes that there is only a single energy level per electrically active impurity atom. It is important to realize that the quantity N is generally less than the metallurgically measured impurity concentration since not all the impurities are electrically active due to precipitation, complexing, etc. Nevertheless, we tested for an inverse relation between bulk lifetime and metallurgical impurity concentration, picking chromium as an example. The expected relationship, Fig. 6, holds surprisingly well for the as-grown silicon material. Assuming that the capture cross-section for chromium is also 5×10^{-15} , the density of centers necessary for a recombination lifetime of $1 \text{ } \mu\text{sec}$ is 2×10^{13} . This value is in close agreement with the metallurgical concentration of 1.2×10^{13} shown on the curve. In this case, apparently all the chromium impurity atoms are electrically active. While these calculations are speculative considering the assumed value of σ , the results seemed important enough to include here as preliminary findings. The same inverse linear relationship does not hold for the diffused material since the density of electrically active centers has changed due to thermal processing, Fig. 6, although τ does diminish with increasing impurity content. Apparently, precipitation and/or gettering which occur during the phosphorus diffusion cycle has influenced the behavior of the diffused material. Experiments to be presented in Section 3.4.4 suggest that both gettering and precipitation (or complexing) participate in the deactivation of chromium recombination centers.

3.4.4 Gettering and Precipitation Studies

Since solar cell processing can have significant effects on recombination lifetime and hence cell performance, we have carried out several kinds of studies directed toward understanding the effects of

Fig. 6. Correlation between recombination lifetime and metallurgical concentration of chromium impurity.

heat treatment and phosphorus diffusion on the electrical activity and distribution of metal impurities in silicon. In this way we hope to unravel the relative effects of such mechanisms as gettering and precipitation on cell behavior. We report below the initial results of continuing studies in two areas, lifetime variation with processing and impurity redistribution during diffusion.

3.4.4.1 Effect of Heat Treatment and Diffusion on Wafer Lifetime

During this program the recombination lifetime has been measured on wafers in both the as-grown conditions and after the phosphorus diffusion cycle. These results, Table 9, show that the lifetime changes appreciably during processing. The lifetime for some impurity types increases with processing while for others it decreases. The changes in lifetime may be assumed to occur during one or both of the two high temperature treatments employed during cell processing: (1) phosphorus diffusion, and (2) sintering of the contacts. Mechanisms responsible for lifetime changes include gettering, precipitation, complexing (chemical combination with other impurities such as oxygen), dissolution of impurities and structural damage to the original crystalline material. A knowledge of which mechanism predominates for each impurity element would be extremely valuable for predicting the changes to be expected from multiple doping.

Photoconductive decay lifetime measurement techniques are especially useful for determining the responsible mechanisms since specimens can be prepared without any heat treatment, but moderate heat treatment can be applied to the finished test piece. Table 10 contains a summary of data from samples subjected to various heat treatments. Annealing for 5 hrs at 550°C should stimulate considerable metal precipitation since the solid solubility of typical impurities at 550°C is extremely low, yet thermal diffusion over small distances should be possible for the faster diffusing metal atoms, viz. Table 11. The

Table 10. Effect of Heat Treatment on
Recombination Lifetime τ_r (μ sec)

Specimen Identification	As-Grown	Annealed 5 Hrs @ 550°C	Annealed, 50 Min @ 825°C	Phosphorus Diffused 50 Min @ 825°C
W001 Baseline	5.0	5.3	5.7	7.2
W004-CR001	0.4	1.0	0.7	1.1
W021-CR002	0.5	---	3.2	4.5
W010-NI002	4.1	7.4	4.5	9.4
W013-MN002	<0.3	2.1	4.1	10.5
W009-V001	<0.3	<0.3	<0.3	< 0.3
W007-CU001	5.4	8.5	3.6	4.4
W008-TI001	2.0	1.8	0.3	0.5

Impurity Elements	Diffusion Rate	Solid Solubility At Melting Temperature	Precipitation Rate	Gettering Rate	Degradation of Cell Efficiency
Cu, Ni	Fast	High	Fast	Fast	Low
Cr, Mn, Fe	Medium	Low	Slow	Slow	High
Al	Slow	High	Negligible	Very Slow	Medium
Ti, V	Medium-Slow	Low	Slow	Slow	High

Table 11

Qualitative Behavior of Various Metal Impurities in Silicon

lifetime data suggest that precipitation occurs at 550°C for all the impurities except titanium and vanadium. Qualitatively, annealing at 825°C may be expected to precipitate the slower diffusing elements while possibly redissolving some precipitates formed by the fast diffusing elements. A hypothesis which might explain the titanium and copper data is that the impurities themselves are relatively innocuous but that complexes formed at high temperature, e.g., TiO_2 and CuO , produce energy levels near the intrinsic fermi level. The phosphorus diffusion cycle should produce both gettering and precipitation. The difference between the data in columns 4 and 5 of Table 10 may be attributed to the additional gettering effect. Gettering is clearly indicated for most of the impurities. The data for copper and titanium are exceptions to the trend and may indicate dissolution of the original precipitates and/or complex formation.

Some of the apparent anomalies in the data of Table 10 showed up in our first series of lifetime/heat treatment studies. The data in the table verify those first results indicating that the results are valid but that we must seek better models to explain these trends.

3.4.4.2 Auger Analysis of Impurity Redistribution During Diffusion

To assess the extent of metal atom gettering during the phosphorus diffusion cycle, the impurity profiles of several diffused wafers have been evaluated by Auger spectroscopy. The analysis was performed by sputtering through the phosphorus glass source ($\sim 360 \text{ \AA}$) and the n^+ layer ($\sim 0.25 \text{ }\mu\text{m}$) formed on each wafer during diffusion. The peak-to-peak heights of the Auger signals were measured as a function of depth on a Ni, a Cu, and a Cr-doped wafer. The data were collected and plotted together in Fig. 7. Since the phosphorus, silicon, and oxygen profiles are similar for all the samples, a composite plot for these elements is shown in the figure.

Fig. 7. Auger depth profiles of phosphorus diffused wafers showing pile-up of impurities in the n⁺ region.

The data points for the metal impurities are from ingots W010-NI, W004-CR, and W017-CU. The relative detectability of Cr, Ni, and Cu in the same matrix differs by less than 50% for the Auger method. Therefore, the data points can be compared on a semiquantitative basis. The salient features of the data can be summarized as follows. The phosphorus and metal signals peak strongly near the glass-silicon interface indicating the gettered impurities have piled up in the n^+ region rather than in the phosphorus glass. This is consistent with gettering profiles measured by Rutherford back scattering.⁵ The Cu signal persists to depths where the Ni and Cr signals have disappeared. This might be expected since the Cu-doped sample contained almost an order of magnitude more metal than Cr and Ni doped samples. The Ni signal at the glass/ n^+ layer interface is much stronger than that of Cu or Cr perhaps indicating more efficient gettering. The oxygen level in the Cr-doped samples does not drop to zero in the n^+ -layer, suggesting the possible chemical association of oxygen with Cr.

While the gettering and precipitation studies are somewhat preliminary, a qualitative understanding of the trends is suggested in Table 11. The faster diffusing impurities which are readily gettered or electrically deactivated by precipitation (viz, Fig. 2) have little effect on cell performance while those slower diffusing impurities which resist gettering/precipitation remain electrically active and tend to degrade cell efficiency. The relative interstitial and substitutional solubilities of the impurities will also influence the degree of electrical activity.

3.5 Solar Cell Characterization

Since the last report we have completed the solar cell evaluation of wafers from six ingots, three doubly-doped and three multiply-doped. The complete set of I-V curve parameters are compiled for each ingot in the Appendix, Section 7.

3.5.1 Doubly-Doped Ingots

Figure 8, updated with information from lightly-doped ingots W021-MG001, W033-TI002, and W035-V002, depicts the general trends in solar cell performance with impurity content. The additional data reinforce previous conclusions² regarding the extremely harmful effects of Ti and V on cell performance. Ingots containing nominally 10^{13} atoms cm^{-3} (probably less in reality) Ti produce cells only 83% as efficient as the baseline material; a similar V level produces cells only 75% as efficient as the baseline. As before, reducing the metal content of the ingots has improved cell performance, however.

3.5.2 Multiply-Doped Ingots

The data from the first complete set of multiply-doped ingots, W027-CU/MN001, W030-CR/CU001, and W003-CR/MN001, are collected in summary form below.

The relative cell efficiencies (η doped ingot/ η baseline) for W027 and W030 are both about 0.69. These values are very close to the relative efficiencies of ingots doped to the same level with Cr and Mn

Table 12. Average Solar Cell Characteristics of Multiply-Doped Ingots

Ingot ID	$V_{OC}(V)$	$I_{SC}(ma)$	V_p	I_p	I_0	n	FF	$\eta(\%)$	$\tau(\mu\text{sec})$
027 CU/MN	.5100	16.87	.4235	15.65	1.12 E-08	1.269	.7704	6.63	0.780
Base 002	.5500	22.88	.4533	21.12	2.35 E-08	1.454	.7608	9.57	4.212
030 CR/CU	.5026	16.70	.4188	15.53	3.26 E-09	1.213	.7749	6.51	0.780
Base 002	.5470	21.95	.4599	20.53	1.02 E-09	1.225	.7863	9.44	4.94
031 CR/MN	.4706	12.10	.3772	10.66	1.89 E-04	2.052	.7162	4.06	0.163
Base 002	.5500	22.30	.4652	20.93	7.49 E-10	1.171	.7939	9.74	4.875

Fig. 8

Variation in solar cell performance with metal doping
No AR coating, AM1 quartz-iodine illumination.

alone, viz. W004-CR (0.75) and W005-MN (0.71). This behavior would be predicted if each impurity acted independently, since Cu itself hardly affects cell efficiency. The relative cell efficiency of W031-CR/MN is only about 0.42 illustrating the combined effect of the two harmful impurities, i.e., they seem to act in an additive fashion.

4. CONCLUSIONS

The initial trends identified regarding impurity effects in cell performance still seem valid. Except for Cu, and perhaps Ni, reducing the metal content of silicon increases the lifetime and cell efficiency. However, for V and Ti even when the metal impurity concentration is as low as 10^{13} atoms/cm³ cell performance is still relatively poor, only 75 and 83% of the baseline values respectively.

Studies of the variation in carrier lifetime with heat treatment and the redistribution of impurities during the solar cell diffusion cycle indicate that both precipitation and gettering play key roles in the ultimate performance of the cell. For example, Cr, Cu, and Ni are gettered to some extent during diffusion and tend to pile up in the n⁺ region of the device. For Cu and Ni evidence for precipitation is strong both from x-ray topographic studies and lifetime measurements. This work will be reported in greater detail later.

5. PROGRAM STATUS

5.1 Present Status

All elements of the program are on schedule except the web studies, viz. revised program schedule, Fig. 9.

During this quarter we have:

- Completed the growth of all second generation ingots.
- Completed the growth of nearly all third generation ingots.
- Completed growth of about half the multiply-doped ingots.
- Completed the growth of baseline boron-doped web material.
- Initiated the growth of doubly-doped web material.
- Completed lifetime measurements on all ingots grown to date.
- Completed solar cell evaluation of all first and second generation ingots.
- Conducted an initial study of the effects of heat treatment and phosphorus diffusion on cell lifetimes.

5.2 Future Activity

During the next quarter we expect to:

- ~~Complete the growth of all remaining third and fourth generation~~ ingots and webs.
- Complete all outstanding ingot and web analyses.
- Complete lifetime measurements and electrical evaluation on all ingots.
- Complete cell evaluation of all ingots and webs.
- Complete growth and evaluation of multiply-doped ingots.
- Complete data analysis and synthesis.
- Recommend preliminary solar silicon definition.

Figure 9. Revised Program Schedule

6. REFERENCES

1. R. H. Hopkins, et al., First Quarterly Report, Silicon Materials Task (Part 2), JPL Contract #954331, February 20, 1976.
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3. A. Many, Y. Goldstein, and N. B. Grover, Semiconductor Surfaces, Amsterdam (1965).
4. A. E. Bakanowski and J. H. Forster, Bell System Tech. Journal 39, 87 (1960).
5. R. L. Meek and C. F. Gibbon, J. Elect. Soc. 121, 444 (1974).

7. APPENDIX

PHOTOVOLTAIC CHARACTERISTICS OF SOLAR CELLS UNDER AM1-EQUIVALENT QUARTZ-IODINE ILLUMINATION

Test Conditions: No anti-reflective coatings, cell area = 1 cm^2 and
illumination = 100 mw/cm^2 .

Key to Abbreviations:

R	--	calibrated reference devices
C	--	wafers from ingot center
T	--	wafers from tang end
S	--	wafers from seed end
E or N	--	end
*	--	item deleted from average

60427 W02IMG001 (5.9E14) W002 00 000

ID	VOC	ISC	VP	IP	IO	N	FF	EFF	TAU
1B	.5450	22.30	.4584	20.86	7.09E-10	1.214	.7869	9.56	4.550
2B	.5500	22.30	.4658	20.95	2.51E-10	1.156	.7955	9.76	5.200
3B	.5500	22.00	.4656	20.66	2.67E-10	1.161	.7950	9.62	4.940
4B	.5500	22.30	.4603	20.80	1.45E-09	1.278	.7806	9.57	5.200
1C	.5370	21.40	.4619	20.28	1.45E-11	.978	.8153	9.37	4.160
2C	.5400	22.50	.4537	21.04	8.37E-10	1.214	.7856	9.55	4.550
3C	.5400	21.40	.4774	20.57	1.43E-14	.741	.8498	9.82	5.200
4C*	.5320	21.50	.4359	19.77	1.86E-08	1.466	.7535	8.62	2.600
5C	.5370	21.80	.4507	20.37	9.53E-10	1.219	.7842	9.18	4.160
1S	.5400	22.30	.4495	20.72	3.04E-09	1.314	.7735	9.32	4.550
2S	.5400	22.00	.4479	20.40	4.68E-09	1.352	.7691	9.14	4.290
3S	.5400	22.00	.4548	20.60	5.79E-10	1.190	.7886	9.37	5.200
1T	.5400	22.40	.4522	20.90	1.34E-09	1.248	.7814	9.45	5.200
2T	.5400	22.50	.4510	20.96	1.98E-09	1.278	.7778	9.45	4.550
3T	.5400	22.40	.4522	20.90	1.34E-09	1.248	.7814	9.45	4.550
4T	.5400	22.40	.4522	20.90	1.34E-09	1.248	.7814	9.45	4.940

AVERAGES: 60427 BASELINE W002 00 000

.5488	22.23	.4625	20.82	6.68E-10	1.202	.7895	9.63	4.973
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60427 W02IMG001 (5.9E14)

.5395	22.10	.4549	20.69	1.46E-09	1.185	.7898	9.41	4.668
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PERCENT CHANGE

-1.69	-.56	-1.65	-.59	118.87	-1.45	.04	-2.24	-6.12
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60608 W033TI002 (1E13) W003 00 000

ID	VOC	ISC	VP	IP	IO	N	FF	EFF	TAU
1B	.5550	22.50	.4741	21.24	5.78E-11	1.079	.8065	10.07	5.200
2B	.5550	22.25	.4645	20.75	1.46E-09	1.290	.7806	9.64	4.550
3B	.5550	22.50	.4689	21.11	3.72E-10	1.191	.7924	9.90	4.550
4B	.5550	22.00	.4672	20.59	6.31E-10	1.229	.7878	9.62	4.940
5B	.5550	22.25	.4645	20.75	1.46E-09	1.290	.7806	9.64	4.290
1C	.5350	19.80	.4527	18.59	2.49E-10	1.131	.7946	8.42	2.210
2C	.5350	19.60	.4426	18.14	5.83E-09	1.369	.7656	8.03	1.690
3C	.5350	19.60	.4525	18.40	2.63E-10	1.135	.7941	8.33	2.210
4C	.5350	19.40	.4450	18.02	2.93E-09	1.310	.7726	8.02	1.950
5C	.5350	19.60	.4525	18.40	2.63E-10	1.135	.7941	8.33	2.340
1S	.5340	19.00	.4501	17.80	4.50E-10	1.170	.7895	8.01	2.210
2S	.5340	19.20	.4473	17.92	1.13E-09	1.234	.7815	8.01	1.950
3S	.5340	19.20	.4473	17.92	1.13E-09	1.234	.7815	8.01	2.210
1T	.5370	19.30	.4498	18.01	1.13E-09	1.240	.7815	8.10	1.950
2T	.5370	18.90	.4494	17.63	1.26E-09	1.250	.7804	7.92	1.690
3T	.5370	19.60	.4516	18.34	6.38E-10	1.198	.7867	8.28	1.950
4T	.5370	19.60	.4516	18.34	6.38E-10	1.198	.7867	8.28	2.080

AVERAGES: 60608 BASELINE W003 00 000

.5550	22.30	.4678	20.89	7.94E-10	1.216	.7896	9.77	4.706
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60608 W033TI002 (1E13)

.5354	19.40	.4494	18.12	1.33E-09	1.217	.7841	8.14	2.037
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PERCENT CHANGE

-3.53	-13.00	-3.95	-13.24	66.95	.08	-.70	-16.66	-56.72
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60609 W035V002 (1E13) W003 00 000

ID	VOC	ISC	VP	IP	IO	N	FF	EFF	TAU
1B	.5540	22.70	.4700	21.35	1.90E-10	1.146	.7978	10.03	5.460
2B	.5540	22.50	.4662	21.06	6.68E-10	1.229	.7875	9.82	5.200
3B	.5540	22.50	.4662	21.06	6.68E-10	1.229	.7875	9.82	5.200
4B	.5540	22.50	.4662	21.06	6.68E-10	1.229	.7875	9.82	5.200
1C	.5250	18.30	.4424	17.14	4.55E-10	1.153	.7891	7.58	1.300
2C*	.4890	18.20	.3588	15.11	2.33E-05	2.824	.6092	5.42	.200
3C	.5230	18.00	.4388	16.81	8.19E-10	1.190	.7838	7.38	1.040
4C	.5230	18.00	.4388	16.81	8.19E-10	1.190	.7838	7.38	1.300
1S	.5160	18.20	.4296	16.92	2.41E-09	1.253	.7739	7.27	1.300
2S	.5210	18.60	.4355	17.33	1.45E-09	1.225	.7789	7.55	1.040
3S	.5210	18.60	.4355	17.33	1.45E-09	1.225	.7789	7.55	1.300
1T	.5250	18.00	.4420	16.85	5.02E-10	1.161	.7880	7.45	1.300
2T	.5250	18.00	.4420	16.85	5.02E-10	1.161	.7880	7.45	1.300
3T	.5250	18.00	.4420	16.85	5.02E-10	1.161	.7880	7.45	1.300
4T	.5250	18.00	.4420	16.85	5.02E-10	1.161	.7880	7.45	1.300

AVERAGES: 60609 BASELINE W003 00 000

.5540 22.55 .4671 21.13 5.48E-10 1.208 .7901 9.87 5.265

60609 W035V002 (1E13)

.5229 18.17 .4389 16.97 9.42E-10 1.188 .7841 7.45 1.248

PERCENT CHANGE

-5.61 -19.42 -6.05 -19.66 71.75 -1.70 -.76 -24.53 -76.30

60423 W027CU-MN001 (1E15,1.4E15) W002 00 000

ID	VOC	ISC	VP	IP	IO	N	FF	EFF	TAU
1B	.5500	22.40	.4604	20.89	1.42E-09	1.276	.7808	9.62	4.160
2B	.5500	23.00	.4449	20.95	7.27E-08	1.670	.7370	9.32	3.900
3B	.5500	23.00	.4563	21.33	4.74E-09	1.374	.7694	9.73	4.550
4B	.5500	23.00	.4485	21.08	3.37E-08	1.575	.7472	9.45	3.900
5B	.5500	23.00	.4563	21.33	4.74E-09	1.374	.7694	9.73	4.550
1C	.5100	17.00	.4250	15.81	1.99E-09	1.229	.7750	6.72	.910
2C	.5100	17.00	.4250	15.81	1.99E-09	1.229	.7750	6.72	.780
3C	.5100	17.00	.4250	15.81	1.99E-09	1.229	.7750	6.72	.780
4C	.5100	16.70	.4278	15.60	8.02E-10	1.164	.7834	6.67	.780
5C	.5100	17.40	.4277	16.25	8.47E-10	1.165	.7832	6.95	.910
6C	.5100	16.50	.4231	15.30	3.43E-09	1.275	.7693	6.47	.650
1S	.5100	17.60	.4190	16.21	1.16E-08	1.379	.7567	6.79	.910
2S	.5100	17.40	.4277	16.25	8.47E-10	1.165	.7832	6.95	.910
1T	.5100	16.00	.4190	14.74	1.06E-08	1.379	.7567	6.17	.650
2T	.5100	16.50	.4097	14.95	9.69E-08	1.629	.7282	6.13	.520
3T	.5100	16.30	.4238	15.13	2.73E-09	1.257	.7715	6.41	.780
4T	.5100	17.00	.4293	15.91	4.81E-10	1.129	.7880	6.83	.780

AVERAGES: 60423 BASELINE W002 00 000

.5500 22.88 .4533 21.12 2.35E-08 1.454 .7608 9.57 4.212

60423 W027CU-MN001 (1E15,1.4E15)

.5100 16.87 .4235 15.65 1.12E-08 1.269 .7704 6.63 .780

PERCENT CHANGE

-7.27 -26.28 -6.57 -25.90 -52.34 -12.72 1.27 -30.75 -81.48

60510 W030CR-CU001 (1E15-1.4E15) W002 00 000

ID	VOC	ISC	VP	IP	IO	N	FF	EFF	TAU
1B	.5470	22.00	.4643	20.70	1.68E-10	1.126	.7985	9.61	4.940
2B	.5470	22.00	.4574	20.50	1.65E-09	1.283	.7793	9.38	4.940
3B	.5470	21.80	.4605	20.41	6.18E-10	1.211	.7880	9.40	4.940
4B	.5470	22.00	.4574	20.50	1.65E-09	1.283	.7793	9.38	4.940
1C	.4970	16.00	.4122	14.83	3.45E-09	1.245	.7689	6.11	.650
2C	.4930	15.60	.4043	14.35	1.22E-08	1.349	.7547	5.80	.520
3C	.5000	16.50	.4162	15.33	2.27E-09	1.217	.7735	6.38	.780
4C	.5060	17.20	.4276	16.14	2.74E-10	1.084	.7928	6.90	.910
5C	.5000	16.50	.4162	15.33	2.27E-09	1.217	.7735	6.38	.650
1S	.5100	17.40	.4297	16.29	4.40E-10	1.121	.7889	7.00	.910
2S	.5100	17.10	.4294	16.01	4.69E-10	1.127	.7882	6.87	1.040
3S	.5100	17.40	.4214	16.09	5.90E-09	1.317	.7642	6.78	.910
2T	.5000	16.50	.4150	15.31	3.20E-09	1.244	.7700	6.35	.650
3T	.5000	16.80	.4164	15.62	2.11E-09	1.210	.7743	6.50	.780

AVERAGES: 60510 BASELINE W002 00 000

.5470 21.95 .4599 20.53 1.02E-09 1.225 .7863 9.44 4.940

60510 W030CR-CU001 (1E15-1.4E15)

.5026 16.70 .4188 15.53 3.26E-09 1.213 .7749 6.51 .780

PERCENT CHANGE

-8.12 -23.92 -8.93 -24.34 218.56 -1.00 -1.45 -31.05 -84.21

60511 W031CR-MN001 (1E15-2.5E15) W002 00 000

ID	VOC	ISC	VP	IP	IO	N	FF	EFF	TAU
1B	.5500	22.30	.4603	20.80	1.45E-09	1.278	.7806	9.57	4.550
2B*	.5200	22.30	.3761	18.18	4.97E-05	3.274	.5898	6.84	.520
3B*	.5000	22.60	.3462	17.30	2.04E-04	4.076	.5302	5.99	.260
4B	.5500	22.30	.4701	21.06	5.16E-11	1.064	.8072	9.90	5.200
1C	.4650	11.50	.3831	10.62	5.40E-09	1.227	.7606	4.07	.390
2C*	.4260	12.20	.3074	9.92	2.98E-05	2.723	.5864	3.05	.130
3C	.4730	12.80	.2905	7.42	1.51E-03	8.099	.3560	2.16	.130
4C	.4650	11.50	.3881	10.71	1.09E-09	1.106	.7771	4.16	.130
5C	.4730	12.90	.3924	11.96	2.68E-09	1.182	.7694	4.69	.130
1S	.4790	12.60	.3993	11.72	1.42E-09	1.151	.7754	4.68	.130
2S*	.4450	11.70	.3261	9.70	1.59E-05	2.592	.6074	3.16	.130
3S	.4700	11.50	.3910	10.69	1.65E-09	1.147	.7730	4.18	.130
1T	.4700	12.00	.3868	11.07	6.52E-09	1.253	.7590	4.28	.130
2T	.4700	12.00	.3868	11.07	6.52E-09	1.253	.7590	4.28	.130
3T*	.4700	13.20	.3554	11.33	4.36E-06	2.255	.6491	4.03	.130
M1A2	.5730	23.60	.4792	22.00	1.68E-09	1.339	.7797	10.54	5.850
M1A4	.5730	23.60	.4822	22.08	7.14E-10	1.273	.7874	10.65	5.200
C1A1	.3950	21.40	.2588	14.77	7.41E-04	4.472	.4521	3.82	.140
C1A2	.3600	22.20	.2364	15.40	7.31E-04	4.018	.4555	3.64	.140

AVERAGES: 60511 BASELINE W002 00 000

.5500 22.30 .4652 20.93 7.49E-10 1.171 .7939 9.74 4.875

60511 W031CR-MN001 (1E15-2.5E15)

.4706 12.10 .3772 10.66 1.89E-04 2.052 .7162 4.06 .163

PERCENT CHANGE

-14.43 -45.74 -18.91 -49.09 ***** 75.28 -9.79 -58.29 -96.67