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LOW-COST SILICON SUBSTRATES BY DIRECTIONAL SOLIDIFICATION

Third Quarterly Report for September 1—November 30, 1980

By
Frederick Schmid
Chandra P. Khattak
Metin Basaran

December 1980
Date Issued

Work Performed Under Contract No. AC02-77CH00178

Crystal Systems, Inc.
Salem, Massachusetts

U.S. Department of Energy

Solar Energy

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Third Quarterly Report
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SOLAR ENERGY RESEARCH INSTITUTE
Subcontract No. XS-Ø-9171-1

by

Frederick Schmid, Chandra P. Khattak and Metin Basaran

Report Issued: December 1980

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This work was performed for the Solar Energy Research Institute, a Division of Midwest Research Institute, under Contract EG-77-C-01-4042 for the U.S. Department of Energy, Chicago Operations Office

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OBJECTIVE

The objective of this work is to identify and develop low-cost processes for fabricating large grain-size polycrystalline silicon substrates. Specifically, the studies will involve the directional solidification of silicon ingots using the Heat Exchanger Method (HEM). The investigations will examine the use of metallurgical grade silicon as the feedstock for the casting process, and will study both prior and *in situ* purifications to obtain high purity, low-cost polycrystalline silicon substrates.

ABSTRACT

Plane front solidification and refining of metallurgical grade silicon (MG-Si) by the Heat Exchanger Method (HEM) were examined experimentally and theoretically.

Better crystallinity was obtained from the MG-Si if melt turbulence occurs during solidification. This was also verified by theoretical analyses.

Experimental measurements indicate that vacuum purification of MG-Si can be improved by increasing the holding time under vacuum, by using silica powder and/or increasing its amount.

It has been shown that simple directional solidification by HEM of commercially available MG-Si can produce material for 7.2% conversion efficiency solar cells.

LOW-COST SILICON SUBSTRATES BY DIRECTIONAL SOLIDIFICATION

Process development was continued toward the utilization of metallurgical grade silicon (MG-Si) for solar cell application. Major emphasis in this quarter was refining of MG-Si by directional solidification, vacuum refining and slagging. Significant developments were made in these areas.

Experimental Results

Directional Solidification. Experiments were carried out to study the solidification behavior of different grades of silicon and to refine the MG-Si by directional solidification. The experiments are shown in Table I.

Nearly single crystal structure was achieved in run S1-37. A 4 kg ingot was solidified. During growth a medium range of turbulence was observed in this run; however, it did not cause breakdown of crystallinity. Figure 1 shows the ingot cross-section.

In run S1-42 no seed meltback was achieved; hence, large grains were formed. The central portion of this ingot was directionally solidified again by HEM in run S1-45. No improvement in resistivity was observed on double solidification. This may be due to the columnar solidification achieved in the experiments. The effect of double solidification with a

TABLE I. Tabulation of Solidification Experiments

Run No.	Maximum Superheat During Meltdown (Furnace Temp. °C)	Growth Time (Hrs)	Structure	Bulk Resistivity (Ω-cm)	Remarks
S1-36	> 120	9	Nearly single crystal	0.240	-
S1-37	> 120	5.5	Nearly single crystal	0.090	-
S1-42	> 120	13	Polycrystalline	0.075	No seed meltback.
S1-43	> 120	10	Polycrystalline	0.040	Fast initial growth.
S1-45	> 120	10	Polycrystalline	0.055	Double solidification of run S1-42.
S1-47	> 120	12	Polycrystalline	0.040	-
S1-49	120	15	Polycrystalline	0.050	Much superheat in the melt caused seed loss.
S1-51	120	15	50% single crystal	0.040	Lower half of the ingot is single, upper half is polycrystalline
S1-52	112	31	Polycrystalline	0.180	Seed melted out.

single crystal interface is under investigation to verify this.

In runs S1-43, 47, 49 and 51, meltstock from the same source was used. In the first run 7.5 kg meltstock was used and polycrystalline structure was obtained. This is attributed to fast initial growth. Very low superheat in the melt during growth results in fast solidification early in the growth. In run S1-47 superheat in the melt was increased and growth rate was kept low. Interface breakdown was still observed early after initiating the growth. This indicates that temperature gradient in the liquid to growth rate ratio (G/R) was not high enough to prevent the constitutional supercooling. One important observation made in these experiments using different MG-Si, was that no turbulence was found during growth. As it will be discussed later in theoretical portions of this report, this necessitates a higher G/R ratio. In order to achieve turbulence in the next run, run S1-49, it was stabilized at a higher superheat. However, since the superheat was higher than intended, it caused seed loss. Hence, polycrystalline structure was obtained.

In run S1-51, lower superheat was used. Mild turbulence was observed during the first half of the growth. In the later part, however, temperature was gradually dropped in order to solidify the melt. This did not result in any convections in the liquid. The structure of this ingot is shown in Figure 2. It is seen that the solid-liquid interface has broken approximately half way along the ingot. This is in close agreement with theory. As it will be discussed further, more experiments were planned to

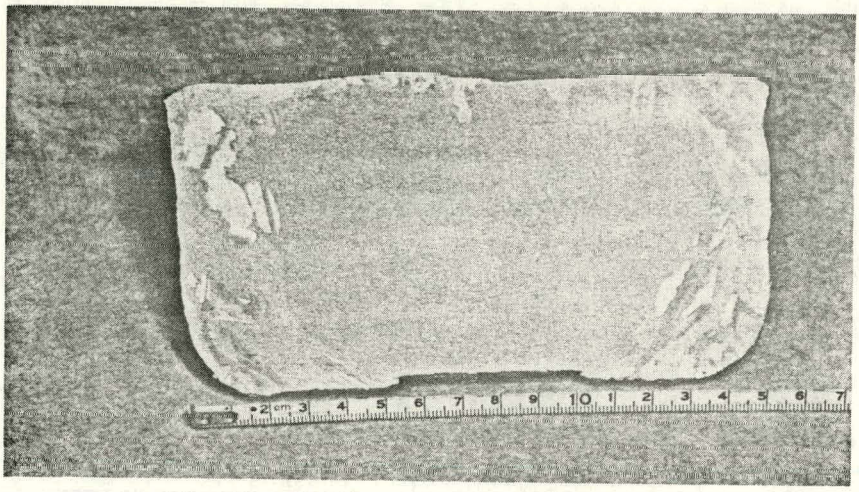

Figure 1. Cross-section of ingot cast in run S1-37.

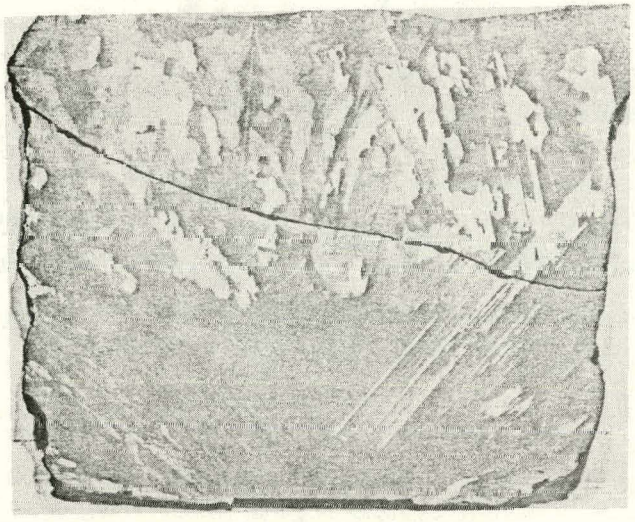

Figure 2. Cross-section of ingot cast in run S1-51.

obtain turbulence, hence to improve refining and crystallinity.

Refining

The experiments shown in Table II were carried out in order to determine the refining effect of vacuum processing and slagging. The effects of time, temperature and the amount of silica were studied.

Vacuum Refining. During the last quarter[1] it was reported that vacuum refining was not a sensitive function of temperature in the range of 28-70°C superheat and a holding time of 5 hours. Run S1-44 was held for 24 hours to study the effect of time on vacuum refining. Refining temperature was 35°C above the melting point. A small amount of material, 170 gms for a 5 kg ingot, was lost during refining. No seed was used and columnar solidification was achieved. Chemical analyses together with the meltstock analyses are given in Table III. These have been determined by spark source emission spectroscopy. Two samples were selected from the two different locations of the ingot for chemical analyses. Sample #1 is from the area at the bottom of the ingot, #2 is from near the top edge of the ingot, first and last material to solidify. Meltstock analysis has been determined twice. These samples were taken from the different locations of batch.

Results of chemical analyses indicate that impurities after 24 hours vacuum processing are well below the meltstock. If these results are compared with previously reported analyses,[1]

TABLE II. Tabulation of Vacuum Refining Experiments

Run No.	Maximum Superheat During Refining	Refining Time (Hrs.)	Bulk Resistivity (Ω-cm)	Remarks
S1-38	35	5	0.041	No seed; 0.25% silica added
S1-39	53	5	0.042	Same as S1-38
S1-40	80	5	0.050	Same as S1-38
S1-41	50	5	0.041	No seed; 0.5% silica added
S1-44	35	24	0.040	No seed. Study effect of vacuum refining for 24 hours.
S1-46	-	-	-	Various slag mixtures were melted in separate crucibles.
S1-48	35	-	0.050	Develop technique to add silica powder after melting silicon
S1-50	18	-	0.040	Same as S1-48

TABLE III. Impurities in the meltstock and ingot that were purified by vacuum processing. All values in ppm. Sample #1 was taken at the bottom of the ingot, #2 near crucible wall at the top of the ingot

Element	Meltstock		Run No. S1-44		Run No. S1-27		Run No. S1-28		Run No. S1-29	
	1st	2nd	#1	#2	#1	#2	#1	#2	#1	#2
B	5	10	< 5	< 5	< 5	< 5	< 5	< 5	< 5	< 5
Mn	56	5.3	< 4	25	< 2	167	< 2	160	< 2	200
Mg	10.9	2.6	2.4	1.1	0.3	0.8	0.4	2	1.3	< 0.2
Fe	580	900	4.3	330	< 1	3600	10	3800	10	4500
Al	660	680	10.9	220	2	2460	17	2440	13	2030
V	320	640	< .5	98	< 0.5	3300	< 0.5	3200	< 0.5	2300
Ti	107	113	4.7	45	< 0.5	1160	< 0.5	1280	< 0.5	680
Cu	32	62	3.8	26	2.6	210	2.8	210	1.7	340
Ni	145	210	.32	620	< 0.5	1480	1.0	1480	1.0	240
Ca	60	24	1.9	14	1.7	16	0.5	18	1.5	7.3
Cr	9.8	39	2	6	< 1	64	< 1	64	< 1	34
Ba	< 1	< 0.4	< 0.2	< .2	< 0.2	< 0.2	< 0.2	< 0.2	< 0.2	< 0.2
P	< 50	< 50	< 50	< 50	< 50	< 50	< 50	< 50	< 50	< 50
Mo	13	7.5	< 1	< 6	< 5	< 5	< 5	< 5	< 5	109
Sr		< 1			< 5	< 5	< 5	< 5	< 5	< 5
Sn		< 5	< 5	< 5	< 20	10	10	< 5	< 5	< 5
Zr	23	12.4	< 2	< 2	< 5	30	< 5	32	< 5	15

runs S1-27 through S1-29, longer holding time under vacuum shows better purification for the last material to solidify. From this it can be concluded that kinetic aspects of vacuum processing have a very important affect of refining. In this respect, more experiments, such as mechanical stirring and purging, are planned for the next quarter of this program. Stirring and purging will be carried out before and during solidifications.

Slagging. In order to refine the silicon, high purity silica powder was added to meltstock and solidification experiments were carried out at different temperatures. The experiments are summarized in Table II. No seed was used in these experiments and the melt was held at temperatures for five hours prior to directional solidification. Columnar type grains were formed. In runs S1-38, S1-39 and S1-40, silicon powder (0.25% by weight of meltstock) was used for refining. During the experiments considerable turbulence was observed in the melt indicating reactions with the silica powder. In run S1-38 no loss of meltstock was observed during refining. In run S1-39 no significant losses occurred; however, in run S1-40, in the vigorous reaction at a high temperature about 25% of the starting material was splattered in the furnace. Using this data a temperature close to that in run S1-39 was used in run S1-41 and the silica powder addition was doubled.

Chemical analyses of the samples from these experiments are shown in Table IV. Comparison of experiments S1-38, S1-39 and S1-40 with S1-27, S1-28 and S1-29 respectively, show that

Table IV. Impurities in the meltstock and ingots that were purified by slagging. All values in ppm. Sample #1 was taken at the bottom of the ingot; #2 near the crucible wall at the top of the ingot. Meltstock chemistry was determined twice.

Element	Meltstock		Run No. S1-38		Run No. S1-39		Run No. S1-40		Run No. S1-41	
	1st	2nd	#1	#2	#1	#2	#1	#2	#1	#2
B	< 5	<10	<10	<10	<10	<10	<10	<10	<10	<10
Mn	56	5.3	19	41	16	210	12	110	4	32
Mg	10.9	2.6	2.1	1.7	< 0.2	8.6	< 0.2	1.3	2.4	2.1
Fe	580	900	86	260	34	1500	4.1	830	11	280
Al	660	680	24	170	15	11	1.9	580	10	112
V	320	640	37	130	19	720	2	530	2	139
Ti	107	113	14	47	6.8	370	< 1	192	< 1	37
Cu	32	62	12.6	21	34	128	2.1	110	36	38
Ni	145	210	16	86	9.8	580	< 1	250	10	81
Cu	60	24	3.2	2.0	5.8	7.3	0.9	3.1	2.6	3.6
Cr	9.8	39	< 0.2	0.34	< 0.2	3.2	< 0.2	18	< 0.2	5.6
Ba	< 1	< 0.4	< 0.4	<0.4	< 0.4	<0.4	<0.4	<0.4	<0.4	<0.4
P	<50	<50	<50	<50	<50	<50	<50	<50	<50	<50
Mo	13	7.5	<4	4.5	<4	34	<4	21	<4	38
Sr		<1	<1	<1	<1	<1	<1	<1	<1	<1
Sn		<5	<5	<5	<5	<5	<5	<5	<5	<5
Zr	23	12.4	<10	43	<10	54	<10	15	<10	<10

by using a silicon powder for refining, better refining can be achieved at the later stage of solidification. This may be attributed to the increasing effect of impurity activity when liquid silicon is enriched with impurities by solute rejection. This is not effective at the beginning of solidification where activity of impurities is low.

Once again, as it was mentioned for vacuum processing,[1] experiments Sl-38, Sl-39 and Sl-40 show that slagging is not a very sensitive function of temperature. The effect of temperature on refining is further discussed in the theoretical portion of this report.

When the slag amount was doubled, run Sl-41, generally lower impurity was detected in comparison to run Sl-39. This is reasonable, because the increase of the amount of silica results in lower impurity activities, hence more impurity attractions from the silicon.

In the experiments mentioned above, silica powder was added to the meltstock in the crucible prior to melting. It was found that some of this powder sintered to the crucible and, therefore, was not very effective. In run Sl-48 a system was developed to add silica powder after melting the feedstock. The addition of powder into the 35°C superheated melt under these conditions caused vigorous reaction in the melt and approximately half the charge was splattered from the crucible. In the next run, run Sl-50, the same experiment was carried out for a 18°C superheated melt. A negligible amount of melt

loss occurred in this case.

Various slag compositions were examined in run S1-46 by melting them in silica crucibles. Oxides such as SiO_2, BaO, CaO, Na_2O, MgO and combinations such as $65SiO_2/10MgO/25CaO$, $75SiO_2/25Na_2O$, and $45BaO/55SiO_2$ were heated in separate crucibles to about 1435°C, held for about 4 hours and cooled. It was found that some of the oxides such as Na_2O reacted with the crucible; the crucibles with the other slag compositions did not show signs of deterioration.

Solar Cell Performance. Bars of 2.5 cm x 2.5 cm cross-section were sectioned out of ingots from runs 14, 18, 20, 33, 34 and 37 so that the growth direction was parallel to the axis of the bar. Solar cells of 2 cm x 2 cm size were fabricated from this material by Spectrolab, Inc., using their baseline fabrication procedure. During the initial testing a very thick layer of AR coating was deposited due to instrument malfunction. Evaluation of samples showed that samples from run 33, 34 and 37 were superior to those from 14, 18, and 20. During subsequent processing only one cell was processed from lot 14, 18 and 20.

(i) Baseline Cell Fabrication Procedure

The crystal sections were cut into slices of 15 mil thickness using multiblade slurry saws. Slices were numbered according to their position in the section, with increasing numbers going from top to bottom of the crystal section. Slices were then selected from the mid-portion of each crystal and polish-etched in an acid solution to a thickness of 9 mils. This was

done to remove the saw damage and to obtain the baseline cell thickness. The etched wafers were then cleaned to remove organic and metallic impurities from their surfaces. Within one hour wafers were placed on a clean quartz boat and placed in the diffusion furnace. The wafers were diffused in a phosphine source at 850°C in a two-step procedure of pre-dep and drive.

After diffusion, the wafers were masked on the front surface and passed through a spray etch. This etch removed the n region from the back surface. The mask was removed and the wafers cleaned in preparation for metallization. The cleaned wafers were placed in metal mask fixtures and the whole assembly was then placed in a diffusion-pumped vacuum chamber.

After pumping the system down to 1-5 x 10^{-6} torr titanium, palladium silver layers were deposited sequentially from crucibles that had been heated by e-beam. The layer thicknesses for the titanium, palladium and silver were 400Å, 200Å and 35,000Å respectively. Thicknesses were monitored *in situ* by a quartz crystal sensor.

Upon completion of metal evaporation (both sides of the wafers), the wafers were placed on a quartz boat and sintered in a hydrogen atmosphere at 590°C for four minutes. The wafers were placed in fixtures, the fixtures placed in a high vacuum system and an antireflecting Ta_2O_5 film deposited from e-beam heated source. Upon removal from the AR evaporator the wafers were annealed in air at ∿ 350°C for one minute.

The wafers were then masked on the top and bottom surfaces,

such that the edges were left uncoated. The wafers were passed through an etch to remove any stray metallic impurities that could cause leakage. After removal of the edge etch masks the completed cells were ready for measurement.

The current-voltage characteristics of the cells were measured at AMO, 28°C on a Spectrolab, Xenon-source, solar simulator. Spectral response measurements were made over the range of 410 nm to 1050 nm using a filter wheel containing narrow-band pass filters. During these measurements the text fixture was maintained at 28°C.

Throughout the processing and measurement each lot included six control wafers. Control wafers are nominally 2 Ω-cm, p type, (100), CZ silicon.

There is a decline in efficiency for cells made from each crystal, and this decline follows the order: 37, 34, 33, 18, 20 and 14. This decline in efficiency can be correlated with the decline in short circuit current and open circuit voltage. The reduced short circuit current is indicative of short minority carrier diffusion lengths in the base. One can also observe a similar grouping for curve fill factors. In many cases the low fill factor is caused by low shunt resistance (leakage). This could be associated with precipitates that might be found in the junction region of the cell. This latter statement is highly speculative and is based upon the effect one expects to find when metal precipitates are present and the fact that the silicon material is not highly purified.

The encouraging aspect of these measurements is that by a simple directional solidification of commercially available MG-Si by HEM it has been demonstrated that 7.2% efficient solar cells can be fabricated. In comparison, the best solar cell reported in literature after single CZ pulling of MG-Si is 2%.[2] The improved performance may be due to the submerged interface that is stable enough to produce a single crystal structure and better purification. Additional purification of material, such as slagging, purging, stirring, etc., can also be carried out during HEM processing to improve the quality of the cast silicon.

(ii) Results

The solar cell (2 cm x 2 cm, AR coated) results were measured under AMO illumination and data are shown in Table V and the spectral response of the same cells is shown in Figure 3.

TABLE V. Results of 2 cm x 2 cm, AR coated solar cells measured under AMO illumination

Run #	Cell #	V_{oc} mV	I_{sc} mA	cff	AMO η	AMI $\eta^{\dagger}$	Remarks
14							Cell broken
18	B-68	538	55	0.567	3.1	3.7	
20	C-62	456	47	0.455	1.8	2.1	
33	D-56	540	66	0.533	3.5	4.1	
	D-58	546	69	0.508	3.5	4.1	
	D-62	548	71	0.523	3.8	4.5	
34	E-54	534	76	0.529	4.0	4.7	
	E-55	581	75	0.690	5.6	6.6	
	E-56	581	78	0.660	5.5	6.5	
	E-64	581	76	0.682	5.6	6.6	
	E-65	582	76	0.683	5.6	6.6	
	E-66	541	73	0.180	1.3	1.5	Chipped cell
37	F-58	470	79	0.442	3.0	3.5	
	F-60	588	79	0.683	5.9	7.0	
	F-61	587	78	0.718	6.1	7.2	
	F-62	563	73	0.444	3.4	4.0	Chipped cell
	F-63	559	78	0.555	4.5	5.3	Chipped cell
Control	X-1	540	127	0.771	9.8	11.6	
	X-2	592	135	0.772	11.4	13.5	
	X-3	586	135	0.767	11.2	13.2	
	X-4	584	134	0.697	10.1	11.9	
	X-5	585	134	0.760	11.0	13.0	
	X-6	587	132	0.757	10.8	12.7	

†Estimated.

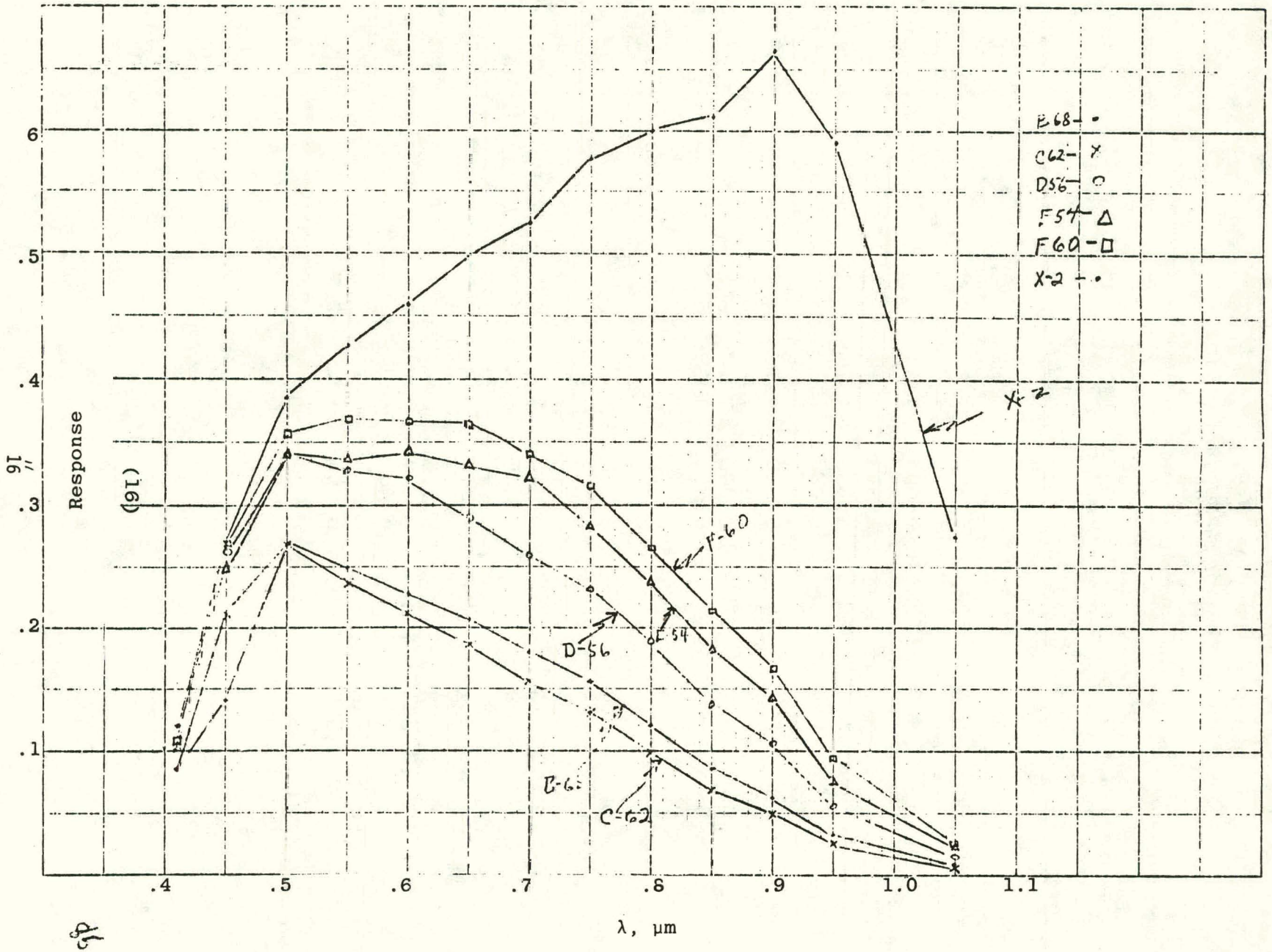
E-68 - •
C-62 - X
D-56 - ○
F-54 - △
F-60 - □
X-2 - •
X-2
F-60
D-56
F-54
C-62
Response
(16)
16
.1
.2
.3
.4
.5
6
.4
.5
.6
.7
.8
.9
1.0
1.1
λ, μm

Figure 3.

THEORETICAL ANALYSIS

Directional Solidification

The MG-Si can be grown into nearly single crystal structure by the HEM; however, in some cases, after some growth has been achieved, there is breakdown in the single crystallinity.

Constitutional supercooling accounts for breakdown in single crystallinity at the interface as impurities build up. It can be prevented by keeping high temperature gradients in the liquid at the interface, G_L , and/or by lowering the growth rate, R. According to the theory[3] it is required that

$$\frac{G_L}{R} \geqslant -\frac{m_L C_s^* (1-k)}{kD_L} \qquad (1)$$

where m_L is the slope of the liquidus line, C_s^* is the composition of solid at the interface, k is the equilibrium partition ratio and D_L is the diffusion coefficient of solute in the liquid. Equation (1) is valid regardless of the presence or absence of convection. If there is no convection, at the steady state $C_s^* = C_o$, where C_o is the initial crystal composition. Hence, equation (1) can be written as

$$\frac{G_L}{R} \geqslant -\frac{m_L C_o (1-k)}{kD_L} \qquad (2)$$

If there is strong enough convection $C_L^* = C_L$, where C_L is the bulk liquid composition. Considering that $C_s^* = kC_L$, from equation (1) it can be written that

$$\frac{G_L}{R} \qquad - \frac{m_L C_L (1-k)}{D_L} \tag{3}$$

The right side of equation (3) is always smaller than the right side of equation (2) because C_L is always smaller than C_o/k as shown below:

From Scheil equation,

$$C_L = C_o(1-f_s)^{k-1} \tag{4}$$

where this equation is valid if there is complete diffusion in the liquid. For $f_s = o$, $C_L = C_o < C_o/k$, and for $f_s = 1$, $C_L = 0 < C_o/k$, where k is always smaller than 1.

Thus, theory shows that if convection is achieved, a lower gradient is required to maintain the planar interface. Any kind of convection, such as obtained thermally or mechanically (by stirring or by purging) should help to achieve single crystal structure.

Vacuum Refining

As mentioned in the experimental results vacuum processing is effective in refining the MG-Si. However, chemical analysis

results do not clearly indicate the effect of temperature on purification. This problem is further discussed below.

The requirement for purification was expressed as

$$\frac{\chi_i}{x_s} = \frac{\gamma_i p_i^o}{p_s^o} \cdot \frac{N_i}{N_s} \qquad (5)$$

Purification under vacuum occurs if the ratio of impurity to silicon in the gas phase is higher than in the liquid, *i.e.*,

$$\frac{\chi_i}{x_s} > \frac{N_i}{N_s} \qquad (6)$$

where p_i^o and p_s^o are the standard partial pressure of impurity and solvent, respectively, γ_i = activity coefficient of impurity at infinite dilution, N_i and N_s are the mole fraction of impurity and solvent in the solution respectively, and χ_i and x_s are the mole fraction of impurity and solvent in the gas phase, respectively. In equation (5) the variables to affect purification are γ_i, p_i^o and p_s^o. The dependence of γ_i on total pressure is extremely small; however, its value is a strong function of temperature. The values of p and p are also dependent on temperature. Therefore, purification is affected by temperature and the change of $\gamma_i p_i^o / p_s^o$ is a measure of the effect of temperature on purification.

In order to make calculations, the activity coefficient of impurities has to be known. Unfortunately there is not adequate

information in the literature related with the activity coefficients of impurities in silicon. The only available information[5,6] is related with Fe and Mn. At 1437 and 1482°C, respectively, γ_{Mn}[5] is 7.01×10^{-3} and 7.58×10^{-3}, γ_{Fe}[6] is 1.572×10^{-2} and 7.58×10^{-3}, and the standard vapor pressures[7] are p^o_{Mn} is 8×10^{-3} and 10×10^{-3}, p^o_{Fe} is 2.7×10^{-5} and 5.3×10^{-5}, and p^o_{Si} is 8×10^{-5} and 1.75×10^{-5} atm. The calculations of $\gamma_i p^o_i/p^o_s$ at 1437 and 1482°C, respectively, are 0.701 and 0.433 (for Mn) and 5.31×10^{3} and 5.02×10^{-3} (for Fe). It is seen that the value of the ratio of $\gamma_i p^o_i/p^o_s$ increases by decreasing the temperature. Since this ratio is smaller than unity in both temperatures, no purification is expected for Fe and Mn.

The Mn content in MG silicon is not high, usually 3-300 ppm[8], and the two samples analyzed from the same meltstock showed values of 5.3 and 56 ppm (Table III). However, the Fe content is quite high. The experimental data shows that during HEM processing considerable purification of Fe is achieved (*e.g.*, Table III) which is contradictory to the above theoretical analysis. The cause for this discrepancy may be associated with parameters such as limited data available in literature for activity coefficients, the interaction of other impurities affecting the values of Fe, formation of complexes instead of vaporization of elemental Fe, variation of localized pressure conditions in experimental arrangement compared to uniform dispersion assumed in theoretical analysis.

SUMMARY

1. It has been shown that simple directional solidification by HEM of commercially available MG-Si can produce material for 7.2% conversion efficiency solar cells.
2. Columnar solidification following the vacuum treatment of MG-Si shows that: (a) longer holding times under vacuum gives better purification; (b) the use of silica powder helps the refining, especially for the later solidified portion of the ingot; (c) the increase in the amount of silica powder results in better refining; (d) vacuum processing with slagging does not seem a sensitive function of temperature.
3. Theoretical analyses of the solidification of MG-Si by HEM as a single crystal shows that lower temperature gradient to growth rate ratio can be allowed if turbulence is achieved in the melt.
4. Theoretical analysis of vacuum refining shows that purification is a strong function of temperature.

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