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LOW-COST EPITAXIAL TECHNIQUES FOR SOLAR-CELL FABRICATION

Quarterly Technical Progress Report No. 1 for September 26—December 24, 1979

By
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January 1980

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RCA Laboratories
Princeton, New Jersey

MASTER

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Solar Energy

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PREFACE

The objective of this work is to extend epitaxial techniques for the growth of silicon films on the lowest cost silicon substrates consistent with the material and device requirements for the fabrication of high-efficiency solar cells. The specific approaches toward this goal are:

- The characterization of several candidate silicon substrate materials to determine impurity level, doping distribution, grain boundary morphology, and defect state;
- Optimization of epitaxial growth parameters in relation to the substrates; and
- Solar-cell and device fabrication and evaluation to provide feedback relating to the epitaxial growth and the substrate quality.

SUMMARY

During the first quarter, a candidate low-cost silicon substrate, designated upgraded metallurgical grade (UMG) silicon, was obtained. The electrical, physical, and structural properties of three grades of this material were measured. An initial epitaxial growth technique was standardized and qualified for application to the growth of diagnostic and solar-cell structures. Such structures were grown on the three UMG grades of substrates and the properties of these layers were compared to those of the substrates. It was generally found that the defect density in the epitaxial films was lower than in the substrates, but greater aerial variation was observed for the less pure, multicrystalline grades of substrates. Solar cells were fabricated in epitaxial structures grown on all three UMG grades of substrates. An AM-1 efficiency of 12.9% was obtained when the purest substrate grade was used, and 10.3% was achieved with a 15- μ m-thick film grown on the material of lowest purity.

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SECTION 1.0

INTRODUCTION

1.1 BACKGROUND

Previous work [1,2] has demonstrated the feasibility and technical advantages of using chemically vapor deposited (CVD) epitaxy for the growth of silicon solar-cell structures. Silicon solar cells having nearly ideal electrical characteristics and very high open-circuit voltage were fabricated in epitaxial layers grown on high-quality silicon substrates [1]. This technique was also explored for potential application in the low-cost manufacturing of solar cells by the use of epitaxial growth in conjunction with substrates such as edge-defined film-fed growth (EFG) ribbon [3] and several forms of simply purified metallurgical grade silicon [4,5]. This latter work identified the need for a full technical qualification and the establishment of supply for the lowest cost silicon substrate consistent with high efficiency, good yield, and reproducibility of epitaxial solar-cell fabrication.

1.2 OBJECTIVES AND APPROACH

The major objective of this contract work is to refine and apply epitaxial growth techniques to several candidate low-cost silicon substrates in order to define the growth technique and substrate combination which will allow the fabrication of low-cost and efficient solar cells. The planned approach is:

- Define and characterize several potentially low-cost silicon substrates in terms of physical, chemical, and electronic properties.
- Refine and explore suitable epitaxial growth methods in conformance with the substrate characteristics so that the resultant epitaxial layers can be used in the fabrication of high-efficiency solar cells. The efficiency goal for such cells is 14%, AM-1.
- To fabricate solar cells and diagnostic devices and to perform electronic characterizations so that the influence of the epitaxial growth and structure, as well as the substrate, can be evaluated.

Sections 2.1 and 2.2 below describe the specific material characterization applied to the first group of substrates selected for study. This material consists of three forms of upgraded metallurgical grade (UMG) silicon, which was purchased from the Hemlock Semiconductor Corporation.* The characterization consists of physical and chemical impurity analysis involving mass spectroscopy and several forms of x-ray topography. The impurity content, crystallographic structure, and defect state obtained from these analyses are to be later

*Hemlock Semiconductor Corporation, Hemlock, Michigan, a subsidiary of the Dow Corning Corporation.

compared with those of the epitaxial layers grown on the substrates. Attempts will also be made to correlate the electronics properties of solar cells with the material properties found in the epitaxial layers and substrates.

Section 2.3 describes the epitaxial techniques applied to date in the growth of diagnostic and solar-cell structures. Also presented in this section are the results of an x-ray topographic study comparing the defect structure in the substrates with that of the epitaxial layers.

In Section 2.4, the electrical characteristics of epitaxial solar cells fabricated on the three grades of UMG silicon are given and compared.

A discussion and summary of the results obtained to date are presented in Section 3.0 and plans for the next quarter of work are given in Section 4.0.

SECTION 2.0

PROGRESS

2.1 DESCRIPTION OF SUBSTRATES

An initial supply of silicon substrate material, designated upgraded metallurgical grade (UMG) silicon, was purchased from Hemlock Semiconductor Corporation. This material was selected on the basis of extensive research and development conducted by Hemlock, their willingness to cooperate in its evaluation, and their projection of its eventual low cost. Three grades of silicon were supplied in the form of 2-in.-diameter wafers and 3-in.-diameter boules. The nomenclature associated with each grade along with some pertinent characteristics of each are given in Table 2-1.

This material was produced by remelting and applying chemical purification techniques to metallurgical grade silicon as obtained from an arc furnace. The three grades differ primarily in the amount of purification or upgrading applied to each. Grade 1S received the highest degree of purification and is single crystal in structure. Grades 1P and 2P have large-grained multicrystalline structures as illustrated by the photograph in Fig. 2-1. The chemical purity of grade 1P is similar to that of 1S, and grade 2P is of substantially lower purity than either 1P or 1S.

2.2 SUBSTRATE CHARACTERIZATION

2.2.1 Electrical Resistivity

The electrical resistivity of the three substrate grades was measured by two-point spreading resistance. Surface and bulk scans were made on wafers arbitrarily selected from each grade to determine the absolute resistivity and the uniformity across large areas. Typical spreading resistance measurements for the three grades of material are shown in Fig. 2-2, where the average resistivity is seen to be $\sim 0.01 \Omega\text{-cm}$. Variation in resistivity across a 2-in.-diameter wafer was no more than $\pm 15\%$.

2.2.2 Impurity Analysis

Emission mass spectrographic analysis was performed on all three grades of material, and the results are shown in Table 2-1. The detected impurity levels for the elements listed and the relative order among the three grades of silicon are consistent with Hemlock's internal analyses and their description of the relative degree of purification employed in the production of this material.

2.2.3 X-Ray Analyses

The orientation of grade 1S material was determined by Laue and was found to be within 1° of $\langle 100 \rangle$. Measurements are underway to determine the relative orientation of selected grains in the 1P and 2P material.

Table 2-1. PROPERTIES OF UMG SILICON

<u>UMG Grade</u>	<u>Crystallinity</u>	<u>Resistivity^a Ω-cm</u>	<u>Type</u>	<u>Impurities^b (ppm)</u>					
				<u>Al</u>	<u>B</u>	<u>P</u>	<u>O</u>	<u>C</u>	<u>Ge</u>
1S	Single Crystal <100> Orientation	0.01 ± 15%	p	4	71	5	58	3	0.6
2P	Large Grain Polycrystalline	0.01 ± 15%	p	40	105	11	240	10	1
1P	Large Grain Polycrystalline	0.01 ± 15%	p	0.6	53	4	44	4	0.7

^aFrom two-point spreading resistance measurements.

^bFrom spark-source mass spectroscopy.

Figure 2-1. PHOTOGRAPH OF THE ETCHED SURFACE OF A 2-in.-DIAMETER WAFER. THE MATERIAL IS UMG GRADE 1P

Figure 2-2. TWO-POINT SPREADING RESISTANCE TRACES MADE ON THE THREE UMG SILICON SUBSTRATES

2.3 EPITAXIAL GROWTH

2.3.1 Growth Procedures

The growths were carried out using dichlorosilane in a standard horizontal reactor. The quartz tube has a cross section of 5 x 10 cm and held a silicon carbide-coated graphite susceptor that was 30 cm long. Heating was accomplished by rf induction into the susceptor, which was inclined horizontally, and the walls of the reactor were air-cooled. Hydrogen was obtained from a Pd-Ag diffusion cell. The doping gas is diborane diluted with hydrogen at the 10- to 20-ppm level and further diluted as needed before insertion into the reactant gas stream. Dichlorosilane was metered as a gas directly from the cylinder and temperatures were measured with an optical pyrometer and corrected for emissivity and quartz adsorption effects. Typically, the temperature during growth is 1100°C and the growth rate is 5 $\mu\text{m}/\text{min}$. The epitaxial growth cycle used in our first experiments is listed below.

- (1) Substrates are chem-etched, polished, cleaned, and then spun dry and placed on a susceptor which is inserted into the epitaxial reactor.
- (2) Reactor is flushed with hydrogen, flow rate 30 liter/min, for 10 min. All other lines to be used are flushed to vent line for 5 min and turned off.
- (3) rf generator is turned on and substrates heated to 1150°C with hydrogen flowing for 5 min.
- (4) Substrates are etched in 1% HCl for 5 min at 1150°C; this removes about 5 μm of material
- (5) HCl is turned off and system is flushed with hydrogen for 5 min.
- (6) Temperature of substrates is reduced to growth temperature of 1100°C.
- (7) Flow rates of gases (SiH_2Cl_2 , AsH_3 , or B_2H_6) to give desired growth rate, doping level and conductivity type are metered to vent line.
- (8) Automatic timer is set to give predetermined thickness, and growth is started.
- (9) Growth is stopped automatically and reactor is flushed with hydrogen for 3 min.
- (10) rf power is turned off and system cools with hydrogen flowing.

This procedure was used successfully in the past to grow solar-cell structures on several different types of silicon substrates. For this reason it was selected as a starting point for the present investigations. The resultant layers and the devices fabricated in them will be analyzed as described below, and changes in the above growth procedures, where necessary, will be made as dictated by deficiencies which can be related to the epitaxial growth. It is anticipated that other reactor types, modified epitaxial techniques, and raw materials may be used to explore lower cost methods of layer growth.

2.3.2 Properties of Epitaxial Layers Grown on UMG Substrates

For purposes of diagnostic measurements described below, 100- μm -thick, undoped layers were grown on the three UMG grades of substrates. The objective in growing such layers is to make comparisons of the impurity content and defect structure in the grown layers with that of the starting substrates. Spark-source mass spectrographic analysis was attempted on such layers during this period; however, difficulties were encountered in reproducibility and correlation of this data with previous data.

Studies of the defect structure by means of x-ray topography have revealed interesting differences between the grown layers and the substrates. From these studies the following observations were made:

- The crystal perfection of the epitaxial layer on average is significantly better than the starting substrate material. This is seen in the section topograph presented in Fig. 2-3. For the grade 1P and 2P material, there are some grains where no improvement in crystal quality is observed. More studies concerning the nature of the imperfections within these grains are planned.
- A network of misfit dislocations near the epitaxial-substrate interface is observed using the single-crystal and polycrystalline UMG substrates. This is shown in Figs. 2-4 and 2-5.
- Grain boundaries present in the substrate material propagate into the epitaxial layer as depicted in Fig. 2-6.

Figure 2-3. SECTION X-RAY TOPOGRAPH OF A 100- μm -THICK EPITAXIAL FILM GROWN ON GRADE 1S SUBSTRATE

Figure 2-4. PROJECTION X-RAY TOPOGRAPH OF A 100- μm EPITAXIAL FILM GROWN ON GRADE 1S SUBSTRATE. CROSS-HATCHED LINES ARE THE STRAIN FIELDS ASSOCIATED WITH MISFIT DISLOCATIONS AT THE FILM-SUBSTRATE INTERFACE

2.4 SOLAR-CELL FABRICATION

In this section, the details of the solar-cell fabrication starting from as-grown epitaxial wafers are described.

2.4.1 General Procedures

The general procedures for fabrication of the solar cells and test structures are:

- (1) Grow epitaxial layers.
- (2) Characterize grown layers by spreading resistance.
- (3) Clean wafers - $\text{H}_2\text{SO}_4:\text{H}_2\text{O}_2$; $\text{H}_2\text{O}_2:\text{NH}_4\text{OH}$; $\text{H}_2\text{O}_2:\text{HCl}$ boiling solutions. For epitaxial structures without a junction layer: POCl_3 diffusion at 875°C for 30 min followed by a slow pull (10 min). Wafers are then stripped of oxide and a four-point sheet resistance measurement is made. Sheet resistance values average $75 \Omega/\square$ with a junction depth of $\sim 0.3 \mu\text{m}$.
- (4) Metallize by evaporation of Ti/Ag ($0.2 \mu\text{m}/5 \mu\text{m}$) - front and back.
- (5) Spin-on AR coating - bake and sinter.
- (6) Screen-on wax to define cell and test structure areas.
- (7) Etch silicon to form mesa structures delineating cell area.
- (8) Remove wax, rinse, and bake.

These processing steps are described in more detail below.

Figure 2-5. PROJECTION X-RAY TOPOGRAPH OF A 100- μ m-THICK
EPITAXIAL FILM GROWN ON GRADE 1P SUBSTRATE

Figure 2-6. SECTION X-RAY TOPOGRAPH OF A 100- μ m-THICK EPITAXIAL FILM GROWN ON GRADE 1P SUBSTRATE. FILM GROWTH IS SEEN (ARROW) TO FOLLOW GRAIN BOUNDARY

2.4.2 Mask Design

The contact mask used to delineate the metallization pattern for all cells reported here is shown in Fig. 2-7. The large cell is 2.1 x 2.1 cm, having 11 current collecting fingers and a single bus-bar with a total 7% metal area coverage. The two small cells and ten diodes are for diagnostic purposes such as the measurement of lifetime and area dependence of electrical characteristics. The parallel bar structures A and A' are included for measurement of surface-layer sheet resistance and metal-to-silicon contact resistivity, and the line structure B is for the measurement of metal sheet resistance.

Figure 2-7. SOLAR-CELL MASK DESIGN INCLUDING DIAGNOSTIC CELLS AND DEVICES

2.4.3 Metallization

Metallization was done by E-gun evaporation of 0.2 μm of titanium followed by 5 μm of silver. These metals are evaporated over the entire front and back of the wafers and the pattern shown in Fig. 2-7 is then defined photolithographically. The silicon substrates were not heated during evaporation; sintering was done in a separate furnace at 500°C for 15 min in argon after the photoresist and metal etching.

2.4.4 Antireflection Coating

A commercially available* titaniumsilica film spin-on antireflection (AR) coating was used because of its ease in handling and good optical properties. The type-C spin-on film we purchased and used has a reported index of refraction of 1.96. The liquid is spun onto the silicon wafer and then baked successively at 100°C and 400°C for 15 min in air.

2.4.5 Mesa Etching

We use mesa etching to define the cell and test structure areas and to delineate and clean the peripheral junction areas. This is accomplished by screening a presoftened wax onto the wafers through a metal mask. The exposed silicon is then etched to a depth of 1 mil (25 μm) using a hydrofluoric acid, acetic acid, nitric acid (1:2:2) solution. The resulting cell area varies somewhat from run to run but is generally close to 4.4 cm^2 . This process results in a clean, damage-free peripheral mesa area including the junction at the cell edge.

2.5 EPITAXIAL STRUCTURES AND SOLAR CELLS

2.5.1 Baseline Epitaxial Solar Cells

In order to establish a baseline for the epitaxial film quality and solar-cell performance, the structures shown in Fig. 2-8 were grown on high-quality Czochralski (CZ) single-crystal p^+ substrates. Solar cells were fabricated on each structure as described in Section 2.3 above. The thicker structures (III, IV, and V) were included to assess the effect of diffusion length on cell performance. The measured AM-1 cell characteristics for cells made in each structure are summarized in Table 2-2.

Figure 2-8. BASELINE EPITAXIAL STRUCTURES

The results show first that a 15- μm -thick epitaxial layer is sufficient to obtain an AM-1 efficiency of 13% with excellent cell characteristics. Second, these data show that with the current epitaxial growth techniques, the performance of solar cells of thickness greater than 20 μm is diffusion-length-limited. This is indicated by the saturation in short-circuit current and

*Titaniumsilica film-type C, purchased from Emulsitone Co., Whippany, NJ.

Table 2-2. SUMMARY OF AM-1 PARAMETERS FOR BASELINE
EPITAXIAL SOLAR CELLS

<u>Structure</u>	<u>W_{epi}</u> <u>(μm)</u>	<u>J_{sc}</u> <u>(mA/cm²)</u>	<u>V_{oc}</u> <u>(mV)</u>	<u>FF</u> <u>-</u>	<u>η</u> <u>(%)</u>
I	15	27.0	602	0.801	13.0
II	20	26.8	590	0.790	12.5
III	50	26.2	570	0.785	11.7
IV	75	IN PROCESS			
V	100	25.8	560	0.785	11.0

the decrease in open-circuit voltage for the thicker structures. Analysis of spectral response data for these samples yielded an estimate of 30 μm for the diffusion length in the epitaxial layer. Although thin epitaxial structures are desirable for economy in the epitaxial growth, and the above results show that with the thinnest structures solar-cell efficiencies of approximately 13% are obtainable, an increase in diffusion length will raise the level of efficiency for all structures.

As a result of these experiments, changes are being made in the epitaxial reactor and growth cycle to include slow-cooling and gettering in an attempt to obtain longer diffusion lengths. Slow-cooling capability has been added to our epitaxial reactor by providing a linear voltage ramp on the power supply output to the rf generator. This allows the system to be cooled at a rate of 3°C/min from the growth temperature (1100°C) down to 800°C. Preliminary measurements on thick epitaxial diode structures prepared with and without slow cooling indicate a three- to fourfold increase in diode recovery time for the slow-cooled samples.

Until the results of the slow-cooling are established, only the thin (<50 μm) structures will be grown on the low-cost UMG substrates. The initial results obtained with these substrates are described below.

2.5.2 Solar Cells on UMG Silicon Substrates

Epitaxial structures I and III of 15- and 50-μm were grown on the three UMG grades described in Section 2.1. For comparative purposes, solar cells were also fabricated directly into these substrates by performing the diffusion of the junction layer along with the epitaxial structures. Solar cells were completed as described above and their AM-1 characteristics were measured.

A summary of the measured AM-1 solar-cell parameters is given in Table 2-3. For each structure, the data on the first line give the parameters for the cell

Table 2-3. COMPARISON OF AM-1 SOLAR-CELL PARAMETERS

STRUCTURE	SUBSTRATE	W_{epi} (μm)	J_{sc}^{a} (mA/cm^2)	V_{oc} (mV)	FF	η (%)	DATA
I	P ⁺ CONTROL	15	28.2	605	0.810	13.2	MAX
I	P ⁺ CONTROL	15	27.0	575	0.778	12.3	AVE
III	P ⁺ CONTROL	50	28.5	593	0.806	13.1	MAX
III	P ⁺ CONTROL	50	27.6	570	0.785	12.3	AVE
I	DOW GRADE 1S	15	26.5	600	0.792	12.6	MAX
I	DOW GRADE 1S	15	25.9	604	0.797	12.5	AVE
III	DOW GRADE 1S	50	27.1	592	0.800	12.9	MAX
III	DOW GRADE 1S	50	26.7	581	0.791	12.3	AVE
I	DOW GRADE 2P	15	24.8	570	0.734	10.3	SAMPLE 1
I	DOW GRADE 2P	15	24.7	562	0.713	9.9	SAMPLE 2
III	DOW GRADE 2P	50	26.0	540	0.655	9.2	SAMPLE 1
III	DOW GRADE 2P	50	24.2	510	0.644	8.0	SAMPLE 2
I	DOW GRADE 1P	15	24.7	550	0.713	9.7	SAMPLE 1
I	DOW GRADE 1P	15	24.4	490	0.560	6.7	SAMPLE 2
III	DOW GRADE 1P	50	23.8	533	0.731	9.3	SAMPLE 1
III	DOW GRADE 1P	50	23.8	522	0.729	9.1	SAMPLE 2
DIFF. N ⁺ /P	DOW GRADE 1S	0	14.9	570	0.501	4.2	TYPICAL

^a Cell Area = 4.5 cm², AR Coated

having the highest efficiency while the average values are given on the second line when the number of samples is four or greater.

These data show the following:

- Aside from the slightly (~4%) reduced short-circuit current density, the performance of the epitaxial cells made on the grade 1S substrates is comparable to that of the control samples made on high-quality CZ silicon.
- An efficiency of 10.3% was achieved with the polycrystalline substrates.
- The similarity of results for cells on the multicrystalline grades 1P and 2P is important because grade 2P material is considerably less pure than grade 1P.
- Solar cells made on the epitaxial layers have significantly higher efficiencies than those made directly in the substrate material.

As noted above no significant difference in efficiency level was noted between the cells fabricated on grades 1P and 2P material, and compared to grade 1S and the controls, the open-circuit voltages and fill-factors are lower, and show more sample-to-sample variation. A set of typical illuminated I-V curves for structure I cells on the three substrates grades is shown in Fig. 2-9.

Figure 2-9. TYPICAL ILLUMINATED I-V CURVES FOR EPITAXIAL AND BULK SOLAR CELLS FABRICATED ON UMG SUBSTRATES

An explanation for the reduced short-circuit current as well as the reduced voltage and fill-factors for the grades 1P and 2P substrates is complicated because these substrates are of low purity and are polycrystalline. Some indication of the effect of the polycrystalline structure was obtained by measuring the spectral response with both a large and small light spot. These data (Fig. 2-10) show the expected lower average quantum efficiency for grade 2P cells when compared to that of grade 1S; however, considerable variation in the quantum efficiency from grain to grain was noted as is also shown in Fig. 2-10.

For comparative purposes, the performance data for cells made by direct diffusion into the grade 1S substrates are also given in Table 2-3 and Figs. 2-9 and 2-10. The base diffusion length in the substrate was estimated from the data of Fig. 2-10 and is only about 3 μm , which is consistent with the very low short-circuit current and efficiency obtained.

Figure 2-10. SPECTRAL RESPONSE CURVES FOR EPITAXIAL AND BULK CELLS FABRICATED ON UMG SUBSTRATES

SECTION 3.0

DISCUSSION AND SUMMARY OF RESULTS

During the first quarter, a candidate low-cost upgraded metallurgical grade (UMG) silicon substrate material has been procured and examined. The impurity content of three grades of this material was measured and found to be in agreement with the degree of purification and with similar measurements made by the manufacturer. Examination by x-ray topographic techniques of epitaxial layers grown on these substrates has shown that in most areas of the films, the defect density in the grown layer is substantially less than that of the substrates. However, in the multicrystalline grades, the defect density and strain in the epitaxial film showed a great degree of aerial variation. We plan to investigate possible correlations of these defects with the photo-response of individual solar-cell areas.

Epitaxial solar cells were fabricated on high-quality CZ silicon to establish baseline performance for structures 15 to 100 μm thick. It was found that 13% cell efficiency was obtainable with the 15- μm -thick films, but that the efficiency of thicker cell structures is limited to 11-12% by a diffusion length of about 30 μm . Slow-cooling capability was added to our epitaxial reactor, and initial experiments indicate that longer diffusion length results. This technique along with simple gettering techniques will be further explored.

Epitaxial solar-cell structures were grown on the three UMG substrate grades. The best solar-cell performance, $\eta = 12.9\%$, was obtained when the purest grade single-crystal UMG silicon was used as the substrate. An efficiency of 10.3% was achieved with a 15- μm -thick epitaxial layer grown on one of the multicrystalline substrates. Although the impurity levels in the two multicrystalline grades differ greatly, the efficiency of epitaxial cells made on them show little difference. This implies that grain boundary effects are playing a major role in limiting the efficiency of cells made on the multicrystalline-grade substrates. However, it should be noted that the density and aerial distribution of point defects in the multicrystalline grades 1P and 2P are quite different from the single-crystal grade 1S substrate, so that it is too early to judge the relative degradation mechanisms associated with these materials.

That the epitaxial layers are required for good cell efficiency was shown by fabricating solar cells directly in the UMG substrates; such cells have an efficiency of only about 4%. We expect to concentrate future efforts on finding the limitations imposed by the multicrystalline substrate grades.

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SECTION 4.0

PLANS FOR NEXT QUARTER

The work planned for next quarter includes:

Task I Substrate and Materials Characterization

- Chemical and physical impurity analysis and comparison of epitaxial film grown on UMG silicon.
- Crystallographic x-ray analysis to determine relative orientations of grains in grades 1P and 2P UMG substrates.
- Section x-ray topographic studies of selected grains from solar-cells grown on 1P and 2P substrates.

Task II Epitaxial Growth

- Evaluate the effect of slow-cooling on solar-cell properties.
- Initiate growth experiments with lower temperatures and the use of silane.
- Prepare epitaxial solar-cell structures on grade 2P substrates which have been cut perpendicular to the crystal growth axis.

Task III Solar-Cell Fabrication and Evaluation

- Perform detailed evaluation by laser scanning and light-spot spectral response measurement on selected solar cells. Attention to be concentrated on cells grown on multicrystalline grades 1P and 2P substrates.
- Continue cell fabrication and evaluation to build statistical data on epitaxial cells grown on UMG substrates.

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SECTION 5.0

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