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LOW-COST THIN-FILM POLYCRYSTALLINE
SILICON SOLAR CELLS

QUARTERLY PROJECT REPORT NO. 1
FOR THE PERIOD
March 15, 1979 to June 30, 1979

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J. D. Crownover
and
Gerard A. van der Leeden

July, 1979

Work Performed Under Contract DE-AC03-79ET-23408

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PREPARED FOR THE
U.S. DEPARTMENT OF ENERGY
SOLAR ENERGY
UNDER CONTRACT DE-AC03-79ET-23048

CONTENTS

List of Illustrations	11
Summary	1
I. Introduction	2
II. Review of the SMU Technology	4
II. 1. Purification of Metallurgical Silicon	
II. 2. Preparation of Substrates	
II. 3. Deposition of Silicon Films and Solar Cell Structures	
III. Preparation of Substrates and Deposition of Silicon Films	11
IV. Acid Extraction of Metallurgical Silicon	15
V. References	17
VI. Personnel	18
VII. Plan for the Next Period	19
VIII. Report Distribution List	20

List of Illustrations

Figure 1	Schematic diagram of the apparatus for the preparation of metallurgical silicon substrate by the moving-coil zone-melting technique.	12
Figure 2	Schematic diagram of the apparatus for the chemical vapor deposition of silicon by the thermal reduction of trichlorosilane.	14
Figure 3	Concentration of iron in solution after successive extractions of pulverized metallurgical silicon with acids.	16

Summary

This is the First Quarterly Project Report of a research and development program " Thin Films Polycrystalline Silicon Solar Cells" supported by the U. S. Department of Energy under Contract No. DE-AC03-79ET-23048. The objectives of this contract is to advance the thin film silicon solar cell technology developed at Southern Methodist University, Dallas, Texas, with a view toward achieving photovoltaic conversion efficiencies greater than 10% at a cost of \$100 to \$300/kWe.

The technology developed at SMU consists of: (1) the purification of metallurgical silicon by acid extraction and phosphorus pentoxide treatment, (2) the preparation of metallurgical silicon substrates by the unidirectional solidification of the melt on graphite, (3) the deposition of a silicon p-n junction structure on metallurgical silicon substrates by the thermal reduction of trichlorosilane, and (4) the application of grid contacts and anti-reflection coatings. Major efforts during this period have been directed to the organization and planning of the overall technical approaches, particularly in regard to the preparation of metallurgical silicon substrates. The zone-melting technique has been selected for the preparation of substrates, and a moving-coil zone-melting apparatus has been designed and constructed. A chemical vapor deposition apparatus for the deposition of the active region of solar cell has also been designed and constructed. In addition, the purification of metallurgical silicon by acid-extraction has been investigated semi-quantitatively.

I. Introduction

This is the First Quarterly Project Report of a research program "Thin Films Polycrystalline Silicon Solar Cells" supported by the U. S. Department of Energy under Contract No. DE-AC03-79ET-23048. The objective of this contract is to advance the thin film silicon solar cell technology developed at Southern Methodist University, Dallas, Texas, with a view toward achieving photovoltaic conversion efficiencies greater than 10% at a cost of \$100 to \$300/kWe.

The technology developed at SMU consists of: (1) the purification of metallurgical silicon by acid extraction and phosphorus pentoxide treatment, (2) the preparation of metallurgical silicon substrates by the unidirectional solidification of the melt on graphite, (3) the deposition of a silicon p-n junction structure on metallurgical silicon substrates by the thermal reduction of trichlorosilane, and (4) the application of grid contacts and anti-reflection coatings. Solar cells of 9 cm² area with AMI efficiencies of up to 9.75% have been produced. The SMU approach is a potentially low cost process and will be reviewed briefly in Section II. Its advantages include:

- (1) Lower-cost metallurgical silicon and graphite are substituted for high cost single crystalline silicon.
- (2) Costly processing operations, such as crystal growth, slicing, surface preparation, diffusion, and back ohmic contact making, are eliminated.
- (3) Rectangular geometries permit a high cell packing factor.

- (4) Any technical innovation which lowers the cost of operations such as anti-reflection coating applications, interconnection, array packaging, etc., will apply equally well to thin film polycrystalline silicon solar cells.

The program at Poly Solar Incorporated is directed to the further development of the SMU process, including (1) the scaling-up and process optimization to produce large area cells of high conversion efficiency with good reproducibility and yields, and (2) the assessment of the feasibility of using this process for manufacturing purposes. During the first quarter of this program, efforts have been directed to program planning, and design and construction of apparatus. Since the structural property of the substrate is a most important parameter affecting the characteristics of solar cells, a moving coil apparatus for the unidirectional solidification of metallurgical silicon has been designed and constructed for the preparation of large area substrates (approximately 190 cm^2) in a controlled manner. An apparatus for the chemical vapor deposition of silicon with controlled dopant concentration and distribution by the thermal reduction of trichlorosilane has also been designed and constructed. Furthermore, a series of experiments has been carried out to investigate quantitatively the removal of iron, a major impurity, from metallurgical silicon by acid-extraction, and the extracts were analyzed for iron spectrophotometrically. These activities are discussed in Sections III and IV.

II. Review Of The SMU Technology

Various aspects of the thin film polycrystalline silicon solar cell technology developed at Southern Methodist University have been described in several publications.⁽¹⁻⁶⁾ The overall process is briefly reviewed here for completeness.

II. 1 Purification of Metallurgical Silicon

Metallurgical silicon is manufactured by the reduction of sand with carbonaceous reducing agents in a submerged electric arc furnace at 1700⁰-1900⁰C, and the resulting silicon melt is discharged into molds for solidification. It is of about 98% purity and contains iron and aluminum at concentrations of a few tenths of one percent and other metallic impurities (such as boron, chromium, copper, nickel, titanium, zinc, etc.) at concentrations of up to several hundred parts per million. The impurity content in metallurgical silicon is excessive for device purposes. The current process used in semiconductor industry for the purification of metallurgical silicon is tedious and expensive. Several simple techniques have been developed for the partial purification of metallurgical silicon. They include: (A) the treatment of molten silicon with gaseous chemical reagents to selectively remove specific impurities from the melt,⁽⁷⁾ (B) the treatment of pulverized metallurgical silicon with acids,⁽⁸⁾ and (C) the melting of metallurgical silicon under a layer of fused silicate to getter various impurities,⁽⁹⁾

It has been claimed that molten silicon could be purified by treatment with a mixture of halogen (or a halogen derivative) and oxygen, and that

3 kg of technical grade silicon (98%) was purified to 99.99% purity in only 3 minutes by the reaction with an equimolar mixture of oxygen and hydrogen chloride. The chemical treatment technique has been studied in detail by passing chlorine, hydrogen chloride, or chlorine-oxygen mixtures through molten metallurgical silicon until about 10% of the silicon had been reacted and volatilized.⁽⁵⁾ This treatment has been found to be effective in reducing the concentration of impurities, such as aluminum and titanium, which are chemically more reactive than silicon. However, impurities less reactive than silicon, such as copper and iron, cannot be removed in this manner. These results are in line with thermodynamic data and do not support the effectiveness of chemical treatment reported earlier.⁽⁷⁾ This discrepancy could be due to the improper sampling technique used for chemical analysis; the solidified material after chemical treatment is highly inhomogeneous due to the small segregation coefficient of impurities in silicon and the use of a small amount of sample could result in a considerable error. The low segregation coefficients of most metallic impurities in silicon suggest that unidirectional solidification is an effective technique for the removal of these impurities from metallurgical silicon. A combination of chemical treatment and unidirectional solidification has been used for the purification of metallurgical silicon. The chemical analysis of chlorine-treated plus unidirectional solidified metallurgical silicon indicate that the concentrations of aluminum and iron in the bottom half of the charge are reduced substantially, 10-100 ppm.

The chemical treatment and unidirectional solidification techniques

are energy intensive, not economical for large scale operations, and the acid-extraction technique, a low temperature purification technique, has been investigated. Although many metallic impurities such as iron, aluminum, copper, gold, nickel, titanium, etc., are highly soluble in molten silicon, their solubilities in solid silicon are relatively low. For example, the solubilities of iron, copper, and aluminum in silicon at 850°C are approximately 2×10^{15} , 7×10^{16} , and 10^{19} cm^{-3} , respectively, and are lower at lower temperatures.⁽¹⁰⁾ Thus, during the solidification of molten metallurgical silicon in the manufacturing process, a major portion of the low-solubility impurities should precipitate at grain boundaries or at interstitial positions. When the cast metallurgical silicon blocks are pulverized, the breakage occurs mostly at grain boundaries. The small grains, 1 mm for example, are either single crystalline or are aggregates of several single crystals. Therefore, a major portion of the metallic impurities are concentrated at the surface of the grains, and the treatment of pulverized metallurgical silicon with acids should be effective in removing these impurities as well as the impurities at grain boundaries. By repeated refluxing of pulverized metallurgical silicon with aqua regia, the concentration of iron, the major impurity, has been reduced to 600 ppma or below.

The fact that acid treated metallurgical silicon still contains an appreciable concentration of iron indicates that metallic impurities also precipitate within the grains. To further remove these impurities, acid-treated metallurgical silicon was heated with phosphorus pentoxide in air

at 1050°C for several days. After removing the phosphorite glass and refluxing with aqua regia, the iron content in silicon has been found to be 200-250 ppma. However, a relatively high concentration of phosphorus is introduced into metallurgical silicon during the gettering process.

II. 2 Preparation of Substrates

The preparation of metallurgical silicon substrates is the most important processing step in the fabrication of thin film silicon solar cells. The chemical and structural defects in the substrate greatly affect the properties of the deposited silicon layer, the active region, and therefore the characteristics of the solar cell. A unidirectional solidification technique has been developed to yield metallurgical silicon sheets on graphite plates with relatively large crystallites. Briefly, this technique is as follows. The purified metallurgical silicon is placed on a graphite plate with a sandblasted surface of 5 cm x 15 cm area; the roughened surface is used to increase the graphite-molten silicon interfacial tension. This assembly is then placed in a fused silica tube of 65 mm ID and heated externally by an rf generator. The spacings between the turns of the rf coil are adjusted to yield a unidirectional temperature gradient of 50-80°C along the length of the specimen. The entire specimen is first melted, and the input power reduced for the solidification to take place from one end of the specimen to the other.

The rate of solidification is the most important parameter in the unidirectional solidification technique. At relatively high rates of solidification, 3-5 cm/min, the solidification initiates from the surface

of the melt and proceeds inwards to minimize the orienting effects of polycrystalline graphite. Under proper thermal conditions, crystallites as long as 10 cm have been obtained. However, the surface of the substrate is not planar but has ridges, faces, and valleys. At low rates of solidification 0.5-1 cm/min, planar surfaces are obtained. By adjusting the thermal conditions to maintain a flat solid-liquid interface, elongated crystallites of several centimeters in length have also been obtained. In both type of substrates, multiple twinning, grain boundaries, and dislocations are the most common structural defects.

The stationary unidirectional solidification technique described above requires the melting of the entire silicon charge and is not suitable for large scale operations. A zone-melting technique has also been used. In this technique, a molten zone of silicon was formed by using closely-spaced turns of a small section of the rf coil, and the remainder of silicon charge was heated at 1000° - 1200° C. The silicon/graphite assembly was pulled at a rate of 0.5-1 cm/min to allow the molten zone to traverse through its length. Planar surfaces similar to that of unidirectional solidified substrate prepared at similar rates were obtained. However, the vibration of the melt associated with uneven pulling must be improved to minimize the formation of growth steps.

Because of the use of phosphorus pentoxide treatment, all substrates are n-type with a resistivity of 0.02-0.03 ohm-cm. The iron distribution in the substrate along the direction of solidification has been found to be essentially uniform. Although considerable segregation of iron is expected, the high diffusion coefficient of iron in silicon is presumably

responsible for the observed distribution.

II. 3 Deposition of Silicon Films and Solar Cell Structures

Silicon films of controlled thickness, conductivity type, and dopant profile were deposited on metallurgical silicon substrates by the thermal reduction of trichlorosilane containing appropriate dopants. The deposited films are epitaxial with respect to the substrate by metallographic examinations.

Potential probe measurements indicate the presence of a potential barrier at grain boundaries. The magnitude of the potential gradient at a grain boundary decreases with decreasing electrical resistivity of the film and becomes negligible in films of 0.01 ohm-cm resistivity.

Solar cells of the configuration n^+ -silicon/p-silicon/ p^+ -metallurgical silicon (acid treated)/graphite have been prepared by the trichlorosilane process. The low resistivity substrate serves to minimize the contact resistance between silicon and graphite and also provides a back-surface field. The conductivity type and electrical resistivity of deposited silicon were controlled by using diborane or phosphine as a dopant, and the p-n junction was formed during the deposition process by varying the composition of the reactant mixture. Typically, 20-30 μm of 0.2-1 ohm-cm p-type silicon was deposited at 1100⁰-1150⁰C at a rate of about 1 $\mu\text{m}/\text{min}$ followed by the deposition of 1-2 μm of low resistivity n-type silicon of uniform or graded dopant concentration at a rate of about 0.1-0.2 $\mu\text{m}/\text{min}$ (the graded profile was used to provide a drift field). The sheet resistance of the n^+ -layer, measured by the four point probe technique, was 30-50 ohms/square. The graphite plate serves as the ohmic contact to the p-region.

The grid contact to the n^+ -surface was made by evaporating about 1000 Å⁰ of titanium and 3-5 μm of silver to the cell surface through a metal mask, followed by annealing in hydrogen at 525°C. Subsequently, about 700 Å⁰ of tin oxide was deposited on the surface by the oxidation of tetramethyltin at 400°C as an antireflection coating.

Solar cells of the configuration p^+ -silicon (uniform or graded)/ n -silicon/ n^+ -metallurgical silicon/graphite have been prepared in a similar manner. In some cases, the solar cells were subjected to a prolonged heat treatment at 500-600°C and a brief heat treatment at 850-950°C before further processing. In nearly all cases, the solar cell subjected to heat treatment exhibits better electrical characteristics than the one without heat treatment. It is possible that during the heat treatment, the dopant diffused from the surface region along grain boundaries into the base region to form a p-n junction at the boundaries. Thus, the grain boundary is converted into a charge collection boundary, and the minority carriers reaching the grain boundary are collected by the junction rather than lost by recombination. The best solar cell (9 cm² area) has an open-circuit voltage, short-circuit current density, and fill factor of 0.575 V, 23 mA/cm², and 74%, respectively, under AMI illumination, corresponding to a conversion efficiency of 9.75%.

III. Preparation Of Substrates And Deposition Of Silicon Films

The technical approach used in this program is the same as that developed at SMU, i.e., the deposition of a silicon film containing a p-n junction on a metallurgical silicon substrate. The preparation of metallurgical silicon substrates with low concentrations of chemical and structural defects and the deposition of silicon films of controlled thickness and dopant profile are most important parameters affecting the characteristics of solar cells. Efforts during this quarter have been directed to the design and construction of apparatus required for these processes.

III. 1 Preparation of Substrates

Extensive studies at SMU has concluded that the zone-melting technique using a moving rf coil is best suited for the recrystallization of metallurgical silicon on graphite. Thus, a moving coil arrangement shown schematically in Figure 1 has been designed and constructed. The separation between the turns of the rf coil can be adjusted at will. The closely spaced section of the coil is used to produce a molten zone of 3 cm-5 cm width, and the remainder of silicon is heated at 1000⁰-1200⁰C. The coil powered by a 25 kw rf generator can be moved at rates from 1 cm/min to 10 cm/min. The fused silica tube is of 95 mm ID and 101 mm OD to accommodate a substrate of 7.5 cm width and 28 cm length. Process parameters of this moving coil technique for the preparation of metallurgical silicon substrates are under study.

Fig. 1 Schematic diagram of the apparatus for the preparation of metallurgical silicon substrate by the moving-coil zone-melting technique.

III. 2 Deposition of Silicon Films

The thermal reduction of trichlorosilane with hydrogen is the most economical process for the deposition of silicon films. An apparatus for the deposition of silicon films by the trichlorosilane process as shown schematically in Figure 2 has been designed and constructed. The flow of various gases is directed by appropriate valves and measured by flowmeters. Trichlorosilane is kept in a double-wall stainless steel cylinder provided with gas inlet and outlet tubes, and a constant temperature fluid is circulated through the jacket of the container to maintain the temperature of trichlorosilane at 15°C. Trichlorosilane is introduced into the reaction tube by bubbling hydrogen through the liquid. Hydrogen is purified by diffusion through a palladium silver alloy. By varying the flow rate of hydrogen through trichlorosilane, deposition rates in the range of 0.2 $\mu\text{m}/\text{min}$ to 2 $\mu\text{m}/\text{min}$ can be readily obtained. Two cylinders of phosphine, 500 and 10 ppm in hydrogen, and two cylinder of diborane, 100 and 25 ppm in hydrogen, are used as the n- and p-type dopants, respectively, so that resistivities in the range of 0.003 - 1 ohm-cm can be obtained. The deposition of silicon is carried out in a fused silica reaction tube of 95 mm ID and 101mm OD. The silicon/graphite substrate, 7.5 cm x 28 cm, is heated externally by an rf generator. The effects of various process parameters are under study.

Fig. 2 Schematic diagram of the apparatus for the chemical vapor deposition of silicon by the thermal reduction of trichlorosilane.

IV. Acid Extraction Of Metallurgical Silicon

The results of SMU studies indicate that acid-extraction is the most cost-effective technique for removing a major portion of metallic impurities from metallurgical silicon. The effectiveness of this technique depends on the particle size of metallurgical silicon, the temperature and duration of the treatment, and the chemical reactivity of the acid.

A series of experiments has been carried out to investigate the relative effectiveness of various acids, e.g., aqua regia, an equivolume mixture of sulfuric acid and nitric acid, and hydrochloric acid. Metallurgical silicon of 30-80 mesh size with an iron concentration of 0.35% was used. Typically, 1 kg of pulverized metallurgical silicon was refluxed with 1 liter of the acid in a 3 liter flask, and the acid was changed every six days for 4-5 times. The concentration of iron in each acid-extracted solution and in the resulting silicon was determined by the spectrophotometric analysis. The data obtained by using aqua regia and a sulfuric acid-nitric acid mixture are shown in Figure 3. It is seen that the concentration of iron in the extract decreases rapidly with each extraction. Also, aqua regia is more effective than the sulfuric acid-nitric acid mixture. After four extractions, the concentration of iron in silicon is 750-850 and 550-650 ppma for sulfuric acid-nitric acid and aqua regia extraction, respectively. It is believed that the iron can be more readily removed and the iron concentration further reduced by using metallurgical silicon of smaller particle size.

Fig. 3 Concentration of iron in solution after successive extractions of pulverized metallurgical silicon with acids.

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VII Plan For The Next Period

1. Continuation of the purification of metallurgical silicon
2. Preparation of metallurgical silicon substrate by the moving-coil zone-melting technique
3. Deposition of solar cell structures by the thermal reduction of trichlorosilane containing appropriate dopants .
4. Fabrication and characterization of solar cells

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