Purity and Minority Carrier Lifetime in Silicon Produced by Direct Electrolytic Reduction of SiO₂ in Molten CaCl₂

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ABSTRACT
Si powder was produced by direct electrolytic reduction of SiO₂ in molten CaCl₂ at 1123 K. From the Si powder, Si ingots were obtained by a floating zone method. The concentrations of most metallic elements and of P in the Si ingots were lower than the acceptable levels for solar grade Si. The minority carrier lifetimes in the Si ingots were measured using a microwave photo conductivity decay method. The obtained values of ca. 1.0 µs were two orders of magnitude shorter than those observed in an Si ingot prepared from 10N purity Si.

Keywords: Electrolysis, Molten Salt, Silicon, Purity

1. Introduction
As a clean and inexhaustible source of renewable energy, solar energy has attracted a great deal of attention and has many applications in daily life and industry.1-4 The photovoltaic (PV) industry has undergone rapid development in the past decade. The global annual installed capacity of PV cells reached 50.6 gigawatts in 2015, which represents a 100-fold increase over the capacity in 2000.5-6 Therefore, high yield production processes are required for solar-grade Si (SOG-Si, 99.9999% purity), which is the dominant raw material used for solar cells. In the conventional SOG-Si production process, metallurgical-grade Si (MG-Si, 99% purity) is first produced by high-temperature carbothermal reduction of SiO₂ at approximately 2000 K. Then, MG-Si is converted into trichlorosilane (SiHCl₃) gas and purified by distillation. Finally, SOG-Si is obtained by the Siemens-C process, which involves chemical vapor deposition (CVD) of the purified SiHCl₃ gas. 7-9 However, this process, especially the Siemens-C process, has the drawbacks of low productivity and low energy efficiency, and is environmentally hazardous.

From this starting point, we proposed the direct electrolytic reduction of solid SiO₂ in molten CaCl₂ at the relatively low temperature of 1123 K.10-26 In contrast to traditional molten salt electrolysis, the cathodic reduction of SiO₂ proceeds through a solid-state reaction. We performed a series of studies in which we investigated the reduction of SiO₂ plates,10,11 sintered SiO₂ pellets,12 and stratiﬁed SiO₂ granules,12-26 and clariﬁed the reaction mechanisms involved. The purities of the obtained Si powders were also measured, as shown in Table 1. Although relatively high purity was achieved, the impurity levels in our previous studies did not reach the acceptable levels for SOG-Si. In addition, the semiconductor properties of the Si powder were not measured, because the amount of powder obtained was insuﬃcient to produce Si ingots.

In the present study, a greater amount of Si powder was prepared by the electrolytic reduction of SiO₂ granules with a scaled-up working electrode in molten CaCl₂. Subsequently, Si ingots were produced from the obtained Si powder using a floating zone method. The impurity concentrations of the Si powder and ingot were analyzed. Finally, Si wafers were cut from the Si ingots, and the minority carrier lifetimes were measured using a microwave photo conductivity decay method and compared with those of Si wafers prepared from commercial 5N Si powder and 10N Si rods.

2. Experimental
2.1 Electrolysis
Figure 1 shows a schematic illustration of the electrolysis cell. Approximately 20 g of SiO₂ granules with a size of less than 100 µm (Taiheiyo Cement Corp., 4N purity) were placed at the bottom of a Si crucible (Furuchi Chemical Corp., o.d. 105 mm, i.d. 90 mm, height 150 mm, 7N purity). Next, ca. 700 g of CaCl₂ (Kojundo Chemical Lab. Co., Ltd., 99%) was added to the Si crucible. Before undergoing electrolysis, the SiO₂ and CaCl₂ in the Si crucible were first dried under a vacuum at 453 K for 24 hours inside a quartz vessel, and further dried at 773 K for 48 hours to remove residual moisture. The Si crucible had sufﬁciently high electric conductivity at 1123 K to be used as the working electrode (W.E.). A Pt wire (Tanaka Kikinzoku Kogyo, Co., Ltd., diameter 1 mm, 99.95%) threaded through an Al₂O₃ tube (Nikkato Corp., o.d. 2.5 mm, i.d. 1.5 mm, length 160 mm, 99.6%) was used as an electric lead to the Si crucible. To avoid the formation of a Pt–Si alloy, a graphite plate (Nilaco Corp., thickness 0.5 mm, 5N purity) was inserted beneath the Si crucible.

A glass-like carbon rod (Tokai Carbon Co., Ltd., diameter 5.2 mm, 99.9998%) was used as the counter electrode (C.E.). An Ag wire (Japan Metal Service, Co., Ltd., diameter 1 mm, 4N purity) was used as a current lead for the C.E. in Experiment 1 (Sample 1), and a Pt wire (Tanaka Kikinzoku Kogyo, Co., Ltd., diameter 1 mm, 99.95%) was used in Experiment 2 (Sample 2). The reference electrode (R.E.) was an Ag⁺/Ag electrode prepared by immersing an Ag wire (Japan Metal Service, Co., Ltd., diameter 1 mm, 4N purity) into molten CaCl₂ containing 0.5 mol% AgCl (Wako Pure Chemical Co., Ltd., 99.5%) contained in a mullite tube (Nikkato Corp., o.d. 6 mm, i.d. 4 mm, length 450 mm, 96%). A Mo wire (Nilaco Corp., diameter 1.0 mm, 99%) was used for the measurement of the Ca²⁺/Ca potential. The potential of the Ag⁺/Ag reference electrode was calibrated with reference to the Ca²⁺/Ca potential.
The unreduced SiO2 was removed using hydrofluoric acid (Wako Pure Chemical Co., Ltd., 35%), followed by washing with distilled water, and then with hydrochloric acid (Tama Chemical Co., Ltd., 38%). The impurity concentration of the obtained Si powder after the washing treatments was analyzed using glow discharge mass spectrometry (GD-MS, VG Scientific Co., Ltd., VG–9000).

Table 1. Impurity concentrations in the obtained Si powders and Si ingots analyzed by GD-MS, with the impurity residual ratios calculated from the Si powder to the Si ingot. For reference, the results of previous studies18–20 acceptable levels for SOG-Si27–29 and segregation coefficients of Si30–33 are listed.

| Element | Powder | Ingot | Previous studies18–20 | Acceptable levels for SOG-Si27–29 | Impurity residual ratio after the floating zone method | Segregation coefficient30–33 |
|---------|--------|-------|----------------------|-------------------------------|-----------------------------------------------|--------------------------|
|         | No. 1  | No. 2 | No. 1  | No. 2 | No. 1 | No. 2 | No. 1 | No. 2 | No. 1 | No. 2 | No. 1 | No. 2 | No. 1 | No. 2 |
| B       | 270    | 150   | 1.8    | 2.1   | 1.3   | 0.1–0.3 | 6.7 × 10⁻³ | 1.4 × 10⁻² | 8 × 10⁻¹ |
| P       | 3.7    | 5.6   | 0.40   | 0.14  | 0.39  | 0.03–0.14 | 1.1 × 10⁻¹ | 2.5 × 10⁻² | 3.5 × 10⁻¹ |
| Ca      | 7000   | 7500  | 830    | 400   | 520   | <10      | 1.2 × 10⁻¹ | 5.3 × 10⁻² | 7 × 10⁻² |
| Al      | 820    | 3400  | 2.7    | 0.61  | 19    | <0.1     | 1.3 × 10⁻³ | 2.5 × 10⁻⁵ | 1 × 10⁻² |
| Ti      | 28     | 86    | 0.44   | <0.1  | 1.2   | <1 × 10⁻³| 3.3 × 10⁻³ | 1.8 × 10⁻⁴ | 2 × 10⁻³ |
| Cr      | 19     | 38    | <0.1   | <0.1  | <0.1  | <0.1     | <5.3 × 10⁻³| <2.6 × 10⁻³| 3 × 10⁻⁵ |
| Fe      | 120    | 240   | 1.8    | <0.1  | 1.1   | <0.1     | 1.5 × 10⁻² | 4.2 × 10⁻⁴ | 8 × 10⁻⁶ |
| Mn      | 1.3    | 3.3   | 0.33   | <0.1  | <0.1  | <0.1     | 2.5 × 10⁻¹ | 3.0 × 10⁻² | 1 × 10⁻⁵ |
| Ni      | 24     | 54    | <0.1   | <0.1  | <0.1  | <0.1     | <4.2 × 10⁻³| <1.9 × 10⁻³| 3 × 10⁻⁶ |
| Cu      | 1.0    | 0.66  | <0.1   | <0.1  | <0.1  | <0.1     | <1.0 × 10⁻¹| <1.5 × 10⁻¹| 4 × 10⁻⁴ |
| Mo      | 83     | 170   | <0.1   | <0.1  | <0.5  | <1 × 10⁻⁴| <1.2 × 10⁻³| <5.9 × 10⁻⁴| 4.8 × 10⁻⁸ |
| Ag      | 930    | 73    | <0.1   | <0.1  | 51    | <0.1     | <1.1 × 10⁻⁴| <1.4 × 10⁻³| 1 × 10⁻⁶ |
| N       | 150    | 160   | 420    | 210   | —     | —        | 2.8 × 10⁰  | 1.3 × 10⁰  | <1 × 10⁻⁷ |
| O       | 3100   | 2600  | 220    | 80    | —     | 10–20    | 7.1 × 10⁻² | 3.1 × 10⁻² | 5 × 10⁻¹ |
| F       | 42     | 330   | 0.42   | 0.14  | —     | —        | 1.0 × 10⁻² | 4.2 × 10⁻⁴ | —       |
| Na      | 300    | 14    | 2.5    | 0.38  | —     | —        | 8.3 × 10⁻³ | 2.7 × 10⁻² | —       |
| Mg      | 54     | 520   | 0.33   | <0.1  | —     | —        | 6.1 × 10⁻³| <1.9 × 10⁻⁴| 3.2 × 10⁻⁶ |
| S       | 16     | 20    | 2.4    | 1.6   | —     | —        | 1.5 × 10⁻¹ | 8.0 × 10⁻² | 10⁻⁵   |
| Cl      | 210    | 360   | 7.6    | 2.0   | —     | —        | 3.6 × 10⁻² | 5.6 × 10⁻³ | —       |
| K       | 29     | 4.3   | 0.2    | 0.35  | —     | —        | 6.9 × 10⁻³ | 8.1 × 10⁻² | —       |

Bold entries indicate values are within the acceptable levels for SOG-Si.

2.2 Preparation of Si ingots and measurement of minority carrier lifetimes

Figure 2 depicts a schematic of the floating zone method. Approximately 2 g of the Si powder obtained after the washing treatment was loaded into a rubber case (Canon Machinery Inc., diameter 8 mm, length 70 mm), and then pressed into a Si rod with a diameter of approximately 7 mm by isostatic pressing under a pressure of 60 MPa for 1 minute. The prepared Si rod was installed as a feed rod inside a quartz tube in the floating furnace (Canon Machinery Inc., Desk top type A), as shown in Fig. 2(a). The feed rod was hung from the top holder by a Pt alloy wire (Furuya Metal Co., Ltd., GS-FPO, diameter 0.3 mm), and a seed Si rod prepared by a compact Si rod (Furuuchi Chemical Corp., diameter 5 mm, 10N purity) was fixed to the bottom holder. The feed Si rods were lowered together in an inverse Ar stream with a variable speed under manual operation since the feed Si rod was fragile. After the first melting and adjustment of the position of the Si rods, the Si rods were again lowered together in an inverse Ar stream with a uniform speed of 3 mm h⁻¹ by automatic operation. For comparison, Si ingots were also prepared from commercial Si powder (Strem Chemicals Inc., <5 μm, 5N purity), which is denoted as Sample 3, and a commercial Si rod (Furuuchi Chemical Corp., diameter 5 mm, 10N purity), denoted as Sample 4. The impurity concentrations of the Si rods were analyzed using GD-MS. Table 2 summarizes the preparation conditions for Samples 1 to 4.

The electrochemical experiments were carried out at 1123 K in a dry Ar atmosphere with a flow rate of 300 mL min⁻¹. The potentiostatic electrolysis of SiO2 granules was conducted at 0.6 V vs. Ca²⁺/Ca for 96 hours. In this condition, no formation of Ca–Si alloy was confirmed in the previous studies.21,25 After electrolysis, the products and the solidified CaCl₂ in the crucible were washed with distilled water, and then with hydrochloric acid (Wako Pure Chemical Co., Ltd., 35–37%), followed by filtration. The unreduced SiO2 was removed using hydrofluoric acid (Tama Chemical Co., Ltd., 38%). The impurity concentration of the obtained Si powder after the washing treatments was analyzed using glow discharge mass spectrometry (GD-MS, VG Scientific Co., Ltd., VG–9000).

Figure 1. Schematic illustration of the electrolysis cell.
Technology Inc., Model 650), as shown in Fig. 2(b). According to a procedure previously reported in the literature, the wafers were first etched using a mixed acid (HF:HNO$_3$:CH$_3$COOH = 3:5:3, volume ratio) for 10 minutes, then etched with another mixed acid (HF:HNO$_3$:CH$_3$COOH = 4:10:35, volume ratio) for 2 hours, and finally washed with diluted hydrofluoric acid (HF:H$_2$O = 1:1, volume ratio) for 20 minutes to remove the surface SiO$_2$ layer. The surface of the Si wafers was observed using an optical microscope before and after etching. After immersing the Si wafers in a solution of iodine in ethanol (0.05 mol L$^{-1}$), the minority carrier lifetimes were measured using a microwave photoconductivity decay (µ-PCD) method (Kobelco Research Institute, Inc., LTA-1600SP).

3. Results and Discussion

3.1 Electrolytic reduction of the stratified SiO$_2$ powder

Figure 3 shows the time-current curves measured during the electrolysis. The dashed curve represents Sample 1, in which an Ag lead wire was used for the counter electrode, and the black curve corresponds to Sample 2, in which a Pt lead wire was used. For Samples 1 and 2, the average currents are 0.541 A and 0.454 A with total charges of 1.87$x10^5$ C and 1.57$x10^5$ C, respectively. These total charges correspond to 145% and 123% of theoretical values for the reduction of 20 g SiO$_2$. During the first 24 hours, the reduction current is approximately 0.6–0.7 A for both samples. After this time, the reduction current of Sample 1 became unstable due to corrosion of the Ag wire by Cl$_2$ gas generated during the electrolysis. The Cl$_2$ generation was confirmed by reaction in a chemical trap connected to the gas outlet, and this phenomenon was also reported in the previous study. In contrast, the reduction current of Sample 2 are stable due to the corrosion resistance of Pt for chlorine and decreases gradually to 0.179 A at the end of the electrolysis. After electrolysis, Si was found at not only the bottom but also on the sides of the Si crucible. The Si generated at the bottom was directly reduced from solid SiO$_2$. The Si deposited on the sides was reduced from dissolved SiO$_2$, because SiO$_2$ is soluble in CaCl$_2$ at 1123 K at high concentration of O$^2-$ ions introduced by the cathodic reaction. The weights of the obtained Si were 4.12 g and 3.50 g for Samples 1 and 2, respectively. Considering the total charges, the current efficiencies for both Samples 1 and 2 were 30.3%.

3.2 Impurity concentrations in the obtained Si powder and Si ingots

Photographs of the obtained Si powder, Si ingot, and Si wafer are shown in Figs. 4(a), (b), and (c), respectively. The color of the Si powder after washing is ash black. The surface of the Si ingot is glossy. The surface of the Si wafer before etching is somewhat rough.

Table 1 lists the impurity concentrations in the Sample 1 powder, Sample 1 ingot, Sample 2 powder, and Sample 2 ingot. Here, the impurity residual ratio after the floating zone method, which is defined as (impurity concentration in the ingot)/impurity concentration in the powder), is also listed for each element. For reference, Table 1 also includes the impurity concentrations achieved in our previous studies, the acceptable impurity levels for SOG-Si, and the segregation coefficients for the various elements at the melting point of Si.
To display this data in a more visually understandable manner, Fig. 5 shows a plot of the concentration of each element. The impurity concentrations in the obtained Si powders, which are represented by black circles (Sample 1) and black triangles (Sample 2), are higher than the acceptable levels for SOG-Si, which are indicated with blue dots. The concentrations of C, Ca, Al, and O are greater than 1000 ppmw, and the concentrations of B, Fe, Ag, Mo, N, F, Na, Mg, and Cl are greater than 100 ppmw in the Si powder. The impurity concentrations in the obtained Si powders are higher than those achieved in our previous studies, which are marked with by the X symbols in Fig. 5. One possible reason for the higher levels of impurities in the present study is the longer electrolysis time, which could introduce more metal impurities due to the corrosion of cell components such as the stainless steel lid, lead wires (Ag and Pt), and working electrode (Mo).

However, the impurity concentrations decrease greatly in the Si ingots, which are represented by red circles (Sample 1) and red triangles (Sample 2) in Fig. 5. The concentrations of P, Ca, Cr, Fe, Mn, Ni, Cu, and Ag are achieved or even lower than the acceptable levels for SOG-Si which are represented by blue dots in Fig. 5. For most elements, the impurity residual ratios are larger than the segregation coefficients, as shown in Table 1. This phenomenon can be attributed to nonequilibrium solidification.

3.3 Minority carrier lifetimes in the Si wafer

Figure 6 shows the optical images of the surface of the Sample 2 wafer (a) before and (b) after etching. The surface becomes lustrous after etching. As can be seen in the local microscopic image of Fig. 6(c) and the simulated 3D image of Fig. 6(d), the surface is covered with craters with lengths of approximately 10–50 µm and depths of less than 5 µm. This morphology is similar to that observed by Osinniy for metallurgical multicrystalline SOG-Si. A similar luster and similar surface morphology were observed for other samples in the present study.

The minority carrier lifetimes in the Si wafers were measured using a µ-PCD method, and are summarized in Fig. 7. The measurements were conducted at random locations on the samples, and for several samples, a wide variation in the lifetime was observed. This can be explained by the different crystallite sizes in polycrystalline Si. The maximum carrier lifetimes are 1.0 and 0.64 µs for Samples 1 and 2, respectively. For comparison, the maximum carrier lifetime of Sample 3, which was prepared from 5N Si, reaches 8.7 µs. This value is 10 times longer than those of Samples 1 and 2. Meanwhile, for reference, the maximum carrier lifetime of Sample 4, which was produced from 10N purity Si, is 54.9 µs. These results confirmed that carrier lifetimes increase with increasing Si purity. A longer carrier lifetime, i.e., a higher purity, is still required for the Si samples prepared by electrolysis. Our next goal is to achieve a carrier lifetime of ca. 10 µs, with the ultimate goal being ca. 100 µs. To achieve this, a longer quartz vessel and a lower moving speed of floating zone would be effective.

4. Conclusion

20 g of SiO2 powder was reduced to Si by direct electrolytic reduction in molten CaCl2 at 1123 K. More than 4 g of Si powder was obtained with a current efficiency of 30.3%. Si ingots were produced from the obtained Si powders by a floating zone method. The impurity concentrations in the obtained Si powders were higher than those achieved in our previous studies. However, the concentrations of most impurities in the Si ingots were lower than those in the Si powders in our previous studies. The concentrations
of most metallic elements and of P in the Si ingot were below the acceptable levels for SOG-Si. The minority carrier lifetime in the Si wafer prepared from the obtained Si powder reached 1 µs. This value was one order of magnitude shorter than the carrier lifetime in the Si wafer made from 5N Si. In order to improve the carrier lifetime, higher purity is required for the electrochemically produced Si powder.

Acknowledgments

This study was partly supported by JST-CREST, Japan Science and Technology Agency and Scientific Research (A), Japan Society for the Promotion of Science. The minority carrier lifetime measurement by a µ-PCD method was supported by assistant professor Kenji Ishizaki, Graduate School of Engineering, Kyoto University.

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