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Recovery of Silicon from Silica Fume

M. Barati*, S. Sarder, A. McLean, R. Roy

University of Toronto, Dept. of Materials Science and Engineering
140 – 184 College Street, Toronto, ON
Canada M5S 3E4
Tel: (416) 978 – 5637
Fax: (416) 978 – 4155
Email: mansoor.barati@utoronto.ca

Feasibility of producing high purity silicon from amorphous silica fume, using a low temperature magnesiothermic reduction was demonstrated. Commercial silica fume containing 97.5% amorphous silica was first purified by acid leaching and roasting to remove large quantities of transition metals and carbon. The product was then reduced using magnesium as the reductant. Effect of the amount of reductant, initial temperature, and dwell time were investigated on the quantity and type of the reaction products. The optimum reduction conditions were decided based on the maximum yield of the Si metal. These corresponded to Mg/SiO₂ molar ratio of 2.0, preheating temperature of 750 °C, and holding time of 2 hours. High purity silicon (>99 wt%) containing <3 ppmw B and 12 ppmw P was obtained after leaching–purification of the reduction products, showing that the material is superior to metallurgical grade silicon, for use as solar–grade silicon feedstock.

1. INTRODUCTION

Silica fume, sometimes referred to as Microsilica^(R)¹, consists of very fine amorphous SiO₂ particles and is generated as a by-product in silicon metal and ferrosilicon manufacturing processes.

Reactions (1) to (3) summarize the basic steps involved in the formation of silica fume. During the carbothermic reduction of silica to silicon in the electric furnace (Reaction 1), silicon monoxide gas is also produced at the process temperatures typically exceeding 1800 °C (Reaction 2). Silicon monoxide escapes the furnace with other gases and is oxidized upon mixing with air to form fine particles of silica (Reaction 3). Condensed silica that is typically non-crystalline is then collected with other dust and condensed particles in the gas cleaning system.

A silicon furnace produces between 200–400 kg of silica fume per tonne of silicon metal, [1, 2], representing 8–15% of the quartz feed. The material has typically a specific surface area (SSA) of 15–30 m²/g, particle size smaller than 1 µm, and SiO₂ content greater than 85 wt% [3]. Because of such characteristics, silica fume exhibits pozzolanic properties, making it a suitable addition to high strength concretes [4], where it is primarily used. The present work aims at recovering high purity silicon from silica fume, as an alternative use of the material, and also investigating the suitability of the resultant silicon as feedstock for producing solar grade silicon (SoG–Si).

¹ Microsilica is the trade name of silica fume by Elkem

In the past decade, the PV industry has experienced a growth rate of over 30%, increasing the demand for SoG–Si substantially. A variety of processing routes are being investigated to achieve the required purity of 6N (99.9999%) while keeping the cost below \$15/kg [5]. One of the approaches towards production of SoG–Si is utilizing high purity starting materials to reduce the contamination of the silicon product. Amongst the materials investigated are ultrapure quartz [6] and rice husk ash [7-10] and a variety of reductants including carbon black, magnesium and calcium.

Formation of silica fume from the gas phase suggests that it may not be significantly contaminated with those impurities that are not highly volatile at the furnace temperature. Therefore, it is expected that silica fume could be a high purity source for SoG–Si feedstock. A mass balance study by Myrhaug and Tveit [11] confirms that the distribution of various elements between silica fume and silicon metal is largely consistent with their boiling temperatures. In particular, only 25% of phosphorus and 5% of boron ended up in the off–gas. Moreover, it is anticipated that the majority of the impurities can be removed from silica, by a simple acid leaching step, on the assumption that they are likely to condense at temperatures different from the oxidation of silicon monoxide, and are thus not intimately mixed with SiO₂ particles. In order to investigate the potential use of silica fume for the production of high purity silicon, the current research was undertaken. A hybrid process consisting of hydrometallurgical and pyrometallurgical steps was developed on a laboratory scale. The process steps and optimized conditions of each will be discussed in this article.

2. EXPERIMENTAL PROCEDURE

The silica fume of Norwegian origin was first characterized to determine physical properties as well as chemical composition. The major components were quantified by X–ray

fluoroscopy while the trace elements were measured by ICP–MS analysis. X–ray diffraction (XRD) was performed to determine the crystal structure of the major components and the total carbon content was quantified using a LECO analyzer. The material was also subjected to particle size analysis by Laser diffraction (using a Malvern S Mastersizer) and the specific surface area was determined with the BET method [12], using a SA3100 Surface Area and Pore Size Analyzer (Coulter).

The process for extraction of Si from silica fume relies on three major treatments; purification of silica fume, conversion of purified SiO_2 to silicon (*i.e.* reduction), and recovery of Si from the reduction products.

Figure 1 depicts the detailed process flow diagram. The purification consists of two sub-processes; acid leaching for removing the soluble impurities, followed by high temperature roasting for evaporation and combustion of the volatile matter and carbon. The leaching purification was carried out by dispersing SF in a 10 wt% hydrochloric acid solution for 1–4 hours at 60 and 90 °C. The leach residue with minimum overall impurity was selected for roasting. The solids were heated to 700 °C in a muffle furnace in the presence of air and maintained at this temperature for 2 hours.

Figure 1. Process flowsheet for production of SoG-Si from silica fume .

For reduction of purified silica fume, metallic reductants were preferred over carbon to lower the temperature of the reduction and also avoid contamination of the silicon product with the impurities associated with carbon. Low temperature reduction of SF is beneficial in that operation is easier, also the large surface area of the SiO_2 particles are preserved so that the product will remain responsive to subsequent leaching treatment. Magnesium was selected as the reductant by considering factors such as cost, availability, ease of removing the reduction by-products, and the effect of residual metal on silicon properties. To determine the minimum temperature required to activate the reactions, a 60 mg mixture of magnesium and silica fume was heated to 1150 °C at a uniform rate of 10 °C/min, in a differential thermal analyzer (Netzsch

STA-409). The reduction onset temperature was identified by a sharp heat release peak at around

620 $^{\circ}\text{C}$ (

Figure 2).

Figure 2. DTA spectrum for a mixture of silica fume and Mg.

For the reduction experiments, mixtures of purified silica fume and Mg granules of 98% purity (Sigma Aldrich, Canada) with appropriate ratios were prepared. Batches of 2 g were loaded into an alumina tube that was inserted inside a steel capsule. The reaction cell was then placed inside a muffle furnace and heated at the rate of 5 °C/min to the target temperature. After a pre-set duration, the furnace was cooled to room temperature. The reaction products were analyzed by XRD to establish the mineralogical composition and the quantity of each phase was determined by processing the XRD patterns using the Rietveld method [13].

The recovery of Si from the reaction products required dissolution of the by-product phases including MgO, Mg₂SiO₄, Mg₂Si, and un-reacted silica. This was achieved by two stages of leaching. In the first stage, most of the magnesium-containing compounds were removed by hydrochloric acid. The effects of HCl concentration (1, 2, and 4M), temperature (30–60°C) and

reaction time (10–60 minutes) were investigated on the extraction kinetics of Mg compounds. Acetic acid (CH_3COOH) was added at a fixed concentration of 25wt% to enhance the wetting behaviour of the fine particles, by lowering the surface tension of the aqueous solution. The second stage of acid leaching was carried out to remove any SiO_2 and remaining Mg_2SiO_4 , using a mixture of HF (4.8 wt%) and acetic acid (25 wt%) for 1 hour at 70 °C. The recovered silicon was thoroughly rinsed with de-ionized water and digested in a mixture of HF and HNO_3 for analysis by ICP–AES.

3. RESULTS AND DISCUSSION

3.1. Characteristics of silica fume

Table 1 presents the chemical analysis of as-received silica fume used in this work. For comparison, the analysis of rice husk ash from a parallel study is provided. The silica fume is of higher purity and is therefore a more suitable feed material for generation of SoG–Si.

Figure 3a) shows a bell–shape distribution with mean particle size of 9 μm . Specific surface area measurement indicated a large specific surface area of 17.8 m^2/g . This is

advantageous for surface-sensitive treatment processes such as acid leaching. X-ray diffraction

analysis (

Figure 3b) provided a spectrum with a broad peak around $2\theta=20^\circ$, confirming the amorphous structure of the material.

Table 1. Chemical analysis of silica fume and rice husk ash

Component	Unit	Silica Fume	Rice Husk Ash [10]
P	ppmw	294	1600
B	ppmw	<2	51
Al	ppmw	2094	4000
Fe	ppmw	1603	2400
K	ppmw	7265	7900
Ca	ppmw	1161	3300
Mg	ppmw	3004	1600
Mn	ppmw	93	294
C	wt%	0.50	1.87
SiO₂	wt%	>97	91.5

Figure 3. (a) Particle size distribution and (b) XRD spectrum of as-received silica fume.

3.2. Purification of silica fume

3.1.1. Leaching Treatment

The majority of the impurities associated with silica fume such as phosphorus, aluminum, iron, calcium, magnesium, sodium and potassium exist as oxides, although because of their low concentration their representative peaks may not be revealed by XRD analysis [14]. It is anticipated that some of the oxide impurities can be removed by acid leaching. The efficiency of this treatment was evaluated by normalizing the impurity concentration with respect to the initial

concentration, and comparing the value for various conditions (

Figure 4). The results indicate that there is reduction in the concentration of majority of the impurities.

Figure 4. Effect of leaching treatment on removal of impurities.

3.1.2. Roasting

The leach residue with minimum impurity level was roasted as described earlier. A relatively low temperature of 700 °C was selected to maintain the amorphous structure of silica. On roasting, the material changed from a greyish colour to white, while carbon was reduced from 0.5 wt% to 0.17wt%.

The purification process increased the SSA to 27.2 m²/g, due to the formation of micro-pores on the particles caused by acid leaching and combustion of carbon. The mean particle size remained the same.

3.3. Reduction

The reduction experiments included investigating the effects of temperature (750–950 °C), hold time (1–3 hours) and magnesium to silica molar ratio (1.5–3.0) in order to establish the

conditions that yield the maximum amount of silicon. The reaction products typically contained elemental silicon, magnesia (MgO), and magnesium silicide (Mg_2Si) as the major phases together with minor amounts of Mg_2SiO_4 and SiO_2 . The X-Ray diffractograms for various experimental conditions were obtained and later quantified using the Rietveld method. As an example, the XRD results for samples with various ratios of Mg to silica (heated to 750 °C and held for 2 hours) are displayed in

Figure 5. As seen from the relative heights of the peaks, the formation of MgO, Mg_2Si and silicon is affected by the ratio of Mg/SiO₂. For example, when the ratio is 3:1 Mg_2Si peaks are very prominent, whereas by reducing Mg, their relative intensity is decreased. Clearly, a decrease in the amount of magnesium silicide gives rise to producing larger amounts of Si. The Si yield was calculated from the quantified XRD results and used as a measure for the optimum reduction conditions, as will be discussed later.

Figure 5. Effect of magnesium/silica ratio on the XRD spectrum of samples reduced at 750°C for 2 hours.

3.4. Recovery of silicon from reaction products

The recovery of silicon from reaction products consisted of a two-step acid leaching to remove magnesium-containing compounds and unreduced silica. Both MgO and Mg_2Si are soluble in HCl ; magnesium silicide reacts with HCl solution and produces silane gas [15] that combusts spontaneously in the presence of air. On the other hand, MgO is readily dissolved in dilute HCl and remains in the solution as MgCl_2 . The effect of concentration of HCl on the

dissolution of a mixture of magnesium compounds was investigated and the results are shown in

Figure 6. **Error! Reference source not found.**The highest dissolution of magnesium compounds was observed for 2M HCl solution followed by 1M and 4M.

Figure 6. Dissolution of magnesium compounds with hydrochloric acid. Leaching temperature was 60°C.

Although the leaching in hydrochloric acid proved to be efficient in removing MgO and Mg₂Si, XRD analysis of the leach residue revealed the presence of forsterite (Mg₂SiO₄) and silica. The second stage of leaching in a mixture of HF+CH₃COOH under the previously discussed conditions removed these components.

3.5. Characteristics of the silicon product

A representative sample of the final silicon product after the above treatments is shown in

Figure 7. Size analysis of the powder shows particles within the range 0.2–250 μm with a mean size of 56 μm . Clearly, the Si grains are larger than the starting silica fume particles, which is because of agglomeration of the particles during the reduction step. The exothermic nature of the reduction reaction can raise the temperature of the products well above the sintering temperature of silicon.

Figure 7. Silicon powder produced from silica fume.

Table 2 presents the ICP–AES analysis of a typical silicon sample obtained after acid purification. The overall purity is >99.0 wt% Si, while some critical impurities such as phosphorous, boron and arsenic are favourably low, 12, <3, and 1 ppmw respectively.

Table 2. Chemical analysis of the Si product

Elements	Concentration	Elements	Concentration
B	<3 *	Ba	6
P	<100* / 12**	Mg	3637
Ca	1041	Cr	9
Fe	1203	Mo	10
Al	2255	Mn	150
Ni	40	Zn	67
K	1420	V	1
As	1	Cd	3

* BDL: Below detection limit, **: Separate analysis using ICP–MS

4. DISCUSSION

4.1. Effectiveness of Primary Leaching–Roasting Purification

Figure 4 presents the ratio of impurity concentration for several elements before and after the leaching treatment. A smaller than unity ratio indicates removal of the element, the smaller the ratio, the greater the removal. As illustrated in this figure, some elements such as P, Pb, and As are removed to a large extent; Fe, Mg, Na, and K are partially removed, while Al and Ti were not responsive to the treatment. The different response of various elements to acid leaching may be related to their respective condensation behaviour. While a large group of impurities (Fe, Mg, Al, etc.) are oxidized and condensed immediately after the mixing of off-gas with air, precipitation of the oxides of P, Pb, and As is delayed, due to their high vapour pressure (low boiling point). As a result, the former group of oxides are dispersed within the SiO₂ particles while the latter group form a layer on the surface of the particles and become more responsive to

the leaching treatment. Leaching at 90 °C for 4 hours provided maximum purification under the studied experimental conditions although the difference with other conditions was not substantial. Therefore, for economic reasons one may choose to perform the leaching at the milder conditions of 60 °C for 1 h.

As discussed earlier, the final carbon content of the silica fume was relatively large, 0.17 wt% after roasting at 700 °C. It is believed that this unburnt carbon is in the form of silicon carbide, as otherwise it would have been readily oxidized under the experimental conditions.

4.2. Optimum reduction conditions

The yield of silicon, defined as the percent of initial Si (as SiO₂) reduced to Si metal, was used as the measure for the determination of the optimum reduction conditions. Although the mechanism of the reduction is complex, involving gaseous Mg and reduction by Mg₂Si as an intermediate phase, the following overall reactions are considered for the formation of Si, MgO, Mg₂Si, and Mg₂SiO₄.

Formation of magnesium silicide should be minimized as it lowers the Si yield and increases the consumption of magnesium. Although magnesium silicide is not thermodynamically stable in the presence of SiO₂, its formation is due to kinetic limitations that prevent complete reduction of silica. Therefore, it is a combination of the kinetic factors (time and temperature) together with availability of Mg that leads to various amounts of Mg₂Si. The effects of these variables are discussed in the following sections.

4.2.1. Effect of Mg/SiO₂ ratio

The Si yields calculated from the quantified XRD results show that a maximum is reached at a Mg/SiO₂ ratio of about 2:1 or the stoichiometric proportion, as depicted in

Figure 8a. This is because at a lower ratio, Mg is not adequate to reduce SiO₂ completely, whilst at Mg/SiO₂>2.0, excess magnesium consumes Si to form Mg₂Si (Reaction 5). This is

validated in

Figure 8b that shows formation of larger amounts of magnesium silicide when Mg/SiO₂ exceeds 2.0, for all temperatures.

Figure 8. Effect of magnesium/silica ratio and temperature on (a) Si yield and (b) Mg₂Si amount.

4.2.2. Effect of temperature

It is evident from

Figure 8a that for mixtures with Mg/SiO₂ below or equal to the stoichiometric ratio (2.0), temperature has a relatively small effect on the Si yield. On the other hand, when the ratio exceeds this value, the effect of temperature becomes significant, yielding more silicon at higher temperatures.

Figure 8b reveals that this is because of a decrease in amount of Mg₂Si formed at higher temperatures. Due to the accelerated kinetics of the reactions at higher temperatures, reduction by Mg₂Si (Reaction 7) that requires solid state reaction or diffusion can take place to a greater extent, giving rise to more reduction of SiO₂ at the expense of Mg₂Si. This is also consistent with

the larger amounts of Mg_2Si at lower temperatures (

Figure 8b).

From

Figure 8a, it appears that for the highest Si yield, the optimum reduction temperature and Mg/SiO₂ ratio are 750 °C and 2.0 respectively.

4.2.3. Effect of hold time

For several samples with fixed Mg/SiO₂=2.0, the hold time at 750 °C was varied from 0.5 to 3 hours. It was found that the Si yield increases from 55% at 0.5 hour to 90% at 2.0 hours and then tends to plateau for longer times. The increase in Si yield is associated with further completion of the reduction reactions that at some stages are controlled by solid state diffusion. Extending the time beyond 2 hours however, does not appear to significantly improve the yield because of the near complete reduction of SiO₂ within the first two hours.

4.3. Kinetics of leaching of Mg compounds

4.3.1. Effect of Acid Concentration

It was shown in

Figure 4 that the 2 M HCl leachant was more effective than the 1 M and 4 M solutions in dissolving the Mg compounds from the reaction products. The decrease in the rate of transfer of the magnesium to the solution observed with 4M HCl concentration could be because of two effects (a) insufficient availability of hydrolyzed water to dissolve Mg compounds and (b) a quick reaction in the beginning that causes formation of an inhibiting Mg-saturated layer around the particles.

4.3.2. Effect of Temperature

Figure 9 illustrates, temperature has a positive effect on both the extent and the rate of extraction of magnesium compounds. An analysis of the results showed that the leaching kinetics is consistent with mass transfer of Mg in the solution acting as the rate limiting step of the overall

dissolution process. The dependence of the rate constant on temperature (

Figure 10) showed an activation energy in the range of 13–15 kJ/mol for various acid concentrations, confirming that the transport of Mg into the solution controls the dissolution rate.

Figure 9. Effect of leaching temperature on dissolution of magnesium compounds (2M HCl).

Figure 10. Temperature dependence of the rate constant.

4.4. Suitability of the Si product as SoG–Si feedstock

The chemical analysis of the produced Si in Table 2 presents much lower impurity levels than the metallurgical grade silicon, that is conventionally used for SoG–Si generation and contains typically 25–50 ppmw phosphorus and 6–50 ppmw boron [16].

Figure 11 **Error! Reference source not found.** shows correlation of solid solubility and segregation coefficient for different impurities in silicon [17]. Impurity level in the silicon powder produced in the present study is also superimposed on this figure (square symbols). It shows that for some of the most deleterious impurities such as As, P, and B, the concentration is well below their solubility in Si. For other impurities such as Mg, Al, Fe, etc, the concentrations are substantially greater than the solubility limits. This indicates that these elements are most likely present along the grain boundaries or at the surface of the silicon grains. Therefore, they

can be removed by melting, regrinding, and leaching of Si or controlled solidification methods such as unidirectional solidification. Furthermore, as seen in Table 2, the majority of the elements in high concentration (Ca, Al, K, Mg) are those that form oxides more stable than silicon. It is thus anticipated that a simple oxidative treatment such as slag refining [18] can effectively eliminate these elements to levels required for SoG–Si application.

Figure 11. Impurity level in the silicon powder produced in this study (■) and maximum solid solubility (○) of various elements in Si (collected from [19-26])

5. CONCLUSIONS

1. Characteristics of silica fume including large surface area, high silica content, low boron concentration and small particle size indicate its suitability as feedstock for production of high purity silicon.
2. Leaching of silica fume for 4 hours at 90 °C by 10% HCl leachant proved more effective for impurity removal than lower temperatures and leaching time examined. Elements with low boiling point are more responsive to leaching.

3. Under the experimental conditions of this research, the optimum conditions for reduction by magnesium of silica fume include: Mg/SiO₂ molar ratio of 2.0, temperature of 750 °C, and hold time of 2 hours
4. Silica fume is a suitable feedstock for the generation of SoG-Si. Through the multi-step process of this work, Si with overall purity of over 99% with low P, B, and As content was produced.
5. The major impurities present in the Si product are Ca, Al, Mg, and K that can be readily removed by metallurgical refining techniques.

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