

SiO- generation for H₂ reduction with SiH₂ process

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Abstract The SiH₂ process is a concept for production of silicon by H₂ as reductant. The idea is to produce SiO gas by reaction between SiO₂ and internal recycled Si. Produced SiO is reduced by H₂ to Si and H₂O. The two reactions take place at different temperatures. To realise the proposed concept, knowledge about the kinetics for the involved reactions is needed. Reaction between SiO₂ and Si to produce SiO is discussed here. In the experiments, flow of warm SiO gas from the reaction has been measured continuously by a new method. This method shows that the reaction rate varies with time and have a peak with very high SiO generation that start as the temperature reach around 1750 °C to 1800 °C and then reduces rapidly until a lower rate is reached. Effects on reaction SiO₂ + Si ⇌ 2SiO of various other parameters such as gas composition, gas flow rates, temperature and properties of SiO₂ and Si source is also discussed.

1. INTRODUCTION

The SiH₂ project proposes a novel process concept, SiH₂ process shown in Figure 1 where hydrogen substitutes carbon as reductant resulting in Si-production with no CO₂ emissions (Ringdalen et al., 2024).

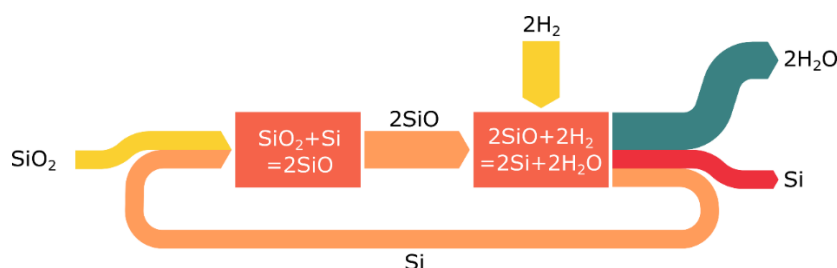
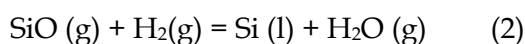


Figure 1 Concept for SiH₂ process.

The process is based on two main reactions. First SiO-gas is produced by reaction (1) between liquid Si and solid or liquid SiO₂. Produced SiO is then reduced by hydrogen by reaction (2) to water (H₂O) and Si. Half of the produced Si is recirculated back to be used as reductant for production of SiO from SiO₂ by reaction (1). Remaining produced Si go to the final product. If other Si-sources are used for reaction (1), more of the produced Si will end up in the final product. Final Si production by reaction (2) require that SiO gas is available as reactant. It is thus crucial for the process that SiO produced by reaction (1) do not react back to SiO₂ and Si by the condensation reaction, reaction (3). This is the reverse of reaction (1).

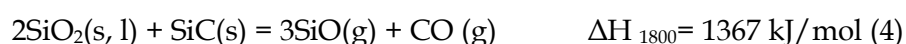


Reaction (1) and reaction (2) will at a pressure of 1 atm not be in equilibrium or run to left within the same temperature range. Production of SiO by reaction (1) in reasonable amounts will take place above around 1750 °C, close to the melting point of SiO₂, while reaction (2) between SiO and H₂ will take place below around 800 °C. Production of Si by this process will thus require either to find conditions where SiO can be produced by reaction (1) with lower temperatures, conditions where SiO can be reduced by H₂ by reaction (2) at higher temperatures, or conditions where SiO can be cooled down to a temperature where it can be reduced with H₂ according to reaction (2) before it reacts back to SiO₂ by reaction (3). Based on the known slow kinetics for reaction (3) (Schei et al., 1998),(Broggi A, 2021), cooling of produced SiO gas is regarded as the most probable alternative. Production of Si-H compounds instead of Si is another alternative.

Revealing the potential for the SiH₂ process require knowledge of all the involved reactions and how they are affected by different parameters. Reaction (1) is an important part of existing Si-production. It is also important for producing SiO to be used in investigations of reactions involving SiO. Results from different studies not targeted towards, but involving reaction (1) between SiO₂ and Si are here summarised together with studies of reaction (1) done as part of the SiH₂ project

2. SiO generation from reaction between SiO₂ and Si, background.

The main reaction studied here is reaction (1), production of SiO gas from SiO₂ and Si. This is an endothermic reaction with $\Delta H_{1800\text{ °C}} = 598 \text{ kJ/mol}$. Produced SiO gas is stable only at high temperatures and will react back to SiO₂ + Si during cooling(Broggi A, 2021). The reaction is mostly studied at conditions where the gas phase contain CO-gas in addition to SiO, as is the case in industrial Si-production. Since there are limited data available about SiO-generation from SiO₂ + Si in a carbon free system, data from investigations in a system with carbon and at 1 atm total pressure is included in the discussion. The system will then include both SiO-generating and SiO consuming reactions. The most important are



The equilibrium content of SiO in a CO-SiO gas mixture at a total pressure of 1 atm at different temperatures is given by Figure 2

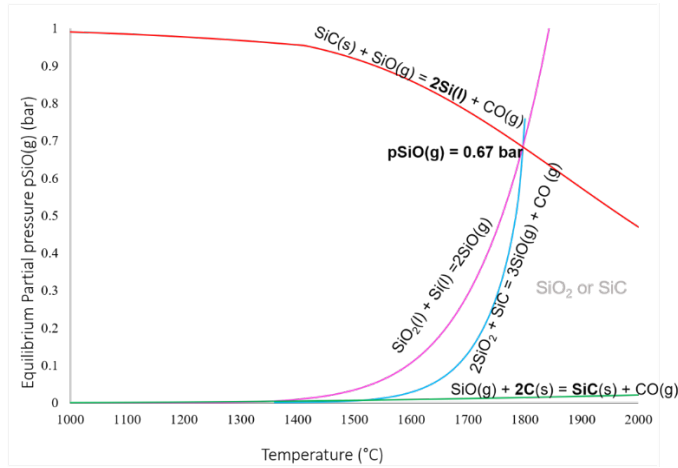


Figure 2 Stability diagram showing equilibrium pSiO for different reactions of importance for the Si process (Jayakumari and Ringdalen, 2022) The gas phase is a mixture of SiO+CO with a total pressure of 1 atm. Adapted from (Schei et al., 1998).

This illustrates that the stable partial pressure of SiO above a mixture of SiO₂+ Si is very low at temperatures below around 1650°C but increase fast with increasing temperature. In a system containing carbon, produced SiO can be consumed by reactions with carbon. SiO generated above around 1500 °C, reacts to SiC according to reaction (5) at low partial pressures of SiO. Produced SiC can react further with SiO₂ according to reaction (4) producing SiO. Above a temperature of 1800 °C and a partial pressure of SiO between around 0.5 to 0.67 bar, SiO reacts with SiC to Si according to reaction (6) Si. For the SiH₂ process where the aim is a carbon free system, reaction (4) to (6) will not take place. Since most of the investigations of reaction between SiO₂ and Si are done in a carbon containing system, they and especially the consumption of produces SiO will be an important part of the evaluation of the obtained results.

SiO generation from SiO₂ + Si by reaction (1) has been found (Andersen, 2010) (Bao et al., 2013) to have a slow reaction rate at 1550 °C. The rate increased with temperature with a slight decrease around melting point for SiO₂, followed by a fast increase in rate at temperatures above the melting point. Sindland (Sindland and Tangstad, 2021) found that the rate of reaction (1) increased with reaction area and temperature. It was not affected by quartz source. The rate could be expressed by equation (7). Reaction area is corrected for melting of SiO₂ by an agglomeration factor F described by (Sindland and Tangstad, 2021).

$$\frac{d\alpha}{dt} = 6.25 \times 10^8 A \exp\left(-\frac{557 \times 10^3}{RT}\right) \quad (7)$$

A similar expression was found (Tangstad et al., 2019) for rate for SiO-generation by reaction (4) between SiO₂ and SiC. Kinetic parameters as activation energies and frequency factors found in different other investigations are summarised in the paper. Comparison of rate for reaction (1) and reaction (4) by Sindland (Sindland and Tangstad, 2021) show that with the same reaction areas, the rate for SiO generation from the two reactions are within the same range, but that SiO generation from SiO₂ + Si is faster than SiO generation from SiO₂ + SiC at all temperatures with largest difference at the highest temperatures. Rate for SiO-generation by reaction (4) SiO₂+ SiC was in the investigations by Tangstad (Tangstad et al., 2019) and by Folstad (Folstad et al., 2021) found not to be affected by ratio between the different SiO₂ polymorphs for the investigated quartz source.

Physical and chemical properties of quartz and their changes during heating vary between commercial available quartz sources (Ringdalen, 2015). This might have an effect on both design and operating conditions for reactors for SiO generation from reaction between SiO₂ and Si and also for investigations of this reaction. Parameters that are found to vary are thermal stability, (Ringdalen, 2014) (Jusnes, 2020), rate for transformations to different SiO₂ polymorphs (Ringdalen and Tangstad, 2016), (Jusnes, 2020), (He et al., 2021), volume changes during heating and melting properties (Ringdalen et al., 2020). Molten silica has a high viscosity which can be lowered by content of minor elements in the quartz source. (Dalaker, 2025). Operating conditions as heating rates, gas flow rates and temperature gradients may affect the observed differences between quartz sources. They may also affect agglomeration and segregation between the reacting phases and the rate for SiO-generation.

2. Investigations of reaction between SiO₂ and Si

SiO has been generated by reaction (1) as a part of other studies where this reaction was not the main focus. Results and observations from such investigations will give useful information about reaction (1) between SiO₂ and Si. Parameters, conditions and results from different investigations are here summarised and compared. The presented results are generated as a “byproduct” from other investigations targeted at other objectives and does not systematically investigate reaction (1). Results from the following studies are included: Production of SiO gas from SiO₂+ Si with different partial pressures of SiO for bio carbon investigations (Ringdalen, E and Jayakumari S, 2025), investigations of properties of remaining SiO₂ after reactions between SiO₂ and Si, and investigations of SiO₂ + Si reaction for the SiH₂ process. The presented investigations are done in two different resistance heated graphite tube furnaces for investigations of 10-40 gram samples, with on-line gas measurement. The main difference is that the two-zone furnace has two chambers with individually controlled temperatures and the possibility for on-line measurement of SiO-content in the flow of warm gas between these. Set-up in the furnaces varied between the different investigations and are presented as a part of these.

2.1 Production of SiO gas from SiO₂+ Si with different partial pressures of SiO

Methodology

As a part of an investigation of reactions between SiO and carbon (Ringdalen, E and Jayakumari S, 2025), SiO-gas was produced in the ReSiNa furnace by reaction between SiO₂ and Si by two different set-ups shown in Figure 3. The main difference between these is the larger size of the crucible and higher amount of SiO₂+ Si in series B compared to series A. There is a temperature gradient in the lower part of the crucible of around 15°C/cm. Reported temperature is measured at the top of the graphite sieve. Temperature in the bottom of the Si + SiO₂ bed is around 75-85 °C higher than this. Gas, either Ar or CO was introduced at the top of the graphite sieve. Gas flow varied between the experiments. The materials were heated by 25 °C/min to 1750 °C and held at this temperature for the designated time of 5 or, 15 but most often 30 min and then cooled down to 25 °C. SiO generation was determined through mass loss of the sample.

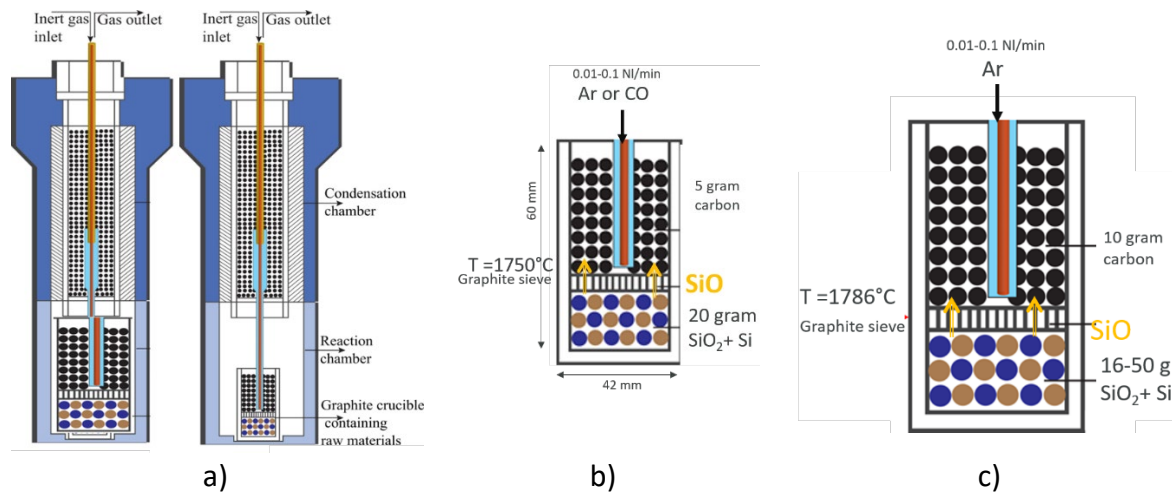


Figure 3 Set-up for investigations of $\text{SiO}_2 + \text{Si}$ reaction to produce different p_{SiO} . (a) furnace set-up, b) Set-up series A, c) set-up series B.

Two different sources of $\text{SiO}_2 + \text{Si}$ was used: 1) SiO granules, which is commercially available 2-4 mm granules of an amorphous mixture of $\text{SiO}_2 + \text{Si}$. Particle size of the individual grains is too small to be seen in EPMA and thus in μm size or smaller. 2) Pieces of $\text{SiO}_2 + \text{Si}$ is a stoichiometric mixture of individual 1-4 mm particles of quartz and pure Si.

The aim of this work was to generate a known partial pressure of SiO from the lower chamber in order to investigate how partial pressure of SiO would affect its reaction with carbon. In series A the aim was to have the same SiO flow from the lower chamber in all the experiments and to obtain different SiO content in the gas in the various experiments by varying the flow of introduced Ar or CO gas. Both total gas flow and SiO-content in the gas are then varied. In Series B, the aim was to have the same total gas flow in all the experiments and to vary the SiO-content in the gas, by vary both flow of generated SiO-gas and flow of introduced gas. Flow of generated SiO-gas was varied by varying amount of $\text{SiO}_2 + \text{Si}$ and thus contact areas between $\text{SiO}_2 + \text{Si}$. Planned flow of SiO was for both cases calculated by the kinetic equation found by Sindland (Sindland and Tangstad, 2021), equation (7). The experiments behind the work of Sindland is done in the apparatus described in Figure 3 with a set up similar to set-up b) with 20 grams of separate 1-5 mm pieces of $\text{SiO}_2 + \text{Si}$. Gas was introduced above the $\text{SiO}_2 + \text{Si}$ bed in both the experiments by Sindland and those discussed here, but in the experiments by Sindland there were no graphite sieve and no carbon bed in the crucible. In the experiments by Sindland the temperature was measured at the bottom of the crucible in contrast to the current experiment where it was measured at the top of the sieve.

Results

Planned and obtained combinations of gas flow and partial pressure of SiO for the current experiments are compared in Figure 4. For Series A, the obtained combination of gas flow and p_{SiO} are close to the planned trend although it is a lot of scatter. SiO-generation and consequently total gas flows are in general lower when CO gas is used instead of Ar-gas. The slightly higher than planned SiO-generation for experiments with Ar is assumed to be a consequence of the different locations for temperature measurement, just above the $\text{SiO}_2 + \text{Si}$ bed in series A and at the bottom of the layer in the experiments by Sindland. In series B all experiments had a higher generation of SiO gas than expected and consequently a higher total gas flow than planned. SiO-generation was lower from pieces of $\text{SiO}_2 + \text{Si}$ than for granules with $\text{SiO}_2 + \text{Si}$.

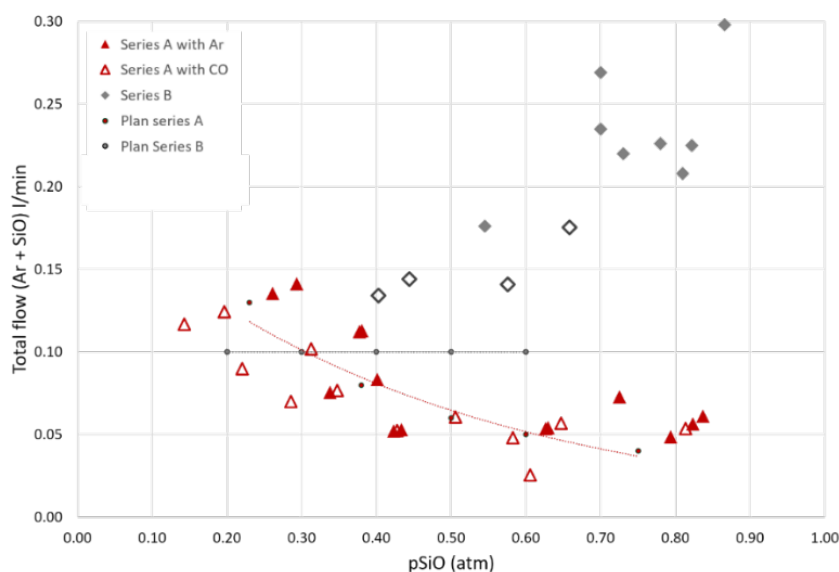


Figure 4 Planned and obtained combinations of gas flows and partial pressure of SiO for the experiments of series A and Series B. In series A markers for experiments with CO instead of Ar gas are not filled. In series B, markers for experiment with pieces of SiO₂ + Si instead of granules are not filled. A trendline is included for the planned combinations. One point in series B (pSiO= 0.93, gas flow= 0.53) is regarded as an outlier and not included.

In the kinetic equation (equation 7) for reaction (1), gas flow is not included as a variable. The varying gas flows used in the experiments will thus not affect the calculated combination of total gas flow and partial pressure of SiO. The relatively good correlation between planned and obtained values in series A indicate that this assumption is valid and that the flow of gas introduced above the Si + SiO₂ bed will not affect the rate for SiO-generation. The lower rate for SiO-generation when CO was used instead of Ar indicates that the gas atmosphere will be of importance for rate of SiO-generation. This should thus be taken into account when the kinetic equation is used in systems with CO-gas, as in industrial furnaces for Si-production. For SiH₂ process that is carbon free, the higher rate for SiO-generation in Ar is expected to be an advantage. The higher than calculated SiO-generation in all experiments in series B shows that for other conditions and other raw materials, another kinetic expression with other parameters must be used.

The relative effects of different variables are compared in Figure 5. Average SiO flow during the experiments were expected to increase with increasing amount of material since this gives a higher total contact area between SiO₂ and Si. This seems not to be the case. For granules of SiO₂+Si, average flow of SiO did not increase when the amount of material was doubled. The comparison of two different amounts of pieces of SiO₂ + Si indicates the same also for this source, although the difference might be within the experimental uncertainty. Both Figure 4 and Figure 5 indicate that SiO-generation from SiO₂ + Si granules are much higher than from separate pieces of SiO₂ + Si. This might be attributed to the very low grain size for particles in the granules of SiO₂+Si giving a higher contact area, or to a higher reactivity of the amorphous SiO₂ in the granules compared to SiO₂ in the larger SiO₂ pieces from quartz origin. Amorphous silica in granules of SiO₂+ Si may also have other melting properties than crystalline SiO₂ in the pieces of SiO₂ and Si. Other explanation might also be possible. The main parameter that affects average SiO flow seems to be reaction time. Average flow of SiO gas is higher when the holding time is shorter. Average flow rate was nearly 3 times higher with 5 minutes holding time than with 30 minutes holding time. This indicates that after an initial high rate for generation of SiO, the reaction will slow and maybe stop completely.

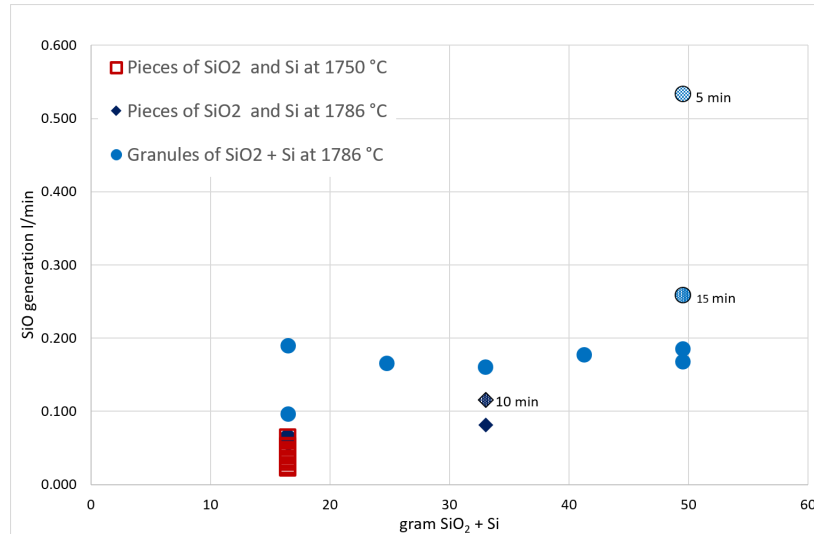


Figure 5 Generation of SiO gas in l/min from different amounts of either pieces of SiO₂ and Si or granules of SiO₂+Si in 1750 or 1786 °C. When nothing else is specified in the figure, the holding time was 30 minutes.

2.2 Investigations of reaction between SiO₂ + Si

Methodology

Reaction between SiO₂ and Si is currently investigated in an on-going work in SiH2 project to study SiO-generation. Results from this will be published separately, but some initial results are presented here. Preliminary investigations of slag formation from quartz, done with the same methodology, generated as a side result information about the reaction between SiO₂ and Si that also will be presented

The experiments were done in the two zone furnace, shown in Figure 6. It has two separately controlled heating zones, both resistances heated. Composition of gas flowing from lower to upper crucible are measured on-line with FTIR (Hestnes Bakke, 2026). In the current work, SiO-content was measured. Composition of cold off gas are measured on-line with μ GC. Changes in material during the experiments are found through visual assessments, weight changes and characterisation by EPMA. In both sets of experiments, the raw materials were a mixture of industrial quartz (Qz42) with more than 99 % purity and PV grade Si. In each experiment 75 gram of a 1:1 molar mixture of SiO₂+ Si was used. In the slag formation experiments, the raw materials had a size of 1-3 mm and in the SiH2 experiments either 0.5-1 mm or 1-1.5 mm. Ar with a flow of 0.2-0.4 l/min was introduced at the bottom of the crucible and flushed through the SiO₂+Si bed. The materials were heated with 50 °C/min to the designated temperature varying between the experiments from 1650 °C to 2000 °C and held at this temperature for the designated time varying between 5 and 30 minutes in the slag formation experiments and 180 minutes in the SiH2 experiments. Temperature difference between bottom and top of the 80 mm high lower crucible was in the newer experiments found to be around 10 °C. The apparatus was improved both between and within the two series. The exact values of data generated in the different experiments are thus not directly comparable, but general trends can be found.

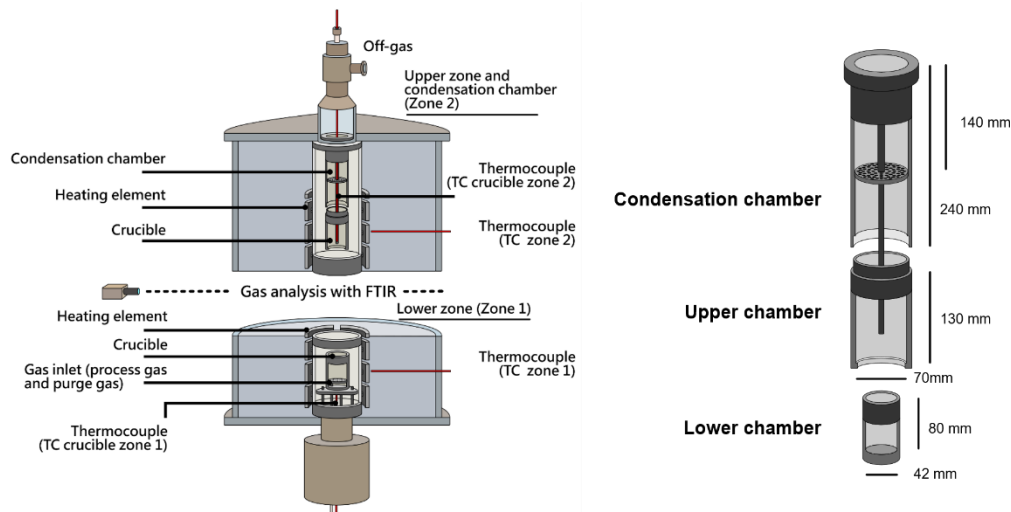


Figure 6 Sketch of two zone furnace. Overview to the left, Crucible sizes to the right.

Results

When the $\text{SiO}_2 + \text{Si}$ mixture is heated to above approximately 1400°C , Si will melt, and above 1750°C SiO_2 will melt. Contact area between the two materials will then change as Si flows down. Examination of the crucibles after the experiments revealed that at 1800°C , Si and SiO_2 was fully separated with a layer of molten SiO_2 above a layer of Si, as seen in Figure 7. This will lower contact area and are thus expected to reduce the reaction rate. Since this is a graphite furnace, carbon is present in the system. A layer of SiC had, as shown in Figure 8, formed around the Si particles in most of the experiments. Such a layer was also seen in the experiments for SiO formations and in the experiments by Sindland. This layer between the SiO_2 and the Si particles is expected to affect the rate of reaction between these.



Figure 7 Crucible after experiment at 1800°C showing segregation with Si at bottom with melted SiO_2 above. Diameter of crucible is 42 mm.

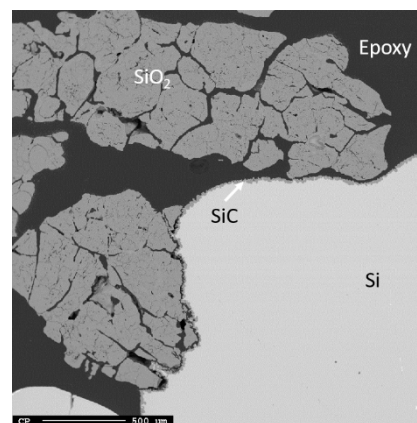


Figure 8 EPMA picture showing SiC formed around the molten Si at the interface between SiO_2 and Si.

Since the only product from the reaction is a gas, weight loss will, if side reactions are disregarded, show amount of reacted material. As expected, this increased with temperature as shown in Figure 9 for preliminary experiments for slag formation and in Figure 10 for SiH_2 experiments. They both show the same trends with very little reaction until the temperature is above 1750°C and that not all material had reacted when the experiments were finished, even

after 3 hours reaction time. The main difference between the two sets of results is the longer holding time for the SiH₂ experiments and the difference in particle size. The different sizes of raw materials used in the SiH₂ process experiments did not seem to affect amount of reacted. The small difference in amount of reacted after 5 minutes and 30 minutes seen in Figure 9 indicate that the reaction rate is much faster in the start and then slow down. This might also explain the relatively small difference between amount of reacted after 30 minutes seen in Figure 9 and after 180 minutes seen in Figure 10.

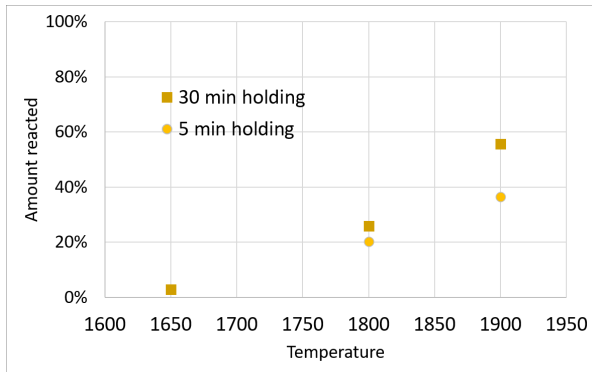


Figure 9 Amount of reacted SiO₂ + Si at different temperatures and holding times in slag formation experiments, 1-3 mm particle size.

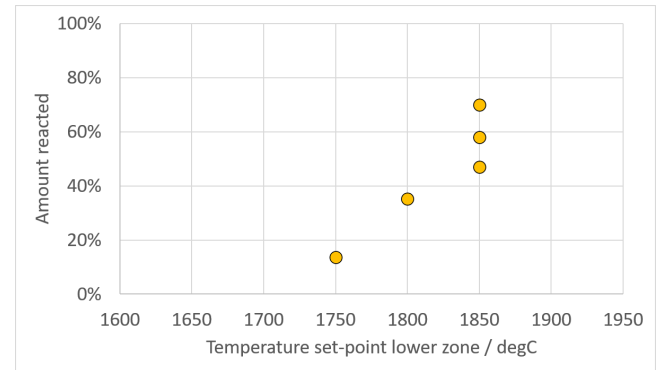


Figure 10 Amount of reacted SiO₂ + Si in SiH₂ process experiments with 180 minutes holding time and 0.5-1 and 1-1.5 mm particle size.

Continuous measurement of SiO formation with time in the slag formation experiments presented in Figure 11 shows that generation of SiO is very fast in the beginning with the highest rates and most of the SiO evolution in the first 10 minutes after the rapid reaction starts. After 15- 20 minutes holding time, there is very little SiO generation indicating that the reaction has nearly stopped. The high rate of SiO generation starts around 1750 °C to 1800 °C. Similar behaviour is also indicated from the on-going SiH₂ experiments. Also, in these there seems to be a peak in SiO generation when a temperature around 1800 °C is reached with a very high generation of SiO in the first 15-30 minutes of the holding time but declining with time in this period.

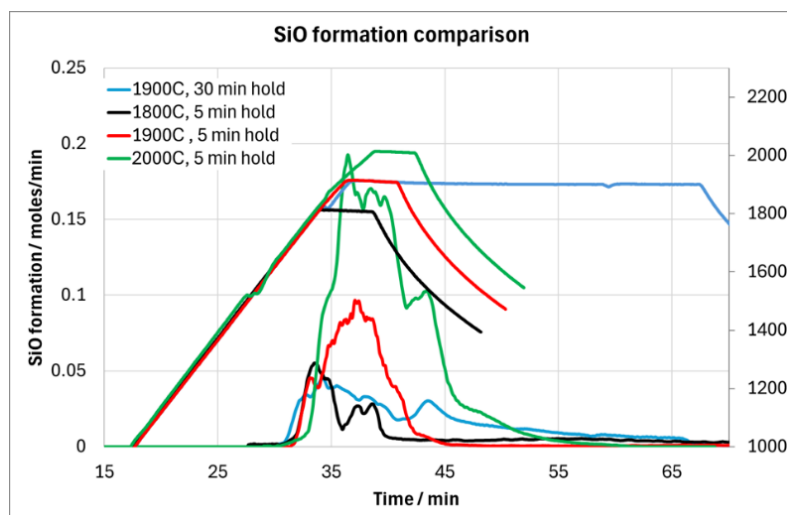


Figure 11 Variation with time in flow of warm SiO gas from reaction between SiO₂+ Si at different temperatures.

3. Discussion

The presented results cover different experimental set-ups, variations in raw material sources and sizes, gas atmospheres and flows as well as reaction temperatures. Based on this, some

general features and effects of parameters on reaction (1) between SiO_2 and Si, can be discussed. There are not enough directly comparable experiments to generate kinetic parameters or to quantify uncertainties in the measurements.

The kinetic equation and parameters for rate for reaction (1) found by Sindland (Sindland and Tangstad, 2021) was found to be valid for reaction between pieces of quartz and Si with a similar set-up to that used by Sindland. It was found also to be valid for different flow rates of gas although this is not a parameter in the equation. This indicates that mass transfer in the gas phase is not rate determining. Substituting Ar-gas with CO gave a lower rate showing that that gas atmosphere can be of importance for the rate of reaction. In further development of the SiH₂ process this can be useful in finding a process design that gives the desired partial pressures of SiO. For SiO_2 and Si pieces a set-up with larger crucibles and more materials gave a higher SiO-generation than predicted in the kinetic equation. This might be within the uncertainty of the experiments or a result of other parameters such as segregation or variation in rate with time.

Segregation between SiO_2 and Si with accumulation of Si at the bottom of the crucible below SiO_2 has been observed in several experiments. This will reduce the contact area between SiO_2 and Si and is thus expected to reduce the reactions rate. The mechanism behind the segregation is believed to be that at temperatures above 1400 °C, Si melts and may start to flow downwards out of the quartz particles while quartz is either solid or acting as a solid due to its high viscosity.

When granules of SiO_2 + Si were used as raw material instead of pieces of quartz and Si, the reaction rate was much higher than predicted. The granules are a 1:1 mol mixture of ultrafine particles of Si and amorphous SiO_2 . The higher reaction rate can either be a result of the ultrafine particle size or a higher reaction rate for amorphous SiO_2 compared to pieces of quartz. At temperatures where SiO_2 is molten, the raw material source is not expected to have any impact. With the slow melting of quartz (Ringdalen et al., 2020), the quartz is expected to be partly molten at 1750 °C, and a difference in reaction rates for different sources at this temperature can not be excluded. Since Folstad (Folstad et al., 2021) found that this is not the case for reaction (4) between SiO_2 and SiC, and Sindland reported that quartz source will not affect reaction between SiO_2 and Si, the higher rate for reaction with granules is expected to be a result of the ultra-fine grain size. This will both give a higher contact area between the phases and most likely lower the downward flow of Si and thus give less segregation. If the case is that amorphous silica gives a higher reaction rate, this can be an advantage for the SiH₂ process. Amorphous SiO_2 as microsilica, could then be a good alternative as SiO_2 source. Higher amount of SiO_2 + Si will give a higher contact area between the two phases and was thus expected to give a higher reaction rate and generation of more SiO. This was as seen in Figure 5 not the case for granules of SiO where SiO generation in l/min was independent of amount of material.

Reaction rate will as shown in the kinetic equation increase exponentially with increased temperature. This was also seen in the experiments shown in Figure 9 and Figure 10. Very little SiO is formed below 1750 °C. Total amount of reacted material depends in addition to the temperature on the holding time. With a constant rate of SiO generation, measured average rate for SiO formation in l/min SiO should at the same temperature be the same independent of holding time. Total amount of reacted will then increase approximately linearly with time. This is not case here. SiO generation in l/min is higher with shorter holding times, and it is as seen in Figure 9 not much difference between amount reacted after 5 minutes and 30 minutes. This indicates a higher reaction rate when the reaction temperature is reached and that this

slows down with time. This is confirmed with on-line measurements of generated SiO as seen in Figure 11. The on-set temperature for the rapid reaction and high generation of SiO is around 1750 °C to 1800 °C. This is assumed to be connected to melting of SiO₂ and its lower viscosity with increased temperature. This has not been investigated and the mechanism behind this change in rate with time is thus not established. The variation in SiO generation with time has an impact on experiments where a known or stable SiO flow is needed for upstream reactions, and for evaluation and modelling of processes where SiO is involved. For design of the SiH₂ process where partial pressure of SiO will affect both condensation of SiO and its further reaction with H₂, the variation in SiO generation with time must be included in the evaluation.

4. Conclusions

The kinetic equation and kinetic parameters for reaction (1) between SiO₂ and Si developed by Sindland (Sindland and Tangstad, 2021) was found to be valid for reaction between pieces of SiO₂ and Si in comparable conditions to the experiments they are based on. Flow of Ar gas is not included in the equation, but this was shown not to affect rate of SiO generation. CO-gas will slightly reduce the rate of SiO-generation. Granules with a 1:1 molar mixture of SiO₂+Si will give a higher reaction rate than predicted. This will not increase with increasing amount of granules. Rate for reaction (1) between SiO₂ and Si is not constant with time for a given temperature. At a temperature between 1750 °C and 1800 °C it will become very fast with a high peak and then decline rapidly during the next 15-30 minutes until the reaction nearly stop. As a consequence of this average SiO-flow for a given period vary strongly with length of the period it is averaged over and will be higher for shorter periods averaged around the peak.

For the SiH₂ process the aim is to first have a high productivity of SiO, then cool the gas down without condensation and finally reduce it with H₂ to Si with a high yield. The partial pressure of SiO is critical in all these parts of the process. Having the knowledge that partial pressure vary considerably with time is thus important for the further development of the process, and methods to mitigate this should be pursued. If the reaction rate for granules is due to its content of amorphous SiO₂, use of amorphous SiO₂ sources such as microsilica could be beneficial for the SiH₂ process.

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