

# Effect of Different SiO<sub>2</sub> Polymorphs on the Reaction Between SiO<sub>2</sub> and SiC in Si Production



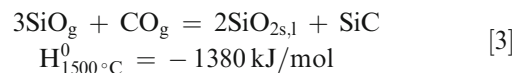
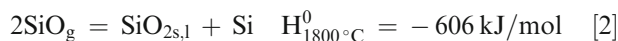
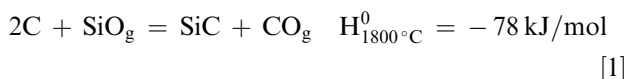
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This work investigates the phase transformations in silica (SiO<sub>2</sub>) during heating to a target temperature between 1700 °C and 1900 °C and the effect of SiO<sub>2</sub> polymorphs on the reduction reaction  $2\text{SiO}_2 + \text{SiC} = 3\text{SiO} + \text{CO}$  in silicon production. Different heating rates up to target temperature have been used to achieve the different compositions of quartz, amorphous silica and cristobalite. The different heating rates had a minor effect on the final composition, and longer time at temperatures > 1400 °C were necessary to achieve greater variations in the final composition. Heating above the melting temperature gave more amorphous silica and less cristobalite, as amorphous silica also may form from  $\beta$ -cristobalite. Isothermal furnace experiments were conducted to study the extent of the reduction reaction. This study did not find any significant difference in the effects of quartz, amorphous silica or cristobalite. Increased temperature from 1700 °C to 1900 °C increased the reaction rate.

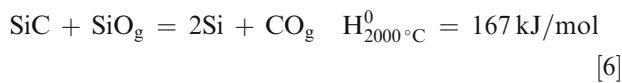
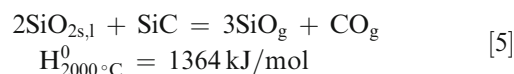
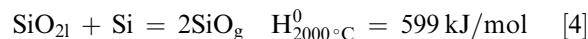
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## I. INTRODUCTION

METALLURGICAL silicon is produced industrially by carbothermic reduction of SiO<sub>2</sub> in a submerged arc furnace. The main reactants are SiO<sub>2</sub> in the form of quartz and carbon (C) in the form of coke, coal, charcoal and woodchips. The overall reaction is  $\text{SiO}_2 + 2\text{C} = \text{Si} + 2\text{CO}_g$ , but the actual reaction process is much more complex. It takes place in several steps with different reactants. These are normally not in equilibrium. The kinetics of sub-reactions, as studied here, is thus important for the overall reactions in the furnace. Silicon carbide (SiC) and silicon monoxide gas (SiO) are necessary intermediate products formed in the furnace, and SiO gas is one of the main variables deciding the silicon yield.<sup>[1]</sup> The raw materials are added at the top of the furnace, and descending SiO<sub>2</sub> and C meet ascending SiO and CO gas. SiC is formed according to Reaction [1], and SiO gas dissociates to Si and SiO<sub>2</sub> according to Reaction [2]. SiO gas will also react with CO gas to produce SiO<sub>2</sub> and SiC as in Reaction [3].



In the high temperature zone, where the temperature exceeds 2000 °C, SiO<sub>2</sub> and SiC will form SiO gas according to Reactions [4] and [5]. Silicon is produced from SiC and SiO gas as in Reaction [6]. The Si yield and thus also the energy consumption of the process are determined by the capture of SiO gas in the outer zone. Unreacted SiO will ascend further up and react with oxygen to form microsilica, which is captured from the off-gas.



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**Table I. Amount of Quartz, Amorphous Silica, Cristobalite and SiC in the Initial Pellets, Given in Wt Pct**

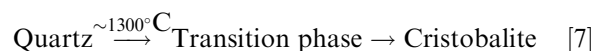
Sample	Quartz [Wt Pct]	Amorphous Silica [Wt Pct]	Cristobalite [Wt Pct]	Sum SiO <sub>2</sub> [Wt Pct]	SiC [Wt Pct]
Initial powder					
1	73.8	1.2	0	75.0	25.0
2	72.4	3.4	0	75.8	24.1

Fig. 1—The 1.25 to 3 mm SiO<sub>2</sub> and SiC pellets after sintering.

An optimized silicon production depends on good understanding of the chemistry in the process. One of the main reactions of the SiO<sub>2</sub> in the furnace is with SiC, and several researchers have studied the reaction rate of Reaction [5] at different temperature intervals.<sup>[2–6]</sup> In addition, the effects of the gas composition and stoichiometry of the reaction are studied, and reaction mechanisms have been proposed. The main goal in this investigation is to study how various polymorphs of quartz affect the formation of SiO from SiC according to Reaction [5]. In industrial furnaces, the amounts of different polymorphs in the sample will depend on the heating profile for quartz in the furnace as well as the kinetics for the reaction. The investigations are thus not based on equilibrium conditions. If SiO gas is produced at lower temperatures higher up in the furnace, it may have an impact on the total silicon yield. When quartz is added to the furnace, it will undergo an immediate temperature increase from room temperature to 700 °C to 1300 °C.<sup>[7]</sup> Furthermore, the quartz will continue to heat as it is descending in the furnace. Pure silica melts at around 1720 °C, but for industrial quartz sources exposed to conditions as in industrial furnaces, found softening and melting temperatures are between 1600 °C and 1800 °C.<sup>[8]</sup> The reaction between SiO<sub>2</sub> and SiC may therefore occur with various polymorphs and different solid, softened and melted stages. Heating of quartz will change its structure into different SiO<sub>2</sub> polymorphs.  $\alpha$ -Quartz transforms into  $\beta$ -quartz at 573 °C.<sup>[9]</sup> This phase transformation is a displacive transition and reversible. The phase transformation from quartz to

cristobalite is a reconstructive phase transformation, meaning that the bonds between the atoms are broken and rearranged. Cristobalite in the high temperature form is a more open structure than  $\beta$ -quartz with densities of 2.20 and 2.53 g/cm<sup>3</sup>, respectively.<sup>[10]</sup> During the transformation from quartz to cristobalite, the volume will increase by about 17 pct.<sup>[8]</sup> The increased volume with an increased reaction area was believed to give a higher reaction rate. Wiik<sup>[11]</sup> found a higher percentage weight loss for cristobalite than quartz in his study with graphite. However, Tangstad *et al.*<sup>[6]</sup> found no significant difference between quartz and cristobalite in their study with SiC. This was also confirmed by Folstad,<sup>[12]</sup> who did not find any difference using both cristobalite formed in a separate study and cristobalite formed in the same furnace experiment.

The transformation from quartz to tridymite is debated, and several researchers have found the transformation from quartz to cristobalite to go *via* an amorphous intermediate state.<sup>[13–17]</sup> Equation [7] shows the suggested transformations.<sup>[14]</sup> The extent of the amorphous phase has been found to vary between different quartz types.<sup>[13,14,18]</sup> Amorphous silica may also form from molten  $\beta$ -cristobalite.



Amorphous silica has no long-range, periodic atomic structure and has weaker bindings. Hakamada *et al.*<sup>[19]</sup> believed that amorphous silica would be more reactive as they found that amorphous silica with graphite was more reactive to forming SiC compared to quartz. The unstructured surface of amorphous silica is expected to give a heterogeneous energetic surface that may have an impact on the reactivity. The quartz undergoes several phase transformations before entering the high temperature part of the Si furnace, which may affect Reaction [5] and the furnace operation.

## II. MATERIALS, EQUIPMENT AND EXPERIMENTAL PROCEDURE

Powder of an industrial quartz, Qz20, with an average initial particle size of 0.4 mm and a purity of 98.9 pct SiO<sub>2</sub> was used in these experiments. Commercial 97.50 pct SiC was used in the form of powder and particles with an average size of 22.8  $\mu\text{m}$  ( $\pm 1.5\mu\text{m}$ ) and size range 0.5 to 10 mm, respectively. Quartz was mixed together with SiC powder in the molar ratio 2:1 (weight ratio 3:1), according to the stoichiometry of

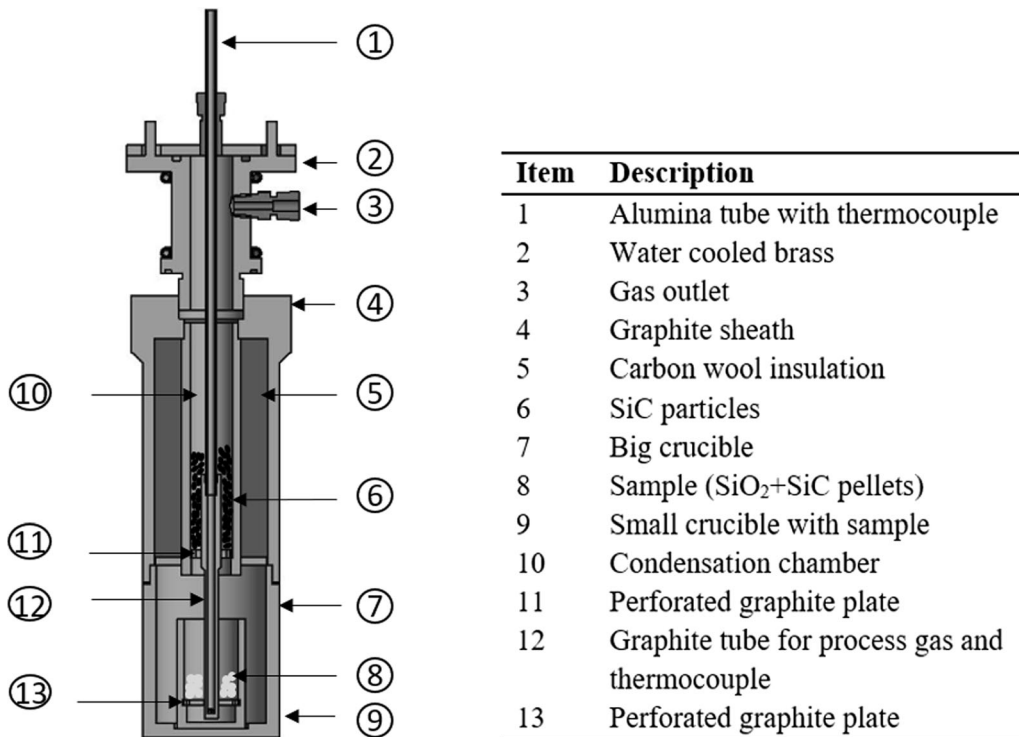


Fig. 2—Schematic figure of the crucibles with reaction chamber and condensation chamber.

Reaction 2.6. Green pellets in the size range of 1.25 to 3.35 mm were made from the powder with a steel-rotating mill using only water as a binder. Since Reaction [4],  $2\text{SiO}_2 + \text{SiC} = 3\text{SiO} + \text{CO}$ , is believed to be very slow at temperatures  $< 1800\text{ }^\circ\text{C}$ , pellets are used to obtain a higher reaction area and hence a higher reaction rate. The pellets were first dried overnight at  $110\text{ }^\circ\text{C}$  to remove any volatile matter and then sintered in a muffle furnace at  $1200\text{ }^\circ\text{C}$  for 15 minutes with a heating rate of  $20\text{ }^\circ\text{C}/\text{min}$  to increase the strength of the pellets by the formation of solid-state bonds. Table I gives the composition found with XRD before heating, and Figure 1 shows a batch of pellets after sintering.

To run the phase transformations in  $\text{SiO}_2$  and to study the reaction between  $\text{SiO}_2$  and  $\text{SiC}$ , a graphite tube furnace was used. A closed graphite crucible is placed inside the furnace, which consists of two main chambers: a reaction chamber and a condensation chamber. The pellets are placed on a perforated graphite plate in a smaller graphite crucible so that the pellet bed is approximately 5 cm high, and the smaller crucible is then placed in the reaction chamber. The process in-gas goes through an alumina tube connected to a graphite tube down to the reaction chamber. In this study, a process gas flow of 0.8 L/min was used, with 100 pct Ar gas during heating and cooling and a mix 25 pct CO and 75 pct Ar at higher temperatures. CO gas is used to create similar conditions as in the industrial furnace, which has  $\sim 33$  pct of this gas. A smaller amount is added to the system since CO gas also forms from Reaction [5]. CO gas was only used at temperatures  $> 800\text{ }^\circ\text{C}$  to avoid the deposit of carbon. Developed SiO gas from the reaction chamber condensates on the SiC particles in the

condensation chamber. Figure 2 shows the schematic drawing of the entire graphite crucible set-up. Fifteen non-isothermal experiments with different heating rates up to the target temperature were conducted to obtain different compositions of  $\text{SiO}_2$  polymorphs. Different heating rates gives different time intervals at the transformation temperatures and were expected to give significantly different amounts of the different  $\text{SiO}_2$  polymorphs. Amorphous silica forms both from quartz and softened/melted  $\beta$ -cristobalite. However, the transformation from quartz to amorphous silica is faster than the transformation from amorphous silica to cristobalite.<sup>[20]</sup> The effect decreases as more quartz is transformed. This would give an initial increasing amount of amorphous silica in the longer time interval with heating  $> 1300\text{ }^\circ\text{C}$  up to  $1700\text{ }^\circ\text{C}$  until a certain point where the transformation rates are more similar. This time interval is not known, and different heating rates must therefore be investigated. When the temperature reaches  $1800\text{ }^\circ\text{C}$  and  $1900\text{ }^\circ\text{C}$ , the  $\beta$ -cristobalite starts to soften/melt and amorphous silica forms from both quartz and  $\beta$ -cristobalite. This gives more amorphous silica than heating to  $1700\text{ }^\circ\text{C}$ . It must also be mentioned again that different quartz types have different transformation rates to amorphous silica and cristobalite. Since the back-reaction from cristobalite to quartz is slow, the number of different polymorphs after cooling is regarded as representative for the amounts at the investigated temperatures, although the samples were not quenched.

The amounts of the different phases present in the pellets were measured using X-ray diffraction. Tridymite was not found in any of the samples analyzed in this study, and it is believed that tridymite needs specific

**Table II. Overview of the Heating Experiments and the Resulting Compositions of Quartz, Amorphous Silica and Cristobalite**

Furnace Parameters			XRD Results		
Temp. [°C]	Heating rate [°C/min]	Time above 1300 °C [min]	Quartz [Wt Pct]	Amorphous silica [Wt Pct]	Cristobalite [Wt Pct]
1700	8	55	48.2	28.7	23.1
1700	16	30	28.1	30.5	41.4
1700	23	22	21.6	35.8	42.6
1700	23	22	43.2	36.1	20.7
1700	28	16	45.2	29.6	25.3
1700	106	9	41.8	38.5	19.7
1800	13	45	0	46.3	53.7
1800	23	29	3.8	39.7	56.4
1800	34	22	13.5	43.9	42.5
1800	34	22	13.5	47.5	37.1
1800	90	13	20.8	58.2	21.1
1800	90	13	20.9	53.1	25.9
1900	32	27	21.4	60.3	18.3

**Table III. Overview of Isothermal Experiments Performed**

Temp [°C]	Heating Rate [°C/min]	SiO <sub>2</sub> Polymorphs			Exp.	Weight Loss Pellets [Pct]
		Quartz [Wt Pct]	Amorphous Phase [Wt Pct]	Cristobalite [Wt Pct]		
1700	106	41.8	38.5	19.7	22	35.61
					24	39.48
					31	32.80
1700	23	21.6/43.2	35.8/36.1	42.6/20.7	9	38.71
					23	37.62
					28	28.89
1700	8	48.2	28.7	23.1	27	40.26
					34	29.89
					35	29.07
1800	90	20.8/20.9	58.2/53.1	21.1/25.9	21	77.51
					26	63.34
					32	58.11
1800	34	13.5/15.5	43.9/47.5	42.5/37.1	11	89.62
					19	79.17
					29	63.73
1800	23	3.8	39.7	56.4	10	60.33
					20	89.90
					33	67.84
1900	32	21.4	60.3	18.3	36	88.86
					37	90.13

impurities to be stable.<sup>[13,21]</sup> To be able to measure any amorphous content, the internal standard method was used. Amorphous silica does not have any crystallinity and long-range order, and the quantification of phases becomes more complicated. The internal standard method relies on all crystalline phases being included in the analysis. An exact known amount of crystalline internal standard material is added to each sample. Corundum (Al<sub>2</sub>O<sub>3</sub>) was used as a standard material in these experiments. The pellets were ground to obtain powder using a boron carbide mortar, and Al<sub>2</sub>O<sub>3</sub> was

added with an exact known amount. The ratio of SiO<sub>2</sub>/SiC and Al<sub>2</sub>O<sub>3</sub> was 4:1, and to ensure accurate measurement both the spike material and the pellets were heated separately overnight in a muffle furnace at 250 °C to evaporate any water. The two powders were thoroughly mixed and then prepared in a backloading sample holder for the XRD investigation. Backloading sample holders were used to minimize the preferred orientation in the powder. The software Diffrac. Eva was used to identify which phases were present in the sample. Then, the quantitative analysis of the diffraction

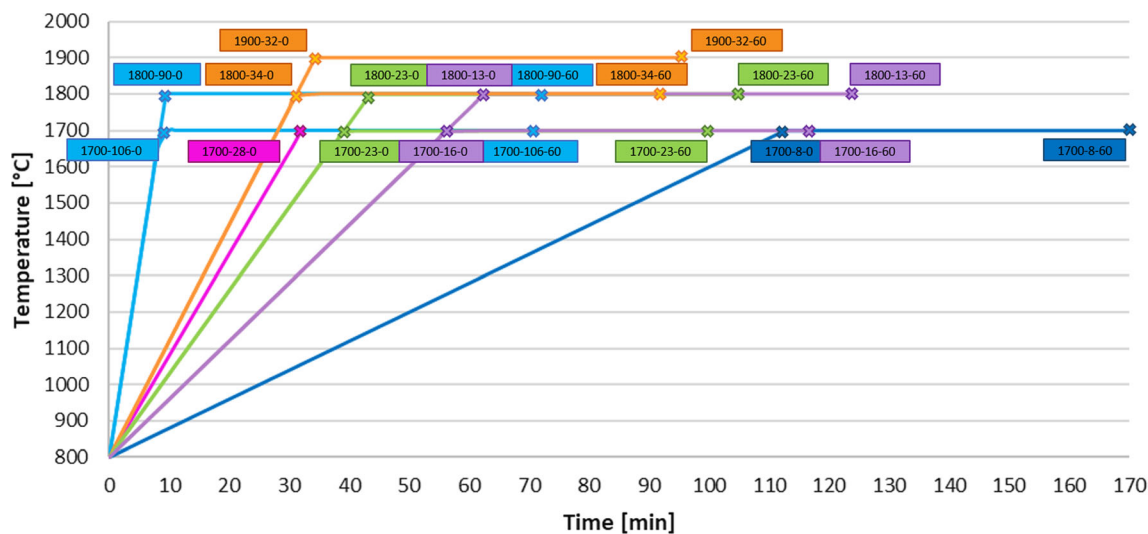


Fig. 3—Graphic overview of the heating experiments in the graphite tube furnace. The first number in the boxes gives the maximum temperature in °C; the next gives the heating rate in °C/min, and the last number gives the number of minutes at isothermal temperature. “0” denotes a non-isothermal experiment.

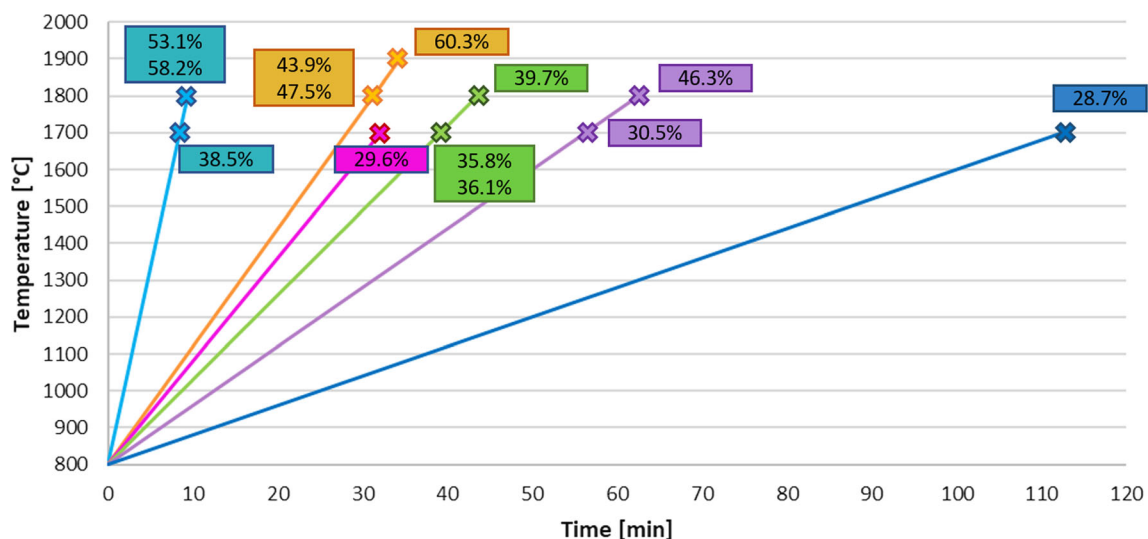


Fig. 4—Graphic overview of the heating experiments and the resulting wt pct of amorphous silica.

patterns was performed using the Topas V5 software. The exact percentage of the spike was put into the program, which then calculates the absolute amount of the crystalline phases after the peaks have been fitted.

Different heating rates gave different compositions of quartz, amorphous silica and cristobalite in the pellets. Table II shows an overview of the non-isothermal experiments and the measured XRD results. Based on the results from the non-isothermal heating experiments, isothermal experiments with a selection of the different heating rates were conducted to study the effect of different amounts of amorphous silica on the reaction between  $\text{SiO}_2$  and SiC. Sixty-minute isothermal hold times in the temperature interval 1700 °C to 1900 °C were used in this study. The main results after reduction of  $\text{SiO}_2$  with SiC are the measured weight loss of the pellets expressed as percent weight loss.

Table III shows an overview of the isothermal experiments. A graphic overview of all the experiments can be seen in Figure 3, and the resulting amorphous silica can be seen in Figure 4. The remaining pellets after the heating experiments were studied with electron probe micro-analysis (EPMA). Si, O and C elemental mapping was performed to see the spatial distribution of  $\text{SiO}_2$  and SiC.

### III. RESULTS AND DISCUSSION

#### A. Effect of Different Heating Rates on Phase Transformations in $\text{SiO}_2$

The  $\text{SiO}_2$  composition results after the heating experiments are shown in Table II. Generally, the amounts of the different phases are similar, and no clear trend can be observed for the non-isothermal experiments at each

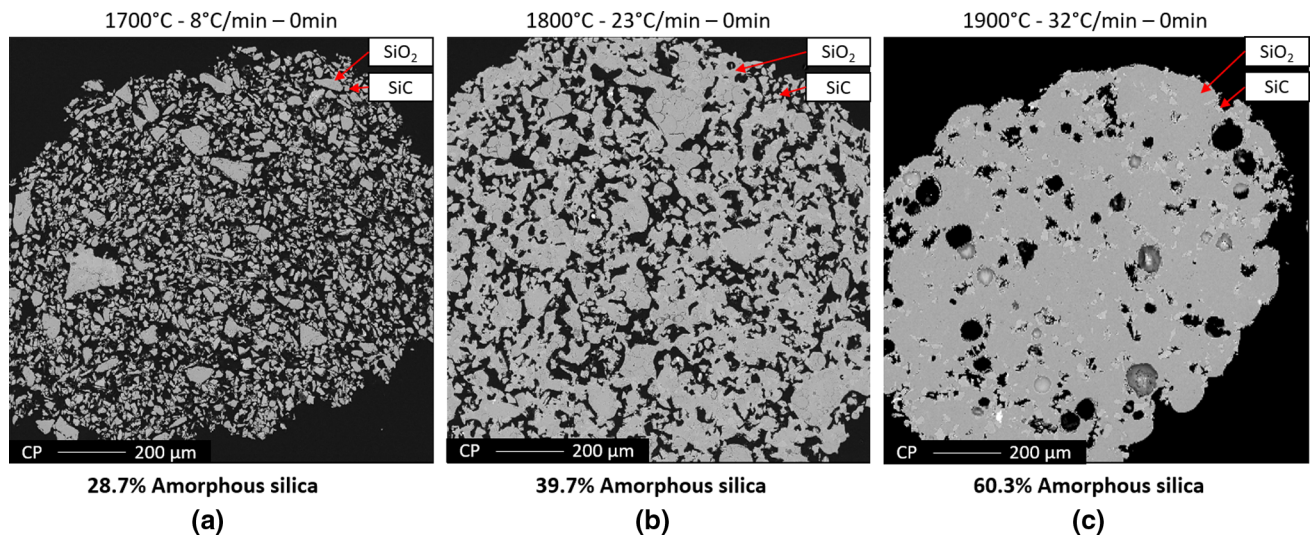


Fig. 5—Morphology images from non-isothermal experiments heated to (a) 1700 °C, (b) 1800 °C and (c) 1900 °C.

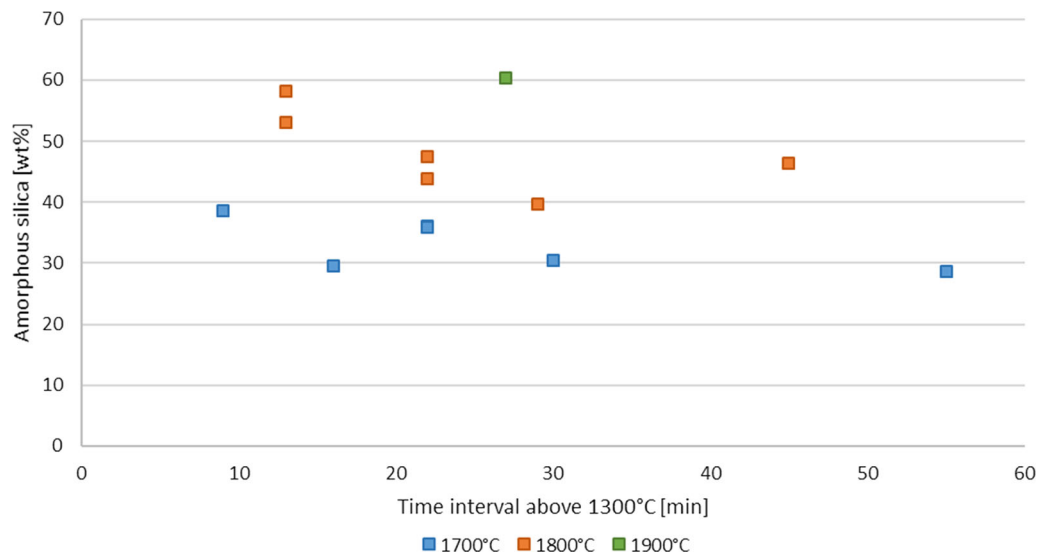


Fig. 6—Amount of amorphous silica as a function of time with temperature > 1300 °C for the non-isothermal furnace experiments. The cooling time to 1300 °C is also included.

temperature. The time interval differences with temperatures > 1300 °C are up to 45 minutes, which was expected, based on previous results,<sup>[16,17]</sup> to give a significant difference in phase composition. However, as the rate of phase transformation is slow and increases with increasing temperature, it may be that the time interval at the highest temperatures is not great enough to give significantly different SiO<sub>2</sub> phase compositions.

The amount of amorphous silica as a function of time interval > 1300 °C is shown in Figure 6. The initial transformation rate from quartz to amorphous silica is faster than from amorphous silica to cristobalite, and a higher concentration of amorphous content at the steepest heating rate was expected. As the amount of

quartz decreases, the quartz → amorphous silica transformation rate decreases, and the amorphous silica content decreases as it transforms to cristobalite. It can also be observed that the amount of amorphous silica increases with increasing target temperature. As mentioned previously, amorphous silica may form from both β-quartz and softened/melted β-cristobalite. Pure SiO<sub>2</sub> melts at around 1720 °C, but a sufficient time interval at the melting temperature is needed for the SiO<sub>2</sub> to melt completely.<sup>[22]</sup> A different degree of melting was observed at 1700 °C, 1800 °C and 1900 °C, as shown in Figure 5. These images clearly show the effect of the temperature difference from 1700 to 1900 °C, which includes the softening and melting temperature of

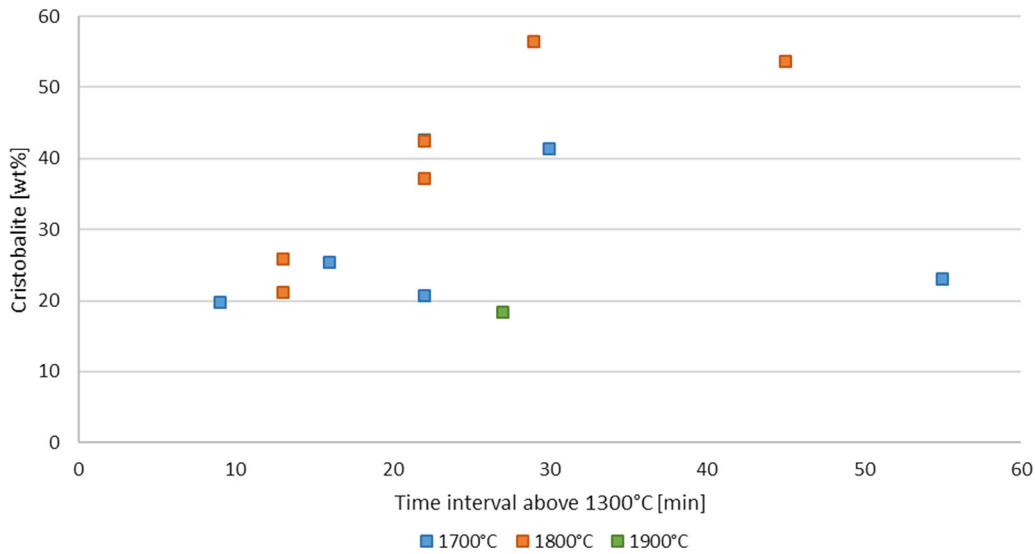


Fig. 7—Amount of cristobalite as a function of time with temperature > 1300 °C for the non-isothermal experiments. The cooling time to 1300 °C is also included.

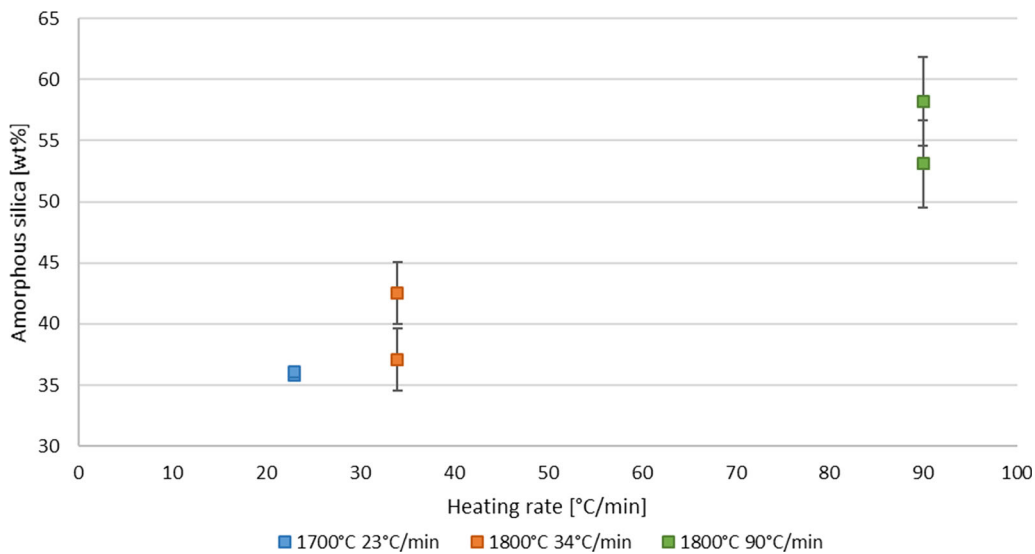


Fig. 8—Amount of amorphous silica as a function of heating rate found in the pellets from the parallel non-isothermal furnace experiments 1700 °C to 23 °C/min, 1800 °C to 34 °C and 1800 °C to 90 °C/min.

SiO<sub>2</sub>. The higher amount of amorphous content found in samples heated to 1800 °C and 1900 °C is therefore probably from softened/melted  $\beta$ -cristobalite.

The amount of cristobalite as a function of the time interval > 1300 °C is also shown in Figure 7.  $\beta$ -Cristobalite starts to form at approximately 1400 °C,<sup>[13]</sup> and the transformation rate increases with increasing the temperature to the softening/melting temperature. The XRD results show lower amounts of cristobalite at 1900 °C. This is consistent with the theory that  $\beta$ -cristobalite melts and transforms into amorphous silica. The highest content of cristobalite is observed for the heating experiments to 1800 °C. This is probably because the transformation from softened/melted  $\beta$ -cristobalite has not yet accelerated as the pellets are not completely

softened, which is confirmed in the morphology image of the pellets heated to 1800 °C in Figure 5. More time at melting temperature is needed.

Two parallel analyses were conducted for three different heating experiments to ensure reproducible results. Figures 8 and 9 show the amount of amorphous silica and cristobalite for those experiments as a function of heating rate. The standard deviations are included in the graphs. The amount of amorphous silica and cristobalite is similar for both parallel experiments heated to 1800 °C. The standard deviations are approximately 3 pct, which are within the uncertainty of the method. For the 1700 °C to 23 °C/min experiments, the amounts of amorphous silica are similar. However, the amount of cristobalite deviates by 15.5 pct. This is not

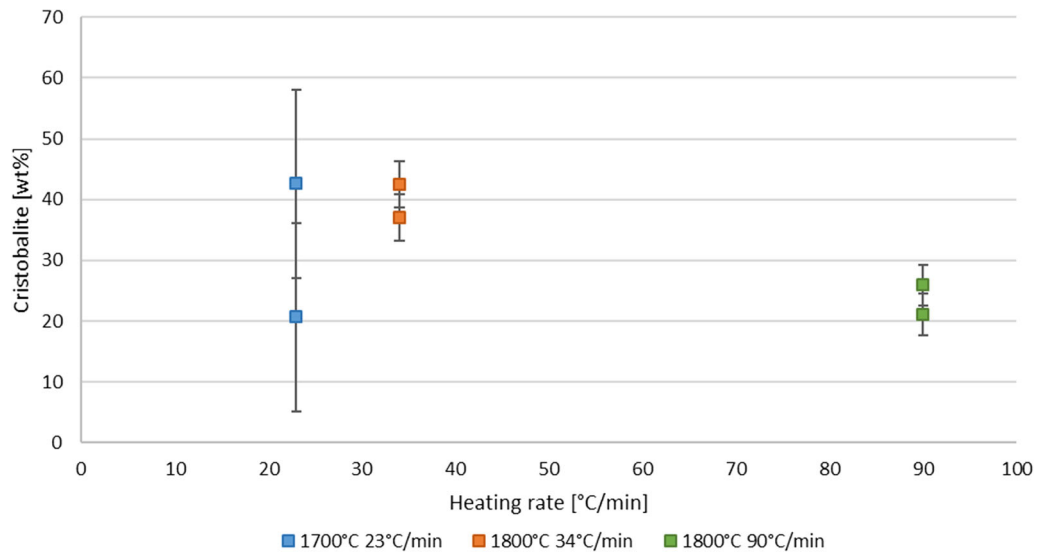


Fig. 9—Amount of cristobalite as a function of heating rate found in the pellets from the parallel non-isothermal furnace experiments 1700 °C to 23 °C/min, 1800 °C to 34 °C and 1800 °C to 90 °C/min.

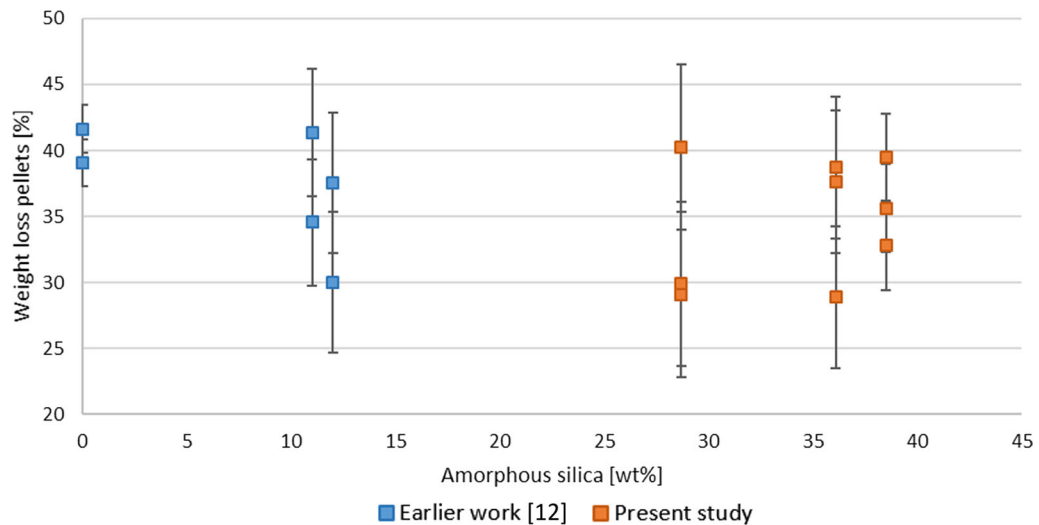


Fig. 10—Percent weight loss as a function of the amount of amorphous silica compared to results from Folstad.<sup>[12]</sup>

believed to be caused by the analyzing method, but because even with identical heat treatment the extent of the phase transformations may vary for the same quartz type. The natural materials that are used here have a high inhomogeneity, and relatively large variations are thus expected. Similar variations have been found previously for other quartz types.<sup>[16,17]</sup>

### B. Reduction Reaction Between $\text{SiO}_2$ and $\text{SiC}$

The main result after the reduction experiments was the measured weight loss of the pellets. Three parallel experiments were performed for each heating rate to 1700 °C and 1800 °C to ensure reproducible results. The conversion given in percent weight loss for all the reduction experiments is shown in Table III.



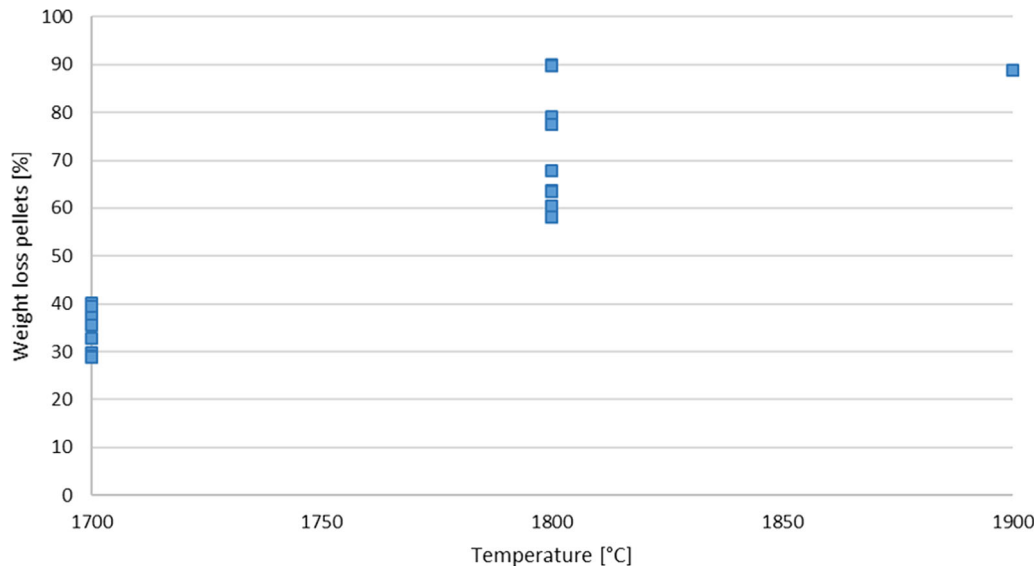


Fig. 11—Percent weight loss as a function of increasing temperature. The experiments were performed at temperature 1700 °C, 1800 °C and 1900 °C. The difference in heating rate is not considered.

### C. Effect of SiO<sub>2</sub> Polymorphs

The experiments conducted in this study showed no significant difference in the reaction rate between SiO<sub>2</sub> and SiC in Reaction [5] regarding the effect of quartz, amorphous silica or cristobalite. For all the isothermal furnace experiments, the overall variation is not larger than the variation between the parallels performed with identical conditions. Cristobalite has a more open structure than quartz, which gives a lower density. This might give a higher reaction rate. Earlier investigations<sup>[6,12]</sup> did not find any differences. This study confirms these results. The heterogeneous energetic surface of amorphous silica and weaker bindings was expected to give a higher reactivity. Figure 10 shows the percent weight loss as a function of the amount of amorphous silica for both of the experiments performed in this study and results from the earlier study of Folstad<sup>[12]</sup> at 1700 °C. All results are within the same area, 28 to 40 pct conversion, and the effect of amorphous silica is therefore concluded to be negligible.

### D. Effect of Temperature

Figure 11 gives the percentage weight loss as a function of temperature for all the isothermal furnace experiments, regardless of the heating rates. The conversion of the pellets increases with increasing temperature. Andersen<sup>[5]</sup> also found that the reaction rate increased with increasing temperature. After a sufficient reduction of the molten SiO<sub>2</sub> viscosity at 1770 °C, a rapid increase of the reaction rate was observed. The melting temperature of pure silica is 1720 °C, but the softening and melting temperatures for different industrial quartz are found to vary between 1600 °C to 1800 °C. Melting of SiO<sub>2</sub> increases the contact area between the reactants and makes the gas transports

through the sample easier, which gives an increased reaction rate. Figures 12 and 13 show pellet morphology images after isothermal furnace experiment at 1700 °C and 1800°C, respectively. The remaining sample after the experiment at 1800 °C has a much higher degree of melting. SiC particles are found in the melted SiO<sub>2</sub> matrix, which confirms a greater contact area and results in a much higher percent weight loss.

## IV. CONCLUSION

Experiments were performed to investigate the effect of different heating rates on the phase transformations quartz → amorphous silica → cristobalite → amorphous silica and the effect of quartz, amorphous silica and cristobalite on the reaction  $2\text{SiO}_2 + \text{SiC} = 3\text{SiO} + \text{CO}$ . The raw materials used were SiO<sub>2</sub>/SiC pellets. The concluding remarks from this study are summarized below.

- The transformation rate to the different SiO<sub>2</sub> polymorphs may be different for different quartz types, and information on how much a property may affect the main reaction of SiO<sub>2</sub> in the furnace is very important when selecting the most suitable quartz for the silicon production.
- Relative amounts of the SiO<sub>2</sub> polymorphs quartz, amorphous silica and cristobalite have no effect on the reaction  $2\text{SiO}_2 + \text{SiC} = 3\text{SiO} + \text{CO}$ .
- The final amounts of quartz, amorphous silica and cristobalite depend on both the target temperature and heating rate.
- The amount of amorphous silica increases with increasing temperature from 1700 °C to 1900 °C. This is because amorphous silica forms from both quartz and softened/melted  $\beta$ -cristobalite.

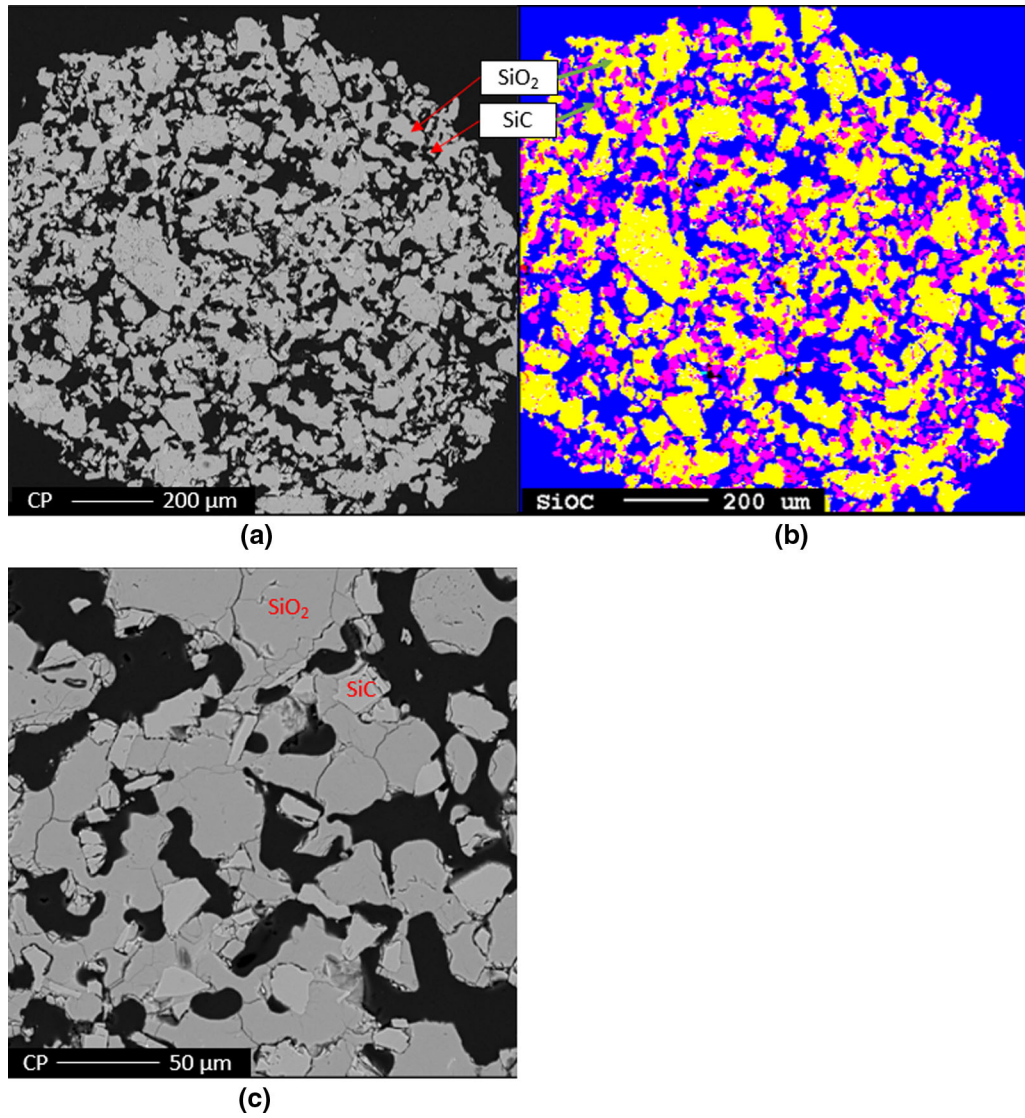


Fig. 12—(a) Backscattered image and (b) Si–O–C chemical composition of remaining pellets with magnification  $\times 80$  for the isothermal experiment heated to 1700 °C, 23 °C/min. The yellow particles are  $\text{SiO}_2$ , and the pink particles are SiC. A  $\times 400$  magnification is shown in (c). Percent weight loss for this experiment was 29 pct (Color figure online).

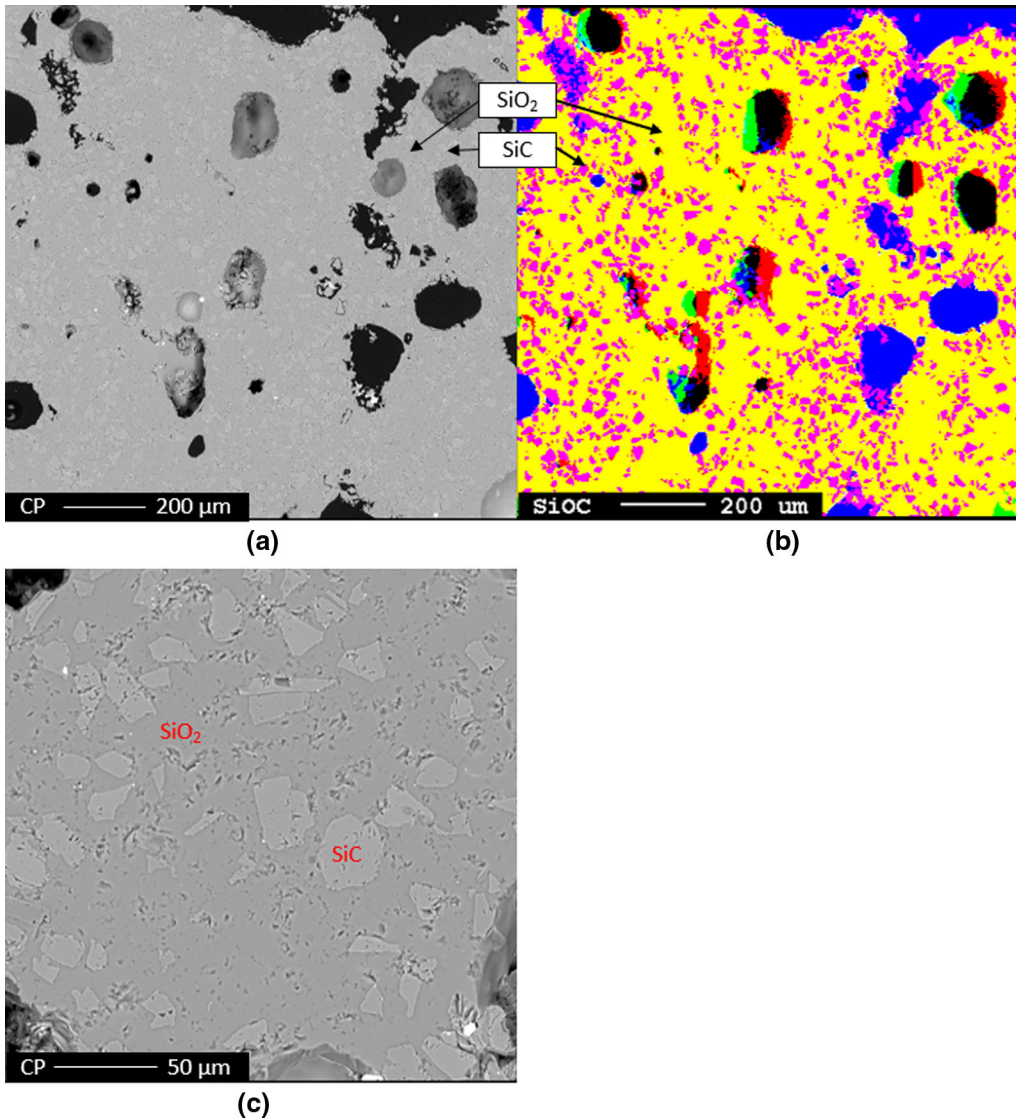


Fig. 13—(a) Backscattered image and (b) Si–O–C chemical composition of remaining pellets with magnification  $\times 80$  isothermal experiment heated to 1800 °C, 34 °C/min. The yellow matrix is SiO<sub>2</sub>, and the pink particles are SiC. A  $\times 400$  magnification is shown in (c). Percent weight loss for this experiment was 90 pct (Color figure online).

– The degree of conversion increases with increasing temperature from 1700 °C to 1900 °C.

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