

Article

Disintegration of Six Different Quartz Types during Heating to 1600 °C

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Abstract: This work investigates the high temperature behavior of six different quartz types A, B, C, D, F and G during heating to elevated temperatures. These quartz types contain different amounts of impurities, from 1% to 0.1%, and have earlier shown different degree of disintegration during heating. Melting experiments to 1750 °C have been performed to find softening and melting temperatures, and it is found that there are significant variations between the different quartz types. A higher content of impurities lowers the softening and melting temperature. The quartz surface has also been studied during heating to 1600 °C in a high temperature confocal microscope. From this, it is found that disintegration mainly happens at two temperature intervals, ~300–600 °C and ~1300–1600 °C, and is mostly due to volume changes in either the SiO₂ matrix or the impurity areas. The cracks occur from impurity areas, expanding grain boundaries, damaged or uneven SiO₂ surface and in some cases from the cavities from the escaped fluid inclusions.

Keywords: quartz; SiO₂ disintegration; fines production; melting temperature; silicon production



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1. Introduction

Metallurgical silicon and ferrosilicon are produced industrially by carbothermic reduction of quartz (SiO₂). The production takes place in a submerged arc furnace, and the overall chemical process can be written as in reaction 1. In ferrosilicon furnaces, it is also added to an iron ore or other iron sources.

To ensure an optimal furnace operation, it is important to have raw materials with properties suited for the production. Several types of quartz can be used, and how it behaves at elevated temperatures is important when selecting quartz for the production. The SiO₂ melting and degradation properties have a great impact on the charge in the furnace. A limited formation of fines and sufficient high softening and melting temperature of the SiO₂ are beneficial to promote stable production. Coarse particles ensure a good charge permeability and enables the ascending SiO and CO gas to interact and react with the carbon materials.



When quartz is added to the furnace, it will experience a steep temperature increase from room temperature to the charge top temperature around 1300 °C [1–3]. The shock heating might cause the SiO₂ to disintegrate, and this may have a negative effect on the furnace operation. Too much fine particles in the charge will decrease the gas permeability. The fine particles can melt together and may hinder an even flow of materials and gases [4,5]. Clogging of the furnace leads to the creation of gas channels and can cause small outbursts of gas. The created gas channels have less interaction with the descending raw materials and more SiO gas will escape the furnace. A good permeability of the charge mixture requires raw materials with a good size distribution, giving more space around the raw

materials and enables an even gas flow. Ideal lump size for the quartz used in Si and FeSi furnaces is 10–150 mm [6], and fine particles are defined as materials below 10 mm.

Quartz is the second most abundant mineral in the earth's crust and is found in various forms. Hydrothermal quartz can be very pure and is therefore often used in silicon production. It is formed by the precipitation of quartz from hot aqueous fluids which flows through fractures or other openings in rocks. Quartz is a hard mineral and it also impacts good corrosion resistance and high chemical stability. The void space in quartz is limited and results in a low tolerance for impurity atoms, often <1% [7]. Quartz can carry impurities in three different forms: structural impurities, fluid inclusions and minerals (solid inclusion). Structural impurities are interstitial or substitutional point defects in the lattice structure. The most common impurities in quartz are aluminum, iron, lithium, sodium and potassium. Al^{3+} is the most generally abundant impurity due to its similar ionic radius to Si and common appearance in the earth's crust [8]. Impurities in the quartz will affect the properties upon heating to elevated temperatures. The impurity content in quartz is also an important factor in the operational process and the final silicon product. Impurities in quartz will follow the same path in the furnace as the quartz. Quartz is not a porous material, and it is therefore believed that the impurities embedded in quartz are not accessible for the reducing atmosphere before the quartz melts. Impurities in quartz can affect the softening and melting temperature. The effect will depend on the impurity, but contaminants are expected to give lower softening and melting temperatures. Contaminants will break up the SiO_2 network structure and create bindings between dislocation cores, which will decrease the system energy and thus lower the melting temperature [9].

There are three main factors which contribute to crack formation in the SiO_2 [10]: (1) low density high temperature silica polymorphs, (2) presence of liquid inclusions and (3) mineral inclusions at the grain boundaries.

1. When quartz is heated, it will transform into different SiO_2 polymorphs. α -quartz transforms to β -quartz at 573 °C, and then to the high temperature polymorph β -cristobalite through β -tridymite or an amorphous phase. The crystal structure of tridymite is debated. It is suggested that the tridymite phase is not a pure silica phase, but needs certain impurity elements to form [11,12], but this is also debated [13,14]. The changes in the SiO_2 structure during heating cause tensions and may again influence the melting process. Cristobalite in the high temperature form has a more open structure than α -quartz with densities 2.20 g/cm³ and 2.65 g/cm³, respectively [15]. During the transformation from quartz to cristobalite, the theoretical volume increase is about 17%. In experimental studies it has also been found that the volume of quartz increased up to 44% [16]. Crack formation occurs when grains expand differently in different directions and induce stress in the crystal group [6].
2. The internal partial pressure of fluid inclusions in the quartz will increase with increasing temperature until they escape from the SiO_2 , normally in the temperature range from 200–600 °C. The fluid inclusions in the quartz mainly consist of water or water + CO_2 . Water inclusions have a higher internal pressure and will decrepitate at a lower temperature than water+ CO_2 . With increasing internal pressure, cracks will develop in the SiO_2 and enable degassing of water and CO_2 from the inclusions [17].
3. Mineral inclusions in the quartz are most often present at the grain boundaries. Typical impurities in quartz are muscovite ($\text{KAl}_2(\text{Si}_3\text{Al})\text{O}_{10}(\text{OH},\text{F})_2$) and feldspar (KAlSi_3O_8 - $\text{NaAlSi}_3\text{O}_8$ - $\text{CaAl}_2\text{Si}_2\text{O}_8$). These minerals have a lower melting temperature than SiO_2 , and heating can therefore lead to crack formation.

Several studies [4,6,18,19] have shown that different quartz types will disintegrate differently, but the reason for this is not clear. Mechanical handling before heating and thermal strength during heating could both be contributors to the production of fines. Jusnes [18] studied the disintegration for ten different quartz types used in Si and FeSi production after shock heating to 1500 °C. Large variations in the production of fines were observed between the different quartz types, from around 5 wt% up to 85 wt%. She found that quartz with larger grain sizes created more fines than the quartz with smaller grains,

and that the increasing amount of cracks gave an increasing amount of fines <10 mm. Jusnes did not find any correlation between the amount of amorphous phase and cristobalite, and the amount of fines after shock heating. Large variations between different quartz types were also observed by Ringdalen [4] and Aasly et al. [20], which studied fines production after shock heating to 1500 °C and 1300 °C, respectively. In addition, Aasly et al. studied the SiO₂ surface during heating and found that heating of the samples opened the grain boundaries in most samples, leading to micro cracks. A network of micro cracks was also observed in the fluid inclusion planes as a result of fluid inclusion decrepitations.

The main goal in this investigation is to verify earlier experiments on quartz disintegration and melting properties with comparable results. In addition, a new method has been used to study the surface structure, crack occurrence, the impurities behavior and other phenomena during heating. More knowledge about the different quartz types is important when selecting the desired quartz for production. The behavior at high temperatures has a great impact on the initial reacting process in the higher parts of the furnace, and hence also the further production of silicon that happens in the high temperature zone of the furnace.

2. Materials and Methods

Six different quartz types used as raw materials in Si and FeSi production have been studied in this report. Picture of each quartz type is shown in Figure 1. Table 1 lists the general ICP-MS analyzes, which gives the initial chemical composition of the quartz types. It is important to note that when working with natural materials, inhomogeneity must be expected, and large variations may therefore occur. The analyses will still provide an indication of the degree of impurities in the quartz. B and C have the highest degree of impurities around ~1%, D in the medium range ~0.5%, while A, F and G contain ~99.9% SiO₂.

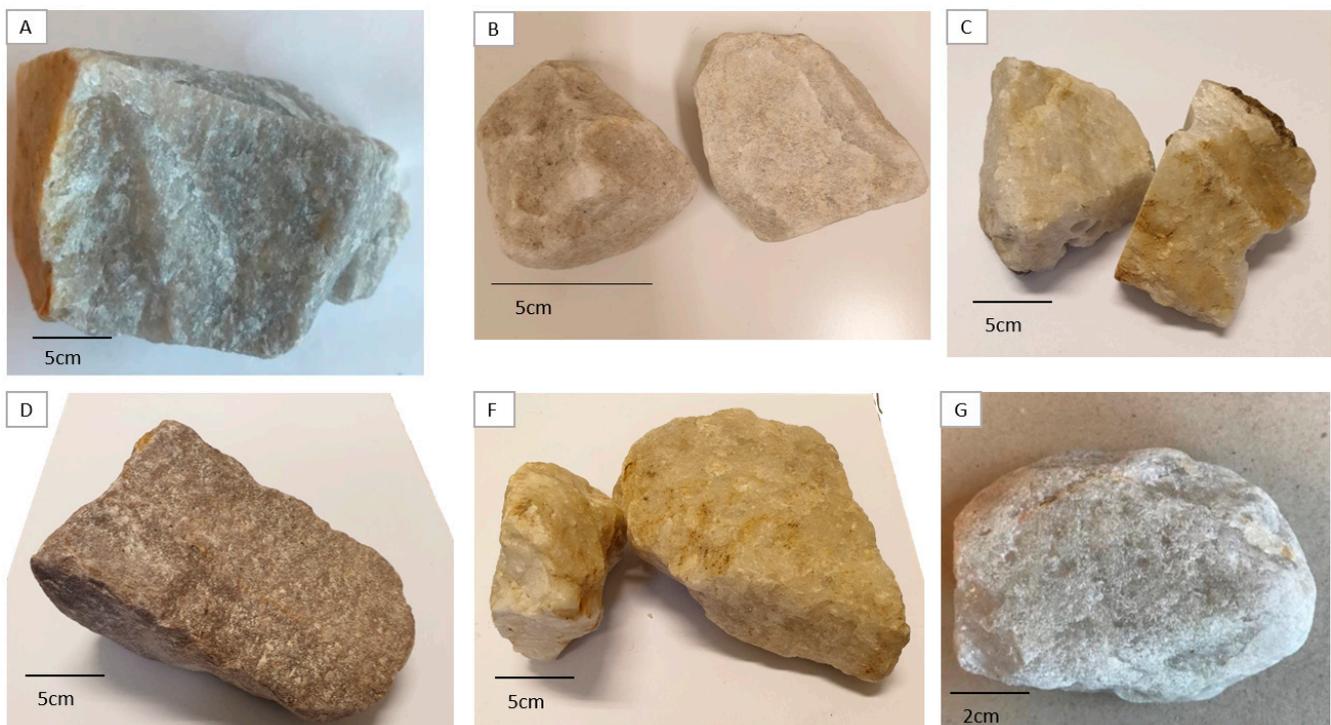
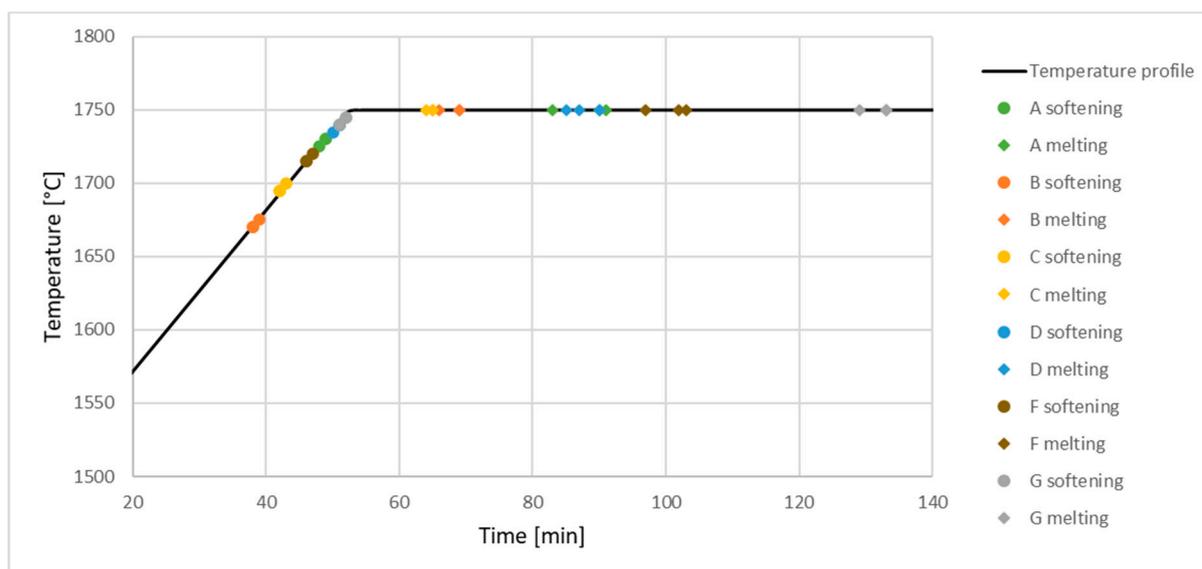


Figure 1. Pictures of the quartz types used in this study: (A,B,C,D,F,G), used as raw material in Si and FeSi production.

Table 1. ICP-MS analyses for quartz type A, B, C, D, F and G [18,21], used as raw material in Si and FeSi production.

Quartz Type	%Al ₂ O ₃	%K ₂ O	%FeO/Fe ₂ O ₃	%CaO	%MnO
A	0.020	0.002	0.003	0.0004	~0.4 ppm
B	0.613	0.154	0.0771	0.0084	0.9 ppm
C	1.19	0.180	0.09	0.09	0.140
D	0.492	0.075	0.314	0.005	<0.1 ppm
F	0.017	0.005	0.003	0.001	~1 ppm
G	0.015	0.002	0.003	0.004	~0.4 ppm

Earlier heating experiments have found that different quartz sources may have different softening and melting point [22] and that increasing heating rate increases the softening and melting temperature. This suggests that melting of quartz takes time [23]. Softening of quartz is defined as when the shape has lost its sharp edges [24], i.e., the temperature at which the SiO₂ starts to melt. Softening and melting experiments to 1750 °C were performed in a sessile drop furnace, which is right above the theoretical melting temperature of pure SiO₂ around 1720 °C. Three parallels of each quartz type were done to ensure reproducible results. The heating profile is shown in Figure 2. After reaching 1750 °C, the temperature is held until the SiO₂ sample is completely melted.

**Figure 2.** Softening and melting temperature for quartz type A, B, C, D, F and G.

Experiments with a high temperature confocal microscope (HTCM) were performed to study the behavior of the SiO₂ during heating, which possesses the function of metallographic analysis and laser confocal real-time analysis. In total, 21 experiments were conducted. All samples were coated with a thin carbon layer on the SiO₂ surface prior to and after heating due to SEM and EDS investigations. The sample surface was observed and monitored from room temperature and up to 1600 °C. Continuous information of the surface structure, crack occurrence, the impurities behavior and other phenomena during heating were obtained. The HTCM consists of a laser confocal microscope, type VL2000DX and a high temperature heating furnace, type SVF17SP. Quartz cuboids with length and width 5.5 mm × 5.5 mm and height 3.5 mm were placed in an alumina crucible with diameter 8 mm and then placed in the microscope. A heating rate 100 °C/min up to 1000 °C, and 25 °C/min up to 1600 °C in argon atmosphere were used for all the experiments.

3. Results and Discussion

3.1. Quartz Impurities during Heating

The softening and melting temperatures for A, B, C, D, F and G are summarized in Figure 2, and from this, it can be observed that there are variations in softening and melting temperatures between the different quartz types. B and C have the lowest softening temperatures around 1670–1700 °C, while G has the highest at 1745 °C. Quartz C only needs 12 min at 1750 °C before it melts completely, while G needs around 80 min. It is found that the impurities have an impact on the softening and melting temperatures. The more impurities in the SiO₂, the lower the softening and melting temperatures. From the ICP-MS analyses [18,21] listed in Table 1, it is found that A and C have the highest impurity content ~1%, and these two quartz types have a significant lower softening temperature and need significantly less time at 1750 °C to be completely melted. This agrees with earlier findings [16,23]. The impurity content in the quartz D samples is expected to be around 0.5%, and around 0.1% in quartz A, F and G. Quartz A, D and F have similar softening/melting temperatures, while G needs 30 min more than F at 1750 °C before it is completely melted. This indicates that there are more factors affecting the softening and melting temperatures. Viscosity of a melt has earlier been found to have an influence on its melting kinetics [25], but more experiments should be performed. During the high temperature confocal microscope (HTCM) experiments, it could be observed that some of the impurity areas expand or change colors, as highlighted in Figure 3. This indicates that chemical reactions are happening, e.g., phase transformations and melting. Table 2 lists the different oxide impurities analyzed in the SiO₂ together with their melting temperature. A temperature of 1600 °C is above the melting temperature for several of these oxides, and a contribution of these in the system will lower the liquidus temperature.

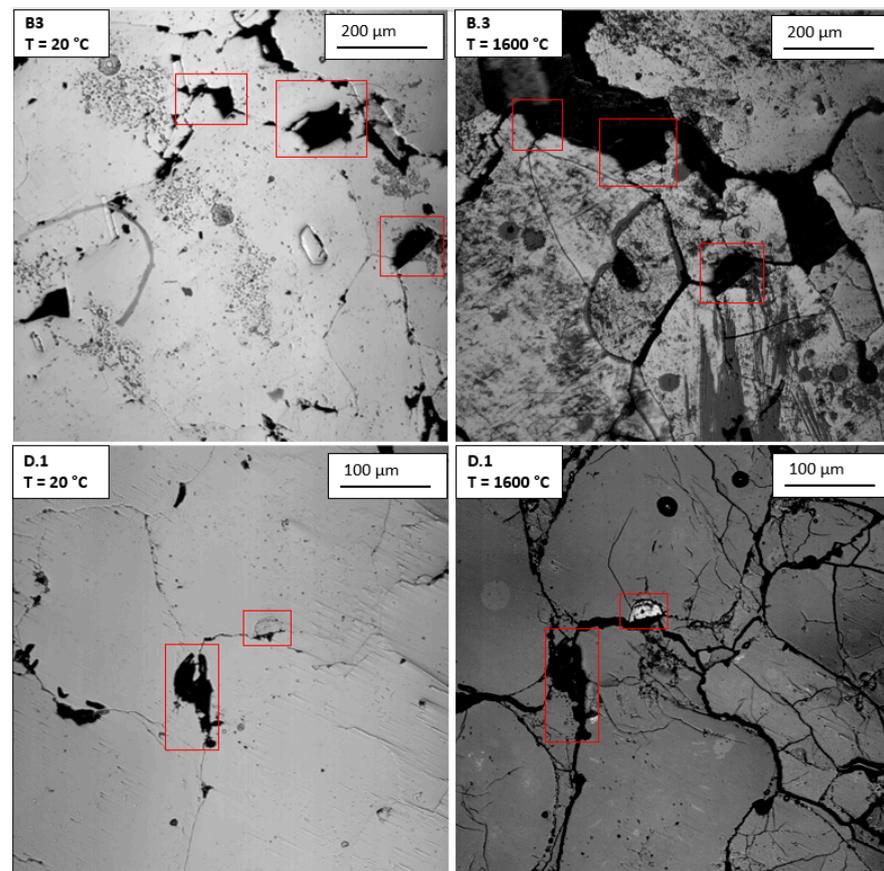


Figure 3. Images from high temperature confocal microscope (HTCM) experiment B.3 and D.1 at 20 °C and 1600 °C, showing the visual changes in the impurity areas after heating.

Table 2. Oxide impurities found in the quartz samples and their melting temperature.

Oxide	T _m [°C]
Al ₂ O ₃	2072
FeO	1377
ZrO ₂	2715
K ₂ O	350
Na ₂ O	1132
MnO	1945

3.2. Disintegration of SiO₂ upon Heating

The main result after the heating experiments is the quantification of crack formation during heating. Three parallel experiments of each quartz type were performed to ensure reproducible results.

3.2.1. Crack Formation during Heating

When quartz is added to the Si/FeSi furnace, it will experience shock heating and it might disintegrate. Different quartz sources have earlier shown different behavior upon heating, and the amount of fines produced from the SiO₂ in the furnace will have a great influence on the charge properties and hence on the production process. The SiO₂ surface for A, B, C, D, F and G are studied during heating up to 1600 °C, and the crack formation for the experiments are summarized in Figure 4. It can be observed that there are large differences between the different quartz types. C, D and G produce the most, >10 mm/mm², while A and F produce < 8 mm/mm². It is also found that there are variations within the same quartz type, as in for quartz A: 12.2 mm/mm² and 4.7 mm/mm². It should also be noticed that some of the variations within the same quartz types are larger than the differences between the different quartz types. This is likely due to differences in the samples heated, e.g., impurities, grain size, damages in the SiO₂ surface and other textural properties. The crack formation during heating to 1600 °C for A.1 and A.2 is 12.2 mm/mm² and 4.7 mm/mm², respectively. From the initial HTCM images, it can be seen that A.2 has a more even surface. Most of the cracks in A.1 form at temperatures >1300 °C, which most likely are due to the volume change from the phase transformation from β-quartz to β-cristobalite. More damages in the SiO₂ surface are more prone to the stress which causes microcracks. In quartz A, there are two domains with different grain sizes. Domain 1 has a medium- to coarse-grained quartz in the mm and μm scale, while the grains in the second domain are stretched and deformed: 10–200 μm thick and a few mm long [18]. However, as these two samples are from the same stone, this is not likely the reason for the variations.

Crack formation in the SiO₂ mainly happens at two temperature intervals, ~300–600 °C and ~1300–1600 °C. From the HTCM experiments, it can be seen that cracks occur from impurity areas, expanding grain boundaries, damaged or uneven surfaces and, in some cases, from the cavities from the escaped fluid inclusions. A volume increase was observed for all the quartz samples during the softening/melting experiments. However, there were not found any correlation between softening/melting temperatures and the amount of cracks produced. The crack formation from 300 °C is, in most cases, from volume changes in the impurity areas, from an uneven SiO₂ surface or from fluid inclusion cavities, illustrated in Figures 5–7. Cracks occurring from ~1300 °C are most probably due to the SiO₂ phase transformation from β-quartz to β-cristobalite. As cristobalite has a lower density than quartz, the volume will increase as more quartz are transformed to cristobalite. The grains will then expand and induce stress that causes microcracks, illustrated in Figure 8. The volume increase also causes more tension in the existing cracks, leading to more crack formation.

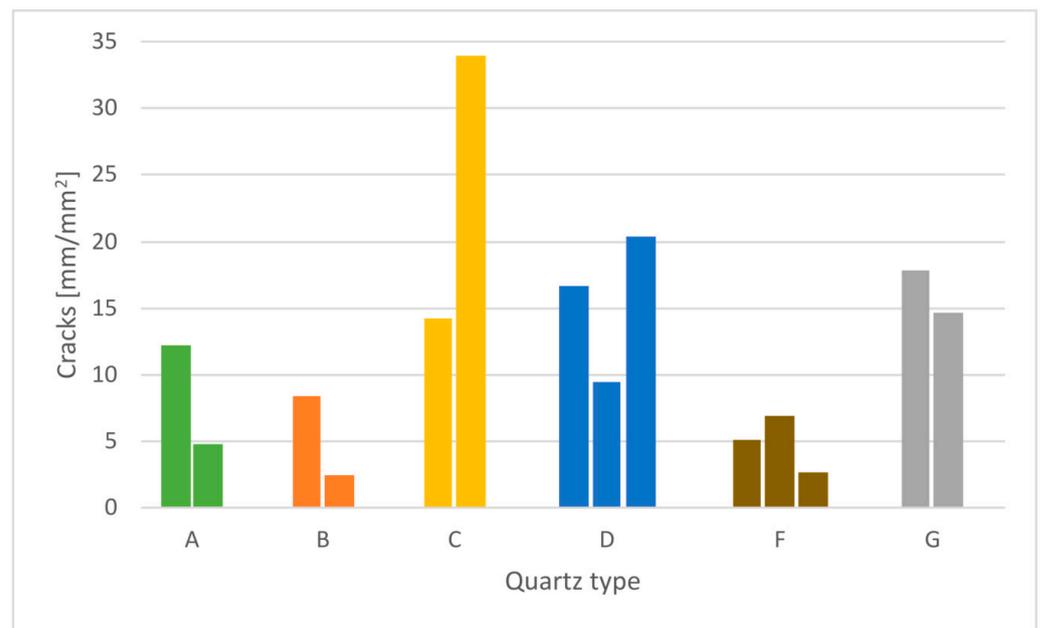


Figure 4. Summary of the crack formation for all the quartz types. The amount of cracks is given in mm/mm².

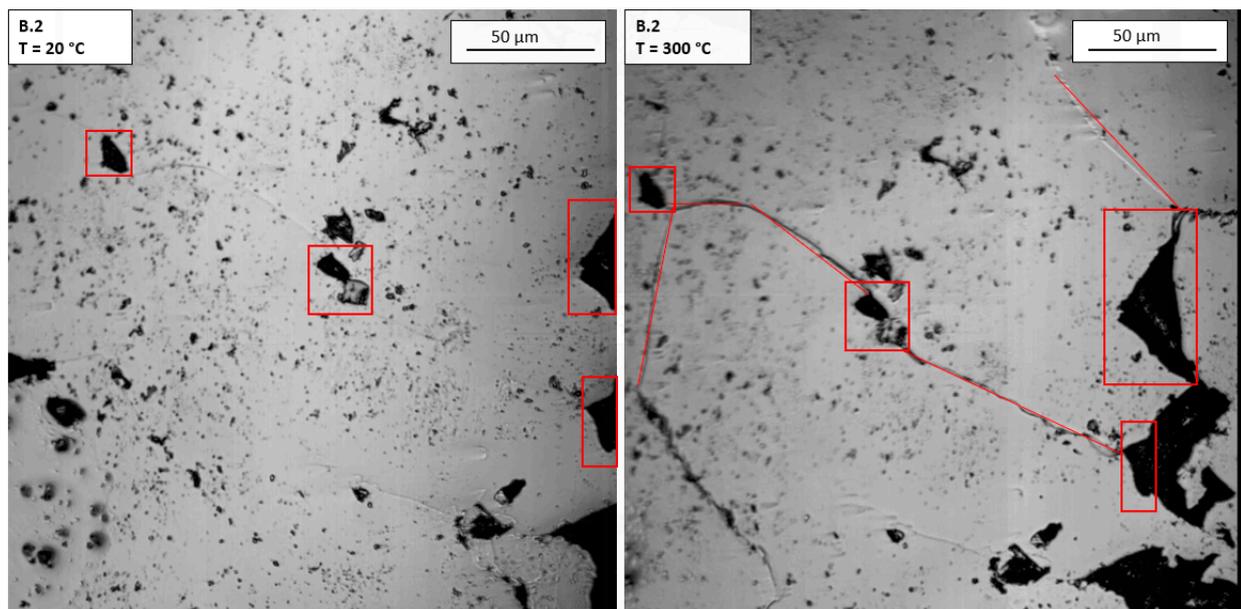


Figure 5. Images from high temperature confocal microscope (HTCM) experiment for B.2 at 20 °C and 300 °C, showing crack formation from impurity areas.

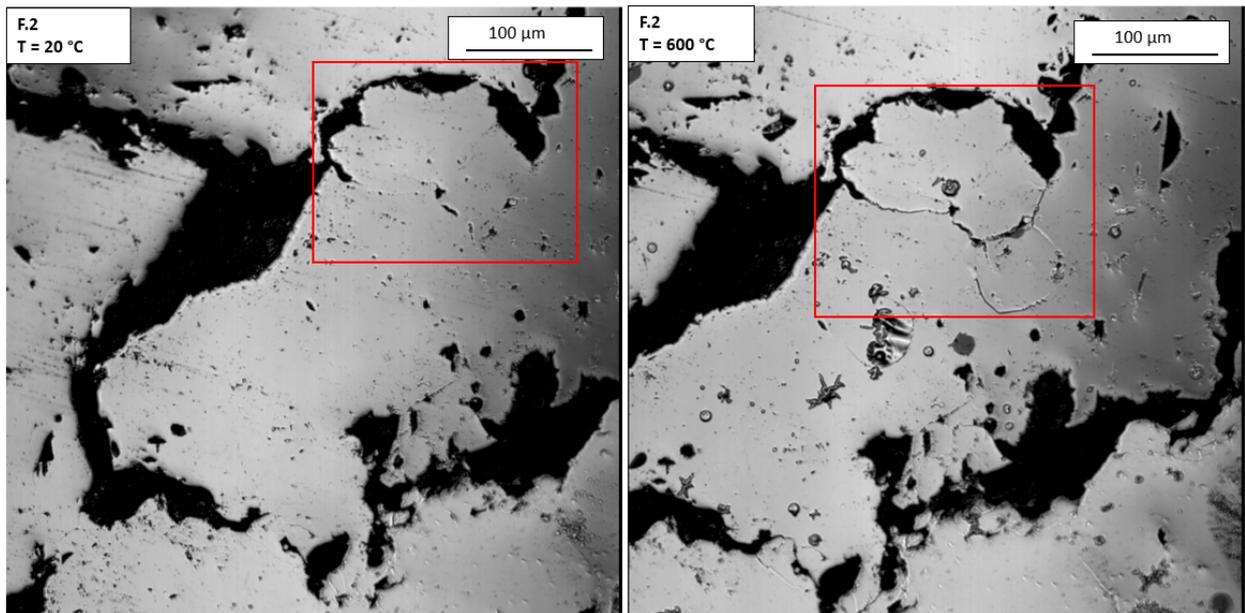


Figure 6. Images from high temperature confocal microscope (HTCM) experiment for F.2 at 20 °C and 600 °C, showing crack formation from an uneven SiO₂ surface. No impurity areas were found in this sample.

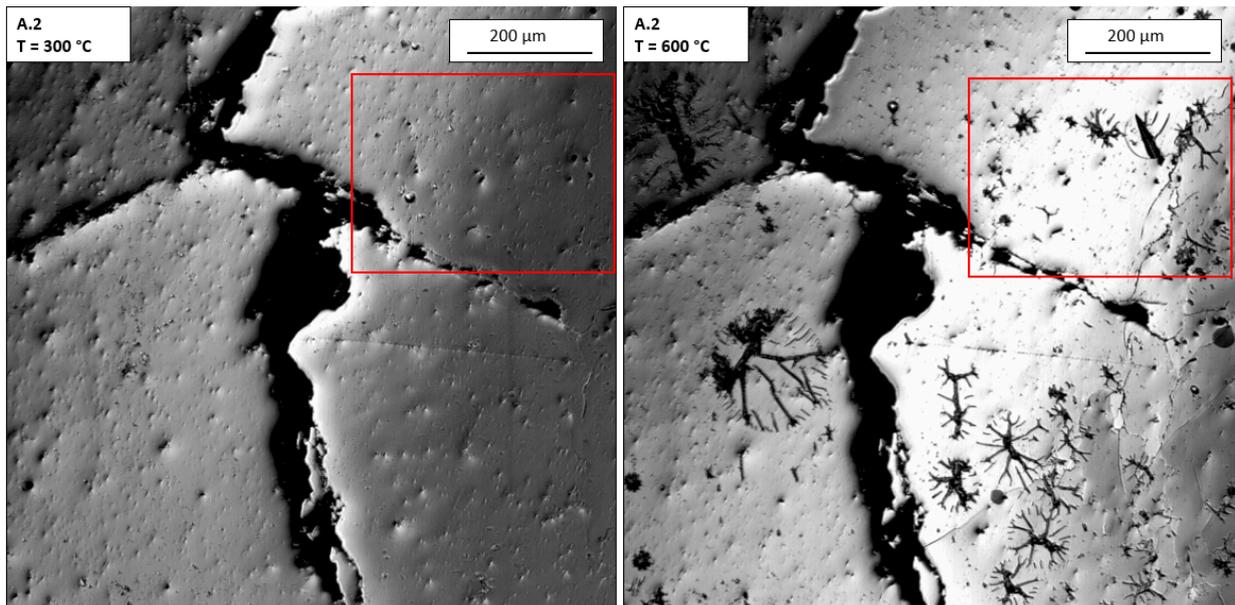


Figure 7. Images from high temperature confocal microscope (HTCM) experiment for A.2 at 300 °C and 600 °C, showing crack formation from cavities from escaped fluid inclusions.

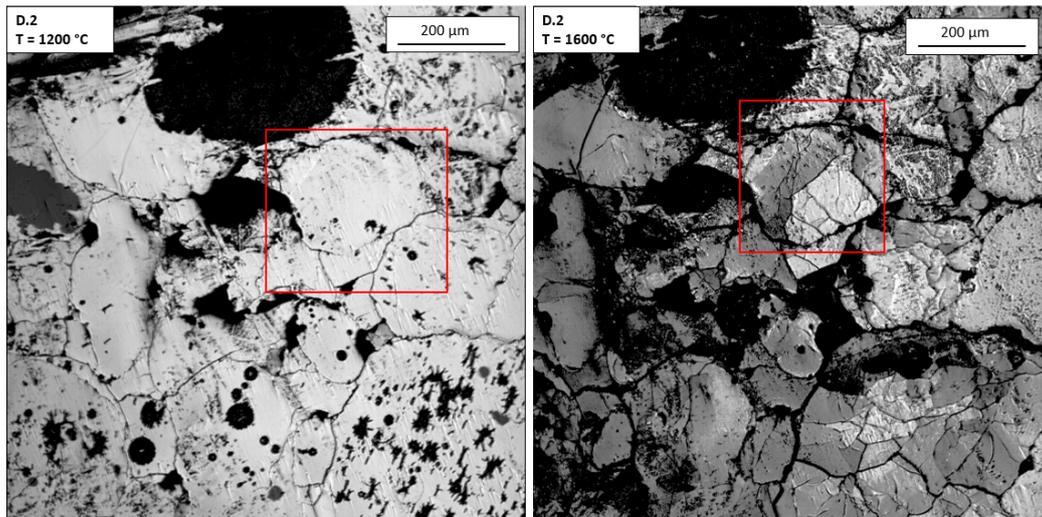


Figure 8. Images from high temperature confocal microscope (HTCM) experiment for D.2 at 1200 °C and 1600 °C, showing crack formation from expanding grain boundaries.

3.2.2. Crack Formation and Fines Production

Disintegration of SiO_2 to fine particles may have a negative effect on the furnace operation in Si and FeSi production. Too much fine particles in the charge will agglomerate or melt together and may hinder an even flow of materials and gases [4,5]. Jusnes [18] investigated in her work four of the quartz types used in this study. She found the amount of fines for quartz A, D, F and G after shock heating. Figure 9 compares the amount of cracks produced during heating to 1600 °C in this study with amount of fines produced from shock heating to 1500 °C. From this, it can be noticed that quartz D, which has a high degree of cracking, has the lowest wt% of fines produced. Quartz F has the highest formation of fines, but the lowest formation of cracks. This is not in agreement with Jusnes, who found an increasing amount of fines with increasing amounts of cracks. However, this could be explained by the different nature of these two quartz types. Quartz D has more impurities on the grain boundaries. These melt and bind the grains together and will also absorb a lot of the stress from the SiO_2 volume increase. Less fines will then be produced, even with a higher degree of crack formation. Quartz F has larger grain boundaries and the impurities have therefore no binding effect in the SiO_2 matrix.

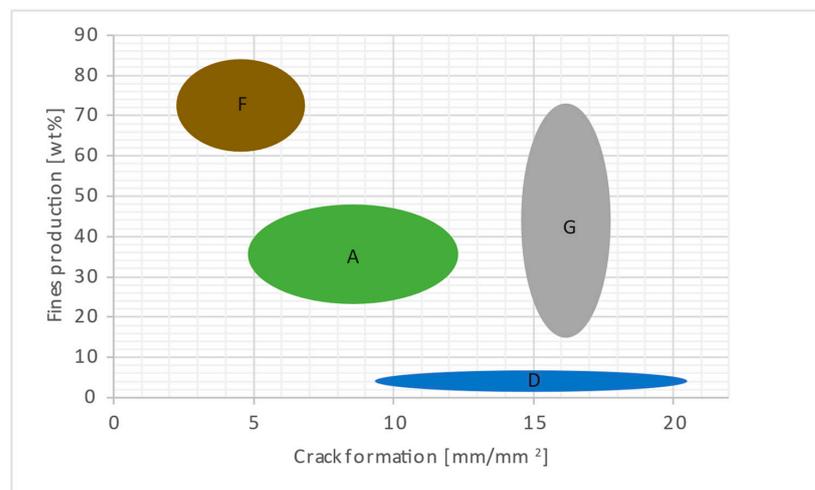


Figure 9. Crack formation at 1600 °C and fines production from shock heating to 1500 °C [11] for quartz type A, D, F and G. Fines are defined as particles <10 mm. Crack formation is given in mm/mm^2 , while fines production is given in wt%.

4. Conclusions

An increased knowledge of how the different quartz types behave at higher temperatures is crucial when selecting the raw materials for Si and FeSi production. The melting properties, the crack formation and further the fines production have a great impact on the charge properties in Si and FeSi production. Quartz samples from six different quartz types, A, B, C, D, F and G, have been studied, all used in Si and FeSi production. The different quartz types show different behaviors during heating, and these results supply answers and raise new questions for further research.

- Crack formation in the quartz during heating to 1600 °C mainly happens at two temperature intervals, ~300–600 °C and ~1300–1600 °C, and is mostly due to volume changes in the sample. The cracks occur from impurity areas, expanding grain boundaries, damaged or uneven surfaces and in some cases from the cavities from the escaped fluid inclusions.
- Amount of cracks produced at 1600 °C varies between the different quartz types from 2.7 mm/mm² to 34.0 mm/mm². However, variations are also found within the same quartz types. This is believed to be due to natural differences in the heated samples.
- The amount of cracks produced during heating is not necessarily correlated to the amount of fines produced. This is seen for quartz type D and F and shows that there are more factors than the amount of cracks that affect the fines production. Earlier research has suggested an increased amount of fines from increased crack formation.
- An increased amount of impurities in the quartz lowers the softening and melting temperature. This effect was observed with an impurity content ~1% but was not found for the samples containing 0.1–0.5% impurities. Images from high temperature confocal microscope (HTCM) experiments show that some of the impurity areas expand or change colors, which indicates that chemical reactions are happening, e.g., phase transformations or melting.

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