

## Research status of composition and formation conditions of compound calcium ferrite (SFCA)

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### Abstract

The research progress of compound calcium ferrite SFCA phase is reviewed in this paper. It is a kind of high basicity sinter and plays a significant role in the quality of blast furnace ironmaking. The first part introduces the chemical composition, characteristics, morphology and types of SFCA. The second part points out the factors that affect the formation of SFCA, such as gangue, oxygen partial pressure and some other conditions. Through the summary of previous research results, we can have a further understanding of these mechanisms, which is of great significance to improve the efficiency of sintering process, so that we can predict the best sintering conditions (such as chemical composition, temperature, alkalinity, oxygen partial pressure, etc.), and then predict the chemical and physical changes of specific iron ore fines mixture needed to produce high-quality products.

### Keywords

Iron ore; sinter; SFCA; chemical composition; morphology and type; influencing factors.

## 1. Introduction

Sinter is the main raw material of blast furnace. The development of iron and steel industry promotes the development of sinter quality, especially the development of high basicity sinter quality, which directly affects the economic and technical indicators of blast furnace ironmaking. Its quality is closely related to the mineral composition and mineral phase structure formed in it, and affects the properties of sinter products, such as strength and reducibility. SFCA is a kind of bonding phase which can bond iron oxide and silicate. The binder phase is composed of Fe, Ca, Si, Al and O, it is formed by iron ore powder, coke and flux at high temperature. Although researchers have carried out extensive research on various scales and obtained a large number of literature data, and have a certain understanding of the composition range, stability, crystal structure, texture, morphology, formation mechanism of "SFCA" and its influence on sinter properties, there are still uncertainties in many aspects of the understanding of "SFCA" stage on the practical and basic level.

## 2. Chemical composition, characteristics, morphology and types of SFCA

### 2.1. Chemical composition of SFCA

The SFCA phase formed in the system of  $\text{Fe}_2\text{O}_3$  (FeO)-CaO- $\text{Al}_2\text{O}_3$ - $\text{SiO}_2$  contains a lot of  $\text{Fe}_2\text{O}_3$  and CaO, which is formed in the early stage by high temperature reaction. The low temperature calcium ferrite phase is formed between minerals containing impurities of  $\text{Al}_2\text{O}_3$  and  $\text{SiO}_2$  gangue elements.

Hidayat *et al.*[1] summarized the thermochemical properties and stability of calcium ferrite phase. For example:  $\text{CaFe}_2\text{O}_4$  (generally referred to as CF),  $\text{Ca}_2\text{Fe}_2\text{O}_5$  ( $\text{C}_2\text{F}$ ) and  $\text{CaFe}_4\text{O}_7$  ( $\text{CF}_2$ ).

However, in iron ore sinter, due to the process conditions beyond its thermal stability limit, and the presence of  $\text{SiO}_2$  and  $\text{Al}_2\text{O}_3$  impurities, it reacts with calcium ferrite to form more complex phases. These simple binary or pseudo binary calcium ferrite phases are rarely observed.

Hancart *et al.* detected the complex phase of compound calcium ferrite (SFCA) from sinter, and studied its composition between  $7 \text{ Fe}_2\text{O}_3 \cdot 2 \text{ SiO}_2 \cdot 3 \text{ Al}_2\text{O}_3 \cdot 5 \text{ CaO}$  and  $5 \text{ Fe}_2\text{O}_3 \cdot 2 \text{ SiO}_2 \cdot 5 \text{ Al}_2\text{O}_3 \cdot 5 \text{ CaO}$ . Except that the CaO content is always 5 mol, the other oxides add up to about 12 mol, which is a relationship of mutual substitution. The chemical formula is  $x \text{ Fe}_2\text{O}_3 \cdot y \text{ SiO}_2 \cdot z \text{ Al}_2\text{O}_3 \cdot 5 \text{ CaO}$ , which satisfies  $x+y+z=12$ , the amount and crystal form of SFCA affect the strength and reducibility of sinter.

Whether in the laboratory or in industrial production, the sintering temperature of iron ore should reach above 1073 K (800 °C) to form various phases, such as calcium ferrite phase, hematite phase, magnetite phase, silicate phase, glass phase and some melts. In the industrial preparation of iron ore sinter, the previous research results show that there are great differences in the composition of SFCA phase[2-4]. The composition of SFCA in iron ore sinter is shown in Table 1[3,5,6], and its composition range (mass percentage) is 60%~76%  $\text{Fe}_2\text{O}_3$ , 13%~17% CaO, 3%~10%  $\text{SiO}_2$ , 3%~11%  $\text{Al}_2\text{O}_3$  and 0%~3% MgO. The change of composition may be caused by the different composition of charge and the different operation conditions of sintering heating and cooling stage.

Table 1 Composition (mass percent) of "SFCA" in different literatures

Oxide	$\text{Fe}_2\text{O}_3$	CaO	$\text{SiO}_2$	$\text{Al}_2\text{O}_3$	MgO
Hancart et al.	60~74	14~16	3~10	5~11	-
Ahsan et al.	63~76	13~16	7~9	4~10	0.7~1.5
Hsieh and Whiteman.	77	14	5	4	-
Yang and Loo.	70~76	13~15	6~8	3~8	0.3~1.5
Stenlake et al.	66~76	14~17	4~8	5~10	0.1~0.8
Ostwald	66~76	12~16	3~10	3~10	0~3
Dawson et al.	72~85	12~18	0.5~11	-	-

## 2.2. Characteristics of SFCA

Pownceby[5] thinks that the formation of SFCA must be based on the existence of  $\text{Al}_2\text{O}_3$ . The content of  $\text{Al}_2\text{O}_3$  affects the formation of SFCA. Generally, the mass fraction of  $\text{Al}_2\text{O}_3$  is between 4% and 8%, and the content of  $\text{SiO}_2$  determines the morphology of SFCA. When the content of  $\text{SiO}_2$  is more than 15%, fine fibrous SFCA is formed. There is a cation exchange reaction in the complex calcium ferrite system. Researchers have different views on the spatial structure of the complex calcium ferrite system. Inoue and Ikeda[6] think that  $\text{Fe}_2\text{O}_3$ ,  $\text{SiO}_2$ ,  $\text{Al}_2\text{O}_3$  and CaO are located in the four vertices of the tetrahedron of the composite calcium ferrite system, and the composite calcium ferrite is located in the plane composed of three vertices, which are  $\text{CaO} \cdot 3 \text{ Fe}_2\text{O}_3$ ,  $\text{CaO} \cdot 3 \text{ Al}_2\text{O}_3$  and  $\text{CaO} \cdot 3 \text{ SiO}_2$  respectively. Dawson[3] thought that the three vertices were composed of  $\text{CaO} \cdot 2 \text{ Fe}_2\text{O}_3$ ,  $\text{CaO} \cdot 2 \text{ Al}_2\text{O}_3$  and  $\text{CaO} \cdot 3 \text{ SiO}_2$  respectively. In addition, Hamilton[7] and others believe that when there is no  $\text{Al}_2\text{O}_3$  in the composite calcium ferrite, the composite calcium ferrite exists in the two dimensional plane composed of  $\text{CaO} \cdot 3 \text{ Fe}_2\text{O}_3$  and  $4 \text{ CaO} \cdot 3 \text{ SiO}_2$ , as shown in Fig. 1[7].

## 2.3. Morphology of SFCA

The most common descriptive names of "SFCA" minerals in the literature include but are not limited to monocalcium ferrite, acicular calcium ferrite, acicular SFCA, tabular SFCA, SFCA-I, fibrous SFCA, fine fibrous SFCA, irregular SFCA, low temperature SFCA, acicular SFCA, dendritic SFCA, columnar SFCA, flat SFCA, massive SFCA, prismatic SFCA, lath SFCA, crystalline SFCA,

eutectic SFCA Compound calcium ferrite. XRD plays a significant role in distinguishing SFCA and SFCA-I, but in general, the most commonly used method for researchers is to observe the microstructure with light microscope or electron microscope. Researchers usually divide "SFCA" into two kinds of microstructure, one is prismatic or columnar SFCA, the other is flat SFCA-I[8,9]. Recent research work of mezibricky[10] has determined the difference between needle like and columnar structure types, indicating that different SFCA phases can form similar microstructure under certain conditions. The similarity of the two phases gives them similar colors, while the contrast between optical microscope and electron microscope makes it difficult to distinguish different phases and microstructures.

### 2.4. Types of SFCA

The results show that[11,12]: although SFCA is defined as a single phase in the sintering process of iron ore, there are still some phases similar to SFCA. Moreover, these SFCA like phases have many different morphologies and chemical properties. Pownceby found a kind of calcium ferrite (SFCA-I) which is very similar to SFCA in structure and chemical composition[5]. Two types of SFCA are shown in Fig. 2. SFCA belongs to low iron and high silicon calcium ferrite with columnar or block shape. The main molecular composition is  $Ca_5Si_2(Fe,Al)_{18}O_{36}$ , while SFCA-I belongs to high iron and low silicon calcium ferrite with main molecular composition of  $Ca_{3.18}Fe_{15.48}Al_{1.34}O_{28}$ , most of them are fibrous or acicular in shape, which makes them superior to SFCA in strength. Therefore, most ironmaking workers and researchers hope to obtain SFCA-I calcium ferrite.

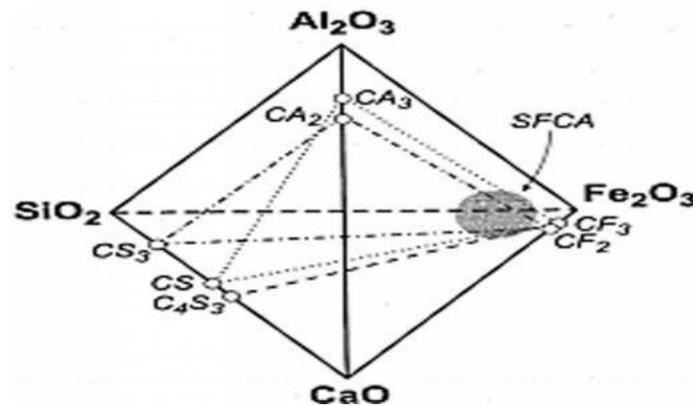


Fig. 1 The complex calcium ferrite solid solution in tetrahedron is in different planes

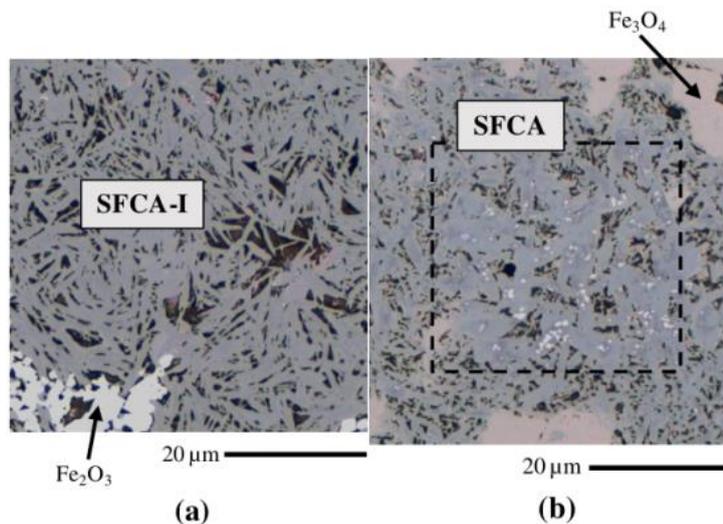


Fig. 2 Two different structural forms of compound calcium ferrite

Mumme et al.[11] prepared a phase  $\text{Ca}_{3.18}\text{Fe}^{3+}_{14.66}\text{Al}_{1.34}\text{Fe}^{2+}_{0.82}\text{O}_{28}$  containing Ca, Al, O and Fe ions, and determined its crystal structure. It was found that this phase is similar to SFCA and named SFCA-I. Although there is  $\text{Fe}^{2+}$  in the crystal structure prepared by Mumme et al., there is no chemical analysis method to determine whether there is  $\text{Fe}^{2+}$  in SFCA-I phase. However, Webster[8] found that SFCA-I samples with mass fraction of 83.5%  $\text{Fe}_2\text{O}_3$ , 12.9% CaO and 5%  $\text{Al}_2\text{O}_3$  contained 1.38wt%  $\text{Fe}^{2+}$ . The content of  $\text{Fe}^{2+}$  in SFCA (volume fraction 80.0%  $\text{Fe}_2\text{O}_3$ , 13.5% CaO, 3.0%  $\text{SiO}_2$  and 4.7 % $\text{Al}_2\text{O}_3$ ) is 0.46%, so SFCA-I can hold more  $\text{Fe}^{2+}$  than SFCA. Mumme *et al.*[11] synthesized the third SFCA, SFCA- II , on the basis of the first two SFCA, and analyzed it by XRD. The phase contains a composition of  $\text{Ca}_{5.1}\text{Al}_{9.3}\text{Fe}^{3+}_{18.7}\text{Fe}^{2+}_{0.9}\text{O}_{48}$ . SFCA- II is considered to be the symbiotic phase of SFCA and SFCA-I, its composition (mass fraction) is 67.3%  $\text{Fe}_2\text{O}_3$ , 12.3% CaO and 20.4 % $\text{Al}_2\text{O}_3$ , and it is named SFCA-II.

### 3. Factors influencing the formation of SFCA

#### 3.1. The influence of aluminum oxide

$\text{Al}_2\text{O}_3$  is an important component of composite calcium ferrite, which can promote the formation of composite calcium ferrite and determine its strength and reducibility [13,14]. Before sintering, there are different modes of occurrence of alumina. With the sintering process, the content of alumina increases continuously due to the enrichment of alumina in the composite calcium ferrite. Because calcium ferrite has good reducibility, and  $\text{Al}_2\text{O}_3$  can promote the formation of calcium ferrite in sinter, it can be seen that appropriate amount of  $\text{Al}_2\text{O}_3$  can also improve the reducibility of sinter[15,16].

Webster *et al.*[17] determined the formation mechanism of SFCA and SFCA-I in the sintering mixture of synthetic iron ore with 1%, 5% and 10%  $\text{Al}_2\text{O}_3$  content by *in-situ* XRD in the temperature range of 298 K to 1623 K (25 °C to 1350 °C) and oxygen partial pressure of  $5 \times 10^{-3}$  atm. They believed that the initial reaction of  $\text{Fe}_2\text{O}_3$  and CaO at 1044 K (771 °C) resulted in the formation of  $\text{Ca}_2\text{Fe}_2\text{O}_5$ . Then, the further reaction of  $\text{Ca}_2\text{Fe}_2\text{O}_5$  with  $\text{Fe}_2\text{O}_3$  results in the formation of  $\text{CaFe}_2\text{O}_4$  in the temperature range of 1241 K to 1252 K (968 °C to 979 °C). The formation of SFCA-I occurs in the temperature range of 1327 K to 1392 K (1054 °C to 1119 °C), which is earlier than that of SFCA in the temperature range of 1380 K to 1437 K (1107 °C to 1164 °C). The temperature range of SFCA-I and SFCA formation changes, which reflects the difference of alumina content. High alumina content increases the temperature range of forming stable SFCA-I, stabilizes SFCA to a higher temperature, and forms  $\text{Fe}_3\text{O}_4$  and melt. Lu *et al.*[18] discussed how  $\text{Al}_2\text{O}_3$  affects the sintering performance of hematite, and found that increasing the content of  $\text{Al}_2\text{O}_3$  in sinter will cause low-temperature reduction pulverization of sinter. Kalenga *et al.*[19] found that with the increase of  $\text{Al}_2\text{O}_3$  content, the concentration of SFCA phase in sinter will increase, but the physical and chemical properties of sinter will also deteriorate sharply. It is found that Al is easy to be dissolved in the composite calcium ferrite by controlling the mass fraction of  $\text{Al}_2\text{O}_3$  between 1.5% and 4%. With the increase of Al content, the materials with strong binding force to Al in calcium ferrite will be attracted. The composition of calcium ferrite is directly related to the content of Al.

#### 3.2. The influence of silicon dioxide

Ding X[20] studied the effect of  $\text{SiO}_2$  on liquid phase formation of sinter at 1473 K (1200 °C). The results show that the increase of  $\text{SiO}_2$  can reduce the melting temperature of CF when the mass fraction of  $\text{SiO}_2$  varies from 0.14% to 6.49%. Matsyno F[21-22] studied the mineral formation process of the mixture of iron ore and limestone with or without coke in the temperature range of 1473K to 1623K (1200 °C to 1350°C). The results show that gangue is easy to dissolve in the melt, and the oxygen partial pressure or  $\text{SiO}_2$  content of the melt determines whether the precipitation form of iron oxide is magnetite or hematite. At higher

temperature, iron oxide coexists with the solution and solidifies as the characteristic structure. Umadevi T[23] studied the change of SiO<sub>2</sub> content in MgO bearing iron ore to compare the change of reducibility after sintering. The experimental results show that SiO<sub>2</sub> promotes the formation of composite calcium ferrite, which is more conducive to the formation of SFCA-I when the silicon content is relatively low. In terms of reducibility, low silicon is better than high silicon. Debrincat D[24] found that increasing the content of SiO<sub>2</sub> in the sinter can make the minerals form pore structure similar to sinter, thus changing the reaction ability of the binder phase with iron oxide.

### 3.3. The influence of magnesium oxide

The results of Yadav U S[25] show that the addition of MgO accelerates the thermal decomposition of hematite, which is conducive to the formation of magnetite phase which is not easy to reduce. Furthermore, the addition of MgO limits the formation of melt and increases the porosity of sinter, which improves the reducibility and reduction pulverization index. Heish L H[26] found that the increase of basicity is beneficial to the formation of calcium ferrite at lower sintering temperature. The content of calcium ferrite decreases with the decrease of the content of alumina. The addition of dolomite increases the content of MgO and decreases the content of calcium ferrite slightly. When MgO is added as serpentine, the content of calcium ferrite increases significantly, and the increase of MgO reduces the amount of secondary hematite. Zhou M *et al.*[27] found that the increase of MgO is not conducive to the formation of calcium ferrite, but the good aspect is to increase the formation of silicate. When the mass fraction of MgO changes from 1.95% to 2.63%, most Mg<sup>2+</sup> is adsorbed by magnetite phase, which stabilizes the magnetite crystal and improves the low-temperature reduction pulverization performance of sinter.

### 3.4. The influence of oxygen partial pressure

In order to study the effect of oxygen partial pressure on the formation of sintered phase, Hsieh and Whiteman[28,29] carried out experiments. Under the control of gas atmosphere, the flake samples made of powder materials were quickly heated from 1453 K to 1283 K (1180 °C to 1255 °C) in a tubular furnace. They found that a moderate oxygen partial pressure of 5×10<sup>-3</sup> atm maximizes the formation of SFCA during heating. In the cooling stage, magnetite can be oxidized to hematite and react with silicate melt to form a large number of columnar SFCA. Webster *et al.*[17] studied the effect of oxygen partial pressure on the formation of SFCA and SFCA-I by *in-situ* XRD, which showed that if the oxygen partial pressure was too low, SFCA-I would not be formed, and both of them were stable in the oxidation state. Webster[17] also studied the effect of alkalinity on the formation of SFCA and SFCA-I, and found that higher alkalinity was conducive to the formation of SFCA-I.

At 1273 K (1000 °C), Edstrom[30] controlled the stage reduction reaction of calcium ferrite by controlling the oxygen partial pressure through the concentration of CO and CO<sub>2</sub> mixture. His research found that the reduction reaction of calcium ferrite was mainly divided into seven stages. When the CO concentration was less than 0.3%, calcium ferrite decomposed into C<sub>2</sub>F with high Ca content and (Fe,Ca)<sub>3</sub>O<sub>4</sub> with high Fe content. When the concentration of CO is between 0.3% and 2.4%, the (Fe,Ca)<sub>3</sub>O<sub>4</sub> is decomposed into C<sub>2</sub>F and Fe<sub>1-x</sub>Ca<sub>x</sub>Fe<sub>2</sub>O<sub>4</sub>. When CO is more than 2.4%, Fe<sub>1-x</sub>Ca<sub>x</sub>Fe<sub>2</sub>O<sub>4</sub> formed in the previous reaction stage reacts with part of C<sub>2</sub>F to form CWF. When CO concentration is higher than 31%, CWF decomposes into C<sub>2</sub>F with high Ca content and CW<sub>3</sub>F with high Fe content. When CO concentration is more than 45%, CW<sub>3</sub>F decomposes into C<sub>2</sub>F and Fe<sub>1-x</sub>Ca<sub>x</sub>O with high Ca content. When CO concentration is higher than 75.8%, Fe<sub>1-x</sub>Ca<sub>x</sub>O is decomposed into calcium ferrite and metallic iron. When the concentration of CO is 78.4%, C<sub>2</sub>F is reduced to metallic iron and calcium oxide containing ferrous oxide. Among them, CWF and CW<sub>3</sub>F are the intermediate products of ternary iron oxides. Ganguly[31] analyzed the reduction behavior of pure ferrite in the gas mixture of H<sub>2</sub>, CO and CO-CO<sub>2</sub>. Weiss

*et al.*[32] studied the reduction process of  $\text{Fe}_2\text{O}_3$  to  $\text{Fe}_3\text{O}_4$ , and found that the system gradually formed micropores.

### 3.5. The influence of other conditions

The results of Li *et al.*[33] show that SFCA can be divided into nickel containing region and nickel free region due to the existence of nickel. The increase of nickel content will greatly reduce the content of SFCA and promote the formation of calcium aluminum silicate. A large amount of  $\text{Fe}_2\text{O}_3$  participates in the crystal transformation of  $\text{Fe}_3\text{O}_4$ , which reduces the amount of  $\text{Fe}_2\text{O}_3$  involved in the formation of calcium ferrite. When the ratio of NiO used to provide nickel is less than 3%, calcium ferrite, hematite and magnetite are basically in cross corrosion state. The porosity and the phase content of silicate glass are low, which is beneficial to sintering production.

Wang and others[34] focused on the formation of SFCA phase under different sintering temperature, CaO/SiO<sub>2</sub> ratio, sintering atmosphere and cooling process conditions. It is found that temperature has a significant effect on the phase content of SFCA, especially in the temperature range of 1523 K to 1573 K (1250 °C to 1300 °C), the formation of SFCA increases with the increase of temperature. The further increase of sintering temperature promotes the reduction of  $\text{Fe}^{3+}$  to  $\text{Fe}^{2+}$  in SFCA crystal structure, which promotes the decomposition of SFCA. Slow cooling in oxidizing gas atmosphere strongly promotes the formation of SFCA. In addition, increasing alkalinity and oxygen partial pressure is beneficial to the formation of SFCA phase. High oxygen partial pressure inhibits the transformation of hematite into magnetite and promotes the formation of SFCA.

Luo *et al.*[35] studied the effect of potassium sodium composite silicate gangue on the formation of composite calcium ferrite by using small-scale experimental device. It was found that when the content of ( $\text{K}_2\text{O} + \text{Na}_2\text{O}$ ) in sinter was low, the CaO content in SFCA decreased with the increase of ( $\text{K}_2\text{O} + \text{Na}_2\text{O}$ ) content in sinter, while when the content of ( $\text{K}_2\text{O} + \text{Na}_2\text{O}$ ) was high, the  $\text{Fe}_2\text{O}_3$  content in SFCA decreased. These two results are not conducive to the reaction of CaO with  $\text{Fe}_2\text{O}_3$  to form calcium ferrite. Hence, the complex silicate gangue containing potassium and sodium in iron ore concentrate has a strong inhibition on the formation of SFCA. When the content of ( $\text{K}_2\text{O} + \text{Na}_2\text{O}$ ) in sinter is low, the content of compound calcium ferrite is high, which mainly exists in fine needle and columnar shape, and is interwoven with iron bearing minerals in network structure. However, when the content of ( $\text{K}_2\text{O} + \text{Na}_2\text{O}$ ) in the sinter is high, some  $\text{K}_2\text{O}$  and  $\text{Na}_2\text{O}$  can enter into the calcium ferrite, which makes the acicular and columnar calcium ferrite with fine grains agglomerate into larger particles and distribute between the iron bearing minerals and the glass phase, thus weakening its binding effect on iron oxide.

Fang and others[36] found that increasing the basicity of low silicon sinter is conducive to the formation of composite calcium ferrite. The needle like SFCA increased with the increase of alkalinity, and reached the peak at alkalinity of 2.8, followed by the formation of columnar or plate-like SFCA, and the binding strength decreased;  $\text{Al}_2\text{O}_3$  is beneficial to the formation of acicular composite calcium ferrite in low silicon sinter of Baotou Steel. When the Al /Si ratio is 0.35, the undissolved  $\text{Al}_2\text{O}_3$  reagent remains, while the composite calcium ferrite is still needle like. There is a common rule that the increase of SFCA is accompanied by the increase of hematite and the decrease of porosity, but the content of glass phase is stable.

## 4. Summary

Calcium ferrite and its composite system SFCA are the main binder phase of high basicity sinter, which directly affects the quality of sinter. SFCA can present different types and structures because of its different chemical composition, gangue content and oxygen partial pressure. The size of gangue and oxygen partial pressure determines the type of SFCA. In recent years, the

research on the formation and crystal structure of calcium ferrite has attracted people's attention. High basicity, low temperature sintering and controlling the reasonable ratio of  $\text{Al}_2\text{O}_3$  and  $\text{SiO}_2$  are the main means to generate SFCA in the sintering process. A series of studies have been carried out by relevant researchers at home and abroad, so that they have some basic understanding of SFCA. However, the relationship between different types of SFCA and sinter quality needs further investigation, which is of great significance for actual production.

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