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Authors: Ömer Saltuk BÖLÜKBAŞI

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A Study on Analysis of Sinter Microstructure and Phase Morphology

Ömer Saltuk BÖLÜKBAŞI*[1]

Abstract

Sinter is a blast furnace input material obtained by temperature to 900-1200 °C without full melting and adhering to each other with superficial melting. It is considered as a multi-phase material with its heterogeneous microstructure. In general, the main mineral phases are hematite, magnetite, silicoferrite of calcium and aluminium (SFCA) and silicates. By determining the SFCA structure in the sinter material, the sintering process will be made more stable and important parameters affecting the quality in the sintering process will be examined. Sinter material consists of iron ore, iron and steel industry by-products and auxiliary materials. The scope of this project is the determination of the amount of SFCA formed by bonding SiO_2, CaO, Fe_2O_3, Al_2O_3 and MgO compounds and monitoring this value as a parameter by sinter manufacturers. Sinter samples having different characteristic features were made ready for X-ray diffraction (XRD) and optical microscopy inspections by polishing, etching and cold mounting in epoxy for mineralogical analyses. Before raw data obtained from the analysis were evaluated at Autoquan, they were converted into Autoquan format and then, read in XRD device and mineralogical composition of the sinter was revealed by XRD analyses. Detailed imaging of mineralogical compounds were made so as to complement scanning electron microscope (SEM) analyses and XRD analyses; elemental composition of the compounds and valence conditions of the elements were determined by energy dispersive spectroscopy (EDS) method. Phase structures such as hematite, magnetite, and calcium ferrite were qualitatively determined by mineralogical investigations on sinter samples. Furthermore, the variations of SFCA phases (SFCA, SFCA-I and SFCA-II) were studied through Rietveld method.

Keywords: Sintering, iron ore, microstructure, mineralogy, SFCA, formation process

1. INTRODUCTION

Sinter is a porous material obtained from partial melting of a mixture of fine iron ore, limestone, dolomite and iron-containing waste materials (flue dust, BOF slag, mill scale, gas cleaning mud). In sinter research; it is important for the technological quality of the sinter to pay attention to the chemical structure, minerology and the distribution of different mineral phase structure forms in the matrix during the sintering process [1]. Sinter production has a dynamic operating parameter and a complex structure. By determining the SFCA structure in the sinter material, the sintering process will be made more

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*Corresponding author: osaltuk.bolukbasi@iste.edu.tr

[1] Iskenderun Technical University, Faculty of Engineering and Natural Science, Department of Metallurgical and Materials Engineering, 31200, İskenderun/Hatay.

E-Mail: osaltuk.bolukbasi@iste.edu.tr

ORCIDhttps://orcid.org/0000-0002-8862-009X

stable to examine important parameters affecting the quality during the sintering process. In the production of the sinter, the bonding phase (SFCA) formed through chemical reactions of SiO_2, CaO, Fe_2O_3, Al_2O_3 and MgO requires to be quantified and this value may be considered by the sinter manufacturers as a parameter to ensure high sinter quality [2].

The researchers agree that the sinter quality depends on the SFCA mineral phase formed during the sintering process [2-5]. During studies on the effects of ore mixtures of different compositions on sinter quality, it has been observed that the reducibility and cold strength of the sinter depends on the presence of calcium ferrite in the structure of the sinter [5-7]. These studies explain that the microstructure consisting of hematite cores surrounded by SFCA-I, one of the SFCA types, is the desired structure for high sinter quality [2, 3, 7-9]. SFCA, the main bond phase of the sinter, is defined as a solid solution $CaO.2Fe_2O_3$ accompanied by Al_2O_3 and SiO_2. This phase is considered to be the SFCA quadruple bond phase complex. SFCA phase is series of solid solutions that can contain Fe^{+2} and Mg^{+2}. [9, 11, 12, 13].

Figure 1-a) shows the typical sintering process of iron ore, fluxes and coke breeze. SFCA phases, calcium-rich ferrite structures, calcium silicates and glassy structures are observed within a typical sinter produced with ore grains in the sinter matrix [9, 14-17]. At low temperatures (<1220 °C) in the sinter body, assimilation is expected to be low and contains a significant amount of residual ore resulting in a heterogeneous texture. A more homogeneous structure is formed in the sinter material due to the high degree of overheating and melt mobility, which occurs at high temperatures (>1300 °C) [18-21]. The homogeneous and heterogeneous formation of the sinter is given in the Figure 1-b).

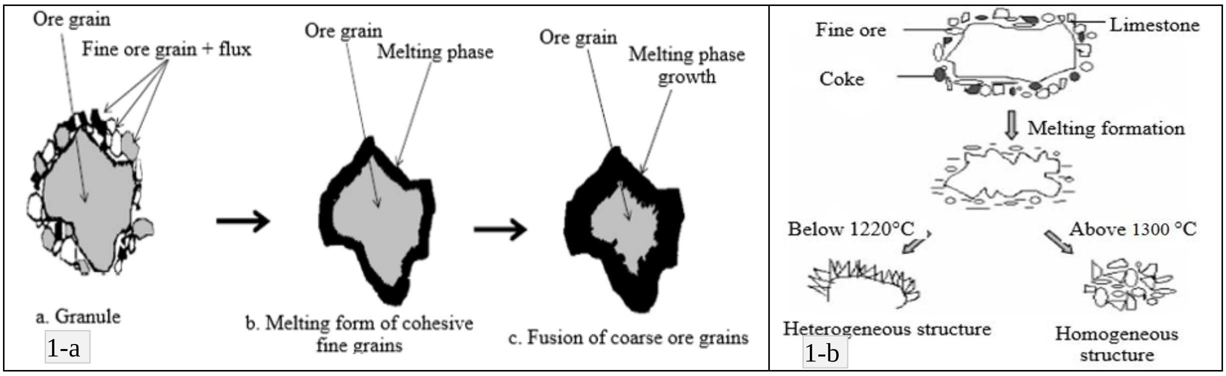

Figure 1-a) Sintering scheme of iron ore, flux and coke breeze [5, 7, 9], b) Homogeneous and heterogeneous formation of sinter [5, 19, 20, 21]

Studies show that the percentage of iron content in raw materials was found to affect the microstructure of the SFCA in the sintering process. While a SFCA form bond structure is obtained in sinter made with iron ore with <62% Fe grade, a form consisting of SFCA and SFCA-I mixture were obtained in the sinter made with 62-65% Fe. SFCA-I was mostly formed in sinter made with 65-68% Fe grade iron ores [14, 22].

In practice, SFCA-I compound structure and SFCA phase structure are seen together. SFCA-I is characterized by a high content of Fe and a low content of Si. The morphology, plate and sections of SFCA-I can be in acicular structures. However, the view of SFCA under the optical microscope is columnar and block-shaped, containing less Fe and it is more stable at high temperature (>1300 ^{o}C). The third type of SFCA phase, as known SFCA-II, can be observed in scattered appearance dendritic structure [9, 19]. In some studies, a third type of SFCA phase structure observed in dendritic morphology and stated that this structure took shape in very thin SFCA-II form and was the first type of SFCA that took shape during the sintering process [7, 18]. This structure is very thin in the form of SFCA-II and it is called the first type of SFCA species that take shape during the sintering process [7, 9, 19, 23]. The studies took into account the microstructure of sinter, and its chemical structure, mineralogy, morphology, and the distribution of different mineral phase structures within the sinter matrix during the sintering process [24, 25].

SFCA phase has been the subject matter of many studies due to its effects on the sinter product such as high strength (TI-Tumbler index), high reducibility (RI-Reduction index) and low temperature degradation (RDI-Reduction degradation index) and has been accepted as the main mineralogical bond in the sintering process [2, 23, 26, 27]. This study will provide information on how to use the Rietveld Method to calculate the ratio of the phases in the sinter SFCA, how to determine the percentage of all compounds in the sinter structure, as well as the quantitative content of the existing phase structures.

2. MATERIAL AND METHODS

The chemical reactions between iron ores and fluxes in the sinter process take place at high temperature (900-1200 °C), and iron ore particles (cores) surrounded with silicoferrite of calcium and aluminium, dicalcium silicate and glassy structure form the basic compound of the sinter cake. Sinter reactions also regulate the volume fraction of different minerals that may affect the quality of the sinter and thus the performance of blast furnace [25].

Sinter matrix structure consists of reactions of flux formers and fine iron ore in the melting phase formation where coarse grains are assimilated and solid-liquid reactions take place. In parallel to preparation of sample in epoxy, sinter samples are powdered through micronizing mill device and before it placed into the device. Iron ores form sinter cake compound in the form of composition consisting of SFCA and dicalcium silicate and glassy structure [28]. Sinter reactions are a special process that continues its activity by controlling the microstructure and concentration of SFCA during the sintering process [15, 29]. A typical industrial scale sinter plant is shown in Figure 2.

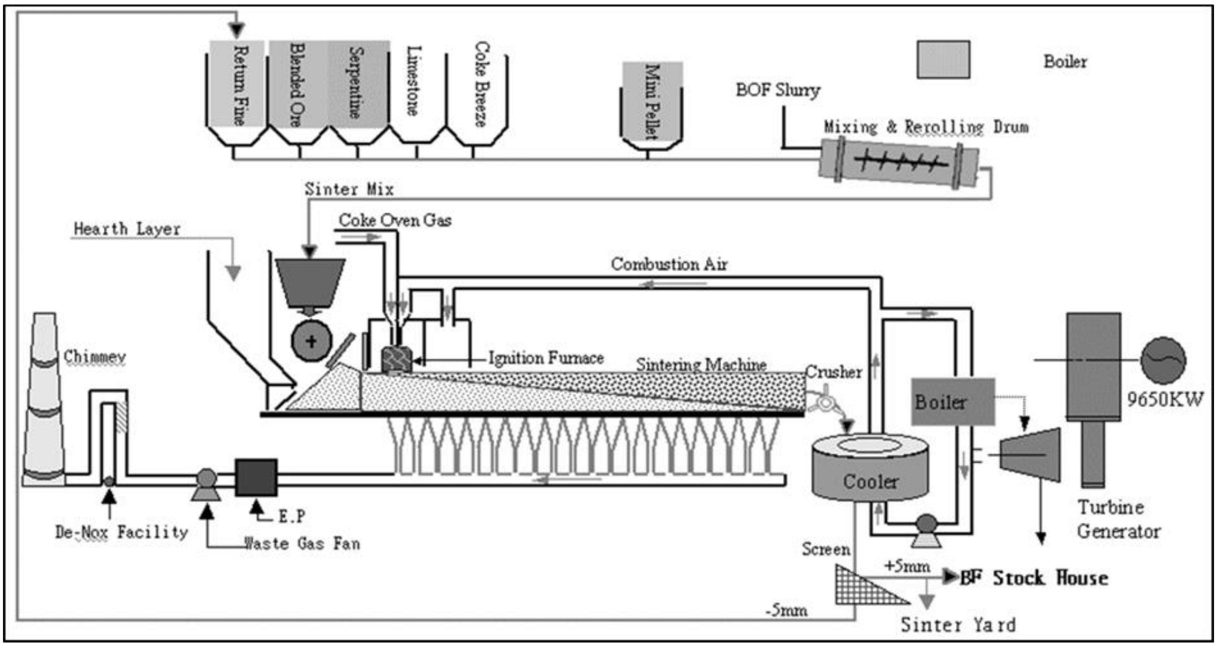

Figure 2 Sinter production flow chart on a typical industrial scale [32].

Sintering parameter: Ignition temperature 1055 °C, sintering time 25 min., pressure drop 1100 (mmH_2O), sintering rate 23 mm/min. Sinter raw material usage values: Domestic iron ore 45%, foreign iron ore 55%, total fuel 72 (kg/t. sinter), Limestone 8.50%, dunite 3.20%, sinter return dust 25.60%, Basicity (CaO/SiO_2) 1.85. Chemical analysis of the sinter is seen in Table 1.

Table 1
Chemical analysis of the sinter (%)

Fe(tot)	**FeO**	**Fe_2O_3**	**CaO**	**MgO**	**SiO_2**	**Al_2O_3**	**K_2O+Na_2O**	**TiO_2**	**MnO**	**S**	**CaO/SiO_2**
55.14	7.00	70.94	9.81	1.57	5,63	1.58	0.25	0.11	0.89	0.02	1.74

Sinter is considered as a multi-phase material due to its heterogeneous microstructure. In general, the main mineral phases include hematite, magnetite, SFCA, ferrite structures and silicates. Sinter mineral formation mainly consists of SFCA, SFCA-I and SFCA-II phases. The key binding phases that provide the strength of the material are SFCA and SFCA-I. These structures were examined considering the contents of mineralogical structures and operational practices (used blend, temperature, sintering time, etc.). Rietveld software programs are designed to accurately define peak profiles as much as possible. The rietveld method directly reports the relative weight fractions of all phase crystals included in the model. The system calculates a series of measurement factors that are directly proportional to the relative quantities of the phases using the least squares method. The results of XRD analysis of the sinter material performed using the Autoquan program by the Rietveld method is displayed in Table 2.

Table 2
Phase structures of the sinter using the Autoquan program (Rwp: error in %).

Sinter	**Phase composition mass%**	**Error%**
	Grade%	**Rwp=2.44%**
C_2S-Larnite	5.87	0.58
Hematite_	21.76	0.35
Magnetite	39.34	0.50
SFCA-I	5.95	1.02
SFCA	26.83	1.07
Wuestite	0.25	0.06
	100.00	

Sinter samples having different characteristic feature will be made ready for optical microscopy inspections by polishing, etching and freezing in epoxy for mineralogical researches (indicated in Figure 3). After polishing, the specimen is analyzed by visual examination using an optical microscopy. Resin, hardener were used by 10 ml and 2,6 ml respectively. Sinter samples were made polishing with 9 micron, 3 micron and 1 micron respectively.

Figure 3 Preparation of sinter samples for mineralogical researches.

The six sample was prepared for XRD analysis and given the standard of verify correct quantification by laboratory of Pretoria. In order to ensure that described sample preparation was adequate, XRD were conducted on the sinter samples. In the XRD analysis of sinter samples, an X-Ray tube with Co K-alpha radiation, which makes measurements more precisely than tubes with Cu K-alpha radiation in iron-containing phases, was used. It was then micronized in a micronizing mill, and then prepared for analysis using a back loading preparation method. It was analyzed with using a Panalytical X'Pert Pro powder diffractometer with X'Celerator detector. The data was collected in the angular range $5^o \leq 2\theta \leq 90^o$ with a step size 0,008^o 2θ and a 13-s scan step time. Phases were identified using X'Pert Highscore plus software and quantification was accomplished with Autoquan/BGMN software. Image of X-ray diffraction instrument was seen in Figure 4.

Figure 4 Sinter sample preparation of X-ray diffraction analysis.

2.1. Sintering reactions and description of bonding phase

When sinter blend consisting of iron ore, limestone, coke breeze and dunite mixture is heated in sinter machine, iron oxide, SFCA and silicate phases are formed in the sinter formation at 1220-1300 ^oC. Dicalcium ferrite (C_2F-$2CaO.Fe_2O_3$) is formed between 750 and 780 ^oC as the first product. With increased sintering temperature, dicalcium ferrites react with hematite to take the form of calcium ferrite (CF-$CaO.Fe_2O_3$). Quartz begins to react with the SFCA-I and SFCA form at 1050 ^oC. SFCA-I breaks down between 1220 ^oC and 1240 ^oC and takes the form of SFCA [17, 23, 30].

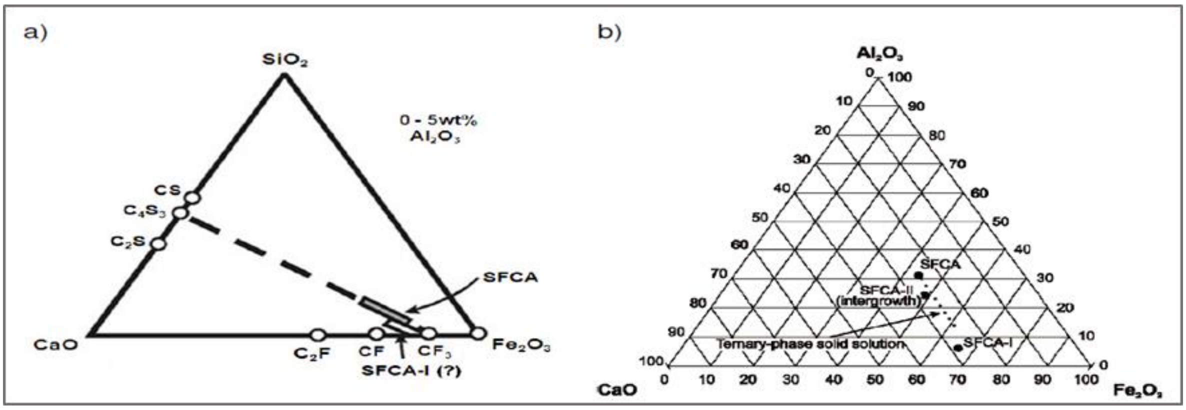

Figure 5-a) Reveals the composition relationship between SFCA and SFCA-I in the quadruple bond system Fe_2O_3-Al_2O_3-CaO-SiO_2. The diagram b) exhibits the formation relationship among SFCA, SFCA-I and SFCA-II [4].

Scientists use the CaO-Fe_2O_3-SiO_2 phase diagram shown in Figure 5 to indicate the mineral compositions of the SFCA phase. The shaded area shown in the Figure 5-a) represents the SFCA composition encountered in the industrial sinters. The SFCA forms shown in the Figure 5-b) are named as SFCA, SFCA-I and SFCA-II (dendritic) solid solution series. In composition, CF_3 (CaO.3Fe_2O_3), C_2F (2CaO.Fe_2O_3) and C_4S_3 (4CaO.3SiO_2) or Fe_2O_3-AI_2O_3-CaO compounds are designed to create a link within a planar structure.

According to many studies, the composition of the phases in the sinter were reported to consist of iron oxides by 40-70%, ferrites by 20-50% (mostly SFCA, silico ferrite of calcium and aluminium), glassy phases by more than 10% and dicalcium silicates by more than 10% [9, 14, 22, 31].

3. RESULTS AND DISCUSSION

The sinter material, its microchemical structure, mineralogy, morphology and the location of different mineral phase structure forms in the matrix during the sintering process have been studied by many scientists [18, 26, 29]. Our study analyses the microstructure of the SFCA phase and the effects of this microstructure on the chemical composition and morphology of the sinter. As a result of the tests, it was found that the SFCA bond structure coexists with the SFCA-I and SFCA-II main phase structures, and the SFCA-I phase structure contains high Fe and low Si. During microscopic examinations, it was observed that the morphology of SFCA-I has a plate-like appearance and its cross sections are acicular (Figure 5-a) and b). It has been determined that SFCA's morphology is columnar and block-shaped, contains less Fe, and it is more stable at higher temperature (>1300 ^{o}C).

A study by Scarlett et al. [17] suggests that the phase compositions in the sinter matrix structure consist of iron oxide by 35 to 60%, ferrites (mostly SFCA) by 20 to 45%, glassy phases by more than 10% and dicalcium silicates by more than 10%. They emphasize that the bond phase morphology is typically composed of SFCA phase composition as well as iron oxides and silicates, forming the most important bond phase structure since SFCA has a major impact on the quality properties of the sinter [18, 19, 26]. Chemical composition of the sinter phases was given in Table 3.

Table 3
Chemical composition of the sinter phases (%).

Sinter Phases	**MgO**	**Al_2O_3**	**SiO_2**	**K_2O**	**CaO**	**MnO**	**Fe_2O_3**	**TiO_2**
Amorphous	0.36	2.17	38.38	0,77	41.32	0.18	16.81	0.00
Larnite	0.31	1.59	38.24	0.65	44.25	0.19	14.78	0.00
Hematite	1.45	0.76	0.08	0.02	1.36	1.99	94.34	0.02

Magnetite	2.41	0.73	0.17	0.01	1.47	1.76	93.45	0.05
SFCA-I	1.58	2.38	4.43	0.05	11.09	0.92	79.56	0.05
SFCA	0.57	3.02	7.99	0.03	15.33	0.49	72.57	0.00
Wuestite	0.00	0.00	0.00	0.00	0.00	0.00	88.13	0.00

In our investigation, it was observed that the quality of the sinter depends on the mineral phase form generated during the sintering process and SFCA is the strongest bond phase that affects the sinter quality. During studies on the effects of ore mixtures of different compositions on sinter quality, it has been observed that the reducibility and cold strength of the sinter depends on the presence of calcium ferrite in the microstructure of the sinter. The literature studies also suggest that the form consisting of hematite cores surrounded by SFCA-I is the desired structure for high sinter quality [7, 18, 19]. During the reduction reactions, the porous structure of the acicular SFCA has been proven to provide a wide surface contact to prevent spread of cracks [10, 30].

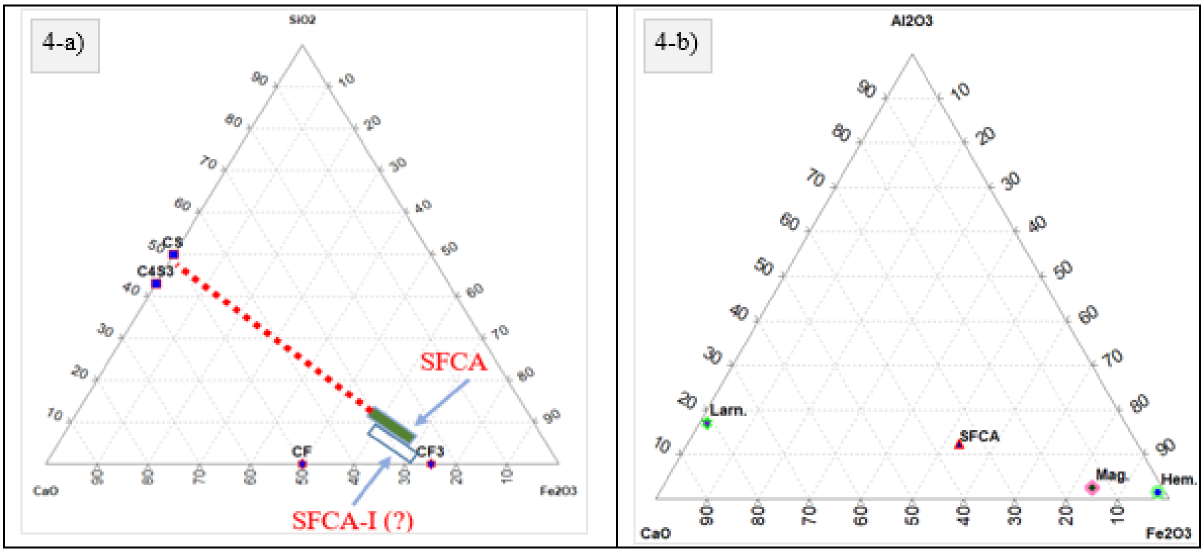

Figure 6-a) exhibits compositional link between SFCA and SFCA-I in the CaO-SiO$_2$-Fe$_2$O$_3$ triple phase diagram in the sinter, b) indicates compositional relationship among the CaO-SiO$_2$-Fe$_2$O$_3$ triple phase diagram and hematite, magnetite, larnite and SFCA.

In the examination of the sinter phase structure (shown in Figure 6); it has been observed that start of mineral compositions, CF$_3$ (CaO.3Fe$_2$O$_3$), CA$_3$ (CaO.3AI$_2$O$_3$) and C$_4$S$_3$ (4CaO.3SiO$_2$) or Fe$_2$O$_3$-AI$_2$O$_3$-CaO compounds can be designed to be able to create a link in a planar structure [13, 18, 19, 30]. In the study, the SFCA composition encountered in the industrial sinter was observed and triple phase diagrams were drawn as SFCA forms, SFCA, SFCA-I and SFCA-II (dendritic) solid solution series [13, 19].

The Figure 7 demonstrates both main SFCA phase structures together. SFCA-I is characterized by a high content of Fe and a low content of Si. As seen in the Figure 7-a), the morphology of SFCA-I can be plate-like and its sections can be acicular. However, as seen in the Figure 7-b), the morphology of SFCA is columnar and block-shaped, containing less Fe and it is more stable at higher temperature (>1300 ^{o}C). The third type of SFCA phase structure can be observed in dendritic morphology. This structure is very thin, in the form of SFCA-II and is the first type of SFCA that took shape during the sintering process [7, 13, 18- 21]. Figure 7-a) and 7-b) show the typical SFCA and SFCA-I phase structures in the sinter material.

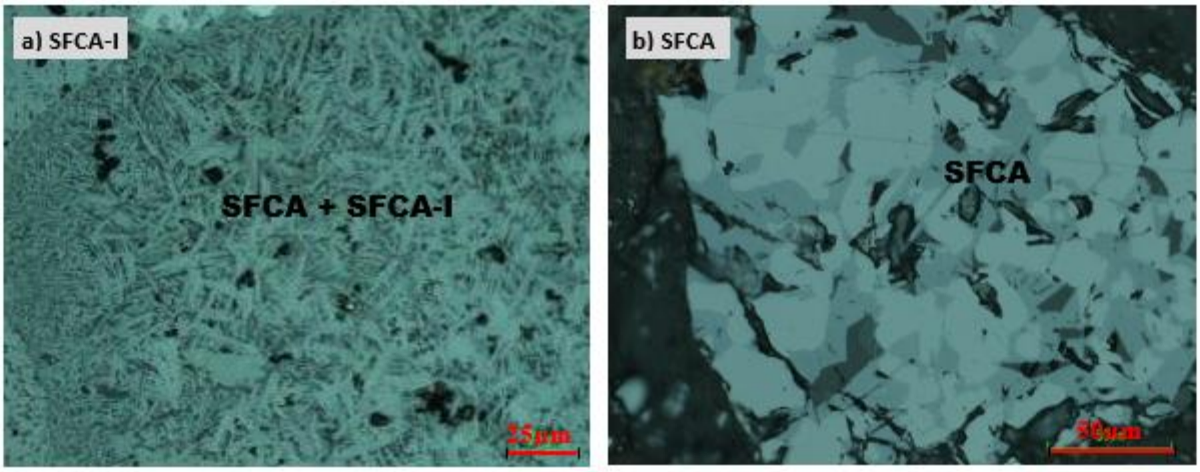

Figure 7-a) and b) show the typical SFCA and SFCA-I phase structures in the sinter material.

Our study also investigated the formation and crystal size of SFCA. Phase structures determined by EDS analysis and the size of each were measured with Image J program. In the examinations, it has been observed that approximately 13% of the structures to be called dendritic crystals are smaller than 4 µm. Where the sintering temperature is 1200-1300 °C, the crystal structure grows, and such large crystals are called acicular SFCA. It has been determined that the SFCA crystals of approximately 24% of the structures to be called acicular structures are larger than 4 µm (microns) and less than 10 µm. During the heating process of acicular SFCA sinter process, it collapsed in the form of columnar SFCA during the cooling phase upon completion of melting at 1300 °C. In this formation, crystal structures were larger and approximately 30% were found to be larger than 10 µm.

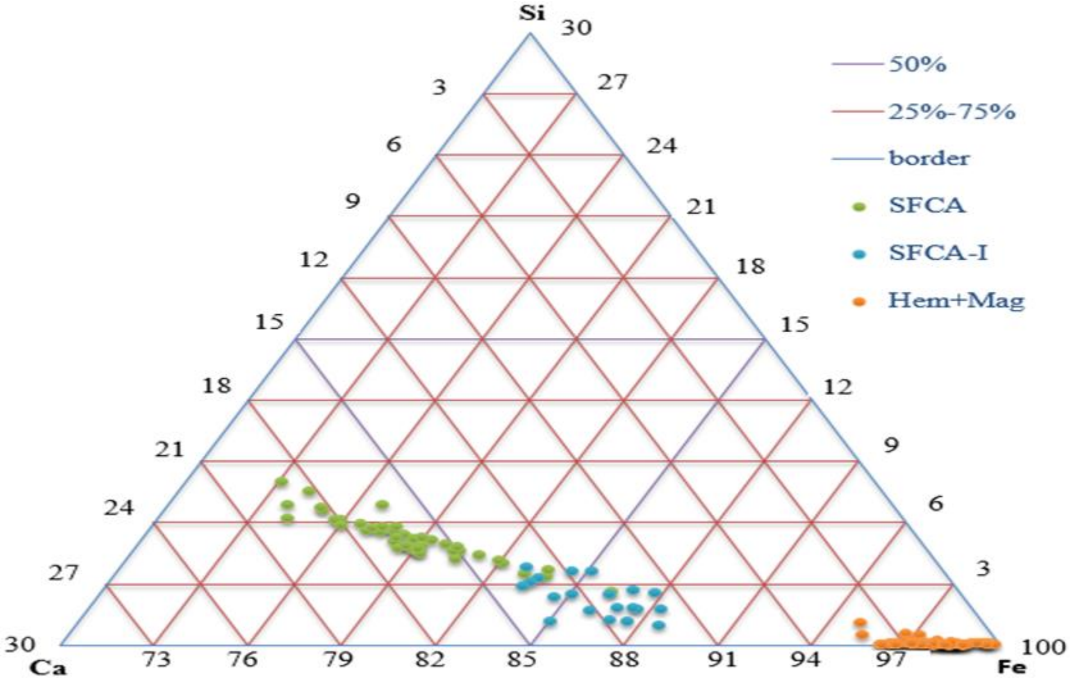

Figure 8 Matrix bond image of hematite, magnetite, SFCA, and SFCA-I phase structures in the sinter structure of the Ca-Si-Fe triple phase diagram.

A ternary phase diagram was obtained with detailed EDS analysis on sinter samples. Sinter types are determined according to the element content of the phase structures according to SEM-EDS analysis. The phase structures of hematite, magnetite and SFCA were characterized with the help of element values (especially Si, Ca, Fe) in sinter. In the Figure 8, the orange colored dots represent hematite and magnetite phase structures, the blue colored dots represent SFCA-I phase structures and the green colored dots clusters represent SFCA phase structures.

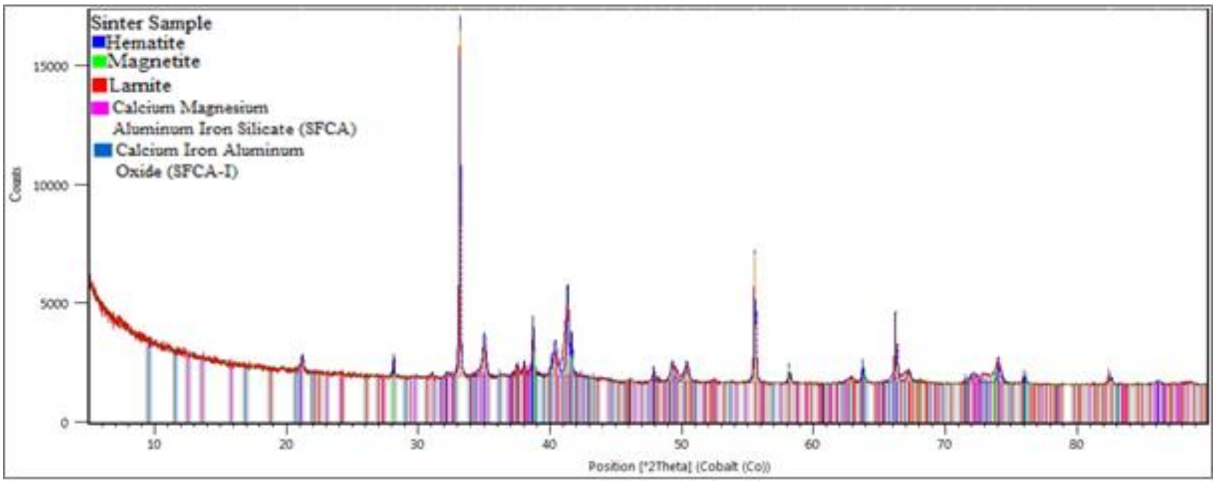

Figure 9 XRD research on Sinter sample with High Score Plus and Rietveld Program.

Figure 9 reveals the Rietveld measurement of the sinter sample. In this study, Autoquan program working with Rietveld method was used to define the phase structures in sinter samples. In this study, the contents of the sinter phase structures were obtained quantitatively (%) by the Autoquan software using the Rietveld method.

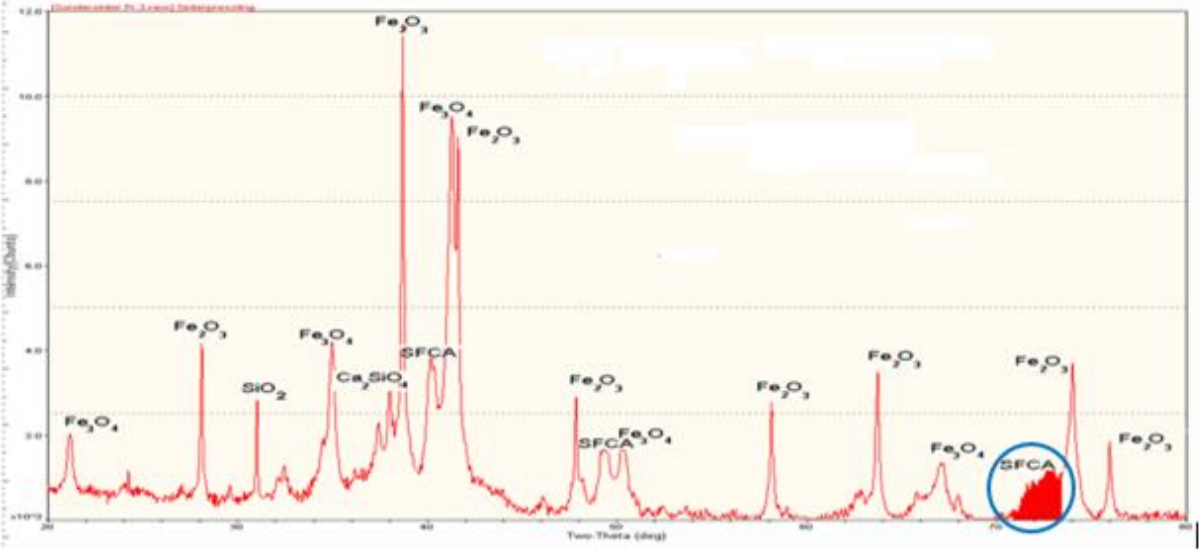

Figure 10 The region where SFCA calculation is made by means of XRD scanning.

Another study we conducted on the determination of the phase structures in the sinter material is given in the Figure 10. In this study, SFCA value is required to be at the rate of 70 %, the area of the region below the angle of 2 Theta between 71°-73°, the region (the red zone in Figure 8) gives quantitative information about the SFCA phase content. The program calculates the area in this region using the integral. If the value is low (<60%), lime is added to the system. If the value is high (>85%), the lime ratio is reduced. It is ensured that the SFCA value in this region remains between 60-85%. However, in this research; it was determined that magnetite (Fe_3O_4), hematite (Fe_2O_3), larnite (Ca_2SiO_4), quartz (SiO_2) and SFCA phases could not be quantitatively determined but only qualitatively. This method will give more suitable results for sinter plants with stable production of raw material and process parameters. Raw material and process conditions may vary in industrial sinter production. For this reason, each facility has to determine its ideal sinter phase morphology and SFCA operating parameters according to its production conditions.

In our study, we mainly focused on two approaches in determining the phase content of the sinter. In the first one, quantitative results related to SFCA is found by using the method of calculating the areas under the peaks containing the SFCA structure. Detailed phase morphology information could not be obtained about SFCA structures. However, changes that may occur in sinter material morphology or process conditions affect the accuracy of the results with the current calculation method. The present method gives proper results in very variable sinter input and more stable process conditions. This approach demonstrates that the results obtained are not healthy, as a result of absorbing the curves obtained with the tube that radiates Cu-alpha, with fluorescence and absorption effect especially in iron-containing materials. Another method was to calculate the ratios of the phases in SFCA with the rietveld method using the tube that radiates Co-alpha (indicated in Tables 2 and 3). Interpretation of the results with the Rietveld method was only possible with the Autoquan program. In the first method, only the area under the SFCA peaks in the region obtained by XRD scanning is calculated by the software. The SFCA value in this region is required to be 70%. In the second method, all parameters (ferrite structures, SFCA types, Fe_2O_3 etc.) that are important in determining the quality in the sinter process are examined. With this method, results can be obtained quantitatively in percentage [33]. Thanks to this method, all changes that may occur in the sinter structure or process conditions can be examined and the sinter phase structures that are likely to occur can be tracked in a more controlled way.

4. CONCLUSION

By calculating the quantitative values of the phases within the sinter, the monitoring and directing of the sinter quality were made more precise with SFCA. A detailed SFCA value cannot be obtained by calculating the area under the peaks in the sinter structure. With the Rietveld Method, the sinter mineralogical structure was fully controlled by calculating all phases in the sinter. Optimum sintering parameters were controlled by the SFCA was more stable blast furnace charge sintering was provided better process control and cost advantage. In the study, it is important to find an optimum SFCA quantity according to mineral compositions and sinter quality which complies with the generally accepted specifications. Sintering plants in the integrated iron and steel plants was followed quality on the basis of small basicity (CaO/SiO_2) ratio. The effects of ferrite structures, wustite (FeO) and aluminum oxide (Al_2O_3), which are important in determining the quality in sinter production, on sinter quality are not examined. By controlling the SFCA in the sinter structure, this quaternary phase matrix (CaO, Al_2O_3, Fe_2O_3, SiO_2) will be taken under control and all parameters that may affect the quality of the process will be examined. With the detection of SFCA in the sinter internal structure, the control of the sinter mineralogical structure will be ensured, and the sinter quality will be improved. Sinter is an important material that is charged to blast furnaces and affects blast furnace efficiency. Mineralogically more stable sinter will be produced with the control of SFCA within the sinter. In this way, more sinter will be charged to the blast furnaces and the raw material costs of the blast furnace will be reduced. Because, the production cost of sinter material is lower than the pellet material charged to the high furnace. With the control of SFCA, sinter quality parameters (basicity, RDI, sieve analysis, etc.) will improve and blast furnace production performance will increase.

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The Declaration of Conflict of Interest/ Common Interest

No conflict of interest or common interest has been declared by the author.

Authors' Contribution

The author solemly performed the computations and wrote the manuscript.

The Declaration of Ethics Committee Approval

The author declare that this document does not require an ethics committee approval or any special permission.

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