

Characterization of Minerals, Metals, and Materials 2020

Edited by

Jian Li

Mingming Zhang

Bowen Li

Sergio Neves Monteiro

Shadia Ikhmayies

Yunus Eren Kalay

Jiann-Yang Hwang

Juan P. Escobedo-Diaz

John S. Carpenter

Andrew D. Brown

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The Minerals, Metals & Materials Series

Jian Li · Mingming Zhang ·
Bowen Li · Sergio Neves Monteiro ·
Shadia Ikhmayies · Yunus Eren Kalay ·
Jiann-Yang Hwang · Juan P. Escobedo-Diaz ·
John S. Carpenter · Andrew D. Brown
Editors

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Editors

Jian Li
CanmetMATERIALS
Hamilton, ON, Canada

Mingming Zhang
ArcelorMittal Global R&D
Scherrerville, IN, USA

Bowen Li
Michigan Technological University
Houghton, MI, USA

Sergio Neves Monteiro
Military Institute of Engineering
Rio de Janeiro, Brazil

Shadia Ikhmayies
Al Isra University
Amman, Jordan

Yunus Eren Kalay
Middle East Technical University
Ankara, Turkey

Jiann-Yang Hwang
Michigan Technological University
Houghton, MI, USA

Juan P. Escobedo-Diaz
University of New South Wales
Canberra, BC, Australia

John S. Carpenter
Los Alamos National Laboratory
Los Alamos, NM, USA

Andrew D. Brown
United States Army Research Laboratory
Adelphi, MD, USA

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Preface

Developing and optimizing industrial processes to form materials to their desired size, shape, and microstructure are in turn deciding the products' properties. This composition–process–structure–property relationship is the key for any materials processing routine. Characterization of properties and microstructure is an important element in determining critical processing parameters. Recent advances in characterization instruments have contributed to an in-depth understanding of materials properties and structure that has enabled much precise process control.

The Characterization of Minerals, Metals and Materials Symposium is sponsored by the Materials Characterization Committee of The Minerals, Metals & Materials Society (TMS). The main focus of this symposium includes but is not limited to advanced characterization of extraction and processing of minerals and the process–microstructure–property relation of metal alloys, ceramics, polymers, composites, and electronic materials. New characterization methods, techniques, and instrumentations are also emphasized.

The Characterization of Minerals, Metals and Materials symposia are one of the largest at each TMS Annual Meeting and attract researchers in the field of materials, minerals, mechanical engineering, chemistry, and physics. At the TMS 2020 149th Annual Meeting & Exhibition held in San Diego, California, USA, this symposium received 222 abstract submissions, of which 122 submissions were accepted for oral presentation in 13 technical sessions, and 100 were accepted as poster presentations. This proceeding volume includes 72 peer-reviewed manuscripts of original research. The authors of the papers represent more than 24 countries from North America, South America, Asia, Europe, Australia, and Africa.

This proceeding publication is a valuable reference for academia and industry that includes advanced characterization methods and instrumentations that cover a wide range of research fields. Readers will enjoy the diversity of topics with novel approaches to process and characterize materials at various length scales.

The editors of this proceeding volume are very grateful to the authors for their contributions and willingness to share their research findings. The editors would also like to thank TMS for providing this valuable opportunity to publish this

stand-alone volume. Appreciations also extend to the Materials Characterization Committee and Extraction and Processing Division (EPD) for sponsoring the symposium. The editors also thank the publisher, Springer, for its timely publication of this book.

Jian Li
Lead Organizer

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About the Editors



Jian Li is a senior research scientist and program manager at CanmetMATERIALS in Natural Resources Canada. He obtained his B.Sc. in Mechanical Engineering from Beijing Polytechnique University, M.Sc. in Metallurgical Engineering from Technical University of Nova Scotia, and Ph.D. in Materials and Metallurgical Engineering from Queen’s University, Kingston, Ontario, Canada. He has a broad experience in materials processing and characterization including alloys deformation, recrystallization, and micro-texture development. He has extensive experience in focused ion beam (FIB) microscope techniques. He is also an expert in various aspects of SEM-EDS and EPMA techniques. He has authored three chapters and published more than 160 papers in scientific journals and conference proceedings.



Mingming Zhang is a lead research engineer at ArcelorMittal Global R&D in East Chicago, Indiana. He has more than 15 years of research experience in the field of mineral processing, and metallurgical and materials engineering. He obtained his Ph.D. degree in Metallurgical Engineering from the University of Alabama and his master’s degree in Mineral Processing from General Research Institute for Non-ferrous Metals in China. Prior to joining ArcelorMittal, he worked with Nucor Steel in Tuscaloosa, Alabama, where he was a metallurgical engineer leading the development of models for simulating slab solidification and secondary cooling processes. He has conducted a number of research projects involving mineral beneficiation,

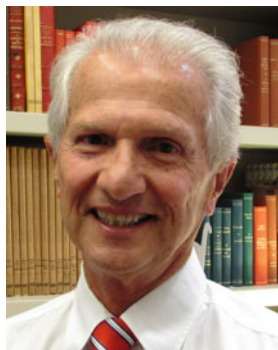
thermodynamics and kinetics of metallurgical reactions, electrochemical processing of light metals, metal recycling, and energy efficient and environmentally cleaner technologies. He has published more than 50 peer-reviewed research papers and is the recipient of several US patents. He also serves as an editor or reviewer for a number of prestigious journals including *Metallurgical and Materials Transactions A and B*, *JOM*, *Journal of Phase Equilibria and Diffusion*, and *Mineral Processing and Extractive Metallurgy Review*. He has made more than 30 research presentations at national and international conferences including more than ten keynote presentations. He was the recipient of the 2015 TMS Young Leaders Professional Development Award. He has served as a conference/symposium organizer and technical committee chair in several international professional organizations including The Minerals, Metals & Materials Society (TMS), Association for Iron & Steel Technology (AIST), and the Society for Mining, Metallurgy & Exploration (SME).



Bowen Li is a research professor in the Department of Materials Science and Engineering and Institute of Materials Processing at Michigan Technological University. His research interests include materials characterization and analysis, metals extraction, ceramic process, antimicrobial additives and surface treatment, porous materials, applied mineralogy, and solid waste reuse. He has published more than 120 technical papers in peer-reviewed journals and conference proceedings, authored/co-authored three books, and edited/co-edited eight books. He also holds 15 patents and has delivered more than 30 invited technical talks.

He received a Ph.D. degree in Mineralogy and Petrology from China University of Geosciences Beijing in 1998 and a Ph.D. degree in Materials Science and Engineering from Michigan Technological University in 2008. He has been an active member in The Minerals, Metals & Materials Society (TMS), Society for Mining, Metallurgy & Exploration (SME), and China Ceramic Society. At TMS, he currently serves as the Past Chair of the Materials Characterization Committee and as a member of the Powder Materials Committee and Biomaterials

Committee. He also has served as an EPD Award Committee member, a *JOM* subject advisor, and a key reader for *Metallurgical and Materials Transactions A*. He has been organizer/co-organizer of a number of international symposia and sessions. He also served as an editorial board member of the *Journal of Minerals and Materials Characterization and Engineering*, *Reviews on Advanced Materials Science*, and *FUTO Journal Series*.



Sergio Neves Monteiro graduated as a metallurgical engineer (1966) at the Federal University of Rio de Janeiro (UFRJ). He received his M.Sc. (1967) and Ph. D. (1972) from the University of Florida, followed by a 1975 course in energy at the Brazilian War College, and a postdoctorate (1976) at the University of Stuttgart. In 1968, he joined the Metallurgy Department of UFRJ as full professor of the postgraduation program in engineering (COPPE). He was elected head of department (1978), coordinator of COPPE (1982), and UnderRector for Research (1983) and was invited as Under-Secretary of Science for the State of Rio de Janeiro (1985) and Under-Secretary of College Education for the Federal Government (1989). He retired in 1993 from the UFRJ and joined the State University of North Rio de Janeiro (UENF), from where he retired in 2012. He is now professor at the Military Institute of Engineering (IME), Rio de Janeiro, and has published more than 1,200 articles in journals and conference proceedings and has been honored with several awards including the ASM Fellowship. He is a top researcher (1A) of the Brazilian Council for Scientific and Technological Development (CNPq) and top scientist of State of Rio de Janeiro (FAPERJ). He was the president of the Superior Council of the State of Rio de Janeiro Research Foundation, FAPERJ (2012), and currently is the coordinator of the Engineering Area of this foundation. He also has served as the president of the Brazilian Association for Metallurgy, Materials and Mining (ABM, 2017–2019), a consultant for the main Brazilian R&D agencies, and a member of the editorial board of five international journals as well as associate editor of the *Journal of Materials Research and Technology*.



Shadia Ikhmayies received a B.Sc. and M.Sc. from the physics department at the University of Jordan in 1983 and 1987, respectively, and a Ph.D. in producing CdS/CdTe thin film solar cells from the same university in 2002. She now works at Isra University in Jordan as an associate professor. Her research is focused on producing and characterizing semiconductor thin films and thin film CdS/CdTe solar cells. She also works in characterizing quartz in Jordan for the extraction of silicon for solar cells and characterizing different materials by computation. She has published more than 50 research papers in international scientific journals, more than 70 research papers in conference proceedings, and three chapters in books. She is the editor of the books *Advances in II-VI Compounds Suitable for Solar Cells Applications* (Research Signpost) and *Advances in Silicon Solar Cells* (Springer), and the eBook series *Advances in Materials Research and Technology* (Springer). She has also served as editor for several TMS Annual Meeting proceedings publications.

She is a member of The Minerals, Metals & Materials Society (TMS) and the World Renewable Energy Network (WREN). She is a member of the international organizing committee and the international scientific committee of the European Conference on Renewable Energy Systems (ECRES2015-ECRES2020). She is a member of the editorial board of the *International Journal of Materials and Chemistry* and has served as guest editor or field editor for six topics published in *JOM* (2014, 2019, and 2020), topical collections published in the *Journal of Electronic Materials* (ECRES 2015–2018), the topical collection “Energy, Power, and Materials Modelling” (SN Applied Sciences), and the journal *Energy Systems*. She is a reviewer for 24 international journals and was the Chair of the TMS Materials Characterization Committee (2016–2017).



Yunus Eren Kalay is an associate professor in the Metallurgical and Materials Engineering Department and an Assistant to the President at Middle East Technical University (METU), Ankara, Turkey. He received his Ph.D. degree from Iowa State University in 2009. His Ph.D. topic was related to the metallic glass formation in Al-based metallic alloy systems. Following his Ph.D., he pursued postdoctoral research at Ames National Laboratory, where he practiced atom probe tomography. In 2011, He joined the Department of Metallurgical and Materials Engineering (METE) of METU as an assistant professor, and in 2014 he was promoted to associate professor. His research interests span microstructural evolution in metallic alloys, rapid solidification of metallic alloys, nanostructured and amorphous alloys, lead-free solders, electronic packaging, and advanced characterization techniques such as scanning and transmission electron microscopy, electron and X-ray spectroscopy, and application of synchrotron X-ray scattering in materials research. He was awarded the “METU Prof. Dr. Mustafa Parlar Foundation Research Incentive Award”, which is a prestigious award that recognizes young scientists in Turkey with exceptional achievements and research productivity. He is also an active member of the TMS Materials Characterization Committee and Phase Transformations Committee and served on organizing committees of three international and one national congress including IMMC, MS&T, and TMS. He is a *JOM* guest editor (2019–2020). He also has been involved in many synergistic activities such as being founding editor of Turkey’s first undergraduate research journal, *MATTER* (<http://matter.mete.metu.edu.tr/>) and organizing materials science camps for K-12 students.



Jiann-Yang Hwang is a professor in the Department of Materials Science and Engineering at Michigan Technological University. He is also the Chief Energy and Environment advisor at the Wuhan Iron and Steel Group Company, a Fortune Global 500 company. He has been the editor-in-chief of the *Journal of Minerals and Materials Characterization and Engineering* since 2002. He has founded several enterprises in areas including water desalination and treatment equipment, microwave steel production, chemicals, fly ash processing, antimicrobial materials, and plating wastes treatment. Several universities have honored him as a guest professor, including the Central South University, University of Science and Technology Beijing, Chongqing University, Kunming University of Science and Technology, and Hebei United University. He received his B.S. from National Cheng Kung University in 1974, and his M.S. in 1980 and Ph.D. in 1982, both from Purdue University. He joined Michigan Technological University in 1984 and served as its director of the Institute of Materials Processing from 1992 to 2011 and the Chair of Mining Engineering Department in 1995. He has been a TMS member since 1985. His research interests include the characterization and processing of materials and their applications. He has been actively involved in the areas of separation technologies, pyrometallurgy, micro-waves, hydrogen storage, ceramics, recycling, water treatment, environmental protection, biomaterials, and energy and fuels. He has more than 28 patents and has published more than 200 papers. He has chaired the Materials Characterization Committee and the Pyrometallurgy Committee in TMS and has organized several symposia. He is the recipient of the TMS Technology Award and of Michigan Tech's Bhakta Rath Research Award.



Juan P. Escobedo-Diaz is a senior lecturer in the School of Engineering and Information Technology (SEIT) at UNSW Canberra. He obtained his doctoral degree in Mechanical Engineering at Washington State University. Prior to taking up this academic appointment, he held research positions at the Institute for Shock Physics at Los Alamos National Laboratory. His main research interests center on the dynamic behavior of materials under extreme conditions, in particular high pressure and high strain rate. His focus has been on investigating the effects of microstructural features on the dynamic fracture behavior of metals and metallic alloys. He has published primarily in the fields of shock physics and materials science. He has been a member of The Metals, Minerals & Materials Society (TMS) since 2011. During this time, he has co-organized more than five symposia at the Annual Meetings including the symposium on Characterization of Minerals, Metals and Materials since 2014. He was awarded a 2014 SMD Young Leaders Professional Development Award.



John S. Carpenter is a scientist within the manufacturing and metallurgy division at Los Alamos National Laboratory, USA. He received his Ph.D. in Materials Science and Engineering from The Ohio State University in 2010 after performing his undergraduate studies at Virginia Tech.

His research focus is on enabling advanced manufacturing concepts through experiments employing novel processing techniques, advanced characterization, and small-scale mechanical testing. Currently, he is working on projects related to the qualification of additively manufactured components and using high-energy X-rays to study phase transformations during solidification in MIG cladding. Throughout his career, he has utilized many characterization techniques including neutron scattering, X-ray synchrotron, XCT, PED, TEM, EBSD, and SEM.

He has more than 55 journal publications, one chapter, and 35 invited technical talks to his credit.

With regard to TMS service, he currently serves as the EPD representative on the Program Committee, SMD representative on the Content Development and Dissemination Committee, and EPD liaison on the

Additive Manufacturing Bridge Committee. He is a participating member of the Mechanical Behavior of Materials Committee and has served as Chair of both the Materials Characterization Committee and the Advanced Characterization, Testing, and Simulation Committee in the past. He serves as a Key Reader for *Metallurgical and Materials Transactions A* and has co-edited special sections in *JOM* related to neutron diffraction, coherent X-ray diffraction imaging methods, and modeling in additive manufacturing. He is the 2012 recipient of the TMS EPD Young Leaders Professional Development Award. He also received the 2018 Distinguished Mentor Award at Los Alamos National Laboratory.



Andrew D. Brown is a mechanical engineer with the Soldier Protection Sciences Branch (FCDD-RLW-PB) of the Weapons and Materials Research Directorate at the Army Research Laboratory (ARL), Aberdeen Proving Grounds, Maryland, USA. He obtained his B.S. in Mechanical Engineering at North Carolina State University (2009) and his doctoral degree in Mechanical Engineering at Arizona State University (2015). He spent three years (2015–2018) at UNSW Canberra as a postdoctoral researcher where he oversaw the daily operations of the Impact Dynamics Laboratory, mentored undergraduate and graduate student research projects, and performed teaching duties. His research expertise is understanding the microstructural effects on the mechanical performance and damage processes of materials subjected to high dynamic pressures (shock) and high strain rates. Since joining ARL in 2018, he has shifted focus to high-rate injury biomechanics research to improve injury outcome prediction and protect the US Warfighter. He has been an active member of TMS since 2011, was a co-organizer for the symposium on Characterization of Minerals, Metals, and Materials in 2018; and has been the symposium's EPD Award Representative from 2017–present. He has published more than 35 peer-reviewed articles in the fields of mechanical engineering, materials science, and biomedical engineering.

Part I
Mineral Processing and Analysis I

Characterization of Iron Ore Sinter Samples by Automated SEM



Mingming Zhang and Marcelo Andrade

Abstract This paper presents a new mineralogical and textural characterization solution for iron ore sinter samples using an automated single scanning electron microscope. It employs a motorized and computer-controlled multiple sample stage. Mosaic images covering large areas of polished sections are acquired to measure the volume/weight fraction of mineral compositions (phase fraction calculation is based on user-defined density per phase). Different classifiers discriminate hematite, calcium ferrite, calcium silico-ferrite, magnesioferrite, calcium silicate and glasses. The entire process is automatic and produces a full pdf report containing typical images and the quantification of mineral phases and grain size. This study represents a convenient method for analyzing mineral phase and grain size in iron ore sinter samples that can allow for quicker results in sintering process control and optimization.

Introduction

Iron ore sinter constitutes an important part of total ferrous burden in modern blast furnace ironmaking operations. Nowadays, most sinter plant operates with large amount of fine materials and reverts; the characteristics of available sintering feed and operation conditions of sinter plant vary from time to time, making it difficult to control and predict production rates as well as sinter quality. Characterization of mineral phases of sinter is a cost-effective and complementary tool to conventional physical and metallurgical testing of iron ore sinter in evaluating and predicting sinter quality. The understanding of sinter mineralogy also can help improve blast furnace operation stability and productivity [1]. This is because the porosity of sinter affects the reducibility of iron ore to pig iron, while its morphological characteristics affect the reducibility and strength of the iron-bearing material in blast furnaces.

Over the years, many schemes have been developed for characterizing iron ore sinter to classify primary sinter phases such as unreacted and partially reacted hematite

M. Zhang (✉) · M. Andrade
ArcelorMittal Global R&D, East Chicago, IN 46312, USA
e-mail: Mingming.Zhang@ArcelorMittal.com

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and magnetite and secondary phases, including prismatic high-temperature silico-ferrite of calcium and alumina (SFCA), secondary hematite and magnetite, glass, larnite (dicalcium silicate; Ca_2SiO_4) and remnant fluxes [2]. Quantification of these phases has traditionally been carried out by visual point counting method under a reflective light microscope; recently, new technologies based on automated optical image analysis, quantitative X-ray diffraction and scanning electron microscopy have been reported and made available for evaluation. Particularly, mineralogical quantification through automated SEM including QEMSCAN[®] can give consistent and reproducible phase recognition because of its automation and may be more robust than point counting reflective light microscopy because of its chemically and backscattered electron (BSE)-based identification [3].

For all SEM-based systems, potential difficulties in mineral phase analysis occur when phases of similar chemistry such as hematite and magnetite also have similar average atomic numbers (i.e., similar BSE intensities). It is therefore important to develop an automated SEM method to differentiate magnetite and hematite based on surface characteristics of the iron oxides being measured (gray scales, porosity, texture, etc.) and the degree of ionic substitution in the iron oxides (Mg, Ca, Al and Mn et al.).

This paper presents an automated SEM method based on stereology and point counting principle to discriminate and measure major phases and grain size in iron ore sinter samples. The automated SEM method makes use of software paradigm to differentiate iron oxide mineral phases with similar chemistry and atomic numbers. In addition, BSE stabilizers, gun alignment, and magnification field size, stage height (working distance) are optimized to determine gray scale of interested phases. The third point BSE calibration standards are adopted and beam dwell times on specific point can be increased to enhance identification to improve discrimination between similar phases. Image analysis and post-processing are also used to eliminate certain artifacts of analysis. Two sinter samples of different chemistry were prepared and characterized using the developed method, and the implication of the results obtained from this quantification method is also assessed. The method presented in this study can be an expedient way to analyze mineral phase and grain size in iron ore sinter samples that can allow for quicker results in sintering process control and optimization.

Materials and Methods

Sample Selection and Preparation

Two set of pot-grate sinter samples were selected as iron ore samples for this study. For each set of sinter samples, ten sinter pieces in the granulometric fraction of 10 to 15 mm were examined. All the samples were cold mounted with epoxy resin and subsequently ground and polished. After an initial examination under a reflective

Table 1 List of samples and their chemical composition

Sinter sample set	Total Fe	FeO	SiO ₂	CaO	Al ₂ O ₃	MgO	CaO/SiO ₂
2-1	53.06	9.37	5.06	13.80	1.5	3.40	2.73
2-2	53.75	9.67	5.00	12.90	1.3	3.11	2.58

light microscope, the cross-section was covered by an evaporated carbon layer to make it conductive and suitable for SEM analysis (Table 1).

Mineral Phase Analysis Method by Automated SEM

A JEOL-JXA 50 scanning electron microscope (SEM) with a Bruker Quantax 200 Energy Dispersive X-ray Spectroscopy (EDS) system was used to acquire a backscattered electron image of each sample. The SEM is also equipped with a multiple sample holder, which makes it easy to analyze multiple samples in one session. The SEM investigation was carried out with 20 keV energy. The chemical analyses could, therefore, be used to determine the different types of mineral phases by comparing these compositions to those reported in the literature [4].

Stereology and point count method were used to correlate the two-dimensional (2D) SEM elemental analysis data with three-dimensional mineral phase volume fraction. The classical rules of stereology are a set of relationships that connect the various measures obtained with the different probes with the structural parameters. The most fundamental rule is that the volume fraction (VV) of a phase within the structure is measured by the area fraction (AA) on the image, or $VV = AA$ [2]. Another way to describe the relationship is, $\langle PP \rangle = VV$ where VV is the volume fraction occupied by the phase being counted and the brackets around $\langle PP \rangle$ signify the expected value for this normalized count, in this case the point fraction. For example, if 100 points are sampled, and 50 are hematite, 35 are calcium ferrite and 15 are glasses, then the volume fraction is estimated at 50% hematite, 35% calcium ferrite and 15% glasses. A key aspect of point counting is in the sampling; this criterion is realized by using a rectangular grid of points with equivalent space between all adjacent rows and columns of points. The point count method is most efficient when the grid spacing is such that adjacent points rarely fall within the same feature, cell or region in the image (they are then said to be independent samples of the structure). An advantage of this method is that when the hits produced by the point grid are all independent, the number of hits can be used directly to estimate the measurement precision. After the volumetric fractions were obtained from point counting method, the mass fractions of the mineral phases were computed based on their theoretical densities as shown in Table 2.

During the automated SEM analysis, the grid points in each field are analyzed. Point counting method uses program logic for moving to a sample, subdividing sample surface into fields and moving to fields, collecting field images, collecting

Table 2 Mineral phase distinction in automated SEM [4]

Mineral phases	Formula	Density (g/cm ³)	Approx. chemical composition	Automated SEM
Magnetite	FeO · Fe ₂ O ₃	5.1–5.2	72% Fe, 28% O	Fe and O signals above threshold value
Magnesioferrite	MgO · Fe ₂ O ₃	4.8–5.0	56% Fe, 32% O, 12% Mg	Mg, Fe and O signals
Calciomagnetite	(Ca, Fe)O Fe ₂ O ₃	5.2	68% Fe, 28% O, 4% Ca	Fe and O signals with low Ca signatures
Wustite	FeO	5.9	78% Fe, 22% O	Fe and O signals close to threshold value
Hematite	Fe ₂ O ₃	5.2–5.3	70% Fe, 30% O	Fe and O signals below threshold value
Calcium ferrite	CaO · 2Fe ₂ O ₃	4.4–4.5	61% Fe, 30% O, 9% Ca	Fe and O signals with high Ca signatures
Calcium silico-ferrite	3CaO · SiO ₂ · 5Fe ₂ O ₃	4.0–4.3	56% Fe, 32% O, 9% Ca, 3% Si	Ca, Si, Fe and O signals that may contain Al
Calcium silicates	xCaO · ySiO ₂	2.8–3.3	34% Ca, 24% Si, 42% O	Ca, Si and O signals (may be different according to CaO/SiO ₂ ratio)
Glass	SiO ₂	2.7	47% Si, 53% O	Si and O signals

and processing EDS spectra, storing particle data, processing stopping criteria, etc. At each point, an EDS x-ray spectrum is first collected, then its elemental composition is quantified, and backscatter intensity is measured with long-term averaging and finally, its mineral species is determined via classification rules. The points can be excluded via thresholding (e.g., exclude sample mounting material). Pseudo-geometry is stored for each point to facilitate post-analysis computation of phase fractions, assay, deportment, etc.

Results and Discussion

Modal mineralogical results obtained by SEM-based image analysis for the two set of sinter samples are shown in Fig. 1 and Tables 3 and 4. The most common elements in iron ore sinter were analyzed including O, Mg, Al, Si, K, Ca, Mn and Fe.

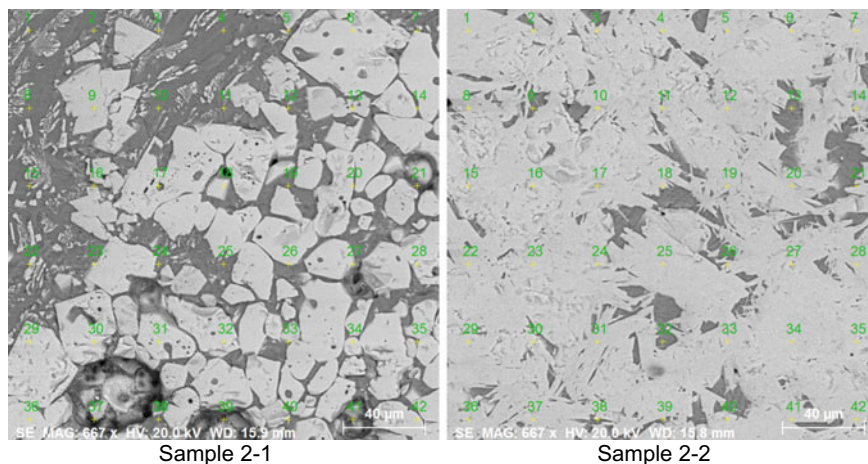


Fig. 1 SEM micrographs of two iron ore sinter samples with point spectrum analyses

Elemental results obtained from each grid point were recalculated to determine the compositions of the different phases present in the sinter samples. The results were clustered according to the elemental analyses and the results recalculated to fit the phases present (i.e., hematite, magnetite, calcium ferrites, calcium silicates, calcium silico-ferrite, calcium silicates and glasses et al.). The main phase composition and grain size of sinter samples are given in Table 5.

Most iron minerals in both sets of sinter samples are hematite/magnetite (47.10% and 48.40%, respectively), followed by calcium ferrite/SFCA (30.80% and 26.22%, respectively), calcium silico-ferrite (14.7% and 16.50%, respectively) and wustite (5.86% and 7.77%, respectively). Minor iron minerals are carried by magnesioferrite (0.05% and 0.01%, respectively), while other iron-bearing minerals are less than 1%.

The mineral phase analysis results based on the automated SEM method agreed well with chemical analysis results on the two sets of sinter samples. The higher mass fraction of hematite/magnetite and wustite in sample 2-2 is consistent with the higher iron content in chemical analysis results of sample 2-2. The higher basicity of sample 2-1 was also confirmed by more calcium ferrite/SFCA mineral phases in sample 2-1 comparing to that of sample 2-2. The differentiation between magnetite and hematite is primarily based on the surface characteristics of iron oxides being measured (gray scale values, porosity and texture) and the degree of ionic substitution being encountered in the iron oxides (Mg, Ca, Al and Mn). However, detailed calcium ferrites and SFCA classification were not easy because classifications of the two phases were based primarily on morphological schemes rather than chemical schemes in addition to inconsistent definitions reported on SFCA and calcium ferrites [5, 6]. Particularly, the definition of SFCA and SFCA-I phases on the basis of chemical analysis was only partially successful, with uncertainty due to limited resolution of data for individual point analyses and problems in direct correlation with

Table 3 Semi-quantitative elemental analysis results on sample 2-1 (all values are given in wt%, normalized)

Spectrum	O	Na	Mg	Al	Si	P	K	Ca	Fe	Possible phases
1	37.48	0.49	1.25	1.13	17.63	0.16	1.18	34.82	5.86	Calcium silicates
2	36.91	0.98	0.67	2.28	19.01	0.34	3.25	26.52	10.04	SFCA
3	32.90	0.03	1.10	0.16	12.62	0.03	0.12	33.89	19.16	Calcium silico-ferrite
4	38.45	0.80	1.73	1.77	17.80	0.23	1.66	28.04	9.50	SFCA
5	36.78	0.55	0.59	0.67	9.00	0.13	0.52	18.89	32.87	Calcium ferrite
6	28.95	0.01	0.01	0.00	0.01	0.00	0.00	0.19	70.83	Hematite
7	32.43	0.22	0.37	0.24	9.97	0.02	0.20	24.66	31.89	Calcium ferrite
8	31.85	0.16	0.58	0.60	18.34	0.11	0.67	44.03	3.66	Calcium silicates
9	27.78	0.43	4.72	0.26	0.58	0.00	0.00	2.14	64.09	Magnesioferrite
10	34.18	0.09	0.34	0.26	17.15	0.03	0.41	45.89	1.63	Calcium silicates
17	36.35	0.38	0.59	1.17	4.95	0.04	0.23	11.32	44.96	Calcium silico-ferrite