

The background of the cover is a photograph of an industrial setting. On the right side, there is a bright, glowing stream of molten metal, likely steel, being poured. The metal is a vibrant yellow-orange color. To the left, there are dark, metallic structures, possibly parts of a furnace or a casting machine, with some steam or smoke rising from the area. The overall scene is dimly lit, with the primary light source being the molten metal.

8th International Symposium on

High-Temperature Metallurgical Processing

EDITED BY

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Tao Jiang
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TMS

 **Springer**

The Minerals, Metals & Materials Series

Jiann-Yang Hwang · Tao Jiang
Mark William Kennedy · Onuralp Yücel
P. Chris Pistorius · Varadarajan Seshadri
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8th International Symposium on High-Temperature Metallurgical Processing

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Preface

This book collects selected papers presented at the 8th International Symposium on High-Temperature Metallurgical Processing organized in conjunction with the TMS 2017 Annual Meeting & Exhibition in San Diego, California, USA. More than 142 abstracts were submitted. Among them, 72 were selected for oral presentation and 60 were provided with poster presentation opportunity. After reviewing the 98 submitted manuscripts, 77 of them were accepted for publication on this book.

As the title of symposium suggests, the interest of the symposium is on thermal processing of minerals, metals, and materials that intends to promote physical and chemical transformations of materials to enable the extraction and production of valuable materials such as metals, alloys, ceramics, and compounds.

The symposium was open to participants from both industry and academia and focused on innovative high-temperature technologies including those based on nontraditional heating methods as well as their environmental aspects such as handling and treatment of emission gases and by-products. Because high-temperature processes require high energy input to sustain the temperature at which the processes take place, the symposium intends to address the needs for sustainable technologies with reduced energy consumption and reduced emission of pollutants. The symposium also welcomed contributions on thermodynamics and kinetics of chemical reactions and phase transformations that take place at elevated temperatures.

This is the seventh book exclusively dedicated to this important and burgeoning topic. We hope the book will serve as a reference for both new and current metallurgists, particularly those who are actively engaged in exploring innovative technologies and routes that lead to more energy efficient and environmental sustainable solutions.

There could not be this book without contributions from the authors of included papers, time and effort that reviewers dedicated to the manuscripts, and help from the publisher. We thank them all! We also want to thank Mrs. Feng Chen for her assistance in collating the submitted abstracts and manuscripts.

 Jiann-Yang Hwang
 Tao Jiang
Mark William Kennedy
 Onuralp Yücel
 P. Chris Pistorius
Varadarajan Seshadri
 Baojun Zhao
 Dean Gregurek
 Ender Keskinilic

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Dr. Hwang received his B.S. degree from National Cheng Kung University 1974, M.S. in 1980 and Ph.D. in 1982, both from Purdue University. He joined Michigan Technological University in 1984 and has 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, microwaves, hydrogen storages, 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

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Tao Jiang received his M.S. in 1986 and Ph.D. in 1990, both from Central South University of Technology. He then joined the university and served as Assistant Professor (1990–1992) and Full Professor (1992–2000). From 2000 to 2003, he was a Visiting Scientist to the Department of Metallurgical Engineering, the University of Utah. Since 2003, Dr. Jiang has been a Professor in the School of Minerals Processing & Bioengineering at Central South University. He has been Specially-Appointed Professor of Chang Jiang Scholar Program of China since 2008 and dean of the school since 2010.

Dr. Jiang's research interests include agglomeration and direct reduction of iron ores, and extraction of refractory gold ores. He has undertaken more than 50 projects from government and industry, including the National Science Fund for Distinguished Young Scholars Program. He and co-workers invented the direct reduction process of composite binder pellets and three plants were set up in China based on the invention. He proposed the innovative composite agglomeration process of iron ore fines, which was put into production in Baotou Steel Company, China. He is actively involved in the areas of utilization of nontraditional ferrous resources such as complex ores and various solid wastes. Dr. Jiang has published 340 technical papers, 6 books including *Direct Reduction of Composite Binder Pellets and Use of DRI*, *Principle & Technology of Agglomeration of Iron Ores*, *Chemistry of Extractive Metallurgy of Gold*, *Electrochemistry and Technology of Catalytical Leaching of Gold*. He holds 39 patents and has more than 35 conference presentations.

Mark William Kennedy is Chief Technology Officer at Proval Partners SA of Lausanne Switzerland and a part time Associate Professor at the Norwegian University of Science and Technology (NTNU), Department of Materials Science and Engineering (as of January 2017).



Onuralp Yücel completed his technical education with a Ph.D. in Metallurgical Engineering from Istanbul Technical University (ITU) where he has held the post of Professor since 2002. He was a Visiting Scientist in Berlin Technical University between 1987 and 1988. He carried out postdoctoral studies at New Mexico Institute of Mining and Technology, Socorro, USA between 1993 and 1994. Professor Yücel has as many as 250 publications/presentations to his credit, which include topics such as technological developments in the production of wide range of metals, ferroalloys, advanced ceramic powders and application of carbothermic and metallothermic processes among others. He was the vice chairman of ITU, Metallurgical and Materials Engineering Department between 2004 and 2007. He was director of ITU, Applied Research Center of Material Science & Production Technologies between 2006 and 2012.

Dr. Yücel's areas of interest include:

Pyrometallurgy: Pretreatment of concentrates (production of WO_3 , Sb_2O_3 , As_2O_3 , MoO_3), smelting and reduction of slags, production ferroalloys, alloys and metals carbothermic and metallothermic processes in EAF or in ladle (copper, cobalt, vanadium, chromium, ferroboration, cobalt boron, nickel boron, ferromolybdenum, ferromanganese, silicomanganese, ferrovandium, ferrotungsten, ferrochromium, nickel-chromium-molybdenum-iron and aluminum-titanium-boron alloys)

Ceramic Powder Production and Processing: Production of carbide, nitride, boride powders and their processing by explosive consolidation or sintering techniques (B_4C , TiB_2 , ZrB_2 , SiC , CrB_2).

Beneficiation of Industrial Wastes; Production of metals and compounds from galvanizing ash, brass production wastes and vanadium sludges produced aluminum production. Grit production from aluminum, copper and steel slags.



P. Chris Pistorius is POSCO Professor and Co-Director of the Center for Iron and Steelmaking Research in the Department of Materials Science and Engineering at Carnegie Mellon University, Pittsburgh, Pennsylvania. His research focuses on pyrometallurgy and electrochemistry. He was Associate Professor (1991–1996) and professor (1997–2008) in the Department of Materials Science and Metallurgical Engineering, University of Pretoria, South Africa, and served as the chair of that department from May 2002 to June 2008. Chris has a Master's degree in Metallurgical Engineering from the University of Pretoria, and a Ph.D. from the University of Cambridge, United Kingdom.



Varadarajan Seshadri had his early education in Madras University, India, did postgraduation in metallurgy from Indian Institute of Science Bangalore in India and he received his doctorate from Technical University of Freiberg in Germany. He served in the Department of Metallurgical Engineering, Indian Institute of Technology, Kanpur and became Professor and Head of the Department of Metallurgical Engineering in Maharaja Sayajirao University of Baroda (1971). He was invited visiting professor in 1978 by the Department of Metallurgical Engineering and Materials, Universidade Federal de Minas Gerais, Belo Horizonte, Brazil, where he has continued ever since. He was also senior Humboldt Fellow in Max-Planck-Institut für Eisenforschung, Düsseldorf, Germany (1971–1973), Visiting Professor/Scientist in Technical University, Aachen, Germany, University of Alberta, Canada and University of Missouri, Rolla, USA, and has also served as UNDP specialist in the National Metallurgical Laboratory India. He has won many awards for his publications in the field of iron and steelmaking, transport phenomena, physical and mathematical modeling. In these areas he has guided many masters and Ph.D. students and has authored more than 250 papers in addition to 3 books. He received the “Distinguished Professor” award from the Universidade Federal de Minas Gerais, Brazil in 2006 for his contributions to undergraduate and postgraduate education as well as industry oriented research in the field of metallurgical engineering. He has served as

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Zhao has received the Spriggs Phase Equilibria Award from The American Ceramic Society (2014), the Billiton Gold Medal from Institute of Materials, Minerals and Mining, UK (2009) and the Non-Ferrous Pyrometallurgy Best Paper Award from Metallurgical Society of CIM, Canada (2009). He has long-term close collaborations with international metallurgical companies including Baosteel, Codelco, Dongying Fangyuan Nonferrous, Rio Tinto Iron Ore, Shougang and Luzhong Refractories.



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International Symposiums on High-Temperature Metallurgical Processing (TMS Annual Meetings 2016 and 2017).



Ender Keskinilic earned his undergraduate degree from the Department of Metallurgical and Materials Engineering of Middle East Technical University (METU), Ankara—the capital city of Turkey—in 1999. He continued M.S. and Ph.D. studies in the same department. He worked as a research assistant in METU between 1999 and 2003. After receiving the master's degree in 2001, he progressed further in the field of extractive metallurgy. During the Ph.D. period, he moved to Ereğli-Zonguldak in 2003 and he worked in Quality Metallurgy & RD Department of Ereğli Iron and Steel Works Co. (ERDEMİR), which is the leading steel company in Turkey regarding the qualities produced and the production capacity. After earning the Ph.D. degree in 2007, Dr. Keskinilic returned to university and he started to work in the Department of Metallurgical and Materials Engineering of Atilim University, Ankara, in 2008. He has been working as a faculty in Atilim since then. He was Assistant Professor between 2009 and 2014. He has been working as an associate professor since 2014. Dr. Keskinilic's primary field of interest is extractive metallurgy and more specifically pyrometallurgical processes such as iron- and steelmaking, ladle metallurgy, ferroalloy production and nonferrous extractive metallurgy.

Part I
Energy Efficient Clean
Metallurgical Technology

Flash Ironmaking from Magnetite Concentrate in a Laboratory Reactor: Experimental and CFD Work

M. Elzohiery, D.Q. Fan, Y. Mohassab and H.Y. Sohn

Abstract A flash ironmaking process is being developed at the University of Utah in which iron is produced directly from magnetite concentrate. The kinetics of magnetite reduction by a mixture of H₂ and CO gases was determined in the temperature range 1150–1600 °C. Over 90% reduction degree was achieved at a temperature as low as 1250 °C within 4–6 s which is the typical residence time available in a flash reactor. The kinetics results were applied to experimental data obtained in a laboratory flash reactor in which H₂ and CO gas mixtures were produced from in situ partial oxidation of natural gas with oxygen. CFD was used to simulate the laboratory reactor, and the results were in good agreement with the experimental data.

Keywords Reduction kinetics · Magnetite concentrate · Hydrogen reduction · Ironmaking · Flash reduction

Introduction

As a part of developing the ironmaking industry, a lot of efforts has been dedicated to reduce the energy consumption as well as the greenhouse gas CO₂ production. The blast furnace (BF) is most dominant ironmaking process which produces over 90% of the world iron from iron ore. The novel flash ironmaking process being developed at the University of Utah [1–8] aims at producing iron directly from iron oxide concentrates by a gas-solid flash reaction utilizing natural gas as the reductant and fuel in temperature range 1150–1600 °C. The direct use of iron ore concentrate (<100 μm) in the flash process will bypass pelletizing and sintering steps in blast

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furnace lowering the energy consumption by 30% [9]. Using natural gas will eliminate the cokemaking step in the blast furnace reducing the CO₂ gas emission by 39–51% [6]. In order to develop and design the industrial flash ironmaking reactor, the reduction kinetics of magnetite concentrates by hydrogen and carbon monoxide gas mixture in temperature range 1150–1600 °C was studied. Using the developed single rate equations, a rate equation for the reduction kinetics by the H₂ and CO gas mixture was established. CFD simulation was used to verify the results of the developed rate equations on the results of a laboratory-scale flash reactor which was built and used for producing iron directly from iron oxide concentrate using natural gas partially combusted with oxygen. The results showed good agreement between the developed rate equations and the experimental results.

Experimental Work

The kinetics of magnetite reduction by H₂ and CO gas mixture was investigated in the temperature range 1150–1600 °C which is the proposed temperature range for the flash ironmaking technology. Most of the previous kinetics studies were either at lower temperature ranges or using hematite ore. In order to get the rate equation for the gas mixture reduction kinetics, it is important to study the reduction kinetics by the single gases.

The magnetite concentrate particles used in this study are of irregular shape and contain oxide gangues. This gangue content results in the particle experiencing fusion at temperatures higher than 1350 °C as was found by experiments. Therefore, it was important to study the kinetics at temperature lower than 1350 °C separately from that at higher temperatures.

Experiments were designed and rate equations were formulated for the reduction of magnetite by H₂, CO and H₂ + CO gas mixture in the temperature range 1150–1350 °C in which the particles remain as solids, and at 1350–1600 °C in which the particles melt. Different size fractions of concentrate particles were used in this work in order to study the effect of particle size. High temperature laminar-flow reactor (LFR) was used to determine the reduction rate of the individual particles. The reactor had a dilute particle-gas conveyed system in which the gas composition was maintained essentially uniform throughout the reactor by using a large excess amount of reducing gas. Water cooled feeding line was inserted into a ceramic tube to avoid any soot formation due to the catalytic effect of the copper tubing that enhances the reduction of CO by H₂ gas and that results in negligible amount of soot formed. A schematic diagram is shown in Fig. 1.

An ICP method, which was developed in our laboratory, was used to determine the iron content in the produced samples. This method has high accuracy in determining iron content in the samples containing high amounts of oxide gangues as in the concentrate used in this work [10].

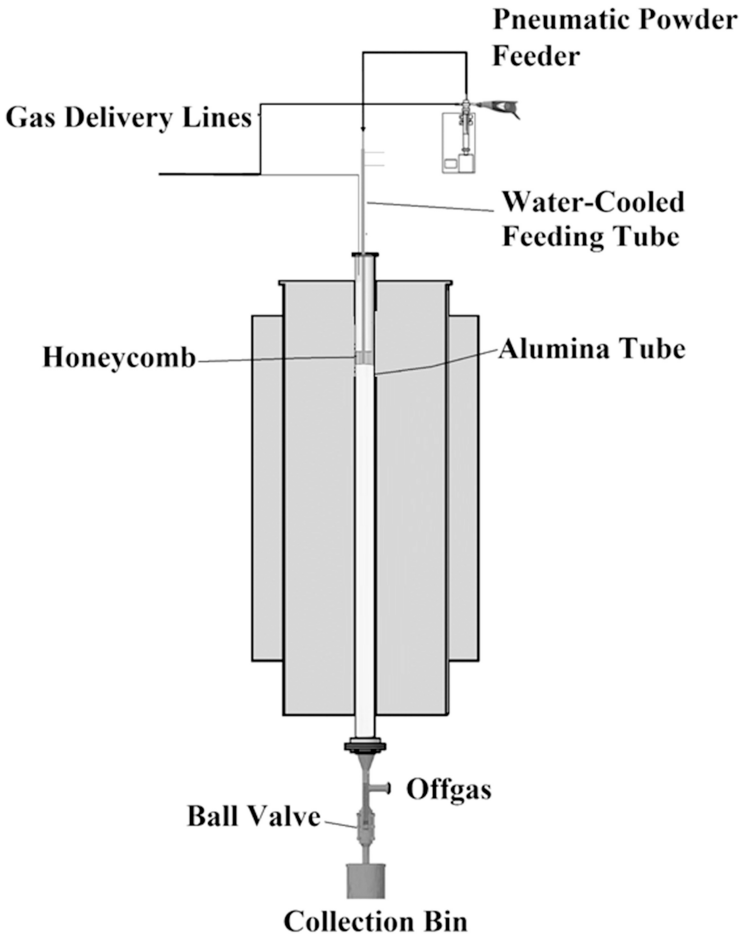


Fig. 1 Schematic diagram for the laminar flow reactor (LFR)

Different experimental conditions were designed in a way to mimic the most used industrial ratios of the syngas such as: $\text{H}_2:\text{CO} = 2:1, 1:1, \text{ and } 0.5:1$ (molar ratio). Similar detailed experimental work, relevant equations for calculating the nominal particles residence time, % excess hydrogen, excess driving force (EDF), reduction degree (RD%), and the length of the reaction zone are described in previous work [8, 11]. The average temperature was calculated up to a certain position where the difference between the average and the actual temperatures was greater than $30\text{ }^\circ\text{C}$. The reaction zone was defined as the zone with temperature $T_a \pm 30\text{ }^\circ\text{C}$ where T_a was the average temperature.

The developed single rate equations were used to formulate a combined rate equation describing the reduction kinetics by the gas mixture. CFD simulation was used to verify the results of these rate equations with the experimental results

obtained from a laboratory scale flash reactor. In this reactor, oxygen/methane form a non-premixed flame where the heat produced is used to heat up the reactor up to 1175 °C. Due to the high volume to surface ratio, heat loss was compensated by electric heating. The partially oxidized methane produced a hydrogen and carbon monoxide mixture that performed the reduction reaction. Different experimental conditions were investigated as the feeding ports and the flame configuration.

Results and Discussion

The aim of this work is to formulate rate equations for magnetite reduction by H₂ and CO gas mixture at the temperature ranges 1150–1350 and 1350–1600 °C. The rate equations for single gas reduction were determined and used for formulating the mixture rate equation. In all rate equations, the effect of reducing gas partial pressure, temperature, particle size, and residence time were studied. These factors represent the main controlling factors in the flash ironmaking technology. The reduction degree of each experiment was calculated based on the amount of oxygen removed after reduction. The reproducibility of the experiments were found to be within ±5%.

Reduction Kinetics Determination

The rate equation for magnetite reduction by hydrogen was found to follow the nucleation and growth kinetics equation with the Avrami parameter $n = 1$. The rate equation had a first order dependence the H₂ gas partial pressure with no particle size effect. The complete rate equation in the temperature range 1150–1350 °C was formulated and the activation energy was 193 kJ as shown in Eq. (1):

$$[-\ln(1 - X)] = 8.65 \times 10^6 \times e^{\frac{-193,000}{RT}} \times \left[p_{H_2} - \frac{p_{H_2O}}{K_{eq}} \right]_{\ln} \times t \quad (1)$$

where R is 8.314 J/mol K, T is in K, p is in atm, and t is in s.

The kinetics of magnetite reduction by CO was found to be much slower than that with H₂. The nucleation and growth equation was used to describe the reduction kinetics with the Avrami parameter $n = 0.4$. Although the Avrami parameter is typically greater than 1, there are some examples of reactions in which it is less than 1. This phenomena occurs when the nuclei growth rate decreases with growth as described by Kim et al. [12] and Hedmark et al. [13]. Wunderlich [14] has analyzed a large amount of data on the rates that follow the Avrami equation, and reported that the Avrami parameter can range from lower than 1 to greater than 6.

The rate equation for magnetite reduction by CO had a first order dependence on CO partial pressure and the activation energy was 479 kJ/mol. There was no effect also for the particle size. The complete rate equation obtained for the reduction of magnetite with carbon monoxide gas in the temperature range 1150–1350 °C was

$$[-\ln(1 - X)]^{1/0.4} = 3.41 \times 10^{14} \times e^{\frac{-479000}{RT}} \times \left[p_{CO} - \frac{p_{CO_2}}{K_{eq}} \right]_{lm} \times t \quad (2)$$

where R is 8.314 J/mol K, T is in K, p is in atm, and t is in s.

The absence of size effect at that temperature range was explained as the particles experience thermal stress and develop cracks of similar dimensions when rapidly heated as they are fed into the reactor at a high temperature. The reaction rate thus depends on the dimension of the solid between the cracks not on the original size. Figure 2 shows that good agreement was obtained between the experimental results and the results obtained by the rate equations.

These rate equations were used to develop a rate equation for the reduction kinetics by CO + H₂ gas mixtures. The presence of CO increased the reduction rate to be higher than the sum of the rates by individual gases. In order to account for the enhanced effects, the reduction contribution due to H₂ was amplified by a constant for the case of reduction by H₂ + CO mixtures. The rate expression for the CO + H₂ gas mixtures in the temperature range 1150–1350 °C was obtained as

$$\frac{dX}{dt} = 1.3 \cdot \frac{dX}{dt} \Big|_{H_2} + \frac{dX}{dt} \Big|_{CO} \quad (3)$$

The comparison of the calculated and experimental results was shown in Fig. 3.

The 1st order model in term of the fraction of solid remaining unreacted describes the reduction kinetics of magnetite concentrate by H₂ in the temperature range 1350–1600 °C. The reaction kinetics had a first-order dependence on the

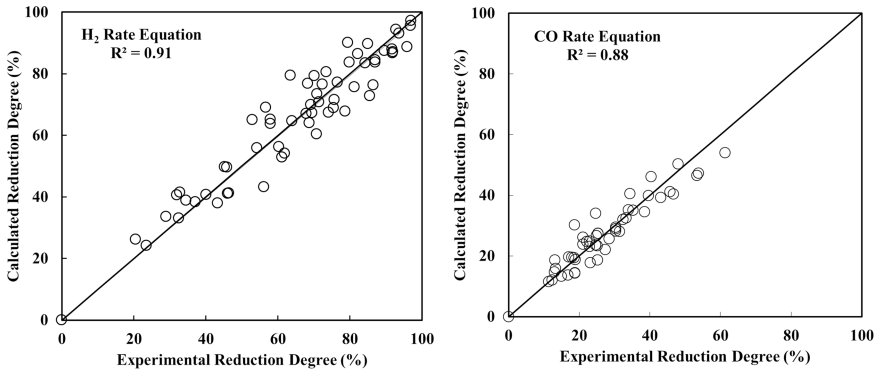


Fig. 2 The agreement between the experimental results and the obtained results by the developed rate equations

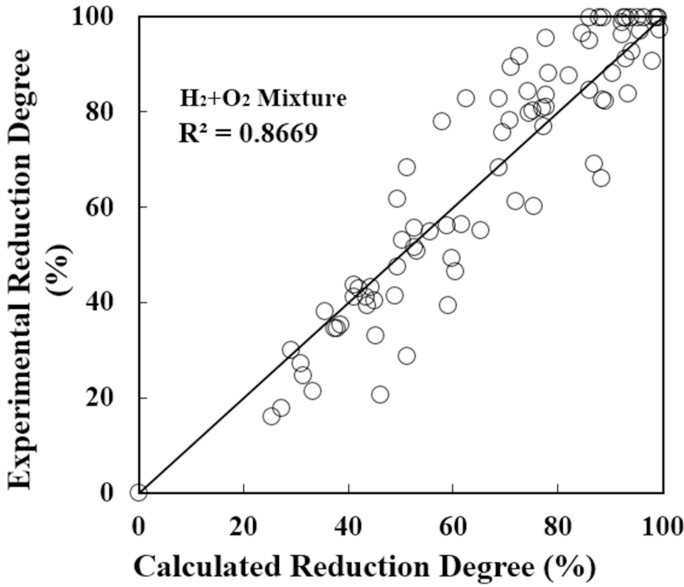


Fig. 3 Comparisons between the calculated reduction degrees versus experimental results with $H_2 + CO$ mixtures as the reducing gas

hydrogen partial pressure with a significant particle size effect on the reduction due to the fusion status of the particles. The activation energy was 177 kJ/mol and the complete rate equation obtained for the reduction of magnetite with hydrogen gas in the temperature range 1350–1600 °C is

$$[-\ln(1 - X)] = 4.2 \times 10^7 \times e^{\frac{-177,000}{RT}} \times (d_p)^{-1} \left[p_{H_2} - \frac{p_{H_2O}}{K_{eq}} \right]_{lm} \times t \quad (4)$$

where R is 8.314 J/mol K, T is in K, d_p is in μm , p is in atm, and t is in s.

The nucleation and growth model described the reduction kinetics of magnetite by CO gas and it was similar to the lower temperature range with an Avrami parameter less than 1 ($n = 0.5$). The dependence on CO partial pressure was found to be of the first order and the activation energy was 49 kJ/mol. The kinetics of reduction by CO had the same dependence on particle size as in the case of reduction by hydrogen. The complete rate equation obtained for the reduction of magnetite with carbon monoxide gas in the temperature range 1350–1600 °C is

$$[-\ln(1 - X)]^{1/0.5} = 4.2 \times 10^2 \times e^{\frac{-49,000}{RT}} \times (d_p)^{-1} \left[p_{CO} - \frac{p_{CO_2}}{K_{eq}} \right]_{lm} \times t \quad (5)$$

where R is 8.314 J/mol K, T is in K, d_p is in μm , p is in atm, and t is in s.

Figure 4 shows good agreement between the experimental and the calculated results.

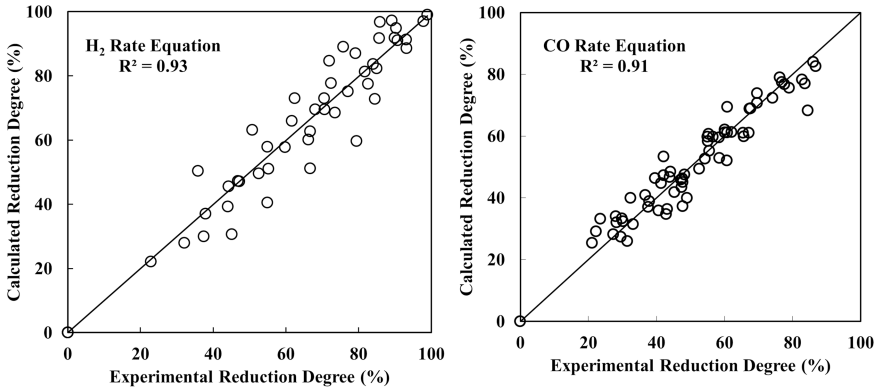


Fig. 4 Agreement between the experimental and calculated reduction degree by the developed rate equations in the temperature range 1350–1600 °C

Table 1 Experimental and calculated reduction degree (%)

CH ₄ flow rate (L h ⁻¹)	O ₂ flow rate (L h ⁻¹)	Experimental (pct)	Calculated (pct)	Residence time (s)
300	270	83 ± 5	90	7.2
600	570	75 ± 5	80	4.9

CFD Simulation of Laboratory Scale Flash Reactor

A laboratory scale flash reactor was used to verify the rate expressions under an actual flash reaction condition. Two typical experimental conditions were simulated and the calculated reduction degrees were compared with the experimental values, which is shown in Table 1.

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