# Characterization of Minerals, Metals, and Materials 2024:

Process–Structure–Property Relations and New Technologies

#### **EDITED BY**

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





## The Minerals, Metals & Materials Series

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

## Characterization of Minerals, Metals, and Materials 2024

Process–Structure–Property Relations and New Technologies





*Editors* Zhiwei Peng Central South University Changsha, China

Jian Li CanmetMATERIALS Hamilton, ON, Canada

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

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

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

Andrew D. Brown Army Research Office Durham, NC, USA Mingming Zhang Baowu Ouyeel Co. Ltd. Shanghai, China

Bowen Li Michigan Technological University Houghton, MI, USA

Rajiv Soman Chem Service Inc. West Chester, PA, USA

Yunus Eren Kalay Middle East Technical University Ankara, Turkey

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

Shadia Ikhmayies Amman, Jordan

ISSN 2367-1181 ISSN 2367-1696 (electronic) The Minerals, Metals & Materials Series ISBN 978-3-031-50303-0 ISBN 978-3-031-50304-7 (eBook) https://doi.org/10.1007/978-3-031-50304-7

© The Minerals, Metals & Materials Society 2024

This work is subject to copyright. All rights are solely and exclusively licensed by the Publisher, whether the whole or part of the material is concerned, specifically the rights of translation, reprinting, reuse of illustrations, recitation, broadcasting, reproduction on microfilms or in any other physical way, and transmission or information storage and retrieval, electronic adaptation, computer software, or by similar or dissimilar methodology now known or hereafter developed.

The use of general descriptive names, registered names, trademarks, service marks, etc. in this publication does not imply, even in the absence of a specific statement, that such names are exempt from the relevant protective laws and regulations and therefore free for general use.

The publisher, the authors, and the editors are safe to assume that the advice and information in this book are believed to be true and accurate at the date of publication. Neither the publisher nor the authors or the editors give a warranty, expressed or implied, with respect to the material contained herein or for any errors or omissions that may have been made. The publisher remains neutral with regard to jurisdictional claims in published maps and institutional affiliations.

This Springer imprint is published by the registered company Springer Nature Switzerland AG The registered company address is: Gewerbestrasse 11, 6330 Cham, Switzerland

Paper in this product is recyclable.

### Preface

This book is a compilation of selected papers from the symposium Characterization of Minerals, Metals, and Materials 2024: Process-Structure-Property Relations and New Technologies at the TMS 2024 153rd Annual Meeting & Exhibition held in Orlando, Florida, USA in 2024. The symposium aimed to provide an international intellectual platform for people to present the state-of-the-art research results in characterization of minerals, metals, and materials and applications of the results on the processing of these materials. It covered a broad range of areas, including, for example, novel methods and techniques for characterizing materials across a spectrum of systems and processes, characterization of mechanical, thermal, electrical, optical, dielectric, magnetic, physical, and other properties of metals, polymers, and ceramics, characterization of structural, morphological, and topographical natures of materials at micro- and nanoscales, characterization of extraction and processing including process development and analysis, and advances in instrument developments for microstructure analysis and performance evaluation of materials, such as computer tomography, X-ray and neutron diffraction, electron microscopy, spectroscopy, and modeling. At the TMS 2024 Annual Meeting & Exhibition, the symposium received 132 abstracts from all over the world, of which 67 abstracts were accepted for submitting manuscripts for publication in the book after peer reviewing.

This book provides a valuable guide and a collection of new and exciting insights for characterization of various minerals, metals, and materials. Scientists, engineers, educators, and students worldwide will enjoy the diverse topics that reflect the authors' brilliant achievements in clarifying the process-structure-property relations and developing novel characterization technologies. The editors of this book wish to extend their sincere appreciation to all the authors and reviewers for their invaluable contributions. They would also like to thank the Materials Characterization Committee and Extraction and Processing Division of TMS for sponsoring the symposium and the publisher, Springer, for its great efforts in making this publication possible.

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

## Contents

#### Part I Advanced Characterization Methods I

Effect of Si on Microstructural and Magnetic Behaviour of Heat-Treated High Carbon Steel Negin Sarmadi, Farshid Pahlevani, Sanjith Udayakumar, Smitirupa Biswal, Clemens Ulrich, and Veena Sahajwalla	3
Estimation Prediction of CaO–SiO <sub>2</sub> –Fe <sub>x</sub> O Slag System Based on Microstructure Analysis Rui Zhang, Ting-an Zhang, Zhihe Dou, and Mao Chen	15
Optimization of Energy Efficiency of Electric Arc Furnace Steelmaking A. Xu, R. Zhu, G. Wei, H. Zhang, and R. Zhao	23
Part II Advanced Characterization Methods II	
Numerical Multi-field Coupling Simulation of Multiple Slab Stacks Heated by Natural Gas Combustion in a Trolley Furnace Bo Liu, Dong Yue, Jiulin Tang, and Liangying Wen	37
Characterization of Mechanical Properties of Viscoelastic Materials Through Experimental Modal Tests Using an Inverse Technique	53
Jagesh Kumar Prusty and Sukesh Chandra Mohanty Part III Mineralogical Analysis and Process Improvement	
Microwave and Conventional Carbothermic Reduction of Chromite Ore: A Comparison Huimin Tang, Zhiwei Peng, Tianle Yin, Lei Ye, Qiang Zhong, and Mingjun Rao	65

Upgrading Iron Ore by Microwave Desulfurization with Reduction of Harmful SO <sub>2</sub> Emission	75	
Lei Ye, Ran Tian, Guanwen Luo, Huimin Tang, Jian Zhang, Mingjun Rao, and Zhiwei Peng	15	
Characterisation and Pre-concentration of a Pegmatite Columbite Ore for Niobium Extraction	. 85	
Abraham Adeleke, Samson Adegbola, and Ayodele Daniyan	05	
Part IV Metallurgical Processing Analysis and Characterization		
Soda-Ash Roasting Behavior of Ludwigite Ore Under Different Oxygen Concentrations	97	
Jinxiang You, Mingjun Rao, Zhiwei Peng, and Guanghui Li		
Effect of Pretreatment During Leaching of Chambishi Copper–Cobalt Air Roast–Leach Calcine Residue Alexander Oniel Noel Old, Yotamu Rainford Stephen Hara, Phenny Mwaanga, Geshom Mwandila, Bawemi Sichinga Mtonga, and Emmanuel Chibwe	107	
Assessing MgO/Al <sub>2</sub> O <sub>3</sub> Effect on Limonitic Laterite Sintering Process Yikang Tu, Yuanbo Zhang, and Zijian Su	117	
Separation of Iron and Phosphorus from High-Phosphorus OoliticHematite Using Direct Reduction and Magnetic SeparationGuangheng Ji, Xu Gao, and Wanlin Wang	127	
<b>Technology Development and Process Optimization</b> <b>of Bottom-Blowing O<sub>2</sub>–CO<sub>2</sub>–CaO in Dephosphorization Converter</b> Xin Ren, Kai Dong, Rong Zhu, and Lingzhi Yang	139	
Production of Zinc Oxide from Willemite Containing Ore from Kabwe Town in Zambia Yotamu Rainford Stephen Hara, Daliso Tembo, Rainford Hara, Ronald Hara, Alexander Oniel Noel Old, and Stephen Parirenyatwa	151	
In Situ Method to Study the Dissolution of Ti(C,N) Inclusion in Molten Mold Flux Li Zhang, Wanlin Wang, Lei Zhang, and Xu Gao	161	
Developing Bottom-Blowing O2–CaO Control Model for Converter         Steelmaking Process         Botao Xue, Kai Dong, Rong Zhu, Lingzhi Yang, and Hang Hu	171	
<b>Emission Characteristics of SO<sub>2</sub> During Roasting of Iron Ore Pellets</b> Rongguang Xu, Yunqing Tian, Dawei Sun, Ruiqing Qian, Xiangjuan Dong, Wenwang Liu, Chengwei Ma, Xiaojiang Wu, Luyao Zhao, Tao Yang, and Li Ma	181	

Reduction of Zn-Bearing Dust Using Biomass Char Jianbo Zhao, Xiaohua Liu, Fupeng He, Yongjie Liu, and Zhixiong You	191
Part V Materials Processing Analysis and Characterization	
Boron Removal from Prepared Rice Hulls Ash Metallurgical-Grade Silicon via Solvent Refining Process B. O. Ayomanor, C. Iyen, G. Ofualagba, J. Umukoro, O. Enamuotor, and E. Omughele	203
Purification of Rutile Ore by HCl and HF Leaching Tong Zhang, Zhiwei Peng, and Shangyong Zuo	213
Quantitative Phase Analysis and Structural Investigation of Graphite Anode for Lithium-Ion Batteries Hammad Farooq, Hilde Johnsen Venvik, and Sulalit Bandyopadhyay	223
Part VI Characterization of Metals	
New Method for the Production of Medium-Mn Steel with Micro-segregation Bands Induced by Sub-rapid Solidification Hui Xu, Wanlin Wang, Peisheng Lyu, and Lankun Wang	237
Part VII Characterization of Polymers, Composites, Coatings and Ceramics	
Preparation of Boronized Ti6Al4V/HA Composites by Powder Sintering for Dental Applications: Effect of Mixing Method Shangyong Zuo, Qian Peng, Tong Zhang, Ting Luo, Yuehong Wang, and Zhiwei Peng	249
Mechanical Properties and EMI-Shielding Efficiencies of Graphite and Iron(II) Oxide-Filled Polypropylene and Polyethylene-Based Polymer Composites Hülya Kaftelen-Odabaşı, Elshod Khakberdiev, Akın Odabaşı, and Selçuk Helhel	259
Phosphoric Acid Leaching of Ni–Co–Fe Powder Derived from Limonitic Laterite Ore Jing Chen, Ding Xu, Zhongxiao Qin, Meishi Hu, Jun Luo, Guanghui Li, Tao Jiang, Xin Zhang, Zhiwei Peng, and Mingjun Rao	269
Preparation of Forsterite-Spinel Refractory from MgO-Rich Residue Derived from Ludwigite Ore Jing Wang, Tao Xiao, Jinxiang You, Jun Luo, Zhiwei Peng, and Mingjun Rao	279
Chemically Bonded Phosphate Ceramics and Their Composites Henry A. Colorado and Mery Gomez-Marroquín	289

Contents
----------

Characterization and Modelling of Triply Periodic Minimum Surface (TPMS) Lattice Structures for Energy Absorption in Automotive Applications N. D. Cresswell, A. A. H. Ameri, J. Wang, H. Wang, P. Hazell, and J. P. Escobedo-Diaz	295
Microwave-Assisted Reduction Behaviors of Spent Cathode Material with Biochar Zhongxiao Qin, Jinxiang You, Mingjun Rao, Xin Zhang, Jun Luo, and Zhiwei Peng	307
Part VIII Poster Session	
Characterisation of 3D-Printed Auxetic Structures Under Low Velocity Blunt Force Impact for the Minimisation of Traumatic Brain Injury in Sport	317
Gracie Jeffrey, Jianshen Wang, Ali Ameri, Paul Hazell, Hongxu Wang, and Juan Pablo Escobedo-Diaz	517
Characterization of a Zeolite Obtained by Means of a Hydrothermal Synthesis Process F. R. Barrientos-Hernández, M. García-Ramírez, María I. Reyes-Valderrama, Julio Juárez-Tapia, M. Reyes-Pérez, X. Álvarez-Álvarez, and K. L. Fuentes-Trejo	333
Characterization of Bacterial Cellulose from Kombucha as a Potential Resource for Its Application on Biodegradable Films R. N. Hernández-Hernández, R. A. Vázquez-García, J. R. Villagómez-Ibarra, R. Velasco Azorsa, N. Islas-Rodríguez, S. Vázquez-Rodríguez, and M. A. Veloz Rodríguez	343
Characterization of Properties of Ceramic Mass Structural Masonry N. A. Cerqueira, J. A. P. Madalena, B. S. Silva, and A. R. G. Azevedo	353
<b>Concrete Using Crushed Rubber as a Substitute for Fine Aggregate</b> Niander Aguiar Cerqueira, Victor Barbosa de Souza, and Afonso Rangel Garcez de Azevedo	361
<b>Cryogenic Toughness of Austenitic Stainless Steels After Aging</b> Maribel L. Saucedo-Muñoz, Victor M. Lopez-Hirata, and José D. Villegas-Cárdenas	369
Effect of Aqueous Ferrous Ion on Collectorless Flotation of Pyrite Martín Reyes Pérez, Esmeralda Camacho Gutierrez, Ramiro Escudero García, Mizraim U. Flores Guerrero, Miguel Pérez Labra, Iván A. Reyes Domínguez, Julio Cesar Juárez Tapia, Francisco Raúl Barrientos Hernández, and Ángel Ruiz Sánchez	379

#### Contents

Effect of Hematite Concentrate on Iron Ore Pellet Quality Yun Wu, Simin Xiang, Fanqiu Zou, Zhiwei Peng, Gaoming Liang, Luben Xie, Xiaoyi Wang, and Qiang Zhong	389
Effect of Raw Material Size on Sintering Quality Jie Liu, Xianguo Ma, Jizhong Tang, Qiang Zhong, Wenzheng Jiang, Hui Zhang, Libing Xv, and Jin Xun	399
<b>Effects of the Rice Husk Ashes and Titanium Dioxide on Properties</b> <b>of ABS Composites Parts Obtained by 3D Printing</b> Gustavo F. Souza, Rene R. Oliveira, Durval Rodrigues Jr, Rita C. L. B. Rodrigues, and Esperidiana A. B. Moura	409
<b>Evaluation of Geopolymer Composites, Based on Red Mud</b> <b>and Metakaolin, for Building Application</b> Cássia Mirelly Milward de Souza, Beatryz Cardoso Mendes, Leonardo Gonçalves Pedroti, and Carlos Maurício Fontes Vieira	421
Evaluation of the Mechanical Properties of GeopolymersManufactured in Molds of Different SizesJ. A. T. Linhares Jr, L. B. Oliveira, D. V. André Jr, T. P. R. de Mello,M. T. Marvila, C. M. Vieria, S. N. Monteiro, and A. G. de Azevedo	431
Evaluation of the Performance of Sustainable Paints Using RedMudJean Carlos Bernardes Dias, Leonardo Gonçalves Pedroti,Márcia Maria Salgado Lopes,Hellen Regina de Carvalho Veloso Moura, and Júlia Lopes Figueiredo	437
<b>Evaluation of the Properties in the Fresh and Hardened State</b> <b>of a Metakaolin Geopolymeric Mortar Reinforced with Açaí Fibers</b> L. B. Oliveira, E. R. G. Júnior, D. V. A. Júnior, J. A. T. L. Júnior, M. T. Marvila, S. N. Monteiro, C. M. F. Vieira, and A. R. G. Azevedo	447
Evaluation of the Properties of Red Ceramics Preparedwith Ornamental RockE. B. Zanelato, A. R. G. Azevedo, M. T. Marvila, J. Alexandre,and S. N. Monteiro	457
Experimental Investigation of the Factors Affecting Performance of Firefighters' Protective Clothing J. Lu, M. Ghodrat, and J. P. Escobedo-Diaz	465
Experimental Investigation on Electroslag System for C-HRA-3 Heat-Resistant Alloy Tengchang Lin, Longfei Li, and Yong Yang	477

Formation of Solid Solutions of BaTiO <sub>3</sub> Doped with Eu <sup>3+</sup> by Solid-State Reaction J. P. Hernández-Lara, A. Hernández-Ramírez, J. A. Romero-Serrano, M. Pérez-Labra, F. R. Barrientos-Hernández, R. Martinez-Lopez, and M. I. Valenzuela-Carrillo	487
<b>Homogenizing Treatment of AISI 420 Stainless and AISI 8620 Steels</b> Victor M. Lopez-Hirata, Maribel L. Saucedo-Muñoz, Karina Rodríguez-Rodríguez, and Héctor J. Dorantes-Rosales	497
Improving the Reduction Swelling Behavior of Fired HematitePellets by Increasing BasicityDeqing Zhu, Bohua Li, Jian Pan, Zhengqi Guo, Congcong Yang,and Siwei Li	507
Influence of Ordinary Portland Cement (OPC) During Collectorless Flotation of Galena Martín Reyes Pérez, Saúl García Pérez, Ramiro Escudero García, Iván A. Reyes Domínguez, Miguel Pérez Labra, Francisco Raúl Barrientos Hernández, Julio Cesar Juárez Tapia, Gustavo Urbano Reyes, and Mizraim U. Flores Guerrero	517
Intensifying Acid Leaching Behaviors of Fe, Ni, and Cr from Stainless-Steel Scraps via Ultrasonic Treatment Yifei Zhang, Qianqian Chu, Bingbing Liu, Guihong Han, and Yanfang Huang	527
<b>Modelling and Simulation of the Scrap Melting in the Consteel EAF</b> Hongjin Zhang, Guangsheng Wei, Afan Xu, Chunyang Wang, and Rong Zhu	537
Mortar Rheology with Partial Replacement of Lime with Dredging Residue I. D. Batista, M. T. Marvila, J. Freitas, E. B. Zanelato, S. N. Monteiro, J. C. Carneiro, G. C. Xavier, L. G. C. H. Silva, J. Alexandre, and A. R. G. Azevedo	549
Nucleation of One Single Sn Droplet on Al Thin Film Explored by Nanocalorimetry Bingjia Wu, Chenhui Wang, Jiayi Zhou, Kai Ding, Bingge Zhao, and Yulai Gao	559
Numerical Simulation of Scrap Preheating with Flue Gas in EAF Steelmaking Process Hang Hu, Lingzhi Yang, Guangsheng Wei, Yuchi Zou, Botao Xue, Feng Chen, Shuai Wang, and Yufeng Guo	569

Contents

Obtaining Ferroelectric Tetragonal Phase Type $Ba_{1-3x}La_{2x}Ti_{1-3x}Bi_{4x}O_3$ ( $0 < x < 0.0075$ ) Using the MechanicalGrinding MethodMaría Inés Valenzuela Carrillo, Miguel Pérez Labra,Francisco Raúl Barrientos Hernández, Ricardo Martínez López,and Martín Reyes Pérez	579
Performance Evaluation of Açaí Fiber as Reinforcement in Coating Mortars J. F. Natalli, I. S. A. Pereira, E. R. G. Júnior, S. A. A. Malafaia, I. D. Batista, M. V. Barbosa, M. T. Marvila, F. M. Margem, T. E. S. Lima, S. N. Monteiro, and A. R. G. Azevedo	587
<ul> <li>Phase Equilibrium in Solid Solutions of BaTiO<sub>3</sub> Doped with Eu<sup>3+</sup></li> <li>and Gd<sup>3+</sup></li> <li>R. Martínez López, M. Pérez Labra, F. R. Barrientos Hernández,</li> <li>M. I. Valenzuela Carrillo, M. Reyes Pérez, J. A. Romero Serrano,</li> <li>A. Hernández Ramírez, and J. P. Hernández Lara</li> </ul>	595
<b>Development of Artificial Granite with Epoxy Resin Matrix Mixed</b> <b>with Cashew Nut Shell Liquid</b> Pablo Barbosa Jacintho, Maria Luiza Pessanha Menezes Gomes, José Lucas Decotê de Carvalho Lírio, Elaine Aparecida Santos Carvalho, Afonso Rangel Garcez de Azredo, Sérgio Neves Monteiro, and Carlos Maurício Fontes Vieira	603
Preparation and Characterization of 3D Printed Biobased Composites from a PBAT/PLA Blend with Lignin and Titanium Dioxide	615
Process Mineralogical Analysis of a Typical Vanadium Titano-magnetite Concentrate Jian Pan, Xin Wang, Deqing Zhu, Zhengqi Guo, Congcong Yang, and Siwei Li	629
Production and Characterization of Artificial Stone for the Making of Permeable Pavement	641
Reducing MgO Content of Blast Furnace Slag Jie Liu, Dongming Zhao, Qiang Zhong, Hui Zhang, Libing Xv, and Jin Xun	653

Reaction Mechanism in EAF Steelmaking Process Based on Selective Oxidation, Bath Stirring and Furnace Body Heat Transfer	663
Lingzhi Yang, Zeng Feng, Yinghui Zhao, Yang Peng, Hang Hu, Yuchi Zou, Shuai Wang, Feng Chen, and Yufeng Guo	
Surface Activation and Directional Modificationin the Technological Properties of Natural PerovskiteUnder the Action of High-Power Electromagnetic PulsesIgor Zh. Bunin, Irina A. Khabarova, and Maria V. Ryazantseva	673
Synthesis and Characterization of TiO2 Nanoparticles by GreenChemistry, Using Aloe VeraR. H. Olcay, I. A. Reyes, E. G. Palacios, L. García, P. A. Ramírez,L. Guzmán, and M. U. Flores	685
Use of Red Mud in Soil Stabilization for Pavement Through Alkali Activation Sarah Souza Silva, Beatryz Cardoso Mendes, Taciano Oliveira da Silva, Emerson Cordeiro Lopes, Flávio Antônio Ferreira, and Leonardo Gonçalves Pedroti	693
Author Index	701 705

## **About the Editors**



**Zhiwei Peng** is a professor in the School of Minerals Processing and Bioengineering at Central South University, China. He received his B.E. and M.S. degrees from Central South University in 2005 and 2008, respectively, and his Ph.D. degree in Materials Science and Engineering from Michigan Technological University, USA, in 2012. His research interests include dielectric characterization, ferrous metallurgy, microwave processing, comprehensive utilization of resources, waste valorization, powder agglomeration, low-carbon technology, process simulation, electromagnetic shielding, and synthesis of functional materials. Dr. Peng has published over 200 papers, including more than 150 peer-reviewed articles in journals such as International Materials Reviews; Journal of Hazardous Materials; ACS Sustainable Chemistry & Engineering; Resources, Conservation & Recycling; Journal of Cleaner Production; Waste Management; Metallurgical and Materials Transactions A; Metallurgical and Materials Transactions B; JOM; Journal of Power Sources; Fuel Processing Technology; Energy & Fuels; IEEE Transactions on Magnetics; IEEE Transactions on Instrumentation and Measurement; Ceramics International; Powder Technology; and Separation and Purification Technology. He holds 68 Chinese patents and has served as an associate editor for Mining, Metallurgy & Exploration, as a guest editor for JOM and Metals, and as an editor for PLOS ONE. He has also been a member of editorial boards of Scientific Reports, Journal of Central South University, and Journal of Iron and Steel Research International, and has served as a reviewer for more than 70 journals. Dr. Peng is an active member of The Minerals, Metals & Materials Society (TMS). He has co-organized 13 TMS symposia and co-chaired 25 symposia sessions since 2012. He is a member of the Pyrometallurgy Committee and the chair of the Materials Characterization Committee. He was a winner of the TMS EPD Young Leaders Professional Development Award in 2014 and the TMS EPD Materials Characterization Award Best Paper—1st Place in 2020.



Mingming Zhang is currently a principal technical officer at Baowu Ouyeel Co. Ltd. in Shanghai, China. He has more than 20 years of experience in the field of mining, mineral processing, smelting, refining, and materials engineering. Before joining Baowu Ouyeel, Dr. Zhang held a principal consultant position at Wood Mackenzie in Chicago and the lead engineer position at ArcelorMittal Global R&D in East Chicago, Indiana. He obtained his Ph.D. in Metallurgical Engineering from The University of Alabama and his master's degree in Mineral Processing from the General Research Institute for Non-ferrous Metals in China. Prior to joining Arcelor-Mittal, 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 process. Dr. Zhang 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 he is the recipient of several U.S. patents. Dr. Zhang also serves as editor and 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.

Dr. Zhang has made more than 30 research presentations at national and international conferences including more than 10 keynote presentations. He was the recipient of the 2015 TMS Young Leaders Professional Development Award. He has served as conference/symposium organizer and technical committee chair in several international professional organizations including The



Minerals, Metals & Materials Society (TMS), the Association for Iron & Steel Technology (AIST), and the Society for Mining Metallurgy & Exploration (SME).

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 broad experience in materials processing and characterization including alloys deformation, recrystallization, and micro-texture development. Dr. Li has experience in Focused Ion Beam (FIB) microscope techniques. He is also an expert in various aspects of SEM-EDS and EPMA techniques. Dr. Li has authored three book chapters and published more than 180 papers in scientific journals and conference proceedings.



**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 150 technical papers in peerreviewed journals and conference proceedings, authored/ co-authored 3 books, and edited/co-edited 15 books. He also holds 16 patents. Dr. Li 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 has served as the chair of the Materials Characterization Committee and as a member of TMS CDDC Committee, Powder Materials Committee, Biomaterials Committee, and EPD Award Committee. He has also served as JOM subject advisor, and 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*. He is the recipient of the AIME Hal W. Hardinge Award (2022).

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 post doctorate (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 as head of department (1978), coordinator of COPPE (1982), Under-Rector for Research (1983), and was invited as Under-Secretary of Science for the State of Rio de Janeiro (1985) and Under-Secretary of the 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), where he retired in 2012. He is now a professor at the Military Institute of Engineering (IME), Rio de Janeiro. Dr. Monteiro has published more than 2,000 articles in journals and conference proceedings and has been honored with several awards including the ASM Fellowship and several TMS awards. He is the top researcher (1A) of the Brazilian Council for Scientific and Technological Development (CNPq) and Emeritus Scientist of State of Rio de Janeiro (FAPERJ). He was president of the Superior Council of the State of Rio de Janeiro Research Foundation, FAPERJ (2012), and currently is coordinator of the Engineering Area of this foundation. He has also served as president of the Brazilian Association for Metallurgy, Materials and Mining (ABM, 2017–2019), as a consultant for the main Brazilian R&D agencies, and as a member of the editorial board of five international journals as well as Executive Editor of the Journal of Materials Research and Technology. He is the author of 150 patents and a top world researcher in "Natural Fiber Composites" and "Ballistic Armor", Scopus 2022.





Rajiv Soman currently serves as Director of Operations, CHEM SERVICE Inc., and is a Scientific Fellow at AnalytiChem Group, USA. Prior to joining AnalytiChem Group, he served as Director, Purity Survey, Materials Science Division, Eurofins EAG Laboratories. He has over 33 years of professional experience in analytical chemistry and materials sciences. He earned a doctorate in Analytical Chemistry from Northeastern University, Boston, USA, and M.Sc. in Applied Chemistry from the Faculty of Technology & Engineering, Maharaja Sayajirao University of Baroda, India. He commenced his professional career as an Advanced Analytical Chemist in the Engineering Materials Technology Laboratories of General Electric Aircraft Engines. Dr. Soman served as Professor (Full) of Chemical Engineering, Chemistry, and Chemical Technology, and as a faculty member for 21 years. He also served as Department Head and Associate Dean. He has received numerous awards for excellence in teaching and twice has been listed in *Who's Who* Among America's Teachers. Dr. Soman's research interests are in the areas of atomic and mass spectrometry, with an emphasis on trace element determination and chemical speciation in a wide range of complex sample matrices. He was an invited guest scientist at the prestigious Forschungszentrum Jülich, Germany, where he conducted research in elemental mass spectrometry. He has co-authored several peer-reviewed publications in international journals and has made numerous presentations at national and international conferences. He holds two U.S. patents. Dr. Soman has been a member of the Society for Applied Spectroscopy (SAS) and the American Chemical Society (ACS) since 1986 and has served in numerous leadership positions in the Professional Societies. He is a member of TMS, where he serves as a member of the Materials Characterization Committee and as Chair of the Poster Awards Committee. He also served as co-organizer for several TMS Symposia and is a reviewer for the TMS proceedings volume Characterization of Minerals, Metals, and Materials and JOM.



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. Dr. Hwang received his B.S. from National Cheng Kung University in 1974, 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, microwaves, 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.

#### About the Editors



Yunus Eren Kalay is an associate professor in the Metallurgical and Materials Engineering Department and assistant to the president at Middle East Technical University (METU), Ankara, Turkey. Dr. Kalay received his Ph.D. with Research Excellence award 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. In 2011, Dr. Kalay 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, high-entropy alloys, electronic packaging, and advanced characterization techniques such as scanning and transmission electron microscopy, electron and X-ray spectroscopy, atom probe tomography, and synchrotron Xray scattering. Dr. Kalay was awarded the METU Prof. Dr. Mustafa Parlar Foundation Research Incentive Award, which is a very 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 served on organizing committees of three international and one national congress including IMMC, MS&T, and TMS. Dr. Kalay has also been involved in many synergistic activities such as being founding editor of Turkey's first undergraduate research journal, MATTER, and organizing the Materials Science Camps for K-12 students.



Juan P. Escobedo-Diaz is a senior lecturer in the School of Engineering and 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 and 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 Minerals, Metals & Materials Society (TMS) since 2011. During this time, he has been a co-organizer of the symposium on Characterization of Minerals, Metals, and Materials since 2014. He was awarded a 2014 SMD Young Leaders Professional Development Award.



John S. Carpenters is a scientist within the manufacturing science and metallurgy division at Los Alamos National Laboratory. Dr. Carpenter received his Ph.D. in Materials Science and Engineering from The Ohio State University in 2010 after performing his undergraduate studies at Virginia Tech. Dr. Carpenter's research focuses 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, the development of new materials for high field magnets through severe plastic deformation and the use of 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 75 journal publications and 50 invited technical talks to his credit. With regard to TMS service, Dr. Carpenter is a past chair for the Materials Characterization Committee, the Advanced Characterization, Testing & Simulation Committee, and the Joint Commission for Metallurgical and Materials Transactions. He is also the Program Committee Representative for the MS&T Conference and the current Vice Chair for the Extraction and Processing division. He is the 2021 recipient of the McKay-Helm Award from the American Welding Society and is the 2018 recipient of the Distinguished Mentor Award at Los Alamos National Laboratory.



Andrew D. Brown serves as Technical and Core Mission Support for the Sciences of Extreme Materials Competency Branch at the Army Research Office, a directorate of the U.S. Army Combat Capabilities Command Army Research Laboratory (ARL). Prior to joining ARO in 2022, Dr. Brown served in the Terminal Effects Division at ARL since 2018. Dr. Brown's research expertise is in the areas of advanced materials characterization, the mechanical response of structural materials subjected to high strain rate loading, and injury biomechanics. He obtained a B.S. in Mechanical Engineering at North Carolina State University (2009) and a doctoral degree in Mechanical Engineering at Arizona State University (2015). He then worked as a postdoctoral researcher in the Impact Dynamics Laboratory at the University of New South Wales Canberra at the Australian Defence Force Academy (2015–2018). He has been an active member of TMS since 2011, a co-organizer for the Characterization of Minerals, Metals, and Materials symposium (2018, 2020-present), and was the symposium's EPD Awards Representative from 2017-2022. Dr. Brown has published over 40 peer-reviewed articles in the fields of mechanical engineering, materials science, and biomechanics.



**Shadia Ikhmayies** received her B.Sc. and M.Sc. from the physics department in the University of Jordan in 1983 and 1987 respectively, and her Ph.D. in producing CdS/CdTe thin film solar cells from the same university in 2002. Her research is focused on producing and characterizing semiconductor thin films, and thin film CdS/CdTe solar cells. She works in characterizing quartz in Jordan for the extraction of silicon for solar cells and characterizing different materials by computation. Dr. Ikhmayies published 59 research papers in international scientific journals, 86 research papers in conference proceedings, and 3 chapters in books. She is the founder and editor of the book series "Advances in Material Research and Technology" published by Springer, and the editor in chief/editor of several books.

Dr. Ikhmayies is a member of The Minerals, Metals & Materials Society (TMS) where she was the chair of the Characterization Committee in 2016 and 2017, and the leading organizer of three symposiums; Solar Cell Silicon 2017-2020, Mechanical Characteristics

and Application Properties of Metals and Non-metals for Technology: An EPD Symposium in Honor of Donato Firrao, and Green Materials Engineering: An EPD Symposium in Honor of Sergio Monteiro. Dr. Ikhmayies is also a member of the World Renewable Energy Network/Congress (WREN/WREC) 2010present. She is a member of the international organizing committee and the international scientific committee in the European Conference on Renewable Energy Systems (ECRES2015-ECRES2023). She is a guest editor and a member of the editorial board of several journals including JOM and the Journal of Electronic Materials. Dr. Ikhmayies has served as a reviewer for 24 international journals and several international conference proceedings. She has received several international awards including the TMS Frank Crossley Diversity Award 2018 and World Renewable Energy Congress 2018 Pioneering Award.

## Part I Advanced Characterization Methods I

## Effect of Si on Microstructural and Magnetic Behaviour of Heat-Treated High Carbon Steel



Negin Sarmadi, Farshid Pahlevani, Sanjith Udayakumar, Smitirupa Biswal, Clemens Ulrich, and Veena Sahajwalla

**Abstract** Silicon (Si) is a versatile alloying element that can enhance the performance of high carbon steel by contributing to deoxidation, grain refinement, hardenability, strength, and toughness. The study investigates the influence of Si (0.8-3.6%)on the microstructural and magnetic properties of high carbon steel. The arrangement and density of magnetic moments, which are affected by the microstructure determines the saturation magnetisation of the material. Industrial-grade high carbon steel samples with varying silica were microstructurally tailored to obtain multiphase steel microstructures. The optical and SEM analyses revealed a combination of cementite and plate martensite, wherein the martensitic structures became finer with higher Si content. The evolution of martensitic microstructures along with mild oxidation of Si inclusions in the grain boundaries of the surface were observed using Confocal microscopy. An increase in the cementite with an accompanied decrease in the martensitic fraction was determined by the quantitative analysis of the Xray diffractograms. The saturation magnetisation  $(M_S)$  of the samples witnessed a gradual decrease with increase in the Si content. Silicon's role in high carbon steel is quite notable, therefore, the nature of heat treatment and level of Si addition can be adjusted to limit the formation of cementite and increase the ferritic martensite or austenite in the microstructure.

Keywords High carbon steel  $\cdot$  Heat treatment  $\cdot$  Saturation magnetisation  $\cdot$  Silicon  $\cdot$  Martensite

© The Minerals, Metals & Materials Society 2024

Z. Peng et al. (eds.), *Characterization of Minerals, Metals, and Materials 2024*, The Minerals, Metals & Materials Series, https://doi.org/10.1007/978-3-031-50304-7\_1

N. Sarmadi (⊠) · F. Pahlevani · S. Udayakumar · S. Biswal · V. Sahajwalla Centre for Sustainable Materials Research and Technology, SMaRT@UNSW, School of Materials Science and Engineering, UNSW Sydney, NSW 2052, Australia e-mail: n.sarmadi@unsw.edu.au

#### Introduction

Steel stands as humanity's most vital structural resource, finding utility across all aspects of our daily existence and within the realms of industry. Its prevalence throughout various applications has led to its growing significance over time, establishing it as an indispensable alloy [1]. Dual-phase steels represent a recent category of steels. Their defining feature is a structure comprising tough martensite particles evenly spread within a pliable, soft matrix. The descriptor "dual phase" highlights the prevalence of two main components in the structure [2, 3]. High carbon dual-phase steel usually encompasses martensite and retained austenite phases, providing remarkable toughness, hardness, and resistance to abrasion. These characteristics make it well-suited for use in extreme operating conditions and environments [4].

Silicon (Si) is an important alloying element for the enhancement of properties in various grades of steel. Si generally enhances steel's strength and hardness, though it's less effective than manganese in this regard. It improves acid resistance, promotes larger grain sizes for higher magnetic permeability, aids crystal orientations in electrical steels, and bolsters oxidation resistance in high-temperature service steels [5]. High carbon steel with increased Si content can be quenched to form martensite more easily, resulting in greater hardness and wear resistance. Introducing Si raises the strength of dual-phase steels following annealing and gradual cooling, owing to the formation of a substantial amount of uniformly distributed, easily hardenable austenite. This process results in a significant presence of martensite within the final structure [6]. In recent advancements in high-strength steel development, a notable emphasis has been placed on incorporating retained austenite into the microstructure of martensitic steels. The objective is to elevate the ductility of the steel without excessively compromising its strength. Si is recognized for its capacity to hinder the development of cementite, likely attributed to its notably limited solubility within cementite. Theoretically, by impeding the precipitation of carbides, a greater quantity of carbon becomes accessible for allocation into retained austenite. Consequently, the introduction of Si has been documented to augment the ultimate proportions of retained austenite or elevate the carbon content within carbon and lowalloy steels [7-10]. However, high carbon steel typically contains cementite (Fe<sub>3</sub>C) in its microstructure, which contributes to its hardness. Excessive Si can lead to the formation of excess cementite, which can affect the steel's properties. Moreover, Si is ineffective in retarding the precipitation of cementite if the parent phase is highly supersaturated with carbon [11]

The current work explores the effect of Si addition with variable amounts on the microstructural aspect of high carbon steel. The steel samples with varying Si content were subjected to heat treatment followed by quenching. Various characterisation techniques were employed to understand the evolution of microstructural phases along with magnetic behaviour with respect to Si content. Subsequently, the magnetic behaviour of the heat-treated steel was assessed, as the arrangement of magnetic moments within a material, and consequently, its saturation magnetization ( $M_S$ ), can be significantly influenced by the microstructural characteristics of the material.

#### **Materials and Methods**

The study was conducted for industrial-grade high carbon steel with a chemical composition of 1.0%C-1.0%Mn-0.4%Cr (in wt.%) with three different concentrations of Si. The microstructure of these steels was initially a combination of martensite and a significant amount of retained austenite. The concentration of Si varied between 0.8 and 3.6 wt.%. The homogenised steel samples were subject to heat treatment, wherein the mode of heat treatment aimed to limit the microstructure to a dual-phase steel. The varying concentrations of Si in the high carbon steel may have both positive and negative effects on the desired microstructure. The sample composition, as determined using Laser-induced breakdown spectroscopy (LIBS) is provided in Table 1.

#### *Heat Treatment Using High-Temperature Confocal Microscopy (HTCM)*

The high carbon steel samples were first mirror polished and then subjected to heat treatment in an IR furnace SVF17-SP from Yonekura Corporation (Kanagawa, Japan) coupled with a confocal microscopy set-up—CSLM VL2000DX from Lasertec Corporation (Yokohama, Japan) for in-situ observations. To prevent the sample from undergoing oxidation at high temperatures during the experiment, the chamber was thoroughly purged of oxygen and moisture well in advance. This was accomplished by initially filling the chamber with purified argon gas, which had been filtered, for ~ 3 min. Subsequently, the chamber was evacuated for 3 min using an oil-sealed rotary vacuum pump. These procedures were iterated three times, resulting in oxygen and water concentrations of < 1.0 ppm. The samples were heated to 1100 °C in Ar atmosphere with a 15-min dwell period followed by rapid cooling with a rate of 50 °C/s. The confocal images of the steel during the heat treatment were recorded using a laser beam having a wavelength of 408 nm at a frame rate of 30 fps and a resolution of 1024 × 1024 pixels.

Sample name	Percentage (%)				Percentage (%)		Percentage (%)	
	С	Cr	Mn	Si				
Low-Si (L-Si)	1.1	0.4	1.0	0.8				
Medium-Si (M-Si)	_			2.0				
High-Si (H-Si)				3.6				

Table 1 High carbon steel: sample name and composition

#### **Characterisation of Heat-Treated Samples**

Semi-quantitative analysis of the sample's microstructure was conducted using Xray diffraction. This involved using Co-K radiation at 45 kV and 40 mA current, employing a step size of  $\Delta \theta = 0.0260^\circ$ , and scanning the diffraction angle from  $30^\circ$  to  $130^\circ$  for detection. To examine the surface morphology of the steel samples, we utilized a Hitachi S3400 scanning electron microscope equipped with energydispersive X-ray spectroscopy (EDS) [15]. The steel samples used for XRD analysis were prepared in accordance with standards for bulk solid analysis. For quantitative phase analysis, Rietveld refinement fits were carried out using PANalytical X'Pert High Score Plus software. This allowed us to determine the volume fractions of both the original and newly transformed phases in the specimens subjected to various test conditions. A series of automated wet grinding and polishing procedures were performed for the metallographic examination of the heat-treated samples using Optical and Scanning Electron Microscopy (SEM).

The magnetic characteristics (saturation magnetization, remanence, and coercivity) of the samples (1 mm × 5 mm) were assessed by advanced SQUID (Superconducting Quantum Interference Device) Quantum Design MPMS-3 magnetometer using the vibrating sample mode (VSM) to achieve highest sensitivity. The magnetic hysteresis loops were measured with a sensitivity control of magnetic field with  $\leq 10^{-8}$  emu at ambient temperature.

#### **Results and Discussion**

The samples subjected to heat treatment were characterised using metallographic, microscopic, and diffraction analyses. During the austenitization and quenching processes in the heat treatment of high carbon steel, Si plays an important role in influencing the transformation of the steel's microstructure. The effects of Si addition in high carbon steel have been briefly discussed below.

#### **Optical and SEM Analysis**

The optical and SEM micrographs of the high carbon steel samples before and after heat treatment are shown in Figs. 1a, b and 2a, b. The optical micrographs of the Si-added samples before heat treatment were characterised by a pearlitic microstructure. Cementite grains were identified as dark black spots segregated at grain boundaries. A dominant martensitic microstructure was observed after heat treatment. The homogenised samples of Si-added steel exhibited mild surface oxidation, which increased with higher Si. From the SEM images, the sample morphology before

heat treatment showed lamellar ferritic structures and cementite. After heat treatment, the ferritic structures transformed into a combination of plate martensites. The martensitic structures evolved into a much finer structure with an increase in the Si content. The cementites were identified as small dark spots along the boundaries of the martensitic plates.

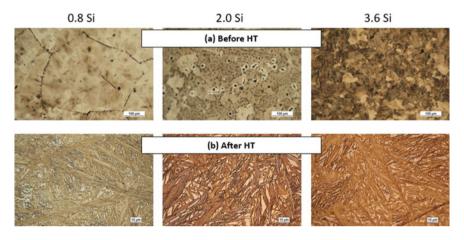


Fig. 1 Optical microscopy images of the steel samples with different Si concentrations before and after heat treatment

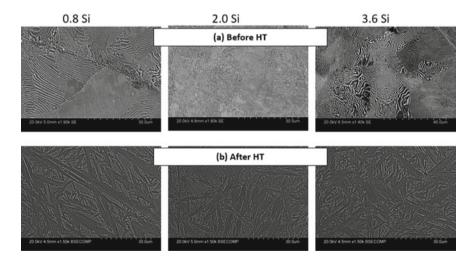


Fig. 2 SEM analysis of steel samples with different Si concentrations before and after heat treatment