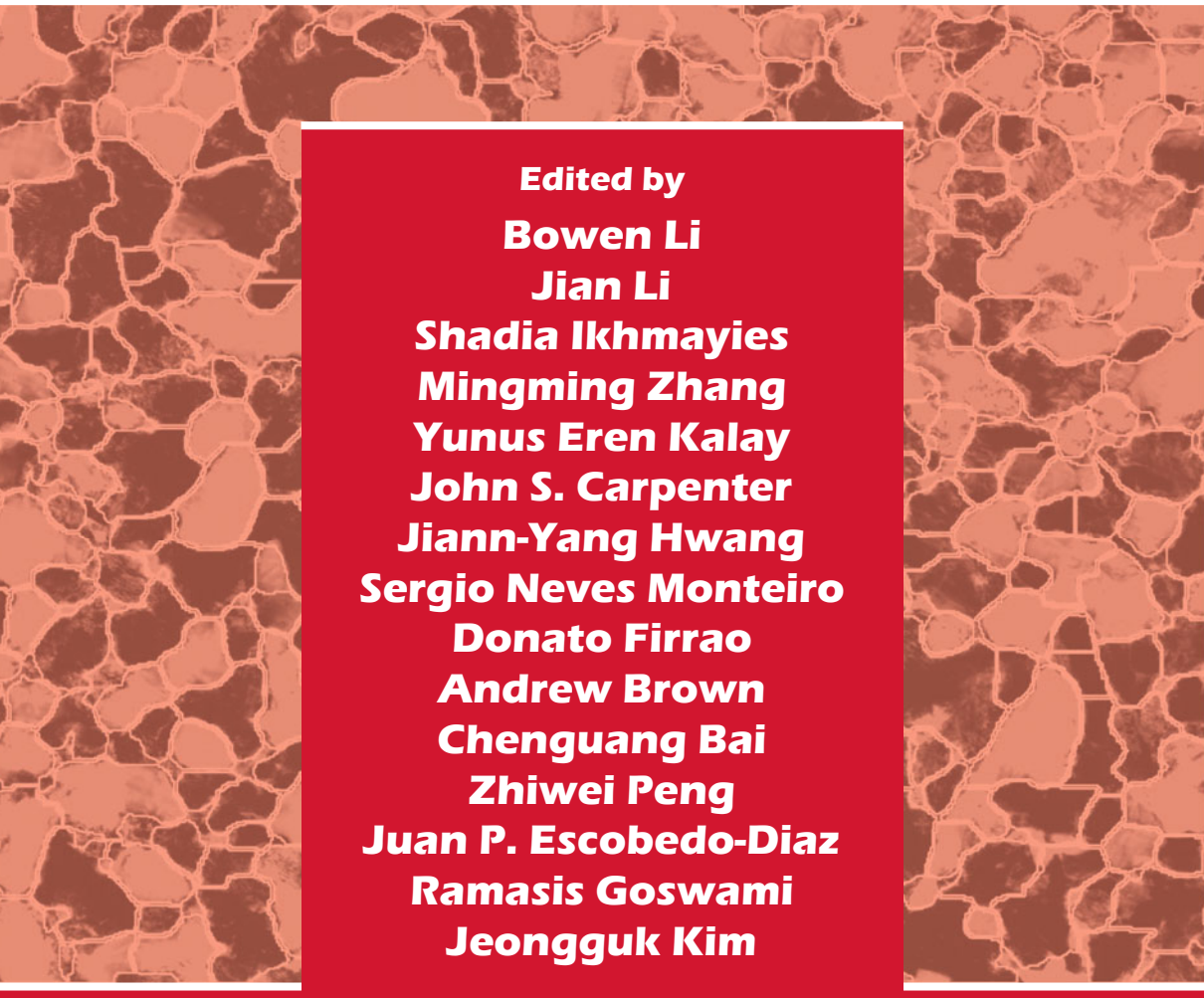
Characterization of Minerals, Metals, and Materials 2018



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Donato Firrao
Andrew Brown
Chenguang Bai
Zhiwei Peng
Juan P. Escobedo-Diaz
Ramasis Goswami
Jeongguk Kim

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The Minerals, Metals & Materials Series

Bowen Li · Jian Li · Shadia Ikhmayies
Mingming Zhang · Yunus Eren Kalay
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Preface

The interrelationships among composition, structure, property, process, and performance of a material are fundamental for materials research, development, manufacturing, and application. Materials characterization is the key to reveal these relationships throughout the entire circulation process of materials, from raw materials selection, through various process stages, final products, and applications, up to materials recycling and reuse. Characterization provides accurate and realistic information for the in-depth understanding of a material, such as how the material fails, how to improve the performance, how to simulate a material, and what is the lifetime of the material.

The symposium Characterization of Minerals, Metals, and Materials is sponsored by the Materials Characterization Committee of The Minerals, Metals & Materials Society (TMS) and focuses on the advancements of characterization of the minerals, metals, and materials from the bulk down to the nanoscale and the applications of characterization results on the processing of these materials. The subjects of the symposium include extraction and processing of various minerals, process–structure–property relationship of metal alloys, glasses and ceramics, polymers, composites, and carbon used as functional and structural materials. All characterization methods and techniques and their applications are covered in this symposium. The methodology and instrumentation for materials characterization are emphasized.

The symposium on which this volume is based is one of the largest—and also one of the broadest in terms of scientific coverage—held during the TMS Annual Meeting, which attracts materials scientists, mineralogists, metallurgists, mechanical engineers, chemists, physicists, microscopists, and instrumental experts from academia and industry across the world. In the TMS 2018 Annual Meeting held in Phoenix, AZ, USA, the symposium received 175 abstract submissions; of these 121 were accepted for oral presentation in 14 technical sessions, and 54 will be presented as posters.

This proceedings volume includes 76 manuscripts of original research. The peer-reviewed manuscripts were invited or contributed by the researchers from the fields of materials science, engineering, metallurgy, physics, chemistry,

manufacturing, and applications. The authors of the papers represent diversity from more than 20 countries in the North America, South America, Asia, Europe, Australia, and Africa. Although the papers were divided into 14 sections based on the technical sessions of the symposium, the topics of this volume cover a wide range of materials characterization from composition, structure, process, property, performance, and their interrelations in the materials from bulk-scale down to microscale and nanoscale. The material sequence and related processes were widely covered and include minerals, metals and alloys, ceramics, polymers and composites, semiconductors, optical, electronic, magnetic, environmental materials, and concrete. Among these papers, metallic materials and various composite materials take the major portion of the proceedings.

This book is a valuable reference for academic and industry readers from advanced undergraduates to experienced professionals who wish to learn about all types of characterization methods, their development, and applications in general, specifically in minerals, metals, and materials. It also provides up-to-date achievements on many types of materials for the scientists and engineers engaged in research, development, and production. The readers will enjoy the diversity of topics in this book with novel approaches and contributions on materials, micro- and nanostructures, performance, and relationships in practical uses.

The editors of this book are very grateful to the authors for their contribution of the manuscripts and willingness to share their new findings with the materials community. The editors would also like to express appreciation to TMS for giving this symposium the opportunity to publish a stand-alone volume. We also thank the Materials Characterization Committee and Extraction and Processing Division for sponsoring this symposium. The editors also thank the publisher, Springer, for their production of this book. Finally, we acknowledge the efforts by the past chairs and members of the Materials Characterization Committee, who continuously built this great symposium and who attracted talented and creative people and research groups from around the world to the committee and symposium.

Bowen Li

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Part XIII Poster Session

About the Editors

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 110 technical papers in peer-reviewed journals and conference proceedings, authored/coauthored 3 books, editor/coedited 7 books, hold 15 patents, and delivered more than 30 invited technical talks.

He received a Ph.D. in Mineralogy and Petrology from China University of Geosciences Beijing in 1998, and a Ph.D. in Materials Science and Engineering from Michigan Technological University in 2008. He has been an active member of The Minerals, Metals & Materials Society (TMS), Society for Mining, Metallurgy, and Exploration (SME), and China Ceramic Society. At TMS, he is the current Chair of the Materials Characterization Committee and member of the Powder Materials Committee and Biomaterials Committee, and former Extraction and Processing Division Award Committee member, *JOM* Subject Advisor, and Key Reader of *Metallurgical and Materials Transactions A*. He is the organizer/co-organizer of a number of international symposia and sessions. He also served as the editorial board

member of the *Journal of Minerals and Materials Characterization and Engineering, SciFed Journal of Metallurgical Science,* and *FUTO Journal Series.*

Jian Li is a senior research scientist at Canmet MATERIALS in Natural Resources Canada. He obtained his B.Sc. in Mechanical Engineering from Beijing Polytechnic University, M.Sc. in Metallurgical Engineering from Technical University of Nova Scotia (TUNS), 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. 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 holds a patent, authored three book chapters, and published more than 120 papers in scientific journals and conference proceedings.

Shadia J. Ikhmayies received a B.Sc. from the physics department in the University of Jordan in 1983, an M.Sc. in molecular physics from the same university in 1987, and a Ph.D. in producing CdS/CdTe thin film solar cells from the same university in 2002. Now she works at Al 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 published 44 research papers in international scientific journals, three chapters in books, and 73 research papers in conference proceedings. She is the author of two books for Springer *Silicon for Solar Cell Applications*, and *Performance Optimization of CdS/CdTe Solar Cells*, which are in production, the editor-in-chief of the book *Advances in Silicon Solar Cells* for Springer, and the e-book series *Material Science: Current and Future Developments* for Bentham Science Publishers, where these two books are in production.

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 in the European Conference on Renewable Energy Systems (ECRES2015-ECRES2017). She is a member of the editorial board of the *International Journal of Materials and Chemistry* for Scientific & Academic Publishing, the editor-in-chief of a book for the Research Signpost. She was a technical advisor/subject editor for *JOM*. She is a guest editor for two topical collections published in the *Journal of Electronic Materials*: European Conference on Renewable Energy Systems (2015 and 2016). She is a reviewer for 24 international journals and she is Chair of the TMS Materials Characterization Committee (2016–2017).

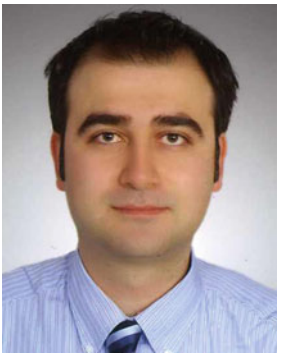
Mingming Zhang is currently a lead research engineer at ArcelorMittal Global R&D in East Chicago, Indiana. His main responsibilities include raw material characterization and process efficiency improvement in mineral processing and ironmaking areas. He also leads a technical relationship and research consortium with university and independent laboratory members and manages pilot pot-grate sintering test facility at ArcelorMittal Global R&D East Chicago.

He has over 15 years of research experience in the field of mineral processing, metallurgical and materials engineering. He obtained his Ph.D. 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 Tuscaloosa, Alabama where he was metallurgical engineer leading the development of models for simulating slab solidification and secondary cooling process.

He has conducted a number of research projects involving mineral beneficiation, thermodynamics and kinetics of metallurgical reactions, electrochemical processing of light metals, energy efficient, and environmental cleaner technologies. He has published over 50 peer-reviewed research papers and is the recipient of

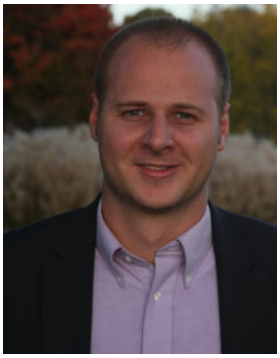
several U.S. patents. He also serves as editor and reviewer for a number of prestigious journals including *Metallurgical & Materials Transactions A* and *B*, *JOM*, *Journal of Phase Equilibria and Diffusion*, and *Mineral Processing and Extractive Metallurgy Review*.

Dr. Zhang has made more than 20 research presentations at national and international conferences including more than 10 keynote presentations. He is the recipient of 2015 TMS Young Leaders Professional Development Award. He has been invited by a number of international professional associations to serve as conference organizer and technical committee member. These associations include The Minerals, Metals & Materials Society (TMS) and the Association for Iron & Steel Technology (AIST).



Yunus Eren Kalay is an Associate Professor of Metallurgical and Materials Engineering and Assistant to the President at METU Ankara, Turkey. He 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 his 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 Middle East Technical University (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, 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 Materials Characterization Committee and Phase Transformations Committee of TMS, and served on the organizing committees of three international and one national congress including IMMC, MS&T, and TMS. He has also been involved in many synergistic activities such as being founder editor of Turkey's first undergraduate research journal, *MATTER* (http://matter.mete.metu.edu.tr/) and organizing the Materials Science Camps for K-12 students.



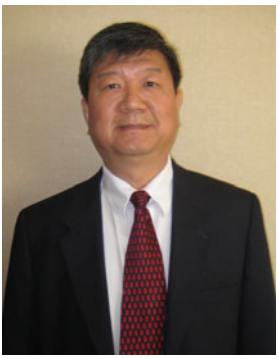
John S. Carpenter is a scientist within the manufacturing and metallurgy division at Los Alamos National Laboratory. He received his Ph.D. in Materials Science and Engineering from The Ohio State University in 2010 after performing his undergraduate studies at Virginia Tech.

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, 1 book chapter, and 25 invited technical talks to his credit.

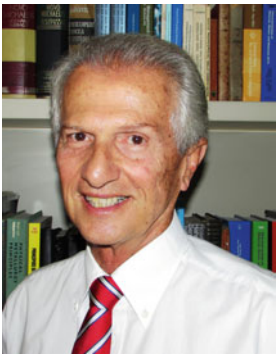
With regard to TMS service, he currently serves as the Extraction & Processing Division (EPD) representative on the Program Committee, the Structural Materials Division representative on the Content Development and Dissemination Committee, chair of the Advanced Characterization, Testing, and Simulation Committee, and the 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 the Characterization Committee in the past. John serves as a Key Reader for *Metallurgical and Materials Transactions A* and has coedited 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 EPD Young Leaders Professional Development Award.



Jiann-Yang (Jim) 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, flyash 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, Hebei United University, etc.

He received his B.S. 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 Materials Characterization Committee and the Pyrometallurgy Committee in TMS and has organized several symposiums. He is the recipient of TMS Technology Award and the Michigan Tech Bhata Rath Research Award.



Sergio Neves Monteiro graduated as 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 course 1975 in Energy at the Brazilian War College and Post-doctorate (1976) at the University of Stuttgart. He joined (1968) the Metallurgy Department as full professor of the postgraduation program in engineering (COPPE) of the UFRJ; was elected head of department (1978), coordinator of COPPE (1982) and Under-Rector for Research (1983); and 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 over 1200 articles in journals and conference proceedings and has been honored with several awards including the ASM Fellowship. He is top researcher (1A) of the Brazilian Council for Scientific and Technological Development (CNPq) and Top Scientist of State of Rio de Janeiro (FAPERJ); President of the Superior Council of the State of Rio de Janeiro Research Foundation, FAPERJ; (2012) and currently coordinator of the Engineering Area of this foundation. He is also 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*.

Donato Firrao received his Laurea in Chemical Engineering at the Politecnico di Torino, Turin, Italy, in 1968 and his M.Sc. in metallurgical engineering at The Ohio State University (OSU) on a Fulbright Scholarship in 1970. He began teaching in 1968 as an assistant professor of ferrous extractive metallurgy and a lecturer of chemistry at the Politecnico di Torino since 1971. In 1983 he became associate professor of Technology of metallic materials, gaining full professorship in the subject 3 years later. He also stayed as visiting fellow from 1978 to 1979 at the OSU Materials Science and Engineering Department, where he was named Distinguished Alumnus in 2003. He has authored more than 230 papers, primarily in the fields of physical and mechanical metallurgy and surface heat treatments. He is a member of Associazione Italiana di Metallurgia (AIM), ASTM International, ESIS, TMS, and the Turin Academy of Sciences, Fellow of ASM International (2011) and member of FAS. A founding partner of the Italian Group on Fracture (IGF) he was its secretary since its establishment in 1982, and the President between 1988 and 1994. He was cochair of the ESIS Technical Committee I (Elasto-Plastic Fracture Mechanics) from 1987 to 1996 and was named ESIS Fellow in 2016. He was President of the Federation of European Materials Societies (FEMS) from 2000 to 2001. Since 1993, he has been the president of the board of trustees of the Collegio Universitario di Torino (a private nonprofit university student housing foundation). He served as Dean of the First College of Engineering at the Politecnico di Torino from 2005 to 2012 and retired in November 2015. He is an expert in failure analysis and has acted as technical advisor to the judge in national and international trials (such as the Ustica aircraft crash, the Mattei affair, and the Sgrena/Calipari cases).

Andrew D. Brown is a Research Associate in the School of Engineering and Information Technology (SEIT) at the University of New South Wales (UNSW) Canberra, Australia. He received his B.S. in Mechanical Engineering from N.C. State University in 2009 and Ph.D. in Mechanical Engineering from Arizona State University in 2015. He has previously held research positions at the Nanoscale Tribology Lab at N.C. State and the Multiscale Materials Characterization and Multiphysics Modelling Lab at Arizona State.

His primary research interests encompass the dynamic behavior of materials under high pressures and strain rates. The focus of past research has been on investigating the microstructural weak links in shock-loaded metals. Current and future research interests include the dynamic response and fracture mechanisms in metals and metallic alloys, metallic and polymeric foam structures, novel metallic additive manufactured structures, bulk metallic glasses, and synthetic tissue materials.

He has been a member of TMS since 2011 and has served as a panel chair and poster contest judge for the Characterization of Minerals, Metals and Materials symposium since 2016 and currently serves as the symposium's Awards Committee representative.

Chenguang Bai is a Professor in the Department of Metallurgical Engineering, School of Materials Science and Engineering at Chongqing University, China. He received his B.S. in 1982, M.S. in 1987, and Ph.D. in 2003 from Chongqing University. He also furthered his study in Department of Metallurgy and Materials, University of Toronto as a visiting scholar between October 1995 to January 1997. He has been actively involved in the teaching and scientific research works in ferrous metallurgy, especially in the field of comprehensive utilization of vanadium–titanium magnetite resources. He has more than 20 patents, published more than 200 research articles, about 60 of which were in the international metallurgical periodicals. He also is Vice Chairman of Chongqing Society for Metals, and was a Member of Advisory

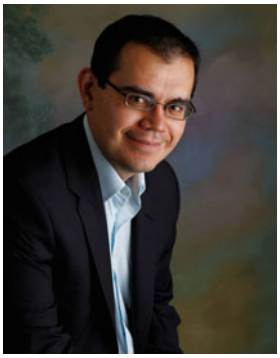
Committee of Experts, Department of Engineering and Materials Science, NSFC. He was the Vice President from 2009 to 2011, and the Vice Chairman of University Council of Chongqing University from 2011 to 2016.

Zhiwei Peng is an Associate Professor in the School of Minerals Processing and Bioengineering at Central South University. He received his B.E. and M.S. degrees from Central South University in 2005 and 2008, respectively, and his Ph.D. in Materials Science and Engineering from Michigan Technological University in 2012. His research interests include heat transfer in microwave heating, dielectric characterization of materials, nonthermal microwave effects, extractive metallurgy, computational electromagnetics, microwave absorbing materials, and biomaterials.

He has published 102 papers, including 71 peer-reviewed articles in multiple journals such as *International Materials Reviews*, *Metallurgical and Materials Transactions A*, *Metallurgical and Materials Transactions B*, *JOM*, *Journal of Cleaner Production*, *Journal of Power Sources*, *Fuel Processing Technology*, *Energy & Fuels*, *IEEE Transactions on Magnetics*, *IEEE Transactions on Instrumentation and Measurement*, *Ceramics International*, and *Annals of Medicine*. He has served as a key reviewer for a number of journals and been on the editorial board of the *Journal of Minerals and Materials Characterization and Engineering* since 2012. He received a TMS Travel Grant Award for the 141st TMS Annual Meeting & Exhibition, the Doctoral Finishing Fellowship and Dean's Award for Outstanding Scholarship of Michigan Technological University in 2012 and the Bhakta Rath Research Award of Michigan Technological University in 2013.

He is an active member of The Minerals, Metals & Materials Society (TMS). He has co-organized 7 TMS symposia (Characterization of Minerals, Metals and Materials in 2013, 2014, 2015, 2016, 2017 and 2018, and the 9th International Symposium on High Temperature Metallurgical Processing in 2018) and cochaired 14 TMS symposia sessions since 2012. He is

a member of the Pyrometallurgy and Materials Characterization committees, the chair of the Continuing Education Sub-Committee of the Materials Characterization Committee, a *JOM* advisor for the Pyrometallurgy Committee, and a winner of the TMS EPD Young Leaders Professional Development Award in 2014.



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 from Washington State University. Prior to taking up this academic appointment, he held research positions at the Institute for Shock Physics and 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 co-organized more than 5 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 Award.

Ramasis Goswami is a scientist with the Multi-functional Materials Branch of the Materials Science and Technology Division at Naval Research Laboratory, Washington, DC, USA. He obtained his bachelor degree in Metallurgical Engineering from Bengal Engineering College, Shibpur, India. He then earned his master's and Ph.D. degrees in Materials Engineering from Indian Institute of Science, Bangalore. He is a recipient of the Alexander von Humboldt fellowship. His current areas of research include the study of dislocation structures ahead of the crack tip, the microstructure and property relationship in metals, alloys and in multilayered thin films, and the

study of interfaces and defects in semiconducting thin films. He has published over 90 peer-reviewed articles in scientific literature.

Jeongguk Kim received his Ph.D. in Materials Science and Engineering, at the University of Tennessee, Knoxville, in 2002. The title of his Ph.D. thesis was "Nondestructive Evaluation (NDE) and Mechanical Behavior of Continuous Fiber Reinforced Ceramic Matrix Composites (CFCCs)." Currently, he is a director at the Future Transportation Systems Research Division, Korea Railroad Research Institute (KRRI), Korea. He is also a professor in the Railway Systems Engineering Department, the KRRI campus, at the University of Science and Technology, Korea.

His research interests include testing and certification of railroad components and systems, failure and safety analyses of railroad materials and systems based on fracture mechanics and several different types of NDE techniques including ultrasonic testing, acoustic emission, infrared thermography, magnetic particle testing, etc., and mechanical behavior of advanced railway materials.

His recent research efforts include development of future transportation systems such as rail–canal system based on multi-axle bogies, an innovative train–ferry system, and the smart container lift. He also enlarged his research on the development of effective maintenance technologies for high-speed train systems.

He has been a member of TMS since 1996, and he has been a regular contributor at TMS meetings as an author and session chair at the Characterization of Minerals, Metals and Materials sessions since 2005.

Part I
Characterization Methods

On FIB Milling Parameters

Jian Li and Pei Liu

Abstract In recent years, focused ion beam (FIB) has become a powerful microscopy tool. Creating large FIB cross sections are often required to characterize important microstructure features e.g. the depth and morphology of pitting corrosion and stress corrosion cracking in natural gas pipeline steels. Large FIB sectioning often takes long time that not only occupy valuable FIB microscope time, but also consume often expensive consumables that include gallium ion source, aperture and deposition gas etc. The new plasma FIB developed by FEI can speed up milling process by using much larger beam current. However, for most of the laboratories that is limited by conventional Ga ion FIB, the milling speed is limited by the relatively small beam current. In this paper, FIB milling rate are studied against milling parameters that include beam dwell time and pixel overlap. By optimizing the milling parameters, milling rate can increase significantly.

Keywords FIB · Milling rate · Sputter yield

Introduction

Soon after the development of liquid metal ion source (LMIS) in the 1960's, focused ion beam (FIB) system was invented. Small probe size of a few nanometers are available for high-resolution ion beam imaging, and high beam current density are now available with modern FIB columns for high speed ion beam milling [1]. In the early days, FIB microscopes were mostly used for IC device modification [2], failure analysis [3], and lithographic mask modification [4]. In the past decade, with increased resolution, FIB microscopy has been widely used as an effective materials science research instrument [5–7]. Examples includes FIB sectioning and imaging to reveal subsurface corrosion and stress corrosion cracking of pipeline steels, crack

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