Characterization of Minerals, Metals, and Materials 2017

Edited by

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Chenguang Bai
Yunus Eren Kalay
Ramasis Goswami
Jeongguk Kim

TMS

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The Minerals, Metals & Materials Series

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Preface

The characterization of materials is an important step to be taken before utilizing the materials for any purpose. It starts from the production of the material, and continues after each processing and/or engineering stage to explore its influence on the structure and properties of the material. Depending on the purpose, one can subject the material to mechanical, thermal, chemical, optical, electrical, and other characterizations to make sure that the material under consideration can function without failure for the life of the final product. Therefore, advances in the materials science are strongly correlated with advances in the characterization technologies. The Characterization of Minerals, Metals, and Materials symposium sponsored by the Materials Characterization Committee of TMS focuses on material characterization from the bulk down to the nano-scale. All characterization techniques and their applications are covered in this symposium. Developments in these techniques and their application in the quantification of the microstructure of materials are essential facets of this symposium. Specific characterization focus areas include catalyst structure, waste and failure characterization, besides structure-property relationships in metals, minerals and materials.

The characterization symposium is a cornerstone symposium in the TMS annual meeting, which attracts materials scientists, metallurgists, mechanical engineers, microscopists, metallographers, from academia and industry from all over the globe. In the TMS 2017 Annual Meeting & Exhibition held in San Diego, California, USA, the characterization symposium received 229 submissions, of which are 137 oral presentations, and 67 will be presented as posters. Of the presented papers, 93 are published in this book after being peer reviewed. The topics of these papers cover a wide range of materials science, metallurgy, physics, chemistry, and engineering of materials. Minerals, ferrous and nonferrous metals, semiconductors, clays, ceramics, alloys, composites, electronic, magnetic, environmental, soft, and advanced materials are widely covered. In addition, research papers about extraction, processing, welding, solidification, corrosion and method development represent a large portion of the presented papers.

This book features original articles and the state-of-the-art reviews on theoretical and practical aspects of the characterization, extraction, processing, structure, and

behavior of minerals, metals, and materials. It is a good reference for academic and industry audiences from advanced undergraduates to seasoned professionals who wish to learn about all types of characterization methods in general, and specifically about real-world applications in the minerals, metals and materials. This book will also be relevant for scientists and engineers engaged in research, development, and production. This book will provide the industry audience with up-to-date information on many types of materials and their characterization with an underlying theme of explaining the behavior of materials using novel approaches.

The reader of this book will learn about all types of characterization methods, their development, and their applications. The reader will enjoy the diversity of topics in this book. He/she will find in this book up-to-date information about bulk materials, thin films, joints and interfaces, powders, slags, micro and nanostructures. The beautiful thing is that this book pays attention to the relationship between production, extraction, processing, recycling, and loading of materials and alloys in practical use. The knowledge gained from this book can be used to prompt innovations in characterization methods and techniques, and to produce new materials with the specific desired properties.

The editors of this book express their sincere thanks and appreciations to the TMS for giving this symposium the opportunity to publish a stand-alone volume. The editors also thank the Materials Characterization Committee for sponsoring this symposium. They also thank the publisher, Springer, for the production of this book, and the authors, who are the core of this scientific work. Finally the editors express their appreciation for the past chairs and members of the Materials Characterization Committee, who built this great symposium and who attracted talented and creative people to the committee, and attracted scientists and research groups from around the world to this symposium.

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About the Editors

Shadia Ikhmayies had received the B.Sc. degree from the Physics Department in the University of Jordan in 1983, the M.Sc. degree in Molecular Physics from the same university in 1987 and the Ph.D. working on producing and characterizing CdS/CdTe thin film solar cells from the same university in 2002. She worked in the Applied Science University from 2004 to 2009 as Assistant Professor, and now she works in Al Isra University as Associate Professor. Her research is focused on producing and characterizing semiconductor thin films such as SnO_2: F, ZnO, CdS, CdTe, $CuInS_2$, thin film bilayers such as SnO_2:F/CdS:In, 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 40 research papers in international scientific journals, three chapters in books, and 62 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 development.

Shadia is a member of The Minerals, Metals & Materials Society (TMS) and a member of the steering committee of the World Renewable Energy Network (WREN/WREC). She is a member of the international organizing committee and the international scientific committee in the Third and Fourth European Conference on Renewable Energy Systems (ECRES2015 and ECRES2016). She was an

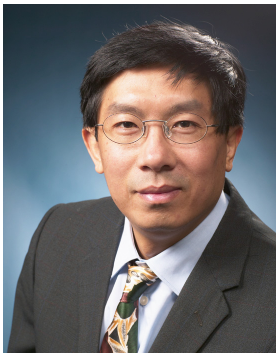
associate editor in the journal of *Physics Express* published by Simplex Academic Publishers. She is an associate editor for the journal *Peak Journal of Physical and Environmental Science Research* (*PJPESR*) published by Peak Journals. She is a member of the editorial board of the *International Journal of Materials and Chemistry* for Scientific & Academic Publishing, the editor-in-chief of the book *Advances in II-VI Compounds Suitable for Solar Cell Applications* for the Research Signpost, the editor-in-chief of the book *Advances in Silicon Solar Cells* for Springer, and the eBook series *Material Science: Current and Future Developments* for Bentham Science Publishers, where the last two books are under construction. She was the technical advisor/subject editor for *JOM* as a representative of the Materials Characterization Committee for the year 2014. She is a guest editor for a special section in the *Journal of Electronic Materials*: Third European Conference on Renewable Energy. Shadia is a reviewer in 22 international journals and five international conferences, and she is the 2016–2017 Chair of the TMS Materials Characterization Committee.

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, metals extraction, ceramic processing, antimicrobial additives, applied mineralogy, and solid waste reuse. He has more than 100 publications and 14 patents. Bowen 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 TMS, SME, and China Ceramic Society. At TMS, he has served as a member in Materials Characterization Committee, Powder Materials Committee, Biomaterials Committee, EPD Award Committee, and *JOM* Subject Advisor, as well as symposium co-organizer and session chair.

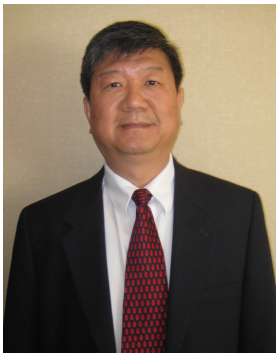
John S. Carpenter is a technical staff member in the Materials Science and Technology Division at the Los Alamos National Laboratory. Dr. Carpenter received his Ph.D. in Materials Science and Engineering from The Ohio State University in 2010 after performing his undergraduate studies at Virginia Tech.

His research interests include the characterization, processing, and mechanical testing of metallic nanocomposites fabricated via severe plastic deformation as well as additive manufacturing. Currently, his work focuses on understanding the relationship between plastic strain, texture, and the mechanical properties of bimetallic nanocomposites fabricated via accumulative roll bonding and joined using friction stir processing. This research involves the use of several characterization techniques including neutron scattering, X-ray synchrotron, PED, TEM, EBSD, and SEM. Mechanical testing for this work includes methods such as micropillar compression, microtension, and nanoindentation. He has more than 45 journal publications, one book chapter, and more than 20 invited technical talks to his credit

With regard to TMS service, Dr. Carpenter currently serves as the past chair for the Materials Characterization Committee, a programming representative for the Extraction and Processing Division (EPD), and the chair for the Advanced Characterization, Testing & Simulation Committee. He is also a participating member of the Mechanical Behavior of Materials, Content Development and Dissemination, and the Nanomechanical Behavior committees. He serves as a Key Reader for *Metallurgical and Materials Transactions A* and has co-edited special sections in *JOM* related to neutron characterization, coherent X-ray diffraction imaging methods, and modeling in additive manufacturing. He is the 2012 recipient of the Young Leaders Professional Development Award for the EPD of TMS. Dr. Carpenter was also awarded an honorable mention for the 2012 Los Alamos National Laboratory Postdoctoral Distinguished Performance Award.



Jian Li is a senior research scientist 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 (TUNS) and Ph.D. in Materials and Metallurgical Engineering from Queen's University, Kingston, Ontario. He has broad experience in materials processing and characterization including alloys deformation, recrystallization and micro-texture development. Dr. Li has extensive experience in focused ion beam (FIB) microscope techniques. He is also an expert in various aspects of SEM-EDS and EPMA techniques. Dr. Li holds a patent, authored three book chapters, and published more than 120 papers in scientific journals and conference proceedings.



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. Dr. Hwang 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.

Dr. Hwang received his B.S. degree 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 has served as Director of the Institute of Materials Processing from 1992 to 2011 and Chair of the Mining Engineering Department in 1995. He has been a TMS member since 1985. His research interests include the characterization and processing of

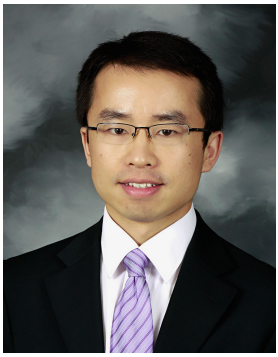
materials and their applications. He has been actively involved in the areas of separation technologies, pyrometallurgy, microwaves, hydrogen storages, ceramics, recycling, water treatment, environmental protection, biomaterials, and energy and fuels. He has more than 28 patents and has published more than 200 papers. He has chaired the Materials Characterization Committee and the Pyrometallurgy Committee in TMS (The Minerals, Metals & Materials Society) 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), and received his M.Sc. (1967) and Ph.D. (1972) from the University of Florida. This was followed by a course (1975) in Energy at the Brazilian War College and postdoctorate (1976) at the University of Stuttgart. He joined (1968) the Metallurgy Department (1977) 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). Dr. Monteiro was invited as Under-Secretary of Science for the State of Rio de Janeiro (1985) and Under-Secretary of College Education for the Federal Government (1989). He retired in 1993 from the UFRJ and joined the State University of North Rio de Janeiro (UENF), from where he retired in 2012. He is now Professor at the Military Institute of Engineering (IME), Rio de Janeiro. He has published over 1200 articles in journals and conference proceedings and has been honored with several awards including the ASM Fellowship, top researcher (1A) of the Brazilian Council for Scientific and Technological Development (CNPq) and top scientist of State of Rio de Janeiro (FAPERJ). He served as 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. Dr. Monteiro is the president of the Brazilian Association for Metallurgy, Materials and Mining—

ABM (2017–2019), a consultant for the main Brazilian R&D agencies and member of the Editorial board of five international journals as well as Associate Editor of the *Journal of Materials Research and Technology*.

Donato Firrao earned 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 Assistant Professor of ferrous extractive metallurgy and 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 three 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. Firrao 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 the Failure Analysis Society (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. Firrao was co-chair 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, Firrao has been the president of the board of trustees of the Collegio Universitario di Torino (a private nonprofit university student housing foundation). Firrao served as the Dean of the First College of Engineering at the Politecnico di Torino from 2005 to 2012; he 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).

Mingming Zhang is currently a senior research engineer at ArcelorMittal Global R&D in East Chicago, Indiana. His main responsibilities include raw material characterization and process efficiency improvement in the mineral processing and ironmaking areas. He also leads technical relationships and research consortia with university and independent laboratory members and manages pilot pot-grate sintering test facility at ArcelorMittal Global R&D East Chicago. Dr. Zhang has more than 15 years of research experience in the field of mineral processing, metallurgical and materials engineering. He obtained his Ph.D. degree in Metallurgical Engineering from The University of Alabama and his Master 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 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, energy efficient and environmental cleaner technologies. He has published more than 30 peer-reviewed research papers and is the recipient of several U.S. patents. Dr. Zhang also serves as editor and key reviewer for a number of prestigious journals including *Metallurgical 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 Leader 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 Association for Iron & Steel Technology (AISTech) and The Minerals, Metals & Materials Society (TMS).



Zhiwei Peng is Associate Professor in the School of Minerals Processing and Bioengineering at Central South University and Adjunct Assistant Professor in the Department of Materials Science and Engineering at Michigan Technological University. 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 in 2012. His research interests include heat transfer in microwave heating, dielectric characterization of materials, non-thermal microwave effects, extractive metallurgy, computational electromagnetics, microwave absorbing materials, and biomaterials.

He has published more than 70 papers, including 45 peer-reviewed articles in multiple journals such as *International Materials Reviews*, *Metallurgical and Materials Transactions A*, *JOM*, *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.

Dr. Peng is an active member of The Minerals, Metals & Materials Society (TMS). He has co-organized five TMS symposia (Characterization of Minerals, Metals and Materials in 2013, 2014, 2015, 2016, and 2017) and co-chaired twelve 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 Leader Professional Development Award in 2014.

Juan P. Escobedo-Diaz is a senior lecturer in the School of Engineering and Information Technology (SEIT) at the University of New South Wales (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 Los Alamos National Laboratory.

His main research interests center on the dynamic behavior of materials under extreme conditions, in particular high pressure and high strain rate. His focus has been on investigating the effects of microstructural features on the dynamic fracture behavior of metals and metallic alloys. He has published primarily in the fields of shock physics and materials science.

He has been a member of The Metals, Minerals & Materials Society (TMS) since 2011. During this time he has co-organized more than five symposia at the Annual Meetings including the symposium on Characterization of Minerals, Metals and Materials in 2014. He was awarded a 2014 SMD Young Leader Award.

Chenguang Bai is Professor in the Department of Metallurgical Engineering, School of Materials Science and Engnieering 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 continueed his studies in the Department of Metallurgy and Materials, University of Toronto as a visiting scholar between October 1995 to January 1997. Dr. Bai 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 the Chongqing Society for Metals, and was a member of the Advisory Committee of Experts, Department of Engineering and Materials Science, National Science Foundation of China (NSFC). He was the Vice President from 2009 to 2011, and the Vice Chairman of University Council of Chongqing University from 2011 to 2016.

Yunus Eren Kalay is 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. degree 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 in Ames National Laboratory, where he was given the opportunity to practice "Atom Probe Tomography". In 2011, Dr. Kalay joined the Department of Metallurgical and Materials Engineering (METE) of METU as 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. 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 Materials Characterization Committee and Phase Transformations Committee of TMS, and served in organizing committees of three international congresses and one national congress including IMMC, MS&T and TMS. Dr. Kalay 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.

Ramasis Goswami is a scientist with the Multifunctional Materials Branch of the Materials Science and Technology Division at Naval Research Laboratory, Washington, D.C., 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. Dr. Goswami 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 more than 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 Professor in the Railway Systems Engineering Department, the KRRI campus, at the University of Science and Technology, Korea.

Dr. Kim's 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 nondestructive evaluation (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 session since 2005.

Part I
Soft Materials

Charpy Toughness Behavior of Fique Fabric Reinforced Polyester Matrix Composites

Artur C. Pereira, Sergio N. Monteiro, Foluke S. Assis and Henry A. Colorado

Abstract The fique fiber is an important natural fiber, originally from Colombia where it is used for sacks and crafts while its plant is used to contain slopes. However, few studies were realized with the fiber obtained from the fique plant leaf, its mechanical properties are superior in many aspects in comparison to some other lignocellulosic fibers. This work investigated the toughness behavior of polyester matrix composites reinforced with up to 30% in volume of a fabric made of fique fiber by means of Charpy impact tests. It was found that the addition of fique fabric results in a marked increase in the absorbed energy by the composites. Macroscopic observation of the post-impact specimens and SEM fracture analysis showed that transversal rupture through the fique fabric interface with the polyester matrix is the main mechanism for the remarkable toughness of these composites.

Keywords Fique fabric · Polyester composites · Charpy impact test · Rupture mechanism

Introduction

The interest of this research is to develop composites with polyester resin matrix reinforced with continuous and aligned fique fibers, for applications in various industries, including construction and automotive industry. Conflicts related to the use of non-renewable forms of energy are increasing the interest to enter the market to replace natural materials; synthetic materials synthetics have a higher power consumption in its manufacture [1–4].

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