Characterization of Minerals, Metals, and Materials 2019

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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 is the 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 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.

Sponsored by the Materials Characterization Committee of The Minerals, Metals & Materials Society (TMS), the symposium Characterization of Minerals, Metals, and Materials is focused on the advancements of characterization of various minerals, metals, and materials from the bulk down to the nanoscale, and on the applications of characterization results on the processing of these materials. The subjects of the symposium include extraction and processing of various minerals, and the 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. Advanced methodology and instrumentation for materials characterization are emphasized.

The characterization symposium is one of the largest and broadest symposia in terms of scientific coverage during TMS Annual Meetings, which attract materials scientists, mineralogists, metallurgists, mechanical engineers, chemists, physicists, microsporidians, and instrumental experts from academia and industry across the world. For the TMS 2019 Annual Meeting held in San Antonio, Texas, USA, the characterization symposium received 188 abstract submissions, of which 107 were accepted for oral presentation in 13 technical sessions, and 74 accepted as posters.

This volume includes 79 peer-reviewed manuscripts of original research. The 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 25 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 collection cover a wide range of materials characterization from composition, structure, process, property, and performance, and their interrelations in the materials from bulk-scale down to microscale and nanoscale. The material sequence and related processes were widely covered which include minerals, metals, and alloys, ceramics, polymers and composites, semiconductors, energy, optical, electronic, magnetic, environmental materials, and concrete. Among these papers, metallic materials and various composite materials make up 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 the minerals, metals, and materials. The collection provides up-to-date achievements on many types of materials for the scientists and engineers engaged in research, development, and production. Readers will enjoy the diversity of topics in this book with novel approaches to 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 the TMS for giving this symposium the opportunity to publish a stand-alone volume. They 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, they 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
Lead Organizer

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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 three books, and edited/coedited seven books. He also holds 15 patents and has delivered more than 30 invited technical talks.

Dr. Li. 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, a former Extraction and Processing Division Award Committee member, a *JOM* Subject Advisor, and a Key Reader for *Metallurgical and Materials Transactions A*. He is the organizer/co-organizer of a number of international symposia and sessions. He also served as the

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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, has authored three book chapters, and has published more than 140 papers in scientific journals and conference proceedings.

Shadia Ikhmayies received a B.Sc. and M.Sc. from the physics department at the University of Jordan in 1983 and 1987 respectively, and a Ph.D. on the topic of producing CdS/CdTe thin film solar cells from the same university in 2002. She now works at Isra University in Jordan as an associate professor. Her research is focused on producing and characterizing semiconductor thin films, and thin film CdS/CdTe solar cells. She also works in characterizing quartz in Jordan for the extraction of silicon for solar cells and characterizing different materials by computation. She has published 48 research papers in international scientific journals, 73 research papers in conference proceedings, and 3 chapters in books. She is the author of two books for Springer, *Silicon for Solar Cell Applications* and *Performance Optimization of CdS/CdTe Solar Cells* (both in production), editor of the book *Advances in II-VI Compounds Suitable for Solar Cell Applications* (Research Signpost), the book *Advances in Silicon Solar Cells* (Springer), an eBook series about material science (in development with Springer), and several TMS proceedings publications. She is the winner of the

TMS Frank Crossley Diversity Award (2018), and the World Renewable Energy Congress 2018 (WREC-18) Pioneering Award.

Dr. Ikhmayies is a member of the 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–ECRES2018). She is a member of the editorial board of the *International Journal of Materials and Chemistry* (Scientific and Academic Publishing), and has served as a technical advisor/subject editor for *JOM* (2014 and 2019). She has been a guest editor for topical collections from the European Conference on Renewable Energy Systems in the *Journal of Electronic Materials*, and an editorial advisory board member for *Recent Patents on Materials Science* (Bentham Science). She is a reviewer for 24 international journals, was the Chair of the TMS Materials Characterization Committee (2016–2017), and has been lead organizer of more than four symposia at the TMS Annual Meeting and Exhibition.

Mingming Zhang is 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.

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. 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 more than 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 post-doctoral 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, and application of synchrotron X-ray scattering in materials research. He was awarded the METU Prof. Dr. Mustafa Parlar Foundation Research Incentive Award, which is

a prestigious award that recognizes young scientists in Turkey with exceptional achievements and research productivity.

Dr. Kalay is an active member of the Materials Characterization Committee and Phase Transformations Committee of TMS, and has 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 serving as founder editor of Turkey's first undergraduate research journal, *MATTER* (http://matter.mete.metu.edu.tr/), and has organized materials science camps for K-12 students.

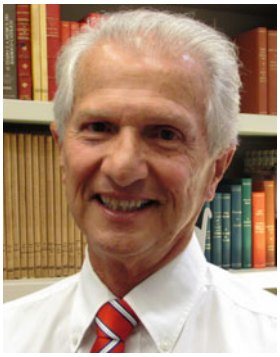
John S. Carpenter is a scientist within the manufacturing and metallurgy division at Los Alamos National Laboratory. He received his Ph.D. in Materials Science and Engineering from The Ohio State University in 2010 after performing his undergraduate studies at Virginia Tech. His research focus is on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

Dr. Carpenter 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 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, and Hebei United University.

Dr. Hwang 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 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 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 postdoctorate (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 was invited as Under-Secretary of Science for the State of Rio de Janeiro (1985) and Under-Secretary of College Education for the Federal Government (1989). He retired in 1993 from the UFRJ and joined the State University of North Rio de Janeiro (UENF), from where he retired in 2012. He is now Professor at the Military Institute of Engineering (IME), Rio de Janeiro, and has published 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). 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 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*.

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 the Department of Metallurgy and Materials, University of Toronto as a visiting scholar between October 1995 and 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 and has 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.

Juan P. Escobedo-Diaz is a Senior Lecturer in the School of Engineering and Information Technology (SEIT) at UNSW Canberra. He obtained his doctoral degree in Mechanical Engineering at Washington State University. Prior to taking up this academic appointment, he held research positions at the Institute for Shock Physics 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 since 2014. He was awarded a 2014 SMD Young Leaders Professional Development Award.

Pasquale Russo Spena is assistant professor of Manufacturing Technology and Systems in the Faculty of Science and Technology of the Free University of Bozen/Bolzano (Italy). Dr. Russo Spena received his M.Sc. in Materials Engineering and his Ph.D. in Industrial Production Systems Engineering (topic: metallurgical engineering) from Politecnico di Torino (Italy). In 2009, he started working as research assistant in the Department of Management and Production Engineering of the Politecnico di Torino; his work was mainly focused in metal processing and characterization and in developing models and tools for the management of product information in manufacturing processes. In 2010, Dr. Russo Spena was appointed as assistant professor of Manufacturing Technology and Systems at the Faculty of Science and Technology of the Free University of Bozen/Bolzano.

Dr. Russo Spena's research interests are in the field of manufacturing and materials engineering, particularly concerning joining techniques for metals, metals and welded joints characterization, and product quality assessment. Currently, his work focuses on understanding the relationship between welding processes (e.g., joining parameters) and mechanical/microstructural properties of welded joints, especially for the automotive industry. This research involves the use of several characterization techniques including, X-ray diffraction, EBSD, SEM, image analysis by digital image correlation techniques.

Dr. Russo Spena is member of several national and international scientific organizations. He has published more than 50 articles in refereed journals, conference proceedings, and book chapters. With regard to TMS service, he has served as a member of the Materials Characterization Committee, as well as a symposium co-organizer and session chair.

Ramasis Goswami is a scientist with the Multifunctional 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 more than 90 peer-reviewed articles in scientific literature.

Part I
Characterization Method Development

Commentary—Are There Still Places for Gallium FIB?

Jian Li

Abstract Since developed in the mid-1980s, focused ion beam (FIB) technology has become quite mature. From the initial single Ga ion beam system to nowadays dual-beam FIB, and from application only in semiconductor industry to applications to biological, mineral and materials engineering, FIB systems seemed to have almost maxed out their capability. The question arises is if the Ga FIB will still hold its place after the release of the new generation of plasma FIB in 2015. This paper provides a high-level overview of the two FIB systems.

Keywords FIB · Microscopy · Milling · PFIB · LMIS · Resolution

Evolution of FIB Microscopy

The concept of focused ion beam (FIB) technique using the liquid gallium ion source (LMIS) was first introduced by Seliger and Fleming [1] in 1974, where they were able to focus a 60 keV ion beam down to 3.5 μm diameter for doping semiconductor devices. In the span of the next 10 years, the technology improved and allowed to focus ion beams produced by LMIS down to about 50 nm. Orloff and Sudraud [2] proposed a design of a FIB system for maskless ion implantation, etching and deposition. One of the most successful early FIB system from Micrion was widely used by semiconductor industry. In the late 1990s, Phaneuf [3] demonstrated the application of FIB microscopy in materials science related research using a 50 kV, 10 nA Micrion-2500 FIB system. With the invention of TEM lift out technique, Giannuzzi and Stevie [4] opened up a new era of FIB applications. The application of FIB microscopy techniques in materials science bloomed in the early 2000s with a flurry of publications [e.g. 5–10].

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In 1993, FEI introduced the first commercial DualBeam™ instrument that integrated a Ga ion FIB column into a SEM column. In the next 20 years leading to today, dual-beam FIBs have almost completely replaced single beam FIB systems, and has become a powerful microscopy instrument for imaging, analysis and prototype fabrication at the nanometer scale. Nowadays, FIB microscopy has been widely used in various types of studies including but not limited to the following areas:

- Semiconductor micro fabrication, device modifications and failure analysis
- Cross sectioning and imaging
- Site-specific TEM specimen preparation
- Micro–Nanostructure deposition
- Cryo FIB for biological and nonconductive specimens
- Tomography (serial sectioning and imaging)
- 3D EDS and EBSD

Recently, there has been lots of interest to develop capabilities for ultrafine milling steps for nanostructures and biomedical related applications. Phaneuf demonstrated an impressive automated ~3 nm per slice milling process using a Zeiss CrossBeam™ system [11]. However, on the other side of spectrum, there has also been a general need to speed up the milling process to remove a large amount of materials to investigate larger areas. Unfortunately, FIBs with LMIS sources can only provide limited beam current. Table 1 lists the beam current range of a few state-of-the-art FIB microscopes on the market. When milling structural materials, large ion doses are usually required for milling due to limited sputtering yield. For example, under a 30 kV, 65 nA beam current, it takes about 8 h to mill a $100 \times 50 \times 50$ μm trench on Ni-based superalloys.

To expedite milling, FEI released the first plasma FIB (Vion) in 2015 that can produce beam current up to 2000 nA. This new plasma FIB, using an ionized Xe beam, can cut through much larger volume of materials within much shorter time. Such high milling rate has enabled research in new areas like investigating concrete structure that requires serial sectioning of large areas (larger than 500×500 μm $\times$ 300 μm sections).

Table 1 Beam current range of selected FIB systems with LMIS

FIB systems	Beam current
Zeiss CrossBeam	1 pA–100 nA
FEI Helios NanoLab	1 pA–65 nA
JEOL JIB-4610F	Up to 90 nA
Hitachi NX2000	Up to 100 nA
Tescan S8000G	Up to 100 nA

Do We Still Need Ga FIB?

Since its release, Plasma FIB (PFIB) has gained lots of attention in the "FIB world". Its ability to perform rapid ion beam milling at speed that "old timer" FIB operators cannot even imagine a few years back. The simplicity of ion column design and the "unlimited" ion source supply shows superior potential over Ga FIB at which typical LMIS source life is only about 1000 h. A common question is if we are at the stage where we see the end of glorious era of Ga FIB. To answer the question, we need to look into a few aspects that the two types of FIBs can offer.

Resolution

Smith et al. [12] showed spot sizes (d_{50}) as a function of ion beam currents. At current below 60 nA, Ga LMIS provide smaller spot size than that of the Xe source. Shown in Fig. 1, under 30 keV, Xe ion beam diameters are about five times larger than the Ga ion beam in low current range. This can translate to about 80 times beam density difference. The smaller Ga ion beam diameter yields to much better resolution at low current range. For ion beam current above 60 nA, Ga ion beam becomes much diffused compared to that of Xe beam in PFIB.

It is obvious that PFIB presents great advantage at high beam current range. With tighter beam and large beam current, PFIB can cut through materials extremely fast. In contrast, in fine polishing stage to achieve high-quality cross-sectioned surfaces, Ga FIB is preferred when tighter beam and higher beam density is desired. After the rough cutting with large beam current, finely focused beam with tighter spot size is always required to produce high-quality FIB polished sections for imaging or 3D EBSD work. The beam current required for final polishing is usually on the order

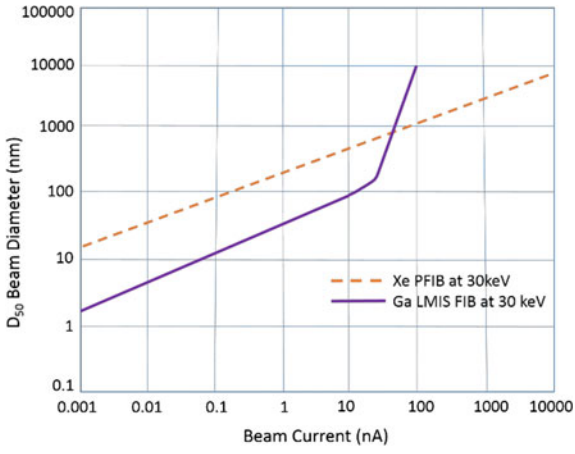

Fig. 1 Comparison of the spot size of Ga FIB and PFIB [12]. d_{50} is the beam diameter containing 50% of the primary ions