Characterization of Minerals, Metals, and Materials 2022

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Sergio Neves Monteiro · Shadia Ikhmayies ·
Yunus Eren Kalay · Jiann-Yang Hwang ·
Juan P. Escobedo-Diaz · John S. Carpenter ·
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Preface

Materials characterization is a vital part in all science and engineering practice, as it is a fundamental process to achieve a good understanding of the processing-microstructure-property relationship. Various advances in characterization techniques and instruments in recent years have contributed, in a significant way, to an in-depth understanding of materials properties and structure on different scales. This enhanced understanding has also made profound impacts on process efficiency of existing industrial processes, and the ways of minerals, metals, and materials application in many fields.

This year, the Characterization of Minerals, Metals, and Materials symposium received 126 abstract submissions, of which 46 papers were accepted in 8 technical sessions. This symposium is among one of the largest in the TMS Annual Meeting & Exhibition. The proceedings volume includes state-of-art techniques used in modern minerals, metals, and materials characterization, and the latest research in the field of materials engineering and technologies. This proceedings publication is a valuable reference for academia scholars and industry professionals who are interested in advanced characterization methods and instrumentations that cover a wide range of research subjects. Readers will enjoy the diversity of topics in this book with innovative approaches to process and characterize materials at various scales and levels.

The Characterization of Minerals, Metals, and Materials 2022 symposium is sponsored by the Materials Characterization Committee under the Extraction & Processing Division (EPD) of TMS. The main focuses of this symposium include, but are not limited to, advanced characterization of extraction and processing of minerals, process-microstructure-property relation of metal alloys, ceramics, polymers, and composites. New characterization methods, techniques, and instrumentations are also emphasized.

As a lead organizer of this symposium, I would like to take this opportunity to express my sincere gratitude to all authors for their contribution and generosity to share their research work. On behalf of the organizing committee, I would like to thank TMS for providing us the valuable opportunity to publish this stand-alone proceedings volume. Much appreciation is also extended to the EPD for sponsoring this symposium.

Most importantly, the success of this proceedings publication would not be possible without the fabulous contribution and support from all members of the Materials Characterization Committee. I also would like to thank our publisher, Springer, for their timely and quality publication of this book.

Mingming Zhang
Lead Organizer

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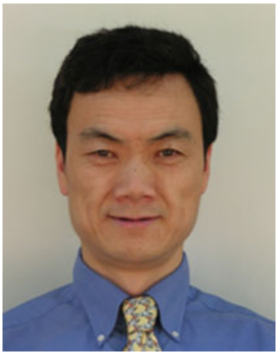
About the Editors

Mingming Zhang is currently a principal consultant at Wood Mackenzie. He has more than 15 years of experience in the field of mining, mineral processing, smelting and refining, and materials engineering. Before joining Wood Mackenzie, Dr. Zhang held 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 ArcelorMittal, 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 the 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 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 has authored three book chapters and published more than 160 papers in scientific journal 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 140 technical papers in peer-reviewed journals and conference proceedings, authored/co-authored 3 books, and edited/co-edited 12 books. He also holds 16 patents and has delivered more than 30 invited technical talks.

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 the Powder Materials Committee and the Biomaterials Committee. He has also served as an EPD Award committee member, a *JOM* subject advisor, and a 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*, *Reviews on Advanced Materials Science*, and *FUTO Journal Series*.

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 postdoctorate (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 1,900 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 associate editor-in-chief of the *Journal of Materials Research and Technology*. He is the author of 130 patents and a top world researcher in "Natural Fiber Composites" and "Ballistic Armor", Scopus 2020.

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. 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 also works in characterizing quartz in Jordan for the extraction of silicon for solar cells and characterizing different materials by computation. She has published 59 research papers in international scientific journals, 85 research papers in conference proceedings, and 3 chapters in books. She is the founder and editor of the eBook 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 TMS Materials Characterization Committee (2016–2017), and the leading organizer of three symposiums; Solar Cell Silicon 2017–2020, Mechanical Characteristics and Application Properties of Metals and Nonmetals 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) 2010–present. She is a member of the international organizing committee and the international scientific committee in the European Conference on Renewable Energy Systems (ECRES2015–ECRES2022). 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 is a reviewer of 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.

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. 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 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 X-ray 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.

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.

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John S. Carpenter is a scientist within the manufacturing 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 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, development of new materials for high field magnets using severe plastic deformation, 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 60 journal publications, one book chapter, and 45 invited technical talks to his credit.

With regard to TMS service, Dr. Carpenter is a past chair for both the Materials Characterization and Advanced Characterization, Testing & Simulation Committees. He is current chair for the Joint Commission for *Metallurgical and Materials Transactions A* and the SMD representative on the Content Development and Dissemination Committee. He is also the Program Committee Representative for the MS&T Conference. He recently received 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 is a mechanical engineer at the U.S. Army Combat Capabilities Development Command (CCDC) Army Research Laboratory (ARL), Aberdeen Proving Grounds, Maryland, USA. He obtained his B.S. in Mechanical Engineering at North Carolina State University (2009) and his doctoral degree in Mechanical Engineering at Arizona State University (2015). He spent three years (2015–2018) at UNSW Canberra as a postdoctoral researcher where he oversaw the daily operations of the Impact Dynamics Laboratory, mentored undergraduate and graduate student research projects, and performed teaching duties. His research expertise is understanding microstructural effects on the mechanical performance and damage processes of materials subjected to high dynamic pressures (shock) and high strain rates. Since joining ARL in 2018 he has shifted focus to high-rate injury biomechanics research to improve injury outcome prediction and protect the U.S. Warfighter. He has been an active member of TMS since 2011; was a co-organizer for the symposium on Characterization of Minerals, Metals, and Materials in 2018; and has been the symposium's EPD Award Representative from 2017–present. Dr. Brown has published 41 peer-reviewed articles in the fields of mechanical engineering, materials science, and biomechanics.

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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 56 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* and *Cogent Chemistry*. 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 10 TMS symposia and co-chaired 24 symposia sessions since 2012. He is a member of the Pyrometallurgy Committee and the vice 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.

Part I
Advanced Microstructural Characterization Methods

Challenges Concerning the Characterization of Cementite in Low Carbon Steel Using Electron Backscatter Diffraction

M. K. O'Brien, S. K. Lawrence, and K. O. Findley

Abstract Understanding the location and morphology of cementite precipitation in steel has long been addressed almost exclusively with transmission electron microscopy, particularly in low carbon steels. However, scanning electron beam techniques such as electron backscatter diffraction (EBSD) could leverage automated diffraction pattern analysis and high scan rates to improve statistics in cementite analysis. This paper discusses challenges specific to EBSD through the lens of a case study concerning low carbon microalloyed pipeline steel. Correlations between secondary electron (SE) and EBSD micrographs of a low carbon, microalloyed steel etched with 2 pct nital could not effectively verify the presence of cementite identified using EBSD. Pixels identified as cementite by the EBSD software often held a 43.6° || <100> axis/angle relationship reflected in grain boundary texture misorientation distributions. Further investigations of the ferrite/cementite interfaces displaying this 43.6° || <100> relationship were undertaken by utilizing pole figures of three well-known orientation relationships (OR) between ferrite and cementite, which resulted in the best match with the Bagaryatskii OR. Some questions concerning interaction volumes and indexing/phase identification algorithms are presented with respect to cementite characterization, as well as a proposal for future work to consider possible pseudosymmetric phenomena in specific ferrite orientations.

Keywords EBSD · Cementite · Phase identification

Introduction

Low carbon, microalloyed steels are often used in applications where weldability is a concern. Strength is achieved using complex thermo-mechanical processing

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schemes. Microalloying elements such as niobium and vanadium are added with the intention of high temperature precipitation in austenite during rolling such that subsequent rolling at temperatures below the temperature for recrystallization results in grain refinement as the precipitates pin grain boundaries. Further strengthening can also be achieved by accelerated cooling upon completion of finish rolling, which can result in non-equiaxed ferrite with M/A constituents, also known as granular bainite.

There are several techniques that have traditionally been used to characterize the size and spatial distribution of cementite in low carbon steels. The predominant technique is transmission electron microscopy (TEM), utilizing diffraction, bright field, and dark field modalities. If the cementite precipitates in known orientation relationships with ferrite, a combination of diffraction and dark field techniques can be used to unequivocally identify cementite. However, due to the small sample size inherent in TEM samples, it is time consuming and difficult to obtain a statistically significant number of precipitates. Capitalizing on nital etchant response and secondary electron micrographs to provide larger sampling regions and thus a higher number of precipitates is another common technique of cementite characterization, but this technique suffers from both resolution limits and ambiguity regarding the interpretation of cementite as opposed to retained austenite, or other precipitates. Lastly, the use of TEM carbon extraction replicas offers some reduction in ambiguity relative to SEM etching techniques, and an increase in the number of precipitates analyzed relative to thin foils, but removes the ability to relate the location and size of cementite precipitates to any characteristics of the original matrix. Improvement of scanning electron beam diffraction techniques of bulk samples could overcome the small statistical sampling size inherent in TEM-based techniques, while providing options for confirming cementite identification by means of dynamical diffraction techniques such as EBSD.

Orientation Relationships Between Cementite and Ferrite

Cementite precipitation can occur by several different mechanisms tied to different orientation relationships between the parent ferrite and daughter cementite. Determining the orientation relationships could assist in identifying when the cementite precipitated during thermo-mechanical processing, in addition to being a verification tool for understanding the veracity of cementite phase identification using EBSD. Cementite is orthorhombic with lattice parameters a = 0.452 nm, b = 0.674 nm, and c = 0.509 nm [1, 2]. The cementite unit cell has 12 iron atoms and 4 carbon atoms, as shown schematically in Fig. 1. Three orientation relationships, the Bagaryatskii, Isaichev, and Pitsch-Petch relationships, are shown in Table 1. In pearlitic steels, the cementite and ferrite lamellae nucleate and grow simultaneously according to a Pitsch-Petch relationship [3–5]. The Pitsch-Petch relationship is also observed when cementite forms at the austenite/ferrite interphase boundary in upper bainite [6]. When cementite precipitation occurs in supersaturated ferrite or martensite during

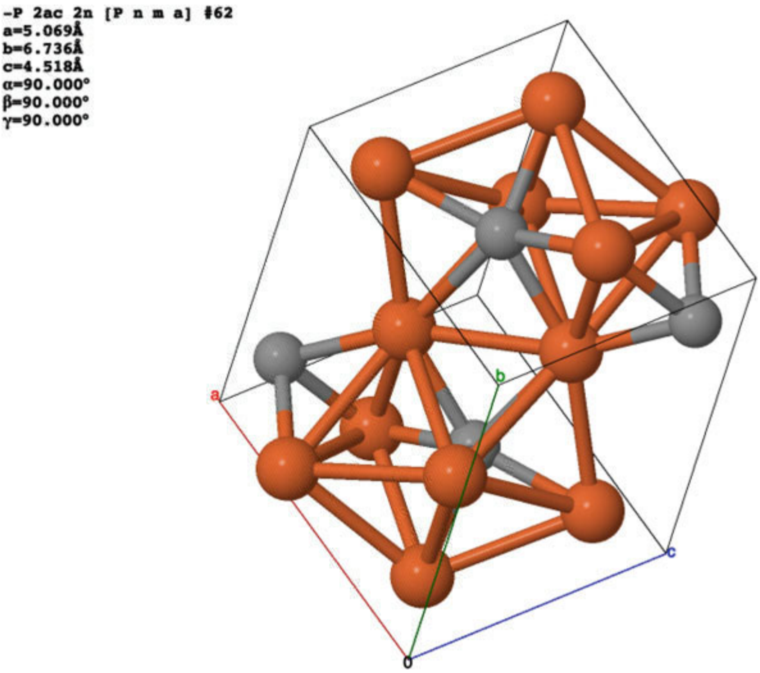


Fig. 1 Three-dimensional representation of the cementite unit cell where the orange atoms represent iron and the grey atoms represent carbon [2] (Color figure online)

Table 1 Three orientation relationships between ferrite and cementite

Bagaryatskii	Isaichev	Pitsch-Petch
[100]c‖ [110]f	[100]c‖ [111]f	[100]c 2.6° from [311]f
[010]c‖ [111]f	(031)c≈‖(101)f	[010]c 2.6° from [131]f
(001)c‖(112)f		(001)c‖(215)f

tempering, the Bagaryatskii and Isaichev ORs are often observed [5, 6]. The Bagaryatskii and Isaichev ORs are closely related; the Isaichev OR deviates from the Bagaryatskii OR by a rotation of 3.8° about the *a*-axis of the cementite lattice [3, 7–12].

Experimental

The chemical compositions of the X70 steel used in this investigation are shown in Table 2. The thermo-mechanical processing path for the X70 steel is not known, although steels of this grade are usually assumed to be accelerated cooled upon finish rolling. The samples were mounted in Bakelite®, ground and polished to 1 μm

Table 2 Chemical composition of X70 plate steel [13, 14]

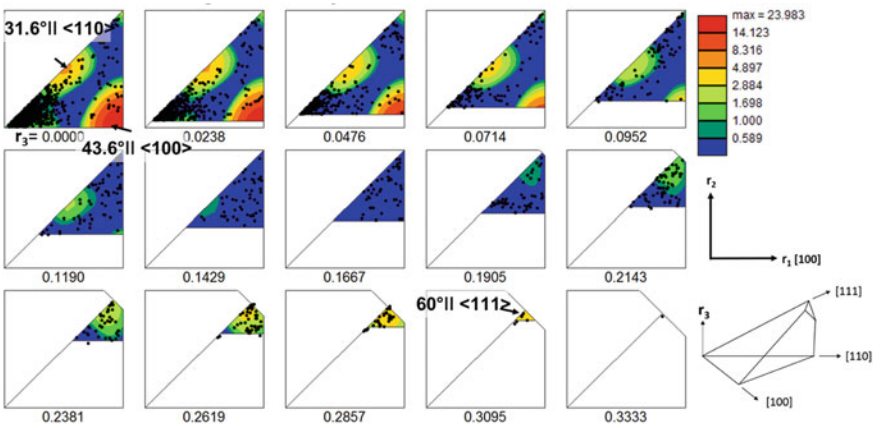
wt pct	C	Mn	Si	Ni	Cr	Ti	Mo	Nb
X70	0.050	1.59	0.3	0.01	0.26	0.013	0.09	0.066
wt pct	V	Al	N	S	P	Cu	Ca	
X70	0.005	0.026	0.0081	0.003	0.010	0.01	–	

grit using standard metallographic procedures, and vibro-polished using 0.02 μm colloidal silica suspension for several hours. Electron backscatter diffraction patterns were obtained using a JEOL®-7000 field emission scanning electron microscope (FE-SEM). All EBSD scans were obtained using an accelerating voltage of 20 kV. Step sizes were varied depending on the size of the scan. All data analysis was performed using OIM Data Analysis and no clean-up procedures were applied to the data sets.

Results and Discussion

Questions about the efficacy of using EBSD to reliably characterize cementite distribution in low carbon steels arose from work in which the effect of microstructure on hydrogen induced cracking in pipeline steels was assessed [13]. In particular, the work focused on characterizing intergranular cracks by comparing misorientation distributions of the uncracked microstructure to misorientations across cracks [13]. Using a misorientation distribution function (MDF) in Rodriguez-Frank (RF) space allowed for the identification of grain boundaries that occur with a higher frequency than others, as shown in Fig. 2. The MDF of the uncracked microstructure is represented by a color gradient map where red represents boundaries that occur at ~24 multiples of a random distribution (MRD) and blue represents boundaries present in amounts equivalent to a random distribution. Individual cracked grain boundaries measured by manually assessing misorientations across the crack are overlayed and represented by black dots. The boundary that appears with the highest MRD in the uncracked microstructure is the 43.6° || <100> axis/angle pair. This axis/angle pair is of particular interest due to the lack of cracked boundaries (black dots in Fig. 2) measured upon hydrogen introduction, which could indicate that there are particular boundaries that are less susceptible to cracking than others in the low carbon steel of interest.

However, when attempting to characterize the spatial distribution of these 43.6° || <100> boundaries, it was discovered that these boundaries appeared as clusters within particular ferrite grains, indicating that they are not grain boundaries. Figure 3a is an image quality (IQ) map from a scan obtained using a 50 nm step size in the ND-TD plane, with a region rich in 43.6° || <100> boundaries outlined in white. Figure 3b includes a PRIAS™ top micrograph with cementite and austenite phases with >0.2 confidence index (CI) overlaid in green and red, respectively. PRIAS™ is an imaging modality available in EDAX® EBSD systems that uses the variation



(a)

Fig. 2 MDF in RF space for the steel with discrete misorientation angle/axis pairs from cracked boundaries overlaid as black dots. Locations of three special boundaries with relatively high multiples of a random distribution are also overlaid for reference [13]

in the intensity of the diffracting electrons on the phosphor screen to create micrographs with varying diffraction contrast modes [15]. When comparing the EBSD micrographs in Fig. 3a and b, it is clear that the same regions that are rich in 43.6° || <100> boundaries are also rich in regions classified as cementite by the EBSD phase identification. It is also interesting to note that the regions rich in 43.6° || <100> boundaries in Fig. 3a are directly adjacent to grains that do not have any 43.6° || <100> boundaries present, indicating that the pixels identified as cementite are likely not universal errors in phase identification, but rather either correct indexing of cementite that precipitated in specific grains or a pseudosymmetry related error. The term pseudosymmetry is often used when discussing multiple orientation solutions possible for a given experimental Kikuchi pattern, which in practice often results in "speckling", or systematic color variation within a single grain.

In order to attempt to verify the accuracy of the EBSD software phase identification, fiducial marks were created near the region of interest using a focused ion beam, and the samples were etched with 2 pct nital. A secondary electron (SE) micrograph was then obtained, which is the bottom micrograph in Fig. 3b. Several raised and light-colored constituents are apparent on the surface due to the nital etchant. It is assumed that nital preferentially attacks the ferrite grains and leaves carbon-rich constituents like austenite and cementite in relief. Several observations come about as a result of comparing the light-colored constituents in the SE micrograph to the phase map produced by EBSD. Constituents identified as austenite in the EBSD phase map correlate well with constituents visible in SE upon nital etching and are identified by black arrows. In contrast, isolated pixels identified as cementite occur along boundaries and sporadically within the ferrite grains and do not correlate well to individual light-colored constituents in the SE micrograph. Several constituents

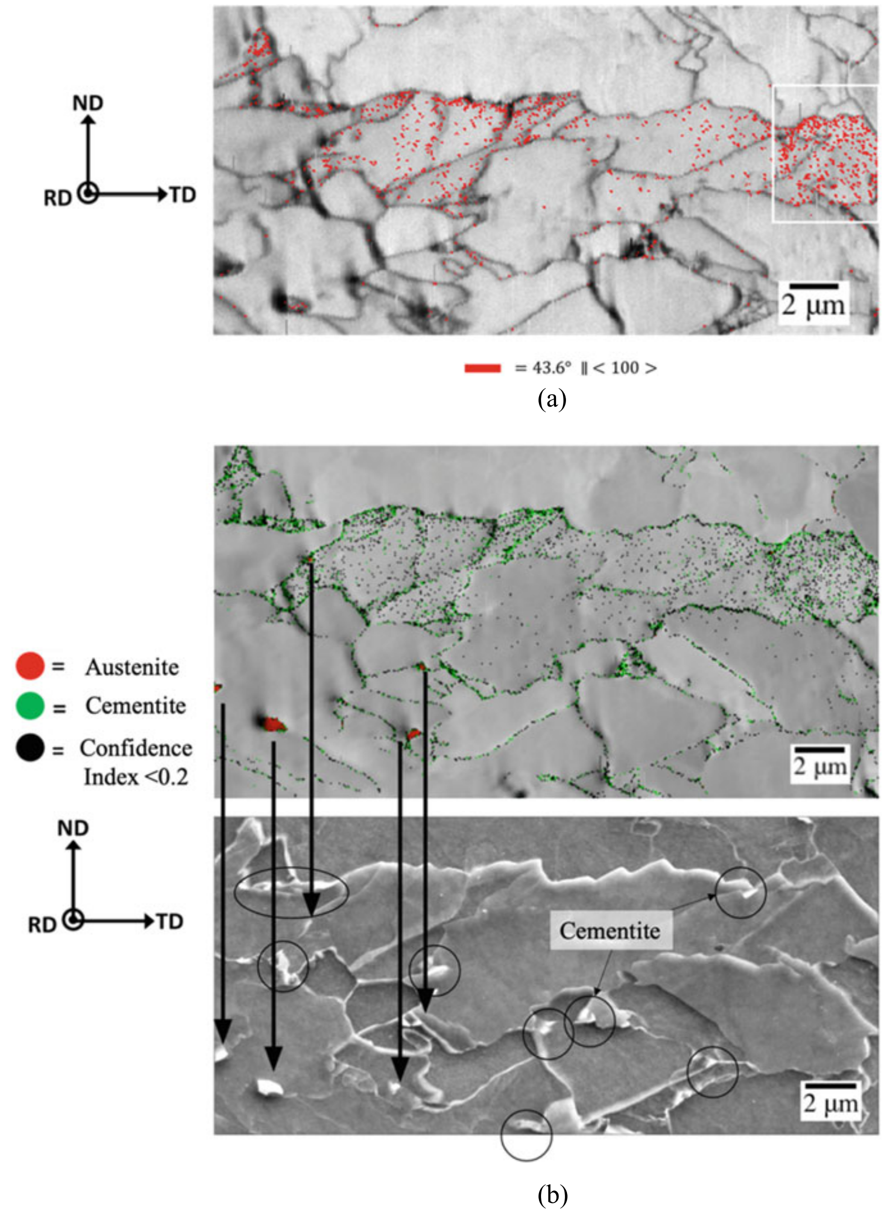
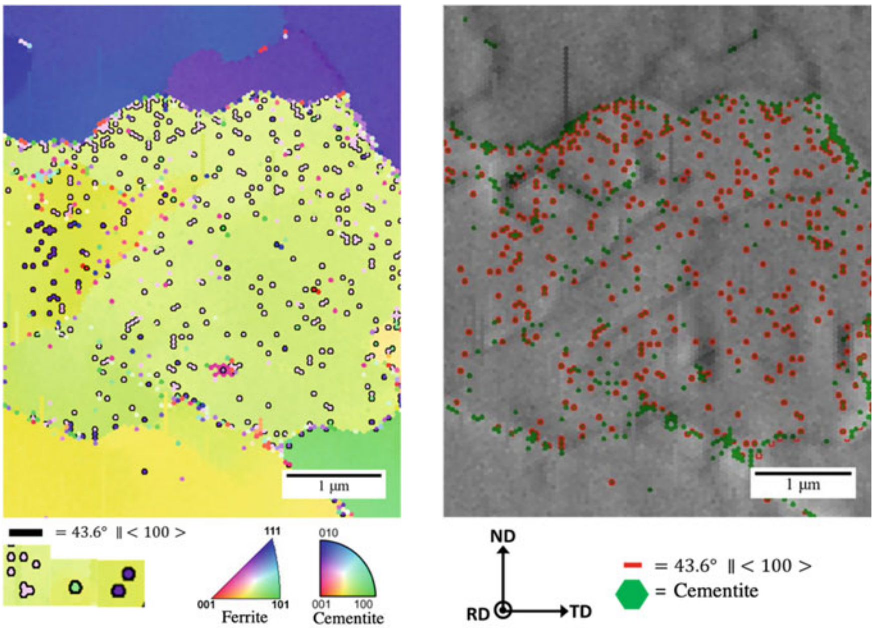


Fig. 3 **a** EBSD image quality map with red regions representing 43.6° ∥ <100> boundaries and subsequent region of interest outlined in white and **b** EBSD PRIAS™ top micrograph (top) with austenite and cementite phases indicated by red and green, respectively, and (bottom) secondary electron micrograph of 2 pct nital etched steel, with arrows pointing to austenite regions and circles indicating possible cementite precipitates (Color figure online)

that might be classified as cementite using SE micrographs of the nital etched steel are indicated by black circles in Fig. 3b, but cannot be as convincingly tied to regions identified as cementite in the EBSD maps. Therefore, a separate, diffraction-based verification modality was undertaken.

Figure 4a is an inverse pole figure (IPF) map of the white outlined region in Fig. 3a with boundaries within 5° of a 43.6° || <100> boundary indicated in black. Figure 4b is a PRIAS™ top micrograph of the same region in Fig. 4a with pixels indexed as cementite shown in green and 43.6° || <100> boundaries shown in red. Data points indexed as cementite appear largely as isolated individual pixels in Fig. 4b and often have red boundaries indicating a 43.6° || <100> boundary surrounding the pixel. The three different colors (e.g. orientations) of cementite are shown below the IPF micrograph in Fig. 4a. The fact that cementite surrounded by the special boundaries only has 3 different colors in the IPF map may be indicative of 3 separate variants. This observation might then in turn indicate that the cementite data points have an orientation relationship with the ferrite, which is described by the 43.6° || <100> axis/angle pair. To investigate if the axis/angle pair correlates to one of the three most common ferrite/cementite orientation relationships discussed in the background section, pole figures were created from several adjacent data points with a common 43.6° || <100> boundary.



(a) (b)

Fig. 4 EBSD maps from a region selected from Fig. 3a where **a** is an IPF map with 43.6° || <100> boundaries indicated as black outlines and **b** is a PRIAS top micrograph with data points indexed as cementite in green and 43.6° || <100> boundaries indicated in red. Each pixel represents data indexed by the EBSD software (Color figure online)