Characterization of Minerals, Metals, and Materials 2021

EDITED BY
Jian Li
Mingming Zhang
Bowen Li
Sergio Neves Monteiro
Shadia Ikhmayies
Yunus Eren Kalay
Jiann-Yang Hwang
Juan P. Escobedo-Diaz
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Alex Moser

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The Minerals, Metals & Materials Series

Jian Li · Mingming Zhang · Bowen Li ·
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Editors

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Editors
Jian Li
CanmetMATERIALS
Hamilton, ON, Canada

Bowen Li
Michigan Technological University
Houghton, MI, USA

Shadia Ikhmayies
Amman, Jordan

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

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

Rajiv Soman
Eurofins EAG Materials Science, LLC
Liverpool, NY, USA

Mingming Zhang
ArcelorMittal
Schererville, IN, USA

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

Yunus Eren Kalay
Middle East Technical University
Ankara, Turkey

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

Andrew D. Brown
United States Army Research Laboratory
Abingdon, MD, USA

Alex Moser
United States Naval Research Laboratory
Washington, DC, USA

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Preface

Characterization of properties and microstructure are important elements in determining critical processing parameters. Recent advances in characterization instruments have contributed, in a significant way, to an in-depth understanding of materials properties and structure that enabled much precise process control.

The Characterization of Minerals, Metals, and Materials Symposium is sponsored by the Materials Characterization Committee under the Minerals, Metals & Materials Society (TMS). The main focus of this symposium includes, but is 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.

The Characterization of Minerals, Metals, and Materials Symposia are among one of the largest in the TMS Annual Meetings that attract researchers in the field of materials, minerals, mechanical engineering, chemistry, and physics. In the TMS 2021 Virtual Annual Meeting & Exhibition, this symposium received 168 abstract submissions, of which 78 submissions were accepted for oral presentation in 8 technical sessions, and 83 were accepted as poster presentations. This proceedings volume includes 62 peer-reviewed manuscripts from original research.

This proceedings publication is a valuable reference for academia and industry that includes advanced characterization methods and instrumentations that cover a wide range of research fields. Readers will enjoy the diversity of topics in this book with novel approaches to process and characterize materials at various length scales.

The editors of this proceedings volume are very grateful to the authors for their contribution and willingness to share their research findings. The editors would also like to thank TMS for providing this valuable opportunity to publish this stand-alone volume. Appreciations also extend to the Materials Characterization Committee and Extraction and Processing Division (EPD) for sponsoring the symposium. The editors also thank the publisher, Springer, for their timely publication of this book. Last but not least, the editors would like to thank the contribution and support from the members of the Materials Characterization Committee.

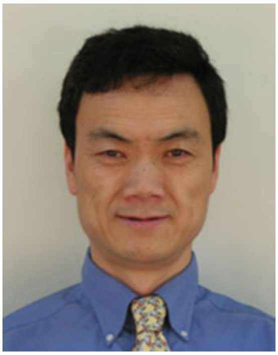
Jian Li
Lead Organizer

Contents

About the Editors

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 journals and conference proceedings.

Mingming Zhang is currently a lead research engineer at ArcelorMittal Global R&D in East Chicago, Indiana. Dr. Zhang has more than 15 years of research experience in the field of mineral processing and 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 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).



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 120 technical papers in peer-reviewed journals and conference proceedings, authored/co-authored 3 books, and edited/co-edited 8 books. He also holds 15 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 currently serves as the past 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 1,500 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 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 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 30 patents and a top world researcher in "Natural Fiber Composites" and "Ballistic Armor", Scopus 2029.

Shadia J. 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 56 research papers in international scientific journals, 80 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–ECRES2020). 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.

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* (http://matter.mete.metu.edu.tr/), 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.

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 at Los Alamos National Laboratory. His main research interests center on the dynamic behavior of materials under extreme conditions, in particular high pressure and high strain rate. His focus has been on investigating the effects of microstructural features on the dynamic fracture behavior of metals and metallic alloys. He has published primarily in the fields of shock physics and materials science. He has been a member of The Minerals, Metals & Materials Society (TMS) since 2011. During this time, he has 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.

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 55 journal publications, one book chapter, and 35 invited technical talks to his credit.

With regard to TMS service, Dr. Carpenter currently serves as the EPD representative on the Program Committee and the SMD representative on the Content

Development and Dissemination Committee. He is a participating member of the Mechanical Behavior of Materials Committee and has served as chair of both the Characterization Committee and the Advanced Characterization, Testing, and Simulation Committee in the past. John serves as a Key Reader for *Metallurgical and Materials Transactions A* and has co-edited 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 TMS EPD Young Leaders Professional Development Award. He also received the 2018 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.

Rajiv Soman currently serves as Director, Purity Survey Analysis, Materials Sciences Division at Eurofins EAG Laboratories, USA. He has over 30 years of professional experience in analytical chemistry and materials sciences. He earned a doctorate in Analytical Chemistry from Northeastern University, Boston. He received his B.Sc. (Chemistry—*Principal*; Physics—*Subsidiary*) with Honors, from Bombay University, India, and M.Sc. in Applied Chemistry from the Faculty of Technology & Engineering, Maharaja Sayajirao University of Baroda, India. He commenced his professional career as an Advanced Analytical Chemist in the Engineering Materials Technology Laboratories of General Electric Aircraft Engines. Prior to joining EAG Laboratories, Dr. Soman served as professor (Full) of Chemical Engineering, Chemistry, and Chemical Technology, and served as a faculty member for 20 years. He has received numerous awards for excellence in teaching and twice has been listed in *Who's Who Among America's Teachers.*

Dr. Soman's research interests are in the areas of atomic and mass spectrometry, with an emphasis on trace element determination and chemical speciation in a wide range of sample matrices. He was an invited guest scientist at the prestigious research institute, Forschungszentrum Jülich, Germany, where he conducted research in elemental mass spectrometry. He has co-authored several publications in international journals and has made numerous presentations at national and international conferences. He holds two U.S. patents.

Dr. Soman has been a member of the Society for Applied Spectroscopy (SAS) and the American Chemical Society (ACS) since 1986 and has served in numerous leadership positions in the societies. He was an invited panel member for the American Chemical Society's National Committee on Preparing for the Workforce 2020. He is also a member of ASM International, ASTM, and TMS, where he serves as a member of the Materials Characterization Committee.

Alex Moser of the U.S. Naval Research Laboratory has over 25 years of experience in materials development, shock-physics modeling, high speed videography, and armor development. He has over 40 publications, 5 patents, and patents pending, and has led research in carbon nanotube sensors.

Dr. Moser has investigated and developed blast and ballistic amour systems for the military resulting in multiple patents (10295310, 10281242, 9879946, 9696120, 9534870), patents pending, and a +$200M procurement for lightweight amour based on his design requirements. He has also contributed to projects concerning blast testing of vests, helmets, and goggles and a novel approach for quantifying the vulnerability of helmet designs.

He has augmented ballistic and blast experimental data with high strain rate simulations using CTH, a shock-physics code developed by Sandia National Laboratories. These endeavors resulted in a better understanding of the mechanisms of blast and ballistic phenomena related to amour materials and systems.

Part I
Advanced Microstructure Characterization

Insights into the Formation of Al–Cu Intermetallic Compounds During the Solid–Liquid Reaction by High-Resolution Transmission Electron Microscopy

Jie Chen, Yongqiong Ren, and Bingge Zhao

Abstract During Al/Cu solid–liquid reaction, different intermetallic compounds (IMCs) are expected, which can affect the mechanical and electrical properties of Al/Cu joints. To tackle this challenge, it is then necessary to tune the interface structure, which requires an insight into the formation mechanism of IMCs. In the current study, Al/Cu liquid–solid reaction was used to fabricate different IMCs. With the aid of a focused ion beam (FIB) and high-resolution transmission electron microscopy (HRTEM), the distribution of IMCs along the Al/Cu interface was determined. Meanwhile, the orientation relationship among different IMCs and Cu with coherent structure was identified. This study provides a fundamental understanding of the mechanism behind the Al/Cu reaction, which may guide the performance improvement of Al/Cu dissimilar weld.

Keywords Al/Cu interface · Solid–liquid reaction · Intermetallic compounds · HRTEM · Crystallographic orientation

Introduction

Materials with different physical and mechanical properties can be combined with dissimilar welding, and the advantages of each material can be fully utilized [1–3]. Al/Cu dissimilar welding has been widely used in power generation and transfer fields [4, 5]. This approach can produce a satisfying effect in lowering cost without much expense in transmission efficiency with respect to pure Cu. However, the physical, chemical, and mechanical properties of Al and Cu are significantly different [6,

J. Chen · Y. Ren
Yonggu Co. Ltd., 328 Wei Ershi Road, Yueqing City, Zhejiang Province 325600, People's Republic of China

B. Zhao (✉)
School of Materials Science and Engineering, Shanghai University, 99 Shangda Road, Shanghai 200444, People's Republic of China
e-mail: binggezhao@shu.edu.cn

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7], which can cause defects during dissimilar welding. Among these issues, intermetallic compounds (IMCs) are essential in influencing the property of Al/Cu joints. According to the Al–Cu binary phase diagram [8], a large number of IMCs are expected in this system. Up to now, many studies have paid attention to this area. In work by Garg, Al_2Cu, and Al_4Cu_9 are the dominant IMCs during the friction stir spot welding of AA6061-T6 and Cu [9]. Nevertheless, Al_2Cu, AlCu, and Al_3Cu_4 were the main phases in Pierpaolo's research, where AA2024-T3 and Cu were joined by friction stir welding [10]. Hwang, on the other hand, utilized cold rolling to join Al and Cu, where Al_2Cu, AlCu, Al_3Cu_4, and Al_4Cu_9 were produced after annealing [11]. In a recent work by Wang, a high-temperature phase, $Cu_{15}Al$, was deemed to form during Al/Cu solid–liquid reaction [12]. These studies suggest that the formation of IMCs is rather complex. To tune the interface and optimize the property of Al/Cu joints, understanding the structural evolution during Al/Cu reaction becomes the impending issue.

In this study, Al/Cu interface structure with different IMCs was prepared by a solid–liquid reaction. To reveal the structural evolution during this process, a focused ion beam (FIB) was used to machine the interface, which was then followed by the structure characterization with high-resolution transmission electron microscopy (HRTEM) . With these efforts, the distribution of IMCs along the Al/Cu interface was determined, and the crystallographic orientation among Cu and IMCs was revealed.

Materials and Methods

Al and Cu, with the purity of 99.99%, were used in Al/Cu solid–liquid reaction. Pure Al ingot was loaded in an Al_2O_3 crucible and then melted in a muffle furnace (SXL-1200C, Shanghai Jujing) by heating it to 780 °C and holding for 5 min. A copper rod with a diameter of 6 mm was ground by sandpaper and then washed by 4 vol.% HCl solution to remove the contaminant and oxide. After that the Cu rod was treated by KF aqueous solution with a concentration of 4 wt% for 60 s to improve the wettability of Al on Cu. After drying in air, Cu was dipped into Al melt for 60 s, which was quenched by water to prepare Al/Cu solid–liquid reaction specimen.

FIB (FEI Helios Nano 600i) was then used to machine the Al/Cu interface. The interface structure was then characterized by HRTEM (JEOL-2010F) equipped with an energy dispersive spectrometer (EDS, Oxford).

Results and Discussion

As revealed in our previous study, IMCs can form in the area close to Cu during the Al/Cu solid–liquid reaction. Figure 1 displays the morphology of the Al/Cu interface. Two different IMCs are expected to form based on the image contrasts. To demonstrate this hypothesis, the interface containing different phases was machined by FIB, as schematically indicated by the yellow frame.

Figure 2 is the bright-field (BF) TEM image of the specimen. Three different areas are obviously identified. The contrast of BF image is related to the structure orientation. Accordingly, the orientation relationship between these three areas should be different.

It was reported that diffusion plays the dominant role in the Al/Cu solid–liquid reaction, suggesting the composition discrepancy in Al/Cu interface. As a result, a high-angle annular dark-field (HAADF) image whose contrast is associated with atom number was employed to reveal this phenomenon. Figure 3 displays the HAADF image of the interface as well as the EDS line scanning results. In agreement with Fig. 2, three different layers are observed in Fig. 3a, suggesting the composition

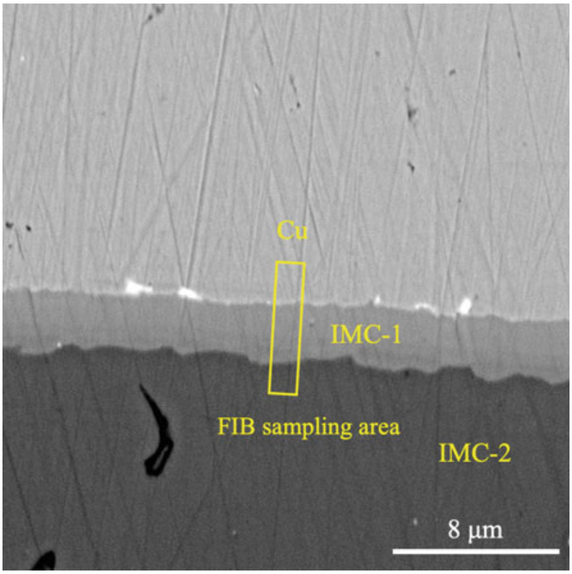

Fig. 1 Scanning electron microscopy (SEM) image of the Al/Cu interface. (Color figure online)

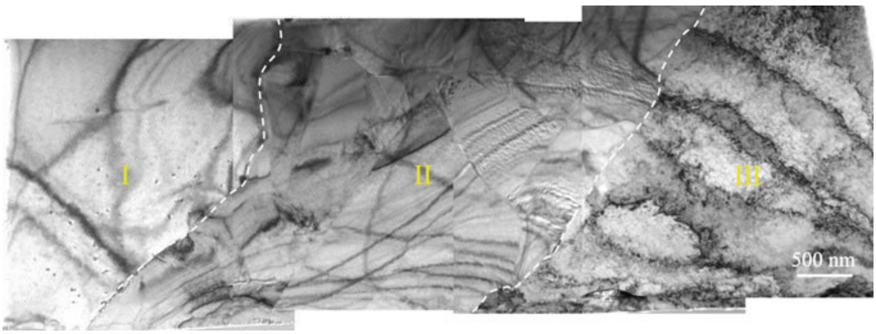

Fig. 2 Bright-field image of the Al/Cu interface. (Color figure online)

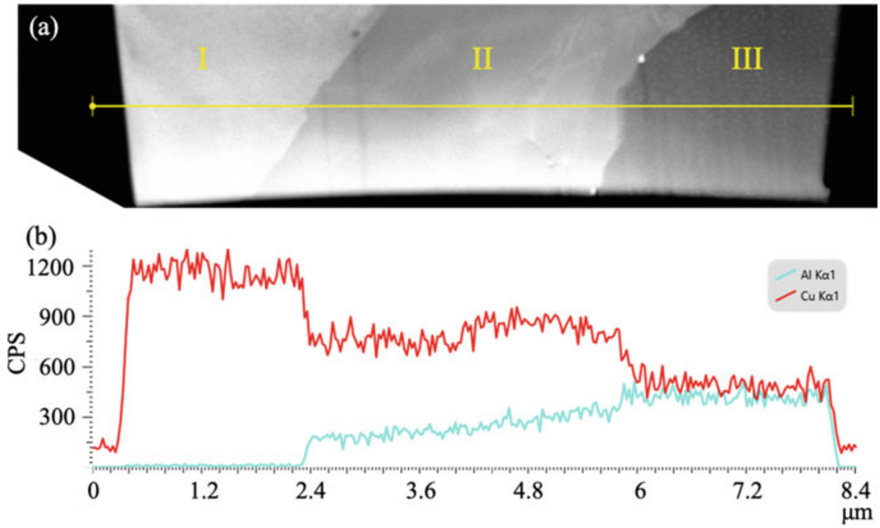

Fig. 3 HAADF image and EDS line scanning results of the specimen. (Color figure online)

difference in the specimen. As suggested by the EDS result in Fig. 3b, the area I is the Cu matrix with minor Al. During Al/Cu solid–liquid reaction, intensive diffusion of Al into Cu can occur. According to Al–Cu binary phase diagram [8], the maximum solubility of Al in Cu can reach 19.7%. That means the Cu matrix can be replaced by Cu solid solution with Al. With the distance away from Cu, the concentration of Cu decreases while it increases for Al. Meanwhile, different IMCs can form with the interdiffusion of Cu and Al.

As mentioned above, the formation of IMCs in Al/Cu system is significantly varied. Although current research uncovers the composition discrepancy in Al/Cu interface by HAADF and EDS, structure characterization is still essential to demonstrate the phase evolution. Figure 4 is the selected area electron diffraction (SAED) pattern (Fig. 4a) and HRTEM image with Fast Fourier Transformation (FFT) pattern (Fig. 4b) of area I. With the help of the SAED pattern, the area I is identified as Cu (PDF card number: 65-9743) without any IMC. Additionally, the interplanar spacing is nearly the same as that of pure Cu. Although the solid solution of Al in Cu is deemed to occur in this area, its determination is hindered by the error of SAED, which can be figured out by X-ray diffraction in our on-going work.

The structure characterization result of area II is displayed in Fig. 5. In contrast to Fig. 4a, two sets of diffraction spots with different brightness are observed: there are two weak diffraction spots between the bright ones. This phenomenon can still be detected with another incident axis (Fig. 5b). This seems like the occurrence of the superlattice, which is, however, seldom reported in the Al–Cu system. In our previous work, the kinetics of AlCu formation cannot be depicted by a single diffusion mechanism. Together with the current result, different IMCs may form in area II rather than the formation of the superlattice. By indexing the SAED patterns

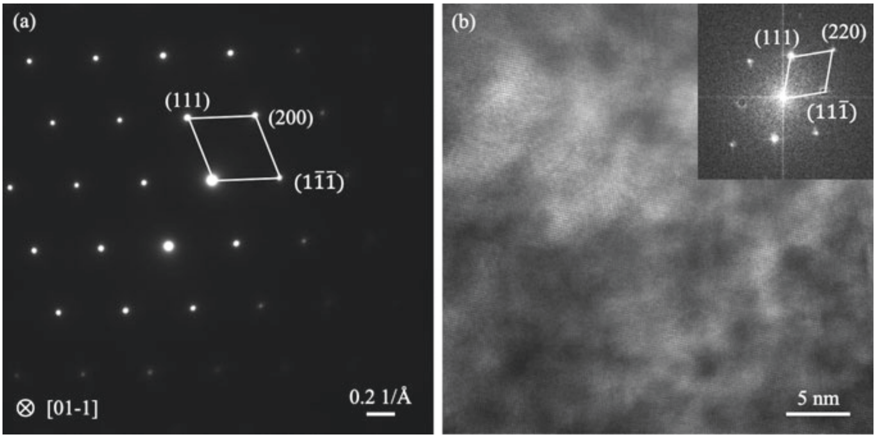

Fig. 4 Structure characterization of area I. **a** SAED pattern of area I. **b** HRTEM image with FFT pattern

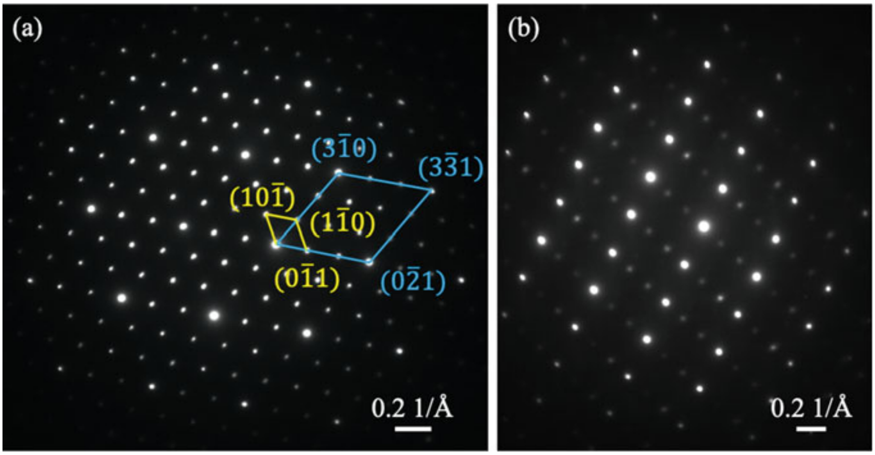

Fig. 5 SAED patterns of area II. Two sets of diffraction spots are detected. (Color figure online)

in Fig. 5a, the bright spots denote the AlCu phase with orthogonal structure (PDF card number: 39-1371) while the weak spots come from Al_4Cu_9 with primitive cubic structure (PDF card number: 65-7542). Moreover, these two IMCs are completely coherent in the microstructure. Although the Al_4Cu_9 IMC layer has been observed in Al/Cu solid reaction [13, 14], its orientation relationship with AlCu is rarely reported in solid–liquid reaction.

To further evidence this result, HRTEM image of area II is presented in Fig. 6. Based on the HRTEM image, FFT and inverse FFT patterns are obtained. By estimating the interplanar spacing, this area is identified as Al_4Cu_9.